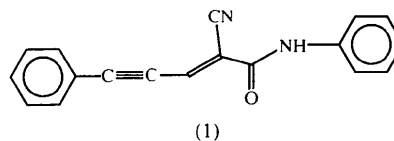


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## Comment

In continuation of systematic study of the structure and reactivity of derivatives of 2-cyano-pentadien-2,4-oic acid (Borbulevych *et al.*, 1998; Golding *et al.*, 1999; Khrustalev *et al.*, 1996), we performed an X-ray study of a similar compound, (1), that contains a triple C≡C bond in an aliphatic chain. The interest in such compounds is explained by their ability to undergo anionic polymerization and copolymerization under mild conditions. In addition, topochemical reactions can occur in these compounds.



*Acta Cryst.* (1999). **C55**, 1019–1020

## The *N*-phenylamide of 2-cyano-5-phenylpent-2-en-4-ynoic acid

OLEG YA. BORBULEVYCH,<sup>a</sup> IGOR R. GOLDING,<sup>a</sup> ANDREY B. KHARITONKIN,<sup>b</sup> OLEG V. SHISHKIN<sup>c</sup> AND YURIY G. GOLOBOV<sup>a</sup>

<sup>a</sup>*A. N. Nesmeyanov Institute of Organoelement Compounds of the Russian Academy of Sciences, 28 Vavilov Str., Moscow 117813, Russia*, <sup>b</sup>*Higher Chemical College of the Russian Academy of Science, 9 Miusskaia Sq., Moscow 125190, Russia*, and <sup>c</sup>*Scientific Research Department of Alkali Halide Crystals, STC 'Institute for Single Crystals' of the National Academy of Sciences of Ukraine, 60 Lenina Ave., Khar'kov 310001, Ukraine. E-mail: oleg@xrlab.ineos.ac.ru*

(Received 6 January 1999; accepted 3 February 1999)

## Abstract

The central 2-cyano-carboxyaminoprop-2-enylic fragment of the title compound, 2-cyano-5-*N*-diphenylpent-2-en-4-ynamide, C<sub>18</sub>H<sub>12</sub>N<sub>2</sub>O, is planar. The phenyl rings are rotated with respect to this plane. Molecules are linked by N—H···O hydrogen bonds and form stacks by translation along the crystallographic *c* axis.

Bond lengths in molecule (1) do not differ from other derivatives of 2-cyanopentadien-2,4-oic acid (Borbulevych *et al.*, 1998; Golding *et al.*, 1999).

The 2-cyano-carboxyaminoprop-2-enylic fragment (N2/C1/O1/C2/C3/C6/N1) in (1) is planar despite the shortened intramolecular contacts H2···C6 2.44 (2) Å (van der Waals radii sum is 2.87 Å; Zefirov & Zorky, 1989) and O1···H3 2.40 (2) Å. The maximum deviations from the least-squares mean plane passing through all non-H atoms of this fragment is 0.038 (1) Å for O1 atom. The C13···C18 and C7···C12 phenyl rings are twisted with respect to 2-cyano-carboxyaminoprop-2-enylic fragment [angles between planes of these fragments are −40.55 (8) and 25.73 (9)°, respectively]. For the C13–C18 ring, this may be linked to short intramolecular contacts O1···C14 2.887 (2) and C1···H14 2.81 (2) Å.

In the crystal phase, molecules of (1) are linked by N2—H2···O1<sup>i</sup> hydrogen bonds [symmetry code: (i)  $x, \frac{1}{2} - y, z - \frac{1}{2}$ ]. Thus, molecules of (1) do not form centrosymmetric dimers in the crystal due to intermolecular hydrogen bonds unlike previously investigated related compounds (Borbulevych *et al.*, 1998; Golding *et al.*, 1999), but form stacks along the crystallographic *c* axis.

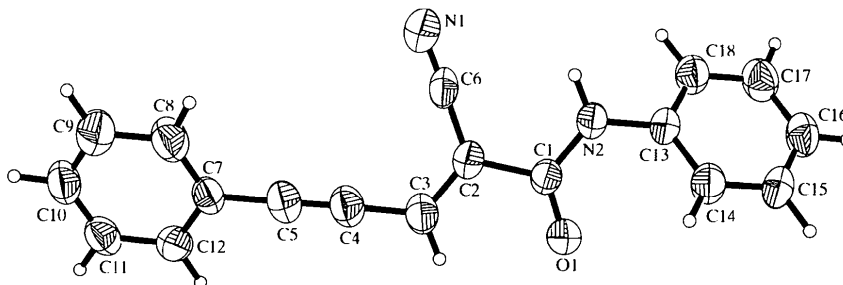


Fig. 1. View of the title compound (1). The non-H atoms are shown with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as small circles of an arbitrary radius for clarity.

## Experimental

Crystals were obtained by isothermic evaporation of solvent from a solution of (1) in hexane.

### Crystal data

C <sub>18</sub> H <sub>12</sub> N <sub>2</sub> O	Mo K $\alpha$ radiation
$M_r = 272.30$	$\lambda = 0.71073 \text{ \AA}$
Monoclinic	Cell parameters from 24 reflections
$P2_1/c$	$\theta = 10\text{--}11^\circ$
$a = 11.662 (5) \text{ \AA}$	$\mu = 0.079 \text{ mm}^{-1}$
$b = 14.323 (7) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 8.700 (4) \text{ \AA}$	Needle
$\beta = 94.57 (4)^\circ$	$0.60 \times 0.25 \times 0.20 \text{ mm}$
$V = 1448.5 (11) \text{ \AA}^3$	Yellow
$Z = 4$	
$D_x = 1.249 \text{ Mg m}^{-3}$	
$D_m$ not measured	

### Data collection

Siemens P3/PC diffractometer	$R_{\text{int}} = 0.03$
$\theta/2\theta$ scans	$\theta_{\text{max}} = 27.06^\circ$
Absorption correction: none	$h = -14 \rightarrow 14$
3381 measured reflections	$k = -18 \rightarrow 0$
3171 independent reflections	$l = 0 \rightarrow 11$
1683 reflections with $I > 2\sigma(I)$	2 standard reflections every 98 reflections
	intensity decay: 5%

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.113$	$(\Delta/\sigma)_{\text{max}} = -0.001$
$S = 1.092$	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
3113 reflections	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
226 parameters	Extinction correction: none
All H-atom parameters refined	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N1—C6	1.144 (2)	C2—C3	1.342 (3)
N2—C1	1.352 (2)	C2—C6	1.421 (3)
N2—C13	1.424 (2)	C3—C4	1.414 (3)
O1—C1	1.214 (2)	C4—C5	1.189 (2)
C1—C2	1.498 (2)	C5—C7	1.439 (2)
C1—N2—C13	123.19 (15)	C6—C2—C1	122.0 (2)
O1—C1—N2	124.08 (15)	C2—C3—C4	125.0 (2)
O1—C1—C2	119.8 (2)	C5—C4—C3	176.3 (2)
N2—C1—C2	116.05 (15)	C4—C5—C7	179.5 (2)
C3—C2—C6	120.0 (2)	N1—C6—C2	177.9 (2)
C3—C2—C1	117.9 (2)	C1—N2—C13—C14	40.55 (8)

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$N2\text{---}H2\cdots O1'$	0.93 (2)	2.14 (2)	2.985 (2)	152 (2)

Symmetry code: (i)  $x, \frac{1}{2} - y, z - \frac{1}{2}$ .

Data collection: P3 (Siemens, 1989). Cell refinement: P3. Data reduction: SHELXTL (Sheldrick, 1994). Program(s) used to solve structure: SHELXTL. Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL.

This work was supported by the Russian Foundation for Basic Research (grant Nos. 97-03-33783 and 96-15-97367) and International Fund 'Vidrodzhennya' (grant No. YSU 083067).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1318). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 1020–1023

## Study of a diastereoisomeric dihydrothiophene derivative at 150 K

MARÍA JESÚS DIÁNEZ,<sup>a</sup> MARÍA DOLORES ESTRADA,<sup>a</sup> AMPARO LÓPEZ-CASTRO<sup>b</sup> AND SIMEÓN PÉREZ-GARRIDO<sup>a</sup>

<sup>a</sup>Departamento de Física de la Materia Condensada, Universidad de Sevilla, Apartado 1065, 41080 Sevilla, Spain, and <sup>b</sup>Instituto de Ciencias de Materiales de Sevilla, CSIC, 41080 Sevilla, Spain. E-mail: dianez@cica.es

(Received 3 November 1998; accepted 19 January 1999)

## Abstract

The crystal and molecular structure of (4*R*,5*R*)-4-(1',2',3',4',5'-penta-*O*-acetyl-D-manno-pentitol-1-yl)-5-[*N*-(4-methoxyphenyl)carbamoyl]-2-(*N*-methylbenzylamino)-3-nitro-5-phenyl-4,5-dihydrothiophene {IUPAC name: 1-(2*R*,3*R*)-5-(*N*-benzyl-*N*-methylamino)-2-[*N*-(4-methoxyphenyl)carbamoyl]-4-nitro-2-phenyl-2,3-dihydro-3-thienyl]-1,2,3,4,5-penta-*O*-acetyl-D-manno-pentitol}, C<sub>41</sub>H<sub>45</sub>N<sub>3</sub>O<sub>14</sub>S, has been investigated at 150 K. The analysis of the crystal structure allows the definition of the absolute configuration of the products of the cycloaddition reaction between 3,5-diphenyl-2-(*N*-methylbenzylamine)-1,3-thiazolium-7-olate