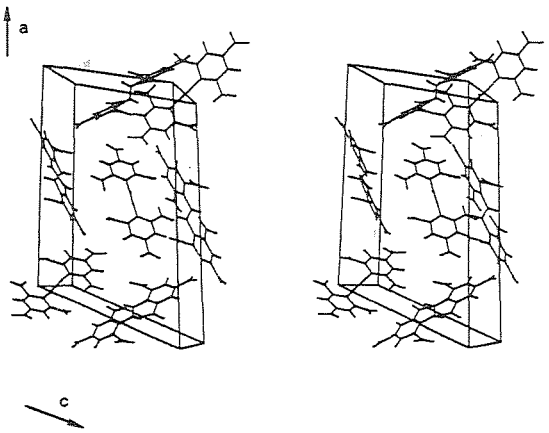
09.2-6 CRYSTAL STRUCTURE OF HNS, 2,2',4,4',6,6'-hexanitrostilbene.

By F.GERARD and A.HARDY, laboratoire de cristallochimie minérale, université de Poitiers, 40 av. Recteur Pineau, F - 86022 POITIERS CEDEX, France.

Monoclinic, $P2_1/c$, a=22.326(7)Å, b=5.5706(9)Å, c=14.667(2)Å, β=110.04(1)°, V=1714(1)Å^3, Z = 4, D_m=1.74(1), D_x=1.745(1), Cu $K\alpha_1$ λ=1.54051 Å , μ = 13.30 cm^{-1}, F(000) = 912, room temperature, R=.060 for 2345 independent reflections, Rw=.057



Two different molecules have a symmetry centre either in 2(d) or in 2(c). Their benzene planes are parallel and respectively 1.298Å and 1.428Å apart. NO_2 groups are twisted in the range 5.51° to 48.64° with respect to carbon rings. Molecules are tilted with regards to the axes and make an herringbone pattern

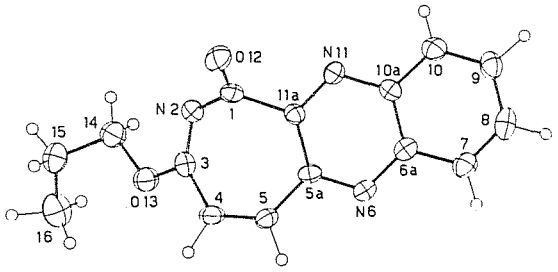
The most compact molecules stacking is along b.

09.2-7 CRYSTAL AND MOLECULAR STRUCTURE OF 1-OXO-3-PROPOXYAZEPINO[7,6-b]QUINOXALINE. By Bruna Bovio, Dipartimento di Chimica Generale, Università di Pavia, Italy.

In the course of investigations of photochemical decomposition of 2-azido-1-(3,5-dimethylpyrazolyl)phenazine in n-propylalcool solution, a compound $C_{15}H_{13}N_3O_2$ was isolated from several reaction products. Since the determination of the structural formula by chemical means appears to be not smooth and IR, ^{1}H NMR, and mass spectra do not permit to attribute unambiguously the structure to the title compound, it was deemed necessary to carry out a single-crystal X-ray analysis.

Crystals are triclinic: space group $P\bar{1}$ with a = 7.289(2) b = 14.414(5) c = 6.797(2) Å α= 83.56(3) β= 68.73(3) γ= 86.58(4)° Z = 2.

The structure was solved by direct methods and refined by full-matrix least-squares to a final R value of 0.047 (Rw = 0.024) for 908 reflections having $I \geq 2\sigma(I)$.



The seven-membered ring exhibits a marked puckering: the puckering parameters, calculated according to Cremer and Pople (J.Am.Chem.Soc., 1975, 97, 1354) are

q_2 = 0.604 ϕ_2 = 358.8°
q_3 = 0.161 (ϕ_3 = 188.5°)
Q^3 = 0.626 θ^3 = 75.0°

These puckering parameters describe a distorted boat. The direction of the distortion is given by θ, which is smaller than 90°; therefore the ring is distorted from the pure boat in the direction of a chair. Indeed, the bow angle is 44.5°, whereas the stern angle is 24.9°. The double bonds are clearly localized at N(2)-C(3) = 1.277(5) and C(4)-C(5) = 1.326(6) Å, whereas the C(5a)-C(11a) bond = 1.418(5) which hinges the two condensed heterocycles, is longer than a double bond, because it takes part in the conjugation within the quinoxaline moiety. The shortening of the C(1)-N(2) bond, 1.382(5), suggests that there is some electron delocalization between the CO group and the adjacent N(2)-C(3) double bond; on the contrary the long C(1)-C(11a) bond, 1.519(5) Å, rules out any electron delocalization between the CO group and the quinoxaline moiety. All the bonds in the quinoxaline moiety have a partial double-bond character, thus reflecting the aromatic character of the quinoxaline: indeed the two condensed rings are nearly coplanar (dihedral angle 1.1°) in spite of their individual nonplanarity. With regard to the propoxy chain, it is worth while to remark the short C(3)-O(13) ether bond (1.335(5) Å) which suggests that there is some electron delocalization between O(13) and the adjacent N(2)-C(3) double bond.