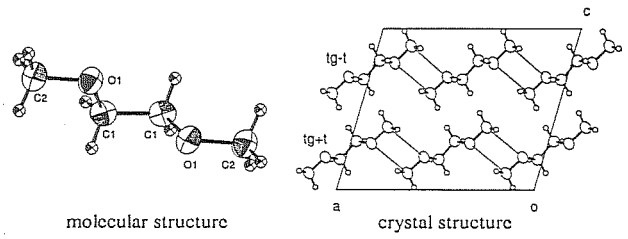
PS11.02.07 STRUCTURE DETERMINATION OF 1,2-DIMETHOXYETHANE AND THE RELATED COMPOUNDS BY USING A NEW DIFFRACTOMETER. Yoshihiro Yokoyama, Hidehiro Uekusa, and Yuji Ohashi, Department of Chemistry, Faculty of Science, Tokyo Institute of Technology, Ookayama, Meguro-ku, Tokyo 152

The crystal structure of 1,2-dimethoxyethane(DME) and the related compounds, which are low-melting-point materials, have been determined. Although these structures have long been subject of interest for conformational studies as the monomer model of poly(oxyethylene), the crystal structures have not been reported from difficulty in getting the single crystal at low temperatures.

Recently a new imaging plate-Weissenberg camera type diffractometer with a new low temperature equipment was designed and made. This equipment enable us to perform the rapid intensity data collection before ice is formed on the surface of the crystal.

The crystal of DME belongs to the monoclinic system and the space group is C2/c. There are four molecules in an unit cell. The DME molecule lies on a two-fold axis and has a TGT conformation, which is not most stable among the possible conformations, in the crystalline state.

When the two oxygen atoms were replaced with two sulfur atoms, the conformation was changed to GTG' conformation.



molecular structure crystal structure

Dynamic Properties in Molecular Crystals

MS11.03.01 VIBRATION AND DIFFUSION IN CRYSTALS OF SMALL MOLECULES AND PROTEINS: SIMULATION EXPERIMENT. Jeremy C. Smith, Simulation Moleculaire, SBPM/DBCM, CEA-Saclay, 91191 Gif-sur-Yvette Cedex, France

Molecular dynamics simulation and normal mode analysis are combined with neutron and far-infrared spectroscopy and X-ray diffuse scattering to examine picosecond-timescale dynamics in molecular crystals and proteins. Hydrogen-bond vibrations, lattice modes and molecular rotational and translational diffusion are characterised in small molecule crystals. Collective and diffusive motions in globular proteins are also determined and their functional roles discussed.

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A. Micu, D. Durand, M. Quilichini, M.J. Field & J.C. Smith, Lattice Vibrations in Lalanine, *Chem J. Phys. Chem.*, 99, 5645-5657 (1995). G.R. Kneller& J.C. Smith, Liquid-like Side-chain Dynamics in Myoglobin, *Chem J. Molec. Biol.*, 242, 181185, (1994).
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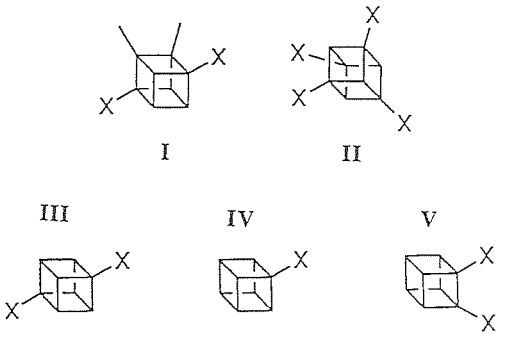
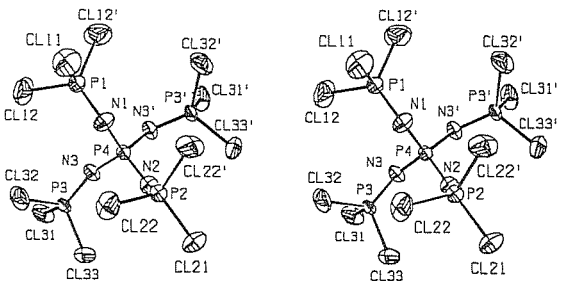
MS11.03.02 MOLECULAR DYNAMICS IN SUBSTITUTED CUBANES- A JOINT CRYSTALLOGRAPHIC AND SOLID STATE NMR STUDY. Alison J. Edwards, Michaele J. Hardie and Frances Separovic, School of Chemistry, University of Melbourne, Parkville, Vic., Australia; John Tsanaktsidis, CSIRO Division of Chemicals and Polymers, Clayton, Vic., Australia

As part of a systematic search for n-fold reorientational dynamics in molecular crystals, a range of halogen substituted cubane compounds I - V (X = Cl, Br or I) are being examined by joint application of solid state Nuclear Magnetic Resonance spectroscopy and X-ray Crystallographic techniques. These compounds were chosen for the wide range of possible symmetry elements about which dynamic behaviour might be observed and for their relative ease of synthesis. Studies of the molecules of type I are furthest advanced and results to date for these dimethyl compounds will form the basis of this presentation.



MS11.03.03 ANALYSIS OF RIGID BODY AND NON-RIGID BODY MOTIONS IN X-RAY CRYSTALLOGRAPHY. F. Belaj, Institut für Anorganische Chemie, Universität Graz, Austria

Due to the high flexibility at the nitrogen atoms the $[P(NPCl_3)_4]^+$ cation as a whole cannot be described as a rigid body not even with allowance for intramolecular torsions about bonds (J.D. Dunitz, D.N.J. White, *Acta Cryst.*, 1973, **A29**, 93-94). The analysis of the thermal motion of this cation and of related species shows that the librations can best be described as almost independent motions of rigid tetrahedra, coupled by 'ball joints'.

An accurate low-temperature structure determination of $[P(NPCl_3)_4]^+ ICl_2$ was hindered by phase transition and the room-temperature structure, the cations lying on 4, could not be interpreted without some difficulties, too (F. Belaj, *Acta Cryst.*, 1995, **B51**, 65-71). Recently the structure determination of $[P(NPCl_3)_4]^+PCl_6^-$ succeeded at 93K: The cations are located on mirror planes, thus three different $NPCl_3$ groups can be compared (see figure). The P-N bond lengths and P-N-P bond angles do not show such exceptionalities as in the ICl_2^- structure, but similar to the latter the PN_4 and $NPCl_3$ tetrahedra of the cation as well as the anion behave as rigid bodies with $R_u<0.05$ $\{R_u = [\sum w(\Delta U)^2/\sum w(U_{obs})^2]^{1/2}\}$, whereas for the cation as a whole an R_u value of 0.35 is obtained.