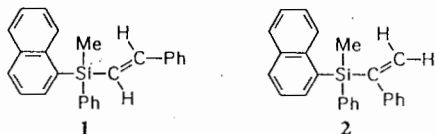


## 06-Crystallography of Organic Compounds

**PS-06.03.18** CRYSTAL STRUCTURES AND ABSOLUTE CONFIGURATIONS OF TWO OPTICALLY ACTIVE METHYLNAPHTHYLPHENYLSILYL(PHENYL)ETHYLENES. By S.V.Belyakov\*, L.I.Borisova, A.R.Vjater, A.F.Mishnev, E.Lukevics, Latvian Institute of Organic Synthesis, Riga, Latvia

In the course of stereochemical investigation of chiral silicon derivatives crystals of optically active enantiomers of the title compounds were obtained.



The crystal structures of 1 and 2 were determined by a single crystal X-ray diffraction technique. Crystal data are:

	a(Å)	b(Å)	c(Å)	Space group
1	8.467	13.210	18.413	P 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub> (Z=4)
2	7.519	11.853	22.428	P 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub> (Z=4)

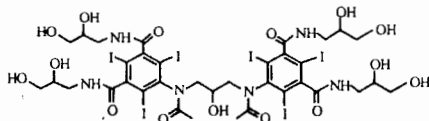
The absolute configurations of silicon atoms were obtained by anomalous X-ray scattering. Conformational analysis using molecular mechanics method and crystallographic data will be also presented.

#### 06.04 – Crystal and Molecular Packing, Hydrogen Bonding

**PS-06.04.01** MOLECULAR AND CRYSTAL STRUCTURE OF A NEW X-RAY CONTRAST AGENT, C<sub>35</sub>H<sub>44</sub>I<sub>6</sub>N<sub>6</sub>O<sub>15</sub>.

By L. Enehom\* and B. Fjærtøft Pedersen, Institute of Pharmacy, University of Oslo, Norway.

The title compound, Iodixanol, is a water soluble non-ionic dimeric contrast agent for use in diagnostic imaging. The structure of a non-ionic monomer has previously been reported (Ganazzoli, F. (1983). *Acta Cryst.* C39, 1570-1572.).



The intensity data from a single crystal were collected on a Nicolet P3 diffractometer at liquid nitrogen temperature. 12 132 reflections were measured, out of which 4779 were considered observed [ $I > 2.5\sigma(I)$ ]. Iodixanol crystallizes in the triclinic space group  $P\bar{1}$ ,  $a=16.864(3)\text{Å}$ ,  $b=18.164(3)\text{Å}$ ,  $c=19.360(4)\text{Å}$ ,  $\alpha=95.68(2)^\circ$ ,  $\beta=102.43(2)^\circ$ ,  $\gamma=114.56(1)^\circ$ ,  $V=5147(2)\text{Å}^3$ ,  $Z=4$ ,  $\lambda(\text{MoK}\alpha)=0.71069\text{Å}$ . The structure was solved by direct methods (Gilmore, C.J. (1983). *MITHRIL, Version 1.0*. University of Glasgow, Glasgow, Scotland.), and refined by full-matrix least-squares minimization of  $\sum w(\Delta F)^2$ , where  $w=1$  (Mallinson, P.R. and Muir, K.W. (1985). *J. Appl. Cryst.* 18, 51-53.). The structure was corrected for absorption, and anisotropic vibration was included for the iodine atoms. The high flexibility of the side chains makes their determination difficult. The structure has an R-factor of 7.5%, and an e.s.d. in the bond distances from 0.04 Å to about 0.15 Å for the outermost atoms. There are two different conformations of the molecule in the asymmetric unit. One of them has an intramolecular hydrogen bond, which gives it a folded conformation.

**PS-06.04.02** CRYSTAL AND MOLECULAR STRUCTURE OF A MESOGENIC COMPOUND By P.Mandal\* and S.Paul, Department of Physics, North Bengal University, Siliguri-734430, India and K.Goubitz and H.Schenk, Crystallography Laboratory, University of Amsterdam, 1018WV Amsterdam, The Netherlands

The molecular arrangement in the crystalline state is one of the factors which predetermines the occurrence of thermal mesomorphism in organic compounds. As a part of a series of structural investigations of mesogenic compounds we have determined the crystal structure of Bis(p-butoxybenzylidene)-p-toluidine (BBBT). Under crossed polarizing microscope pretransition effect was observed both before Cr-Sm and N-I transitions. An unspecified varieties of mosaic texture in smectic regions and marbled texture in nematic regions were observed. Supercooling was noticed as indicated below. X-ray diffraction photographs were taken in mesomorphic range with and without magnetic field. These indicate the smectic phase as SmecticG. Observed transition temperatures ( $^\circ\text{C}$ )

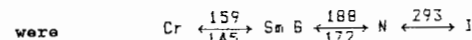


Plate shaped single crystals were grown by slow evaporation from benzene solution. Intensity data were measured in a CAD-4 diffractometer. Cell dimensions are  $a=6.1162(29)$ ,  $b=7.9157(25)$ ,  $c=31.4213(81)$ ,  $\alpha=92.39(2)^\circ$ ,  $\beta=92.35(3)^\circ$ ,  $\gamma=96.59(2)^\circ$ ,  $Z=2$  and space group P1. 5108 reflections were measured of which 2129 were treated as observed with  $I > 2.5\sigma(I)$ . The structure was solved by the symbolic phase extension program SIMPEL and was refined to a R-value of 0.12 and  $R_w$  of 0.08. At this stage a  $\Delta-F$  map showed no maxima higher than 0.41  $e/\text{Å}^3$ . Bond lengths and angles were normal. However temperature factors of a few atoms of butoxy chains were very high. Higher R value may be due to structural disorder of butoxy chains. The symmetrical fractions of the molecule C1-C18 and C19-C36 are planar (Fig.1), but the angle between them is about  $60^\circ$ . It is observed that molecules are arranged in layers inclined to ab-plane and the layers are stacked along the c-axis (Fig. 2). This tilted layer like structure is precursor to the tilted smectic phase. Twisted molecular geometry and van der Waals' interactions result in very efficient packing of the centro symmetrically related molecules. Few intermolecular short contacts observed in overlapping region supports this conclusion. The high melting point of the compound may be due to this packing. At the Cr-Sm transition temperature the molecules gain sufficient energy to move within the layers and to undergo rotations about the long molecular axis. The Cr-Sm transition is likely to be displacive type, details will be discussed.

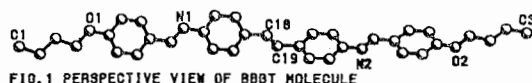


FIG.1 PERSPECTIVE VIEW OF BBBT MOLECULE

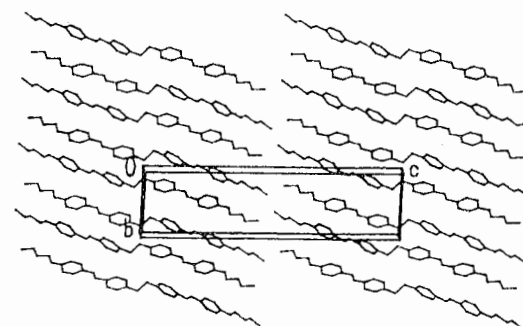


FIG.2 CRYSTAL STRUCTURE OF BBBT VIEWED ALONG X-AXIS