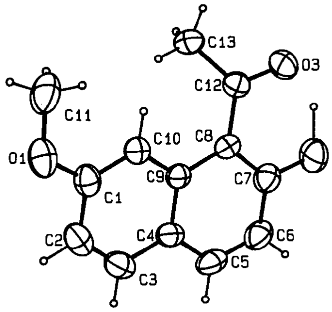

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with arbitrary radius.

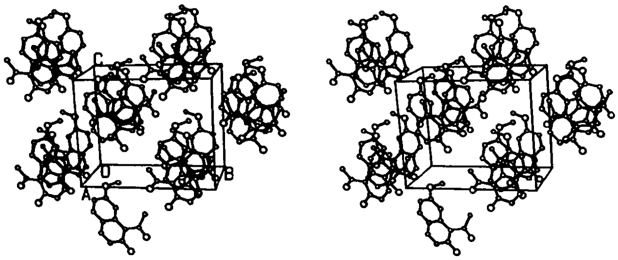

Fig. 2. Stereoview of the unit cell.

Related literature. The literature contains seven crystal structures of organic compounds containing the 2-hydroxyacetophenone fragment, and forming an intramolecular hydrogen bond between the hydroxy group and the acetyl O atom (Cohen *et al.*, 1989; Li, Li, Yang, You, Huang & Wang, 1986; Nowshad & Ul-Haque, 1976; Ray & Sen Gupta, 1981; Schlemper, 1986; Wajsman, Grabowski, Stępień & Cygler, 1978; Wu, Wang, Jong, McPhail, McPhail & Lee, 1989). The average O···O distance is 2.52 Å.

Support for this work was provided by a grant from the National Science Foundation.

References

Cohen, N., Weber, G., Banner, B. L., Lopresti, R. J., Schaer, B., Focella, A., Zenchoff, G. B., Chiu, A.-M., Todaro, L., O'Donnell, M., Welton, A. F., Brown, D., Garippa, R., Crowley, H. & Morgan, D. W. (1989). *J. Med. Chem.* **32**, 1842–1860.

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Frenz, B. A. & Okaya, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Li, C., Li, D., Yang, X., You, X., Huang, J. & Wang, M. (1986). *Youji Huaxue*, pp. 365–368.

Nowshad, F. & Ul-Haque, M. (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 623–626.

Prince, P., Fronczek, F. R. & Gandour, R. D. (1989). *Acta Cryst.* C**45**, 1256–1258.

Ray, T. & Sen Gupta, S. P. (1981). *Cryst. Struct. Commun.* **10**, 1123–1128.

Schlemper, E. O. (1986). *Acta Cryst.* C**42**, 755–757.

Wajsman, E., Grabowski, M. J., Stępień, A. & Cygler, M. (1978). *Cryst. Struct. Commun.* 7, 233–236.

Wu, T.-S., Wang, M.-L., Jong, T.-T., McPhail, A. T., McPhail, D. R. & Lee, K.-H. (1989). *J. Nat. Prod.* **52**, 1284–1289.

Yao, J.-X. (1981). *Acta Cryst.* A**37**, 642–644.

Acta Cryst. (1992). C**48**, 2227–2229

Structure of 2-Phenylpyrimidinium 1-Oxide

By Ivan Leban and Slovenko Polanc

Department of Chemistry and Chemical Technology, University of Ljubljana, 61001 *Ljubljana, POB* 537, *Slovenia*

(*Received* 9 *January* 1992; *accepted* 20 *March* 1992)

Abstract. $C_{10}H_8N_2O$, $M_r = 172.2$, orthorhombic, $P2_12_12_1$, $a = 7.189\,(1)$, $b = 10.141\,(2)$, $c = 11.302\,(2)$ Å, $V = 824.0\,(3)$ Å^3, $Z = 4$, $D_x = 1.388$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.87$ cm^{-1}, $F(000) = 360$, $T = 293\,(2)$ K, final $R = 0.058$ and $wR = 0.064$ for 1104 observed reflections. The benzene and pyrimidine rings are planar to within 0.020 (2) and 0.007 (3) Å respectively. The dihedral angle between the rings is 25.0 (5)°. The bond lengths and angles agree with expected values.

Experimental. A prismatic single crystal (0.63 × 0.50 × 0.47 mm) was used for X-ray data collection. Cell dimensions were determined from the angular settings of 25 reflections with $10 < \theta < 12°$, measured on an Enraf–Nonius CAD-4 diffractometer. Space

0108-2701/92/122227-03$06.00

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors* (Å^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O	0.1045 (3)	0.2025 (2)	−0.2297 (1)	0.064
N1	0.1259 (3)	0.1103 (2)	−0.1516 (2)	0.042
C2	0.1532 (3)	0.1370 (2)	−0.0331 (2)	0.039
N3	0.1821 (3)	0.0397 (2)	0.0434 (2)	0.047
C4	0.1798 (5)	−0.0851 (2)	0.0073 (2)	0.054
C5	0.1448 (4)	−0.1181 (2)	−0.1093 (2)	0.053
C6	0.1180 (4)	−0.0177 (3)	−0.1879 (2)	0.050
C11	0.1490 (3)	0.2732 (2)	0.0133 (2)	0.037
C12	0.1891 (4)	0.3857 (2)	−0.0532 (2)	0.049
C13	0.1865 (5)	0.5088 (3)	0.0005 (3)	0.060
C14	0.1424 (5)	0.5230 (3)	0.1182 (3)	0.059
C15	0.1020 (5)	0.4132 (3)	0.1839 (2)	0.059
C16	0.1065 (4)	0.2883 (2)	0.1331 (2)	0.047

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

O—N1	1.295 (3)	C5—C6	1.366 (4)
N1—C2	1.381 (4)	C11—C12	1.396 (4)
N1—C6	1.362 (4)	C11—C16	1.396 (4)
C2—N3	1.328 (4)	C12—C13	1.387 (4)
C2—C11	1.478 (3)	C13—C14	1.375 (5)
N3—C4	1.330 (3)	C14—C15	1.369 (5)
C4—C5	1.382 (4)	C15—C16	1.391 (4)
O—N1—C2	122.4 (2)	N1—C6—C5	120.5 (2)
O—N1—C6	118.5 (2)	C2—C11—C12	124.7 (2)
C2—N1—C6	119.0 (2)	C2—C11—C16	116.8 (2)
N1—C2—N3	120.5 (2)	C12—C11—C16	118.5 (2)
N1—C2—C11	121.6 (2)	C11—C12—C13	119.8 (3)
N3—C2—C11	117.8 (2)	C12—C13—C14	121.4 (3)
C2—N3—C4	120.3 (2)	C13—C14—C15	119.2 (3)
N3—C4—C5	121.7 (2)	C14—C15—C16	120.8 (3)
C4—C5—C6	117.7 (2)	C11—C16—C15	120.3 (2)

group $P2_12_12_1$ was determined from systematic absences. Intensity data were collected in the range $1 < 2\theta < 60°$ with variable-speed ω–2θ scans using graphite-monochromated Mo $K\alpha$ radiation: maximum scan time 60 s; scan width (2θ) $(0.7 + 0.3\tan\theta)°$; aperture $(2.4 + 0.9\tan\theta)$ mm; background measured for one quarter of scan time at each scan limit.

Three reflections were monitored at intervals of 400 reflections. Orientation was checked after every 2 h. Data were corrected for intensity variation (−5.4%) and Lp effects, but absorption was ignored. A total of 5222 reflections ($+h$, $\pm k$, $\pm l$) were collected. On averaging ($R_{\rm int} = 3.7\%$), 1104 reflections were considered observed [$I > 3.5\sigma(I)$, $\sigma(I)$ based on counting statistics] out of 1393 unique reflections; range of hkl: h 0/9, k 0/13, l 0/15. The structure was solved in a routine manner with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The structural model obtained was refined by full-matrix least squares minimizing the function $\sum w(|F_o| - |F_c|)^2$ with *SHELX*76 (Sheldrick, 1976). Final R and wR values were 0.058 and 0.064, respectively, with $w = 69.1/[\sigma^2(F_o)]$ and 119 variables. H-atom positions were found in the difference synthesis and included in the refinement with constraints imposed only on the C—H bonds. A common isotropic temperature factor U for all H atoms was 0.066 (2) Å^2 at convergence. A final difference Fourier map showed residual electron density within 0.37 and −0.22 e Å^{-3}. The maximum value of Δ/σ at convergence was 0.029 (overall scale factor). Scattering factors for C, N and O atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and for H atoms from Stewart, Davidson & Simpson (1965). All calculations were performed on the VAX 8550 cluster at the University Computer Centre, Ljubljana. The *GX* package (Mallinson & Muir, 1985) was

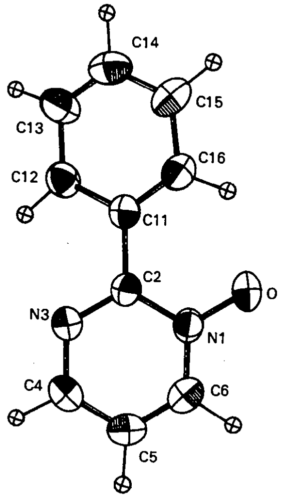

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule with the atomic numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

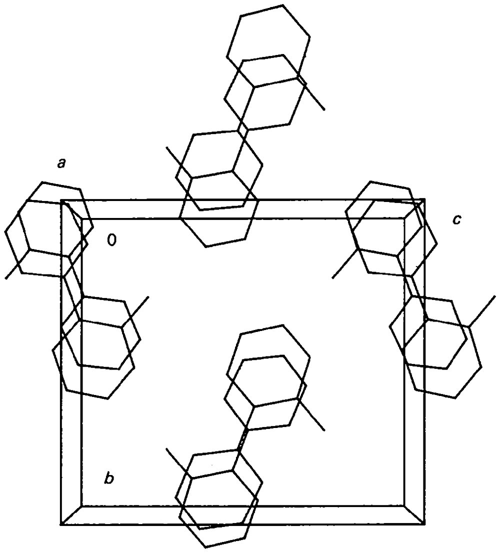

Fig. 2. View of the molecular packing.

used for data processing and final interpretation of molecular geometry. The final atomic parameters are listed in Table 1.* Bond lengths and angles are given in Table 2. A view of the molecule with the atomic numbering, and of the molecular packing are presented in Figs. 1 and 2.

Related literature. The reaction of 2,6-dichlorobenzamide oxime with 1,3-dichloropropane results in the formation of 4,5,6,7-tetrahydro-1,2,4-oxadiazepine as a stable product (Shell Research Ltd, 1963). However, the thermal reaction of 4,7-dihydro-5,7-diphenyl-1,2,4-oxadiazepin-3(2*H*)-one gave 4,6-diphenylpyrimidin-2(1*H*)-one showing that ring contraction had occurred (Lassalvy, Petrus & Petrus, 1981). As part of our research on carboxamide oxime chemistry [see, *e.g.*, Kočevar, Polanc, Sollner, Tišler & Verček (1988) and Kočevar, Polanc, Sollner & Verček (1988)] we have heated carboxamide oximes in organic solvents with C_3 synthon in the presence of the appropriate acid catalyst. In general, one could expect the formation of 1,2,4-oxadiazepine or a pyrimidine ring. In order to support the pyrimidine *N*-oxide type structure of the isolated products we have performed X-ray analysis of crystals prepared from benzamide oxime and 1,1,3,3-tetramethoxypropane.

The financial support of the Ministry of Science and Technology, Slovenia, is gratefully acknowledged.

References

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

Kočevar, M., Polanc, S., Sollner, M., Tišler, M. & Verček, B. (1988). *Synth. Commun.* **18**, 1427–1432.

Kočevar, M., Polanc, S., Sollner, M. & Verček, B. (1988). *J. Serb. Chem. Soc.* **53**, 75–78.

Lassalvy, C., Petrus, C. & Petrus, F. (1981). *Gazz. Chim. Ital.* **111**, 273–278.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

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

Shell Research Ltd (1963). Belgian Patent 623714.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, selected torsion angles and the results of mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55314 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1001]

Acta Cryst. (1992). C**48**, 2229–2231

Structure of Tris(5-acetyl-3-thienyl)methane–Ethanol (2/1) Inclusion Compound

By Jan L. M. Dillen and H. Marita Roos

Department of Chemistry, University of Pretoria, Pretoria 0002, *South Africa*

(*Received* 27 *December* 1991; *accepted* 13 *March* 1992)

Abstract. $2C_{19}H_{16}O_3S_3.C_2H_6O$, $M_r = 823.1$, triclinic, $P1$, $a = 12.488$ (5), $b = 10.372$ (5), $c = 8.335$ (5) Å, $\alpha = 84.95$ (5), $\beta = 108.74$ (5), $\gamma = 98.51$ (5)°, $V = 1010$ (1) Å^3, $Z = 1$, $D_x = 1.35$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 3.34$ cm^{-1}, $F(000) = 430$, $T = 298$ K, final $wR = 0.065$, $R = 0.097$ for 3786 observed reflections with $F > \sigma(F_o)$ and 483 variable parameters. The structure consists of two tris(5-acetyl-3-thienyl)methane host molecules and one ethanol guest molecule.

Experimental. The host molecule, tris(5-acetyl-3-thienyl)methane (TATM), is formed by the reaction of chloroform and 2-acetylthiophene (Yakubov, Sudarushkin, Belenki & Gold'Farb, 1973) and crystallization with many solvents yields 2/1 solute–solvent complexes (Bin Din & Meth-Cohn, 1977). Crystallization of TATM with ethanol resulted in suitable crystals for diffraction with approximate dimensions of 0.3 × 0.3 × 0.3 mm; m.p. 381–387 K. A Philips PW1100 diffractometer, with graphite-monochromated Mo radiation, was used for data collection. Unit-cell parameters were determined from 25 reflections in the range $12 \leq \theta \leq 15°$. 4387 reflections were measured for $3 \leq \theta \leq 28°$ in the range $-15 \leq h \leq 15$, $-13 \leq k \leq 13$, $0 \leq l \leq 10$, using $\omega/2\theta$ scans with scan speed 0.072° s^{-1} and scan width 1.8°. Three standard reflections were measured

0108-2701/92/122229-03$06.00