

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

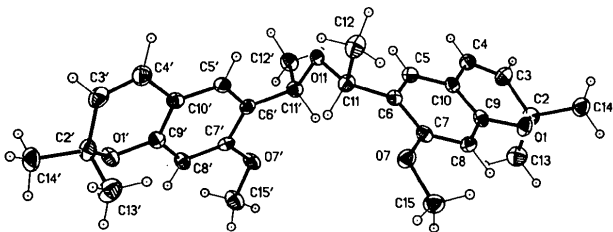


Fig. 2. Molecular structure of *B*, with 50% probability ellipsoids, showing the atomic numbering scheme.

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.

Funding for this project was supplied by the Robert A. Welch Foundation (F-017 to SHS and F-130 to TJM) and the National Institutes of Health (Grant GM-35710 to TJM).

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*Acta Cryst.* (1990). **C46**, 1566–1567

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

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(Received 18 December 1989; accepted 19 March 1990)

**Abstract.** C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>·0.5CH<sub>4</sub>O, *M<sub>r</sub>* = 224.28, monoclinic, *P*<sub>2</sub><sub>1</sub>/*n*, *a* = 6.201 (3), *b* = 7.7111 (9), *c* = 12.975 (3) Å, β = 95.68 (3)°, *V* = 617.4 Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.20 Mg m<sup>−3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.081 mm<sup>−1</sup>, *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

Table 1. Atomic coordinates and equivalent isotropic temperature factors for the non-H atoms with e.s.d.'s in parentheses (atomic labeling as in Fig. 1)

X refers to the equally distributed C and O atoms of the methanol molecule with an overall occupation factor of 0.5.

$$U_{eq} = (U_1 U_2 U_3)^{1/3}$$

	x	y	z	$U_{iso}$
N(1)	-0.161 (1)	0.4265 (8)	0.1443 (3)	0.0832
C(1)	0.036 (1)	0.497 (1)	0.1517 (5)	0.0861
C(2)	0.1738 (9)	0.5019 (8)	0.2416 (5)	0.0720
C(3)	0.1121 (8)	0.4268 (7)	0.3315 (4)	0.0579
C(4)	-0.0931 (9)	0.3505 (8)	0.3244 (5)	0.0700
C(5)	-0.217 (1)	0.3550 (9)	0.2297 (6)	0.0783
C(6)	0.2469 (9)	0.4236 (8)	0.4294 (4)	0.0641
C(7)	0.4367 (9)	0.5047 (8)	0.4504 (4)	0.0607
X(8)	0.447 (2)	0.440 (1)	0.0216 (9)	0.099 (4)

Table 2. Bond lengths (Å) and bond angles (°)

N(1)—C(1)	1.334 (8)	C(3)—C(6)	1.450 (7)
N(1)—C(5)	1.314 (8)	C(4)—C(5)	1.382 (9)
C(1)—C(2)	1.375 (8)	C(6)—C(7)	1.337 (7)
C(2)—C(3)	1.389 (7)	C(7)—C(7')	1.44 (1)
C(3)—C(4)	1.397 (7)	C(8)—O(8)	1.29 (2)
C(5)—N(1)—C(1)	115.2 (6)	C(6)—C(3)—C(4)	119.5 (6)
C(2)—C(1)—N(1)	124.0 (6)	C(5)—C(4)—C(3)	118.0 (6)
C(3)—C(2)—C(1)	120.1 (6)	C(4)—C(5)—N(1)	126.2 (6)
C(4)—C(3)—C(2)	116.4 (5)	C(7)—C(6)—C(3)	126.2 (6)
C(6)—C(3)—C(2)	124.0 (5)	C(7)—C(7')—C(6)	123.3 (7)

occupation of 0.5; the presence and the amount of this molecule were confirmed by NMR and chemical analysis. In order to take into account the equal distribution of C and O atoms at this position, this peak was refined with the nitrogen scattering factors. H atoms were placed in calculated positions (C—H 0.99 Å) after each refinement cycle, except those of the disordered atoms which were ignored. Full-matrix least-squares refinement (CRYSTALS; Watkin, Carruthers & Betteridge, 1985) on 78 parameters. Unit weights were used throughout. At the end of the refinement the maximum least-squares shift to e.s.d. was 0.01, the minimum and maximum

*Acta Cryst.* (1990). **C46**, 1567–1569

## Structure of 3,3',3''-Nitrilotripropionamide

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(Received 26 October 1989; accepted 2 March 1990)

**Abstract.**  $C_9H_{18}N_4O_3$ ,  $M_r = 230.3$ , rhombohedral,  $R3c$ ,  $a = 8.338 (2) \text{ \AA}$ ,  $\alpha = 85.27 (2)^\circ$ ,  $V =$

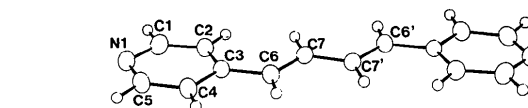


Fig. 1. Thermal ellipsoids drawn at the 30% probability level, excepting those of H which have  $U_{iso} = 0.025 \text{ \AA}^2$  for clarity. The atoms with a superscript ' are related to those without a superscript through an inversion center.

values of the residual electron density were  $-0.6$  and  $0.3 \text{ e \AA}^{-3}$ , respectively, and  $R = 0.067$ ,  $wR = 0.060$ . Final positional parameters for the non-H atoms are listed in Table 1,\* and bond lengths and bond angles in Table 2. An ORTEP (Johnson, 1976) drawing of the centrosymmetric molecule is shown in Fig. 1.

**Related literature.** The title compound has been synthesized in the course of a study of electron transfer through  $\alpha,\omega$  dipyridylpolyenes. The synthesis and spectral data have been described by Woitellier, Launay & Spangler (1989).

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, all bond angles and details of selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52864 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$574.1 (4) \text{ \AA}^3$ ,  $Z = 2$ ,  $D_m$  (by flotation) =  $1.36 (1)$ ,  $D_x = 1.332 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 0.09 \text{ mm}^{-1}$ ,  $F(000) = 248$ ,  $T = 173 (5) \text{ K}$ ,  $R = 0.0198$  for 664 observed reflections. The central nitrogen