

Table 2. Bond lengths (Å), bond angles (°) and selected torsion angles (°)

O—C(1)	1.215 (3)	C(7)—C(8)	1.513 (3)
C(1)—C(2)	1.497 (3)	C(8)—C(9)	1.382 (3)
C(1)—N(4)	1.361 (3)	C(8)—C(13)	1.374 (3)
C(2)—C(3)	1.535 (3)	C(9)—C(10)	1.377 (3)
C(2)—C(5)	1.286 (3)	C(10)—C(11)	1.366 (3)
C(3)—N(4)	1.494 (3)	C(11)—C(12)	1.367 (3)
C(3)—C(7)	1.502 (3)	C(12)—C(13)	1.379 (3)
C(5)—C(6)	1.301 (3)		
O—C(1)—C(2)	136.3 (2)	C(2)—C(5)—C(6)	178.3 (2)
O—C(1)—N(4)	132.4 (2)	C(3)—C(7)—C(8)	112.7 (2)
C(2)—C(1)—N(4)	91.3 (2)	C(7)—C(8)—C(9)	121.5 (2)
C(1)—C(2)—C(3)	88.3 (2)	C(7)—C(8)—C(13)	120.8 (2)
C(1)—C(2)—C(5)	135.5 (2)	C(9)—C(8)—C(13)	117.7 (2)
C(3)—C(2)—C(5)	136.2 (2)	C(8)—C(9)—C(10)	121.3 (2)
C(2)—C(3)—N(4)	85.0 (2)	C(9)—C(10)—C(11)	120.0 (2)
C(2)—C(3)—C(7)	116.7 (2)	C(10)—C(11)—C(12)	119.5 (2)
N(4)—C(3)—C(7)	115.3 (2)	C(11)—C(12)—C(13)	120.3 (2)
C(1)—N(4)—C(3)	95.3 (2)	C(8)—C(13)—C(12)	121.2 (2)
C(2)—C(3)—C(7)—C(8)	176.6 (2)	C(3)—C(7)—C(8)—C(9)	96.3 (2)
C(3)—C(7)—C(8)—C(13)	-82.8 (2)	N(4)—C(3)—C(7)—C(8)	79.1 (2)

are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Bond lengths, selected bond angles and torsion angles with their standard deviations are given in Table 2. The packing of the molecules in the unit cell is shown in the stereoscopic drawing in Fig. 2.

Related literature. The title compound was prepared (Buynak, Mathew, Rao, Haley, George & Siriwardane, 1987) by sequential treatment of the α -(α' -trimethylsilyl)ethylidene- β -lactam (Buynak, Rao, Chandrasekaran & Haley, 1985) with hypochlorous acid, potassium fluoride and ceric ammonium nitrate. The related structural studies include 2-azetidinones (De Meester, Buynak & Chu, 1986), allenedicarboxylic acid (Berkovitch-Yellin, Leiserowitz & Nader, 1977) and diphenylvinylidene cyclobutane (Berkovitch-Yellin, Lahav & Leiserowitz, 1974).

This work was supported by the Robert A. Welch Foundation under grants to JDB and SSC, and by the donors of the Petroleum Research Fund, administered by the American Chemical Society.

Acta Cryst. (1987). **C43**, 2243–2245

Structure of *o*-Anisic Acid

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(Received 19 January 1987; accepted 9 June 1987)

Abstract. *o*-Methoxybenzoic acid. $C_8H_8O_3$, $M_r = 152.15$, monoclinic, $P2_1/c$, $a = 7.719(2)$, $b = 14.911(3)$, $c = 6.994(2)$ Å, $\beta = 115.52(2)^\circ$, $V = 726.5(7)$ Å³, $Z = 4$, $D_x = 1.39$ Mg m⁻³, Mo $K\alpha$ (graphite monochromator), $\lambda = 0.71073$ Å, $\mu = 0.10$ mm⁻¹, $F(000) = 320$, $T = 293(1)$ K, $R = 0.039$

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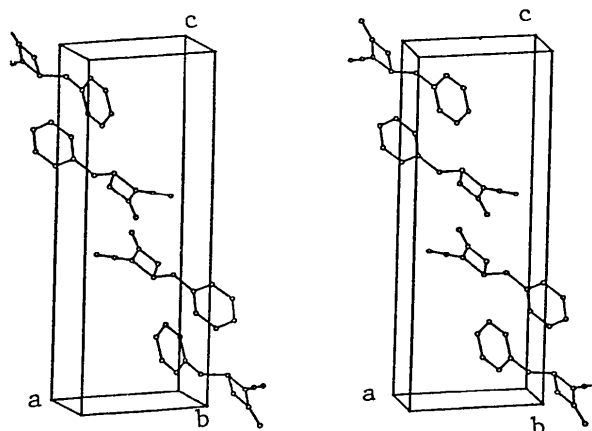


Fig. 2. Stereoscopic drawing of the molecular packing in the unit cell.

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Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2) with e.s.d.'s in parentheses

	x	y	z	B_{eq}^*
O(1)	0.3330 (1)	0.1545 (1)	-0.0952 (1)	3.84 (2)
O(2)	0.4194 (1)	0.0712 (1)	-0.3732 (1)	4.75 (2)
O(3)	0.7027 (1)	0.0052 (1)	-0.2521 (1)	4.79 (2)
C(1)	0.5160 (2)	0.1419 (1)	0.0501 (2)	3.04 (3)
C(2)	0.6391 (2)	0.0922 (1)	-0.0102 (2)	3.06 (3)
C(3)	0.8262 (2)	0.0763 (1)	0.1415 (2)	3.72 (3)
C(4)	0.8930 (2)	0.1094 (1)	0.3440 (2)	4.45 (3)
C(5)	0.7710 (2)	0.1600 (1)	0.3974 (2)	4.63 (4)
C(6)	0.5853 (2)	0.1758 (1)	0.2545 (2)	3.96 (3)
C(7)	0.2044 (2)	0.1985 (1)	-0.0281 (2)	5.04 (4)
C(8)	0.5781 (2)	0.0551 (1)	-0.2260 (2)	3.22 (3)

$$* B_{\text{eq}} = \frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

Table 2. Bond lengths (\AA), bond angles ($^\circ$) and important torsion angles ($^\circ$) with estimated standard deviations in parentheses

O(1)—C(1)	1.352 (1)	C(2)—C(3)	1.396 (2)
O(1)—C(7)	1.427 (2)	C(2)—C(8)	1.481 (2)
O(2)—C(8)	1.237 (2)	C(3)—C(4)	1.372 (2)
O(3)—C(8)	1.290 (2)	C(4)—C(5)	1.378 (2)
C(1)—C(2)	1.405 (2)	C(5)—C(6)	1.369 (2)
C(1)—C(6)	1.388 (2)	O(3)...O(2')	2.631 (1)
C(1)—O(1)—C(7)	117.8 (2)	C(2)—C(3)—C(4)	122.0 (1)
O(1)—C(1)—C(2)	118.0 (2)	C(3)—C(4)—C(5)	118.6 (1)
O(1)—C(1)—C(6)	122.6 (1)	C(4)—C(5)—C(6)	121.3 (1)
C(2)—C(1)—C(6)	119.4 (1)	C(1)—C(6)—C(5)	120.5 (1)
C(1)—C(2)—C(3)	118.2 (1)	O(2)—C(8)—O(3)	121.8 (1)
C(1)—C(2)—C(8)	123.0 (2)	O(2)—C(8)—C(2)	122.8 (2)
C(3)—C(2)—C(8)	118.8 (1)	O(3)—C(8)—C(2)	115.5 (1)
O(3)—H(O3)...O(2)	169 (2)		
C(7)O(1)—C(1)C(6)	-4.9 (2)	C(1)C(2)—C(8)O(2)	5.9 (2)
O(1)C(1)—C(2)C(8)	1.4 (2)	C(2)C(3)—C(4)C(5)	-0.3 (2)
C(6)C(1)—C(2)C(3)	2.0 (2)	C(3)C(4)—C(5)C(6)	1.5 (2)
C(1)C(2)—C(3)C(4)	-1.5 (2)	C(4)C(5)—C(6)C(1)	-0.9 (2)

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

for 132 variables and 1020 independent reflections with $I > 3\sigma(I)$. The crystal structure consists of dimers which are H-bonded through carboxyl groups. Both the methoxy and carboxy groups are essentially coplanar with the six-membered ring.

Experimental. Prismatic crystal $0.54 \times 0.40 \times 0.27$ mm from toluene/carbon tetrachloride (1:1), cell dimensions and orientation matrix from 25 reflections with $10 < \theta < 15^\circ$, intensity data $2 < \theta < 25^\circ$ on an Enraf-Nonius CAD-4 diffractometer, $\omega/2\theta$ scans, ω -scan width $(0.80 + 0.35 \tan\theta)^\circ$, $0 \leq h \leq 9$, $0 \leq k \leq 17$, $-8 \leq l \leq 8$, 1249 unique reflections, 1020 with $I > 3\sigma(I)$, no significant variation in intensities of three standard reflections measured every 2 h, Lp correction, empirical absorption correction (North, Phillips & Mathews, 1968), min. and max. transmission factors 0.9648 and 1.000, respectively. Structure solved by MULTAN82 (Main, Fiske, Hull, Lessinger, Germain,

Declercq & Woolfson, 1982), refinement by full-matrix least-squares calculations on F^2 's, H atoms from ΔF map, C and O anisotropic, H isotropic in least-squares refinement. Final $R = 0.039$, $wR = 0.057$, $w = [\sigma^2(F_o) + (0.080F_o)^2]^{-1}$, $(\Delta/\sigma)_{\text{max}} = 0.02$, $\Delta\rho \pm 0.17 \text{ e \AA}^{-3}$ in final difference map, goodness of fit = 1.31. Scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs from the Enraf-Nonius *Structure Determination Package* (B. A. Frenz & Associates Inc., 1985), ORTEP (Johnson, 1976). The atomic parameters are given in Table 1 and molecular dimensions in Table 2.* Fig. 1 shows the dimeric molecules with the crystallographic numbering scheme; Fig. 2 shows the packing of molecules in the unit cell.

Related literature. The crystal structure of *p*-methoxybenzoic acid has been described by Colapietro & Domenicano (1978).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a full list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44130 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

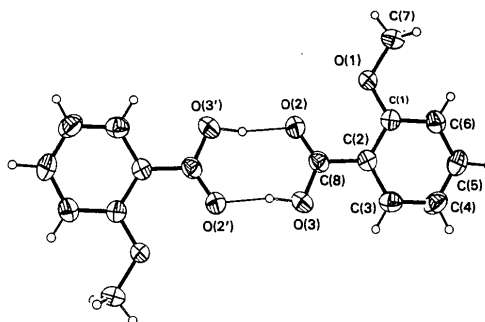


Fig. 1. Perspective drawing of the dimeric structure of *o*-anisic acid showing the atomic numbering scheme.

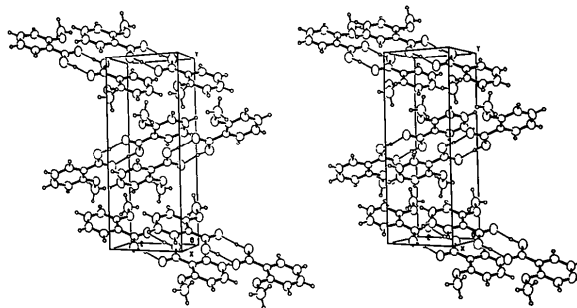


Fig. 2. Stereoview of the unit cell of *o*-anisic acid.

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Acta Cryst. (1987). C43, 2245–2246

1,6-Dioxacyclodeca-3,8-diene

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(Received 19 May 1987; accepted 23 June 1987)

Abstract. C₈H₁₂O₂, *M_r* = 140.18, triclinic, *P* $\bar{1}$, *a* = 7.263 (15), *b* = 7.683 (15), *c* = 7.225 (12) Å, α = 72.62 (5), β = 137.37 (8), γ = 130.36 (7)°, *V* = 195.33 Å³, *Z* = 1, *D_x* = 1.19 Mg m⁻³, $\lambda(\text{Cu } K\alpha)$ = 1.5418 Å, μ = 0.6 mm⁻¹, *F*(000) = 76, *T* = 293 K, *R* = 0.062 for 350 photometric reflexions. The centrosymmetric molecule adopts a chair-like conformation. The two C–O–C groups forming the 'seat' are coplanar (r.m.s. deviation 0.003 Å). The angle between the normals to the 'back' and 'seat' planes is 94.8 (2)°. A rigid-body calculation shows that the molecule is not flexible (RG 0.055).

Experimental. Colourless equidimensional crystals from methanol, volatile at room temperature. Equi-inclination Weissenberg photographs, unit cell refined from *hk0*, *h0l* and *0kl* pictures, intensity data from *h* 0–5 and *k* 0–4. SERC Microdensitometer Service, Daresbury Laboratory. [Reduced cell *a* = 6.285 (15), *b* = 6.903 (16), *c* = 5.266 (13) Å, α = 103.00 (8), β = 99.41 (6), γ = 113.76 (6)°.] Capillary-mounted crystal 0.4 × 0.5 × 0.4 mm. 683 measured reflexions gave 353 unique data (*R*_{int} 0.063), all observed with *I* > 0, 2 θ _{max} 133°. Index range |*h*| ≤ 8, |*k*| ≤ 8, |*l*| ≤ 8. Programs used: *SHELXS* (Sheldrick, 1986), *SHELX76* (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Rigid-body calculation by method of Schomaker & Trueblood (1968).

Structure solution by routine direct methods (20 visually estimated intense streaky reflections added to

Table 1. 1,6-Dioxacyclodeca-3,8-diene: coordinates × 10⁴ for non-H atoms with e.s.d.'s in parentheses, *U*_{eq}¹ Å² × 10³

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} [*]
O1	4452 (9)	7065 (6)	6274 (8)	56 (1)
C2	6166 (13)	8114 (8)	5420 (12)	53 (1)
C3	9341 (12)	8270 (8)	7382 (11)	47 (1)
C4	9654 (12)	7113 (8)	6628 (10)	43 (1)
C5	6864 (13)	5354 (8)	3708 (11)	46 (1)

$$* U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

Table 2. 1,6-Dioxacyclodeca-3,8-diene: interatomic distances (Å) and angles (°)

C2–O1	1.454 (5)	C3–C2–O1	113.1 (4)
C3–C2	1.508 (7)	C4–C3–C2	127.2 (5)
C4–C3	1.369 (6)	C5–C4–C3	127.3 (4)
C5–C4	1.499 (7)	O1'–C5–C4	113.1 (4)
O1'–C5	1.439 (5)	C2–O1–C5'	112.4 (4)
Torsion angles (°)			
O1–C2–C3–C4	117.1 (5)	C4–C5–O1'–C2'	60.3 (4)
C2–C3–C4–C5	–2.5 (5)	C5'–O1–C2–C3	–58.3 (5)
C3–C4–C5–O1'	114.5 (5)		

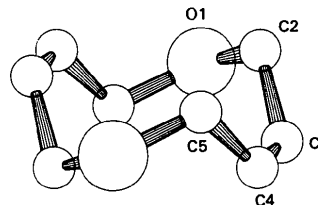


Fig. 1. 1,6-Dioxacyclodeca-3,8-diene.

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