

**Graduate Student Recruitment and Training Support**

**Report for**

**One Ajahn, One Project**

**1 January 2005 through 31 December 2005**

**Calendar Year 2548**

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This project is titled "Graduate Student Recruitment and Training Support", and had the goal of attending an International Conference in chemistry or crystallography, accompanied by at least one SUT graduate student, to make presentations related to the research my group is working on at SUT. During the calendar year 2005, our research was presented at two international conferences and three domestic conferences. five of my SUT students presented papers at international, and thirteen of my SUT group members (including a guest researcher from the University of Peradeniya, Sri Lanka) presented papers at domestic conferences. Six of our international abstracts are published and searchable in the Chemical Abstracts/SciFinder Scholar database. Six of our domestic papers were published in conference proceedings volumes. I visited King Mongkut's University of Technology in Thonburi and Mahidol University International School in Nakhon Phatom for recruitment purposes.

#### Results:

##### I. Attended two International Conferences and represented at one other.

- a. Represented by a student attending Nanotec, Nano Material Fabrication Technologies held in Chiang Mai, Thailand, 28-29 July, 2005. (documents available: 1 abstract)

Attending student: Kittipong Chainok.

Financial support for partial payment of expenses from SUT.

- i. "Synthesis and Characterization of Organic-Inorganic Hybrid Cobalt Vanadates:  $[\text{Co}(\text{Im})_4\text{V}_4\text{O}_{12}]$ , Im = imidazole", Kittipong Chainok; Kenneth J. Haller, NANOTEC, Nano Material Fabrication Technologies, Chiang Mai, Thailand: 28-29 July, 2005.

- b. Attended IUCr2005, the XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005. (documents available: 6 abstracts)

Accompanying students: Kittipong Chainok, Samroeng Krachodnok, Ratchadaporn Puntharod, Nongnaphat Khosavithikul, and Weenawan Somphon

Financial support for payment of student registration and accomodation expenses and one student for air travel from the International Union of Crystallography student travel awards, partial support for poster preparation and travel expenses for two students from SUT.

- ii. "Noncovalent Interactions in a Three Component Supramolecular Structure", Kittipong Chainok; Kenneth J. Haller; Herman H.-Y. Sung; Ian D. Williams, IUCr2005, XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005.
- iii. "A New Organically Templated Vanadium Arsenate", Samroeng Krachodnok; Kenneth J. Haller, IUCr2005, XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005.
- iv. "An Order-Disorder Phase Transition in  $[\text{Ag}(\text{bipy})\text{NO}_3]_n$ ", Weenawan Somphon; Kenneth J. Haller; A. David Rae, IUCr2005, XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005.
- v. PRIZE: Ms. Weenawan Somphon won the Oxford Cryosystems instrumentation prize for the Best Use of Low Temperature Crystallography for her work
- vi. PRIZE: Ms. Weenawan Somphon also won the International Union of Crystallography best student research presentation prize for the same presentation.
- vii. "Preparation and Characterization of  $\text{NiCl}(\text{NO})(\text{PPh}_3)_2$ ", Ratchadaporn Puntharod; Kenneth J. Haller, IUCr2005, XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005.

- viii. "Crystal Structure of  $[\text{Sn}(\text{Bu})_3(\text{O}=\text{PPh}_3)_2][\text{BPh}_4]$ ", Nongnaphat Khosavithikul; Kenneth J. Haller; Seik Weng Ng, IUCr2005, XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005.
  - ix. "Structure of 19-Hydroxyneohopane", Kenneth J. Haller, IUCr2005, XX International Union of Crystallography Congress and General Assembly held in Florence, Italy 23-31 August 2005.
  - x. All six abstracts for this meeting are published in *Acta Crystallographica A* and are searchable through Chemical Abstracts/SciFinder Scholar abstract database.
- c. Sent one student to The Eleventh Triuniversity Conference, TRI-UNIV2004, Chiang Mai, Thailand: 21-26 November 2004. (documents available: 1 abstract and one 4 page proceedings paper)  
 Student: Nongnaphat Khosavithikul.  
 Financial support from the conference organizers.
- xi. "Supramolecular Interactions in Four-Coordinate Azidonitrosylbis(Triphenylphosphine) Nickel", (with Nongnaphat Khosavithikul) Eleventh Triuniversity Conference, Chiang Mai University, Thailand, 21-26 November 2004.

## II. Attended three National Conferences.

- d. Attended the 9th Annual National Symposium on Computational Science and Engineering (documents available: eight abstracts, six conference proceedings papers, Crystallography Minisymposium paper list, Crystallography Workshop schedule) Accompanying students: Nongnaphat Khosavithikul, Weenawan Somphon, Aungkana Chatkon, Samroeng Krachodnok, and Kittipong Chainok.
- xii. Member of the Conference Organizing Committee.
  - xiii. (Sole) organizer of a Crystallography Minisymposium for the Computational Chemistry and Biology section of the conference.
  - xiv. (Sole) organizer of the half-day Conference Workshop of the title Crystallography Workshop.
  - xv. "Analyzing Concerted Noncovalent Interactions in Structures Dominated by Phenyl Rings", ORAL, Nongnaphat Khosavithikul; Aungkana Kiatpichitpong; Kenneth J. Haller, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB1-4, 23-25 March 2005.
  - xvi. "Refinement of a Commensurate Displacive Modulated Structure", ORAL, Kenneth J. Haller; Weenawan Somphon; A. David Rae; Ian D. Williams, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB1-5, 23-25 March 2005.
  - xvii. "Unraveling the Solvent/Cation Disorder in the Structure of a Vanadium Borate Cluster,  $[\text{V}_6\text{B}_{20}\text{O}_{50}\text{H}_8]^{n-}$ ", ORAL, Aungkana Chatkon; Kenneth J. Haller; Ian D. Williams, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB2-1, 23-25 March 2005.
  - xviii. "Supramolecular Structure Analysis: Detection of a Novel Carbonyl-Carbonyl Interaction in the Structure of Pyridine-3,5-Dicarboxylic Acid", ORAL, Samroeng Krachodnok; Kenneth J. Haller, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB2-6, 23-25 March 2005.
  - xix. "Supramolecular Structure Analysis: Trispicolinato Cobalt(III) Monohydrate,  $[\text{Co}(\text{C}_6\text{H}_4\text{O}_2\text{N})_3]\cdot\text{H}_2\text{O}$ ", ORAL, Kittipong Chainok; Kenneth J. Haller, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB2-7, 23-25 March 2005.
  - xx. "Refining a Multiply Twinned Modulated Structure: Triethyl Phosphine Sulfide", ORAL, Weenawan Somphon; Kenneth J. Haller; A. David Rae, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB5-3, 23-25 March 2005.
  - xxi. "Introduction to Twinning", ORAL, Kenneth J. Haller, ANSCSE9 Workshop on Crystallography, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand, 25 March 2005.
  - xxii. "Introduction to the Cambridge Structural Database", ORAL, Kenneth J. Haller, ANSCSE9 Workshop on Crystallography, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand, 25 March 2005.

- e. Attended the Royal Golden Jubilee PhD Congress VI held in Pattaya 28-30 April 2005. (documents available: two abstracts) Accompanying students: Weenawan Somphon and Samroeng Krachodnok.
- xxiii. "Organically Templated Transition Metal Vanadium Arsenates", Samroeng Krachodnok; Kenneth J. Haller, Royal Golden Jubilee PhD Congress VI, Pattaya, Thailand: Abstract S2-P9.
  - xxiv. "Effect of pH, Ionic Strength, and Poly(diallyldimethyl ammonium chloride) on Solubility of Barium-Arsenic Compounds", ORAL, Winya Dungkaew; Kenneth J. Haller; Adrian E. Flood; John F. Scamehorn, Royal Golden Jubilee PhD Congress VI, Pattaya, Thailand: Abstract S2A-O9.
- f. Attended the 31st Congress on Science and Technology of Thailand, held at SUT, Nakhon Ratchasima, 18-20 October 2005. (documents available: eight abstracts) Attending group members: Pushpakumara Herath, Winya Dungkaew, Saiphon Chanpaka, Ratchadaporn Puntharod, Kittipong Chainok, Sitthapagorn Srisobha, Amonrat Phetsopa, Samroeng Krachodnok, and Kadsada Sala
- xxv. "Environmental Health Risk for the Chronic Renal Failure in Sri Lanka", K. R. P. K. Herath; Oliver A. Ileperuma; H. A. Dharmagunawardhane; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract H0057, 18-20 October 2005.
  - xxvi. "Phase Precipitation and Solubilization Study of Barium-Arsenate Compounds", Winya Dungkaew; Kenneth J. Haller; Adrian E. Flood; John F. Scamehorn, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0070, 18-20 October 2005.
  - xxvii. "Synthesis of Difuctose Dianhydrides and Disorbose Dianhydrides by Acid Dehydration", Saiphon Chanpaka; Adrian E. Flood; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0125, 18-20 Oct 2005.
  - xxviii. "Structural Characterization of the Benzene Solvate of  $\text{NiCl}(\text{NO})(\text{PPh}_3)_2$ ", Ratchadaporn Puntharod; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0130, 18-20 October 2005.
  - xxix. "Organic-Inorganic Hybrid Supramolecular Materials: Synthesis and Characterization of Layer Structure Metal Vanadate Compounds", Kittipong Chainok; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0231, 18-20 October 2005.
  - xxx. "Hydrothermal Synthesis and Structural Characterization of an Organic-Inorganic Hybrid Vanadium Oxide Material", Sitthapagorn Srisobha; Kittipong Chainok; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0233, 18-20 October 2005.
  - xxxi. "Cocrystallization Studies of Caffeine", Amornrat Phetsopa; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0253, 18-20 October 2005.
  - xxxii. "Hydrothermal Synthesis and Characterization of a New Organically Templated Vanadium Arsenate", Samroeng Krachodnok; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0254, 18-20 October 2005.
  - xxxiii. "Cocrystallization Studies of Acetylsalicylic Acid (Aspirin) and Carboxylic Acids", Kadsada Sala; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract not printed in the abstract book, 18-20 October 2005.

III. Visited Mahidol University International College, Salaya Campus, Nakhon Phatom, in February and in May.

g. Discussed possible collaboration on graduate student projects with faculty.

IV. Visited King Mongkut's University of Science and Technology, Thonburi in February.

h. Discussed possible collaboration on graduate student projects with faculty.

i. Discussed potential workshop presentations in crystallography and use of the crystallographic databases with Dr. Kenji Okada.

V. Hosted Prof. Oliver Ileperuma, Department of Chemistry, University of Sri Lanka, Peradeniya Campus, Kandy, Sri Lanka to give seminars and lectures at SUT from 28 August to 3 September 2005.

- j. Prof. Ileperuma gave a four hour series on Bioinorganic Model Compounds.
- k. Prof. Ileperuma gave a six hour series on Solar Energy Conversion.
- l. Prof. Ileperuma and I held discussions on the possibilities for recruiting Sri Lankan students for graduate study at SUT.
- m. Prof. Ileperuma and I discussed possibilities for exchanging graduate students between our two research groups to enhance the graduate experience for all our students.
- n. We finalized arrangements for one of his graduate students (Mr. Pushpakumara Herath) to come to SUT for three months to work on an environmental chemistry project of mutual interest.
- o. Prof. Ileperuma engaged in discussion with my graduate students and research associates, and toured the National Synchrotron Research Center.
- p. Mr. Pushpakumara Herath, graduate student of Dr. Ileperuma, joined my research group from the middle of July until 22 October. Mr. Herath presented a poster at the STT31 meeting at SUT.

VI. Visited the Hanoi University of Technology in Hanoi as a guest of Dr. Pham Thanh Huyen from the Petrochemical and Catalysis Material Laboratory, Faculty of Chemical Technology from 21-30 September 2005.

- q. Dr. Huyen arranged several meetings for faculty and deans to present their research interests as well as the structure of programs, etc. at HUT to me.
- r. I gave seminars to three groups of undergraduate and graduate students and two groups of faculty to present basic information about SUT, more specific information about the Institute of Science and the School of Chemistry, and quite specific information about my own research group.
- s. I attended a dissertation presentation on bismuth clusters by one member of the Faculty of Chemical Technology.
- t. I toured laboratory facilities and met several of the support staff. Some of these scientists have M.Sc. degrees and could be potential chemistry students for SUT.
- u. Dr. Huyen and I discussed possibilities for exchanging graduate students between our two research groups to enhance the graduate experience for our students.
- v. Dr. Huyen and I worked on results and interpretation for collaborative research we are engaged in.
- w. I met Mr. Pham Gia Toan, an upper level undergraduate student in chemistry and talked to him about opportunities for graduate study in chemistry at SUT.
- x. I visited a group of Mathematics faculty for discussion. Some of them have been at SUT campus in the past. One remembered meeting me at SUT about eight years ago.

VII. Follow-up visit to the Hanoi University of Technology in Hanoi as a guest of Dr. Pham Thanh Huyen from the Petrochemical and Catalysis Material Laboratory, Faculty of Chemical Technology from 29 November to 4 December 2005.

- y. My primary activity was to attend the Regional Symposium on Chemical Engineering that was held in Hanoi from 30 November to 2 December 2005.
- z. I discussed results from collaborative research Dr. Huyen and I are engaged in.
- aa. I met Mr. Pham Gia Toan (see VI.w. above) again and talked.

VIII. Re-established contact with Dr. Bayden Wood at Monash University. He will be a coadvisor for Ms. Ratchadaporn Puntharod, and will host her in his laboratories at Monash University for approximately one year of her research time.

IX. Hosted Professor A. David Rae, RGJ coadvisor for Weenawan Somphon, three separate occasions for Ms. Weenawan's thesis defense and to work on papers. Prof. Rae talked with other group members. He is keen to work with Mr. Kittipong Chainok for about six months as part of his Australian government Endeavour Scholarship Grant to spend one year in Australia.

- ab. First visit: 15-26 March 2005.
- ac. Second visit: 10-14 April 2005.
- ad. Third visit: 11-14 September 2005.
- ae. Additionally, secured Prof. Rae as a Keynote Speaker for ANSCSE9, the Annual National Symposium on Computational Science and Engineering.
- af. Prof. Rae participated as a Speaker for the Crystallography Workshop at the Annual National Symposium on Computational Science and Engineering.
- ag. Prof. Rae worked with Mr. Kittipong and myself related to new features in his RAELS crystallographic refinement program.
- ah. Prof. Rae participated in lively discussions with my research group on scientific topics of interest.

X. Hosted Associate Professor Ian Williams, RGJ coadvisor for Samroeng Krachodnok, two separate occasions, first for Ms. Weenawan's thesis defense, then for proposal defenses by Mr. Kittipong Chainok and Ms. Samroeng Krachodnok. He will work with Ms. Samroeng Krachodnok as an RGJ coadvisor, and with Mr. Kittipong Chainok as an informal coadvisor in Hong Kong in 2006.

- ai First visit: 20-28 March 2005.
- aj Second visit: 8-10 December 2005.
- ak. Additionally, with his research group, contributed three papers to ANSCSE9, the Annual National Symposium on Computational Science and Engineering.
- al. Dr. Williams participated as a Speaker and Trainer for the Crystallography Workshop at the Annual National Symposium on Computational Science and Engineering.
- am. Dr. Williams, Mr. Kittipong, and I discussed proposed work Mr. Kittipong will carry out at Hong Kong University of Science and Technology in the summer of 2006.
- an. Dr. Williams, Ms. Samroeng, and I discussed proposed work Ms. Samroeng will carry out during her Ph.D. research work.
- ao. Dr. Williams participated in discussions with my research group on scientific topics of interest.
- ap. Dr. Williams and I discussed ongoing collaborative research projects, and worked on joint abstracts and manuscripts.
- aq. Three members of Dr. Williams' research group accompanied him and interacted with students and faculty at SUT and at ANSCSE9.

XI. Notes on some individual students recruited.

- ar. Mr. Pham Gia Toan, undergraduate student in chemistry at Hanoi University of Technology. Mr. Toan plans to study abroad. If he cannot go to the US or Europe, he is interested in SUT.
- as. Ms. Ratchadaporn Puntharod, (One Ajahn, One Project, 2004, VI.n.) M.Sc. degree in chemistry from Chiangmai University, is a faculty member at Meijo University. Ms. Ratchadaporn became a graduate student at SUT in Term 3/2547. has been accepted and will begin her studies at SUT in Term 3/2547.
- at. Ms. Panada Tansupo, (One Ajahn, One Project, 2004, VI.o.) M.Sc. in Chemistry from Khonkaen University. Still working and still considering graduate school.
- au. Mr. Ngo Xuan Dong, (One Ajahn, One Project, 2004, VI.r.) B.Sc. in Chemistry from Hanoi University of Technology (contact from activity I.b.xi above). Mr. Dong decided against coming to SUT (I believe because of concerns that there was not enough financial support for a foreign student).

- av. Mr. Sitthapagorn Srisobha, third year (B.Sc. in Chemistry) undergraduate research project student from Ratchaphat Ubon Ratchathani. He prepared an abstract and presented his work at STT31 at SUT. He wants to attend graduate school.
- aw. Ms. Wilaiporn Kidrob, B.Sc. in Chemistry from Meijo University. Ms. Wilaiporn was interested in Polymer Chemistry, but joined my group as a researcher for about three months. She decided to work a while before graduate school and did not apply to SUT in 2548.
- ax. Ms. Sa-ard Dangna, B.Sc. in Chemistry from Meijo University, seemed quite interested in pursuing an M.Sc. degree at SUT. She joined my group as a researcher for about six months, but frequently told me her mother thought she should get a job. She decided to become a high school science teacher (Loei) rather than go to graduate school and consequently, did not apply to SUT in 2548.
- ay. Ms. Amonrat Phetsopa B.Sc. in Chemistry from Meijo University. Wanted to go to Chula, but joined my group as a researcher for about six months first. She did not apply to SUT graduate school in 2548.
- az. Mr. Pushpakumara Herath, B.Sc. in Chemistry from the University of Sri Lanka, is finishing writing his M.Sc. thesis. He currently has a job, but is interested in further study sometime in the future. I will maintain contact with him and continue to try to attract him to SUT.

**XII. Attachments List (19 pages):**

List of Published Works – 2005 (1 page)

List of Conference Presentations –2005 (4 pages)

Eight abstracts from the Crystallography Minisymposium of the 9th Annual National Symposium on Computational Science and Engineering held at Mahidol University, Bangkok, 23-25 March 2005. (8 pages)

Six abstracts from the International Union of Crystallography Congress and General Assembly, IUCr2005, held at Florence, Italy, 23-31 August 2005. (6 pages)

(additional abstracts, conference proceedings, and papers are available if required)

**Published Works -- 2005**

- p1. X-Ray Structural Characterization of Disordered Epifriedelin-3-ol and Friedelin-3-one. Auphatham Phothikanith; Kenneth J. Haller, *Suranaree J. of Sci. and Tech.* **2005** 12(3) 211-222.
- p2. Noncovalent Interactions in a Three Component Supramolecular Structure. Kittipong Chainok; Kenneth J. Haller; Herman H.-Y. Sung; Ian D. Williams, *Acta Cryst. Sect. A* **2005** 61, C306.
- p3. Crystal Structure of  $[\text{Sn}(\text{Bu})_3(\text{O}=\text{PPh}_3)_2][\text{BPh}_4]$ . Nongnaphat Khosavithikul; Kenneth J. Haller; Seik Weng Ng, *Acta Cryst. Sect. A* **2005** 61, C307.
- p4. An Order-Disorder Phase Transition in  $[\text{Ag}(\text{bipy})\text{NO}_3]_n$ . Weenawan Somphon; Kenneth J. Haller; A. David Rae, *Acta Cryst. Sect. A* **2005** 61, C321.
- p5. A New Organically Templated Vanadium Arsenate. Samroeng Krachodnok; Kenneth J. Haller, *Acta Cryst. Sect. A* **2005** 61, C306.
- p6. Preparation and Characterization of  $\text{NiCl}(\text{NO})(\text{PPh}_3)_2$ . Ratchadaporn Puntharod; Kenneth J. Haller, *Acta Cryst. Sect. A* **2005** 61, C306.
- p7. Structure of 19-Hydroxyneohopane. Kenneth J. Haller, *Acta Cryst. Sect. A* **2005** 61, C281.
- p8. Packing and Polytypism in 1,10-Phenanthroline-1-ium (2-Carboxyethyl)(2-carboxylatoethyl)dichlorostannate(IV). Weenawan Somphon; Kenneth J. Haller; A. David Rae; Seik Weng Ng, *Acta Cryst. Sect. B* **2005**, accepted for publication.
- p9. The Commensurately Modulated Bis(aquachlorotriphenyltin 1,10-Phenanthroline) Crystal Structure. A. David Rae; Kenneth J. Haller; Ng Seik Weng, *J. Sci. Tech. Tropics* **2005** 1, accepted for publication.
- p10. The Order-Disorder Transition at 150 K of Polymeric  $\text{Ag}(\text{bipy})\text{NO}_3$ , Weenawan Somphon; Kenneth J. Haller; A. David Rae, *Acta Cryst. Sect. B* **2005**, submitted for publication.



**Conference Presentations -- 2005**

- c1. "Analyzing Concerted Noncovalent Interactions in Structures Dominated by Phenyl Rings", ORAL, Nongnaphat Khosavithikul; Angkana Kiatpichitpong; Kenneth J. Haller, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB1-4, 23-25 March 2005.
- c2. "Refinement of a Commensurate Displacive Modulated Structure", ORAL, Kenneth J. Haller; Weenawan Somphon; A. David Rae; Ian D. Williams, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB1-5, 23-25 March 2005.
- c3. "Unraveling the Solvent/Cation Disorder in the Structure of a Vanadium Borate Cluster,  $[V_6B_{20}O_{50}H_8]^{n-}$ ", ORAL, Aungkana Chatkon; Kenneth J. Haller; Ian D. Williams, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB2-1, 23-25 March 2005.
- c4. "Supramolecular Structure Analysis: Detection of a Novel Carbonyl-Carbonyl Interaction in the Structure of Pyridine-3,5-Dicarboxylic Acid", ORAL, Samroeng Krachodnok; Kenneth J. Haller, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB2-6, 23-25 March 2005.
- c5 "Supramolecular Structure Analysis: Trispicolinato Cobalt(III) Monohydrate,  $[Co(C_6H_4O_2N)_3] \cdot H_2O$ ", ORAL, Kittipong Chainok; Kenneth J. Haller, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB2-7, 23-25 March 2005.
- c6 "Refining a Multiply Twinned Modulated Structure: Triethyl Phosphine Sulfide", ORAL, Weenawan Somphon; Kenneth J. Haller; A. David Rae, ANSCSE9, 9th Annual National Symposium on Computational Science and Engineering, Bangkok, Thailand: Abstract CCB5-3, 23-25 March 2005.
- c7. "Introduction to Twinning", ORAL, Kenneth J. Haller, ANSCSE9 Workshop on Crystallography, 9th Annual National Symposium on Computational Science and Engineering, Abstract CCB-W-2, Bangkok, Thailand, 25 March 2005.
- c8. "Introduction to the Cambridge Structural Database", ORAL, Kenneth J. Haller, ANSCSE9 Workshop on Crystallography, 9th Annual National Symposium on Computational Science and Engineering, Abstract CCB-W-3, Bangkok, Thailand, 25 March 2005.
- c9. "Organically Templated Transition Metal Vanadium Arsenates", Samroeng Krachodnok; Kenneth J. Haller, Royal Golden Jubilee PhD Congress VI, Pattaya, Thailand: Abstract S2-P9, 28-30 April 2005.
- c10. "Effect of pH, Ionic Strength, and Poly(diallyldimethyl ammonium chloride) on Solubility of Barium-Arsenic Compounds", ORAL, Winya Dungkaew; Kenneth J. Haller; Adrian E. Flood; John F. Scamehorn, Royal Golden Jubilee PhD Congress VI, Pattaya, Thailand: Abstract S2A-O9, 28-30 April 2005.

- c11. "Synthesis and Characterization of Organic-Inorganic Hybrid Cobalt Vanadates:  $[\text{Co}(\text{Im})_4\text{V}_4\text{O}_{12}]$ , Im = imidazole", Kittipong Chainok; Kenneth J. Haller, Nanotec, Nano Material Fabrication Technologies, Chiang Mai, Thailand: Abstract xx-xx, 28-29 July, 2005.
- c12. "Noncovalent Interactions in a Three Component Supramolecular Structure", Kittipong Chainok; Kenneth J. Haller; Herman H.-Y. Sung; Ian D. Williams, IUCr2005, XX International Union of Crystallography Congress and General Assembly, Florence, Italy: Abstract P07.04.7, 23-31 August 2005.
- c13. "A New Organically Templated Vanadium Arsenate", Samroeng Krachodnok; Kenneth J. Haller, IUCr2005, XX International Union of Crystallography Congress and General Assembly, Florence, Italy: Abstract P07.04.6, 23-31 August 2005.
- c14. "An Order-Disorder Phase Transition in  $[\text{Ag}(\text{bipy})\text{NO}_3]_n$ ", Weenawan Somphon; Kenneth J. Haller; A. David Rae, IUCr2005, XX International Union of Crystallography Congress and General Assembly, Florence, Italy: Abstract P08.06.12, 23-31 August 2005. *This poster won the IUCr student research presentation prize and the Oxford Cryosystems Use of Low Temperature student award.*
- c15. "Preparation and Characterization of  $\text{NiCl}(\text{NO})(\text{PPh}_3)_2$ ", Ratchadaporn Puntharod; Kenneth J. Haller, IUCr2005, XX International Union of Crystallography Congress and General Assembly, Florence, Italy: Abstract P07.04.8, 23-31 August 2005.
- c16. "Crystal Structure of  $[\text{Sn}(\text{Bu})_3(\text{O}=\text{PPh}_3)_2][\text{BPh}_4]$ ", Nongnaphat Khosavithikul; Kenneth J. Haller; Seik Weng Ng, IUCr2005, XX International Union of Crystallography Congress and General Assembly, Florence, Italy: Abstract P07.04.9, 23-31 August 2005.
- c17. "Structure of 19-Hydroxyneohopane", Kenneth J. Haller, IUCr2005, XX International Union of Crystallography Congress and General Assembly, Florence, Italy: Abstract P06.04.2, 23-31 August 2005.
- c18. "Environmental Health Risk for the Chronic Renal Failure in Sri Lanka", K. R. P. K. Herath; Oliver A. Ileperuma; H. A. Dharmagunawardhane; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract H0057, 18-20 October 2005.
- c19. "Phase Precipitation and Solubilization Study of Barium-Arsenate Compounds", Winya Dungkaew; Kenneth J. Haller; Adrian E. Flood; John F. Scamehorn, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0070, 18-20 October 2005.
- c20. "Synthesis of Difuctose Dianhydrides and Disorbose Dianhydrides by Acid Dehydration", Saiphon Chanpaka; Adrian E. Flood; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0125, 18-20 October 2005.
- c21. "Structural Characterization of the Benzene Solvate of  $\text{NiCl}(\text{NO})(\text{PPh}_3)_2$ ", Ratchadaporn Puntharod; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0130, 18-20 October 2005.

- c22. "Organic-Inorganic Hybrid Supramolecular Materials: Synthesis and Characterization of Layer Structure Metal Vanadate Compounds", Kittipong Chainok; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0231, 18-20 October 2005.
- c23. "Hydrothermal Synthesis and Structural Characterization of an Organic-Inorganic Hybrid Vanadium Oxide Material", Sitthapagorn Srisobha; Kittipong Chainok; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0233, 18-20 October 2005.
- c24. "Cocrystallization Studies of Caffeine", Amornrat Phetsopa; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0253, 18-20 October 2005.
- c25. "Hydrothermal Synthesis and Characterization of a New Organically Templated Vanadium Arsenate", Samroeng Krachodnok; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract C0254, 18-20 October 2005.
- c26. "Cocrystallization Studies of Acetylsalicylic Acid (Aspirin) and Carboxylic Acids", Kadsada Sala; Kenneth J. Haller, STT2005, 31st Congress on Science and Technology of Thailand, Nakhon Ratchasima, Thailand: Abstract mistakenly not printed in the abstract book, 18-20 October 2005.

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- c27. "Barium-Arsenate Phase Precipitation and Solubilization Study: Effect of pH, Ionic Strength, and PDADMAC", Winya Dungkeaw; Kenneth J. Haller; Adrian E. Flood; John F. Scamehorn, International Conference Hazardous Waste Management for a Sustainable Future, NRC-EHWM, Chulalongkorn University, Bangkok, Thailand, 10-12 January 2006.
- c28 "Carbonyl-Ether Supramolecular Building Block in 25,26-Oxidofriedel-1,3-dione", INVITED ORAL, Kenneth J. Haller; Auphatham Phothikanith, session 10.02, *Natural Products and Drugs*, American Crystallographic Association Annual Meeting, Honolulu, Hawaii, USA, Abstract 10.02 W0499, 22-27 July 2006, to be presented.
- c29 "Variable Low-Temperature Data Collection and Hierarchical Refinement to Study an Order-Disorder Phase Transition", INVITED ORAL, Weenawan Somphon; Kenneth J. Haller; A. David Rae, session 10.01, *Applications of Crystal Growth and Low Temperature Techniques*, American Crystallographic Association Annual Meeting, Honolulu, Hawaii, USA, Abstract 10.01 W0588, 22-27 July 2006, to be presented.
- c30 "Crystal Structure of the 2:1 Adduct of 1,2-Benzenediol and Hexamethylenetetramine", Kadsada Sala; Kenneth J. Haller, American Crystallographic Association Annual Meeting, Honolulu, Hawaii, USA, Abstract 10.03 W0335, 22-27 July 2006, accepted for presentation.
- c31 "Packing Interactions of 2,3,7,8,12,13,17,18-Octaethylporphyrinato(picrato)iron (III)", Rachadaporn Puntharod; Kenneth J. Haller, American Crystallographic Association Annual Meeting, Honolulu, Hawaii, USA, Abstract 10.03 W0382, 22-27 July 2006, accepted for presentation.
- c32 "Hydrothermal Synthesis and Structural Characterization of an Open-Framework Arsenic Vanadate,  $As_2V_{10}O_{26}$ ", Samroeng Krachodnok; Kenneth J. Haller, American Crystallographic Association Annual Meeting, Honolulu, Hawaii, USA, Abstract 10.03 W0430, 22-27 July 2006, to be presented.

c33 "Synthesis and Characterization of Novel 2D Organic-Inorganic Hybrid Cobalt and Nickel Vanadates", Kittipong Chainok; Kenneth J. Haller; Herman H.-Y. Sung; Ian D. Williams, American Crystallographic Association Annual Meeting, Honolulu, Hawaii, USA, Abstract 13.12 W0327, 22-27 July 2006, accepted for presentation.

## Refinement of a Commensurate Displacive Modulated Structure

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### Abstract

Solvothermal reaction of copper sulfate, terephthalic acid, and 4,4'-bipyridine gave yellow crystals;  $[\text{Cu}(\text{C}_{10}\text{H}_8\text{N}_2)]_4(\text{SO}_4)_2 \cdot 2(\text{C}_8\text{H}_6\text{O}_4)$ , monoclinic  $C2/c$ ,  $Z = 8$ ,  $a = 19.597(4)$ ,  $b = 34.771(3)$ ,  $c = 16.274(3)$  Å,  $\beta = 107.52(3)^\circ$ ,  $T = 295$  K. Initial positions for the nonhydrogen atoms were obtained easily from direct methods, and refined to give an average structure with parameters that did not make chemical sense.

The structure can be understood as a commensurate displacive modulation of an idealized  $Z = 2$  parent structure (also  $C2/c$ ) with  $\mathbf{a}_p = \mathbf{a}/2$ ,  $\mathbf{b}_p = \mathbf{b}/2$ ,  $\mathbf{c}_p = \mathbf{c}$ . In the parent, polymeric  $[-\text{Cu}^{\text{I}}\text{-bipy-Cu}^{\text{I}}\text{-bipy-}]$  chains are propagated by an  $n$  glide,  $1/2+x, 1/2-y, -1/2-z$ , and are cross linked (Cu-O-S-O-Cu) by the sulfate groups which lie on 2-fold axes. The terephthalic acids lie on centers of inversion and hydrogen bond to the free oxygen atoms of the bridging sulfate ions. The Cu atom and the 4,4'-bipyridine are at general positions; the apparent local 2-fold through the Cu atom and the apparent inversion center in the 4,4'-bipyridine are not symmetry elements of the parent  $C2/c$  structure. The parent  $n$  glide can not remain a symmetry element of the modulated structure.

The structure was refined using RAELS [1], employing a (3+1) dimensional approach with modulation vector  $(\mathbf{a}_p^* + \mathbf{b}_p^*)/2$ , suggesting the possibility of a twinned triclinic crystal. Observations of weak reflections with  $h$  even,  $k$  even,  $h+k=4n+2$ , preclude this option. A (3+2) dimensional approach with modulation vectors  $\mathbf{a}_p^*/2$ ,  $\mathbf{b}_p^*/2$  allows for space group  $C2/c$  with four options for the selection of the quarter of the parent structure symmetry elements to be retained. Reverting to the standard setting this allows four origin options for the asymmetric unit but origins  $1/4, 1/4, 0$  apart do not correspond to alternative origins for the asymmetric unit of the parent structure. In the correct model, the terephthalic acids no longer sit on inversion centers. Some residual disorder about a 2-fold axis remains but an ordered structure in a lower symmetry space group was shown to be inappropriate.

[1] A. David Rae (2000). RAELS00, Australian National University, Canberra, Australia.

## Unraveling the Solvent/Cation Disorder in the Structure of a Vanadium Borate Cluster, $[V_6B_{20}O^{50}H^8]^{n-}$

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### Abstract

Hydrothermal reaction of vanadium pentoxide, boric acid, ethylenediamine, and hydrofluoric acid (1:10:3:3) at 180° C for 3 days gave the title anionic cluster which has been characterized by single crystal X-ray crystallography. Charge balance is maintained by conversion of amine groups to ammonium groups on associated ethylenediamine molecules in the monoclinic crystal lattice (space group  $C2/c$ ). The cluster, which consists of a central  $V_6O_{18}$  band of six square-pyramidal vanadyl groups capped top and bottom through the basal oxygen atoms by raft-like  $B_{10}O_{16}H_4$  polyborate ligands, was easily located by conventional direct methods using SIR97. Electron density difference maps based on this preliminary model showed a plethora of peaks in the intercluster region. Some peaks were rational for the organic amine component, but several regions were not recognizable due to disorder of cations and solvent water molecules. Successful solution of this structure was only possible through an iterative process of examination/interpretation of electron density maps followed by model building and constrained/restrained refinement. The resulting three dimensional supramolecular array of cations, anions, and solvent water molecules, interconnected by extensive hydrogen bonding will be described.

## Refining a Multiply Twinned Modulated Structure: Triethyl Phosphine Sulfide

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### Abstract

The published structure [1] of  $S=P(C_2H_5)_3$  is described as having a 1:1 disorder in space group  $P6_3mc$  with  $Z = 2$  with  $P3_1c$  describing a proposed ordered structure rather than  $P6_3$ . Synthetic precession photographs obtained from data collection on a Nonius KappaCCD diffractometer clearly indicate a doubling of the  $a$  and  $b$  axes. The parent  $P6_3mc$  structure can also be described as a  $C$ -centred orthorhombic cell of  $Ccm2_1$  symmetry with  $a_p' = 2a_p + b_p$ ,  $b_p' = b_p$ ,  $c_p' = c_p$  with four molecules per cell. The dominant modulation creating the extra reflections is a combination of displacements parallel to  $c$  and not an ordering of the ethyl ligands. This mode lowers the  $Ccm2_1$  symmetry to  $Pnm2_1$  with three fold twinning restoring hexagonal diffraction symmetry. Further modulations of the parent structure seem necessary, and partial ordering in a mode of  $Pna2_1$  symmetry is a reasonable option. This suggests a probable six fold twinning since local symmetry of an average structure would then be lowered to  $Pn$ . Absences in the diffraction pattern suggest that the  $n$  glide is a dominant feature with adjacent layers displacing in opposite directions along  $c$ .

[1] M. van Meerssche & A. Leonard, (1959) Acta Cryst. **12** 1053-1054)

## Supramolecular Structure Analysis: Detection of a Novel Carbonyl-Carbonyl Interaction in the Structure of Pyridine-3,5-Dicarboxylic Acid

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### Abstract

A previous report [1] of the structure of pyridine-3,5-dicarboxylic acid, based on a twinned crystal, details the network of strong hydrogen bonds creating a two-dimensional layer structure. Untwinned (single) crystals were obtained by hydrothermal crystallization and the structure redetermined; monoclinic space group  $P2_1/c$ ,  $a = 9.6756(4)$ ,  $b = 11.1166(7)$ ,  $c = 6.5808(4)$  Å,  $\beta = 107.766(4)^\circ$ ,  $V = 674.07(7)$  Å<sup>3</sup>,  $Z = 4$ ,  $T = 298$  K.

The molecules are interconnected in two-dimensional layers (parallel to the  $ab$  plane) through O–H $\cdots$ N hydrogen bond interactions between adjacent molecules related by a  $2_1$  screw axis, and O–H $\cdots$ O hydrogen bond interactions between adjacent molecules related by the other independent  $2_1$  screw axis. Thus, the possibility of the ubiquitous inversion related carboxylic acid dimer structure is precluded.

Further analysis of the crystal structure reveals previously unreported supramolecular features. The layer structure is reinforced by weak C–H $\cdots$ O hydrogen bond interactions involving all three aromatic hydrogen atoms. Layers related by the  $c$  glide and inversion centers are held together by face-to-face,  $\pi$ – $\pi$  interactions. It is also noted that the two, nominally chemically equivalent, carboxylic acid groups have considerably different bonding parameters. An antiparallel noncovalent interaction between inversion related pairs of carbonyl groups in adjacent layers is the likely cause of the differences. The  $>C(\delta^+) \cdots O(\delta^-)$  interaction distance is 3.015 Å, considerably shorter than the sum of van der Waals radii, and near the optimum value recently found [2] for this type of interaction.

[1] F. Takusagawa, K. Hirotsu, A. Shimada (1973) Bull. Chem. Soc. Jpn. **46** 2292-2299.

[2] F. H. Allen, C. A. Baalham, J. P. M. Lommerse, and P. R. Raithby (1998) Acta Cryst. **B54** 320-329.



## Analyzing Concerted Noncovalent Interactions in Structures Dominated by Phenyl Rings

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### Abstract

With the current interest in nanotechnology, considerable effort is being focused on analysis of supramolecular structure, and the building blocks of extended solid state structures. One such building block for molecular compounds is the multiple phenyl-phenyl edge-to-face (*ef*) C–H $\cdots$  $\pi$  attractive noncovalent interactions of the concerted sextuple phenyl embrace (6PE), which give a sum of interaction energy sufficient to make it a dominant supramolecular motif for crystals of complexes containing triphenylphosphine or similar ligands. [1]. Analysis of these features is often requires a combination of the evaluation of derived geometric values, and examination of graphical images of local and extended portions of the crystal structure.

This work reports the analysis of the supramolecular structures of a series of three complexes, the four coordinate Ni(X)(NO)(P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>)<sub>2</sub> complexes where X = NCS<sup>-</sup>, N<sub>3</sub><sup>-</sup>, and Cl<sup>-</sup>. Although the molecular complexes are quite closely related, their supramolecular structures are different. The primary extended interactions in the isothiocyanato complex are chains of the expected 6PE between adjacent triphenylphosphine ligands. Adjacent chains are connected together into a three dimensional network by additional weaker concerted interactions. The chloro complex contains distorted 6PE chains. However, it crystallizes with a benzene molecule of solvation, which is able to accommodate considerably more C–H $\cdots$  $\pi$  interactions, and therefore becomes the major link between surrounding molecules. While the azido complex does not contain 6PE, it does contain one-dimensional chains of highly concerted noncovalent interactions involving the azido group as well as phenyl rings. Thus, as the strongest hydrogen bond acceptor, the azido ligand becomes perhaps the most important determiner of the supramolecular structure. Phenyl-phenyl interactions are still abundant and still important in determining finer details of the crystal structure. The supramolecular structures of these three closely related molecular complexes will be presented.

[1] Ian Dance and Marcia Scudder (1995) *J. Chem. Soc., Chem. Commun.*, 1039-1040

**Supramolecular Structure Analysis:  
Trispicolinato Cobalt(III) Monohydrate, [Co(C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>N)<sub>3</sub>]·H<sub>2</sub>O**

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**Abstract**

The single crystal X-ray structure was solved by direct methods and refined with the conventional SHELX crystallographic program system, using intensity data collected on a Bruker Nonius KappaCCD autodiffractometer equipped with a molybdenum X-ray source. The molecular structure of the complex exhibits distorted octahedral coordination geometry about the cobalt atom with three *mer* pyridine nitrogen atoms and three *mer* carboxylate oxygen atoms. Comparison of intramolecular distances and angles in the molecular structure with values derived from structure correlation [1] show them to be completely normal.

Analysis of the intermolecular interactions is more complicated. Standard program packages, such as SHELX, include algorithms to analyze strong hydrogen bond interactions. However, when the packing is determined by weak noncovalent interactions (including weak hydrogen bonds such as C–H···O or C–H··· $\pi$ ), or by a combination of strong hydrogen bonds and weak noncovalent interactions, routine application of the standard packages fails to give complete results. The results of the analysis of the analysis of the supramolecular structure will be presented. The molecules pack as a series of offset phenyl-phenyl face-to-face ( $\pi$ ··· $\pi$ ) interactions between ring type 1, propagated by alternate 2-fold sites and inversion sites in the *c* direction giving an interdigitated double comb motif. The water molecules bridge the rings of type 1 to rings of type 2 on adjacent  $\pi$ ··· $\pi$  stacks generating a two-dimensional network. The third dimension is achieved by phenyl ring 1 to ring 3 edge-to-face interactions between adjacent  $\pi$ ··· $\pi$  stacks.

[1] A. G. Orpen, L. Brammer, F. H. Allen, O. Kennard, D. G. Watson, R. Taylor (1994). Typical Interatomic Distances in Organic Compounds and Organometallic Compounds and Coordination Complexes of the *d*- and *f*-block Metals. in Structure Correlation, H.-B. Burgi & J. D. Dunitz, eds, VCH, Table A.2, pp 785-848.

## **Introduction to Twinning**

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### **Abstract**

A twinned crystal is an aggregate made up of domains of the same species that are joined together in some definite mutual orientation. The diffraction patterns derived from the different domains are rotated, reflected, or inverted with respect to each other, depending on the specific relationship between the domains. The relationship is expressed as a symmetry operation, called the twin law. The patterns are weighted according to the relative amount of each domain that is present in the aggregate. Thus, the diffraction patterns measured during data collection are superpositions of multiple patterns resulting from the different orientations and with the different relative intensities for each of the patterns. Reflections from different domains in the aggregate may overlap, leading to the broad classes of twins, 1) those for which all reflections overlap, and 2) those for which only certain zones of reflections overlap. The first class generally occur when a crystal lattice belongs to a higher point group than the crystal structure itself, and the second class often occurs when the twin law is a symmetry operation belonging to a higher symmetry supercell.

General terms related to twinning and terms relating to twinning operations, reference frames, and classes of twins will be given. The relationship between twins and diffraction patterns will be illustrated

## Introduction to the Cambridge Structural Database

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### Abstract

The Cambridge Crystallographic Data Center, CCDC, grew out of a small group that was set up by J. D. Bernal and Olga Kennard at Birkbeck College in London in 1959 to collect crystal structures of organic compounds and compounds containing organic parts. The group moved to Cambridge in 1962. In 1965 the CCDC was formally established with a grant from the British Office for Scientific and Technical Information, and is thus 40 years old this year.

By 1970 they were beginning to validate structure and enter them into a database, which has become the Cambridge Structural Database, CSD, we know today. The initial effort included some 5000 structures. The resulting Version 1 of the CSD was released to academics in the UK, USA, Japan, and Italy in the mid 1970s. As with many computational related aspects of crystallography, the database was among the first of the electronic numerical information databases to be established. It incorporated an earlier electronic bibliographic database that had already been published several years as the book series, *Molecular Structures and Dimensions* (also among the first—one of the earliest handbooks to be typeset directly by computer). It was also realized that to increase the usefulness of the database, scientists needed facile ways to search 2D and 3D substructures, and connectivity files with associated search software were created. These three components, bibliographic information, the validated crystallographic data, and 2D and 3D substructure connectivity, were initially provided as three separated database files, and have subsequently been combined to form what we know today as the Cambridge Structural Database.

The early work with structural correlation by Hans-Beat Bürgi and Jack Dunitz inspired use of the CSD for fundamental research beginning in the late 1970s. One of the early well-known results was the 1982 Taylor and Kennard publication which established the weak C–H···O interaction as truly a hydrogen bond (*J. Am. Chem. Soc.* **1982** 104, 5063-5070). Currently, the CCDC website lists about 1200 references with substantial portions of the results derived from the CSD by what is often referred to as 'data mining'. In the late 1980s, the first tabulations of mean bond lengths, another valuable tool derived from the database appeared (*J. Chem. Soc. Perkin Trans.* **1987** S1-S9; *J. Chem. Soc. Dalton Trans.* **1987** S1-S83).

Version 5.26, released 1 January 2005 contained 335,276 structures. About 29,000 were added in 2004, and the CCDC estimates another 33,000 will be added in 2005. If current trends continue the database will reach half a million entries during 2009. Another group of structures is also being held at the CCDC, those being direct deposits of Private Communications. Some of these are for papers in the publication process for the 84 journals that now require electronic submission of the crystallographic information to the CCDC for validation as the first step in the referee process, and a growing number are structures that the author prefers not be made public. Although these communications are not included in the CSD, efforts are underway to be able to use them in creating derivative information such as the bond length tabulations.

### Structure of 19-Hydroxyneohopane

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Several fused multicyclic natural product ring systems, especially those that are saturated or nearly saturated, are poorly represented in the Cambridge Structural Database of crystallographic determinations of organic compounds.

19-hydroxyneohopane, C<sub>30</sub>H<sub>48</sub>O, is one such compound consisting of a five fused ring system (rings 1 to 4 containing six carbons and ring 5 containing five carbons) with two double bonds trans across the 2-3 ring junction. The compound was obtained from the rhizome of *Davallia solida* Sw and crystallizes in the monoclinic space group, *P*2<sub>1</sub>, with two molecules in a cell of dimensions: *a* = 12.587(3), *b* = 7.558(3), *c* = 13.620(3) Å, and  $\beta$  = 102.68(3)° at *T* = 113(2) K.

Crystal Data: C<sub>30</sub>H<sub>48</sub>O, *MW* = 424.68, clear colorless plate crystal, 0.50 x 0.50 x 0.02 mm, monoclinic, *P*2<sub>1</sub>, *a* = 12.587(3), *b* = 7.558(3), *c* = 13.620(3) Å,  $\beta$  = 102.68(3)°, *V* = 1264.10, *Z* = 2 *T* = 113(2) K, *d*<sub>calc</sub> = 1.116 Mg m<sup>-3</sup>,  $\mu$  = 0.48 mm<sup>-1</sup>, CuK $\alpha$  radiation, *F*(000) = 472.,  $\sin\theta/\lambda_{\max}$  = 0.545 Å<sup>-1</sup>, *R*<sub>int</sub> = 0.0688, 3876 unique data, 3404 observed *F*<sub>o</sub> > 4 $\sigma$ (*F*<sub>o</sub>), *R*<sub>1</sub> = 0.0737, *goof* = 1.126.

**Keywords:** crystal structure, natural product, hopane

Abstract P08.06.12 presented at the International Union of Crystallography Congress and General Assembly, IUCr2005, held at Florence, Italy, 23-31 August 2005. This poster won the IUCr student research presentation prize and the Oxford Cryosystems Use of Low Temperature student award.

### An Order-Disorder Phase Transition in $[\text{Ag}(\text{bipy})\text{NO}_3]_n$

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The 160-296 K *Fddd* form of  $\text{Ag}(\text{bipy})\text{NO}_3$  contains a disordered  $\text{NO}_3$  ion and shows previously unreported planes of diffuse scattering indexed as  $3k\pi l=4n$ ,  $a$  12.8424(2),  $b$  9.9429(1),  $c$  34.4621(4) Å. Synthetic precession photographs show *Fddd* symmetry at  $\geq 160$  K but a loss of systematic absences indicated *F12/d1* (i.e. *C2/c*) at  $\leq 150$  K. The 100 K structure, *C2/c*,  $a$  12.751(1),  $b$  9.860(1),  $c$  18.379(2) Å,  $V$  109.98(1) Å<sup>3</sup> has twin components related by a rotation around  $c^*$ . The cell for *F12/d1* has  $a'=a$ ,  $b'=b$ ,  $c'$  34.547(2) Å,  $V'$  89.68(1) Å<sup>3</sup>. Refinement gave a 3:1 twin with no disorder and  $R(F)=0.022$  for 2294  $F_{obs}$ . Along a chain, alternate Ag atoms,  $(3b\pi c)/4$  apart, are displaced 0.221(1) Å in opposite directions perpendicular to the chain. Chains are cross linked by Ag–Ag contacts of 2.958(1) Å and Ag–O contacts of 2.749(2) and 2.747(2) Å. The chains zig-zag so that Ag atoms avoid closer contact with the  $\text{NO}_3$ . The 200 K structure, *Fddd*,  $a$  12.823(1),  $b$  9.937(1),  $c$  34.450(1) Å was refined as a 1:1 disorder of all atoms initiated by disordering the 100 K structure. Constrained refinement gave  $R(F)=0.028$  for 1004  $F_{obs}$ . The Ag displacements reduced to 0.123(3) Å. The alternative nitrate orientation gave three Ag–O contact distances indicating an intermediate step for a change of local ordering. The diffuse scattering indicates mistakes in  $\text{NO}_3$  positions cause a localized straightening of the adjacent chains that moving substantial amounts of these chains along their lengths. In contrast, the actual position of a  $\text{NO}_3$  only affects the closest Ag atoms.

**Keywords:** polymorphic structure, phase transition, order-disorder transition

### **A New Organically Templated Vanadium Arsenate.**

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Materials of open-framework and microporous structures are extensively studied because of their potential applications in catalysis, ionic conductivity, ion-exchange, and magnetic devices. Here we report a compound synthesized under hydrothermal conditions as part of a study of organically templated vanadium arsenates.  $V_2O_5$ ,  $Na_2HAsO_4 \cdot 7H_2O$ ,  $H_2N(CH_2)_3NH_2$  and  $H_2O$  in the molar ratio of 2:1:2.4:556 were heated at 180 °C for 3 days to give black crystals.

The product was characterized by X-ray diffraction, SEM/EDX, FTIR, and TGA. The EDX spectrum indicates presence of vanadium and arsenic in a 3:1 ratio. The IR spectrum has a strong peak at 999  $cm^{-1}$  that can be assigned to the  $\nu(V=O)$  vibration, strong bands at 973, 800, 634 and 567  $cm^{-1}$  characteristic of  $\nu(M-O-M)$  ( $M = V$  or  $As$ ) and  $\nu(As-O)$  [1], and bands in 1623-1383  $cm^{-1}$  region are due to the  $NH_2$  and  $CH_2$  bending of 1,3-dap. The broad band at 3438  $cm^{-1}$  can be attributed to O-H stretching.

[1] Beneš, L., Melánová, K., Zima, V., Trchová, M., Uhlřová, E., Matějka, P., *Eur. J. Inorg. Chem.*, 2000, 895-900.

**Keywords:** hydrothermal synthesis, vanadium arsenates, organic template

### **Noncovalent Interactions in a Three Component Supramolecular Structure.**

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Preparation of  $[(\text{ImH}_2)^+]_2[\text{Co}(\text{H}_2\text{O})_6]^{2+}[\text{Co}(\text{TMA})_2(\text{H}_2\text{O})_4]^{4-}$  by hydrothermal synthesis, and characterization by single crystal X-ray diffraction is reported. There are distinct cationic and anionic cobalt complex ions. The coordination environment of the cobalt atom in the cationic complex contains essentially octahedral, water molecules, while the coordination environment of the cobalt atom in the anionic complex, is distorted octahedral with four water molecules and two trans trianionic trimesate ligands. Two imidazolium monocations complete the structure and provide charge balance. The three moieties interact in the lattice through noncovalent attractions. The carboxylate groups are good hydrogen bond acceptors, while the solvate water molecules and imidazolium cations are good hydrogen bond donors. Thus, it is not surprising to find strong hydrogen bonds between the imidazolium N-H groups and the carboxylate oxygen atoms from the trimesate ligand. The N-H $\cdots$ O hydrogen bonds are in the range 2.693-2.777 Å, and the O-H $\cdots$ O hydrogen bonds are in the range 2.666-2.948 Å. The two imidazolium cations exhibit a pair of N-H $\cdots$  $\pi$  hydrogen bonds ( $\pi$ - $\pi$  stacking) across an inversion center, and additional  $\pi$ - $\pi$  interactions to the trimesate ligands.

Crystal Data:  $P-1$   $a = 9.1175(9)$ ,  $b = 9.3507(9)$ ,  $c = 10.6299(11)$  Å,  $\alpha = 79.224(2)$ ,  $\beta = 87.448(2)$ ,  $\gamma = 71.995(2)^\circ$ ,  $V = 845.59(15)$  Å<sup>3</sup>;  $Z = 1$ ;  $T = 298(2)$  K;  $R_I = 0.042$ , 2688 observed data.

**Keywords: noncovalent bonding, supramolecular structures**



### **Preparation and Characterization of NiCl(NO)(PPh<sub>3</sub>)<sub>2</sub>.**

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Previous work [1] on the title compound reports an intractable disorder problem, the complex was prepared again following the method of Feltham [2], and purple crystals prepared by vapor diffusion of hexane into a benzene solution. The compound crystallizes in a C-centered monoclinic space group in a cell of dimensions:  $a = 17.399(3)$ ,  $b = 13.136(3)$ ,  $c = 16.954(3)$  Å,  $\beta = 104.74(1)^\circ$ ,  $Z = 4$  and  $T = 296$  K.

FTIR spectral absorptions attributed to aromatic  $\nu(\text{C-H})$  at  $3050\text{ cm}^{-1}$ ,  $\nu(\text{N-O})$  at  $1716\text{ cm}^{-1}$ , aromatic  $\nu(\text{C-C})$  at  $1434\text{ cm}^{-1}$ , and bands for CH in- and out-of-plane bending at  $1095\text{ cm}^{-1}$  and  $693\text{ cm}^{-1}$ , respectively. The supramolecular structure contains the expected [3] six-fold phenyl embrace, 6PE, chains linking nickel bis(phosphine) moieties into one-dimensional zigzag chains. However, the lattice also contains a benzene solvate. The benzene molecule is able to form considerably more C-H $\cdots\pi$  interactions than a phenyl ring and becomes the major link, interconnecting three of the 6PE chains via ten C-H $\cdots\pi$  interactions centered about the benzene of crystallization. Additional weak noncovalent interactions supplement these major interactions to form an extensive three-dimensional crystal structure.

[1] Haller K.J., Enemark J.H., *Inorg. Chem.*, 1978, **17**(12), 3552.

[2] Feltham R.D., *Inorg. Chem.*, 1964, **3**(1), 116.

[3] Dance I., Scudder M., *J. Chem. Soc., Chem. Comm.*, 1995, **10**, 1039.

**Keywords: supramolecular structure, nitric oxide complexes, cooperative phenomena**

### Crystal Structure of $[\text{Sn}(\text{Bu})_3(\text{O}=\text{PPh}_3)_2][\text{BPh}_4]$ .

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The crystal structure of the title complex was determined by single crystal X-ray analysis. Data were collected on a Nonius KappaCCD diffractometer equipped with a fine focus molybdenum X-ray source, 0.3 mm *ifg* capillary collimator, and an Oxford Cryosystems crystal cooler. Structure solution and refinement utilized SIR97, MaXus, and the SHELXTL system.

The cation consists of trigonal bipyramidal tin(IV), with three butyl groups coordinated in the three equatorial positions and triphenylphosphine oxide ligands in the axial positions. The supramolecular structure is dominated by extensive concerted phenyl-phenyl interactions [1] among the six phenyl groups of the cation triphenylphosphine moieties and the four phenyl groups of the anion tetraphenylborate moieties.

*Crystal data:*  $\text{C}_{72}\text{H}_{77}\text{BO}_2\text{P}_2\text{Sn}$ ;  $M_r = 1165.78$  Daltons; transparent colorless flat slabs; 0.17 x 0.20 x 0.23 mm; orthorhombic;  $Pna2_1$ ;  $a = 19.2421(3)$ ,  $b = 14.9077(4)$ ,  $c = 21.9461(5)$  Å,  $V = 6295.4$  Å<sup>3</sup>;  $Z = 4$ ;  $D_{\text{calc}} = 1.230$  Mg/m<sup>3</sup>;  $\lambda_{\text{MoK}\alpha} = 0.71073$  Å;  $\mu = 5.0$  cm<sup>-1</sup>;  $T = 200$  K. Data collection: Bruker-Nonius KappaCCD, 0.3 mm *ifg* capillary collimator, 60,132 data collected,  $R_{\text{int}} = 0.0775$ , 8209 unique data, 6431 observed data ( $I_o > 4\sigma(I_o)$ ).

[1]Dance, I., Scudder, M., *J. Chem. Soc., Chem. Commun.*, 1995, 1039.

**Keywords:** noncovalent bonding; crystal structure; tin compound