09.2.56 MOLECULAR AND CRYSTAL STRUCTURE OF AN OPEN CYCLAMPHOSPHORANE ADDUCT: BIS(BORANE)CYCLAMPHOSPHANE, by J.N. Dupart, A. Grand, S. Face and J.G. Bles, DRF, Laboratoires de Chimie, Centre d'Etudes Nucleaires, 38, F. 38041 Grenoble Cedex, France.

The first X-ray structure determination of an "open tautomeric form" derivative of a tetracyclic tetramethinophosphorane, bis(borane)cyclamphosphan, is reported. The borane groups are coordinated to the P and N atoms, confirming the NMR data in solution, which pointed to the presence of only one of the two possible diastereoisomers; this has now been identified as 5', in which the P=B and N=B bonds are oriented trans to each other with respect to the molecule's mean plane. The P=B bond length (1.859(5) Å) and N=B bond length (1.619(6) Å) are in the usual range. There is no linear correlation between the sum (ΣN) of the bond angles around the three tertiary nitrogen atoms (N1, N2 and N3) and the three corresponding P=N bond lengths. A very short P=N3 bond (1.653(4) Å) is found, in spite of the distinctly pyramidal geometry of the N2 atom (ΣN2 = 341.6° ± 1°). The five- and six-membered rings adopt the low energy "envelope" and "chair" conformations, indicating the absence of noticeable constraints in the polycyclic structure. The results presented in this paper are expected to be relevant to most open tautomeric forms of the tetracyclic tetramethinophosphoranes, whether uncomplexed or acting as mono or bidentate ligands towards Lewis acids.

09.2.57 THE CRYSTAL AND MOLECULAR STRUCTURE OF 4-BENZYLIDENE-4'-BENZOXYFANE-5-ONE, by A. Katrusiak, M. Ratajczak-Sitarz and Z. KIausingi, Institute of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-760 Poznań, Poland.

This work is the continuation of our previous X-ray studies on unsaturated ketones /Z. KIausingi et al., Bull. Acad. Polon. Chim. (1976), 11 A.; Hoefer et al., Acta Cryst. (1980), B36, 1256/60/. Our crystallographic works were set up to establish both the effects of different substituents of the phenyl group close to carbonyl on the conjugation of alternative π-electron system and the influence of strains in the molecule on its conformation.

Crystals of the title compound were obtained by slow evaporation of its ethanol solution. The measurement was carried out on a SYNTEX P21 diffractometer using graphite monochromated Cu Kα. The crystals are orthorhombic, space group P2_1 2_1 2_1, a=11.208/12 Å, b=12.082/2 Å, c=9.978/10 Å, D_n=1.139 Mgm⁻³, Z=4.

The structure was solved by direct methods using MULTAN-80. Final R=0.047 for 992 observed reflections using isotropic thermal parameters for hydrogen atoms and anisotropic thermal parameters for all other atoms.

The bond lengths of the 1,3-azone system are in a very good agreement with those observed for other similar structures of unsaturated ketones. The observed bond lengths correspond to no conjugated bonds. However the bond lengths in the ether group indicate a slight conjugation with the neighbouring phenyl ring.