

Table 1. Fractional atomic coordinates ($\times 10^4$) and B_{eq} values (\AA^2)
$$B_{\text{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
C(1)	5100 (2)	2765 (3)	3498 (1)	4.15
C(11)	5843 (2)	1858 (3)	3968 (1)	4.11
N(12)	5568 (2)	1297 (3)	4456 (1)	5.18
C(13)	6225 (2)	495 (4)	4865 (2)	5.54
C(14)	7151 (3)	217 (4)	4808 (2)	6.46
C(15)	7436 (3)	805 (5)	4313 (2)	7.98
C(16)	6779 (2)	1641 (4)	3879 (2)	6.41
C(21)	5021 (2)	4449 (3)	3664 (1)	4.26
N(22)	4619 (2)	5353 (4)	3188 (1)	7.14
C(23)	4537 (3)	6856 (5)	3293 (2)	8.51
C(24)	4826 (3)	7520 (5)	3852 (2)	6.96
C(25)	5229 (3)	6599 (4)	4339 (2)	6.03
C(26)	5327 (2)	5043 (4)	4239 (2)	4.76
C(31)	4095 (2)	1999 (3)	3358 (2)	4.39
N(32)*	3898 (3)	1010 (4)	2873 (1)	6.43
C(33)	2979 (4)	334 (5)	2738 (3)	7.96
C(34)	2304 (4)	656 (6)	3060 (3)	8.51
C(35)	2538 (3)	1660 (7)	3543 (2)	8.42
C(36)*	3439 (2)	2343 (6)	3690 (2)	6.49

* Site occupancy factor 0.58 (5).

Related literature. The title compound was examined as part of a wider study of compounds of this type including the ligands (2-py)₃P (Keene, Snow & Tiekink, 1988a) and (2-py)₃COH (Keene, Snow & Tiekink, 1988b), and their complexes (Keene *et al.*, 1988). In complexes both bidentate and tridentate coordination have been shown by X-ray crystallography in [AuMe₂{(2-py)₃CH}]NO₃ (Canty, Minchin, Healy & White, 1982) and [Ru{(2-py)₃CH₂}]₂(tosylate)₂ (Keene *et al.*, 1988), respectively.

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Structure of (±)-Encecanescin

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Abstract. (R^* , R^*)-(±)-6,6'-(Oxydiethylidene)bis[7-methoxy-2,2-dimethyl-2H-1-benzopyran] [compound (1), (±)-encecanescin], C₂₈H₃₄O₅, $M_r = 450.57$, monoclinic, $P2_1/n$, $a = 27.35$ (1), $b = 8.293$ (2), $c = 22.133$ (7) Å, $\beta = 94.68$ (4)°, $V = 5003$ (3) Å³, $Z = 8$ (two molecules, A and B , in the asymmetric unit), $D_x = 1.196$ g cm⁻³, $\text{Mo } K\alpha$ ($\lambda = 0.71069$ Å), $\mu =$

Table 2. Selected interatomic distances (Å) and bond angles (°)

C(1)—C(11)	1.517 (4)	C(1)—C(21)	1.515 (4)
C(1)—C(31)	1.522 (4)	C(1)—H(1)	1.11 (2)
C(11)—N(12)	1.329 (3)	N(12)—C(13)	1.335 (4)
C(21)—N(22)	1.338 (4)	N(22)—C(23)	1.332 (5)
C(31)—C(32)*	1.361 (5)	N(32)—C(33)	1.383 (6)
C(31)—C(36)*	1.338 (5)	C(36)—C(35)	1.363 (6)
C(11)—C(1)—C(21)	113.8 (2)	C(11)—C(1)—C(31)	111.9 (2)
C(11)—C(1)—H(1)	109 (1)	C(21)—C(1)—C(31)	110.7 (2)
C(21)—C(1)—H(1)	106 (1)	C(31)—C(1)—H(1)	105 (1)
C(1)—C(11)—N(12)	118.5 (2)	C(1)—C(11)—C(16)	119.6 (3)
C(1)—C(21)—N(22)	114.1 (3)	C(1)—C(21)—C(26)	124.8 (3)
C(1)—C(31)—N(32)	116.6 (3)	C(1)—C(31)—C(36)	120.5 (3)
C(11)—N(12)—C(13)	118.0 (3)	C(21)—N(22)—C(23)	117.9 (3)
C(31)—N(32)—C(33)	116.7 (4)		

* N(32) and C(36) have site occupancies 0.58 (5).

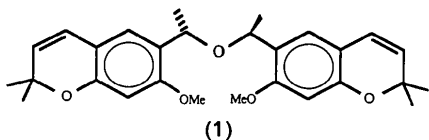
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and differ mainly in the geometry around C2'. The rings containing C2' in *A* and *B* exhibit a half-boat conformation but differ in the orientation of the flag atom C2'. In molecule *A*, C2' is $-0.426(4)$ Å from the plane containing atoms O1', C3', C4', C9' and C10' [distances of other atoms: C13' $0.249(5)$ Å; C14' $-1.941(5)$ Å], while in molecule *B* it is at a distance of $0.504(4)$ Å to the opposite side of the plane [distances of other atoms: C13' $2.018(5)$ Å; C14' $-0.068(5)$ Å]. The different C2' positions in *A* and *B* cause a change in the positions of C13' and C14' from equatorial/axial to axial/equatorial.

Experimental. Crystals of (1) were grown by slow evaporation, at room temperature, from a 1:1 mixture of ethyl acetate and cyclohexane. A colorless plate of dimensions $0.16 \times 0.20 \times 0.27$ mm was chosen for X-ray diffraction. Data were collected on a Syntex P2₁ diffractometer equipped with a graphite monochromator utilizing Mo K α radiation ($\lambda = 0.71069$ Å). 45 reflections with $15.48 \leq 2\theta \leq 22.23^\circ$ were used to refine the cell parameters. 17 950 reflections ($h, 0 \rightarrow 32; k, -9 \rightarrow 9; l, -26 \rightarrow 26$) were collected using the ω -scan method, 8798 unique reflections, $R_{\text{int}} = 0.027$; 2θ range $3.0 \rightarrow 55.0^\circ$, $1^\circ \omega$ -scan at $3-6^\circ \text{ min}^{-1}$. Four reflections (110, 220, 210, 212) were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was $< 1.03\%$). Absorption corrections were applied based on measured crystal faces (Riley & Davis, 1976); absorption coefficient $\mu = 0.755 \text{ cm}^{-1}$.



(1)

The structure was solved by direct methods using MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) from which the locations of all non-H atoms were obtained. The structure was refined using block-matrix least squares in SHELX76 (Sheldrick, 1976) and the positions of all H atoms were determined from a difference Fourier map. Non-H atoms were treated anisotropically, whereas the H atoms were refined with isotropic thermal parameters. 867 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma(F_o))^2$, $\sigma(F_o) = 0.5 k(I^{-1/2})\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$, $\sigma(I) = (\text{scan rate}) \times (I_{\text{peak}} + I_{\text{background}})^{1/2}$ and $I(\text{intensity}) = (\text{scan rate}) \times (I_{\text{peak}} - I_{\text{background}})$; k is the correction due to decay and Lp effects, and 0.02 is a factor used to downweight intense reflections and to account for instrument instability. Final $R = 0.048$, $wR = 0.042$ for 4015 reflections having $F_o \geq 6\sigma(F_o)$, and goodness-of-fit = 1.27. Maximum $\Delta/\sigma < 0.01$ in the final refinement cycle and the minimum and maxi-

Table 1. Fractional coordinates and equivalent isotropic thermal parameters (\AA^2) for the non-H atoms

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

Molecule <i>A</i>	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
O1	-0.14031 (8)	0.3816 (3)	0.25896 (10)	0.0254 (9)
O1'	0.27640 (9)	0.5501 (3)	0.37813 (11)	0.0255 (9)
O7	0.02544 (9)	0.1923 (3)	0.26543 (10)	0.0254 (9)
O7'	0.11929 (9)	0.8066 (3)	0.35866 (11)	0.0253 (9)
O11	0.05677 (8)	0.4040 (3)	0.43393 (10)	0.0226 (9)
C2	-0.17182 (13)	0.5175 (5)	0.2726 (2)	0.0267 (14)
C2'	0.31062 (13)	0.4436 (5)	0.4137 (2)	0.0257 (13)
C3	-0.16372 (13)	0.5662 (5)	0.3383 (2)	0.0270 (14)
C3'	0.28638 (14)	0.2873 (5)	0.4276 (2)	0.0285 (14)
C4	-0.12257 (14)	0.5314 (5)	0.3717 (2)	0.0276 (14)
C4'	0.23846 (14)	0.2785 (5)	0.4332 (2)	0.0287 (14)
C5	-0.03751 (13)	0.4164 (5)	0.3751 (2)	0.0213 (13)
C5'	0.15742 (13)	0.4265 (5)	0.4276 (2)	0.0215 (13)
C6	-0.00121 (12)	0.3320 (4)	0.34850 (15)	0.0165 (12)
C6'	0.12830 (13)	0.5556 (5)	0.40773 (15)	0.0207 (13)
C7	-0.01213 (13)	0.2733 (4)	0.2900 (2)	0.0194 (12)
C7'	0.15029 (13)	0.6839 (4)	0.37846 (15)	0.0190 (12)
C8	-0.05828 (13)	0.2935 (4)	0.2592 (2)	0.0220 (13)
C8'	0.20028 (13)	0.6820 (5)	0.3718 (2)	0.0219 (13)
C9	-0.09373 (12)	0.3760 (5)	0.2883 (2)	0.0204 (13)
C9'	0.22843 (13)	0.5506 (5)	0.3917 (2)	0.0215 (13)
C10	-0.08433 (13)	0.4408 (5)	0.3459 (2)	0.0213 (13)
C10'	0.20751 (13)	0.4194 (5)	0.4193 (2)	0.0231 (13)
C11	0.04729 (13)	0.2954 (5)	0.3835 (2)	0.0237 (13)
C11'	0.07400 (13)	0.5592 (5)	0.4169 (2)	0.0223 (13)
C12	0.0476 (2)	0.1264 (5)	0.4105 (2)	0.032 (2)
C12'	0.0621 (2)	0.6751 (6)	0.4662 (2)	0.030 (2)
C13	-0.1605 (2)	0.6556 (6)	0.2305 (2)	0.037 (2)
C13'	0.3531 (2)	0.4208 (6)	0.3740 (2)	0.038 (2)
C14	-0.2234 (2)	0.4556 (6)	0.2587 (2)	0.029 (2)
C14'	0.3267 (2)	0.5293 (6)	0.4728 (2)	0.035 (2)
C15	0.0186 (2)	0.1498 (6)	0.2022 (2)	0.029 (2)
C15'	0.1392 (2)	0.9314 (5)	0.3233 (2)	0.033 (2)
Molecule <i>B</i>				
O1	0.13395 (8)	0.5772 (3)	0.22495 (10)	0.0252 (9)
O1'	-0.28838 (8)	0.3640 (3)	0.08184 (10)	0.0249 (9)
O7	-0.03221 (8)	0.7641 (3)	0.22262 (10)	0.0250 (9)
O7'	-0.12673 (8)	0.1559 (3)	0.13159 (10)	0.0243 (8)
O11	-0.06544 (8)	0.5538 (3)	0.05441 (10)	0.0197 (8)
C2	0.16658 (12)	0.4471 (5)	0.2088 (2)	0.0246 (13)
C2'	-0.31753 (13)	0.5134 (5)	0.0760 (2)	0.0254 (13)
C3	0.15772 (13)	0.4027 (5)	0.1425 (2)	0.0282 (13)
C3'	-0.29978 (14)	0.6169 (5)	0.0264 (2)	0.0296 (14)
C4	0.11566 (13)	0.4344 (5)	0.11095 (15)	0.0241 (13)
C4'	-0.25257 (13)	0.6176 (5)	0.0161 (2)	0.0293 (14)
C5	0.02976 (12)	0.5449 (4)	0.11095 (15)	0.0216 (12)
C5'	-0.16730 (13)	0.5105 (5)	0.0481 (2)	0.0224 (13)
C6	-0.00629 (12)	0.6268 (4)	0.13859 (15)	0.0176 (12)
C6'	-0.13690 (12)	0.3949 (4)	0.07594 (15)	0.0189 (12)
C7	0.00540 (13)	0.6837 (4)	0.1973 (2)	0.0217 (12)
C7'	-0.15895 (12)	0.2685 (4)	0.10574 (14)	0.0199 (12)
C8	0.05189 (13)	0.6630 (4)	0.22680 (15)	0.0213 (12)
C8'	-0.20942 (13)	0.2610 (4)	0.1084 (2)	0.0212 (12)
C9	0.08724 (12)	0.5820 (4)	0.1965 (2)	0.0205 (12)
C9'	-0.23848 (13)	0.3803 (4)	0.0804 (2)	0.0216 (12)
C10	0.07676 (12)	0.5189 (4)	0.1390 (2)	0.0197 (12)
C10'	-0.21847 (13)	0.5063 (4)	0.0492 (2)	0.0217 (12)
C11	-0.05602 (13)	0.6634 (4)	0.1046 (2)	0.0224 (12)
C11'	-0.08143 (12)	0.3980 (4)	0.0727 (2)	0.0214 (12)
C12	-0.0568 (2)	0.8313 (5)	0.0773 (2)	0.0303 (15)
C12'	-0.06489 (15)	0.2774 (5)	0.0273 (2)	0.0267 (15)
C13	0.1579 (2)	0.3034 (6)	0.2490 (2)	0.035 (2)
C13'	-0.3126 (2)	0.6019 (7)	0.1362 (2)	0.038 (2)
C14	0.2178 (2)	0.5158 (6)	0.2220 (2)	0.033 (2)
C14'	-0.37002 (15)	0.4546 (6)	0.0611 (2)	0.035 (2)
C15	-0.0244 (2)	0.8066 (6)	0.2859 (2)	0.029 (2)
C15'	-0.1466 (2)	0.0310 (6)	0.1673 (2)	0.034 (2)

imum peaks in the ΔF map were -0.25 and 0.22 e \AA^{-3} , respectively. Minimum and maximum transmissions are 93.44 and 98.63%, respectively. The linear absorption coefficient was calculated from values from the *International Tables for X-ray Crystallography* (1974); scattering factors for non-H atoms were taken from Cromer & Mann (1968) with

Table 2. Bond lengths (Å) and angles (°) of the non-H atoms

Molecule A			Molecule B						
1	2	3	1—2	1—2—3	1	2	3	1—2	1—2—3
C2	O1	C9	1.465 (5)	117.7 (3)	C2	O1	C9	1.463 (4)	118.1 (3)
C9	O1		1.382 (4)		C9	O1		1.378 (4)	
C2'	O1'	C9'	1.468 (4)	117.7 (3)	C2'	O1'	C9'	1.474 (4)	116.5 (3)
C9'	O1'		1.369 (4)		C9'	O1'		1.375 (4)	
C7	O7	C15	1.376 (4)	117.5 (3)	C7	O7	C15	1.382 (4)	117.0 (3)
C15	O7		1.440 (5)		C15	O7		1.443 (5)	
C7'	O7'	C15'	1.373 (4)	117.2 (3)	C7'	O7'	C15'	1.376 (4)	117.4 (3)
C15'	O7'		1.432 (5)		C15'	O7'		1.435 (5)	
C11	O11	C11'	1.440 (4)	113.6 (3)	C11	O11	C11'	1.442 (4)	112.9 (2)
C11'	O11		1.431 (5)		C11'	O11		1.433 (4)	
C3	C2	C13	1.509 (5)	111.6 (3)	C3	C2	C13	1.513 (5)	110.9 (3)
C3	C2	C14		110.4 (3)	C3	C2	C14		110.5 (3)
C3	C2	O1		111.2 (3)	C3	C2	O1		111.4 (3)
C13	C2	C14	1.525 (6)	111.3 (3)	C13	C2	C14	1.519 (6)	111.6 (3)
C13	C2	O1		107.4 (3)	C13	C2	O1		107.9 (3)
C14	C2	O1	1.510 (6)	104.6 (3)	C14	C2	O1	1.518 (5)	104.3 (3)
C3'	C2'	C13'	1.499 (6)	112.4 (4)	C3'	C2'	C13'	1.504 (5)	110.7 (3)
C3'	C2'	C14'		109.2 (3)	C3'	C2'	C14'		112.2 (3)
C3'	C2'	O1'		110.9 (3)	C3'	C2'	O1'		109.8 (3)
C13'	C2'	C14'	1.524 (6)	111.9 (3)	C13'	C2'	C14'	1.517 (6)	111.0 (4)
C13'	C2'	O1'		104.4 (3)	C13'	C2'	O1'		108.7 (3)
C14'	C2'	O1'	1.521 (6)	107.8 (3)	C14'	C2'	O1'	1.526 (5)	104.0 (3)
C4	C3	C2	1.326 (5)	122.0 (3)	C4	C3	C2	1.322 (5)	121.8 (3)
C4'	C3'	C2'	1.329 (5)	121.5 (4)	C4'	C3'	C2'	1.329 (5)	120.2 (3)
C10	C4	C3	1.442 (5)	120.2 (3)	C10	C4	C3	1.455 (5)	120.4 (3)
C10'	C4'	C3'	1.460 (5)	119.7 (4)	C10'	C4'	C3'	1.465 (5)	119.9 (3)
C6	C5	C10	1.385 (5)	122.5 (3)	C6	C5	C10	1.380 (5)	122.9 (3)
C10	C5		1.401 (5)		C10	C5		1.398 (5)	
C6'	C5'	C10'	1.384 (5)	122.4 (3)	C6'	C5'	C10'	1.381 (5)	122.7 (3)
C10'	C5'		1.398 (5)		C10'	C5'		1.402 (5)	
C7	C6	C11	1.393 (5)	121.3 (3)	C7	C6	C11	1.396 (5)	121.2 (3)
C7	C6	C5		117.7 (3)	C7	C6	C5		117.5 (3)
C11	C6	C5	1.512 (5)	120.9 (3)	C11	C6	C5	1.530 (5)	121.2 (3)
C7'	C6'	C11'	1.406 (5)	120.5 (3)	C7'	C6'	C11'	1.401 (5)	120.2 (3)
C7'	C6'	C5'		118.3 (3)	C7'	C6'	C5'		117.6 (3)
C11'	C6'	C5'	1.515 (5)	121.2 (3)	C11'	C6'	C5'	1.525 (5)	122.2 (3)
C8	C7	O7	1.395 (5)	122.7 (3)	C8	C7	O7	1.391 (5)	123.5 (3)
C8	C7	C6		121.9 (3)	C8	C7	C6		121.7 (3)
O7	C7	C6		115.3 (3)	O7	C7	C6		114.8 (3)
C8'	C7'	O7'	1.387 (5)	124.2 (3)	C8'	C7'	O7'	1.388 (5)	123.9 (3)
C8'	C7'	C6'		120.2 (3)	C8'	C7'	C6'		121.4 (3)
O7'	C7'	C6'		115.6 (3)	O7'	C7'	C6'		114.7 (3)
C9	C8	C7	1.386 (5)	118.2 (3)	C9	C8	C7	1.393 (5)	118.5 (3)
C9'	C8'	C7'	1.386 (5)	120.1 (3)	C9'	C8'	C7'	1.384 (5)	119.1 (3)
C10	C9	O1	1.390 (5)	120.8 (3)	C10	C9	O1	1.385 (5)	121.6 (3)
C10	C9	C8		122.2 (3)	C10	C9	C8		121.6 (3)
O1	C9	C8		116.9 (3)	O1	C9	C8		116.6 (3)
C10'	C9'	O1'	1.394 (5)	121.9 (3)	C10'	C9'	O1'	1.389 (5)	121.2 (3)
C10'	C9'	C8'		121.1 (3)	C10'	C9'	C8'		121.7 (3)
O1'	C9'	C8'		116.7 (3)	O1'	C9'	C8'		117.0 (3)
C4	C10	C5		123.9 (3)	C4	C10	C5		124.2 (3)
C4	C10	C9		118.6 (3)	C4	C10	C9		118.1 (3)
C5	C10	C9		117.4 (3)	C5	C10	C9		117.6 (3)
C4'	C10'	C5'		124.3 (3)	C4'	C10'	C5'		124.8 (3)
C4'	C10'	C9'		117.7 (3)	C4'	C10'	C9'		117.5 (3)
C5'	C10'	C9'		117.8 (3)	C5'	C10'	C9'		117.5 (3)
C12	C11	O11	1.523 (6)	106.1 (3)	C12	C11	O11	1.517 (6)	106.0 (3)
C12	C11	C6		111.3 (3)	C12	C11	C6		111.2 (3)
O11	C11	C6		111.5 (3)	O11	C11	C6		110.4 (3)
C12'	C11'	O11	1.511 (6)	106.6 (3)	C12'	C11'	O11	1.513 (6)	107.1 (3)
C12'	C11'	C6'		112.3 (3)	C12'	C11'	C6'		111.9 (3)
O11	C11'	C6'		111.4 (3)	O11	C11'	C6'		110.8 (3)

anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1, and the bond lengths and angles in Table 2.* Ther-

mal-ellipsoid drawings of molecules *A* and *B* with the atomic labelling schemes are shown in Figs. 1 and 2, respectively.

Related literature. An investigation of the aerial parts of *Ageratina asenii* (Astereaceae, tribe Eupatorieae) afforded eight chromenes and one *p*-hydroxyacetophenone derivative (Fang, Yu & Mabry, 1988). One of these compounds was reported to be '(+)-encecanescin', a skeletal type previously reported by Bohlmann, Tsankova, Jakupovic, King & Robinson (1983). The NMR analysis of the (+)-encecanescin sample by Fang *et al.* (1988) suggested previously

* Tables of anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, crystallographic data and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52831 (65 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

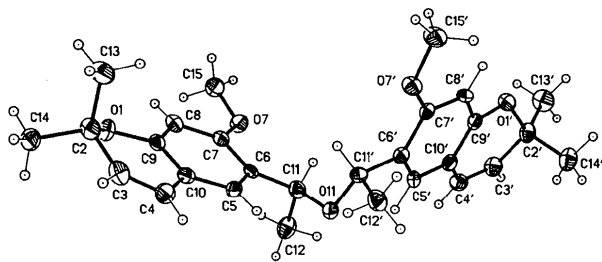


Fig. 1. Molecular structure of *A*, with 50% probability ellipsoids, showing the atomic numbering scheme.

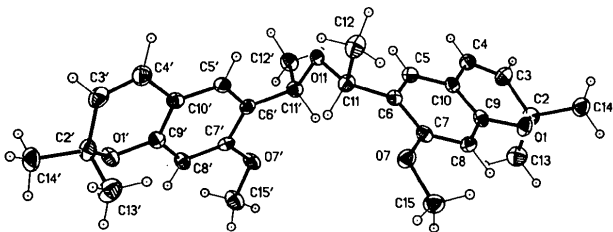


Fig. 2. Molecular structure of *B*, with 50% probability ellipsoids, showing the atomic numbering scheme.

reported stereochemistries should be revised. Surprisingly, however, the available enecanescin material was found to crystallize as a racemic mixture. It is not known at this time if racemization of the (+) isomer occurred after isolation of the material from

the plant or if preferential crystallization of the racemic mixture occurs from an unequal naturally occurring mixture of the (+) and (−) isomers in which the (+) isomer is dominant.

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Structure of (*E,E*)-1,4-Bis(4-pyridyl)-1,3-butadiene Methanol Solvate

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Abstract. C₁₄H₁₂N₂·0.5CH₄O, *M_r* = 224.28, monoclinic, *P*_{2₁}/*n*, *a* = 6.201 (3), *b* = 7.7111 (9), *c* = 12.975 (3) Å, β = 95.68 (3)°, *V* = 617.4 Å³, *Z* = 2, *D_x* = 1.20 Mg m^{−3}, λ(Mo *K*α) = 0.71069 Å, μ = 0.081 mm^{−1}, *F*(000) = 238, *T* = 293 K, *R* = 0.067 for 484 unique observed reflections. The angle between the best-squares planes through the phenyl groups and the butadiene chain is 7.0°. The alternation of short and long C—C distances in the chain [C(3)—C(6) = 1.450 (7), C(6)—C(7) = 1.337 (7), C(7)—C(7′) = 1.44 (1) Å] shows a partial delocalization of the π electrons along this chain.

Experimental. Poorly diffracting crystals were obtained by slow cooling of a water/methanol solution. Lath-shaped crystal, 0.55 × 0.20 × 0.15 mm.

Enraf–Nonius CAD-4F diffractometer with graphite-monochromated Mo *K*α radiation. Unit-cell parameters were refined by least-squares fitting procedure using 25 reflections with 22 < 2θ < 50°. For data collection, θ–2θ scans, 4 ≤ 2θ ≤ 50°, −7 ≤ *h* ≤ 7, 0 ≤ *k* ≤ 9, 0 ≤ *l* ≤ 15, two standard reflections used for scaling the data, no significant crystal movement or decay, no absorption correction, 1309 reflections, giving 484 with *I* ≥ 3σ(*I*) for refinements. Corrections for Lorentz and polarization effects were applied. The structure was determined by direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980).

After isotropic refinement, the Fourier peak at 0.65 Å from the second inversion center was attributed to a disordered methanol molecule with an