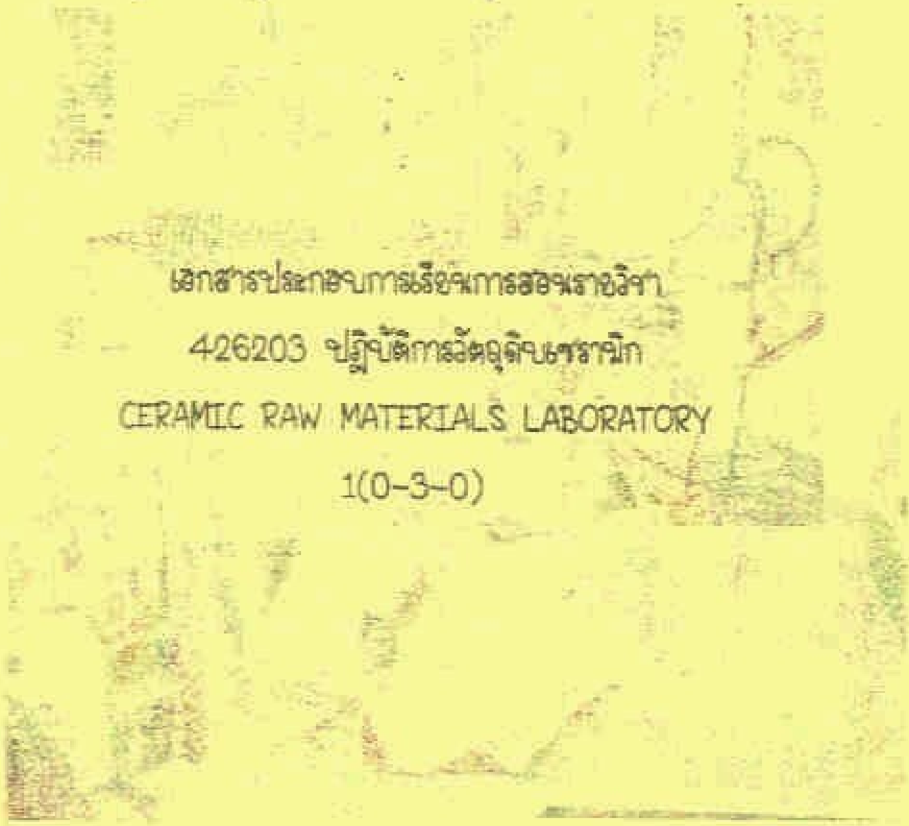
Sirirat

คู่มือปฏิบัติการวัตถุดิบเซรามิก



เอกสารประกอบการเรียนการสอนรายวิชา
426203 ปฏิบัติการวัตถุดิบเซรามิก
CERAMIC RAW MATERIALS LABORATORY
1(0-3-0)

สาขาวิชาวิศวกรรมเซรามิก สำนักวิชาวิศวกรรมศาสตร์
มหาวิทยาลัยเทคโนโลยีสุรนารี
ปรับปรุงเมื่อ กันยายน 2550

คำนำ

คณาจารย์ประจำสาขาวิศวกรรมเซรามิก สำนักวิชาวิศวกรรมศาสตร์ มหาวิทยาลัยเทคโนโลยีสุรนารี ได้จัดทำคู่มือปฏิบัติการเล่มนี้ขึ้นเพื่อเป็นการรวบรวมเนื้อหาและใช้ประกอบการเรียนการสอนในรายวิชา **Ceramic Raw Materials Laboratory** เพื่อให้นักศึกษาได้ทำความเข้าใจวิธีการทดลองก่อนเข้าทำปฏิบัติการและใช้ประกอบในการเขียนรายงาน คู่มือเล่มนี้ประกอบด้วย **2** ส่วนคือ ส่วนที่ **1** เป็นการทดลองในรายวิชาจำนวน **8** การทดลอง สำหรับเตรียมวัตถุดิบ การตรวจสอบสมบัติทางฟิสิกส์และเคมีของวัตถุดิบต่างๆ เพื่อให้นักศึกษาได้คุ้นเคยกับวัตถุดิบที่เป็นดิน หินจากธรรมชาติ และวัตถุสังเคราะห์ ส่วนที่ **2** เป็นการแนะนำวิธีการเขียนรายงานการทดลองในห้องปฏิบัติการให้ถูกต้อง

หนังสือเล่มนี้ได้มีการปรับปรุงเนื้อหาหลายครั้งเพื่อให้เนื้อหาสมบูรณ์และถูกต้องยิ่งขึ้น แต่ยังมิได้จัดทำเพื่อจำหน่ายแก่บุคคลภายนอก จึงขอสงวนลิขสิทธิ์ทุกประการ

คณะผู้จัดทำ
ผศ.ดร.ศิริรัตน์ ทับสูงเนิน รัตนจันทร์ (ผู้ประสานรายวิชา)
อาจารย์ ดร.วีระยุทธ์ ลอประยูร
รศ.ดร. จรัสศรี ลอประยูร
ผศ.ดร.สุธรรม ศรีหล่มสัก
ผศ.ดร.สุขเกษม กังวานตระกูล

สารบัญ

นื้อหา หน้า

าคผนวก

ง. มาตรฐานการทดสอบ ASTM

426203 ปฏิบัติการวัตถุดิบเซรามิก 1(0-3-0)
Ceramic Raw Materials Laboratory
สาขาวิชาวิศวกรรมเซรามิก สำนักวิศวกรรมศาสตร์
มหาวิทยาลัยเทคโนโลยีสุรนารี

วิชาบังคับก่อนหรือเรียนร่วม : 426 202, 506202 วัตถุดิบเซรามิก หรือเรียนควบคู่

เนื้อหา: วิเคราะห์และทดสอบคุณสมบัติทางกายภาพและทางเคมีของวัตถุดิบชนิดต่างๆ

จุดประสงค์: เพื่อให้นักศึกษาเข้าใจกระบวนการเตรียมวัตถุดิบเซรามิก สามารถตรวจวิเคราะห์ ทดสอบสมบัติทางกายภาพและทางเคมี และเขียนรายงานการทดลองอย่างเป็นระเบียบ

เนื้อหารายวิชา

การทดลองที่	ชื่อปฏิบัติการ
1	Crushing, Grinding & Dry Sieve Analysis
2	Clay preparation & Wet Sieve Analysis
3	Density Measurement
4	Methylene Blue Absorption
5	Feldspar Test
6	Green Strength Test
7	Physical Properties of Fired Clay Products
8	Refractory Raw Materials

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วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 1
CRUSHING, GRINDING & DRY SIEVE ANALYSIS

อาจารย์ ดร. วีระยุทธ์ ลอประยูร

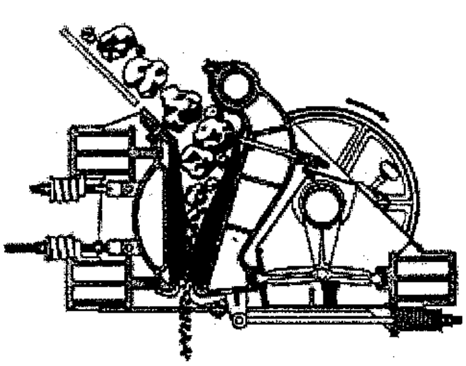
บทนำ

Crushing and Grinding

วัตถุดิบเซรามิกบางชนิดมีขนาดอนุภาค (Particle size) ที่ใหญ่เกินกว่าที่จะนำไปใช้ในอุตสาหกรรม จึงจำเป็นต้องถูกนำไปบดย่อย (Crushing & Grinding) ก่อนด้วยเครื่องมือที่ใช้ในการบดย่อยแบ่งออกได้เป็น 3 ชนิดคือ

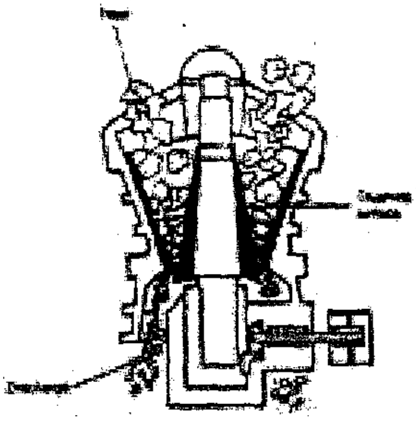
1. Primary Crusher ใช้บดวัตถุดิบที่มีขนาด 3 ฟุตขึ้นไป ให้เหลือขนาด 2-3 นิ้ว แบ่งเป็น 2 ชนิดใหญ่ๆ คือ

1.1 Jaw Crusher บดโดยอาศัยการขบกันของแผ่นบดตามแนวนอน (Horizontal squeezing or chewing motion) ดังรูป 1



รูป 1 The PCC **Pennsylvania Jaw** crushes by compression without rubbing. Hinged overhead and on the centerline of the crushing zone, the swinging jaw meets the material firmly and squarely. **There is no rubbing action** to reduce capacity, to generate fines or to cause excessive wear of jaw plates.

1.2 Gyratory crusher บดโดยอาศัยแรงเหวี่ยง eccentric rotation motion ดังรูปที่ 2



Gyratory Crusher

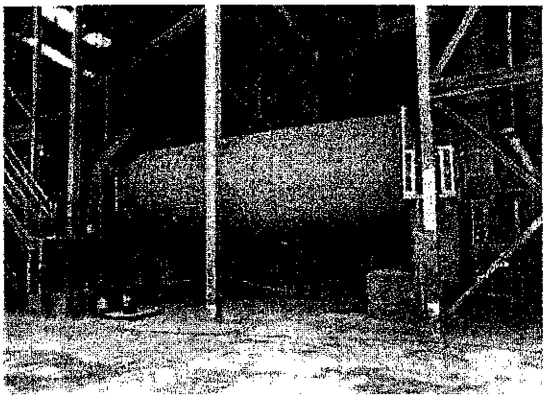
รูป 2 Gyratory crusher is similar in principle to a jaw crusher, but a stationary inverted bowl and eccentrically gyrating, inner cone are used to apply the “chewing” pressure to the rock

2. Secondary Crusher ใช้บดอนุภาคที่มีขนาด 2-3 นิ้วให้เหลือ 0.1 นิ้ว เครื่องบดชนิดนี้ที่เหมาะกับดินได้แก่ Dry-pan crusher ดังรูปที่ 3 โดยอาศัยการหมุนของมูเล่ย์ (Muller) กดทับบน rotation pan ส่วน secondary crusher สำหรับวัตถุดิบที่แข็งได้แก่ Hammer mill และ Impact mill

รูป 3 A dry-pan crusher

3. Grainder ได้แก่เครื่องบดที่ใช้ในการบดอนุภาคเล็กประมาณ 0.1 นิ้วให้มีขนาดละเอียดที่เหมาะสมในการใช้ในส่วนต่างๆของผลิตภัณฑ์ มักใช้ในส่วนการเตรียมวัตถุดิบของโรงงานอุตสาหกรรม เช่น ใช้ในการบดผสมน้ำดิน สำหรับทำน้ำดินหรือเพื่อ spray dryer ตัวอย่างเครื่องบดชนิดนี้ที่ใช้กันมากคือ tumbling mill ซึ่งมีลักษณะเป็นรูปทรงกระบอกวางตัวแนวนอน ภายในบรรจุลูกบดซึ่งทำจาก Heavy steel หรือ dense ceramic ทำหน้าที่เป็นตัวบด เมื่อ tumbling mill หมุน ลูกบดจะทำหน้าที่บดอนุภาคให้ละเอียดยิ่งขึ้น

Tumbling mill แบ่งออกเป็น 2 ชนิดตามรูปทรงของลูกบดที่ใช้ ถ้าลูกบดที่ใช้มีลักษณะทรงกลมเรียก tumbling mill นั้นว่า ball mill ถ้าลูกบดที่รูปทรงเป็นทรงกระบอกเรียกว่า Rod mill รูปที่ 4 แสดง Tumbling mill ขนาดใหญ่ที่ใช้ในอุตสาหกรรม



รูป 4 Large Ball Mill
(Courtesy Machine & Equipment Company, Inc)

SIEVE METHOD

ขนาดของอนุภาคของวัตถุดิบเซรามิกมีส่วนสำคัญในการควบคุมคุณสมบัติของผลิตภัณฑ์ก่อนเผา และคุณสมบัติของผลิตภัณฑ์หลังเผา ดังนั้นการควบคุมขนาดอนุภาคของวัตถุดิบจึงเท่ากับเป็นการควบคุมการผลิตให้เป็นไปโดยสะดวก มีการเสียหายระหว่างการผลิตน้อย

การควบคุมขนาดอนุภาคเริ่มต้นจากการเรียนรู้การวัดขนาดของอนุภาค

ตารางที่ 1 แสดงวิธีการวัดขนาดอนุภาคด้วยวิธีการต่างๆ ซึ่งจะเลือกใช้ให้เหมาะสมกับขนาดอนุภาค วิธีการวัดขนาดอนุภาคที่ใช้กันในอุตสาหกรรมเซรามิกได้แก่

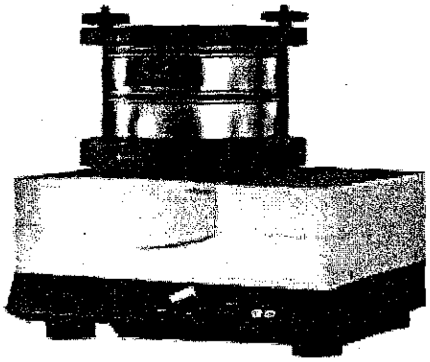
1. เครื่องมือสมัยใหม่ เช่น Light scattering method วิธีนี้จะใช้แสงเลเซอร์ยิงเข้าไปนับจำนวนอนุภาค สามารถใช้ได้กับอนุภาคที่มีขนาด 2000-0.1 ไมครอน เป็นวิธีที่สะดวกทำได้รวดเร็ว ค่าที่ได้บอกเป็นจำนวนอนุภาคที่มีขนาดต่างๆ (number base) ไม่สามารถบอกเป็นสัดส่วนโดยน้ำหนัก (Weight base) ได้
2. กล้องจุลทรรศน์ (Microscopic method) ใช้วัดอนุภาคที่มีขนาด 100-1 ไมครอน ความสามารถในการวัดขึ้นอยู่กับกำลังขยายและชนิดของกล้อง
3. การวัดพื้นที่ผิว (Specific surface area method) ใช้วัดขนาดของอนุภาคที่มีขนาดเล็กมากๆ ซึ่งอยู่ในช่วงคอลลอยด์ ระหว่าง 0.2-0.001 ไมครอน ตัวอย่างวิธีการนี้ได้แก่ Methylene blue adsorption method ในการทดลองที่ 4 ค่าที่วัดได้โดยวิธีนี้จะบอกเป็นพื้นที่ผิวจำเพาะ ไม่ใช่ขนาดของอนุภาค

4. การตกตะกอน (Sedimentation or hydrometer method) วิธีการนี้ใช้กันมากทางธรณีวิทยา ในการวัดขนาดอนุภาคที่มีขนาด 100-0.2 ไมครอน ซึ่งเป็นขนาดที่ตะแกรงร่อนไม่สามารถกระทำได้ วิธีนี้ใช้หลักการการตกตะกอนของดินได้แก่ Andersen pipet และ hydrometer method
5. ตะแกรงคัดขนาด (Sieve method) เป็นวิธีการที่สะดวกที่สุด ลงทุนไม่มาก เหมาะสำหรับวัดขนาดอนุภาคที่มีขนาดตั้งแต่ 45 ไมครอนขึ้นไป ซึ่งสามารถแบ่งออกได้เป็น 2 วิธีคือ
    a. ร่อนแบบแห้ง (Dry sieve) เหมาะสำหรับวัดอนุภาคที่มีขนาดใหญ่สะอาด มีเม็ดละเอียดปนอยู่น้อยเป็นพวก non-plastic raw materials เช่นหินฟันม้า และทราย
    b. ร่อนแบบเปียก (Wet sieve) เหมาะสำหรับอนุภาคที่มีขนาดเล็กมาก ส่วนใหญ่เป็น plastic raw materials เช่น ดินขาว ดินเหนียว

<u>เครื่องมือที่สำคัญในวิธี sieve method หรือ sceen</u>

<u>ตะแกรง (Screen)</u>

ขนาดของตะแกรงบอกเป็น mesh number ซึ่งเป็นจำนวนช่องเปิดในระยะ 1 นิ้วของตะแกรง ดังนั้นตะแกรงที่มีความถี่มากค่า mesh number จะสูง ตารางที่ 2 แสดงค่าขนาดของช่องเปิดของตะแกรงที่มี mesh number ต่างๆ ถ้าอนุภาคที่สามารถผ่านตะแกรงเบอร์ 8 ได้เรียกอนุภาคนั้นว่า –8 mesh หรือ minus 8 mesh หรือ through 8 mesh ถ้าอนุภาคผ่านตะแกรงเบอร์ 8 ได้แต่ไม่สามารถผ่านตะแกรงเบอร์ 10 เราเรียกว่า –8+10 mesh หรือ 8 through on 10 mesh หรือ 8/10 mesh ตะแกรงจะสามารถวัดอนุภาคที่มีความละเอียดมากที่สุด 38 ไมครอน ปัญหาของการทดสอบแบบตะแกรงคือ ถ้าใช้ตัวอย่างปริมาณมากเกินไป หรือตัวอย่างไม่แห้งดีพอ ตัวอย่างจะอุดตันรูของตะแกรงได้ ดังนั้นเครื่องตะแกรงร่อนที่ดี จึงมักมี mechanical shaking (รูปที่ 5)เครื่องบางเครื่องอาจจะมี frequency air หรือ suction เพื่อช่วยให้อนุภาคร่อนผ่านตะแกรงได้สะดวกดีขึ้น



รูปที่ 5 ตะแกรงร่อน และเครื่องเขย่า (Courtesy Pharmaceutical International)

ถ้าอนุภาคที่วัดมีลักษณะเป็นรูปยาวรี อาจจะทำให้การทดสอบโดยวิธีนี้มีข้อผิดพลาดได้มากกว่า อนุภาคที่มีลักษณะเป็นทรงกลม

Table 1

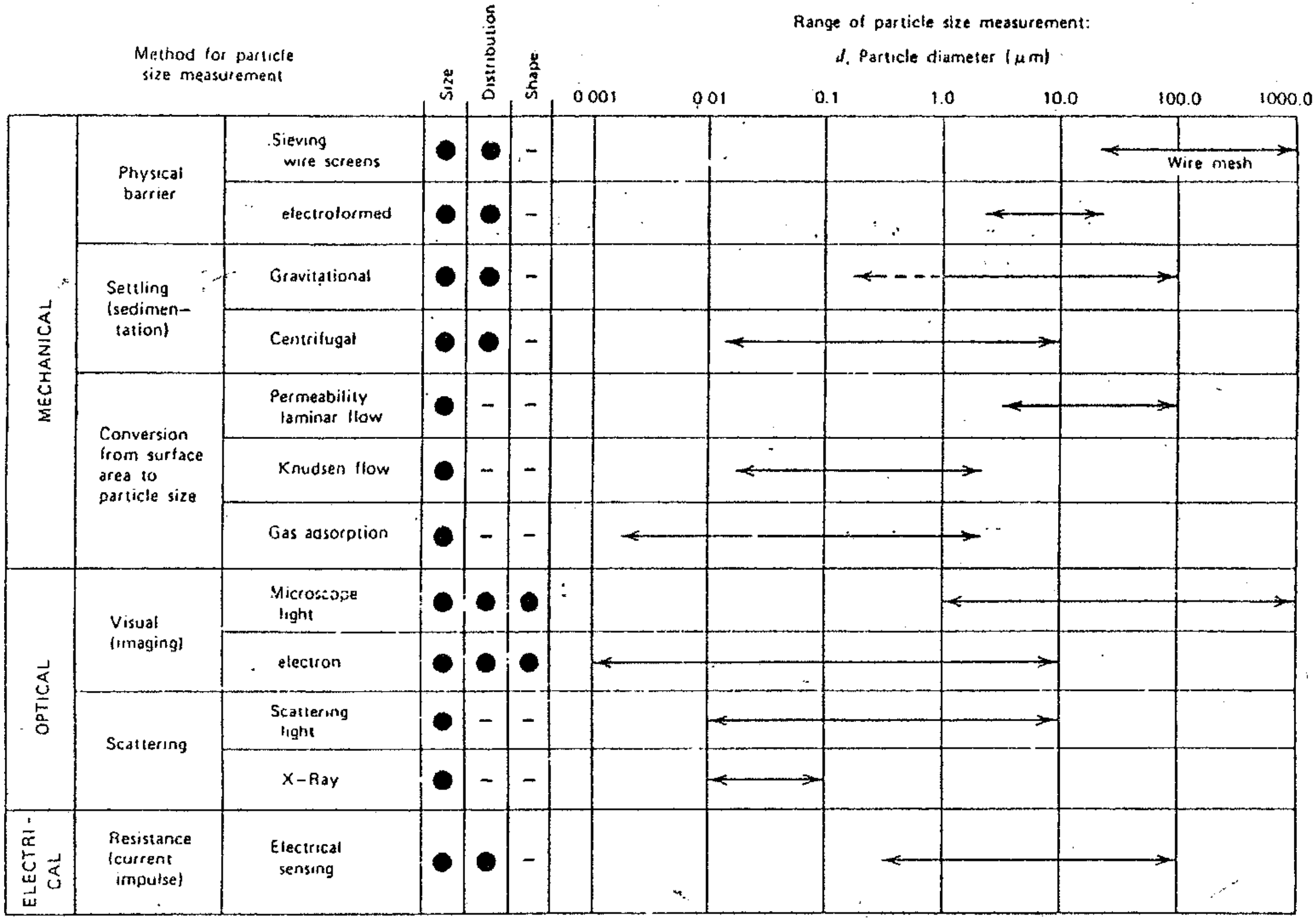


Fig. 1. Graph showing range of particle size measurement and general applicability for various common methods of particle size measurement.

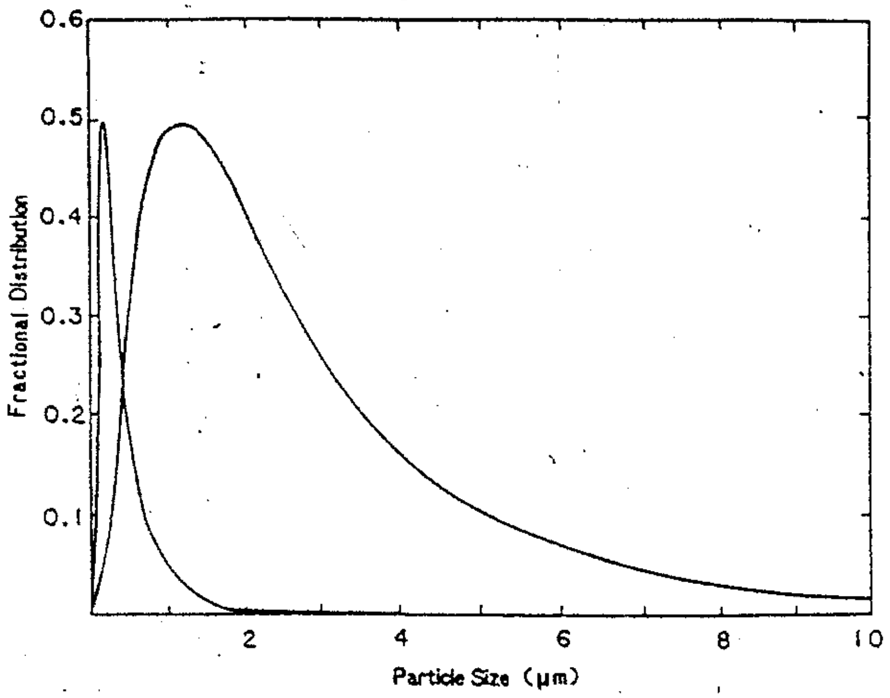
Table II

ขนาด หรือเบอร์	ขนาดช่องลอด (มม หรือไมครอน)	ขนาด หรือเบอร์	ขนาดช่องลอด (มม หรือไมครอน)
(4")	100.0 มม		
(3")	75.0	(3")	75.0 มม
(2$\frac{1}{2}$")	63.0	(2$\frac{1}{2}$")	63.0
(2")	50.0	(2")	50.0
(1$\frac{3}{4}$")	45.0		
(1$\frac{1}{2}$")	37.5	(1$\frac{1}{2}$")	37.5
(1$\frac{1}{4}$")	31.5		
(1")	25.0		28.0
			20.0
($\frac{3}{4}$")	19.0		
($\frac{5}{8}$")	16.0		
			14.0
($\frac{1}{2}$")	12.5		
			10.0
($\frac{3}{8}$")	9.5		
($\frac{5}{16}$")	8.0		
($\frac{1}{4}$")	6.3		6.3
			5.0
เบอร์ 4	4.75		
5	4.00		
		เบอร์ 5	3 353
เบอร์ 6	3.35		
7	2.80	เบอร์ 6	2.812
		7	2.411
8	2.36		
10	2.00	8	2.057
12	1.70	10	1.676
14	1.40	12	1.405
		14	1.204
16	1.18		
18	1.00	16	1.003
20	850 ไมครอน	18	853 ไมครอน
25	710	22	699
30	600	25	599
35	500	30	500
40	425	36	422
45	355	44	353
50	300	52	295
60	250	60	251
70	212	72	211
80	180	85	178
100	150	100	152
120	125	120	124
140	106	150	104
170	90	170	89
200	75	200	76
230	63	240	66
270	53	300	53
325	45		
400	38		

การรายงานผลการวัดขนาดของอนุภาคด้วยตะแกรง สามารถแสดงได้ 2 แบบคือ

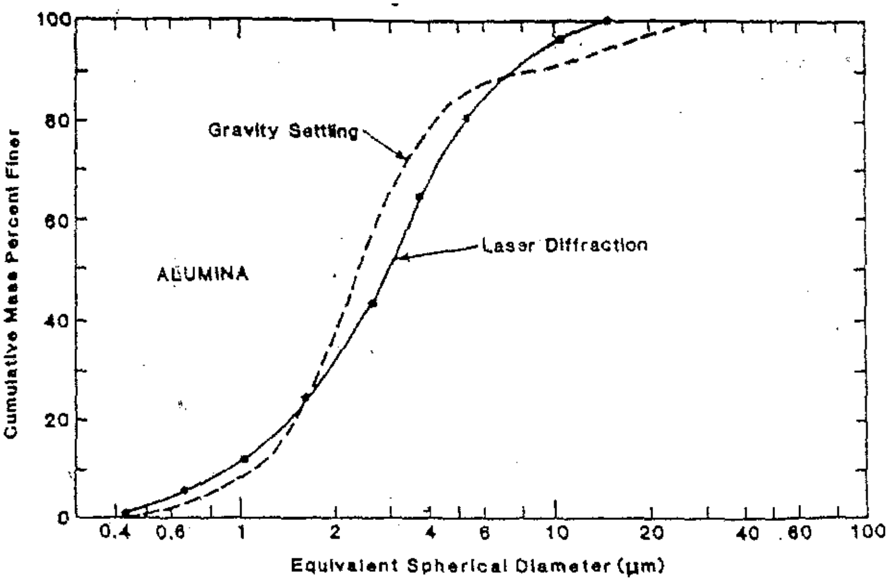
1. mass หรือ number fraction/size
2. cumulative massหรือ number % / size

กราฟ Mass หรือ number fraction/size เป็นการแสดงน้ำหนักหรือจำนวน อนุภาคที่มีขนาดใดขนาดหนึ่งต่อขนาดนั้น ดังตัวอย่างรูปที่ 6 กราฟ Cumulative % mass หรือ number/size เป็นการแสดงน้ำหนักหรือจำนวนอนุภาคที่มีขนาดเล็กกว่าหรือใหญ่กว่าขนาดใดขนาดหนึ่งต่อขนาดนั้น ดังตัวอย่างในรูปที่ 7 และ 8 สังเกตว่าแกนนอนจะแสดงเป็นแกน log ถ้าเขียนกราฟแบบมาตรฐานอังกฤษนิยมจัดสเกลขนาดตะแกรงใหญ่อยู่ทางด้านขวามือ (รูป 7) แต่มาตรฐานอเมริกาจัดสเกลใหญ่อยู่ทางด้านซ้ายมือ (รูป 8)



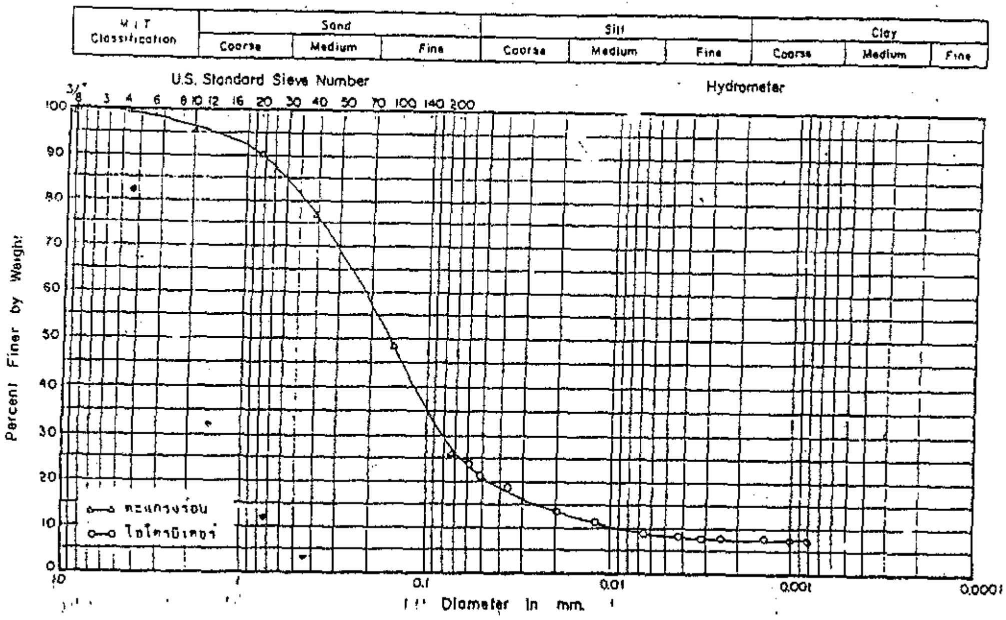
Fractional number and mass size distributions for a milled zircon powder

รูป 6 Fractional number and mass size distributions for a milled zircon powder



รูป 7 Comparative size data for a calcined alumina obtained by gravity settling and laser light diffraction (After H. Frock and P. Plantz, Processing Consequences of Raw Materials Variables, Alfred University, Alfred, NY., 1985)

Figure 8



รูป 8 กราฟแสดงการกระจายของเม็ดิน หลุมเจาะ BH-9-2 ตัวอย่างที่ UD-1 ความลึก 0.20-0.10 ม.

กราฟแสดงน้ำหนักหรือจำนวนอนุภาคที่มีขนาดเล็กกว่าขนาดใดขนาดหนึ่งเรียกว่า Cumulative mass (number) Persent finer หรือเรียกย่อว่า CMPF หรือ CNPF ส่วนกราฟที่แสดงน้ำหนักหรือจำนวนอนุภาคที่มีขนาดใหญ่กว่าขนาดใดขนาดหนึ่ง เรียกว่า Cumulative mass (number) Percent larger เรียกย่อๆว่า CMPL หรือ CNPL ตัวอย่างตารางการบันทึกข้อมูลและการหาค่า CMPF หรือ CMPLแสดงในตารางที่ 3

Presentation of Particle Size Data

Table III

Table 7.2 Particle Size Distribution of Ball Milled Petalite $LiAlSi_4O_{10}$

Sieve Size (μm)	Mass on Sieve (g)	Mass on Sieve (%)	CMPF (%)	CMPL (%)
1000	0	0.0	100.0	0.0
500	0.18	0.9	99.1	0.9
355	0.50	2.5	96.6	3.4
250	1.45	7.3	89.3	10.7
180	3.00	15.0	74.3	25.7
125	3.96	19.8	54.5	45.5
90	3.26	16.3	38.2	61.8
63	3.18	15.9	22.3	77.7
44	1.94	9.7	12.6	87.4
0	2.52	12.6	0.0	100.0
Total	20.00	100.0		

จุดประสงค์

เพื่อให้นักศึกษาเรียนรู้วิธีการและสามารถแปรสภาพวัตถุดิบทางเซรามิกที่เป็นก้อนใหญ่ให้มีขนาดเล็กลงโดยใช้เครื่องบดย่อยชนิดต่างๆ และตรวจขนาดและการกระจายขนาดของวัตถุดิบที่บดได้โดยใช้วิธีการร่อนผ่านตะแกรงที่มีขนาดรูเปิดตามกำหนดและเปรียบเทียบขนาดที่ได้จากการบดแต่ละเครื่อง

การทดลอง

อุปกรณ์การทดลอง

1. กล่องแบ่งตัวอย่างดิน 1 กล่อง
2. ถาดใส่ตัวอย่าง 1 ถาด
3. ตะแกรงร่อนตามเบอร์ที่อาจารย์ผู้สอนกำหนดให้สำหรับแต่ละกลุ่ม
4. แปรงลวดสำหรับทำความสะอาดตะแกรงหยาบ
5. แปรงขนอ่อนสำหรับทำความสะอาดตะแกรงละเอียด
6. ภาชนะตักดิน
7. เตาอบแห้งตั้งอุณหภูมิที่ 100°C จำนวน 2 เตา
8. เครื่องชั่งละเอียดความผิดพลาดไม่เกิน 0.05 กรัม จำนวน 2 เครื่อง
9. ค้อนยางสำหรับเคาะตะแกรงร่อนดิน
10. ผ้าเช็ดมือ
11. แผ่นพลาสติกและเทปกาวติด

<u>อุปกรณ์ที่ใช้ในการทำการทดลอง</u>

1. เครื่องบดย่อยขนาดหยาบ (Jaw crusher)
2. เครื่องบดย่อยขนาดละเอียด (Disc crusher)
3. ตะแกรง (Sieve) มาตรฐานเบอร์ต่างๆดังนี้

ASTM sieve no.	Opening (mm.)
12	1.405
16	1.003
30	0.500
50	0.300
100	0.150
200	0.075
Pan	

วัตถุดิบ

Na-feldspar, Quartz, Lime stone, Milled sand

วิธีการทดลอง

กรณีใช้ non-plastic raw materials

1. ให้บดวัตถุดิบตามตารางที่แนบ sample ละ 500 กรัม ด้วยเครื่องบดในห้องทดลอง
2. อบ sample ที่บดแล้ว 100°C เป็นเวลา 15 นาที
3. ชั่งน้ำหนัก sample ที่บดแล้ว
4. ทำความสะอาดตะแกรง (ตะแกรงหยาบใช้แปรงลวด ตะแกรงละเอียดใช้แปรงขน)
5. ร่อน sample ในข้อ 3 ตามตะแกรงขนาดที่อาจารย์ผู้สอนกำหนดให้
6. ชั่งน้ำหนัก sample ที่ค้างบนตะแกรง (เคาะลงบนแผ่นพลาสติกแล้วนำไปชั่ง)
7. บันทึกข้อมูลตามตัวอย่างในตารางที่ 3
8. คำนวณ CMPF แล้วเขียนกราฟตามตัวอย่างรูปที่ 8

ผลการทดลอง

บันทึกข้อมูลตามตารางที่ 3 และคำนวณ CMPF พร้อมเขียนกราฟตามตัวอย่างกราฟในรูปที่ 8

เอกสารอ้างอิง

1. อ.สถาพร คูวิจิตรจารุ, คู่มือการทดสอบดิน เอกสารประกอบวิชา 410322 soil mechanics laboratory มหาวิทยาลัยเทคโนโลยีสุรนารี
2. J.T.Jones, M.F. Berard, Ceramic Industrial Processing and Testing
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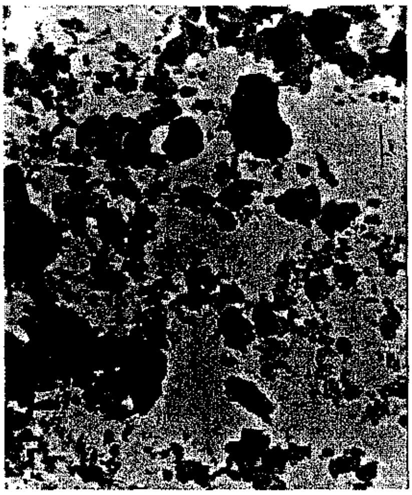
วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 2
CLAY PREPARATION AND WET SIEVE ANALYSIS

รศ.ดร. จรัสศรี ลอประยูร

บทนำ

แร่ดินจากธรรมชาติเกิดจากการแปรสภาพ (Weathering) มาจากหินอัคนี (Igneous rock) เช่น Feldspathic rock และแกรนิต ซึ่งมี Feldspar, quartz และ mica จึงมักพบดินอยู่รวมกับส่วนที่ยังไม่แปรสภาพ ดินบางแห่งถูกน้ำพัดพาไปไกลจากหินแห่งเดิม กระแสน้ำช่วยล้างทำให้ดินมีสภาพบริสุทธิ์สูงขึ้น มีเม็ดละเอียด ดินเหล่านี้จะไปตกตะกอน (Sedimentation) ทับถมปะปนกับสารอินทรีย์ในที่ที่ไกลออกไป ดินเหล่านี้ยังคงมีเนื้อละเอีนดและมีสารอินทรีย์ที่เน่าเปื่อยผุพังปะปนกัน ดินบางชนิดเกิดจากกระบวนการที่มีแก๊สร้อน ร่วมกับน้ำ (Hydrothermal action) ดินที่มีมากที่สุดคือ Kaolinite [$Al_2Si_2O_5(OH)_4$] มีรูปผลึกที่แสดงในรูปที่ 1 เป็นภาพถ่ายจาก Transmission electron microscope แผ่นบาง ๆ มีรูปแบบ Hexagonal ที่มีความหนาประมาณ 0.1 μm



รูปที่ 1 ภาพถ่ายของผลึก Kaolinite จาก Transmission electron microscope [1]

โดยทั่วไปถ้านำดินมาผสมน้ำในปริมาณที่พอเหมาะ ดินจะมีความเหนียว (Plasticity) เกิดขึ้น สามารถนำมาขึ้นรูปได้โดยใช้แรงกด อัดหรือรีด และเมื่อทิ้งไว้ก็ยังคงรักษารูปร่างไว้ได้ Kaolinite ที่มีขนาดอนุภาคเล็กมากๆ มีความเหนียวมาก เรียกว่า Ball clay ซึ่งมักจะนำไปผสมกับดินที่มีความเหนียวไม่มากเพื่อให้สามารถขึ้นรูปเป็นผลิตภัณฑ์ที่ต้องการได้

จากสภาวะการเกิดและการเคลื่อนย้ายแหล่งดินดังกล่าว ทำให้ดินที่นำมาใช้ในอุตสาหกรรมเซรามิกมีสมบัติทางกายภาพและทางเคมี แตกต่างกัน การนำไปใช้งานก็แตกต่างกันด้วย

การแบ่งประเภทของดินตามการใช้งาน โดย Norton [1]

A. White-burning clays (used in whiteware)
    a. Kaolin:
        i. Residual
        ii. Sedimentary
    b. Ball clays
B. Refractory clays (having a fusion point above 1600°C but not necessarily white burning)
    a. Kaolin (sedimentary)
    b. Fire clays:
        i. Flint
        ii. Plastic
    c. High alumina clays:
        i. Gibbsite
        ii. Diaspore
C. Heavy clay-products clays (of low plasticity but containing fluxes)
    a. Paving brick clays and shales
    b. Sewer-pipe clays and shales
    c. Brick and hollow tile clays and shales
D. Stoneware clays (plastic, containing fluxes)
E. Brick clays (Plastic, containing iron oxide)
    a. Terra-cotta clays
    b. Face and common brick
F. Slip clays (containing more iron oxide)

ดินที่พบเห็นโดยทั่วไปจะเกิดปะปนกับส่วนที่ไม่ใช่ดิน โดยปรกติก็จะมีความหยาบมากกว่าเนื้อดิน จึงจำเป็นต้องทำการล้างดินและกรองขนาดอนุภาคของตัวอย่างดิน

จุดประสงค์

เพื่อให้นักศึกษาสามารถเตรียมดินจากวัตถุดิบ โดยการล้างเพื่อแยกเนื้อดินออกจากแร่อื่นๆและได้เนื้อดินที่มีความสม่ำเสมอ สำหรับตรวจสมบัติทางกายภาพและเคมีเบื้องต้น และวิเคราะห์ความละเอียดของดินโดยวิธี wet sieve analysis ร่อนดินผ่านตะแกรงที่มีขนาดรูเปิดตามกำหนด

การทดลอง

การล้างดินเพื่อตรวจสมบัติต่างๆ

ให้นักศึกษาแต่ละกลุ่มนำตัวอย่างดินที่เตรียมไว้ ประมาณกลุ่มละ 2-5 กิโลกรัม ไปล้างดินโดยการกระจายเนื้อดินในน้ำ ทำให้กรวด ทราย ที่มีอนุภาคใหญ่ตกตะกอน เนื้อดินซึ่งประกอบด้วยอนุภาคขนาดเล็กจะแขวนตัวอยู่ในน้ำ แยกน้ำดินออกจากส่วนที่ตกตะกอย โดยการเทน้ำดินผ่านตะแกรงเบอร์ 200 เมช ทำน้ำดินให้ตกตะกอนโดยใช้สารเคมี รินส่วนที่เป็นน้ำใสออก เทส่วนที่ข้นลงในถังเกรอะดินที่ทำด้วยปูนปลาสเตอร์ที่มีผ้ารองรับอีกชั้นหนึ่งทิ้งไว้จนน้ำดินหมาด มีลักษณะที่นำไปนวดได้ จึงเก็บใส่ถุงพลาสติก ดินที่เก็บไว้ส่วนนี้จะใช้ในการทดลองอื่นๆต่อไป

อุปกรณ์และสารเคมี

1. เครื่องกวนดิน (Blunger)
2. Deflocculating agent เช่น sodium polyphosphates หรือ Sodium silicate
3. Flocculating agent เช่น $Al_2(SO_4)_3$ และ $AlCl_3$
4. ภาชนะแยกทรายและใส่ clay suspension
5. ผ้าดิบ
6. กะบะปูนปลาสเตอร์สำหรับเกรอะดิน
7. เครื่องชั่ง
8. ตะแกรงมาตรฐานเบอร์ต่างๆ ตามที่อาจารย์กำหนด

ASTM sieve no.	Opening (mm.)
50	0.297
100	0.105
200	0.074
325	0.045
Pan	

วิธีการทดลอง

1. นำตัวอย่างดิน 500 กรัม อบที่ 80-100°C เป็นเวลา 3-4 ชั่วโมง (ถ้าดินที่อบมาให้แล้ว ให้นำมา 400 กรัม)
2. ใส่ Deflocculating agent เช่น sodium silicate 0.2% ของน้ำหนักดินแห้ง
3. ใส่น้ำ 1 ลิตร กวนให้เป็นเนื้อเดียวกัน นำน้ำดินที่ได้ไปผ่านตะแกรงที่เรียงเป็นชั้นๆ เรียงจากหยาบไปหาละเอียดตามที่ผู้สอนกำหนดให้
4. ค่อยๆปล่อยน้ำจากก๊อกน้ำให้ไหลผ่านขณะที่เทน้ำดินร่อนบนตะแกรง
5. นำดินที่ค้างบนตะแกรงแต่ละเบอร์ไปเคาะบนแผ่นพลาสติก ส่วนที่เหลือติดตะแกรงให้ใช้แปรงค่อยๆ แปรงออกหรือใช้น้ำฉีดไล่ดินออกมาให้หมด แล้วจึงนำไปรวมกับดินที่เคาะออกมา
6. นำดินและน้ำดินจากข้อ 5 ใส่กระจกนาฬิกาแล้วนำไปอบจนแห้งสนิท
7. ชั่งน้ำหนักดินที่ค้างบนตะแกรงแต่ละเบอร์ เมื่ออบแห้งแล้ว
8. บันทึกข้อมูลตามตัวอย่างในตารางที่ 3 ในการทดลองที่ 1 และคำนวณ CMPF พร้อมเขียนกราฟตามตัวอย่างในรูปที่ 8 การทดลองที่ 1

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Clay Preparation and Wet Sieve Analysis (ส่วนเพิ่ม)

ผศ.ดร. สุธรรม ศรีหล่มสัก

แบ่งการทดลองนี้ออกเป็นสองส่วนคือ 1) การล้างดิน (clay preparation) และ 2) การวิเคราะห์ขนาดอนุภาคแบบเปียก (wet sieve analysis)

1) การล้างดิน Clay Preparation

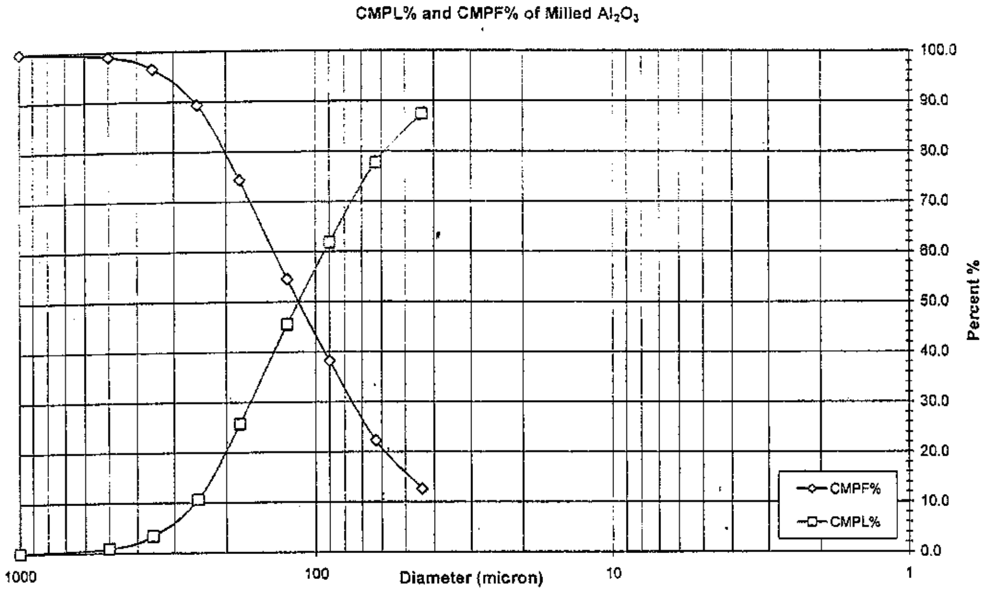
ส่วนนี้นักศึกษาจะได้ฝึกหัดการล้างดินที่มีอยู่ตามธรรมชาติให้สะอาดและเหมาะสมที่จะนำมาใช้งานทางเซรามิก แต่ละกลุ่มจะได้รับตัวอย่างดินกลุ่มละ 1 กิโลกรัม ให้นักศึกษาล้างแล้วเก็บตัวอย่างของดินและกากค้างตะแกรงแล้วนำมาส่งพร้อมกับรายงาน ส่วนดินที่ล้างเสร็จแล้วให้นักศึกษาเก็บไว้ใช้ในการทดลองอื่นต่อไป

2) การวิเคราะห์ขนาดอนุภาคแบบเปียก Wet Sieve Analysis

ส่วนนี้นักศึกษาจะได้หัดทำการวิเคราะห์หาขนาดอนุภาคแบบเปียก โรงงานทั่วไปจะเก็บข้อมูล wet sieve analysis ของเนื้อดินปั้นและเคลือบทุก batch ที่บดผสมเพื่อให้แน่ใจว่าการบดผสมเป็นไปตามมาตรฐาน ในการทดลองนี้นักศึกษาจะได้ดินกลุ่มละ 100 กรัม ให้ไปทำการวิเคราะห์หาขนาดอนุภาคของดินโดยแบบเปียกโดยนำดินไปผสมน้ำแล้วนำน้ำดินไปร่อนผ่านตะแกรงที่มีช่องเปิดต่างกัน 4 เบอร์ แล้วชั่งน้ำหนักกากค้างตะแกรงที่อบแห้งแล้วและเก็บตัวอย่าง ต่อจากนั้นนำข้อมูลน้ำหนักของกากค้างตะแกรงทุกเบอร์ไปคำนวณ cumulative percent finer than (CMPF%) และ cumulative percent larger than (CMPL%) แล้วนำไปเขียนกราฟ CMPF% และ CMPL% ส่งในรายงานพร้อมกับตัวอย่างของกากค้างตะแกรงทุกเบอร์

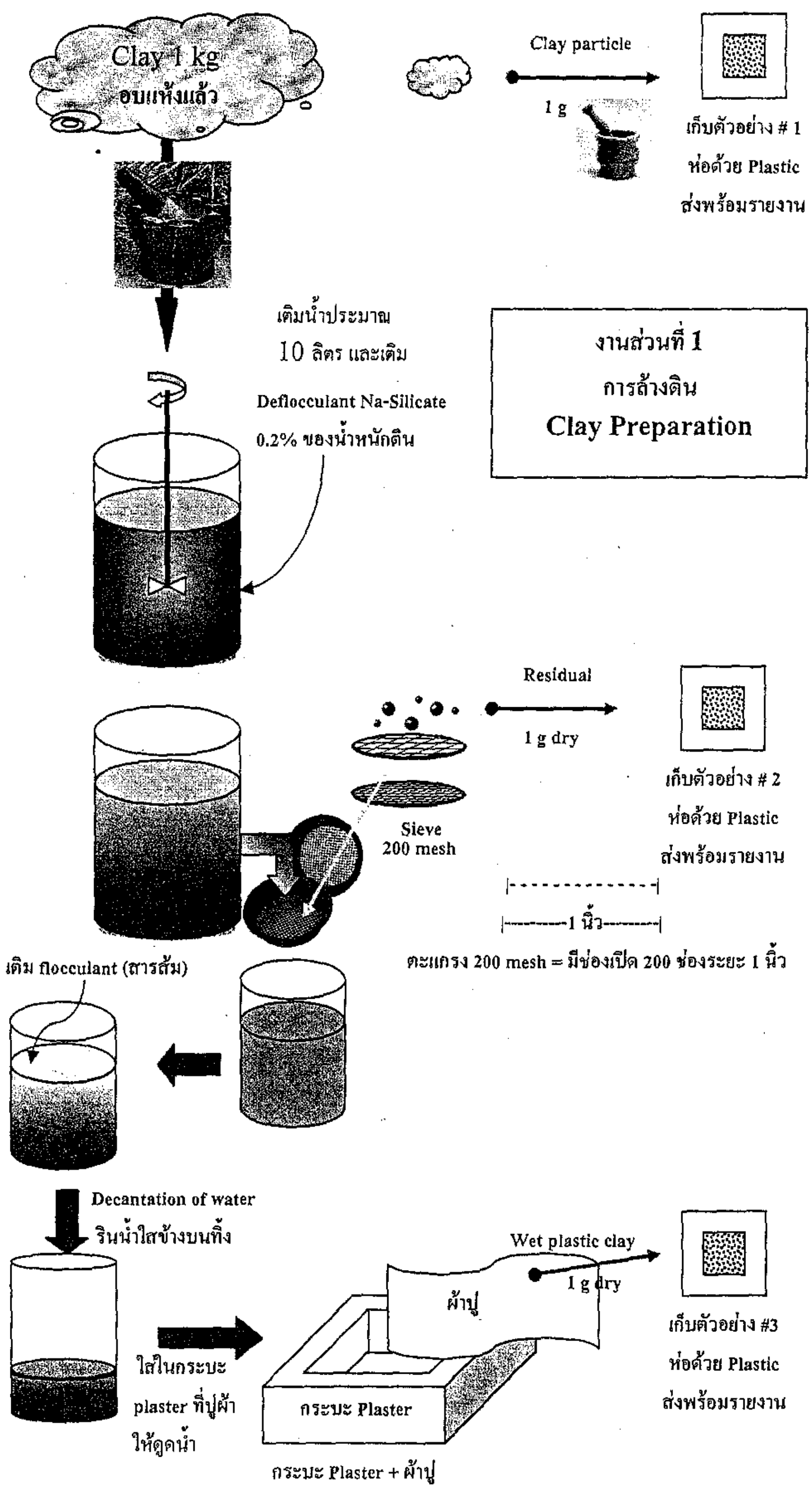
<u>ตัวอย่างการคำนวณ</u> CMPL% และ CMPF% สังเกตว่า CMPF% = 100 – CMPL%

sieve size	mass on sieve		CMPL%	CMPF%
	g	%		
1000	0.00	0.0	0.0	100.0
500	0.18	0.9	0.9	99.1
355	0.50	2.5	3.4	96.6
250	1.45	7.3	10.7	89.3
180	3.00	15.0	25.7	74.3
125	3.96	19.8	45.5	54.5
90	3.26	16.3	61.8	38.2
63	3.18	15.9	77.7	22.3
44	1.94	9.7	87.4	12.6
0	2.52	12.6	100.0	0.1
total	19.99	100.0		



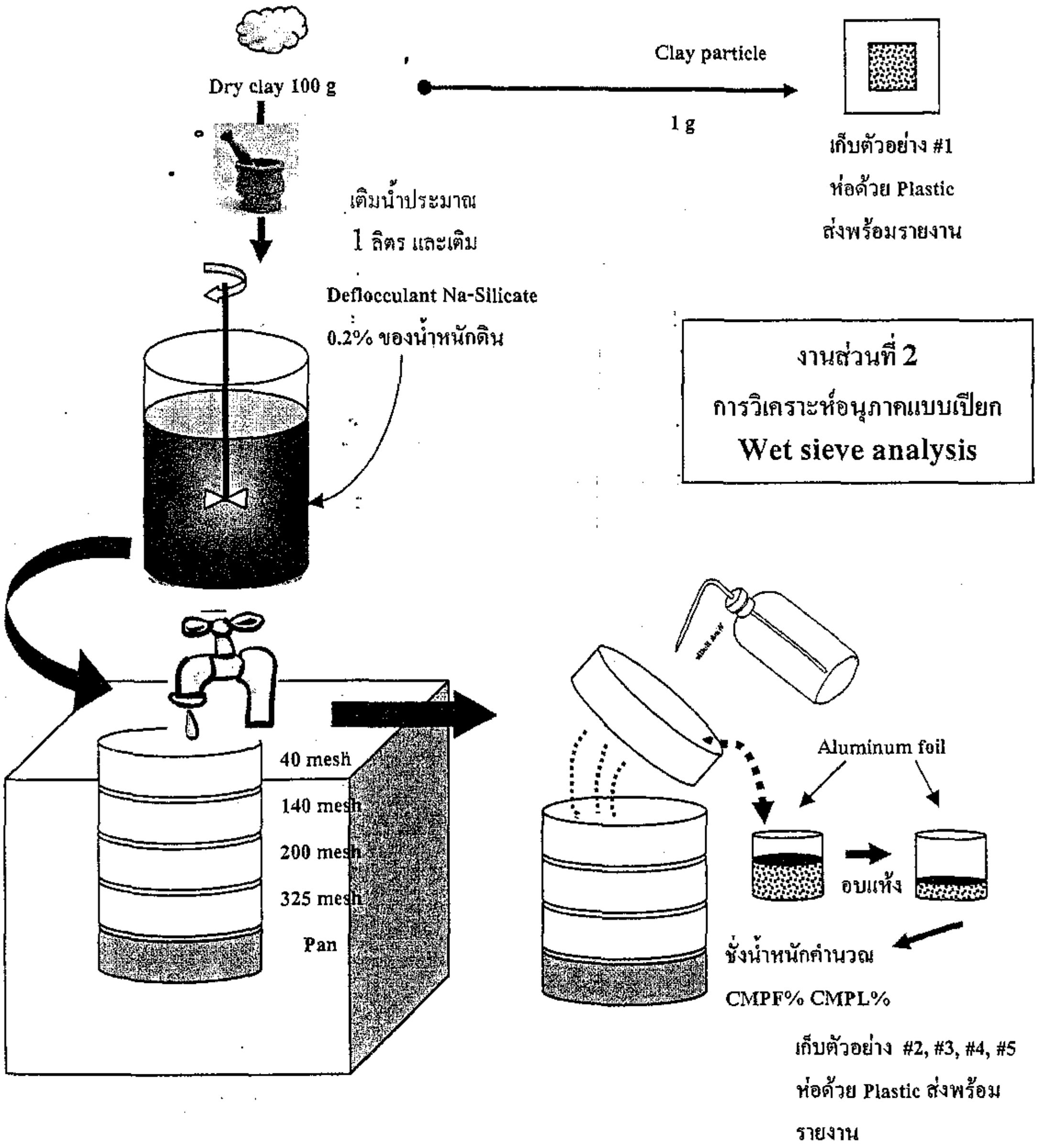
ตัวอย่างกราฟ CMPL% และ CMPF% สังเกตว่าแกนนอนเป็นแกน log แต่แกนตั้งเป็นแกนธรรมดา

แผนผังแสดงการทดลอง



<u>หมายเหตุ</u> อย่าเติม Na-silicate ในรูปของผง!!! แต่ให้เติม Na-silicate ในรูปของสารละลาย สารละลายที่ใช้ทำจาก Na-silicate 20 gm + น้ำ 500 cc

งานส่วนที่ 2
การวิเคราะห์อนุภาคแบบเปียก
Wet sieve analysis



<u>ตัวอย่าง</u> การคำนวณเปอร์เซ็นต์กากค้างตะแกรง (percent retained), เปอร์เซ็นต์กากค้างตะแกรงสะสม (cumulative % retained) และเปอร์เซ็นต์ที่เล็กกว่า (percent finer)

1) percent retained = น้ำหนักดินบนตะแกรงแต่ละเบอร์ ÷ น้ำหนักดินทั้งหมด
2) cumulative % retained ของตะแกรงเบอร์ใด = percent retained ของตะแกรงนั้น + percent retained ของตะแกรงที่เบอร์เล็กกว่าทุกเบอร์
   cumulative % retained = cumulative % larger than = CMPL%
3) percent finer = 100- cumulative % retained
   percent finer = cumulative % finer than = CMPF%

ตัวอย่างตารางเก็บข้อมูล

Sieve Analysis Sample Data

Analyst Name:______________________ Date:__________ Test No.:__________

Project Information:
Project Name:______________________
Project Location:______________________
Boring No:________ Sample No.________ Depth:________
Notes:______________________

Sample Description:
Source:______________ Condition:__________
Vis. Class. & USCS Symbol:______________
Color:__________ Grain Shape:__________
G.S.D.:__________ Mineral Comp.__________

Oven dry weight of sample: __500 g________

Sieve No.	Sieve Opening (mm)	Wt. Retained (g)	Percent Retained	Cumulative % Retained	Percent Finer
4	4.75	0	0	0	100
10	2.0	40.2	8.04	8.04	91.96
20	.850	84.6	16.92	24.96	75.04
40	.425	90.2	18.04	43.00	57.00
60	.250	106.4	21.28	64.28	35.72
140	.106	108.8	21.76	86.04	13.96
200	.075	59.4	11.88	97.92	2.08
pan	—	8.7			

Σ= __498.3____ = W1 Soil Loss: 0.34___

Sieve Analysis Data Sheet

Analyst Name:__________ Date:__________ Test No.:__________

Project Information:

Project Name:__________

Project Location:__________

Boring No:__________ Sample No.__________ Depth:__________

Notes:__________

Sample Description:

Source:__________ Condition:__________

Vis. Class. & USCS Symbol:__________

Color:__________ Grain Shape:__________

G.S.D.:__________ Mineral Comp.__________

Oven dry weight of sample:__________

Sieve No.	Sieve Opening (mm)	Wt. Retained (g)	Percent Retained	Cumulative % Retained	Percent Finer

$\Sigma=$__________$=W_1$ Soil Loss:__________

วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 3

DENSITY MEASUREMENTS

ผศ.ดร. สิริรัตน์ ทับสูงเนิน รัตนจันทร์ และ ผศ.ดร. สุขเกษม กังวานตระกูล

บทนำ:

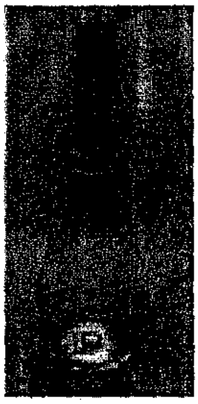
ความหนาแน่นของผงเซรามิก (Particle density) คือ น้ำหนักของผงหรืออนุภาคต่อปริมาตร ซึ่งในงานทางด้านเซรามิกจะวัดความหนาแน่นของดินหรือวัตถุดิบอื่นๆ การวัดความหนาแน่นของอนุภาคที่แท้จริง **(True density)** เป็นการวัดความหนาแน่นของผงหรือวัตถุดิบเซรามิกที่เป็นความหนาแน่นของเนื้อสารที่แท้จริง ซึ่งไม่ได้รวมปริมาตรของรูพรุนแบบเปิดหรือแบบปิดในเนื้อของวัสดุดังเช่นการหาความหนาแน่นที่ปรากฏ (Bulk density) และเป็นค่าที่ใช้ในการอ้างอิงทางฟิสิกส์และเคมีของผงวัตถุดิบนั้นๆ

วิธีการหาค่า True density

ในการหาค่าความหนาแน่นที่แท้จริง สามารถทำได้ 2 วิธีคือ การวัดโดยการแทนที่น้ำ เรียกว่า Pycnometer และการวัดปริมาตรของผงตัวอย่างด้วยอากาศหรือแก๊สที่เรียกว่า Air pycnometer ในการทดลองนี้นักศึกษาจะได้เรียนรู้เกี่ยวกับการวัดความหนาแน่นที่แท้จริงของของแข็งโดยการแทนที่ด้วยน้ำ

วิธีที่ 1 การวัดโดย Pycnometer method

วิธีนี้เป็นการวัดความหนาแน่นของของแข็งและของเหลวได้ และเป็นวิธีที่สะดวกและทำได้โดยง่าย วิธีนี้มีมาตรฐานการวัด เช่น British Standard BS 4550, ASTM D 854-00 Standard Test for Specific Gravity of Soil Solids by Water Pycnometer. เป็นวิธีการวัดปริมาตรโดยการแทนที่น้ำโดยใช้ขวด ถ.พ. ที่ทราบปริมาตรที่แน่นอน ขนาดไม่เกิน 50 ml. สำหรับผงที่มีขนาดละเอียดและใช้ขวดขนาดใหญ่ขนาด 1000 ml. สำหรับวัตถุดิบชนิดหยาบกว่า 2 mm. ขวด ถ.พ. ประกอบด้วยจุกที่มีรูเปิดเพื่อให้น้ำหรือของเหลวที่เกินล้นออกมาได้ ดังรูป



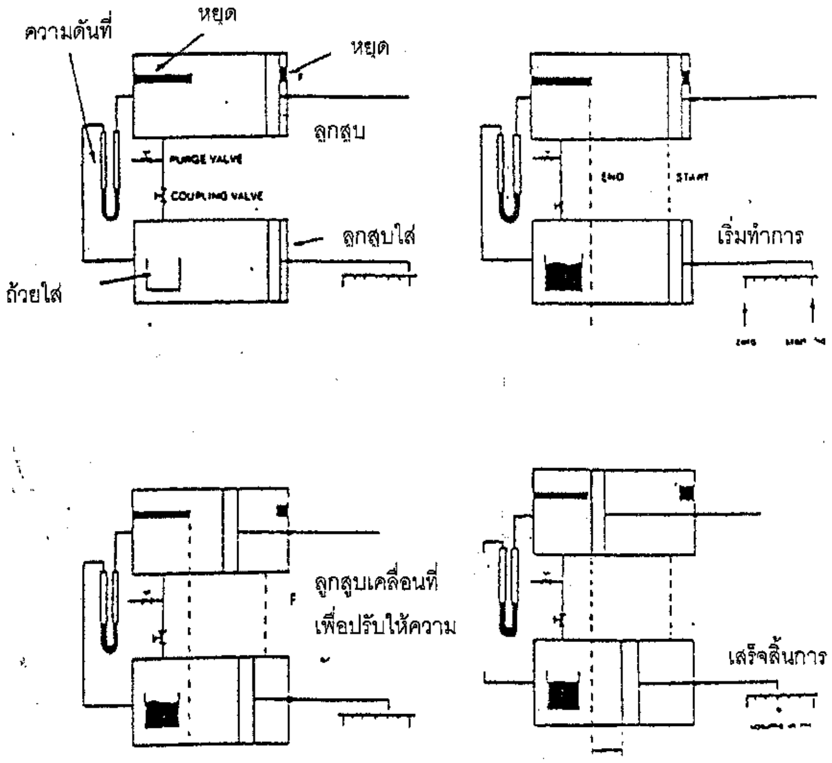
รูปที่ 1 ขวด pycnometer

การวัดทำได้โดยการหาปริมาตรของผงตัวอย่างโดยการแทนที่น้ำ ความหนาแน่นของผงหาได้จากน้ำหนักของผงแห้งต่อปริมาตรของผง ซึ่งปริมาตรของผงแห้งหาได้จากผลต่างของปริมาตรของน้ำที่เต็มขวดและปริมาตรของน้ำที่เติมลงไปในผงตัวอย่างจนน้ำเต็มขวด ในกรณีที่สารตัวอย่างสามารถละลายน้ำหรือทำปฏิกิริยากับน้ำได้ ควรจะเลือกใช้สารละลายชนิดอื่นแทนน้ำ

วิธีที่ 2 การวัดโดย Air pycnometer

เป็นการวัดความหนาแน่นของของแข็งเท่านั้น โดยเฉพาะใช้ในงานที่ต้องการหาความหนาแน่นของดิน หรือความถ่วงจำเพาะของดิน โดยการแทนที่ด้วยอากาศหรือแก๊ส ตัวอย่างที่กล่าวนี้เป็นเครื่องมือของ Beckman model 930 air comparison pycnometer หรือเครื่อง air pycnometer ที่ใช้หลักการของ Langer [1]

Air Pycnometer ประกอบด้วยกระบอกสูบ พร้อมทั้งลูกสูบ 2 ชุด ดังรูปที่ 2 ชุดหนึ่งเป็นลูกสูบอ้างอิงและปล่อยให้ว่างเปล่า อีกชุดหนึ่งมีที่สำหรับใส่ถ้วยผงตัวอย่างเข้าไป เมื่อใส่ตัวอย่างลงไป ตัวอย่างจะเข้าแทนที่แก๊สหรืออากาศที่บรรจุอยู่ในกระบอกสูบ สามารถหาปริมาณแก๊สที่ถูกแทนที่ โดยการปรับระยะที่เลื่อนของลูกสูบทั้งสองนั้น เราสามารถอ่านปริมาตรของผงตัวอย่างได้โดยตรง



รูป 2 Air Pycnometer

จุดประสงค์การทดลอง

เพื่อให้นักศึกษาได้เรียนรู้ และสามารถหาค่าความหนาแน่นของผงเซรามิกชนิดต่างๆได้โดยวิธีการแทนที่น้ำ หรือใช้วิธี Pycnometer

การทดลอง

วัสดุและอุปกรณ์

ดินตัวอย่าง

ขวด ถ.พ. พร้อมจุก (pycnometer)

Hot plate หรือ Ultrasonic bath

เครื่องชั่งละเอียด
น้ำกลั่น

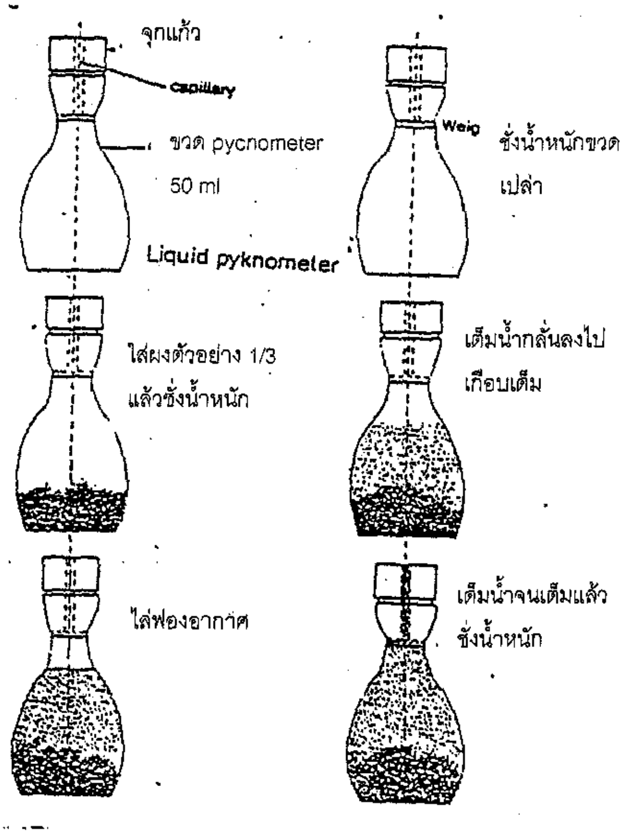
วิธีการทดลอง

1. เตรียมดินและขวด pycnometer

นำดินตัวอย่าง (อาจมาจากการทดลองที่ 1) มาบดให้ละเอียดผ่านตะแกรงเบอร์ 150 mesh แล้วนำไปอบแห้ง พร้อมทั้งอบขวด ถ.พ. จนทั้ง 2 ขวดมีน้ำหนักคงที่ จึงเก็บเข้า Desiccators

2. ชั่งขวด pycnometer ด้วยเครื่องชั่งละเอียด 4 ตำแหน่ง 2 ครั้ง นำมาหาค่าเฉลี่ย เป็นน้ำหนักขวดเปล่า W_1

3. บรรจุผงดินลงในขวดประมาณ 1/3 ของปริมาตรขวด(ประมาณ 10 กรัม) บันทึกน้ำหนักที่อ่านได้ 2 ครั้งนำมาหาค่าเฉลี่ย เป็น W_2



รูป 3 การหาความหนาแน่นด้วย pycnometer

4. เทน้ำกลั่นลงในขวดที่มีผงตัวอย่าง จนระดับน้ำอยู่ที่ปริมาณ 4/5 ของปริมาตรขวด นำไปไล่ฟองอากาศโดยการนำไปต้มใน water bath หรือนำไปใส่ในเครื่อง ultrasonic bath เป็นเวลาประมาณ 30 นาที แล้วเติมน้ำกลั่นให้เต็มขวด ปิดขวดด้วยจุก รักษาระดับน้ำในขวดให้เต็มพอดี โดยน้ำที่เต็มเกินจะล้นออกมาทางรูเปิดที่จุก เช็ดขวดให้แห้ง นำไปชั่งน้ำหนัก 2 ครั้งเพื่อหาค่าเฉลี่ย ได้เป็น W_3

5. นำ pycnometer ไปล้างให้สะอาด นำไปอบให้แห้งแล้วเติมน้ำกลั่นจนเต็ม ค่อยๆปิดด้วยจุกเพื่อให้น้ำล้นออกมา รักษาระดับน้ำให้เต็มขวดพอดี นำไปชั่งน้ำหนัก 2 ครั้งหาค่าเฉลี่ยได้เป็น W_4

ขั้นตอนการทดลองแสดงดังรูปที่ 3

การคำนวณ

ความหนาแน่น ρ = น้ำหนักของผงดินแห้ง / ปริมาตรของผงดินนั้น

$$\rho = \frac{W_2 - W_1}{(W_4 - W_1) - (W_3 - W_2)}$$

รายงานผล

รายงานผลการวัดความหนาแน่นจากการทำการทดลอง 2 ครั้งแล้วหาค่าเฉลี่ย (ดัง Data sheet) ที่แนบมาด้านหลัง

เอกสารอ้างอิง

1. Svarovsky, L. Powder Testing Guide: Methods of Measuring the Physical Properties of Bulk Powders, Elsevier Applied Science, London and New York.

2. Air pycnometer according to Langer, Eijkelkamp agrisearch equipment, 2003

3. Ryan, W. and Radford, C., Whitewares Production, Testing and Quality Control, Pergamon Press, 1987, pp. 232.

4. คณาจารย์สาขาวิชาวิศวกรรมเซรามิก, คู่มือปฏิบัติการวัตถุดิบเซรามิก พ.ศ. 2542

การหาความหนาแน่นของผงตัวอย่าง (Density determination)

ใบบันทึกผลการทดลอง

วันที่ทดลอง:

ทดลองโดย:

ชื่อสมาชิก:

ชื่อผงตัวอย่าง:

การทดลอง	ครั้งที่ 1	ครั้งที่ 2
น้ำหนักขวดเปล่า (W_1)		
น้ำหนักขวด + ผงตัวอย่าง (W_2)		
น้ำหนักขวด + ผงตัวอย่าง + น้ำเต็มขวด (W_3)		
น้ำหนักขวด + น้ำกลั่นเต็มขวด (W_4)		
ความหนาแน่นของผง		

วิธีการคำนวณ :

วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 4

METHYLENE BLUE ADSORPTION

ผศ.ดร. ศิริรัตน์ ทับสูงเนิน รัตนจันทร์ และ ผศ.ดร.สุธรรม ศรีหล่มสัก

บทนำ:

วัตถุดิบเซรามิกที่ใช้ผสมเป็นเนื้อดินปั้น โดยทั่วไปจะมีขนาดเล็ก ซึ่งขนาดของอนุภาคดินจะมีผลต่อคุณสมบัติต่างๆ เช่น ความเหนียว การหดตัวและความแข็งแรงเมื่อแห้งของเนื้อดิน การกระจายลอยตัว ดังนั้นในการควบคุมขนาดอนุภาคของดินจะช่วยให้สามารถควบคุมขั้นตอนการผลิต และได้ผลิตภัณฑ์ที่มีคุณภาพสม่ำเสมอได้ โดยปรกติอนุภาคของดินที่มีขนาดใหญ่กว่า 100 ไมครอน สามารถหาได้ด้วยการคัดขนาดผ่านตะแกรงร่อน แต่สำหรับอนุภาคที่มีขนาด 100-0.2 ไมครอนนั้น สามารถหาได้ด้วยเทคนิคอื่น เช่น Hydrometer method, Andreasen pipette และสำหรับอนุภาคที่มีขนาดเล็กมากๆ ในช่วงคอลลอยด์ คือ 0.2-0.001 ไมครอน ต้องใช้วิธีทางเคมีที่ยุ่งยากเช่น nitrogen adsorption หรือ Water vapor adsorption และเนื่องจากอนุภาคของดินโดยทั่วไปเมื่อกระจายในน้ำจะมีสมบัติเป็นอนุมูลลบ จึงสามารถดูดซับอิออนบวกอื่นได้ เช่น การดูดซับ methylene blue ซึ่งเป็น cationic dye เราจึงสามารถหาขนาดอนุภาคของดิน พื้นที่ผิวและความสามารถในการแลกเปลี่ยนอิออนของดินได้ด้วยเทคนิค mehtylene blue adsorption ดังนั้นในปฏิบัติการนี้นักศึกษาจะได้เรียนรู้เกี่ยวกับเทคนิค Methylene blue adsorption

Methylene Blue Adsorption

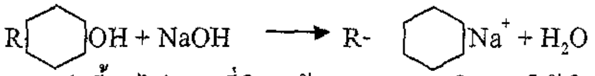
หลักการ

1. เมื่ออนุภาคของดินโดยทั่วไปกระจายตัวในน้ำ จะมีคุณสมบัติเป็นอนุมูลลบซึ่งสามารถดูดซับอนุมูลบวกของ methylene blue ($C_{16}H_{18}N_3SCl.3H_2O$) ได้ ดินที่มีอนุภาคขนาดคอลลอยด์มากเช่น ดินเหนียว (ball clay) จะมีพื้นที่ผิวจำเพาะ (Specific surface area) สูง จึงสามารถดูดซับ metylene blue ได้มากกว่าดินที่มีอนุภาคขนาดคอลลอยด์น้อยกว่า

2. F.A. Faruqi, et.al [4], M.M. Kanonova [5] และ C. Steelink [6] พบว่า Organic impurity ที่มีในดิน โดยเฉพาะพวกที่มีน้ำหนักโมเลกุลสูง และประกอบด้วยหมู่ carboxyl, phenolic hydroxyl สามารถแตก

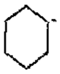
ตัวออกมาเป็นอนุมูลบวก และลบได้ดี เมื่ออยู่ในสภาวะที่เป็นด่างหรือเมื่อมี alkaline cation อยู่มาก ดังสมการ 1 และ 2

ที่ pH 6-8 $\quad R\text{-}COOH + NaOH \longrightarrow RCOO^- + Na^+ + H_2O$ (1)

ที่ pH 9-11  (2)

อนุมูลที่แตกออกมาเหล่านี้จะไปเกาะที่ผิวหน้าของอนุภาคดิน ทำให้เกิดเป็น Protective colloid ที่ขอบของอนุภาคดินดังแสดงในรูปที่ 1

(a)ในสภาพเป็นด่าง (b) ในสภาพเป็นกรด

รูปที่ 1 อนุภาคของดินในสภาพเป็นด่าง (a) $RCOO^-$, R-  จะเกิดเป็น Protective layer เกาะที่ผิวของอนุภาคดิน แต่ในสภาพที่เป็นกรด (b) จะไม่เกิด

ดังนั้นเมื่อนำน้ำดินที่มี Organic impurity และมีความเป็นด่างมาทำการทดสอบการดูดซับ Methylene blue จะได้ค่า Methylene blue index (MBI) สูงกว่าความเป็นจริงมาก ดังนั้นในการวิเคราะห์ด้วยวิธีนี้จะต้องควบคุมให้ pH ของสารละลายน้ำดินอยู่ในช่วง 3-4

จุดประสงค์การทดลอง

เพื่อให้นักศึกษาได้เรียนรู้ และสามารถหาค่า MBI และ specific surface area โดยวิธี Methylene blue adsorption

วิธีการทดสอบ

วิธีการ methylene blue adsorption ทำได้โดยการไทเทรตน้ำดินที่ปรับ pH ให้อยู่ในช่วง 3-4 ด้วย methylene blue เมื่อดินดูดซับ Methylene blue จนถึงจุดอิ่มตัวจนไม่สามารถดูดซับได้อีกต่อไป ถ้าเรานำน้ำดินที่ไทเทรตมาหยดลงบนกระดาษกรอง Methylene blue ที่เหลือและไม่ได้ถูกดูดซับจะเกิดการแพร่กระจาย

ลงบนกระดาษกรองเป็นสีฟ้าอมเขียว มีลักษณะเป็นวงรอบๆจุด เรียกว่า Halo ซึ่งเป็นปริมาณของ methylene blue ที่ปกคลุมอิ่มตัวบนผิวหน้าของอนุภาคดินซึ่งเรียกว่า “Optimum flocculation”

P.T. Hang และ G.W. Brindley [1] สามารถคำนวณพื้นที่ผิวจำเพาะของดิน 1 กรัมได้จาก

$$\boxed{\text{Specific surface area } (m^2/g) = M \times A \times 6.02 \times 0.01}$$

เมื่อ M คือ Methylene blue index (MBI) มีค่าเท่ากับ milliequivalent of methylene blue ที่ถูกดูดซับจนถึงจุด Optimum flocculation และ A คือ พื้นที่ผิวของ methylene blue cation ซึ่งมีค่าเท่ากับ 129.2 square angstroms

วิธีนี้มักรายงานผลเป็นค่า MBI คือ milliequivalent ของ methylene blue ที่ใช้ต่อดินแห้ง 100 กรัม ในการไทเทรตให้ถึงจุด optimum flocculation ค่าที่บอกว่าวัตถุดิบนั้นสามารถดูดซับเมทีลีนบูลได้มากน้อยเพียงใด ซึ่งสามารถจะบอกเป็นค่าความละเอียดของวัตถุดิบและสมบัติอื่นๆ เช่น ค่า MBI มาก แสดงว่าวัตถุดิบนั้นมีความละเอียดมาก และมีพื้นที่ผิวมาก มีความสามารถในการแลกเปลี่ยนอนุมูลได้มาก ซึ่งทำให้ทราบคุณสมบัติอื่นๆของเนื้อดินปั้นเมื่อเรานำวัตถุดิบนั้นๆไปเป็นส่วนผสม

อุปกรณ์การทดลอง

1. ครกบดดิน
2. เตาอบไฟฟ้าเพื่อไล่น้ำและความชื้นในดิน
3. ตะแกรงร่อนขนาด 120 เมช
4. ช้อนตักสาร
5. เครื่องชั่งที่มีความละเอียด 4 ตำแหน่ง
6. Magnetic Stirrer หรือเครื่องปั่นผสม
7. Beaker ขนาด 600 ml
8. กระบอกฉีดน้ำแบบพลาสติกบีบได้สำหรับใช้ฉีดเอาดินลงในบีกเกอร์
9. pH meter หรือกระดาษวัด pH ใช้วัดเพื่อปรับค่า pH ของน้ำดิน
10. กระดาษกรอง ชนิด Harden (Baroid เบอร์ 987 หรือเทียบเท่า)
11. Pipette หรือ Dropper หรือแท่งแก้วคน สำหรับจุ่มสารละลายน้ำดินที่กำลัง titrate มาเช็คจุดอิ่มตัว

12. Buret 25 ml

สารเคมีที่ต้องใช้

1. น้ำกลั่นหรือ deionized water สำหรับผสมกับดินในการ titrate
2. สารละลายกรด H_2SO_4 ความเข้มข้น 0.1 normal เตรียมโดยการละลาย H_2SO_4 ลงใน deionized wate จนมีความเข้มข้น 0.1 normal
3. Methylene blue solution ความเข้มข้น 0.01 normal เตรียมโดยการละลาย Methylene blue cholride ($C_{16}H_{18}N_3SCl.3H_2O$) 1.87 gm ลงใน deionized water 500 cc

การคำนวณสารละลายกรด 1 normal

ความเข้มข้นของสารละลาย 1 Normal หมายถึง สารละลายปริมาตรหนึ่งลิตรจะมีเนื้อสารละลายอยู่ 1 กรัมสมมูลย์ หรือสารละลายปริมาตร 1 มิลลิลิตร จะมีเนื้อสารอยู่ 1 มิลลิกรัมสมมูลย์ (milligram-equivalence)

$$\text{จำนวนกรัม-สมมูลย์} = \frac{\text{น้ำหนักโมเลกุลของสาร (กรัม)}}{\text{จำนวนวาเลนซี}}$$

เนื่องจากกรดเป็นของเหลวที่มีฤทธิ์กัดกร่อนสูง จึงมักนิยมเจือจางสารละลายกรดเข้มข้นให้มีความเข้มข้นที่ต่ำกว่า โดยใช้สูตร

$$V = \frac{100xMWxN}{\%xExSp.gr.}$$

เมื่อ V = ปริมาตร (มิลลิลิตรของกรดเข้มข้น) ที่ต้องการจะเจือจางให้มีปริมาตรครบ 1 ลิตร

MW = น้ำหนักโมเลกุลของกรด

N = ความเข้มข้นเป็น Normal ที่ต้องการเตรียม

% = เปอร์เซ็นต์ความเข้มข้นของกรดที่นำมาเจือจาง

E = จำนวนวาเลนซีของกรด

Sp.gr. = ความถ่วงจำเพาะของกรด

คำถาม: นักศึกษาลองคำนวณการเตรียมสารละลายกรด HCl ความเข้มข้น 1 normal ปริมาณ 100 มิลลิลิตร จากกรด HCl เข้มข้น 36% ความถ่วงจำเพาะ 1.19 น้ำหนักโมเลกุลของกรด HCl = 36.47 (ตอบ 86 มิลลิลิตร)

การคำนวณการเตรียมสารละลาย Methylene Blue เข้มข้น 0.01 normal จำนวน 1000 cc

methylene blue มีสูตรเป็น ($C_{16}H_{18}N_3SCl.3H_2O$) molecular weight = 373.905 g เมื่อแตกตัวจะเสียน้ำ 3 mole ออกไป ∴ methylene blue 0.01 normal คือ นำ methylene blue มา 0.01 mole แล้วเติมน้ำให้เป็น 1000 cc

∴ เตรียม methylene blue 0.01 normal โดยใช้ MB = 373.905 x 0.01 g แล้วเติมน้ำให้เป็น 1000 cc

ขั้นตอนการทดลอง

1. ดินที่นำมาทดสอบควรจะแห้งสนิท (ผ่านการอบประมาณ 70-80°C) และผ่านการบดให้มีอนุภาคเล็ก

2. นำดินที่จะทดสอบมาร่อนผ่านตะแกรงเบอร์ 120

3. แบ่งดินที่ได้จาก 2 มาโดยวิธีตัดกองเหมือนแบ่งขนมเค็ก เพื่อที่จะได้ดินที่มีขนาดเฉลี่ยเป็นตัวอย่างที่ดีของกลุ่มได้

4. ชั่งดินที่ได้จาก 4 ด้วยเครื่องชั่งที่มีความผิดพลาดไม่เกิน 0.001 g โดยเลือกดินที่จะทำการทดลองดินขาว, ดินเหนียว , montmorrillonite อย่างละ 2 กรัม

5. ผสมดินตามข้อ 4 กับ deionized water 300 cc โดยเครื่องปั่นผสมตามระยะเวลาปั่นผสมดังนี้

1/ดินขาว	5	นาที
2/ดินเหนียว	10	นาที
3/ดิน montmorillonite	15	นาที

เทน้ำดินลงใน Beaker 600 cc น้ำดินที่เหลือติดเครื่องปั่นให้ใช้กระบอกฉีดน้ำที่บรรจุ deionize water ฉีดลงมาให้หมด

6. ปรับ pH น้ำดินจาก ข้อ 5 ให้มีค่าระหว่าง 2.5-3.8 ด้วยสารละลายกรดที่เตรียมให้จะต้องคอยกวนผสมน้ำดินอย่างต่อเนื่องประมาณ 10-15 นาที

7. นำสารละลาย Methylene blue ที่เตรียมมาใส่ในบิวเรต อ่านสเกลที่ระดับสารละลาย จดบันทึกไว้

8. หยดสารละลายเมทิลีนบูล ความเข้มข้น 0.01 N จากปิเปตที่เตรียมให้ครั้งละ 5 ml กวนให้เข้าเนื้อกันอย่างน้อย 1-2 นาที ก่อนหยดสารละลาย ครั้งต่อไป

8. ใช้แท่งแก้วแตะสารละลายมาหยดลงบนกระดาษกรองที่เตรียมให้ 1 หยด ทุก ๆ ครั้งที่เติมสารละลายเมทิลีนบูล ลงไป 5 ml ช่วงแรก ๆ ขณะยังไม่ถึงจุด end point สีเมทิลีนบูล จะยังไม่ซึมเข้าไปในกระดาษกรอง เมื่อสังเกตเห็นว่ามีเมทิลีนบูลส่วนเกินที่ไม่ถูกดินดูดซับจะซึมออกมาติดเนื้อกระดาษกรองเป็นสีฟ้าอมเขียว จึง

ค่อยๆเติมสารละลาย Methylene blue ครั้งละ 1 ml แล้วคนต่อเนื่องประมาณ 2 นาที ก่อนที่จะทดสอบหยดบนกระดาษกรองอีกครั้งหนึ่ง ลักษณะที่ถึงจุดสุดท้าย จะมีลักษณะเป็น halo

9. เพื่อให้มั่นใจว่าถึงจุด End point จริง ให้กวนผสมน้ำดินต่อไปอีก 2 นาที แล้วหยดน้ำดินลงบนกระดาษกรองอีกครั้ง ถ้าคราวนี้ไม่มีลักษณะ halo ให้ titrate ต่อไปอีกจนถึงจุด end point

วิธีการคำนวณค่า Methylene blue index (MBI)

$$\mathrm{MBI} = \frac{ExV}{W}x100$$

เมื่อ MBI = Methylene blue index สำหรับดินนั้นๆมีค่า meq/100 กรัมของดิน

E = Miliequivalents ของ Methylene blue ต่อ 1 มิลลิลิตร

V = ปริมาณ Methylene blue ที่ใช้ในการไทเทรต (มิลลิลิตร)

W = น้ำหนักของดินแห้ง (กรัม)

เมื่อใช้ดินแห้ง 2 กรัม และไทเทรตด้วย Methylene blue ความเข้มข้น 0.01 N ได้ดังนี้

$$\mathrm{MBI} = \frac{0.01xV}{2}x100 = 0.5V$$

การคำนวณและรายงาน

ให้แสดงการคำนวณค่า MBI และ specific surface area ของดินที่ทำการทดสอบ

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11) C 837-99 (2003) Standard Test Method for Methylene Blue Index of Clay

วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 5

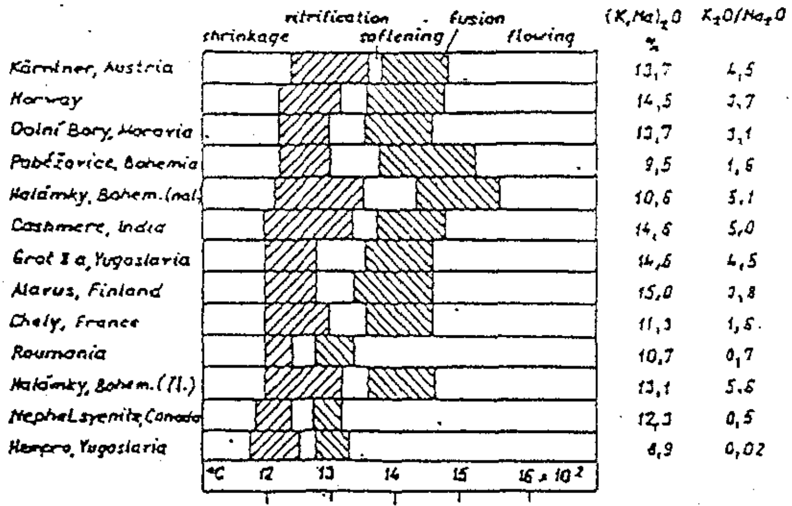
FELDSPAR TEST

รศ.ดร.จรัสศรี ลอประยูร

บทนำ

Feldspar เป็นวัตถุดิบที่มีความสำคัญมากในอุตสาหกรรมเซรามิก ใช้ในการผสมเป็นเนื้อดินปั้น (Body) ชนิดต่างๆและผสมในน้ำเคลือบ ซึ่ง Feldspar ทำหน้าที่เป็นตัวลดจุดสุกตัวของเนื้อดินปั้น (หรือทำหน้าที่เป็น Flux) ซึ่งเป็นตัวที่ก่อให้เกิดแก้วขึ้นในเนื้อผลิตภัณฑ์ที่ทำให้เกิดเนื้อแน่นและมีความโปร่งแสงดี แต่ความสามารถในการลดจุดหลอมขึ้นอยู่กับชนิดและปริมาณของ Cations ที่มีอยู่ใน feldspar นั้นๆ Feldspar จัดเป็นแหล่งของ Alkalies และ Alumina ในเคลือบและแก้ว

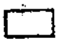
Feldspar คือสารประกอบพวก Aluminosilicates ของ Sodium, Potassium และ Calcium นอกจากนี้ยังประกอบด้วยธาตุหายากอื่นๆ ปะปนอยู่ในปริมาณเล็กน้อย และจัดเป็นสารประกอบ Alkalies ที่ไม่ละลายน้ำ โครงสร้างอะตอมของ Feldspar เป็นโครงร่างตาข่ายของ Oxygen-silicon tetrahedron ที่เชื่อมต่อมุมทั้งสี่ด้าน Al^{3+} อาจแทนที่ Si^{4+} บางส่วนและมี K^+, Na^+ หรือ Ca^{2+} เข้าไปอยู่ในช่องเปิดนั้น ดังนั้นโครงสร้างจึงเปลี่ยนแปลงได้ขึ้นอยู่กับ cations ที่เข้าไปอยู่ในโครงสร้าง เช่น orthoclase เป็น monoclinic ถ้าใน feldspar มี orthoclase และ albite ปะปนกัน จะได้โครงสร้างแบบ monoclinic หรือ triclinic ขึ้นอยู่กับปริมาณของ K และ Na ที่มีอยู่ และนอกจากนี้โครงสร้างของ Feldspar สามารถเปลี่ยนได้เมื่ออุณหภูมิสูงขึ้น โดยทั่วไปสูตรของ Feldspar คือ MZ_4O_8 เมื่อ M เป็น Na^+, K^+ หรือ Ca^{2+} (บางทีก็อาจเป็น NH^{4+}, Ba^{2+} และ Fe^{2+} ปนอยู่ด้วย ซึ่งสามารถเขียนสูตรได้เป็น $K(AlSi_3O_8)$, $Na(AlSi_3O_8)$ และ $Ca(Al_2Si_2O_8)$ ที่มีชื่อเรียกว่า Orthoclase, Albite และ Anorthite ตามลำดับ) ในธรรมชาติมักเกิดร่วมกันใน ternary system เมื่อกลุ่มแร่ $K(AlSi_3O_8)$ อยู่ร่วมกับ $Na(AlSi_3O_8)$ เรียกว่า Alkali feldspar ถ้า $Na(AlSi_3O_8)$ อยู่ร่วมกับ $Ca(Al_2Si_2O_8)$ เรียกว่า Plagioclase feldspar มีชื่อย่อเรียกตามความแตกต่างของอัตราส่วนโมลของ Na/Ca จากมากไปน้อยดังนี้ Albite, Aligonite, Andesite, Labradorite, Bytownite และ Anorthite แร่ feldspar มีปริมาณ cations ต่างกันจึงทำหน้าที่เป็น flux ที่ต่างกัน ปริมาณ Alkalies มากจะหลอมง่ายและทำปฏิกิริยากับสารอื่นๆในเนื้อดินปั้นได้อย่างรวดเร็ว K-feldspar หลอมที่อุณหภูมิสูงกว่า 1150°C ขณะที่ Na-feldspar หลอมที่อุณหภูมิต่ำกว่านั้น ปริมาณ K_2O/Na_2O มีค่าต่ำ (มี Na-feldspar มาก) ก็หลอมได้ที่อุณหภูมิต่ำ



Changes of the original rectangular silhouette of a cylindrical pellet (2 mm × 2 mm) of alkali feldspar visible in the heating microscope with increasing temperature (after Obst, 1964), modified).

รูปที่ 1 แสดงส่วนประกอบทางเคมีและพฤติกรรมของ Feldspar แหล่งต่างๆ

ในการใช้งาน K-feldpar หลอมแล้วจะมีความหนืดสูงกว่า Na-feldspar จึงนิยมใช้ K-feldspar ในอุตสาหกรรมเซรามิก โดยผสมทำเป็นเนื้อดินปั้น และใช้ Na-feldspar ทำน้ำเคลือบของเซรามิก และใช้ในอุตสาหกรรมแก้ว ถ้าเพิ่มความร้อนให้กับ Feldspar เราจะสังเกตเห็นความเปลี่ยนแปลงของรูปทรงที่เกิดขึ้น จากการศึกษาโดยใช้กล้องจุลทรรศน์ชนิดที่เพิ่มความร้อนได้ ทำการเพิ่มความร้อนในอัตรา 10°K /min จากอุณหภูมิ 20 ถึง 1700°C เราสามารถสังเกตเห็นการเปลี่ยนแปลงต่างๆเกิดขึ้น เช่น shrinkage, vitrification, softening, fusion และการไหลของ Feldspar ที่หลอมจึงมีการกำหนดลักษณะที่เห็นกับการเปลี่ยนแปลงไว้ดังนี้ การเปลี่ยนแปลงรูปร่างแต่ละขั้นตอนของ Feldspar เมื่อได้รับความร้อนแตกต่างกันเนื่องจาก feldspar มี Na,K และ Ca แตกต่างกัน เราสามารถเปรียบเทียบปริมาณ Flux ใน Feldspar โดยสังเกตลักษณะที่เปลี่ยนไปนี้ ส่วนในประเทศไทยนั้นพบแหล่ง Feldspar หลายแห่งได้แก่ จังหวัด ตาก อุทัยธานี และราชบุรี

Deformation points	**Characteristics Features**
Shrinkage point 	จุดสุดท้ายของการคงรูปเดิมของ feldspar ต่อจากนี้ จะเริ่มเปลี่ยนขนาด ได้
Vitrification point 	เริ่มกลายเป็น liquid phase และมีขนาดเล็กลงอย่างรวดเร็ว แต่ยังมีรูปร่างเหมือนเดิม
Softening point 	มี liquid phase เกิดขึ้นมากมาย มีรูปร่างลักษณะภายนอกเปลี่ยนไป มุมหายไป ด้านบนเริ่มโค้ง
Fusion point 	ผิวและขอบหลอมละลาย เปลี่ยนรูปร่างจากเหลี่ยมเป็นมน มีลักษณะ " Drop like shape "
Advanced fusion point 	มีความสูงลดลง เพิ่มส่วนกว้างออกไป
Flowing point 	ฐานเริ่มแผ่ออกเป็นรูปครึ่งวงกลม

รูป 2 ลักษณะรูปร่างของการเปลี่ยนแปลงขณะหลอม

จุดประสงค์

เพื่อให้นักศึกษาสามารถตรวจดูสภาพการหลอมของ Feldspar และเปรียบเทียบปริมาณ Flux กับลักษณะการหลอม

การทดลอง

อุปกรณ์และสารเคมี

1. Feldspar
2. กระบอกตวง ขนาด 100 ml.
3. เครื่องบด
4. ตะแกรงร่อน
5. เครื่องชั่งละเอียด
6. PVA ใช้เป็น binder
7. น้ำกลั่น
8. บีกเกอร์ ขนาด 200 ml.
9. เครื่องอัด (Hydraulic press)

10. แบบที่ใช้สำหรับอัดชิ้นตัวอย่าง
11. เตาอบ
12. วัสดุทนไฟสำหรับรองชิ้นตัวอย่าง
13. เตาเผา

การบดผสม

นำ Feldspar ที่บดแล้ว ร่อนผ่านตะแกรงเบอร์ 150 mesh ผสมกับสารเคมีตามตารางข้างล่างนี้

ตัวอย่าง	Feldspar	K_2CO_3	Na_2CO_3	CaCO3	Firing temperture (°C)
1	100	-	-	-	1250
2	98	2	-	-	
3	95	5	-	-	
4	90	10	-	-	
1	100	-	-	-	1250
2	98	-	2	-	
3	95	-	5	-	
4	90	-	10	-	
1	100	-	-	-	1250
2	98	-	-	2	
3	95	-	-	5	
4	90	-	-	10	
1	100	-	-	-	1250
2	98	-	1	1	
3	95	-	2.5	2.5	
4	90	-	5.0	5.0	
1	100	-	-	-	1250
2	98	1	1	-	
3	95	2.5	2.5	-	
4	90	5.0	5.0	-	
1	100	-	-	-	1250
2	98	1	-	1	
3	95	2.5	-	2.5	
4	90	5.0	-	5.0	

การอัดชิ้นตัวอย่าง

ชั่งส่วนผสมอย่างละ 10 กรัม ใส่ในกระจกนาฬิกาผสม binder (ความเข้มข้น 10% โดยน้ำหนัก) เพื่อช่วยให้สารเกาะติดกัน คลุกเคล้าให้เข้ากันแล้วเทลงในแบบอัด เส้นผ่านศูนย์กลางประมาณ 1.3 ซ.ม. อัดให้เป็นชิ้นตัวอย่างรูปทรงกระบอกสูง 1.3 ซ.ม.

การเผาชิ้นตัวอย่าง

อบชิ้นตัวอย่างให้แห้ง แล้วจัดวางบนแผ่นรองทนไฟให้แต่ละชิ้นอยู่ห่างกันประมาณ 3 ซ.ม. นำเข้าเตาเผาให้ความร้อนเพิ่มด้วยอัตรา 10°C /min จนถึงอุณหภูมิที่ต้องการ แล้วปิดเตา นำชิ้นตัวอย่างออกมาจากเตาเมื่อเตาเย็นลง

การตรวจสอบแท่งตัวอย่างหลังเผา

ศึกษาลักษณะของแต่ละตัวอย่าง เขียนรายงานลักษณะที่ปรากฏว่าอยู่ในสถานภาพใด รายงานผลการทดลองเปรียบเทียบอิทธิพลของการเติม Fluxing agent ลงใน Feldspar ธรรมชาติที่ผ่านการเผาตามอุณหภูมิที่กำหนด โดยพล็อตกราฟระหว่าง h/w กับปริมาณ Fluxing agent เมื่อ h คือความสูงของชิ้นตัวอย่าง และ w คือเส้นผ่านศูนย์กลางของแท่งตัวอย่าง

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Feldspar Test (ส่วนเพิ่ม)

จุดประสงค์ เพื่อให้เข้าใจความสัมพันธ์ของการหลอมและส่วนผสมทางเคมีของ feldspar

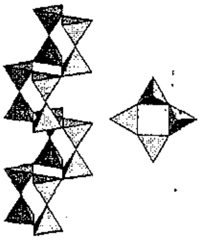
แร่ Feldspar มีหลายชนิด เช่น

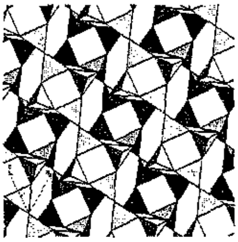
Albite $Na_2O.Al_2O_3.6SiO_2$

Orthoclase $K_2O.Al_2O_3.6SiO_2$

Anothite $CaO.Al_2O_3.6SiO_2$

เป็น Framework silicate O:Si = 2:1 ที่ Al^{3+} ion แทนที่ Si^{4+} ion แล้วสมดุลประจุโดยการดูดไอออนบวก เช่น Na^{1+}, K^{1+}, Ca^{2+} เข้าไป





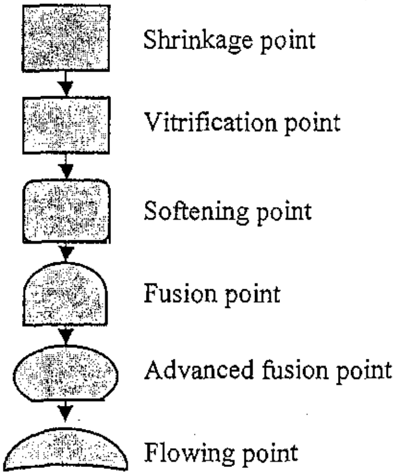
สูตรเคมี (Chemical formula) เขียนอีกแบบหนึ่งได้ว่า

$$K_2O.Al_2O_3.6SiO_2 \rightarrow 2K(AlSi_3O_8)$$

การนำไปใช้ (Application)

ทำหน้าที่เป็น fluxing agent ใน whiteware body และ glaze

เมื่อเพิ่มความร้อนให้กับ feldspar จะเกิดการเปลี่ยนแปลงต่างๆ เช่น หดตัว (shrinkage), สุกตัว (vitrification), อ่อนตัว (softening), หลอมตัว (fusion), ไหลตัว (flowing) ตามลำดับดังภาพต่อไปนี้



การทดลอง

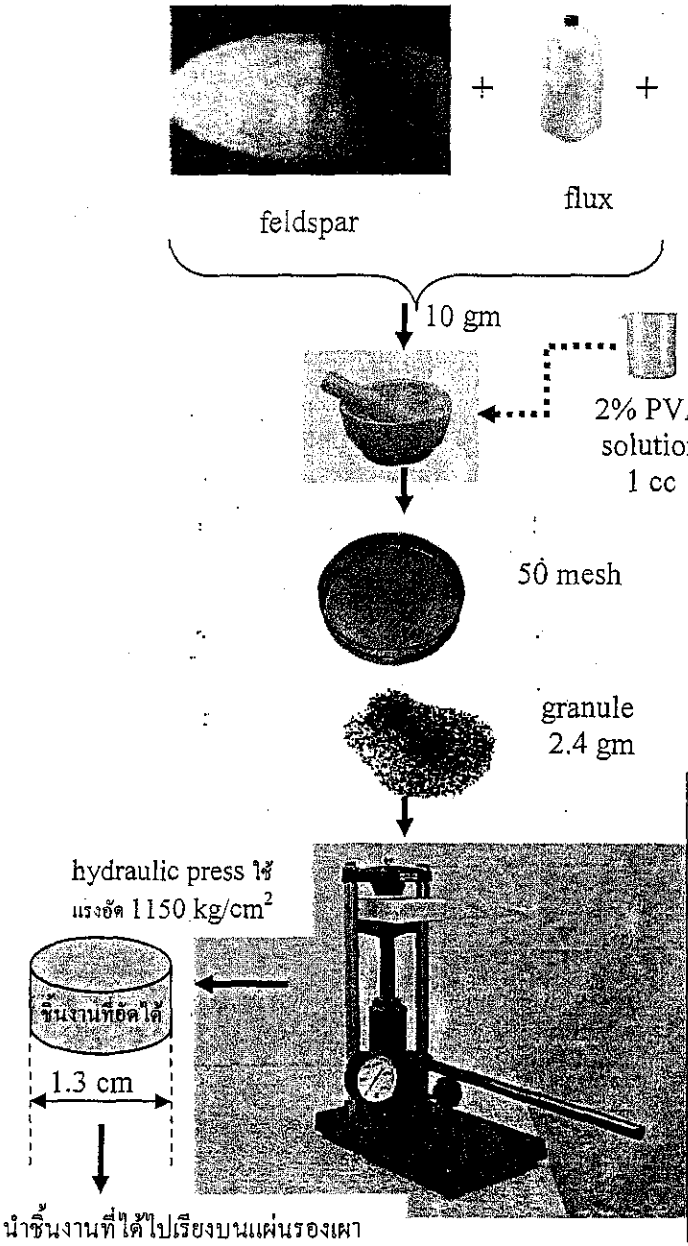
1) ผสม Feldspar + สารเคมีต่างๆ เช่น Na_2O, K_2O, CaO ฯลฯ ที่ทำขึ้นแทนการมี feldspar หลายๆ ชนิด
2) ร่อนผ่านตะแกรง
3) อัดชิ้นตัวอย่างด้วย Hydraulic press

4) เตรียมแผ่นรองเผา

5) เผาชิ้นตัวอย่าง

6) วัดความสูง : high (H) และความกว้าง : width (W) แล้วเขียนกราฟ H/W ต่อปริมาณสารเคมีที่เติม เพื่อเปรียบเทียบการหลอมตัวต่อ % flux เช่น Na_2O หรือ K_2O หรือ CaO

ส่วนเสริม Flow chart แสดงวิธีการทดลอง



วิธีเตรียมสารละลาย 2% PVA

1) ชั่งผง PVA มา 20 gm
2) นำน้ำกลั่นมา 100 cc
3) ต้มน้ำกลั่นที่ 80°C อย่าใช้สูงกว่านี้ เพราะจะทำให้ PVA เสื่อมคุณภาพ
4) ค่อยๆ ใส่ผง PVA ลงไปในน้ำร้อน พร้อมๆ กับคอยคนให้ PVA ละลาย เมื่อส่วนแรกละลายแล้วจึงเติมส่วนใหม่ลงไป จนใช่ผง PVA หมด
5) ทิ้งในสารละลายเย็นจึงนำไปใช้ ส่วนที่เหลือใส่ในขวดที่ ปิดฝาได้สนิท

ใช้ได้

ประมาณ 2-3 เดือน จะหมดอายุ

วิธีการคำนวณแรงอัดว่าต้องใช้แรงอัดกี่กิโลกรัม สมมุติว่า แม่แบบมี ∅ 1.3 cm และต้องใช้แรงอัด $P = 1150 kg/cm^2$

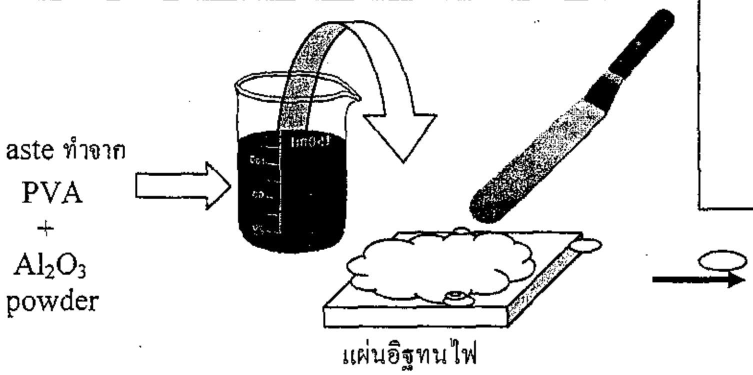
1) พ.ท. หน้าตัด $= \pi\left(\frac{D}{2}\right)^2 = \left(\frac{22}{7}\right)\left(\frac{1.3}{2}\right)^2$

2) $Pressure\left({}^{kg}\!/_{cm^2}\right) = \frac{Force\,(kg)}{area\,(cm^2)}$

$$Force(kg) = 1150\left({}^{kg}\!/_{cm^2}\right) \times area\,(cm$$

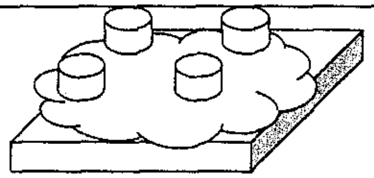
$$= 1527\ kg$$

ห้โรยผง Al_2O_3 หรือฉาบ Al_2O_3 paste ก่อนวางชิ้นงาน



<u>วิธีเตรียมแผ่นรองเผา</u>

1) ใช้ spatula ตัก paste ปาดให้เกิดความหนา ฉาบบนผิวของแผ่นวัสดุทนไฟอย่างสม่ำเสมอ
2) วางชิ้นงานที่อัดได้ห่างกันและห่างจากขอบ ประมาณ 3 cm.



เผาชิ้นตัวอย่างที่ 1250 °C โดยเพิ่มอุณหภูมิ 10 °C/นาที ยืนไฟ 15 นาที

ให้ส่งชิ้นตัวอย่างพร้อมกับรายงาน กลุ่มละ 1 ชุด

วิชา 426 203 CERAMIC RAW MATERIAL LABORATORY

การทดลองที่ 6
GREEN STRENGTH TEST

ผศ.ดร.สุขเกษม กังวานตระกูล

บทนำ:

การอบแห้งเป็นขั้นตอนสำคัญในขบวนการผลิตทางอุตสาหกรรมเซรามิก เนื่องจากในการขบวนการผลิตต้องเกี่ยวข้องกับน้ำ เช่น ในขั้นตอนการบด ในขั้นตอนการผสมวัตถุดิบชนิดต่างๆให้เข้ากันเป็นเนื้อดินปั้นและขั้นตอนต่อไปก็คือการขึ้นรูป การอบแห้งจึงเป็นขั้นตอนในการเตรียมวัสดุก่อนที่จะเข้าสู่ขบวนการต่อไป เช่น การถอดแม่แบบ และการอบชิ้นงานหลังจากการชุบเคลือบ ซึ่งจะเป็นขั้นตอนที่ต้องกระทำก่อนการเผา และยังเป็นขั้นตอนสุดท้ายของขบวนการผลิตบางอย่างเช่น การแต่งแร่ดินขาวและการผลิตผงสีเซรามิก เป็นต้น จะเห็นได้ว่าการอบมีส่วนร่วมในขบวนการผลิตตั้งแต่ขั้นการเตรียมวัตถุดิบจนกระทั่งได้มาซึ่งผลิตภัณฑ์เซรามิกที่ต้องการ การอบแห้งคือการกำจัดน้ำที่มีอยู่ในผลิตภัณฑ์โดยการระเหย (Evaporization) ออกไปก่อนนำไปเผา

โดยทั่วไปการอบแห้งสามารถทำได้ 2 วิธีคือ Mechanical method คือการใช้เครื่องอัดกรอง (Filter press) เพื่อไล่น้ำออก และอีกวิธีคือ Thermal vaporization โดยใช้เครื่องอบ (Dryer) ซึ่งเป็นวิธีที่ได้รับความนิยมมาก ในการกำจัดความชื้นซึ่งเนื่องมาจากน้ำหรือของเหลวอื่นๆที่มีอยู่ในชิ้นงานนั้นจะมี condition ของการอบแห้งที่แตกต่างกัน ทั้งนี้ขึ้นอยู่กับค่าจุดเดือด ความร้อนจำเพาะ ความร้อนแฝงของการกลายเป็นไอ ความดันไอ และน้ำหนักโมเลกุลของๆเหลวชนิดนั้นๆ แต่กลไกที่เกิดจากการอบแห้งนั้นจะคล้ายคลึงกัน ชิ้นงานเซรามิกที่ผ่านการอบแห้งจะมีความแข็งแรงมากกว่าตอนหลังจากที่ขึ้นรูปใหม่ๆ เรียกว่า Leather hard บางที่เรียกว่า Green body สิ่งที่มีผลต่ออัตราการอบแห้งได้แก่ อุณหภูมิ ความชื้นของอากาศ และความเร็วของอากาศที่อยู่รอบๆชิ้นงาน รวมทั้งพลังงานความร้อนที่ให้แก่ชิ้นงานขณะอบแห้งด้วย

กลไกการอบแห้งสามารถอธิบายได้ว่า การที่อนุภาคมาจับตัวรวมกันในขณะเปียก เพื่อขึ้นรูปจะมีชั้นของน้ำบางๆฉาบอยู่ที่ผิวของอนุภาค เพื่อยึดให้อนุภาคมาอยู่ใกล้กัน เมื่อให้ความร้อนเพื่อไล่ความชื้นหรือน้ำนี้ออกไป จะทำให้เกิดการหดตัวเนื่องจากน้ำที่มีอยู่ระเหยไป ทำให้ชิ้นงานลดขนาดลง การที่บริเวณผิวของชิ้นงานหรือผิวที่บางกว่ามีการระเหยของน้ำมากกว่าบริเวณภายในชิ้นงานหรือบริเวณที่มีความหนามากกว่าจะทำให้การหดตัวของบริเวณทั้งสองนี้ไม่เท่ากัน ซึ่งจะมีผลทำให้รูปร่างของชิ้นงานเกิดการบิดเบี้ยว โค้งงอ และเกิดรอยแตก (crack) เนื่องจากอัตราการอบแห้งเร็วเกินไป ถ้าอัตราการระเหยน้ำจากผิวของชิ้นงานมากกว่าอัตราการแพร่ของน้ำที่แพร่ผ่านรูพรุนที่อยู่ในเนื้อไปสู่ผิว

จะทำให้ที่ผิวของชิ้นงานแห้งเร็วเกินกว่าภายในชิ้นงาน สภาวะนี้เรียกว่า Hardening ซึ่งเกิดขึ้นเมื่อผิวด้านนอกหดตัว ในขณะที่ผิวด้านในยังคงไม่เปลี่ยนแปลง ทางแก้ไขคือ พยายามไม่ให้มีการระเหยของน้ำจากผิวของชิ้นงานเร็วเกินไป ทำได้โดยการให้ความร้อนแก่ชิ้นงานในบริเวณที่มีการรักษาให้มีความชื้น (Humidity) ค่อนข้างสูง การที่มีความชื้นของอากาศรอบๆชิ้นงานค่อนข้างสูงนี้จะทำให้อัตราการระเหยจากผิวของชิ้นงานต่ำลง ในกระบวนการอบแห้ง ถ้าให้อุณหภูมิค่อยๆสูงขึ้น และให้ความชื้นในอากาศค่อยๆลดลงก็จะได้การระเหยและอัตราการแพร่คงอยู่สูงเท่าๆกัน ทำให้ชิ้นงานที่แห้งมีการแห้งอย่างสม่ำเสมอทั่วชิ้นงาน ส่วนขั้นตอนสุดท้ายของการอบแห้งคือจะทำการอบที่อุณหภูมิสูงกว่า 100 °C และมีความชื้นต่ำมากๆ ก็จะได้ชิ้นงานที่ไม่มีการแตกร้าวเลย ในทางปฏิบัติอาจทำได้ง่ายๆโดยการเอาผ้าชุบน้ำคลุมที่ผิวของชิ้นงานเพื่อเพิ่มความชื้นรอบชิ้นงานและไม่ให้การระเหยที่ผิวเกิดขึ้นรวดเร็วเกินไป

ในการอบแห้งผลิตภัณฑ์เซรามิกในสภาพแวดล้อมที่คงที่ โดยอุณหภูมิและความชื้นในอากาศ ความเร็วลมใน Dryer ไม่เปลี่ยนแปลง สามารถแบ่งได้เป็น 3 ขั้นตอนคือ

1. ช่วงที่ 1 อัตราการระเหยคงที่ (Constant rate period) ช่วงนี้อัตราการระเหยจะไม่ขึ้นกับ moisture content
2. ช่วงที่ 2 อัตราการระเหยลดลงในช่วงแรก (First falling-rate period) อัตราการอบแห้งจะแปรตาม moisture content เป็นลักษณะเชิงเส้น
3. ช่วงที่ 3 อัตราการระเหยค่อยลดช้าลงในช่วงที่ 2 (Second falling-rate period) เส้น curve ความสัมพันธ์ระหว่างอัตราการอบแห้ง และ moisture content จะเป็นเส้นโค้ง

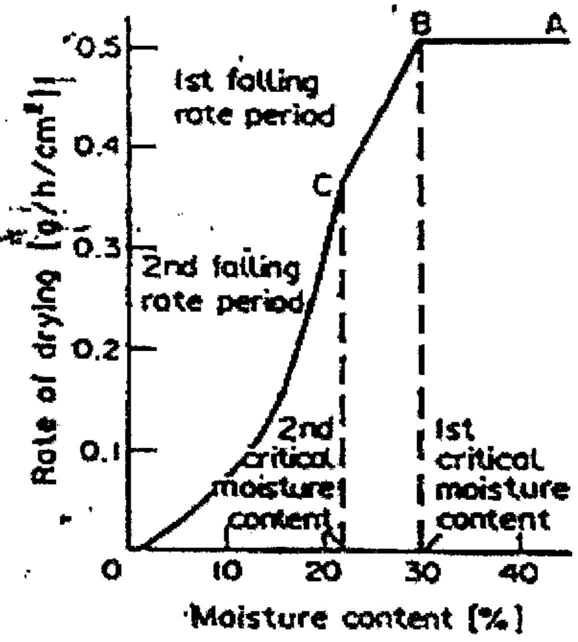


Fig.1 Drying curve for ceramics (courtesy Institute of Ceramics)

จากรูป 1 ถ้าเรา plot curve ระหว่างน้ำหนักที่เปลี่ยนไป และเวลาที่ใช้ในการอบ จะพบว่าช่วงแรกของการอบจะไม่ขึ้นกับความชื้นที่มีอยู่ (แสดงด้วยเส้น A-B) นั่นคืออัตราการระเหยจะคงที่ แต่เมื่อถึงจุดหนึ่งคือจุด B ซึ่งถือว่าเป็น First critical moisture content อัตราการอบแห้งจะเริ่มลดลงคงที่ (ตามเส้น B-C) จนกระทั่งในที่สุดเมื่อถึงจุดอีกหนึ่งจุดคือ จุด C เรียกว่า Second critical moisture content จะได้ curve ที่ลดลงแบบไม่เป็นเส้นตรงซึ่งเนื่องมาจากอัตราการระเหยจากผิวของชิ้นงานขึ้นกับอัตราการแพร่ของน้ำในชิ้นงาน

ในการอบแห้งโดยการให้ความร้อนจะใช้เตาอบในกรณีที่ต้องการอบผลิตภัณฑ์เป็นครั้งคราว ดังแสดงในรูปที่ 2 ซึ่งต้องมีพัดลมเป่าอากาศให้มีการหมุนเวียนไปรับความร้อนจากขดลวดความร้อนและให้มีการถ่ายเททั่วเตาอบแต่ในกรณีที่ต้องการอบผลิตภัณฑ์จำนวนมากและต่อเนื่องกัน จะใช้ Tunnel dryer ดังรูปที่ 3 ซึ่งจะเรียงผลิตภัณฑ์ใส่รถ และรถจะเคลื่อนที่เข้าไปผ่านบริเวณที่มีอุณหภูมิและความชื้นคงที่ และเมื่อผลิตภัณฑ์แห้งก็จะเข้าไปยัง Tunnel kiln เพื่อเผาผลิตภัณฑ์ต่อไป

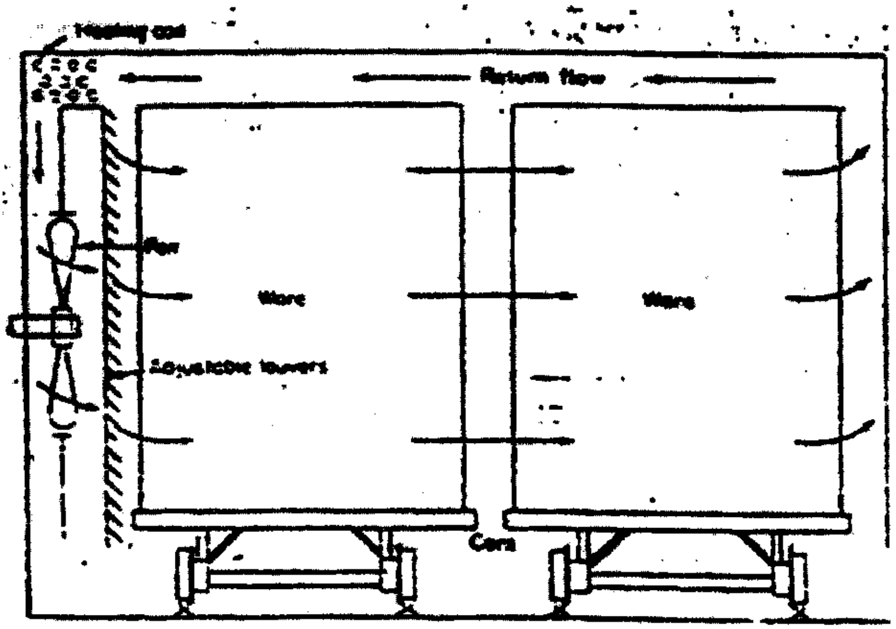


Fig.2 Fan-type humidity dryer.

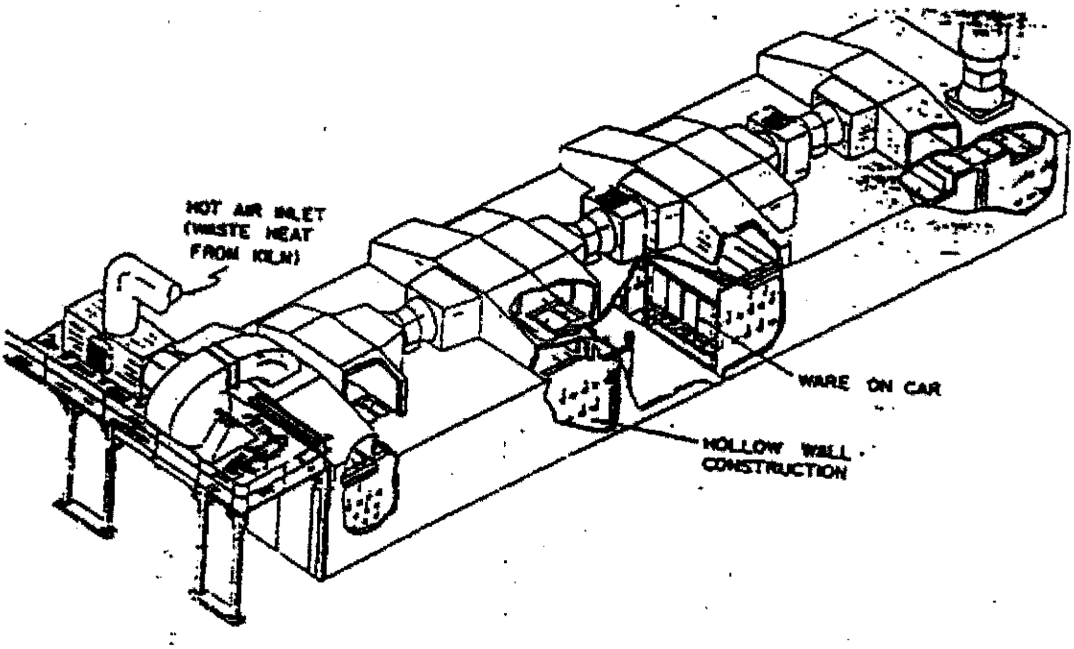


Fig.3 Tunnel dryer (courtesy Swindell-Dressler Co. Ltd.)

เนื่องจากความแข็งแรงของผลิตภัณฑ์ก่อนเผา (Green Strength) มีความสำคัญในการช่วยให้อัตราการแตกหักของผลิตภัณฑ์ที่จะนำไปเผาลดลง ดังนั้นในการทดลองนี้จะหาและวัดคุณสมบัติของดินที่ยังไม่ได้เผา ซึ่งในการทดลองนี้จะให้ศึกษาคุณสมบัติ 4 ประการคือ

1. % moisture content (Dry basis)
2. Drying shrinkage
3. Green density
4. Green strength

1. การคำนวณหา % moisture content

สิ่งที่เกิดขึ้นภายหลังจากการอบแห้งคือ moisture content ทำได้โดยชั่งน้ำหนักของชิ้นงานก่อนอบ แล้วนำไปอบที่ 105-110 °C แล้วนำค่าน้ำหนักที่ได้มาแทนในสูตร ซึ่งสามารถคิดได้ 2 แบบ คือ Dry weight basis และ Wet weight basis

$$\%\text{ moisture content (dry basis)} = \frac{(\text{weight before drying} - \text{weight after drying})}{\text{weight after drying}} \times 100$$

$$\%\ \text{moisture content (wet basis)} = \frac{\text{(weight before drying - weight after drying)}}{\text{weight before drying}} \times 100$$

2. การหา **Drying shrinkage (% linear drying shrinkage, base on dried length)**

ทำได้โดยการวัดขนาดของแท่งดินหลังจากขึ้นรูปแล้ว และวัดขนาดอีกครั้งหลังจากผ่านการอบแห้งแล้ว ตามสูตร

$$\%\ \text{Linear drying shrinkage} = \frac{\text{(length before drying - length after drying)}}{\text{length after drying}} \times 100$$

3. การหา **Green density (bulk density)**

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}}\ (\text{g/cm}^3)$$

4. การหา **Green strength**

ทำได้โดย นำชิ้นงานหลังอบมาวัด three-point bending วัดอย่างน้อย 10 ชิ้น โดยขึ้นรูปชิ้นงานที่มีพื้นที่หน้าตัดเป็นรูปสี่เหลี่ยม แล้วแทนค่าต่างๆ ในสูตร

$$\text{MOR} = \frac{3PL}{2bd^2} \quad \text{psi (lb/in}^2\text{), ksi (1000 lb/in}^2\text{), MPa}$$

เมื่อ P = load ที่ทำให้ชิ้นงานแตกหัก
L = Span (ระยะระหว่าง Supporter)
b = ความกว้างของ Specimens
d = ความสูงของ Specimens

หรือทำได้โดยขึ้นรูปชิ้นงานที่มีขนาดตาม Specification ของเครื่องวัด แล้วสามารถอ่านค่า strength ได้จาก scale ของเครื่องเลย

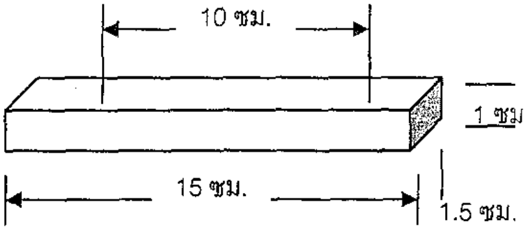
จุดประสงค์การทดลอง :

เพื่อให้นักศึกษาได้เรียนรู้วิธีการ และการวัดคุณสมบัติของชิ้นงานเซรามิกหลังจากอบแห้ง และสามารถบอกได้ว่าปัจจัยใดบ้างที่มีผลต่อขบวนการอบแห้งของวัตถุดิบเซรามิก

การทดลอง :

อุปกรณ์ในการทดลอง

1. วัตถุดิบที่นักศึกษาเลือก กลุ่มละ 1 ชนิด ที่เคยใช้ในการทดลองที่ 1
2. Roller ไม้สำหรับขึ้นรูป
3. โต๊ะสำหรับนวดดินที่มีความเรียบและได้ระนาบ
4. ตู้อบ (Dryer)
5. เครื่อง Bending strength tester
6. เครื่องชั่งที่มีความละเอียด 2 ตำแหน่ง
7. เวอร์เนีย คาร์ลิบเปอร์ (Verniers Caliper)



วิธีทำการทดลอง

- นำดินสำเร็จรูปมาขึ้นรูปให้เป็นแผ่นโดยการรีดด้วยมือและ roller ไม้ช่วยนวด
- ทำแท่งดินตัวอย่างขนาดกว้าง 1.5 ซ.ม. ยาว 15 ซ.ม. สูงหรือหนา 1.0 ซ.ม. จำนวน 20 แท่ง ตกแต่งให้เรียบร้อย (อาจทำมากกว่า 30 แท่ง เพื่อป้องกันชิ้นงานเสีย)

แท่งดินตัวอย่าง 30 แท่ง

Green Strength = 10 แท่ง

Fired Clay = 20 แท่ง

1000°C, 1 ชม. = 10

1100°C, 1 ชม. = 10

ทำไว้สำหรับการทดลองที่ 4

↓

วัดระยะ Mark ก่อนอบ ได้ Length before drying (L_b)

ชั่งน้ำหนักก่อนอบ ได้ Weight before drying (W_b)

↓

อบ 100 °C จนแห้ง

↓

วัดระยะ Mark หลังอบ ได้ Length after drying (L_a)

ชั่งน้ำหนักหลังอบ ได้ Weight after drying (W_a)

↓

นำมาทดลอง 3-point bending

บันทึกผล คำนวณหา % moisture content

Drying shrinkage

Green density

Green strength

และเขียนรายงานการทดลอง

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การทดลองที่ 7

PHYSICAL PROPERTIES OF FIRED CLAY PRODUCTS

ผศ.ดร.สุธรรม ศรีหล่มสัก

บทนำ

การหดตัวของดินเมื่อถูกเผา

เมื่อดินถูกความร้อนจะมีการหดตัว เรียกการหดตัวช่วงนี้ว่า “firing shrinkage” ซึ่งอาจมีค่าแตกต่างกันซึ่งขึ้นอยู่กับปัจจัยหลายอย่าง เช่น ความละเอียดของดิน ปริมาณของอินทรีย์สารในดิน วิธีการขึ้นรูปหรือทำชิ้นตัวอย่างและอุณหภูมิที่ใช้เผาดิน โดยทั่วไปดินที่เหนียวมากจะหดตัวมาก ทั้งขณะที่ทำให้แห้งและเผา มากกว่าดินที่เหนียวน้อย สารอินทรีย์ในดินมีส่วนทำให้เกิดการหดตัวในช่วงที่เผามากขึ้น การหดตัวของดินจะเพิ่มมากขึ้นเมื่ออุณหภูมิสูงขึ้น ในช่วงที่อุณหภูมิที่ดินยังไม่แตกตัวหรือหลอมละลาย ดินบางชนิดจะหดตัวอย่างรวดเร็วที่อุณหภูมิต่ำ แต่ดินบางชนิดจะหดตัวอย่างช้าๆ ดินที่สุกตัวที่อุณหภูมิสูงจะมีการหดตัวน้อยกว่าดินที่มีจุดสุกตัวที่อุณหภูมิต่ำ

การใช้ดินที่มี firing shrinkage สูงจะทำให้ผลิตภัณฑ์บิดเบี้ยว ยุบตัว และแตกง่าย เราจำเป็นต้องทราบค่าการหดตัวในการเผาเพื่อการกำหนดขนาดก่อนเผา ให้ได้ขนาดผลิตภัณฑ์ตามต้องการ เนื้อดินผสมเมื่อทำเป็นเป็นผลิตภัณฑ์ประเภทต่าง ๆ ก็ต้องมีการทดสอบค่านี้เช่นกัน

การวัดและการคำนวณ % firing shrinkage ทำได้หลายวิธี เช่นถ้าเทียบกับขนาดชิ้นตัวอย่างที่แห้งก็จะใช้สูตร

$$\text{\% Linear firing shrinkage} = \frac{(Dry\ length - Fired\ length)}{Dry\ length} \times 100$$

$$\text{\% Volume firing shrinkage} = \frac{(Dry\ volume - Fired\ volume)}{Dry\ volume} \times 100$$

แต่ถ้าคำนวณ % firing shrinkage เทียบกับขนาดของชิ้นตัวอย่างที่ยังไม่อบ (green) ก็จะใช้สูตร

$$\text{\% Linear shrinkage} = \frac{Green\ length - Fired\ length}{Green\ length} \text{ x } 100$$

$$\text{\% Volume shrinkage} = \frac{Green\ volume - Fired\ volume}{Green\ volume} \text{ x } 100$$

หมายเหตุ 1) ค่า % shrinkage แบบวิธีหลังค่าจะเป็นเปอร์เซ็นต์การหดตัวจากการอบและการเผารวมกัน จึงมักจะเรียกว่า % shrinkage มากกว่าเรียกว่า % firing shrinkage

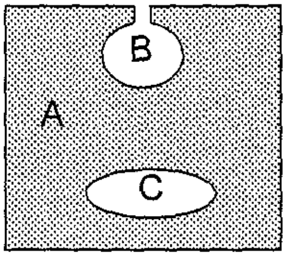
2) เราอาจจะคำนวณ volume shrinkage จาก linear shrinkage โดยใช้สูตร

$$\text{\% Volume shrinkage} = \left[1 - \left(1 - \frac{\%Linear\ firing\ shrinkage}{100}\right)^3\right] \times 100$$

พฤติกรรมของดินเมื่อผ่านความร้อน

เมื่อดินถูกเผาจะมีการเปลี่ยนแปลงหลายอย่างเช่น การเปลี่ยนแปลงสี, น้ำหนัก, ปริมาตร, ควา หนาแน่น, ความถ่วงจำเพาะ, ความพรุนตัว, การดูดซึมน้ำ, ความแข็ง, ความแข็งแรง ฯลฯ ในการทดลองนี้นักศึกษาจะได้ศึกษาพฤติกรรมการเปลี่ยนแปลง 1) ปริมาตร(volume), 2) ความพรุนตัว (porosity), 3) การดูดซึมน้ำ(water Absorption), 4) ความหนาแน่น (density), 5) ส่วนที่ไม่ซึมน้ำ (apparent specific gravity) และ 6) หนักที่หายไประหว่างเผา (weight loss) ของดินเมื่อผ่านความร้

1. ปริมาตร (Volume : V) เนื้อดินหลังจากเผาจะมีความพรุนตัว ดังแสดงในภาพที่ 1 จากภาพจะเห็นว่าเนื้อผลิตภัณฑ์หลังเผาประกอบด้วย ส่วนประกอบ 3 ส่วนคือ



1) เนื้อวัสดุจริง ๆ คือ ส่วนที่แรเงา A ในภาพ
2) รูพรุนชนิดมีรูเปิดที่ผิว (open pore) คือ B ในภาพ
3) รูพรุนชนิดไม่มีความต่อเนื่องที่ผิว (close pore) คือ C ในภา

ในเนื้อผลิตภัณฑ์จริงๆ จะมีรูพรุนที่กล่าวมาแล้วข้างต้นเป็นล้านๆ รู ฝังอยู่ใ เนื้อและที่ผิวของผลิตภัณฑ์ เราบอกปริมาตรของส่วนประกอบฯ ข้างต้นในรูปของ volume โดยนิยามดังนี้ :-

1) Pore volume (V_p) คือ volume ของ close pores + volume ของ open pore
$$V_p = V_{cp} + V_{op}$$
2) Bulk volume (V_b) คือ volume ของเนื้อวัสดุจริง (true volume) + pore volume
$$V_b = V_t + V_p = V_t + V_{op} + V_{cp}$$
3) Apparent volume (V_a) คือ volume ของเนื้อวัสดุจริง ๆ (V_t) + close pore
$$V_a = V_t + V_{cp} = V_b - V_{op}$$
4) Open pore volume
$$V_{op} = V_b - V_a = V_p - V_{cp}$$
5) Close pore volume
$$V_{cp} = V_b - V_t - V_{op} = V_a - V_t$$
6) True volume (V_t) คือ volume ของเนื้อวัสดุจริง ๆ
$$V_t = V_b - V_p = V_a - V_{cp}$$

โดยทั่วไปเรานิยมรายงานค่า V_b, V_a และ V_{op} เป็นหลัก ในทางปฏิบัติเราอาจจะหา V_b , V_a , V_{op} โดยการชั่งน้ำหนักชิ้นตัวอย่างในสภาพแห้ง (Dry weight, W_D), สภาพอิ่มตัวด้วยน้ำ (Saturated weight, W_S) และสภาพแขวนลอยในน้ำ (Suspended weight W_{SS}) แล้วนำน้ำหนักข้างต้นมาคำนวณหา V_b , V_{op} และ V_a จากสูตร

$$V_b = \frac{W_S - W_{SS}}{\rho_L}$$ เมื่อ ρ_L = density ของน้ำที่ suspended มีค่า ~1.00 g/cm^3

$$V_{op} = \frac{W_S - W_D}{\rho_L}$$

$$V_a = V_b - V_{op} = \frac{W_S - W_{SS}}{\rho_L} - \frac{W_S - W_D}{\rho_L} = \frac{W_D - W_{SS}}{\rho_L}$$

2. ความพรุนตัว (Porosity : P) จากข้างต้นจะเห็นว่าเราสามารถบอกปริมาณรูพรุนตัวของผลิตภัณฑ์หลังเผาในรูปของ open pore และ close pore อย่างไรก็ตาม โดยส่วนใหญ่แล้วนักเซรามิกมักจะรายงาน porosity ของผลิตภัณฑ์ในรูปของ apparent porosity โดย apparent porosity (P) คือ volume ของ open pore (V_{op}) ในแท่งตัวอย่างต่อ bulk volume

$$P = \frac{(W_S - W_D)}{V_b} \times 100$$

3. การดูดซึมน้ำ (Water Absorption : A) คือความสามารถในการดูดซึมน้ำของชิ้นตัวอย่าง ส่วนใหญ่เรามักจะบอกเทียบกับน้ำหนักของผลิตภัณฑ์ โดยรายงานในรูปของ % Water absorption (A)

โดย $$A = \frac{(W_S - W_D)}{W_D} \times 100$$

F.H. Norton จำแนกผลิตภัณฑ์เซรามิกตาม % Water absorption ดังแสดงในตารางที่ 1.1

4. ความหนาแน่น (Density : D) คือ ความหนาแน่น มีสูตรว่า $D = \frac{M}{V}$ เมื่อ M คือ mass และ V คือ volume มีหน่วยเป็น g/cm^3 หรือ kg/m^3 Density ที่นักเซรามิกนิยมใช้ในการรายงานคุณสมบัติของผลิตภัณฑ์หลังเผาคือ

1) Bulk density (ρ_b) คือ density หาได้จาก dry weight ÷ bulk volume

$$\rho_b = \frac{W_D}{V_b} = \frac{W_D \rho_L}{(W_S - W_{SS})}$$

Bulk density เป็น density ชนิดที่ใช้กันมากที่สุดโดยทั่วไป ถ้าบอกว่า density เฉย ๆ โดยไม่บอกว่าเป็น density อะไร มักถือว่าเป็น density ชนิดนี้

2) True density (ρ_t) คือ density ของชิ้นตัวอย่างที่มี porosity = 0

$$\rho_t = \frac{W_D}{V_t}$$

3) Apparent density คือ density ที่ได้จาก dry weight ÷ apparent volume

$$\rho_a = \frac{W_D}{V_a} = \frac{W_D \rho_L}{(W_D - W_{SS})}$$

4) %Theoretical density เป็นค่าที่ใช้เปรียบเทียบ bulk density ของวัสดุที่สนใจเทียบกับ theoretical density ของวัสดุ

$$\% \text{ Theoretical density } = \frac{\rho_b}{\rho_t} \times 100$$

5. ส่วนที่ไม่ซึมน้ำ (Apparent specific gravity : T) คือเปอร์เซ็นต์ส่วนของวัสดุที่น้ำซึมผ่านไม่ได้ หาได้จาก

$$T = \frac{W_D}{W_D - W_{SS}} \times 100$$

6. **น้ำหนักที่หายไประหว่างเผา (% Weight loss)** หาได้จากน้ำหนักชิ้นที่หายไประหว่างเผาเทียบ
น้ำหนักอบแห้งก่อนเผา (oven dry weight)

$$\%\ \text{Weight Loss} = \frac{(W_{OD} - W_F)}{W_{OD}} \times 100$$

เมื่อ W_{OD} คือน้ำหนักของชิ้นตัวอย่างหลังอบแห้งก่อนเผา และ W_F คือน้ำหนักของชิ้นตัวอย่างหลังเ
(fired weight)

Table 1.1 Classification of Fine Ceramics

Class and type	Absorption %	Products
Earthenware:		
Natural	+15	Tableware, artware, tiles
Fine	10-15	Tableware, artware, kitchenware, tiles
Talc	10-20	Artware, tiles, ovenware
Semivitreous	4-9	Tableware, artware
Stoneware:		
Natural	0-5	Drain pipe, kitchenware, artware, tiles
Fine	0-5	Cookware, artware, tableware
Technical vitreous	0-0.2	Chemical ware
Jasper	0-1	Artware
Basalt	0-1	Artware
China:		
Hotel	0.1-0.3	Tableware, heavy
Household	0.0-0.2	Tableware
Bone	0.3-2.0	Tableware, artware
Frit	0.0-0.5	Tableware, artware
High strength	0.0-1.0	Tableware
Low expansion	1.0-5.0	Ovenware, stoveware
Porcelain:		
Hard	0-0.5	Tableware, artware
Technical vitreous	0-0.2	Chemicalware, ball mill balls and linings
Triaxial electrical	0-0.2	Low-frequency insulators
High-strength electrical	0-0.2	Low-frequency insulators
Alumina	0-0.1	Spark plug cores, valve seats, cutting tools, substrates, abrasion-resisting ware
Steatite	0-0.5	High-frequency insulators, low-loss dielectric
Ferrites	0-0.05	High K-dielectrics constant
Ferromagetics	0-0.05	Permanent magnets, transformer cores, memory units, antennas
Vitreous plumbing fixtures	0.1-0.3	Lavatories, closet bowls, flush tanks, urinals
Dental	0-0.1	Dentures
Parian	0-0.5	Sculpture
Refractory	0-10	pyrometer tubes, combustion boats, furnace parts, nuclear fuel elements

จุดประสงค์การทดลอง :

เพื่อนักศึกษาได้เห็นการเปลี่ยนแปลงของลักษณะดินเมื่อผ่านการเผาที่อุณหภูมิต่าง ๆ และสามารถตรวจสอบสมบัติทางกายภาพที่เปลี่ยนไป เช่น สี การเปลี่ยนขนาด ความหนาแน่น และความพรุนตัว

การทดลอง

อุปกรณ์

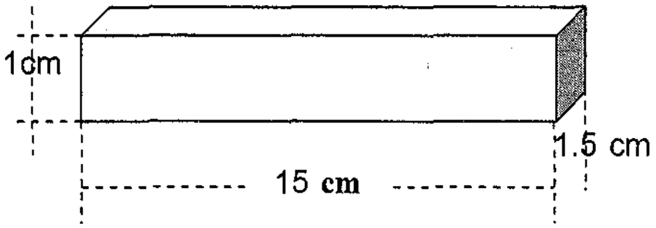
1) เครื่องชั่งอย่างหยาบ ± 0.5 g ชนิดชั่งได้สูงสุด 10 กก 1 เครื่อง ชนิดชั่งแบบ submerged ได้ พร้อมตระแกรงใส่วัสดุ
2) Vernier caliper
3) หม้อต้มน้ำ พร้อมเตา gas และอุปกรณ์
4) มีด และ spatula ตัดดิน กลุ่มละ 1 ด้าม
5) ไม้บรรทัดเหล็กความยาว กลุ่มละ 1 อัน
6) เตาอบไฟฟ้า เปิดเตาไว้ที่ 100°C 2 เตา
7) เนื้อดินปั้นชนิดต่าง ๆ ที่มีความชื้นพอเหมาะที่จะขึ้นเป็นแท่ง ๆ ตามภาพที่ 2 กลุ่มๆ ละ 1-2 กก. (เช่น Compound Clay, ดินด่านเกวียน ฯลฯ)
8) เตาเผา
9) Desiccators
10) ถาดอลูมิเนียม
11) ผ้าซับน้ำ และ ฟองน้ำเช็ดโต๊ะ
12) ชิ้นตัวอย่าง Ceramic ชนิดต่าง ๆ
    1) กระเบื้องปูพื้น 8" x 8"
    2) กระเบื้องผนัง 4" x 4" หรือ 8" x 8"
    3) กระเบื้องโมเสด
    4) กระเบื้อง mosaic
    5) อิฐ บ.ป.ก.
    6) อิฐมอญ
    7) กระเบื้องดินเผาด่านเกวียน
    8) โอ่งราชบุรี
    9) ลูกถ้วยไฟฟ้า
    10) ถ้วยกาแฟ
    11) จาน
    12) อื่นๆ (ถ้ามี)

วิธีการทดลอง

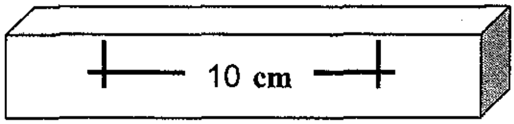
ตอน ก) การหดตัวหลังเผา

1) ทำชิ้นตัวอย่างเป็นแท่งรูปเหลี่ยม ดังภาพที่ 2 จำนวน 20 แท่ง
2) นำชิ้นตัวอย่างอบจนแห้งสนิท (หากชิ้นตัวอย่างแห้งสนิทแล้วน้ำหนักชิ้นตัวอย่างจะไม่ลดลงอีกต่อไปไม่ว่าจะอบต่อไปอีกกี่ชั่วโมง)บันทึกน้ำหนักชิ้นตัวอย่างที่แห้งสนิท (W_{OD}) เก็บเอาไว้ใช้ในข้อ 6 ต่อไป

3) ทำเครื่องหมายระยะ 10 ซม. dry length บนชิ้นตัวอย่างตามภาพที่ 3
4) นำชิ้นตัวอย่างไปเผาที่อุณหภูมิ 1000 และ 1100 ^{O}C อุณหภูมิละ 10 ชิ้นตัวอย่าง ใช้ heating rate 10°C/นาที และเมื่อถึงอุณหภูมิที่ตั้งไว้แล้วให้ off ไฟฟ้าทันที ทิ้งไว้จนชิ้นตัวอย่างเย็นตัวเท่าอุณหภูมิห้อง
5) นำชิ้นตัวอย่างออกมาจากเตาไปทิ้งให้เย็นใน desiccator
6) เมื่อชิ้นตัวอย่างเย็นแล้ววัดความยาวระยะที่บริเวณซึ่งทำเครื่องหมายไว้ตอนแรกได้เป็น fired length แล้วชั่งน้ำหนักชิ้นตัวอย่างหลังเผาบันทึกค่าไว้เป็น fired weight (W_F) คำนวณ % weight loss
7) คำนวณ % Linear firing shrinkage และ % Volume firing shrinkage



ภาพที่ 2 แสดงขนาดของชิ้นตัวอย่างที่เตรียมขึ้น



ภาพที่ 3 เครื่องหมายระยะ 6 ซม. dry length บนชิ้นตัวอย่างหลังจากอบแห้งแล้ว

ตอน ข) หา Volume, Porosity, Water Absorption, Density และ Apparent specific gravity

ตอน ข.1 ของวัสดุที่ทำขึ้นมาเอง

1) นำชิ้นตัวอย่างจากข้อ 6 ตอน ก) ไปต้มในน้ำเดือด 2 ชั่วโมง ปิดฝาด้วย (ประหยัดพลังงาน) ต้องคอยดูแลและเติมน้ำให้น้ำเดือดท่วมชิ้นตัวอย่างตลอดเวลาที่ต้ม
2) ครบ 2 ชั่วโมง แล้วยกภาชนะต้มออกจากเตาทิ้งไว้ให้เย็น 24 ชั่วโมง (ตาม ASTM ให้ต้ม 5 ชั่วโมง แต่ในการทดลองนี้ให้ต้มเพียง 2 ชั่วโมงจะได้ไม่กลับบ้านเย็น)
3) นำชิ้นตัวอย่างไปชั่งแบบแขวนลอยในน้ำได้เป็น suspended Weight (W_{SS})
4) นำชิ้นตัวอย่างออกมาเอาผ้าเช็ดน้ำที่ไหลเยิ้มออก (เฉพาะที่ไหลเยิ้ม อย่าเช็ดน้ำที่ซึมอยู่ตาม open pore ที่ผิวออกไปด้วย) แล้วชั่งน้ำหนักได้เป็น saturated weight (W_S)
5) นำชิ้นตัวอย่างไปอบจนแห้งสนิทแล้วปล่อยให้เย็นใน desiccator และชั่งน้ำหนักได้ dry

วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 8
REFRACTORY RAW MATERIALS

อาจารย์ ดร. วีระยุทธ์ ลอประยูร

จุดประสงค์

เพื่อให้นักศึกษาทำความรู้จักและคุ้นเคยกับวัตถุดิบหลักๆ ที่ใช้ในการผลิตวัสดุทนไฟ และสามารถแยกแยะความแตกต่างเบื้องต้นได้

การทดลอง

1. ตรวจดูลักษณะและข้อแตกต่างโดยทั่วไปของวัตถุทนไฟต่อไปนี้
2. ตรวจดูด้วยตา และเครื่อง Stereo-microscope
3. ทดลองผสมน้ำ ดูความเหนียว การแข็งตัว ฯลฯ

Raw Materials for Fireclay
Refractories
  a. Refractory clay
  b. Chamotte (Calcined Clay Aggregate)

Raw Materials for high alumina refractories
  a. Bauxite
  b. Calcined Alumina
  c. Andalusite

Raw Materials for Basic Refractories
  a. Calcined Natural Magnesite
  b. Sea Water Magnesia Clinker

Special Raw Materials
  a. Silicon Carbide
  b. Spray Dry Granules (ส่วนผสมวัตถุดิบซึ่งเตรียมเป็นเม็ดโดยเครื่อง Spray dry)

วิธีการใช้กล้อง Stereomicroscope (ดูรูปประกอบ)

1. ปรับระยะห่างระหว่างเลนส์ทั้งสอง (7) ให้เหมาะสมกับระยะห่างระหว่างตาของผู้ใช้
2. ปรับเครื่องหมาย " O" ที่เลนส์ตาด้านที่ไม่มีกากบาทใส่ที่จุดขาว (2) และปรับเครื่องหมาย " O" ที่เลนส์ตาด้านที่มรกากบาทใส่ที่จุดแดง (3)

weight (W_D)

6) นำค่า W_D , W_S และ W_{SS} ที่ได้ไปคำนวณหาค่า
    a) Bulk volume
    b) Open pore volume
    c) Apparent volume
    d) Apparent porosity
    e) Water absorption
    f) Bulk density
    g) Apparent density
    h) Apparent specific gravity

เขียนกราฟระหว่างคุณสมบัติที่หาได้ในตอน ก) และ ข) กับอุณหภูมิ แล้วตอบว่าคุณสมบัติเหล่านี้มีการเปลี่ยนแปลงกับอุณหภูมิอย่างไร? เพราะอะไรจึงเป็นเช่นนั้น?

ตอน ข.2 *ของวัสดุที่นำมาจากภายนอก*

1) นำชิ้นตัวอย่างของวัสดุที่ทำมาจากภายนอกไปต้มน้ำ ทิ้งไว้ให้เย็นแล้วชั่งน้ำหนัก W_{SS}, W_S แล้วอบจนแห้งสนิทและชั่งน้ำหนัก W_D ด้วยวิธีเดียวกันที่ทำในตอน ข.1 จากนั้นคำนวณคุณสมบัติในข้อ 6 ตอน ข. 1
2) เปรียบเทียบ bulk density ของวัสดุที่จัดให้กับตารางที่ 1.1 และระบุว่าวัสดุที่จัดให้เป็นผลิตภัณฑ์เซรามิกประเภทใด earthenware, stoneware, china, porcelain, หรืออื่นๆ

เอกสารอ้างอิง

1) รศ.ดร.จรัสศรี ลอประยูร. “Physical Properties of Fired clay Products,” ใน คู่มือ Lab วิชา Ceramic Raw Material Lab Experiment IV, มหาวิทยาลัยเทคโนโลยีสุรนารี, 2540.
2) J.T. Jones, M.F. Berard, Ceramics Industrial Processing and Testing. 2nd Edition, IOWA State University Press, Ames, 1993.
3) F.H. Norton, Fire Ceramic. McGraw-Hill, New York. 1970.
4) ASTM: C326-82 (Reapproved 1997) Standard Test Method for Drying and Firing Shrinkage of Ceramic Whiteware Clays.
5) ASTM: C373-88 (Reapproved 1994) Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products.

วิชา 426203 CERAMIC RAW MATERIALS LABORATORY

การทดลองที่ 8
REFRACTORY RAW MATERIALS

อาจารย์ ดร. วีระยุทธ์ ลอประยูร

จุดประสงค์

เพื่อให้นักศึกษาทำความรู้จักและคุ้นเคยกับวัตถุดิบหลักๆ ที่ใช้ในการผลิตวัสดุทนไฟ และสามารถแยกแยะความแตกต่างเบื้องต้นได้

การทดลอง

1. ตรวจดูลักษณะและข้อแตกต่างโดยทั่วไปของวัตถุทนไฟต่อไปนี้
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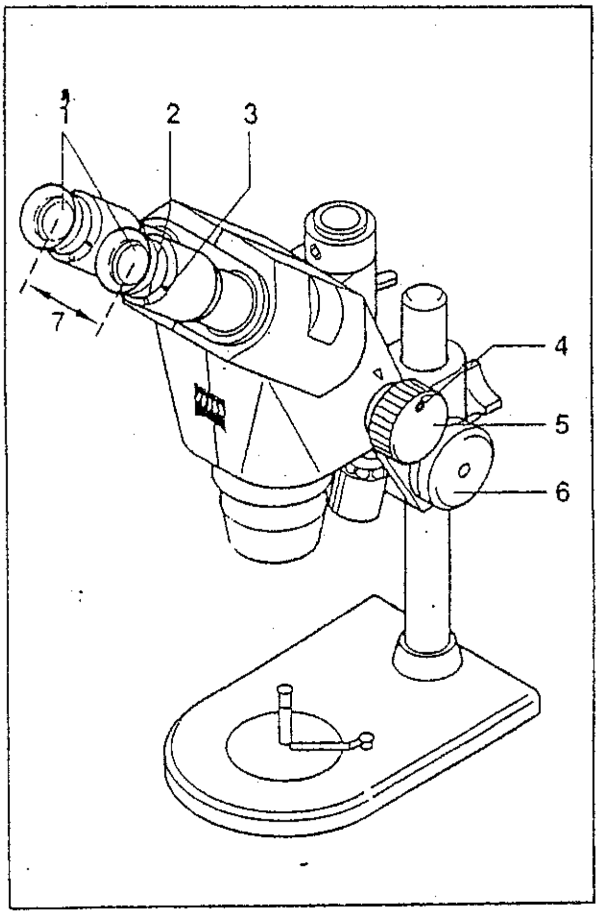
วิธีการใช้กล้อง Stereomicroscope (ดูรูปประกอบ)

1. ปรับระยะห่างระหว่างเลนส์ทั้งสอง (7) ให้เหมาะสมกับระยะห่างระหว่างตาของผู้ใช้
2. ปรับเครื่องหมาย " O" ที่เลนส์ตาด้านที่ไม่มีกากบาทใส่ที่จุดขาว (2) และปรับเครื่องหมาย " O" ที่เลนส์ตาด้านที่มรกากบาทใส่ที่จุดแดง (3)

3. วาง sample เข้าที่.
4. ตั้งกำลังขยายเลนส์ objective (5) สูงสุดแล้วปรับ zoom (6) ให้ได้ภาพคมชัดที่สุด
5. ตั้งกำลังขยายเลนส์ objective (5) ต่ำสุดแล้วปรับ zoom (6) ให้ได้ภาพคมชัดที่สุด [ถ้าจำเป็นให้ปรับ focus ที่เลนส์ตาอีกที (ทำ error correction)]
6. ขยับ sample ไปทางซ้าย-ขวา เพื่อดูรายละเอียดในตัวอย่าง



รูป 1 ส่วนประกอบของ Stereomicroscope

วิธีการบำรุงรักษากล้อง microscope

1. เมื่อใช้งานเสร็จแล้ว เช็ดด้วยผ้าแห้ง (อย่าเช็ดที่เลนส์) คลุมด้วยผ้าคลุมกันฝุ่น
2. อย่าเก็บกล้องไว้ในห้องที่มีความชื้นมากๆ เพราะเลนส์ด้านในจะเป็นรา
3. ปิดฝาช่องเปิดทุกช่องด้วยฝาปิด dust cap ที่บริษัทผู้ผลิตกล้องให้มา
4. ถ้ามีฝุ่นติดที่ Optical surface ให้กำจัดด้วยลูกยางเป่าฝุ่นหรือแปรงพิเศษ ทำความสะอาดแปรงนี้ด้วยแอลกอฮอล์ แล้วปล่อยให้แห้ง แต่ถ้ามีรอยนิ้วมือหรือเศษสกปรกที่ลูกยางหรือแปรงให้ใช้ผ้านุ่มๆที่สะอาดและไม่มีขนมาเช็ด (เป็นผ้าที่ใช้ทำความสะอาดเลนส์โดยเฉพาะ) ถ้าจำเป็นจริงๆให้เอาผ้าชุบ Petroleum ether เช็ดได้ แต่อย่าชุบ alcohol

5. เมื่อใช้เสร็จให้เก็บกล้องพร้อมทั้งอุปกรณ์ประกอบไว้ในที่แห้ง มีแสงส่องถึง อากาศถ่ายได้ ไม่มีฝุ่น ถ้าต้องเก็บนานๆ ควรมีเม็ดดูดความชื้นที่ชุบน้ำยาฆ่าเชื้อราเก็บไว้ด้วยกัน

การเขียนรายงาน

วัตถุประสงค์ของรายงาน เพื่อให้นักศึกษารู้จักสิ่งที่แตกต่างในวัตถุดิบชนิดต่างๆ ถ้าพบเห็นวัตถุดิบเหล่านี้ในโรงงานวัสดุทนไฟโดยไม่ทราบว่าเป็นวัสดุใด

ฉะนั้นในรายงานควรบอกถึงข้อแตกต่างของวัตถุดิบแต่ละชนิดเท่าที่สังเกตเห็นโดยละเอียด เช่น สี รูปร่างลักษณะ Plasticity น้ำหนัก ความอ่อนแข็ง ฯลฯ รวมถึงค้นคว้าจากตำราและบทความด้วยว่า วัตถุดิบแต่ละชนิดมีข้อแตกต่าง ที่สำคัญอย่างไร และจะทดสอบได้ด้วยวิธีใด ถ้านักศึกษาสามารถออกแบบตารางเพื่อดูความแตกต่างขั้นต้นของวัตถุดิบแต่ละชนิดได้ง่ายๆ ก็จะเป็นประโยชน์ต่อนักศึกษาเองต่อไปในอนาคต

	Refractory clay	Chamote	Bauxite	Cal. Alumina	Nature MgO	Sea Water MgO	High alumina cement		SiC	Zircon sand
สูตรเคมี							FONDU	CA25R		
สี										
ลักษณะทางกายภาพ										
โครงสร้าง										
Density										
ความทนไฟ										
ปฏิกิริยากับน้ำ										
ความแข็ง										
Stereo Microscope										

การเขียนรายงานการทดลอง (Lab Report Writing)

ผศ.ดร. สุธรรม ศรีหล่มสัก

วิศวกรและนักวิทยาศาสตร์ ที่จะประสบความสำเร็จในการทำงานต้องมีความสามารถในการสื่อสารได้ดี (Good communication) การสื่อสารทั่วไปต้องทำได้หลายวิธีเช่น สื่อสารโดยการพูด (verbal หรือ oral) และสื่อสารโดยการเขียนรายงาน (report writing)

ในการเรียนวิชาปฏิบัติการ (Laboratory) ในระดับมหาวิทยาลัย นอกจากจะได้เรียนรู้เทคโนโลยีในสาขาวิชาที่เรียนแล้ว นักศึกษายังจะได้รับความรู้และฝึกฝนความสามารถในการเขียนรายงาน (report writing) ในการเขียนรายงานการทดลอง (Lab report writing) ของนักศึกษาเอง จึงสมควรอย่างยิ่งที่นักศึกษาได้ให้ความสนใจในการเขียนรายงานการทดลอง เพื่อจะได้จบออกมาเป็นวิศวกร หรือนักวิทยาศาสตร์ที่ประสบความสำเร็จในอนาคตต่อไป รายงานทางวิทยาศาสตร์ที่ดีจะทำให้ผู้อ่านคล้อยตามและเข้าใจสิ่งที่ผู้เขียนต้องการถ่ายทอดให้ได้อย่างรวดเร็ว จึงสมควรอย่างยิ่งที่จะต้องใช้ความวิริยะ อุตสาหะ ในการเขียนรายงาน ไม่เขียนอย่างลวก ๆ เพราะจะทำให้ผู้อ่าน อ่าน แล้วไม่รู้เรื่อง แล้วยังทำให้ผู้อ่านเกิดความรู้สึกไม่ดีต่อผู้เขียนอีกด้วย

โครงร่างรายงานคร่าว ๆ ที่จะกล่าวต่อไปนี้เป็นแนวทางในการเขียนรายงานการทดลอง (Lab report) สำหรับส่งให้คณาจารย์ของสาขาวิชา การให้คะแนนของคณาจารย์จะให้ตามแนวให้คะแนนที่แนบอยู่ตอนท้าย นักศึกษาจะต้องศึกษาและทำความเข้าใจอย่างดีเพื่อจะได้เขียน Lab report ให้ถูกต้องตามแนวทางนี้ เพื่อจะได้คะแนนรายงานการทดลอง (Lab report) สูงสุดต่อไป

โครงร่างของรายงานการทดลอง (Outline for Lab Report)

รายงานการทดลองประกอบด้วยส่วนประกอบทั้งหมด 7 ส่วนคือ

1. ปกรายงาน (Title page)
2. บทคัดย่อ (Abstract)
3. สารบัญ (Table of contents)
4. คำนำ (Introduction)
5. อุปกรณ์และวิธีการทดลอง (Equipment & Procedure)
6. ผลการทดลอง (Result) และบทวิเคราะห์ (Discussion)
7. สรุปผลการทดลองและข้อเสนอแนะ (Conclusions and Recommendation)
8. เอกสารอ้างอิง (Reference)
9. ภาคผนวก (Appendices)

ส่วนที่ 3-6 นี้รวมกันเป็นส่วนหลักของรายงาน(Main Body)

ในแต่ละส่วนมีรายละเอียดดังนี้

1. **ปกรายงาน (Title Page)** ควรมีรายละเอียดของสถานศึกษา, ลำดับที่ของการทดลอง, รหัสวิชา, วันที่ทดลอง, หัวข้อเรื่องที่ทดลอง (Title) , บทคัดย่อ (Abstract), กลุ่มนักศึกษาที่ทดลอง, ชื่อและเลขประจำตัวนักศึกษา ผู้เขียนรายงานการทดลองและเพื่อนที่เป็น partner lab, วันส่งรายงานทดลองและชื่ออาจารย์ผู้สอนการทดลอง ตามตัวอย่างในหน้าที่ 4)

ปกรายงานให้ใช้กระดาษสีพื้นอ่อน ๆ เช่น ขาว หรือ เขียวอ่อน เหลืองอ่อน ฯลฯ ไม่จำเป็นต้องใช้ปกแข็ง (เปลืองเงิน) *ใช้กระดาษสีขาว A4 ธรรมดาก็ได้*

2. **บทคัดย่อ (Abstract)** เป็นส่วนที่พูดถึงการทดลองอย่างย่อที่สุดเพื่อให้ผู้อ่านที่ไม่มีเวลาอ่านในรายละเอียดทั้งหมดของรายงาน ได้อ่านในส่วนนี้แล้วจะได้ทราบได้ว่า การทดลองนี้*1)ทดลองอะไร 2) ทดลองอย่างไร 3)ได้ผลอย่างไร และ 4)สรุปว่าอะไร* จะต้องมีความยาวประมาณ 10-15 บรรทัด ไม่เกินครึ่งหน้ากระดาษพอดี ทั้งยังบรรจุในตอนกลางของหน้าปกรายงาน

3. **สารบัญ (Table of Contents)** เพื่อแสดงหัวข้อของรายงาน พร้อมแสดงเลขหน้า

4. **คำนำ (Introduction)** เป็นส่วนที่ใช้บอกวัตถุประสงค์ (objective or statement of purpose) ของการทดลอง พูดถึงทฤษฎี (Theory) จัดว่าส่วนนี้เป็น Literature survey ซึ่งจะพูดถึงสูตร ทฤษฎี และการทดลองของคนอื่น ๆ ที่มีก่อนหน้าที่เกี่ยวข้องกับการทดลองที่ทำครั้งนี้

นักศึกษาจะคัดลอกบางส่วนของ Lab Instruction มาก็ได้แต่ไม่ให้คัดลอกทั้งหมดมาจาก Lab Instruction แต่ควรจะค้นคว้าเพิ่มเติมจากหนังสือหรือแหล่งข้อมูลอื่นมาเพิ่มเติมด้วย

5. อุปกรณ์และวิธีการทดลอง (Equipment & procedure) ประกอบด้วย 2 ส่วนคือ *ส่วนที่พูดถึงอุปกรณ์ (Equipment)* กับ *ส่วนที่พูดถึงวิธีการทดลอง (Procedure)*

ส่วนที่พูดถึงอุปกรณ์ (Equipment) ที่ใช้ในการทดลอง ถ้าใช้เครื่องมือที่ไม่ well known จะต้องอธิบาย พร้อมทั้งมีภาพคร่าวๆ แสดงถึงเครื่องมือ มีคำอธิบายประกอบชัดเจนอาจจะต้องโยงลูกศร บอกว่าส่วนไหนเรียกว่าอะไร

ส่วนที่พูดถึงวิธีการทดลอง Procedures ควรจะ*เขียนขึ้นมาเองตามวิธีการทดลองที่นักศึกษาทำจริง ๆ ในการทดลอง* **ไม่ใช่ลอก Lab Instruction มา**

ในการเขียนวิธีการทดลอง นักศึกษาต้องมีความละเอียดละออในการเขียน โดยเฉพาะอย่างยิ่งการบอกค่าต่าง ๆ เป็นตัวเลขที่ใช้ในการปรับตั้งเครื่องมือเป็นสิ่งที่วิศวกรในอนาคตต้องให้ความสำคัญ เช่น บอกว่าตั้งแรงดันเครื่อง Filter press ไว้ที่ 80 ปอนด์ ***แบบนี้ผิด*** ที่ถูกจะต้องบอกให้ชัดเจนว่าตั้งแรงดันไว้ที่ 80 ปอนด์ต่อตารางนิ้ว

6. ผลการทดลองและบทวิจารณ์ (Result & Discussion)

ประกอบด้วย 2 ส่วนย่อยคือ (1) ผลการทดลอง (Result) และ (2) บทวิจารณ์ (Discussion)

6.1) ส่วนผลการทดลอง เป็นส่วนแสดงผลการทดลองที่ได้วิศวกรส่วนใหญ่มักจะนิยมแสดงผลการทดลองในรูปของกราฟมากกว่าในรูปของตาราง ยกเว้นแต่ว่าตารางมีความสำคัญในตัวของมันเอง รายละเอียด หลักการในการเขียนกราฟ และตารางในรายงานหรือจะพูดถึงอีกในตอนต่อไป

ในส่วนนี้ถ้าต้องมีการคำนวณข้อมูลมาประกอบด้วย จะต้องแสดงสมการและตัวอย่างการคำนวณประกอบด้วย ข้อมูลละ 1 ตัวอย่าง

6.2) ส่วนวิจารณ์ เป็นส่วนที่จะวิจารณ์ว่าผลการทดลองเป็นไปตามทฤษฎีหรือไม่เป็นไปตามทฤษฎี ถ้าไม่เป็นไปตามทฤษฎีเนื่องจากสาเหตุอะไร

สังเกตุว่า ส่วนนี้ของ Lab Report มีคะแนนสูงที่สุด นักศึกษาจึงต้องทำงานเป็นทีมทุก ๆ คน คือ ***ทุกคนต้องมีส่วนร่วมในการช่วยกันวิจารณ์*** ไม่ปล่อยให้เป็นหน้าที่ของนักศึกษา ผู้เขียนรายงานการทดลองวิจารณ์แต่คนเดียว เพราะถ้าวิจารณ์มาน้อยๆ หรือวิจารณ์มาผิด ๆ มาจะเสียคะแนนมาก

กลุ่มการทดลอง_____ Section__

สาขาวิชาวิศวกรรมเซรามิก
สำนักวิชาเทคโนโลยีทรัพยากร
มหาวิทยาลัยเทคโนโลยีสุรนารี
111 ถ.มหาวิทยาลัย ต.สุรนารี อ.เมือง จ.นครราชสีมา 30000

การทดลองที่_________

รหัสวิชา____________________ วันที่ทดลอง________________

หัวข้อเรื่องที่ทดลอง

บทคัดย่อ

ผู้เขียนรายงานทดลอง ชื่อ ______________________________ เลขประจำตัว ______________

ผู้ร่วมทำการทดลอง

1) ชื่อ ____________________________________ เลขประจำตัว ____________________

2) ชื่อ ____________________________________ เลขประจำตัว ____________________

3) ชื่อ ____________________________________ เลขประจำตัว ____________________

4) ชื่อ ____________________________________ เลขประจำตัว ____________________

5) ชื่อ ____________________________________ เลขประจำตัว ____________________

วันที่ส่งรายงานการทดลอง____________________อาจารย์ผู้สอน_________________________

หลักการในการเขียนรูปภาพและกราฟใน Lab report

1. ปกติควรมีรูปภาพหรือกราฟ 1 กราฟ ในกระดาษ 1 แผ่น ดังตัวอย่างในรูปที่ 1-3

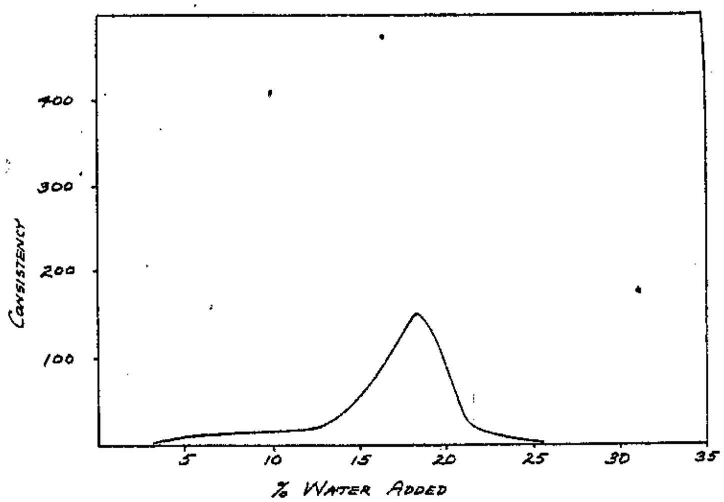
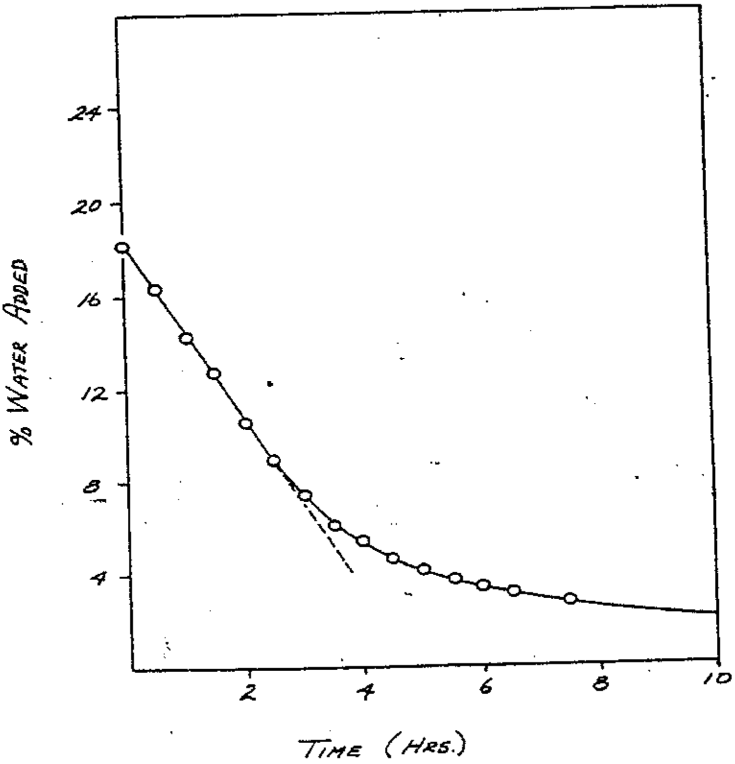


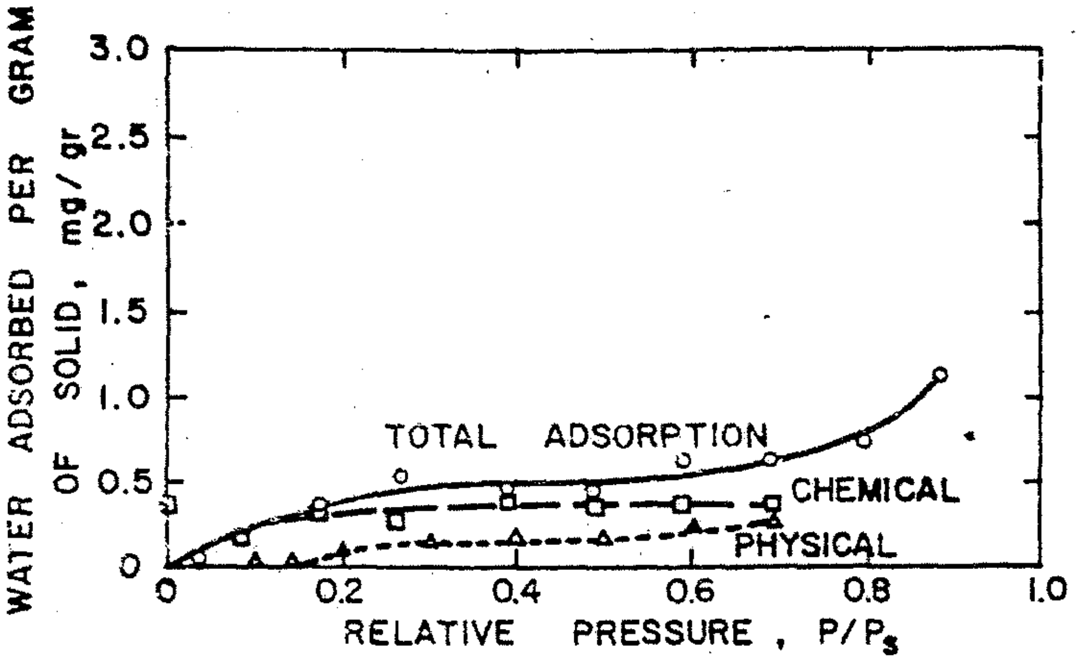
Fig. 1
Plastograph of a Red Burning Brick Clay

รูป 1 ตัวอย่างการเขียนกราฟแนวนอน



รูป 2 ตัวอย่างการเขียน กราฟแนวตั้ง

Drying Curve of a Red Burning Brick Body at 50°C and 50%R.H.



รูป 3 Comparative physical and chemical effects in adsorption of water vapor on the 1250°C Kaolin specimen at 35°C

2. ควรเว้นขอบกระดาษทางซ้ายและล่างอย่างน้อย 1.5 นิ้ว และขอบบนและขวาอย่างน้อย 1 นิ้ว ดังตัวอย่างในรูปที่ 1-3
3. ถ้าในรายงานการทดลองฉบับที่เขียนมีรูปภาพและกราฟมากกว่า 1 ต้องลำดับเลขที่ภาพด้วยเลข Arabic เช่น 1,2,3,4,... ดังตัวอย่างในรูปที่ 1-3
4. คำอธิบายภาพต้องเขียนไว้ใต้ภาพ ดังตัวอย่างในรูปที่ 1-3
5. ปกติแกนนอน หรือแกน x ของกราฟมักเป็นแกนที่ไม่สำคัญเท่ากับ y จึงมักเขียนเป็นแกนเวลา หรืออุณหภูมิ
6. ต้อง label แกนให้ชัดเจน บอกหน่วย ถ้าต้องคูณ 10 หรือ 100 ที่แกนใดแกนหนึ่งให้บอกให้ชัดเจน การ label แกน x ให้ label ใต้ตรงกึ่งกลางของแกน x ส่วนการ label แกน y ควรเขียนอักษรในแนวนอนตามแกน เขียนในแนวที่ให้ผู้อ่านอยู่ทางด้านขวามือของหน้ากระดาษ ดังตัวอย่างในรูปที่ 1-3
7. ถ้ามีข้อมูลหลายชุด ในกราฟต้องใช้เครื่องหมายที่ต่าง ๆ กัน แสดงตัวแปรแต่ละชุด เช่น เครื่องหมาย ○,□, △ ดังในตัวอย่างในรูปที่ 3
8. ถ้าจำเป็นต้องวางกราฟ ตามแนวยาวของหน้ากระดาษจะต้องวางนอนตามยาวในลักษณะ อ่านอยู่ทางขวามือของหน้ากระดาษตามตัวอย่างในรูปที่ 1

9. ผู้เขียน ควรใช้วิจารณญาณในการเลือกแกนของกราฟว่า ควรใช้แกน linear ธรรมดา หรือแกน log และในการต่อเส้นของกราฟว่าควรจะต่อด้วยเส้นตรง หรือเส้นโค้ง smooth กราฟมากน้อยเพียงใด
10. ถ้าคัดลอกกราฟหรือรูปภาพจากแหล่งข้อมูลมาต้องใส่ footnote บอกแหล่งที่มาของรูปภาพหรือกราฟนั้น ๆ ด้วย

หลักการในการเขียนตารางในรายงานการทดลอง

๑) ตารางที่มีไว้เพื่อแสดงผลการทดลองให้ชัดเจนขึ้นเท่านั้น ดังนั้นการมีตารางแล้ว ยังต้องมีคำอธิบายบอกว่าตารางอะไร แสดงข้อมูลดังตัวอย่างในตารางที่ I

๒) คำอธิบายตารางจะเขียนไว้บนตาราง **ไม่เขียนไว้ข้างล่าง** ดังตัวอย่างในตารางที่ I และ II

๓) ถ้าในรายงานการทดลองฉบับหนึ่งมีตารางมากกว่า 1 ตารางต้องลำดับเลขที่ ของตารางด้วยเลขโรมัน I, II, III, IV, V, VI, VII, VIII, IX, X,.......

๔) ตารางรายงานผลการทดลองไม่ใช่บัญชีข้อมูลดิบ ไม่ต้องลงรายละเอียดทุกสิ่งทุกอย่าง เช่น วัดความหนาของ Specimen ที่เผาเสร็จ วัด 3 ตำแหน่งคือ หัว, กลาง, ท้าย ได้ความหนา 2.0, 2.1 และ 2.2 ม.ม. ก็ไม่ต้องลงทั้ง 3 ค่า ในตาราง แต่ให้ลงค่าเดียวคือค่า mean ที่เกิดจาก 3 ค่า เป็น 2.1 ม.ม. เป็นต้น

๕) ข้อมูลที่ plot เป็นกราฟทุกชุดไม่จำเป็นต้องเขียนลงเป็นตาราง

๖) หากจำเป็นต้องเขียนตารางข้อมูลละเอียดบางส่วนที่ไม่มีความสำคัญมากในรายงานให้นำตารางนั้นไปไว้ในส่วนที่เป็นภาคผนวกไม่นำมาปนกับส่วนที่เป็น main body ของรายงานและลำดับเลขที่ตารางนั้นต่างหาก แยกจากตารางใน main body

๗) ถ้าคัดลอกตารางหรือบางส่วนของตารางจากแหล่งข้อมูลอื่นมาต้องใส่ footnote บอกแหล่งที่มาของตารางหรือข้อมูลด้วย

๘)ข้อมูลที่ได้จากการคำนวณในตาราง ควรแสดงตัวอย่างในการคำนวณด้วย

๙)ต้องแสดงหน่วยของข้อมูลทุกข้อมูลในตารางย่างชัดเจน ดังตัวอย่างในตารางที่ II

ตารางที่ 1

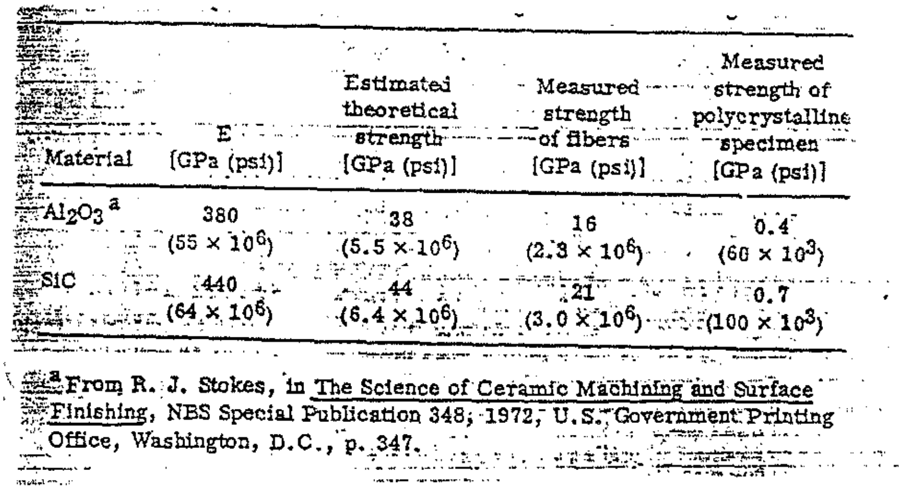
Factors for Calculating the Thermal Expansion of Glasses and Enamels

	Thermal expansion (cubical)				
Composition	M. & H. $\times 10^{-7}$	W. & S. $\times 10^{-7}$	E. & T. $\times 10^{-7}$	Composition	M. & H. $\times 10^{-7}$
SiO_2	0.8	0.8	0.15	SnO_2	2.0
Al_2O_3 . . .	5.0	5.0	0.42	TiO_2	4.1
B_2O_3 . . .	0.1	0.1	−1.98	ZrO_2	2.1
Na_2O . . .	10.0	10.0	12.96	Na_3AlF_6 . . .	7.4
K_2O	8.5	8.5	11.7	NaF	7.4
PbO	4.2	3.0	3.18	AlF_3	4.4
ZnO	2.1	1.8	2.1	CaF_2	2.5
CaO	5.0	5.0	4.89	Cr_2O_3	5.1
MgO	0.1	0.1	1.35	CoO	4.4
BaO	3.0	3.0	4.2	CuO	2.2
As_2O_3 . . .	2.0	2.0		Fe_2O_3	4.0
P_2O_5 . . .	2.0	2.0		NiO	4.0
Sb_2O_5 . . .	3.6			MnO_2	2.2

E. & T.—Corrected to English and Turner, J. Am. Ceram. Soc. 12, 760 (1929).
W. & S.—Winkelmann and Schott, Jena Glass.
M. & H.—Mayer and Havas.

ตารางที่ 2

Comparison of Theoretical Strength and Actual Strength



Material	E [GPa (psi)]	Estimated theoretical strength [GPa (psi)]	Measured strength of fibers [GPa (psi)]	Measured strength of polycrystalline specimen [GPa (psi)]
Al_2O_3 [a]	380 (55×10^6)	38 (5.5×10^6)	16 (2.3×10^6)	0.4 (60×10^3)
SiC	440 (64×10^6)	44 (6.4×10^6)	21 (3.0×10^6)	0.7 (100×10^3)

[a] From R. J. Stokes, in The Science of Ceramic Machining and Surface Finishing, NBS Special Publication 348, 1972, U.S. Government Printing Office, Washington, D.C., p. 347.

7. สรุปผลการทดลองและข้อเสนอแนะ (Result and Recommendation)

เป็นบทที่จะแสดงให้เห็นถึงความสัมพันธ์ระหว่าง ผลสรุปจากการทดลองและวัตถุประสงค์ของการทดลอง โดยอาจจะสรุปออกมาเป็นข้อๆ ตามลำดับความสำคัญ และเสนอแนะให้เห็นถึงข้อควรปรับปรุงในการทดลองเรื่องนี้ หากจะทดลองเรื่องนี้อีกหรือหากจะศึกษาเพิ่มเติมในขั้นต่อไป

8. เอกสารอ้างอิง (References)

เป็นบทที่บอกที่มาของทฤษฎี, ข้อมูล และเนื้อหาที่เกี่ยวข้องที่นำมาเขียนในรายงาน ซึ่งอาจจะยกมาจากหนังสือ หรือวารสารฉบับใดฉบับหนึ่ง ซึ่งผู้เขียนต้องบอกแหล่งที่มา เพื่อให้ผู้อ่านที่สนใจจะไปค้นคว้าต่อไปได้

แบบมาตรฐานในการเขียนเอกสารอ้างอิง จะแตกต่างกันไปในที่นี้จะให้นักศึกษาใช้ตามมาตรฐานของสถาบันวิจัยและพัฒนา มหาวิทยาลัยเทคโนโลยีสุรนารี ซึ่งมีตัวอย่างดังนี้

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1) หนังสือ : ผู้แต่ง. (ปีที่พิมพ์). <u>ชื่อหนังสือ</u>. เล่มที่(ถ้ามี). ครั้งที่พิมพ์(ถ้ามี). ชื่อชุดหนังสือและลำดับที่(ถ้ามี). สำนักพิมพ์:สถานที่พิมพ์. ***ตัวอย่างเช่น***

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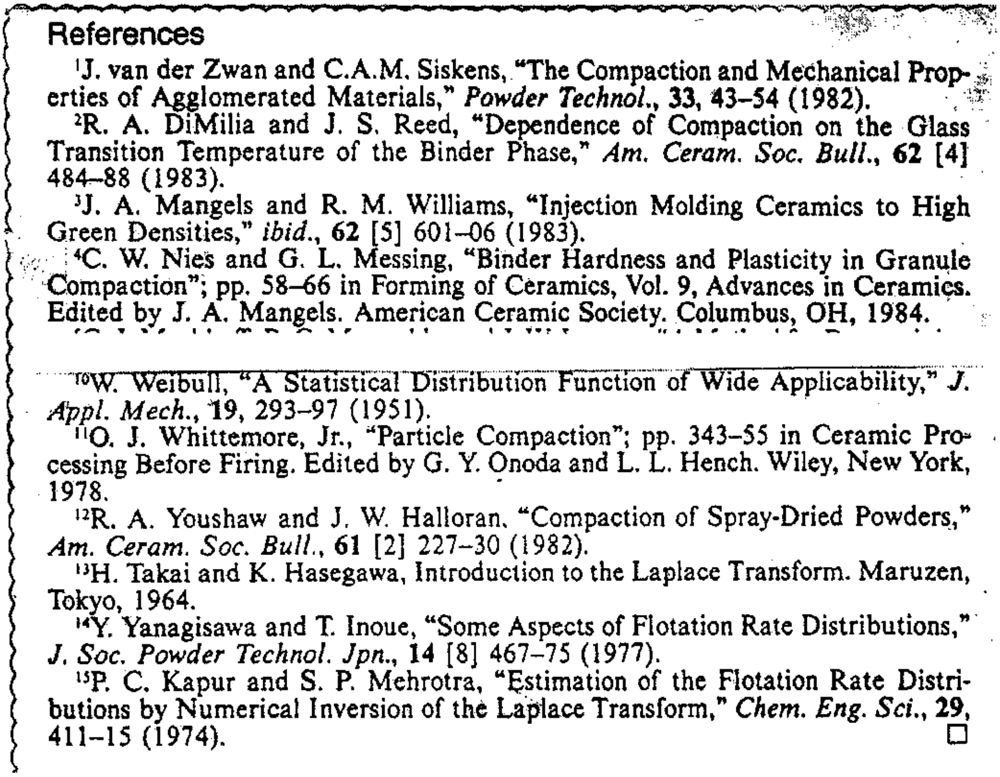

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8) จุลสาร เอกสารอัดสำเนา และเอกสารที่ไม่ได้ตีพิมพ์อื่นๆ

ให้เขียนแบบเดียวกับหนังสือหรือวารสารแล้วแต่กรณี แต่ให้มีวงเล็บว่า “อัดสำเนา” ท้ายข้อความ

9) อ้างอิงถึงเอกสารอื่น

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11) สารสนเทศจากสื่ออิเลคทรอนิกส์ คล้ายกับการอ้างอิงจากเอกสารทั่วไป แต่เพิ่มวงเล็บประเภทของสื่อ และระบุแหล่งที่จะสืบค้นได้ในตอนท้าย เช่น

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9. ภาคผนวก (Appendices)

อาจจะมีหรือไม่มีก็ได้ เป็นบทที่บรรจุข้อมูลต่าง ๆ ที่มีมากเกินความจำเป็นที่จะลงใน main body ของ report เช่น ข้อมูลดิบ, ตัวอย่างการคำนวณที่ยาว ๆ

ทั้ง 9 ส่วน ที่กล่าวมาเป็นโครงร่างทั้งหมดของรายงานที่นักศึกษาเซรามิกจะได้ฝึกฝนให้เขียนให้ถูกต้องเพื่อจะเป็นวิศวกรหรือนักวิทยาศาสตร์ที่ประสพความสำเร็จต่อไปในอนาคต

ท้ายที่สุด แต่สำคัญไม่น้อยที่สุดคือ การเขียนรายงานนักศึกษาต้องยึดหลักที่ว่า

1) ต้องเขียนรายงานมีความกระชับ ไม่ยืดยาว ตรงประเด็น และมีความต่อเนื่องที่สุด

2) ต้องหลีกเลี่ยงการใช้ภาษาพูด และคำสรรพนาม (ผม, เรา .... ฯลฯ) เช่น "ต่อจากนั้น ผมปรับวาล์วไปที่ค่าสูงสุด" เป็นต้น แต่ต้องใช้ภาษาเขียนว่า "วาล์วถูกปรับไปที่ค่าสูงสุด"

3) จะพิมพ์ หรือเขียนด้วยลายมือก็ได้ ถ้าเขียนด้วยลายมือตัวหนังสือต้องมีขนาดพอเหมาะและอ่านออกง่าย ถ้าพิมพ์จะต้อง Proof Read มาด้วย การพิมพ์ผิด ๆ มามาก แสดงให้เห็นถึงความหยาบของงานที่ทำ

4) จัดรูปเล่มให้เป็นระเบียบเรียบร้อยตามลำดับโครงร่างรายงาน

5) สำคัญที่สุดต้องตรวจคำสะกด (Spelling) ทั้งภาษาไทยและอังกฤษให้ถูกต้อง ใช้หลักไวยากรณ์ที่ถูกต้องด้วย

ภาพที่4 แสดงตารางการให้คะแนนของอาจารย์ผู้ตรวจรายงานซึ่งเต็ม 100 คะแนน/Report 1 ฉบับ เป็นการให้คะแนนกรณีที่นักศึกษาส่ง Lab Report ตรงตามเวลา *คือส่งภายใน 7 วันหลังจากการทดลอง คื* *ส่งก่อนเข้าเรียน Lab ถัดไป* **ถ้านักศึกษาส่งรายงานผลการทดลองช้าไป 1-7 วัน อาจารย์จะหักคะแนน Lat 50 คะแนน** ***หากส่งช้ากว่ากำหนด 7-14 วัน อาจารย์จะตรวจ Report ให้แต่ไม่มีคะแนน (ถูกหัก 100 คะแนน*** **แต่ถ้าส่งช้ากว่ากำหนด 14 วัน อาจารย์จะไม่รับ Report ฉบับ นั้น**

หลักการและเหตุผลในการให้คะแนนการเขียนรายงาน

1. รายงาน (คะแนน 50 %) เพื่อให้นักศึกษาเขียนรายงานเพื่อนำเสนอได้อย่างมีคุณภาพและได้มาตรฐาน ได้มีการกำหนดแบ่งส่วนการให้คะแนน (จากเต็ม 100%) ได้ดังนี้ คือ

- รายงานจะต้องส่งถึงอาจารย์ผู้สอนปฏิบัติการนั้น ในวันที่จะทำปฏิบัติการต่อไป (1 อาทิตย์หลังจากที่ทำปฏิบัติการนั้นๆ) ถ้าไม่ส่งภายในกำหนด จะถูกหักคะแนนวันละ **5 คะแนน**
- บทนำ (Introduction)….. **20%** เนื่องจากเป็นส่วนที่ต้องมีการค้นคว้าข้อมูลเพิ่มเติมเกี่ยวกับการทดลองและทฤษฎีต่างๆที่เกี่ยวข้อง เป็นส่วนที่ใช้บอกวัตถุประสงค์ของการทดลอง และการทดลองที่มีมาก่อนที่เกี่ยวข้องกับการทดลองครั้งนี้ นอกเหนือจากที่ให้ในคู่มือการทดลอง ถ้าลอกบทนำในคู่มือมาทั้งหมดจะไม่ได้คะแนน
- รูปแบบ……….**15%** รายงานควรจะมีรูปแบบถูกต้อง ครบถ้วนตามที่กำหนดไว้ นั่นคือรายงานการทดลองประกอบด้วยส่วนประกอบทั้งหมด 7 ส่วนคือ

1. ปกรายงาน (Title page) มีรูปแบบดังแสดงในหน้าถัดไป ปกควรใช้กระดาษสีพื้นอ่อน

2. บทคัดย่อ (Abstract) ความยาวประมาณ 300 คำ สามารถบรรจุในตอนกลางของหน้าปกรายงาน เป็นส่วนที่เขียนการทดลองแบบย่อๆ เช่น การทดลองอะไร วิธีการทดลองเป็นอย่างงัย ได้ผลการทดลองเป็นอย่างงัย และสรุปได้อย่างไร

3. สารบัญ (Table of contents) แสดงหัวข้อ พร้อมแสดงเลขหน้า
4. บทนำ (Introduction)
5. อุปกรณ์และวิธีการทดลอง (Equipments and Experimental procedure)
6. ผลการทดลอง (Result) และบทวิเคราะห์ (Discussion)
7. สรุปผลการทดลองและข้อเสนอแนะ (Conclusions and Recommendation)

ส่วนหลักของรายงาน

8. เอกสารอ้างอิง (Reference)
9. ภาคผนวก (Appendices)

- ความสะอาดและความเรียบร้อย……..**15%** รายงานไม่จำเป็นต้องพิมพ์ ถ้าเขียนด้วยลายมือต้องมีความเรียบร้อยเป็นระเบียบ อ่านง่าย และน่าอ่าน มีหน่วยต่างๆ ถูกต้อง ครบถ้วน ไม่มีคำผิด ซึ่งจะสำคัญมากในการเสนอรายงาน เพราะแสดงถึงความตั้งใจและมาตรฐานของผู้นำเสนอ
- วิธีการทดลองและการแสดงผล (Experimental Procedure & Results) ……..**20%** เป็นส่วนที่มีความสำคัญมาก วิธีการทดลองเป็นส่วนที่เขียนขึ้นมาเองจากที่ได้ทำการทดลองจริง (ไม่ได้คัดลอกมาจากคู่มือ) พร้อมทั้งมีภาพคร่าวๆแสดงถึงเครื่องมือ และส่วนประกอบของเครื่องและการแสดงผลการทดลองต้องชัดเจน ดูง่าย และมีหน่วย และคำอธิบายต่างๆถูกต้อง ผลการทดลองอาจจะเสนอในรูปของตารางหรือกราฟ ในการคำนวณข้อมูลออกมา จะต้องมีการแสดงสมการ และตัวอย่างการคำนวณประกอบด้วย
- วิจารณ์และสรุปผลการทดลอง (Discussion & Conclusion) ……**30%** เป็นส่วนที่สำคัญที่สุดในรายงาน ให้คะแนนมากที่สุด ควรมีการวิเคราะห์และวิจารณ์ผลที่ได้ แสดงความคิดเห็น

เกี่ยวกับความถูกต้องของวิธีการวัด การทดสอบ ข้อดี และข้อเสียของวิธีการ ฯ หรือผลการทดลองเป็นไปตามทฤษฎีหรือไม่ ถ้าไม่เป็นต้องหาเหตุผลว่าเนื่องมาจากอะไร ดังนั้นส่วนนี้ทุกคนในกลุ่มต้องมีส่วนร่วมในการวิจารณ์ เพราะจะทำให้เสียคะแนนมาก

- การเขียนเอกสารอ้างอิง (Reference) บอกที่มาของทฤษฎี ข้อมูลและเนื้อหาที่เกี่ยวข้องที่นำมาใช้เขียนในรายงาน อาจจะมาจากหนังสือ หรือวารสารฉบับใดฉบับหนึ่ง ซึ่งต้องอ้างถึงและเพื่อให้ผู้อ่านที่สนใจได้ไปค้นคว้าต่อไป

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ตัวอย่างการเขียนบรรณานุกรมจากแหล่งที่มาต่างๆ สามารถค้นคว้าได้จากหนังสือแนะนำการเขียนรายงาน ที่เกี่ยวข้อง หรือจากตัวอย่างการเขียนบรรณานุกรมจากหนังสือหรือวารสารทั่วๆ ไป

การฝึกเขียนรายงานที่ดี เพื่อประโยชน์ของนักศึกษาเองในอนาคต และ ถ้ารายงานดี มีคุณภาพ นักศึกษาสามารถนำมาอ่านทบทวนความรู้ได้ง่าย

การทำงานเป็นกลุ่ม ขอให้มีการแบ่งงานกันอย่างยุติธรรม และเรียนรู้การทำงานเป็นทีม รู้จักการปรับตัว ช่วยเหลือผู้อื่น ซึ่งจะมีความสำคัญมากในการทำงานจริงในอนาคต

NAME	General Appearance	Format	Abstract	Introduction	Procedure	Results & Discussion	Summary & Conclusion	Tables & Figures	Total	COMMENTS
	10	10	10	10	10	30	10	10	100	

รูป 5 ตัวอย่างการให้คะแนนการเขียนรายงาน

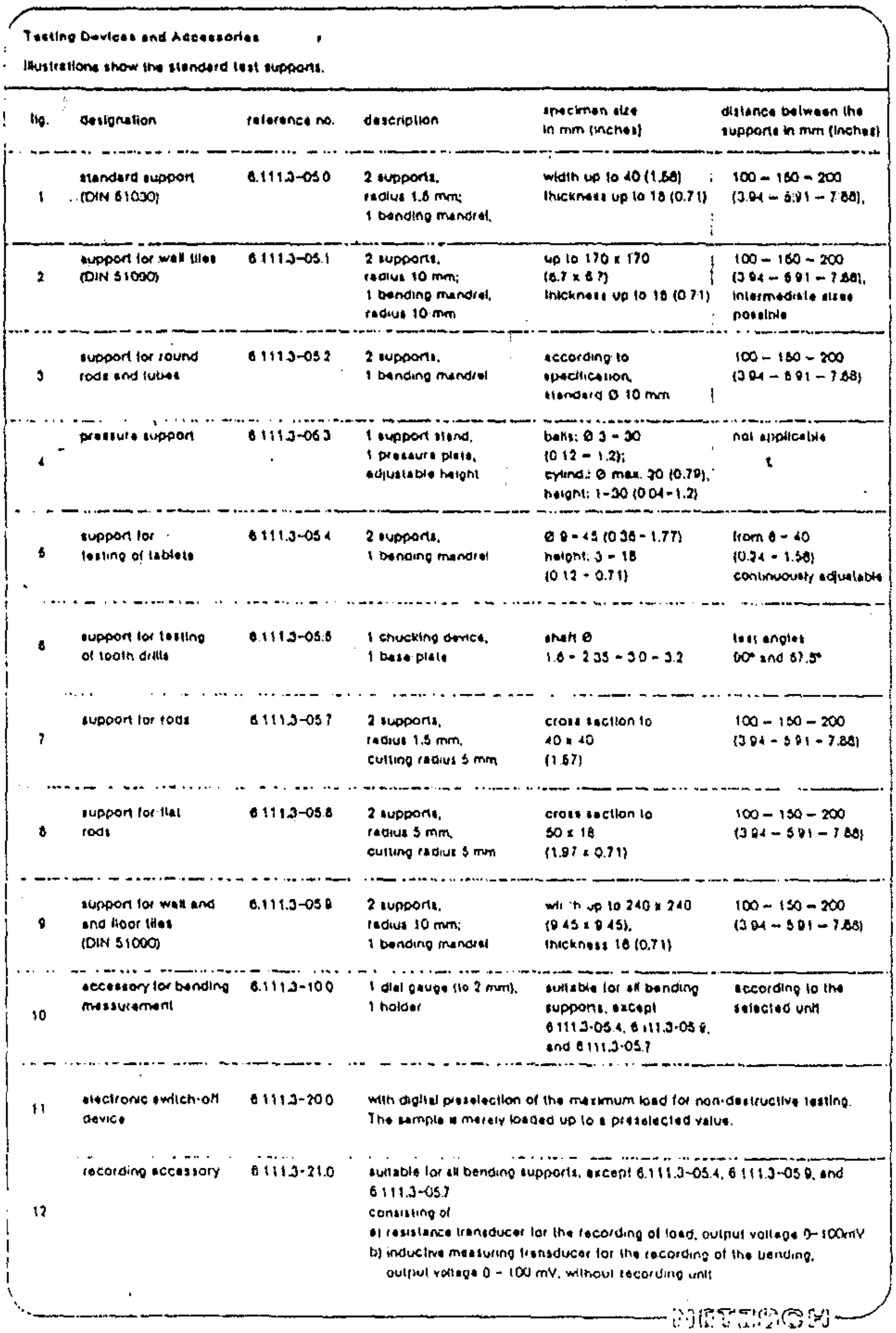
เครื่องวัด Bending Strength

1. General

The Bending Strength Testers, series 401, are used for the determination of the bending strength and the static modulus of elasticity at ambient temperature of mainly ceramic raw materials and ceramics. Using different standard supplements measurements on test bodies of different shapes, dimensions and materials can be carried out (compare fig. 1). The determination follows DIN 51030 and EN 100. Depending on application three basic models are available with four load ranges each (compare table 1)

Table 1 Load Ranges of the Bending Strength tester

Type	Load range / N			
401/1	20	40	80	240
401/2	20	120	240	480
401/3	75	150	300	600



Testing Devices and Accessories

Illustrations show the standard test supports.

fig.	designation	reference no.	description	specimen size in mm (inches)	distance between the supports in mm (inches)
1	standard support (DIN 51030)	6.111.3-05.0	2 supports, radius 1.5 mm; 1 bending mandrel,	width up to 40 (1.58) thickness up to 18 (0.71)	100 – 150 – 200 (3.94 – 5.91 – 7.88),
2	support for wall tiles (DIN 51090)	6.111.3-05.1	2 supports, radius 10 mm; 1 bending mandrel, radius 10 mm	up to 170 x 170 (6.7 x 6.7) thickness up to 18 (0.71)	100 – 150 – 200 (3.94 – 5.91 – 7.88), intermediate sizes possible
3	support for round rods and tubes	6.111.3-05.2	2 supports, 1 bending mandrel	according to specification, standard Ø 10 mm	100 – 150 – 200 (3.94 – 5.91 – 7.88)
4	pressure support	6.111.3-06.3	1 support stand, 1 pressure plate, adjustable height	balls: Ø 3 – 30 (0.12 – 1.2); cylind.: Ø max. 20 (0.79), height: 1–30 (0.04–1.2)	not applicable
5	support for testing of tablets	6.111.3-05.4	2 supports, 1 bending mandrel	Ø 9 – 45 (0.35 – 1.77) height: 3 – 18 (0.12 – 0.71)	from 6 – 40 (0.24 – 1.58) continuously adjustable
6	support for testing of tooth drills	6.111.3-05.5	1 chucking device, 1 base plate	shaft Ø 1.6 – 2.35 – 3.0 – 3.2	test angles 90° and 67.5°
7	support for rods	6.111.3-05.7	2 supports, radius 1.5 mm, cutting radius 5 mm	cross section to 40 x 40 (1.57)	100 – 150 – 200 (3.94 – 5.91 – 7.88)
8	support for flat rods	6.111.3-05.8	2 supports, radius 5 mm, cutting radius 5 mm	cross section to 50 x 18 (1.97 x 0.71)	100 – 150 – 200 (3.94 – 5.91 – 7.88)
9	support for wall and and floor tiles (DIN 51090)	6.111.3-05.9	2 supports, radius 10 mm; 1 bending mandrel	width up to 240 x 240 (9.45 x 9.45), thickness 18 (0.71)	100 – 150 – 200 (3.94 – 5.91 – 7.88)
10	accessory for bending measurement	6.111.3-10.0	1 dial gauge (to 2 mm), 1 holder	suitable for all bending supports, except 6.111.3-05.4, 6.111.3-05.9, and 6.111.3-05.7	according to the selected unit
11	electronic switch-off device	6.111.3-20.0	with digital preselection of the maximum load for non-destructive testing. The sample is merely loaded up to a preselected value.		
12	recording accessory	6.111.3-21.0	suitable for all bending supports, except 6.111.3-05.4, 6.111.3-05.9, and 6.111.3-05.7 consisting of a) resistance transducer for the recording of load, output voltage 0–100mV b) inductive measuring transducer for the recording of the bending, output voltage 0 – 100 mV, without recording unit		

Fig. 1 available standard test supplement

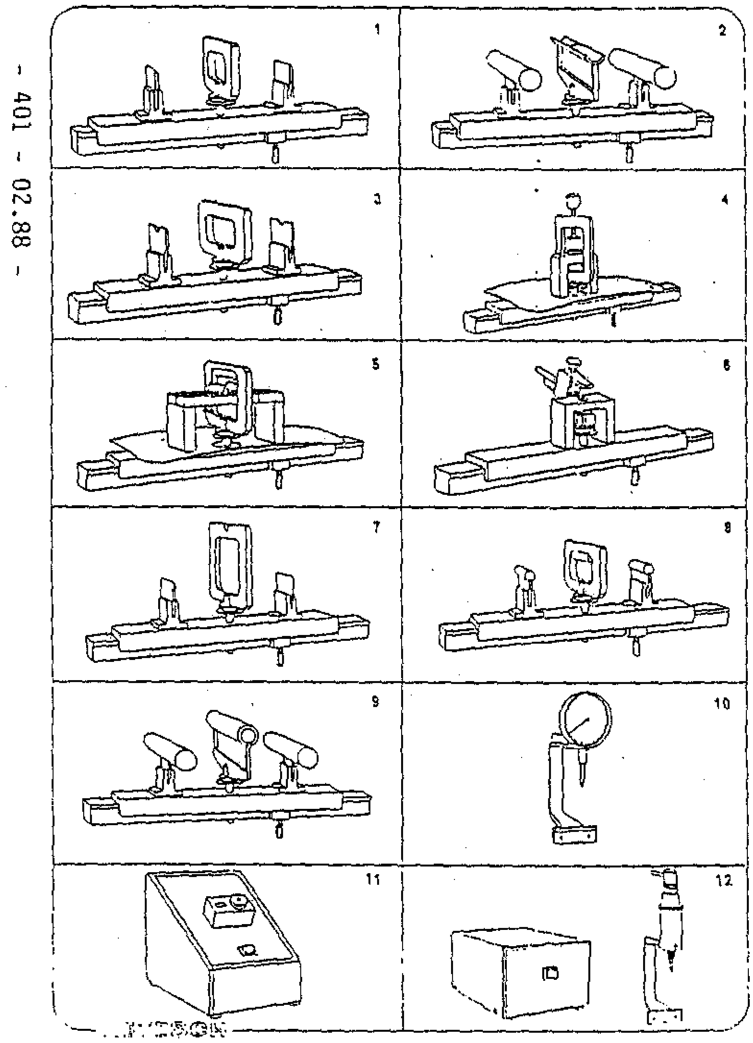


Fig. 2 Supports for Bending Strength Test

2. Construction and mode of operation (see in fig.3)

2.1 Principle of Load Transmitting

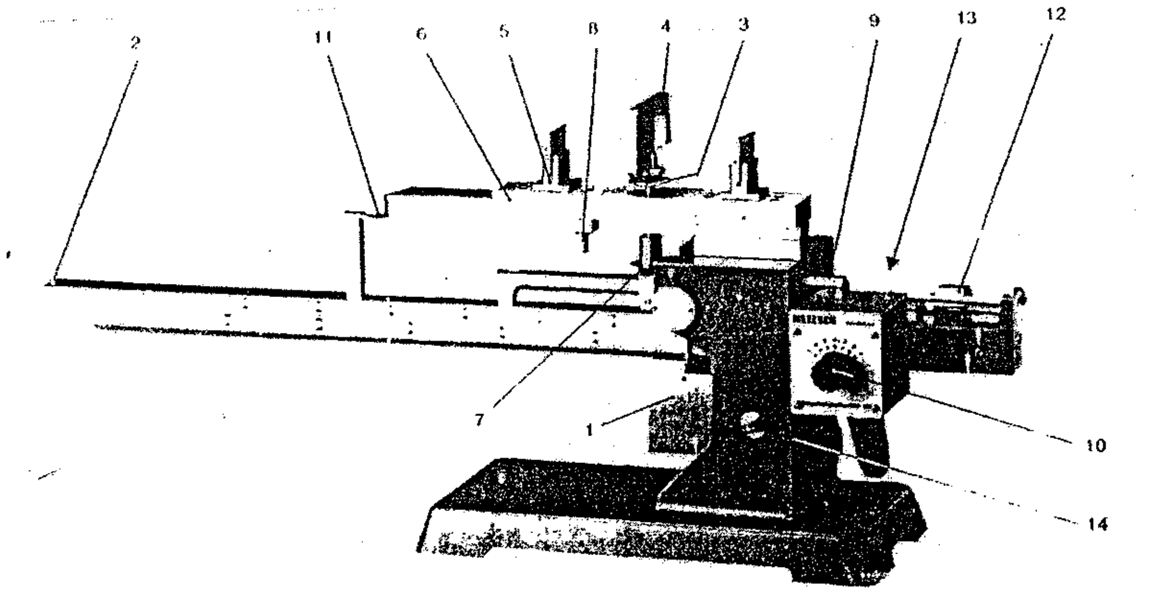
Load is affected by the electro-motive displacement of the sliding weight (1) along the swing boom (2). The arising by unbalancing the sliding weight is transmitted by the load pin (3) to the load frame (4) (principle of the one-armed lever). This force acts onto the centre of the test body which is deposited on two supports (5) (three-point method).

2.2 Variation of Load Range and Load Rate

The four available load ranges of the bending strength tester (compare table 1) can be set by loosening the index pin (8) and displacing the sled (6). The ranges can be read off on the markings on the sled. Each of the four load ranges is represented by an own division on the scale of the swing boom. In case of changing the measuring range the compensation weight (9) is displaced simultaneously by a cable line in such way, that the equilibrium position of the swing boom (2) is maintained.

A synchronous motor with a change gearing moves by means of a toothed belt the sliding weight (1) inside the swing boom (2) in accordance with increasing load. Four different speeds can be selected (compare table 2).

Whenever the selector switch (10) is on position "0", the motor is disconnected and the sliding weight (1) can be moved manually. On the right edge of the swing boom (2) there is a threaded pin. By means of this pin the stop of the sliding weight (1) for load "0" (start of the measurement) can be exactly calibrated. If the sliding weight is on position "0" the taring pointers (11) must be in accordance. Deviations are corrected with the taring weight (12). The driving motor is switched on and off by the power switch.



Erläuterungen:

(1)	Laufgewicht	(8)	Index-Stift
(2)	Ausleger	(9)	Ausgleichsgewicht
(3)	Belastungsbolzen	(10)	Schaltknopf
(4)	Belastungsbügel	(11)	Tarierzungen
(5)	Prüfkörperstützen	(12)	Tariergewicht
(6)	Schlitten	(13)	Rändelmutter
(7)	Höhenverstellung	(14)	Hutmutter für Anschlag

Explanations:

(1)	sliding weight	(8)	index pin
(2)	swing boom	(9)	compensation weight
(3)	load pin	(10)	gear switch
(4)	load frame	(11)	taring pointers
(5)	test specimen supports	(12)	taring weight
(6)	sled	(13)	knurled head nut for micro switch
(7)	elevation adjustment	(14)	cap nut for limit stop

รูป 3 Bending Strength Tester 401

Load Range		Load Increase N/s			
Type	N	1st gear	2nd gear	3rd gear	4th gear
401/1	20	0.0833	0.25	0.5	1
	40	0.1670	0.50	1	2
	80	0.3330	1	2	4
	240	1	3	6	12
401/2	60	0.125	0.25	0.5	1
	120	0.250	0.50	1	2
	240	0.50	1	2	4
	480	1	2	4	8
401/3	75	0.125	0.25	0.50	1
	150	0.25	0.50	1	2
	300	0.50	1	2	4
	600	1	2	4	8

Table 2 load and load increase ranges of the bending strength tester

2.3 Automatic determination of tensile stress

The switch case in the rear of the bending strength tester contains a micro-switch, which sensor touches the knurled head nut (13). The thread bolt with the knurled head nut (14) move together with the swing boom (2). The micro-switch disconnects the motor at a defined deflection of the swing boom, normally when the test body breaks. The switch point is adjusted by turning the knurled head nut (13). The deflection of the swing boom (2) is limited by a stop. Which can be displaced after loosening the cap nut (14)? When switching off the driving motor the sliding weight (1) stops immediately on the last load value, which can be easily read off on the scale of the swing boom. If the final load does not lead to breakage of the test body on the adjusted load range, the motor is switched off automatically.

3. Commissioning

Prior to first putting into operation the entrained rope (for the automatic weight compensation when changing the measuring range) has to be put over the corresponding rolls!

1. Set gear switch (10) to "0"
2. Push the sliding weight (1) to "0"
3. Check whether the sliding weight (1) stops at "0"
4. Bring the pointers (11) in accordance by use of the taring weight (12)
5. Switch the gear switch (10) to any gear
6. Switch on the power switch. As soon as the sliding weight (1) has moved a little to the left, the swing boom (2) lowers up to stop. The stop must be adjusted by means of the cap nut (14) in such way, that a test body will surely break, but the compensation weigth does nowhere strike.
7. The knurled head nut (13) must be adjusted in such way, that it is guaranteed that the micro-switch disconnects the drive in case of breakage of the test body, but not already in case of bending.

4. Determination of the tensile stress

4.1 Measurement

1. Push the sliding weight (1) to "0"
2. Adjust the sledge (6) to the required load range. In case of doubt select the lowest load range.
3. Adjust the desired load increase on the gear switch (10) suing table 2.
4. Put the specimen supports (5) to the desired width between the supports, symmetrically to the load frame (4).
5. Put on the test body.
6. In case of test bodies which are plane on top loosen the knurled head nut situated on the upper edge of the load pin (3), in case of concave test bodies the knurled head nut must be tightened.

7. Adjust the load frame (4) to the height of the test body using the elevation adjustment (7), that if the pointers are in accordance the knife of the load frame (4) just touched the test body.

Remark: If a higher bending is to be expected, the lift taring pointer is adjusted a bit higher (maximum up to the stop).

8. Switch on the power switch. Wait for the breakage of the test body and thus stoppage if the sliding weight (1). Read off the break load.
9. Repeat the measurement in a higher load range if no breakage occurred. Put back the sliding weight (1) carefully to “0” (gear switch (10) must be on “0”).

4.2 Evaluation

The tensile bending stress can be calculated from the read-off breaking load F_B, the width between the supports L_s and the moment of resistance W of the test body:

$$\sigma = \frac{F_B . L_s}{4W}$$

For specimen with a rectangular cross section of height h and width b the moment of resistance is calculated as follows:

$$W = \frac{b. h^2}{6}$$

and thus the tensile bending stress is

$$\sigma = F_B . \frac{3 L_s}{b}$$

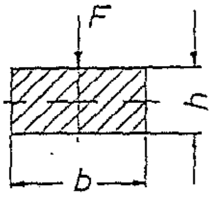
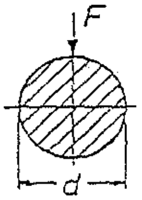
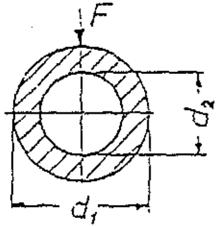
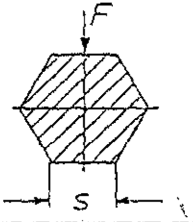
According to DIN 51030 there follows with the dimension h = 15 mm, b = 21 mm:

$$\sigma = F_B . \frac{L_s}{3150 \text{ mm}^3}$$

With the standard width between the supports $L_s = 200$ mm the following relation for the determination of the tensile bending stress results:

$$\sigma = F_B \cdot 0.06349\ \mathrm{mm}^{-2}$$

Table 3 Moment of resistance of some essential cross section forms

shape of cross sect.	*moment of resistance*
	$W = \frac{h^2 \cdot b}{6}$
	$W = \frac{\pi \cdot d^3}{32}$
	$W = \frac{\pi \cdot (d_1^4 - d_2^4)}{32 \cdot d_1}$
	$W = \frac{5 \cdot s^3}{8}$

5. Instruction to renew the entrainer rope for the automatic weight compensation when changing the measuring range

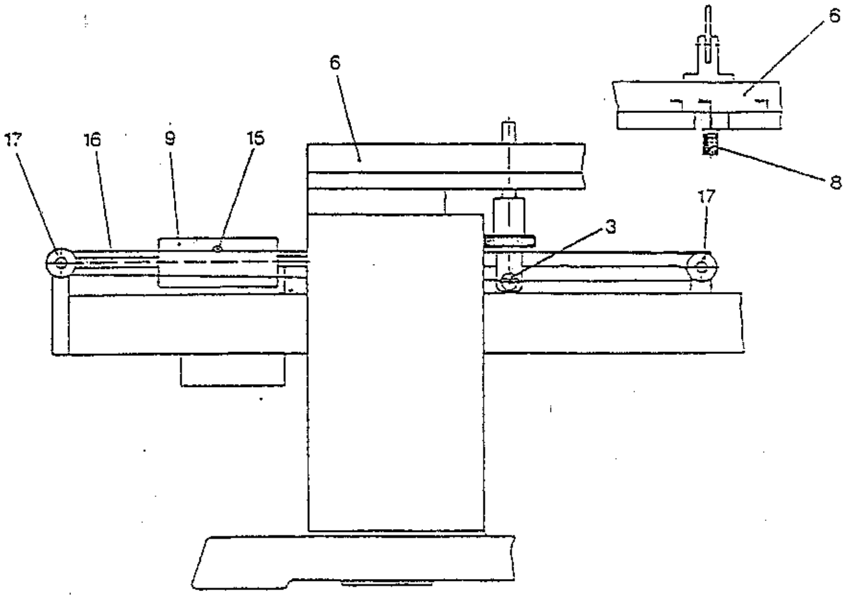


Fig. 4 Exchange of the entrainer rope for the automatic weight compensation

1. Select a measuring range as low as possible. For this move the sledge (6) until the locating mark on the sledge matches the loading mark of the index device. Tighten the index pin (8) in this position.
2. Adjust the compensation weight (9) on its guide bar in such way, that both taring pointers (11) of the bending strength tester agree.

Attention! Do not change the position of the compensation weight anymore when mounting the entrainer rope.

3. Loosen the clamping screw (15) on the compensation weight by some turns, afterwards wrap the rope (16) with one turn around the shaft of the clamping screw. Retighten the clamping screw. No the rope is firmly connected with the compensation weight (9). The clamping point should be in about in the centre of the rope.
4. Now put the rope over the both rope pulleys (17), then the two ends of the rope have to be thread on the left and the right through the cross hole of the bearing pin (3) and must be knotted first over the surface area of the pin. To avoid that this knot will be pulled through the bore the free ends of the rope have to be knotted once more several times over the surface area of the bearing pin (3).
5. The free ends of the rope above the knot now can be cut off. Fix the knots by use of a suitable adhesive.

Attention! Do not operate the sledge adjustment before the fixing adhesive has hardened.

Check: If the mounted has been done corrected, the taring pointers of the instrument remain always in agreement- independent on the selection of the measuring range. No supplementary adjustment by the use of the installed counter-weights is necessary.

1. Specific surface area หาโดยวิธี Methylene blue

   Specific surface area คือพื้นที่ผิวจำเพาะของดินหนัก 1 กรัม

   MBI = millimole equivalent ของดินแห้งหนัก 100 กรัม

   ดังนั้น ดินแห้ง 1 กรัม จะมี MBI/100 millimole eauivalent

   หรือ ดินแห้ง 1 กรัม จะมี $\frac{\text{MBI}}{100\text{x}1000}$ mole equivalent

   1 mole = 6.02×10^{23} อนุภาค

   $\frac{\text{MBI}}{100{,}000}$ mole = $\frac{6.02 \times 10^{23} \times \text{MBI}}{100{,}000}$ อนุภาค

   1 อนุภาค methylene blue มีพื้นที่ผิว 129.2×10^{-20} m^2

   ดังนั้น $6.02 \times 10^{23} \times \text{MBI} \times 10^{-5}$ อนุภาค มีพื้นที่ผิว

   $$= 6.02 \times 10^{23} \times \text{MBI} \times 10^{-5} \times 129.2 \times 10^{-20} \text{ m}^2$$
   $$= 6.02 \times \text{MBI} \times 0.01 \times 129.2 \text{ m}^2$$

   ดังนั้น **Specific surface area ของดิน 1 กรัม = $6.02 \times \text{MBI} \times 0.01 \times 129.2$ m^2/g**

ภาคผนวก

Annual Ceramic Mineral Resources Review

Because of the changing nature of materials requirments for ceramics, such as the synthetic materials used in advanced ceramics, the ACerS Ceramic Minerals Resources Committee, chaired by Hendrik Heystek, requests additional input to the Annual Ceramic Mineral Resources Review. The committee will consider reports on the availability, technology, and economic status of all materials used in ceramics today. Authors wishing to contribute to the May 1986 Ceramic Mineral Resources Review should contact Hendrik Heystek, U.S. Department of the Interior, Bureau of Mines Tuscaloosa Research Center, Box L, University, AL 35486.

Alumina

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"Alumina" is a specific chemical term defining the oxide of aluminum, Al_2O_3. It occurs abundantly in nature, most often as impure hydroxides which are the essential constituents of bauxites and laterites. Numerous high purity grades of alumina are manufactured from these ores as major chemical products, including hydrated, activated, calcined, low soda, tabular, and fused. Most raw bauxite is refined by the Bayer process to remove impurities (e.g., SiO_2, Fe_2O_3, and TiO_2) and produces a nominal 99.5% Al_2O_3 product with Na_2O as the dominant impurity. Virtually all commercial production is obtained by the Bayer process with Western World alumina production averaging 25 million metric tons during the decade ending 1983. About 90% of alumina is used in the production of aluminum metal. Ceramic and refractory applications consume slightly more than 50% of the nonaluminum production. Major nonaluminum calcined alumina markets in 1982 are estimated: refractories=30%; abrasives=25%; whitewares and spark plugs=25%; ceramics (80+% Al_2O_3)=10%; glass and enamels=5%; and miscellaneous=5%.

The future availability of special Bayer aluminas for the ceramics and refractories industry depends upon the availability of metal grade bauxite and the refining capacity to convert bauxite into high purity alumina. Western World reserves of metal grade bauxite are generally located around the equatorial belt and total 22.4 billion metric tones. At average operating rates for aluminum metal production during the past decade, these reserves are adequate to supply the Western World for over 200 years.

Although a near supply-demand balance occurred in 1979 with regard to refining capacity and aluminum metal and nonmetal requirements, there appears to be plenty of available alumina for future nonmetal requirements. Current refining capacity exceeds 30 million metric tons and is forecasted to exceed 40 million metric tons of alumina by 1990.

Although a wide variety of high purity aluminas is used in ceramics/refractories, specialty calcined alumina powders, tabular alumina refractory aggregate and calcium aluminate cement (CAC) are the most important. Specialty calcined aluminas are prepared in a very wide range of grades to suit a similarly wide range of applications. Both chemical and physical properties may be adjusted during preparation. The normal 0.5% Na_2O Bayer alumina can be reduced to make specialty low soda grades during refining and/or calcination. Physical properties may be adjusted during rotary kiln calcination and by subsequent grinding. Precision control of crystal size, degree of calcination and level of impurities are required in the manufacture of commercial aluminas during heating to obtain alpha alumina, the stable form of anhydrous alumina known also as the naturally occurring substance corundum.

The availability of specialty grades of calcined alumina in the unground form is basically a function of rotary kiln capacity with the appropriate auxiliary equipment necessary to provide the correct chemical and temperature controls. Excess kiln capacity exists at many alumina refiners because of the adoption of fluid calciners in the manufacture of metal grade alumina. Thus, there will be an adequate supply of calcined aluminas for ceramics/refractories through 1990 when projecting a 10% compound annual growth pattern which is overoptimistic based on historical patterns. The availability of ground calcined aluminas is simply a matter of installing the necessary grinding facilities to accommodate the growth.

Tabular alumina is produced from calcined alumina in large-scale production centers having a capacity of 50 000 to 125 000 metric tons annually. This sintered product is a high purity aluminum oxide that has been recrystallized to the massive corundum state by heating 1.9 cm (0.75-in.) pellets at 1925°C (3500°F), just below the fusion temperature to convert the fine alpha alumina crystallites into large hexagonal elongated table-shape crystals ranging from 40 to 200μm median.

Tabular alumina is crushed and graded to offer a wide variety of particle sizes from 3 mesh aggregate to −325

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*Member, the American Ceramic Society.

Recent information on Andalusite, Arsenic, Barium Carbonate, Lanthanides and Yttrium, Limestone and Dolomite, Silicon Nitride, and Titanium Products, which are not covered here, can be found in last year's Annual Ceramic Mineral Resources Review (cf. pages 668–91, May 1984 *Ceramic Bulletin*).

mesh powder. Metallic iron picked up in the crushing and screening process is removed by electromagnetic separation.

Recent expansions have boosted U.S. capacity by an estimated 60 000 metric tons. A new tabular plant became operational in early 1985 in Ludwigshafen, Germany, adding 50 000 metric tons of capacity in Europe. Retrofitting older operations with the latest production technology will push total capacity to above 250 000 metric tons. Assuming an optimistic growth of 10% annually, these expansions provide an adequate supply of tabular until 1990–95.

High purity CAC is characterized by its low iron and silica and its high alumina content. Low cement castable technology may limit CAC growth, but expanding monolithic applications tend to counter this trend. The increasing need for more sophisticated high quality monolithic refractories should continue to expand the growth and the demand for these products which are made by reacting high purity alumina with limestone in production centers ranging from 15 000–60 000 metric tons. Availability is, therefore, tied to the capacity of calcium aluminate cement production centers. Recent expansions in the U.S. by Alcoa and Lone Star Lafarge combined with a new high purity CAC plant in Rotterdam, increases total capacity to about 125 000 metric tons. This should be adequate for anticipated growth and demand through 1990.

Market prices were relatively stable for Bayer alumina products until 1974 when prices rose to reflect the rapid increase in costs of fuel and bauxite. Prices from 1974 to 1980 were cost-driven, for the most part reflecting inflationary factors and were generally accepted in the marketplace. Prices from 1980 to 1984 began to again stabilize as a result of low inflationary pressures and market competitiveness. The 1984 year-end price range in the U.S. for carload quantities of Bayer process aluminas follows: alpha alumina trihydrate —\$0.24 to \$1.35/kg (\$0.11 to \$0.61/lb); normal calcined aluminas—\$0.40 to \$0.62/kg (\$0.18 to \$0.28/lb); low soda calcined aluminas—\$0.49 to \$1.74/kg (\$0.22 to \$0.79/lb); tabular alumina—\$0.51 to \$1.73/kg (\$0.23 to \$0.33/lb); and CAC (60%–80% Al_2O_3)—\$0.33 to \$0.53/kg (\$0.15 to \$0.24/lb).

Although there are numerous methods for producing alumina of increasing purity, commercial production is relatively small compared to the Bayer process alumina production and may be priced as much as 10 times higher than the most equivalent Bayer product.

The Western World market for special aluminas is supplied primarily by Alcoa, Kaiser, and Alcan in North America; PUK, Martinswerk (Alusuisse), Alcoa Chemie, Vereinigte Aluminium-Werke (VAW), and British Aluminium Company (BACO) in Europe; and Nippon Lightmetal, Showa Denko, Sumitomo Chem-icals, and Mitsui Alumina and Moralco in Japan.

Although the depressed 1982–1984 economy reduced the overall demand for all ceramic/refractory aluminas, the general trend by industry to adopt advancing technologies ensures their future growth. This is depicted by the previously cited example about the steel industry turning to more expensive, yet more cost-effective, refractories in the production of higher quality steels using the latest available technologies in order to meet competition and to survive. □

Ball Clay

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Ball clay is a fine-grained, highly plastic, and essentially white-burning sedimentary clay that is used as a bonding agent for whitewares and refractory production. These clays are composed mineralogically of kaolinite, quartz, feldspar and/or mica, and organic (carbonaceous) matter. There can be trace to minor amounts of other clay minerals such as illite and/or montorillonite present in certain commercial grades.

The main function of ball clay in ceramic compositions is to contribute plasticity or workability, provide green strength, and fire to a reasonable degree of whiteness. They must also possess certain rheological properties that enable them to be deflocculated easily for slip cast applications.

The major ball clay sources are in the U.S., most of the ball clays are mined in Kentucky and Tennessee with lesser quantities extracted from Mississippi, Texas, and Maryland. The major producers are Kentucky-Tennessee Clay Co., Mayfield, KY; H. C. Spinks Clay Co., Paris, TN; Cyprus Industrial Minerals, Gleason, TN; and Old Hickory Clay Co., Mayfield, KY. K-T operates open pit mines in the three states. The other producers restrict their operations to one or two states.

U.S. and world production of ball clay minerals increased, for the second consecutive year and continues to reverse the downward trend in production that persisted from 1978 to 1982 (according to preliminary figures from BuMines). In 1984, major domestic uses for ball clays were estimated as 32% dinnerware and pottery, 13% floor and wall tile, and 18% sanitary ware.

Nearly all U.S. imports of ball clay are from England and probably represent less than 1% of all ball clay consumed in the U.S. Most of the world supplies are provided from sources in the central U.S. (Kentucky and Tennessee) and England. There are other ball-clay-like materials produced in central and eastern Europe.

The use of U.S. ball clay increased every year between 1975 and 1979 and was sparked essentially by the demand for housing in that time period. That growth declined significantly since 1979 to a low of 582 000 metric tons (642 000 short tons) in 1982. In 1983 and 1984 increases in home and commercial building, and automobile production increased the demand for ball clays again. For the first nine months of 1984 production is reported at 758 000 metric tons (835 000 tons) with a value of \$31 894 000.

Prices for U.S. ball clays have increased moderately over the last five years. The price range is a high of \$55 for air-floated bagged clay to a low of \$21 for shredded, bulk material. Generally, foreign sources are more expensive at FOB point of origin. All U.S. prices are expected to increase slowly, reflecting higher quality requirements and increasing costs associated with land acquisition, land rehabilitation, and environmental and energy factors.

There have been no significant new developments in processing ball clays. The processing still consists of shredding, blending, drying, and grinding. Blending is extremely important to the producer and consumer in making available blends of specific technical properties, as well as making possible better control of properties. (This also extends the reserve of important grades.)

Other forms commercially available include: shredded and semidried, air-floated, and slurry (in the U.S. and England). Air-floated production accounts for over 65% of U.S. production.

There has been a trend in recent years to use more dried clay and less shredded clay by the consumers to offset higher transportation costs. This means more air-floated and semidried production and less shredded is being produced. In processing the clay, many of the producers are moving to larger, more energy efficient mills. In producing semidried or partially dried ball clays, fluid-bed-type driers have been more popular than rotary driers from an efficiency standpoint and also in cleanliness of operation.

There has been a trend in the U.S. and more recently in England for certain industries to convert from conventional handling methods of dried clays to a liquid of slurry clay delivery. This means the ball clay industry now has mixing, screening, and storage equipment for liquid clay.

Table I. Chemical Analysis of Typical U.S. Ball Clays

	A	B	C	D	E
SiO_2	61.7%	54.7%	57.5%	54.0%	61.0%
Al_2O_3	24.9	31.0	25.9	30.7	24.5
Fe_2O_3	1.0	1.0	1.2	1.6	1.0
TiO_2	1.7	1.1	1.3	1.1	1.3
CaO	0.3	0.3	0.5	0.4	0.1
MgO	0.2	0.2	0.7	0.5	0.1
K_2O	2.1	1.2	1.6	0.2	1.7
Na_2O	0.5	0.2	0.4	0.1	0.4
LOI	7.7	10.4	10.8	11.5	9.7
Total	100.1	100.1	99.9	100.1	99.8

Ball clays and ball clay mixes are available in a wide variety of chemistries, particle size distributions, organic content, and soluble salt levels. Clays and clay blends are formulated to develop desired properties depending on the application and attempts are made to control the properties within agreed-upon realistic limits. Table I shows some chemical analysis for typical U.S. ball clays.

Because of the complex factors that have to be considered, the user needs to consider more than the chemical analysis in determining his specific needs. Consulting with a supplier can be beneficial in determining all important properties for the application. Some important physical and chemical properties include: physical properties (MOR, plasticity), casting properties (rheology), soluble salt content (SO_4), type and amount of organic matter (colloidal vs massive), and chemical analysis.

Most grades are in excellent supply. Many producers have excess capacity and are not expected to have shortages even during a full economy. More truck shipping is used presently than in the past due to the deregulation of the trucking industry in the U.S.

There is little change in ball clay technology. In general, better equipment and greater recovery are achieved by using newer, more efficient equipment. More quality control is possible today due to better knowledge of the clays in the ground, better blending after mining, and better rotation of stock.

The major U.S. markets for ball clay minerals are ceramics, pottery, and related products. The ball clay for these products is used as a plasticizing and bonding agent and must be essentially white-firing. For refractory products, the principal function of ball clays is as a bonding agent. There are some nonceramic and specialty uses for ball clay minerals, but most of this information is withheld by the clay producers as proprietary information. There are minor amounts of ball clays used in adhesives, feeds, and drilling muds according to BuMines.

Ball clay producers have several problem areas that effect their production efforts. Some of these problems include:

1. Higher overburden/clay rations.
2. Longer hauling distances from new producing mines to older producing plants.
3. Higher energy costs (diesel, natural gas, gasoline, and electrical costs).
4. More governmental regulations involving pollution control, permits, etc., required to open mines, work rules, etc.
5. Reclamation.
6. Greater emphasis and awareness by the general public of the effects of mining industry on the quality of life.

All of the above add up to higher costs to the producer and ultimately to the consumer. □

Bauxite

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This overview will be limited to calcined, refractory-grade bauxites consumed from imports into the U.S. Statistics are unavailable for domestically produced bauxites due to the limited number of producers as reported by BuMines.

The 1984 imports of refractory-grade bauxite (85% to 90% alumina range, calcined basis) totalled 189 000 metric tons. About 58% of these imports were produced in Guyana; another 40% came from the Peoples Republic of China. Both sources are large-scale mining operations with the object being premium grade calcined materials. Production and imports from Surinam have been curtailed. Five-year historical bauxite imports for refractory production are given in Table I.

Calcined bauxites continue to find application in a wide variety of high alumina refractory brick and monolithics. Usage trends are steady.

Typical technical features of major imported bauxite are shown in Table II.

Suppliers representing each major bauxite type are continually attempting to upgrade and build on the strengths of their respective material positions while optimizing production efficiencies. Availability appears to be no problem provided delivery lead times are planned in advance. Ample production capacities for refractory grade bauxites are described by the major suppliers.

Prices for calcined Guyanese material are $165 per metric ton FOB rail (Baltimore) or barge (Gulf Coast) according to *Industrial Minerals*. Chinese material runs about $120 to $145 per ton, CIFFO Burnside, depending on production method, grade, and size. □

Table I. U.S. Bauxite Imports

Year	Metric tons
1980	285 000
1981	237 000
1982	131 000
1983	147 000
1984	189 000

Table II. Commercial Bauxite Grades

	Source	
Chemistry (calcined)	Guyana	China
Alumina	88.30	87.8
Silica	6.50	6.0
Iron oxide	1.76	1.5
Titania	3.20	3.75
Alkalies	0.03	0.15
Bulk density (g/cc)	3.10+	3.10–3.30
Primary mineral form	Gibbsite	Diaspore

Beryllia

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Beryllium oxide (BeO), or beryllia, is an outstanding conductor of heat as well as an excellent electrical insulator. Because of its unique combination of thermal, electrical, and mechanical properties, its use as a ceramic material has greatly expanded. The largest use of beryllium oxide is as the raw material for the manufacture of beryllium-copper alloys, followed closely as the raw material for conversion to the metal.

BeO has been used as a ceramic material since the 1930s. Its initial metallurgical usage was for crucibles and thermocouple protection tubes because of BeO's inertness against reactive molten metals. BeO was also used in the phosphor formulation of early fluorescent tubes. With the advent of the nuclear age in the 1940s, the extremely small neutron-absorption cross section of beryllium was exploited for neutron moderation and to limit contamination in metal processing associated with nuclear technology.

During the early 1960s, beryllia's excellence as a heat sink ceramic material for electronics was recognized. Its specific heat of 1088 J/kg·K at room temperature is the highest of any oxide ceramic and is greater than that of all metals except beryllium. The thermal conductivity of beryllia is also the highest of any oxide ceramic; at room temperature the thermal conductivity of 250 W/m·K (145 Btu·ft/h·ft²·F) is about two-thirds that of copper and six to seven times that of alumina. Although its thermal conductivity decreases rapidly as temperature rises, the conductivity of beryllia at 100°C (a common operating temperature for electronic components), is still half that of copper and 10 times that of alumina. Even at 1000°–1500°C, thermal conductivity of BeO is three to eight times higher than that of alumina. Power transistors for most communications equipment and high-powered communication tubes use beryllia heat sinks to avoid overheating. Likewise, heat generated in resistor cores and substrates is rapidly dissipated, thereby minimizing hot spots and reducing temperatures of critical components. Such products are manufactured by Kyocera, Metaramics, National Beryllia Div. of General Ceramics, Inc., Brush Wellman, Inc., and NGK.

Cold-pressed-and-sintered BeO ceramics are manufactured by Brush Wellman, Inc., Consolidated Beryllium LTD, and the National Beryllia Div. of General Ceramics, Inc. High purity beryllium oxide powder is mixed with fluxing agents and grain-growth inhibitors before compacting at relatively high pressure. The moderating chemicals are commonly oxides of magnesium, aluminum, silicon, or a combination of two or three of these. The industry standard has become 99.5% BeO in the ceramic. One of the most common applications of BeO ceramics is in the electronic ignition modules of a large number of standard automobiles and trucks. In this module, the metallized BeO plate is a heat sink under the Darlington integrated circuit chip. Similar metallized BeO ceramic products for use in other electronic and electrical applications are manufactured by Ceradyne, Kyocera, Brush Wellman, Inc., National Beryllia Div., Varian, Metaramics, NGK, Coors, and others.

Because of complex configurations or special dimensions, many beryllia parts must be machined to close tolerances. Beryllia has a hardness of about 65 $R_{45\,N}$ or 9 Mohs; grinding is usually done with diamond-impregnated wheels. Lapping or polishing is usually done with diamond or silicon carbide powder. Precision BeO substrates which are polished, laser scribed, or both, are available from Laser Services, Lasermation, Accumet, Laserage Technology Corp., and P/M Industries, Inc. Grinding shops which shape precision parts from already-fired BeO ceramic stock are operated by Ceradyne, Accuratus Ceramics, San Jose Delta, Brush Wellman, National Beryllia, and Speedring.

In 1946, a chronic lung disorder found among many fluorescent tube workers in Massachusetts was described in the literature by Drs. Hardy and Tabershaw. This disease was later identified with beryllium exposure and is now referred to as berylliosis. Berylliosis is now accepted by medical opinion to be a disease of delayed immunological response. In other words, an individual must be "allergic" to beryllium to contract the disease. The experience in the beryllium and fluorescent lamp industries, where large numbers of employees were massively exposed before the toxicity of beryllium was recognized, indicates that only about 1% of those exposed contracted berylliosis.

There is no record of any ill effects resulting from ingestion of beryllia. However, the same care should be taken to avoid swallowing beryllium compounds as one would take with any other chemical compound. Beryllia ceramics utilized in their solid form for electronic applications are completely safe. The only known potential problem with beryllia is inhalation of excessive amounts of respirable beryllia (particulates less than 10 μm in size) by individuals who are hypersensitive reactors to beryllium. Respirable beryllia might be created as dust during drilling, machining, filing, crushing or other mechanical operations, as fumes and mists when metallizing, chemical etching, or wet grinding beryllia, or when chips, burrs, and slivers are pulverized into finer particles. In 1949, the U.S. Atomic Energy Commission (AEC) established three levels of maximum respirable airborne beryllium concentrations to safeguard against potential health hazards. Two covering in-plant conditions are 2 micrograms of beryllium per m³ maximum atmospheric concentrations over an 8-h day and 25 micrograms of beryllium per cubic m³ of air for periods less than 30 min. These two limits were adopted as a consensus standard by OSHA. The third AEC recommendation of an out-plant standard limiting the level to 0.01 micrograms of beryllium per m³ of air averaged monthly was established by the EPA for the immediate area surrounding a beryllium plant.

The standards adopted for airborne beryllium concentrations and the problems associated with air sampling and beryllium analysis have led to a decrease in the number of companies willing to manufacture beryllium oxide powders. The only source of BeO powder in the western world is Brush Wellman. Beryllia powder and ceramics are believed to be manufactured in the communist block countries of Poland, Russia, and China.

The utilization of BeO ceramics in the communications and electronic microcircuits continues to grow at a rapid pace in spite of the high cost of BeO over that of alumina. Raw stage BeO powder sells for $120/kg ($55/lb), FOB Elmore, OH, in quantities greater than 9100 kg (20 000 lb). The reserves of raw materials for BeO ceramic materials are quite encouraging. Since 1969, Brush Wellman has supplied its beryllium oxide requirements for alloy, metal, and ceramics from domestic deposits of bertrandite ore processed in its Delta, UT, mill. Although domestic mineral production would be capable of meeting all domestic requirements well beyond the year 2000, there is a need to rejuvenate the search for and recovery of beryl ore. Toward this goal, Brush Wellman resumed processing of beryl ore in its Delta mill in 1978. With both bertrandite and beryl processing capabilities installed, any increase in world demand for beryllium products can be met with pegmatic or domestic resources. □

Boron

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Domestic production of boron minerals and chemicals increased 7% in 1984 to an estimated 617 000 metric tons B_2O_3 (680 000 tons). Imports of colemanite, ulexite and boric acid (mostly from Turkey) were estimated at 36 250 metric tons B_2O_3 (40 000 tons) while exports of boric acid and refined borates were estimated at 235 000 product metric tons (260 000 tons). Reported domestic consumption increased 1.2% in 1984 to 313 000 metric tons (345 000 tons). All U.S. production is based in California.

The ceramic industry represents almost 60% of the domestic borate market on a B_2O_3 basis. Insulation fiber glass is the largest market segment consuming about 30% of total domestic B_2O_3 in the form of borax pentahydrate and ulexite. Textile fiber glass, the fastest growing ceramic segment, consumes about 15% of the total B_2O_3. About 10% of total B_2O_3 goes into other borosilicate glass manufacture; this market has declined recently because of pressure from imported glass. Other ceramic uses include vitreous enamels, frit, boron carbide, boron nitride and refractories. Overall market growth is expected to average 3%–4% through 1990.

The largest domestic producer, United States Borax & Chemical Corp., operates an open-pit mine at Boron, California. The adjacent refinery complex produces borax in decahydrate, pentahydrate, and anhydrous forms. Boric acid is produced in a unique direct ore processing plant. Specialty borate chemicals are produced at the firm's Wilmington, CA, plant.

Kerr-McGee Corp. recovers boron from the brines of Searles Lake. Borax decahydrate, borax pentahydrate, and anhydrous borax are produced at West End, CA; boric acid and anhydrous borax are produced at Trona, CA.

The third U.S. producer, American Borate Co., a subsidiary of Owens-Corning Fiberglas Corp., is mining colemanite (calcium borate) and ulexite/probertite (sodium-calcium borate) from an underground mine in Death Valley. Colemanite is used domestically in textile fiber glass production and ulexite is used as a raw material in making insulation fiber glass.

Etibank, the state trading company of Turkey, is the largest producer of boron minerals and chemicals outside of the United States. Etibank was to begin producing refined sodium borates at Kirka. This plant did not come on stream in 1984 and start-up operations are continuing. When completed, Etibank will be a full-line supplier with colemanite, ulexite, refined sodium borates and boric acid.

Other world producers of borates are the USSR, China, Argentina, Peru, and Chile. Several other countries produce boric acid from imported boron-containing materials.

With adequate capacities and inflation remaining under control, prices of major boron minerals and chemicals remained stable during the past year. A 3% price increase for major products was announced by U.S. Borax for April 1, 1985 and included the first increase for boric acid prices in over three years. Announced prices in dollars per metric ton for bulk railcars, FOB California are: borax decahydrate \$206; borax pentahydrate \$236; boric acid \$627; and anhydrous borax \$664.

The U.S. ceramic industry is in a favorable position for long term supply of all boron-containing materials. Current capacities are adequate and economic reserves, while not definitely known, are typically published as being at least 100 years. In any case, the life of U.S. reserves will be affected by the timing and rate of development of the sodium borate deposits in Turkey. □

Chromite

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Chromite has three principal uses, metallurgical, refractory, and chemical. Many years ago, 25 or more, each use had its own type of chromite with the types being designated according to its usage. As the refractory application is the only one strictly within the ceramic industry, this review previously could have been confined to refractory chromite. But at present, there is considerable interchangeability of the different chromite types among the uses. Therefore, in discussing the supply-demand situation, it is desirable to cover all of the wide range of chromites and the consumption patterns affecting them. The U.S. has no domestic production and is totally dependent on imports.

Table I shows the U.S. consumption of chromite by industry for the 1980–84 period.

All three categories declined, reaching a low point in 1983. In that year the long dominant metallurgical usage was number three. This was caused by enormous increase in use of imported ferrochrome by the steel industry over domestic ferrochrome produced from chromite. The increase of chromite for metallurgical use in 1984 to 108 000 metric tons (207 000 tons) was caused mainly by the conversion of government stockpile chromite to ferrochrome by way of a contract let out by the federal government. Since the ferrochrome will be returned to the strategic stockpile, the chromite consumption pattern is not normal. The second line for 1984 in Table I presents the normal distribution, excluding the special production from stockpile chromite. This line shows 1983 to 1984 increases of 67%, 32%, and 2% for metallurgical, refractory, and chemical industries, respectively. The chemical industry has emerged as the number one consumer of chromite and will likely remain so for the foreseeable future.

It should be emphasized, however, that the metallurgical industry always has been and will continue to be the largest consumer of chromium, most of which is provided by imported ferrochrome produced in countries that mine chromite.

The increase in refractory chromite consumption in 1984 was influenced substantially by the 10% increase in steel production from the previous year. A disproportionate share of the chromite increase, however, was caused by the 40% increase in open hearth steel production, as that process consumes about 10 times more chromite per ton of steel as does total steel by all processes. Open hearth steel rose to 9.1% of total steel in 1984 as compared to 7.1% in 1983. This is probably a temporary interruption in the long decline of the open hearth.

In 1984 chromite was imported from five countries. Table II shows sources

Table I. U.S. Chromite Consumption by Industry

	Metallurgical		Refractory		Chemical		
Year	Amt.[*]	%	Amt.[*]	%	Amt.[*]	%	Total
1980	520	59.2	141	16.0	218	24.8	879
1981	456	56.6	134	16.6	216	26.8	806
1982	245	49.5	73	14.7	177	35.8[*]	495
1983	58	19.7	65	22.1	171	58.2	294
1984[†]	207[‡]	44.1	86	18.3	176	37.6	469
1984	97[§]	27.0	86	24.0	176	49.0	359

[*] Thousands of metric tons. [†] Preliminary estimates for all 1984 figures. [‡] Includes G.S.A. chromite converted into ferrochrome for strategic stockpile. [§] Excludes converted stockpile chromite.

Table II. Chromite Imports into the U.S.[*]

Country	Gross weight[†]	%	Cr_2O_3 content	%	No. of grades	Avg. wt % Cr_2O_3
South Africa	201 822	78.4	92 914	81.4	3	46.0
Philippines	36 989	14.4	12 961	11.4	2	35.0
Finland	10 186	3.9	4 482	3.9	1	44.0
New Caledonia	3 150	1.2	1 764	1.5	1	56.0
Canada[‡]	5 408	2.1	2 047	1.8	3	37.9
Total	257 555		114 168			

[*] First 11 months, 1984. [†] Metric tons. [‡] Transshipment from a producing country.

and amounts of imports.

It is seen from the table that South Africa is by far the largest source of chromite, 78%, with the Philippines as second. The two countries supply 93% of the total imports. The chromite imports of Table II actually represent a minor portion of the total imports of chromium. In the same 11-month period shown in Table II, there was 2.6 times more chromium imported in ferrochrome. In this period 202 484 metric tons of chromium contained in ferrochrome and 78 119 metric tons of chromium contained in chromite were imported. This translates to 72% of the chromium from ferrochrome and 28% from chromite. Ferrochromium is imported from 15 countries. But only five countries supplied 93% of the total. These are: South Africa, 62%; Zimbabwe, 12%; Turkey, 9.2%; Yugoslavia, 6.3%; Brazil, 3.7%. South Africa supplied 67.4% of total chromium from chromite and ferrochrome.

Prices for chromite remained flat for most of 1984. EMJ market quotation for Transvaal 44% Cr_2O_3, no ratio, chromite was $56 to $61/metric ton ($51 to $55/ton) for January-February and $53 to $57/metric ton ($48 to $52/ton) for the remainder of the year and January, 1985. Turkish 48% Cr_2O_3, 3:1 Cr:Fe ratio, was quoted at $121/metric ton ($110/ton) for the same period. Actual prices for chromite imported during the first 11 months of 1984 ranged from $40.53 to $113.34 per metric ton FOB port of shipment. The weighted average price of total chromite imports in the same period was $55.91 per metric ton. The wide range in prices attests to the need for special grades commanding premium prices. For example, Philippine high alumina grade with only 33% Cr_2O_3, sold for $106.79/metric ton, while high iron South African with 44% Cr_2O_3 brought the lowest price of $40.53/metric ton. Another Philippine grade with low iron and 59.5% Cr_2O_3 had the highest price of $120.56/metric ton, but this chromite comprised only 1.1% of the total. Physical properties, such as grain size and hardness, as well as chemistry, affects the price. Prices of chromium in ferrochrome remained constant during 1984. These materials have been caught in the great metals-minerals commodities recession that has been going on for about three years, much longer for chromite.

Chromite usage in refractories declined very steeply from the 1970s and from as recently as 1981. The decline from 1981 to 1983 was 51%. In that period, raw steel production, a major user of refractory chromite, declined only 31%. Open hearth steel production, which uses the most chromite per ton of steel, declined 56% in that period.

It appears that the downtrend in refractory chromite consumption has flattened out and will probably continue in the present range for a number of years. One reason for this is that the greatest erosion of use in the steel industry has occurred, although it is not completely over in the U.S. Another reason is that chromite usage in nonsteel industries now accounts for a larger percentage of the total than formerly. These uses have remained more or less the same as a percent of the output of these industries.

Supplies remain very large in South Africa and Zimbabwe. Reserves in the other countries from which the U.S. obtains chromite, while small in comparison to southern Africa, are adequate for a number of years. □

Dolomite

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Dolomite is a double carbonate of calcium and magnesium ($CaMg(CO_3)_2$) which theoretically contains 54.3% $CaCO_3$ (30.4% CaO) and 45.7% $MgCO_3$ (21.8% MgO), or a rock that is composed mainly of the mineral dolomite along with a certain percentage of impurities. These impurities are typically silica, iron, alumina, sulfur, and phosphorous. Dolomite that is used for ceramic purposes is usually of high purity. A high purity dolomite is generally considered to be one that contains at least 98% total carbonates (over 43% $MgCO_3$) and less than 2% impurities.

The reserves of dolomite in the U.S. and Canada are extremely large, although high purity deposits are restricted to certain geographical areas, and many deposits have been and continue to be lost due to restrictive zoning regulations, urban development, and the continuing public opposition to mining in general. The largest reserves of high purity dolomite are contained in the Niagara Group in the states of the Great Lakes region, notably Ohio, Indiana, Illinois, and in the Canadian province of Ontario. Other less extensive deposits occur scattered throughout many other states. Reserves of dolomite for construction material and reserves of high purity dolomite for most applications should be more than adequate for many years to come, although some deposits more remote from market areas may have to be considered.

The exact tonnage of dolomite produced in the U.S. is difficult to determine, but BuMines is again attempting to differentiate between limestone and dolomite in its annual statistics. According to BuMines, in 1983 (which is the last year for which complete statistics are available), dolomite was produced exclusively in 84 quarries. These quarries produced an estimated 22 million metric tons (24.2 million tons). An additional 46 quarries produced both limestone and dolomite without indicating the volume of each. A total of 61 companies in 23 states was involved in the production of dolomite. Leading states in dolomite production include New York, Pennsylvania, Michigan, Tennessee, and Virginia.

In 1983, 28 plants in 13 states produced dolomitic quicklime or dead-burned dolomite. The total lime production in 1984 is estimated to be approximately 14.4 million metric tons (15.9 million tons): of this amount, about 20% or around 2.9 million metric tons (3.2 million tons) was dolomitic including approximately 517 000 metric tons (570 000 tons) of dead-burned dolomite. This represents nearly a 6% increase in total lime production, and over a 20% increase in refractory dolomite production over the 1983 figures.

Approximately 1% of the total lime production and over 1.2 million metric tons (1.3 million tons) of crushed limestone and dolomite were used to manufacture glass. The amount of dolomite used for other ceramic applications; although important, make up a relatively minor tonnage. These uses include the production of refractory dolomite brick, a bonding agent in silica brick, and other specialized refractories in the process to produce periclase from sea water or brine, and as a filler or flux in the production of some whiteware and pottery. Other major nonceramic applications of dolomite include agricultural stone, flux stone, filler material, and for environmental applications.

Prices of dolomite are highly variable depending on the proposed application and the degree of processing of the material required. Prices may vary from a few dollars per ton for crushed dolomite used for construction purposes, to hundreds of dollars a ton for special varieties of dead-burned dolomite.

Although some rationalization of the industry is still occurring, 1984 was a relatively stable year after the rash of plant sales and shutdowns that occurred in 1983. Efforts are still continuing in order to reduce the cost of processing dolomite, especially in regard to optimizing kiln efficiencies, and research continues in order to improve the quality of various ceramic products manufactured from dolomite. This is in response to a more competitive marketplace, and more strict customer specifications. It is anticipated that 1985 should be on par with 1984, with some slight improvement possible. No supply problem can be seen for dolomite, as the U.S. is, and should continue to be, self-sufficient in this commodity. □

Feldspar

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Feldspar is the name given to a group of minerals which are the most abundant constituents of the igneous rocks. Feldspar is found in pegmatites and granites throughout the U.S. The principal types of feldspar are orthoclase and microcline (the K-Spars), albite (NA-Spar), and anorthite (CA-Spar). All of these types are found in varying ratios in the granites and pegmatites exploited for commercial production of feldspar in the U.S. Potash spar refers to feldspar which is predominantly orthoclase or microcline (with a K_2O content of 8% or higher). Soda spar describes feldspar which is predominantly albite (usually containing about 7% Na_2O).

Of the more than 40 countries around the world which produce feldspar, the U.S. is the largest. Production in the U.S. of feldspar and feldspar-silica (figures for feldspar-silica mixtures reflect feldspar content only) mixtures in 1984 was estimated at 644 000 metric tons (710 000 tons) with a production value of $23 million, according to preliminary statistics from BuMines. 1984 results were comparable to those of 1983, with 644 000 metric tons (710 000 tons) valued at 22.5 million. Potash feldspar (K_2O content 8% or higher) production for 1984 was estimated at 65 000 metric tons (72 000 tons) valued at $3.7 million, and was an improvement over 1983, 59 200 metric tons (65 200 tons) valued at $4.2 million. Exports accounted for an estimated 9072 metric tons (10 000 tons) in 1984 vs 8490 metric tons (9360 tons) in 1983.

The improved economic conditions during 1984 maintained the demand for feldspar and related materials experienced in 1983. Strong demand for housing and commercial construction further increased the requirement for pottery plumbing fixtures, tile, and structural clay products in which feldspar is used. Fiber glass production was up with the continued improvement in construction, but further decline in glass container production resulted in reduced demand for feldspar in 1984, which offset gains in the other areas.

Feldspar for glassmaking is ground to nominal 20-mesh and 40-mesh, depending on the application. Feldspar provides alumina and soda ash for the glass batch, which improves the melting and forming characteristics of the glass. Among the end uses are glass containers, fibrous glass, TV picture tubes, specialty glass, and art glass.

Feldspar is the most widely used fluxing agent for ceramics, and can be found in formulations for both bodies and glazes, as well as for enamels and frits. End uses include pottery plumbing fixtures, wall and floor tile, dinnerware, electrical porcelain, and artware. Feldspar has also been found to be very suitable as an inert extender/filler in latex foam, paints, caulks, and plastics, and as an abrasive agent in cleanser compounds. End use distribution in 1984 of feldspar was estimated at 58% for glass, 38% for ceramics, and 4% for other applications, including enamels and fillers.

The major producing states were (in order of volume): North Carolina, Connecticut, Georgia, California, Oklahoma, and South Dakota, with the top three providing 90% of the output. Primary producers of feldspar and feldspar-silica mixtures are shown in Table I.

By-product and co-product producers of feldspar and/or feldspar/silica mixtures are shown in Table II.

The outlook for 1985 is for production volume somewhat better than in 1984 on the strength of continued stability in housing and construction related markets. Overall decline in demand for glass grade feldspar is expected to be offset by increased demand for feldspar for ceramic and filler applications. The U.S. government estimate for production in 1985 is 653 000 metric tons (720 000 short tons). □

Table I. Producer of Feldspar and Silica-Feldspar Mixtures in the U.S.

Producer	Location	Facilities
Feldspar Corp.	Spruce Pine, NC	Mine and plant
	Middletown, CT	Mine and plant
	Monticello, GA	Mine and plant
Indusmin, Ltd.	Spruce Pine, NC	Mine and plant
Imcore Div., International Minerals & Chemical Corp.	Spruce Pine, NC	Mine and plant
Feldspar Div., Pacer Corp.	Custer, SD	Mine and plant
Calspar Corp.	Santa Fe Springs, CA	Mine and plant

Table II. By-Product and Co-Product Producers of Feldspar and Feldspar-Silica Mixtures in the U.S.

Producer	Location	Facilities
Arcola Sand & Gravel Div. of Ashland Oil Co.	Muskogee Co., OK	Dredge and plant
Foote Mineral Co.	Kings Mountain, NC	Mine and plant
Kings Mountain Mica Co.	Kings Mountain, NC	Mine and plant
Ottawa Silica Co.	Crystal City, CA	Mine and plant
Owens-Illinois Glass Co.	Mission Viejo, CA	Mine and plant
Spartan Minerals Div.	Bessemer City, NC	Mine
Lithium Corp. of America	Pacolet, SC	Plant

Fluorspar

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Consumers and producers experienced a good year for fluorspar in 1984; 1985 looks to be just as good.

Consumers have been treated to adequate supplies of fluorspar as well as stable if not lower prices for the entire year.

Producers have seen increased sales not only reduce some very large stockpiles that accumulated over the last several years of recession but also increase their operating rates from 40%–45% to the 95%–98% range. There are a few small remaining producer stockpiles, but these will shortly be worked off.

Supplies may tighten by the end of 1985 but we do not see any shortages on the horizon. Although fluorspar consumption has grown with the economic recovery in the past year and a half, it has not yet reached its prerecession level, partly due to the decline of raw steel production, some EPA considerations, and the general deindustrialization of the U.S.

There remains only one domestic mine/mill to supply an acid grade fluorspar for the ceramic industry; however, that facility exists primarily as a supplier of acid grade fluorspar to its parent company for the production of hydrofluoric acid and downstream compounds. Only 15%–20% of its production is sold to the general commercial market. The other large domestic mine has been closed for two years on a standby basis; it has now stopped the maintenance of the underground workings.

The high valued dollar has been a major factor in the closing of this mine and other industrial mineral mines in this country. The following change in ratio of (fluorspar-producing) foreign currencies are shown in Table I.

Table I. Rates of Exchange* Affecting U.S. Fluorspar Production

Currency	Year 1980	 1985
Rand	1 to 0.75	1 to 2.2
Peso	1 to 12.5	1 to 225
Peseta	1 to 90	1 to 175

*Ratio of dollar to . . . .

Ceramic fluorspar which is a 90%–95% CaF_2 product is generally not available, as it has been replaced by acid grade fluorspar. Due to the U.S. Tariff Code, less than 97% CaF_2 has an advolorum duty of 13.5% vs a $2.10 gross ton duty for over 97% (acid) acid (or a difference of from $10 to $12 a ton). Almost all dried fluorspar shipped to ceramic users is now over 97% CaF_2. Small amounts of ceramic or subacid grade are imported from Mexico, where poor metallurgy or complex ores prevent raising the grade to the U.S. Tariff grade of plus 97% CaF_2.

Total U.S. production of fluorspar in 1985 should be in the range of 39 000–68 000 metric tons (65 000–75 000 tons) of acid grade whereas apparent consumption will be from 680 000–817 000 metric tons (750 000–900 000 tons) of all grades. Imports from South Africa should be in the range of 154 000–172 000 metric tons (170 000–190 000 tons); acid plus some subacid grades from Mexico should be 200 000–245 000 metric tons (220 000–270 000 tons), from Spain 18 000–27 000 metric tons (20 000–30 000 tons) and perhaps as much as 14 000 metric tons (15 000 tons) from Italy. Most of this acid and subacid tonnage will be consumed by hydrofluoric plants and the balance will go into ceramics, steel, welding rods, abrasives, and metallurgical fluxes.

Wet concentrate prices, world-wide FOB vessel, for 1984 were in the range of $95 to $100 per metric ton. Ocean rates for 30 000 or 40 000 ton vessel lots have been very competitive and in some cases fluorspar bulk shipments have gotten "ballast" rates for ships coming to U.S. ports for grain cargos. The excess vessel capacity in the world should continue at least through this year maintaining the low vessel rates to U.S. ports. Most present nonoperating domestic fluorspar producers would need price increases in the range of $44/metric ton ($40/ton) to motivate them to restart their mines and mills.

Mexico's effort to form a cartel totally collapsed in 1982–83 when buyers resumed control of the market; as a result Mexico lost market share that they have not, and probably won't, recover. South Africa took over a large part of that market with Spain and Italy following, based on good price, good grade, and on-time deliveries.

China keeps threatening to be a force in the market but they haven't seemed to be able to prove consistency of delivery even though their grade and price have been good. Until China can improve its domestic infrastructure and reliability it will only be an occasional part of the fluorspar picture in the U.S.

Fluorspar has suffered from the environmental reaction to fluorine; the worst example was the fluorocarbon/ozone theory several years ago that destroyed a propellent market that was growing at the rate of 7% per year. Fluorine reduction projects in the glass and ceramic industry are working for 25% to 50% or more reductions in fluorine. For those industries continuing to use fluorspar, their specification for arsenic, phosphorus, mercury, and other possible hazardous residuals have tightened considerably, so that producers are constantly improving their techniques for purity to maintain their market share.

World reserves of fluorspar have remained fairly constant, as reduced consumption has practically eliminated the need for further exploration. Total operating mines have been reduced worldwide and there have not been any new large mines brought into production for over ten years.

Based on present and low projected future consumption, the present foreign sources of fluorspar are equipped to provide the U.S. industries with sufficient quantities of fluorspar for some years to come. □

Graphite

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Natural graphite, a strategic industrial mineral, sometimes referred to as "plumbagos" or "black leads," is a soft, black, natural-occurring form of carbon which varies in hardness from 1 to 2 on the Mohs scale and has a specific gravity between 2.1 and 2.3 g/cc. It is resistant to attack by ordinary chemical agents and is chemically inert and stable at ordinary temperatures. Natural graphite is an excellent conductor of heat and electricity and will sublime at approximately 3500°C. It is furthermore characterized by an extremely low coefficient of friction and thermal expansion. This is primarily due to the atomic arrangement of the base structure of the graphite crystals. The crystals being held together both by van der Waal forces and covalent bonding.

The classifications of natural graphite fall into three general groups. They are flake, veined (high crystalline), and amorphous graphite. The basis for variant modes of formation are contributed to both the base raw material and to the method in which the graphite was formed. The differences being both in physical properties and appearance as well as chemical composition.

Flake graphite consists of flat platelike particles that occur in a disseminated form through layers of regional metamorphosed silica-rich sedimentary rocks such as quartz and mica schists, felspathic or micaceous quartzites, gneisses, and marbles. The particle size can vary greatly depending on the nature of the formation. Commercial importance of each deposit will vary considerably, depending on the carbon content and the size of the flake.

Vein graphite (high crystalline) deposits are found in fissures, fractures, or other cavities, transverse, igneous, or metamorphic rock. This form is typically massive and the size of the particle can vary from fine grain to large lumps. Minerals that may be present as impurities include feldspar, quartz, mica, zircon, rutile, apatite, and iron sulfides. The vein deposits exhibit wide variation in width from a few millimeters to over two meters. There has been some debate over the formation of these deposits which display a high degree of density and toughness as well as high crystallinity in respect to the *d* spacing.

Amorphous graphite is formed by metamorphism of coal seams or carbon-rich deposits which have been exposed to extremely high pressure and moderate temperatures. This particular type of graphite has a soft black earthy appearance in contrast to the crystallized graphites which have the striking metallic luster. In fact, the term "amorphous" is not strictly correct in this case at all, due to the fact that this graphite has small crystallites when examined under a microscope. The more appropriate term would be "microcrystalline graphite."

In common the vein, flake, and amorphous deposits vary considerably in size and purity. The purity is dependent upon the type of material it was before metamorphosis and determines to what application the graphite is eventually put. For example, the low grade Korean graphite is often used in industrial fuel because it is high in nongraphitic carbon.

The importation of natural flake graphite has shown a steady increase in all areas except the battery industry. The total imported natural flake graphite to the U.S. exceeded 18 000 metric tons in the year 1983 and is expected to grow at approximately 5% for the next several years, but the long term growth rate is expected to decrease to somewhere in the 3% to 4% range.

Although in 1979 a major change oc-

Table I. Major U.S. Imports of Natural and Artificial Graphite Powders (Metric Tons)

	Year					
Country of Origin	1979	1980	1981	1982	1983	1984[*]
Brazil[†]	2 097	4 863	3 585	4 720	3 233	3 750
Canada[†]			2 839	250	4 825	5 500
China[†]	2 449	3 342	3 926	7 640	5 368	5 800
Germany[†]	1 965	958	836	1 006	1 368	1 560
India[†]	60	55	458	392	425	415
Madagascar[†]	3 165	2 571	4 015	4 169	3 367	3 070
Russia	4 866	1 954	620			
Sri Lanka[‡]	2 022	1 223	919	1 195	743	760
China[§]	2 695	2 220	5 042	5 470	4 307	12 500
Korea[§]	11 574					
Mexico[§]	46 382	36 736	35 879	28 441	26 335	22 560
Switzerland[∥]	1 316	1 699	1 796	1 959	2 555	2 765

[*] Estimate. [†] Flake, price range: $200–$3500 per metric ton (FOB); media: $929. [‡] Price range: $500–$1500 per metric ton (FOB); media: $1025. [§] Amorphous, price range: $60–$120 per metric ton (FOB); media: $929. [∥] Synthetic, price range: $1650–$3300 per metric ton (FOB); media: $2475.

curred in the importation of natural graphite, due to the decrease usage in the battery industry, the overall requirements have constantly increased due to recent developments in the refractory industry, both in the magnesite refractory and in the high alumina developments. It would be good to mention at this time that two new deposits have been reported in the last two years, one of them in Canada and the other one in China. Neither one of these deposits is presently being mined, but as the demand increases, these will start operation.

The importation of vein graphite (high crystalline graphite) in the U.S. has declined during the last several years. In 1982 1195 metric tons were imported, but in 1983 it declined to 743 metric tons. The decline has been primarily due to the low quality, erratic shipments and increase in price. It is due to the above factors that the majority of the U.S. consumers spent monies on research to develop new products without vein graphite.

In many research programs it was discovered that a superior product could be made with flake graphite at considerable savings.

It appears that the general decline will continue in the usage of vein graphite over the next several years or until the prices are put into proper perspective. Since vein graphite has a single source, Sri Lanka, it is not considered a desirable graphite for the development of new products. There will be more than an adequate supply until the year 2000 and the Sri Lanka mining company continues to search for new deposits and tries to upgrade its process.

Amorphous natural graphite has maintained its long historical standing as being the major volume material of the U.S. and the worldwide natural graphite consumption. The major uses for amorphous graphite are in the hot metal industries.

The general decline in the usage which has occurred in the last year has been primarily due to the lack of production in the hot metal industries. This setback is only expected to be a temporary situation, and new developments in refractories will add to the increase in the consumption during the next several years. The one factor that makes amorphous graphite very acceptable in the hot metal and refractory industry is the fact that it is one of the cheapest graphitic materials on the market. It would be well to point out that there are various grades of amorphous graphite according to particle size and carbon content.

In Table I, four major categories have been listed on natural graphite and synthetic graphite powders which were imported into the U.S. Also included in this table is the general price range for each of these types. One must consider the fact that carbon content, type of graphite, and size of graphite are all factors which can affect the cost. It can easily be seen from Table I that there has been a general decline in the total usage of graphite since 1981, but 1985 projections will almost equal 1981 consumption. It can be also easily seen from Table I that as one source seems to disappear another one appears. Note that we no longer import graphite from Russia, primarily due to trade relations; but the new operation in Canada more than offsets what was lost from Russia. It can also be seen that the market area is constantly growing for the synthetic high purity graphite powders. The major consumers for the synthetic high purity powders are the battery industries.

In summary, there are three major uses of natural graphite: refractories, hot metals, and pencil industries. Other applications consist of batteries, powdered metals, plastics, magnetic tapes and disks, carbon brushes, seals, gaskets, conductive coatings, paints, and friction products. The general growth in all uses of natural graphite is expected to be in the range of 3% to 5% over the next five to ten years.

With respect to the world supply there appears to be an abundance of all types with the exception of vein (high crystalline graphite—Sri Lanka) graphite. This does not mean shortages will not occur in certain types, due to development in new products. The supply line on the sources is very long, and many times new products can be on the market before mining operations can increase their production. As graphite is essential to a number of industrial processes, the demand is dependent upon the worldwide industrial activity.

The major industries which import and refine natural graphite in the U.S. are: Asbury Graphite Mills, Inc., the largest processor and its subsidiaries throughout the U.S., Canada, and Mexico; Superior Graphite Co. has processing plants in Chicago and Kentucky and mines graphite through its affiliates in Mexico; Southwestern and Dixon also have plants in the U.S. and import and process natural graphites in Texas and New Jersey. □

Kyanite

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Kyanite, Al_2SiO_5, is a mineral widely used in the refractories, glass, and ceramic industries. Most kyanite is used to make mullite refractories which cost more but have a much longer life than fireclay refractories under similar conditions. The U.S. has huge reserves of kyanite ore with proven workable kyanite-quartzite deposits containing 10% to 30% recoverable kyanite exceeding 91 million metric tons (100 million tons). The U.S. produces and consumes more kyanite than any other country.

Kyanite and the related aluminum silicate minerals, sillimanite and andalusite, became important during World War I with the critical need for porcelain spark plug insulators for airplane engines. Almost simultaneously, their value as raw materials for the production of mullite and mullite refractories was recognized. By 1925, four domestic mines were in operation, but slow development of the mullite refractories industry and competition from imported Indian kyanite eventually forced most of these mines to close. Since 1955 only two mines have produced kyanite in the U.S.: Kyanite Mining Corp., in Virginia, and C-E Minerals, in Georgia. Through competition between these two companies, the price of domestically produced kyanite has enabled the U.S. to change from a major importer to a major exporter over the past 20 years. Kyanite was removed from the list of strategic materials for stockpiling by the U.S. government in March 1970.

Kyanite is an important refractory raw material because: 1) it provides an Al_2O_3 content of 55% to 60%, 2) it expands irreversibly upon heating, 3) it decomposes to mullite at high temperatures, and 4) it does not melt until about 1800°C. In order to be commercially useful, kyanite ore must be crushed and extensively beneficiated to obtain a product that contains over 55% Al_2O_3 and below 1% Fe_2O_3. Kyanite is marketed in size ranges of 35, 48, 100, 200, and 325 mesh (Tyler). Calcined kyanite (mullite) is also produced in similar size ranges.

Approximately 75% of the kyanite consumed in the U.S. is used to make refractories for the metals industry; an additional 15% is used in the glass industry, with the remainder being used in whitewares, such as spark plug insulators, catalyst substrates, dinnerware, sanitary ware, etc. Statistics on the production and consumption of kyanite in the U.S. are not made public because of the competitive postures of the two producing companies, but estimates suggest that 91 000 metric tons (100 000 tons) of kyanite are consumed and about 136 000 metric tons (150 000 tons) are produced annually in the U.S. at this time. Obviously significant quantities of kyanite are exported every year. The U.S. is the major supplier of kyanite to the world market.

India has produced and exported kyanite for many years, but a recent decision by the Indian government states that it does not intend to permit future export of refractory-grade minerals such as kyanite. Brazil has begun to market kyanite and will probably increase production somewhat in the near future. It has also been reported that a kyanite deposit in Bulgaria is in the process of being developed with significant production expected in several years.

The major producer of commercial kyanite in the U.S. (and the world) is Kyanite Mining Corp. operating the East Ridge and Willis Mountain Mines in central Virginia. Their production capacity is not public knowledge, but they have recently completed a new plant that increases their capacity greatly. The other producer of kyanite is C-E Minerals, which operates the Graves Mountain mine in northeast Georgia.

It is interesting to note that kyanite consumption in the U.S. has decreased slightly in recent years. Apart from the recession, this is primarily a result of the process change to continuous casting in the steelmaking industry, a change that requires fewer refractories. New applications using kyanite, such as volume stable monolithic mixes, investment casting molds, brake linings, welding rod fluxes, ceramic fiber blankets, flex insulating blankets, etc., generally use small volumes and do not offset the decline in steel usage.

The main competition or substitute for kyanite is andalusite, primarily produced in South Africa. At this time, production and shipping costs prevent andalusite from being cost-competitive with kyanite in the U.S.; however, it does appear to be cost-competitive in Europe, Asia, and the Far East. There are significant differences in andalusite and kyanite that may overshadow their price differences. Andalusite, for example, is available in coarse grades up to 1.27 cm (0.5 in.), whereas kyanite is not available coarser than 35 mesh. Andalusite does not expand irreversibly upon heating as kyanite does. There appears to be a trend toward mixing kyanite and andalusite where possible for many refractory applications, especially in the area of unfired monolithic mixes.

Extensive research in mining, beneficiation and product development of kyanite and kyanite-containing products has been conducted over the years, primarily by BuMines, National Bureau of Standards, and U.S. Geological Survey. In recent years all these programs have become essentially inactive. There continues to be the need for research, especially in developing improved methods of beneficiation, quality control, and development of techniques to make volume stable mullite grog. □

Lead Products

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Lead may well be the first metal to be smelted and used by ancient man. Artifacts made of lead are dated as early as 6500 B.C. The use of lead oxides in glass and glazes dates at least to the period from 2000 B.C. to 1600 B.C., while its use in enamels for application to glass and metals may date from the first century B.C.

The largest single use for lead is in the manufacture of storage batteries of all types. This industry uses, in an average year, about 60% of all lead produced. While this lead is used, it is not consumed. Most of the lead used in storage batteries is recovered as secondary lead and is used again. This is one reason why any fear that we may run out of lead in the foreseeable future is unfounded. There are other reasons as well.

The U.S. is the world's leading producer of primary lead. Australia, Canada, Peru, Mexico, and Yugoslavia follow in that order. The latest year for which U.S. production figures for pure refined lead are available is 1983 (see Table I).

Production of antimonial lead and other alloys make the grand totals of 1 019 000 metric tons (1 123 000 short tons) in 1983. Recovery of secondary lead in the U.S. reached a high of 801 368 metric tons in 1979, but fell to 675 578 metric tons in 1980. Figures for 1981 and 1982 will show further declines because of the recession. U.S. production of lead is about 20% of the world total. Table I shows the effect of the recession on both the demand for and the supply of lead.

Both production and price of lead metal and lead products have been highly variable in the past few years (Table II). With reduced demand, prices have fallen from an all-time high of $1.39/Kg ($0.63/lb) in October 1979 to $0.40/Kg ($0.18/lb) for a brief period in January 1985, with frequent ups and downs between those extremes. Many factors affect the price of lead, chief of which are supply and demand. World politics which result in wars, or such inconveniences as the closing of the Suez Canal a few

Table I Lead Production (Pure Refined) U.S.*

Year	Metric tons	Short tons
1972	648 160	714 463
1973	691 537	762 277
1974	646 278	712 388
1975	581 869	641 390
1976	597 276	658 374
1977	555 233	612 030
1978	517 831	629 191
1979	581 617	461 113
1980	550 087	606 358
1981	501 897	533 238
1982	512 817	565 288
1983	515 203	567 913

*Source: Minerals Yearbook; BuMines, 1983.

Table II Lead Supplies in the U.S.* (1983)

	Metric tons	Short tons
Mine production, metal content of ores	419 038	494 979
Refined primary lead	515 203	567 913
Secondary lead	503 501	555 104
Imports:		
Refined lead	176 000*	194 006
In ores and concentrates	20 000	22 046
Consumption, U.S.	1 148 487	1 265 989

*Includes 42 000 metric tons of U.S. brands returned from the London Metal Exchange.

years ago, have their effect. Strikes or smelter closures force prices up by interfering with supply. Business recession drives prices down by cutting demand. Long range prediction of price trends, with any hope of accuracy, is difficult, if not virtually impossible.

Lead monoxide (litharge), or its equivalent in the form of lead silicates or frits, is the principal lead product used in ceramic industries (Table III).

Glass products represent the greatest usage. Glass for television, lighting, various electronic products, radiation absorbing glasses, optical and ophthalmic glasses, and tableware are all substantial consumers of lead compounds. While from year to year the quantity is variable, it can exceed 91 000 metric tons (100 000 tons) of world consumption in a single year. Lead oxides for glazes for dinnerware and for floor and wall tile are substantial consumers of lead products. Some of this is provided in the form of lead silicates of very low solubility and some is supplied by the manufacturers of frits and colors. Some of these frits have very low solubility and thus present little hazard to the workers.

Dry process enamels, used largely on sanitary ware such as bathtubs, also account for a substantial usage of lead products. This use is directly related to the rate of housing construction and rehabilitation and therefore is quite variable.

Lead compounds and compositions that are soluble in body acids such as gastric juice are toxic if ingested or inhaled in quantity. The average person, not industrially exposed, is not at risk from the normal environment. Much work has been done to assure safe foodware of all types. Concern for the industrial worker has resulted in development of safe lead compounds and frits which are not absorbed significantly and pass through the body if ingested or inhaled. Engineering controls in the plants also are designed to minimize or remove the chance of exposure in the plant. The results of this effort to provide safe work conditions in the plants are shown by the near-absence of lead poisoning symptoms in the ceramic industries since World War II. In Britain, the potteries industry reported as many as 450 lead poisoning cases per year in the early part of this century; there have been none reported since World War II. □

Table III. Litharge Used in the U.S.* Ceramic Industry

Year	Metric tons	Short tons
1979	37 620	41 469
1980	36 560	40 300
1981	34 732	38 285
1982	30 980	34 150
1983	37 143	40 943

*All data are provided by BuMines Minerals Yearbook: 1983.

Lithium

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Continued improvement was noted in the world consumption of lithium chemicals and metal during 1984 (See Table I). It is estimated that in 1984, 27 million kg (60 million lb) of lithium carbonate equivalents were consumed vs approximately 24.4 million kg (53.7 million lb) in 1983. Demand by the traditional consuming industries strengthened during the year.

During 1984, lithium chemicals continued to be produced in the U.S. by Foote Mineral Co. (a subsidiary of Newmont Mining Corp.) and Lithium Corp. of America (a subsidiary of Gulf Resources & Chemical Corp.). Chemetall (a wholly-owned subsidiary of Metallgesellschaft AG) of West Germany and Honjo Chemical of Japan, as well as the USSR and the Peoples Republic of China, also continued lithium chemical production. Lithium ore production continued in the U.S., Brazil, Australia, and Zimbabwe.

Lithium Corp. of America has an open-pit mining operation near Bessemer City, NC, where it produces spodumene ore as feed for its chemical plant in Bessemer City. A complete line of lithium chemicals and metal are manufactured. The announced capacity at this facility is 16 million kg (36 million lb) of lithium carbonate equivalents annually. Lithium Corp. of Europe, a wholly-owned subsidiary of Lithium Corp. of America, located near Liverpool in the U.K., produces butyllithium and lithium metal.

Foote Mineral Co. has two sources of lithium in the U.S.—an operation at Kings Mountain, NC, where lithium carbonate is produced from spodumene, and at Silver Peak, NV, where lithium carbonate is produced from brine. Other downstream lithium chemicals are produced at Sunbright, VR, New Johnsonville, TN, and Frazer, PA. Foote Mineral's U.S. operations have a total capacity of 13 million kg (34 million lb) a year of lithium carbonate equivalents.

Sociedad Chilena de Litio Ltda. (SCL) began commercial production of lithium carbonate in July 1984. The feed material is supplied from a brine deposit located in the Salar de Atacama in northern Chile. This material is transported to a chemical facility located near the port city of Antofagasta. The initial capacity of this plant is rated at 6.4 million kg (14 million lb) of lithium carbonate annually. SCL is a joint venture, owned 55% by Foote Mineral Co. and 45% by the Chilean government development corporation, Corporacion de Fomento de la Produccion (CORFO).

Chemetall of West Germany produces a variety of lithium chemicals at its Langelsheim operation and is the largest producer of secondary lithium chemicals in Europe. The principal producer of downstream lithium products in Japan is Honjo Chemical Corp.

Higher internal consumption of lithium values is believed to be the principal reason for the lower level of exports from the USSR during 1984. The Peoples Republic of China had a lower level of activity with exports of lithium carbonate and lithium hydroxide in 1984 vs the previous year.

Lithium chemical prices rose by approximately 5% at midyear. The price trend over the past few years has roughly paralleled the rate of U.S. inflation.

Lithium carbonate and lithium ores continued to be used in black-and-white television tubes and in the production of glass/ceramics.

Research efforts continue on programs utilizing lithium to produce glass that will decrease or contain fuel and

Table I. Estimated 1984 Western World Consumption Lithium Metal and Chemicals*

Market	Kilograms	Pounds
North America	11 300	25 000
Western Europe	6 400	14 000
Far East	4 500	10 000
South America	2 500	5 500
All other countries	2 500	5 500
Total	27 200†	60 000

*Thousands of kg and lb of Li_2CO_3 equivalents.
†5116 metric tons Li.

manufacturing costs. Chemical boosting of glasses with lithium can result in increased capacity by as much as 10% with only minor adjustments to the batch composition. Other advantages are decreases in temperature and refractory erosion.

The overall consumption of lithium values in the western world in 1984 increased by approximately 11% vs 1983. In 1985, consumption is projected to improve by an estimated 10%. The additional 6.4 million kg (14 million lb) of annual capacity that came on stream in midyear in Chile assures the consuming industries of long-term availability of lithium. ☐

Magnesia/ Magnesite

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Magnesium oxide (MgO) is the most important product of the magnesium compounds industry. It occurs naturally as the mineral periclase, but those occurrences are rare and of no commercial value. The principle sources of magnesium oxide for commerce are magnesite ($MgCO_3$), dolomite ($CaCO_3 \cdot MgCO_3$), seawater, and magnesium-rich brines.

Major deposits of magnesite occur in Austria, Brazil, Canada, China, Czechoslovakia, Greece, India, North Korea, U.S., USSR, and Yugoslavia. The ores contain varying amounts of silica, iron, alumina, and lime and are usually mined selectively and often beneficiated. Beneficiation methods vary according to location but can include crushing and size separation, hand sorting, optical sorting, magnetic separation, heavy media separation, and froth flotation. Crude magnesite has very limited application and the bulk of it is used for the production of magnesium oxide.

The most important commercial process for the production of magnesium oxide from seawater and brines takes advantage of the fact that magnesium hydroxide $MgO(OH)_2$ can be precipitated from solutions of magnesium salts by the addition of a strong base. The magnesium hydroxide is washed, thickened, filtered and then calcined to produce magnesium oxide. Calcined limestone (CaO) or calcined dolomite [$CaO \cdot MgO$] are usually used as the source of alkali. If calcined dolomite is used, half of the magnesium hydroxide in the precipitate comes from the dolomite. In another commercial process, concentrated magnesium chloride brine is sprayed into a thermal reactor where hot gases convert it to magnesium oxide and hydrochloric acid. The magnesium oxide is slurried and forms magnesium hydroxide, which is washed, filtered and calcined to produce magnesium oxide.

In the U.S., the main source of magnesium oxide is seawater and brines. Other countries which are major producers of magnesia from seawater and/or brine are Ireland, Israel, Italy, Japan, Mexico, UK, and the USSR.

The physical properties of magnesium oxide (magnesia) are governed by the precursor, time and temperature of calcination, and impurities. With increasing calcining time and temperature, the magnesium oxide crystallites increase in size and this is accompanied by decreases in porosity and reactivity.

Caustic-calcined magnesia is produced at temperatures below about 900°C and characterized by its moderate-to-high chemical reactivity. Typical end uses are in the manufacture of chemicals, rayon, petroleum additives, fertilizers, oxychloride cements for construction, and mineral supplements for animal feeds. Dead-burned magnesia is produced at temperatures above about 1400°C and characterized by low chemical reactivity and resistance to basic slags. Dead-burned magnesia is consumed almost exclusively in refractory applications.

BuMines estimates that in 1984 over 80% of the magnesia consumed in the U.S. was dead-burned magnesia for refractories; principally those used in the iron and steel industry. The remainder was used for caustic-calcined and specified magnesias and other magnesium compounds. In most other countries, the proportion of magnesia consumed in refractory applications is even greater.

Domestic production of magnesia for 1984 was estimated by BuMines to be 590 000 metric tons (650 000 tons), up slightly from 1983's 567 000 metric tons (625 000 tons), but down significantly from 1980's 726 000 metric tons (800 000 tons). Over the same period (1980–1984) imports increased from 47 000 to 59 000 metric tons (52 000 to 65 000 tons) while exports decreased from 47 000 to 18 000 metric tons (65 000 to 20 000 tons). Import sources over the period 1980–1983 were Ireland—41%, Greece—21%, Canada—10%, Japan—9% and other—19%. Canada became a major source in 1983. BuMines estimates for 1985 are 612 000 metric tons (675 000 tons) domestic production and 649 000 metric tons (715 000 tons) domestic consumption.

World magnesium oxide capacity is estimated to be approximately 8.1 million metric tons (8.9 tons); approximately 70% derived from natural magnesite and 30% from seawater/brines. Dead-burned magnesia accounts for approximately 85% of that total.

In recent years the world's magnesia industry has been plagued by overcapacity. There are a number of reasons for this, principal among them are 1) the decline in world steel production, which resulted in reduced refractory consumption, 2) a reduction in the specific consumption (lb/ton of steel) of refractories brought about by (a) improvements in steelmaking process technology and operating practices and (b) improvements in refractory technology and product applications, 3) overly optimistic estimates of future growth in world steel production during the 1960s and 1970s that led to major expansions in magnesia production capacity, 4) major increases in the price of energy during the early 1970s that reduced the competitiveness of synthetic magnesia and led to capacity expansions in natural magnesite operations and 5) the entry of North Korea and China, both with vast reserves of high grade natural magnesite, into the world market.

Magnesia resources are virtually unlimited and magnesia production capacity is more than adequate to meet probable demand for many years. World prices are depressed and range from less than $110/metric ton ($100/ton) to more than $441/metric ton ($400/ton) depending on grade. Domestic prices recently reported by the Chemical Marketing Reporter for synthetic magnesia, bulk in carload lots FOB plant, were: $364/metric ton ($330/ton) for chemical grade (caustic-calcined) and $432/metric ton ($392/ton) for refractory grade (dead-burned). ☐

Nepheline Syenite

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Nepheline syenite is a holocrystalline, granular, igneous rock made up of nepheline ($K_2O \cdot 3Na_2O \cdot 4Al_2O_3 \cdot 9SiO_2$), potash feldspar (microcline), soda feldspar (albite), and such minor accessory minerals as mica, hornblende, and magnetite. It is found in Canada, India, Norway, and the USSR. It resembles granite in texture but contains no free quartz. An analysis of the commercial ceramic nepheline syenite from Blue Mountain, ON, is:

SiO_2	60.4%
Al_2O_3	23.6
Fe_2O_3	0.08
CaO	0.7
MgO	0.1
Na_2O	9.8
K_2O	4.6
LOI	0.7
Total	99.98%

This analysis represents the commercial material after removal of iron. Iron content of the original rock is about 2%.

For this composition, nepheline syenite has a molecular weight of 447, specific gravity of 2.61 in the crystalline form, and 2.28 in the glossy state; hardness of about 6 in Mohs scale. It starts to sinter at cone 08 and has a PCE of about cone 7. There is an eutectic between soda feldspar and nepheline which is a factor in the wide sintering range of nepheline syenite.

In sanitary ware bodies, the substitution of nepheline syenite for potash feldspar makes possible a much lower firing temperature. A longer firing range with decreased warpage is noted when firing the ware in commercial tunnel kilns. Recent research in the development of low fired vitreous ware has demonstrated the fact that a cone 4 sanitary ware body can be made which will provide a very large reduction in the cost of fuel and refractories plus the added advantage of a fast firing cycle.

In floor and wall tile bodies, the lower fusibility and increased fluxing action of nepheline syenite permit the formulation of bodies maturing at lower temperatures. Direct substitution of nepheline syenite for potash feldspar in wall tile bodies lowers the absorption and moisture expansion and increases the shrinkage and mechanical strength. Floor tile bodies show less variation in thermal expansion with differences in thermal treatment than the corresponding feldspar bodies. Thermal expansion of talc wall tile bodies is lowered by the direct substitution of nepheline syenite for feldspar, although bodies fluxed with nepheline syenite alone have a higher thermal expansion than those fluxed with potash feldspar only.

In electrical porcelain the same general results as noted above may be expected; substitution of nepheline syenite for potash feldspar increases firing range, increases strength, decreases absorption, and increases shrinkage at the lower firing temperature.

In semivitreous bodies, nepheline syenite produces increased vitrification. There is a long range which results in less warpage. In bodies fluxed with nepheline syenite, the thermal expansion is greater than in corresponding feldspar bodies. This tends to further a state of compression in the glaze and reduces crazing tendencies.

Low temperature vitreous bodies maturing at cone 3 to 5 can be formulated from clays and nepheline syenite without recourse to an auxiliary flux. Such bodies have a long firing range and good strength. These bodies are highly translucent when prepared by wet milling, and by combining low temperature with a fast firing cycle a large savings is made in fuel, refractories, and ware lost from warpage; furthermore, a wider color range in both body and glaze is made possible.

Table I. Proposed Batch[*] for Glass Using Nepheline Syenite

Sand	1000 lb
Nepheline syenite	153 lb
Burned dolomite lime	169 lb
Barium sulfate	12 lb
Fused borax (pyrobor)	27 lb[†]
Soda ash	339 lb
Arsenic	2 lb
Decolorizer	Sufficient quantity
Cullet	35% of weight of batch

[*] Not considering weight of cullet, the foregoing batch will produce approximately 1548 lb of glass.

[†] Prefer using fused borax or pyrobor, but if ordinary borax, which contains waters of crystallization, is used, it will be necessary to use 54 lb instead of 27 lb.

The higher the nepheline syenite content of the bodies, the higher the thermal expansion and the smaller the variation in expansion with differences in thermal history. The addition of 5% to 10% flint raises the thermal expansion of the bodies so that typical semivitreous dinnerware glazes are placed under adequate compression and can be used for one fired ware. Also, special high compression glazes have been developed for this use. For two fired ware excellent glazes are available which have good service characteristics and which mature at cone 01.

A typical low temperature body formula is shown below:

Nepheline syenite	54%
Flint	6
Kaolin	24
Ball Clay	16

Due to its high alumina content, nepheline syenite is a good material for introducing alumina into a glass batch. It contains considerable alkali, a desirable constituent of the batch, and melts at a relatively low temperature. These advantages, together with the fact that it is taken into the melt very readily make it a desirable addition to tank glasses. Substitution of nepheline syenite for potash feldspar on a chemical analysis basis in a typical opal glass batch is said to permit melting at a lower temperature, thereby affording the possibility of fuel economy and longer life of refractories. The resultant glass will have the same thermal expansion as the comparable feldspar glass, but softens at about 50°C lower temperature. The iron-alumina ratio of this material makes it useful in glasses where low iron oxide content is of primary importance.

Table II. Glass Grade Nepheline Syenite

Silica	70.30%
Iron and alumina	2.36
Calcium oxide	6.32
Magnesium oxide	4.54
Barium oxide	.50
Sodium oxide	14.26
Potassium oxide	.48
Boric oxide	1.20
Total	99.96%

A typical container batch is shown in Table I which is rather high in aluminum oxide and lime, but it has been found that this glass has great durability and strength, and the rate of production is increased due to the higher aluminum oxide content.

Glass-grade nepheline syenite is now refined by means of magnetic separation, making possible a granular concentrate with a constant low iron oxide content (Table II).

In porcelain enamels, the obvious advantage again is the decreased fusion temperature possible. However, when substituting directly for feldspar, increased viscosity due to increased alumina makes it necessary to fire at nearly normal temperature to get proper maturity. It is also possible, through the use of nepheline syenite, to incorporate considerably more alumina in an enamel without increasing the hardness of the enamel. This is desirable because increasing alumina generally reduces the solubility of the enamel.

Recent tonnage figures indicate 413 000 metric tons (455 000 tons) of nepheline syenite shipped by the two North American producers on an annual basis. Of this total, 54 000 metric tons (60 000 tons) were fine grind, 309 000 metric tons (340 000 tons) were glass grade and 50 000 metric tons (55 000 tons) were export offshore.

Pricing for nepheline syenite has been depressed in recent years due to excess production capacity. In addition, glass containers, the major consumer of tonnage, has been affected by severe competition from both P.E.T. bottles and cans. ☐

Pyrophyllite

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The mineral pyrophyllite is in appearance similar to talc and is grouped with talc in many statistical presentations. In its pure form, it is characterized chiefly by its micaceous habit, cleavage, and greasy feel. It is a comparatively rare mineral found in metamorphic rocks, frequently with kyanite. It occurs in considerable amounts in Guilford and Orange counties, North Carolina.

Pyrophyllite is ahydrous aluminum silicate with the formula Al_2O_3, $4SiO_2$ H_2O, and like talc, rarely occurs in its pure form. For example, pyrophyllite mined in Robbins and Glendon, North Carolina, has a distinct mineralogy which makes it suitable for whitewares and refractories.

According to BuMines' *Mineral Facts and Problems*, 1985 Ed., using a table differentiating end uses for ground talc and pyrophyllite in 1983, we can extract the following data for the United States: 25 000 metric tons (27 000 tons) of pyrophyllite used in ceramics and 21 000 metric tons (23 000 tons) in refractories with a total of 84 000 tons. These figures account for 32.1% and 27.4% for ceramics and refractories respectively. In 1978 the use of pyrophyllite for all uses amounted to 103 000 metric tons (113 000 tons) with 15 000 metric tons (17 000 tons) (15%) used in ceramics and 35 000 metric tons (39 000 tons) (34.5%) in refractories. This indicates that the total tonnage for all uses decreased by 25% over the last 5 years. The use of pyrophyllite in refractories decreased by 41% since 1978 while the use for ceramics increased 59%.

The pyrophyllite reserves in North Carolina alone are said to total at least 12 million metric tons. Estimates of quantities are not available for California and Pennsylvania. Those states also have commercial deposits of lesser importance.

The whiteware grades contain, in addition to major pyrophyllite, major sericite and quartz with minor kaolin. High sericite pyrophyllite shows the following properties:

- Lowers firing temperature.
- Produces low moisture expansion bodies resulting in good craze resistance.
- Produces bodies with little or no shrinkage and reduced warpage.
- Increases thermal shock resistance due to its high thermal conductivity and low coefficient of thermal expansion
- Greatly increases firing strength in vitreous bodies.

These properties result in the principal use of pyrophyllite ceramic wall tile bodies, with little or no shrinkage, greater uniformity in tile size, and less warpage. The high sericite content also contributes to the formation of mullite at comparatively low temperatures. Mullite, with its interlocking grain structure, results in greatly increased fired strength in vitreous bodies, where it substitutes for all the flint or all of the flint and some of the feldspar. The shrinkage and absorption properties are not affected by this substitution, while thermal shock resistance is increased.

Another type of industrial pyrophyllite, mined at Glendon, is of particular interest for refractory applications, due to the following properties:

- Permanent expansion.
- Excellent reheat stability.
- Low reversible thermal expansion.
- Low bulk density.
- Low thermal conductivity.
- Highly resistant to corrosion by molten metals and basis slugs.

The minerals present are major pyrophyllite, major-to-moderate quartz, minor kaolin, and trace quantities of sericite. The properties of this type pyrophyllite make it particularly suitable for insulating fire brick class 2300, 2500, and 2600.

Other applications include metal pouring refractories, castables, stiff plastic refractory compositions, gunning mixes, and kiln car refractories. In mold dressing, this type of pyrophyllite suppresses or totally inhibits the formation and structure of a skin on iron castings.

Another type of pyrophyllite is the andalusite-pyrophyllite deposit at Hillsborough. Here, quartz is floated away from pyrophyllite and andalusite, increasing the Al_2O_3 content and PCE. ☐

Refractory Clays

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BuMines estimates 1984 clay usage for refractories in the U.S. was 1 700 000 metric tons (1 900 000 tons) categorized as fire clay, kaolin, bentonite, and ball clay.

Usage trends are dominated by production and technology requirements of the iron and steel industry, which consumes approximately 50% of U.S. refractories. Higher refractory clay consumption requirements caused by a 10% increase in raw steel production in 1984 was offset by the continued shift toward the use of higher alumina, basic, and nonoxide refractories. Increased usage of monolithic refractories has emphasized the importance of calcined aggregates and further decentralized specialty refractory production to regional locations.

The highest quality refractory clays—kaolin, and bauxitic kaolin—are mined and processed primarily in the Andersonville, GA, and Eufaula, AL, areas. Other kaolin producing areas are in central and northern Georgia, South Carolina, and Ione, CA. Most of the super duty flint clay is mined and processed in Missouri. There has been a further reduction of the lower quality refractory clays in Pennsylvania, Ohio, Kentucky, West Virginia, and Alabama.

The poor economic condition of the steel industry continues to hold prices down. Production focus is on cost reduction without adversely affecting quality. The most important process innovation to reduce costs has been the conversion of rotary kilns to coal firing. Production capacity exceeds demand with ample clay and calcined aggregate available to the refractory industry. However, the larger operations must have sufficient volume to maintain profitable operations without price increases. Smaller companies with less capital and overhead can operate at lower capacity levels but cannot meet the demand requirements of the refractory industry. This will result in further restructuring if volume continues to decline.

Maintaining quality control standards is being increasingly stressed by refractory consumers. This situation has in turn reflected renewed attention on both the quality and long term consistency of all raw materials used to produce refractory products. High quality clays and calcined aggregates are rapidly replacing lesser quality materials—especially in monolithics—as more attention is given to hot load and corrosion resistance properties. ☐

Silica

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The mineral industrial silica is distinguished from sand and gravel by its chemical purity—high SiO_2 content and a low percentage of impurities such as Fe_2O_3, Al_2O_3, and refractory minerals. For certain applications, industrial silica sand with an SiO_2 content exceeding 90% is adequate. For most uses, however, the purer grades of industrial silica containing SiO_2 in the 98% to 99+% range are specified. Unlike sand and gravel, industrial silica is washed, sometimes beneficiated to remove impurities, dried, and carefully screened to specific mesh ranges determined by its intended use. Industrial silica is also ground into grades such as 98% passing a 120 mesh, 98% passing a 325 mesh, etc. Finer micron-sized grades are offered by a few producers, with one supplier offering grades with average particle sizes as fine as 1.1 μm.

Production and consumption of industrial sand, as reported by the BuMines, declined steadily from a high of 30 047 000 metric tons (33 120 000 tons) in 1979 to only 24 147 000 metric tons (26 617 000 tons) in 1983. Sales volume in 1983 fell 3.9% from 1982 tonnage and 17.1% from 1979 results, per the Bureau's statistics. Preliminary estimates from the Bureau indicate that 26 000 000 metric tons (29 000 000 tons) were produced in 1984, an increase of 8.9% over 1983.

Another source of industrial sand production data is the National Industrial Sand Assn., which reportedly represents producers accounting for approximately 85% of the industrial sand (excluding gravel) sold in the U.S. This association reports its members produced 22 702 000 metric tons (25 025 000 tons) in 1979, but only 15 823 628 metric tons (17 442 271 tons) in 1983. No tonnage estimates are available as yet for 1984.

According to BuMines' latest annual report, the average price of industrial sand in 1983 varied by region (as it has historically). The 1983 reported prices were $15.22/net metric ton ($13.81/net ton) in the northeast, $11.89/net metric ton ($10.79/net ton) in the north central states, $14.93 net metric ton ($13.55/net ton) in the south and $16.81/net metric ton ($15.25/net ton) in the west. The national average price was $13.93/net metric ton ($12.64/net ton), while total revenues in 1983 are reported to have been $329 500 000. Based on the latest information from the Bureau, 1984 total sales revenues are estimated at approximately $370 000 000 with average national prices last year projected to be about ($13.00/net ton).

Major consuming industries for industrial silica are glass (all types), foundry, metallurgical, abrasive, chemical, ceramics, filtration, coatings, and oil/gas well fracturing sand. Demand for industrial silica products is, of course, tied to their overall economic performance. The 1985 market will, therefore, mirror the conditions that prevail within these major markets during the year.

Expansion and modernization of various industrial silica producing facilities and the startup of new plants in recent years assure adequate supplies of silica products for at least the immediate future. □

Silicon Carbide

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The first silicon carbide crystals were made accidentally by Edward Acheson while experimenting with a mixture of clay and carbon in an electric furnace. Silicon carbide does not occur naturally; it is a synthetic material made by combining silica and carbon. The Acheson resistance furnace remains as the basic manufacturing process, though it has been modified and updated in recent years to improve productivity and meet environmental standards.

High-quality silica sand (99.5% SiO_2), low-sulfur petroleum coke, and electricity (3.5 KWh/lb) are the major ingredients. The reaction takes place in a trough-like furnace with removable refractory sides, or some similar configuration, and permanent refractory ends holding carbon electrodes. When the furnace is started, these electrodes are joined by a graphite core laid the length of the furnace near the center of the mixture which fills the furnace. The voltage passing through the resistance core generates a temperature of about 2400°C, and in about 36 hours the chemical reaction of silica and carbon to silicon carbide is complete. The reaction requires five to seven pounds of raw material for one pound of SiO. The highest purity (light green in color) material is found near the core. The outside layers of uncombined material are removed before further processing of the crude SiC begins. The crude is then selected for purity, crushed and graded with screens to meet the various requirements.

The most critical manufacturing issues in recent years have been the cost and availability of coke, cost of electricity, and environmental considerations. Petroleum coke, a by-product of petroleum refining, is also used by the aluminum industry and as a result in periods of high aluminum demand, it is in tight supply. Prices also have risen sharply in line with other petroleum-based products. Most plants are located near sources of low-cost hydroelectric power, but energy related cost pressures will continue to have an impact on SiC costs.

The issue that will continue to draw a great deal of the industry's attention and captial resources is the need to meet tighter environmental regulations. Costs and availability of material will be impacted as manufacturers decide which of the older plants will be closed, which can be effectively modified, and when to build new plants in order to meet the stricter standards.

Purity of silicon carbide determines the color. Light green material is about 99.8% pure and as the purity declines to 99.5%, the color changes to dark green, then to black at about 99% and finally to grey at 90% SiC. The purities generally available are 90% which is used for metallurgical and some refractory applications; 98%–99% is used for most abrasive and refractory applications. High-purity green SiC is used in a few special grinding wheel applications and special ceramic applications and as resistance heating rods. Ultrapure SiC is in growing demand for reaction-bonded products.

The requirement for high purity green material is relatively small and as a result, all manufacturers have rationalized their production to the point where it is manufactured only in selected plants and only in Europe or Japan.

For the manufacture of high performance ceramics by sintering or hot pressing, other methods of synthesis are sometimes used to prepare high purity, very fine reactive powders. Such processes include: plasma arc synthesis, continuous feed-through and induction furnace, batch reactions of silica and carbon in CO or inert gas, decomposition of polycarbosilanes, and chemical vapor deposition.

Six firms in North America currently produce silicon carbide by the Acheson process. This is one less than 1983 because ESK (USA) and Exolon have announced plans to combine their operations into a new company. Imports from Europe account for less than 6% of the total usage and are generally confined to specialty items such as green SiC and micrograts. Prices vary widely from $496 to $6614 per metric ton ($450 to $6000 per ton), depending on chemical and physical specifications. The bulk of materials for refractory uses sell for between $661 and $1102 per metric ton ($600 and $1000 per ton). Prices have been depressed for about three years—industry wide. As the economy picks up, price increases are likely.

According to BuMines, North American production in 1984 was 110 000 metric tons (121 000 tons) which is the same as 1983. The largest portion, about 45%, of SiC production is used in metal melting as additives in iron and steelmaking. Aside from acting as a source of silicon and carbon, the silicon carbide imparts other desirable properties to the melt. It acts as a deoxidant, but more importantly, once the material is melted, the silicon carbide dissociates in an exothermic reaction which makes production of thin castings easier.

Abrasive applications account for

about 35% of silicon carbide usage. Large quantities are used in grinding wheels and in wire sawing of granite. Smaller applications include the manufacture of coated paper and cloth, lapping, and a wide variety of specialty jobs.

The most promising growth area of SiC is in refractory and ceramic applications. This segment now accounts for about 20% of total production. Silicon carbide has a number of properties that are desirable in these applications. Properties such as high thermal conductivity, high thermal shock resistance, high hot strength, and low coefficient of thermal expansion make it useful in applications such as kiln furniture, cements, crucibles, etc. Its extreme hardness is utilized in applications such as furnace skid rails where resistance to abrasion is critical.

Resistance to chemical attack even at high temperatures make it effective in corrosive environments such as those encountered in blast furnaces, aluminum reduction cells, and coal-fired power plants. The unique nonlinear electrical resistance characteristics of SiC make it useful in lightning arrestor blocks and heating elements.

Silicon carbide products of high strength can also be made by reaction bonding in which carbon and molten silicon are reacted in situ in form shapes made from carbon with or without silicon carbide. The strongest and most wear-resistant silicon carbide products are made by sintering or hot pressing of fine high purity powders. Such products are finding applications in areas of high wear such as mechanical seals and pump parts. Properties of wear and corrosion resistance and high hot-strength also make them useful for some engine components.

Silicon nitride, Si_3N_4, is a synthetic raw material which is made by high temperature chemical reactions between 1000° and 1600°C. Because of the unique properties of dense products of silicon nitride, it is beginning to find commercial uses after many years of research. Sintered and hot pressed silicon nitride bodies have high hardness, high strength, low density (3.2 Mg/m^3) low thermal expansivity, and good oxidation resistance.

The first silicon nitride products were made not from silicon nitride raw material, but from silicon. Powder metallurgy techniques were used to fabricate shapes of silicon metal which were then fired in nitrogen to produce a porous silicon nitride product. Silicon carbide products bonded with silicon nitride have been made commercially for about 30 years. Like reaction bonded silicon nitride, the silicon nitride is manufactured in situ by firing a silicon carbide and silicon mixture in nitrogen.

High strength silicon nitride shapes were obtained after it was learned how to hot press or sinter silicon nitride powders to theoretical density. The three most important methods for manufacturing silicon nitride powder are: reacting silicon metal powder with nitrogen; reacting silica, carbon, and nitrogen; and reacting chlorosilanes with a gas containing nitrogen or a nitrogen compound. The newest synthesis method is to spray fused silicon in nitrogen and quench.

For hot pressing or sintering, these powders are normally mixed with a sintering aid such as calcia, magnesia, or yttria. The sialons (combinations of silicon, aluminum, oxygen, and nitrogen) are often fabricated with silicon nitride powder as a raw material.

Published production figures for the U.S. and the world for silicon nitride powders are not available. Many of the manufacturers of silicon nitride products make their own powder. Estimated annual production of silicon nitride powder is 9.1 to 13.6 metric tons (10 to 15 tons) in the United States and 18 to 27 metric tons (20 to 30 tons) worldwide. Apparently, there is no domestic supplier of commercial quantities of silicon nitride powder.

Powder can be purchased from German, Japanese, U.K., and Canadian sources. Prices vary depending on specifications for particle size and purity. They range from $20 to $350/kg.

Silicon nitride has been studied as a potential material for engines for the last 15 years. It is being used today to produce prechamber cups for a diesel application in quantities of about 120 000 pieces annually. This is the order of about 1000 kg annually, but it is a start. Tool bits of silicon nitride, sialon, and mixtures of silicon nitride with other materials are finding increasing use. There are at least five domestic suppliers of tool bits. They are also manufactured in Japan, the U.K., and Germany. Rolling contact bearings are being used in small quantities for high performance application.

Now that commercial uses are beginning to develop for this material, there should be a dramatic growth of these products made from silicon nitride. Fortunately, both silica and nitrogen are plentiful sources for the manufacture of silicon nitride. □

Sodium Carbonate

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Production of soda ash in the U.S. in 1984 was 7.8 million metric tons, essentially unchanged from 1983. Despite the strength of the dollar, however, the export market remained strong particularly in the Asian markets. Latin America, Southeast Asia, China, and Japan continued to be major importers of U.S. soda ash. Exports represented 20% of U.S. production. Worldwide soda ash demand was 28 million metric tons, supplied by some 90 manufacturing plants in 36 countries. The U.S., with almost 30% of world capacity, was the leading supplier (Table I). There are seven soda ash producing plants in the U.S. All except one (Allied's Syracuse plant) are based on the refining of naturally occurring ores or brines in Green River, WY, and Searles Lake, CA. Outside of the U.S. the synthetic process based on ammonia and salt still dominates.

Overcapacity in the U.S. and Western Europe continues to be a problem. U.S. pricing, although improved somewhat in 1984, is still down substantially from the 1982 high of $101/metric ton. At the end of 1984, the price was $87/metric ton at Green River, WY, with somewhat higher pricing by the east and west coast producers.

Table II summarizes the present U.S. soda ash end-use market. Glass containers dominate the market, but are experiencing severe competition from aluminum cans and from plastics. Soda ash consumption in glass container manufacture has also been adversely affected by the increased use of eco-

Table I. U.S. Domestic Producers of Soda Ash

Producer	Location	Nameplate capacity (Millions of metric tons)
FMC	Green River, WY	2.6
Allied Corp.	Green River, WY	2.0
Stauffer	Green River, WY	1.7
TexasGulf	Green River, WY	0.9
Tenneco	Green River, WY	0.9
Kerr-McKee	Searles Lake, CA	1.3
Allied Corp.	Syracuse, NY	0.7
Total		10.0

Table II. U.S. Soda Ash Markets (1984)

End use	Consumption (Millions of metric tons)
Glass and ceramics	
Glass containers	2.10
Flat glass	0.60
Fiber glass	0.20
Other glass and ceramics	0.20
Total glass and ceramics	3.10
Chemicals	1.20
Detergents	0.60
Pulp	0.15
Water treatment	0.20
All other	1.05
Total U.S. demand	6.30
Exports.	1.45
Total U.S. production	7.75

logical cullet. State container deposit laws and environmental pressures have made cullet more available for recycle. Glass container production peaked in 1978 and has declined each year since. This end use for soda ash is expected to show no growth and probably will decline.

Soda ash is also used in the production of a wide variety of chemicals, many of which find use in the ceramic industry. These include sodium silicates, phosphates, and lithium compounds. Other chemicals, such as baking soda and chrome compounds for pigments and auto use, are also based on soda ash. Two use areas showing good growth are builders for household detergents and the scrubbing of sulfur-containing wastes. Overall, though, growth of the U.S. soda ash industry is expected to be less than one percent per year.

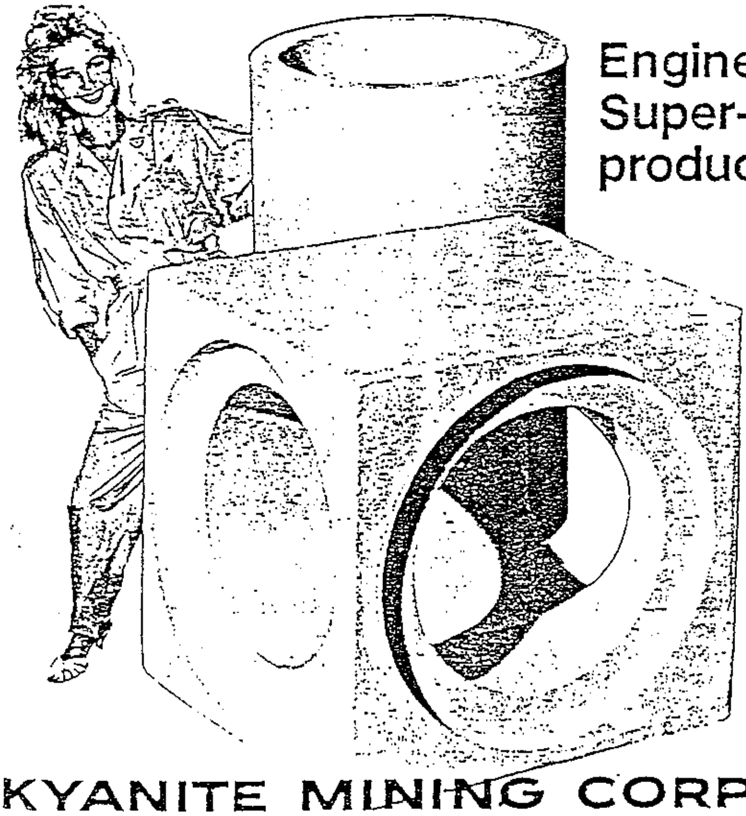
With overcapacity in many parts of the world, considerable attention has been devoted to methods of reducing soda ash production costs. In the U.S., FMC Corp. has undertaken a major development program to apply solution mining techniques to the mining of trona (soda ash) ore. Solution mining in its simplest form consists of drilling two parallel wells, injecting a solution to dissolve the trona in one, developing an underground cavity, and removing the dissolved trona through the second well. This basic technique had been used for a number of years in salt and potash mining. It has the advantage of lower labor costs than dry mining, provides a higher percentage of ore recovery, and allows mining at depths that normally would be too deep to safely mine by conventional methods. By the fall of 1985, FMC plans to have a full scale soda ash plant on stream at Green River, WY, using this technique.

The synthetic soda ash plants outside of the U.S. have been particularly troubled by the high cost of energy. To help combat this problem, several of the European synthetic plants have recently been converted from oil and gas based energy to coal or lignite. Two synthetic process soda ash plants within Europe have been closed.

Another result of this never-ending search for ways to reduce production costs has been an increasing interest in the development of foreign natural based soda ash deposits which might have lower energy requirements.

Recently, the discovery of large deposits of trona ore near Ankara, Turkey, has been reported. These deposits are receiving considerable attention because of their proximity to the EEC market. Studies to determine the optimum method for commercializing these deposits are now under way.

The Peoples Republic of China has reported the finding of extensive deposits of soda-ash-containing ore in Honan Province. Soda ash brine deposits in Mongolia have been known for many years. At present both of these deposits are too remote from their major markets for large scale develop-



ment. However, the Peoples Republic of China has a rapidly increasing demand for soda ash to supply its fast growing glass industry, and new synthetic process plants have been announced for Shandong, Xiamen, and Lianyungang. Completion is scheduled over the next three years. Present capacity there is 1.9 million metric tons.

In Africa, ICI has incrementally expanded its natural soda ash plant at Lake Magadi from 250 000 to 300 000 metric tons per year. British Petroleum is conducting studies to determine the economics of producing soda ash in Botswana from the Sua Pan deposits. Lake Natron in Tanzania contains major amounts of soda ash, but the cost of developing an infrastructure has slowed its commercialization.

Most recently, there have been reports of the finding of potentially interesting soda-containing brines in Queensland, Australia. Because of its large alumina industry, Australia must import major quantities of alkalis in the form of caustic soda. Should these new deposits prove economically attractive, the resulting soda ash could supplement a portion of these import requirements. ICI produces soda ash synthetically in Australia at Botany Bay.

In the area of synthetic soda ash capacity, there have been few changes. India has announced a new 400 000 metric ton plant scheduled for completion in 1986–87. Although India already has a capacity to produce 750 000 metric tons of soda ash per year, their requirements are rapidly increasing. Brazil has had plans to construct a new 200 000 metric ton per year plant at Macau for six years. The equipment has been received and delivered to the site, but funds are lacking for its completion. The ASEAN Group (Assn. for Southeast Asian Nations) proposed erecting 300 000 metric ton soda ash plant at Sattahip, Thailand. This plant too has been subject to numerous delays. Recent press releases indicate renewed interest.

A summary of soda ash capacity and demand is shown in Table III.

The North American and European market is mature and supply is more than adequate to supply the demands of the glass and ceramic industry. In Latin America and Asia (with the exception of Japan), the reverse is true, and these regions are net importers of soda ash. Japan has adequate soda ash capacity to supply its domestic needs, but has high production costs and hence imported soda ash has become highly competitive with locally produced material. Africa and the Middle East are essentially in balance, although Africa both imports and exports material.

Another new development during the past year is the foundation of the American Natural Soda Ash Corp. (ANSAC). All of the U.S. soda ash producers have joined in the association to pool export sales and better serve their overseas customers.

Table III. World Soda Ash Capacity and Demand (Millions of Tons)

Area	Capacity	Demand
North America	10.8	7.4
Africa/Middle East	0.6	0.6
Asia/Oceana	5.0	5.6
Eastern Europe	10.5	8.3
Latin America	0.4	0.9
Western Europe	7.3	5.8
Total	34.6	28.6

In summary, there is adequate world soda ash capacity to serve all markets. Little new synthetic production capacity will be built over the next few years except in regional markets such as the Peoples Republic of China. Emphasis of the major soda ash producers will be on cost reduction projects. As part of this program, the industry may be expected to continue to seek out new natural brine and trona sources having potentially lower costs. □

Talc

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The mineral talc is a soft, hydrous magnesium silicate, $3MgO4SiO_2-H_2O$. Commercial talcs range from mineral products approaching the near-theoretical mineral composition to mineral products that have physical and/or pyrophysical properties in common with pure talc but contain very little of the actual minerals. The mineral composition of talc mined in Montana, for example, is essentially talc with minor chlorite, dolomite, calcite, and quartz. In Texas, depending on the district of mining, associated minerals include quartz, magnetite, chlorite, tremolite, and anthophyllite. In Vermont, magnesite, serpentine, chlorite, and sulfides are associated with the talc rock. We can see in just reviewing a few locations that talc minerals from different sources may present notable differences, which in turn shape the consumption pattern.

According to BuMines, the latest figures show the production of talc in 1982 for the U.S. was 1 million metric tons (1.1 million tons) and 1983 estimated at 0.95 metric tons (1.04 million tons), a drop of approximately 8%.

Vermont, Montana, Texas, and New York, in order of volume, produced together 83% of the yearly total. There were 21 talc producing companies in 11 states in 1983; the three largest supplied 50% of the output. The ceramic industry consumed 34% of the ground talc.

Estimated reserve bases of talc-group minerals remain at 136 million metric tons (150 million tons) in the U.S. Exports of talc and related minerals from the U.S. during 1983 are estimated to outweigh imports by more than 11 to 1. Apparent consumption of talc-group minerals in the U.S. was 2% lower than 1982. However, using 1979 as a base, demand for talc and related minerals is expected to increase at an annual rate of about 2% through 1990. It is further estimated that in 1984, figures will show domestic mine production of talc will be 1.2 million metric tons (1.3 million tons) and U.S. apparent consumption will be 0.91 million metric tons (1.0 million tons). U.S. exports of talc minerals has shown cyclical growth in prior years, but the tonnage in 1983 was the same as 1982, and the lowest since 1976.

BuMines reports production worldwide for 1982 totalled 6.9 million metric tons (7.6 million tons), with 1983 tonnage estimated at 6.4 million metric tons (7.1 million tons.) The present world reserve base is listed at 299 million metric tons (330 million tons). World resources are estimated to be approximately five times the quantity of reserves. Shortages of talc, therefore, are not expected. The supply situation could be adversely affected, however, by lack of financial capital for expansion or stringent regulations on mining.

Petrographic and X-ray examinations of ores from the talc mining areas of Gouverneur, NY, show them to contain three principal minerals: talc, tremolite (nonasbestiform), and serpentine (in the form of antigorite). Due to the presence of 30%–50% prismatic tremolite, New York talc provides unique properties for the ceramic industry. Tremolitic talc is used in whitewares (semivitreous ware and vitreous ware), glazes, and refractories.

Typical semivitreous bodies include wall tile art pottery, and dinnerware. Tremolitic talc has been found to be an ideal raw material for the following reasons:

- Produces high thermal expansion bodies through the formation of enstatic, resulting in glazes being put in compression which, in turn, tends to prevent crazing.
- Low moisture expansion bodies are produced resulting in good resistance to delaying crazing.
- Aids dry-pressing because of the presence of 30%–50% prismatic tremolite.
- Permits faster pressing, eliminates lamination, and improves product density.

- Especially suitable for high talc, low temperature casting bodies through control of specific resistance on certain grades.
- Low firing temperatures and fast firing schedules are possible, which conserve energy and, in many cases, reduce cost.

The use of talc as an auxiliary flux in vitreous bodies (high alumina, electrical porcelain, vitreous china, sanitary ware, and translucent artware) to form a eutectic with feldspar and nepheline syenite is well known. The result is a tighter and stronger body when fired to the same temperature, or the maturing temperatures may be reduced to effect fuel impact on the maturing temperature. Pieper described a low-thermal-expansion ceramic composition in his patent application, using tremolite (5%–20%) as a constituent to make "cordierite-like ceramics" suitable for thermal shock resistance cookware. This body composition has advantages when compared with a cordierite body such as reduced firing temperatures and wider firing range. □

Wollastonite

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Wollastonite, a calcium silicate (48.3% CaO 51.7% SiO), has received much attention over the last decade. Twelve years ago, world production of wollastonite was about 40 000 metric tons/yr, a rise of 275%. With the market for wollastonite to grow at about 10% per year for at least the next five years, the increase may create a shortage.

The U.S. is the leader in wollastonite production with an output in excess of 50% of the estimated world production. Other producers include Finland, Mexico, India, Turkey, and Namibia. West Germany is producing synthetic wollastonite. The wollastonite ore bodies vary from location to location and for the most part need to be beneficiated. Associated minerals in the largest wollastonite operation (at the Fox Knoll Mill near Willsboro, NY), are primarily up to 40% garnet and other impurities. High intensity magnetic separation is used to separate the garnet. In the Gouverneur region of New York, a rather pure wollastonite is being mined, which contains only minor calcite and traces of diopside, and does not have to be beneficiated. In Finland, the deposit contains limestone, dolomite, and an average wollastonite content of 18%–20%. Froth flotation and optical ore sorting is being used in this operation to upgrade the wollastonite.

Why has wollastonite become so popular for ceramic application? In wall tile, its major use area, it assists toward uniformity of dimensions, low shrinkage, good strength, low warpage, low moisture expansion, and fast firing with decreased gas evolution. In semivitreous ware, substituting 1% to 3% wollastonite for flint and/or flux decreases shrinkage and moisture expansion and increases strength. In vitrified bodies, replacing 2% to 5% feldspar and quartz with wollastonite results in decreased shrinkage and lower vitrification temperature. In glazes, wollastonite may be used to substitute for limestone and flint, reducing the volatiles and increasing the gloss and texture of the glaze. In porcelain enamels, wollastonite acts as a natural frit reducing gas evolution.

In the production of glass and fiber glass, wollastonite shows advantages of reduced energy requirements, if used in place of limestone and sand. In addition, the presence of "scum" (floating layer of unmelted material on the molten glass surface) and "seeds" (extremely small gaseous inclusion in glass) are also lessened. We can see that most benefits are related to reduced energy consumption, a basic reason for the sudden popularity of wollastonite.

Wollastonite also finds use in glaze and body stains, in frits for glazes, in bonds for vitrified grinding wheels, in ultralow ceramic insulating bodies, and an auxiliary flux in electrical insulators. In other applications, such as in slag treatment for the steel industry, wollastonite is being investigated and could potentially contribute to a greater increase than projected. Overall, wollastonite has unique properties which will push up the demand. □

Zinc Oxide

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Consumption of zinc oxide in the U.S. over the last 10 years has averaged 182 000 metric tons (200 000 tons) and most of this has been supplied by three large domestic producers. In 1984, U.S. consumption was about 186 000 metric tons (205 000 tons), a 10% increase over 1983.

Production capacity for U.S. zinc oxide is presently 227 000 metric tons per year (250 000 tons per year) and is adequate for today's market. Imported zinc oxide has made substantial gains in recent years. Currently, it accounts for about 17% of U.S. consumption.

The U.S. has ample zinc oxide capacity for the short term future, even though raw material availability remains a concern. The supply of zinc concentrates for primary smelting is adequate for the present time, but many metal producers have started using high quality zinc secondary (recycle) materials that were traditionally reserved for zinc oxide and zinc dust producers. The problem of recycle raw material availability is becoming serious as auto shredders begin to process the generation of automobiles which contain less recoverable zinc.

Price of zinc oxide is largely influenced by the price of slab zinc (metal). The slab zinc market has approximately 10 times the volume of zinc oxide on a world basis. That factor is significant in the area of feed materials. Zinc oxide producers compete with slab zinc producers for the same raw materials which are all based on slab zinc price. Therefore, the production costs and, eventually, the price of all zinc products generally move in the same direction, at about the same time.

Zinc oxide does not exactly track the price of slab zinc on a month-to-month basis. However, long term pricing trends of both materials are similar. Last year, we saw the price of slab zinc move downward at a very sharp rate. The price of zinc oxide followed at a lagging pace.

With regard to properties, zinc oxide is a fine, white powder and is both a pigment and a chemical. It is nontoxic, amphoteric, and relatively inexpensive. All these properties have resulted in zinc oxide appearing in a wide range of important large tonnage applications.

Rubber is the most important market for zinc oxide, accounting for over 50% of industry sales. Zinc oxide continues to serve an important function as a rubber vulcanization activator. No substitute products are anticipated to replace zinc oxide, even though much effort has been put into assessing replacements. The recent recession has hit the rubber industry hard, but it is expected to recover as economic recovery becomes apparent.

Within the rubber industry, the major tire companies use most of the zinc oxide. In 1984, tire production was excellent and resulted in a very good year for zinc oxide consumption. The present year is expected to remain strong for the rubber industry—following the trends of automobile production.

The paint industry accounts for approximately 10% of zinc oxide consumption. After significant declines in the late 1970s, the paint industry usage for zinc oxide seems to be holding steady at slightly less than 10% of the total market. The loss of tonnage was attributed to a customer preference for latex paints in place of oil base house paints. Latex house paints use less zinc oxide.

The use of zinc oxide in glass and ceramics has accounted for about 5% of the zinc oxide market. Some of the traditional markets have remained constant, and some growth is expected in new specialty ceramic markets.

Zinc oxide is consumed in the manufacture of a variety of zinc chemicals with diverse end-use markets including conversion coatings, catalysts, fungicides, pharmaceuticals, pigments, and lubricating oil additives. The chemical market accounts for about 15% of all zinc oxide consumed. A wide range of new potential applications for zinc oxide is encouraging, and modest growth is forecast for this segment of the market in the future.

Consumption of photoconductive zinc oxide for coated office copy paper peaked in the mid-1970s and has been declining steadily. Currently, it accounts for less than 5% of the total market. A continued decrease is forecast as plain paper copiers gain popularity. New plain paper copiers are quite low-cost, which makes them affordable for even small businesses. Photoconductive zinc oxide used for offset masters and repeat charge masters appears to be stable and offers some growth possibilities for this market segment.

Zinc oxide is a mature market with reasonably good balance between supply and demand. The versatility of zinc oxide along with its stable position in the rubber industry assure continued high volume use in the foreseeable future. Ample production capacity exists in the U.S. to handle the expected demands from the marketplace. ☐

Zircon

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The mineral zircon (zirconium silicate ($ZrSiO_4$) occurs as a small percentage (0.5%–3.0%) in special sand deposits with small percentages of other heavy minerals ilmenite, rutile, and monazite. The refined product and its compounds are widely used in ceramic applications such as refractories, glazes for ceramic tile and sanitary ware, and as the raw material for the production of zirconia, (zirconium oxide, ZrO_2) and alumina-zirconia abrasives.

It is estimated that approximately 90% of world wide zircon consumption was accounted for by refractory, ceramic, and foundry uses. Approximately 90 720 metric tons (100 000 short tons) of zircon were consumed in the U.S. in 1983, almost half of it in foundry applications. With a few exceptions, for more than a decade, the annual consumption of zircon in the U.S. was within 10% of 141 000 metric tons (155 000 tons). A major decline was reported for 1982. BuMines estimate 1984 consumption to have recovered to more normal levels of 118 000 metric tons (130 000 tons) (See Table I).

Zircon sand is a commodity being sold to mature and well developed world wide markets. The economics of mining zircon is closely related to the fact that zircon occurs with rutile and other heavy minerals in ratios predetermined by nature. Rutile has been the economically dominant product, with zircon usually considered to be a by-product. Thus if the demand for rutile is low, and mining production is curtailed, the result is a shortage of zircon since its demand is fairly consistent. Prices can fluctuate widely with shifts in the balance between supply and demand. In 1983, the large tonnage prices of zircon sand in bulk were stable. Premium prices were paid for premium quality sands.

Zircon is produced in 10 countries with Australia, South Africa, and the U.S. accounting for about 84% of world production (see Table II).

Present overall mine capacity in these three countries is estimated to be approximately 680 000 metric tons (750 000 short tons). Considerable consolidation has taken place in the past decade (23 operating mines in 1976 versus 12 mines in active production in 1984). A major shift also occurred with South Africa becoming a major producer and with the decline of production on the east coast of Australia (see Table III).

Three companies, DuPont, General Mining, and Consolidated Goldfields now control 85% of zircon mine capacity in

Table II. 1983 World Zircon Production

Source	Amount: Metric tons	Short tons	%
Australia	463 000	510 000	60%
South Africa	127 000	140 000	16%
U.S.	62 000	68 000	8%
Subtotal	652 000	718 000	84%
USSR	82 000	90 000	10%
China	15 000	17 000	2%
India	12 000	13 000	2%
Others	14 000	15 000	2%
Grand total	775 000	853 000	100%

Source: BuMines, 1983 Minerals Year Book; (except U.S. data).

Table III. Zircon Mine Capacity, Metric Tons (Number of Mines)

Location	Year: 1976		1984	
East Australia	385 000	(15)	100 000	(4)
West Australia	215 000	(6)	325 000	(5)
South Africa			150 000	(1)
U.S.	80 000	(2)	105 000	(2)
Total	680 000	(23)	680 000	(12)

Source: TAM Ceramics, Inc., Market Research.

Table I. Estimated U.S. Zircon Consumption (short tons)*

End use	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983
Zircon refractories	30 000	28 000	29 000	31 000	27 000	26 000	25 000	25 000	15 000	17 000
AZS refractories	10 000	12 000	10 500	11 000	11 000	12 000	8 000	5 000	4 400	4 000
Zirconia and AZ abrasives	22 000	21 800	14 000	14 000	17 000	20 000	18 000	13 000	8 000	8 500
Alloys	1 600	2 800	1 800	3 000	3 000	3 000	2 000	5 000	3 100	4 500
Foundry applications	80 000	46 200	67 000	69 000	72 000	75 000	55 000	75 000	46 000	49 000
Other	30 400	18 000	32 700	34 000	34 000	32 000	32 000	27 000	16 500	17 000
Total	174 000	128 800	155 000	162 000	164 000	168 000	140 000	150 000	93 000	100 000

*To convert to metric tons, multiply by 0.9072.
Source: BuMines, Annual Mineral Year Books; 1974–1983.

Table IV. Major Zircon Producers and Capacity

Producer	Amount (metric tons)	
Allied Eneabba, West Australia	160 000	
E. I. du Pont de Nemours & Co., Inc., Florida, U.S.	70 000	
DuPont Group	230 000	(34%)
Richards Bay Minerals, South Africa	150 000	
Consolidated Rutile, Ltd., East Australia	40 000	
General Mining Group	190 000	(28%)
Associated Minerals Consolidated Ltd., West Australia	110 000	
Associated Minerals Consolidated Ltd., East Australia	15 000	
Associated Minerals (U.S.A.) Ltd., Inc., Florida, U.S.	35 000	
Consolidated Goldfields Group	160 000	(23%)
Others, East and West Australia	100 000	(15%)
Total Mine Capacity, Australia, South Africa, U.S.:	680 000	(100%)

Source: TAM Ceramics, Inc., Market Research.

Australia, South Africa, and the U.S. (See Table IV).

For many ceramic applications, it is desirable to further process zircon sand by calcining, acid washing, magnetic treatment, dry ball-milling or wet ball-milling. Up-graded zircon sand and milled zircon products are produced in the U.S. by American Minerals, Inc. (formerly C-E Minerals), Continental Minerals Processing Corp., M & T Chemicals, Inc., and TAM Ceramics, Inc.

Of the zircon normally consumed annually in the U.S., about 15% goes for nonceramic uses such as metals, alloys, chemicals, and welding fluxes. A little under 50% is typically used in foundry sands, mold washes, and investment casting molds. Foundry uses take advantage of zircon's stability, high thermal conductivity, lack of reaction with molten metals, low toxicity, and availability in a sand or milled form. Foundry uses are quite cost sensitive and when zircon prices get too high zircon is often replaced by chromite, olivine, or aluminum silicates.

About 20% to 25% of zircon is used in refractories. This application utilizes zircon's refractoriness, thermal stability, and lack of reaction with many slags and glasses. Dense zircon shapes formed by casting or isostatic pressing are used for glass contact areas in glass tanks. Alumina-zirconia-silica (AZS) refractories made with zircon are used in a variety of applications. Zircon and AZS shapes are used in feeder parts or glass, kiln furniture, crucibles, and furnace linings. Usage in the U.S. steel industry is quite small, particularly when compared with the 91 000 metric tons (100 000 tons) of zircon used for lining Japanese steel ladles.

About 9% to 15% of the zircon consumed in the U.S. goes into the manufacture of zirconium oxide and alumina-zirconia abrasives.

Approximately 8%–10% of the zircon consumed in the U.S. goes into whitewares industry, largely as a glaze opacifier for tile and sanitary ware. Zircon has a high index of refraction, a light color, compatibility with ceramic colors, and a low solubility in glass. These properties make it the dominant opacifier for glazes. Very finely milled zircon is desirable for good opacification, and most zircon opacifiers have an average particle size of 1 to 2 μm. Zircon is also used in some glaze frits where the presence of zircon contributes to glaze opacification.

About 1% of the U.S. zircon consumption goes into miscellaneous ceramics, such as special glasses and porcelain enamels where the zircon contributes a high index of refraction, alkali resistance, radiation stability or opacity. Small quantities of zircon are also used in a variety of ceramic bodies. Zircon is added to fiber glass to improve the alkali resistance of fiber used for reinforcing concrete. ☐

ACerS Membership Profile

Grades of Membership

To meet the needs of all ceramists, the ACerS has established ten grades of membership. Table I shows the number of members in each grade as of March 31.

Regular Members

This represents the largest grade of membership in the ACerS. These members receive all the benefits of the ACerS and are eligible to join as many as three divisions.

Student Members

Student members must be enrolled for at least ten hours per week in a recognized college or university. They are involved with many activities of the ACerS, not only at the student branches located at various schools, but also at Annual Meetings, where they may represent their schools in the Student Speaking Contest and/or are employed as pages to assist in managing the Annual Meeting. Many graduate students present technical papers at the Annual and Divisional Meetings. Students also participate in section meetings and activities. Student members, however, do not qualify for Division affiliation, nor may they vote in Society elections beyond the student branch level.

Associate Members

Associate Members are 26 years of age or under, who do not qualify for student membership. Associate members are eligible for affiliation with as many as three ACerS divisions.

Table I. Grades of Membership

Grade, as of March 31	Number
Regular Members	7080
Student Members	1357
Emeritus Members	384
Associate Members	329
Paid Life Members	249
Corporate Members	237
Senior Members	58
Complimentary Members	46
Distinguished Life Members	32
Honorary Members	3*
Total	9775

*Honorary Membership first bestowed during the ACerS Annual Meeting, May 7, 1985.

Paid Life Members

Persons interested in paid life membership pay one lump sum for permanent membership based on their age. Payments may be spread out over a two-year period. By paying in advance, members protect themselves from inevitable dues increases. Paid life members also enjoy the full benefits of Society membership.

Corporation Members

Some 237 corporations materially aid and support the objectives of the ACerS through their corporation membership. Corporate members select a representative to act as voter in Society affairs.

Other Members

The remaining five types of membership include senior member, emeritus member, distinguished life member, complimentary member, and, most recently added, honorary member. These grades are bestowed for long service to the Society and the ceramic community and/or for notable professional achievements.

Information on joining is available from the American Ceramic Society, 65 Ceramic Dr., Columbus, OH 43214 (614/268-8645).

Designation: C 775 – 79 (Reapproved 1997)$^{\epsilon 1}$

Standard Method for Particle-Size Analysis of Whiteware Clays[1]

This standard is issued under the fixed designation C 775; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

$^{\epsilon 1}$ Note—Section 16 was added editorially in October 1997.

1. Scope

1.1 This method covers the quantitative determination of the distribution of particle sizes in kaolins and ball clays in the size range 44 to 0.1 µm by the sedimentation process. Particles are allowed to settle under normal gravitational forces and concentrations of particles in the size range 44 to 2 µm are measured using a hydrometer. Centrifuging is used to cause the smaller particles to settle more rapidly so that concentrations of particles in the size range 2 to 0.1 µm may be measured using a pipet.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[2]

E 100 Specification for ASTM Hydrometers[3]

3. Definition

3.1 *equivalent spherical diameter*—the diameter of a spherical particle having the same density and sedimentation rate in the same fluid as the clay particles being tested.

4. Apparatus

4.1 *Stirring Apparatus*—A mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 r/min without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate at not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

Note 1—A few clays are difficult to disperse and for these clays the use of a Waring blender[4] or equivalent is recommended.

4.2 *Centrifuge*—The centrifuge shall have bottles with a capacity of approximately 250 mL, and the rotational speed shall be closely controlled and measured using a tachometer. A timing device should be used to stop the centrifuge after predetermined time intervals.

4.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for Hydrometer 152H in Specification E 100.

4.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 360 ± 20 mm from the bottom on the inside.

4.5 *Sieve*—A No. 325 (45-µm) sieve conforming to the requirements of Specification E 11.

4.6 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the clay suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspensions at a convenient constant temperature at or near 25°C (77°F). Such a device is illustrated in Fig. 3. In cases where the work is performed in a room at an automatically controlled temperature, the water bath is not necessary.

5. Records

5.1 *Dispersing Agent*—Dissolve 2 g of sodium hexametaphosphate (sometimes called sodium metaphosphate ($(NaPO_3)$)) in 1 L of distilled or demineralized water.

Note 2—Some fine-grained clays require more of the dispersant and up to 6 g/L of water may be added after trials have been run to determine the percentage of dispersant causing the maximum fluidity using a viscometer suitable for clay-water suspensions.

Note 3—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

5.2 *Water*—All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail

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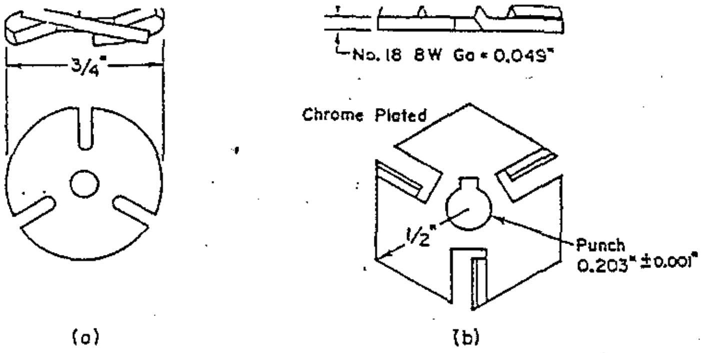
[1] This method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.04 on Clays.

Current edition approved Aug. 31, 1979. Published November 1979. Originally published as C 775 – 74. Last previous edition C 775 – 74.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] Available from Waring Products Div., Dynamics Corp. of America, New Hartford, CT 06057.



(a) (b)

Metric Equivalents

in.	0.001	0.049	½	¾
(mm)	(0.03)	(1.24)	(12.7)	(19.0)

FIG. 1 Detail of Stirring Paddles

during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 25°C (77°F). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

6. Hydrometer Test and Sieve Analysis

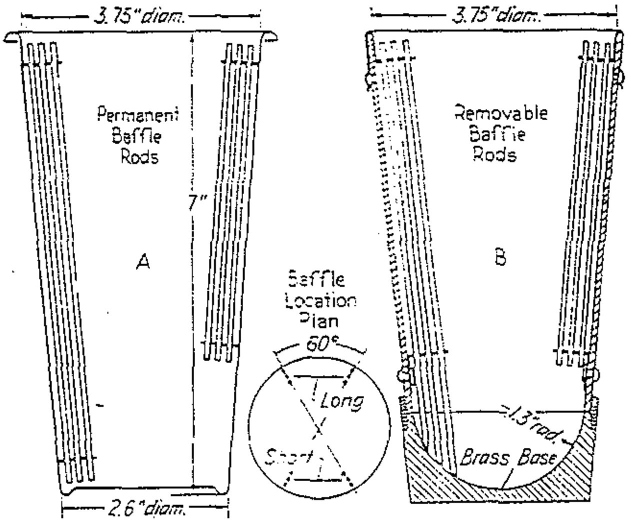
6.1 *Determination of Composite Correction for Hydrometer Reading:*

6.1.1 Equations for percentages of clay remaining in suspension, as given in Section 13, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

6.1.2 Calibrate the hydrometer at 20°C (68°F). Variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings.

NOTE 4—The amount of the inaccuracy increases as the variation from the standard temperature increases.

6.1.3 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid



Metric Equivalents

in.	1.3	2.6	3.75
(mm)	(33)	(66)	(95.2)

FIG. 2 Dispersion Cups of Apparatus

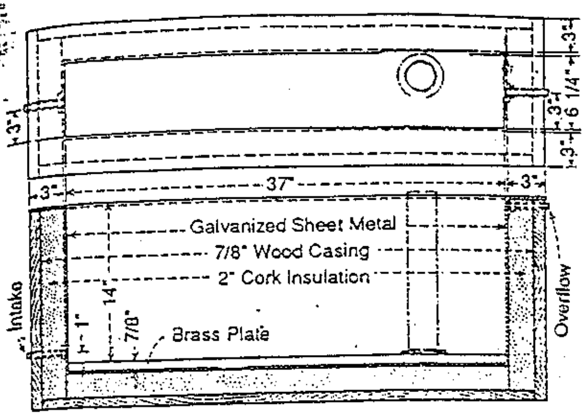


FIG. 3 Insulated Water Bath

on the stem. Since it is not possible to secure readings of the clay suspensions at the bottom of the meniscus, readings must be taken at the top and a correction factor applied.

6.1.4 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

6.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperature, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

6.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and the dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in the sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For Hydrometer 152H, it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

7. Hygroscopic Moisture

7.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion from 10 to 15 g in a small metal or glass container, dry the sample to a constant weight in an oven at 110 ± 5°C (230 ± 9°F), and weigh again. Record the weights.

8. Dispersion of Clay

8.1 Weigh out a sample of 50 g of air-dry clay. Place the sample in a 500-mL beaker and cover with 250 mL of the dispersing agent (2 g/L). Stir until the clay is thoroughly wetted. Allow to soak for at least 24 h.

8.2 At the end of the soaking period disperse the sample further, using the stirring device. Transfer the clay-water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the special dispersion cup using distilled or demineralized water. Add more of the distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 10 min.

9. Sieve Analysis

9.1 Transfer the suspension to a No. 325 (45-μm) sieve, collecting the suspension passing through the sieve for use in the hydrometer test. Rinse the special dispersion cup with a small amount of distilled or demineralized water and pour through the sieve. Transfer the material on the No. 325 sieve to a suitable container, dry in an oven at 110 ± 5°C (230 ± 9°F) and weigh. Record the weight.

10. Hydrometer Test

10.1 Immediately after dispersion and passing through a No. 325 (45-μm) sieve, transfer the clay-water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 5). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 240, and 360 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2 and 5-min readings.

NOTE 5—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any clay remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due, to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 6—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting a thermometer into the suspension.

11. Pipe Test After Centrifuging

11.1 Weigh out a sample of approximately 50 g of air-dry clay. Disperse the clay in 250 mL of the dispersing agent (2 g/L) and allow to soak for 24 h in the same manner as described for the hydrometer sample in Section 8. Disperse the sample further using the stirring device and pass through a No. 325 (45-μm) sieve as described in Section 9. Transfer the suspension passing through the No. 325 sieve to a glass 1000-mL graduate and then dilute with distilled or demineralized water to within ½ in. (12.7 mm) of the 1000-mL graduation mark. Place the suspension in the graduate in the constant-temperature water bath maintained at a temperature of 25°C (77°F). When the suspension reaches the temperature of the bath fill the graduate to the 1000-mL graduation using distilled or demineralized water.

11.2 The centrifuge bottles used in this portion of the test are illustrated in Fig. 4. Place a mark on the side of each bottle at a level that represents a condition of the bottle filled with 250 mL of the clay-water suspension. This may be accomplished by using a 250-mL volumetric flask filled with water to add to each to the centrifuge bottles for marking of the desired level.

11.3 Using the palm of the hand over the open end of the graduate (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min as described in 10.2. Fill one of the centrifuge bottles to the 250-mL calibration mark placed there previously.

11.4 Centrifuge the suspension in the bottle for 24.45 min at a corrected centrifuge speed of 500 r/min. Measure the temperature of the suspension after centrifuging and record the average temperature of the suspension during centrifuging. Place the end of the pipet 75 mm below the surface o the clay-water suspension as shown in Fig. 4 and withdraw 5-mL specimen. Place the specimen in a tared weighing disl and evaporate to dryness at a temperature of 110 ± 5°C (23 ± 9°F) and weigh to determine the concentration of the cla in the suspension after centrifuging in grams per cubic centimetre.

11.5 Prepare three additional specimens of the clay-wate suspension in the same manner as described in 11.3 an centrifuge at a speed of either 1000, 2000, or 2500 r/min fo a period of either 23.50, 36.70, or 93.90 min, respectively After each centrifuging, take specimens at a depth of 75 mm below the surface of the suspension, dry and weigh a described in 11.4 to determine the concentration of the cla in the suspension in grams per cubic centimetre.

12. Calculation

12.1 *Hygroscopic Moisture Correction Factor*—The hy-

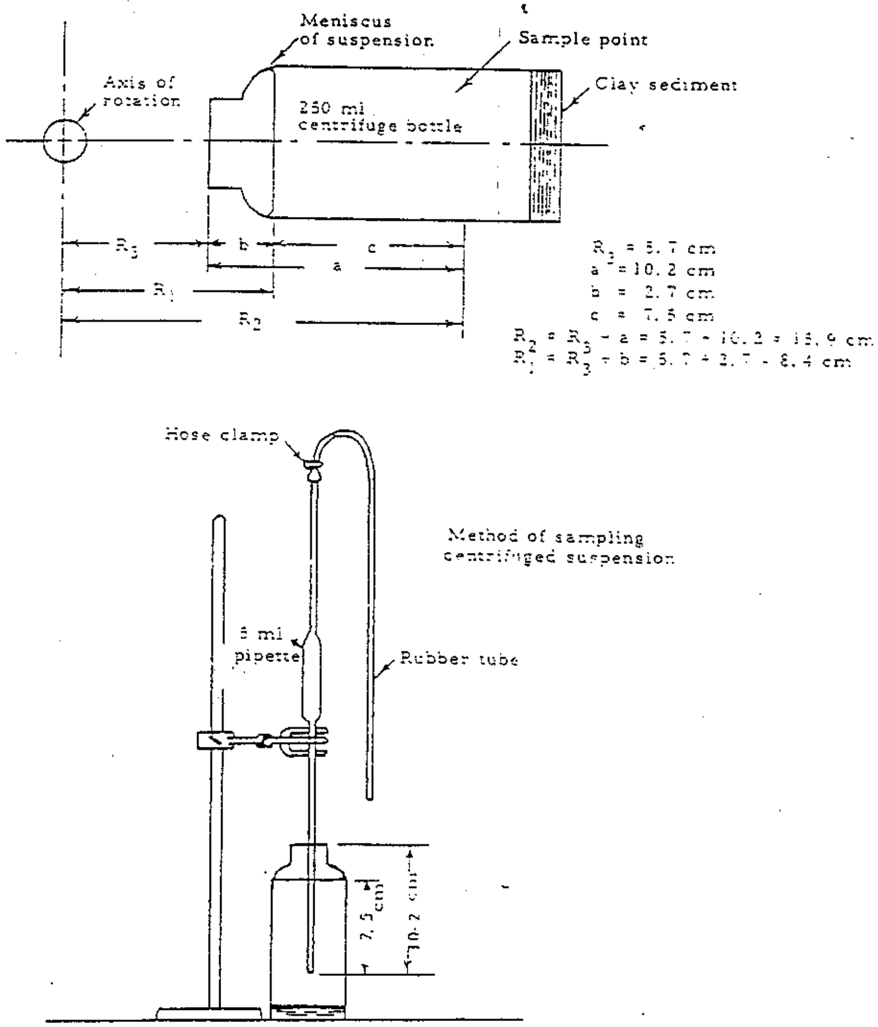


FIG. 4 Centrifuge Bottle and Sampling Pipet

groscopic moisture correction factor is the ratio between the weight of the oven-dried sample and the air-dry weight before drying. It is a number less than one, except when there is no hygroscopic moisture.

12.2 *Percentage of Coarse Material in Clay Sample:*

12.2.1 Calculate the oven-dry weight of clay used in the hydrometer analysis by multiplying the air-dry weight by the hygroscopic moisture correction factor.

12.2.2 Calculate the percentage of the clay sample that is coarser than 45 µm by dividing the weight of the material remaining on the No. 325 (45-µmm) sieve by the oven-dry weight of clay used for the hydrometer experiment, and multiplying the result by 100.

12.3 *Percentages of Clay in Suspension in Hydrometer Experiment*—Calculate the percentage of clay remaining in suspension at the level at which the hydrometer is measuring the density of the suspension as follows (Note 7):

NOTE 7—Hydrometers are easily broken and sometimes require considerable time for purchase. The instructions for sampling using the pipet (Section 11) may be substituted after appropriate time interval for sedimentation or centrifuging has elapsed.

For Hydrometer 152H:

$$P_h = (Ra/W) \times 100$$

where:

a = a correction factor of 1.01 to be applied to the reading of Hydrometer 152H because the hydrometer is calibrated for solids having a specific gravity of 2.65 and clay is considered to have a specific gravity of 2.60,

P_h = percentage of clay remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with the composite correction applied (Section 6), and

W = oven-dry weight of clay in total test samples as represented by weight of clay dispersed (see 12.2.1), g.

12.4 *Diameter of Clay Particles:*

12.4.1 Calculate the diameter of a particle corresponding to the percentage indicated by a given hydrometer reading in accordance with Stokes' law (Note 8) on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. The particle diameter may be calculated in accordance with Stokes' law as follows:

$$D = \sqrt{[30\eta/980\,(G - G_1)] \times (L/T)}$$

where:

D = diameter of particle, µm.

η = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This depth is known as effective depth. See Table 1.), cm,

T = interval of time from beginning of sedimentation to the taking of the reading, min,

G = specific gravity of clay particles, which is considered to be 2.60, and

G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 8—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the clay particles.

12.4.2 For convenience in calculations, the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

TABLE 1 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
0	16.3	31	11.2
1	16.1	32	11.1
2	16.0	33	10.9
3	15.3	34	10.7
4	15.6	35	10.5
5	15.5		
6	15.3	36	10.4
7	15.2	37	10.2
8	15.0	38	10.1
9	14.3	39	9.9
10	14.7	40	9.7
11	14.5	41	9.6
12	14.3	42	9.4
13	14.2	43	9.2
14	14.0	44	9.1
15	13.3	45	8.9
16	13.7	46	8.8
17	13.5	47	8.6
18	13.3	48	8.4
19	13.2	49	8.3
20	13.0	50	8.1
21	12.9	51	7.9
22	12.7	52	7.6
23	12.5	53	7.6
24	12.4	54	7.4
25	12.2	55	7.3
26	12.0	56	7.1
27	11.9	57	7.0
28	11.7	58	6.8
29	11.5	59	6.5
30	11.4	60	6.5

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,

L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,

L_2 = overall length of the hydrometer bulb, cm,

V_B = volume of hydrometer bulb, cm³, and

A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table 1 are as follows:

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/L

= 2.3 cm for a reading of 50 g/L

L_2 = 14.0 cm

V_B = 67.0 cm³

A = 27.8 cm²

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TABLE 2 Values of K for Use in Equation for Computing Diameter of Particles Having a Specific Gravity of 2.60 in Hydrometer Analysis

Temperature, °C	K Values
16	0.01457
17	0.01439
18	0.01421
19	0.01403
20	0.01386
21	0.01369
22	0.01353
23	0.01337
24	0.01321
25	0.01306
26	0.01291
27	0.01277
28	0.01264
29	0.01249
30	0.01236

K = constant depending on the temperature of the suspension and the specific gravity of the clay particles. Values of K for a range of temperatures and the specific gravity of the clay particles considered as 2.60 are given in Table 2. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

12.5 *Percentages of Clay in Suspension After Centrifuging*—Calculate the percentage of clay remaining in suspension at the level at which the pipet specimen is withdrawn 75 mm below the surface as follows:

$$P_p = [(200 \times W_c)/W] \times 100$$

where:

W_c = oven-dry weight of clay in the 5-mL test specimen removed at a depth of 75 mm below the surface of the suspension after centrifuging, g.

W = oven-dry weight of clay in the total test sample as represented by weight of clay dispersed (see 12.2.1), g.

P_p = percentage of clay remaining in suspension at the level at which the pipet specimen is withdrawn 75 mm below the surface of the suspension.

12.6 *Diameter of the Clay Particles*—Calculate the diameter of a particle corresponding to the percentage indicated by a given pipet specimen in accordance with Stokes' law, reduced to the following form, on the basis that a particle of this diameter was at the surface of the suspension before centrifuging and had settled to the level at which the pipet specimen is withdrawn after centrifuging:

$$D = \sqrt{[18n/(G - G_1)tw^2][\ln(R_2/R_1)]}$$

where:

D = diameter of particle, µm,

n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

G = specific gravity of clay particles, which is considered to be 2.60,

TABLE 3 Centrifuging Conditions[A]

Equivalent Spherical Diameter, µm	Corrected Centrifuge Speed, r/min	Angular Velocity, rad/s	Centrifuging Time at 25.0°C, min
1	500	52.4	23.45
0.5	1000	104.7	23.50
0.2	2000	209.4	36.70
0.1	2500	261.8	93.90

[A] Calculated on the basis that R_1 = 8.4 cm and R_2 = 15.9 cm.

G_1 = specific gravity (relative density) of suspending medium (value changes very slightly depending upon the average test temperature);

t = centrifuging time, min,

w = angular velocity, as listed in Table 3, rad/s, and

R_1 and R_2 = radii of rotation of the container placed in the centrifuge as shown in Fig. 4.

13. Graph

13.1 Make a graph of the test results, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate.

14. Report

14.1 Report the following information:

14.1.1 Name or number of the clay sample,

14.1.2 Form of the sample (lumps, granular, air floated),

14.1.3 Percentage of hygroscopic moisture,

14.1.4 Equipment used for dispersion,

14.1.5 Time of storage in water,

14.1.6 Time of dispersion,

14.1.7 Type of dispersant used, and

14.1.8 Cumulative weight percent of the sample finer than 44 µm, 10 µm, 5 µm, 2 µm, 1 µm, 0.5 µm, 0.2 µm, and 0.1 µm, respectively.

14.1.9 Comparison of different samples of the same clay is often simplified by reporting only a single size. Thus china clays might be compared by reporting only the cumulative weight percent of the clay finer than 2 µm. Ball clays might be compared by reporting only the cumulative weight percent of the clay finer than 0.5 µm.

15. Precision and Bias

15.1 The precision for values of cumulative weight percent finer than 44 µm, 10 µm, 5 µm, and 2 µm is ±3 %. However, for the values of cumulative weight percent finer than 1 µm and 0.5 µm, the precision is ±5 %.

15.2 The geometric mean diameter calculated from these measurements for one clay should vary less than ±0.2 µm from laboratory to laboratory.

16. Keywords

16.1 clay; particle size

Designation: C 325 – 81 (Reapproved 2007)

Standard Test Method for Wet Sieve Analysis of Ceramic Whiteware Clays[1]

This standard is issued under the fixed designation C 325; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method covers the wet sieve analysis of eramic whiteware clays. This test method is intended for use testing shipments of clay as well as for plant control tests.

1.2 *This standard does not purport to address all of the afety concerns, if any, associated with its use. It is the esponsibility of the user of this standard to establish appro-riate safety and health practices and determine the applica-ility of regulatory limitations prior to use.*

Referenced Documents

2.1 *ASTM Standards:* [2]

C 322 Practice for Sampling Ceramic Whiteware Clays

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Apparatus

3.1 *Stirring Device*—A mechanical stirrer with a three-bladed propeller 2 in. (51 mm) in diameter and having a speed of approximately 1700 r/min, or the equivalent, shall be provided.

3.2 *Sieves*—The sieves shall conform to Specification E 11 and shall include the No. 100 (150-μm), No. 140 (106-μm), No. 200 (75-μm) and No. 325 (45-μm) sieves (Note 1). The wire cloth for these sieves shall be woven (not twilled) and shall be mounted in circular metal frames 8 in. (203 mm) in diameter, which shall be so constructed as to permit nesting of two or more sieves. A pan and cover for the sieves shall be provided.

Note 1—Equivalent sieves from other standard series, such as the Tyler series, may also be used. If results are to be compared with those obtained with sieves from the ASTM series, it is important that the openings of the sieves used fall within the tolerances specified in Specification E 11 for the corresponding ASTM sieves.

4. Sampling

4.1 The sample shall be obtained in accordance with Practice C 322.

4.2 The sample as received shall be placed in a drying oven at 100 to 110°C for a period of not less than 5 h prior to testing.

5. Procedure

5.1 Transfer duplicate portions, of approximately 250 g of the dried clay sample, weighed to the nearest 0.1 g, to containers of at least 2-L capacity. Wet the clay with 1 L of water and allow to slake for 2 h. If a free-flowing slurry is not produced by this treatment, add another 500 mL of water.

5.2 To ensure complete separation of clay from nonplastic impurities, agitate the slurry by means of a mechanical stirrer (3.1). Continue the stirring between 5 and 10 min.

5.3 Transfer the slaked and stirred sample, without loss, to the finest sieve to be employed in the test, and wash by means of a small jet of water from a ¼-in. (6.4-mm) soft rubber hose attached to a water supply having a pressure not in excess of that of an ordinary city main. The force of the jet may be controlled by compressing the end of the hose between the thumb and forefinger. Take care to avoid loss of sample from splashing. Continue washing until water passing through the sieve shows only traces of sample. Should lumpy material remain on the screen, return the residue to the stirrer container by careful washing with a gentle jet of water, and agitate in approximately 1 L of water for 10 min, then wash the slurry as previously described.

5.4 Wash the residue remaining on the finest sieve into the pan. Thoroughly wet the remaining sieves to be used in the test with clear water, and nest them in the proper sequence on the finest sieve. Wash the residue in the pan quantitatively onto the top sieve, and give the stack a preliminary washing.

5.5 Nest the top sieve on the pan, which shall contain about ½ in. (12.7 mm) of clear water. Wash the residue by holding the pan and sieve firmly in the hands, and by a sidewise movement, causing water to splash up through the sieve and into the residue. This movement, coupled with interspersed

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[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.04 on Raw Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

circular motions, allows thorough washing. Wash the residue and water remaining in the pan onto the top sieve of the stack.

5.6 Again fill the pan with the proper amount of water, nest the top sieve and its residue on the pan, and repeat the operation. Continue this until the finest sieve has been washed. Carefully blot each sieve on its underside with a soft, damp sponge, and place the sieve either in a drying oven at 100 to 110°C or under infrared lamps until thoroughly dry. Approximately 2 h is required with a drying oven, but only about 30 min with an infrared lamp set 12 in. (305 mm) above the sieve.

5.7 Nest the dried residues and sieves in the proper order, with due care to prevent dusting of the residues. Close the stack of sieves with a dry pan and cover, and tap the assembly lightly for 1 min on a table top.

5.8 Separate the nested sieves and carefully brush the residue from each onto a weighing paper. Weigh the residues to the nearest 0.001 g on an analytical balance.

6. Calculation and Report

6.1 Calculate the sieve analysis for test sample on the dry weight basis, and report the results to the nearest 0.01 % of the material retained on each sieve. Report the percentage passing the finest sieve as the difference between 100 % and the sum of the percentages retained on the various sieves.

7. Precision and Bias

7.1 The true value of the particle size can be defined only in terms of a test method. Within this limitation, this test method has no known bias.

8. Keywords

8.1 clay; sieve analysis; wet sieve

Designation: C 326 – 82 (Reapproved 1997)$^{\epsilon 1}$

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Drying and Firing Shrinkages of Ceramic Whiteware Clays[1]

This standard is issued under the fixed designation C 326; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—Section 7 was added editorially in October 1997.

Scope

1.1 This test method covers the determination of linear rinkage of ceramic whiteware clays, both unfired and fired.

1.2 *This standard does not purport to address all of the fety concerns, if any, associated with its use. It is the ponsibility of the user of this standard to establish appropiate safety and health practices and determine the applicaility of regulatory limitations prior to use.*

Significance and Use

2.1 The purpose of this test is to obtain values of shrinkage ter drying and firing of clays or bodies or both, under various ocessing conditions to enable designers to determine the oper size of mold or die so as to produce a predetermined of fired ware.

Test Specimens

.1 Test specimens shall be either round bars approximately mm in diameter by 115 mm in length, or bars of square s section approximately 25 by 25 by 115 mm in dimens.

2 Test specimens may be prepared either by casting or tic forming, as circumstances require. At least five specis shall be prepared. For cast specimens the molds may be r one-piece, or two- or more-part molds; in either instance cient space shall be provided to allow solid casting without g. Where plastic-forming is employed, the clay-water s shall be brought to a consistency that permits the making pecimens rigid enough to allow careful handling without rtion immediately after the test specimen is made. Plasticed test specimens shall be made either by extrusion or by ing in a suitable metal mold. Where a vacuum pugmill is a vacuum of not less than 25 in. (635 mm) Hg shall be tained during the forming operation. Where no vacuum ment is used, the plastic clay shall be thoroughly handed to eliminate entrapped air as a preliminary to forming pieces.

The test specimens, cast or plastic-formed, shall be suitably identified and marked with shrinkage reference lines 100 mm apart on the long axis of the specimen.

3.4 The marked specimens shall then be placed on a lightly oiled pallet and allowed to dry at 20 to 40°C for 24 h. During this preliminary drying period, bars of square cross section shall be turned 90 deg several times at 2-h intervals to eliminate possible warping. After the initial drying period, the specimens shall be placed in a drying oven at 100 to 110°C and further dried for 24 h.

3.5 The drying shrinkage of the test specimens should be determined in accordance with Sections 4 and 5. The specimens can now be fired according to a suitable firing schedule to the desired temperature and the procedures of Sections 4 and 5 applied to determine the total shrinkage.

4. Shrinkage Measurement

4.1 Measure the distance between shrinkage reference marks on dried or fired specimens to the closest 0.1 mm with vernier calipers. Record the average of at least five measurements (one measurement on each of five specimens).

5. Calculation

5.1 Calculate the linear drying shrinkage as a percentage of plastic length, as follows:

$$S_d = \frac{L_p - L_d}{L_p} \times 100 \qquad (1)$$

where:

S_d = linear drying shrinkage, %,
L_p = plastic length of test specimen, and
L_d = dry length of test specimen.

5.2 Calculate the total linear shrinkage after drying and firing of clay shrinkage specimens as a percentage of plastic length, as follows:

$$S_t = \frac{L_p - L_f}{L_p} \times 100 \qquad (2)$$

where:

S_t = total linear shrinkage after drying and firing, %,
L_p = plastic length of test specimen, and
L_f = fired length of test specimen.

5.3 When desired, volume shrinkage may be calculated from linear shrinkage, as follows:

$$\text{Volume shrinkage, \%} = [1 - (1 - S/100)^3]\ 100 \qquad (3)$$

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s test method is under the jurisdiction of ASTM Committee C-21 on Whitewares and Related Products and is the direct responsibility of mittee C21.04 on Clays.

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where:

S = linear shrinkage, %.

5.4 The shrinkage factor is used to convert fired sizes to plastic sizes or mold sizes. Calculate as follows:

$$\text{Shrinkage factor} = L_p/L_f \tag{4}$$

where:

L_p = plastic length of test specimen, and

L_f = fired length of test specimen.

6. Precision and Bias

6.1 The direction of flow in forming influences the orientation of the clay particles and the shrinkage. The shrinkage value should be related to the method of forming as well as t dimension of the ware.

6.2 When the original reference points are 100 mm apa the precision of the shrinkage measurement is ± 0.1 %.

6.3 Measurements of drying shrinkage on one dimension specimens all formed by the same method should yield standard deviation of 0.1 % or less.

6.4 Measurement of total linear shrinkage after drying a firing on one dimension of specimens should yield a standa deviation of 0.25 % or less.

7. Keywords

7.1 clay; drying shrinkage; firing shrinkage

Designation: C 689 – 03a

Standard Test Method for Modulus of Rupture of Unfired Clays[1]

This standard is issued under the fixed designation C 689; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the modulus of rupture of ceramic whiteware clays both dry and after conditioning at 50 or 80 % relative humidity, or both.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 322 Test Method for Sampling Ceramic Whiteware Clays

3. Apparatus

3.1 *Conditioning Cabinet*—Any suitable airtight cabinet with means for circulating air, or vacuum desiccator, wherein prescribed specimens can be conditioned as desired before testing. Specimens for dry strength shall be stored with silica gel desiccant. Specimens for 50 % relative humidity shall be stored over a saturated solution of sodium dichromate ($Na_2CR_2O_7 \cdot 2H_2O$), and specimens for 80 % relative humidity shall be stored over a saturated solution of ammonium chloride (NH_4Cl).

3.2 *Testing Machine*—Any suitable testing machine may be used, provided a uniform rate of direct loading can be maintained at no more than 1 lb/min (4.4 N/min) using the prescribed specimens. For the support of the test specimen, two steel bars having a diameter of 0.5-in. (12.7-mm) shall be provided. The load shall be applied by means of a third steel bar having a diameter of 0.5-in. (12.7-mm). All three bars are to be smooth polished steel without surface defects such as scratches or gouges.

4. Test Specimen Preparation

4.1 *Preparation of Extruded Specimens*—100 % clay specimen preparation: The test sample shall be blunged with sufficient distilled water to give complete dispersion and produce a slip of sufficient viscosity to avoid noticeable settling of particles (usually in the range of 25 to 50 percent solids). Slip shall be sieved through a No. 120 (125 µm) sieve or equivalent. After aging 24 h the slip shall be dewatered to a plastic condition preferably by filter pressing. At this point the moisture content must be adjusted to optimum conditions for forming by some measure of plasticity such as Pfefferkorn apparatus, plumb bob penetration, or other acceptable means. After the water of plasticity has been adjusted (usually will range from 20 to 50 %), test specimens shall be formed by extrusion. Where hand-operated extruder is used without vacuum, the plastic clay shall be thoroughly hand-wedged to eliminate entrapped air as a preliminary to forming test pieces. Where a vacuum extruder is used, a vacuum of not less than 25 in. (635 mm) Hg shall be maintained during forming operation.

4.1.1 *Clay-Flint Specimens*—to be used in the case of strong clays that are considered bonding materials. Prepare specimens as in 4.1 using a mixture of clay and 200 mesh potters flint in a 1/1 ratio blend that has been thoroughly dry mixed.

4.1.2 *Solid Cast Specimens*—100 % clay and clay-flint specimens should be prepared by making a high solids slurry deflocculated to minimum viscosity. Adjust the solids content of the slurry to obtain a viscosity between 100-500 cps. The slurry should be cast in plaster molds until solid, and the bars then dried in accordance to the procedure outlined in 4.3 and 4.4.

4.2 *Dimension*—The test specimens shall be round bars of 0.50 (12.8 mm) diameter, and at least 4.5 in. (114 mm) in length to permit an overhang of at least 0.25 in. at each end when mounted on the supports.

4.3 *Handling and Warpage*—All due precaution shall be observed in the forming and drying to produce straight test specimens. No specimen shall be used that shows a warpage greater than 1 % of its overall length. The bars shall be checked from time to time during drying for straightness, and before they stiffen, corrective straightening may be encouraged by

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

rotating the bars so that drying occurs from another surface region. Defective bars due to warpage, flaws, or voids shall be rejected.

4.4 *Drying and Storage*—The extruded specimens shall be placed on a lightly oiled pallet and allowed to dry at room atmospheric conditions 68 to 104°F (20 to 40°C) for 24 h. Then the specimens shall be dried in a drier at 140°F (60°C) and low relative humidity for 6 h, or until moisture content is less than 0.5 %. The bars shall be loosely stacked in the desiccator to permit rapid cooling. Bars should be cooled to near room temperature but not longer than one (1) hour. After the initial drying period, the specimens for dry strength shall be further dried at 212 to 230°F (100 to 110°C) for 24 h and then cooled in a desiccator before testing. The bars shall be loosely stacked in the desiccator to permit rapid cooling. Bars should be cooled to near room temperature but not longer than one (1) hour. After the initial drying, the specimens for testing at 50 or 80 % relative humidity shall be placed into the conditioning cabinet with circulating air (or in partial vacuum) with a saturated solution of sodium dichromate or ammonium chloride, respectively, and in each case, stored for 24 h to allow equilibrium conditions to be reached.

5. Procedure

5.1 Test at least ten specimens at room temperature for each condition, whether dry or conditioned, at 50 or 80 % relative humidity.

5.2 Remove the test specimen from storage, one at a time, and immediately place on the round bar supports of the testing machine. These supports shall be spaced 4 in. (102 mm) apart, depending on the type machine and type clay, and test specimen must overlap each support by at least 0.25 in. (6.4 mm). Apply the load at right angles to the specimen and midway between the supports. Apply the load at a uniform rate not to exceed 1 lb-f/min (4.4 N/min) until failure occurs. The loading rate should be such as to cause failure in approximately one minute. Measure the diameter at the break to the nearest 0.001 in. (0.03 mm). Use the average of at least three diameter readings around the bar.

6. Calculation

6.1 Calculate the modulus of rupture for each specimen as follows:

$$M = 8PL/\pi d^3 \tag{1}$$

where:

M = modulus of rupture, psi (or MPa),
P = load at rupture, lbf (or N),
L = distance between supports, in. = 2.0 or 4.0 (51 or 102 mm), and
d = diameter of specimen, in. (or mm).

7. Report

7.1 Report the following information:

7.1.1 Identification of the material tested,

7.1.2 Data and computed modulus of rupture for each specimen,

7.1.3 Adjusted average of the computed modulus of rupture values (discarding those values from the bars which, upon inspection, show obvious defects), or deviate more than ± 10% from the adjusted avaerage. Average again.

7.1.4 Drying and storage conditions; that is, whether the tes specimens were bone dry, or conditioned at 50 or 80 % relative humidity,

7.1.5 Percent moisture of extruded bars,

7.1.6 Nominal diameter of bars,

7.1.7 Length of span (distance between supports).

7.2 The report may also include:

7.2.1 Percent moisture of each specimen at testing,

7.2.2 Description of type of fracture and the behavior unde load of each specimen,

7.2.3 Name and rating of the machine used to make the tes and

7.2.4 Curve sheet showing the individual values of modulu of rupture arranged in ascending order.

8. Precision and Bias

8.1 *Precision*—The precision of this test method is approx mately ±10 % of the average modulus of rupture value for t bone dry specimens when the measurements are made by a experienced operator.

8.2 *Bias*—The bias of this test method cannot be specifie

9. Keywords

9.1 clay; modulus of rupture

Standard Test Method for
Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products[1]

This standard is issued under the fixed designation C 373; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers procedures for determining water absorption, bulk density, apparent porosity, and apparent specific gravity of fired unglazed whiteware products.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 Measurement of density, porosity, and specific gravity is a tool for determining the degree of maturation of a ceramic body, or for determining structural properties that may be required for a given application.

3. Apparatus and Materials

3.1 *Balance*, of adequate capacity, suitable to weigh accurately to 0.01 g.

3.2 *Oven*, capable of maintaining a temperature of 150 ± 5°C (302 ± 9°F).

3.3 *Wire Loop, Halter, or Basket*, capable of supporting specimens under water for making suspended mass measurements.

3.4 *Container*—A glass beaker or similar container of such size and shape that the sample, when suspended from the balance by the wire loop, specified in 3.3, is completely immersed in water with the sample and the wire loop being completely free of contact with any part of the container.

3.5 *Pan*, in which the specimens may be boiled.

3.6 *Distilled Water.*

4. Test Specimens

4.1 At least five representative test specimens shall be selected. The specimens shall be unglazed and shall have as much of the surface freshly fractured as is practical. Sharp edges or corners shall be removed. The specimens shall contain no cracks. The individual test specimens shall weigh at least 50 g.

5. Procedure

5.1 Dry the test specimens to constant mass (Note 1) by heating in an oven at 150°C (302°F), followed by cooling in a desiccator. Determine the dry mass, *D*, to the nearest 0.01 g.

NOTE 1—The drying of the specimens to constant mass and the determination of their masses may be done either before or after the specimens have been impregnated with water. Usually the dry mass is determined before impregnation. However, if the specimens are friable or evidence indicates that particles have broken loose during the impregnation, the specimens shall be dried and weighed after the suspended mass and the saturated mass have been determined, in accordance with 5.3 and 5.4. In this case, the second dry mass shall be used in all appropriate calculations.

5.2 Place the specimens in a pan of distilled water and boil for 5 h, taking care that the specimens are covered with water at all times. Use setter pins or some similar device to separate the specimens from the bottom and sides of the pan and from each other. After the 5-h boil, allow the specimens to soak for an additional 24 h.

5.3 After impregnation of the test specimens, determine to the nearest 0.01 g the mass, *S*, of each specimen while suspended in water. Perform the weighing by placing the specimen in a wire loop, halter, or basket that is suspended from one arm of the balance. Before actually weighing, counterbalance the scale with the loop, halter, or basket in place and immerse in water to the same depth as is used when the specimens are in place. If it is desired to determine only the percentage of water absorption, omit the suspended mass operation.

5.4 After the determination of the suspended mass or after impregnation, if the suspended mass is not determined, blot each specimen lightly with a moistened, lint-free linen or cotton cloth to remove all excess water from the surface, and determine the saturated mass, *M*, to the nearest 0.01 g. Perform the blotting operation by rolling the specimen lightly on the wet cloth, which shall previously have been saturated with water and then pressed only enough to remove such water as will drip from the cloth. Excessive blotting will introduce error

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[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns.

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by withdrawing water from the pores of the specimen. Make the weighing immediately after blotting, the whole operation being completed as quickly as possible to minimize errors caused by evaporation of water from the specimen.

6. Calculation

6.1 In the following calculations, the assumption is made that 1 cm^3 of water weighs 1 g. This is true within about 3 parts in 1000 for water at room temperature.

6.1.1 Calculate the exterior volume, V, in cubic centimetres, as follows:

$$V = M - S \tag{1}$$

6.1.2 Calculate the volumes of open pores V_{OP} and impervious portions V_{IP} in cubic centimetres as follows:

$$V_{OP} = M - D \tag{2}$$

$$V_{IP} = D - S \tag{3}$$

6.1.3 The apparent porosity, P, expresses, as a percent, the relationship of the volume of the open pores of the specimen to its exterior volume. Calculate the apparent porosity as follows:

$$P = [(M - D)/V] \times 100 \tag{4}$$

6.1.4 The water absorption, A, expresses as a percent, the relationship of the mass of water absorbed to the mass of the dry specimen. Calculate the water absorption as follows:

$$A = [(M - D)/D] \times 100 \tag{5}$$

6.1.5 Calculate the apparent specific gravity, T, of th portion of the test specimen that is impervious to water, follows:

$$T = D/(D - S)$$

6.1.6 The bulk density, B, in grams per cubic centimetre, a specimen is the quotient of its dry mass divided by t exterior volume, including pores. Calculate the bulk density follows:

$$B = D/V$$

7. Report

7.1 For each property, report the average of the val obtained with at least five specimens, and also the individ values. Where there are pronounced differences among individual values, test another lot of five specimens and, addition to individual values, report the average of all determinations.

8. Precision and Bias

8.1 This test method is accurate to ±0.2 % water absorpt in interlaboratory testing when the average value recorded all laboratories is assumed to be the true water absorption. precision is approximately ±0.1 % water absorption on m surements made by a single experienced operator.

9. Keywords

9.1 apparent porosity; apparent specific gravity; bulk sity; fired whiteware products; water absorption

Designation: C 329 – 88 (Reapproved 2006)

Standard Test Method for Specific Gravity of Fired Ceramic Whiteware Materials[1]

This standard is issued under the fixed designation C 329; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of specific gravity of fired ceramic whiteware materials under prescribed conditions.

NOTE 1—This test method is not applicable to materials attacked by water.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 153 Test Methods for Specific Gravity of Pigments

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Significance and Use

3.1 Measurement of specific gravity is a tool for determining the degree of maturation of a ceramic body.

4. Apparatus and Materials

4.1 *Analytical Balance and Weights.*

4.2 *Pycnometers*, of 50-mL capacity, consisting of suitable bottles with capillary tube stoppers.

4.3 *Thermometer*, calibrated at 0.5°C intervals in the room temperature range.

4.4 *Drying Oven.*

4.5 *Weighing Bottle.*

4.6 *Desiccator.*

4.7 *Vacuum Source*—A suitable apparatus to produce a vacuum equivalent to an absolute pressure of less than 1.0-in. (25.4-mm) Hg.

4.8 *Distilled Water*, that has been freshly evacuated, or boiled and cooled, to remove dissolved air.

5. Sample Preparation

5.1 When possible, the sample for test shall consist of at least two pieces totaling 100 to 150 g taken from different portions of the material in such a way as to exclude skin surfaces in so far as possible. The sample shall be selected so as to be representative of the material to be tested.

5.2 The pieces shall be crushed, if necessary, between hardened steel surfaces. The specimen shall then be reduced to 25 to 50 g by quartering, and any magnetic material introduced by crushing shall be removed. This specimen shall be ground in a suitable mortar so that it will pass a 150-μm (No. 100) sieve, conforming to Specification E 11, or its equivalent. Care shall be taken at all stages of the crushing, grinding, and quartering to minimize the introduction of impurities and retain all material even though difficult to grind.

6. Procedure

6.1 Make all determinations in duplicate. Determine all weights in this procedure to the nearest 0.0001 g.

6.2 Place the ground specimen in a glass weighing bottle and dry to constant weight at 105 to 110°C. Close the bottle with a glass stopper immediately upon removal from the oven.

6.3 Dry the pycnometer and stopper at 105 to 110°C, cool to room temperature in a desiccator, weigh on an analytical balance, and record the weight as p. Fill the pycnometer bottle with distilled water at room temperature, t_1, insert the stopper, and remove the excess water on the tip of the capillary by means of filter paper. Weigh the pycnometer and contents and record the weight as W_1. Empty and dry the pycnometer.

6.4 Place about 8 to 12 g of the dried specimen in the dry pycnometer; weigh the pycnometer, stopper, and specimen and record the weight as W. Add distilled water until the bottle is approximately one half full, and, to remove entrapped air, first stir the specimen and water thoroughly with a glass rod. Then remove the glass rod, using a small quantity of distilled water to wash back into the pycnometer any particles of specimen adhering to the rod. Finally subject the specimen and water to a reduced air pressure of less than 1.0-in. (25.4-mm) Hg (abs).

NOTE 2—A suitable method for evacuation of gas is described in 5.5 of Test Methods D 153.

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[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Absolute Density of Water

Temperature, °C	Absolute Density, g/cm^3									
	0	1	2	3	4	5	6	7	8	9
0	0.999 841	0.999 900	0.999 941	0.999 965	0.999 973	0.999 965	0.999 941	0.999 902	0.999 849	0.999 78
10	0.999 700	0.999 605	0.999 498	0.999 377	0.999 244	0.999 099	0.998 943	0.998 774	0.998 595	0.998 40
20	0.998 203	0.997 992	0.997 770	0.997 538	0.997 296	0.997 044	0.996 783	0.996 512	0.996 232	0.995 94
30	0.995 646	...	...	...	...	...	...	...	...	...

6.5 Fill the bottle, after evacuation with distilled water at room temperature, t_2, insert the stopper, and remove the excess water on the tip of the capillary by means of filter paper. Weigh the pycnometer and contents, and record the weight as W_2.

6.6 Temperatures t_1 and t_2 shall be reported to the nearest 0.5°C and shall not differ by more than 5°C.

7. Calculation

7.1 Calculate the specific gravity with respect to water at 4°C, as follows:

$$\text{Specific gravity} = \frac{d_1 d_2 (W - p)}{0.999973[d_2(W_1 - p) - d_1(W_2 - W)]} \quad (1)$$

where:

d_1 = absolute density of water (from Table 1) at temperature t_1 (6.3),

d_2 = absolute density of water (from Table 1) at temperature t_2 (6.5),

p = weight of the stoppered pycnometer (6.3),

W = weight of the stoppered pycnometer and specimen (6.4),

W_1 = weight of the stoppered pycnometer filled with water (6.3), and

W_2 = weight of the stoppered pycnometer, specimen, and water (6.5).

7.2 The absolute density of the specimen may be determined by following the directions in Section 6, but making certain that all weighings are made at identical temperatures and in a dry atmosphere. If this precaution is taken, the absolute density may be calculated as follows:

$$G = (W - p)/[(W_1 - p) - (W_2 - W)]$$

$$\text{Absolute density} = [G\,(d - a)] + a$$

where:

G = specific gravity with respect to water at temperature

d = absolute density of water (from Table 1) at temperatur t,

a = absolute density of air (from Table 2) at temperature and

t = temperature at which all weighings were made.

8. Report

8.1 Report the following information:

8.1.1 Designation of the material tested,

8.1.2 Data sheet showing all weights and water tempe tures. If the absolute density is required, air temperatures sh also be shown, and

8.1.3 Specific gravity (or absolute density, if require Duplicate determinations shall be reported to the nearest 0.0

9. Precision and Bias

9.1 It is generally accepted that duplicate determinatio should not differ by more than 0.005. Additional speci information to support a precision and bias statement is not available.

10. Keywords

10.1 fired ceramic whiteware materials; specific gravity

TABLE 2 Absolute Density of Dry Air at 760-mm Hg

Temperature, °C	Absolute Density, g/cm^3									
	0	1	2	3	4	5	6	7	8	9
0	0.001 293	0.001 288	0.001 284	0.001 279	0.001 274	0.001 270	0.001 265	0.001 261	0.001 256	0.001 2
10	0.001 247	0.001 243	0.001 239	0.001 234	0.001 230	0.001 226	0.001 221	0.001 217	0.001 213	0.001 2
20	0.001 205	0.001 201	0.001 196	0.001 192	0.001 188	0.001 184	0.001 180	0.001 176	0.001 173	0.001 1
30	0.001 165	...	...	...	...	...	...	...	...	...

Designation: C 773 – 88 (Reapproved 2006)

Standard Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials[1]

This standard is issued under the fixed designation C 773; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers two test procedures (A and B) for the determination of the compressive strength of fired whiteware materials.

1.2 Procedure A is generally applicable to whiteware products of low- to moderately high-strength levels (up to 150 000 psi or 1030 MPa).

1.3 Procedure B is specifically devised for testing of high-strength ceramics (over 100 000 psi or 690 MPa).

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 4 Practices for Force Verification of Testing Machines
E 6 Terminology Relating to Methods of Mechanical Testing
E 165 Test Method for Liquid Penetrant Examination

3. Significance and Use

3.1 Resistance to compression is the measure of the greatest strength of a ceramic material. Ideally, ceramics should be stressed this way in use. This test is a measure of the potential load-bearing usefulness of a ceramic.

PROCEDURE A

4. Apparatus

4.1 *Testing Machine*—Any testing machine conformin Practices E 4 and to the requirements for speed of tes prescribed in Sections 5 and 12 of this test method, ma used.

4.2 *Spherical Bearing Block*—In vertical testing machi the spherical bearing block shall be spring-suspended from upper head of the machine in such a manner that the u platen of the machine (lower face of the spherical bea block) remains in a central position (spherical surfaces in contact) when not loaded. The spherical surfaces shall be lubricated, and the center of curvature shall lie on the l face of the platen. The diagonal or diameter of the platen s be only slightly greater than the diagonal of the 1 (38.1-mm) square contact blocks to facilitate accurate ce ing of the specimens.

4.3 *Contact Blocks*—Cold-rolled steel contact blocks s be used between the test specimen and the platens of machine. These blocks shall be 1½ in. (38.1 mm) square b to ¾ in. (15.9 to 19.1 mm) thick, and the contact faces sha surface ground until plane and parallel. The contact bl shall be resurfaced, if necessary, after each strength test, may be reused only so long as the thickness remains over (12.7 mm). If the contact block is cracked during testin shall be replaced.

4.4 *Cushion Pads*—Cushion pads shall be used betwee test specimens and the contact blocks to aid in distributing load. New cushion pads shall be used for each speci Suitable materials for cushion pads, selected in accord with the compressive strength range of the material b tested, are shown in the following table:

Compressive Strength Range, psi (MPa)	Cushion Pad
5000 to 50 000 incl (34.5 to 345)	blotting paper, $\frac{1}{64}$ in. (0.4 mm) thi
Over 50 000 to 150 000 incl (345 to 1030.0)	mild steel, $\frac{1}{32}$ in. (0.8 mm) thick (HRB max)

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[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns.

Current edition approved Feb. 15, 2006. Published February 2006. Originally approved in 1974 to replace C 407 and C 528. Last previous edition approved in 1999 as C 773 – 88 (1999).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

5. Procedure

5.1 Dye-check specimens in accordance with Test Method E 165 before testing. Discard any pieces exhibiting cracks or flaws visible to the unaided eye.

5.2 Clean the test specimens with a suitable solvent after grinding and immerse in an ultrasonic bath filled with hot detergent solution. Then rinse specimens in hot water, dry at 110 ± 2°C (230 ± 4°F) for 2 h and cool to room temperature in a desiccator.

5.3 Carefully center the specimen in the machine between the contact blocks. Place an appropriate guard around the specimen to contain flying fragments at failure; eye protection should be used by the operator.

5.4 Apply the load continuously and without impact shock until ultimate failure. The rate of loading to be used shall depend on the compressive strength of the material being tested, as shown in Table 1.

6. Calculation

6.1 Calculate the compressive strength of each specimen as follows:

$$C = P/A \tag{1}$$

where:

C = compressive strength of the specimen, psi or MPa;
P = total load on the specimen at failure, lbf or N; and
A = calculated area of the bearing surface of the specimen, in.^{2} or mm^2.

7. Report

7.1 Report the following information:

7.1.1 The procedure used,

7.1.2 Type of testing machine (hydraulic or screw),

7.1.3 Material and size of contact blocks or of cushioning materials,

7.1.4 Description of material being tested (Note 1),

7.1.5 Rate of loading,

7.1.6 Number of specimens tested,

7.1.7 Dimensions and load at failure of each specimen, and

7.1.8 Compressive strength of each specimen tested, rounded off to the nearest 100 psi (1.0 MPa), together with the average compressive strength of the sample tested and the standard deviation.

NOTE 1—It is desirable to include details of the origin of the specimen and subsequent treatment.

TABLE 1 Typical Loading Rates to Cause Failure in 1 min

NOTE 1—The loading rate of 16 000 lbf/min (70 kN/min) shall be used for the first three tests of an unknown material to determine the general strength classification group. Some specimens crack before ultimate failure; the load at which the first audible crack occurs shall be noted, but only the load on the specimen at ultimate failure shall be used for calculation of compressive strength.

Compressive Strength, psi (MPa)	Specimen Diameter, in. (mm)	Loading Rate, lbf/min (kN/min)
10 000(69)	1.00(25.4)	8000(35)
50 000(345)	0.64(16.3)	16 000(70)
150 000(1034)	0.45(11.5)	24 000(105)

8. Precision and Bias

8.1 *Interlaboratory Test Data*—An interlaboratory test was run in 1979 in which randomly drawn samples of six materials were tested in each of five laboratories. One operator in each laboratory tested ten specimens of each material. The components of variance for compressive strength results expressed as coefficients of variation were calculated as follows:

Single-operator component	1.50 % of the average
Between-laboratory component	8.80 % of the average

8.2 *Critical Differences*—For the components of variance reported in 8.1, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the following critical differences listed below:

Number of Observations in Each Average	Critical Difference, % of Grand Average[A] Single-Operator Precision	Between-Laboratory Precision
10	4.16	24.40

[A] The critical differences were calculated using $t = 1.960$ which is based on infinite degrees of freedom.

8.3 *Confidence Limits*—For the components of variance noted in 8.1, single averages of observed values have the following 95 % confidence limits:

Number of Observations in Each Average	Width of 95 % Confidence Limits, Percent of the Grand Average[A] Single-Operator Precision	Between-Laboratory Precision
10	±2.94	±17.26

[A] The confidence limits were calculated using $t = 1.960$ which is based on infinite degrees of freedom.

8.4 *Bias*—No statement on bias is being made due to lack of an accepted standard reference material.

9. Test Specimens

9.1 *Preparation*—The test specimens shall be right cylinders. They may be formed and matured for the purpose of compression testing, or they may be cut from matured whiteware by sawing or coredrilling. The ends of all specimens shall be ground or lapped to yield plane and parallel faces. These faces shall be perpendicular to the axis of the specimen, and parallel within 15 min of arc (0.044 rad).

9.2 *Size*—The size of the specimen should be no larger than to require more than 80 % of the rated capacity of the testing machine. Examples of specimen size limitations are shown in Table 2.

TABLE 2 Maximum Specimen Diameter in Inches (Millimetres) to Use 80 % of Rated Capacity of Testing Machine

NOTE 1—The ratio of length to diameter of the test specimens may vary between 1.9 and 2.1. Diameters shall be measured to the nearest 0.001 in. (0.03 mm).

Maximum Compressive Strength, psi (MPa)	Testing Machine Capacity, lbf (kN) 10 000 (44)	20 000 (89)	30 000 (134)
	in. (mm)	in. (mm)	in. (mm)
10 000(69)	1.0(25.4)	1.43(36.3)	1.75(44.4)
50 000(345)	0.45(11.5)	0.64(16.3)	0.78(19.8)
150 000(1034)	0.26(6.6)	0.37(9.4)	0.45(11.5)

9.3 *Number of Specimens*—The number of specimens shall not be less than ten.

PROCEDURE B

10. Apparatus

10.1 *Testing Machine*—Any fixed-head testing machine conforming to Practices E 4 and to the requirements for speed of testing prescribed in 12.3 may be used. A spherical head must not be used.

10.2 *Bearing Plates*—Hardened steel 60 HRC bearing plates shall be used between the contact cylinders and the platens of the machine. These plates shall be approximately 2.5 in. (63.5 mm) in diameter by 1 in. (25.4 mm) thick. The contact faces shall be surface ground until flat and parallel within 0.001 in. (0.025 mm) total indicator reading. The bearing plates shall be resurfaced as necessary to retain their tolerance and to remove any surface damage resulting from testing high-strength materials.

10.3 *Contact Cylinders*—Ceramic contact cylinders of the same material as the specimens to be tested shall be used between the bearing plates and the test specimen to aid in distributing the load and to minimize detrimental "end effects." These contact cylinders shall be ½ in. (12.7 mm) high and ⅝ in. (15.9 mm) in diameter. The contact faces shall be flat and parallel to within 0.0005 in. (0.013 mm) total indicator reading. Two new contact cylinders should be used for each specimen to prevent a damaged contact cylinder failing prematurely and thereby giving an erroneous reading. By using contact blocks made of the same, or similar, material as the test specimen itself there is less deformation and less frictional resistance at the interfaces.

NOTE 2—Ceramic contact cylinders of similar composition to that of the test specimen may be used so long as the contact cylinders have a similar elastic modulus and equal or higher tensile strength to that of the test specimen.

11. Test Specimens

11.1 *Preparation*—Grind the test specimens to right cy ders. Grind the ends of all specimens with a 100-grit or fi diamond wheel, until parallel and perpendicular to the a within 0.0005 in. (0.013 mm) total indicator reading.

11.2 Clean the test specimens with a suitable solvent a grinding and follow by immersion in an ultrasonic bath fi with hot detergent solution. Then rinse the specimens in water, dry at 110 ± 2°C (230 ± 4°F) for 2 h and cool to ro temperature in a desiccator.

NOTE 3—In the event that water-sensitive specimens, such as MgO being cleansed, a substitute for water should be used.

11.3 *Size*—Specimens shall be 0.250 ± 0.001 in. (6.35 0.025 mm) in diameter and 0.500 ± 0.002 in. (12.70 ± 0 mm) in length.

11.4 *Number of Specimens*—The number of test specim shall be not less than ten.

12. Procedure

12.1 Dye-check specimens and contact cylinders in acc dance with Test Method E 165 before testing. Discard any p exhibiting cracks or flaws visible to the unaided eye.

12.2 Center the specimen carefully in the machine betw the bearing plates (Fig. 1) to avoid eccentric loading. Place appropriate guard around the specimen to contain fly fragments at failure; eye protection should be used by operator.

12.3 Apply the load continuously and without impact sh at a rate of 10 000 lbf/min (45 kN/min), within 20 %. Use c the load on the specimen at ultimate failure for calculatio the compressive strength.

13. Keywords

13.1 compressive strength; fired whiteware materials

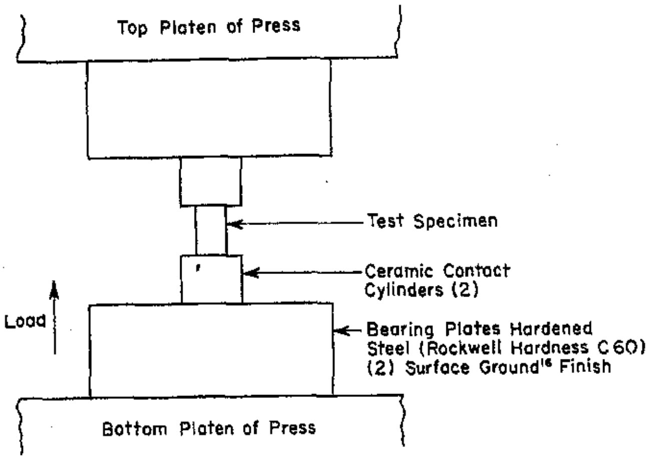

FIG. 1 Apparatus for Testing Compressive Strength

Designation: C 837 – 99 (Reapproved 2003)

Standard Test Method for Methylene Blue Index of Clay[1]

This standard is issued under the fixed designation C 837; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the adsorption of methylene blue dye by a clay, which is calculated as a methylene blue index for a clay.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 324 Test Method for Free Moisture in Ceramic Whiteware Clays[2]

3. Significance and Use

3.1 Tests run on many clays generally indicate that a straight-line relationship exists between the methylene blue index (MBI) and such fundamental clay properties as cation exchange capacity, dry bond strength, and casting rate. Where the colloidal portion of the clay is kaolinite, there is also a direct correlation with specific surface (as determined by nitrogen adsorption). Where the colloidal portion contains significant amounts of illite or montmorillonite, the same close correlation does not exist. The MBI better correlates with the ceramic-forming properties than does the specific surface.

3.2 That portion of a clay lying within the colloidal range (generally defined as the 0.5- to 0.001-µm range), determines the strictly colloidal properties of the clay and, together with the amount and type of organic material associated with the clay and the 2- to 0.2-µm fraction, largely determines the properties of the clay when used in ceramic-forming processes. While the specific surface of a clay is a function of the particle size and morphology and a relationship exists between dye adsorption and specific surface, the MBI should not be considered to be a particle size analysis since the value obtained is dominated by the character of only the very fine end of the particle size distribution. This procedure describes the determination of the dye adsorption (in this case, methylene blue) of a clay.

4. Apparatus

4.1 *Balance*, accurate to 0.01 g.

4.2 *Mixer*.[3]

4.3 *pH Meter or pH Paper.*

4.4 *Beaker*, 600 mL.

4.5 *Buret*, 25 mL.

4.6 *Medicine Dropper or Glass Stirring Rod.*

4.7 *Filter Paper*, Baroid No. 987.[4]

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall b used in all tests. Unless otherwise indicated, it is intended tha all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[5] Other grades may b used, provided it is first ascertained that the reagent is o sufficiently high purity to permit its use without lessening th accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean distilled water or water o equal purity.

5.3 *Methylene Blue Solution (1 mL = 0.01 meq)*[4]—Store i darkness.

5.4 *Sulfuric Acid (0.1N).*

6. Procedure

6.1 Weigh out 2.00 g of clay that has been dried i accordance with the procedure in Test Method C 324 and plac

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[1] This test method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C 21.04 on Clays.

Current edition approved Oct. 1, 2003. Published October 2003. Originally approved in 1976. Last previous edition approved in 1999 as C 837 – 99.

[2] *Annual Book of ASTM Standards*, Vol 15.02.

[3] This test method is based on the use of the Model F Lightnin mixer, which available from Mixing Equipment Co., Inc., Rochester, NY. However, it has bee found that some clays are not completely dispersed in this apparatus, and so resul may be spurious. It has been found that the Waring Blender, available from Warir Products Division, Dynamics Corporation of America, New Hartford, CT 06057, successful in dispersing all clays.

[4] Available from Baroid Div., National Lead Co., No. 425-15.

[5] *Reagent Chemicals, American Chemical Society Specifications*, Americ Chemical Society, Washington, DC. For suggestions on the testing of reagents n listed by the American Chemical Society, see *Analar Standards for Laborato Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacope and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockvil MD.

the 600-mL beaker. If the clay cannot be tested immediately ter drying, it should be stored in a desiccator.

6.2 Add 300 mL of distilled water to the beaker and stir with e mixer until the clay is uniformly dispersed.

6.3 Determine the pH of the slurry and add sufficient lfuric acid to bring the pH within the range from 2.5 to 3.8. ontinue stirring while the pH is being adjusted and continue irring for 10 to 15 min after the last addition of acid.

6.4 Again test the slurry for pH, adding additional acid if ecessary to restore the pH to the 2.5 to 3.8 range.

6.5 With the slurry still under the mixer, fill the buret with e methylene blue solution, add 5 ml of the solution to the urry, and stir for 1 to 2 min.

6.6 Remove a drop of the slurry, using the dropper or the lass stirring rod, and place on the edge of the filter paper.

6.7 Observe the appearance of the drop on the filter paper. he end point is indicated by the formation of a light blue halo ound the drop. Continue adding the methylene blue solution the slurry in 1.0-mL increments with 1 to 2 min of stirring fter each addition, then testing, until the end point is reached. or ball clays with relatively high methylene blue indexes, sting may start after two or even three 5-mL additions have een made to save time. Allow 1 to 2 min of stirring after each -mL increment.

6.8 After the end point is reached, continue stirring for 2 in and retest.

NOTE 1—Greater precision can be obtained by using larger samples with only minor influence on the value obtained.

7. Calculation

7.1 Calculate the methylene blue index as follows:

$$\mathrm{MBI} = \frac{E \times V}{W} \times 100$$

where:

MBI = methylene blue index for the clay in meq/100 g clay,
E = milliequivalents of methylene blue per millilitre (see 5.3),
V = millilitres of methylene blue solution required for the titration, and
W = grams of dry material.

7.2 The calculations may be facilitated by using a multiplication factor where the specimen size is 2.00 g and the methylene blue titrating solution is 0.01N:

$$\mathrm{MBI} = \frac{0.01 \times V}{2} \times 100$$
$$= 0.5\,V$$

7.3 Record the methylene blue index for the clay.

8. Precision and Bias

8.1 The precision of this test method is the calculated methylene blue index (MBI) ± 0.25 meq/100 g clay. The MBI value cannot be directly related to any single, simply measured, characteristic of the clay; thus the bias of this test method cannot be specified.

Designation: D 1921 – 06

Standard Test Methods for Particle Size (Sieve Analysis) of Plastic Materials[1]

This standard is issued under the fixed designation D 1921; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 These test methods cover the measurement of the particle size of plastic materials in the powdered, granular, or pelleted forms in which they are commonly supplied. As these test methods utilize dry sieving, the lower limit of measurement is considered to be about 38 µm (No. 400 sieve). For smaller particle sizes, sedimentation test methods are recommended.

1.2 Two test methods are described:

1.2.1 *Test Method A*—This test method uses multiple sieves selected to span the particle size of the material. This method is used to determine the mean particle diameter and particle size distribution.

1.2.2 *Test Method B*—This test method is an abbreviated version of Test Method A conducted with a few specific sieves. This test method determines "percent passing" or "percent retained" on a given sieve. Test Method B is applicable to materials which do not have a normal particle size distribution such as pellets and cubes.

1.3 The values stated in SI units shall be considered standard for dimensions of the wire cloth openings and the diameter of the wires used in the wire cloth. The values stated in inch-pound units shall be considered standard with regard to the sieve frames.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no technically equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 11 Specification for Wire Cloth and Sieves for Testin Purposes

E 691 Practice for Conducting an Interlaboratory Study t Determine the Precision of a Test Method

3. Summary of Test Methods

3.1 A dry mass of plastic material is placed on a series sieves arranged in order of increasing fineness and the mass divided into fractions corresponding to the sieve opening.

4. Significance and Use

4.1 These test methods can be used to determine partic size distribution and therefore are useful for determini lot-to-lot uniformity.

4.2 The particle sizes of plastic materials affect the handli characteristics and sometimes will affect the processing ch acteristics of some polymers.

5. Interferences

5.1 Some materials develop a static charge during sievi This charge interferes with the sieving process and results i coarse bias. Use of an antistat is necessary to obtain meani ful results.

5.2 The choice of antistat (or slip agent) has been know affect the coarse bias. Some materials are more effective aiding the fines to separate from the mass.

5.3 Too much material on a sieve causes mass blinding results in a coarse bias. The sieve selection and charge wei must be chosen to avoid overloading any sieve.

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[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

Current edition approved April 1, 2006. Published June 2006. Originally approved in 1961. Last previous edition approved in 2001 as D 1921 - 01.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.or contact ASTM Customer Service at service@astm.org. For *Annual Book of A Standards* volume information, refer to the standard's Document Summary pa the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

5.4 Wavy, improperly stretched wire-cloth may allow wires to separate without being visually damaged. Sieves with wavy or torn wires shall not be used, as they no longer conform to Specification E 11.

6. Apparatus

6.1 *Balance*, 500-g minimum capacity with the capability of reading to the nearest 0.1 g.

6.2 *Mechanical Sieving Device and Time Switch*—A mechanical sieve-shaking device equipped with an automatic time switch. This device shall be capable of imparting uniform rotary motion and a tapping action at a rate of 150 ± 10 taps/min.

6.3 *Wire Cloth Sieves*, woven wire cloth conforming to Specification E 11, as shown in Table 1, mounted in 8-in. [203-mm] frames. The number of sieves and the choice of sizes shall be selected for the material being tested. A cover and a bottom pan are also required.

6.4 *Accessories for Cleaning the Screens*:

6.4.1 *Brush*[3],

6.4.2 *Vacuum Cleaner*, and

6.4.3 *Air Hose*.

7. Reagents and Materials

7.1 Antistat (or slip) agent suitable to the material being tested.

8. Hazards

8.1 The sieving operation and cleaning of the sieves sometimes introduce fine plastic particles and antistat agent into the atmosphere. Take precautions to avoid breathing these particles.

9. Sampling

9.1 Homogenize the lot where possible before removing the test sample to avoid segregation of particles during handling.

10. Preparation of Apparatus

10.1 Thorough cleaning and inspection of the sieve are required prior to initiating a test. Carefully clean the sieves with a brush and vacuum cleaner or compressed air, or both. Periodic washing with soap and water or suitable solvent is recommended with some materials.

10.2 Tare each sieve and the pan. Record tare weights to the nearest 0.1 g.

10.3 Assemble sieves so that the sieve openings decrease in size in sequence from the top of the stack. Place the pan at the bottom.

10.4 Use full- or half-size screens to accommodate the holder in the shaker.

11. Conditioning

11.1 The plastic material must be in a free-flowing condition.

11.2 If possible, condition the material to the laboratory temperature and humidity.

TEST METHOD A

12. Procedure

12.1 Select sieves in sufficient number to cover the expected range of particle sizes, and nest them together in order of diminishing opening with the coarsest sieve on top and the pan on the bottom.

12.2 Select sieves in sufficient number to have significant measurable quantities on four or more sieves. Weigh the sieves on a balance reading to the nearest 0.1 g. Record these sieve masses as their tare masses, respectively.

12.3 Weigh 50 g of sample to the nearest 0.1 g and transfer it to the top of the stack. Record the sample weight used. Large sample size causes screen blinding and will skew the results to the coarse particle size. A screen is considered blinded if it is holding 20 or more g. For repeatable results, use a smaller sample size.

NOTE 2—For some materials an antistat (or slip agent) is needed. Add 1 % of the antistat (or slip agent) to the sample and mix in with a spatula. With polyvinyl chloride resins, it has been found that the distribution will skew to either the fine or the coarse particle size depending on the antistat used. Record the antistat (or slip agent) used.

12.4 Cover the stack and place it in the mechanical sieve shaker. Start the shaker and run for 10 min. Run the shaker for a longer time if it is required by the material and the efficiency of the shaker.

12.5 After shaking, carefully separate the stack of sieves, beginning at the top, and weigh each sieve with powder to the nearest 0.1 g. Determine the net weight of the powder remaining in each sieve by subtracting the sieve tare masses from the total weight of the sieve and the powder in that sieve.

12.6 If the cumulative total of actual weight is less than 98 %, carefully check the weights and operations and repeat the work if necessary.

13. Analysis of Particle Distribution

13.1 *Calculation of Particle Distribution*:

13.1.1 Obtain net weight of material retained on each sieve Calculate percentage by dividing net weight by total sampl weight × 100.

13.1.2 Repeat for each sieve.

13.2 *Calculation of Mean Particle Size*:

13.2.1 Obtain net weight of material retained on each sieve

13.2.2 Determine an average particle size for each sieve The average particle size is defined as the nominal opening siz of that sieve plus the nominal opening size of the next large sieve in the stack divided by two.

NOTE 3—Options are recommended to determine the average mesh si of the top sieve and the pan. On the coarse end, if the particles ha already been through a coarser screen, the screen size of the "throug screen" can be used as the upper limit of the top screen (first screen). commonly used method is to place a set of sieves with openings great than the desired top sieve. Select the lowest sieve where there is r particle on or the amount of particles on the sieve is insignificant. Use tl opening size of this sieve for calculation. Similar analog can be used to tl pan by using smaller opening sieves. A more practical way is to divide tl

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[3] The sole source of supply of the Type 8577 (W. S. Tyler) Brush known to the committee at this time is W. S. Tyler, Inc., 8750 Tyler Blvd., Mentor, OH 44060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

TABLE 1 Nominal Dimensions, Permissible Variations for Wire Cloth of Standard Test Sieves (U.S.A.) Standard Series

Sieve Designation		Nominal Sieve Opening, in.[A]	Permissible Variation of Average Opening from the Standard Sieve Designation	Opening Dimension Exceeded By Not More Than 5 % of the Openings	Maximum Individual Opening	Nominal Wir Diameter, mm
Standard[C]	Alternative					
(1)	(2)	(3)	(4)	(5)	(6)	(7)
125 mm	5 in.	5	±3.70 mm	130.0 mm	130.9 mm	8.00
106 mm	4.24 in.	4.24	±3.20 mm	110.2 mm	111.1 mm	6.30
100 mm[D]	4 in.[D]	4	±3.00 mm	104.0 mm	104.8 mm	6.30
90 mm	3½ in.	3.5	±2.70 mm	93.6 mm	94.4 mm	6.30
75 mm	3 in.	3	±2.20 mm	78.1 mm	78.7 mm	6.30
63 mm	2½ in.	2.5	±1.90 mm	65.6 mm	66.2 mm	5.80
53 mm	2.12 in.	2.12	±1.60 mm	55.2 mm	55.7 mm	5.00
50 mm[D]	2 in.[D]	2	±1.50 mm	52.1 mm	52.6 mm	5.00
45 mm	1¾ in.	1.75	±1.40 mm	46.9 mm	47.4 mm	4.50
37.5 mm	1½ in.	1.5	±1.10 mm	39.1 mm	39.5 mm	4.50
31.5 mm	1¼ in.	1.25	±1.00 mm	32.9 mm	33.2 mm	4.00
26.5 mm	1.06 in.	1.06	±800 mm	27.7 mm	28.0 mm	3.55
25.0 mm[D]	1.00 in.[D]	1	±800 mm	26.1 mm	26.4 mm	3.55
22.4 mm	⅞ in.	0.875	±700 mm	23.4 mm	23.7 mm	3.56
19.0 mm	¾ in.	0.750	±600 mm	19.9 mm	20.1 mm	3.15
16.0 mm	⅝ in.	0.625	±500 mm	16.7 mm	17.0 mm	3.15
13.2 mm	0.530 in.	0.530	±410 mm	13.83 mm	14.05 mm	2.80
12.5 mm[D]	½ in.[D]	0.500	±390 mm	13.10 mm	13.31 mm	2.50
11.2 mm	7⁄16 in.	0.438	±350 mm	11.75 mm	11.94 mm	2.50
9.5 mm	⅜ in.	0.375	±300 mm	9.97 mm	10.16 mm	2.24
8.0 mm	5⁄16 in.	0.312	±250 mm	8.41 mm	8.58 mm	2.00
6.7 mm	0.265 in.	0.265	±210 mm	7.05 mm	7.20 mm	1.80
6.3 mm[D]	¼ in.[D]	0.250	±200 mm	6.64 mm	6.78 mm	1.80
5.6 mm	No. 3½ [E]	0.223	±180 mm	5.90 mm	6.04 mm	1.60
4.75 mm	No. 4	0.187	±.150 mm	5.02 mm	5.14 mm	1.60
4.00 mm	No. 5	0.157	±.130 mm	4.23 mm	4.35 mm	1.40
3.35 mm	No. 6	0.132	±.110 mm	3.55 mm	3.66 mm	1.25
2.80 mm	No. 7	0.110	±.095 mm	2.975 mm	3.070 mm	1.12
2.36 mm	No. 8	0.0937	±.080 mm	2.515 mm	2.800 mm	1.00
2.00 mm	No. 10	0.0787	±.070 mm	2.135 mm	2.215 mm	0.900
1.7 mm	No. 12	0.0661	±.060 mm	1.820 mm	1.890 mm	0.800
1.4 mm	No. 14	0.0556	±.050 mm	1.505 mm	1.565 mm	0.710
1.18 mm	No. 16	0.0469	±.045 mm	1.270 mm	1.330 mm	0.830
1.00 mm	No. 18	0.0394	±.040 mm	1.080 mm	1.135 mm	0.560
850 µm[F]	No. 20	0.0331	±35 µm	925 µm	970 µm	0.500
710 µm	No. 25	0.0278	±30 µm	775 µm	815 µm	0.450
600 µm	No. 30	0.0234	±25 µm	660 µm	695 µm	0.400
500 µm	No. 35	0.0197	±20 µm	550 µm	585 µm	0.315
425 µm	No. 40	0.0165	±19 µm	471 µm	502 µm	0.280
355 µm	No. 45	0.0139	±16 µm	396 µm	426 µm	0.224
300 µm	No. 50	0.0117	±14 µm	337 µm	363 µm	0.200
250 µm	No. 60	0.0098	±12 µm	283 µm	306 µm	0.160
212 µm	No. 70	0.0083	±10 µm	242 µm	263 µm	0.140
180 µm	No. 80	0.0070	±9 µm	207 µm	227 µm	0.125
150 µm	No. 100	0.0059	±8 µm	174 µm	192 µm	0.100
125 µm	No. 120	0.0049	±7 µm	147 µm	163 µm	0.090
106 µm	No. 140	0.0041	±6 µm	126 µm	141 µm	0.071
90 µm	No. 170	0.0035	±5 µm	108 µm	122 µm	0.063
75 µm	No. 200	0.0029	±5 µm	91 µm	103 µm	0.050
63 µm	No. 230	0.0025	±4 µm	77 µm	89 µm	0.045
53 µm	No. 270	0.0021	±4 µm	66 µm	76 µm	0.036
45 µm	No. 325	0.0017	±3 µm	57 µm	66 µm	0.032
38 µm	No. 400	0.0015	±3 µm	48 µm	57 µm	0.030
32 µm	No. 450	0.0012	±3 µm	42 µm	50 µm	0.028
25 µm[D]	No. 500	0.0010	±3 µm	34 µm	41 µm	0.025
20 µm[D]	No. 635	0.0008	±3 µm	29 µm	35 µm	0.020

[A] Only approximately equivalent to the metric values in Column 1.

[B] The average diameter of the wires in the *x* and *y* direction, measured separately, of any wire cloth shall not deviate from the nominal values by more than

[C] These standard designations correspond to the values for test sieve openings recommended by the International Standards Organization, Geneva, Switzerland where noted.

[D] These sieves are not in the standard series, but they have been included because they are in common usage.

[E] These numbers (3½ to 635) are the approximate number of openings per linear inch, but it is preferred that the sieve be identified by the standard desig millimetres or micrometres.

[F] 1000 µm—1 mm.

opening size of the sieve above the pan by two and use it as the average particle size of the pan.

13.2.3 For materials that have a normal distribution, calculate the mean particle size as

$$D_m = \Sigma(P_i \times D_i)$$

where:

D_m = mean particle diameter, µm,
P_i = material retained on sieve (or pan), %, and
D_i = average particle size of material on sieve, µm.

14. Report

14.1 Report the following information:

14.1.1 Percentage of material retained on each sieve, with its corresponding sieve size,

14.1.2 Sample weight,

14.1.3 Antistat (or slip agent) used, and

14.1.4 Mean particle size and method used for calculation.

TEST METHOD B

15. Procedure

15.1 Choose the sieve(s) to be used and weigh each of them to the nearest 0.1 g. Record these sieve masses as their tare masses, respectively. If a single sieve is being used, stack it on the pan and transfer a sample weighing 100 ± 0.1 g to that sieve. If two sieves are to be used in the analysis of the sample, stack the coarse sieve over the fine sieve and transfer the weighed sample to the coarse sieve. For those finely divided powders which tend to clog the sieves, add 1.0 % of an antistat (see Note 2).

15.2 Cover the stack and place it in the mechanical sieve shaker. Start the shaker and run for 10 min ± 15 s.

15.3 After shaking, carefully separate the stack of sieves, beginning at the top, and weigh each sieve with powder to the nearest one tenth of a gram.

16. Report

16.1 Report the following information:

16.1.1 Percentage of material retained on each sieve, with its corresponding sieve size,

16.1.2 Sample weight, and

16.1.3 Antistat (or slip agent) used.

17. Precision and Bias

17.1 *Precision*—Table 2 and Table 3 are based on a round robin conducted in 1985 in accordance with Practice E 691, using D 1921 – 89, involving four materials tested by three laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the result of one individual determination. Each laboratory obtained two

TABLE 2 Percent Retained on Each Sieve of Screen Pack

Material	USA Sieve Number	Values in Units of Percent				
		Mean Retained on Sieve	S_r[A]	S_R[B]	I_r[C]	I_R[D]
Acrylic powder	40	13.5	0.1	1.2	0.3	3.4
	60	15.0	0.2	1.4	0.6	4.0
	80	9.3	0	0.4	0	1.1
	100	6.6	0.1	0.7	0.3	2.0
	200	22.9	0.1	1.2	0.3	3.4
	325	16.2	0.2	1.6	0.6	4.5
	PAN	16.5	0.1	3.6	0.3	10.2
Polyethylene powder	40	11.4	0.6	2.6	1.7	7.4
	60	47.8	0.6	2.9	1.7	7.6
	80	18.0	0.1	0.8	0.3	2.3
	100	7.5	0.1	0.7	0.3	2.0
	PAN	15.3	0.1	1.3	0.3	3.7
Polyvinyl chloride powder with carbon black antistat	40	0	...	...	...	...
	60	0.1	0.1	0.1	0.3	0.3
	80	5.4	0.9	0.9	2.5	2.5
	100	28.4	2.1	2.2	5.9	6.2
	140	53.7	1.3	1.6	3.7	4.5
	200	11.4	1.7	1.5	4.8	4.2
	PAN	0.9	0.3	6.3	0.9	0.9
Polyvinyl chloride powder with hi-sil antistat	40	0	...	...	...	...
	60	0	...	...	...	...
	80	4.2	0.7	0.6	2.0	1.7
	100	30.3	2.9	2.9	8.2	8.2
	140	52.6	2.0	2.2	5.7	6.2
	200	11.3	1.3	1.3	3.7	3.7
	PAN	1.5	0.3	0.3	0.9	6.9
Polystyrene cubes	20	99.9	...	...	...	...
	PAN	0.06	...	0.04	...	0.1

[A] S_r = within-laboratory standard deviation of the average (median/other function),
[B] S_R = between-laboratories standard deviation of the average median,
[C] I_r = 2.83 S_r, and
[D] I_R = 2.83 S_R.

TABLE 3 Calculated Mean Particle Diameter

Material	Average of Calculated Mean Particle Diameters	Values, µm			
		S_r[A]	S_R[B]	I_r[C]	I_R[D]
Acrylic powder	196	0.8	13	2.3	36.8
Polyethylene powder	291	0.6	4.8	1.7	13.6
PolyVinyl chloride with carbon black	137	2.2	1.8	6.2	5.1
PolyVinyl chloride with hi-sil	137	2.2	1.6	6.2	4.5

[A] S_r = within-laboratory standard deviation of the average,

[B] S_R = between-laboratories standard deviation of the average,

[C] I_r = 2.83 S_r, and

[D] I_R = 2.83 S_R.

test results for each material. (**Warning**—The explanations of *r* and *R* (17.2-17.2.2) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 2 and Table 3 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test metho should apply the principles outlined in Practice E 691 t generate data specific to their materials and laboratory (c between specific laboratories). The principles of 17.2-17.2. would then be valid for such data.)

17.2 *Concept of r and R in Table 3*—If S_r and S_R had bee calculated from a large enough body of data, and for test resul that were averages from testing seven specimens for each te result, then the following would apply:

17.2.1 *Repeatability*—Two test results obtained within on laboratory shall be judged not equivalent if they differ by mo than the *r* value for that material; *r* is the interval representin the critical difference between two test results for the sam material obtained by the same operator using the same equip ment on the same day in the same laboratory.

17.2.2 Any judgment in accordance with 17.2.1 would hav an approximate 95 % (0.95) probability of being correct if a adequate number of laboratories had participated.

17.3 There are no recognized standards by which to est mate the bias of these test methods.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue, D 1921 - 01, that may impact the use of this standard. (April 1, 2006)

(*1*) Changed old Note 2 to 12.2, and renumbered subsequent Notes accordingly.

(*2*) Added Note 3.

(*3*) Five-year review and removed permissive language.

Designation: C 371 – 89 (Reapproved 2003)

Standard Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders[1]

This standard is issued under the fixed designation C 371; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the particle size distribution of nonplastic ceramic powders such as alumina, silica, feldspar, pyrophyllite, nepheline syenite, talc, titanates, and zircon using wire cloth sieves.

1.2 Materials containing a large amount of fines, containing agglomerates, or that are nonfree-flowing, are wet-sieved to remove excessive fines or to disperse agglomerates before performing the test. This technique is not applicable to materials that are, to any degree, water soluble.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 322 Practice for Sampling Ceramic Whiteware Clays[2]

E 11 Specification for Wire Cloth and Sieves for Testing Purposes[2]

3. Significance and Use

3.1 Sieve analyses are carried out to determine the particle size distribution of powders which, in turn, are used to qualify those materials as to their usefulness in the process under consideration. Since particle size analyses have only relative significance, the results should be considered only where they correlate with process characteristics. The parameter that is being measured in this test is the amount of material that will pass through a cloth having theoretically square openings. It must be remembered that all the holes are not square, nor uniform in size, and the question of whether a given particle will go through is a statistical one. Since each particle size analysis method measures a unique physical parameter, the results from one method may not agree with those from another. Particle size distributions play a role in such properties as bulk density, dustiness, and handling characteristics. Care should be taken, however, when interpretations are made from one or two points (sieves) on the distribution curve.

4. Apparatus

4.1 *Balance*, having a sensitivity of 0.05 g.

4.2 *Sieves*, clean, unblinded, 205 mm (8 in.) in diameter, and conforming to Specification E 11. At all times they shall be certified by, or shall be calibrated with sieves certified by, the National Institute of Standards and Technology. For wet-sieving, use full-height 50-mm (2-in.) sieves; these sieves and pan may be used for dry-sieving also. Half-height 25-mm (1-in.) sieves and pan shall be used for dry-sieving only. The sieves to be used may range from 45 μm (No. 325) through 212 μm (No. 70).

4.3 *Drying Pans*, about 205 mm (8 in.) in diameter and 25 or 50 mm (1 or 2 in.) high.

4.4 *Dryer*—For drying, the use of an oven maintained automatically at 100 to 110°C is recommended.

4.5 *Mechanical Shaking Device*[3]—The shaking device shall be such as to produce a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample moving continuously over the surface of the sieve.

4.6 In wet-sieving, the water should be slightly above room temperature (for example, a hot-cold mixer tap) and should be supplied by means of a fixed or hand-held spray.

5. Sampling

5.1 *Unit for Sampling*—Each carload shall be considered a unit for sampling.

5.2 *Gross Sample* (See Practice C 322)—In collecting the gross sample from a carload shipped in bags, select a number of bags equivalent to not less than 1 % of the total number of bags in the car. Bags taken for sampling shall be from locations evenly distributed, horizontally and vertically, throughout the car. The gross sample shall consist of equal increments of not less than 227 g (0.5 lbs) from each of the bags taken for sampling. In collecting the gross sample from a carload

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[1] This method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.07 on Nonplastics.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The Tyler Ro-Tap mechanical shaking device, available from W.S. Tyler Co., Inc., Gastonia, NC 28053-9065 and major scientific supply houses, or equivalent, has been found satisfactory for this purpose.

shipped in bulk, take equal increments of not less than 227 g each from points well distributed both horizontally and vertically.

5.3 *Test Sample*—Obtain the test sample, of not less than 110 g, by mixing the gross sample and then riffling or hand quartering. Dry the entire test sample in an oven at 100 to 110°C to constant weight. Take a 100-g (Note 1) specimen weighed to the nearest 0.1 g shall be taken for sieve analysis.

NOTE 1—Alternatively, if less than 50 % passes the finest sieve, use a 50-g specimen.

6. Procedure

6.1 Transfer the 100-g specimen to glazed paper and proceed to 6.5, except as noted in 6.2.

6.2 Alumina, talc, pyrophillite, other nonfree-flowing materials, and those materials containing agglomerates require a preliminary wet sieving. Place the 100-g specimen into a glass beaker. Add 200 g of distilled water together with a suitable dispersion agent (Note 2) and stir the suspension with a rod until complete dispersion is obtained (Note 3). Pour the suspension onto the finest sieve to be used. Wash any residue in the beaker onto the sieve with a wash bottle.

NOTE 2—One suitable dispersion agent is 0.1 % sodium pyrophosphate. A 0.1 % sodium hexametaphosphate should be used for alumina.

NOTE 3—Dispersion aids, such as ultrasonic vibration and high shear mixers (food blenders) may be necessary to eliminate agglomerates. Care must be taken that undesirable grinding does not take place.

6.3 While agitating, thoroughly wash the sieve under the sprayer head, the flow being sufficiently gentle to avoid splashing. Sieving is complete when no more material passes. Determine this by catching the washings and examining for the solids.

6.4 When sieving is complete, pass a sponge over the underside of the sieve-cloth to draw out as much of the water as possible. Rinse the retained solids with acetone, to speed up drying, and dry off any excess with a sponge. Do this thoroughly to avoid dangerous levels of acetone in the oven. Dry the sieve and specimen thoroughly at 100 to 110°C and then brush onto glazed paper.

6.5 Assemble all the sieves to be used in the test with the coarsest sieve on top and the others under it in order of decreasing sieve opening size. Transfer the specimen from the glazed paper to the top or coarsest sieve and shake the entire series of sieves. It is recommended that this agitation be done mechanically. For hand-sieving, hold the nest of sieves, with pan and cover attached, in one hand and gently strike the side about 150 times/min against the palm of the other hand. The side of sieves next to the striking hand should be slightly higher than the other side so that sample will be well distributed over sieves. Turn the sieve-nest every 25 strokes about one sixth of a revolution in the same direction. With either mechanical or hand agitation, the agitation should continue to the point where not more than 0.05 g passes a given sieve in 1 min.

6.6 Separate the nested sieves and carefully remove the residue from each and weigh to the nearest 0.1 g. These weights in grams will equal the percent of residue on each sieve (if a 100-g specimen was used).

7. Calculation and Report

7.1 Calculate and report the cumulative percent passing, or the residue on each sieve, as required, to the nearest whole percent.

8. Precision and Bias

8.1 *Precision*—The precision of the procedure in Test Method C 371 for measuring particle size is being determined.

8.2 *Bias*—The true value of the particle size can only be defined in terms of a test method. Within this limitation, this method has no known bias.

9. Keywords

9.1 alumina; particle size distribution; sieve analysis; silica; wet-sieving

Designation: C 92 – 95 (Reapproved 2005)

Standard Test Methods for Sieve Analysis and Water Content of Refractory Materials[1]

This standard is issued under the fixed designation C 92; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover a wet and a dry method for sieve analysis of refractory materials.

1.1.1 *Wet Sieve Analysis*—Water promotes the slaking of clays and helps to separate fine particles, washing them from the larger grains. This method is recommended for use with materials that require water addition, and that slake in normal industrial use.

1.1.2 *Dry Sieve Analysis*—The dry method is not as effective as the wet method in determining the amount of material present in the smaller particle sizes. It is recommended (*1*) for clays, when the slaking action of water is undesirable, (2) when the material is in the form of coarsely ground grog and calcine, and (*3*) when the clay is to be used in such a way that the ultimate particle size is of secondary importance.

1.2 These test methods also cover determination of the water content of refractory materials in the wet condition and of air-dried samples as received, so that the sieve analysis can be calculated on the dry basis. Included is a method for obtaining the water content of other refractory materials, such as plastic refractories and wet mixes.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture

E 11 Specification for Wire-Cloth and Sieves for Testing Purposes

E 105 Practice for Probability Sampling Of Materials

E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for Characteristic of a Lot or Process

2.2 *Other Document:*

ASTM STP 447 *Manual on Test Sieving Methods*[3]

3. Significance and Use

3.1 Particle size distribution has a major affect upon most of the refractory properties. These test methods provide a means of measuring the distribution for the purpose of comparison to the desired distribution.

3.2 These test methods also cover determination of the water content of refractory materials in the wet condition and of air-dried samples received, so that the sieve analysis can be calculated on the dry basis.

3.3 These methods can produce data for specification acceptance, design purposes, manufacturing control, and research and development.

3.4 A reference set of standard matched or calibrated sieves[4] shall be provided for use in checking the set of sieves used in the actual sieve analysis of samples. The sieves for use in sieve analysis may also be standard matched sieves or may be unmatched sieves conforming to the Specification Table in Specification E 11, provided that such sieves will give results that differ by no more than 5 % from those obtained with the reference set when the two sets are compared in accordance with the section of Test Method C 429 on testing of sieves and samples splitters.

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[1] These test methods are under the jurisdiction of ASTM Committee C08 on Refractories and are the direct responsibility of Subcommittee C08.03 on Physical Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[4] The sole source of supply of matched sieves known to the committee at this time is W. S. Tyler, Inc., Mentor, OH 44060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

4. Apparatus

4.1 ASTM sieves, or the equivalent Tyler Series listed in Table 1, shall be used. The wire cloth for the sieves, described in Specification E 11, shall be woven (not twilled) and mounted without distortion or looseness in 8-in. (200-mm) diameter circular frames. Pans and covers shall be provided for the sieves.

5. Sampling, Test Specimens, and Test Units

5.1 A representative sample of the material to be tested shall weigh at least four to five times the required weight of the actual test specimen. Material on which the water content is to be determined shall be packed in a watertight container.

5.2 *Water Content*:

5.2.1 *Wet-Type Air-Setting Refractory Mortars*—Remove approximately 50 g of the material immediately after opening the original container and after carefully mixing the contents. To facilitate handling the specimen, place it on a tared piece of waxed paper or aluminum weighing dish. Weigh the test specimen to the nearest 0.1 g both before and after drying for 24 h at 220 to 230°F (105 to 110°C). Calculate the percentage of water to the nearest 0.1 % on the as-received basis.

5.2.2 *Materials Other Than Wet-Type Air-Setting Refractory Mortars* (ground fire clays, fireclay mortars, dry-type air-setting mortars, plastic refractories, and similar materials)—If the material is shipped in the wet condition, prevent loss of water before obtaining a test specimen of approximately 250 g. Weigh the test specimen to the nearest 0.1 g both before and after drying for 3 h at 220 to 230°F (105 to 110°C). Calculate the percentage of water to the nearest 0.1 % on the as-received basis. The dried specimen may be required for further tests (see Sections 6 and 7).

WET SIEVE ANALYSIS

6. Dry Materials

6.1 If the material is received in the dry condition, the test specimen (Note 1) shall consist of the dried and weighed test specimen prepared in accordance with 5.2.2.

NOTE 1—The size of the test specimen may be changed by reason of the nature of the material. For example, some clays tend to pack or cake on the sieves when ground to exceedingly fine particle size, in which case a 100-g sample may be used. For plastic refractories or coarsely ground mixes, the weight of the specimen could be increased to 500 g.

TABLE 1 ASTM Sieves and the Equivalent Tyler Standard Series

ASTM Sieves (U.S. Standard Series) No.	Tyler Standard Series (Mesh Designation)	Sieve Opening
0.265 in.	3	6.7 mm
4	4	4.75 mm
6	6	3.35 mm
8	8	2.36 mm
12	10	1.70 mm
16	14	1.18 mm
20	20	850 μm
30	28	600 μm
40	35	425 μm
50	48	300 μm
70	65	212 μm
100	100	150 μm
140	150	106 μm
200	200	75 μm

7. Wet Materials

7.1 Materials prepared with water (plastic refractories, wet-type high-temperature bonding mortars, etc.) shall be tested as received. Take two test specimens immediately after opening the original container and, in the case of mortars, after carefully mixing the contents. Use one specimen for determining the water content in accordance with either 5.2.1 or 5.2.2. Obtain approximately 250 g of the other specimen (Note 1) for sieve analysis. Weigh the test specimen to the nearest 0.1 g and transfer to the 1-dm^3 container (see Section 8). Wash the utensils used during weighing (to which a small part of the sample may adhere) with a small jet of water from a ¼-in (6-mm) hose to ensure a quantitative transfer of the weighed specimen to the container.

8. Procedure

8.1 Place the test specimen into a container of about 1-dm capacity. Add sufficient water to form a slurry. Allow slaking to proceed for 1 h, after which a further addition of water may be necessary. Then transfer the test specimen (without loss) to the finest sieve to be used in the analysis. Wash with a small jet o water from a ¼-in. (6-mm) rubber hose until the water passing through the sieve contains only traces of the specimen Exercise care during washing to prevent loss by splashing. I may be necessary to break up lumps by gently rubbing between the fingers, but never by rubbing or pressing against the sieve Then dry the washed residue in the sieve to constant weight at 220 to 230°F (105 to 110°C). This usually requires about 2 h. If desired, a preliminary drying period at a lower temperature may be used. Then transfer the dried residue to the top or coarsest sieve of the series to be used. Complete the sieving and weighing operations in accordance with Section 10 or 11.

9. Calculation and Report

9.1 Calculate the wet sieve analysis for the test specimen on the dry weight, and report the results to the nearest 0.1 % of the material retained on each sieve (Note 2). Report the percentage passing the finest sieve as the difference between 100 % and the sum of the percentages retained on the other sieves.

NOTE 2—As an alternative, the results of sieve analysis may be reported on the cumulative basis, either as the total percentage retained on o passing each sieve.

DRY SIEVE ANALYSIS

10. Mechanical Sieving

10.1 When the sieving is to be done mechanically, arrang the sieves in the order of size with the coarsest sieve at the to of the series. The specimen for sieving (Note 1) shall consist o a dried and weighed material prepared in accordance wit 5.2.2. Transfer the specimen to the top sieve of the series, an mechanically sieve until less than 0.1 g passes through eac sieve after hand sieving for 1 min, as described in Section 11 The machine-sieving operation usually requires about 15 min Then carefully separate the sieves and determine the amount o material retained on each by weighing to the nearest 0.1 g.

11. Hand Sieving

11.1 The specimen for sieving (Note 1) shall consist of a dried and weighed material prepared in accordance with 5.2.2. Use one sieve at a time beginning with the coarsest and then successively to finer sizes. Alternately tap and rotate the sieve, with pan and cover attached, while holding it in a slightly inclined position so that the test sample will be well distributed over the sieve. Continue the operation until less than 0.1 g of the material passes through each sieve during 1 min of continuous sieving. Determine the amount of material retained on each sieve by weighing to the nearest 0.1 g.

12. Calculation and Report

12.1 Calculate the dry sieve analysis for the test specimen on the dry weight, and report the results to the nearest 0.1 % of the material retained on each sieve (Note 2). Include dust loss with the material passing the finest sieve.

13. Precision and Bias

13.1 *Interlaboratory Testing*—An interlaboratory study was conducted among seven laboratories in 1986. The same samples of −2.5, +28 mesh tabular alumina and −14 mesh silicon carbide were tested by each laboratory to eliminate sampling variability. A third material, wet bauxite mortar, was split into separate samples for each laboratory for wet sieve analysis and moisture content tests.

13.1.1 The sponsoring laboratory used matched sieves and ran the initial and final dry sieve analyses on the tabular alumina and the silicon carbide samples in order to evaluate sample loss or sample breakdown in the repeated runs. The total specimen loss at this end of the study was 3.7 % of the tabular alumina and 4.3 % of the silicon carbide. Each of the other 6 laboratories, with one exception, ran two repetitions on each specimen. Thus, a total of seven sets of data with two replicates each and one set of data with one replicate were analyzed.

13.1.2 The wet mortar results consisted of one set of data from each of six laboratories. Each set of data included sieve analyses and water content on two separate samples split from the main sample received by each laboratory.

13.1.3 Analysis of the sieve analyses data was based on the positive accumulated difference (PAD), which is the sum of the absolute values of the difference between the percent held on each sieve in one repetition and the grand average of the percent on each sieve for all repetitions. The PAD divided by the total number of sieves within the particle size range of the particular material gave the average PAD per sieve. Hereafter, the value will be referred to as the average difference.

13.1.4 As shown in Table 2 and Table 3, the standard deviations within and between laboratories for the average difference was similar for the dry tabular alumina and silicon carbide. The standard deviations of the average difference were slightly greater for the dry-sieved portion (+150 mesh) and much greater for the wet-sieved portion (−150 mesh) of the wet mortar.

13.2 *Precision*:

13.2.1 Precision is based on the average difference only. For dry sieve analyses in one laboratory, the average difference for two materials is significantly different at a probability of 95 % ($t = 1.96$) if it exceeds the repeatability (I_r) listed for precisior in Table 2 or for relative precision (% I_r) in Table 3. That is, th average difference between two sieve analysis on the sam material obtained in the same laboratory will be expected t exceed the I_r value only about 5 % of the time. If the averag difference exceeds I_r, there is reason to question the test results Likewise, the average difference for two materials obtained b two laboratories is considered to be significantly different if i exceeds the applicable reproducibility intervals (IR and % IR in Table 2 and Table 3.

TABLE 2 Precision

Material	Average Difference from Grand Average X	Standard Deviation Within-Between		Repeatability-Reproducibility Intervals	
		Sr	SL	*Ir*	IR
Tabular alumina (−2.5× 28 m)	0.74	0.064	0.293	0.18	0.85
SiC (−14 m)	0.70	0.059	0.306	0.17	0.88
Grand average	0.72	0.062	0.300	0.175	0.865
Wet mortar (28 × 150 m)	0.35	0.123	0.0221	0.35	0.35
(−150 m)	1.25	0.860	0.700	2.40	3.10
	Average				
Water content	12.4	0.11	1.15	0.31	3.25

TABLE 3 Relative Precision

Material	Coefficient of Variation Within-Between		Repeatability-Reproducibility Intervals	
	Vr	VL	% *Ir*	% *IR*
Tabular alumina	8.7	39.8	24.7	115.1
SiC	8.4	43.7	23.7	125.8
Average	8.6	41.8	24.2	120.5
Wet mortar				
(28 × 150)	34.7	5.9	98.2	99.6
(−150 m)	68.6	56.0	194.0	250.4
H_2O content	0.88	9.2	2.5	26.2

13.2.2 The precision of the sieve analysis of wet materials i treated in two parts, the dry-sieved +150 mesh and th wet-sieved −150 mesh. For dry sieve analyses in one labora tory, the average difference for two materials is significantl different at a probability of 95 % ($t = 1.96$) if it exceeds th repeatability intervals listed for precision in Table 2 or f relative precision in Table 3. Likewise for comparing th average differences of two wet materials tested in two labor tories.

13.2.3 The precision and relative precision for the wat content of wet materials are shown in Table 2 and Table respectively, and are used in the same fashion as described 13.2.2.

13.3 *Bias*—No justifiable statement on bias can be ma since the true values for particle size and water content different materials cannot be established by an accepted ref ence method.

14. Keywords

14.1 dry sieve analysis; refractories; water content; w sieve analysis

Designation: C 429 – 01

Standard Test Method for
Sieve Analysis of Raw Materials for Glass Manufacture[1]

This standard is issued under the fixed designation C 429; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the sieve analysis of common raw materials for glass manufacture, such as sand, soda-ash, limestone, alkali-alumina silicates, and other granular materials used in glass batch.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 92 Test Methods for Sieve Analysis and Water Content of Refractory Materials[2]

C 325 Test Method for Wet Sieve Analysis of Ceramic Whiteware Clays[3]

C 371 Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders[3]

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

E 105 Practice for Probability Sampling of Materials[5]

E 122 Practice for Choice of Sample Size to Estimate the Average Quality for a Lot or Process[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *unit for sampling*—a carload lot or truckload lot of bulk material, or the entire shipment of bagged material.

3.1.2 *sublot*—a fraction of a shipment of bagged material, such as 1/10 or 1/20 of the lot.

3.1.3 *gross sample*—the total number of sample increments taken from the lot.

3.1.4 *sample increment*—an individual portion of the gross sample taken from the lot at a definite time or location, or both; increments shall be of nearly equal weight or volume, or both.

3.1.4.1 *Discussion*—A 2.2 to 4.5-kg (5 to 10-lb) increment generally is satisfactory in sampling raw materials for glass manufacture, for determining particle size distribution.

3.1.5 *laboratory sample*—a 0.9 to 1.8-kg (2 to 4-lb) representative fraction of the gross sample.

3.1.6 *test specimen*—a 100 to 150-g representative fraction of the laboratory sample.

4. Significance and Use

4.1 The purpose of this test method is to determine the particle size distribution of the glass raw materials.

5. Apparatus

5.1 *Testing Sieves*:

5.1.1 Sieves shall conform to Specification E 11 with particular reference to Table 1 and Section 4 on Frames. Sieves shall be designated by the U. S. Standard Series of sieve numbers and shall vary in opening size by the ratio of the $\sqrt{2}$:1, in accordance with frames 1 in. (25 mm) deep (half height) are recommended for mechanical shaking. The following sieves shall be provided:

Sieve Designation	Sieve Designation
No. 8 (2.36-mm)	No. 50 (300-µm)
No. 12 (1.70-mm)	No. 70 (212-µm)
No. 16 (1.18-mm)	No. 100 (150-µm)
No. 20 (850-µm)	No. 140 (106-µm)
No. 30 (600-µm)	No. 200 (75-µm)
No. 40 (425-µm)	

5.1.2 *Standard Matched Sieves*—A reference set of standar matched sieves[6] shall be provided for use in checking the set o sieves used in the actual sieve analysis of samples. The sieve for use in sieve analysis of samples may also be standar matched sieves or may be unmatched sieves conforming t 5.1.1, provided that such sieves will give results that differ b not more than 5 % from those obtained with the reference se when the two sets are compared in accordance with Section

5.2 *Sieve Shaker*—A mechanically operated sieve shak that imparts to the set of sieves a rotary motion and tappin

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[1] This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.02 on Chemical Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] *Annual Book of ASTM Standards*, Vol 05.05.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Matched sieves, available from the following scientific supply companies, ha been found satisfactory for this purpose: VWR Scientific Co., P.O. Box 62 Bridgeport, NJ 08014; Fisher Scientific Co., 585 Alpha Dr., Pittsburgh, PA 1523 and W. S. Tyler Co., 8570 Tyler Blvd., Mentor, OH 44060.

action of uniform speed shall be provided. The number of taps per minute shall be between 140 and 150. The sieve shaker shall be fitted with a wooden plug or rubber stopper to receive the impact of the tapper. Other types of mechanical shakers may be used, provided they can be adjusted to duplicate within 5 % results obtained by the type specified above, when tested with the same sample and standard matched sieves. The shaker shall be equipped with an automatic timer accurate to ½ min.

5.3 *Sample Splitters*:

5.3.1 For the reduction of the gross sample to laboratory size, either a large riffle with 25-mm (1-in.) openings or a sample splitter of the type that cuts out a fractional part (for example, a twelfth or a sixteenth) of the gross sample may be used. Sample splitters are available commercially or may be constructed by the user. The criterion for their use is that they shall produce a representative sample.

5.3.2 Riffles with openings of 6.4 to 13 mm (¼ to ½ in.) are required for reducing the laboratory sample to test size. The riffle opening must be at least three times the width of the largest particle diameter. This restricts use of a riffle with 6.4-mm openings to materials passing a No. 8 sieve.

5.4 *Balance*—A suitable balance or scale capable of weighing accurately to 0.1 g shall be used. A more sensitive balance may be used for weighing small fractions when they are considered critical.

6. Testing of Sieves and Sample Splitters

6.1 Since standard matched sieves are specified for the purpose of this test method, calibration as such by the tester is obviated. However, the tester must have a method to check the precision of the sieves. This shall be accomplished by having available at least two sets of sieves: a reference set and a working set. The reference set shall consist of standard matched sieves and shall be reserved for testing the working set. The working set also may consist of standard matched sieves or of sieves the tester has proven to be satisfactory (see 6.2). The testing of the working sieves is necessary because sieves will gradually change their characteristics after long usage from clogging and wear. The working set should be tested after every 100 to 150 sieve analyses. The test shall be made by sieving a suitable test sample through the working set as directed in Section 10, and then sieving the same test sample through the reference set. The results shall be calculated and compared. All testing sieves of the working set that give results within 10 % of the reference set shall be considered satisfactory for use. (See Appendix X1 for an example of this test.)

6.2 A new unmatched sieve can be used if it is proven by testing that it will produce results within 5 % of a standard matched sieve. To test an unmatched sieve, it should be substituted for the equivalent sieve in a standard matched set and a sieve analysis made with a sample previously sieved with the complete matched set. If agreement is satisfactory, the new unmatched sieve can be used as a working sieve.

6.3 A sample splitter for reducing a gross sample should be tested for reproducibility before it can be considered reliable. A minimum test shall be to take three gross samples of materials, weighing 45 kg (100 lb) or more, with different particle size distribution, and obtain four laboratory-size samples of each by repeated splitting. The laboratory samples shall be riffled to test size and sieved. The same set of sieves shall be used for all tests. Duplication of results within each group should be 5 % or better.

7. Care and Cleaning of Testing Sieves

7.1 Testing sieves must be properly cared for if reproducible and reliable results are to be obtained from them. The life of a sieve is materially lengthened by proper care and careful handling. It is inevitable that some particles will become fastened in the sieve cloth, but excessive clogging can be controlled by brushing the underside of the wire cloth with a stiff bristle or bronze wire brush every time the sieve is used in testing. A nylon bristle paint brush 51 mm (2 in.) in width, with the bristles cut back to about 25 mm (1 in.) long, is recommended for brushing, although any short-bristle brush that will not stick in the wire cloth is satisfactory. A bronze wire brush should be used only for sieves No. 60 and coarser. Brushing shall be firm enough to remove the majority of clogging particles but not so vigorous as to distort the sieve cloth. Sieves shall be washed periodically with a mild detergent or soap, brushing on the underside of the cloth. They should be washed immediately after sieving hygroscopic materials, such as alkali carbonates, and dried before storing. They may be dried in a drying oven at 105 to 110°C. A properly cared for sieve will be clean and free of patina. It will have a minimum of clogged openings. The wire cloth will be taut in the frame and free of distortion. The solder joint will be firm. A loosened joint on an otherwise satisfactory sieve may be repaired by carefully resoldering with resin-core solder. Additional cleaning methods are contained in ASTM STP 447B.[7]

8. Sampling

8.1 *General Considerations*—Follow the principles of probability sampling as given in Practice E 105. To estimate the size (mass and number of increments) of the gross sample, follow Practice E 122. The methods used for other necessary statistical calculations are given in ASTM STP 15D.[8]

8.2 *Sampling Plan*—The sampling plan shall be such that the sample obtained will represent as nearly as practicable the average particle size distribution of the lot. Sampling bulk material and bagged material will each present a different problem.

8.2.1 Some segregation or nonuniformity will always exist in a bulk lot of material. At rest, this nonuniformity can and probably will be multidirectional, with some layers of segregation in the lot that are nearly perpendicular to each other. The exact degree is never completely known. To obtain a representative cross section of the lot is difficult, if not impossible. In motion, however, some mixing occurs, and segregation will tend to become unidirectional with layers of segregation generally parallel to the direction of flow. Therefore, a sample increment taken by uniformly cutting across the flowing stream is generally much more nearly representative than an increment taken with the material at rest. An entire lot should be sampled

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[7] *ASTM STP 447B, Manual on Test Sieving Methods*, ASTM, 1985.

[8] *ASTM STP 15D, Manual on Presentation of Data and Control Chart Analysis*, ASTM, 1986.

by taking a number of increments spaced at nearly equal intervals during the whole time of loading or unloading of the car or truck. To take frequent cuts (sample increments) of all of the stream part of the time reduces the danger of a biased sample.[9] Furthermore, when sampling a moving stream, the requirement for randomness is more nearly met at the time and place of sampling since the chance of taking one grain instead of another is about equal. The total number of increments required for a desired precision can be estimated statistically as in Practice E 122. Some simple device is required to sample the stream. This may consist of a box-type cutter for sampling the stream discharging from the end of a belt or spout, or a scoop for sampling the stream being transported on the belt. (See Appendix X2 for illustrations of simple stream samplers.) For the purpose of this test method, a sampling plan that provides for sampling the moving stream is recommended. The sampling of a car or truckload lot of material at rest, by shovel, scoop and cylinder, or thief is not recommended.

8.2.2 In sampling bagged material, an added problem is presented—that of choosing which bags of the lot will be taken for sampling and how the bags taken are to be sampled. A suitable plan for taking bags for sampling would be to divide the lot into sublots and then to take at random one bag from each sublot. This would afford a simple cross section of the lot and a random selection in each sublot. The number of sublots in which to divide the lot should be calculated using the same considerations as for estimating the number of increments to be taken when sampling bulk material. The consideration of segregation within bags must not be overlooked. If a suitable sample splitter is available, the entire contents of the bag can be taken and segregation ignored. However, if the bag is sampled with a thief, or by some other method, it must be made certain that any segregation is taken into account. A bag of granular material, particularly after shipping, can show visible evidence of segregation. If stratification or segregation has occurred, care must be taken to sample so as not to obtain a biased or "weighted" sample. The samples obtained from the bags are mixed to constitute the gross sample.

8.3 *Gross Sample Requirement*—Because of the many ways of handling materials and, in many cases, the limitations so imposed on sampling, and because of the several kinds of materials used for glass making, a single sampling plan is not prescribed. Only certain minimum considerations are presented and recommendations made. However, for the purpose of this test method, any plan devised or used shall produce—or as near thereto as it is practicable to obtain—a gross sample that has a 99.7 % probability that the minimum precision of the estimate will be 10 % relative for the critical particle size fraction (Note 1). (See Appendix X3 for calculation and discussion of this requirement.)

NOTE 1—A critical particle size fraction is considered to be one upon which a specification for purchase or use is based.

9. Reduction of the Sample for Analysis

9.1 The gross sample obtained by combining all of the increments shall be reduced to laboratory sample size of 0.9 to 1.8 kg (2 to 4 lb) by use of a large riffle with 25-mm (1-in.) openings or by a sample splitter. If the material is too moist to flow freely in a small riffle, it shall be dried before further handling (9.1.2). The laboratory sample shall be reduced to test specimen size, using a riffle with 6.4 to 13-mm (¼ to ½-in.) openings. It shall be divided until the fractional portion weighs approximately 100 to 150 g. This whole fraction constitutes the test specimen. An exception to the above weight for the test specimen is burned dolomite. Because of its light density, the dolomite shall be riffled to a test size weighing 50 to 75 g. The test specimen shall be weighed to the nearest 0.1 g before sieving.

9.1.1 When reduction of the gross sample or laboratory sample to test size by the means described in Section 8 is not feasible, hand reduction by the cone and quarter method may be used. The applicable portions of this method as described in Method D 346 shall be followed.

9.1.2 Most materials can be dried at 105 to 120°C. However, naturally hydrated materials such as gypsum, if dried, must not be heated above the critical temperature of the hydrate. Gypsum would best be dried in a stream of dry air or a desiccator.

10. Procedure for Mechanical Sieving

10.1 Assemble in order the selected sieves, which shall vary in opening size by the ratio of $\sqrt{2:1}$, with the coarsest on top and a pan on the bottom. Place the test specimen on the top sieve, close the nest of sieves with a cover, and place the entire assembly on the shaker. Shake the sieves for the time specified in 10.2. After shaking for the specified time, stop the shaker, remove the sieves, and weigh each separated fraction to the nearest 0.1 g.

10.2 *Shaking Time*—The shaking time for this test method is as follows:

	Time, min
Sodium carbonate (soda ash)	10
Potassium carbonate (potash)[A]	5
All other materials	15

[A] The finest sieve used shall be the No. 50.

11. Procedure for Burned Dolomite (Note 2)

11.1 Before shaking, mix 1 g of tricalcium phosphate[10] into the test specimen of burned dolomite by rolling back and forth on a sheet of glazed paper. Sieve the specimen in the regular manner. Subtract the added gram from the pan for calculation. The addition of tricalcium phosphate imparts a free-flowing character to the burned dolomite, preventing balling and blinding of the sieve. This additive will permit sieving through a No. 140 sieve. If sieving through a No. 200 sieve is desired, an additional 10 min of shaking may be necessary. However i may be found that sieving through a No. 200 will not be successful. In any case, remove all fractions except tha remaining on the No. 200 sieve and weigh before continuing

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[9] Taggart, A. F., *Handbook of Mineral Dressing, Ores & Industrial Minerals*, John Wilcox & Sons, New York, NY, 1945.

[10] The tricalcium phosphates supplied by Fisher Scientific Co., N. F. grade, an technical grade, are satisfactory. Analytical Reagent and other comparable grade from phosphate chemical manufacturers and other laboratory supply houses als should prove satisfactory.

NOTE 2—Within the lime industry burned dolomite is classified also as ground, screened, or pulverized quicklime.

12. Procedure for Hand Sieving

12.1 Hand sieving is not a standard procedure for the purpose of this test method. However, if necessity requires hand sieving a material, follow the procedure described in Section 8 on Hand Sieving in Test Methods C 92.

13. Procedure for Wet Sieving

13.1 When the sizing of finely ground materials on sieves finer than No. 200 is required, they shall be wet sieved. For the purpose of this test method the following test methods are considered suitable: Test Methods C 325 and C 371.

14. Calculation and Report

14.1 Weigh each fraction recovered to the nearest 0.1 g. When all fractions are recovered and weighed, take the sum of the fractions as the test specimen mass for calculation; the sum of the fractions and the original sample mass should agree to within 1 g or a weighing error is indicated. Calculate the percent retained on each sieve and report to the nearest 0.1 %. When a fraction retained on a sieve is definite, but is less than 0.1 %, and is of importance because of specification requirements, weigh it to the nearest 1 mg, and report to the nearest 0.01 % or 0.001 % as required.

15. Precision and Bias

15.1 *Precision—Repeatability and Reproducibility Standard Diviations*

15.1.1 Fifteen laboratories preformed dry sieve analysis on six commonly used glass melting sands according to Test Method C 429, following Practice E 691. The statistical results are summarized in Table 1. Complete results are given in Reasearch Report C14–1001[11], Interlaboratory Study to Update the Precision Statistics of Sieve Analysis for Glass Melting Sand.

15.1.2 Table 1 provides with-in lab "repeatability" standard deviations (Sr) and between-labs "reproducibility" standard deviations (SR) for six sands of varying partial size. For natural whole grain sands consisting primarily of particles larger than 200 mesh, Material A-D, Table 1 show relatively better sieve analysis precision with Srs less than 1.4% retained and SRs less than 3.2%. Finely ground sands containing more than 80% of particles smaller than 200 mesh, Materials E-F, exhibited significantly higher Sr's and SRs, that is, relatively poorer sieve analysis precision. These results support the recommendation in Section 13, that wet sieving is appropriate for finely ground sands.

15.2 *Precision-Repeatability and Reproducibility 95% Limits:*

15.2.1 Table 2 lists repeatability and reproducibility 95% Limits for the Difference Between Pairs of Results, "r" and "R". Computationally, Table 2 is derived from the Sr and SR values of Table 1 by the relationship: 95% limit = 2.8 × standard deviation. ASTM Form and Style and Practice E 177 prescribe that the 95% r and R limits be included in the precision statement of test methods, to serve as a reference for the numerous practical laboratory situations involving the comparison of the two test results. Appendix 4 provides an illustration of the use of the quantities "r" and "R".

15.3 *Bias:*

15.3.1 The National Institute of Standards and Technology provides samples of Materials A, C, D allowing users of Test Method C 429 to evaluate the accuracy of their sieves. Sample bottles may be obtained from NIST by requesting Reference Material(s) 8010, Sand for Sieve Analysis.

16. Keywords

16.1 glass raw materials; sampling; sieve analysis; sieve shaker; splitters; standard sieves; testing of sieves

TABLE 1 Dry Sieve Analysis Results for Six Glass Melting Sands Determined by C 429, Including Repeatability and Reproducibility Standard Deviations; Sr, SR respectively (% Retained)

Sieve Mesh #	Material-A			Material-B			Material-C			Material-D			Material-E			Material-F		
	Ave.	Sr	SR	Ave.	Sr	SR	Ave.	Sr	SR	Ave.	Sr	SR	Ave.	Sr	SR	Ave.	Sr	SR
30	0.3	0.2	0.2	0.1	0.1	0.1	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.1	0.1	0.0	0.1
40	22.4	0.8	2.7	5.3	0.5	0.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.1
50	42.0	1.1	3.2	21.0	0.9	1.6	0.2	0.1	0.1	0.0	0.0	0.1	0.0	0.0	0.1	0.1	0.0	0.1
70	19.5	1.1	1.8	38.7	0.9	3.0	5.7	0.1	0.9	0.1	0.0	0.1	0.1	0.1	0.2	0.1	0.2	0.2
100	9.2	0.4	0.7	28.8	0.8	2.7	41.4	0.8	2.3	5.4	1.4	1.6	0.7	0.9	1.1	1.7	2.1	2.7
140	5.0	0.2	0.3	5.2	0.6	0.7	41.4	0.7	2.8	28.5	0.7	1.5	3.0	1.0	2.4	7.6	2.2	11.2
200	1.5	0.1	0.1	0.5	0.1	0.1	10.4	0.3	1.0	27.7	0.9	2.1	9.9	1.4	4.8	7.0	2.9	3.8
270	0.2	0.1	0.1	0.1	0.1	0.1	0.7	0.1	0.3	23.3	0.5	1.9	24.8	1.6	10.7	18.8	3.9	12.7
325	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	7.2	0.5	2.0	14.8	2.7	6.3	18.6	4.5	8.9
Pan	0.1	0.1	0.1	0.0	0.0	0.0	0.1	0.0	0.1	8.2	0.5	1.4	46.1	3.1	17.9	43.6	8.5	21.6

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[11] Available from ASTM Headquarters, request Research Report : C14–1001.

TABLE 2 Repeatability and Reproducibility 95% Limits, r and R respectively, for Dry Sieve Analysis of Six Glass Melting Sands Determined by C 429 (% Retained)

Sieve Mesh #	Material-A		Material-B		Material-C		Material-D		Material-E		Material-F	
	r	R	r	R	r	R	r	R	r	R	r	R
30	0.4	0.4	0.2	0.2	0.1	0.1	0.1	0.2	0.1	0.2	0.1	0.3
40	2.2	7.6	1.3	2.6	0.1	0.1	0.0	0.1	0.1	0.1	0.1	0.2
50	3.1	9.1	2.5	4.5	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.3
70	3.2	5.0	2.5	8.4	0.4	2.4	0.1	0.3	0.3	0.5	0.4	0.5
100	1.1	1.9	2.4	7.6	2.2	6.4	4.1	4.6	2.6	3.1	5.9	7.6
140	0.4	0.9	1.6	2.0	2.0	7.7	2.0	4.2	2.9	6.7	6.2	31.5
200	0.2	0.4	0.3	0.3	0.8	2.7	2.6	6.0	4.0	13.5	8.1	10.6
270	0.2	0.3	0.1	0.1	0.4	0.7	1.3	5.2	4.5	30.0	10.9	35.6
325	0.1	0.1	0.1	0.1	0.2	0.3	1.4	5.7	7.5	17.6	12.7	25.0
Pan	0.1	0.2	0.1	0.1	0.1	0.3	1.4	4.0	8.6	50.2	23.7	60.4

APPENDIXES

(Nonmandatory Information)

X1. TESTING THE WORKING SET AGAINST THE REFERENCE SET OF STANDARD MATCHED SIEVES

X1.1 It is specified in 6.1 that each working set of sieves shall be tested periodically against a reference set of standard matched sieves. This will give the necessary assurance that the working set of sieves is reliable, or provide the data for discarding any of the sieves. An example of a hypothetical comparison is given in Table X1.1 and the reasoning for the discarding of one of the sieves shown in the following paragraphs. In comparing the sieve analyses, it is necessary to calculate only the percent accumulative for each sieve to show which sieve (or sieves) is defective.

X1.2 If the percent retained were alone considered, a cursory examination would indicate that both the Nos. 70 and 100 sieves in the working set were unsatisfactory as they differ by more than 10 percent relative from the reference set:

$$\text{No. } 70\ (3.4/23.3) \times 100 = 14.5\ \% \quad \text{(X1.1)}$$

$$\text{No. } 100\ (4.3/15.7) \times 100 = 27.4\ \% \quad \text{(X1.2)}$$

However, the amount of material retained on a sieve is directly influenced by the amount that has been retained on the next larger sieve as well as by its own sieving characteristics. Examination of the tabulated accumulative columns reveals this relationship and is the guide used to judge the accuracy of a sieve.

TABLE X1.1 Hypothetical Comparison of Reference Set Versus Working Set

Sieve No.	Reference Set		Working Set	
	Retained, %	Accumulative, %	Retained, %	Accumulative, %
30	1.4	1.4	1.5	1.5
40	16.6	18.0	16.0	17.5
50	24.2	42.2	25.4	42.9
70	23.3	65.5	26.7	69.6
100	15.7	81.2	11.4	81.0
140	7.8	89.0	8.0	89.0
−140 (pan)	11.0	100.0	11.0	100.0

X1.3 From the data in Table X1.1, it is noted that Nos. 30, 40, and 50 sieves are satisfactory but the No. 70 sieve is suspect. The No. 70 differed by + 4.1 between the reference and working set, so the percent accumulative error in No. 70 is calculated as follows:

$$\text{Real Error: } (4.1/23.33) \times 100 = 17.6\ \% \quad \text{(X1.3)}$$

This No. 70 sieve is to be discarded.

X1.4 However, in the case of the No. 100 sieve, it is obvious after inspection of the accumulative column that the error in the retained column is almost entirely due to the No. 70 sieve being too retentive. If the No. 70 were to pass the excess 4.1 % it retained, the retained column would show a percent of 15.5, only 0.2 % less than that shown for the reference set. The No. 100 sieve is a satisfactory sieve, and is not discarded from the working set.

X1.5 When testing a working set of sieves for accuracy, a test sample should be chosen that will have approximately a minimum of 10 % for a given sieve size fraction for judging any particular sieve. In the example given above, the test sample would not be one to use for judging the accuracy of the No. 30 sieve and those larger, and possibly also the No. 200 sieve which is not shown. Practically, two test samples of a hard nonfriable material, one of coarse size and one of fine size, will most generally serve best as test samples. They can be used over and over, and tailor-made to exact chosen size distribution if so desired.

X1.6 It is suggested that when a sieve is discarded from the working set, that it be replaced by its like number from the reference set. A new standard matched sieve is then added to the reference set to fill the vacancy. This practice will assure that the reference set will consist of the newer and least used sieves.

X2. STREAM SAMPLERS

X2.1 The design of stream samplers is generally of simple box construction so as to conform to the environmental restrictions of the sampling location. The chief requirement in their construction is that they will not overflow while passing through or cutting the stream and that they are large enough in one direction to catch the full stream. To sample, the cutter should be passed through and out of the stream at a constant rate so as to cut as evenly as possible each section of the stream. A double pass may be made if a single pass does not catch an increment of at least 2.2 kg (5 lb).

X2.2 Fig. X2.1 shows a design for a sampler to pass across a free-falling stream transverse to the general direction of flow from a belt or spout. Fig. X2.2 is a simple open box or trough for use where it is not practical to use a cutter type as shown in Fig. X2.1. Fig. X2.3 is a simple scoop for sampling across a belt. This is probably the least satisfactory way of sampling a stream, but if done quickly and smoothly, and if the material is picked up completely from the bottom of the belt, it should prove adequate. Automatic samplers of proper design can be of much help when many samples must be taken. A straightlin cutter sampler as shown in Fig. X2.4 should prove satisfa tory.[12]

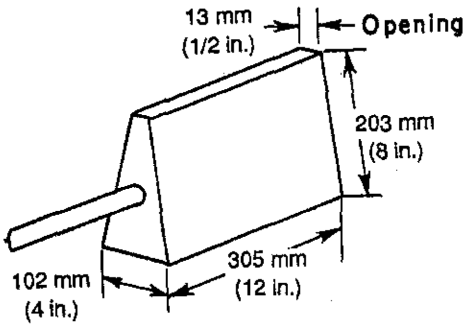

FIG. X2.1 Sampler for Free Falling Stream Transverse to Direction of Flow

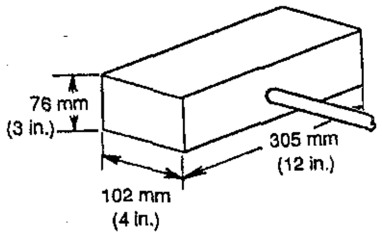

FIG. X2.2 Open Box Sampler

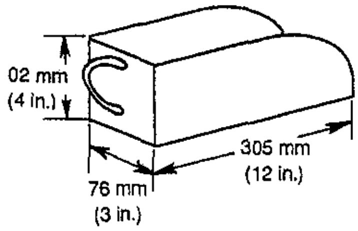

FIG. X2.3 Scoop Sampler

X2.3 Automatic samplers can be tested out statistically f sampling error as described for hand sampling methods Appendix X3. If many more than ten sampling cuts are take from a lot, the cuts may be grouped into eight or ten subgrou for individual testing for the estimate of the sampling error.

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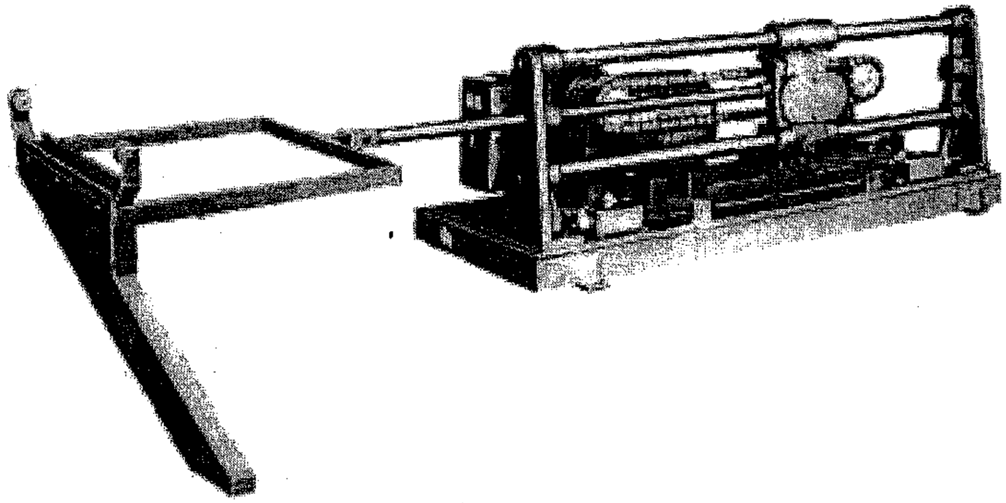
[12] Automatic samplers manufactured by Denver Equipment Div., Joy Manufa turing Co., P.O. Box 340, Colorado Springs, CO 80901, and by Gustafson, Inc., P. Box 660065, Dallas, TX 75266-0065, among others, have been found satisfacto for this purpose.



NOTE 1—This illustration shows the sampler with the cover removed. The cutter (left) reciprocates horizontally and is supported by rollers on a track.

FIG. X2.4 Automatic Sampler

X3. ESTIMATE OF GROSS SAMPLE SIZE REQUIREMENT

X3.1 The use of Eq. 1 in Section 5 on Equations for Calculating Sample Size of Practice E 122 permits the estimation of the sample size within a certain probability of a gross error. In the case of sampling by increments from bulk material, the calculation arrives at the number of increments to be taken.

X3.2 To arrive at a sampling plan that will meet the requirements of 8.3 of this test method, each particle-size fraction must be considered separately. If the increments of a sample are tested individually, each particle-size fraction will have a distribution around the average, and the standard deviation can be calculated (*Part 1, Section 20* of ASTM STP 15D).[8] Prior knowledge of the standard deviations is desirable, although it can be estimated using the example in Section 6 of Practice E 122. However, the penalty of an estimate of the standard deviation versus more exact knowledge of its value usually results in taking a larger sample than necessary.

X3.3 An example is given in Table X3.1 of data assembled and the calculation of n, using Eq. 1 in Section 5 on Equations for Calculating Sample Size of Practice E 122. Five cars of sand were sampled, taking ten 45-kg (10-lb) increments from across the flowing stream while emptying each car. Each increment was sieved separately with the same set of sieves.

TABLE X3.1 Example of Data Assembled and Calculation of n[A]

Sieve No.	$\bar{X}'$ %	$\bar{R}$	$\bar{\sigma}'$	n
40	3.13	2.1	0.65	40
50	27.26	7.6	2.4	7
70	29.61	4.6	1.2	2
100	26.77	4.3	1.4	8
140	12.05	5.6	1.8	20
200	0.73	0.6	0.18	55
−200 (pan)	0.43	0.3	0.087	37

[A] The symbols are defined as follows:
$\bar{X}'$ = average of the averages for the five lots,
$\bar{R}$ = average range of the five lots,
$\bar{\sigma}'$ = average standard deviation of the five lots corrected for sample size (ten increments), and
n = sample size as increments required for a probability of 99.7 % that the estimate of the average will not exceed 10 % error.

X3.4 The calculations for n were computed using Eq. 1 of Practice E 122:

$$n = (3\sigma'/E)^2; \tag{X3.1}$$

where E = 10 % of a given particle size fraction.

For the No. 50 sieve fraction,

$$n = [(3 \times 2.4)/2.726]^2 = 6.97 \text{ or } 7 \tag{X3.2}$$

X3.5 After calculating for the number of increments required, the other considerations for the sampling plan can be taken into account—the number of increments that can be taken practically, and the specifications or critical particle size fractions of the material. To illustrate, the following assumptions are made:

X3.5.1 The maximum number of increments that can be taken is 20, although 10 is preferable.

X3.5.2 It is specified that the percent of material remaining on the No. 40 sieve does not exceed 5.0.

X3.5.3 It is specified that the percent of material that passes the No. 100 sieve does not exceed 15.0.

X3.6 It is apparent that the requirement for a 99.7 % probability of not exceeding a 10 % sampling error cannot be met for the Nos. 40, 200, and pan sieve fractions. However, the No. 200 and pan fractions are quite small and are disregarded. More properly they should be added into the No. 140 sieve fraction; however, in either case the resulting estimation would not be much different. By substituting in Eq. 1 of Practice E 122 and using Fig. 14 in Part 1 of ASTM STP 15D,[8] the probability of not exceeding a 10 % sampling error, or the percent of error for a 99.7 % probability for a 10 or 20-increment sample can be estimated where P = probability (factor), and e = sampling error. P is translated to percent, using Fig. 14 of ASTM STP 15D,[8]

X3.7 For the No. 40 sieve, a 10-increment sample and a 10 percent sampling error, Eq. 1 is written to solve for P:

$$10 = [(P \times 0.65)/0.31]^2 \qquad \text{(X3.3)}$$

and for a 10-increment sample but to solve for e with a 99.7 % probability:

$$10 = [3 \times 0.65)/e]^2 \qquad \text{(X3.4)}$$

X3.8 Solving for both a 10 and 20-increment sample, the following estimates are made:

X3.8.1 For the No. 40 sieve:

X3.8.1.1 The probability is 86.7 % not to exceed a 10 % sampling error for a 10-increment sample; or 95 % not to exceed a 10 % sampling error for a 20-increment sample.

X3.8.1.2 The estimated error is 1.6 % (52 % of the average fraction percent) for a 10-increment sample, and 0.4 % (14 % of the average fraction percent) for a 20-increment sample.

X3.8.2 For the No. 140 sieve:

X3.8.2.1 The probability is 96 % not to exceed a 10 % sampling error for a 10-increment sample.

X3.8.2.2 The estimated error is 1.7 % (14 % of the average fraction percent) for a 10-increment sample.

X3.9 The decision as to the number of increments to incorporate into the sampling plan definitely favors taking a 20-increment sample. This satisfies the requirement for the No 140 sieve fraction and still permits an acceptable probability and sample error for the No. 40 sieve fraction. Considering the specification for this fraction, a sampling error of 1.6 % plus the average percent of 3.1 will place the test results near the 5 % specification limit, and if the error is minus, a lot over specification could test as acceptable.

X3.10 The above data, calculations, and discussion were made on test results that included the testing errors of riffling and sieving. The standard deviations of riffling and sieving are quite small when proper technique and care are exercised. One purpose of this test method is to minimize these errors to the negligible point. It is doubtful that much would be gained in attempting to correct the sampling data for testing errors in the way of fewer increment requirements. Actually, there is a gain in a margin of safety by considering all the error as sampling.

X3.11 Finally, these considerations are for the first estimation of the sample size. More data and experience may dictate a change in any sampling plan.

X4. ILLUSTRATION OF THE USE OF PRECISION LIMITS "r" AND "R" (LISTED IN Table 2)

X4.1 To illustrate the use of "r", consider Material A and its 40 mesh screen. Further, assume that a sand supplier produces Material A to the same 40 Mesh target value equal to the average listed in Table 1, 22.4% retained. A single shipment test , like 40 Mesh = 29.0 is suspiciously high. If a retest of the 29.0 done in the same lab yielded 27.0 then both tests are considered valid because they do not differ by more than the repeatability limit r = 2.2 (see Table 2, Sand A, 40 Mesh). The average of the two results, 40 Mesh = 28.0 is then the best single number to characterize the shipment. However, if the retest in the same lab showed 26.0, then the difference between the pair of numbers (29–26) exceeds r = 2.2, meaning one or the other or both tests are suspect and a third test is required to characterize the shipment. If the initial testing is done in the supplier's laboratory and the retest is done by the customer, then the situation is analogous to the above except that "R." is referenced instead of "R.". If a third test is required in such cases of buyer check testing, then the reproduced test is usually done in a separate independent or commercial referee laboratory.

Designation: C 674 – 88 (Reapproved 2006)

Standard Test Methods for Flexural Properties of Ceramic Whiteware Materials[1]

This standard is issued under the fixed designation C 674; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover determination of the modulus of rupture and the modulus of elasticity of fired ceramic whitewares bodies, formed by any fabrication method, and are applicable to both glazed and unglazed test specimens.

1.2 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Methods

2.1 The specimens, either cylindrical, or rectangular, are supported on knife edges over a suitable span and a direct load is applied at the midpoint between the supports at a uniform rate until breakage occurs. The modulus of elasticity may be determined by halting the applied load at definite intervals and measuring the deflection of the specimen at the midpoint to the nearest 0.001 in. (0.025 mm).

3. Significance and Use

3.1 These test methods provide a means for determining the modulus of rupture and the modulus of elasticity, which may be required in product specifications.

4. Apparatus

4.1 *Testing Machine*—Any suitable testing machine may be used, provided uniform rates of direct loading can be maintained.

4.1.1 For all specimens the loading rate should be such that the specimen should fail in approximately 1 min. Table 1 lists loading rates that shall be used for each size specimen. For strengths of specimens intermediate of those specified, interpolation can be used to obtain the equivalent loading rate.

4.1.2 For the prescribed rectangular specimens the loading rate shall be 1000 ± 150 lbs (454 ± 68 kg)/min.

4.2 *Bearing Edges*—For the support of the test specimen, two steel (or high-strength ceramic) knife edges rounded to a radius of 0.125 in. (3.18 mm) shall be provided. The load shall be applied by means of a third steel knife edge rounded to a radius of 0.125 in. When testing rectangular specimens, the supporting members for the bearing edges shall be constructed to provide a means for alignment of the bearing surfaces with the surfaces of the test specimen (it being possible that the rectangular specimen may be wedge-shaped or twisted). The apparatus shown in Figs. 1 and 2 are suggested as suitable devices for ensuring proper spacing and alignment of the bearing edges for rectangular test specimens.

5. Test Specimens

5.1 *Preparation of Specimens*—Form, fire, and finish the specimens by the appropriate methods, following practices used in actual production.

5.2 *Dimensions*—The specimens shall be approximately 1.125 in. (28.6 mm), 0.750 in. (19.2 mm), 0.500 in. (12.7 mm), or 0.250 in. (6.4 mm) in diameter, whichever diameter is most comparable to that of the finished product. The length shall be 6 ± 0.50 in. (153 ± 12.7 mm) to permit an overhang of at least 0.25 in. (6.4 mm) at each end when mounted on the supports. Note that the 0.25-in. specimens may be 3.75 ± 0.25 in. (95 ± 7 mm) long.

5.2.1 The dry-pressed specimens shall be rectangular bars approximately 1 in. (25.4 mm) by 0.50 in. (12.7 mm) in cross section and at least 4.50 in. (114 mm) in length to permit an overhang of at least 0.25 in. at each end when mounted on the supports.

5.3 *Handling*—Observe all due precautions in the forming, drying, and firing to produce straight test specimens of uniform cross section.

5.4 *Storage*—Cool test specimens taken warm from the kiln in a desiccator. If the testing must be delayed, store the bars in

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[1] These test methods are under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and are the direct responsibility of Subcommittee C 21.03 on Methods for Whitewares and Environmental Concerns.

Current edition approved Feb. 15, 2006. Published Febraury 2006. Originally approved in 1971. Last previous edition approved in 1999 as C 674 – 88 (1999).

TABLE 1 Loading Rates for Specimens

Specimen Size, in. (mm)	Span, in. (mm)	Nominal MOR, ksi (MPa)		
		10 (69)	30 (207)	50 (345)
		Loading Rate, lb/min (kg/s)		
1.125 (28.6) diameter	5 (127)	1100 (8.32)	3400 (25.70)	5600 (42.34)
0.750 (19.2) "	5 (127)	300 (2.27)	1000 (7.56)	1700 (12.85)
0.500 (12.7) "	5 (127)	100 (0.76)	300 (2.27)	500 (3.76)
0.250 (6.4) "	3 (76)	20 (0.15)	60 (0.45)	100 (0.76)
1 by 0.50 (25.4 by 12.7)	4 (102)	400 (3.02)	1250 (9.45)	2100 (15.88)

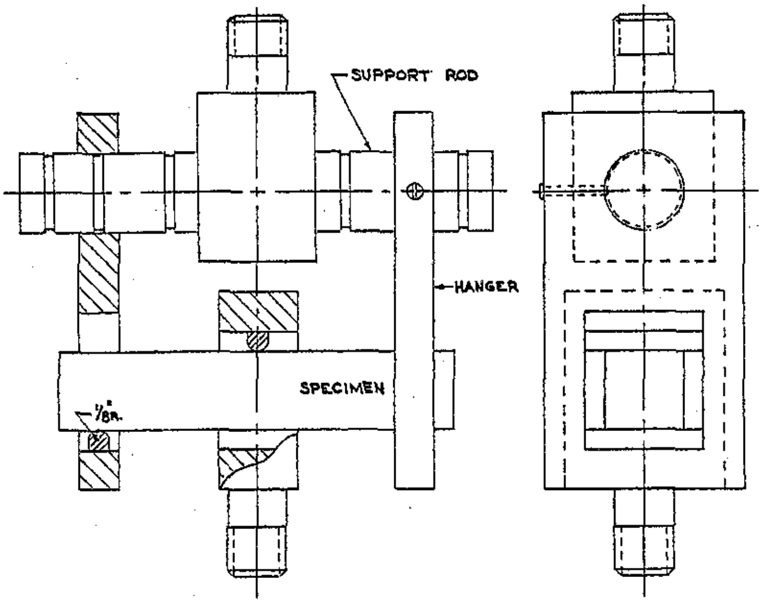

FIG. 1 Suggested Bearing Edge and Specimen Support

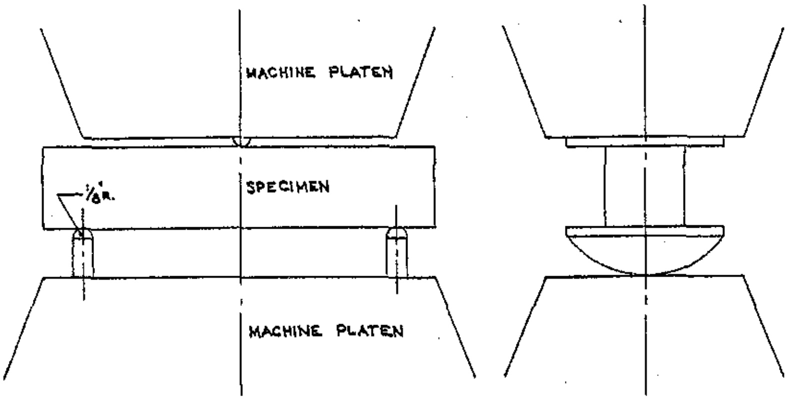

FIG. 2 Alternative Suggested Bearing Edge and Specimen Support

the desiccator, or in an electric oven at 110°C, and then cool in a desiccator before testing. When removing specimens from a hot kiln, take care to avoid thermal shock which will lead to erroneous results.

MODULUS OF RUPTURE

6. Procedure

6.1 Test at least ten dry specimens at room temperature.

NOTE 1—The modulus of elasticity may be determined as described in Sections 10-11 on the same type specimens used for the modulus of rupture test.

6.2 Place the cylindrical specimen on the bearing edge spaced 5 in. (127 mm) ± 2 % between centers, with th specimen overhanging each end by at least 0.25 in. (6.4 mm Apply the load at a right angle to the specimen and midwa (±2 %) between the supporting edges. Apply the load uni formly at the appropriate rate for the specimen size (see 4.1.1 until failure occurs. Measure the diameter at four approxi mately equidistant points at the break and take the average t the nearest 0.001 in. (0.0254 mm) for calculation purposes Break the 0.25-in. diameter rods between 3-in. (76-mm centers and note this in the report.

6.2.1 Place the rectangular test specimen on the bearing edges, spaced 4.0 in. (102 mm) between centers, with the specimen overhanging at each end by at least 0.25 in. (6.4 mm). Apply the load at a right angle to the 1-in. (25.4-mm) surface of the specimen and midway between the supporting edges. Break specimens glazed on only one of the 1-in. surfaces with the glazed surface either up or down, but break all of the specimens from any one lot in the same position and record this position. Apply the load uniformly at the rate of 1000 ± 150 lbs (454 ± 68 kg)/min, until failure occurs. Measure the width and thickness at the break to the nearest 0.001 in. (0.0254 mm).

7. Calculation

7.1 Calculate the modulus of rupture of each circular cross section specimen as follows:

$$M = 8PL/\pi d^3 \qquad (1)$$

where:

M = modulus of rupture, psi (or MPa);
P = load at rupture, lbf (or N);
L = distance between supports, in. (or mm);
d = diameter of specimen, in. (or mm).

7.1.1 Calculate the modulus of rupture of each rectangular specimen as follows:

$$M = 3\,PL/2bd^2 \qquad (2)$$

where:

M = modulus of rupture, psi (or MPa);
P = load at rupture, lbf (or N);
L = distance between supports, in. (or mm);
b = width of specimen, in. (or mm); and
d = thickness of specimen, in. (or mm).

8. Report

8.1 Report the following information:

8.1.1 Identification of the material tested,

8.1.2 Data and computed modulus of rupture for each specimen,

8.1.3 The adjusted average of the computed modulus of rupture values (discarding those values from the bars which, upon inspection, show obvious defect), and

8.1.4 In the case of glazed specimens, the position of the glazed surface or surfaces, with respect to the applied load.

8.2 The report may also include:

8.2.1 A description of type of fracture and the behavior of each specimen under load,

8.2.2 Name and rating of the machine used to make the test, and

8.2.3 A graph showing the individual values of modulus of rupture arranged in ascending order.

9. Precision and Bias

9.1 *Precision*:

9.1.1 *Interlaboratory Test Data*—An interlaboratory test was run in 1978 in which randomly drawn samples of five materials were tested in each of four laboratories. One operator in each laboratory tested ten specimens of each material. The components of variance for flexural strength results expressed as coefficients of variation were calculated as follows:

Single-operator component	2.42 % of the average
Between-laboratory component	18.79 % of the average

9.1.2 *Critical Differences*—For the components of variance reported in 9.1.1, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the following critical differences listed below:

	Critical Difference, % of Grand Average[A]	
Number of Observations in Each Average	Single-Operator Precision	Between-Laboratory Precision
10	6.72	52.10

[A] The critical differences were calculated using $t = 1.960$ which is based on infinite degrees of freedom.

9.1.3 *Confidence Limits*—For the components of variance noted in 9.1.1, single averages of observed values have the following 95 % confidence limits:

	Width of 95 % Confidence Limits, Percent of the Grand Average[A]	
Number of Observations in Each Average	Single-Operator Precision	Between-Laboratory Precision
10	±4.75	±36.84

[A] The confidence limits were calculated using $t = 1.960$ which is based on infinite degrees of freedom.

9.2 *Bias*—No justifiable statement on bias is possible since the true value of flexural strength of ceramic whiteware materials cannot be established and will be different for each specific material.

MODULUS OF ELASTICITY

10. Procedure

10.1 Support the specimen in the same way as for the modulus of rupture determination. Set any type of deflectometer capable of indicating to 0.001 in. (0.0254 mm) to measure deflection at mid-span relative to the ends of the span. Apply the load uniformly (see either 4.1.1 or 4.1.2) in accordance with the shape of the specimen. Stop the loading at 15 % increments of the expected total breaking load, as may previously have been determined in the modulus of rupture tests, and record the corresponding deflections.

11. Calculation

11.1 Plot the load-deflection readings to a convenient scale and draw a straight (stress-strain) line to represent, as nearly as possible, the average of the plotted points below the elastic limit. (In some materials, increasing departures from a straigh line may be noted at the higher stress. Such evidence of plastic flow, or of non-recoverable strain, indicates that the elastic limit has been exceeded.) If the line does not pass through the zero point, draw a corrected line through this point parallel to the stress-strain line.

11.1.1 Calculate the modulus of elasticity, E, for rectangular specimens from the coordinates of some convenient point on the corrected line as follows:

$$E = W^1L^3/4\Delta bd^3 \tag{3}$$

where:

W^1 = load coordinate of the selected point, lbf (or N);
L = length of span, in. (or mm);
Δ = deformation coordinate of the selected point, in. (or mm);
b = width of specimen at the center, in. (or mm); and
d = thickness of specimen at the center, in. (or mm).

11.1.2 Calculate the modulus of elasticity, E, for circular cross-section specimens from the coordinates of some convenient point on the corrected line as follows:

$$E = 4W^1L^3/3\pi\Delta d^4 \tag{4}$$

where:

W^1 = load coordinate of the selected point, lbf (or N);
L = length of span, in. (or mm);
Δ = deformation coordinate of the selected point, in. (c mm); and
d = diameter of specimen, in. (or mm).

12. Report

12.1 Report the following information:

12.1.1 Identification of the material tested,

12.1.2 Stress-strain data and modulus of elasticity for eac specimen, and

12.1.3 In the case of glazed specimens the position of t glazed surface or surfaces.

13. Precision and Bias

13.1 Specific information to support a precision and bi statement for modulus of elasticity is not yet available.

14. Keywords

14.1 ceramic whiteware materials; flexural properties

Designation: C 322 – 03

Standard Practice for Sampling Ceramic Whiteware Clays[1]

This standard is issued under the fixed designation C 322; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures for sampling bulk and bagged shipments of ceramic whiteware clays.

2. Sampling

2.1 Where a bulk shipment consists of lumps, a number of samples shall be taken from different parts of the rail car or truck during unloading, so that the final sample will represent an average of all parts of the shipment from top to bottom. For a 30-ton (27-Mg) unit, no less than 20 samples of approximately 10 lb (4.5 kg) each shall be taken from different parts of the rail car or truck. This may be done by removal during loading or unloading, at spaced intervals. The lumps shall then be broken to pieces no larger than 4 in. (102 mm), in the largest dimension, and the several samples shall be made into a composite lot, by turning with a shovel on a clean floor. The composite shall then be quartered or riffled to provide a 10-lb laboratory sample.

2.2 Sampling of hopper car or hopper truck shipments is to be done during unloading of the car or truck.

2.3 Where a bulk shipment consists of shredded or coarsely ground clay, sampling shall be done at 20 points for a 30-ton (27-Mg) unit; the samples may be taken with a shovel, or with a grain sampler, if the form of the clay permits. The samples so taken shall then be mixed thoroughly, and quartered or riffled to form a 10-lb (4.5-kg) (or proportionately larger) laboratory sample.

2.4 For bagged lots of ground or air-floated clay, the number of samples taken shall depend on the number of units in a shipment. A grain-sampler or similar sampling instrument shall be used to take samples which then shall be combined, mixed, and quartered or riffled to obtain a 10-lb (4.5-kg) laboratory sample. Where a shipment consists of 100 bags or less, the number of bags sampled at random shall be not less than 5 and preferably 10. When the number of bags is greater than 100 but less than 500, the number of bags sampled shall not be less than 15. For lots of from 500 to 1000 bags, 20 bags shall be sampled. For shipments of 1000 to 2000 bags, 30 bags shall be sampled at random.

2.5 When the clay is shipped in slurry form a representative sample may be obtained by taking a single grab sample from any portion of the shipment except by skimming the top surface. If the shipment has been unagitated for a period of two weeks or more the shipment can be agitated by inserting an air lance to the bottom of the vessel for a period of ten minutes. One two-liter sample is required.

2.6 Preshipment samples of slurries and bulk materials from the supplier can be used, replacing the sampling done when the shipment is received.

3. Precision and Bias

3.1 *Precision*— It is not possible to specify the precision of the procedure in this test method beyond stating that the sample size must be +/- 10% of the weight specified.

3.2 *Bias*—No information can be presented regarding the bias of this procedure.

4. Keywords

4.1 clay; sampling

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[1] This practice is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.04 on Raw Materials.

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Designation: C 323 – 56 (Reapproved 2006)

Standard Test Methods for Chemical Analysis of Ceramic Whiteware Clays[1]

This standard is issued under the fixed designation C 323; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analysis of clays used in the manufacture of ceramic whitewares.

1.2 The analytical procedures appear in the following order:

	Section
Moisture	7
Loss on Ignition	8
Silica	9
Iron, Aluminum, and Titanium Oxides	10
Iron Oxide	11
Titania	12
Alumina	13
Lime	14
Magnesia	15
Alkalies	16

NOTE 1—These test methods have been compiled as standard procedures for use in referee analyses. These test methods, however, when the determination of iron oxide as Fe_2O_3 is involved, are not intended to preclude the use of other procedures that give results within the permissible variations. For the sake of uniformity the classical Zimmerman-Reinhardt procedure is specified for the determination of iron oxide. It is recognized that numerous other procedures are equally accurate and often more convenient. The other procedures commonly in use include reduction of an oxidized solution with zinc or other metal, and titration with standard potassium permanganate ($KMnO_4$) or potassium dichromate ($K_2Cr_2O_7$) solution, as well as titration with a standard solution of titanous chloride in an oxidized solution. These procedures shall be considered acceptable, provided the analyst has obtained results by his special procedure that check with the Zimmerman-Reinhardt procedure within the limits specified in Section 17. It is suggested that National Institute of Standards and Technology standard samples be used for checking the accuracy of procedures.

It will be understood that the making of a complete analysis of a ceramic whiteware clay is a difficult procedure requiring a wide knowledge of the chemistry involved in the operations and a thorough training in carrying out the work. A skilled analyst of good training is therefore required to do the work. The descriptions here given cover the vital points of procedure, but frequent reference in regard to the details of the various manipulations should be made to "Applied Inorganic Analysis" by Hillebrand and Lundell[2] and to similar publications. Particularly in the determination of alumina, reference should be made to *Scientific Paper No. 286* of the National Bureau of Standards.[3]

1.3 The values stated in acceptable metric units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [4]

C 322 Practice for Sampling Ceramic Whiteware Clays

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Reagents

3.1 Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Unless otherwise indicated, references to water shall be understood to mean distilled water. Paragraphs 3.1.1-3.1.16 include those reagents common to two or more of the analytical procedures. Other reagents will be found listed with the particular test method in which they are prescribed.

3.1.1 *Concentrated Acids and Ammonium Hydroxide*—Concentrated acids and ammonium hydroxide of approximately the following specific gravities or concentrations will be required:

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[1] These test methods are under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and are the direct responsibility of Subcommittee C 21.04 on Raw Materials.

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[2] Hillebrand, W. F., and Lundell, G. E. F., *Applied Inorganic Analysis*, Wiley and Son, New York, 1929.

[3] Blum, W., "Determination of Alumina as Oxide," National Bureau of Standards, *Scientific Paper No. 286*.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, o contact ASTM Customer Service at service@astm.org. For *Annual Book of AST Standards* volume information, refer to the standard's Document Summary page o the ASTM website.

[5] *Reagent Chemicals, American Chemical Society Specifications*, America Chemical Society, Washington, DC. For suggestions on the testing of reagents n listed by the American Chemical Society, see *Analar Standards for Laborator Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacope and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockvill MD.

Hydrochloric acid (HCl)	1.19 sp gr
Nitric acid (HNO_3)	1.42 sp gr
Sulfuric acid (H_2SO_4)	1.84 sp gr
Hydrofluoric acid (HF)	40 %
Perchloric acid ($HClO_4$)	60 to 70 %, cp[A]
Sulfurous acid (H_2SO_3)	6 % solution[B]
Ammonium hydroxide (NH_4OH)	0.90 sp gr

[A] Lower purity varieties may contain aluminum oxide, (Al_2O_3), as an impurity.

[B] As supplied by reagent manufacturers.

3.1.2 *Diluted Acids and Ammonium Hydroxide*—The diluted acids and ammonium hydroxide referred to are of varying percentages by volume. They shall be made up by mixing proportional volumes of the concentrated reagent and water. The diluted sulfuric acid mixtures shall be made up by slowly stirring the acid into the water. These diluted acids and ammonium hydroxide are designated in the methods as (1 + 4), (1 + 9), and so forth, except very diluted solutions which are referred to by the percent of reagent added. The designation in parentheses indicates the ratio of the volume of the concentrated reagent to the volume of water; for example, H_2SO_4 (1 + 9) contains 10 volume % of H_2SO_4 (sp gr 1.84). The following will be required:

	Volume %
HCl	50
	20
H_2SO_4	50
	10
	5
HNO_3	30
	3
NH_4OH	50

3.1.3 *Ammonium Chloride (2 %)*—Dissolve 2 g of NH_4Cl in 100 mL of water.

3.1.4 *Ammonium Oxalate Solution (Saturated)*—Dissolve 4 g of $(NH_4)_2C_2O$ in 100 mL of water.

3.1.5 *Chloroplatinic Acid Solution* (10 %).

3.1.6 *Diammonium Phosphate Solution*—Dissolve 10 g of $(NH_4)_2HPO_4$ in 100 mL of water.

3.1.7 *Ethyl Alcohol (80 %)*—Prepare a solution containing 80 volume % of ethyl alcohol in water.

3.1.8 *Ethyl Alcohol (Absolute)*—Certain commercial brands of denatured absolute alcohol are satisfactory as well as being considerably less expensive than the reagent grade absolute alcohol.

3.1.9 *Hydrogen Peroxide* (30 %) (H_2O_2).

3.1.10 *Manganese Sulfate Solution*—Dissolve 70 g of crystalline $MnSO_4$ in 500 mL of water. Add 140 mL of phosphoric acid (H_3PO_4, sp gr 1.7), and 130 mL of sulfuric acid (H_2SO_4, sp gr 1.84). Dilute to 1 L.

3.1.11 *Mercuric Chloride Solution (Saturated)*—Prepare a saturated solution of $HgCl_2$.

3.1.12 *Potassium Permanganate, Standard Solution* (0.1*N*)—Dissolve 3.25 g of $KMnO_4$ in 1000 mL of water. Allow to stand for one week, filter through an asbestos mat, porous glass, or porcelain filter, and keep in a dark place. Standardize against the National Institute of Standards and Technology standard Sample No. 40*c* of sodium oxalate.

3.1.13 *Potassium Permanganate, Standard Solution* (0.04*N*)—Dissolve 2.5 g of $KMnO_4$ in water and make up to 2 L. Allow to stand for one week, filter through an asbestos mat, porous glass, or porcelain filter, and keep in a dark place. Standardize against the National Institute of Standards and Technology standard Sample No. 40*c* of sodium oxalate.

3.1.14 *Sodium Arsenite, Standard Solution*— Dissolve 0.908 g of arsenious oxide, (As_2O_3), in a small amount of hot sodium carbonate (Na_2CO_3) solution, cool, filter, and dilute to 1 L. Standardize against a steel of known manganese content.

3.1.15 *Stannous Chloride Solution (50 g/L)*—Dissolve 50 g of $SnCl_2$ in 100 mL of HCl and dilute to 1000 mL. Keep a few pieces of metallic tin in the bottle.

3.1.16 *Titania, Standard Solution*—Weigh out 0.05 g of calcined titanium dioxide (TiO_2). Fuse with 10 g of $K_2S_2O_7$ in a clean platinum crucible, keeping the temperature as low as possible to maintain fluidity. Cool, and dissolve in about 300 mL of H_2SO_4 (1 + 5). Cool, transfer to a 500-mL volumetric flask, dilute to the mark with water, and mix thoroughly. To standardize the solution, take two 50-mL portions in 400-mL beakers, dilute, boil, and precipitate with NH_4OH. Filter, and wash with hot water. Place the papers in the original beakers, add 15 mL of HCl, stir to macerate the paper, dilute, and precipitate again with NH_4OH. Filter, and wash with hot water until free of alkali salts. Ignite carefully, blast, and weigh. From the weight determined, calculate the strength of the solution.

4. Sampling

4.1 *Selection of Sample*—Obtain the sample in accordance with Practice C 322.

4.2 Crush the sample in a small jaw or roll-type crusher with hardened tool-steel faces to pass a 2.36-mm (No. 8) sieve (Note 2). Crush the sample to pass a 850-µm (No. 20) sieve, mix, and quarter to about 50 g. Grind this 50-g sample so that it will all pass a 150-µm (No. 100) sieve, unless otherwise specified, mix thoroughly, and place in a container that will ensure freedom from contamination. Do fine grinding in a suitable mortar (agate, mullite, alumina, or boron carbide) to prevent the introduction of impurities. Take precautions to prevent contamination of the sample by steel particles from the sampling equipment during crushing or grinding.

Note 2—Detailed requirements for these sieves are given in Specification E 11.

5. Method of Analysis

5.1 Determine moisture on the sample in its ordinary air-dried condition. Determine all other percentage compositions on moisture-free samples and report accordingly on a moisture-free basis. The drying temperature recommended for all moisture determinations is 105 to 110°C. Whenever a sample is weighed out for any determination other than moisture, it shall be moisture-free. If preferred, the sample may be dried in a weighing bottle from which the required samples shall be weighed out.

6. Blank Determinations

6.1 Make blank determinations on the reagents for each constituent in the whiteware clay and deduct this blank in each case. For the determination of the silica (SiO_2) blank, approximately 0.25 g of Al_2O_3 should be added as aluminum chloride.

7. Moisture

7.1 Weigh 1.00 g of the sample and heat to constant weight at a temperature not under 105 nor over 110°C. Record the loss in weight as moisture.

8. Loss on Ignition

8.1 Weigh 1.000 g of the moisture-free (105 to 110°C) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000°C. Record the loss in weight as the ignition loss.

9. Silica

9.1 Weigh 0.5000 g of the moisture-free (105 to 110°C) sample into a platinum crucible containing about 5 g of powdered anhydrous Na_2CO_3 and mix well with a platinum wire. Cover the mixture with a little more Na_2CO_3. Heat gradually to the full heat of a good burner (1000 to 1100°C) maintained for about 1 h until complete solution is obtained. Place the crucible cover on a triangle, and when the melt has partially cooled, pour it on the lid (Note 3). When cool, place the crucible and lid in a 150-mL beaker, placing the button on a watch glass above the beaker. Add 30 mL of HCl (1 + 1). When solution is complete wash off the crucible and lid with HCl (1 + 4), taking care to remove all SiO_2. Place the button in the solution. Transfer the contents of the beaker to an evaporating dish and evaporate to dryness on a steam bath. Bake for 1 h at 110°C. Add 20 to 30 mL of HCl (1 + 1) and 50 mL of hot water. When all salts have been dissolved, allow to settle for several minutes and then filter through a general-purpose grade acid-washed medium-retention filter paper. Wash the SiO_2 three times by decantation using 20- to 30-mL portions of first hot water, then HCl (1 + 1), then hot water again. Transfer the precipitate to the filter paper, removing all SiO_2 from the dish with a policeman. Wash the paper and precipitate with hot water until free from salt. To recover the small amount of SiO_2 remaining in the filtrate, evaporate to dryness, using the same procedure for baking and filtering as before. Combine the two precipitates, place in a platinum crucible, and burn off the paper carefully to prevent any loss of SiO_2. Ignite the sample to constant weight at 1100 to 1200°C (15 to 20 min is usually sufficient), cool in a desiccator, and weigh. Moisten the residue with several millilitres of water, add 10 mL of HF and three or four drops of H_2SO_4. Evaporate the solution to dryness, ignite carefully to prevent decrepitation, and blast for several minutes at 1100°C. Cool the crucible in a desiccator, weigh, and repeat blasting to constant weight. The loss in weight from the original silica residue represents the SiO_2 content, except for that part of the SiO_2 which is later recovered from alumina, and so forth.

NOTE 3—Another scheme to aid in subsequent solution of the fused melt is to rotate the crucible as it cools, spreading the mass up the side walls.

10. Iron, Aluminum, and Titanium Oxides

10.1 Fuse the residue with 1 g of fused potassium pyrosulfate ($K_2S_2O_7$) or sodium pyrophosphate ($Na_2S_2O_7$), dissolve in a small amount of water, and add to the filtrate from the silica determination (Section 9). Add 5 g of NH_4Cl and three drops of 0.1 % methyl red solution. Heat the solution almost to boiling, and add slowly NH_4OH (1 + 1) until the indicator has changed to a yellow color. Boil for several minutes to remove the excess ammonia. Allow to settle for 30 min and decant through an open, rapid-filtering acid-washed filter paper, transferring the precipitate to the paper and washing the beaker and paper several times with a warm 2 % NH_4Cl solution. Reserve the filtrate, "A," for the determination of calcium oxide (CaO) and magnesium oxide (MgO) (Section 14). Return the precipitate and paper to the original beaker, add 50 mL of hot water and 10 mL of HCl (sp gr 1.19). Stir until the precipitate is dissolved and the paper is well macerated. Dilute to about 200 mL with hot water, precipitate and filter as before. Combine this filtrate "B" with filtrate" A." Wash the paper and precipitate with a warm 2 % NH_4Cl solution. Place the precipitate in a weighed platinum crucible and ignite. Continue the ignition at 1200°C to constant weight (15 to 20 min is usually sufficient). Cool in a desiccator, and weigh with the crucible covered with the lid. The R_2O_3 consists of the aluminum oxide (Al_2O_3), TiO_2, and Fe_2O_3 present in the sample. In addition, there may be small amounts of phosphoric anhydride (P_2O_5), zirconium oxide (ZrO_2), vanadium pentoxide (V_2O_5), and chromic oxide (Cr_2O_3).

11. Iron Oxide

11.1 *Procedure A: Fe_2O_3 Determined on R_2O_3 Sample*—Heat the R_2O_3 precipitate (Note 4) obtained in the determination of iron, aluminum, and titanium oxides (Section 10), with fused $K_2S_2O_7$ or $Na_2S_2O_7$ until solution is complete. Dissolve the fusion in 50 mL of H_2SO_4 (1 + 9) and evaporate to fumes. Cool, dilute with water, and filter off the SiO_2, washing with hot water. Reserve the filtrate for the determination of Fe_2O_3 and TiO_2. Ignite the SiO_2 in a platinum crucible and weigh. Treat the precipitate with 5 mL of HF and two or three drops of H_2SO_4. Evaporate to dryness, ignite, and weigh. The loss in weight represents extra SiO_2 which should be added to that determined previously and also deducted from the weight of the R_2O_3 precipitate. Evaporate the filtrate obtained in correcting the R_2O_3 precipitate for SiO_2 to about 75 mL. Cool, and dilute to 100 mL in a volumetric flask. Reserve 25 mL for the determination of TiO_2(Section 12). To the remainder, add 25 mL of HCl (1 + 1) and heat to boiling. Reduce the iron by adding $SnCl_2$ solution drop by drop from a pipet with constant swirling of the beaker until the solution is colorless. Then add one drop in excess. Cool quickly in running water, then add at one stroke 15 mL of saturated $HgCl_2$ solution. Allow to stand for 3 min, then transfer with washing to a 1000-mL beaker containing 300 mL of cold distilled water and 25 mL of $MnSO_4$ solution. Titrate with standard 0.04*N* $KMnO_4$ solution, added very slowly while stirring constantly, until a permanent pink end point is obtained.

NOTE 4—Instead of fusing directly in the platinum crucible in which the R_2O_3 was ignited the precipitate may be brushed into a porcelain crucible and then fused with $K_2S_2O_7$ or $Na_2S_2O_7$. This avoids loss of platinum by the action of the pyrosulfate, and no platinum is present in the filtrate to interfere with the iron determination.

11.2 *Procedure B: Fe_2O_3 Determined on a Separate Sample*—Weigh 1.00 g of the finely ground, moisture-free (10 to 110°C) sample into a platinum crucible, add ten drops perchloric acid ($HClO_4$) and 20 mL of HF, and heat almost to dryness on a hot plate. Add 5 to 10 mL of $HClO_4$ and heat until residue has dissolved (Note 5). Cool, place crucible in a 400-mL beaker, add 100 mL of water, and heat to boiling. Any residue present, other than SiO_2, should be filtered off and fused with $K_2S_2O_7$ or $Na_2S_2O_7$ in a porcelain crucible and added to the main solution.

NOTE 5—Decomposition of ceramic whiteware clays and some fired whiteware materials can be effected equally as well by substituting an equal volume of H_2SO_4 (1 + 1) for the $HClO_4$. In this case, heat to fumes once, cool, dilute, filter, and fuse any residue remaining undissolved.

11.3 Determine iron, using one of the approved methods referred to in Note 1 of Section 1.

12. Titania

12.1 Determine TiO_2 colorimetrically by use of a photometer, as follows: Transfer the 25-mL portion reserved for the determination of TiO_2 (Section 11) to a 100-mL volumetric flask. Add 5 mL of H_2SO_4 (1 + 1). Cool to room temperature and dilute to the mark. Transfer exactly half of the solution to another 100-mL volumetric flask. Dilute one of the flasks to the mark with H_2SO_4 (1 + 19). To the other, add 5 mL of 3 % H_2O_2, then dilute nearly to the mark with H_2SO_4 (1 + 19), adjust temperature to 25 ± 1°C, then adjust volume exactly and let stand at least 5 min. Transfer a portion from the first flask to a cuvette and set the potentiometer scale reading at zero. Then measure the absorption of the solution in the second flask. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solution to H_2SO_4 (1 + 19), develop color with 5 mL of 3 % H_2O_2, let stand 5 min and read the absorption, using H_2SO_4 (1 + 19) for the zero setting of the potentiometer scale.

NOTE 6—If a spectrophotometer is used, measure the absorption at mean transmission of 420 nm. If a filter photometer is used, use a glass with a maximum transmission in the region of 420 nm.

NOTE 7—As an alternative method, TiO_2 can be determined in the 25-mL portion reserved for this purpose (Section 11) by oxidizing both the sample and the standard TiO_2 solution with several drops of a 30 % H_2O_2 solution. Compare the colors either in Nessler tubes or in a suitable colorimeter. Use a 5 % H_2SO_4 solution for diluting purposes in matching the colors.

13. Alumina

13.1 Subtract the calculated weight of Fe_2O_3 (see 11.2), TiO_2 (Section 12), and SiO_2 (Section 9) from the weight of R_2O_3 (Section 10). The remainder is the weight of Al_2O_3 plus small amounts of the oxides which may include those previously mentioned in Section 10. These are generally considered as Al_2O_3 in reporting the analysis of ceramic whiteware clays.

14. Lime

14.1 Evaporate the combined filtrates reserved (Section 10) for the determination of CaO and MgO to about 200 mL, add 10 to 15 mL of the saturated ammonium oxalate solution and 2 to 3 mL of NH_4OH. Heat for 1 to 2 h, by which time the volume should be about 75 to 100 mL. Allow the precipitated calcium oxalate to settle. Decant through a dense filter paper, taking care to retain the precipitate in the beaker, wash sever times with warm water by decantation, and then wash the pap until free from soluble salts. Reserve the filtrate for the Mg determination (Section 15). Return the paper to the beak containing the precipitate, add 100 mL of H_2SO_4 soluti (5 %), warm, and titrate to a faint pink end point with standa 0.04*N* $KMnO_4$ solution. A blank should be previously dete mined for the effect of the paper.

NOTE 8—For greater accuracy, a double precipitation should be ma in which case, after precipitating the calcium oxalate as described abo decant the liquid and wash the beaker and paper several times with wa water. Dissolve the precipitate on the paper with warm HCl (1 + allowing it to run into the beaker containing the major portion of calcium oxalate. Wash the paper with hot water. To the solution (about to 100 mL in volume) add several millilitres of saturated $(NH_4)_2C_2$ solution and NH_4OH in slight excess. Heat for 2 h, filter, wash, and titr as described above.

15. Magnesia

15.1 Evaporate the filtrate from the CaO determinati (Section 14) to about 150 to 200 mL and add 2 to 3 g diammonium phosphate ($(NH_4)_2HPO_4$), stir until dissolve and then add NH_4OH until alkaline and then 20 mL in exce Allow the solution to stand overnight. Filter and wash with 5 NH_4OH. Dissolve the precipitate on the paper with hot H (1 + 4), allowing it to run into the beaker containing t precipitate. Wash the paper with hot water. To the solutic which should be not more than 100 mL in volume, add 0.1 0.2 g of $(NH_4)_2HPO_4$. Make ammoniacal, and then add a slig excess while stirring constantly until the precipitate is w formed. Then add 10 mL more of NH_4OH and allow to sta overnight or at least for 4 h. Filter through a dense filter pap Transfer the precipitate to the paper and wash well with 5 NH_4OH. Place the paper in a weighed platinum or porcela crucible, burn off the paper at a low temperature (bel 900°C), and ignite to constant weight at 1050 to 1100°C (15 30 min is sufficient).

16. Alkalies[6]

16.1 Weigh 1.00 g of the moisture-free (105 to 110° sample (ground to an impalpable powder) and 1.0 g of NH_4 into an agate mortar and mix well. Add 7 to 8 g of calci carbonate ($CaCO_3$) (Note 9) and again mix intimately. Plac 3.2-mm (⅛-in.) layer of $CaCO_3$ in the bottom of a platin crucible, and then add the above mixture, tapping the cruci occasionally to obtain a dense mass. Place a 3.2–mm layer $CaCO_3$ on the top. Heat the crucible over a low flame u ammonia fumes are no longer given off, then increase the h so that the bottom half of the crucible is a dull red and maint this temperature for about 1 h. Cool, fill the crucible thr fourths full of water, and heat until the contents can be tak out and crushed in an agate mortar. Transfer to a platinum porcelain dish by means of a jet of water. Evaporate to a l volume, decant through a dense paper, and wash the material the dish several times by decantation with warm wat Transfer to the paper and wash several times with hot wat

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[6] This procedure for the determination of alkali is the J. Lawrence Smith Met

Acidify with several millilitres of HCl and evaporate to a volume of 150 to 200 mL. Add several millilitres of NH_4OH and sufficient $(NH_4)_2CO_3$ to precipitate the lime, keeping the dish covered with a watch glass. Warm until the precipitate settles out. Filter and wash with warm water. Evaporate the solution to a low volume, then add a small lump of $(NH_4)_2CO_3$ to determine whether practically all calcium has been precipitated. If no precipitate forms, evaporate to dryness, otherwise precipitate and filter as before. Drive off the ammonium salts by heating just short of a dull red. Dissolve the residue in water and add a few millilitres of a saturated solution $(NH_4)_2C_2O_4$ and 1 to 2 mL of NH_4OH to precipitate the last trace of calcium. Heat for 30 to 45 min, filter, and wash with water containing $(NH_4)_2C_2O_4$ (0.1 %). Catch the filtrate in a weighed platinum dish. Add several drops of HCl and evaporate to dryness. Ignite gently as before and weigh as sodium chloride (NaCl) and potassium chloride (KCl).

NOTE 9—Calcium carbonate of the ACS grade "low in alkalies and heavy metals" shall be used.

16.2 The separation of the potassium and sodium must be carried out in an atmosphere free from ammonia fumes. Add to the solution of the combined chlorides in a small porcelain dish, sufficient chloroplatinic acid solution to react with all of the sodium and potassium. The necessary amount to use is readily calculated from the known strength of the platinum solution and the weight of mixed chlorides counted as NaCl. The dilution of the resulting solution should be such that when heated on the steam bath any precipitate that may have formed entirely dissolves. This is to prevent inclusion of mother liquor in a mass of crystals suddenly formed. Evaporate until the solution is just syrupy enough to solidify on cooling. Do not evaporate to dryness, as this will dehydrate the sodium salt and render it less soluble in alcohol. Drench the residue with alcohol (80 %), filter by decantation through a small paper, wash by decantation with more of the alcohol, crushing the crystals with a small pestle or a widened and rounded end of a short glass rod. Reserve the filtrate and washings if sodium is to be determined directly. The residue should be golden yellow. An orange-red color indicates incomplete removal of the sodium salt. It is unnecessary to bring the mass of the precipitate upon the filter. Dry the dish and paper for a few moments to remove adhering alcohol. Dissolve the precipitate on the filter with hot water, catching the solution in a weighed crucible or small dish of platinum. Evaporate to dryness and add the salt that is still in the porcelain dish. If the salt is at all lumpy, redissolve it in water and again evaporate to dryness. Heat for 1 h at 130°C in an air oven (100°C suffices for very small amounts of fine-grained precipitate). It is necessary to cover the receptacle at first because the precipitate is prone to decrepitate. When dry, cool and weigh as K_2PtC_6. Calculate the oxides, as follows:

$$KCl = \text{wt of } K_2PtCl_6 \times 0.3068 \quad (1)$$

$$K_{20} = \text{wt of } K_2PtCl_6 \times 0.1941$$

$$NaCl = \text{total chlorides} - KCl$$

$$Na_2O = NaCl \times 0.5303$$

17. Precision and Bias

17.1 *Precision*—In all cases, check determinations shall be made, and the results shall be redetermined if satisfactory checks are not obtained. Results shall be considered satisfactory if the differences between check determinations do not exceed the following limits:

	Permissible Variations Between Check Determinations, max, %
For silica or other constituent amounting to 30 %[A] or over	0.3
For alumina or other constituent amounting to 10 to 30 %[A]	0.2
For any other constituent amounting to under 10 %[A]	0.1

[A] These figures are stated in terms of the whole sample as 100 %.

17.2 *Bias*—The bias of these test methods have not yet been determined.

18. Keywords

18.1 ceramic whiteware clays; chemical analysis

INTERNATIONAL

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Standard Test Method for Free Moisture in Ceramic Whiteware Clays[1]

This standard is issued under the fixed designation C 324; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of free moisture in ceramic whiteware clays. Whiteware clays may be shipped as a bulk shipment in lumps, a bulk shipment of shredded or coarsely ground clay, or in bagged lots of ground or airfloated clay. Directions are given in this test method for obtaining representative samples of the clay shipment to be used in subsequent tests for the properties of the clay in the shipment.

1.2 *This standard does not purport to address all of the safetyconcerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 332 Specification for Lightweight Aggregates for Insulating Concrete

3. Sampling

3.1 The sample shall be obtained in accordance with Specification C 322. The sample submitted for testing shall weigh not less than 1000 g and shall be kept in an airtight containe to prevent loss of water before testing.

4. Procedure

4.1 Remove the sample of clay from its container. Weigh about 500 g of the sample to the nearest 0.1 g. Spread out the weighed portion of the sample in a weighed shallow metal o porcelain container, and dry at 100 to 110°C for 24 h in a drying oven. Reweigh the dried clay, as quickly as possible, to the nearest 0.1 g.

5. Calculation

5.1 Calculate the percentage of free moisture to the nearest 0.1 % as follows:

$$\text{Free moisture, dry weight basis, } \% = [(A - B)/B] \times 100 \quad (1)$$

$$\text{Free moisture, wet weight basis, } \% = [(A - B)/A] \times 100 \quad (2)$$

where:

A = "as-received" weight of the portion of the sample used and

B = weight of sample after drying.

6. Keywords

6.1 ceramic whiteware clays; free moisture

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[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.04 on Raw Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.