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Acta Cryst. (1987). C**43**, 2245–2246

1,6-Dioxacyclodeca-3,8-diene

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(*Received* 19 *May* 1987; *accepted* 23 *June* 1987)

Abstract. $C_8H_{12}O_2$, $M_r = 140{\cdot}18$, triclinic, $P\bar{1}$, $a = 7{\cdot}263\ (15)$, $b = 7{\cdot}683\ (15)$, $c = 7{\cdot}225\ (12)$ Å, $\alpha = 72{\cdot}62\ (5)$, $\beta = 137{\cdot}37\ (8)$, $\gamma = 130{\cdot}36\ (7)°$, $V = 195{\cdot}33$ Å^3, $Z = 1$, $D_x = 1{\cdot}19$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}6$ mm^{-1}, $F(000) = 76$, $T = 293$ K, $R = 0{\cdot}062$ 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 *hk*0, *h*0*l* and 0*kl* pictures, intensity data from *h* 0–5 and *k* 0–4. SERC Microdensitometer Service, Daresbury Laboratory. [Reduced cell $a = 6{\cdot}285\ (15)$, $b = 6{\cdot}903\ (16)$, $c = 5{\cdot}266\ (13)$ Å, $\alpha = 103{\cdot}00\ (8)$, $\beta = 99{\cdot}41\ (6)$, $\gamma = 113{\cdot}76\ (6)°$.] Capillary-mounted crystal 0·4 × 0·5 × 0·4 mm. 683 measured reflexions gave 353 unique data (R_{int} 0·063), all observed with $I > 0$, $2\theta_{\max}$ 133°. Index range $|h| \le 8$, $|k| \le 8$, $|l| \le 8$. Programs used: *SHELXS* (Sheldrick, 1986), *SHELX*76 (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* ×10^4 *for non*-H *atoms with e.s.d.'s in parentheses*, U_{eq} Å^2 × 10^3

	x	*y*	*z*	U_{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)

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

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)		

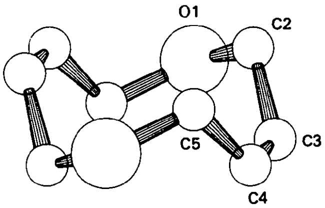

Fig. 1. 1,6-Dioxacyclodeca-3,8-diene.

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data). Refinement minimizing $\sum w\Delta^2$ to R 0·062, wR 0·076. All non-H atoms anisotropic; H atoms in calculated positions, U(H) refined to 0·083 (9) Å^2 for CH_2, 0·073 (11) Å^2 for CH; constant weights, 49 refined parameters, max. Δ/σ 0·002; max. features in final difference map 0·163, −0·180 e Å^{-3}. Atomic scattering curves from *SHELX*76. Final coordinates are given in Table 1,* with bond lengths and angles in Table 2. The molecule is shown in Fig. 1.

Related literature. The synthesis and properties of this compound will be reported by Schroth in a subsequent publication.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44178 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1987). C**43**, 2246–2247

2-(1,4-Dimethoxy-2-naphthyl)-2-phenylacetonitrile

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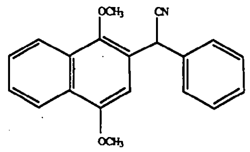
(*Received* 21 *January* 1987; *accepted* 15 *June* 1987)

Abstract. $C_{20}H_{17}NO_2$, $M_r = 303·36$, triclinic, $P\bar{1}$, $a = 7·885\ (4)$, $b = 9·515\ (5)$, $c = 11·591\ (6)$ Å, $\alpha = 84·19\ (4)$, $\beta = 69·95\ (4)$, $\gamma = 99·74\ (4)°$, $V = 793·2\ (7)$ Å^3, $Z = 2$, $D_x = 1·27$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·76 cm^{-1}, $F(000) = 320$, $T = 295$ K. Final $R = 0·038$ for 1631 observed reflections. The naphthyl, phenyl, nitrile and hydrogen moieties are arranged in a distorted tetrahedron with respect to the α-carbon atom. The angle between the planes formed by the phenyl and naphthyl rings is 78·91 (6)°. The bond distances are O—CH_3(ave.) = 1·426 (3), (CH_3)O—C(ave.) = 1·372 (2), $C\equiv N$ = 1·133 (3), C—CN = 1·472 (3) and (CN)C—C(ave.) = 1·524 (3) Å. The structure confirms simple anion addition of the sodium salt of phenylacetonitrile to 3,8-dimethoxynaphthyne in the reaction.

Experimental. Recently, we found a simple method for the preparation of (I) in 70% yield and a variety of structurally related 2-alkyl- and 2-aryl-2-(1,4-dimethoxy-2-naphthyl)acetonitriles in high yields by the addition of sodium alkylnitriles to the aryne intermediate generated from the appropriate haloarene (Khanapure & Biehl, 1987).



(I)

Crystals of (I) are colorless rhombohedral plates; a single crystal of dimensions 0·40 × 0·60 × 0·24 mm was mounted on a goniometer head with an epoxy resin; unit-cell parameters obtained by least-squares refinement of 15 reflections in the range $10 < 2\theta < 25°$, automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, variable scan rate (3·0–14·7° min^{-1}, depending on intensity), 2358 measured reflections, 2094 independent reflections in the range $3 < 2\theta < 45°$, $R_{\rm int} = 0·016$, hkl range h −8 → 8, k −10 → 10, l 0 → 12, 1631 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change (~3%) in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Direct-methods *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978),

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