

Table 1. Fractional atomic coordinates and vibrational parameters (Å²)

	x	y	z	U _{eq} *
C(1)	0.3781 (5)	0.6505 (4)	0.4142 (1)	0.0419 (6)
C(2)	0.1818 (5)	0.7695 (4)	0.4195 (1)	0.0474 (6)
C(3)	0.1129 (5)	0.9351 (5)	0.3656 (2)	0.0512 (6)
N(4)	0.2233 (4)	0.9842 (4)	0.3107 (1)	0.0520 (6)
C(5)	0.4099 (5)	0.8725 (5)	0.3069 (1)	0.0505 (7)
C(6)	0.4954 (5)	0.7000 (5)	0.3568 (1)	0.0476 (6)
N(1)	0.4554 (4)	0.4656 (4)	0.4665 (1)	0.0462 (5)
O(1)	0.4432 (4)	0.2522 (3)	0.4485 (1)	0.0784 (7)
F(2)	0.0636 (3)	0.7284 (3)	0.47497 (9)	0.0720 (5)
F(3)	-0.0757 (3)	1.0550 (3)	0.3693 (1)	0.0789 (5)
F(5)	0.5231 (3)	0.9302 (3)	0.25177 (8)	0.0783 (5)
F(6)	0.6853 (3)	0.5877 (3)	0.35010 (9)	0.0745 (5)

$$*U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Table 2. Bonds (Å) and angles (°)

C(1)—C(2)	1.373 (4)	C(1)—C(6)	1.365 (3)
C(1)—N(1)	1.447 (3)	C(2)—C(3)	1.378 (4)
C(2)—F(2)	1.330 (3)	C(3)—N(4)	1.300 (3)
C(3)—F(3)	1.330 (3)	N(4)—C(5)	1.298 (4)
C(5)—C(6)	1.384 (4)	C(5)—F(5)	1.325 (3)
C(6)—F(6)	1.326 (3)	N(1)—O(1)	1.250 (3)
N(1)—N(1)'	1.320 (4)		
C(6)—C(1)—C(2)	119.4 (2)	N(1)—C(1)—C(2)	120.7 (2)
N(1)—C(1)—C(6)	119.8 (2)	C(3)—C(2)—C(1)	117.5 (2)
F(2)—C(2)—C(1)	121.3 (2)	F(2)—C(2)—C(3)	121.2 (3)
N(4)—C(3)—C(2)	123.7 (3)	F(3)—C(3)—C(2)	119.1 (2)
F(3)—C(3)—N(4)	117.1 (2)	C(5)—N(4)—C(3)	118.1 (2)
C(6)—C(5)—N(4)	123.7 (2)	F(5)—C(5)—N(4)	117.2 (2)
F(5)—C(5)—C(6)	119.1 (3)	C(5)—C(6)—C(1)	117.5 (3)
F(6)—C(6)—C(1)	120.8 (2)	F(6)—C(6)—C(5)	121.7 (2)
O(1)—N(1)—C(1)	121.5 (2)		

A prime denotes an atom generated by 1 - x, 1 - y, 1 - z.

Related literature. Absence of monomer indicates that the title molecule is analogous to 4-bromo-2,3,5,6-tetrafluoronitrosobenzene (Castellano, Green

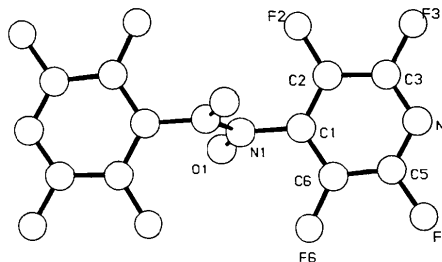


Fig. 1. The title molecule including labelling scheme drawn using *PLUTO* (Motherwell & Clegg, 1978).

& Kauffman, 1966) and not pentafluoronitrosobenzene.

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References

- BANKS, R. E., DU BOISSON, R. A., MARRACCINI, A., SEKHRI, L. & TIPPING, A. E. (1987). *Fluorine Chem.* **37**, 295–297.
- CASTELLANO, J. A., GREEN, J. & KAUFFMAN, J. M. (1966). *J. Org. Chem.* **31**, 821–824.
- CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual*. SERC Daresbury Laboratory, Warrington, England.
- 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*. Univ. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- PROUT, C. K., CODA, A., FORDER, R. A. & KEMENAR, B. (1974). *Cryst. Struct. Commun.* **3**, 39–40.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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N-(4-Fluorophenyl)-C,C-diphenylnitron*

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Abstract. C₁₉H₁₄FNO, *M_r* = 291.33, orthorhombic, *Pbca*, *a* = 17.851 (3), *b* = 8.783 (1), *c* = 18.686 (3) Å, *V* = 2929.7 Å³, *Z* = 8, *D_x* = 1.32 Mg m⁻³, λ(Mo *Kα*)

= 0.71069 Å, μ = 0.053 mm⁻¹, *F*(000) = 1216, *T* = 293 K, *R* = 0.049 for 1511 unique reflexions [*F* ≥ 3σ(*F*)]. The nitron and bonded C atoms are nearly planar [O=N=C—Ph = 3.1 (1)°]. However all the aromatic rings are twisted [C—N—C—C = 67.4 (3), N—C—C—C = 33.0 (3), 57.8 (3)°]. The molecular

* IUPAC name: *N*-(diphenylmethylene-*p*-fluoroaniline *N*-oxide.

Table 1. *Fractional atomic coordinates and vibrational parameters (\AA^2) for non-H atoms*

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C(1)	0.2935 (2)	0.2652 (3)	0.2173 (2)	0.0479 (7)
N(2)	0.2706 (1)	0.2574 (3)	0.2846 (1)	0.0501 (6)
O(3)	0.2019 (1)	0.2377 (3)	0.3046 (1)	0.0838 (8)
C(4)	0.2395 (1)	0.2591 (3)	0.1580 (2)	0.0478 (7)
C(5)	0.2622 (2)	0.1964 (4)	0.0935 (2)	0.0599 (9)
C(6)	0.2141 (2)	0.1905 (5)	0.0357 (2)	0.078 (1)
C(7)	0.1437 (2)	0.2508 (5)	0.0408 (3)	0.082 (1)
C(8)	0.1208 (2)	0.3149 (5)	0.1033 (3)	0.081 (1)
C(9)	0.1671 (2)	0.3189 (4)	0.1624 (2)	0.0633 (9)
C(10)	0.3740 (1)	0.2845 (3)	0.2013 (1)	0.0418 (6)
C(11)	0.3963 (2)	0.4075 (4)	0.1606 (2)	0.0497 (8)
C(12)	0.4705 (2)	0.4302 (4)	0.1440 (2)	0.0565 (8)
C(13)	0.5237 (2)	0.3300 (4)	0.1682 (2)	0.0598 (9)
C(14)	0.5029 (2)	0.2078 (4)	0.2082 (2)	0.0578 (8)
C(15)	0.4285 (2)	0.1830 (4)	0.2247 (2)	0.0509 (7)
C(16)	0.3218 (1)	0.2739 (3)	0.3446 (2)	0.0458 (7)
C(17)	0.3555 (2)	0.4120 (4)	0.3569 (2)	0.0538 (8)
C(18)	0.4024 (2)	0.4272 (5)	0.4152 (2)	0.0628 (9)
C(19)	0.4133 (2)	0.3057 (5)	0.4585 (2)	0.0604 (9)
C(20)	0.3798 (2)	0.1678 (5)	0.4475 (2)	0.0596 (9)
C(21)	0.3327 (2)	0.1521 (4)	0.3893 (2)	0.0536 (8)
F(22)	0.4597 (1)	0.3217 (3)	0.5154 (1)	0.0929 (7)

Table 2. *Bond lengths (\AA) and angles ($^\circ$) for non-H atoms*

C(1)—N(2)	1.324 (4)	C(1)—C(4)	1.469 (4)
C(1)—C(10)	1.477 (4)	N(2)—O(3)	1.293 (3)
N(2)—C(16)	1.454 (3)	C(4)—C(5)	1.386 (4)
C(4)—C(9)	1.398 (4)	C(5)—C(6)	1.381 (5)
C(6)—C(7)	1.367 (6)	C(7)—C(8)	1.360 (6)
C(8)—C(9)	1.379 (5)	C(10)—C(11)	1.380 (4)
C(10)—C(15)	1.390 (4)	C(11)—C(12)	1.376 (4)
C(12)—C(13)	1.371 (4)	C(13)—C(14)	1.360 (5)
C(14)—C(15)	1.382 (4)	C(16)—C(17)	1.373 (4)
C(16)—C(21)	1.372 (4)	C(17)—C(18)	1.381 (5)
C(18)—C(19)	1.354 (5)	C(19)—C(20)	1.367 (5)
C(19)—F(22)	1.355 (3)	C(20)—C(21)	1.379 (4)
C(4)—C(1)—N(2)	120.8 (2)	C(10)—C(1)—N(2)	119.9 (2)
C(10)—C(1)—C(4)	119.3 (2)	O(3)—N(2)—C(1)	125.0 (2)
C(16)—N(2)—C(1)	122.2 (2)	C(16)—N(2)—O(3)	112.7 (2)
C(5)—C(4)—C(1)	118.6 (3)	C(9)—C(4)—C(1)	123.3 (3)
C(9)—C(4)—C(5)	118.1 (3)	C(6)—C(5)—C(4)	120.9 (4)
C(7)—C(6)—C(5)	120.2 (4)	C(8)—C(7)—C(6)	119.7 (4)
C(9)—C(8)—C(7)	121.2 (4)	C(8)—C(9)—C(4)	119.8 (4)
C(11)—C(10)—C(1)	118.8 (3)	C(15)—C(10)—C(1)	122.9 (3)
C(15)—C(10)—C(11)	118.3 (3)	C(12)—C(11)—C(10)	121.0 (3)
C(13)—C(12)—C(11)	120.0 (3)	C(14)—C(13)—C(12)	119.9 (3)
C(15)—C(14)—C(13)	120.7 (3)	C(14)—C(15)—C(10)	120.1 (3)
C(17)—C(16)—N(2)	119.5 (3)	C(21)—C(16)—N(2)	118.8 (3)
C(21)—C(16)—C(17)	121.6 (3)	C(18)—C(17)—C(16)	118.9 (3)
C(19)—C(18)—C(17)	118.8 (3)	C(20)—C(19)—C(18)	123.0 (3)
F(22)—C(19)—C(18)	118.4 (3)	F(22)—C(19)—C(20)	118.6 (3)
C(21)—C(20)—C(19)	118.3 (3)	C(20)—C(21)—C(16)	119.3 (3)

conformation and dimensions agree with those of the unfluorinated compound [Brown & Trefonas (1973). *Acta Cryst.* B29, 237–241; Falshaw, Hashi & Taylor (1985). *J. Chem. Soc. Perkin Trans.* 1, pp. 1837–1843] with which it is isomorphous.

Experimental. The sample was prepared by slow addition of a solution of diphenyldiazomethane in petrol (14.4 mmol) to a stirred solution of 4-fluoro-nitrosobenzene in trichloromethane (14.4 mmol,

278 K) until the green colour discharged. The solvent was removed under reduced pressure and the solid residue triturated with diethyl ether, separated by filtration and washed with diethyl ether prior to recrystallization from aqueous ethanol.

Crystal dimensions 0.15 × 0.30 × 0.40 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centered reflexions ($9.3 \leq \theta \leq 19.8^\circ$), ω - 2θ scan mode, ω -scan width $0.6 + 0.35 \tan \theta$ ($^\circ$) and scan speed ranging from 0.2 to 5° min^{-1} according to the intensity gathered in a pre-scan, $0 \leq h \leq 21$, $0 \leq k \leq 10$, $0 \leq l \leq 22$, $0 \leq \theta \leq 25^\circ$, 3007 reflexions measured, 1951 unique ($R_{\text{int}} = 0.010$), 1511 observed [$F \geq 3\sigma(F)$], intensity standards (238; $2\bar{3}8$; 400) measured every 2.5 h, no systematic drift, Lp corrections applied but absorption ignored *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem, H atoms from ΔF synthesis, full-matrix least squares based on F using *SHELX76* (Sheldrick, 1976), final $R = 0.049$ ($wR = 0.054$, $w = 1.0/[\sigma^2(F) + 0.0006F^2]$), anisotropic thermal parameters for heavier atoms, isotropic for hydrogen. Maximum fluctuation in final ΔF map in range -0.3 to 0.56 e \AA^{-3} , maximum Δ/σ 0.02. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), computation carried out on the Amdahl 5890 system of the University of Manchester Regional Computing Centre. Literature survey performed via the Cambridge Structural Database using the Crystal Structure Search and Retrieval interactive system (CSSR, 1984). Fractional atomic coordinates and vibrational parameters for non-H atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The molecule, including atomic labelling, is displayed in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, and H-atom coordinates, bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53275 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

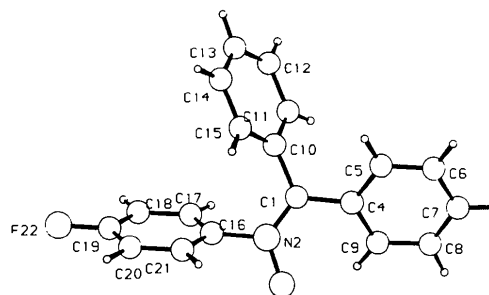


Fig. 1. The title molecule drawn using *PLUTO* (Motherwell & Clegg, 1978).

Related literature. This structure determination was undertaken to establish the course of a reaction, which may also be used to prepare a series of analogous compounds (DuBoisson, 1986).

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References

BROWN, J. N. & TREFONAS, L. M. (1973). *Acta Cryst.* **B29**, 237–241.

- CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual*. SERC Daresbury Laboratory, Warrington, England.
- DUBOISSON, R. A. (1986). PhD Thesis, Univ., of Manchester, England.
- FALSHAW, C. P., HASHI, N. A. & TAYLOR, G. A. (1985). *J. Chem. Soc. Perkin Trans.* **1**, pp. 1837–1843.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, 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. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of N-Methyl-2-(4-methoxyphenylthio)benzylammonium Chloride

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(Received 8 March 1990; accepted 27 June 1990)

Abstract. $C_{15}H_{18}NOS^+Cl^-$, $M_r = 295.8$, orthorhombic, $P2_12_12_1$, $a = 5.167$ (1), $b = 10.748$ (4), $c = 27.278$ (8) Å, $V = 1515.0$ (9) Å³, $Z = 4$, $D_x = 1.297$ Mg m⁻³, Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å), $\mu = 3.456$ mm⁻¹, $F(000) = 624$, $T = 295$ K, $R = 0.065$, $wR = 0.085$ for 1440 observed reflections. The dihedral angle between the phenyl rings is 100.9 (2)°. Strong intermolecular contacts N9—H92...Cl [N9...Cl 3.141 (5) Å, N9—H92...Cl 147 (6)°] and N9—H91...Clⁱ [N9...Clⁱ 3.079 (5) Å, N9—H91...Clⁱ 154 (6)° where i is the equivalent position $x - 1, y, z$] join ammonium cations and chloride anions into chains parallel to the a axis. The methyl group attached to N9 is not in the generally preferred *anti* conformation but is approximately *gauche* [C7—C8—N9—C10 -82.4 (6)°]. The C8 atom lies in the mean plane of the first phenyl ring [deviation 0.004 (5) Å] and the atoms O15, C16 lie near the mean plane of the second phenyl ring [deviations: 0.046 (5) and 0.019 (7) Å]. The atom S1 deviates by -0.106 (2) Å from the first and by 0.172 (2) Å from the second plane.

Experimental. Colorless, needle-shaped and extremely fragile crystals were grown from an ethanol–heptane solution (9:1) by slow evaporation at room temperature. Crystal dimensions 0.40×0.17

$\times 0.14$ mm, Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell constants by least squares using 21 reflections with $9 \leq 2\theta \leq 26^\circ$. Cell dimensions and space group $P2_12_12_1$ independently determined also by Weissenberg and oscillation techniques. Only 1546 unique reflections collected using $\theta/2\theta$ scans, 1440 with $F \geq 3.92\sigma(F)$, $0 \leq 2\theta \leq 132^\circ$, $0 \leq h \leq 6$; $0 \leq k \leq 12$; $0 \leq l \leq 32$. No absorption corrections applied ($\mu r = 0.82$). No significant variation in three standard reflections (200, 016, 008 measured after every 40 reflections) were observed.

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) and refined with *SHELX76* (Sheldrick, 1976). An isotropic refinement based on $|F|$ with all non-H atoms gave $R = 0.15$. H atoms were refined to reasonable positions without any constraints ($R = 0.135$). However, H91 and H92 required fixed temperature factors $U = 0.1$ Å² (Table 1). Anisotropic refinement of non-H atoms and isotropic refinement of H atoms (243 variables) converged at $R = 0.065$, $wR = 0.064$ and $S = 2.4$ using 1440 F 's with $F \geq 3.92\sigma(F)$ (and at $R = 0.069$, $wR = 0.064$ for all unique reflections), $w^{-1} = \sigma^2(F) + 0.0009F^2$, $(\Delta/\sigma)_{\max} = 0.18$. The maximum and minimum peaks in the final difference map were 0.35, -0.85 e Å⁻³. Atomic scattering factors were taken