ORGANIC COMPOUNDS

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Conformational Study of 1,3,5-Tris-(*o*-methoxyphenyl)-1,3,5-triazacyclohexane and 1,3,5-Tris(*p*-methoxyphenyl)-1,3,5-triazacyclohexane

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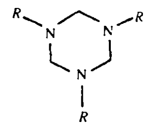
Abstract

The triazacyclohexane ring in both title compounds, $C_{24}H_{27}N_3O_3$ (1*a*) and $C_{24}H_{27}N_3O_3$ (1*b*), adopts a chair conformation with an equatorial–diaxial orientation of the aryl groups. The repulsion between axial groups is relieved by the displacement of the N—C(aryl) axial bonds outwards from ideal tetrahedral positions by between 10 and 24°. The torsion angles around the N—C bonds in the triazacyclohexane ring are in the range 49.5–58.7 (2)° for (1*a*) and in the range 56.6–57.2 (3)° for (1*b*). The N—C ring bond lengths are 1.445–1.481 (2) and 1.444–1.482 (3) Å, and the exocyclic N—C bond lengths are 1.422–1.429 (2) and 1.416–1.421 (3) Å for compounds (1*a*) and (1*b*), respectively. The CH_2—N—CH_2 angles are 108.4–112.4 (2) and 108.9–109.8 (2)°, and the N—CH_2—N angles are 112.3–113.5 (2) and 111.8–112.0 (2)° for compounds (1*a*) and (1*b*), respectively.

Comment

A variety of chair, twist-boat and boat conformations can be considered for 1,3,5-triazacyclohexanes with a pyramidal arrangement of bonds at the N atoms. Four types of chair conformation, *eee*, *eea*, *eaa* and *aaa*, where *e* is equatorial and *a* is axial, are possible and each of these conformations results in axial interactions involving substituents or lone pairs of electrons on the N atoms. Investigation of the conformational properties of 1,3,5-triaryl-1,3,5-triazacyclohexanes has been limited to an inconclusive NMR study of 1,3,5-triphenyl-1,3,5-triazacyclohexane (1*c*) (Farmer & Hamer, 1968) and an X-ray structure analysis of that compound which showed a molecule with the *eaa* chair conformation in the crystal (Giumanini, Verado, Randaccio, Bresciani-Pahor & Traldi, 1985). We undertook X-ray studies of a number of *o*-, *m*- and *p*-substituted phenyl derivatives to provide more information about the conformations of the triaryl compounds; the results for the *o*- and *p*-methoxyphenyl compounds [(1*a*) and (1*b*), respectively] are presented here.



(1*a*) $R = o\text{-}CH_3OC_6H_4$
(1*b*) $R = p\text{-}CH_3OC_6H_4$
(1*c*) $R = C_6H_5$
(1*d*) $R = C_6H_{11}$

Figs. 1 and 2, drawn using *ORTEP*II (Johnson, 1971), show the molecular structures of the title compounds. Each molecule adopts the *eaa* chair conformation, with diaxial repulsion between aryl groups preferred to that between lone pairs of electrons. A similar result was obtained for compound (1*c*) (Giumanini, Verado, Randaccio, Bresciani-Pahor & Traldi, 1985). The alternative *eea* chair conformation has been found in the crystal structure of 1,3,5-tricyclohexyl-1,3,5-triazacyclohexane, (1*d*), in accordance with the greater steric requirements of the cyclohexyl group (Bouchemma, McCabe & Sim, 1988).

In compounds (1*a*) and (1*b*), the N atoms have pyramidal geometry with the N—C(aryl) bonds inclined at 30.8–44.9 (3)° to their respective CH_2—N—CH_2 planes. For comparison, spectroscopic studies indicate that the corresponding out-of-plane angle of the N—C(aryl) bond is 37.5–42.2° in aniline (Larsen, Hansen & Nicolaisin, 1976; Lister, Tyler, Hog & Larsen, 1974), 27.0° in *N*,*N*-dimethylaniline (Cervellati, Borgo & Lister, 1982) and 46.4° in *p*-fluoroaniline (Hastie, Lister, McNeil & Tyler, 1970). The N atoms of aliphatic amines are associated with larger out-of-plane angles than the N atoms of aromatic amines and the out-of-plane angles formed by the N—C bonds in (1*d*) are 46.1–52.2 (2)° (Bouchemma, McCabe & Sim, 1988). The corresponding angle for a tetrahedral arrangement of bonds is 54.7° and the N—C(axial) bonds in compounds (1*a*) and (1*b*) are consequently bent outwards by 10–24° from the ideal chair conformation, reducing the interactions between the axial aryl groups. The orientations of the aryl groups about the N—C(aryl) bonds reflect the increased steric requirements when the aryl group is changed from *p*- to *o*-methoxyphenyl. The angle θ (Table 5) for compound (1*b*) is in the range 62.4–63.6 (3)°, indicating that the dihedral angle between the N atom lone-pair orbital and the aromatic π orbitals is *ca* 26–28°, whereas in (1*a*) θ is 32.8–40.0° and the lone-pair/π-orbital dihedral angle is increased to 50–57°. In the absence of steric effects the lone-pair/π-orbital dihedral angle would be expected to be 0° to provide maximum overlap.

The N atoms deviate from the phenyl ring planes by 0.036–0.160 (3) Å and the O atoms deviate by 0.004–0.079 (3) Å giving out-of-plane angles of 1.4–6.5 (3)° (mean 4.4°) for the C(aryl)—N bonds and 0.2–3.3 (3)°

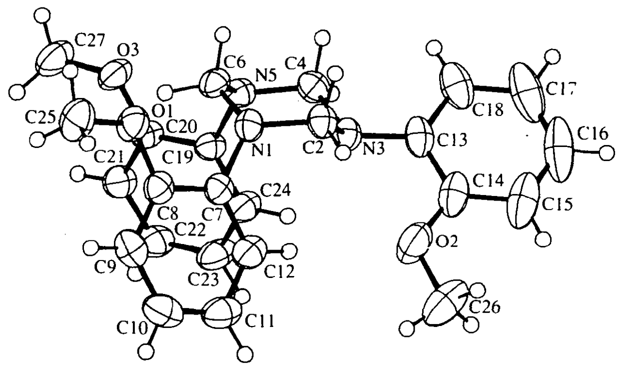


Fig. 1. The molecular structure and atomic numbering scheme for compound (*1a*). The displacement ellipsoids of the C and N atoms are drawn at the 50% probability level and H atoms are represented by spheres of radii 0.1 Å.

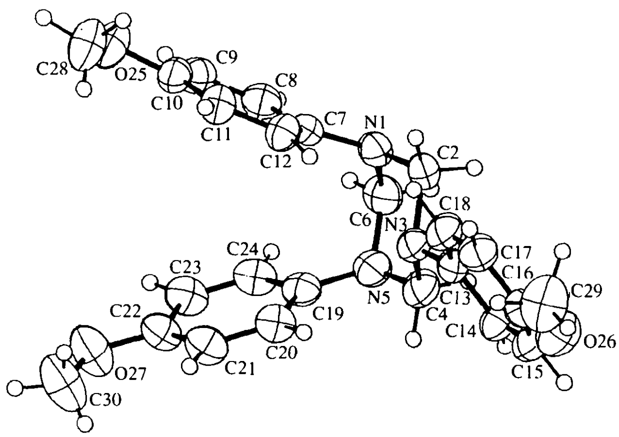


Fig. 2. The molecular structure and atomic numbering scheme for compound (*1b*). The displacement ellipsoids of the C and N atoms are drawn at the 50% probability level and H atoms are represented by spheres of radii 0.1 Å.

(mean 1.1°) for the C(aryl)—O bonds. Molecular-orbital (MO) calculations for aniline give a non-planar equilibrium geometry with the C(aryl)—N bond inclined at 2.4° to the aromatic plane (Niu & Boggs, 1984).

The packing of the molecules is rather more compact for compound (*1a*) than for (*1b*) since the respective mean molecular volumes are 520 and 539 Å^3. Similar results were obtained for the *o*- and *p*-fluorophenyl and the *o*- and *p*-chlorophenyl compounds.

Experimental

Compound (*1a*) was prepared by stirring 2-anisidine (6.16 g, 50 mmol) for 3 h at 319 K in an oil bath with paraformaldehyde (1.58 g, 52.5 mmol) in xylene containing 0.05 g of sodium hydroxide. The xylene was removed under vacuum using an oil pump and the residue was recrystallized from acetone to give the required product (5.94 g, 88%) as needles (m.p. 439–441 K). Analysis: found C 71.17, H 6.79, N 10.35%; $C_{24}H_{27}N_3O_3$ requires C 71.08, H 6.71, N 10.36%. *m/z* 135, 134, 121, 107, 92 and 77; ν_{max} (KBr) 3000, 2838, 1595, 1500, 1465, 1454, 1196 1175, 1112, 1030, 1000, 940 and 770 cm^{-1}; δ_H 3.82 (9H, *s*, CH_3), 4.86 (6H, *s*, CH_2), 6.67–7.00 p.p.m. (12H, *m*, aryl).

Compound (*1b*) was synthesized by adding formalin (8 ml) with stirring to a solution of 4-anisidine (6.18 g, 50 mmol) in ethanol (20 ml) at room temperature over 1 h. The resulting precipitate was filtered and dried to yield the required product (4.88 g, 72%) which was recrystallized from ethanol to give needles (m.p. 406 K). Analysis: found C 70.99, H 6.85, N 10.25%; $C_{24}H_{27}N_3O_3$ requires C 71.08, H 6.71, N 10.36%. *m/z* 135, 134, 107 and 92; ν_{max} (KBr) 2957, 2835, 1510, 1246, 1185, 1157, 1036, 987, 940 and 825 cm^{-1}; δ_H 3.72 (9H, *s*, CH_3), 4.66 (6H, *s*, CH_2) and 7.00 p.p.m. (6H, *d*, aryl).

Compound (1*a*)

Crystal data

$C_{24}H_{27}N_3O_3$	Mo $K\alpha$ radiation
M_r = 405.53	λ = 0.71069 Å
Triclinic	Cell parameters from 25 reflections
$P\overline{1}$	θ = 12.0–17.0°
a = 8.582 (2) Å	μ = 0.09 mm^{-1}
b = 8.996 (2) Å	T = 293 K
c = 15.357 (3) Å	Needle
α = 94.90 (1)°	0.17 × 0.33 × 0.50 mm
β = 93.27 (1)°	White
γ = 117.59 (1)°	
V = 1040 (1) Å^3	
Z = 2	
D_x = 1.29 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.024
ω–2θ scans	θ_{max} = 28.5°
Absorption correction: none	h = 0 → 11
5280 measured reflections	k = −12 → 12
3691 independent reflections	l = −20 → 20
3691 observed reflections [$I > 2.5\sigma(I)$]	2 standard reflections frequency: 60 min intensity decay: 2%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.3
R = 0.038	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
wR = 0.050	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
S = 2.23	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)
3691 reflections	
380 parameters	
Only coordinates of H atoms refined	
$w = 1/\sigma^2(F)$	

Compound (1*b*)

Crystal data

$C_{24}H_{27}N_3O_3$	Cu $K\alpha$ radiation
M_r = 405.53	λ = 1.5418 Å
Orthorhombic	Cell parameters from 25 reflections
$Pca2_1$	θ = 17.0–25.0°
a = 15.518 (2) Å	μ = 0.68 mm^{-1}
b = 16.427 (2) Å	T = 293 K
c = 8.460 (1) Å	

$V = 2157$ (2) Å^3
$Z = 4$
$D_x = 1.25$ Mg m^{-3}
Needle
$0.10 \times 0.35 \times 0.35$ mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2443 measured reflections
2275 independent reflections
2111 observed reflections [$I > 2.5\sigma(I)$]
$\theta_{max} = 72°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 10$
2 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

Refinement on F
$R = 0.027$
$wR = 0.036$
$S = 1.88$
2111 reflections
379 parameters
Only coordinates of H atoms refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.1$
$\Delta\rho_{max} = 0.11$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (1*a*)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N(1)	0.25853 (13)	0.08060 (13)	0.68684 (6)	0.039
N(3)	0.33540 (13)	0.18870 (13)	0.84249 (7)	0.040
N(5)	0.48220 (13)	0.37244 (13)	0.73449 (7)	0.039
C(2)	0.19546 (17)	0.07206 (18)	0.77244 (8)	0.042
C(4)	0.40923 (18)	0.36012 (17)	0.81789 (9)	0.044
C(6)	0.34826 (17)	0.25336 (16)	0.66506 (9)	0.042
C(7)	0.32254 (15)	−0.03352 (15)	0.65823 (8)	0.039
C(8)	0.29521 (16)	−0.09136 (16)	0.56773 (8)	0.043
C(9)	0.3415 (2)	−0.2134 (2)	0.5376 (1)	0.056
C(10)	0.41334 (2)	−0.2798 (2)	0.5964 (1)	0.063
C(11)	0.4415 (2)	−0.2239 (1)	0.6844 (1)	0.060
C(12)	0.39686 (18)	−0.10042 (18)	0.71533 (10)	0.050
C(13)	0.28214 (17)	0.17356 (19)	0.92917 (9)	0.047
C(14)	0.2660 (2)	0.0337 (2)	0.9695 (1)	0.058
C(15)	0.2264 (3)	0.0207 (3)	1.0558 (1)	0.079
C(16)	0.1994 (3)	0.1428 (4)	1.1019 (1)	0.087
C(17)	0.2105 (2)	0.2777 (3)	1.0638 (1)	0.080
C(18)	0.2527 (2)	0.2942 (3)	0.9771 (1)	0.062
C(19)	0.65731 (15)	0.39246 (15)	0.73592 (8)	0.038
C(20)	0.76807 (16)	0.49279 (15)	0.67727 (8)	0.041
C(21)	0.94431 (17)	0.52968 (17)	0.68244 (10)	0.049
C(22)	1.01164 (18)	0.46722 (19)	0.74507 (11)	0.056
C(23)	0.90477 (19)	0.36692 (20)	0.80178 (10)	0.059
C(24)	0.72819 (18)	0.32961 (18)	0.79704 (9)	0.050
C(25)	0.1662 (2)	−0.0947 (2)	0.4252 (1)	0.064
C(26)	0.2786 (3)	−0.2289 (3)	0.9538 (1)	0.090
C(27)	0.8090 (2)	0.6687 (2)	0.5644 (1)	0.071
O(1)	0.21717 (13)	−0.02354 (12)	0.51519 (6)	0.052
O(2)	0.29630 (18)	−0.08078 (16)	0.91908 (7)	0.077
O(3)	0.69286 (12)	0.55057 (13)	0.61719 (7)	0.055

Table 2. *Selected geometric parameters* (Å, °) *for* (1*a*)

N(1)—C(2)	1.445 (2)	C(13)—C(18)	1.387 (3)
N(1)—C(6)	1.459 (2)	C(14)—C(15)	1.388 (3)
N(1)—C(7)	1.422 (2)	C(14)—O(2)	1.365 (3)
N(3)—C(2)	1.481 (2)	C(15)—C(16)	1.372 (4)
N(3)—C(4)	1.464 (2)	C(16)—C(17)	1.359 (4)
N(3)—C(13)	1.429 (2)	C(17)—C(18)	1.402 (3)
N(5)—C(4)	1.448 (2)	C(19)—C(20)	1.404 (2)
N(5)—C(6)	1.460 (2)	C(19)—C(24)	1.384 (2)
N(5)—C(19)	1.427 (2)	C(20)—C(21)	1.386 (2)
C(7)—C(8)	1.408 (2)	C(20)—O(3)	1.365 (2)
C(7)—C(12)	1.386 (2)	C(21)—C(22)	1.380 (3)
C(8)—C(9)	1.384 (3)	C(22)—C(23)	1.368 (3)
C(8)—O(1)	1.368 (3)	C(23)—C(24)	1.388 (2)
C(9)—C(10)	1.386 (3)	C(25)—O(1)	1.425 (2)
C(10)—C(11)	1.368 (3)	C(26)—O(2)	1.424 (3)
C(11)—C(12)	1.390 (3)	C(27)—O(3)	1.427 (3)
C(13)—C(14)	1.403 (3)		
C(2)—N(1)—C(6)	112.4 (2)	N(3)—C(13)—C(18)	123.2 (2)
C(2)—N(1)—C(7)	118.4 (1)	C(14)—C(13)—C(18)	118.1 (2)
C(6)—N(1)—C(7)	118.7 (1)	C(13)—C(14)—C(15)	120.2 (2)
C(2)—N(3)—C(4)	108.4 (2)	C(13)—C(14)—O(2)	115.4 (2)
C(2)—N(3)—C(13)	114.2 (2)	C(15)—C(14)—O(2)	124.3 (2)
C(4)—N(3)—C(13)	114.7 (2)	C(14)—C(15)—C(16)	120.4 (2)
C(4)—N(5)—C(6)	110.6 (1)	C(15)—C(16)—C(17)	120.6 (2)
C(4)—N(5)—C(19)	116.8 (2)	C(16)—C(17)—C(18)	119.9 (2)
C(6)—N(5)—C(19)	118.4 (1)	C(13)—C(18)—C(17)	120.8 (2)
N(1)—C(2)—N(3)	112.6 (2)	N(5)—C(19)—C(20)	118.4 (2)
N(3)—C(4)—N(5)	112.3 (2)	N(5)—C(19)—C(24)	123.2 (2)
N(1)—C(6)—N(5)	113.5 (2)	C(20)—C(19)—C(24)	118.2 (2)
N(1)—C(7)—C(8)	117.8 (2)	C(19)—C(20)—C(21)	120.2 (2)
N(1)—C(7)—C(12)	123.3 (2)	C(19)—C(20)—O(3)	116.5 (2)
C(8)—C(7)—C(12)	118.7 (2)	C(21)—C(20)—O(3)	123.2 (2)
C(7)—C(8)—C(9)	119.9 (2)	C(20)—C(21)—C(22)	120.1 (2)
C(7)—C(8)—O(1)	115.8 (2)	C(21)—C(22)—C(23)	120.4 (2)
C(9)—C(8)—O(1)	124.3 (2)	C(22)—C(23)—C(24)	119.8 (2)
C(8)—C(9)—C(10)	120.1 (2)	C(19)—C(24)—C(23)	121.2 (2)
C(9)—C(10)—C(11)	120.5 (2)	C(8)—O(1)—C(25)	117.4 (2)
C(7)—C(12)—C(11)	120.9 (2)	C(14)—O(2)—C(26)	119.3 (2)
N(3)—C(13)—C(14)	118.6 (2)	C(20)—O(3)—C(27)	116.8 (2)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (1*b*)

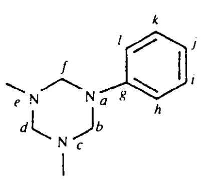
$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N(1)	0.37834 (8)	0.14412 (8)	0.1829 (6)	0.052
N(3)	0.37743 (9)	0.29049 (9)	0.22094 (27)	0.051
N(5)	0.35247 (9)	0.23412 (10)	−0.03926 (27)	0.056
C(2)	0.35725 (11)	0.20975 (11)	0.28969 (31)	0.056
C(4)	0.33129 (12)	0.29913 (12)	0.07002 (30)	0.059
C(6)	0.33300 (11)	0.15532 (12)	0.03307 (33)	0.060
C(7)	0.46494 (10)	0.11632 (9)	0.17650 (28)	0.046
C(8)	0.53060 (10)	0.15129 (10)	0.26301 (28)	0.051
C(9)	0.61279 (11)	0.11674 (11)	0.26896 (28)	0.054
C(10)	0.63054 (11)	0.04740 (10)	0.18310 (27)	0.052
C(11)	0.56629 (12)	0.01400 (10)	0.08891 (29)	0.055
C(12)	0.48465 (11)	0.04713 (9)	0.08727 (29)	0.054
C(13)	0.36895 (10)	0.35614 (11)	0.32827 (29)	0.051
C(14)	0.31741 (11)	0.42410 (11)	0.29970 (30)	0.059
C(15)	0.31560 (12)	0.48872 (11)	0.40392 (34)	0.063
C(16)	0.36425 (10)	0.48801 (11)	0.54013 (33)	0.058
C(17)	0.41455 (11)	0.42071 (12)	0.57181 (29)	0.061
C(18)	0.41683 (11)	0.35632 (11)	0.46778 (30)	0.059
C(19)	0.43304 (11)	0.23880 (10)	−0.11865 (28)	0.050
C(20)	0.49467 (12)	0.29799 (10)	−0.08891 (30)	0.055
C(21)	0.57075 (12)	0.30122 (11)	−0.17439 (30)	0.060
C(22)	0.58791 (13)	0.24402 (11)	−0.28879 (30)	0.059
C(23)	0.52757 (14)	0.18338 (11)	−0.31730 (31)	0.062
C(24)	0.45133 (12)	0.18177 (10)	−0.23615 (30)	0.057
C(28)	0.77506 (14)	0.04029 (19)	0.2752 (4)	0.080
C(29)	0.4121 (2)	0.5599 (2)	0.7682 (5)	0.093
C(30)	0.7259 (2)	0.2984 (3)	−0.3510 (5)	0.101
O(25)	0.70746 (9)	0.00669 (9)	0.18324 (27)	0.070
O(26)	0.35723 (10)	0.55512 (9)	0.63516 (29)	0.077
O(27)	0.66127 (12)	0.24058 (10)	−0.38103 (31)	0.085

Table 4. *Selected geometric parameters* (Å, °) *for* (**1***b*)

N(1)—C(2)	1.444 (4)	C(13)—C(18)	1.395 (4)
N(1)—C(6)	1.462 (5)	C(14)—C(15)	1.380 (4)
N(1)—C(7)	1.420 (2)	C(15)—C(16)	1.378 (4)
N(3)—C(2)	1.482 (3)	C(16)—C(17)	1.380 (3)
N(3)—C(4)	1.471 (4)	C(16)—O(26)	1.369 (3)
N(3)—C(13)	1.416 (3)	C(17)—C(18)	1.376 (4)
N(5)—C(4)	1.450 (3)	C(19)—C(20)	1.387 (3)
N(5)—C(6)	1.463 (3)	C(19)—C(24)	1.395 (3)
N(5)—C(19)	1.421 (3)	C(20)—C(21)	1.386 (3)
C(7)—C(8)	1.380 (3)	C(21)—C(22)	1.375 (4)
C(7)—C(12)	1.398 (3)	C(22)—C(23)	1.388 (3)
C(8)—C(9)	1.397 (3)	C(22)—O(27)	1.381 (4)
C(9)—C(10)	1.379 (3)	C(23)—C(24)	1.368 (4)
C(10)—C(11)	1.389 (3)	C(28)—O(25)	1.418 (4)
C(10)—O(25)	1.368 (3)	C(29)—O(26)	1.413 (5)
C(11)—C(12)	1.379 (3)	C(30)—O(27)	1.405 (5)
C(13)—C(14)	1.394 (3)		
C(2)—N(1)—C(6)	109.8 (2)	N(3)—C(13)—C(18)	119.7 (2)
C(2)—N(1)—C(7)	118.6 (3)	C(14)—C(13)—C(18)	116.8 (2)
C(6)—N(1)—C(7)	117.6 (4)	C(13)—C(14)—C(15)	121.1 (3)
C(2)—N(3)—C(4)	108.9 (2)	C(14)—C(15)—C(16)	121.1 (2)
C(2)—N(3)—C(13)	114.2 (2)	C(15)—C(16)—C(17)	118.6 (3)
C(4)—N(3)—C(13)	116.0 (2)	C(15)—C(16)—O(26)	116.2 (2)
C(4)—N(5)—C(6)	109.8 (2)	C(17)—C(16)—O(26)	125.2 (3)
C(4)—N(5)—C(19)	117.4 (2)	C(16)—C(17)—C(18)	120.4 (3)
C(6)—N(5)—C(19)	115.3 (2)	C(13)—C(18)—C(17)	121.9 (2)
N(1)—C(2)—N(3)	112.0 (2)	N(5)—C(19)—C(20)	124.0 (2)
N(3)—C(4)—N(5)	111.8 (2)	N(5)—C(19)—C(24)	118.6 (3)
N(1)—C(6)—N(5)	112.0 (2)	C(20)—C(19)—C(24)	117.4 (2)
N(1)—C(7)—C(8)	123.0 (3)	C(19)—C(20)—C(21)	121.3 (2)
N(1)—C(7)—C(12)	119.3 (2)	C(20)—C(21)—C(22)	120.4 (2)
C(8)—C(7)—C(12)	117.6 (2)	C(21)—C(22)—C(23)	118.8 (2)
C(7)—C(8)—C(9)	121.6 (2)	C(21)—C(22)—O(27)	125.8 (2)
C(8)—C(9)—C(10)	119.9 (2)	C(23)—C(22)—O(27)	115.4 (2)
C(9)—C(10)—C(11)	119.0 (2)	C(22)—C(23)—C(24)	120.7 (3)
C(9)—C(10)—O(25)	125.3 (2)	C(19)—C(24)—C(23)	121.4 (2)
C(11)—C(10)—O(25)	115.7 (2)	C(10)—O(25)—C(28)	117.1 (2)
C(10)—C(11)—C(12)	120.6 (2)	C(16)—O(26)—C(29)	117.7 (2)
C(7)—C(12)—C(11)	121.1 (2)	C(22)—O(27)—C(30)	117.3 (3)
N(3)—C(13)—C(14)	123.5 (3)		

Table 5. *Conformational angles* (°)



θ is the angle between planes *dagj* and *aghijkl*, γ is between the *a*—*g* bond and the *fab* plane and ζ is between the *a*—*g* bond and the *ghijkl* plane.

	θ	γ	ζ
Compound (1*a*)			
N(3) equatorial	40.0 (3)	44.9 (3)	3.7 (3)
N(1) axial	38.4 (3)	30.8 (3)	5.3 (3)
N(5) axial	32.8 (3)	35.5 (3)	6.0 (3)
Compound (1*b*)			
N(3) equatorial	63.3 (3)	43.1 (3)	3.5 (3)
N(1) axial	63.6 (3)	35.0 (3)	6.5 (3)
N(5) axial	62.4 (3)	39.5 (3)	1.4 (3)

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1979). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *GX*. Molecular graphics: *ORTEP* (Johnson, 1971). Software used to prepare material for publication: *GX*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1074). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Non-Sinusoidal Structure of the 1:1 Complex of Phenothiazine and 7,7,8,8-Tetracyanoquinodimethane

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Abstract

The title compound, $C_{12}H_9NS.C_{12}H_4N_4$, has a different space group to that reported by Kobayashi [*Acta Cryst.* (1974), **B30**, 1010–1017] and does not present any disorder or sinusoidal modulation, in contrast to the first study of this compound. In particular, no N···N distance of about 3.3 Å was observed.

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