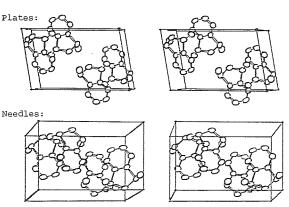
## 09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

BENZENE-o-DISULFINIC ANHYDRIDE. -ONE COMPOUND -TWO CRYSTAL STRUCTURES. By Rita G. Hazell, Chemistry department, Aarhus University, DK8000 Århus C, Denmark.

Recrystallization of benzene-o-disulfinic anhydride from boiling acetonitrile gave colourless plates from the hot solution. When the mother liquor was left at  $-25^{\circ}$ C colourless needles formed, giving a suspicion of cistrans isomerism.

Crystal data: Plates:a=7.377(2), b=7.783(1), c=12.313(2),  $\beta$ =99.78(1), F21/n, Z=4, R=0.033. Needles: a=7.521(2), b=11.860(5), c=8.132(3),  $\beta$ =100.46(2), P2 $_1$ /c, Z=4,R=0.035. Both structures solved by MULTAN.

The cis-conformation was found in both structures, the main difference being that the two S-O bonds in the 5ring were significantly different , 1.686 and 1.662  $\mbox{\AA},$ in the low temperature form, not in the other. This is explained by a fairly short contact to an oxygen atom of another molecule from the sulfur with the long S-O bond.



THE CRYSTAL AND MOLECULAR STRUCTURES OF DIBENZIMIDE AND ITS N-METHYLATED ANALOGUE: STERIC AND HYDROGEN-BONDING EFFECTS. Mizrahi and M. Niven, School of Chemical Sciences, University of Cape Town, Rondebosch, South Africa.

Dibenzimide, C14H11NO2, Mr=225.3, orthorhombic,  $Ib\alpha_2$ ,  $\alpha$ =15.775(8), b=8.471(4), c=9.003(4)Å,  $D_{\alpha}$ =1.24Mgm<sup>-3</sup>, Z=4, μ(Mo $K\alpha$ )=0.05mm<sup>-1</sup>, F(000)=472, R=0.09,  $R\omega$ =0.07 ( $\omega$ =( $\sigma^2F$ )-1) for 196 reflections; N-Methyldibenzimide,  $C_{15}H_{13}NO_{2}$ , Mr=239.3, monoclinic,  $P^{21}/c$ ,  $\alpha=10.107(5)$ , b=15.625(8), c=7.912(4)Å, =96.87(2)°,  $D_{\infty}=1.28 \text{Mgm}^{-3}$ , Z=4,  $\mu$  (Mo $K\alpha$ ) = 0.05 mm<sup>-1</sup>, F(000)=504, R=0.05,  $R\omega=0.04$  ( $\omega=(\sigma^2F)^{-1}$ ) for 1083 reflections; graphitemonochromated Mo $K\alpha$  radiation ( $\lambda=0.7107$ Å) used in both determinations in both determinations.

Both compounds exhibit significant deviation from OCNCO planarity, in addition to substantial aromatic ring twist. The conformation of the secondary imide is  $\simeq (Z,Z)$  with the N-H group located on a two-fold axis. The imide hydrogen is shared in intermolecular hydrogen bonding by the two carbonyl oxygen atoms of an adjacent molecule. In contrast In contrast. the tertiary imide adopts an =(E,Z) conformat-

An important consequence of N-methylation of dibenzimide is that the nitrogen atom pyramidality is distorted, which reduces the conjugation within the OCNCO moiety. This effect manifests itself in weakening the N-C bond with concomitant enhancement of carbonylcarbon electrophilicity (as indicated by comparative <sup>13</sup>C n.m.r. chemical shifts) in the

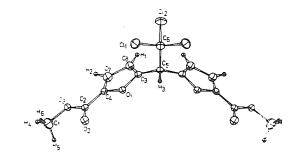
tertiary derivative. These observations are consistent with the greater susceptibility of tertiary imides to nucleophilic attack than their secondary analogues.

09.2-2 CRYSTAL STRUCTURE OF 1,1-BIS(5-CARBOMETHOXY-2-FURYL)-2,2,2-TRICHLOROETHANE. By R. Pomes and F. Fajardo, Academy of Sciences of Cuba and University of Oriente, Santiago de Cuba, CUBA.

The crystals of  $C_{14}H_{11}O_6Cl_3$  are monoclinic, space group  $P2_1/m$  with a = 6.38(1), b = 21.19(1), c = 6.21(1)Å,  $\beta = 102.15(1)^{\circ}, Z = 2.$ 

Data were collected with a perpendicular beams diffractometer using MoKa radiation. Direct methods were used for the determination of the structure. Refinement was made by the full-matrix least-squares method to a final R value of 0.03 for 981 F(hkl).

The structure is stabilized by Van der Waals contacts.



Molecular structure of  $C_{14}H_{11}O_6Cl_3$