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ORGANOSULPHUR COMPOUNDS-XXXVIII[1]
AXIAL PREFERENCE OF A 2-DIMETHOXYPHOSPHORYL SUBSTITUENT IN 1,3,5-TRITHIANE

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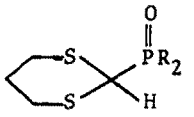
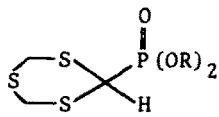
Abstract - ^{1}H-NMR studies and X-ray analysis indicate that 2-dimethoxyphosphoryl-1,3,5-trithiane exists both in solution and in the crystal in a chair conformation with the dimethoxyphosphoryl group at C-2 being axial.

Since the discovery by Lemieux et al.[2] of the tendency of an electronegative substituent at C-2 in a pyranose ring to occupy the axial position, it has become known that a strong anomeric effect also operates in 2-substituted thianes and polythianes (1,3-dithianes, 1,3,5-trithianes)[3]. Extensive studies carried out by Ōki and coworkers[4-7] showed that substituents such as benzyloxy, phenylthio, carboxy and chloro attached to the carbon atom C-2 of the thiane, 1,3-dithiane and 1,3,5-trithiane ring prefer the axial position. In some cases, low temperature ^{1}H-NMR studies revealed the presence of a minor amount of the equatorial conformer at equilibrium with the axial one. An important characteristic for the anomeric effect from the viewpoint of the ^{1}H-NMR spectroscopy is that the axial protons at C-4 and C-6 in axially 2-substituted polythianes are strongly deshielded by the anisotropy effect of the axial bond. Consequently, they are located at lower field than the corresponding equatorial protons, the difference in chemical shifts between the axial and equatorial protons at C-4 and C-6 amounting to 1.20 ppm or more.

Recently, Juaristi at al.[8] have found that 2-diphenylphosphinyl-1,3-dithiane (1), both in solution and in the solid state, adopts a chair conformation with the diphenylphosphinyl group at C-2 being axial. A full analysis of the 200 and 400 MHz ^{1}H-NMR spectra of the structurally related 2-dimethoxyphosphoryl-1,3-dithiane (2) has led us[9] to the similar conclusion that the dimethoxyphosphoryl group in 2 is axial.





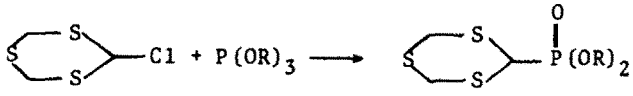
1, R = Ph
2, R = OCH_3

3, R = CH_3
3-d_6, R = Cd_3

Since the diphenylphosphinyl and dimethoxyphosphoryl groups containing tetrahedral phosphorus are bulky substituents, the operation of a strong anomeric effect in 1 and 2 was rather unexpected but interesting especially with regard to the nature of this effect. To obtain more data on the structure of the phosphorylated polythianes, 2-dimethoxyphosphoryl-1,3,5-trithiane (3) was prepared and its solution and solid state conformations were elucidated with the aid of ^{1}H-NMR-spectroscopy and X-ray diffraction, respectively. This paper describes the results obtained.

RESULTS AND DISCUSSION

2-Dimethoxyphosphoryl-1,3,5-trithiane (3) was prepared according to our earlier described procedure[10] by treatment of 2-chloro-1,3,5-trithiane with trimethyl phosphite. The pure product obtained in 70% yield after column chromatography has mp 113-114°C. The use of trimethyl phosphite-d_9 in this reaction afforded the hexadeuterium analogue, 3-d_6.



3, R = CH_3
3-d_6, R = Cd_3

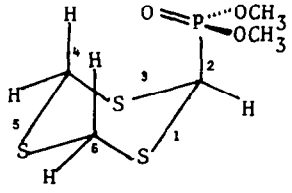
The ^{1}H-NMR spectrum of 3 measured at 90 MHz in $CDCl_3$ solution shows four groups of signals at δ = 3.57, 3.89, 3.91 and 5.20 ppm. That the two middle doublets centered at δ = 3.89 and 3.91 ppm with characteristic coupling constants $^2J_{H-P}$ = = 20.0 Hz and $^3J_{H-P}$ = 10.8 Hz correspond to the methine proton at C-2 and the P-methoxy protons, respectively was confirmed univocally by phosphorus decoupling experiments and comparison of the ^{1}H-NMR spectrum of 3 with that of 3-d_6. Therefore, the remaining two signals at δ = 3.57 and 5.20 ppm must be attributed to the equatorial and axial methylene protons in 3. It should be emphasized that the chemical shift difference, $\Delta_{a,e}$, between these two signals is 1.63 ppm. Such a big difference is characteristic for the anomeric effect and, in accord with all the available literature data, it was assumed that the equatorial protons at C-4 and C-6 absorb at δ = = 3.57 ppm whereas the axial protons resonate at lower field i.e. at δ = 5.20 ppm, as a consequence of the deshielding effect of the axial dimethoxyphosphoryl group in 3. For comparison, the corresponding values, $\Delta_{a,e}$, for 2-phosphoryl-1,3-dithianes 1 and 2 are 1.20 ppm (at 90 MHz) and 1.64 ppm (at 200 MHz), respectively. In the case of 2-phenylthio-1,3,5-trithiane, for which both conformers were detected in the low temperature ^{1}H-NMR spectra[5], the value $\Delta_{a,e}$ = 1.60 ppm was recorded for the conformer with the axial phenylthio group whereas that for the equatorial conformer, 0.45 ppm, was much smaller.

Analysis of a splitting pattern of the methylene proton resonance signals in 3 provides further support for our assignment. In this connection, it should be noted that the best resolved ^{1}H-NMR spectra of 3 and 3-d_6 were obtained in CD_2Cl_2 solution. In this solvent the low-field signal at δ = 5.15 ppm appears as a doublet of doublets with a big geminal coupling constant, $^2J_{H-H}$ = 14.6 Hz, and a small one, $^4J_{H-P}$=2.8 Hz, due to coupling with phosphorus over four bonds. On the other hand,

the high-field signal at δ = 3.61 ppm consists of a doublet of triplets with the same geminal coupling constant $^{2}J_{H-H}$ = 14.6 Hz. In the ^{1}H-NMR spectrum with ^{31}P-decoupling this signal is a doublet of doublets with a small coupling constant equal to 0.9 Hz, This results is best explained by ascribing the high-field signal to the equatorial methylene proton at C-4 or C-6 which couples with the methine proton at C-2 (long range W-type coupling) and with phosphorus with the same coupling constant, $^{4}J_{H-H}$ = $^{4}J_{H-P}$ = 0.9 Hz. Accordingly, both parts of the doublet due to the methine proton at C-2 show a further splitting.

The observation of long-range coupling, which makes all the ring proton signals a little more complex than indicates above, strongly suggests that the trithiane ring in 3 exists largely, if not exclusively, in one conformation with the dimethoxyphosphoryl group in the axial position. This is additionally confirmed by temperature invariance of the ^{1}H-NMR spectrum of 3-d_6 (between +20° and -90° in CD_2Cl_2 and +20° - +95° in dioxane).There was neither decoalescence at low temperature nor any appreciable change in chemical shift with temperature, as would be expected if there existed in palpable equilibrium between two conformations.

In further support of our spectral and configurational assignments a simulation of the ^{1}H-NMR spectrum of 3-d_6 was carried out. Both the experimental spectrum of 3-d_6 at 90 MHz in a CD_2Cl_2 solution and the simulated one are shown in Figure 1. The best accordance of the simulated with the experimental spectrum (at an assumed half-line width of 0.4 Hz)was obtained for the following coupling constants:



$J(P,H_{6a})$ = 2.8 $J(H_{6a},H_{6e})$ = 14.6 $J(H_{6e},H_{4a})$ = 0.4* $J(H_{4a},H_{4e})$ = 14.6
$J(P,H_{6e})$ = 0.9 $J(H_{6a},H_{4a})$ = 0.0* $J(H_{6e},H_{4e})$ = 0.9 $J(H_{4a},H_{2e})$ = 0.6*
$J(P,H_{4a})$ = 2.8 $J(H_{6a},H_{4e})$ = 0.4* $J(H_{6e},H_{2e})$ = 0.9 $J(H_{4e},H_{2e})$ = 0.9
$J(P,H_{4b})$ = 0.9 $J(H_{6a},H_{2e})$ = 0.6*
$J(P,H_{2e})$ = 20.5

It should be noted that some values of the long-range coupling constants(marked with an asterisk) which could not be estimated from our experimental spectra were taken from the simulated spectrum of 2.

As part of the present work, the effect of solvent on the chemical shift difference between the axial and equatorial methylene protons in 3 was also investigated. The results are summarized in Table 1.

Table 1. Solvent Effect on the Chemical Shift Difference, $\Delta_{a,e}$, for the Methylene Protons in 3

Solvent	δ_{eq} (ppm)	δ_{ax} (ppm)	$\Delta_{a,e}$
$(CD_3)_2SO$	3.98	4.87	0.89
$(CD_3)_2CO$	3.79	5.17	1.38
CD_2Cl_2	3.61	5.15	1.54
Dioxane-D_8	3.61	5.18	1.57
$CDCl_3$	3.57	5.20	1.63

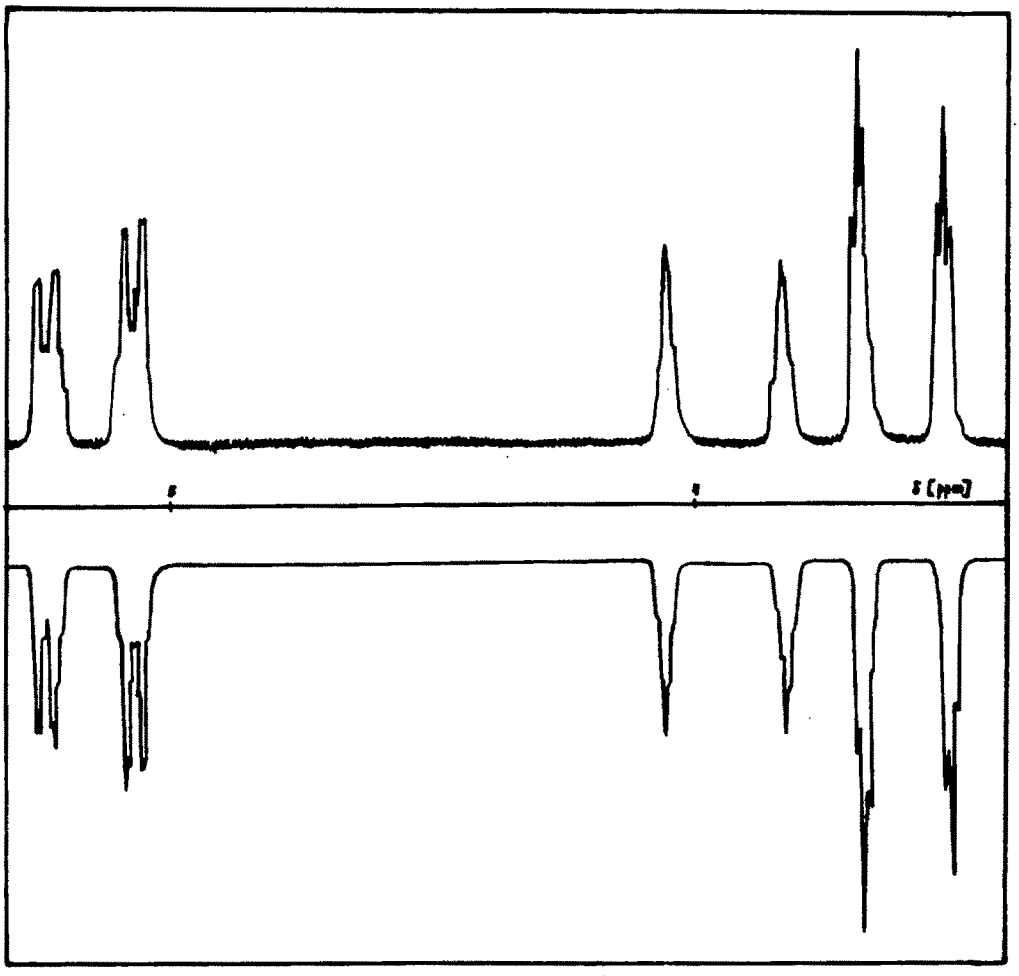


Fig.1. The observed (top) and simulated (bottom) 90 MHz proton spectrum of 2-dimethoxy-d_6-phosphoryl-1,3,5-trithiane (3-d_6).

It is of interest to note that in DMSO the $\Delta_{a,e}$ value is considerably smaller than values observed in other solvents. It was also found that the chemical shift of the methine proton at C-2 in 3 is strongly affected by DMSO as solvent. Thus, whereas the methine proton resonates at 3.91 ppm in $CDCl_3$, in DMSO it is located at δ = 4.86 ppm. These preliminary observations suggest that solvent effect should be taken into account if one compares the NMR spectral data of the phosphorylated polythianes.

Clear-cut evidence for the axial position of the dimethoxyphosphoryl group in 3 was provided by X-ray analysis. The solid state structure of 3 with the numbering system† is shown in Fig.2. The final positional parameters with equivalent temperature factors for nonhydrogen atoms are listed in Table 2††. The bond lengths and angles are given in Table 3.

The six-membered 1,3,5-trithiane ring in 3 adopts a chair conformation which was found to be slightly flattened at the carbon atom C(1) bonded to the axial dimethoxyphosphoryl group. This flattening is illustrated by deviation of the atom

† It should be noted that the numbering system shown in Fig.2 and used in crystallographic analysis is different from that based on chemical nomenclature.

†† The structure factors, anisotropic thermal parameters and positional parameters of hydrogen atoms are deposited with the British Library Lending Division as supplementary publication.

C(1) and S(2) from the plane (*i*) (see Table 4). The sulphur atom S(2) is 0.17 Å more off this plane than the carbon atom C(1). The angle between the normal to this plane (*i*) and the axial P-C(1) bond is 12.3(5)°. The corresponding value for the equatorial C(1)-H bond is equal to 56.9(5)°.

In addition to the most important finding that the dimethoxyphosphoryl group is axial, it was found that the phosphoryl oxygen atom O(1) is almost symmetrically situated above the 1,3,5-trithiane ring. It is 3.378(3) and 3.538(3)Å away from the sulphur atom S(1) and S(3), respectively. The distances between phosphoryl oxygen and the endocyclic carbon atom C(2) and C(3) i.e. O(1)···C(2) and O(1)···C(3) have values of 3.252(4) and 3.256(4)Å, respectively. The corresponding contacts between phosphoryl oxygen and axial hydrogens O(1)···H-(1) of 3.14(3)Å and O(1)···H-C(3) of 2.62(3)Å are observed. According to Taylor and Kennard[11], who have discussed in detail the crystallographic evidence and criteria for the existence of C-H···O hydrogen bonds, these contacts are too long to be classifying as the nearest-neighbour contacts and can not be described as hydrogen bonds (the *d* value in our case is smaller than 0.3Å). However, it is not possible to exclude definitively that some weak electrostatic, attractive interactions between the phosphoryl oxygen atom and axial hydrogens exist, which, in addition to the anomeric effect, can stabilize the axial conformer of 3. Finally, it should be noted that there are no contacts between the molecules of 3 shorter than the sum of van der Waals radii.

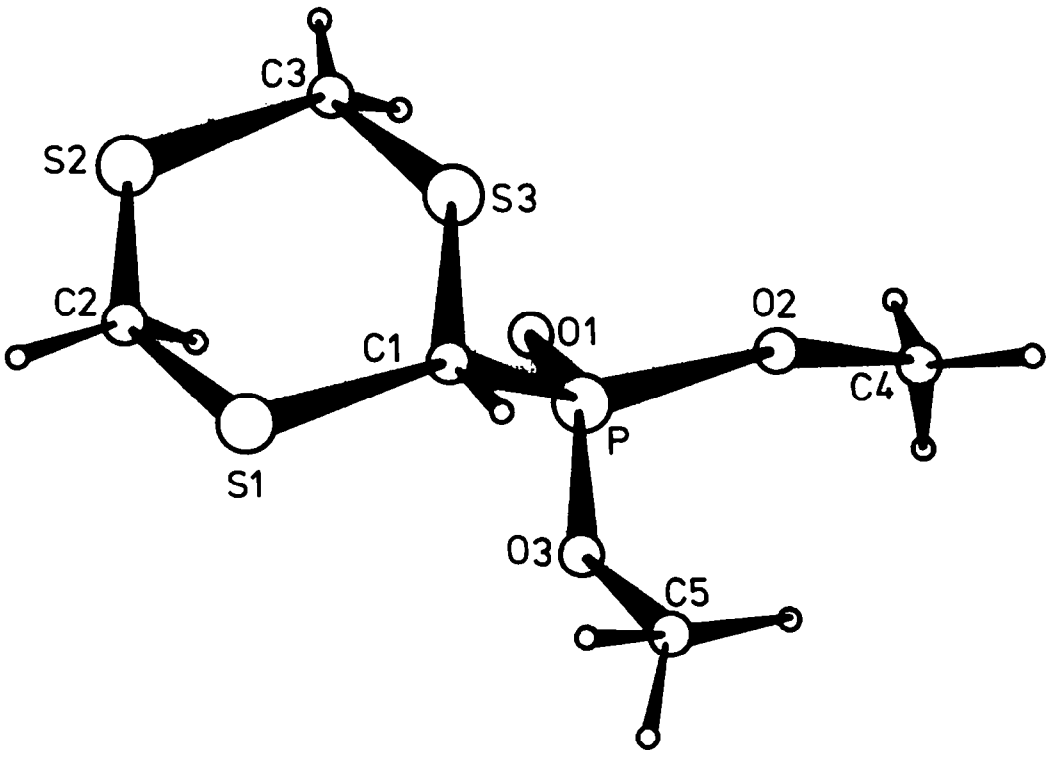


Fig. 2. Atom numbering scheme and solid state conformation of 2-dimethoxyphosphoryl--1,3,5-trithiane (3).

The X-ray crystallographic data of 3 gave no evidence, however, that the observed preference of the dimethoxyphosphoryl group for the axial position in 1,3,5-trithiane is due to a lone pair-antibonding orbital interaction (n_S-δ^*_{C-P}), considered to be the basis of the anomeric effect[3].

The observed lengths for the sulphur-anomeric carbon bonds of 1.806(3) and 1.813(3)Å and the anomeric carbon-phosphorus bond of 1.812(3)Å are normal and the expected bond length changes characteristic of the anomeric effect are not occuring. Interestingly, a similar situation was also observed by Juaristi et al.[8] in the case of 1. Therefore, the rationalization of the anomeric effect in polythianes bearing a phosphoryl substituent at the anomeric carbon atom is still an open question and needs further studies.

Table 2. Positional parameters (x 10^4) and equivalent U values (x 10^3) for the nonhydrogen atoms in 3

$$U_{eq} = \frac{U_{11}+U_{22}\sin^2\beta+U_{13}\cos\beta}{3(1-\cos^2\beta)}$$

	X	Y	Z	U
P	1015(1)	3223(1)	1655(1)	30(0)
S(1)	4553(2)	4043(1)	3586(1)	42(0)
S(2)	1399(2)	5567(1)	3507(1)	46(0)
S(3)	2708(2)	4928(1)	1256(1)	44(0)
O(1)	-1273(4)	3434(2)	2022(2)	43(1)
O(2)	843(4)	3119(2)	298(2)	46(1)
O(3)	1984(4)	2393(1)	2229(2)	43(1)
C(1)	3445(5)	3970(2)	2021(3)	32(1)
C(2)	2025(6)	4550(2)	4051(3)	41(1)
C(3)	366(6)	5337(2)	1987(3)	44(1)
C(4)	-1129(8)	2631(3)	-353(3)	63(1)
C(5)	4011(7)	1953(2)	1902(4)	58(1)

Table 2a. Anisotropic temperature factors (Å^2 x 10^3) in the form, $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}lhc^*a^* + 2U_{12}hka^*b^*)]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
P	21(0)	35(0)	34(0)	-7(0)	7(0)	-1(0)
S(1)	32(0)	46(0)	42(0)	-8(0)	-5(0)	6(0)
S(2)	50(1)	36(0)	57(1)	-9(0)	21(0)	0(0)
S(3)	48(1)	44(0)	44(0)	3(0)	18(0)	-3(0)
O(1)	21(1)	52(1)	61(2)	-12(1)	15(1)	-2(1)
O(2)	48(1)	57(2)	35(1)	-11(1)	10(1)	-13(1)
O(3)	39(1)	37(1)	55(1)	-1(1)	13(1)	2(1)
C(1)	20(1)	39(2)	37(1)	-6(1)	7(1)	-1(1)
C(2)	49(2)	41(2)	36(2)	-2(1)	17(1)	-1(1)
C(3)	43(2)	40(2)	49(2)	2(2)	7(2)	7(1)
C(4)	73(3)	67(3)	44(2)	-14(2)	-3(2)	-24(2)
C(5)	33(2)	46(2)	93(3)	-9(2)	11(2)	9(2)

Table 3. Bond lengths and angles in 3

Bond length (Å)

O(1)-P	1.465(2)	O(2)-P	1.572(2)
O(3)-P	1.565(2)	C(1)-P	1.812(3)
C(1)-S(1)	1.806(3)	C(2)-S(1)	1.809(3)
C(2)-S(2)	1.794(4)	C(3)-S(2)	1.788(4)
C(1)-S(3)	1.813(3)	C(3)-S(3)	1.823(4)
C(4)-O(2)	1.446(4)	C(5)-O(3)	1.454(4)

Bond angles (°)

O(2)-P -O(1)	116.0(1)	O(3)-P -O(1)	109.1(1)
O(3)-P -O(2)	106.3(1)	C(1)-P -O(1)	115.1(1)
C(1)-P -O(2)	101.7(1)	C(1)-P -C(3)	108.1(1)
C(2)-S(1)-C(1)	101.7(1)	C(3)-S(2)-C(2)	98.8(2)
C(3)-S(3)-C(1)	101.7(1)	C(4)-O(2)-P	118.3(2)
C(5)-O(3)-P	122.7(2)	S(1)-C(1)-P	111.8(2)
S(3)-O(1)-P	112.7(2)	S(3)-C(1)-S(1)	115.8(2)
S(2)-C(2)-S(1)	115.5(2)	S(3)-C(3)-S(2)	114.9(2)

Table 4. Geometry of the 1,3,5-trithiane ring in 3

a. Torsion angles

S(1)-C(1)-S(3)-C(3)	-59.0(5)	C(3)-S(2)-C(2)-S(1)	65.9(5)
C(1)-S(3)-C(3)-S(2)	64.5(5)	S(2)-C(2)-S(1)-C(1)	-62.2(5)
S(3)-C(3)-S(2)-C(2)	-67.2(5)	C(2)-S(1)-C(1)-S(3)	58.4(5)

b. Planes

(*i*) Plane containing atoms denoted with asterisk

3.0008X + 12.7841Y + 2.4071Z = 7.4059

C(1)	-0.808(10)	C(3)*	-0.008(10)	S(2)	0.974(3)
C(2)*	0.008(10)	S(1)	-0.007(3)	S(3)*	0.007(3)

(*ii*) Plane containing atoms: C(1), S(1), S(3)

5.5000X + 1.2314Y + 3.8794Z = 1.6093

(*iii*) Plane containing atoms: C(2), C(3), S(2)

5.4981X + 1.2861Y + 3.8843Z = 0.1223

c. Angles between normals to planes (°)

(*i*) (*ii*) = 56.9(5)
(*i*) (*iii*) = 56.7(5)

EXPERIMENTAL

^{1}H-NMR spectra were recorded at 80 and 90 MHz on a Tesla and Bruker HFX-72 instrument, respectively. Tetramethylsilane was used as internal standard. Simulated ^{1}H NMR spectra were obtained on a Bruker HFX-72 spectrometer at 80 and 90 MHz joined with a Nicolet B-NC-12 minicomputer using NMRCAL program. ^{13}C NMR spectra were obtained on a Bruker HFX-72 instrument. ^{31}P NMR spectra were measured on a Jeol JNM-FX 60 Fourier transform spectrometer at 24.3 Hz with 85% H_3PO_4 as external standard. Mass spectra were recorded with a LKB 2091 mass spectrometer.

2-Dimethoxyphosphoryl-1,3,5-trithiane (3). To a stirred suspension of trithiane (4.15 g, 0.03 mol) in dry benzene (100 ml) powdered N-chlorosuccinimide (4.4 g, 0.033 mol) was added portionwise at room temperature (water bath). Stirring was continued at this temperature for 2 hr and the reaction mixture was left to stand overnight. Then, a solution of trimethyl phosphite (3.84 g, 0.031 mol) in dry benzene (20 ml) was added at room temperature and the reaction mixture was heated with stirring for 2-3 hr at 70°C. After cooling to room temperature and filtration, the benzene solution was washed twice with water (40 ml), dried over $MgSO_4$ and evaporated. The residue was chromatographed on silica gel with benzene/hexane (50/1) as eluent to afford 3; 5.2 g (71%); m.p. 113-114° (Found: C,24.39;H, 4.61; S, 38.93. Calc.for $C_5H_{11}O_3PS_3$ (246.31)C, 24.38; H, 4.50; S, 39.05%); ^{31}P NMR (δ): 17.9 (benzene), 18.8 ($CHCl_3$) ppm; ^{13}C NMR (δ,$CDCl_3$): 30.09 (s,$\underline{CH_2}$S), 35.26 (d, J_{P-C}=157.4 Hz, $\underline{CH}$P), 54.08 (br.s,$\underline{CH_3}$O) ppm; MS-70eV (m/e%): 246(21), 169(22), 155 (100), 124(44), 109(34), 91(21), 93(22), 79(35), 45(99), 46(37), 47(30).

2-Dimethoxy-d_6-phosphoryl-1,3,5-trithiane (3-d_6). It was obtained according to the procedure described above starting from trimethyl phosphite-d_9 (4.12 g, 0.031 mol); 5.3 g (70%); m.p. 113-114° (Found: C, 23.81; H + D, 6.96; S, 38.17. Calcd. for $C_5H_5D_6O_3PS_3$ (252.35) C, 23.81, H + D, 6.78; S, 38.12%, MS-70eV (m/e%): 252(29), 175(27), 161(100), 130(32), 115(43), 91(22), 99(25), 45(94), 46(48),47(26).

X-Ray structure determination of 3.

Crystal data: $C_5H_{11}O_3PS_3$. M = 246.31. Monoclinic, $P2_1/c$, a = 5.5692(4), b = = 16.4152(13), c = 11.6423(11) Å, β = 100.553(6)°, V = 1042.79(15)Å^3, D_x = 1.578 g·cm^{-3}, Z = 4, F(000) = 512, μ($M_oK\alpha$) = 7.53 cm^{-1}, λ = 0.071069 Å.

Crystallographic measurements and structure analysis. Suitable crystals of 3 were obtained from aceton-n-hexane solution. The cell dimensions were preliminarily obtained from Weissenberg photographs and were refined by least-squares method on the basis of diffractometric measurements. Intensities were measured for a crystal of

approximate dimensions 0.2 x 0.2 x 0.3 mm sealed into a Lindemann glass capillary tube. The intesity data were collected on a Hilger-Watts four-circle diffractometer with $M_oK\alpha$ radiation (λ = 0.71069 Å). The θ-2θ scan mode was used for $2\theta < 60^o$. Three reflections were used as standards and remeasured during the data collection; no crystal decomposition was detected. Of 2086 reflections measured, 1854 were classifying as observed, i.e. $F > 8\sigma(F)$. Only Lorentz-polarization corrections were applied.

The structure was solved by the MULTAN-76 program. The coordinates of all non-hydrogen atoms from the E-map were used as the starting parameters and the structure was refined by full-matrix least-squares (SHELX 76), first isotropically, then with non-hydrogen atoms anisotropically. Hydrogen atoms were located in a difference map in the expected position. The isotropic thermal parameters of hydrogen atoms were taken initially as 1.5 of isotropic parameters for their parent-carbon atoms. The positions and isotropic parameters of H-atoms were refined (12 reflections per parameter) at convergence, R = 0.035 for 1854 reflections observed. One reflection (020) was suffering badly from extinction and was omitted from the refinement. Weights were proportional to $1/\sigma(F)$. The maximum changes in the parameter was $(\Delta/\delta)_{max}$=0.055.

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