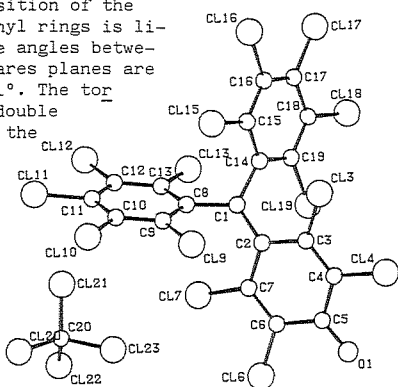


09.2-55 THE CRYSTAL AND MOLECULAR STRUCTURE OF 1:1 COMPLEX PERCHLOROPHOSPHONIC ANHYDRIDE AND CARBON TETRACHLORIDE. By E. Molins, J. Rius and C. Miravittles. Instituto de Geología "Jaime Almera", C.S.I.C. c/. Alcarria, s/n, Aptdo. de Correos 30.102, Barcelona, Spain.

In the course of the structural studies of perchlorinated organic compounds, we have solved the structure of the title compound in order to establish the general conformation and the torsion of the C1-C2 double bond. The compound crystallizes in the triclinic system  $P\bar{1}$  with  $a = 7.256(6)$ ,  $b = 13.663(6)$ ,  $c = 16.341(6)$  Å,  $\alpha = 75.22(4)$ ,  $\beta = 80.18(7)^\circ$ ,  $\gamma = 82.57(5)^\circ$ . Intensity data for 2017 reflections were measured on an automatic CAD4 diffractometer. The structure was solved with the MULTAN 11/82 system (Main, P. et al., 1982) and refined by SHELX-76 (Sheldrick, 1976) program. The final R value is 0.047 and the  $R_w$  is 0.051.

The relative disposition of the three perchlorophenyl rings is like a helix and the angles between their least-squares planes are 70.9, 76.0 and 81.1°. The torsion of the C1-C2 double bond is defined by the torsion angles C8-C1-C2-C7 (34.9°) and C14-C1-C2-C3 (31.5°).

Financial support from C.S.I.C. and C.S.I.C. predoctoral fellowship of E.M. are gratefully acknowledged.



09.2-56 MOLECULAR AND CRYSTAL STRUCTURE OF AN OPEN CYCLAMPHOSPHORANE ADDUCT: BIS(BORANE)CYCLAMPHOSPHANE. By J.M. Dupart, A. Grand, S. Pace and J.G. Riess, DRF, Laboratoires de Chimie, Centre d'Etudes Nucléaires, 85 X, F.38041 Grenoble Cedex, France.

The first X-Ray structure determination of an "open tautomeric form" derivative of a tetracyclic tetraaminophosphorane, bis(borane)cyclamphosphane, 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 (1.858(5) Å) and N-B bond length (1.619(6) Å) are in the usual ranges. There is no linear correlation between the sum ( $\sum N$ ) 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-N2 bond (1.653(4) Å) is found, in spite of the distinctly pyramidal geometry of the N2 atom ( $\sum N2 = 341.6 + 3.0^\circ$ ). 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 tetraaminophosphoranes, whether uncomplexed or acting as mono or bidentate ligands towards Lewis acids.

09.2-57 THE CRYSTAL AND MOLECULAR STRUCTURE OF 4-BENZYLIDENE- $\beta$ -BENZOOXEPANE-5-ONE

A.Katrusiak, M.Ratajczak-Sitarz and Z.Kažuski, Institute of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland.

This work is the continuation of our previous X-ray studies on unsaturated ketones /Z.Kažuski et al., Bull.Ac.Pol.:Chim., (1978), 11 A.Hoser et al., Acta Cryst. (1980), B36, 1256/. 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  $\pi$ -electron system and the influence of strains in the molecule on its conformation.

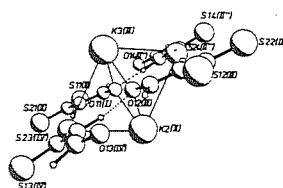
Crystals of the title compound were obtained by slow evaporation of its ethanol solution. The measurement was carried out on a SYNTeX P<sub>2</sub> diffractometer using graphite monochromated CuK $\alpha$ . The crystals are orthorhombic, space group P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>,  $a = 11.208(1)$ ,  $b = 12.828(2)$ ,  $c = 8.987(1)$  Å,  $D_x = 1.29$  Mg/m<sup>3</sup>,  $Z = 4$ .

The structure was solved by direct methods with 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-enone 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.

09.2-58 CRYSTAL STRUCTURE OF POTASSIUM-N-FORMYLDITHIOCARBAMATE. A SUPERSTRUCTURE BASED ON AN H-BOND RINGSYSTEM. By R. Gerner, G. Kiel und G. Gattow. Institut für Anorganische Chemie und Analytische Chemie. Johannes Gutenberg-Universität D-6500 Mainz. Bundesrepublik Deutschland.

We succeeded in synthesizing the title compound, which was unknown before. This compound is of high interest on chemical and structural view as well. Longtime rotation- and WEISSENBERG-photographs indicated the presence of a superstructure-cell. The superstructure is caused by hydrogen-bridge-bonds. They build up a ring of four N-formyl-dithiocarbamate-anions representing the contents of the asymmetric unit. This structure analysis, solved by means of PATTERSON-synthesis, can be dealt with as an example which helps to avoid misinterpretation of unusual structure results, such as fourier-peak-splitting, arising from unobserved superstructures.



Space group: Cc Z=16  
cell constants:  
 $a = 13.187(3)$  Å  
 $b = 12.928(2)$  Å  
 $c = 13.962(2)$  Å  
 $\beta = 101.75(3)^\circ$

$R = 3.39\%$ ,  $R_w = 3.76\%$   
for 1857 independent reflections.