References

CLEGG, W. (1981). *Acta Cryst.* A**37**, 22–28.

JONES, P. G., EDWARDS, M. R. & KIRBY, A. J. (1986). *Acta Cryst.* C**42**, 1370–1372, and references therein.

SHELDRICK, G. M. (1983). *SHELXTL Users Manual*, revision 4. Nicolet XRD Corporation, Madison, Wisconsin.

YUFIT, D. S., YANOVSKII, A. I., FROLOV, Y. L. & STRUCHKOV, YU. T. (1983). *Kristallografiya*, **28**, 84–90.

Acta Cryst. (1987). C**43**, 366–367

1-(4-Nitrophenyl)ethyl *p*-Toluenesulfonate

BY PETER G. JONES, KAREN MEYER-BÄSE AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

(*Received* 14 *November* 1985; *accepted* 15 *August* 1986)

Abstract. $C_{15}H_{15}NO_5S$, $M_r = 321·36$, orthorhombic, $Pca2_1$, $a = 13·311$ (4), $b = 7·643$ (2), $c = 15·511$ (4) Å, $U = 1578$ Å^3, $Z = 4$, $D_x = 1·35$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å^3, $\mu = 2·0$ cm^{-1}, $F(000) = 672$, $T = 293$ K, $R = 0·063$ for 1859 reflections. The torsion angles of the Ar—S—O—C—Ar chain are 76·8 (5) and 117·4 (5)° about the S—O and O—C bonds, respectively.

Experimental. Colourless blocks from diffusion of petrol into CH_2Cl_2 solution. Fragment 0·4 × 0·4 × 0·35 mm cut from larger crystal. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode (Clegg, 1981). $2\theta_{max}$ 50°, 2958 reflections, octants $+h+k+l$ and $-h-k-l$. Three check reflections, no significant intensity variation. No absorption correction. 2766 unique reflections, R_{int} 0·020, index range $|h| \leq 14$, $|k| \leq 9$, $|l| \leq 18$. 1859 reflections with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*; Sheldrick, 1983). Cell constants refined from $\pm 2\theta$ values of 38 reflections in the range 20–23°.

Structure solution by random-start direct methods. Refinement on F to R 0·063, wR 0·059. All non-H

Table 1. *Atom coordinates* (× 10⁴) *and isotropic temperature factors* (Å^2 × 10^3)

	x	y	z	U_{eq}*
S	5299 (1)	5108 (2)	5000	61 (1)
O(1)	5325 (3)	3374 (5)	4417 (2)	74 (2)
O(2)	4315 (3)	5619 (5)	5175 (2)	90 (2)
O(3)	5927 (3)	6213 (5)	4527 (3)	84 (2)
N(4)	2168 (5)	1068 (7)	1594 (4)	78 (2)
O(41)	2589 (4)	897 (7)	898 (3)	108 (2)
O(42)	1268 (4)	1031 (9)	1707 (3)	124 (2)
C(3)	4636 (4)	1951 (7)	4576 (3)	67 (2)
C(4)	5206 (5)	391 (8)	4835 (4)	93 (3)
C(11)	5895 (4)	4494 (6)	5974 (3)	52 (2)
C(12)	6902 (4)	4154 (7)	5971 (3)	62 (2)
C(13)	7362 (4)	3700 (7)	6738 (4)	71 (2)
C(14)	6834 (5)	3552 (7)	7505 (3)	64 (2)
C(15)	5835 (6)	3890 (7)	7465 (4)	73 (2)
C(16)	5330 (4)	4332 (7)	6716 (3)	64 (2)
C(17)	7357 (6)	3085 (9)	8327 (4)	96 (3)
C(21)	4010 (4)	1774 (6)	3794 (3)	51 (2)
C(22)	2984 (4)	2054 (6)	3819 (3)	59 (2)
C(23)	2395 (4)	1832 (7)	3122 (3)	59 (2)
C(24)	2820 (4)	1369 (7)	2354 (3)	53 (2)
C(25)	3823 (4)	1098 (7)	2272 (3)	64 (2)
C(26)	4421 (4)	1292 (7)	3004 (3)	67 (2)

* Equivalent isotropic U calculated from anisotropic U.

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

O(1)—S	1·604 (5)	O(2)—S	1·394 (5)
O(3)—S	1·396 (5)	N(4)—O(41)	1·223 (8)
N(4)—O(42)	1·210 (9)	C(3)—O(1)	1·444 (7)
C(3)—C(4)	1·469 (9)	C(11)—S	1·770 (6)
C(11)—C(12)	1·366 (8)	C(11)—C(16)	1·380 (8)
C(12)—C(13)	1·382 (9)	C(13)—C(14)	1·386 (9)
C(14)—C(15)	1·356 (11)	C(14)—C(17)	1·495 (10)
C(15)—C(16)	1·384 (9)	C(21)—C(3)	1·478 (8)
C(21)—C(22)	1·383 (9)	C(21)—C(26)	1·391 (8)
C(22)—C(23)	1·346 (9)	C(23)—C(24)	1·366 (8)
C(24)—N(4)	1·483 (9)	C(24)—C(25)	1·357 (9)
C(25)—C(26)	1·395 (8)		

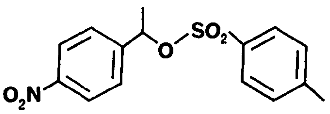
O(1)—S—O(2)	111·2 (3)	O(1)—S—O(3)	101·0 (3)
O(2)—S—O(3)	119·7 (3)	O(1)—S—C(11)	104·6 (3)
O(2)—S—C(11)	109·2 (3)	O(3)—S—C(11)	109·9 (3)
S—O(1)—C(3)	120·8 (4)	O(41)—N(4)—O(42)	125·4 (7)
O(41)—N(4)—C(24)	116·7 (6)	O(42)—N(4)—C(24)	117·9 (6)
O(1)—C(3)—C(4)	109·3 (6)	O(1)—C(3)—C(21)	106·7 (5)
C(4)—C(3)—C(21)	116·2 (6)	S—C(11)—C(12)	119·1 (5)
S—C(11)—C(16)	119·5 (5)	C(12)—C(11)—C(16)	121·4 (6)
C(11)—C(12)—C(13)	118·6 (6)	C(12)—C(13)—C(14)	122·3 (6)
C(13)—C(14)—C(15)	116·2 (6)	C(13)—C(14)—C(17)	121·0 (7)
C(15)—C(14)—C(17)	122·8 (7)	C(14)—C(15)—C(16)	124·1 (7)
C(11)—C(16)—C(15)	117·2 (6)	C(3)—C(21)—C(22)	121·3 (6)
C(3)—C(21)—C(26)	121·7 (6)	C(22)—C(21)—C(26)	116·9 (6)
C(21)—C(22)—C(23)	122·3 (6)	C(22)—C(23)—C(24)	119·4 (6)
N(4)—C(24)—C(23)	119·4 (6)	N(4)—C(24)—C(25)	118·6 (6)
C(23)—C(24)—C(25)	122·0 (6)	C(24)—C(25)—C(26)	117·9 (6)
C(21)—C(26)—C(25)	121·4 (6)		

O(2)—S—O(1)—C(3)	−41·0 (5)	O(3)—S—O(1)—C(3)	−169·0 (5)
C(11)—S—O(1)—C(3)	76·8 (5)	O(1)—S—C(11)—C(12)	69·0 (5)
O(1)—S—C(11)—C(16)	−109·6 (5)	O(2)—S—C(11)—C(12)	−171·9 (5)
O(2)—S—C(11)—C(16)	9·5 (6)	O(3)—S—C(11)—C(12)	−38·8 (6)
O(3)—S—C(11)—C(16)	142·6 (5)	S—O(1)—C(3)—C(4)	−116·3 (5)
S—O(1)—C(3)—C(21)	117·4 (5)	O(41)—N(4)—C(24)—C(23)	170·1 (6)
O(41)—N(4)—C(24)—C(25)	−12·8 (9)	O(42)—N(4)—C(24)—C(23)	−9·0 (9)
O(42)—N(4)—C(24)—C(25)	168·1 (7)	O(1)—C(3)—C(21)—C(22)	−116·6 (6)
O(1)—C(3)—C(21)—C(26)	64·5 (7)	C(4)—C(3)—C(21)—C(22)	121·3 (7)
C(4)—C(3)—C(21)—C(26)	−57·6 (8)	S—C(11)—C(12)—C(13)	179·1 (5)
C(16)—C(11)—C(12)—C(13)	−2·3 (9)	S—C(11)—C(16)—C(15)	−178·5 (5)
C(12)—C(11)—C(16)—C(15)	2·9 (8)	C(11)—C(12)—C(13)—C(14)	1·1 (9)
C(12)—C(13)—C(14)—C(15)	−0·5 (9)	C(12)—C(13)—C(14)—C(17)	−179·0 (6)
C(13)—C(14)—C(15)—C(16)	1·2 (9)	C(17)—C(14)—C(15)—C(16)	179·7 (6)
C(14)—C(15)—C(16)—C(11)	−2·4 (9)	C(3)—C(21)—C(22)—C(23)	−177·3 (6)
C(26)—C(21)—C(22)—C(23)	1·7 (8)	C(3)—C(21)—C(26)—C(25)	178·9 (6)
C(22)—C(21)—C(26)—C(25)	−0·1 (8)	C(21)—C(22)—C(23)—C(24)	−2·2 (9)
C(22)—C(23)—C(24)—N(4)	178·0 (6)	C(22)—C(23)—C(24)—C(25)	1·0 (9)
N(4)—C(24)—C(25)—C(26)	−176·5 (6)	C(23)—C(24)—C(25)—C(26)	0·5 (9)

0108-2701/87/020366-02$01.50

atoms anisotropic; H atoms included using riding model with C—H 0·96 Å, H—C—H 109·5°, U(H) = 1·2U_{eq}(C); weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}00025F^2$; 204 parameters; S = 1·80; max. Δ/σ 0·02; max. and min. features in final $\Delta\rho$ map 0·7, −0·5 e Å^{-3}. Absolute structure (Jones, 1984) weakly indicated by η refinement (Rogers, 1981); η = 1·3 (3). Atomic scattering factors from *SHELXTL*. Final atomic coordinates are given in Table 1, and bond lengths and angles in Table 2.* Fig. 1 shows the atom-numbering scheme.

Related literature. For results of a series of structure determinations of ethers and esters of 1-(4-nitrophenyl)ethanol, see Jones, Edwards & Kirby (1986). The large difference peak near the C—O—S moiety, possibly an unidentified minor disorder or twinning phenomenon, precludes the comparison of the current structure (1).



(1)

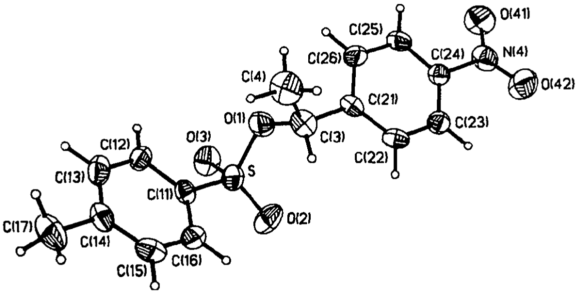


Fig. 1. Molecule of the title compound, showing the atom-numbering scheme.

We thank the Verband der Chemischen Industrie for financial support, and Dr A. J. Kirby and Mr M. R. Edwards for providing the sample.

References

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.
Jones, P. G. (1984). *Acta Cryst.* A**40**, 660–662.
Jones, P. G., Edwards, M. R. & Kirby, A. J. (1986). *Acta Cryst.* C**42**, 1370–1372, and references therein.
Rogers, D. (1981). *Acta Cryst.* A**37**, 734–741.
Sheldrick, G. M. (1983). *SHELXTL Users Manual*, revision 4. Nicolet XRD Corporation, Madison, Wisconsin.

---

* Lists of structure factors, H-atom coordinates, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43068 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1987). C**43**, 367–368

Bis(cyclohexylammonium) Hydrogenphosphate

By Peter G. Jones

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

(*Received* 14 *November* 1985; *accepted* 15 *August* 1986)

Abstract. $2C_6H_{14}N^+.HPO_4^{2-}$, $M_r = 296{\cdot}35$, monoclinic, $P2_1/c$, $a = 14{\cdot}451\ (4)$, $b = 12{\cdot}955\ (4)$, $c = 8{\cdot}157\ (2)$ Å, $\beta = 90{\cdot}11\ (2)$°, $U = 1527{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}29$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 2·0 cm^{-1}, $F(000) = 648$, $T = 293$ K, $R = 0{\cdot}044$ for 2265 reflections. A hydrogen-bonding network connects NH_3^+ and HPO_4^{2-} units and also the phosphate ions amongst themselves; all X—H atoms (X = O or N) are donors and all O atoms (except that bearing H) twofold acceptors. P—O bond lengths 1·609 (2) to (OH), 1·510 (2), 1·514 (2), 1·522 (2) Å.

Experimental. Stoe two-circle diffractometer, monochromated Mo $K\alpha$ radiation, $2\theta_{max}$ 50°, two crystals: (*a*) colourless needle, 0·55 × 0·1 × 0·1 mm, mounted about **b**, layers 0–14, 5034 reflections, and (*b*) 0·7 × 0·2 × 0·1 mm, mounted about **c** (the needle axis), layers 0–2, 1259 reflections. No absorption correction. Interlayer scale factors by analysis of common reflections. 2806 unique reflections (R_{int} 0·023), 2265 with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*; Sheldrick, 1983). Index range after merging: $|h| \le 17$, $k \le 15$, $l \le 9$. Cell constants refined from $\pm 2\theta$ values of 22 reflections [measured on Stoe–Siemens four-circle diffractometer with crystal (*b*)].

Structure solution by routine direct methods, four missing non-H atoms located in difference synthesis.