

without having to know the effects of the Me— and MeNH— substituents. (The linear combination is likely to hold well here as there is no mechanism for through-conjugation.) Let us suppose that substituent parameters are known for two of the three parameters. We have published (DMR) values for 22 substituents, among them NO₂ and Me, but no values are available for NHMe. They have been estimated as follows on the basis of the linear hypothesis. From DMR we have:

$$\begin{aligned}\Delta\alpha(\text{NO}_2) &= 2.9^\circ & \Delta\alpha(\text{Me}) &= -1.9^\circ \\ \Delta\beta(\text{NO}_2) &= -1.9 & \Delta\beta(\text{Me}) &= 1.0 \\ \Delta\gamma(\text{NO}_2) &= 0.3 & \Delta\gamma(\text{Me}) &= 0.4 \\ \Delta\delta(\text{NO}_2) &= 0.4 & \Delta\delta(\text{Me}) &= -0.7.\end{aligned}$$

From (30)–(35) we have

$$\begin{aligned}\Delta\alpha(\text{NHMe}) + 0.3 - 0.7 + 120 &= 117.3^\circ \\ \Delta\beta(\text{NHMe}) - 1.9 + 0.4 + 120 &= 119.6 \\ \Delta\gamma(\text{NHMe}) + 2.9 + 1.0 + 120 &= 124.1 \\ \Delta\delta(\text{NHMe}) - 1.9 - 1.9 + 120 &= 114.3 \\ \Delta\gamma(\text{NHMe}) + 0.3 + 1.0 + 120 &= 122.9 \\ \Delta\beta(\text{NHMe}) + 0.4 + 0.4 + 120 &= 121.7.\end{aligned}$$

Hence

$$\begin{aligned}\Delta\alpha(\text{NHMe}) &= -2.3^\circ \\ \Delta\beta(\text{NHMe}) &= 1.0 \\ \Delta\gamma(\text{NHMe}) &= 0.9 \\ \Delta\delta(\text{NHMe}) &= -1.9.\end{aligned}$$

Acta Cryst. (1982). **B38**, 2825–2829

Structural Studies of Benzene Derivatives.

XI.* The Structure of *p*-Toluidine Hydrochloride

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(Received 2 March 1982; accepted 17 May 1982)

Abstract

Crystals of the title compound, C₇H₁₀N⁺.Cl⁻, are monoclinic, space group *P*2₁/*c*, with *a* = 9.116 (1), *b* =

* Part X: Colapietro & Domenicano (1982).

0567-7408/82/112825-05\$01.00

It is gratifying to note that these values are close to those for —NH₂ and —NMe₂ (DMR):

$$\begin{aligned}\Delta\alpha(\text{NH}_2) &= -1.3^\circ & \Delta\alpha(\text{NMe}_2) &= -2.4^\circ \\ \Delta\beta(\text{NH}_2) &= 0.3 & \Delta\beta(\text{NMe}_2) &= 0.7 \\ \Delta\gamma(\text{NH}_2) &= 1.0 & \Delta\gamma(\text{NMe}_2) &= 1.4 \\ \Delta\delta(\text{NH}_2) &= -1.3 & \Delta\delta(\text{NMe}_2) &= -1.7.\end{aligned}$$

I thank Professor A. Vaciago and Dr A. Domenicano for their interest, and the British Council/CNR (Rome) for a travel grant.

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9.357 (1), *c* = 9.913 (2) Å, β = 108.56 (2)°, *Z* = 4. The structure, originally determined by Von Eller [*Bull. Soc. Fr. Minéral. Cristallogr.* (1955), **78**, 275–300], has been redetermined from 1615 counter intensities and refined to a final *R* value of 0.0330. The benzene

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ring has almost exact *mm* symmetry. The deviations from *6/mmm* symmetry involve bond distances as well as angles; the values of the C—C—C angles are very well reproduced by superimposing independent distortional effects from each substituent. The NH₃⁺ groups and Cl⁻ ions are connected in infinite layers through Cl⁻...H—N hydrogen bonds of length 3.12–3.15 Å.

Introduction

As a contribution to our programme of accurate determinations of the molecular geometries of *para*-substituted anilinium cations (Colapietro, Domenicano, Marciante & Portalone, 1981, and references therein) we report here the structure of *p*-toluidine hydrochloride. This compound was the subject of an early X-ray diffraction study (Von Eller, 1955). The analysis was based on visually estimated photographic data and ignored the contribution of the H atoms. Thus a new structural study was mandatory if the effects of the substituents on the geometry of the benzene ring were to be investigated.

The structures of a few other salts of the *p*-toluidinium cation have been determined by X-ray crystallography (Adman & Margulis, 1967; Denne, Mathieson & Mackay, 1971; Denne & Mackay, 1971); in no case, however, was the precision of the analysis high enough to allow a detailed description of the effects of the substituents on the ring geometry. A preliminary report on a study of *p*-toluidinium bifluoride by neutron crystallography has appeared (Williams & Schneemeyer, 1973). Unfortunately, neither the geometry of the cation nor the atomic parameters were reported.

Experimental

Colourless, well shaped crystals were grown from an ethanolic solution of *p*-toluidine with excess HCl. A crystal *ca* 0.37 × 0.44 × 0.56 mm was mounted on a Syntex P2₁ diffractometer. The cell parameters were determined by least-squares techniques from the measured θ values of 15 reflexions, lying in the θ range 15–17° and well distributed in reciprocal space. The values obtained are in reasonable agreement with those given by Von Eller (1955) (Table 1).

Intensities were measured at room temperature in the θ – 2θ scan mode with graphite-monochromatized Mo K α radiation. The scanning interval was from $2\theta(K\alpha_1) - 1.0^\circ$ to $2\theta(K\alpha_2) + 1.2^\circ$. The scanning speed was varied according to the intensity, from a minimum value of 0.017 to a maximum of 0.49° s⁻¹. The background was measured for a quarter of the scanning time at each end of the scan. A recentring routine was run every 500 reflexions.

Table 1. *Crystal data*

p-Toluidine hydrochloride, C₇H₁₀N⁺·Cl⁻, *M_r* = 143.62, monoclinic, space group *P*2₁/*c* (systematic absences: 0*k*0, *k* = 2*n* + 1; *h*0*l*, *l* = 2*n* + 1), *Z* = 4.

	This work ^(a)	Von Eller (1955)
<i>a</i> (Å)	9.116 (1)	9.07 ^(b)
<i>b</i> (Å)	9.357 (1)	9.33 ^(b)
<i>c</i> (Å)	9.913 (2)	9.86 ^(b)
β (°)	108.56 (2)	108.7
<i>V</i> (Å ³)	801.6 (2)	790
<i>D_x</i> (Mg m ⁻³)	1.190	1.207 ^(c)
<i>D_m</i> (Mg m ⁻³)	1.186 ^(d)	1.193 ^(e)

Notes: (a) Measured at 293 K with Mo K α radiation (λ = 0.71069 Å). E.s.d.'s are given in parentheses as units in the last digit. (b) All \pm 0.4%. (c) Calculated from the given cell volume. (d) Measured at 296 K by flotation in a mixture of chloroform and *n*-hexane. (e) Measured at 291 K.

Three standard reflexions, monitored every 100 reflexions, showed a regular decrease of intensity (6% during the entire data collection). The intensities were corrected for the decrease and for Lorentz and polarization effects, but not for absorption [μ (Mo K α) = 0.392 mm⁻¹].

3250 reflexions in the θ range 1.5–32.5° were measured; 1708 of these, having $I > 4\sigma(I)$, were considered as non-zero.* The reflexions of the layer $h = 0$ were measured both as 0*kl* and as 0*k* \bar{l} ; averaging and merging [internal $R(F_o) = 0.027$] led to a set of 1615 independent observations.

Structure determination and refinement

Preliminary atomic parameters for the non-H atoms, obtained by Patterson and Fourier techniques ($R = 0.284$), confirmed the correctness of the structure given by Von Eller (1955). Isotropic and then anisotropic least-squares refinement followed by the introduction of the H atoms as given by a difference synthesis lowered R to 0.048. Further refinement of all the atomic parameters, with the H atoms treated isotropically, converged to $R = 0.0337$. At this stage a difference synthesis showed that the methyl group also had an alternative, less populated conformation.

The final refinement was by full-matrix least squares, minimizing $\sum w(|F_o| - k|F_c|)^2$. The methyl H atoms of the most populated conformation were allowed to refine independently, with a (fixed) occupancy factor of 0.75. The H atoms of the alternative conformation were introduced as fixed contributors in idealized positions ($d_{C-H} = 0.98$ Å, $\angle C-C-H = \angle H-C-H = 109.5^\circ$), with $B = 3.0$ Å² and an occupancy factor of 0.25.

* The reflexions 11 $\bar{3}$, 11 $\bar{2}$, 111 and 12 $\bar{1}$ were too intense to be measured accurately and were excluded from the data set.

Table 2. Final coordinates ($\times 10^5$) and equivalent values of the anisotropic temperature factors for non-hydrogen atoms

E.s.d.'s from the final full-matrix least-squares cycle are given in parentheses as units in the last digit.

	x	y	z	$B_{eq} (\text{\AA}^2)^*$
Cl ⁻	59445 (5)	62299 (5)	34347 (5)	4.04
N	43301 (18)	80190 (18)	7134 (16)	3.79
C(1)	28245 (19)	73709 (17)	-304 (16)	3.41
C(2)	15039 (22)	79759 (22)	964 (21)	4.27
C(3)	905 (24)	73609 (22)	-6146 (22)	4.61
C(4)	-182 (21)	61485 (21)	-14444 (18)	4.13
C(5)	13408 (24)	55566 (24)	-15318 (20)	4.58
C(6)	27658 (23)	61670 (22)	-8389 (20)	4.30
C(7)	-15670 (29)	55043 (33)	-22325 (27)	5.58

* Defined as $(B_1 \cdot B_2 \cdot B_3)^{1/3}$, where $B_i = 8\pi^2 U_i^2$ ($i = 1, 2, 3$). The U_i^2 are the mean-square amplitudes of vibration along the principal axes of the thermal ellipsoid.

Table 3. Final coordinates ($\times 10^3$) and isotropic temperature factors for the hydrogen atoms

E.s.d.'s from the final full-matrix least-squares cycle are given in parentheses as units in the last digit.

	x	y	z	$B (\text{\AA}^2)$
H(2)	158 (3)	879 (3)	64 (3)	3.9 (5)
H(3)	-82 (3)	775 (3)	-51 (3)	3.6 (5)
H(5)	130 (3)	470 (3)	-207 (2)	3.2 (5)
H(6)	366 (3)	580 (2)	-91 (2)	2.8 (4)
H(1N)	492 (3)	811 (3)	17 (3)	3.5 (5)
H(2N)	421 (3)	889 (3)	104 (3)	3.6 (5)
H(3N)	485 (2)	747 (2)	148 (2)	2.8 (5)
H(71)*	-229 (4)	567 (3)	-176 (3)	3.1 (6)
H(72)*	-148 (4)	438 (5)	-224 (4)	4.9 (8)
H(73)*	-184 (4)	582 (4)	-313 (4)	3.6 (7)
H(71')†	-233	626	-256	3.0
H(72')†	-189	486	-160	3.0
H(73')†	-149	497	-306	3.0

* Occupancy factor 0.75.

† Occupancy factor 0.25.

Weights were given according to the function $w = 1/(a + b|F_o| + c|F_o|^2)$ (Cruickshank, 1965), with $a = 123$, $b = 1.0$ and $c = 0.81$. The merits of this weighting function in accurate structural work have been discussed by Di Rienzo, Domenicano & Riva di Sanseverino (1980). The number of observations per refined parameter was 1613/122 = 13.2.* The final R and R_w were 0.0330 and 0.0444, respectively, with all shifts $\leq 0.01\sigma$. The final atomic parameters are given in Tables 2 and 3.†

* The reflexions 162 and 262 had $|F_o|$ suspiciously different from $|F_c|$ and were excluded from the final refinements.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36957 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Calculations were carried out on the Univac 1100/80 computer of the Università di Roma and on the HP 21MX minicomputer of the CNR Research Area. The computer programs and atomic form factors used were the same as those in part V (Colapietro, Domenicano & Pela Ceccarini, 1979).

Results and discussion

Bond lengths and angles are given in Fig. 1 and Table 4; no corrections have been applied for the effects of thermal motion. Torsion angles involving the NH_3^+ substituent are listed in Table 4. The displacements of all atoms from the least-squares plane through the benzene ring are given in Table 5.

Table 4. Bond lengths (\AA), bond angles ($^\circ$) and torsion angles ($^\circ$) involving the NH_3^+ substituent

E.s.d.'s are given in parentheses as units in the last digit. Signs for the torsion angles have been given according to the convention of Klyne & Prelog (1960).

C(1)—N	1.467 (2)	N—H(2N)	0.90 (3)
N—H(1N)	0.88 (3)	N—H(3N)	0.92 (2)
C(1)—N—H(1N)	113 (2)	H(1N)—N—H(2N)	108 (3)
C(1)—N—H(2N)	111 (2)	H(1N)—N—H(3N)	108 (2)
C(1)—N—H(3N)	110 (1)	H(2N)—N—H(3N)	108 (2)
H(1N)—N—C(1)—C(2)	-133 (2)	H(2N)—N—C(1)—C(6)	168 (2)
H(1N)—N—C(1)—C(6)	47 (2)	H(3N)—N—C(1)—C(2)	106 (1)
H(2N)—N—C(1)—C(2)	-13 (2)	H(3N)—N—C(1)—C(6)	-74 (1)

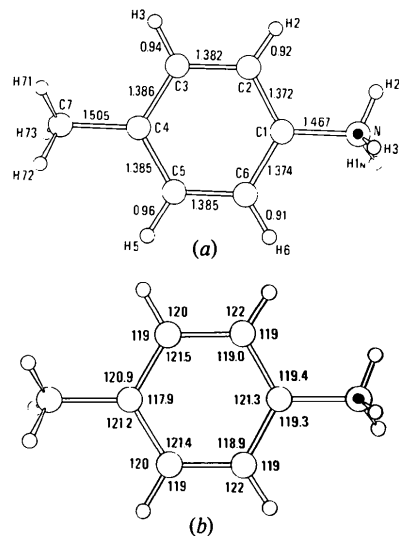


Fig. 1. Geometry of the cation in *p*-toluidine hydrochloride: (a) bond lengths (\AA), (b) bond angles ($^\circ$). E.s.d.'s (calculated from the e.s.d.'s in the atomic parameters given in Tables 2 and 3) are 0.002 \AA for the C(1)—N bond, 0.003 \AA for C—C bonds, 0.03 \AA for C—H bonds, 0.15–0.20 $^\circ$ for N—C—C and C—C—C angles, and ca 1.5 $^\circ$ for C—C—H angles. The methyl group is shown in the most populated conformation.

Table 5. *Deviations (Å) from planarity*

The equation of the least-squares plane through the six C atoms of the benzene ring, referred to the crystallographic axes, is $-1.7189x - 5.3480y + 8.0998z = -4.4498$. The e.s.d.'s in the atomic positions are 0.0028 Å for C(7), 0.0016–0.0021 Å for N and the other C atoms, 0.03–0.04 Å for H(71), H(72), H(73), and 0.02–0.03 Å for the other H atoms.

C(1)*	0.002	H(3)	-0.03
C(2)*	-0.004	H(5)	-0.04
C(3)*	0.000	H(6)	0.02
C(4)*	0.005	H(1N)	0.60
C(5)*	-0.007	H(2N)	0.19
C(6)*	0.003	H(3N)	-0.82
C(7)	0.033	H(71)	-0.39
N	0.005	H(72)	-0.55
H(2)	0.00	H(73)	0.88

* Atoms defining the least-squares plane.

The benzene ring

The bond lengths and angles of the ring conform within experimental error to the axial symmetry observed in most *para*-disubstituted benzene derivatives. Exact C_{2v} (mm) symmetry is not achieved, however, since some of the C atoms of the ring are slightly out of the least-squares plane (Table 5).

The distortions of the benzene ring from D_{6h} ($6/mmm$) symmetry are highly significant and involve bond distances as well as angles.

The ring C–C–C angles are all significantly different from 120° [Fig. 1(b)]. Their values are very well reproduced by superimposing independent distortional effects from each substituent. Indeed, the angles calculated from available angular substituent parameters (Domenicano & Murray-Rust, 1979; Norrestam & Schepper, 1981) are in excellent agreement with the present experimental results (Table 6).

The C–C bonds involving the C atom *ipso* to the NH_3^+ substituent, C(1)–C(2) and C(1)–C(6), are shorter than the central C–C bonds of the ring, C(2)–C(3) and C(5)–C(6), as expected with a substituent that causes an increase of the *ipso* angle (Domenicano, Vaciego & Coulson, 1975). The difference is *ca* 0.010 Å, essentially the same as in *p*-phenylenediamine dihydrochloride (Domenicano, Foresti Serantoni & Riva di Sanseverino, 1977), *p*-fluoroaniline hydrochloride and *p*-cyanoaniline hydrochloride (Colapietro, Domenicano, Marciante & Portalone, 1981). The ring C–C bonds involving the C atom *ipso* to the methyl group, C(3)–C(4) and C(4)–C(5), do not show the lengthening that should accompany a decrease of the *ipso* angle (Domenicano, Vaciego & Coulson, 1975). This is rather surprising, also, because a lengthening of the correct magnitude, 0.013 Å, was observed in *p*-xylene by gas-electron diffraction (Domenicano, Schultz, Kolonits & Hargittai, 1979).

Table 6. *Internal angles ($^\circ$) of the benzene ring: observed and predicted values*

The e.s.d.'s of the observed values are 0.16–0.20°.

	Observed	Predicted	
		(a)	(b)
C(2)–C(1)–C(6)	121.3	121.0	121.3
C(1)–C(2)–C(3)	119.0		
C(1)–C(6)–C(5)	118.9	119.2	119.0
C(2)–C(3)–C(4)	121.5		
C(4)–C(5)–C(6)	121.4	121.4	121.2
C(3)–C(4)–C(5)	117.9	118.0	118.2

(a) From the angular substituent parameters of Domenicano & Murray-Rust (1979).

(b) From the angular substituent parameters of Norrestam & Schepper (1981, Table 4).

The NH_3^+ substituent

The C(1)–N length, 1.467 (2) Å, is in the narrow range of values obtained in this Laboratory for other *para*-substituted anilinium cations, 1.463–1.467 Å. As in previous cases (Colapietro, Domenicano, Marciante & Portalone, 1981, and references therein) one of the H atoms of the substituent is almost eclipsed with the ring (Fig. 2 and Table 4).

The CH_3 substituent

The C atom of the methyl group, C(7), is bent out of the least-squares plane of the benzene ring by 0.033 Å, 1.3° . The length of the C(4)–C(7) bond, 1.505 (3) Å, is in excellent agreement with the mean value of 1.506 (2) Å calculated from 22 *para*-substituted derivatives of toluene (Domenicano, Vaciego & Coulson, 1975). The group occurs in two alternative conformations, both staggered with respect to the ring.

The crystal structure

The $CH_3-C_6H_4-NH_3^+$ and Cl^- ions are connected in infinite layers through $Cl^- \cdots H-N$ hydrogen bonds

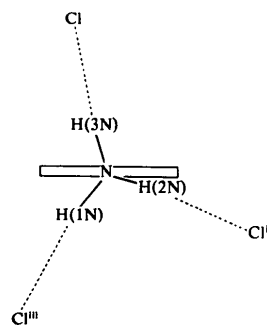


Fig. 2. Projection of the NH_3^+ group along the N–C(1) bond. The system of intermolecular hydrogen bonds is also shown. The superscripts refer to the symmetry operations given in the caption of Fig. 3.

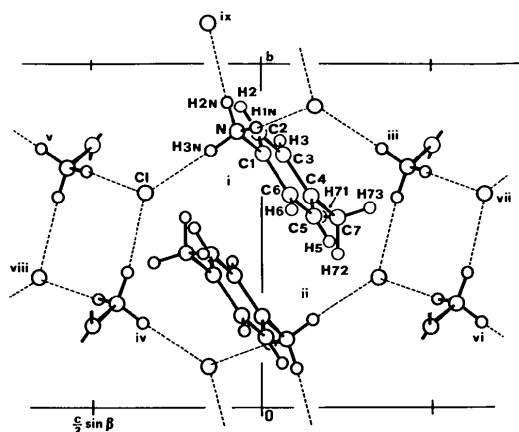


Fig. 3. Projection of the crystal structure down *a*. The broken lines represent intermolecular hydrogen bonds. The methyl groups are shown in the most populated conformation. The symmetry operations relating the various pairs of ions to pair (i) (which is at x, y, z) are: (ii) $1 - x, 1 - y, -z$; (iii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iv) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (v) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (vi) $1 - x, -\frac{1}{2} + y, -\frac{1}{2} - z$; (vii) $x, y, -1 + z$; (viii) $1 - x, 1 - y, 1 - z$; (ix) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Table 7. *Geometry of the hydrogen bonds*

E.s.d.'s are given in parentheses as units in the last digit. The superscripts refer to the symmetry operations given in the caption of Fig. 3.

(a) Distances (Å)			
Cl...N	3.117 (2)	Cl...H(3N)	2.21 (2)
Cl ⁱⁱⁱ ...N	3.143 (2)	Cl ⁱⁱⁱ ...H(1N)	2.29 (3)
Cl ^{ix} ...N	3.152 (2)	Cl ^{ix} ...H(2N)	2.27 (3)
(b) Angles (°)			
Cl...H(3N)-N	174 (2)	Cl ^{ix} ...H(2N)-N	170 (3)
Cl ⁱⁱⁱ ...H(1N)-N	164 (2)		

(Figs. 2 and 3). The layers run parallel to *bc*. The network of hydrogen bonds is different from that occurring in *p*-fluoroaniline hydrochloride and *p*-cyanoaniline hydrochloride, where the $X-C_6H_4-NH_3^+$ ($X = F, CN$) and Cl^- ions are connected in ribbons, which in

turn are joined in layers through $Cl^- \cdots H-N$ contacts (Colapietro, Domenicano, Marciante & Portalone, 1981).

Also the environment of the Cl^- ion is, of course, different. This ion takes part in three $Cl^- \cdots H-N$ hydrogen bonds (Von Eller, 1955); it is not involved, however, in bifurcated interactions, nor does it have close contacts with aromatic protons.* Geometrical details of the three independent hydrogen bonds are given in Table 7.

We thank Mrs C. Marciante for skilful technical help.

* The two shortest $Cl^- \cdots H-C$ contacts involve H(3) at $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$ and H(2) at $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$, and have $Cl^- \cdots H$ distances of 2.96 (3) and 3.14 (3) Å, respectively; compare 2.79 (3) and 2.82 (3) Å in either *p*-fluoroaniline hydrochloride or *p*-cyanoaniline hydrochloride (Colapietro, Domenicano, Marciante & Portalone, 1981).

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