Single Crystal X-Ray Diffraction Unit

Introduction

Single crystal X-ray diffraction allows the determination of the three-dimensional structure of small molecules and macromolecules of any kind, such as organic molecules or biological macromolecules (proteins, DNA segments, viruses and ribosomes), alloys, and minerals. This information can then be used for the clarification of several important issues, such as the nature of chemical bonding, the mechanisms involved in important biological processes, questions related to the development of nanotechnology and so on.

Facilities & Infrastructure

The University of Ioannina has recently acquired an automatic Siemens (now Bruker) P4 single-crystal X-ray diffractometer (Fig. 1). It consists of a 3kW X-ray generator, a four-circle goniometer, a scintillation detector and a computer (Pentium) equipped with the software required to run the diffractometer. A liquid nitrogen cryostat, for use in low-temperature data collection, was also provided. This, however, has never been used, due to the lack of an appropriate container for the liquid nitrogen.

This diffractometer is mainly designed to be used for small-molecule (molecules composed of 400-500 atoms) crystal structure determination. The crystal's unit cell and space group are also determined in the process. The equipment can also be used to determine the morphology of the crystal. Use of the cryostat would allow the collection of data at low temperatures, which is generally more expensive, but permits the determination of difficult structures (involving disorder) and charge density studies.

FUTURE DEVELOPMENT

The most immediate priorities are:

a) purchase of the liquid nitrogen container, to allow use of the cryostat
b) acquiring SHELXTL software (Bruker patent), to be used in resolving crystal structures.

It is also very important that a new generation CCD (charge coupled device), with which all modern crystallography laboratories are equipped, be installed. CCD diffractometers have great capabilities and are frequently necessary in certain areas of research such as nanochemistry and supramolecular chemistry (see Tsoucaris, Current Challenges on Large Supramolecular Assemblies, Kluwer 1999 p. 130, and J. Atwood, Science, 285, 1999, p. 1049).

Lastly, it is appreciated that development of crystallography of biological macromolecules, which is of particular importance to biotechnology and pharmacy, will require the purchase of an X-ray diffraction system appropriate for such substances.

Figure 1: Brüker P4, single crystal X-ray diffractometer.

Contact Information

A Scientific/Administrative Committee is responsible for the Single Crystal X-ray Diffraction Unit. Information can be acquired from S. Skoulika (vskoul@cc.uoi.gr) or A. Michaelides (amihail@cc.uoi.gr) and Dr. Dokorou Vassiliki (dokorou@cc.uoi.gr)

Postal address: Laboratory of Physical Chemistry, Chemistry Department, The University of Ioannina, 451 10 Ioannina, Greece
Tel: +30-26510-08446, 08447, 08445.
Fax: +30-26510-08798

Representative Publications

1. I. Ozturk, S. Filimonova, S.K. Hadjikakou, N. Kourkoumelis, V. Dokorou, E. Manos, J. Tasiopoulos, M.M.Barsan, I.S. Batler, E.R. Milaeva, Jan Balzarini, N. Hadjiliadis 'Structural

Motifs and Biological Studies of New Antimony(III) Iodide Complexes with Thiones' *Inorganic Chemistry* 49 **(2010)** 488-501.
2. A. Michaelides, S. Skoulika, "Crystallo graphic Evidence for Ionic Molecular Building Blocks in the Assembly of a Two- Dimensional Metal - Organic Frame work" *Crystal Growth & Design,* 9 **(2009)** 4998-5002.
3. K.N. Kouroulis, A.K. Metsios, S.K. Hadjikakou, V.Y. Tyurin, N. Kourkoumelis, A.V. Dolganov, M. Kubicki, E.R. Milaeva L. Male, M. Hursthouse, N. Hadjiliadis, S. Skoulika, "Synthesis, structural characterization and *in vitro* cytotoxicity o f new Au (III) and Au (I) complexes with thioamides" *Dalton Trans.*, **(2009)** 10446–10456.
4. A. Michailidis, S. Skoulika, M.G. Siskos, "Designed self-assembly of a reactive metal–organic framework with quasi a-Po topology" *CrystEng Comm* 10 **(2008)** 817–820.
5. Th. Liaskopoulos, S. Skoulika, P.G. Tsoungas, G. Varvounis, "Novel Synthesis of Naphthopyranoisoxazoles and Versatile Access to Naphthopyranoisoxazolines" *SYNTHESIS* 5 **(2008)** 0711–0718.
6. M N. Xanthopoulou, S. Skoulika, S. K. Hadjikakou, T. Bakas, M. Baril, N. Hadjiliadis, I. S. Butler, "Synthesis, Structural Characterization, and Biological Studies of Sixand Five-Coordinate Organotin(IV) Complexes with the Thioamides 2-Mercaptobenzothiazole, 5-Chloro-2-mercaptobenzothiazole, and 2-Mercaptobenzoxazole" *Inorg. Chem*., 46 **(2007)** 1187-1195.
7. P. Supsana, P. Tsoungas, A. Aubry, S. Skoulika, G. Varvounis, "Oxidation of 1-acyl-2-naphthol oximes: peri- and o-cyclisation and spiro cyclodimerisation of naphthoquinone nitrosomethide intermediates". *Tetrahedron* 57 (**2001**) 3445-3453.
8. A. Dimos, D. Tsaousis, A. Michaelides, S. Skoulika, S. Gohlen, L. Ouahab, C. Didierjean, A. Aubry "Microporous Rare Earth Coordination Polymers: The Effect of Lanthanide Contraction on Crystal Architecture and Porosity". *Chem. Mater*.
9. K. Barbarossou, A. E. Aliev, I. P. Gerothanassis, J. Anastassopoulou and T. Theophanides, "Natural Abundance ^{15}N CP MAS NMR as a Novel Tool for Investigating Metal Binding to Nucleotides in the Solid State." *Inorg. Chem* 40 **(2001)**3626-3628.
10. A. Dimos, A. Michaelides, S. Skoulika "A Molecular Bilayer Motif Constructed from a Three-Connected Organic Ligand and Cd2+ Cations: Crystal Structure of $[Cd_3(trimesate)_2(H_2O)_9]\ 2H_2O$", *Chem. Mater*. 12 (**2000**) 3256-3258
11. P. Zachariadis, S. Hadjikakou, N. Hatjiliadis, A. Michaelides, S. Skoulika, Y. Ming, Y. Xiaolin "Synthesis, structure and anti-HIV activity of a water soluble hexanuclesr silver(I) cluster compound with 2-mercapto-nicotinic acid as ligand". *J. Medicinal Chemistry.*