

Monoclinic

 $P2_1/c$ $a = 3.9237 (1) \text{ \AA}$ $b = 11.9223 (2) \text{ \AA}$ $c = 17.7519 (3) \text{ \AA}$ $\beta = 97.034 (2)^\circ$ $V = 824.18 (3) \text{ \AA}^3$ $Z = 4$ $D_x = 2.022 (1) \text{ Mg m}^{-3}$ D_m not measured

Cell parameters from 2682 reflections

 $\theta = 2\text{--}25^\circ$ $\mu = 5.56 \text{ mm}^{-1}$ $T = 173 (2) \text{ K}$

Plate

 $0.50 \times 0.28 \times 0.08 \text{ mm}$

Colorless

The author would like to thank Dr Victor G. Young Jr of the University of Minnesota X-ray Diffraction Laboratory for collecting the data on the SMART system.

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BK1210). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Data collection

Siemens SMART area-detector diffractometer

 ω scans

Absorption correction:

 ψ scans (XPREP in SHELXTL; Sheldrick, 1994) $T_{\min} = 0.259$, $T_{\max} = 0.641$

4108 measured reflections

1519 independent reflections

1282 reflections with

 $I > 2\sigma(I)$ $R_{\text{int}} = 0.203$ (before absorption correction)

and 0.081 (after)

 $\theta_{\max} = 25.1^\circ$ $h = -4 \rightarrow 4$ $k = -14 \rightarrow 14$ $l = -20 \rightarrow 20$ Intensity decay: $< 1\%$

Refinement

Refinement on F $R = 0.046$ $wR = 0.056$ $S = 1.74$

1282 reflections

100 parameters

H atoms not refined

 $w = 4F^2/[\sigma^2(F^2) + (0.05F^2)^2]$ $(\Delta/\sigma)_{\max} = 0.02$ $\Delta\rho_{\max} = 1.02 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -1.35 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)Table 1. Selected geometric parameters ($\text{\AA}$, $^\circ$)

Br4—C4	1.894 (5)	C1—C7	1.449 (7)
Cl2—C2	1.729 (5)	C2—C3	1.394 (7)
Cl6—C6	1.723 (5)	C3—C4	1.384 (7)
N1—C7	1.154 (7)	C4—C5	1.392 (8)
C1—C2	1.391 (7)	C5—C6	1.390 (8)
C1—C6	1.399 (7)		
C2—C1—C6	118.5 (5)	C4—C5—C6	117.7 (5)
C1—C2—C3	121.1 (5)	C1—C6—C5	121.8 (5)
C2—C3—C4	118.6 (5)	N1—C7—C1	179.4 (5)
C3—C4—C5	122.4 (5)		

The data collection nominally covered more than a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω . The crystal-to-detector distance was 4.94 cm. Coverage of the unique set was approximately 98% complete to at least 25° in θ . Crystal decay was monitored by repeating the initial frames at the end of the data collection and analyzing the duplicate reflections.

Data collection: *ASTRO* (Siemens, 1995a). Cell refinement: *SAINT* (Siemens, 1995b). Data reduction: *SAINT*. Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Buerskens, 1984). Program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1985). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Acta Cryst. (1997). **C53**, 227–229

2,4-Dinitro-*trans*-cinnamic Acid

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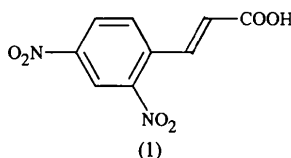
(Received 19 March 1996; accepted 3 October 1996)

Abstract

2,4-Dinitrocinnamic acid [3-(2,4-dinitrophenyl)prop-2-enoic acid], $\text{C}_9\text{H}_6\text{N}_2\text{O}_6$, crystallizes as a normal centrosymmetric carboxy dimer. Both $\text{O} \cdots \text{H}$ and $\text{C} \cdots \text{H}$ hydrogen bonds are found in the structure.

Comment

3,5-Dinitrocinnamic acid, (2), crystallizes as an O—H···O dimer wherein hydrogen-bonded molecules are related not by the more common inversion centre but by a twofold axis (Desiraju & Sharma, 1991). This has led us to investigate the structure of the isomeric 2,4-dinitrocinnamic acid, (1). (Some details of this structure were deposited as a footnote with an earlier publication: Desiraju & Sharma, 1991; CSD refcode KODJAO.) This previous study was based on refinement on *F* and included only 3 σ reflections. This paper presents the results of a refinement on *F*² and includes all reflections. Furthermore, the H atoms are now included in the refinement which is more relevant to the study of C—H···O hydrogen bonding.



Acid (1) crystallizes as a normal inversion symmetry O—H···O dimer. An *ORTEP*II (Johnson, 1976) plot of the molecular structure of (1) is shown in Fig. 1. The 2-nitro group and the styryl moiety are non-coplanar with the aromatic ring as expected (interplanar angles of 25.5 and 40°, respectively). The 2-nitro group does not effectively participate in C—H···O hydrogen bonding, possibly as a result of conformational inflexibility caused by steric hindrance. The overall C—H···O bonding in acid (1), however, is as good as in acid (2) [C···O, H···O and C—H···O ranges in acids (1) and (2) are 3.261(2)–3.629(2), 2.606(19)–2.900(20) Å and 120.8(18)–153.5(15)°, and 3.19–3.69, 2.56–2.99 Å and 123–160°, respectively]. A packing diagram of acid (1) is given in Fig. 2. While the C—H···O hydrogen bonds in acid (1) are as effective as those in acid (2), they do not seem to contradict the formation of an O—H···O centrosymmetric dimer as

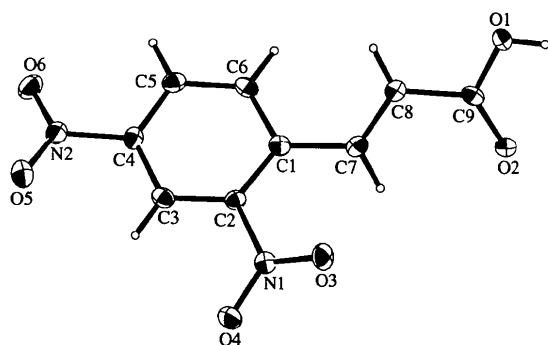


Fig. 1. An *ORTEP*II (Johnson, 1976) diagram of acid (1). Displacement ellipsoids are plotted at the 50% probability level. For clarity, H atoms are drawn as small circles.

was suggested in the case of acid (2) (Desiraju & Sharma, 1991). Such observations could be indicative of the absence and presence, respectively, in (1) and (2) of interference between different types of intermolecular forces. Such interference is a general issue in crystal engineering, which is the designed synthesis of a solid-state structure (Desiraju, 1995).

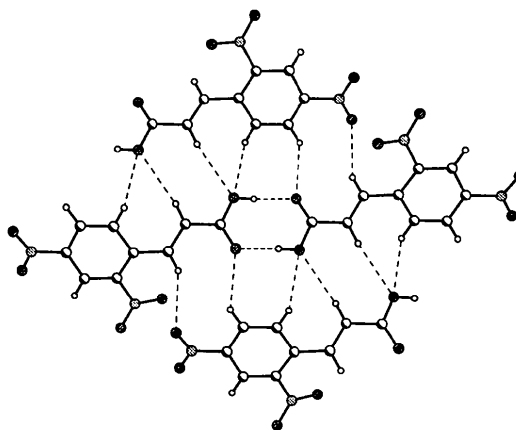


Fig. 2. The crystal structure of acid (1) approximately down [100], showing the O—H···O and C—H···O hydrogen bonds. The central pair of molecules is O—H···O hydrogen bonded. The two other molecules are C—H···O hydrogen bonded to the central pair. These C—H···O bonds involve alkenic and aromatic C—H groups.

Experimental

The title compound was prepared from the Perkin condensation of 2,4-dinitrobenzaldehyde with acetic anhydride and fused sodium acetate.

Crystal data

C₉H₆N₂O₆
M_r = 238.16
 Monoclinic
*P*2₁/*n*
a = 8.760 (9) Å
b = 7.869 (5) Å
c = 14.040 (10) Å
 β = 93.12 (7)°
V = 966.4 (14) Å³
Z = 4
D_x = 1.637 Mg m^{−3}
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 50 reflections
 θ = 2–25°
 μ = 0.141 mm^{−1}
T = 153 (2) K
 Irregular
 0.64 × 0.31 × 0.14 mm
 Light yellow

Data collection

Nicolet R3m diffractometer
 ω –2 θ scans
 Absorption correction: none
 1699 measured reflections
 1699 independent reflections
 1254 reflections with
 $I > 2\sigma(I)$

$\theta_{\max}$ = 24.99°
 h = −10 → 10
 k = 0 → 9
 l = 0 → 16
 3 standard reflections
 every 100 reflections
 intensity decay: none

RefinementRefinement on F^2 $R(F) = 0.040$ $wR(F^2) = 0.091$ $S = 1.024$

1699 reflections

178 parameters

All H atoms refined

 $w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.0374P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.014$ $\Delta\rho_{\max} = 0.169 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.244 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)Table 1. Selected geometric parameters ($\text{\AA}$, $^\circ$)

O1—C9	1.322 (3)	C3—C4	1.381 (3)
O1—H9	0.87 (3)	C3—H3	0.94 (2)
O2—C9	1.227 (3)	C4—C5	1.389 (3)
O3—N1	1.232 (2)	C4—N2	1.473 (3)
O4—N1	1.229 (2)	C5—C6	1.386 (3)
O5—N2	1.232 (2)	C5—H5	0.96 (2)
O6—N2	1.233 (2)	C6—H6	0.98 (2)
C1—C2	1.405 (3)	C7—C8	1.330 (3)
C1—C6	1.409 (3)	C7—H7	0.92 (2)
C1—C7	1.472 (3)	C8—C9	1.477 (3)
C2—C3	1.383 (3)	C8—H8	0.96 (2)
C2—N1	1.477 (3)		
C9—O1—H9	109.1 (18)	C5—C6—H6	119.0 (14)
C2—C1—C6	116.5 (2)	C1—C6—H6	119.4 (14)
C2—C1—C7	124.5 (2)	C8—C7—C1	123.2 (2)
C6—C1—C7	119.0 (2)	C8—C7—H7	120.6 (13)
C3—C2—C1	123.4 (2)	C1—C7—H7	116.1 (13)
C3—C2—N1	116.2 (2)	C7—C8—C9	120.2 (2)
C1—C2—N1	120.4 (2)	C7—C8—H8	122.5 (12)
C4—C3—C2	117.3 (2)	C9—C8—H8	117.2 (12)
C4—C3—H3	121.6 (14)	O2—C9—O1	124.0 (2)
C2—C3—H3	121.1 (14)	O2—C9—C8	123.0 (2)
C3—C4—C5	122.6 (2)	O1—C9—C8	113.0 (2)
C3—C4—N2	118.3 (2)	O4—N1—O3	124.0 (2)
C5—C4—N2	119.0 (2)	O4—N1—C2	117.7 (2)
C6—C5—C4	118.6 (2)	O3—N1—C2	118.3 (2)
C6—C5—H5	122.4 (13)	O5—N2—O6	124.2 (2)
C4—C5—H5	119.0 (13)	O5—N2—C4	117.6 (2)
C5—C6—C1	121.6 (2)	O6—N2—C4	118.2 (2)

Data collection: Nicolet system software. Cell refinement: Nicolet system software. Data reduction: Nicolet system software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL93*.

VRT thanks the DAE for fellowship support. We thank Professor W. T. Robinson, University of Canterbury, Christchurch, New Zealand, for X-ray data collection.

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BK1250). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 229–231

 $[\text{Ph}_3\text{PH}][\text{nido-B}_{11}\text{H}_{14}]$

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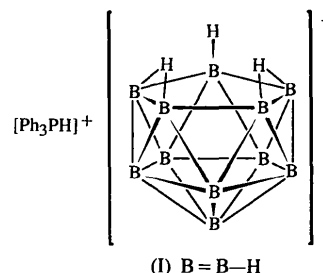
(Received 25 March 1996; accepted 23 October 1996)

Abstract

The structure of the tetradecahydro-*nido*-undecaborate(1−) anion, as its triphenylphosphonium salt, is reported ($\text{C}_{18}\text{H}_{16}\text{P}^+\text{B}_{11}\text{H}_{14}^-$). The borane anion has the expected icosahedral fragment geometry, with crystallographically-imposed mirror symmetry.

Comment

In the course of our synthetic and structural studies of metalla(hetero)boranes, we have isolated and structurally characterized the title salt, (I).



Although the borane anion, $[\text{B}_{11}\text{H}_{14}]^-$, has been known for over 30 years (Aftandilian, Miller, Parshall & Muettterties, 1962), its structure determination was only reported relatively recently (Getman, Krause & Shore, 1988) and the species continues to be the subject of

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