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.

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* 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]

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# 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$^{-3}$, $\lambda$(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

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Table 1. *Fractional coordinates* ( × $10^4$) *and equivalent isotropic thermal factors* ($\AA^2 \times 10^3$)

$U_{iso}$ for atoms C(39), C(40) and O(7); remaining atoms were refined anisotropically; $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C(1) | 1775 | 1739 | 10855 | 36 (1) |
| S(1) | 192 (7) | 4502 (7) | 7768 (8) | 46 (1) |
| C(2) | 1293 (11) | 3695 (13) | 8947 (15) | 32 (1) |
| C(3) | 1017 (11) | 2718 (13) | 9817 (17) | 37 (1) |
| C(4) | −223 (11) | 2483 (13) | 9632 (16) | 36 (1) |
| C(5) | −756 (12) | 3472 (14) | 8515 (14) | 33 (1) |
| C(6) | −1955 (12) | 3593 (14) | 8019 (18) | 42 (1) |
| O(1) | −2292 (10) | 4526 (11) | 6909 (13) | 56 (1) |
| C(7) | −2736 (13) | 2754 (13) | 8868 (19) | 70 (2) |
| S(2) | 4895 (7) | 1991 (8) | 10487 (9) | 67 (1) |
| C(8) | 3480 (13) | 1296 (16) | 10242 (18) | 56 (1) |
| C(9) | 2947 (13) | 2034 (13) | 10876 (15) | 33 (1) |
| C(10) | 3643 (12) | 3187 (15) | 11762 (17) | 49 (1) |
| C(11) | 4757 (16) | 3298 (17) | 11610 (18) | 66 (2) |
| C(12) | 5821 (14) | 4304 (14) | 12077 (19) | 74 (2) |
| O(2) | 6654 (11) | 4283 (13) | 11717 (16) | 80 (1) |
| C(13) | 5741 (13) | 5298 (16) | 13107 (19) | 78 (2) |
| S(3) | 1543 (7) | 1777 (7) | 15623 (8) | 47 (1) |
| C(14) | 1553 (14) | 2332 (15) | 13621 (18) | 62 (2) |
| C(15) | 1651 (12) | 1404 (12) | 12661 (15) | 32 (1) |
| C(16) | 1546 (12) | 216 (13) | 13467 (16) | 36 (1) |
| C(17) | 1573 (12) | 188 (14) | 15066 (17) | 40 (1) |
| C(18) | 1494 (12) | −944 (15) | 16310 (15) | 56 (1) |
| O(3) | 1491 (12) | −556 (12) | 17639 (13) | 73 (1) |
| C(19) | 1394 (13) | −2248 (13) | 15720 (18) | 80 (2) |
| C(20) | −1699 (12) | −1590 (13) | −10910 (17) | 41 (1) |
| S(4) | −155 (7) | −4519 (8) | −7729 (9) | 52 (1) |
| C(21) | −1312 (14) | −3707 (15) | −8776 (17) | 54 (1) |
| C(22) | −913 (12) | −2591 (13) | −9843 (15) | 35 (1) |
| C(23) | 185 (13) | −2595 (14) | −9679 (16) | 45 (1) |
| C(24) | 765 (13) | −3453 (14) | −8607 (18) | 42 (1) |
| C(25) | 1961 (15) | −3723 (14) | −8052 (18) | 51 (1) |
| O(4) | 2334 (11) | −4525 (12) | −7113 (15) | 76 (1) |
| C(26) | 2689 (12) | −2797 (17) | −8689 (18) | 84 (2) |
| S(5) | −4877 (8) | −1979 (8) | −10485 (10) | 73 (1) |
| C(27) | −3549 (13) | −1357 (15) | −10245 (18) | 57 (1) |
| C(28) | −2985 (13) | −2045 (16) | −11031 (18) | 56 (2) |
| C(29) | −3723 (12) | −3152 (13) | −11656 (16) | 39 (1) |
| C(30) | −4744 (11) | −3248 (14) | −11528 (16) | 39 (1) |
| C(31) | −5683 (10) | −4252 (15) | −12135 (16) | 53 (1) |
| O(5) | −6503 (12) | −4251 (16) | −11755 (18) | 107 (2) |
| C(32) | −5552 (12) | −5386 (14) | −13192 (18) | 63 (2) |
| S(6) | −1554 (8) | −1779 (8) | −15605 (9) | 64 (1) |
| C(33) | −1652 (11) | −2432 (13) | −13719 (15) | 36 (1) |
| C(34) | −1638 (12) | −1488 (14) | −12696 (17) | 51 (1) |
| C(35) | −1650 (12) | −161 (15) | −13486 (16) | 42 (1) |
| C(36) | −1509 (13) | −221 (15) | −15128 (18) | 44 (1) |
| C(37) | −1492 (13) | 798 (12) | −16426 (18) | 54 (1) |
| O(6) | −1411 (11) | 686 (11) | −17851 (13) | 63 (1) |
| C(38) | −1653 (12) | 2162 (12) | −15990 (14) | 69 (1) |
| C(39) | 5725 (16) | 9274 (21) | 4074 (27) | 275 (4) |
| C(40) | 4917 (20) | 9765 (18) | 4840 (33) | 275 (4) |
| O(7) | 4068 (14) | 8676 (17) | 4836 (21) | 275 (4) |

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

| | | | |
|---|---|---|---|
| C(1)—C(3) | 1.51 (2) | C(1)—C(9) | 1.45 (2) |
| C(1)—C(15) | 1.57 (2) | S(1)—C(2) | 1.699 (11) |
| S(1)—C(5) | 1.707 (14) | C(2)—C(3) | 1.26 (2) |
| C(3)—C(4) | 1.49 (2) | C(4)—C(5) | 1.41 (2) |
| C(5)—C(6) | 1.44 (2) | C(6)—O(1) | 1.30 (2) |
| C(6)—C(7) | 1.51 (2) | S(2)—C(8) | 1.761 (14) |
| S(2)—C(11) | 1.77 (2) | C(8)—C(9) | 1.33 (2) |
| C(9)—C(10) | 1.47 (2) | C(10)—C(11) | 1.42 (2) |
| C(11)—C(12) | 1.53 (2) | C(12)—O(2) | 1.17 (2) |
| C(12)—C(13) | 1.43 (2) | S(3)—C(14) | 1.720 (14) |
| S(3)—C(17) | 1.76 (2) | C(14)—C(15) | 1.35 (2) |
| C(15)—C(16) | 1.36 (2) | C(16)—C(17) | 1.32 (2) |
| C(17)—C(18) | 1.51 (2) | C(18)—O(3) | 1.21 (2) |
| C(18)—C(19) | 1.45 (2) | C(20)—C(22) | 1.55 (2) |
| C(20)—C(28) | 1.58 (2) | C(20)—C(34) | 1.51 (2) |
| S(4)—C(21) | 1.73 (2) | S(4)—C(24) | 1.761 (13) |
| C(21)—C(22) | 1.52 (2) | C(22)—C(23) | 1.34 (2) |
| C(23)—C(24) | 1.32 (2) | C(24)—C(25) | 1.48 (2) |
| C(25)—O(4) | 1.13 (2) | C(25)—C(26) | 1.41 (2) |
| S(5)—C(27) | 1.643 (14) | S(5)—C(30) | 1.696 (14) |
| C(27)—C(28) | 1.40 (2) | C(28)—C(29) | 1.39 (2) |
| C(29)—C(30) | 1.30 (2) | C(30)—C(31) | 1.44 (2) |
| C(31)—O(5) | 1.166 (14) | C(31)—C(32) | 1.59 (2) |
| S(6)—C(33) | 1.690 (12) | S(6)—C(36) | 1.686 (14) |
| C(33)—C(34) | 1.35 (2) | C(34)—C(35) | 1.47 (2) |
| C(35)—C(36) | 1.44 (2) | C(36)—C(37) | 1.45 (2) |
| C(37)—O(6) | 1.24 (2) | C(37)—C(38) | 1.55 (2) |
| C(39)—C(40) | 1.519 (10) | C(40)—O(7) | 1.429 (10) |

| | | | |
|---|---|---|---|
| C(3)—C(1)—C(9) | 113.7 (10) | C(3)—C(1)—C(15) | 113.9 (11) |
| C(9)—C(1)—C(15) | 113.2 (9) | C(2)—S(1)—C(5) | 91.8 (6) |
| S(1)—C(2)—C(3) | 114.7 (9) | C(1)—C(3)—C(2) | 127.2 (11) |
| C(1)—C(3)—C(4) | 118.6 (11) | C(2)—C(3)—C(4) | 114.0 (11) |
| C(3)—C(4)—C(5) | 107.8 (11) | S(1)—C(5)—C(4) | 111.7 (10) |
| S(1)—C(5)—C(6) | 123.4 (10) | C(4)—C(5)—C(6) | 124.9 (12) |
| C(5)—C(6)—O(1) | 115.7 (12) | C(5)—C(6)—C(7) | 121.3 (12) |
| O(1)—C(6)—C(7) | 122.8 (12) | C(8)—S(2)—C(11) | 91.7 (8) |
| S(2)—C(8)—C(9) | 111.5 (12) | C(1)—C(9)—C(8) | 124.9 (11) |
| C(1)—C(9)—C(10) | 118.8 (11) | C(8)—C(9)—C(10) | 116.2 (13) |
| C(9)—C(10)—C(11) | 109.3 (13) | S(2)—C(11)—C(10) | 111.1 (12) |
| S(2)—C(11)—C(12) | 113.1 (13) | C(10)—C(11)—C(12) | 136 (2) |
| C(11)—C(12)—O(2) | 126 (2) | C(11)—C(12)—C(13) | 113 (2) |
| O(2)—C(12)—C(13) | 121.1 (12) | C(14)—S(3)—C(17) | 88.7 (7) |
| S(3)—C(14)—C(15) | 114.1 (10) | C(1)—C(15)—C(14) | 121.7 (11) |
| C(1)—C(15)—C(16) | 128.9 (10) | C(14)—C(15)—C(16) | 109.3 (11) |
| C(15)—C(16)—C(17) | 117.7 (12) | S(3)—C(17)—C(16) | 109.2 (10) |
| S(3)—C(17)—C(18) | 119.4 (10) | C(16)—C(17)—C(18) | 131.0 (13) |
| C(17)—C(18)—O(3) | 110.4 (13) | C(17)—C(18)—C(19) | 117.2 (11) |
| O(3)—C(18)—C(19) | 132.3 (12) | C(22)—C(20)—C(28) | 111.3 (10) |
| C(22)—C(20)—C(34) | 109.2 (10) | C(28)—C(20)—C(34) | 107.0 (10) |
| C(21)—S(4)—C(24) | 92.9 (7) | S(4)—C(21)—C(22) | 107.5 (11) |
| C(20)—C(22)—C(21) | 123.0 (12) | C(20)—C(22)—C(23) | 126.6 (10) |
| C(21)—C(22)—C(23) | 110.4 (12) | C(22)—C(23)—C(24) | 119.3 (11) |
| S(4)—C(24)—C(23) | 109.8 (11) | S(4)—C(24)—C(25) | 115.4 (10) |
| C(23)—C(24)—C(25) | 134.8 (12) | C(24)—C(25)—O(4) | 126.4 (14) |
| C(24)—C(25)—C(26) | 113.8 (12) | O(4)—C(25)—C(26) | 120 (2) |
| C(27)—S(5)—C(30) | 91.2 (6) | S(5)—C(27)—C(28) | 114.1 (11) |
| C(20)—C(28)—C(27) | 120.8 (13) | C(20)—C(28)—C(29) | 132.2 (13) |
| C(27)—C(28)—C(29) | 106.6 (13) | C(28)—C(29)—C(30) | 116.9 (12) |
| S(5)—C(30)—C(29) | 110.9 (10) | S(5)—C(30)—C(31) | 120.9 (9) |
| C(29)—C(30)—C(31) | 128.3 (13) | C(30)—C(31)—O(5) | 118.8 (14) |
| C(30)—C(31)—C(32) | 119.6 (9) | O(5)—C(31)—C(32) | 121.5 (14) |
| C(33)—S(6)—C(36) | 95.1 (7) | S(6)—C(33)—C(34) | 110.7 (10) |
| C(20)—C(34)—C(33) | 130.1 (11) | C(20)—C(34)—C(35) | 114.8 (12) |
| C(33)—C(34)—C(35) | 115.0 (12) | C(34)—C(35)—C(36) | 107.7 (13) |
| S(6)—C(36)—C(35) | 111.0 (11) | S(6)—C(36)—C(37) | 118.5 (10) |
| C(35)—C(36)—C(37) | 129.9 (13) | C(36)—C(37)—O(6) | 127.4 (12) |
| C(36)—C(37)—C(38) | 115.2 (12) | O(6)—C(37)—C(38) | 117.3 (11) |
| C(39)—C(40)—O(7) | 104.9 (9) | | |

every hour (no decay). Lorentz–polarization corrections, but no correction for absorption, were applied.

The structure was solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement on $F$ was performed using *SHELX*76 (Sheldrick, 1976), with $\sigma^{-2}(F)$ weights. All TATM non-H atoms were refined anisotropically, all solvent atoms with a common isotropic thermal parameter that refined to $U_{iso} = 0.275\ (4)\ \AA^2$, and all H atoms of TATM isotropically in calculated positions (C—H = 1.08 Å, H—C—H = 109.4°) with a common thermal parameter that converged to $U_{iso}(H) = 0.117\ (4)\ \AA^2$. 483 variables were refined, and the function $\sum w|\Delta F|^2$ minimized. Final $wR = 0.065$, $R = 0.097$, $(\Delta/\sigma)_{max} < 1$, $(\Delta\rho)_{max} = 0.83$, $(\Delta\rho)_{min} = -0.38\ e\ \AA^{-3}$. The ethanol molecule was refined in a fixed mode by constraining the C—C bond distance to 1.53 (1) Å, the C—O bond distance to 1.42 (1) Å, and the non-bonded distance to 2.41 (1) Å. The restrictions in the least-squares procedure led to an unusually high $R$ factor and standard deviations. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 gives the atom parameters, Table 2 bond lengths and angles, and Fig. 1 shows the molecular structure and atom-numbering scheme

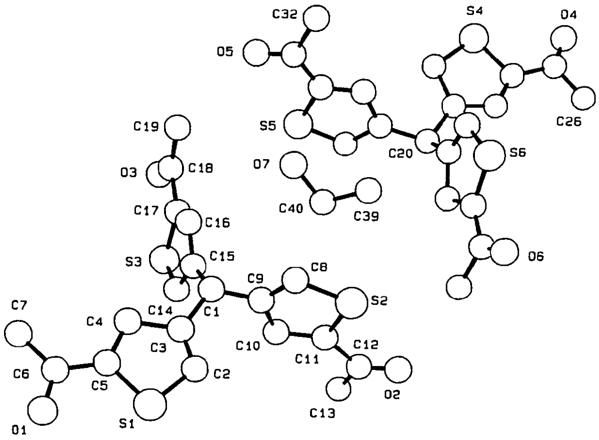

Fig. 1. Perspective drawing showing the numbering scheme.

drawn by *ORTEP* (Johnson, 1965).* The clathrate crystallizes in the common TATM–solvent ratio of 2/1.

**Related literature.** Related clathrates are inclusion compounds of TATM with ethyl acetate (van Rooyen & Roos, 1991*a*), benzene (van Rooyen & Roos, 1991*b*) and hexane (Roos & Dillen, 1992).

The authors wish to thank Dr Meth-Cohn for the title compound.

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* Lists of structure factors, bond lengths and angles involving H atoms, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55295 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0310]

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# 2-Hydroxy-4,4-dimethyl-2-(4-tolyl)-morpholinium Bromide

By J. Gabriel Garcia, Frank R. Fronczek and Richard D. Gandour*

*Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana* 70803-1804, *USA*

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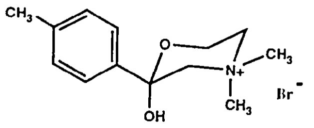
**Abstract.** $C_{13}H_{20}NO_2^+.Br^-$, $M_r = 302.2$, monoclinic, $P2_1/c$, $a = 8.697$ (2), $b = 12.741$ (3), $c = 12.940$ (2) Å, $\beta = 103.39$ (2)°, $V = 1394.9$ (8) Å$^3$, $Z = 4$, $D_x = 1.439$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71073 Å, $\mu = 29.1$ cm$^{-1}$, $F(000) = 624$, $T = 296$ K, $R = 0.038$ for 1662 observations with $I > 2\sigma(I)$ (of 2460 unique data). The morpholinium ring adopts the chair conformation with endocyclic torsion angle magnitudes 49.1 (4)–61.9 (4)°. The hydroxyl group is in the axial position of the morpholinium ring, with C—OH bond distance 1.401 (4) Å. The hydroxy H atom points towards a Br ion; the interaction has O···Br distance 3.292 (2) Å, H···Br distance 2.61 (3) Å, and angle at H 160 (4)°.

**Experimental.** The title compound (I) was prepared as described by Garcia (1986). Crystals were recrystallized from methanol and had m.p. 492–493 K. A



(I)

clear colorless crystal with dimensions 0.28 × 0.32 × 0.36 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections

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* Author to whom correspondence should be addressed.

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