
The first X-ray structure determination of an "open tautomeric form" derivative of a tetracyclic ketenophosphorane, 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', and the powder X-ray patterns confirm the presence of this stereochemistry. The bond lengths of the 1,3-enone system are in a very good agreement with those observed for hydrogen atoms and anisotropic thermal parameters in the structure of the parent compound.


We succeeded in synthesizing the title compound, which was unknown before. This compound is of high interest for 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-ions representing the contents of the asymmetric unit. This structure analysis, solved by means of PATTEN-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:

\[ a = 13.187(3) \]
\[ b = 12.926(2) \]
\[ c = 10.17(2) \]
\[ \beta = 92.9(2) \]

R = 3.93%, R1 = 3.76% for 1857 independent reflections.