Heteroatom Chemistry
Volume 9, Number 6, 1998

Crystal and Molecular Structure of Diastereomeric 2-Diphenylthiophosphinoyl-*cis*-(4,6-dimethyl)-1,3-dithianes

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Received 22 September 1997; revised 31 December 1997

ABSTRACT: *The crystal and molecular structures of a pair of diastereomeric 2-diphenylthiophosphinoyl-cis-(4,6-dimethyl)-1,3-dithianes* 2 *have been determined by the X-ray method. The differences in corresponding bond distances in cis-*2 *and trans-*2 *are discussed in context of the anomeric effect operating in this heterocyclic system. The comparison of selected structural parameters between diastereomeric cis- and trans-dithianes* 2 *and their 5-t-butyl analogs* 1 *is also discussed.* © 1998 John Wiley & Sons, Inc. Heteroatom Chem 9:537–541, 1998

INTRODUCTION

The S–C–P anomeric interactions attracted recently considerable attention, and their nature is still a matter of debate. Although it is accepted on the basis of accumulated data that the n_s-$\sigma^*_{\text{C-P}}$ negative hyperconjugation contributes importantly to the anomeric effect observed in 2-phosphorus substituted 1,3-dithianes, their structural parameters, especially the C–S and C–P bond lengths, are not fully consistent with the operation of this stereoelectronic effect. An inspection of selected bond lengths in *cis*- and *trans*-2-phosphoryl, 2-thiophosphoryl-, and 2-selenophosphoryl-5-*t*-butyl-1,3-dithianes **1** revealed that the C(2)–P bond lengths in diastereomers are not characteristic for the position of the phosphorus and, at first sight, are not consistent with the n_s-$\sigma^*_{\text{C-P}}$ negative [1,2] hyperconjugation.

cis - **1**

a, X = O
b, X = S
c, X = Se

trans - **1**

This is due to the operation of other effects such as, for example, n_s–n_x repulsive interactions. Having in our hands *cis*- and *trans*-2-diphenylthiophosphinoyl-*cis*-(4,6-dimethyl)-1,3-dithianes **2**, we decided to establish their structures by means of X-ray crystallography in the hope that the comparison of structural parameters of the diastereomers of **2** with the axial and equatorial phosphorus substituent will reveal the characteristic features that could be attributed to the negative hyperconjugation.

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Financial support by the State Committee for Scientific Research (KBN) Grant No. 3 T09 088 10.

CCC 1042-7163/98/060537-05

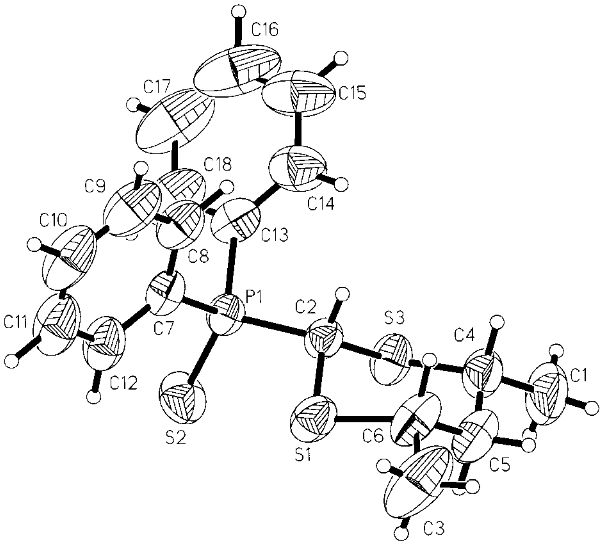


FIGURE 1 Thermal ellipsoidal view with the atom-numbering scheme of the molecule of *cis*-**2**. Ellipsoids are shown with 50% probability.

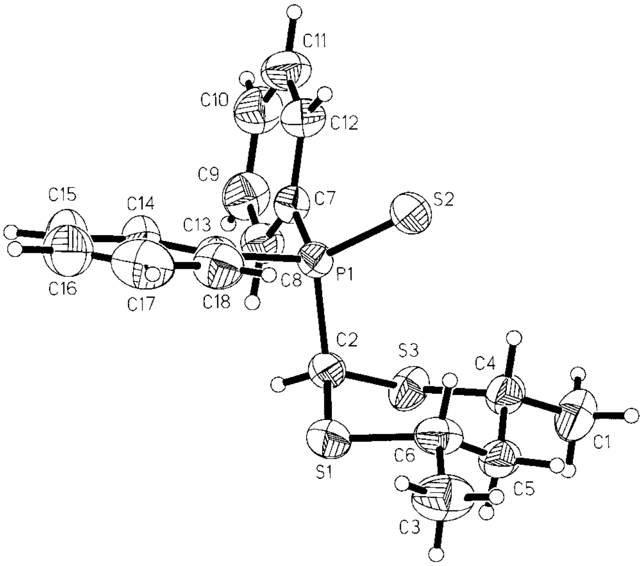
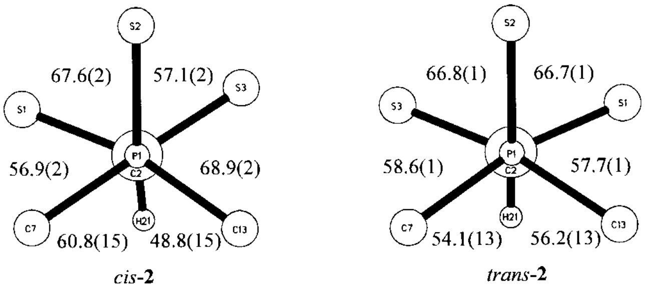


FIGURE 2 Thermal ellipsoidal view with the atom-numbering scheme of the molecule of *trans*-**2**. Ellipsoids are shown with 50% probability.



cis-**2** *trans*-**2**

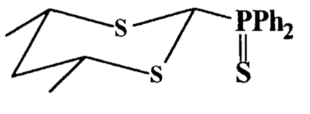
FIGURE 3 The Newman projections perpendicular to the C2–P1 bond.

TABLE 1 Crystal Data and Experimental Details

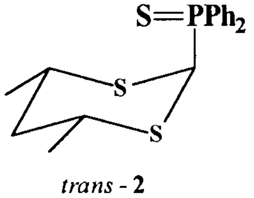
		cis-**2**	*trans*-**2**
Molecular formula		$C_{18}H_{21}PS_3$	$C_{18}H_{21}PS_3$
Formula weight		364.50	364.50
Crystal system		orthorhombic	monoclinic
Space group		*Pna*2_1	*P*2_1/*n*
a (Å)		10.889(3)	9.0173(8)
b (Å)		10.820(5)	20.3408(14)
c (Å)		16.280(5)	10.3519(6)
β (°)		90.00	100.822(6)
V (Å^3)		1918.1(12)	1865.0(2)
Z		4	4
D_c (g/cm^3)		1.262	1.298
μ (cm^3)		42.59	43.80
Crystal dim. (mm)		04 × 0.4 × 0.5	0.07 × 0.09 × 0.5
Maximum 2θ (°)		150	150
Radiation, λ (Å)		CuKα, 1.54178	CuKα, 1.54178
Scan mode		$\omega/2\theta$	$\omega/2\theta$
Scan width (°)		0.70 + 0.14 tanθ	0.70 + 0.14 tanθ
hkl ranges:	*h* =	−13 0	0 11
	k =	0 13	−25 0
	l =	−20 20	−12 12
EAC corr.:	min	0.9296	0.9545
	max.	0.9995	0.9987
	ave.	0.9600	0.9757
No. of refl.: unique		3942	3833
with $I > 0\sigma(I)$		3876	3560
with $I > 2\sigma(I)$		3782	3229
No. of ref. param.		238	284
Largest peak (eÅ^{-3})		0.194	0.290
Largest hole (eÅ^{-3})		−0.244	−0.289
Shift/esd_max		−0.001	−0.001
R_{obs}		0.0383	0.0297
wR_{obs}		0.1060	0.0805
S_{obs}		1.091	1.071
Wght coeff.*	*m*	0.0538	0.0484
	n	0.6716	0.3153
Exti. coeff.*^{b}*	*k*	0.0042(3)	0.003(0)
Flack χ [8]		0.00(2)	—
R_{int}		0.0000	0.0136
$T_{meas.}$		293(2)	293(2)
F_(000)		768	768

[a] Weighting scheme $w = [\sigma^2 (Fo^2) + (mP)^2 + nP]^{-1}$, where $P = (Fo^2 + 2Fc^2)/3$.

[b] Extinction method SHELXL, extinction expression $Fc^* = kFc[1 + 0.001 \times Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$.



cis - **2**



trans - **2**

RESULTS AND DISCUSSION

An overall view of the dithiane *cis*-2 with the atom numbering is shown in Figure 1. Figure 2 shows the solid-state conformation of its isomer *trans*-2. In Table 4, the selected bond lengths for both diastereomeric dithianes 2 are collected. Table 5 contains bond angles for nonhydrogen atoms of *cis*- and

TABLE 2 Fractional Atomic Coordinates and Equivalent Isotropic Displacement Parameters (Å²) for *cis*-**2** $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j$

	x	*y*	*z*	U_{eq}
P1	0.12518(7)	0.60390(6)	0.9694	0.0542(2)
S1	0.21770(9)	0.63098(8)	0.79923(7)	0.0735(2)
S2	−0.03788(8)	0.54298(9)	0.94102(9)	0.0805(3)
S3	0.08276(8)	0.83975(7)	0.88228(7)	0.0712(2)
C1	0.0913(5)	1.0477(4)	0.7937(4)	0.105(2)
C2	0.1873(3)	0.7122(3)	0.8940(2)	0.0558(6)
C3	0.3336(7)	0.7051(7)	0.6631(3)	0.138(3)
C4	0.1715(4)	0.9346(3)	0.8120(3)	0.0779(10)
C5	0.2063(4)	0.8676(4)	0.7341(3)	0.0866(11)
C6	0.2911(4)	0.7580(5)	0.7452(2)	0.0859(11)
C7	0.2411(3)	0.4847(3)	0.9765(2)	0.0584(6)
C8	0.3539(3)	0.5100(4)	1.0119(2)	0.0742(9)
C9	0.4409(5)	0.4172(5)	1.0185(3)	0.0957(14)
C10	0.4138(5)	0.2998(5)	0.9921(3)	0.100(2)
C11	0.3044(5)	0.2747(4)	0.9556(3)	0.0905(12)
C12	0.2152(4)	0.3666(3)	0.9468(2)	0.0751(9)
C13	0.1323(3)	0.6811(3)	1.0682(2)	0.0652(8)
C14	0.2064(4)	0.7818(4)	1.0839(3)	0.0869(11)
C15	0.2152(6)	0.8308(6)	1.1624(3)	0.113(2)
C16	0.1509(7)	0.7777(9)	1.2250(3)	0.134(3)
C17	0.0772(7)	0.6775(9)	1.2121(3)	0.130(3)
C18	0.0678(5)	0.6307(6)	1.1325(3)	0.0946(13)

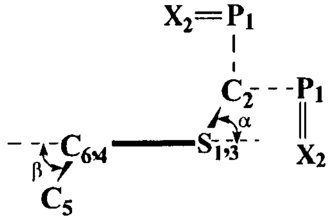
TABLE 3 Fractional Atomic Coordinates and Equivalent Isotropic Displacement Parameters (Å²) for *trans*-**2** $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j$

	x	*y*	*z*	U_{eq}
P1	0.16092(5)	0.83962(2)	0.90464(4)	0.03622(12)
S1	0.21358(6)	0.93521(2)	0.69147(5)	0.05226(14)
S2	0.12445(6)	0.90346(2)	1.03546(4)	0.05201(14)
S3	−0.10046(5)	0.87647(2)	0.68318(4)	0.04736(13)
C1	−0.3054(3)	0.97042(13)	0.6939(3)	0.0641(6)
C2	0.1021(2)	0.86716(8)	0.7325(2)	0.0405(4)
C3	0.2103(4)	1.06623(13)	0.7084(3)	0.0745(7)
C4	−0.1371(2)	0.95877(9)	0.7416(2)	0.0461(4)
C5	−0.0430(2)	1.01128(9)	0.6918(2)	0.0485(4)
C6	0.1237(2)	1.00745(9)	0.7486(2)	0.0505(4)
C7	0.0667(2)	0.76165(8)	0.9172(2)	0.0387(3)
C8	0.0055(2)	0.72293(9)	0.8092(2)	0.0476(4)
C9	−0.0645(3)	0.66409(10)	0.8277(2)	0.0557(5)
C10	−0.0730(3)	0.64321(10)	0.9523(2)	0.0590(5)
C11	−0.0110(3)	0.68070(11)	1.0601(2)	0.0578(5)
C12	0.0579(2)	0.73977(10)	1.0425(2)	0.0479(4)
C13	0.3592(2)	0.81807(9)	0.9201(2)	0.0447(4)
C14	0.4026(2)	0.75447(11)	0.8950(2)	0.0589(5)
C15	0.5543(3)	0.7391(2)	0.9054(3)	0.0775(7)
C16	0.6610(3)	0.7859(2)	0.9385(3)	0.0809(8)
C17	0.6212(3)	0.8485(2)	0.9654(3)	0.0771(8)
C18	0.4695(3)	0.86531(13)	0.9573(2)	0.0602(5)

TABLE 4 Bond lengths (Å) for Nonhydrogen Atoms

		cis-**2**	*trans*-**2**
P1	S2	1.9494(12)	1.9483(6)
P1	C2	1.827(3)	1.848(2)
P1	C7	1.809(3)	1.815(2)
P1	C13	1.814(3)	1.818(2)
S1	C2	1.806(3)	1.808(2)
S1	C6	1.817(4)	1.829(2)
S3	C2	1.799(3)	1.812(2)
S3	C4	1.816(4)	1.831(2)
C1	C4	1.533(5)	1.524(3)
C3	C6	1.526(7)	1.528(3)
C4	C5	1.509(6)	1.514(3)
C5	C6	1.514(6)	1.510(3)
C7	C12	1.394(5)	1.387(2)
C7	C8	1.384(5)	1.394(2)
C8	C9	1.385(5)	1.383(3)
C9	C10	1.373(8)	1.374(3)
C10	C11	1.359(7)	1.380(3)
C11	C12	1.397(5)	1.380(3)
C13	C18	1.374(6)	1.385(3)
C13	C14	1.379(6)	1.390(3)
C14	C15	1.387(6)	1.388(3)
C15	C16	1.364(10)	1.350(4)
C16	C17	1.366(10)	1.367(4)
C17	C18	1.396(8)	1.397(4)

trans-**2**, and Table 6 contains torsional angles for both structures. An inspection of Figure 1 and the data from Table 6 reveals that the 1,3-dithiane ring in *cis*-**2** is almost symmetrical with the mirror plane including the C2 and C5 atoms. Its conformation represents a deformed chair, while the conformation of the 1,3-dithiane ring in *trans*-**2** is almost an ideal chair. Compound *trans*-**2** shows also more symmetrical distribution of atoms around the C2–P1 in comparison to *cis*-**2**, as it is visible from the Newman projections for both isomers (Figure 3).



The comparison of the dihedral angles α (Plane 1/Plane 2 in Table 9) in the 1,3-dithiane rings for both isomers reveals that the ring folding is greater in the isomer *cis*-**2**. This tendency exists also when we compare isomers *cis* and *trans* in compounds **1a**, **1b**, and **1c**.

The bond angles in the 1,3-dithiane ring of *trans*-**2** are distinctly larger than in *cis*-**2**. These differences are shown as follows:

TABLE 5 Bond Angles (°) for Nonhydrogen Atoms

			cis-**2**	*trans*-**2**
C2	P1	S2	113.24(11)	114.38(6)
C7	P1	S2	114.18(12)	112.96(6)
C13	P1	S2	113.89(12)	113.02(6)
C7	P1	C2	104.02(13)	106.29(8)
C13	P1	C2	106.5(2)	104.92(8)
C13	P1	C7	104.00(14)	104.39(8)
C2	S1	C6	97.3(2)	103.85(8)
C2	S3	C4	99.4(2)	103.72(8)
S1	C2	S3	113.5(2)	115.43(9)
S1	C2	P1	109.2(2)	112.78(9)
S3	C2	P1	109.2(2)	113.29(9)
C5	C4	C1	111.3(4)	111.6(2)
C5	C4	S3	113.0(3)	112.65(13)
C1	C4	S3	105.7(3)	105.40(14)
C4	C5	C6	115.5(3)	114.3(2)
C5	C6	C3	112.0(4)	112.1(2)
C5	C6	S1	112.4(3)	112.61(13)
C3	C6	S1	105.9(4)	105.1(2)
C12	C7	C8	120.4(3)	119.0(2)
C12	C7	P1	119.3(3)	117.21(14)
C8	C7	P1	120.3(3)	123.79(13)
C9	C8	C7	119.7(4)	120.0(2)
C8	C9	C10	120.0(5)	120.3(2)
C11	C10	C9	120.7(4)	120.2(2)
C10	C11	C12	120.8(4)	119.9(2)
C7	C12	C11	118.4(4)	120.6(2)
C18	C13	C14	118.1(4)	119.0(2)
C18	C13	P1	118.1(4)	120.0(2)
C14	C13	P1	123.6(3)	121.0(2)
C15	C14	C13	120.9(5)	120.2(2)
C16	C15	C14	119.5(6)	120.4(3)
C17	C16	C15	121.4(5)	120.5(2)
C16	C17	C18	118.3(6)	120.4(3)
C13	C18	C17	121.8(6)	119.4(2)

TABLE 6 Torsional Angles (°) for Nonhydrogen Atoms

				cis-**2**	*trans*-**2**
C6	S1	C2	S3	63.7(2)	48.60(12)
C6	S1	C2	P1	−174.2(2)	−83.82(11)
C4	S3	C2	S1	−61.9(2)	−48.49(11)
C4	S3	C2	P1	175.9(2)	83.68(10)
C7	P1	C2	S1	56.9(2)	−167.98(8)
C13	P1	C2	S1	166.42(15)	−57.75(11)
S2	P1	C2	S1	−67.6(2)	66.66(10)
C7	P1	C2	S3	−178.4(2)	58.56(10)
C13	P1	C2	S3	−68.9(2)	168.80(9)
S2	P1	C2	S3	57.1(2)	−66.79(10)
C2	S3	C4	C5	56.4(3)	54.43(15)
C2	S3	C4	C1	178.4(3)	176.34(15)
C1	C4	C5	C6	177.0(4)	172.9(2)
S3	C4	C5	C6	−64.3(4)	−68.7(2)
C4	C5	C6	C3	−173.3(5)	−172.9(2)
C4	C5	C6	S1	67.6(5)	68.8(2)
C2	S1	C6	C5	−61.3(3)	−54.69(15)
C2	S1	C6	C3	176.0(4)	−177.0(2)
C13	P1	C7	C12	137.1(3)	87.02(15)
C2	P1	C7	C12	−111.6(3)	−162.38(14)
S2	P1	C7	C12	12.4(3)	−36.16(15)
C13	P1	C7	C8	−42.2(3)	−92.2(2)
C2	P1	C7	C8	69.2(3)	18.4(2)
S2	P1	C7	C8	−166.9(2)	144.62(14)
C12	C7	C8	C9	−0.6(5)	0.8(3)
P1	C7	C8	C9	178.7(3)	180.0(2)
C7	C8	C9	C10	−1.9(6)	−0.5(3)
C8	C9	C10	C11	3.4(7)	−0.4(3)
C9	C10	C11	C12	−2.5(6)	0.9(3)
C10	C11	C12	C7	0.0(6)	−0.6(3)
C8	C7	C12	C11	1.5(5)	−0.2(3)
P1	C7	C12	C11	−177.7(3)	−179.5(2)
C7	P1	C13	C18	−84.0(3)	−159.0(2)
C2	P1	C13	C18	166.5(3)	89.4(2)
S2	P1	C13	C18	40.9(3)	−35.9(2)
C7	P1	C13	C14	90.7(3)	20.9(2)
C2	P1	C13	C14	−18.9(3)	−90.7(2)
S2	P1	C13	C14	−144.4(3)	144.08(15)
C18	C13	C14	C15	−0.2(6)	−1.0(3)
P1	C13	C14	C15	−174.9(3)	179.0(2)
C13	C14	C15	C16	0.9(7)	−0.7(4)
C14	C15	C16	C17	−0.5(8)	1.7(4)
C15	C16	C17	C18	−0.6(9)	−0.9(4)
C14	C13	C18	C17	−0.9(6)	1.7(3)
P1	C13	C18	C17	174.1(4)	−178.3(2)
C16	C17	C18	C13	1.3(8)	−0.8(4)

$$\Delta(\text{C2-S1-C6}) = 6.6^\circ \ (33\sigma)$$

$$\Delta(\text{C2-S3-C4}) = 4.3^\circ \ (22\sigma)$$

$$\Delta(\text{S1-C2-S3}) = 1.9^\circ \ (10\sigma)$$

It should be pointed out that the crystal structures for both isomers of **2** were refined with good accuracy. Thus, the R-values for *cis*-**2** and *trans*-**2** were found to be 0.0383 and 0.0297, respectively. Therefore, using these precisely determined structural data, it is possible to analyze in detail the differences between the exocyclic C–P and endocyclic S–C bond distances in *cis*-**2** and *trans*-**2** and to relate them with the stereoelectronic effect operating in this system. It was found that the axial C2–P bond in *trans*-**2** is longer by 0.021 Å than the equatorial one in *cis*-**2**. This difference may be interpreted as being due to $n_s \rightarrow \sigma_{\text{C-P}}$ negative hyperconjugation. Interestingly, in the case of 2-diphenylthiophosphinoyl-5-*t*-butyl-1,3-dithiane **1b**, one observes the same relationship, the difference between axial and equatorial C2–P bond being much smaller (0.009 Å). However, in contrast to expectations, the endocyclic S–C2 bond distances in *trans*-**2** are slightly greater than those in *cis*-**2** in the range of 4σ.

Finally, it is interesting to point out that in *trans*-**2**, the nonbonding distances between the thiophosphoryl sulfur S2 and two axial hydrogens at C4 and C6 are nonequal and differ by 0.023 Å. A similar sit-

TABLE 7 Hydrogen-Bonding Geometry (Å, °) (H···Y not greater than 3.00 Å)

D—H···A	*D—H*	*H···A*	*D···A*	*D—H···A*
*cis-**2***				
C4—H41···S2^{i}	1.026(35)	2.825(34)	3.806(4)	160.1(21)
C12—H121···S2	1.038(39)	2.830(29)	3.353(4)	111.5(19)
*trans-**2***				
C4—H41···S2	0.987(21)	2.879(19)	3.659(2)	136.5(16)
C5—H51···S2^{ii}	0.991(23)	2.825(24)	3.506(2)	126.4(16)
C6—H61···S2	0.962(22)	2.902(21)	3.645(2)	134.9(16)
C12—H121···S2	0.889(24)	2.931(25)	3.387(2)	113.7(18)

Symmetry codes: (i) $0.5 + x, 1.5 - y, z$; (ii) $-x, 2 - y, 2 - z$.

TABLE 8 Asymmetry Parameters for Six-Membered Ring

	*cis-***2**	*trans-***2**
$\Delta C_s^{S1} = \Delta C_s^{C4}$	5.8(6)	14.8(3)
$\Delta C_s^{C2} = \Delta C_s^{C5}$	3.6(6)	0.2(3)
$\Delta C_s^{S3} = \Delta C_s^{C6}$	4.8(5)	14.6(3)
$\Delta C_2^{S1\text{-}C2} = \Delta C_2^{C4\text{-}C5}$	7.9(5)	11.1(3)
$\Delta C_2^{C2\text{-}S3} = \Delta C_2^{C5\text{-}C6}$	5.6(6)	10.7(3)
$\Delta C_2^{S3\text{-}C4} = \Delta C_2^{C6-S1}$	3.2(6)	20.2(4)

TABLE 9 Angles Formed by Least-Squares Planes
Plane1—S1, S3, C4, C6
Plane2—S1, C2, S3
Plane3—C4, C5, C6
Plane4—C7, C8, C9, C10, C11, C12
Plane5—C13, C14, C15, C16, C17, C18

	*cis-***2**	*trans-***2**
Plane1/Plane2	62.52(15)	47.30(9)
Plane1/Plane3	58.11(31)	60.34(13)
Plane2/Plane3	4.50(36)	13.04(15)
Plane1/Plane4	70.22(10)	72.96(5)
Plane2/Plane4	53.17(17)	70.54(7)
Plane3/Plane4	57.46(35)	61.05(11)
Plane1/Plane5	49.45(13)	13.76(7)
Plane2/Plane5	79.42(12)	39.71(10)
Plane3/Plane5	77.28(19)	52.31(13)
Plane4/Plane5	88.16(14)	86.53(7)

uation has been found in a wide range of 1,3-dithianes containing axial phosphoryl group bonded to the anomeric carbon atom [1].

EXPERIMENTAL

Crystal and molecular structures of *cis*-**2** and *trans*-**2** were determined using data collected at room temperature on a CAD4 diffractometer with graphite monochromatized CuKα radiation. The compound *cis*-**2** crystallizes in the orthorhombic system, in space group $Pna2_1$. The compound *trans*-**2** crystallizes in the monoclinic system, space group $P2_1/n$. Both compounds have the unit cells consisting of four molecules. Crystal data and experimental details are shown in Table 1. The lattice constants were refined by least-squares fit of 25 reflections in the θ range 20.2°–27.2° for *cis*-**2** and 20.2°–29.8° for *trans*-**2**. The decline in intensities of three control reflections were 2.1% for *cis*-**2** (4, 0, 6; 4, 3, 4; −4, 5, 0) during 54.6 hours of exposure time and 2.4% for *trans*-**2** (2, −7, −4; 1, −9, 3; 2, −6, −3) during 56.4 hours. An empirical absorption correction was applied by the use of the ψ-scan method (EAC program) [3,4]. A total of 3876 reflections for *cis*-**2** and 3560 for *trans*-**2** [with $I > 0\ \sigma(I)$] were used to solve the structures by direct methods and to refine it by full-matrix least-squares using Fs [5,6]. For the structure *cis*-**2**, hydrogen atoms were placed geometrically at idealized positions, set as riding with C–H distance free to refine, and refined isotropically; for methylene groups, the rotation about the C–C bond was also allowed. For compound *trans*-**2**, all H atoms were found from the difference Fourier maps and refined isotropically. Anisotropic thermal parameters were refined for all nonhydrogen atoms in both structures. The final refinement for *cis*-**2** converged to $R = 0.0383$ for 3782 observed reflections [with $I > 2\sigma(I)$] and 238 refined parameters. For *trans*-**2**, $R = 0.0297$ for 3229 reflections and 284 refined parameters. The all-structural refinements were performed using the weighting scheme $w = [\sigma^2(F_0^2) + (mP)^2 + nP]^{-1}$, where $P = (F_c^2 + 2F_c^2)/3$ (see Table 1).

Full crystallographic data for both structures are deposited at the Cambridge Crystallographic Data Centre [7].

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