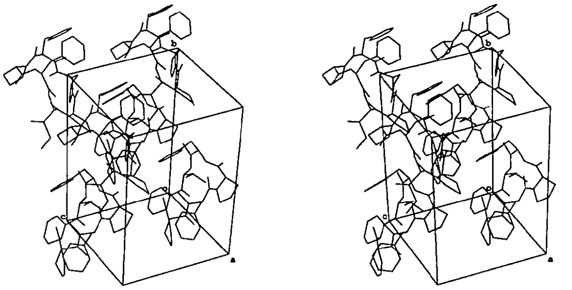

Fig. 3. Stereoview of the molecular packing around a unit cell. The view selected shows more than the contents of a single unit cell.

to 12·9°) with no unusual bond lengths or angles. The χ_1 angles for Ile and the two Phe residues have values indicating standard staggered conformations for the side-chain substituents.

A packing diagram is shown in Fig. 3. There are no intermolecular distances shorter than 3·35 Å and thus no hydrogen bonding between molecules. While this lack of hydrogen bonding is unusual it is not unique and because only N1 and N8 have suitable protons and these are both pointing into the macrocycle it is not that surprising that there are no intermolecular hydrogen bonds. As is evident from the packing diagram there are no apparent stacking interactions of unsaturated systems either and thus it is presumed that only weak van der Waals interactions are present in the crystal structure.

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References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Bardi, R., Piazzesi, A. M., Toniolo, C., Jensen, O. E., Andersen, T. P. & Senning, A. (1988). *Tetrahedron*, **44**, 761–769.

Bock, M. G., DiPardo, R. M., Williams, P. D., Pettibone, D. J., Clineschmidt, B. V., Ball, R. G., Veber, D. F. & Freidinger, R. M. (1990). *J. Med. Chem.* **33**, 2321–2323.

Brown, J. N. & Teller, R. G. (1976). *J. Am. Chem. Soc.* **98**, 7565–7569.

Brown, J. N. & Yang, C. H. (1979). *J. Am. Chem. Soc.* **101**, 445–449.

Enraf–Nonius (1985). *SDP-Plus Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Hansen, H. A. S., Clausen, K. & La Cour, T. F. M. (1987). *Acta Cryst.* C**43**, 519–522, 522–524.

IUPAC–IUB Joint Commission on Biochemical Nomenclature (1984). *Eur J. Biochem.* **138**, 9–37.

Jensen, O. E., Lawesson, S.-O., Bardi, R., Piazzesi, A. M. & Toniolo, C. (1985). *Tetrahedron*, **41**, 5595–5606.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kessler, H., Bats, J. W., Griesinger, C., Koll, S., Will, M. & Wagner, K. (1988). *J. Am. Chem. Soc.* **110**, 1033–1049.

Kessler, H., Klein, M., Wagner, K., Bats, J. W., Zeigler, K. & Frimmer, M. (1986). *Angew. Chem. Int. Ed. Engl.* **25**, 997–999.

Schaumann, E., Kausch, E., Klaska, K.-H., Klaska, R. & Jarchow, O. (1977). *J. Heterocycl. Chem.* **14**, 857–860.

Schmid, U., Heimgartner, H., Schmid, H. & Oberhansli, W. E. (1976). *Helv. Chim. Acta*, **59**, 2768–2785.

Sheldrick, G. M. (1985). *SHELXS*86. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189, Oxford Univ. Press.

Sherman, D. B. & Spatola, A. F. (1990). *J. Am. Chem. Soc.* **112**, 433–441.

Spatola, A. F. (1983). *Chemistry and Biochemistry of Amino Acids, Peptides and Proteins*, Vol. 7, edited by B. Weinstein, pp. 267–357. New York: Decker.

Stout, G. H. & Jensen, L. H. (1968). *X-ray Structure Determination*. New York: Macmillan.

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

Acta Cryst. (1991). C**47**, 1219–1222

Conformation of 1-*p*-Fluorophenyl-3,5-bis(iodomethyl)piperidine

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Abstract. $C_{13}H_{16}FI_2N$, $M_r = 459{\cdot}14$, monoclinic, $P2_1/n$, $a = 16{\cdot}771\ (3)$, $b = 8{\cdot}522\ (2)$, $c = 10{\cdot}782\ (2)$ Å, $\beta = 103.18\ (2)^\circ$, $V = 1500\ (1)$ Å^3, $Z = 4$, $D_x = 2{\cdot}02$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 4{\cdot}2$ mm^{-1}, $F(000) = 864$, $T = 293$ K, $R = 0{\cdot}048$ for 2377 independent observed reflections. The piperidine ring adopts a chair conformation with the three substituent groups in equatorial positions. The N—C(aryl) bond is inclined at 42·6 (7)° to the C(2)—N(1)—C(6) plane of the piperidine. The torsion angles in the piperidine ring are 53·4–60·2 (6)°, with the largest angles adjacent to N(1) and the smallest adjacent to C(4).

Introduction. Lone pairs of electrons strongly influence the conformational properties of heterocyclic

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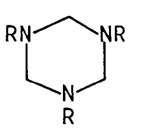
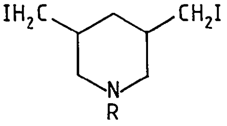
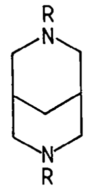
compounds (Riddell, 1980) and various 1,3,5-triaryl-1,3,5-triazacyclohexanes (1) adopt the diaxial–equatorial orientation of substituents in the solid state, thus avoiding 1,3-diaxial lone-pair repulsions (Giumanini, Verardo, Randaccio, Bresciani-Pahor & Traldi, 1985; Bouchemma, McCabe & Sim, 1989). The title compound [(2) $R = p\text{-}FC_6H_4$] was prepared in the course of the synthesis of derivatives of 3,7-diazabicyclo[3.3.1]nonane (3) and an X-ray study of (2) ($R = p\text{-}FC_6H_4$) was undertaken to compare the structural parameters with results available for 1,3,5-tris(*p*-fluorophenyl)-1,3,5-triazacyclohexane [(1) $R = p\text{-}FC_6H_4$] (Bouchemma *et al.*, 1989) and for phenylcyclohexane (Allinger & Tribble, 1971; Eliel, 1985; Hodgson, Rychlewska, Eliel, Manoharan, Knox & Olefirowicz, 1985; Squillacote & Neth, 1987).



(1)



(2)



(3)

Experimental. 1,5-Diiodo-2,4-bis(iodomethyl)pentane (Zefirov & Rogozina, 1974) was stirred with *p*-fluoroaniline in refluxing toluene (bath temperature 400 K) for 72 h. After purification by chromatography on a column of silica gel HF 254 with elution by chloroform–petrol, the product (2) was crystallized from ethanol (yield *ca* 15%). M.p. 346–348 K. Found: C 34·13, H 3·22, N 2·99%; $C_{13}H_{16}FI_2N$ requires C 34·01, H 3·51, N 3·05%. m/z 459 (M), 332 ($M - I$), 205 ($M - 2I$), 162, 147, 137 (C_8H_8FN) and 121. ν_{max} ($CHCl_3$) 3020, 2940, 1505, 1458, 1440, 1422, 972, 920 cm^{-1}. δ_H ($CDCl_3$, 200 MHz) 0·87 [1H, *dt*, J = 12, 12 Hz, C(4)H$_{ax}$], 1·9 [2H, *m*, C(3)H, C(5)H], 2·14 [1H, *dm*, J = 12 Hz, C(4)H$_{eq}$], 2·32 [2H, *dd*, J = 12, 12 Hz, C(2)H$_{ax}$, C(6)H$_{ax}$], 3·07 (2H, *dd*, J = 10, 7 Hz) and 3·16 (2H, *dd*, J = 10, 6 Hz) [C(7)H$_2$, C(8)H$_2$], 3·70 [2H, *dt*, J = 12, 1·8 Hz, C(2)H$_{eq}$, C(6)H$_{eq}$], 6·8–7·1 (4H, *m*, Ar). δ_c ($CDCl_3$, 50·3 MHz) 9·36 [C(7), C(8)], 37·57 [C(3), C(5)], 38·59 [C(4)], 57·11 [C(2), C(6)], 115·66 [*d*, J_{CCF} = 22 Hz, C(11), C(13)], 118·65 [*d*, J_{CCCF} = 8 Hz, C(10), C(14)], 147·32 [C(9)], 157·22 [*d*, J_{CF} = 239 Hz, C(12)].

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

$U_{eq} = (U_1U_2U_3)^{1/3}$.

	x	y	z	U_{eq}
I(1)	0·71464 (3)	0·08936 (7)	0·81648 (4)	0·061
I(2)	1·08759 (3)	−0·11964 (8)	1·35041 (5)	0·065
F	0·4689 (3)	−0·1928 (7)	1·4261 (5)	0·085
N(1)	0·7488 (3)	−0·0801 (6)	1·2499 (5)	0·034
C(2)	0·7410 (4)	−0·0932 (8)	1·1126 (5)	0·038
C(3)	0·8170 (4)	−0·0277 (8)	1·0764 (5)	0·036
C(4)	0·8938 (4)	−0·1127 (9)	1·1488 (6)	0·040
C(5)	0·8997 (4)	−0·1065 (8)	1·2913 (5)	0·036
C(6)	0·8199 (4)	−0·1678 (9)	1·3214 (6)	0·040
C(7)	0·8125 (5)	−0·0421 (11)	0·9337 (6)	0·053
C(8)	0·9690 (4)	−0·2024 (11)	1·3687 (8)	0·056
C(9)	0·6761 (4)	−0·1087 (7)	1·2920 (6)	0·036
C(10)	0·6031 (4)	−0·1534 (9)	1·2087 (6)	0·046
C(11)	0·5329 (4)	−0·1787 (10)	1·2554 (7)	0·053
C(12)	0·5360 (4)	−0·1608 (9)	1·3805 (7)	0·054
C(13)	0·6067 (5)	−0·1144 (9)	1·4650 (7)	0·054
C(14)	0·6755 (5)	−0·0844 (9)	1·4199 (6)	0·047

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

I(1)—C(7)	2·145 (9)	I(2)—C(8)	2·161 (8)
F—C(12)	1·354 (9)	N(1)—C(2)	1·461 (8)
N(1)—C(6)	1·468 (9)	N(1)—C(9)	1·416 (9)
C(2)—C(3)	1·522 (10)	C(3)—C(4)	1·527 (10)
C(3)—C(7)	1·528 (9)	C(4)—C(5)	1·518 (9)
C(5)—C(6)	1·539 (10)	C(5)—C(8)	1·507 (11)
C(9)—C(10)	1·396 (10)	C(9)—C(14)	1·397 (10)
C(10)—C(11)	1·399 (11)	C(11)—C(12)	1·346 (11)
C(12)—C(13)	1·380 (11)	C(13)—C(14)	1·374 (11)

C(2)—N(1)—C(6)	111·6 (5)	C(2)—N(1)—C(9)	115·3 (5)
C(6)—N(1)—C(9)	113·6 (5)	N(1)—C(2)—C(3)	110·2 (5)
C(2)—C(3)—C(4)	110·7 (6)	C(2)—C(3)—C(7)	112·1 (6)
C(4)—C(3)—C(7)	108·6 (6)	C(3)—C(4)—C(5)	110·8 (6)
C(4)—C(5)—C(6)	110·0 (6)	C(4)—C(5)—C(8)	114·0 (6)
C(6)—C(5)—C(8)	107·3 (6)	N(1)—C(6)—C(5)	111·0 (6)
I(1)—C(7)—C(3)	114·0 (6)	I(2)—C(8)—C(5)	112·4 (6)
N(1)—C(9)—C(10)	122·3 (6)	N(1)—C(9)—C(14)	119·4 (6)
C(10)—C(9)—C(14)	118·2 (6)	C(9)—C(10)—C(11)	119·8 (7)
C(10)—C(11)—C(12)	120·0 (7)	F—C(12)—C(11)	119·7 (7)
F—C(12)—C(13)	118·6 (7)	C(11)—C(12)—C(13)	121·7 (8)
C(12)—C(13)—C(14)	119·0 (7)	C(9)—C(14)—C(13)	121·2 (7)

Crystal dimensions 0·15 × 0·35 × 0·50 mm. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, generator settings 50 kV, 20 mA. Cell dimensions from setting angles of 25 reflections with θ 13–20°; 3612 reflections surveyed in range θ 1·5–27·0°; h 0→21, k 0→10, l −13→13; scan width (1·05 + 0·25 tanθ)°; max. scan time 100 s; 2377 independent reflections with $I > 2{\cdot}5\sigma(I)$. Two reference reflections monitored periodically showed no significant variation in intensity. Corrections applied for Lorentz–polarization effects, assuming an ideally imperfect monochromator crystal, and for absorption, using the empirical procedure *DIFABS* (Walker & Stuart, 1983) with transmission factors 0·69–1·49. Structure determined by Patterson and Fourier methods. H atoms initially placed by difference Fourier syntheses and calculation of ideal positions. Full-matrix least-squares calculations on F with anisotropic thermal parameters for C, F, I and N atoms and isotropic for H atoms. Convergence at R = 0·048, wR = 0·059, S = 2·72 for 219 parameters, $\Delta/\sigma < 0{\cdot}25$, $w = 1/\sigma^2(|F_o|)$. Final $\Delta\rho$ max. 1·14, min. = −1·23 e Å^{-3} (in region of I atoms). An alternative set of calculations with no absorption correction converged at R = 0·061 with essentially identical atomic coordinates. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations on a SEL 32/27 computer with the *GX* system of programs (Mallinson & Muir, 1985).

Discussion. Atomic coordinates are listed in Table 1 and molecular dimensions in Table 2.* Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular structure.

All three substituents have the expected equatorial orientation on the piperidine ring, whereas 1,3,5-tris(*p*-fluorophenyl)-1,3,5-triazacyclohexane [(1) R = *p*-FC_6H_4] has diaxial–equatorial orientation of substituents to avoid interactions of lone pairs of electrons at the N atoms (Bouchemma *et al.*, 1989).

The N atom in (2) (R = *p*-FC_6H_4) has pyramidal geometry, the N—C(aryl) bond being inclined at 42·6 (7)° to the C(2)—N(1)—C(6) plane of the piperdine. The analogous angles at the N atoms in 1,3,5-tris(*p*-fluorophenyl)-1,3,5-triazacyclohexane [(1) R = *p*-FC_6H_4] are a little smaller, 36·9–37·4 (3)°, while spectroscopic studies indicate that the N—C(aryl) out-of-plane angle is 46·4° in *p*-fluoroaniline (Hastie, Lister, McNeil & Tyler, 1970), 37·5–42·2° in aniline (Lister, Tyler, Høg & Larsen, 1974; Larsen, Hansen & Nicolaisin, 1976) and 27·0° in *N*,*N*-dimethylaniline (Cervellati, Borgo & Lister, 1982). The N—C(aryl) out-of-plane angles in (1) (R = *p*-FC_6H_4) and (2) (R = *p*-FC_6H_4) are larger than the angles in (1) (R = C_6H_5), 30·9–351·2 (7)° (Giumanini *et al.*, 1985) and (3) (R = C_6H_5), 30·3 and 32·7 (8)° (Levina, Potekhin, Kurkutova, Struchkov, Palyulin & Zefirov, 1984); in conjunction with the spectroscopic results this indicates that the competing mesomeric effect of the *p*-fluoro substituent reduces the overlap between the N lone pair and the aromatic π orbitals.

A conformational comparison can be made with phenylcyclohexane for which force-field calculations and NMR measurements (Allinger & Tribble, 1971; Eliel, 1985; Hodgson *et al.*, 1985; Squillacote & Neth, 1987) have established that the equatorial conformer has the plane of the aromatic ring parallel to the symmetry plane of cyclohexane to avoid repulsive interactions between the *ortho* H atoms of the phenyl group and the equatorial H atoms at C(2) and C(6) of the cyclohexane ring. In compound (2) (R = *p*-FC_6H_4), the angle between the mean plane defined by atoms N(1), C(9), C(10), C(11), C(12), C(13), C(14), F and that defined by atoms C(4), N(1), C(9), C(12), F is 71 (1)° and the preference for a conformation in which the *p*-fluorophenyl group is nearly perpendicular to the symmetry plane of the piperdine can be attributed to a favourable energy term arising from overlap between the N lone pair and the phenyl π orbitals. A measure of such overlap in anilines is provided by the barrier to rotation around the N—C(aryl) bond and this has been estimated from spectroscopic measurements to be 21 kJ mol^{-1} in *N*,*N*-dimethylaniline (Mackenzie & MacNicol, 1970), 24 kJ mol^{-1} in aniline and 19 kJ mol^{-1} in *p*-fluoroaniline (Larsen *et al.*, 1976). The small twist away from the ideal perpendicular conformation in (2) (R = *p*-FC_6H_4) has little effect on the overlap between the N lone pair and the π orbitals of the aromatic ring ($\sin 71° = 0·95$) but increases the H(10)···H(2_{eq}) and H(14)···H(6_{eq}) separations (see Fig. 1).

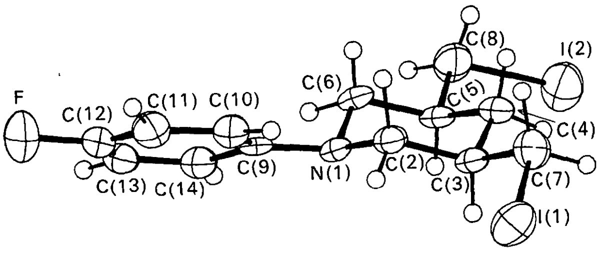


Fig. 1. Molecular structure and atomic numbering of the compound. The thermal ellipsoids of the C, F, I and N atoms are drawn at the 50% probability level and the H atoms are represented by spheres of radius 0·1 Å.

The torsion angles for the piperidine ring are in the range 53·4–60·2 (6)°, with the largest angles adjacent to N(1) and the smallest adjacent to C(4). The bond angles are 110·0–111·6 (5)°, all slightly larger than tetrahedral.

The coupling constants in the ^1H NMR spectrum of compound (2) (R = *p*-FC_6H_4) indicate that in solution the piperidine ring adopts a chair conformation with the iodomethyl groups equatorial. The large downfield shift (δ = 3·70) shown by the equatorial H atoms at C(2) and C(6) relative to the corresponding axial resonances (δ = 2·32) establishes the preferred orientation of the aromatic ring as perpendicular, or nearly perpendicular, to the symmetry plane of the piperidine ring, for in this conformation the induced diamagnetic ring current deshields the C(2), C(6) equatorial H atoms. Though the *p*-fluorophenyl ring in the solid state is tilted by 19° from the ideal perpendicular orientation the aryl H atoms H(10) and H(14) are magnetically equivalent in solution, as are H(11) and H(13). The X-ray and various NMR results are consistent with the aryl ring in solution undergoing hindered rotation around the N—C(aryl) bond with a substantial barrier at the parallel orientation and a small, or zero, barrier at the perpendicular orientation. In the ^1H NMR spectrum in benzene-d_6 the aryl resonances appear as an A_2B_2X system ($X = F$) with a twelve-peak multiplet at δ = 6·82 [H(11), H(13)] and a nine-peak multiplet at δ = 6·54 [H(10), H(14)] showing apparent couplings of 9 Hz [$J_{ortho} + J_{para}$], 8·3 Hz [$J_{H(11)F,H(13)F}$] and 4·6 Hz [$J_{H(10)F,H(14)F}$].

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53570 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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References

ALLINGER, N. L. & TRIBBLE, M. T. (1971). *Tetrahedron Lett.* pp. 3259–3262.

BOUCHEMMA, A., MCCABE, P. H. & SIM, G. A. (1989). *J. Chem. Soc. Perkin Trans.* 2, pp. 583–587.

CERVELLATI, R., BORGO, A. D. & LISTER, D. G. (1982). *J. Mol. Struct.* **78**, 161–167.

ELIEL, E. L. (1985). *J. Mol. Struct.* **126**, 385–400.

GIUMANINI, A. G., VERARDO, G., RANDACCIO, L., BRESCIANI-PAHOR, N. & TRALDI, P. (1985). *J. Prakt. Chem.* **327**, 739–748.

HASTIE, A., LISTER, D. G., MCNEIL, R. L. & TYLER, J. K. (1970). *J. Chem. Soc. Chem. Commun.* pp. 108–109.

HODGSON, D. J., RYCHLEWSKA, M., ELIEL, E. L., MANOHARAN, M., KNOX, D. E. & OLEFIROWICZ, E. M. (1985). *J. Org. Chem.* **50**, 4838–4843.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

LARSEN, N. W., HANSEN, E. L. & NICOLAISIN, F. M. (1976). *Chem. Phys. Lett.* **43**, 584–586.

LEVINA, O. I., POTEKHIN, K. A., KURKUTOVA, E. N., STRUCHKOV, YU. T., PALYULIN, V. A. & ZEFIROV, N. S. (1984). *Dokl. Akad. Nauk SSSR*, **227**, 367–370.

LISTER, D. G., TYLER, J. K., HØG, J. H. & LARSEN, N. W. (1974). *J. Mol. Struct.* **23**, 253–264.

MACKENZIE, R. K. & MACNICOL, D. D. (1970). *J. Chem. Soc. Chem. Commun.* pp. 1299–1300.

MALLINSON, P. R. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

RIDDELL, F. G. (1980). *The Conformational Analysis of Heterocyclic Compounds*, pp. 1–153. London: Academic Press.

SQUILLACOTE, M. E. & NETH, J. M. (1987). *J. Am. Chem. Soc.* **109**, 198–202.

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

ZEFIROV, N. S. & ROGOZINA, S. V. (1974). *Tetrahedron*, **30**, 2345–2352.

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Investigation of the Geometry of Substituted Dinitroanilines. II. Structural Study of *N*-Cyclohexyl-*N*-isopropyl-2,4-dinitroaniline in the Solid State and in Solution*

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Abstract. $C_{15}H_{21}N_3O_4$, monoclinic, $P2_1/n$, $M_r = 307{\cdot}3$, $a = 11{\cdot}601\ (6)$, $b = 11{\cdot}223\ (4)$, $c = 12{\cdot}143\ (4)$ Å, $\beta = 97{\cdot}75\ (2)^\circ$, $V = 1567\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}30$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}89$ cm^{-1}, $F(000) = 656$, room temperature, final $R = 0{\cdot}043$, $wR = 0{\cdot}051$ for 1948 observed independent reflections. The molecule in the solid state is significantly non-planar, the dialkyl-substituted amino and *o*-nitro groups being rotated out of the benzene ring. The isopropyl moiety is *anti* to the *ortho* substituent in order to minimize intramolecular interactions. The cyclohexane ring, perpendicular to the amino group, adopts a chair conformation. Deformations observed in the aromatic ring skeleton could not be accounted for by the additivity of the substituent effects. ^{13}C NMR data allow us to infer that the molecule in solution has the amino and *o*-nitro groups twisted out of the mean ring plane, in agreement with single-crystal results.

Introduction. It has been found that several crystalline substituted nitroanilines exhibit useful quadratic nonlinear optical properties (Barzoukas, Josse, Fremaux, Zyss, Nicoud & Morley, 1987; Panunto, Urbánczyk-Lipowska, Johnson & Etter, 1987). Therefore, we have undertaken the present study as part of an investigation of the effects of bulky substituents on optical properties, geometry and packing of *N*,*N*-dialkyl-substituted dinitroanilines (Nudelman, Socolovsky & De Waisbaum, 1982; Punte, Rivero, Socolovsky & Nudelman, 1987, 1989).

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