substituants. En effet, des calculs de charges atomiques partielles par des méthodes semi-quantique (Cotrait, Bideau, Buisson & Royer, 1985) semblent bien montrer l'importance de la présence et de la nature de ces substituants sur cette distribution.

Références

BIDEAU, J. P., BRAVIC, G. & COTRAIT, M. (1984). *Acta Cryst.* C**40**, 1948–1950.

BIDEAU, J. P., BRAVIC, G. & COTRAIT, M. (1985). *Acta Cryst.* C**41**, 558–560.

BIDEAU, J. P., BRAVIC, G., COTRAIT, M. & COURSEILLE, C. (1983*a*). *Acta Cryst.* C**39**, 1101–1103.

BIDEAU, J. P., BRAVIC, G., COTRAIT, M, & COURSEILLE, C. (1983*b*). *Acta Cryst.* C**40**, 322–324.

BRAVIC, G., BIDEAU, J. P. & COURSEILLE, C. (1982). *Cryst. Struct. Commun.* **11**, 409–412.

CAVIER, R., BUISSON, J. P., LEMOINE, J. & ROYER, R. (1981). *Eur. J. Med. Chem.* **16**, 73–76.

COTRAIT, M., BIDEAU, J. P., BUISSON, J. P. & ROYER, R. (1985). *Eur. J. Med. Chem.* Soumis.

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Tome IV, édité par J. A. IBERS & W. C. HAMILTON. Birmingham: Kynoch Press. (Distributeur actuel D. Reidel, Dordrecht.)

HOFNUNG, M. (1985). *Eur. J. Med. Chem.* Sous presse.

KITAIGORODSKII, A. I. (1973). *Molecular Crystals and Molecules*, p. 12. London, New York: Academic Press.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. de York, Angleterre, et Louvain, Belgique.

ROYER, R. & BUISSON, J. P. (1980). *Eur. J. Med. Chem.* **2**, 275–278.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965), *J. Chem. Phys.* **42**, 3175–3187.

TROTTER, J. (1959). *Acta Cryst.* **12**, 889–892.

WEILL-THÉVENET, N., BUISSON, J. P., ROYER, R. & HOFNUNG, M. (1981). *Mutat. Res.*, **88**, 355–362.

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Structure of *trans*-5-Iodo-5-isopropyl-1,3-diphenyl-1λ^6,3λ^6,2,4,6,5λ^5-dithiatriazaphosphorine 1,3-Dioxide

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Abstract. $C_{15}H_{17}IN_3O_2PS_2$, $M_r = 493{\cdot}33$, orthorhombic, $Pna2_1$, $a = 16{\cdot}895\ (1)$, $b = 10{\cdot}589\ (1)$, $c = 10{\cdot}513\ (1)$ Å, $V = 1880{\cdot}8\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}742$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 19{\cdot}9$ cm^{-1}, $F(000) = 976$, $T = 295$ K, $R = 0{\cdot}036$ for 1363 observed reflections with $I > 2{\cdot}5\sigma(I)$. The inorganic ring skeleton possesses a twist-boat conformation with the phenyl substituents in *trans* positions. The endocyclic bond lengths reflect double-bond character caused by d_π–p_π overlap [S(1)—N(2) 1·602 (6), N(2)—S(2) 1·580 (6), S(2)—N(3) 1·554 (5), N(3)—P 1·608 (6), P—N(1) 1·610 (6), N(1)—S(1) 1·558 (5) Å].

Introduction. In the scope of our investigations into the reactivity of six-membered cyclothiaphosphazenes towards organometallic reagents, reactions of *trans*-$NPCl_2(NSOPh)_2$ with $(Bu_3PCuI)_4/{}^i$PrMgCl were carried out following Allcock & Harris (1979, 1981). Treatment of the reaction mixture with 2-propanol gave *trans*-NP(H)^{i}Pr(NSOPh)$_2$, which was converted into the title compound by reaction with iodine in tetrachloromethane. Although analogous compounds, *i.e.* NPIR(NPCl$_2$)$_2$ (R = alkyl), are known (Allcock & Harris, 1981) this communication presents the crystal structure determination of the first example of a cyclothiaphosphazene containing an I atom. The present work was undertaken in order to examine the

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influence of the I substituent on the characteristics of the ring skeleton, such as the conformation and endocyclic bond lengths and angles.

Experimental. Colourless crystals obtained by recrystallization from diethyl ether. D_m not determined. Enraf–Nonius CAD-4F diffractometer, graphite-monochromated Mo $K\alpha$ radiation; crystal 0·2 × 0·14 × 0·075 mm glued on top of a glass fibre. Total of 3700 reflections measured; $\omega/2\theta$ scan; $\Delta\omega = (0{\cdot}80 + 0{\cdot}35\,\mathrm{tg}\theta)°$; $1{\cdot}2° < \theta < 25°$; h −20→20, k 0→12, l 0→12. Three reference reflections measured every hour ($\bar{2}\bar{2}0$: r.m.s.d. 1·4%; 401: r.m.s.d. 0·7%; $\bar{4}01$: r.m.s.d. 0·7%) showed a small linear decay of 0·3% during the 67·2 h of X-ray exposure time. Cell dimensions from setting angles of 19 reflections ($18{\cdot}1° < \theta < 20{\cdot}2°$) in four alternative settings (de Boer & Duisenberg, 1984). A 360° ψ scan for the reflection 224 showed an intensity variation up to 8% about the mean. The intensities were corrected for Lorentz and polarization effects and the small decay; absorption correction applied with *DIFABS* (Walker & Stuart, 1983), min. and max. correction factors being 0·91 and 1·09 on F, respectively. Data set averaged [$R_1 = \sum(I - \bar{I})/\sum I = 0{\cdot}036$] in unique set of 1754 reflections ($R_2 = \sum\sigma/\sum I = 0{\cdot}069$). The standard deviation $\sigma(I)$ was calculated following McCandlish, Stout & Andrews (1975): $\sigma^2(I) = \sigma^2(I)_{\rm cs} + (P^2I^2)$ with $P = 0{\cdot}017$. Structure solved by standard Patterson and Fourier methods; refinement on F by block-diagonal least-squares techniques with anisotropic thermal parameters for the non-hydrogen atoms. H atoms were introduced at positions, 1·0 Å from their carrier atoms, assuming sp^2 or sp^3 hybridization and refined in the riding mode with an overall isotropic temperature factor. The polarity of the structure was tested by a refinement with $+if''$ and $-if''$ values, respectively, giving only marginally differing R values, probably due to twinning in this respect. The subsequent refinements were carried out for the structure with the polarity that gave the lowest R value. Convergence was reached at $R = 0{\cdot}036$, $wR = 0{\cdot}026$, $w = 1/\sigma^2(F)$; $S = 1{\cdot}005$; 1363 observed reflections with $I > 2{\cdot}5\sigma(I)$; 218 parameters refined; max. $\Delta/\sigma = 0{\cdot}21$. Min. and max. residual densities in the final difference Fourier map −0·46 and 0·46 e Å^{-3} [in the neighbourhood of the probably somewhat disordered C(13)]. No correction for secondary extinction. Scattering factors from Cromer & Mann (1968). Anomalous-dispersion correction from Cromer & Liberman (1970). Calculations carried out on the Cyber 180–855 of the University of Utrecht Computer Center. Programs used include *XTAL* (Stewart & Hall, 1983) and *EUCLID* (Spek, 1982; calculation of geometric data and preparation of illustrations including an extended version of the program *PLUTO*).

Discussion. The final values of the refined parameters are given in Table 1.* Fig. 1 gives a view of the title compound including the adopted numbering scheme. Relevant data on the geometry are given in Table 2. The orthorhombic unit cell contains four discrete molecules (Fig. 2). The observed conformation of the PNS ring approaches that of a twist-boat (Cremer & Pople, 1975) with torsion angles ranging from −38·5 (6) to 16·7 (5)° and lowest asymmetry-parameter value $\Delta C_2 = 1{\cdot}0\,(5)°$ for S(1) and N(3) (Duax, Weeks & Rohrer, 1976). Puckering parameters are: $Q = 0{\cdot}457\,(5)$ Å, $\theta = 90{\cdot}9\,(6)°$, $\varphi = 270{\cdot}7\,(6)°$. As a result of the puckering the I atom becomes an axial substituent with an angle of 13·7 (3)° between the P—I bond and the normal to the least-squares plane of the PNS ring. P and S atoms show distorted tetrahedral geometry. The bond length C(13)—C(14) = 1·58 (1) Å is somewhat longer than the normal value of 1·526 Å for a C(sp^3)—C(sp^3) bond (Lide, 1962), probably due to some disorder of C(13). The P—I bond length is 2·435 (2) Å. Other reported P—I bond distances are in the range 2·426–2·493 Å (Gridunova, Shklover, Struchkov, Vil'Chevskaya, Podgbedova & Krylova, 1982; Wörz, Pritzkow & Latscha, 1984). With regard to the PNS ring plane the I atom and the phenyl ring [C(7) − C(12)] are in a *cis* configuration with a short intramolecular C(12)—H(12)···I contact distance H(12)···I = 3·210 (8) Å (sum of the van der Waals radii of 3·4 Å; Bondi, 1964). The exocyclic bond angle I—P—C(13) is 107·7 (3)°. The endocyclic P angle is 114·1 (3)°. In the closely related compounds *trans*-$NPCl_2(NSOPh)_2$ (van Bolhuis, van den Berg & van de Grampel, 1981) and *trans*-$NP^n Pr^i Pr(NSOPh)_2$ (Meetsma, Spek, Winter, van de Grampel & de Boer, 1985) these values are 102·8 (1)/117·9 (3)° and 104·8 (3)/113·0 (2)°, respectively. Hence the wide exocyclic angle is probably a result of a steric effect and not of the scissoring effect (Ritchie, Harris & Allcock, 1980). The torsion angles about P—C(13) indicate an ethane conformation. Both phenyl rings display normal geometry with average bond lengths 1·376 (6)† and 1·368 (7) Å, respectively. The angles between the phenyl planes and the related NSN-segment plane are 87·5 (4) and 82·5 (5)° respectively. The angle between the bisector of the angle O(1)—S(1)—C(1) and the plane of the segment N(1)S(1)N(2) is 6·6 (3)°; for the S(2)O(2)Ph moiety this angle is 11·0 (3)°. This means

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H and an *ORTEP* plot have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42407 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Throughout this paper the mean values and their standard deviations have been calculated using the formulae given by Domenicano, Vaciago & Coulson (1979).

that the S—O bond consistently makes a smaller angle with the N—S—N plane than does the corresponding S—C(Ph) bond. The torsion angle O(1)—S(1)—C(1)—C(2) is −1·0 (7)°, whereas the torsion angle O(2)—S(2)—C(7)—C(8) is 33·6 (7)°. For comparison, in *trans*-NPCl$_2$(NSOPh)$_2$ (van Bolhuis, van den Berg & van de Grampel, 1981) the corresponding values are −4·4 (6) and 31·5 (6)° respectively. These facts indicate a rotation about a line through S(2), normal to the plane O(2)—S(2)—C(7), and a rotation of the phenyl groups about the S(2)—C(7) bond, which may be explained as packing effects. Short non-bonding distances between I and the phenyl ring [C(7)—C(12)] [$-x$, $1-y$, $\frac{1}{2}+z$] are observed: I···C(8), I···C(9), I···C(10) range from 3·51 to 3·84 Å (sum of the van der Waals radii, 3·90 Å; Bondi, 1964).

The equally substituted SNS segment exhibits slightly unequal bond lengths: 1·602 (6) and 1·580 (6) Å; an analogous but stronger effect was observed in *trans*-NPCl$_2$(NSOPh)$_2$ (van Bolhuis, van den Berg & van de Grampel, 1981). As found for other members belonging to the class of thiatriazaphosphorine oxides (van de Grampel, 1981), the endocyclic bond lengths are shorter than the corresponding single-bond lengths, N—P = 1·77 Å (Cruickshank, 1964) and N—S = 1·76 Å (Sass, 1960). This shortening can be explained from an additional π-bonding system involving *d* orbitals (S,P) and *p* (sp^2) orbitals (N). The difference of 0·053 (6) Å (mean value) between the N—P and N—S bond length in a P—N—S unit can be correlated with the difference in electronegativity between the P and S centre (van de Grampel, 1981).

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters, with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
I	0·05853 (3)	0·21820 (5)	0·9987 (1)	0·0549 (2)
S(1)	−0·1599 (1)	0·1309 (2)	0·8592 (2)	0·0292 (6)
S(2)	−0·1091 (1)	0·3360 (2)	0·7121 (2)	0·0293 (6)
P	−0·0027 (1)	0·1591 (2)	0·7976 (2)	0·0301 (6)
O(1)	−0·1823 (3)	0·1205 (4)	0·9874 (7)	0·042 (2)
O(2)	−0·1399 (3)	0·3350 (4)	0·5863 (5)	0·044 (2)
N(1)	−0·0784 (3)	0·0709 (5)	0·8250 (6)	0·036 (2)
N(2)	−0·1649 (3)	0·2759 (5)	0·8164 (6)	0·033 (2)
N(3)	−0·0228 (3)	0·2859 (5)	0·7198 (6)	0·034 (2)
C(1)	−0·2293 (4)	0·0458 (6)	0·7675 (7)	0·023 (3)
C(2)	−0·2886 (5)	−0·0143 (7)	0·8291 (9)	0·048 (3)
C(3)	−0·3444 (5)	−0·0811 (7)	0·760 (1)	0·058 (3)
C(4)	−0·3385 (5)	−0·0888 (7)	0·629 (1)	0·058 (4)
C(5)	−0·2784 (6)	−0·0261 (7)	0·5691 (9)	0·066 (4)
C(6)	−0·2220 (5)	0·0405 (7)	0·6381 (8)	0·050 (3)
C(7)	−0·1072 (4)	0·4939 (6)	0·7647 (7)	0·030 (3)
C(8)	−0·1662 (4)	0·5730 (6)	0·7226 (8)	0·034 (3)
C(9)	−0·1676 (5)	0·6945 (6)	0·7685 (9)	0·048 (3)
C(10)	−0·1123 (5)	0·7360 (7)	0·8511 (9)	0·051 (3)
C(11)	−0·0542 (5)	0·6586 (7)	0·8926 (9)	0·053 (3)
C(12)	−0·0516 (5)	0·5348 (7)	0·8499 (8)	0·042 (3)
C(13)	0·0693 (4)	0·0651 (6)	0·7079 (9)	0·043 (3)
C(14)	0·1453 (4)	0·1497 (8)	0·6865 (9)	0·055 (3)
C(15)	0·0893 (5)	−0·0555 (7)	0·778 (1)	0·058 (4)

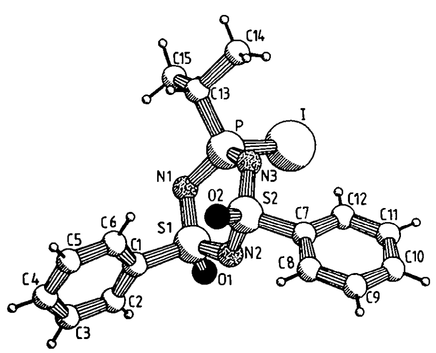

Fig. 1. *PLUTO* (*EUCLID* version) drawing of *trans*-NPI^{i}Pr(NSOPh)$_2$ illustrating the puckering and the adopted numbering scheme.

Table 2. *Bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

P—I	2·435 (2)	P—N(1)	1·610 (6)	C(7)—C(8)	1·38 (1)
S(1)—O(1)	1·404 (7)	P—N(3)	1·608 (6)	C(7)—C(12)	1·37 (1)
S(1)—N(1)	1·558 (5)	P—C(13)	1·833 (8)	C(8)—C(9)	1·37 (1)
S(1)—N(2)	1·602 (6)	C(1)—C(2)	1·35 (1)	C(9)—C(10)	1·35 (1)
S(1)—C(1)	1·765 (7)	C(1)—C(6)	1·37 (1)	C(10)—C(11)	1·35 (1)
S(2)—O(2)	1·421 (6)	C(2)—C(3)	1·38 (1)	C(11)—C(12)	1·39 (1)
S(2)—N(2)	1·580 (6)	C(3)—C(4)	1·38 (1)	C(13)—C(14)	1·58 (1)
S(2)—N(3)	1·554 (5)	C(4)—C(5)	1·37 (1)	C(13)—C(15)	1·51 (1)
S(2)—C(7)	1·760 (7)	C(5)—C(6)	1·39 (1)		

O(1)—S(1)—N(1)	115·3 (3)	S(2)—N(3)—P	120·7 (4)
O(1)—S(1)—N(2)	109·3 (3)	S(1)—C(1)—C(2)	118·1 (6)
O(1)—S(1)—C(1)	107·7 (3)	S(1)—C(1)—C(6)	120·3 (6)
N(1)—S(1)—N(2)	111·9 (3)	C(2)—C(1)—C(6)	121·6 (7)
N(1)—S(1)—C(1)	104·6 (3)	C(1)—C(2)—C(3)	119·6 (9)
N(2)—S(1)—C(1)	107·5 (3)	C(2)—C(3)—C(4)	120·3 (8)
O(2)—S(2)—N(2)	115·1 (3)	C(3)—C(4)—C(5)	118·9 (8)
O(2)—S(2)—N(3)	112·9 (3)	C(4)—C(5)—C(6)	121·0 (9)
O(2)—S(2)—C(7)	107·9 (3)	C(1)—C(6)—C(5)	118·6 (8)
N(2)—S(2)—N(3)	112·7 (3)	S(2)—C(7)—C(8)	117·6 (5)
N(2)—S(2)—C(7)	100·1 (3)	S(2)—C(7)—C(12)	121·2 (5)
N(3)—S(2)—C(7)	106·9 (3)	C(8)—C(7)—C(12)	121·0 (6)
I—P—N(1)	109·3 (2)	C(7)—C(8)—C(9)	118·1 (7)
I—P—N(3)	108·5 (2)	C(8)—C(9)—C(10)	121·2 (7)
I—P—C(13)	107·7 (3)	C(9)—C(10)—C(11)	120·9 (7)
N(1)—P—N(3)	114·1 (3)	C(10)—C(11)—C(12)	119·5 (8)
N(1)—P—C(13)	107·7 (3)	C(7)—C(12)—C(11)	119·3 (7)
N(3)—P—C(13)	109·4 (3)	P—C(13)—C(14)	107·7 (5)
S(1)—N(1)—P	120·4 (3)	P—C(13)—C(15)	110·9 (6)
S(1)—N(2)—S(2)	123·4 (4)	C(14)—C(13)—C(15)	111·5 (6)

P—N(1)—S(1)—N(2)	19·7 (6)	I—P—C(13)—C(15)	62·6 (5)
N(1)—S(1)—N(2)—S(2)	20·2 (6)	N(1)—P—C(13)—C(14)	−177·4 (5)
S(1)—N(2)—S(2)—N(3)	−38·4 (5)	N(1)—P—C(13)—C(15)	−55·2 (6)
N(2)—S(2)—N(3)—P	16·7 (5)	N(3)—P—C(13)—C(14)	58·1 (6)
S(2) N(3) P - N(1)	18·3 (6)	N(3)—P—C(13)—C(15)	−179·7 (5)
N(3)—P—N(1)—S(1)	−38·5 (6)	O(1)—S(1)—C(1)—C(2)	−1·0 (7)
I—P—C(13)—C(14)	−59·6 (6)	O(2)—S(2)—C(7)—C(8)	−33·6 (7)

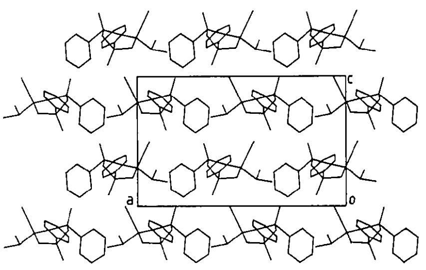

Fig. 2. Projection of part of the structure down the *b* axis.

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References

Allcock, H. R. & Harris, P. J. (1979). *J. Am. Chem. Soc.* **101**, 6221–6229.

Allcock, H. R. & Harris, P. J. (1981). *Inorg. Chem.* **20**, 2844–2848.

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* A**40**, C410.

Bolhuis, F. van, van den Berg, J. B. & van de Grampel, J. C. (1981). *Cryst. Struct. Commun.* **10**, 1031–1035.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Cruickshank, D. W. J. (1964). *Acta Cryst.* **17**, 671–672.

Domenicano, A., Vaciago, A. & Coulson, C. A. (1979). *Acta Cryst.* B**31**, 221–234.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Top. Stereochem.* **9**, 271–383.

Grampel, J. C. van de (1981). *Rev. Inorg. Chem.* **3**, 1–28.

Gridunova, G. V., Shklover, V. E., Struchkov, Yu. T., Vil'Chevskaya, V. D., Podgbedova, N. L. & Krylova, A. I. (1982). *J. Organomet. Chem.* **238**, 297–305.

Lide, D. R. (1962). *Tetrahedron*, **17**, 125–134.

McCandlish, L. E., Stout, G. H. & Andrews, L. C. (1975). *Acta Cryst.* A**31**, 245–249.

Meetsma, A., Spek, A. L., Winter, H., van de Grampel, J. C. & de Boer, J. L. (1985). *Acta Cryst.* Submitted.

Ritchie, R. J., Harris, P. J. & Allcock, H. R. (1980). *Inorg. Chem.* **19**, 2483–2486.

Sass, R. L. (1960). *Acta Cryst.* **13**, 320–323.

Spek, A. L. (1982). The *EUCLID* package. In *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Stewart, J. H. & Hall, S. R. (1983). The *XTAL* system. Tech. Rep TR-1364. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Wörz, H.-J., Pritzkow, H. & Latscha, H. P. (1984). *Z. Naturforsch. Teil B*, **39**, 139–141.

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1,10-Bis(trimethylsilyl)phenothiazine

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Abstract. $C_{18}H_{25}NSSi_2$, $M_r = 343{\cdot}6$, monoclinic, Cc, $a = 13{\cdot}674\,(6)$, $b = 13{\cdot}983\,(5)$, $c = 10{\cdot}186\,(3)$ Å, $\beta = 93{\cdot}8\,(3)^\circ$, $V = 1943\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}17$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}8$ cm^{-1}, $F(000) = 1472$, $T = 296$ K, $R = 0{\cdot}039$ for 1555 observed reflections. The easy preparation and observed geometry of the title compound show that the phenothiazine ring system with the N atom substitutent in the synclinal (quasi-axial or *extra*) conformation (presumed necessary for antidepressant activity) can accommodate very bulky substituents in the 1- and 10-positions simultaneously; steric repulsion is relieved by bending of the bulky groups away from one another.

Introduction. Until very recently, all of the many X-ray studies on phenothiazine derivatives related to the antipsychotic promazine showed the N(10) substituent to be in flattened synperiplanar [quasi-equatorial or *intra* (Malreau & Pullman, 1964)] positions (Bell, Blount, Briscoe & Freeman, 1968; Marsau & Cam, 1973; Marsau & Busetta, 1973; Malmstrom & Cordes, 1973; Chu & van der Helm, 1974, 1975, 1976; Phelps & Cordes, 1976; Chu & Yang, 1977; Chu, Napoleone, Ternay & Chang, 1982*a*,*b*), as depicted in Fig. 1 for 10-methylphenothiazine (1) (Chu & van der Helm, 1974). Even with a 1-isopropyl substituent, the N(10) proton was found in this conformation (Chu, Napoleone, Ternay & Chang, 1982*a*). Recently, however, two derivatives bearing both N(10) and C(1)

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