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A NEW DIAMINO-DERIVATIVE OF AN INORGANIC HETEROCYCLE CONTAINING SILICON, NITROGEN, PHOSPHORUS AND OXYGEN

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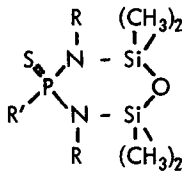
A few years ago WANNAGAT, GIESEN and RABET (1) reported the synthesis of some inorganic heterocycles containing the elements silicon, nitrogen, phosphorus and oxygen e.g. type I :

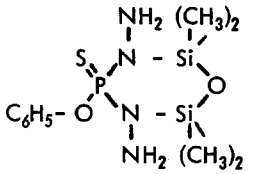


(I)

R = methyl, ethyl

R′ = ethyl

We now report the synthesis of another heterocycle of this type:



(II)

3,5-Diamino-2,2,6,6-tetramethyl-4-phenoxy-4-thioxo-1-oxa-3,5-diaza-4-phospha(λ-5)-2,6-disila-cyclohexane

II is formed by hydrolysis during the reaction of phenoxy-thiophosphoryl-dihydrazide in tetrahydrofurane with dichloro-dimethyl-silane in the presence of triethylamine:

$$C_6H_5O\text{-}(S)P(NH\text{-}NH_2)_2 + 2\ Si(CH_3)_2Cl_2 + H_2O \xrightarrow[-\ 4\ HCl]{(C_2H_5)_3N} \text{II}$$

II is obtained in small amounts by destillation of the reaction mixture at $110^{\circ}C/10^{-4}$mmHg. The pure, colourless, wax-like crystals melt at 100 - 101°C.

The constitution of II was confirmed by analysis, ^{1}H-NMR- , Mass- as well as IR- and RAMAN-spectra.

Analysis (theory in parenthesis) : C : 34.69 (34.46)% , N : 15.55(16.07)% ,

H : 6.38 (6.07)% .

The ^{1}H-NMR-spectrum (solvens $CDCl_3$) indicates the phenyl group $\delta = 7.22$ (multiplett), NH_2-groups $\delta = 3.55$ (singlett) and two different methyl groups (none-equivalent positions below and above flexible ring) $\delta = 0.26$ and $\delta = 0.16$ (singletts). The ratio of intensities corresponds very nearly to the theoretical of 5 : 4 : 6 : 6 .

TABLE 1

Some Fragments of II in the Mass-spectrum

m/e		m/e	
348	M^+	149	$O\text{-}P(N\text{-}Si)_2OH_2^+$
255	$SP(N(NH_2)Si(CH_3)_2)O^+$	132	$(CH_3)_2Si\text{-}OSi(CH_3)_2^+$
225	$SP(N(NH_2)Si(CH_3))_2O^+$	131	$P(N\text{-}Si)_2O^+$
209	$O\text{-}P(N(NH_2)Si(CH_3))_2O^+$	77	$C_6H_5^+$

TABLE 2

Main Peaks in the IR- and RAMAN-spectra of II

IR	RAMAN	Assignement	IR	RAMAN	Assignement
3335 s	3329 w	ν_{as} (NH_2)	1597 s	1595 m	ν (CC) Ar.
3265 m	3255 m	ν_s (NH_2)	1497 s		ν (CC) Ar.
2970 s	2965 m	ν_{as} (CH_3)	1198 s	1195 w	ν (P-O-Ar)
	2909 vs	ν_s (CH_3)	1173 m	1165 m	ν (P-O-Ar)
1630 w sh		δ (NH_2)	1045 vs		ν_{as} (Si-O-Si)

Reference

1. U. WANNAGAT, K. GIESEN and F. RABET, Z. anorg. allg. Chem. 382(1971) 195