

A similar trend has been found in the 2,2'-bipyridyl that is coordinated to Mn in the complex trinitrato-2,2'-bipyridylmanganese(III) (Einstein, Johnson & Sutton, 1972).

The packing of the molecules in the unit cell is shown in Fig. 2. There are no intermolecular distances less than 3.5 Å.

References

CORFIELD, P. W. R., DOEDENS, R. J. & IBERS, J. A. (1967). *Inorg. Chem.* **6**, 197–204.

EINSTEIN, F. W. B., JOHNSON, D. W. & SUTTON, D. (1972). *Canad. J. Chem.* **50**, 3332–3338.

HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040–1044.

MERRITT, L. L. JR & SCHROEDER, E. D. (1956). *Acta Cryst.* **9**, 801–804.

OOL, S., CARTER, D. E. & FERNANDO, Q. (1968). *Progress in Coordination Chemistry*, edited by M. CAIS, p. 293. Amsterdam: Elsevier.

OOL, S. & FERNANDO, Q. (1967). *Inorg. Chem.* **6**, 1558–1562.

SHETTY, P. S. & FERNANDO, Q. (1970). *J. Amer. Chem. Soc.* **92**, 3964–3969.

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1-{1-[3-(2- α,α,α -Trifluoromethyl-10-phenothiazinyl)propyl]-4-piperidinyl}-2-benzimidazolinone

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Abstract. C₂₈H₂₇N₄OF₃S, FW 524.6, monoclinic, C2/c, $a = 24.497$ (6), $b = 5.802$ (1), $c = 36.573$ (9) Å, $\beta = 102.73$ (4)°, $Z = 8$, $t = 25^\circ\text{C}$. The molecules form centrosymmetric dimers linked through the amide groups.

Introduction. The title compound is a neuroleptic phenothiazine derivative. The space group was determined from photographs. The final cell dimensions and intensities were measured on a Hilger & Watts computer-controlled four-circle diffractometer. The instrumental settings are given in Table 1.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). Full-matrix least-squares

refinement was performed with the *SDP 75* program system (Okaya & Frenz, 1975) and gave a final R of 0.10 for all observed reflexions. The scattering factors were those of *International Tables for X-ray Crystallography* (1974). The final coordinates are listed in Table 2. Fig. 1 gives bond lengths and angles and the atomic numbering.*

Discussion. The phenothiazine group is folded along the S—N(1) line, the angle between the planes of the benzene rings being 149°. The conformation of the molecule is defined by the torsion angles given in Table 3.

Folding of the side chain involves van der Waals interactions between the benzimidazolinone group and the F substituents: C(27)—F(1) 3.56, C(27)—F(2) 3.77 Å.

The angle between the least-squares mean plane of

Table 1. *Instrumental settings for the data collection*

Source: Mo $K\alpha$; $\lambda = 0.7107$ Å; ω - 2θ step scan
 $\theta_{\max} = 58^\circ$
 Confidence level: 3.0
 Total number of independent reflexions: 3527
 Total observed: 2221

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32762 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

the piperidine ring and the benzimidazolinone group is 88°. The presence of an aromatic group nearly perpendicular to the mean plane of the piperidine ring seems to be a requirement for strong neuroleptic activity. In spiperone (Koch, 1973), benperidol

(Declercq, Germain & Koch, 1973) and clopimozide (Van Opendbosch, Evrard, Durant & Koch, 1977), this conformation is rigidly fixed.

The molecules form centrosymmetric dimers similar to those found in spiperone (Koch, 1973)

Table 2. *Positional parameters* ($\times 10^4$, for $y \times 10^3$) *and their estimated standard deviations*

	x	y	z		x	y	z
S	2856 (2)	776 (1)	3006 (1)	C(10)	1252 (5)	571 (3)	2795 (3)
F(1)	806 (3)	114 (3)	3334 (3)	C(11)	1280 (5)	378 (3)	3021 (3)
F(2)	374 (4)	385 (2)	3104 (4)	C(12)	1781 (4)	292 (2)	3231 (3)
F(3)	568 (4)	120 (3)	2773 (2)	C(13)	2814 (4)	205 (2)	3799 (3)
O	754 (3)	977 (3)	4950 (2)	C(14)	3124 (4)	331 (2)	4151 (3)
N(1)	2803 (3)	354 (2)	3461 (2)	C(15)	2897 (4)	557 (2)	4211 (3)
N(2)	2302 (3)	544 (2)	4238 (2)	C(16)	2254 (4)	482 (2)	4616 (3)
N(3)	740 (3)	662 (2)	4549 (2)	C(17)	1642 (4)	457 (2)	4646 (3)
N(4)	-35 (3)	752 (2)	4721 (2)	C(18)	4721 (4)	677 (2)	4524 (3)
C(1)	3377 (5)	558 (2)	3110 (3)	C(19)	1388 (4)	743 (2)	4130 (3)
C(2)	3307 (4)	379 (2)	3332 (3)	C(20)	2010 (4)	759 (2)	4127 (3)
C(3)	3730 (4)	213 (2)	3429 (3)	C(21)	515 (4)	814 (2)	4761 (3)
C(4)	4238 (5)	243 (3)	3302 (3)	C(22)	329 (4)	507 (2)	4366 (3)
C(5)	4269 (5)	429 (3)	3073 (3)	C(23)	-153 (4)	567 (2)	4486 (3)
C(6)	3858 (5)	581 (3)	2974 (3)	C(24)	-637 (4)	444 (2)	4366 (3)
C(7)	2283 (4)	414 (2)	3233 (3)	C(25)	-628 (5)	260 (2)	4124 (3)
C(8)	2260 (5)	604 (2)	3004 (3)	C(26)	-151 (5)	202 (2)	4002 (3)
C(9)	1754 (6)	673 (3)	2780 (3)	C(27)	346 (4)	326 (2)	4128 (3)
				C(28)	772 (5)	252 (3)	3046 (3)

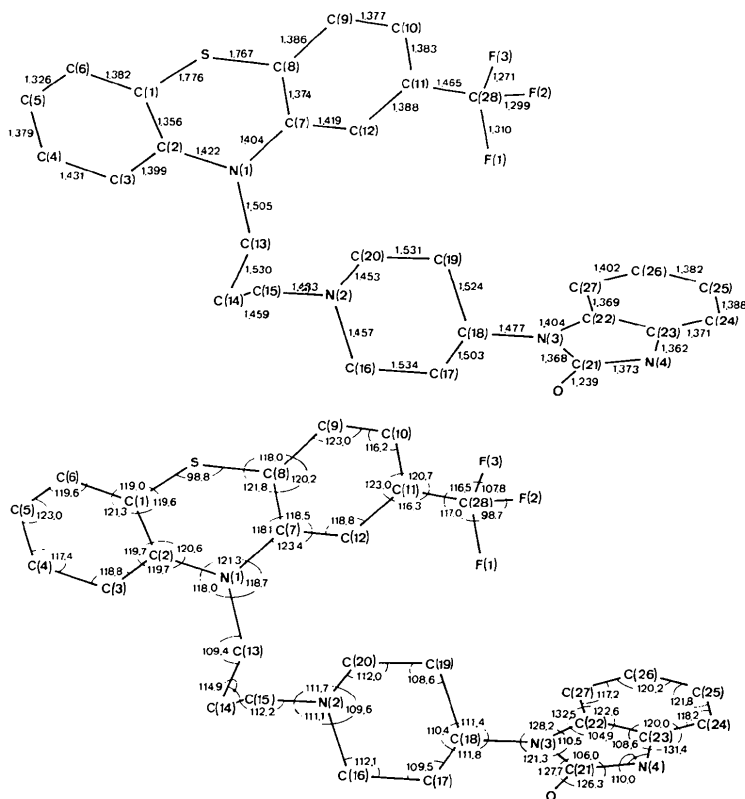


Fig. 1. Bond lengths (Å) and angles (°). The e.s.d.'s for distances and angles involving nonhydrogen atoms are in the ranges 0.005–0.008 Å and 0.2–0.6°.

Table 3. *Torsion angles* (°)

C(16)–N(2)–C(15)–C(14)	83
C(20)–N(2)–C(15)–C(14)	–154
N(2)–C(15)–C(14)–C(13)	58
C(15)–C(14)–C(13)–N(1)	57
C(14)–C(13)–N(1)–C(2)	74
C(14)–C(13)–N(1)–C(7)	–122

and clopimozide (Van Opdenbosch, Evrard, Durant & Koch, 1977). The dimerization is due to the amide groups which are hydrogen-bonded: O–N(4)[–x, –y, –z] 2.82 Å.

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References

- DECLERCQ, J. P., GERMAIN, G. & KOCH, M. H. J. (1973). *Acta Cryst.* **B29**, 2311–2313.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- International Tables for X-ray Crystallography* (1974). Vol. III. Birmingham: Kynoch Press.
- KOCH, M. H. J. (1973). *Acta Cryst.* **B29**, 379–382.
- OKAYA, Y. & FRENZ, B. (1975). Molecular Structure Corporation, PO Box DF, College Station, Texas.
- VAN OPDENBOSCH, N., EVRARD, G., DURANT, F. & KOCH, M. H. J. (1977). *Acta Cryst.* **B33**, 596–599.

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N-Methylphenethylammonium Trichloronickelate(II)

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Abstract. [C₉H₁₄N]NiCl₃, *M_r* = 301.29, orthorhombic, *P*2₁2₁2₁, *Z* = 4. At –35°C, *a* = 7.414 (1), *b* = 26.510 (5), *c* = 6.125 (1) Å, *V* = 1203.7 Å³, *D_x* = 1.662 g cm^{–3}. Mo *K*α radiation, λ = 0.71069 Å, μ = 22.3 cm^{–1}. Full-matrix least-squares refinement using 1116 reflections [*I* > 2σ(*I*)] collected with ω scans on a Syntex diffractometer converged at a conventional *R* of 0.039. The structure consists of (NiCl₃)_n infinite chains in which each of the chloride ions serves as a bridging ligand to effect octahedral coordination of the nickel ions. These chains interact weakly with the cations through N–H⋯Cl hydrogen bonds.

Introduction. The structures of *N*-methylphenethylammonium trichlorocuprate(II) (Harlow, Wells, Watt & Simonsen, 1974*a*) and bis(*N*-methylphenethylammonium) tetrachlorocuprate(II) (Harlow, Wells, Watt & Simonsen, 1974*b*) contain Cu²⁺ ions with unusual coordination geometries. As an extension of these studies, other *N*-methylphenethylammonium [hereafter abbreviated as (nmpH)] chlorometallates are presently being investigated.

Light-orange crystals of the title compound were grown by slow evaporation of an acetone solution under a stream of dry N₂ gas. The crystal selected for this study was a cleaved section of a needle with approximate dimensions of 0.05, 0.1 and 0.5 mm. The

crystal was mounted parallel to the needle axis (crystallographic *c* axis) and placed on a Syntex diffractometer equipped with a low-temperature apparatus which kept the crystal cooled to –35°C. The unit-cell parameters were refined using the Bragg angles of 30 low-angle (18 < 2θ < 27°) reflections.

Intensity data for 1635 unique reflections (4 < 2θ < 55°) were collected by the ω-scan technique. Scans of 1.0° were employed with scan rates which ranged from 1.5 to 5.0° min^{–1} depending on the number of counts accumulated in a rapid preliminary scan. Background measurements were taken at both ends of the scan; the time for each measurement was one-half the scan time. The intensities of four standard reflections were monitored after every 96 reflections; only statistical variations were noted. The intensities were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods and Fourier syntheses. The full-matrix least-squares refinement of 127 variables using only those 1116 reflections for which *I* > 2σ(*I*) converged at a conventional *R* of 0.039. Anomalous dispersion corrections for the scattering factors of Ni and Cl were included in the final stages of the refinement; the enantiomorphic structure converged at *R* = 0.042. The non-hydrogen atoms were refined with anisotropic thermal