Siemens Analytical X-ray Instruments, Inc. (1989). *P3/PC Data Collection*. Version 4.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wahl, P., Krieger, C., Schweitzer, D. & Staab, H. A. (1984). *Chem. Ber.* **117**, 260–276.

Acta Cryst. (1993). **C49**, 833–834

Structure of the Photostable Form of *p*-Nitrocinnamic Acid

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(*Received* 6 *May* 1992; *accepted* 13 *October* 1992)

Abstract

The molecule is planar to within 0.08 (2) Å. The molecules form a centrosymmetric dimer through O—H···O hydrogen bonds between the carboxylic groups. The dimers are stacked along **b** with an interplanar distance of 3.374 (2) Å. The C=C double bonds of the nearest neighbors are related by a **b** translation.

Comment

The structure of the photostable form of *p*-nitrocinnamic acid has been determined as part of studies into solid-state photoreactions (Iwamoto, Kashino & Haisa, 1989). The lattice parameters and space groups of the photostable and photoreactive forms of the title compound have been reported by Schmidt (1964). The crystals used in the present study were obtained by slow evaporation from an ethanol solution. The crystal structure is similar to that of photoreactive *p*-formylcinnamic acid (Nakanishi, Hasegawa & Mori, 1985). However, the length of the *b* axis is 0.206 Å longer than in *p*-formylcinnamic acid.

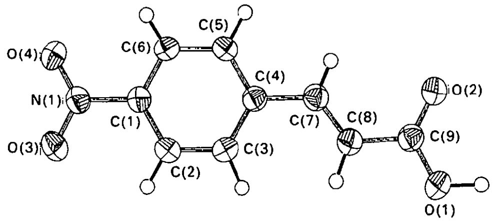

Fig. 1. The thermal ellipsoids with atomic numbering. Ellipsoids are drawn at 50% probability for the non-H atoms; the H atoms are represented as spheres equivalent to $B = 1.0$ Å^2.

Experimental

Crystal data

$C_9H_7NO_4$
$M_r = 193.16$
Monoclinic
$P2_1/a$
$a = 27.729$ (5) Å
$b = 5.0311$ (7) Å
$c = 6.105$ (1) Å
$\beta = 99.57$ (2)°
$V = 839.9$ (3) Å^3
$Z = 4$
$D_x = 1.527$ Mg m^{-3}
$D_m = 1.50$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 18 reflections
$\theta = 8$–$18°$
$\mu = 1.06$ mm^{-1}
$T = 295$ K
Prismatic
0.28 × 0.28 × 0.15 mm
Colorless

Data collection

Rigaku AFC-5 diffractometer
ω-2θ scans
1548 measured reflections
1329 independent reflections
1284 observed reflections
$[F_o > 1\sigma(F_o)]$
$R_{\rm int} = 0.011$ for 138 *hk*0 reflections

$\theta_{\max} = 62.5°$
$h = -31 \rightarrow 30$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 6$
3 standard reflections
monitored every 57 reflections
intensity variation: 1%

Refinement

Refinement on F
Final $R = 0.045$
$wR = 0.055$
$S = 2.91$
1284 reflections
156 parameters
All H-atom parameters refined

$w = 1/[\sigma(F_o)^2 - 0.4488|F_o| + 0.0228|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.23$
$\Delta\rho_{\max} = 0.2$ e Å^{-3}
$\Delta\rho_{\min} = -0.3$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{\rm eq} = (4/3)\sum_i \beta_{ii}/a_{ii}^{*2}$.

	x	y	z	$B_{\rm eq}$
O(1)	0.50023 (4)	1.3330 (2)	0.2491 (2)	4.57 (5)
O(2)	0.45399 (4)	1.2762 (2)	−0.0802 (2)	4.36 (5)
O(3)	0.30860 (4)	−0.0074 (2)	0.6531 (2)	4.33 (5)
O(4)	0.26096 (4)	−0.0400 (2)	0.3379 (2)	4.05 (5)
N(1)	0.29527 (4)	0.0591 (2)	0.4595 (2)	3.09 (5)
C(1)	0.32246 (5)	0.2730 (3)	0.3712 (2)	2.79 (5)
C(2)	0.36232 (5)	0.3803 (3)	0.5065 (2)	3.43 (6)
C(3)	0.38838 (5)	0.5765 (3)	0.4236 (2)	3.55 (6)
C(4)	0.37501 (5)	0.6666 (3)	0.2051 (2)	2.89 (6)
C(5)	0.33429 (5)	0.5554 (3)	0.0745 (2)	3.21 (6)
C(6)	0.30776 (5)	0.3572 (3)	0.1563 (2)	3.12 (6)
C(7)	0.40240 (5)	0.8759 (3)	0.1126 (2)	3.22 (6)
C(8)	0.44095 (5)	1.0036 (3)	0.2189 (2)	3.60 (7)
C(9)	0.46586 (5)	1.2160 (3)	0.1198 (2)	3.41 (6)

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0108-2701/93/040833-02$06.00

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bond geometry* (Å, °)

O(1)—C(9)	1.276 (1)	C(2)—C(3)	1.369 (3)
O(2)—C(9)	1.248 (1)	C(3)—C(4)	1.399 (2)
O(3)—N(1)	1.225 (1)	C(4)—C(5)	1.387 (2)
O(4)—N(1)	1.212 (1)	C(4)—C(7)	1.465 (1)
N(1)—C(1)	1.467 (1)	C(5)—C(6)	1.381 (2)
C(1)—C(2)	1.375 (2)	C(7)—C(8)	1.321 (2)
C(1)—C(6)	1.374 (1)	C(8)—C(9)	1.457 (2)
O(1)—C(9)—O(2)	122.8 (1)	C(1)—C(6)—C(5)	118.5 (1)
O(1)—C(9)—C(8)	116.0 (1)	C(2)—C(3)—C(4)	120.9 (2)
O(2)—C(9)—C(8)	121.2 (1)	C(2)—C(1)—C(6)	122.2 (1)
O(3)—N(1)—O(4)	124.0 (1)	C(3)—C(4)—C(5)	118.6 (1)
O(3)—N(1)—C(1)	117.6 (1)	C(3)—C(4)—C(7)	121.9 (1)
O(4)—N(1)—C(1)	118.4 (1)	C(4)—C(5)—C(6)	121.0 (2)
N(1)—C(1)—C(2)	118.6 (1)	C(4)—C(7)—C(8)	126.1 (1)
N(1)—C(1)—C(6)	119.2 (1)	C(5)—C(4)—C(7)	119.5 (1)
C(1)—C(2)—C(3)	118.8 (2)	C(7)—C(8)—C(9)	123.6 (2)

D	A	$D\cdots A$	$H\cdots A$	$D—H\cdots A$
O(1)	O(2^{i})	2.641 (1)	1.62 (3)	174 (3)

Symmetry code: (i) $1-x, 3-y, -z$.

D_m was measured by flotation in aqueous KI. The scan width was $(1.2+0.15\tan\theta)^\circ$ in ω with a scan rate of 4° min^{-1} in ω and background counts for 4 s on each side of every scan. Refinement was by block-diagonal least-squares methods. Data collection and cell refinement: Rigaku AFC-5 software. Data reduction: *NTDATAIN* (Tanaka, 1979). Program used to solve structure: *RSSFR*-5 (Sakurai, 1967). Program used to refine structure: *HBLS*-V (Ashida, 1973). Software used to prepare material for publication: *DAPH* (Ashida, 1973), *MOLCON* (Fujii, 1979), *ORTEPII* (Johnson, 1971). Computations were carried out at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University, and at the Okayama University Computer Center.

The authors thank the Research Center for Protein Engineering, Institute for Protein Research, Osaka University, for the use of their facilities.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55748 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1006]

References

Ashida, T. (1973). *HBLS*-V and *DAPH. The Universal Crystallographic Computing System – Osaka.* The Computation Center, Osaka Univ., Japan.

Fujii, S. (1979). *MOLCON. The Universal Crystallographic Computing System – Osaka.* The Computation Center, Osaka Univ., Japan.

Iwamoto, T., Kashino, S. & Haisa, M. (1989). *Acta Cryst.* C**45**, 1753–1758.

Johnson, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

Nakanishi, H., Hasegawa, M. & Mori, T. (1985). *Acta Cryst.* C**41**, 70–71.

Sakurai, T. (1967). *RSSFR-5. The Universal Crystallographic Computing System* (I). The Crystallographic Society of Japan, Tokyo.

Schmidt, G. M. J. (1964). *J. Chem. Soc.* pp. 2014–2021.

Tanaka, N. (1979). *NTDATAIN*. Program for data reduction. The Research Center for Protein Engineering, Institute for Protein Research, Osaka Univ., Japan.

Acta Cryst. (1993). C**49**, 834–837

2-Amino-5-nitropyridinium Monohydrogenphosphite

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(*Received* 25 *June* 1992; *accepted* 22 *October* 1992)

Abstract

The crystal structure of this new 2-amino-5-nitropyridinium salt consists of centrosymmetric double layers built up with $C_5H_6N_3O_2^+$ cations and $H_2PO_3^-$ phosphite anions. The main feature of this stacking is the presence of centrosymmetric $(H_4P_2O_6)^{2-}$ clusters holding both layers together through relatively strong hydrogen bonds. Weaker hydrogen bonds maintain the cohesion of the organic–inorganic arrangement within a layer. Direct bonding exists neither between the organic entities, nor between the double layers. The dimension charge compromise of the anionic group is not favourable for inducing a polar packing of cations which would be necessary to originate nonlinear optical properties.

Comment

The design of organic–inorganic polar crystals for quadratic nonlinear optical applications is today oriented and supported by two main observations: (i) the organic molecules containing π-electron systems asymmetrized by electron donor–acceptor groups are highly polarizable entities in which problems of transparency and crystal growth may arise from their molecular crystal packing; (ii) the ionic inorganic host-matrices are able to increase the cohesion of packing, to shift the transparency of organic entities towards blue wavelengths and to originate the acentricity of packing. This approach has been applied to the 2-amino-5-nitropyridinium cation encapsulated in various anionic inorganic subnetworks (Masse, Bagieu-Beucher, Pécaut, Levy & Zyss, 1992).

The attempt using $(H_2PO_4^-)_n$, $(H_2AsO_4^-)_n$ polymeric anions has been successful with the crystalliza-

0108-2701/93/040834-04$06.00