Table 3. *Hydrogen-bond geometry* (Å, °), *least-squares plane referenced to the crystallographic axes and shifts of atoms from the plane* (Å)

Hydrogen bonds

	O/N—H	H···O	O/N···O	O/N—H···O
O(11)—H(2)···O(13)	0.80 (3)	1.77 (3)	2.563 (2)	173 (2)
N(1)—H(7)···O(13)	0.89 (3)	1.89 (3)	2.769 (2)	171 (3)
N(1)—H(8)···O(12)	0.84 (3)	2.03 (3)	2.823 (2)	157 (2)
N(3)—H(3)···O(12)	0.90 (3)	1.75 (3)	2.655 (2)	177 (2)

Equation of the pyridine ring plane

$-12.831\,(9)\,x + 0.369\,(9)\,y - 1.0\,(1)\,z = -5.190\,(7)$

Deviations of atoms used for defining the plane

C(1)	0.005 (2)	C(5)	−0.001 (2)
C(3)	−0.004 (2)	C(6)	−0.004 (2)
C(4)	0.005 (2)	N(3)	−0.002 (2)

Deviations of atoms excluded from the plane calculation

N(1)	0.016 (2)	N(2)	0.019 (2)
H(7)	0.17 (3)	O(21)	−0.101 (2)
H(8)	−0.04 (2)	O(22)	0.149 (3)

Crystals of the title compound were prepared by dissolving 2-amino-5-nitropyridine (0.01 *M* $C_5H_5N_3O_2$) in an aqueous solution of phosphorous acid (0.01 *M* H_3PO_3) at 320 K. Slow evaporation at room temperature yields large prismatic crystals. The chemical formula has been established on the basis of the structural determination. The crystal structure was solved by direct methods using *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). H atoms were located by difference Fourier syntheses. Refinements were run using anisotropic full-matrix least squares for non-H atoms, isotropic for H atoms. The Enraf-Nonius (1977) *SDP* program operating on a MicroVAX II computer was used for all calculations. Molecular graphics were prepared using *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55758 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1015]

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A Twist-Boat 1,3,5-Triaryl-1,3,5-triazacyclohexane: X-ray Analysis of the Inclusion Compound Formed Between 1,3,5-Tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane and Pyridine

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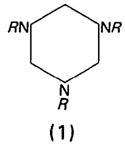
Abstract

The triazacyclohexane ring adopts a twist-boat conformation with torsion angles −25.3 (2), −41.3 (2), 70.3 (2), −27.7 (2), −35.8 (2) and 66.0 (2)°. The N—C(aryl) bonds from the ring have lengths of 1.356 (3), 1.382 (3) and 1.400 (3) Å and are inclined at 3.3 (2), 21.2 (2) and 29.5 (3)° to the CH_2—N—CH_2 planes. The N—C bond lengths in the triazacyclohexane ring are 1.442 (3)–1.474 (3) Å. The pyridine molecules are accommodated in an ordered manner in channels aligned along the screw axes parallel to **b**.

Comment

X-ray investigations of 1,3,5-trialkyl and 1,3,5-triaryl derivatives of 1,3,5-triazacyclohexane (1) have consistantly found the expected chair conformation with pyramidal arrangement of bonds at N atoms (Giumanini, Verardo, Randaccio, Bresciani-Pahor & Traldi, 1985; Zangrando, Poggi, Giumanini & Verardo, 1987; Giumanini, Verardo, Zangrando & Lassiani, 1987; Sim, 1987; Bouchemma, McCabe & Sim, 1988, 1989, 1990). We now report a study which has enabled us to isolate and examine the alternative twist-boat conformation.



(1)

The difference in enthalpy between the chair and twist-boat conformers of cyclohexane is in excess of 20 kJ mol^{-1} (Hendrickson, 1961; Allinger, Hirsch, Miller, Tyminski & Van-Catledge, 1968; Pickett & Strauss, 1970; Wiberg & Boyd, 1972; Komornicki & McIvor, 1973; Squillacote, Sheridan, Chapman & Anet, 1975) and at normal temperatures the twist-boat form is confined to substituted cyclohexanes in which *syn*-axial interactions in the chair form are relieved by conformational conversion to the twist-boat form. The differential is lowered appreciably by introduction of the carbonyl group and various derivatives of cyclohexanone have been shown to exist in the twist-boat form (Eliel, Allinger, Angyal & Morrison, 1965). The twist-boat conformation is predominant in cyclohexane-1,4-dione and has been studied in the crystal structures of derivatives of this compound (Groth & Hassel, 1964, 1965; Mossel & Romers, 1964; Groth, 1968).

The conversion of cyclohexane to cyclohexanone involves the replacement of a tetrahedral sp^3 C atom by a trigonal sp^2 C atom and this raises the question of whether a twist-boat 1,3,5-triaryl-1,3,5-triazacyclohexane can be obtained if the N atoms have a coplanar, rather than a pyramidal, arrangement of bonds. Since the N atom in *N*,*N*-dimethylaniline has a pyramidal bonding pattern (Cervellati, Borgo & Lister, 1982) whereas the corresponding atom in *p*-nitro-*N*,*N*-dimethylaniline has a coplanar arrangement of bonds (Mak & Trotter, 1965), the compound 1,3,5-tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane [(1), R = *p*-$NO_2C_6H_4$] was selected for X-ray crystallographic analysis. The insolubility of the compound precluded its crystallization from most common solvents. However, slow crystallization from pyridine gave large lustrous prisms of the 1:1 inclusion compound of (1) (R = *p*-$NO_2C_6H_4$) and pyridine. The crystal faces of the inclusion compound became cloudy on standing, suggesting slow loss of occluded solvent.

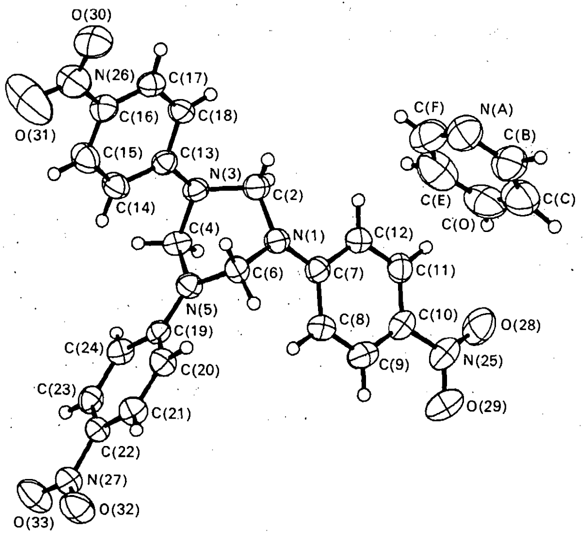


Fig. 1. Molecular structure and atomic numbering of 1,3,5-tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane. The thermal ellipsoids of the C and N atoms are drawn at the 50% probability level and H atoms are represented by spheres of radius 0.1 Å.

Atomic coordinates are listed in Table 1, molecular dimensions in Table 2 and the torsion angles that define the conformation of the triazacyclohexane ring in Table 3. Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular geometry and Fig. 2 illustrates the molecular packing in the crystal.

The triazacyclohexane adopts the twist-boat conformation in contrast to the other triazacyclohexanes that have been examined crystallographically, though the conformation departs a little from the ideal twist-boat form (Table 3), presumably in response to crystal packing requirements. A substituted twist-boat cyclohexane shows a similar skewing (Table 3). The twist-boat conformation of cyclohexane is flexible; there is pseudorotation through a sequence of twist-boat and boat forms with only small differences in energy and the torsion angles are easily altered (Hendricksen, 1961; Pickett & Strauss, 1970; van de Graaf, Baas & van Veen, 1980).

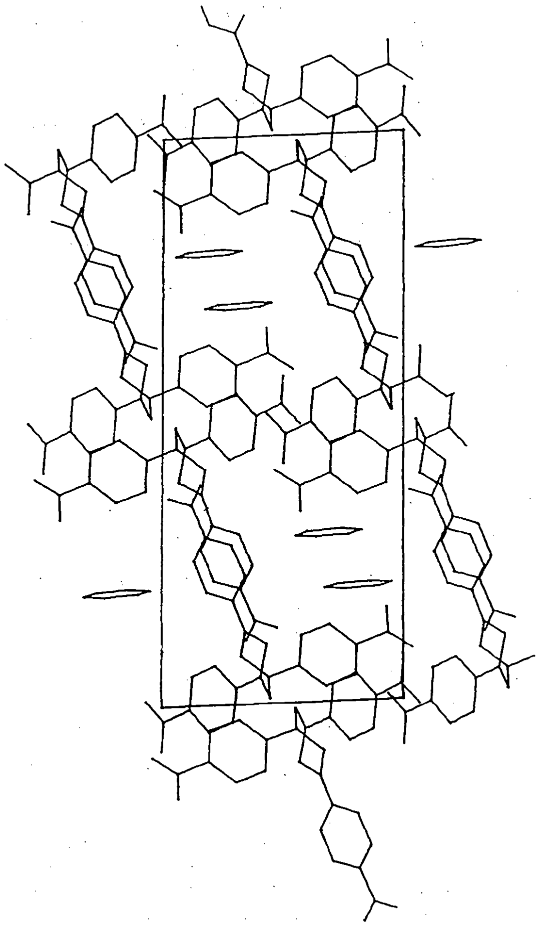


Fig. 2. Packing of molecules in the unit cell, viewed along the *b* axis.

Although the finding of the twist-boat conformation was not unexpected, the argument about coplanar and pyramidal bonding patterns that led us to examine the *p*-nitrophenyl derivative must be regarded as dubious. 1,3,5-Triacetyl-1,3,5-triazacyclohexane [(1), $R = COCH_3$] has coplanar bonds at the N atoms but the molecule adopts a chair conformation in the crystal (Choi, Santoro & Marinkas, 1975). Moreover, the N—C(aryl) bonds from the triazacyclohexane nucleus in the *p*-nitrophenyl compound are tilted at 21.2 (2), 29.5 (3) and 3.3 (2)° to the CH_2-N-CH_2 planes at N(1), N(3) and N(5), respectively, so that the arrangement of N—C bonds is coplanar at only one of the N atoms, N(5). These out-of-plane angles are, nevertheless, smaller than the angles in other triazacyclohexanes, *e.g.* 32.6-48.5 (4)° in the *o*-, *m*- and *p*-fluorophenyl compounds (Bouchemma, McCabe & Sim, 1989). The conformation found in a solid is not necessarily the predominant conformation of a set of molecules in solution or in the vapour phase. Unambiguous examples of packing forces influencing molecular conformation occur in polymorphic forms of crystals (Truter, 1983); two crystalline forms of humulene nitrosite, for example, contain distinctly different conformers (Khan, MacAlpine, Porte & Sim, 1983). Moreover, incorporation of an inclusion compound may cause a molecule to adopt a conformation that differs from the minimum-energy conformation because of the host-guest interactions (Freer, Gilmore, MacNicol & Wilson, 1980). The compound 3,3,6,6-tetramethyl-1,2,4,5-tetrathiane, for example, crystallizes in the twist-boat conformation (Korp, Bernal, Watkins & Fronczek, 1981), whereas the inclusion compound it forms with hexakis(*p*-*tert*-butylphenylthiomethyl)benzene contains the chair conformation (MacNicol & Murphy, 1981); calculations by molecular mechanics indicate that the chair form is less stable than the twist-boat form by 3 kJ mol^{-1} (Allinger, Hickey & Kao, 1976). The adoption of the twist-boat conformation by the 1,3,5-tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane molecule in the pyridine inclusion compound may represent another example of a response to host-guest interactions and crystal-packing forces. An X-ray study of the unsolvated material would throw light on this matter but, unfortunately, suitable crystals have not yet been obtained.

The exocyclic N—C(aryl) bonds from the triazacyclohexane ring have lengths 1.356 (3), 1.382 (3) and 1.400 (3) Å, with the shortest bond associated with the smallest N—C(aryl) out-of-plane angle [3.3 (2)°] and the longest bond associated with the largest out-of-plane angle [29.5 (3)°]. The correlation is connected with the quinonoid character of a *p*-nitroaniline fragment decreasing as the bonding pattern at N(amino) becomes more pyramidal. The N—C(aryl) bonds in the *o*-, *m*- and *p*-fluorophenyl compounds are generally a little larger, 1.398-1.427 (3) Å, mean 1.416 Å, in agreement with the larger N—C(aryl) out-of-plane angles in these compounds. The N—CH_2 bond lengths in the triazacyclohexane nucleus, 1.442(3)-1.474 (3) Å, mean 1.461 Å, are very similar to these in the *o*-, *m*- and *p*-fluorophenyl compounds, 1.443-1.479 (4) Å, mean 1.460 Å (Bouchemma, McCabe & Sim, 1989). The CH_2—N—CH_2 angles are 110.6 (2)-112.5 (2)° and the N—CH_2—N angles are 108.3 (2)-108.9 (2)°, whereas in the *o*-, *m*- and *p*-fluorophenyl compounds the corresponding angles are CH_2—N—CH_2 108.2-110.7 (3)° and N—CH_2—N 110.5-113.0 (3)°. In the pyridine molecule the N—C bonds, 1.300 (5) and 1.312 (5) Å, are a little shorter than the C—C bonds, 1.327 (7)-1.430 (6) Å, mean 1.368 (6) Å.

Experimental

Crystal data

$C_{26}H_{23}N_7O_6$
$M_r = 529.57$
Monoclinic
$P2_1/n$
$a = 9.848\ (3)$ Å
$b = 10.636\ (3)$ Å
$c = 23.216\ (4)$ Å
$\beta = 93.110\ (20)°$
$V = 2482\ (2)$ Å^3
$Z = 4$

$D_x = 1.449$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10.7$-$16.5°$
$\mu = 0.99$ cm^{-1}
$T = 293$ K
Prism
0.30 × 0.40 × 0.40 mm
Yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: none
5022 measured reflections
4432 independent reflections
3026 observed reflections [$I > 2.5\sigma(I)$]

$R_{\text{int}} = 0.024$
$\theta_{\text{max}} = 25°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -27 \rightarrow 27$
2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
Final $R = 0.039$
$wR = 0.050$
$S = 2.42$
3026 reflections
446 parameters
Only coordinates of H atoms refined
Weighting scheme based on measured e.s.d.'s
$w = 1/[\sigma^2(F)]$

$(\Delta/\sigma)_{\text{max}} = 0.001$
$\Delta\rho_{\text{max}} = 0.49$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.20$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Initially all non-H atoms of the pyridine were treated as C; inspection of the thermal parameters and location of the five H atoms unambiguously identifed the N atom and established that the pyridine molecule has an ordered orientation. Refinement of the occupation parameter for the pyridine molecule converged at 0.985 (3), indicating essentially complete occupation of the solvent site.

Data collection: CAD-4 (Enraf-Nonius). Cell refinement: CAD-4 (Enraf-Nonius). Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *GX*. Program(s) used to refine structure: *GX*. Molecular graphics: *ORTEP* (Johnson, 1965; Mallinson & Muir, 1985). Software used to prepare material for publication: *GX*.

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

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

	x	*y*	*z*	$U_{\rm eq}$
C(*B*)	0.7298 (3)	0.6327 (3)	−0.2066 (1)	0.084
C(*C*)	0.8632 (4)	0.6530 (5)	−0.2085 (1)	0.094
C(*D*)	0.9488 (4)	0.5566 (5)	−0.2059 (1)	0.094
C(*E*)	0.8995 (4)	0.4361 (5)	−0.1998 (1)	0.096
C(*F*)	0.7554 (3)	0.4250 (4)	−0.1965 (1)	0.082
C(2)	0.5517 (3)	0.2624 (2)	0.0093 (1)	0.048
C(4)	0.6753 (2)	0.1511 (2)	0.0847 (1)	0.041
C(6)	0.5688 (2)	0.3516 (2)	0.1055 (1)	0.041
C(7)	0.69209 (19)	0.45359 (17)	0.02885 (8)	0.040
C(8)	0.7782 (2)	0.5180 (2)	0.0686 (1)	0.047
C(9)	0.8740 (2)	0.6014 (2)	0.0510 (1)	0.052
C(10)	0.8858 (2)	0.6215 (2)	−0.0072 (1)	0.046
C(11)	0.8024 (2)	0.5608 (2)	−0.0476 (1)	0.047
C(12)	0.7060 (2)	0.4780 (2)	−0.0302 (1)	0.046
C(13)	0.43543 (19)	0.09717 (17)	0.06383 (8)	0.038
C(14)	0.4279 (2)	0.0449 (2)	0.1188 (1)	0.048
C(15)	0.3086 (2)	−0.0072 (2)	0.1356 (1)	0.056
C(16)	0.1959 (2)	−0.0070 (2)	0.0986 (1)	0.047
C(17)	0.2003 (2)	0.0420 (2)	0.0437 (1)	0.044
C(18)	0.3194 (2)	0.0931 (2)	0.0268 (1)	0.043
C(19)	0.70872 (19)	0.24436 (17)	0.18352 (7)	0.036
C(20)	0.6583 (2)	0.3224 (2)	0.2265 (1)	0.042
C(21)	0.7149 (2)	0.3201 (2)	0.2816 (1)	0.044
C(22)	0.82145 (19)	0.23869 (17)	0.29580 (7)	0.039
C(23)	0.8705 (2)	0.1576 (2)	0.2551 (1)	0.041
C(24)	0.81494 (19)	0.16042 (18)	0.19952 (8)	0.039
N(*A*)	0.6745 (2)	0.5217 (3)	−0.2004 (1)	0.077
N(1)	0.59323 (17)	0.37087 (15)	0.04541 (6)	0.042
N(3)	0.55628 (16)	0.14974 (14)	0.04571 (6)	0.040
N(5)	0.65739 (17)	0.24993 (14)	0.12818 (6)	0.040
N(25)	0.99102 (19)	0.70584 (17)	−0.02646 (9)	0.056
N(26)	0.0682 (2)	−0.0553 (2)	0.1179 (1)	0.068
N(27)	0.88302 (18)	0.24072 (16)	0.35350 (7)	0.048
O(28)	0.99965 (19)	0.72266 (16)	−0.07837 (8)	0.077
O(29)	1.06732 (18)	0.75546 (16)	0.01005 (8)	0.075
O(30)	−0.03641 (17)	−0.03841 (17)	0.08927 (7)	0.072
O(31)	0.0705 (2)	−0.1111 (3)	0.1634 (1)	0.163
O(32)	0.84281 (18)	0.31861 (15)	0.38795 (6)	0.066
O(33)	0.97351 (18)	0.16439 (16)	0.36663 (6)	0.069

Table 2. *Geometric parameters* (Å, °)

C(*B*)—C(*C*)	1.334 (6)	C(13)—C(18)	1.393 (3)
C(*B*)—N(*A*)	1.312 (5)	C(13)—N(3)	1.400 (3)
C(*C*)—C(*D*)	1.327 (7)	C(14)—C(15)	1.375 (4)
C(*D*)—C(*E*)	1.380 (7)	C(15)—C(16)	1.367 (4)
C(*E*)—C(*F*)	1.430 (6)	C(16)—C(17)	1.379 (3)
C(*F*)—N(*A*)	1.300 (5)	C(16)—N(26)	1.452 (3)
C(2)—N(1)	1.471 (3)	C(17)—C(18)	1.369 (3)
C(2)—N(3)	1.465 (3)	C(19)—C(20)	1.409 (3)
C(4)—N(3)	1.442 (3)	C(19)—C(24)	1.410 (3)
C(4)—N(5)	1.474 (3)	C(19)—N(5)	1.356 (3)
C(6)—N(1)	1.443 (3)	C(20)—C(21)	1.368 (3)
C(6)—N(5)	1.469 (3)	C(21)—C(22)	1.386 (3)
C(7)—C(8)	1.399 (3)	C(22)—C(23)	1.386 (3)
C(7)—C(12)	1.408 (3)	C(22)—N(27)	1.440 (3)
C(7)—N(1)	1.382 (3)	C(23)—C(24)	1.375 (3)
C(8)—C(9)	1.373 (4)	N(25)—O(28)	1.226 (3)
C(9)—C(10)	1.380 (4)	N(25)—O(29)	1.222 (3)
C(10)—C(11)	1.374 (4)	N(26)—O(30)	1.208 (3)
C(10)—N(25)	1.459 (3)	N(26)—O(31)	1.211 (4)
C(11)—C(12)	1.372 (4)	N(27)—O(32)	1.232 (3)
C(13)—C(14)	1.398 (3)	N(27)—O(33)	1.232 (3)
C(*C*)—C(*B*)—N(*A*)	124.3 (4)	C(20)—C(19)—N(5)	121.2 (2)
C(*B*)—C(*C*)—C(*D*)	119.9 (5)	C(24)—C(19)—N(5)	120.7 (2)
C(*C*)—C(*D*)—C(*E*)	119.7 (4)	C(19)—C(20)—C(21)	120.7 (2)
C(*D*)—C(*E*)—C(*F*)	116.0 (4)	C(20)—C(21)—C(22)	119.9 (2)
C(*E*)—C(*F*)—N(*A*)	122.5 (4)	C(21)—C(22)—C(23)	121.0 (2)
N(1)—C(2)—N(3)	108.3 (2)	C(21)—C(22)—N(27)	119.1 (2)
N(3)—C(4)—N(5)	108.3 (2)	C(23)—C(22)—N(27)	119.9 (2)
N(1)—C(6)—N(5)	108.9 (2)	C(22)—C(23)—C(24)	119.4 (2)
C(8)—C(7)—C(12)	117.6 (2)	C(19)—C(24)—C(23)	120.9 (2)
C(8)—C(7)—N(1)	122.6 (2)	C(*B*)—N(*A*)—C(*F*)	117.6 (3)
C(12)—C(7)—N(1)	119.7 (2)	C(2)—N(1)—C(6)	112.5 (2)
C(7)—C(8)—C(9)	121.5 (3)	C(2)—N(1)—C(7)	121.3 (2)
C(8)—C(9)—C(10)	119.2 (3)	C(6)—N(1)—C(7)	121.1 (2)
C(9)—C(10)—C(11)	121.2 (2)	C(2)—N(3)—C(4)	110.6 (2)
C(9)—C(10)—N(25)	119.6 (2)	C(2)—N(3)—C(13)	120.0 (2)
C(11)—C(10)—N(25)	119.2 (2)	C(4)—N(3)—C(13)	119.4 (2)
C(10)—C(11)—C(12)	119.8 (3)	C(4)—N(5)—C(6)	111.7 (2)
C(7)—C(12)—C(11)	120.8 (2)	C(4)—N(5)—C(19)	124.4 (2)
C(14)—C(13)—C(18)	118.0 (2)	C(6)—N(5)—C(19)	123.8 (2)
C(14)—C(13)—N(3)	121.4 (2)	C(10)—N(25)—O(28)	118.6 (2)
C(18)—C(13)—N(3)	120.6 (2)	C(10)—N(25)—O(29)	118.3 (2)
C(13)—C(14)—C(15)	120.5 (2)	O(28)—N(25)—O(29)	123.1 (2)
C(14)—C(15)—C(16)	119.8 (3)	C(16)—N(26)—O(30)	120.3 (2)
C(15)—C(16)—C(17)	121.1 (3)	C(16)—N(26)—O(31)	117.9 (3)
C(15)—C(16)—N(26)	119.5 (2)	O(30)—N(26)—O(31)	121.8 (3)
C(17)—C(16)—N(26)	119.4 (2)	C(22)—N(27)—O(32)	118.7 (2)
C(16)—C(17)—C(18)	119.0 (2)	C(22)—N(27)—O(33)	118.9 (2)
C(13)—C(18)—C(17)	121.5 (2)	O(32)—N(27)—O(33)	122.4 (2)
C(20)—C(19)—C(24)	118.1 (2)		

Table 3. *Torsion angles* (°) *in twist-boat six-membered rings*

Column *A* contains the results for 1,3,5-tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane, column *B* the results for 1,3-dihydroxymethyl-5-*tert*-butylcyclohexane (van Koningsveld, 1981) and column *C* the results for an ideal twist-boat cyclohexane (Bucourt & Hainaut, 1965).

Bond	*A*	*B*	*C*
1—2	−25.3 (2)	−20.2	−31
2—3	−41.3 (2)	−38.8	−31
3—4	70.3 (2)	62.2	65
4—5	−27.7 (2)	−22.6	−31
5—6	−35.8 (2)	−38.1	−31
6—1	66.0 (2)	61.1	65

1,3,5-Tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane was synthesized in 43% yield by reaction of *p*-nitroaniline and dimethyl sulfoxide in the presence of phosphorus pentoxide (Lerch & Moffatt, 1971). These crystals were unsolvated. Analysis found C 56.11, H 4.11, N 18.82%; $C_{21}H_{18}N_6O_6$ requires C 56.00, H 4.02, N 18.65%; *m*/*Z* 300, 150, 120, 104, 92 and 77; $\lambda_{\rm max}$(DMSO) = 356 nm; $\nu_{\rm max}$ (KBr) = 1596, 1509, 1330, 1233, 950 and 835 cm^{-1}; $\delta_{\rm H}$ (DMSO-d_6, 90 MHz) 5.35 (*s*, CH$_2$), 7.21 and 8.09 (*d*, *JZ* = 9 Hz, Ar) in the area ratio of 1:1:1; (DMSO-d_6, 50.3 MHz) 51.0 (CH$_2$), 111.8 and 128.2 (Ar CH), 138.9 and 153.4 (Ar CN). Slow crystallization (6 weeks) from anhydrous pyridine in a desiccator over silca gel gave the 1:1 pyridine inclusion compound as deep yellow prisms, m.p. (decomp.) 563-564 K. Analysis found C 58.84, H 4.10, N 18.41%; $C_{21}H_{18}N_6O_6.C_5H_5N$ requires C 58.97, H 4.38, N 18.52%; $\delta_{\rm H}$ (DMSO-d_6) 5.33 (6H, *s*, CH$_2$), 7.18 (6H, *d*, *J* = 10 Hz; Ar) and 8.10 (*ca* 6H, *d*, *J* = 10 Hz; Ar) partly obscured by pyridine resonances at δ 7.36 (*ca* 2H), 8.16 (*ca* 1H) and 8.57 (2H).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55779 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1035]

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5-(3-Iodophényl)-2,3-dihydro-5-hydroxy-5*H*-imidazo[2,1-*a*]isoindole

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Abstract

5-(3-Iodophenyl)-2,3-dihydro-5*H*-imidazo[2,1-*a*]isoindol-5-ol. The pentagonal *A* and *B* rings show an envelope conformation and the hexagonal *C* and *D* rings are planar. The dihydroimidazoisoindole group is approximately planar and its least-squares plane makes an angle of 91.3 (2)° with that of ring *D*. The molecules form dimers in which they are related by a centre of symmetry and linked together by the O(19)—H(19)···N(5^{i}) hydrogen bond [(i) $-x$, $1-y$, $-z$; 2.798 (5) Å, 177°]. The rotational barrier around the C(1)—C(3) bond (20.1 kJ mol^{-1}) has been calculated using the AM1 semi-empirical method. Its relatively low value suggests that the molecular geometry may be easily modified in solution.

Commentaire

Le mazindol est un inhibiteur du recaptage de la dopamine. La détermination de sa structure a été entreprise dans le cadre de l'étude des relations entre la structure moléculaire et l'activité pharmacologique. L'exploration fonctionnelle du cerveau à l'aide de la méthode SPECT (Single-Photon-Emission-Computed Tomography) nécessite l'utili-