1·45 (2) Å], and the four remaining C—C bonds are shortened, av. 1·376 (6) Å [1·38 (1) Å].

The three nitro groups display various degrees of twisting, moderate for one of the *o*-nitro groups [9·0 (4)°] and the *p*-nitro group [15·0 (4)°], but large for the other *o*-nitro group [41·8 (4)°].

References

ALLEN, F. H., KENNARD, O. & TAYLOR, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
GRIFFIN, J. F. & COPPENS, P. (1973). Am. Crystallogr. Assoc. Winter Meet. *Program and Abstracts*, p. 30.
HERBSTEIN, F. H., KAPON, M. & WIELINSKI, S. (1977). *Acta Cryst.* **B33**, 649–654.
MAARTMANN-MOE, K. (1969). *Acta Cryst.* **B25**, 1452–1460.
PALENIK, G. J. (1972). *Acta Cryst.* **B28**, 1633–1635.
SHELDRICK, G. M. (1976). *SHELX76*. Program for structure determination. Univ. of Cambridge, England.
SPEK, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.
STAMMLER, M. (1968). *Explosivstoffe*, **16**, 154–163.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1990). C**46**, 64–66

Dimethylbis(5-methylfurfuryl)ammonium Chloride

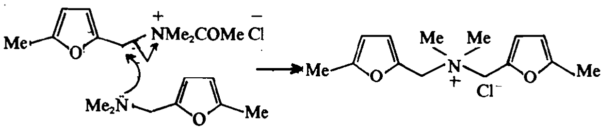
BY D. S. BROWN, C. A. BURNS, H. HEANEY, G. PAPAGEORGIOU AND R. F. WILKINS

*Department of Chemistry, Loughborough University of Technology, Loughborough, Leics. LE*11 3*TU*, *England*

(*Received* 24 *February* 1989; *accepted* 24 *April* 1989)

Abstract. $C_{14}H_{20}NO_2^+.Cl^-$, $M_r = 269{\cdot}8$, monoclinic, $C2/c$, $a = 6{\cdot}24$ (1), $b = 14{\cdot}47$ (1), $c = 15{\cdot}96$ (1) Å, $\beta = 92{\cdot}3$ (2)°, $V = 1439{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}25$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}17$ cm^{-1}, $F(000) = 576$, $T = 293$ K. The structure was refined to $R = 0{\cdot}034$ for 1141 observed reflections [$F/\sigma(F) > 6$]. The compound is a by-product from the interaction of a Mannich base with acetyl chloride resulting in the quaternary ammonium salt. The salt possesses a twofold axis of symmetry with N and Cl occupying special positions, the quaternary nitrogen having two short and two long C—N bonds [1·533 (2) and 1·496 (2) Å].

Introduction. Mannich reactions of furan had been reported not to proceed using classical methodology (Eliel & Peckham, 1950; Eliel & Fisk, 1963). As part of a recent study (Heaney, Papageorgiou & Wilkins, 1988), reactions of pre-formed methyleneimonium salts were carried out using both furan and 2-methylfuran. The normal method of isolating the Mannich bases involves extraction with diethyl ether. However, isolation of 2-dimethylaminomethyl-5-methylfuran (1) by extraction using dichloromethane gave rise to variable quantities of a quaternary ammonium salt which was shown to have the structure (2). Although the majority of the physical properties of (1) and (2) were different, we were unable to establish the molecular weight of the salt (2) using electron-impact mass spectrometry. It was for this reason that X-ray crystallographic studies were initiated. Although the electron-impact mass spectra of the compounds (1) and (2) were essentially identical, we were eventually able to obtain a fast-atom-bombardment spectrum which did establish the structure.



(1) (2)

It is assumed that the quaternary salt (2) is formed by the displacement of dimethylamine from the hydrochloride derived from (1). The salt (2) was subsequently prepared in 98% yield by the reaction of acetyl chloride with 2-dimethylaminomethyl-5-methylfuran (2 mol equivalents).

Experimental. Preparation by the method of Heaney *et al.* (1988); colourless acicular crystal, 1·0 × 0·4 × 0·3 mm, mounted about *c*; Stoe Stadi-2 Weissenberg diffractometer, $(\sin\theta)\lambda < 0{\cdot}6$ Å^{-1}; lattice parameters from optimum fit of axial row reflections ($15 < 2\theta < 40°$); 1631 unique reflections measured of which 1141 had $F/\sigma(F) > 6$ and were classed as observed; h −6→6, k 0→16, l 0→18; standard reflections on each layer measured every 50 reflections, no signifi-

0108-2701/90/010064-03$03.00

cant change; no corrections for absorption or extinction; structure solved by direct methods using *EEES* (Sheldrick, 1976) and refined by full-matrix least squares based on F to $R = 0{\cdot}034$ (unit weights); H atoms found from difference map and refined isotropically, all non-H atoms anisotropic. Max. $\Delta/\sigma = 0{\cdot}003$, $\Delta\rho$ excursions $+0{\cdot}1$ to $-0{\cdot}2$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974), structure refinement using *SHELX76* (Sheldrick, 1976) and geometry calculations using *XTAL83* (Stewart & Hall, 1983) as implemented at Loughborough University Computer Centre and University of Manchester Regional Computer Centre respectively.

Table 1. *Atomic coordinates* ($\times 10^5$) *and equivalent isotropic thermal parameters*

$$B_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i B_{ii}.$$

	x	y	z	$B_{\rm eq}$ (Å^2)
Cl(1)	50000	8885 (6)	75000	4·4 (1)
O(1)	103747 (21)	11710 (9)	4807 (7)	3·3 (1)
N(1)	100000	9375 (14)	25000	2·7 (1)
C(1)	88563 (31)	7409 (13)	9586 (11)	3·1 (1)
C(2)	69462 (36)	7364 (15)	5387 (13)	3·9 (1)
C(3)	72626 (37)	11855 (15)	−2448 (13)	4·0 (1)
C(4)	93179 (33)	14356 (13)	−2568 (11)	3·4 (1)
C(5)	106263 (48)	19242 (18)	−8746 (15)	4·5 (1)
C(6)	95999 (34)	2914 (13)	17485 (11)	3·2 (1)
C(7)	80830 (40)	15324 (18)	26354 (15)	4·3 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·343 (3)	C(4)—C(5)	1·484 (3)
C(1)—C(6)	1·477 (2)	C(4)—O(1)	1·380 (2)
C(1)—O(1)	1·387 (2)	C(6)—N(1)	1·533 (2)
C(2)—C(3)	1·430 (3)	C(7)—N(1)	1·496 (2)
C(3)—C(4)	1·333 (3)		
C(2)—C(1)—C(6)	131·7 (2)	C(5)—C(4)—O(1)	116·3 (2)
C(2)—C(1)—O(1)	109·8 (2)	C(1)—C(6)—N(1)	115·8 (2)
C(6)—C(1)—O(1)	118·0 (2)	C(6)—N(1)—C(7)	111·0 (1)
C(1)—C(2)—C(3)	106·4 (2)	C(6)—N(1)—C(6)	104·8 (1)
C(2)—C(3)—C(4)	107·6 (2)	C(6)—N(1)—C(7)	110·1 (1)
C(3)—C(4)—C(5)	133·9 (2)	C(7)—N(1)—C(7)	109·7 (2)
C(3)—C(4)—O(1)	109·8 (2)	C(1)—O(1)—C(4)	106·3 (1)

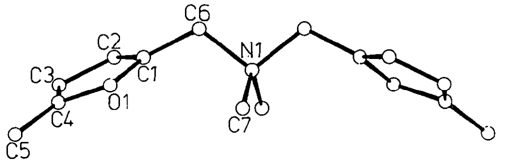


Fig. 1. *ORTEP* (Johnson, 1965) drawing of molecular structure and numbering.

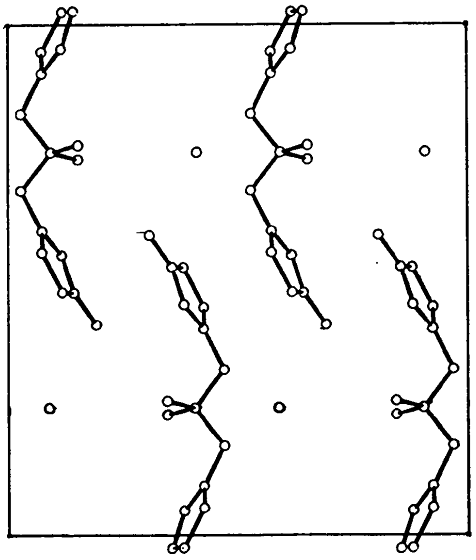


Fig. 2. Unit-cell contents projected down **a**.

Discussion. The final atomic coordinates are listed in Table 1* and bond lengths and angles in Table 2. The molecular structure and atom numbering are shown in Fig. 1; unit-cell contents projected down **a** are represented in Fig. 2.

The structure of the quaternary ammonium salt has been confirmed as that of (2). The molecule possesses a twofold axis of symmetry with N and Cl occupying special positions. The C atoms attached to the nitrogen are arranged in an approximately tetrahedral manner although the bond angles suggest some interaction of the methyl groups with each other and also with the adjacent rings. The bond lengths in the furyl rings are similar to those in furan itself (Gilchrist, 1985). Some double-bond character is observed in the C(1)—C(6) bond [1·478 (2) Å], presumably due to interaction with the furan ring. There is a corresponding lengthening of the C(6)—N(1) bond length to 1·530 (2) Å compared to the average value of about 1·51 Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987).

There are no abnormal intermolecular contacts, the nearest contact involving the chloride ion being one of 3·59 Å with C(6) and C(6′).

---

* Lists of structure factors, hydrogen positions and anistropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52169 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Eliel, E. L. & Fisk, M. T. (1963). *Org. Synth. Collect.* **4**, 626–627.

Eliel, E. L. & Peckham, P. E. (1950). *J. Am. Chem. Soc.* **72**, 1209–1212.

Gilchrist, T. L. (1985). *Heterocyclic Chemistry*, p. 13. London: Pitman.

Heaney, H., Papageorgiou, G. & Wilkins, R. F. (1988). *Tetrahedron Lett.* **29**, 2377–2380.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

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

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

STEWART, J. M. & HALL, S. R. (1983). The *XTAL* system. Tech. Rep. TR-1364. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Acta Cryst. (1990). C46, 66–68

Structure of 2-(*p*-Toluenesulfonyl)-1,2,3,4-tetrahydro-1-isoquinolinecarbonitrile

BY J. WDOWIAK, M. GDANIEC AND Z. KOSTURKIEWICZ*

Department of Crystallography, A. Mickiewicz University, ul. Grunwaldzka 6, 60–780 *Poznań, Poland*

(*Received* 28 *December* 1988; *accepted* 10 *April* 1989)

Abstract. $C_{17}H_{16}N_2O_2S$, M_r = 312·4, monoclinic, $P2_1$, a = 10·278 (1), b = 6·467 (1), c = 11·972 (1) Å, β = 95·46 (1)°, V = 792·1 (2) Å^3, Z = 2, D_x = 1·31 g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 17·74 cm^{-1}, $F(000)$ = 328, room temperature, R = 0·054 for 1088 observed reflexions. The heterocyclic fragment of the molecule exhibits a conformation intermediate between half-chair and sofa. The N atom shows considerable pyramidalization [sum of valency angles = 353 (1)°].

Introduction. This work is a continuation of our studies of the structures of Reissert compounds (Reissert, 1905). It is of interest to determine the effects of substituents on the molecular conformation in such a group of compounds. Our aim is to compare the arrangement of the side fragments relative to the central N(2) atom depending on the character and dimensions of those fragments. So far we have investigated the structure of 2-benzoyl-1,2,3,4-tetrahydro-1-isoquinolinecarbonitrile (I) (Pływaczyk, Tykarska, Jaskólski & Kosturkiewicz, 1984), ethyl 1-cyano-1,2,3,4-tetrahydro-2-isoquinolinecarboxylate (II) (Gzella, Jaskólski, Rychlewska & Kosturkiewicz, 1984) and 2-(*p*-toluenesulfonyl)-1,2,3,4-tetrahydroisoquinoline (III) (Urbaniak, Jaskólski, Rozwadowska & Kosturkiewicz, 1986), which is the product of reductive decyanation of the present Reissert compound.

Experimental. Crystals obtained from methanol, colorless plates. Space group from Weissenberg photographs and the lack of a symmetry center from statistics. Crystal 0·03 × 0·15 × 0·5 mm. Syntex $P2_1$ diffractometer, θ–2θ scan. Cell parameters from least-squares treatment of setting angles of 15 reflexions with $16 \leq 2\theta \leq 26°$. An empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968) (correction factors were in the range 0·77–0·99). 1175 reflexions with $2\theta \leq 115°$, measured in the range h: $-11 \rightarrow 11$, k: $0 \rightarrow 7$, l: $0 \rightarrow 13$. No significant intensity variation (±2·7%) for two standard reflexions ($0\bar{1}1$, 200) recorded every hour. Peak profile analysis according to Lehmann & Larsen (1974), 1091 observed reflexions with $I \geq 2\sigma(I)$. Structure solved by heavy-atom method using *SHELX76* program (Sheldrick, 1976). Full-matrix least-squares refinement (function minimized: $\sum w(F_o - F_c)^2$] of anisotropic non-H atoms. H atoms from molecular geometry, included with fixed isotropic thermal parameters in F_c calculation. R = 0·054, wR = 0·070, S = 4·2, $w = 1/[\sigma^2(F_o) + 0·0001F_o^2]$. $\Delta/\sigma \leq$ 0·2, $\Delta\rho_{\max}$ = 0·22, $\Delta\rho_{\min}$ = −0·17 e Å^{-3}.

Computer programs: *SHELX76* (Sheldrick, 1976) and local programs (Jaskólski, 1982). Scattering factors those stored in *SHELX76*. Molecular illustrations drawn using *PLUTO* (Motherwell & Clegg, 1978) and *ORTEP* (Johnson, 1976).

Discussion. Atomic parameters are given in Table 1, bond lengths and angles in Table 2.† The labeling sequence and a view of the molecule are presented in Fig. 1. The molecule consists of an aromatic ring condensed with a heterocyclic hydrogenated ring containing a cyano group and linked through the ring N atom to a toluenesulfonyl group. Bond lengths and angles in the aromatic rings do not deviate significantly from standard values, and the planes of these rings form an angle of 43·4 (1)°. The C9—C1—N2 angle is tetrahedral [110·4 (4)°] in

---

* To whom correspondence should be addressed.

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52131 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/90/010066-03$03.00