C6—N1—C2	114.4 (3)	C4—C3—C2	106.3 (3)
C6—N1—C5	114.8 (3)	C5—C4—C3	106.6 (4)
C2—N1—C5	104.8 (3)	C4—C5—N1	103.0 (3)
N1—C2—C3	104.2 (3)	O1—C7—O2	127.2 (4)
N1—C2—C7	111.6 (3)	O1—C7—C2	117.3 (4)
C3—C2—C7	114.1 (3)	O2—C7—C2	115.5 (3)

H atoms attached to N and O were located on a difference electron-density map and their coordinates refined with $U_{\rm iso} = 1.3U_{\rm eq}$ of parent atom.

Data collection: *XSCANS* (Siemens, 1993). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1308). Services for accessing these data are described at the back of the journal.

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n-Butyl α-Cyano-4-[2-(4-pyridyl)ethenyl]cinnamate†

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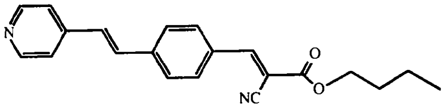
Abstract

The crystal structure of the title compound, $C_{21}H_{20}N_2O_2$, is compared with related compounds. There are two crystallographically independent molecules, which form two respective stacking columns along the *a* axis. Neither stacking modes are favorable for a [2+2] photopolymerization reaction.

Comment

The crystal structures of a number of alkyl esters of α-cyano-4-[2-(4-pyridyl)ethenyl]cinnamates have been analyzed in order to correlate their structures with their photoreactivity. For the methyl ester, photodimerization occurred between pyridyl-side olefinic groups of neighboring molecules. Oligomerization was observed on further photo-irradiation. For the ethyl and *n*-propyl homologs, however, dimerization occurred between the ester-side olefinic groups. Although the reaction terminated at the dimer formation for the *n*-propyl ester, the dimer of the ethyl ester gave a polymer on further irradiation (Maekawa, Kato, Saigo, Hasegawa & Ohashi, 1991; Hasegawa & Hashimoto, 1992; Hasegawa, 1995). This paper reports the structure of the *n*-butyl ester, (I). Recently, preliminary experiments on polymer formation have been performed and a variety of oligomeric compounds obtained. Although the products were investigated thoroughly, no high polymeric compounds were obtained.



(I)

There are two crystallographically independent molecules, *A* and *B*, both of which form infinite columns, *A* and *B*, along the *a* axis. The molecular structures

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† Alternative name: *n*-butyl 2-cyano-3-{4-[2-(4-pyridyl)ethenyl]phenyl}propenoate.

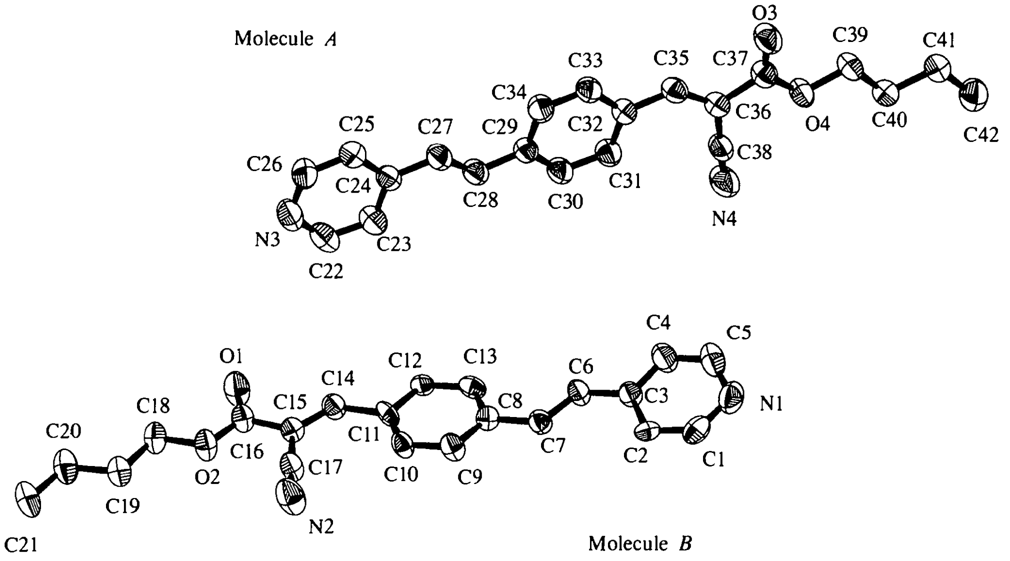

Fig. 1. *ORTEPII* (Johnson, 1976) drawing of the molecular structures and numbering systems of molecules *A* and *B* showing 50% probability displacement ellipsoids.

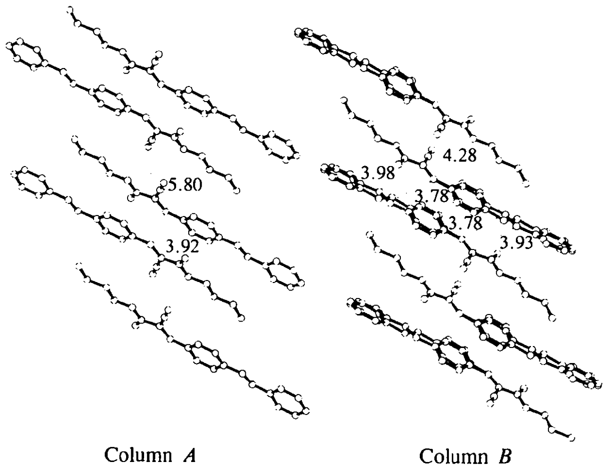

Fig. 2. Stacking patterns of columns *A* and *B*. The C—C distances (Å) between the double bonds are given.

and numbering schemes are shown in Fig. 1. The packing patterns of the *A* and *B* columns are different from each other, as shown in Fig. 2, in which the intermolecular distances between the ethylenic double bonds are given. In column *A*, the dimerization between the ester-side olefinic groups would easily occur easily on irradiation, considering the threshold value of 4.2 Å for the reaction (Schmidt, 1964, 1971). However, the oligomerization or polymerization may be difficult since the ethylenic double bonds of neighboring molecules are far from the pyridyl-side olefinic group. In column *B*, the pyridyl-side double bond is disordered. The intermolecular distances between the double bonds are within the threshold value. One of the disordered conformations is parallel and favorable for [2+2] cycloaddition. This indicates that the cyclic dimer would be produced. Another possibility is that the trimer may be produced since the distance between the ester-side double bonds (4.28 Å) is very close to the threshold value. In either case, polymerization in the crystal of the *n*-butyl ester is not very likely, which is consistent with the preliminary result. The dimers or trimers produced would be converted to various oligomers *via* non-topochemical reactions.

Experimental

The title compound was prepared as described previously (Ichimura & Watanabe, 1982) and recrystallized from a benzene solution.

Crystal data

$C_{21}H_{20}N_2O_2$
$M_r = 332.401$
Triclinic
$P\bar{1}$
$a = 9.072\,(2)$ Å
$b = 26.855\,(8)$ Å
$c = 8.135\,(2)$ Å
$\alpha = 97.419\,(6)^\circ$
$\beta = 110.80\,(2)^\circ$
$\gamma = 83.564\,(13)^\circ$
$V = 1832.5\,(9)$ Å^3
$Z = 4$
$D_x = 1.2048$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from all reflections
$\mu = 0.078$ mm^{-1}
$T = 293\,(2)$ K
Plate
0.3 × 0.2 × 0.03 mm
Yellow

Data collection

Rigaku R-AXIS IICS diffractometer

3934 reflections with $I > 2\sigma(I)$

Area detector scans
Absorption correction: none
15 309 measured reflections
7744 independent reflections

$R_{int} = 0.053$
$\theta_{max} = 27.70°$
$h = -10 \rightarrow 9$
$k = -34 \rightarrow 35$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.156$
$S = 1.000$
7740 reflections
539 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 0.1876P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.160$ e Å^{-3}
$\Delta\rho_{min} = -0.184$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected intermolecular distances (Å)*

C14···C7^{i}	3.780 (3)	C35···C36^{iii}	3.925 (3)
C14···C15^{ii}	4.273 (3)	C35···C36^{iv}	5.798 (3)
C15···C6^{i}	3.936 (3)	C36···C35^{iii}	3.925 (3)
C15···C14^{ii}	4.273 (3)	C36···C35^{iv}	5.798 (3)

Symmetry codes: (i) $1 - x, 2 - y, 2 - z$; (ii) $-x, 2 - y, 1 - z$; (iii) $1 - x, 1 - y, 2 - z$; (iv) $2 - x, 1 - y, 2 - z$.

The data collection conditions by R-AXIS IICS are as follows: number of frames measured 19, oscillation range per frame 10.0°, exposure time per frame 45 min and crystal-to-detector distance 143.0 mm.

Data collection: *OSCILL* in *R-AXIS IICS Software* (Rigaku Corporation, 1994). Cell refinement: *SCALE* in *R-AXIS IICS Software*. Data reduction: *SCALE* in *R-AXIS IICS Software*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994) in *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1061). Services for accessing these data are described at the back of the journal.

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β-D-3′-Deoxyadenosine (Cordycepin)

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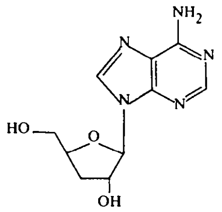
Abstract

In the title compound, $C_{10}H_{13}N_5O_3$, the nucleoside adopts an *anti* conformation with C3′-*endo* sugar puckering. The overall molecular conformation is similar to that of both adenosine and 2′-deoxyadenosine.

Comment

3′-Deoxynucleosides could function as possible inhibitors of RNA synthesis and, thus, may be useful in the treatment of cancer, as well as viral and other infections (Bazin & Chattopadhyaya, 1985; Rainny & Santi, 1983). As part of our program to study their molecular conformations (Karthe, Gautham, Kumar & Katti, 1997), we report here the structure of 3′-deoxyadenosine, (I).



(I)

An *ORTEP*92 (Vicković, 1994) view of the molecule with the atom-numbering scheme is shown in Fig. 1. The conformation of the base with respect to the ribose moiety is *anti* with the glycosidic torsion angle χ_{CN}, O4′—C1′—N9—C4, equal to −172.5 (6)°. In α-D-2′-deoxyadenosine (Watson, Sutor & Tollin, 1965) and also in β-D-adenosine (Lai & Marsh, 1972), the conformation is *anti*. The sugar pucker in the present structure is C3′-*endo*, as observed in adenosine. The pseudorotation phase angle (*P*) and the maximum amplitude of puckering (τ_M) are 4.4 and 38.4°, respectively. In 2′-deoxyadenosine, the sugar pucker is C3′-*exo*. The torsion angles φ_{OO} (O5′—C5′—C4′—O4′) and φ_{OC} (O5′—C5′—C4′—C3′) are 57.3 (8) and 175.5 (6)°, respectively. The conformation about the C4′—C5′ bond

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