Paquette, Moriarty, Meunier, Gautheron, Sornay, Rogers & Rheingold, 1989). For reviews on titanium organometallic chemistry see Cozak & Melnik (1986) and Bottrill, Gavens, Kelland & McMeeking (1982). The methylcyclopentadienyl ligand offers several advantages over the unsubstituted ligand: increased solubility, elements of molecular symmetry from NMR spectra and most importantly increased crystallizability (Darkwa, Giolando, Jones Murphy & Rauchfuss, 1990). The coordination geometry of the title compound is similar to that of $(C_5H_5)TiCl_3$ which was published with some space group ambiguity (Allegra, Ganis, Porri & Corradini, 1961; Ganis & Allegra, 1962; Engelhardt, Papasergio, Raston & White, 1984). Further single-crystal studies of related transition-metal complexes include $[(C_5H_5)_2Co][(C_5H_5)TiCl_3]$ (Hughes, Jimenez-Tenorio & Leigh, 1989), $(CH_3C_5H_4)VCl_3$ (Morse, Hendrickson, Rauchfuss & Wilson, 1988), $[C_5(CH_3)_4(C_2H_5)]VCl_3$ (Hammer & Messerle, 1990), $(C_5H_5)ZrCl_3$ (Engelhardt, Papasergio, Raston & White, 1984) and $[Li(C_5H_5)CrCl_3.2C_4H_8O]_2.C_4H_8O_2$ (Müller & Krausse, 1972).

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7-Methoxy-2-naphthol

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Abstract. $C_{11}H_{10}O_2$, $M_r = 174{\cdot}2$, monoclinic, $P2_1/c$, $a = 17{\cdot}662\ (3)$, $b = 5{\cdot}9287\ (7)$, $c = 8{\cdot}4003\ (10)$ Å, $\beta = 99{\cdot}857\ (13)°$, $V = 866{\cdot}6\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}335$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 7{\cdot}01$ cm^{-1}, $F(000) = 368$, $T = 300$ K, $R = 0{\cdot}045$ for 1137 observations (of 1708 unique data). The average deviation from planarity is 0·010 Å with a maximum of 0·022 (2) Å for the fused rings. The methoxy group adopts a conformation with the methyl group *syn* to the neighboring α-carbon of the ring, with a C—C—O—C torsion angle of 3·8 (3)°. Steric interaction between the methyl group and the adjacent

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
O1	0·42132 (8)	0·4286 (3)	0·6200 (2)	5·28 (4)
O2	0·06235 (8)	0·2100 (3)	0·0656 (2)	4·94 (4)
C1	0·3487 (1)	0·4740 (4)	0·5409 (2)	3·88 (4)
C2	0·3185 (1)	0·6812 (4)	0·5879 (2)	4·21 (5)
C3	0·2474 (1)	0·7486 (3)	0·5188 (2)	3·97 (5)
C4	0·2004 (1)	0·6139 (3)	0·4020 (2)	3·22 (4)
C5	0·1255 (1)	0·6785 (4)	0·3295 (2)	3·86 (4)
C6	0·0811 (1)	0·5433 (4)	0·2197 (2)	3·93 (4)
C7	0·1111 (1)	0·3354 (4)	0·1767 (2)	3·53 (4)
C8	0·1833 (1)	0·2672 (3)	0·2432 (2)	3·32 (4)
C9	0·2304 (1)	0·4043 (3)	0·3574 (2)	3·00 (4)
C10	0·3059 (1)	0·3399 (4)	0·4287 (2)	3·51 (4)
C11	0·4572 (1)	0·2279 (5)	0·5735 (3)	6·09 (7)

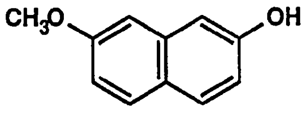
Table 2. *Bond distances* (Å) *and angles* (°)

O1—C1	1·367 (2)	C4—C5	1·412 (3)
O1—C11	1·434 (3)	C4—C9	1·426 (3)
O2—C7	1·374 (2)	C5—C6	1·364 (3)
C1—C2	1·422 (3)	C6—C7	1·413 (3)
C1—C10	1·359 (3)	C7—C8	1·363 (3)
C2—C3	1·349 (3)	C8—C9	1·414 (2)
C3—C4	1·418 (3)	C9—C10	1·418 (3)

C1—O1—C11	116·9 (2)	C5—C6—C7	119·2 (2)
O1—C1—C2	113·6 (2)	O2—C7—C6	115·3 (2)
O1—C1—C10	125·8 (2)	O2—C7—C8	123·4 (2)
C2—C1—C10	120·5 (2)	C6—C7—C8	121·3 (2)
C1—C2—C3	120·1 (2)	C7—C8—C9	120·5 (2)
C2—C3—C4	121·5 (2)	C4—C9—C8	118·5 (2)
C3—C4—C5	122·7 (2)	C4—C9—C10	119·2 (2)
C3—C4—C9	118·2 (2)	C8—C9—C10	122·2 (2)
C5—C4—C9	119·1 (2)	C1—C10—C9	120·4 (2)
C4—C5—C6	121·3 (2)		

hydrogen is responsible for opening the angle between the methoxy group and the ring. The CH_3O—C—C angles are 125·8 (2)° where the methyl group is *syn* to the hydrogen, and 113·6 (2)° where it is *anti*; by contrast, the HO—C—C angles are 123·4 (2) and 115·3 (2)° for the *syn* and *anti* orientations, respectively. In spite of the presence of the hydroxy group, no hydrogen bonds are present in the crystal.

Experimental. Colorless plates of (1), m.p. 389–390 K, were isolated by sublimation of the crude reaction product of 2,7-dihydroxynaphthalene and dimethyl sulfate in dichloromethane/water at room temperature.



(1)

Crystal size 0·02 × 0·50 × 0·52 mm, capillary mounted, space group from systematic absences 0*k*0 with *k* odd and *h*0*l* with *l* odd, cell dimensions from setting angles of 25 reflections having $15 < \theta < 20°$. Data collection on an Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 90 s, scan rates varied 0·82–3·30° min^{-1}. Two quadrants of data having $2 < \theta < 75°$, $-22 \le h \le 22$, $0 \le k \le 6$, $-10 \le l \le 10$ measured, 2790 total data. Data corrected for background, Lorentz and polarization effects. Standard reflections 600, 020, 002 varied randomly and no decay correction was applied. Absorption corrections were based on ψ scans, with a minimum relative transmission coefficient of 0·758. The two equivalent quadrants of data merged, $R_{\rm int} = 0{\cdot}022$, to yield 1708 unique data, 1137 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *SHELXS*86 (Sheldrick, 1985), refined by full-matrix least squares minimizing $\sum w(|F_o| - |F_c|)^2$ with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using the Enraf–Nonius *SDP* system (Frenz & Okaya, 1980), scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1); anomalous coefficients from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). Non-H atoms refined anisotropically; H atoms located by ΔF and refined isotropically. Final $R = 0{\cdot}045$, $wR = 0{\cdot}053$, $S = 2{\cdot}213$ for 159 variables. Maximum shift 0·05σ in the final cycle,

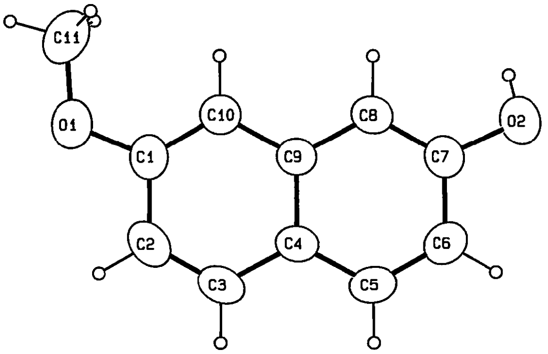


Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 50% probability level. H atoms are drawn as circles of arbitrary radius.

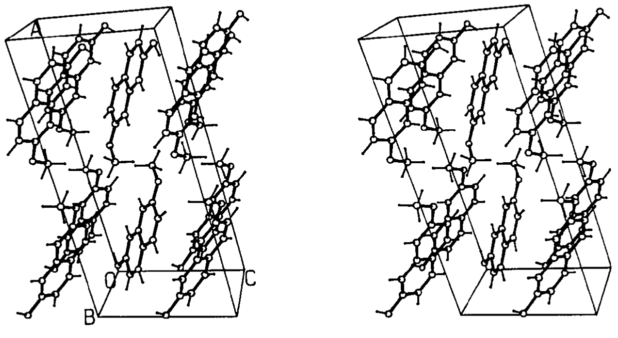


Fig. 2. Stereoview of the unit cell, viewed slightly obliquely to the *b* axis, with **c** horizontal.

max. residual density 0·19, min. $-0{\cdot}14$ e Å^{-3}, extinction coefficient (Larson, 1969) $g = 5{\cdot}0\,(4) \times 10^{-6}$ where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c, maximum correction 16·5% for the $30\bar{2}$ reflection. Table 1 presents the final coordinates* and equivalent isotropic thermal parameters, and Table 2 presents bond distances and angles. Fig. 1 illustrates the molecule and the numbering scheme; and Fig. 2 shows the unit cell.

Related literature. The space group of naphthalene-2,7-diol is discussed in Ahmed (1978). For macrocycles containing the 2,7-dioxynaphthyl group see Hamilton & Van Engen (1987) and Muehldorf, Van Engen, Warner & Hamilton (1988). For the crystal structure of 2,7-dimethoxynaphthalene see Prince, Fronczek & Gandour (1989*a*), for 1-acetyl-2,7-dimethoxynaphthalene see Prince, Fronczek & Gandour (1989*b*), for 1-(1-chlorovinyl)-2,7-dimethoxynaphthalene see Prince, Evans, Boss, Fronczek & Gandour (1990), and for 1-ethynyl-2,7-dimethoxynaphthalene see Prince, Fronczek & Gandour (1990).

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[*] Tables of H-atom coordinates, bond distances and angles involving H atoms, anisotropic thermal parameters, a table of least-squares planes, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54124 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,7-Naphthalenediyl Bis(*p*-toluenesulfonate)

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Abstract. $C_{24}H_{20}O_6S_2$, $M_r = 468{\cdot}6$, triclinic, $P\bar{1}$, $a = 8{\cdot}4835\,(7)$, $b = 11{\cdot}801\,(2)$, $c = 12{\cdot}036\,(2)$ Å, $\alpha = 82{\cdot}321\,(10)$, $\beta = 76{\cdot}560\,(8)$, $\gamma = 73{\cdot}261\,(8)°$, $V = 1119{\cdot}4\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}390$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 24{\cdot}38$ cm^{-1}, $F(000) = 488$, $T = 296$ K, $R = 0{\cdot}037$ for 4025 observations having $I > 3\sigma(I)$ (of 4620 unique data). The average deviation from planarity is 0·018 (2) Å with a maximum of 0·034 (1) Å for the fused rings. One of the toluenesulfonate groups points towards the neighboring α-carbon of the ring, whereas the second one points in the opposite direction forming C—O—S—C torsion angles $-79{\cdot}96\,(12)$ and $+81{\cdot}10\,(11)°$. The dihedral angles between the naphthalene system and the toluenesulfonyl rings are 74·20 (5) and 134·40 (4)° respectively. The S—C distances are 1·743 (2) and 1·753 (1) Å, the S—O distances are identical with length 1·601 (1) Å, and the S=O distances range 1·415 (1)–1·424 (1) Å.

Experimental. Colorless crystals of (1), m.p. 425–426 K, were isolated by recrystallization from THF/hexane of the crude reaction product of 2,7-dihydroxynaphthalene and toluene-4-sulfonyl chloride in dichloromethane/pyridine at 278 K. Crystal size $0{\cdot}10 \times 0{\cdot}32 \times 0{\cdot}48$ mm, mounted on a glass fiber in random orientation on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator, Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 reflections having $25 < \theta <$

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