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 Aarhus 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 5-0 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.

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, \( C_{14}H_{11}N_2O_2 \), Mr=225.3, orthorhombic, \( Iba_2 \), a=15.775(8), b=8.471(4), c=9.003(4) Å, \( D_x=1.24 \) mg/cm\(^3\), \( Z=4 \), \( \beta (MoK\alpha)=0.05 \) mm\(^{-1}\), \( R=0.09 \), \( R_w=0.07 \) (\( w=(\sigma^2 p)_I \)) for 196 reflections; N-Methyl dibenzimide, \( C_{15}H_{14}N_2O_3 \), Mr=239.3, monoclinic, \( P2_1/\alpha \), a=10.107(5), b=15.625(8), c=7.912(4) Å, \( \beta = 102.15(1) \), \( D_x=1.28 \) mg/cm\(^3\), \( \beta (MoK\alpha)=0.05 \) mm\(^{-1}\), \( F(000)=504 \), \( R=0.05 \), \( R_w=0.04 \) (\( w=(\sigma^2 p)_I \)) 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 conformation of the secondary imide is \( \psi = (2,2) \) with the N-R 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 \( \psi = (3,2) \) conformation.

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 carbonyl carbon electrophilicity (as indicated by comparative \( ^{13}C \) 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-CARBOXYTHI0XY-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 \( P2_1/n \) with \( a=6.38(1) \), \( b=21.19(1) \), \( c=6.21(1) \) Å, \( \beta = 102.15(1) \), \( Z=2 \).

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.