$C_{42}H_{40}O_8S_6$

Fig. 1. Perspective view with atomic numbering scheme.

applied, Lorentz–polarization correction. Structure solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement using *SHELX*76 (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 [$U_{iso}(H) = 0·134$ (2) Å^2], 625 variables refined, $\sum w|\Delta F|^2$ minimized. Final wR = 0·056, $R = 0·084$, $(\Delta/\sigma)_{max} = 0·60$, 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.

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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 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-Dimesitylvinylene Bis(mesitylenecarboxylate)

BY B. P. VAN EIJCK

Vakgroep Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3584 CH Utrecht, The Netherlands

Abstract. $C_{40}H_{44}O_4$, $M_r = 588·79$, monoclinic, $P2_1$, $a = 8·436$ (2), $b = 16·739$ (6), $c = 11·698$ (3) Å, $\beta = 97·69$ (2)°, $V = 1637·0$ (8) Å^3, $Z = 2$, $D_x = 1·194$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·7$ cm^{-1}, $F(000) = 632$, $T = 100$ 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 $14 < \theta < 20°$. The probability distribution of the normalized structure factors indicated a noncentrosymmetric space group ($\langle||E|^2 - 1|\rangle = 0·782$); 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 [$\theta < 27·5°$, $\omega/2\theta$ scan; $\Delta\omega = (0·75 +$

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0·35tanθ)°; $-10 \leq h \leq 10$, $0 \leq k \leq 21$, $0 \leq l \leq 15$] were scanned, of which 3871 were unique $(R_{int} = 0.050)$. Three reference reflections (220, 32$\bar{4}$, $\bar{3}$02) showed a decay of 7% during the 70 h of X-ray exposure time. The intensities were corrected for this decay and for Lp, but not for absorption. Variance values $\sigma^2(I)$ were calculated based on counting statistics plus a term $(PI)^2$, where P $(= 0.076)$ is the instability constant as derived from the excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). The 3544 reflections with $I \geq 2.5\sigma(I)$ were used in the subsequent analysis. The structure was solved by direct methods (SHELXS86; Sheldrick, 1990) and refined on F by weighted least-squares techniques with SHELX76 (Sheldrick, 1976) on a MicroVAX II. All non-H atoms were refined with anisotropic thermal parameters. The H atoms were located from a difference Fourier map and refined with two uniform isotropic temperature factors. Convergence was reached at $R = 0.039$ [$wR = 0.055$, $w = 1/\sigma^2(F)$, 530 parameters, $S = 0.50$, $(\Delta/\sigma)_{av} = 0.02$, $(\Delta/\sigma)_{max} = 0.17$]. The final $\Delta\rho$ map did not show features outside the range -0.21 to 0.30 e Å^{-3}. Fig. 1 shows the molecule with the adopted numbering. Atomic coordinates and equivalent isotropic temperature factors of the non-H atoms are given in Table 1. A selection of geometrical parameters is presented in Table 2.* Scattering factors of Cromer & Mann (1968) were used. Geometrical calculations and illustrations were made with the programs PLATON and PLUTON of the EUCLID package (Spek, 1982).

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* Lists of anisotropic thermal parameters, H-atom parameters, all geometrical parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54223 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
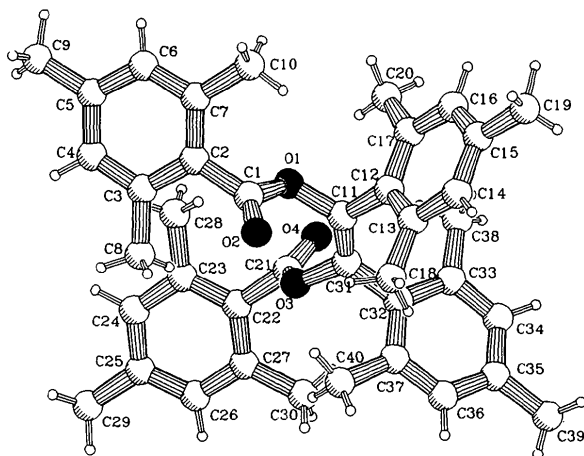

Fig. 1. View of the molecule with adopted numbering.

Table 1. *Final coordinates and equivalent isotropic thermal parameters of the non-H atoms with e.s.d.'s in parentheses*

$U_{eq} = 1/3$ of the trace of the orthogonalized U.

	x	y	z	U_{eq}(Å^2)
O(1)	0·0703 (2)	0·45	0·24831 (13)	0·0182 (4)
O(2)	0·3014 (2)	0·42243 (15)	0·17930 (16)	0·0251 (5)
O(3)	0·0865 (2)	0·30938 (14)	0·35555 (13)	0·0186 (4)
O(4)	−0·1259 (2)	0·34104 (18)	0·44470 (16)	0·0345 (6)
C(1)	0·2267 (3)	0·46419 (16)	0·23529 (18)	0·0154 (5)
C(2)	0·2881 (3)	0·53527 (16)	0·30417 (18)	0·0161 (6)
C(3)	0·4094 (3)	0·52264 (17)	0·39741 (19)	0·0181 (6)
C(4)	0·4605 (3)	0·58717 (18)	0·4670 (2)	0·0208 (6)
C(5)	0·3991 (3)	0·66390 (17)	0·4456 (2)	0·0201 (6)
C(6)	0·2839 (3)	0·67512 (17)	0·3499 (2)	0·0200 (6)
C(7)	0·2262 (3)	0·61201 (17)	0·27828 (19)	0·0190 (6)
C(8)	0·4799 (3)	0·44050 (18)	0·4241 (2)	0·0240 (7)
C(9)	0·4570 (3)	0·73270 (19)	0·5225 (3)	0·0268 (7)
C(10)	0·1045 (3)	0·62883 (19)	0·1744 (2)	0·0263 (7)
C(11)	−0·0027 (3)	0·38330 (16)	0·18799 (19)	0·0171 (6)
C(12)	−0·0789 (3)	0·39895 (17)	0·06797 (19)	0·0175 (6)
C(13)	−0·0273 (3)	0·35914 (17)	−0·02645 (19)	0·0188 (6)
C(14)	−0·1042 (3)	0·37576 (19)	−0·1370 (2)	0·0232 (7)
C(15)	−0·2276 (3)	0·43064 (19)	−0·1567 (2)	0·0269 (7)
C(16)	−0·2741 (3)	0·47038 (18)	−0·0628 (2)	0·0244 (7)
C(17)	−0·2028 (3)	0·45588 (17)	0·0496 (2)	0·0207 (6)
C(18)	0·1091 (3)	0·30085 (18)	−0·0142 (2)	0·0217 (6)
C(19)	−0·3043 (4)	0·4483 (2)	−0·2783 (2)	0·0358 (9)
C(20)	−0·2655 (3)	0·50006 (18)	0·1466 (2)	0·0255 (7)
C(21)	0·0091 (3)	0·31792 (17)	0·44919 (19)	0·0202 (6)
C(22)	0·1198 (3)	0·29609 (18)	0·55600 (19)	0·0199 (6)
C(23)	0·1931 (3)	0·35758 (18)	0·62548 (19)	0·0211 (6)
C(24)	0·3044 (3)	0·3364 (2)	0·7197 (2)	0·0260 (7)
C(25)	0·3408 (3)	0·2566 (2)	0·7471 (2)	0·0281 (7)
C(26)	0·2619 (3)	0·1979 (2)	0·6785 (2)	0·0276 (8)
C(27)	0·1517 (3)	0·21554 (18)	0·5829 (2)	0·0229 (7)
C(28)	0·1538 (4)	0·44395 (19)	0·6001 (2)	0·0284 (8)
C(29)	0·4618 (4)	0·2357 (3)	0·8498 (2)	0·0390 (9)
C(30)	0·0682 (4)	0·1488 (2)	0·5123 (3)	0·0298 (8)
C(31)	−0·0065 (3)	0·31507 (17)	0·24540 (17)	0·0168 (6)
C(32)	−0·0824 (3)	0·23882 (16)	0·20350 (17)	0·0163 (6)
C(33)	−0·2492 (3)	0·23173 (17)	0·17411 (18)	0·0172 (6)
C(34)	−0·3122 (3)	0·15837 (18)	0·1343 (2)	0·0192 (6)
C(35)	−0·2145 (3)	0·09275 (18)	0·1214 (2)	0·0201 (6)
C(36)	−0·0506 (3)	0·10059 (18)	0·1518 (2)	0·0205 (6)
C(37)	0·0180 (3)	0·17241 (17)	0·19300 (19)	0·0175 (6)
C(38)	−0·3626 (3)	0·30021 (18)	0·1843 (2)	0·0201 (6)
C(39)	−0·2854 (3)	0·0152 (2)	0·0740 (3)	0·0305 (8)
C(40)	0·1974 (3)	0·17745 (19)	0·2221 (2)	0·0240 (7)

Table 2. *Selected bond distances (Å), bond angles (°) and torsion angles (°)*

O(1)—C(1)	1·369 (3)	O(3)—C(21)	1·356 (3)
O(1)—C(11)	1·417 (3)	O(3)—C(31)	1·419 (3)
O(2)—C(1)	1·194 (3)	O(4)—C(21)	1·197 (3)
C(1)—C(2)	1·490 (4)	C(21)—C(22)	1·502 (3)
C(1)—O(1)—C(11)	116·3 (2)	C(21)—O(3)—C(31)	117·3 (2)
O(1)—C(1)—O(2)	123·1 (2)	O(3)—C(21)—O(4)	123·8 (2)
O(1)—C(1)—C(2)	110·4 (2)	O(3)—C(21)—C(22)	109·5 (2)
O(2)—C(1)—C(2)	126·4 (2)	O(4)—C(21)—C(22)	126·7 (2)
O(1)—C(11)—C(31)	117·7 (2)	O(3)—C(31)—C(11)	118·0 (2)
O(1)—C(11)—C(12)	115·8 (2)	O(3)—C(31)—C(32)	113·9 (2)
C(31)—C(11)—C(12)	126·3 (2)	C(11)—C(31)—C(32)	127·8 (2)
C(31)—C(11)—C(12)—C(13)	−65·5 (4)	C(11)—C(31)—C(32)—C(33)	−65·1 (3)
C(31)—C(11)—O(1)—C(1)	96·4 (3)	C(11)—C(31)—O(3)—C(21)	101·0 (3)
O(1)—C(1)—C(2)—C(7)	−65·2 (3)	O(3)—C(21)—C(22)—C(27)	−76·9 (3)
O(1)—C(11)—C(31)—O(3)	−11·0 (3)		

Related literature. For the chemical background of the title compound we refer to our recent publication on the (E) isomer (Kouwijzer, van Eijck & Muizebelt, 1991). Apart, of course, from the central C(11)—C(31) bond the structures of the two isomers are quite comparable, the largest difference in a torsion angle being 17°.

We thank Dr W. J. Muizebelt (Akzo Research Laboratories) for donating the crystals and Dr A. J. M. Duisenberg for collecting the data.

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Structure of 5-Chloro-2,4,6-trifluoroisophthalonitrile*

BY DOYLE BRITTON

Department of Chemistry, University of Minnesota, Minneapolis, MN 55455, USA

Abstract. $C_6ClF_3(CN)_2$, $M_r = 216.55$, monoclinic, $P2_1/c$, $a = 10.266$ (3), $b = 14.848$ (6), $c = 11.537$ (3) Å, $\beta = 108.83$ (3)°, $Z = 8$ (two molecules in the asymmetric unit), $V = 1664$ (2) Å^3, $D_x = 1.728$ (2) g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 4.60$ cm^{-1}, $F(000) = 848$, $T = 297$ (2) K, $R = 0.053$ for 2259 unique observed reflections with $I > \sigma(I)$. The bond lengths and angles are normal, except that one ring is slightly puckered and in both molecules about half of the substituents are bent out of the mean plane of the ring by 1.0 to 2.5°. Three short intermolecular distances can be seen in the packing.

Experimental. The compound was obtained from Dr Robert Battershell of the Diamond Shamrock Corp.; crystals suitable for X-ray diffraction were found in the original sample. A colorless crystal 0.10 × 0.25 × 0.60 mm, mounted in air, was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 22 reflections with $10 < \theta < 14°$ were used to determine the cell parameters. Systematic extinctions ($h0l$, l odd; $0k0$, k odd) uniquely determined the space group. Data were collected, using $\omega/2\theta$ scans, in the range $0 < \theta < 25°$ for all of one octant and half of a second (ranges: h, -12 to 4; k, 0 to 17; l, -13 to 13). The intensities of 4429 different reflections were measured. Three check reflections measured every 6000 s of exposure time showed a linear decrease in intensity of 45% over the

course of the data collection; a correction was made for this decay. After absorption corrections were made using the program *DIFABS* (Walker & Stuart, 1983; transmission factors 0.73 to 1.10), equivalent reflections were combined to give 2924 independent reflections ($R_{int} = 0.037$) of which the 2259 with $I > \sigma(I)$ were used in the calculations. The structure was solved by direct methods (*MITHRIL*, Gilmore, 1984) and refined with full-matrix least-squares refinement on F's. There are two molecules in the asymmetric unit. All atoms were given anisotropic thermal parameters. Refinement converged with $R = 0.053$, $wR = 0.056$, and $S = 1.22$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I)_c + (0.05I)^2$, where $\sigma(I)_c$ is the standard deviation in I based on counting statistics alone. In the final cycle of refinement $(\Delta/\sigma)_{max} = 0.03$, $(\Delta\rho)_{max} = 0.23$ and $(\Delta\rho)_{min} = -0.31$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The computer programs used were from *TEXSAN* (Molecular Structure Corporation, 1985). The final positional parameters are given in Table 1.†

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† Lists of anisotropic thermal parameters, complete bond angles, deviations from the mean planes of the benzene rings, intermolecular distances, observed and calculated structure factors, and a figure showing the thermal ellipsoids in molecule *B* have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54237 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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* 5-Chloro-2,4,6-trifluoro-1,3-benzenedicarbonitrile.