

Table 2. Interatomic distances (Å) and angles (°) with *e.s.d.'s in parentheses*

C(1)—O(1)	1.276 (1)	O(1)—H(1A)	0.85 (3)
C(1)—O(2)	1.255 (2)	O(2)—H(1B)	0.85 (4)
C(1)—C(2)	1.456 (2)	C(3)—H(2)	0.92 (2)
C(2)—C(3)	1.345 (2)	C(4)—H(3)	0.95 (2)
C(3)—C(4)	1.411 (2)	C(5)—H(4)	0.97 (2)
C(4)—C(5)	1.339 (2)	O(1)···O(3)	2.691 (1)
C(5)—O(3)	1.359 (2)	O(1)···O(2)	2.635 (1)
O(3)—C(2)	1.366 (2)		
O(1)—C(1)—O(2)	124.5 (1)	C(1)—O(1)—H(1A)	114 (2)
C(2)—C(1)—O(1)	118.2 (1)	C(1)—O(2)—H(1B)	118 (2)
C(2)—C(1)—O(2)	117.3 (1)	C(2)—C(3)—H(2)	125 (1)
C(1)—C(2)—C(3)	131.9 (1)	C(4)—C(3)—H(2)	129 (1)
C(1)—C(2)—O(3)	117.6 (1)	C(3)—C(4)—H(3)	124 (1)
C(2)—C(3)—C(4)	106.3 (1)	C(5)—C(4)—H(3)	129 (1)
C(3)—C(4)—C(5)	106.6 (1)	C(4)—C(5)—H(4)	132 (1)
C(4)—C(5)—O(3)	110.8 (1)	O(3)—C(5)—H(4)	117 (1)
C(5)—O(3)—C(2)	105.7 (1)		
O(3)—C(2)—C(3)	110.5 (1)		

Symmetry code: (i) = 1 - x, -y, -z.

References

- BERKOVITCH-YELLIN, Z. & LEISEROWITZ, L. (1982). *J. Am. Chem. Soc.* **104**, 4052–4064.
- COPPENS, P. & VOS, A. (1971). *Acta Cryst.* **B27**, 146–158.
- DIETERICH, D. A., PAUL, I. C. & CURTIN, D. Y. (1974). *J. Am. Chem. Soc.* **96**, 6372–6380.
- FELD, R., LEHMANN, M. S., MUIR, K. W. & SPEAKMAN, J. C. (1981). *Z. Kristallogr.* **157**, 216–231.
- GOODWIN, T. H. & THOMSON, C. M. (1954). *Acta Cryst.* **7**, 166–173.
- GROTH, P. (1919). *Chem. Krystallogr.* **5**, 498.
- HIRSHFELD, F. L. & HOPE, H. (1980). *Acta Cryst.* **B36**, 406–415.
- HUDSON, P. (1962). *Acta Cryst.* **15**, 919–920.
- International Tables for X-ray Crystallography* (1974). Vol. IV, Table 2.2B. Birmingham: Kynoch Press.
- JOHN, I. G. & RADOM, L. (1978). *J. Am. Chem. Soc.* **100**, 3981–3991.
- MATA, F., MARTIN, M. C. & SØRENSEN, G. O. (1978). *J. Mol. Struct.* **48**, 157–163.

Acta Cryst. (1983). **C39**, 1113–1114

A Redetermination of the Structure of 1,1,2,2-Tetracarboxymethoxyethane,* C₁₀H₁₄O₈, at 145 K

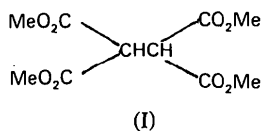
BY FRANK R. FRONCZEK, VINOD K. GUPTA† AND GEORGE R. NEWKOME‡

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

(Received 7 February 1983; accepted 6 April 1983)

Abstract. $M_r = 262.2$, monoclinic, $P2_1/n$, $a = 6.679$ (2), $b = 7.648$ (2), $c = 12.115$ (3) Å, $\beta = 104.36$ (2)°, $V = 599.5$ (5) Å³, $Z = 2$, $F(000) = 276$, $D_x = 1.453$ (2) g cm⁻³, $\lambda = 0.71073$ Å, $\mu(\text{Mo } K\alpha) = 1.20$ cm⁻¹, $R = 0.046$ for 1283 observations (of 1740 unique data). The previously reported structure [Schaefer & Costin (1968). *J. Org. Chem.* **33**, 1677–1678] is confirmed.

Introduction. The structure of 1,1,2,2-tetracarboxymethoxyethane (I) was previously determined (Schaefer & Costin, 1968) using film data, and refined to $R = 0.116$. During our studies of ligands derived from dimethyl malonate, we isolated this tetraester and determined its structure using cryogenic diffractometer data.



* Tetramethyl ethanetetraester.

† On leave from University of Delhi, India, 1980–1983.

‡ To whom correspondence should be addressed.

Experimental. 1,1,2,2-Tetracarboxymethoxyethane was obtained as a by-product (10–20%) during the synthesis of pyridine- or pyrazine-malonate ligands (Newkome, Gupta & Fronczek, 1982) in the form of colorless crystals: § m.p. 407–408 K; ¹H NMR (CDCl₃) δ 3.77 (s, OCH₃, 12H), 4.18 (s, CH, 2H); ¹³C NMR δ 51.1 (CH₃), 53.0 (CH), 167.4 (C=O); MS (70eV) m/e 231 ($M^+ - \text{OCH}_3$, 23), 203 ($M^+ - \text{CO}_2\text{CH}_3$, 28), 171 ($M^+ - \text{C}_3\text{H}_7\text{O}_3$, 100), 159 ($M^+ - \text{C}_4\text{H}_7\text{O}_3$, 48). Other experimental details can be found elsewhere (Newkome, Gupta & Fronczek, 1982; Newkome, Puckett *et al.*, 1982).

Single crystals grown from EtOAc–cyclohexane (1:3). Crystal dimensions 0.24 × 0.32 × 0.48 mm, Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator and a N₂ gas-stream cryostat. Cell dimensions and crystal orientation by least-squares fit of setting angles of 25 reflections with 13° < θ < 16°. Space group $P2_1/n$ was chosen because this indexing yields a more nearly orthogonal cell than the $P2_1/c$ of the original study (Schaefer & Costin, 1968).

§ Analytical data are within acceptable limits.

Cell dimensions at 297 K: $a = 6.822(3)$, $b = 7.675(1)$, $c = 12.163(2)$ Å, $\beta = 103.89(3)^\circ$, $D_x = 1.409(2)$ g cm $^{-3}$. One quadrant of data with $2^\circ < \theta < 30^\circ$ collected, ω - 2θ scan method, scan rates from 0.35 to 5.00 o min $^{-1}$ [in order to measure all significant data with $I \sim 50\sigma(I)$]. Maximum of 180s spent on any single scan. Three standard reflections (200, 040, 002) varied in intensity by less than 40% during the experiment. Data corrected for background, Lorentz, and polarization effects; absorption effects were insignificant. Equivalent data were averaged ($R_{\text{int}} = 0.028$), and data for which $I > 3\sigma(I)$ were used in the refinement.

Refinement by full-matrix least squares based on F with weights $w = \sigma^{-2}(F_o)$, treating nonhydrogen atoms anisotropically. Enraf-Nonius *SDP* programs (Frenz & Okaya, 1980) used throughout; scattering factors were those of Cromer & Waber (1974) with anomalous coefficients of Cromer (1974). H atoms located in initial difference map as peaks of density 0.52–0.74 e Å $^{-3}$, and were refined with isotropic thermal parameters. Secondary-extinction effects were evident, and an extinction coefficient refined to a value of $3.7(7) \times 10^{-6}$. Convergence was achieved (maximum shift = 0.01 σ) with $R = 0.072$ for all data, $R_w = 0.065$, GOF = 1.98 for 111 variables, and a maximum residual of 0.37 e Å $^{-3}$.*

Discussion. The previously reported structure (Schaefer & Costin, 1968) is confirmed. The molecule, illustrated in Fig. 1, has exact $\bar{1}$ symmetry in the crystal and closely approximates local symmetry $2/m$. The distances and angles (Table 1) represent a fourfold increase in precision over the former determination. The H atoms of the methyl groups adopt the expected

* A list of structure factors and a table of coordinates and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38512 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Bond distances (Å) and angles ($^\circ$)

C(1)–C(1')	1.525 (2)	C(4)–O(2)	1.455 (1)
C(1)–C(2)	1.527 (1)	C(4)–H(41)	0.960 (18)
C(1)–C(3)	1.526 (2)	C(4)–H(42)	0.951 (19)
C(1)–H(1)	0.923 (15)	C(4)–H(43)	0.998 (19)
C(2)–O(1)	1.203 (1)	C(5)–O(4)	1.450 (1)
C(2)–O(2)	1.326 (1)	C(5)–H(51)	0.969 (17)
C(3)–O(3)	1.201 (1)	C(5)–H(52)	0.971 (19)
C(3)–O(4)	1.328 (1)	C(5)–H(53)	0.987 (19)
C(2)–C(1)–C(1')	110.51 (11)	C(1)–C(3)–O(3)	123.78 (10)
C(2)–C(1)–C(3)	108.83 (8)	C(1)–C(3)–O(4)	111.33 (9)
C(3)–C(1)–C(1')	110.07 (10)	O(3)–C(3)–O(4)	124.89 (10)
C(1)–C(2)–O(1)	123.90 (9)	C(2)–O(2)–C(4)	116.02 (9)
C(1)–C(2)–O(2)	111.02 (9)	C(3)–O(4)–C(5)	114.92 (9)
O(1)–C(2)–O(2)	125.08 (10)		

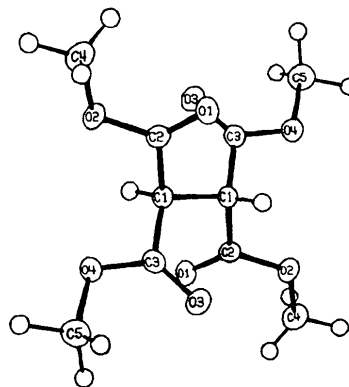


Fig. 1. Drawing of the molecule, illustrating the numbering scheme and H-atom positions.

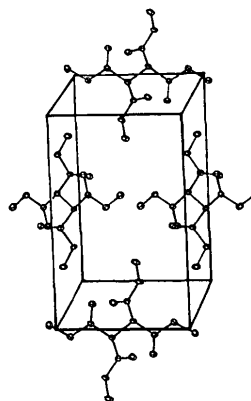


Fig. 2. Crystal packing. The origin is at upper left (foreground), with b horizontal and c vertical.

staggered conformation. The average C–H distance is 0.97 (2) Å. No unusual intermolecular contacts exist, all such distances between heavy atoms being longer than 3.3 Å. The crystal packing is illustrated in Fig. 2.

This work was supported, in part, by the National Science Foundation.

References

- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press.
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press.
- FRENZ, B. A. & OKAYA, Y. (1980). *Enraf-Nonius Structure Determination Package*, Enraf-Nonius, Delft, Holland.
- NEWKOME, G. R., GUPTA, V. K. & FRONCZEK, F. R. (1982). *Organometallics*, **1**, 907–910.
- NEWKOME, G. R., PUCKETT, W. E., KIEFER, G. E., GUPTA, V. K., XIA, Y., COREIL, M. & HACKNEY, M. A. (1982). *J. Org. Chem.* **47**, 4116–4120.
- SCHAEFER, J. P. & COSTIN, C. R. (1968). *J. Org. Chem.* **33**, 1677–1678.