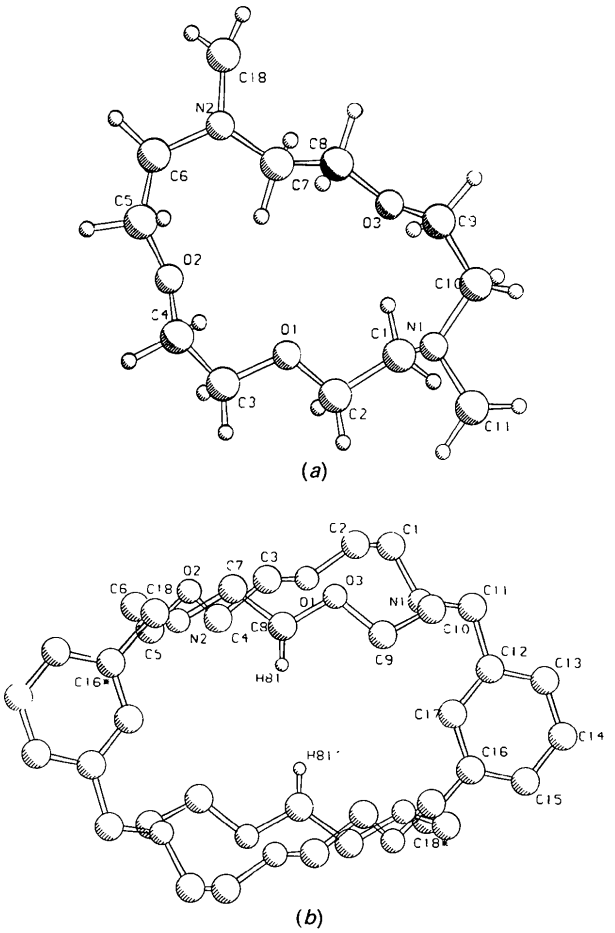

Fig. 1. The numbering scheme for the title compound. (*a*) View of one constituent 15-membered ring; C(18) and C(11) are joined to (centrosymmetrically related) benzene rings. (*b*) View of the whole molecule (hydrogens omitted for clarity) showing that in the crystal conformation there is no large central cavity. [Both views drawn by *PLUTO* (Motherwell, 1976).]

parameters refined for all C, N and O atoms; benzene rings constrained as regular hexagons with C—C = 1·395 Å. $R = 0{\cdot}073$, $wR = 0{\cdot}065$, 174 parameters, $S = 1{\cdot}261$, weight $= 1/[\sigma^2(F) + 0{\cdot}00065\,(F)^2]$, maximum shift/e.s.d. $\leq 0{\cdot}4$, maximum and minimum electron density in final difference map 0·37, $-0{\cdot}41$ e Å^{-3}. The relatively high value of the final R factor and the need to omit many of the weaker intensities in the refinement reflects the overall weakness of the diffraction data obtainable. Refinement with *SHELX* (Sheldrick, 1976) using atomic scattering factors therein. Table 1 gives atom positions* and Table 2 selected information on bond lengths and angles. Views of the molecule are shown in Fig. 1.

Related literature. Like other crown ethers the compound is an efficient and selective multidentate chelating agent, *e.g.* $H_3N^+—CH_2—CH_2—N^+H_3$ are accommodated (Sutherland, 1985).

We acknowledge financial support from the Science & Engineering Research Council.

* 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 54273 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4′-Demethyl-9-(3″-thymidyl)-epipodophyllotoxin

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Abstract. {5*R*-[5α,5aβ,8aα,9β(*R**)]}-5,5a,8a,9-Tetrahydro-5-(4-hydroxy-3,5-dimethoxyphenyl)-9-{[1″-(2″-deoxy-β-D-ribofuranosyl)-5‴-methyluracil]oxy}-furo[3′,4′:6,7]naphtho[2,3-*d*]-1,3-dioxol-6(5a*H*)-one, $C_{31}H_{32}N_2O_{12}$, $M_r = 624{\cdot}60$, monoclinic, $P2_1$, $a = 6{\cdot}724$ (1), $b = 16{\cdot}251$ (2), $c = 13{\cdot}7598$ (8) Å, $\beta = 93{\cdot}292$ (9)°, $V = 1501{\cdot}0$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}382$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 8{\cdot}63$ cm^{-1}, $F(000) = 656$, $T = 296$ K, final $R = 0{\cdot}057$ for 1679 unique reflections.

0108-2701/91/112479-04$03.00

Experimental. The title nucleoside derivative of podophyllotoxin (POD) was synthesized by Derry & Gupta (1991). The compound was crystallized from ethanol by slow evaporation at 277 K. A transparent plate-like crystal, having approximate dimensions of 0·3 × 0·3 × 0·15 mm, was mounted on a glass fibre. Measurements were made on a Rigaku AFC6R diffractometer with graphite-monochromated Cu $K\alpha$ radiation and a 12 kW rotating anode generator. Intensities were measured at 296 K using ω–2θ scans to a $2\theta_{max}$ value of 120·1°. Cell constants were obtained from a least-squares refinement of the setting angles of 24 reflections in the range $50.2 < 2\theta < 59{\cdot}84°$. h,k,l ranged from 0,0,−15 to 8,18,15. 2560 reflections were collected, 2341 were unique (R_{int} = 0·043). Number of unobserved reflections = 662 defined by $I \le 3{\cdot}0\sigma(I)$. The intensities of three representative reflections 112, 103, and 220 remained constant throughout data collection and thus no decay correction was applied. The data were corrected for Lorentz and polarization effects. An empirical absorption correction (North, Phillips & Mathews, 1968) was applied, based on azimuthal scans of several reflections, with transmission factors ranging from 0·65 to 1·00. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984). y axis origin was fixed with respect to atom O(34). H atoms were generated using optimum bonding geometry and included in F_c. H-atom thermal parameters and positions were not refined. Non-H atoms were refined anisotropically. Least squares were refined on F, with function minimized $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/\sigma^2(F_o^2)$. The final cycle of full-matrix least-squares refinement was based on 1679 observed reflections and 405 variable parameters and converged with $R = 0{\cdot}057$ and $wR = 0{\cdot}068$ [R and wR for 2331 observations of $I > 0{\cdot}00\sigma(I) = 0{\cdot}094$, 0·076 respectively]. The max. shift/e.s.d. in the final cycle was 0·03. The standard deviation, S, of an observation of unit weight was 2·15. The weighting scheme was based on counting statistics and included a p factor of 0·03 to downweight the intense reflections. Max. and min. peaks of 0·36 and −0·20 e Å^{-3}, respectively, were observed on the final difference Fourier map. Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964); the values for f' and f'' were those of Cromer (1974). All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1985).

Table 1* lists positional parameters and equivalent isotropic temperature factors. Table 2 lists bond lengths and Table 3 lists bond angles. Fig. 1 displays the title POD derivative using an *ORTEP* (Johnson, 1976) representation. Fig. 2 displays the crystal packing arrangement using *PLUTO* (Motherwell & Clegg, 1978). Crystal packing appears to be stabilized by hydrogen-bonding networks involving the interaction of the phenyl group of molecule $(x,y,z+1)$, the ribose group of molecule $(x+1,y,z)$ and the pyridine group of molecule $[-(x+1), y+0{\cdot}5, -z]$. Important intermolecular distances are given in Fig. 3.

Table 1. *Positional parameters and B_{eq} values with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq} (Å^2)
O(34)	1·461 (1)	0·2719	1·1208 (5)	6·1 (4)
O(35)	1·226 (1)	0·3076 (6)	1·0013 (5)	6·1 (4)
O(36)	0·633 (1)	−0·1044 (6)	1·2065 (4)	5·4 (3)
O(37)	0·9650 (9)	0·0719 (5)	1·3347 (3)	4·5 (3)
O(38)	0·411 (1)	−0·0402 (6)	1·1071 (5)	5·7 (4)
O(39)	0·946 (1)	−0·0638 (6)	0·7955 (4)	5·9 (3)
O(40)	0·615 (1)	−0·0117 (6)	0·6955 (4)	5·9 (3)
O(41)	0·356 (1)	0·0828 (5)	0·7713 (4)	4·7 (3)
O(42)	1·058 (1)	0·1706 (6)	1·5580 (4)	5·5 (3)
O(43)	1·332 (1)	0·0333 (6)	1·5558 (5)	6·0 (4)
O(44)	1·220 (1)	0·3808 (6)	1·4259 (5)	5·8 (4)
O(45)	1·783 (1)	0·3929 (6)	1·6207 (5)	6·7 (4)
N(32)	1·290 (1)	0·2727 (6)	1·5254 (5)	4·1 (3)
N(33)	1·503 (1)	0·3839 (7)	1·5216 (6)	5·1 (4)
C(1)	1·426 (2)	0·3211 (8)	1·0365 (9)	5·8 (6)
C(2)	1·159 (1)	0·2424 (7)	1·0510 (6)	4·0 (4)
C(3)	0·982 (1)	0·2028 (7)	1·0409 (6)	4·2 (4)
C(4)	0·944 (1)	0·1367 (6)	1·1049 (5)	3·4 (4)
C(5)	0·741 (1)	0·0931 (7)	1·0866 (5)	3·7 (4)
C(6)	0·715 (1)	0·0284 (7)	1·1675 (5)	3·7 (4)
C(7)	0·569 (2)	−0·0383 (7)	1·1534 (6)	4·3 (5)
C(8)	0·822 (2)	−0·0879 (7)	1·2560 (7)	5·2 (5)
C(9)	0·909 (1)	−0·0158 (7)	1·1978 (6)	3·7 (4)
C(10)	1·056 (1)	0·0432 (7)	1·2463 (5)	3·7 (4)
C(11)	1·087 (1)	0·1149 (6)	1·1778 (5)	3·7 (4)
C(12)	1·268 (1)	0·1580 (7)	1·1866 (6)	4·2 (4)
C(13)	1·298 (1)	0·2207 (7)	1·1256 (6)	3·9 (4)
C(14)	0·717 (1)	0·0601 (6)	0·9823 (5)	3·3 (3)
C(15)	0·853 (1)	0·0113 (7)	0·9417 (6)	3·9 (4)
C(16)	0·820 (1)	−0·0134 (7)	0·8430 (6)	3·8 (4)
C(17)	0·653 (1)	0·0090 (7)	0·7908 (6)	3·8 (4)
C(18)	0·519 (1)	0·0600 (7)	0·8300 (6)	4·0 (4)
C(19)	0·546 (1)	0·0847 (7)	0·9265 (6)	3·7 (4)
C(20)	1·121 (2)	−0·0926 (7)	0·8410 (7)	5·5 (5)
C(21)	0·219 (1)	0·1410 (8)	0·8055 (6)	5·6 (5)
C(22)	1·091 (2)	0·1132 (8)	1·4042 (6)	5·2 (5)
C(23)	1·070 (2)	0·2079 (8)	1·3955 (7)	7·9 (7)
C(24)	1·091 (2)	0·2392 (7)	1·4983 (6)	5·3 (5)
C(25)	1·020 (1)	0·0960 (7)	1·5041 (6)	4·4 (4)
C(26)	1·120 (2)	0·0253 (7)	1·5557 (6)	5·8 (6)
C(27)	1·332 (2)	0·3480 (7)	1·4882 (7)	4·7 (5)
C(28)	1·635 (2)	0·3540 (7)	1·5958 (7)	4·9 (5)
C(29)	1·579 (2)	0·2750 (7)	1·6345 (6)	4·7 (4)
C(30)	1·413 (1)	0·2382 (7)	1·5987 (6)	4·6 (4)
C(31)	1·709 (2)	0·2408 (8)	1·7153 (8)	6·3 (6)

Related literature. Podophyllotoxin (POD) and its derivatives exhibit two distinct types of biological activity. POD and other naturally occurring POD derivatives, such as epipodophyllotoxin and 4′-demethylepipodophyllotoxin (DMEP), are potent inhibitors of microtubule assembly and arrest cell growth during mitosis (Gupta & Ross, 1989). In contrast to the antimitotic activities of the naturally occurring POD derivatives, semi-synthetic glycoside derivatives of DMEP (*e.g.* etoposide and teniposide)

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54209 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Intramolecular distances* (Å) *with e.s.d.'s in parentheses*

O(34)	C(1)	1·42 (1)	C(3)	C(4)	1·42 (1)
O(34)	C(13)	1·38 (1)	C(4)	C(5)	1·55 (1)
O(35)	C(1)	1·42 (1)	C(4)	C(11)	1·39 (1)
O(35)	C(2)	1·35 (1)	C(5)	C(6)	1·55 (1)
O(36)	C(7)	1·35 (1)	C(5)	C(14)	1·53 (1)
O(36)	C(8)	1·43 (1)	C(6)	C(7)	1·47 (1)
O(37)	C(10)	1·470 (9)	C(6)	C(9)	1·53 (1)
O(37)	C(22)	1·41 (1)	C(8)	C(9)	1·55 (1)
O(38)	C(7)	1·21 (1)	C(9)	C(10)	1·51 (1)
O(39)	C(16)	1·37 (1)	C(10)	C(11)	1·52 (1)
O(39)	C(20)	1·38 (1)	C(11)	C(12)	1·40 (1)
O(40)	C(17)	1·362 (9)	C(12)	C(13)	1·34 (1)
O(41)	C(18)	1·375 (9)	C(14)	C(15)	1·36 (1)
O(41)	C(21)	1·42 (1)	C(14)	C(19)	1·40 (1)
O(42)	C(24)	1·41 (1)	C(15)	C(16)	1·42 (1)
O(42)	C(25)	1·44 (1)	C(16)	C(17)	1·35 (1)
O(43)	C(26)	1·43 (1)	C(17)	C(18)	1·36 (1)
O(44)	C(27)	1·23 (1)	C(18)	C(19)	1·39 (1)
O(45)	C(28)	1·21 (1)	C(22)	C(23)	1·55 (2)
N(32)	C(24)	1·47 (1)	C(22)	C(25)	1·51 (1)
N(32)	C(27)	1·36 (1)	C(23)	C(24)	1·50 (1)
N(32)	C(30)	1·39 (1)	C(25)	C(26)	1·49 (1)
N(33)	C(27)	1·35 (1)	C(28)	C(29)	1·45 (1)
N(33)	C(28)	1·40 (1)	C(29)	C(30)	1·33 (1)
C(2)	C(3)	1·35 (1)	C(29)	C(31)	1·48 (1)
C(2)	C(13)	1·39 (1)			

Table 3. *Intramolecular bond angles* (°) *with e.s.d.'s in parentheses*

C(1)	O(34)	C(13)	106·6 (7)	O(34)	C(13)	C(2)	108·2 (8)
C(1)	O(35)	C(2)	106·4 (8)	O(34)	C(13)	C(12)	129·6 (8)
C(7)	O(36)	C(8)	110·6 (7)	C(2)	C(13)	C(12)	122·2 (8)
C(10)	O(37)	C(22)	116·7 (7)	C(5)	C(14)	C(15)	123·4 (8)
C(16)	O(39)	C(20)	121·2 (7)	C(5)	C(14)	C(19)	117·0 (7)
C(18)	O(41)	C(21)	119·7 (6)	C(15)	C(14)	C(19)	119·6 (7)
C(24)	O(42)	C(25)	113·3 (7)	C(14)	C(15)	C(16)	119·2 (8)
C(24)	N(32)	C(27)	116·2 (8)	O(39)	C(16)	C(15)	124·1 (8)
C(24)	N(32)	C(30)	122·1 (7)	O(39)	C(16)	C(17)	115·1 (7)
C(27)	N(32)	C(30)	120·6 (8)	C(15)	C(16)	C(17)	120·7 (8)
C(27)	N(33)	C(28)	126·1 (8)	O(40)	C(17)	C(16)	123·3 (8)
O(34)	C(1)	O(35)	107·5 (8)	O(40)	C(17)	C(18)	116·2 (7)
O(35)	C(2)	C(3)	129·4 (9)	C(16)	C(17)	C(18)	120·2 (7)
O(35)	C(2)	C(13)	110·2 (8)	O(41)	C(18)	C(17)	117·1 (7)
C(3)	C(2)	C(13)	120·2 (8)	O(41)	C(18)	C(19)	122·6 (8)
C(2)	C(3)	C(4)	119·0 (8)	C(17)	C(18)	C(19)	120·3 (8)
C(3)	C(4)	C(5)	115·8 (7)	C(14)	C(19)	C(18)	119·8 (8)
C(3)	C(4)	C(11)	119·7 (8)	O(37)	C(22)	C(23)	111·8 (8)
C(5)	C(4)	C(11)	124·5 (7)	O(37)	C(22)	C(25)	108·8 (8)
C(4)	C(5)	C(6)	109·4 (6)	C(23)	C(22)	C(25)	102·8 (9)
C(4)	C(5)	C(14)	111·1 (7)	C(22)	C(23)	C(24)	105·1 (8)
C(6)	C(5)	C(14)	115·1 (7)	O(42)	C(24)	N(32)	108·5 (8)
C(5)	C(6)	C(7)	120·7 (7)	O(42)	C(24)	C(23)	105·7 (8)
C(5)	C(6)	C(9)	112·5 (7)	N(32)	C(24)	C(23)	113·5 (9)
C(7)	C(6)	C(9)	104·0 (7)	O(42)	C(25)	C(22)	105·1 (7)
O(36)	C(7)	O(38)	120·3 (9)	O(42)	C(25)	C(26)	110·0 (7)
O(36)	C(7)	C(6)	109·1 (8)	C(22)	C(25)	C(26)	114·8 (9)
O(38)	C(7)	C(6)	130·5 (9)	O(43)	C(26)	C(25)	110·6 (9)
O(36)	C(8)	C(9)	104·2 (7)	O(44)	C(27)	N(32)	121·4 (9)
C(6)	C(9)	C(8)	99·0 (7)	O(44)	C(27)	N(33)	121·7 (9)
C(6)	C(9)	C(10)	110·6 (7)	N(32)	C(27)	N(33)	116·8 (8)
C(8)	C(9)	C(10)	120·6 (7)	O(45)	C(28)	N(33)	119·9 (9)
O(37)	C(10)	C(9)	105·8 (7)	O(45)	C(28)	C(29)	126 (1)
O(37)	C(10)	C(11)	110·4 (6)	N(33)	C(28)	C(29)	114·3 (9)
C(9)	C(10)	C(11)	108·9 (6)	C(28)	C(29)	C(30)	119·3 (9)
C(4)	C(11)	C(10)	121·8 (8)	C(28)	C(29)	C(31)	116·9 (9)
C(4)	C(11)	C(12)	119·6 (8)	C(30)	C(29)	C(31)	123·7 (9)
C(10)	C(11)	C(12)	118·6 (8)	N(32)	C(30)	C(29)	122·7 (8)
C(11)	C(12)	C(13)	119·2 (8)				

show no microtubule inhibitory activity, but cause extensive DNA strand breaks in mammalian cells (Gupta & Ross, 1989; Liu, 1989). This activity results from inhibition of the strand-rejoining reaction of the enzyme DNA topoisomerase II, which plays an important role in DNA replication and transcription. Drugs exhibiting the latter type of activity have also proven clinically very useful in cancer chemotherapy (see Gupta & Ross, 1989).

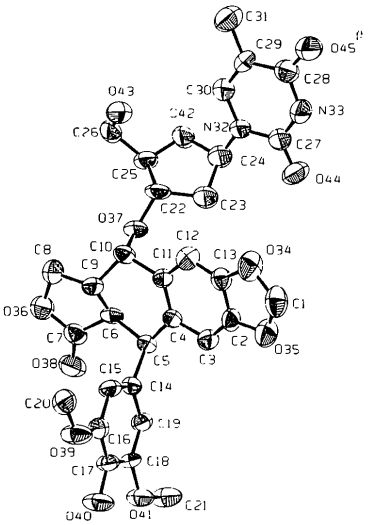

Fig. 1. *ORTEP* (Johnson, 1976) plot of the molecular structure with coordinate atom labelling.

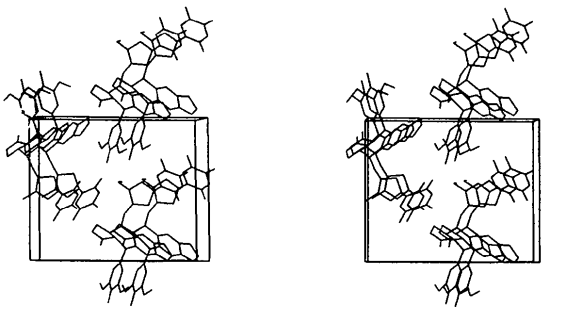

Fig. 2. *PLUTO* stereo plot of packing diagram; view along *a* unit axis.

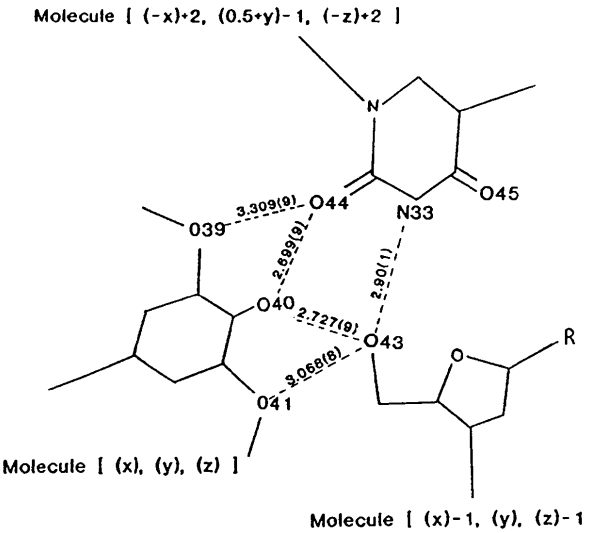

Fig. 3. Hydrogen-bonding network stabilizing crystal packing. Labelled are the molecule designate and intermolecular bond distances (Å) with e.s.d.'s in parentheses.

The structural information on the POD derivatives should prove useful in understanding the structure–activity relationships between this important group of drugs and how they interact with their cellular receptors.

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Acta Cryst. (1991). C**47**, 2482–2484

Structure of a Lactone, (3α,4α,6α)-4-(*tert*-Butyldimethylsiloxy)-3,4,5,6-tetrahydro-6-methyl-3-(2-oxopropyl)-2*H*-pyran-2-one

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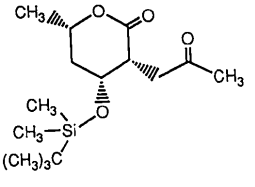
Abstract. $C_{15}H_{28}O_4Si$, $M_r = 300{\cdot}5$, monoclinic, $P2_1/a$, $a = 11{\cdot}194\ (4)$, $b = 10{\cdot}944\ (3)$, $c = 14{\cdot}815\ (5)$ Å, $\beta = 95{\cdot}01\ (3)°$, $V = 1808\ (2)$ Å^3, $D_x = 1{\cdot}104$ g cm^{-3}, $Z = 4$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}43$ cm^{-1}, $F(000) = 656$, $T = 293$ K, final $R = 0{\cdot}052$, $wR = 0{\cdot}058$ for 1630 reflections with $I > 3\sigma(I)$. The valerolactone ring is in a boat conformation and the three ring substituents are *cis*.

Experimental. Colorless crystals from ethyl acetate/hexane, 0·06 × 0·26 × 0·33 mm plate-like specimen; Enraf–Nonius CAD-4 diffractometer; cell parameters and crystal orientation from 25 automatically



centered reflections in the range $6{\cdot}0 < \theta < 16{\cdot}4°$; 2θ–θ scans over $\Delta\theta$ range of $1{\cdot}5(1{\cdot}0 + 0{\cdot}14\tan\theta)°$; variable θ scan speed of 5·49–0·82° min^{-1}; each scan recorded in 96 steps and post-processed with a reflection profile program (Lehmann & Larsen, 1974; Ammon, 1986); diffractometer controlled with Digital Equipment Corp. MicroVAX II computer with version 5.0 of CAD-4 diffractometer control program; six standard reflections monitored at 1 h intervals of X-ray exposure, −2·1–0·1% intensity variation, −0·5% average, correction applied; $2 < \theta < 25°$, *hkl* range for data collection of $h = -13$–13, $k = 0$–13, $l = 0$–17; 3676 total data measured including standards and systematically absent data, 3367 unique data, 1630 data with $I > 3\sigma(I)$; $R_{int} = 0{\cdot}01$ for 135 twice-measured data; absorption correction (*DIFABS*; Walker & Stuart, 1983) following a full isotropic structure refinement, transmission factor range of 0·670–1·220, average of 0·974. All crystallographic calculations performed with the *TEXSAN* (Molecular Structure Corporation, 1989) program system on DEC MicroVAX II or VAXStation II computers; structure solved with the *MITHRIL* (Gilmore, 1983) direct methods program incorporated in *TEXSAN*. Full-matrix least-squares refinement, $\sum[w(F_o - F_c)^2]$ minimized with $w = 1/\sigma^2(F_o)$, reflections with $I < 3\sigma(I)$ excluded from refinement; correction for secondary isotropic extinction (Zachariasen, 1968) applied, $g = 0{\cdot}37\ (4) \times 10^{-6}$; C, O and Si refined with anisotropic temperature factors; H atoms positioned from the C-atom framework, individual isotropic temperature factors refined; 209 total variables; atomic scattering factors

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