

09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

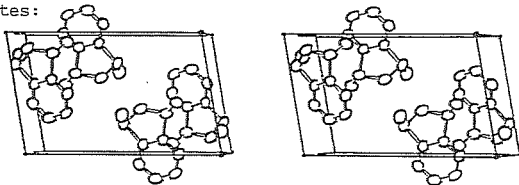
09.1-7 BENZENE-O-DISULFINIC ANHYDRIDE. -ONE COMPOUND
-TWO CRYSTAL STRUCTURES. By Rita G. Hazell,
Chemistry department, Aarhus University, DK8000 Århus C,
Denmark.

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

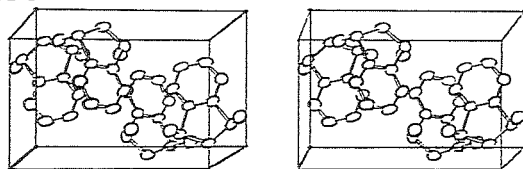
Crystal data: Plates: $a=7.377(2)$, $b=7.783(1)$, $c=12.313(2)$,
 $\beta=99.78(1)$, $P2_1/n$, $Z=4$, $R=0.033$. Needles: $a=7.521(2)$,
 $b=11.860(5)$, $c=8.132(3)$, $\beta=100.46(2)$, $P2_1/c$, $Z=4$, $R=0.035$.
Both structures solved by MULTAN.

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.
Mizrahi and M. Niven, School of Chemical
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 α 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 compar-
ative ^{13}C n.m.r. chemical shifts) in the

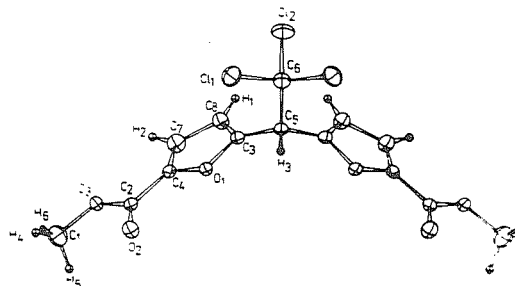
tertiary derivative. These observations are
consistent with the greater susceptibility
of tertiary imides to nucleophilic attack
than their secondary analogues.

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 α 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 $\text{C}_{14}\text{H}_{11}\text{O}_6\text{Cl}_3$