$w_F(|F_o| < 25) = (|F_o|/25)^2$, $w_F(|F_o| > 30) = (30/|F_o|)^2$, and $w_F(25 < |F_o| < 30) = 1{\cdot}0$, and $w_S(\sin\theta < 0{\cdot}40) = (\sin\theta/0{\cdot}40)^{2{\cdot}5}$, $w_S(\sin\theta > 0{\cdot}42) = (0{\cdot}42/\sin\theta)^2$, and $w_S(0{\cdot}40 < \sin\theta < 0{\cdot}42) = 1{\cdot}0$. H atoms were found in the difference map and were included with calculated positions and $U = 0{\cdot}06$ Å^2 as fixed atom contribution to the structure-factor calculation. Final difference Fourier map shows max. height of 1·04 e Å^{-3} close to Ce atom, min. −0·83 e Å^{-3}. The hand of the crystal was established by the refinement of imaginary component of the anomalous dispersion [2·1 (7) e].

All calculations carried out using *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).* Atomic scattering factors for non-H atoms from Cromer & Mann (1968). Anomalous-dispersion correction factors for non-H atoms from Cromer & Liberman (1970).

Table 1 gives the atomic coordinates and Table 2 bond lengths and angles. Fig. 1 shows the coordination sphere around the Ce atom with the labelling of atoms, Fig. 2 shows the packing in the unit cell.

Related literature. The crystal structure of the title compound was determined as part of a systematic study concerning double salts of the rare earths of general formula $M^{\rm I}{\rm Ln}^{\rm III}({\rm SO_4})_2.x{\rm H_2O}$ (Arhar, Golič, Jordanovska & Šiftar, 1981; Kaučič, Bukovec & Golič, 1985, and references therein).

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* Lists of structure amplitudes, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44481 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 374–376

2-(2,5-Dimethoxybenzyl)-3,6-dimethoxy-4-methylbenzonitrile

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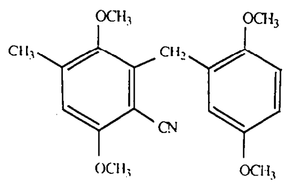
Abstract. $C_{19}H_{21}NO_4$, $M_r = 327{\cdot}41$, monoclinic, $P2_1/c$, $a = 8{\cdot}152$ (3), $b = 21{\cdot}858$ (7), $c = 9{\cdot}872$ (3) Å, $\beta = 103{\cdot}83$ (2)°, $V = 1708{\cdot}1$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}27$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·83 cm^{-1}, $F(000) = 696$, $T = 295$ K, final $R = 0{\cdot}041$ for 1524 observed reflections. The bond distances are H_2C-C(av.) = 1·510 (3), C≡N = 1·144 (4), C—CN = 1·427 (4), $C-CH_3$ = 1·505 (4), H_3C-O(av.) = 1·426 (4), O—C(benz., av.) = 1·372 (4) and C—C (benz., av.) = 1·386 (4) Å. The X-ray structure unambiguously confirms the 4-position of the methyl group. The benzene rings are planar and the angle between the normals to the planes of the rings is 85·0 (1)°.

Experimental. The title compound (I) was prepared in 20% yield by the reaction of lithio-2,5-dimethoxyphenylacetonitrile and 2-bromo-5-methyl-1,4-dimethoxybenzene (generated *in situ* with LDA in THF) involving a tandem addition rearrangement.



(I)

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Table 1. *Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-*H *atoms*

E.s.d.'s are given in parentheses. The standard deviations of U_{eq}'s were calculated according to Schomaker & Marsh (1983).

	x	y	z	$U_{eq}/U(\text{Å}^2)$
C(1)	0·7826 (3)	0·54103 (11)	−0·1624 (2)	0·0414 (6)
C(2)	0·6964 (3)	0·48595 (11)	−0·1938 (2)	0·0429 (6)
C(3)	0·6651 (3)	0·44779 (11)	−0·0906 (3)	0·0466 (6)
C(4)	0·7216 (3)	0·46535 (12)	0·0474 (3)	0·0489 (6)
C(5)	0·8036 (3)	0·51996 (12)	0·0820 (2)	0·0443 (6)
C(6)	0·8350 (3)	0·55780 (11)	−0·0225 (3)	0·0418 (6)
C(7)	0·8098 (3)	0·58305 (11)	−0·2763 (3)	0·0496 (6)
C(8)	0·6715 (3)	0·62988 (11)	−0·3218 (2)	0·0440 (6)
C(9)	0·5427 (3)	0·63686 (11)	−0·2543 (3)	0·0462 (6)
C(10)	0·4158 (3)	0·68003 (12)	−0·2975 (3)	0·0497 (6)
C(11)	0·4172 (4)	0·71749 (12)	−0·4079 (3)	0·0557 (7)
C(12)	0·5468 (4)	0·71172 (12)	−0·4771 (3)	0·0612 (8)
C(13)	0·6720 (4)	0·66783 (12)	0·4356 (3)	0·0540 (7)
O(14)	0·6303 (2)	0·47191 (9)	−0·3322 (2)	0·0623 (5)
C(15)	0·5691 (4)	0·38876 (13)	−0·1257 (3)	0·0683 (8)
O(16)	0·8589 (2)	0·54138 (9)	0·2147 (2)	0·0632 (5)
C(17)	0·9203 (3)	0·61452 (12)	0·0159 (3)	0·0535 (6)
O(18)	0·2955 (2)	0·68057 (9)	−0·2202 (2)	0·0696 (5)
O(19)	0·8012 (3)	0·65743 (9)	−0·4993 (2)	0·0718 (5)
C(20)	0·7229 (4)	0·42767 (15)	−0·3896 (3)	0·0787 (10)
C(21)	0·8084 (4)	0·50920 (16)	0·3243 (3)	0·0728 (9)
N(22)	0·9868 (3)	0·66038 (11)	0·0447 (3)	0·0812 (7)
C(23)	0·1597 (4)	0·72252 (15)	−0·2590 (4)	0·0768 (9)
C(24)	0·8108 (4)	0·69435 (16)	−0·6167 (3)	0·0822 (10)

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

C(1)–C(7)	1·510 (3)	C(13)–O(19)	1·369 (4)
C(2)–O(14)	1·378 (3)	O(14)–C(20)	1·424 (4)
C(3)–C(15)	1·505 (4)	O(16)–C(21)	1·431 (4)
C(5)–O(16)	1·362 (3)	C(17)–N(22)	1·144 (4)
C(6)–C(17)	1·427 (4)	O(18)–C(23)	1·417 (4)
C(7)–C(8)	1·511 (3)	O(19)–C(24)	1·431 (4)
C(10)–O(18)	1·379 (3)		
C(4)–C(5)–O(16)	124·6 (2)	C(12)–C(13)–O(19)	124·8 (3)
C(6)–C(5)–O(16)	115·6 (2)	C(2)–O(14)–C(20)	115·2 (2)
C(1)–C(7)–C(8)	113·8 (2)	C(5)–O(16)–C(21)	118·0 (2)
C(9)–C(10)–O(18)	114·4 (2)	C(6)–C(17)–N(22)	178·8 (3)
C(11)–C(10)–O(18)	125·3 (2)	C(10)–O(18)–C(23)	118·0 (2)
C(8)–C(13)–O(19)	115·0 (2)	C(13)–O(19)–C(24)	118·4 (2)
C(1)–C(2)–O(14)–C(20)	103·6 (3)	C(4)–C(5)–O(16)–C(21)	−9·0 (4)
C(1)–C(6)–C(17)–N(22)	39 (13)	C(8)–C(13)–O(19)–C(24)	−179·9 (3)
C(1)–C(7)–C(8)–C(9)	6·2 (3)	C(9)–C(10)–O(18)–C(23)	−178·9 (2)
C(2)–C(1)–C(7)–C(8)	90·8 (3)		

Crystals of (I) are colorless hexagonal prisms, unit-cell parameters by least-squares fit of 15 reflections in the range $10 < 2\theta < 25°$, crystal dimensions $0{\cdot}22 \times 0{\cdot}31 \times 0{\cdot}56$ mm, space group $P2_1/c$ from systematic absences ($0k0$, k odd, $h0l$, l odd); automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, variable scan rate ($3{\cdot}0$–$14{\cdot}7°\ \text{min}^{-1}$, depending on intensity), 2568 measured reflections, 2250 independent reflections in the range $3 < 2\theta < 45°$, $R_{\text{int}} = 0{\cdot}005$, hkl range $h\ -8\rightarrow 8$, $k\ 0\rightarrow 22$, $l\ 0\rightarrow 9$, 1524 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 (~2%) in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Direct methods *SHELXS*86 (Sheldrick, 1986), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), anisotropic; H atoms located in difference Fourier maps, H atoms included in the refinement with isotropic temperature factors and constrained as benzyl, methyl and $-\text{CH}_2-$; $w = 1/(\sigma^2F + 0{\cdot}000507F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}041$ and $wR = 0{\cdot}045$ for 1524 observed reflections; $S = 1{\cdot}69$, $(\Delta/\sigma)_{\max} = 0{\cdot}04$, $\Delta\rho_{\max,\min} = 0{\cdot}19$, $-0{\cdot}13\ \text{e Å}^{-3}$ in final difference Fourier map. Atomic scattering factors for C, H, N and O were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* selected bond lengths,

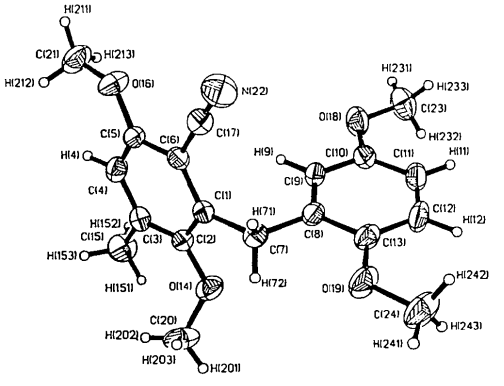

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

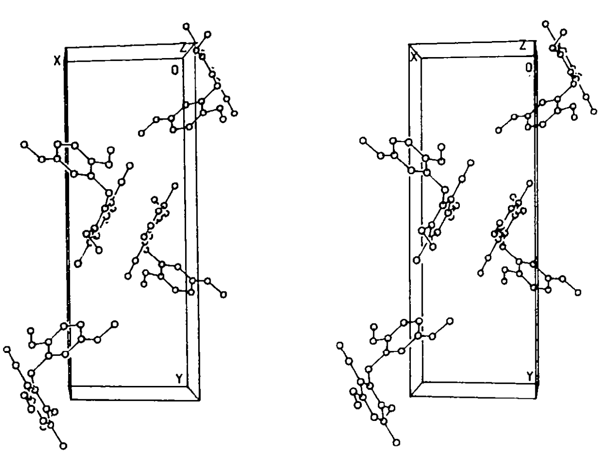

Fig. 2. Stereoscopic drawing showing the packing of molecules in the unit cell. H atoms are removed for clarity.

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* Lists of anisotropic temperature factors, bond lengths, bond angles, torsion angles, deviations from least-squares planes, H-atom parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44412 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles and torsion angles are given in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1; the packing of the molecules is shown in the unit cell in Fig. 2.

Related literature. Polysubstituted benzenes such as (I) are easily hydrolyzed to acids which serve as precursors to naturally occurring anthraquinones and cyanodontin (de Silva, Watanabe & Snickus, 1979).

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Structure of the Ethanol Solvate of 13-Ethyl-11β-methyl-18-norlynestrenol

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Abstract. 13-Ethyl-11β-methyl-18,19-dinor-17α-pregn-4-en-20-yn-17β-ol, $C_{22}H_{32}O.C_2H_6O$, $M_r = 358{\cdot}57$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}888\ (3)$, $b = 10{\cdot}594\ (4)$, $c = 26{\cdot}45\ (2)$ Å, $V = 2210\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}077$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}6$ cm^{-1}, $F(000) = 792$, $T = 293$ K, $R = 0{\cdot}064$ for 1946 observations. The overall molecular conformation is identical to that of 11β-methyllynestrenol [Rohrer, Hazel, Duax & Zeelen (1976). *Cryst. Struct. Commun.* **5**, 543–546]; a least-squares fit of the steroid backbones [C(1)–C(18)] gave a mean deviation of the fitted atoms of 0·033 (4) Å. The steroid backbone is bent by steric interaction between the angular methyl and ethyl groups. The methyl group does not affect the orientation of the ethyl group, which has the preferred *trans* conformation with respect to the *C*/*D* ring junction [van Geerestein, Kanters, Duisenberg & Kroon (1986). *Acta Cryst.* C**42**, 469–472], C(14)–C(13)–C(18)–C(22) = −168·2 (4)°. The ethanol solvent molecule acts as a donor as well as an acceptor in two hydrogen bonds connecting two steroid molecules related by the 2_1 axis parallel to **a**; $\rightarrow$O(17)$\rightarrow$O(*e*)$\rightarrow$O(17′) $(\frac{1}{2}+x, \frac{3}{2}-y, -z)$ with O(17)···O(*e*) = 2·735 (6), O(*e*)···O(17′) = 2·751 (6) Å, O(17)–H···O(*e*) = 158 (5) and O(*e*)–H···O(17′) = 162 (5)°. The shortest intermolecular H···H contact distances are approximately 2·3 Å.

Experimental. Sample obtained from the Scientific Development Group of Organon, Oss, The Netherlands. Large crystals were obtained by evaporation of an ethanol solution. An irregular shaped fragment (1·0 × 1·0 × 0·6 mm) was used for data collection. Lattice parameters refined by least-squares fitting of 2θ values of 12 reflections in range 19–24°; Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation; ω–2θ scan mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}35\tan\theta)°$; 2889 independent reflections measured, $2\theta_{\max} = 55°$, h, k, l (max. range 10, 13, 34), 1946 reflections with $I \geq 2{\cdot}5\sigma(I)$ were considered observed. Four standard reflections showed intensity variations up to 7%; intensities were corrected for these fluctuations. Lp corrections were applied, but no absorption corrections.

Structure solved by Patterson search with a preliminary version of *PATSEE* (Egert & Sheldrick, 1985); the fused *B* + *C* rings of the steroid backbone were used as search fragment, whereafter the best solution was used for tangent expansion. *E* map gave all non-H atoms including those of the solvent molecule. However, the methyl C atom of ethanol was located at very low density.

H atoms were placed at calculated positions riding on their bonded atoms, except both hydroxyl-group H atoms and the H atom bonded to the ethynyl group, which were located on a difference map and refined positionally. 245 parameters were varied in full-matrix least-squares refinement on *F* with *SHELX*76 (Sheldrick, 1976); all non-H atoms refined anisotropically and for H atoms an overall isotropic thermal parameter was varied [$U = 0{\cdot}073\ (3)$ Å^2]; H atoms of the

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