

Table 2. Bond lengths (Å), valency angles (°) and selected torsional angles (°)

N(01)—C(02)	1.453 (5)	C(2)—O(2)	1.237 (5)
C(02)—C(03)	1.496 (5)	C(2)—C(3)	1.490 (6)
C(03)—O(03)	1.232 (5)	C(3)—C(4)	1.307 (6)
C(03)—N(04)	1.327 (5)	C(4)—C(5)	1.470 (8)
N(04)—C(05)	1.448 (5)	C(5)—O(5)	1.201 (10)
C(05)—C(06)	1.517 (6)	C(5)—O(6)	1.317 (7)
C(06)—N(01)	1.463 (5)	O(6)—C(7)	1.452 (7)
N(01)—C(2)	1.357 (4)		
C(02)—N(01)—C(06)	116.2 (3)	O(2)—C(2)—N(01)	120.5 (4)
C(02)—N(01)—C(2)	115.5 (3)	C(3)—C(2)—N(01)	119.0 (4)
C(06)—N(01)—C(2)	126.1 (3)	O(2)—C(2)—C(3)	120.5 (4)
N(01)—C(02)—C(03)	117.0 (3)	C(2)—C(3)—C(4)	120.5 (4)
C(02)—C(03)—N(04)	118.4 (3)	C(3)—C(4)—C(5)	122.7 (5)
C(02)—C(03)—O(03)	118.3 (4)	C(4)—C(5)—O(5)	124.4 (5)
O(03)—C(03)—N(04)	123.3 (4)	C(4)—C(5)—O(6)	111.3 (6)
C(03)—N(04)—C(05)	123.8 (3)	O(5)—C(5)—O(6)	124.3 (5)
N(04)—C(05)—C(06)	110.7 (4)	C(5)—O(6)—C(7)	117.2 (5)
C(05)—C(06)—N(01)	108.0 (3)		
C(02)—N(01)—C(2)—O(2)	7.6 (5)		
C(02)—N(01)—C(2)—C(3)	-170.2 (4)		
C(06)—N(01)—C(2)—O(2)	170.3 (4)		
C(06)—N(01)—C(2)—C(3)	-7.6 (6)		
N(01)—C(2)—C(3)—C(4)	168.4 (4)		
O(2)—C(2)—C(3)—C(4)	-9.4 (7)		
C(2)—C(3)—C(4)—C(5)	-175.1 (5)		
C(3)—C(4)—C(5)—O(5)	6.9 (9)		
C(3)—C(4)—C(5)—O(6)	-175.7 (5)		
C(4)—C(5)—O(6)—C(7)	-173.1 (5)		
O(5)—C(5)—O(6)—C(7)	4.3 (9)		

Andrzejewska-Golec, Broda & Najman, 1977; Graczyk, Pakulska, Groszkowski & Najman, 1980; Groszkowski & Najman, 1986, and references therein). Some showed promising activity in mice. The —CH=CH—C=O fragment which is also present in butenoates is supposed to be responsible for the cytostatic activity of acrylates (Lee, Kim, Piantadosi, Huang & Geissman, 1974; Loeffler, Sajadi & Hall, 1977). Therefore we started an X-ray study on the 4-oxo-2-butenate moiety (Głównka & Iwanicka, 1990; Głównka, Iwanicka & Najman, 1991; Głównka, 1991).

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Structure of Tris(5-acetyl-3-thienyl)methane:Ethyl Acetate Inclusion Compound

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(Received 12 February 1990; accepted 9 May 1991)

Abstract. C₄₂H₄₀O₈S₆, $M_r = 865.1$, triclinic, $P1$, $a = 12.329(5)$, $b = 11.229(5)$, $c = 8.229(5)$ Å, $\alpha = 98.42(5)^\circ$, $\beta = 106.43(5)^\circ$, $\gamma = 99.05(5)^\circ$, $U = 1057(1)$ Å³, $Z = 1$, $D_x = 1.36$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 3.23$ cm⁻¹, $F(000) = 452$, $T =$

The bond lengths and angles in the present structure agree with the previous results to within 3σ limits. In the crystal, the molecules are linked in infinite chains by means of intermolecular N(04)—H \cdots O(03) [$2 - x, y - \frac{1}{2}, \frac{5}{2} - z$] hydrogen bonds with N \cdots O and H \cdots O distances of 2.872(4) and 2.04(2) Å, respectively and an N—H \cdots O angle of 168(3)°. The chains run along the 2₁ axis.

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Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal factors ($\times 10^3 \text{ \AA}^2$) for the non-H atoms in TATM:EtOAc
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
C(1)	9780	6187	7652	52 (1)
S(1)	9721 (2)	6213 (2)	2670 (2)	49 (1)
C(2)	9692 (5)	6865 (5)	4621 (7)	40 (1)
C(3)	9719 (4)	6072 (5)	5724 (7)	39 (1)
C(4)	9732 (5)	4856 (5)	4941 (7)	43 (1)
C(5)	9716 (5)	4815 (5)	3266 (7)	50 (1)
C(6)	9722 (5)	3710 (5)	1994 (7)	47 (1)
O(1)	9818 (4)	3829 (4)	576 (6)	70 (1)
C(7)	9816 (7)	2520 (6)	2619 (9)	76 (1)
S(2)	8330 (1)	9171 (1)	9281 (2)	48 (1)
C(8)	9433 (5)	8280 (6)	9306 (8)	54 (1)
C(9)	9104 (5)	7300 (5)	7977 (6)	40 (1)
C(10)	7969 (5)	7285 (5)	6847 (7)	48 (1)
C(11)	7395 (6)	8209 (5)	7407 (7)	51 (1)
C(12)	6306 (5)	8458 (5)	6600 (7)	40 (1)
O(2)	5953 (4)	9335 (4)	7283 (6)	66 (1)
C(13)	5570 (5)	7650 (6)	4963 (8)	65 (1)
S(3)	13020 (2)	6696 (2)	10762 (3)	71 (1)
C(14)	11554 (5)	6046 (6)	9747 (8)	52 (1)
C(15)	11033 (5)	6779 (5)	8723 (7)	38 (1)
C(16)	11810 (5)	7836 (5)	8764 (7)	45 (1)
C(17)	12913 (5)	7938 (6)	9764 (7)	51 (1)
C(18)	13989 (5)	8909 (6)	10118 (8)	53 (1)
O(3)	14866 (4)	8822 (5)	11229 (7)	94 (1)
C(19)	13933 (6)	9944 (6)	9287 (9)	66 (1)
C(20)	16689 (4)	12658 (4)	15744 (6)	19 (1)
S(4)	18085 (2)	10015 (2)	14092 (2)	56 (1)
C(21)	7106 (5)	10797 (5)	4055 (7)	43 (1)
C(22)	17413 (5)	11789 (5)	15496 (7)	41 (1)
C(23)	18539 (4)	11863 (5)	16556 (6)	35 (1)
C(24)	18997 (4)	10957 (5)	16617 (6)	31 (1)
C(25)	10154 (6)	10685 (6)	6809 (8)	59 (1)
O(4)	10477 (4)	9853 (4)	16154 (6)	77 (1)
C(26)	10911 (6)	11513 (7)	8552 (8)	77 (1)
S(5)	16743 (2)	12980 (2)	20736 (2)	52 (1)
C(27)	16735 (5)	12285 (6)	18679 (7)	53 (1)
C(28)	16771 (5)	13110 (5)	17629 (7)	43 (1)
C(29)	16780 (5)	14321 (5)	18487 (7)	44 (1)
C(30)	16750 (4)	14402 (5)	20161 (7)	36 (1)
C(31)	16682 (5)	15428 (6)	21412 (8)	60 (1)
O(5)	16702 (4)	15284 (4)	22849 (6)	68 (1)
C(32)	16749 (6)	16646 (5)	20877 (8)	73 (1)
S(6)	13436 (2)	12467 (2)	12615 (2)	65 (1)
C(33)	14854 (6)	13060 (6)	13578 (8)	59 (1)
C(34)	15415 (5)	12381 (5)	14631 (7)	46 (1)
C(35)	14638 (5)	11278 (6)	14675 (7)	49 (1)
C(36)	13520 (5)	11214 (5)	13597 (7)	47 (1)
C(37)	12493 (6)	10266 (6)	13199 (8)	60 (1)
O(6)	11583 (4)	10352 (5)	12227 (6)	82 (1)
C(38)	12574 (6)	9195 (6)	14193 (9)	78 (1)
C(39)	3716 (8)	3340 (10)	8404 (12)	144 (1)
C(40)	3022 (9)	3746 (10)	6835 (10)	190 (1)
O(7)	1939 (5)	3544 (7)	6356 (9)	149 (1)
O(8)	3697 (7)	4817 (9)	6646 (11)	190 (1)
C(41)	3103 (10)	5090 (9)	5002 (12)	204 (1)
C(42)	2818 (9)	6225 (9)	5729 (12)	157 (1)

enclathrated by two of the three acetylthienyl substituents of each host molecule. The shortest non-hydrogen non-bonded distance between host and guest molecules is 3.416 Å [C(26)⋯O(7)].

Experimental. The host molecule, tris(5-acetyl-3-thienyl)methane (TATM), is formed by the reaction of chloroform and 2-acetylthiophene (Yakubov, Sudarushkin, Belenkii & Gold'farb, 1973), and crystallization with many solvents yields 2:1 solute:solvent complexes (Bin Din & Meth-Cohn, 1977). Crystallization of TATM with ethyl acetate yielded pale-yellow cubic crystals with approximate dimensions 0.3 × 0.3 × 0.3 mm, m.p. 373–374 K.

Table 2. Bond lengths (Å) and valence angles (°) for the non-H atoms in TATM:EtOAc

C(1)—C(3)	1.552 (6)	C(1)—C(9)	1.638 (6)
C(1)—C(15)	1.529 (5)	S(1)—C(2)	1.677 (6)
S(1)—C(5)	1.712 (7)	C(2)—C(3)	1.359 (8)
C(3)—C(4)	1.426 (8)	C(4)—C(5)	1.367 (9)
C(5)—C(6)	1.503 (8)	C(6)—O(1)	1.230 (8)
C(6)—C(7)	1.510 (9)	S(2)—C(8)	1.807 (7)
S(2)—C(11)	1.729 (5)	C(8)—C(9)	1.352 (7)
C(9)—C(10)	1.441 (8)	C(10)—C(11)	1.437 (10)
C(11)—C(12)	1.410 (9)	C(12)—O(2)	1.260 (8)
C(12)—C(13)	1.467 (7)	S(3)—C(14)	1.747 (6)
S(3)—C(17)	1.723 (7)	C(14)—C(15)	1.366 (9)
C(15)—C(16)	1.393 (8)	C(16)—C(17)	1.353 (8)
C(17)—C(18)	1.506 (8)	C(18)—O(3)	1.231 (7)
C(18)—C(19)	1.434 (10)	C(20)—C(22)	1.453 (8)
C(20)—C(28)	1.529 (7)	C(20)—C(34)	1.533 (7)
S(4)—C(21)	1.597 (6)	C(21)—C(24)	1.738 (4)
C(21)—C(22)	1.420 (7)	C(22)—C(23)	1.397 (7)
C(23)—C(24)	1.319 (8)	C(24)—C(25)	1.490 (8)
C(25)—O(4)	1.189 (9)	C(25)—C(26)	1.541 (8)
S(5)—C(27)	1.754 (6)	S(5)—C(30)	1.730 (6)
C(27)—C(28)	1.360 (9)	C(28)—C(29)	1.434 (8)
C(29)—C(30)	1.378 (9)	C(30)—C(31)	1.458 (9)
C(31)—O(5)	1.211 (9)	C(31)—C(32)	1.495 (10)
S(6)—C(33)	1.681 (6)	S(6)—C(36)	1.725 (7)
C(33)—C(34)	1.353 (9)	C(34)—C(35)	1.454 (8)
C(35)—C(36)	1.397 (8)	C(36)—C(37)	1.444 (9)
C(37)—O(6)	1.207 (8)	C(37)—C(38)	1.551 (11)
C(39)—C(40)	1.509 (13)	C(40)—O(7)	1.253 (12)
C(40)—O(8)	1.406 (14)	O(8)—C(41)	1.446 (13)
C(41)—C(42)	1.46 (2)		
C(3)—C(1)—C(9)	103.6 (3)	C(3)—C(1)—C(15)	106.6 (3)
C(9)—C(1)—C(15)	102.3 (3)	C(2)—S(1)—C(5)	90.9 (3)
S(1)—C(2)—C(3)	113.3 (4)	C(1)—C(3)—C(2)	135.0 (5)
C(1)—C(3)—C(4)	112.5 (4)	C(2)—C(3)—C(4)	112.4 (5)
C(3)—C(4)—C(5)	110.1 (5)	S(1)—C(5)—C(4)	113.2 (4)
S(1)—C(5)—C(6)	119.9 (5)	C(4)—C(5)—C(6)	126.9 (6)
C(5)—C(6)—O(1)	120.0 (5)	C(5)—C(6)—C(7)	118.0 (5)
O(1)—C(6)—C(7)	121.1 (6)	C(8)—S(2)—C(11)	91.7 (3)
S(2)—C(8)—C(9)	113.3 (4)	C(1)—C(9)—C(8)	128.5 (4)
C(1)—C(9)—C(10)	121.4 (4)	C(8)—C(9)—C(10)	110.0 (5)
C(9)—C(10)—C(11)	116.1 (4)	S(2)—C(11)—C(10)	108.7 (4)
S(2)—C(11)—C(12)	120.8 (5)	C(10)—C(11)—C(12)	130.2 (5)
C(11)—C(12)—O(2)	121.1 (4)	C(11)—C(12)—C(13)	118.7 (5)
O(2)—C(12)—C(13)	120.2 (5)	C(14)—S(3)—C(17)	91.6 (3)
S(3)—C(14)—C(15)	110.9 (4)	C(1)—C(15)—C(14)	113.0 (4)
C(1)—C(15)—C(16)	134.8 (5)	C(14)—C(15)—C(16)	111.8 (5)
C(15)—C(16)—C(17)	115.7 (6)	S(3)—C(17)—C(16)	110.1 (5)
C(16)—C(17)—C(18)	118.3 (4)	C(16)—C(17)—C(18)	131.7 (6)
C(17)—C(18)—O(3)	117.5 (6)	C(17)—C(18)—C(19)	119.7 (5)
O(3)—C(18)—C(19)	122.6 (5)	C(22)—C(20)—C(28)	114.7 (4)
C(22)—C(20)—C(34)	119.8 (4)	C(28)—C(20)—C(34)	108.8 (5)
C(21)—S(4)—C(24)	91.6 (3)	S(4)—C(21)—C(22)	113.5 (4)
C(20)—C(22)—C(21)	124.4 (4)	C(20)—C(22)—C(23)	125.1 (4)
C(21)—C(22)—C(23)	110.3 (5)	C(22)—C(23)—C(24)	112.2 (4)
S(4)—C(24)—C(23)	112.1 (4)	S(4)—C(24)—C(25)	118.9 (4)
C(23)—C(24)—C(25)	128.9 (4)	C(24)—C(25)—O(4)	122.1 (5)
C(24)—C(25)—C(26)	117.0 (6)	O(4)—C(25)—C(26)	120.9 (6)
C(27)—S(5)—C(30)	91.8 (3)	S(5)—C(27)—C(28)	112.1 (5)
C(20)—C(28)—C(27)	119.5 (5)	C(20)—C(28)—C(29)	128.9 (5)
C(27)—C(28)—C(29)	111.3 (5)	C(28)—C(29)—C(30)	114.6 (5)
S(5)—C(30)—C(29)	110.2 (4)	S(5)—C(30)—C(31)	118.3 (5)
C(29)—C(30)—C(31)	131.5 (6)	C(30)—C(31)—O(5)	119.5 (6)
C(30)—C(31)—C(32)	115.9 (6)	O(5)—C(31)—C(32)	124.1 (6)
C(33)—S(6)—C(36)	91.5 (3)	S(6)—C(33)—C(34)	114.6 (5)
C(20)—C(34)—C(33)	128.6 (5)	C(20)—C(34)—C(35)	119.8 (5)
C(33)—C(34)—C(35)	111.6 (5)	C(34)—C(35)—C(36)	110.5 (5)
S(6)—C(36)—C(37)	111.8 (4)	S(6)—C(36)—C(37)	118.7 (5)
C(35)—C(36)—C(37)	129.4 (6)	C(36)—C(37)—O(6)	121.1 (6)
C(36)—C(37)—C(38)	117.5 (5)	O(6)—C(37)—C(38)	121.1 (7)
C(39)—C(40)—O(7)	122.5 (9)	C(39)—C(40)—O(8)	107.6 (7)
O(7)—C(40)—O(8)	122.3 (10)	C(40)—O(8)—C(41)	107.2 (7)
O(8)—C(41)—C(42)	95.9 (8)		

Philips PW1100 diffractometer, graphite-mono-chromatized radiation, unit cell from 25 reflections ($\theta < 13^\circ$), 4336 reflections for $3 \leq \theta \leq 23^\circ$ in the range $-15 \leq h \leq 15$, $-14 \leq k \leq 14$, $0 \leq l \leq 9$ using $\omega/2\theta$ scans, scan speed $0.053^\circ \text{ s}^{-1}$, scan width 1.60° . Three standard reflections (012, $\bar{1}\bar{4}\bar{3}$, $\bar{4}34$) were measured every hour, no decay or absorption corrections

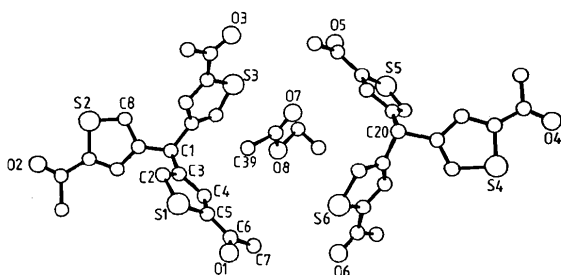


Fig. 1. Perspective view with atomic numbering scheme.

applied, Lorentz-polarization correction. Structure solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement using *SHELX76* (Sheldrick, 1976), F magnitudes, $\sigma^{-2}(F)$ weights, all non-H atoms anisotropic, all thiophene H atoms in experimental positions and all other H atoms isotropic in calculated positions (C—H = 1.08 Å, H—C—H = 109.4°) with a common thermal parameter that was also refined [$U_{\text{iso}}(\text{H}) = 0.134(2) \text{ \AA}^2$], 625 variables refined, $\sum w|\Delta F|^2$ minimized. Final $wR = 0.056$, $R = 0.084$, $(\Delta/\sigma)_{\text{max}} = 0.60$, maximum residual electron density = 0.61 e Å⁻³. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 gives the atom parameters,

and Fig. 1 shows the molecular structure and atomic numbering scheme drawn by *ORTEP* (Johnson, 1965).* Bond lengths and angles involving non-H atoms are given in Table 2.

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

* 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 54245 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (Z)-1,2-Dimesitylvinyene Bis(mesitylenecarboxylate)

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Abstract. C₄₀H₄₄O₄, $M_r = 588.79$, monoclinic, $P2_1$, $a = 8.436(2)$, $b = 16.739(6)$, $c = 11.698(3) \text{ \AA}$, $\beta = 97.69(2)^\circ$, $V = 1637.0(8) \text{ \AA}^3$, $Z = 2$, $D_x = 1.194 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$, $\mu = 0.7 \text{ cm}^{-1}$, $F(000) = 632$, $T = 100 \text{ K}$, $R = 0.039$ for 3544 unique reflections with $I \geq 2.5\sigma(I)$. The molecule has approximate twofold symmetry; torsion angles in the two parts differ by up to 12°.

Experimental. The title compound was prepared by Dr W. J. Muizebelt (Akzo Research) by reduction of mesitoyl chloride with lithium amalgam (Horner & Dickerhof, 1983). Crystals were formed on crystallization from dichloromethane/hexane (m.p. 522.1–

522.4 K). X-ray data for a colourless crystal (0.25 × 0.4 × 0.4 mm), glued on top of a glass fibre, were collected at liquid-nitrogen temperature on an Enraf-Nonius CAD-4F diffractometer using Zr-filtered Mo $K\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the setting angles of 25 reflections with $14 < \theta < 20^\circ$. The probability distribution of the normalized structure factors ($\langle |E|^2 - 1 \rangle = 0.782$); together with the extinctions the space group was determined to be $P2_1$. No other space group is compatible with the structure found; this was confirmed by the method of Le Page (1988). 4536 reflections [$\theta < 27.5^\circ$, $\omega/2\theta$ scan; $\Delta\omega = (0.75 +$