Fig. 1. View of a single molecule of phenylacetic acid. Thermal ellipsoids at the 50% probility level, with H atoms of arbitrary size.
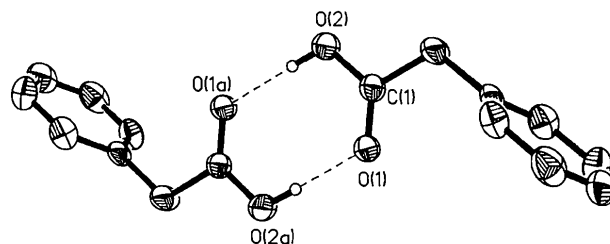

Fig. 2. Hydrogen bonding between pairs of phenylacetic acid molecules. Atoms labelled 'a' are related to the 'parent' atoms by inversion through the center at the origin.

numbering scheme, is shown in Fig. 1, and the intermolecular hydrogen-bonding pattern is depicted in Fig. 2.

Related literature. Decarboxylations of carboxylic acids are not uncommon (Fitzpatrick & Hopgood, 1974; de Meester & Hodgson, 1976, 1978). The structure of phenylacetic acid was initially solved by Patterson (1927) as one of his pioneering crystallographic studies, and the structure of a potassium salt has been reported (Manojlović & Speakman, 1968). Carboxylic acids generally form centrosymmetric hydrogen-bonded pairs (Haas & Brenner, 1966) and, as is shown in Fig. 2, phenylacetic acid is

normal in this regard. In the present case, the $O(2) \cdots O(1a)$ and $H \cdots O(1a)$ distances are 2·679 (3) and 1·61 (3) Å, while the associated $O(2)—H \cdots O(1a)$ angle is 174 (2)°.

References

FITZPATRICK, J. H. JR & HOPGOOD, D. (1974). *Inorg. Chem.* **13**, 568–574.
HAAS, D. & BRENNER, S. A. (1966). *Acta Cryst.* **20**, 709–713.
MANOJLOVIĆ, L. & SPEAKMAN, J. C. (1968). *Acta Cryst.* B24, 323–325.
MEESTER, P. DE & HODGSON, D. J. (1976). *J. Am. Chem. Soc.* **98**, 7086.
MEESTER, P. DE & HODGSON, D. J. (1978). *Inorg. Chem.* **17**, 440–444.
PATTERSON, A. L. (1927). *Strukturbericht* I, pp. 702–704.
SHELDRICK, G. M. (1987). *SHELXTL-Plus.* Version 2. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Structure of *N*-Methylurotropinium Iodide

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Abstract. 1-Methyl-1,3,5,7-tetraazatricyclo[3.3.1.1^{3,7}]-decan-1-ium iodide, [(CH$_2$)$_6$N$_4$CH$_3$]I, $M_r = 282\cdot13$, monoclinic, $P2_1$, $a = 6\cdot630$ (2), $b = 7\cdot254$ (2), $c = 10\cdot878$ (2) Å, $\beta = 105\cdot04$ (7)°, $V = 505\cdot3$ (2) Å^3, $Z = 2$, $D_x = 1\cdot854$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 3\cdot093$ mm^{-1}, $F(000) = 276$, $T = 293$ (2) K, final $R = 0\cdot048$ for 2734 observed reflections. The structure consists of N_1-methylurotropinium cations and I$^-$ anions. The C—N distances and bond angles (C—N—C and N—C—N) vary from 1·415 (8) to 1·572 (8) Å and from 103·0 (5) to 115·9 (5)°, respectively. I$^-$ is surrounded by seven H atoms at distances in the range 2·90 (5)–3·29 (5) Å. N atoms are 4·391 (7) Å away from I$^-$.

Experimental. To a solution of 1·14 g (0·001 mol) of methenamine in 10 ml of methanol was added 1·25 ml (0·02 mol) methyl iodide. The reaction mixture was kept at room temperature for 5 h in the dark and was then evaporated in vacuum at 313 K. The solid product crystallized from methanol (m.p. 477–478 K, yield 99%). The purity of the crystals was monitored by thin-layer chromatography.

A crystal *ca* 0·30 × 0·57 × 0·65 mm was mounted on a CAD-4 diffractometer equipped with a graphite monochromator. Cell constants were refined by a least-squares fit for 50 centred reflections in the range 12 < θ < 18°. Systematic absences 0*k*0: *k* = 2*n* + 1. ω–2θ scan in the range 1·0 < θ < 30·0° with *h*: −9 to 9, *k*: −10 to 10, *l*: −15 to 15. Three standard reflections (512, 432, 24$\bar{5}$) were measured every 2 h. Intensity change was 0·44%. Intensities of 5883 reflections were measured of which 2927 were unique and non zero (R_{int} = 0·058) after merging. Data were corrected for Lorentz and polarization effects but not for absorption. Structure was solved by *SHELXS*86 (Sheldrick, 1986), yielding all non-H atoms. Full-matrix least-squares refinement with *SHELX*76 (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 109 parameters with $w = [\sigma^2(F_o) + 5 \times 10^{-3}F_o^2]^{-1}$ using 2734 reflections with $I > 2\cdot5\sigma(I)$. Final R = 0·048, wR = 0·049. $\Delta\rho_{max}$ = 1·91 e Å^{-3}, $(\Delta/\sigma)_{max}$ = 0·20. Positions of H atoms were generated from assumed geometries. Their positions were taken into account without refinement in structure-factor calculations with common isotropic temperature factor U = 0·053 (4) Å^2. Scattering factors were taken from *SHELX*76 and from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed on a PC/AT computer. The structure is defined in Tables 1 and 2,* and Fig. 1.

Related literature. A synthesized derivative of methenamine has potential antimicrobial activity (Katul & Frank, 1970). Structure of $(CH_2)_6N_4$: Becka & Cruickshank (1963); $[(CH_2)_6N_4CH_3]_2I_8$: Hon, Mak & Trotter (1979); $[(CH_2)_6N_4CH_3]I_3$: Hon, Mak & Trotter (1982); $[(CH_2)_6N_4H]Br$: Mak, Li & Yip (1983); $[(CH_2)_6N_4H][B_5O_6(OH)_4.0\cdot5H_2O]$: Bacanov, Petrova, Struchkov, Akimov, Molodkin & Skvorcov (1986).

Table 1. *Final fractional coordinates for non-H atoms and equivalent isotropic temperature factors* (× 10⁴) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + U_{13}aca^*c^*\cos\beta].$$

	x	*y*	*z*	U_{eq}(Å²)
I	9932 (1)	0	1659 (1)	458 (1)
N1	3736 (4)	− 50 (9)	−1938 (2)	291 (4)
C2	4777 (8)	1586 (7)	−2328 (6)	306 (9)
N3	4636 (6)	1739 (7)	−3656 (6)	283 (8)
C4	2384 (8)	1605 (7)	−4488 (5)	316 (8)
N5	1395 (4)	66 (8)	−4063 (3)	306 (4)
C6	2526 (9)	− 1683 (7)	−4291 (4)	288 (7)
N7	4669 (9)	− 1596 (8)	− 3640 (6)	356 (11)
C8	4795 (10)	− 1847 (8)	−2283 (5)	325 (8)
C9	1462 (4)	67 (10)	−2725 (3)	297 (4)
C10	5654 (5)	75 (10)	−3962 (3)	361 (7)
C11	3795 (7)	−4 (14)	−570 (3)	506 (9)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

|			
N1—C2	1·489 (8)	C4—N5	1·431 (7)
N1—C8	1·572 (8)	N5—C6	1·526 (7)
N1—C9	1·531 (3)	N5—C9	1·445 (5)
N1—C11	1·479 (4)	C6—N7	1·415 (8)
C2—N3	1·428 (9)	N7—C8	1·468 (9)
N3—C4	1·535 (6)	N7—C10	1·462 (9)
N3—C10	1·463 (8)		
C2—N1—C8	108·9 (4)	C4—N5—C6	108·1 (4)
C2—N1—C9	105·0 (4)	C4—N5—C9	115·4 (4)
C2—N1—C11	112·1 (4)	C6—N5—C9	106·0 (4)
C8—N1—C9	110·4 (4)	N5—C6—N7	110·7 (4)
C8—N1—C11	111·2 (4)	C6—N7—C8	106·6 (5)
C9—N1—C11	109·1 (4)	C6—N7—C10	111·7 (5)
N1—C2—N3	115·9 (5)	C8—N7—C10	115·6 (5)
C2—N3—C4	113·0 (5)	N1—C8—N7	103·0 (5)
C2—N3—C10	104·7 (5)	N1—C9—N5	109·4 (4)
C4—N3—C10	104·4 (4)	N3—C10—N7	111·6 (5)
N3—C4—N5	108·4 (4)		
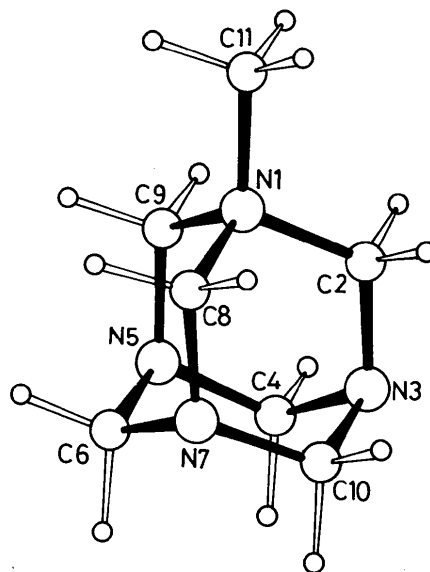
Fig. 1. A perspective view of the $[(CH_2)_6N_4CH_3]^+$ ion. The H atoms are shown but not labelled.

References

BACANOV, A. S., PETROVA, O. V., STRUCHKOV, YU. T., AKIMOV, V. M., MOLODKIN, A. K. & SKVORCOV, V. G. (1986). *Zh. Neorg. Khim.* **31**, 1120–1126.

BECKA, L. N. & CRUICKSHANK, D. W. J. (1963). *Proc. R. Soc. (London) Ser. A*, **273**, 435–454.

HON, P. K., MAK, T. C. W. & TROTTER, J. (1979). *Inorg. Chem.* **18**, 2916–2918.

HON, P. K., MAK, T. C. W. & TROTTER, J. (1982). *Z. Kristallogr.* **158**, 213–220.

KATUL, M. J. & FRANK, I. N. (1970). *J. Urol.* **101**, 320–323.

MAK, T. C. W., LI, W.-K. & YIP, W.-H. (1983). *Acta Cryst.* C**39**, 134–136.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

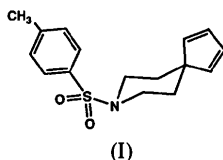# N-Tosyl-8-azaspiro[4.5]deca-1,3-diene

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Abstract. $C_{16}H_{19}NO_2S$, $M_r = 289.4$, orthorhombic, $P2_12_12_1$, $a = 6.444$ (3), $b = 7.3726$ (11), $c = 32.038$ (2) Å, $V = 1522.2$ (8) Å^3, $Z = 4$, $D_x = 1.266$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 18.5$ cm^{-1}, $F(000) = 616$, $T = 296$ K, $R = 0.060$ for 1156 observations with $I > 3\sigma(I)$ (of 1851 unique data). The six atoms of the aromatic ring exhibit a maximum deviation of 0.009 (7) Å from coplanarity, with the sulfur atom and tolyl carbon atom lying 0.108 (2) and 0.104 (9) Å respectively to the same side of this plane. The five atoms of the cyclopentadienyl ring exhibit a maximum deviation of 0.003 (6) Å from coplanarity. The dihedral angle between the aromatic ring and the cyclopentadienyl ring is 105.2 (2)°. The N—S bond distance is 1.631 (5) Å and the S=O bond distances are 1.446 (4) and 1.417 (5) Å. The piperidine ring has a chair conformation.

Experimental. N-(p-Toluenesulfonyl)-8-azaspiro[4.5]-deca-1,3-diene (I) was prepared by treating N-(p-toluenesulfonyl)bis[2-(p-toluenesulfonate)ethyl]amine, $TsN(CH_2CH_2OTs)_2$ (Searle & Geue, 1984) with sodium cyclopentadienide in hexamethylene phosphoramide (Halterman, Vollhardt, Welker, Bläser & Böse, 1987).

(I)

Crystals, m.p. 379–381 K, formed by slow cooling of a 2,2,4-trimethylpentane solution; a clear, colorless crystal with dimensions 0.08 × 0.25 × 0.42 mm was used for the data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $25 < \theta < 30°$. The ω–2θ scans were designed for $I = 25\sigma(I)$, subject to max. scan time = 90 s, scan rates varied from 0.57–3.30° min^{-1}. One octant of data having $2 < \theta < 75°$, $0 \le h \le 8$, $0 \le k \le 9$, $0 \le l \le 40$ was measured and corrected for background, Lorentz, and polarization. Absorption corrections were based on ψ scans; the min. relative transmission coefficient was 0.844. Three standard reflections (200, 020, 006) showed only a random fluctuation in intensity so no correction for decay was applied. Systematic absences $h00$ with h odd, $0k0$ with k odd, and $00l$ with l odd indicated space group $P2_12_12_1$. The structure was solved by direct methods. An 11 atom fragment recognized in an E map from *RANTAN* (Yao, 1981) was expanded using *DIRDIF* (Beurskens, 1984) to the full structure and refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Non-H-atom coordinates were refined with anisotropic thermal parameters; many of the H-atom positions were visible in ΔF maps; however, H atoms were included as fixed contributions, 0.95 Å from the bonded C atom with isotropic $B = 1.3$ B_{eq} for the C atom. Final $R = 0.060$

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