

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

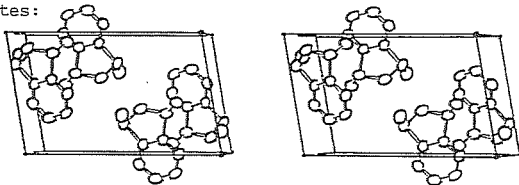
09.1-7 BENZENE-O-DISULFINIC ANHYDRIDE. -ONE COMPOUND  
-TWO CRYSTAL STRUCTURES. By Rita G. Hazell,  
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Denmark.

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

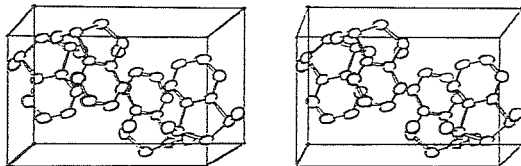
Crystal data: Plates:  $a=7.377(2)$ ,  $b=7.783(1)$ ,  $c=12.313(2)$ ,  
 $\beta=99.78(1)$ ,  $P2_1/n$ ,  $Z=4$ ,  $R=0.033$ . Needles:  $a=7.521(2)$ ,  
 $b=11.860(5)$ ,  $c=8.132(3)$ ,  $\beta=100.46(2)$ ,  $P2_1/c$ ,  $Z=4$ ,  $R=0.035$ .  
Both structures solved by MULTAN.

The cis-conformation was found in both structures, the  
main difference being that the two S-O bonds in the 5-  
ring were significantly different, 1.686 and 1.662 Å,  
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.

Plates:



Needles:



09.2-1 THE CRYSTAL AND MOLECULAR STRUCTURES  
OF DIBENZIMIDE AND ITS N-METHYLATED ANALOGUE:  
STERIC AND HYDROGEN-BONDING EFFECTS. By V.  
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Sciences, University of Cape Town, Rondebosch,  
South Africa.

Dibenzimide,  $\text{C}_{14}\text{H}_{11}\text{NO}_2$ ,  $M_r=225.3$ , orthorhombic,  
 $Iba_2$ ,  $a=15.775(8)$ ,  $b=8.471(4)$ ,  $c=9.003(4)$  Å,  
 $D_x=1.24\text{Mgm}^{-3}$ ,  $Z=4$ ,  $\mu(\text{MoK}\alpha)=0.05\text{mm}^{-1}$ ,  $F(000)=472$ ,  
 $R=0.09$ ,  $R_w=0.07$  ( $w=(\sigma^2 F)^{-1}$ ) for 196 reflect-  
ions; N-Methyldibenzimide,  $\text{C}_{15}\text{H}_{13}\text{NO}_2$ ,  $M_r=239.3$ ,  
monoclinic,  $P2_1/c$ ,  $a=10.107(5)$ ,  $b=15.625(8)$ ,  
 $c=7.912(4)$  Å,  $\beta=96.87(2)^{\circ}$ ,  $D_x=1.28\text{Mgm}^{-3}$ ,  $Z=4$ ,  
 $\mu(\text{MoK}\alpha)=0.05\text{mm}^{-1}$ ,  $F(000)=504$ ,  $R=0.05$ ,  $R_w=0.04$   
( $w=(\sigma^2 F)^{-1}$ ) for 1083 reflections; graphite-  
monochromated MoK $\alpha$  radiation ( $\lambda=0.7107$  Å) used  
in both determinations.

Both compounds exhibit significant deviation  
from OCNCO planarity, in addition to  
substantial aromatic ring twist. The conform-  
ation of the secondary imide is  $\approx(Z,Z)$  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,  
the tertiary imide adopts an  $\approx(E,Z)$  conforma-  
tion.

An important consequence of N-methylation of  
dibenzimide is that the nitrogen atom pyramid-  
ality is distorted, which reduces the conjug-  
ation within the OCNCO moiety. This effect  
manifests itself in weakening the N-C bond  
with concomitant enhancement of carbonyl-  
carbon electrophilicity (as indicated by comp-  
arative  $^{13}\text{C}$  n.m.r. chemical shifts) in the

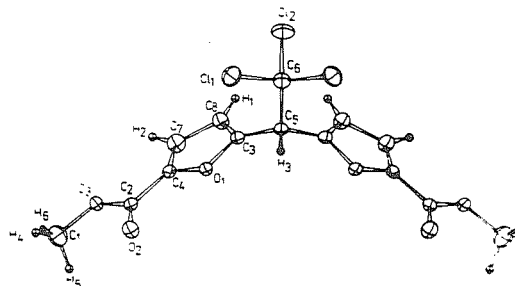
tertiary derivative. These observations are  
consistent with the greater susceptibility  
of tertiary imides to nucleophilic attack  
than their secondary analogues.

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

The crystals of  $\text{C}_{14}\text{H}_{11}\text{O}_6\text{Cl}_3$  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 MoK $\alpha$  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  $\text{C}_{14}\text{H}_{11}\text{O}_6\text{Cl}_3$