

Benzene-1,2,4,5-tetracarboxylic acid–*trans*-cinnamamide (1/2)

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Electronic paper

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Received 12 September 2000

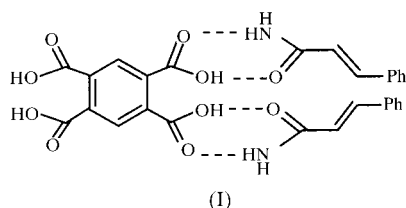
Accepted 27 September 2000

Data validation number: IUC0000271

In the title adduct, C₁₀H₆O₈·2C₉H₉NO, benzene-1,2,4,5-tetracarboxylic acid has a crystallographic twofold axis parallel to **b** and forms a flat zigzag chain along **c** linked by O—H···O cyclic hydrogen bonds with cinnamamide molecules.

Comment

The C=C bonds of the two neighbouring cinnamamide molecules of the title adduct, (I), adopt a twisted arrangement. The distance between the centres of the C13=C14 and C13ⁱ=C14ⁱ bonds is 4.722 (6) Å [symmetry code: (i) 1 - x, y, $\frac{1}{2}$ - z]. Both H atoms of the cinnamamide NH₂ group are involved in intermolecular N—H···O hydrogen bonds, forming a two-dimensional sheet.



Experimental

Crystals of the title compound were grown by slow evaporation of a 2-propanol solution of a mixture of benzene-1,2,4,5-tetracarboxylic acid and *trans*-cinnamamide (1:2).

Crystal data

C₁₀H₆O₈·2C₉H₉NO
M_r = 548.51
Monoclinic, *C*2/*c*
a = 7.163 (2) Å
b = 39.861 (9) Å
c = 10.053 (3) Å
 β = 107.40 (2)°
V = 2739.1 (14) Å³
Z = 4*D_x* = 1.330 Mg m⁻³
Mo *K*α radiation
Cell parameters from 24 reflections
 θ = 12.2–14.4°
 μ = 0.102 mm⁻¹
T = 298 (1) K
Plate, colourless
0.4 × 0.4 × 0.1 mm

Data collection

Rigaku AFC-7R diffractometer
 ω scans
3396 measured reflections
3157 independent reflections
1298 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.028
 θ_{\max} = 27.5°*h* = -9 → 0
k = 0 → 52
l = -13 → 13
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on *F*²
R(*F*) = 0.060
wR(*F*²) = 0.158
S = 0.95
3157 reflections
234 parameters
All H atoms refined $w = 1/[\sigma^2(F_o^2) + (0.0643P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
(Δ/σ)_{max} = 0.001
 $\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$
Extinction correction: *SHELXL97*
(Sheldrick, 1997)
Extinction coefficient: 0.0018 (4)

Table 1

Selected geometric parameters (Å, °).

O1—C7	1.212 (3)	O5—C12	1.254 (3)
O2—C7	1.312 (3)	N6—C12	1.328 (4)
O3—C11	1.231 (4)	C12—C13	1.463 (4)
O4—C11	1.254 (4)	C13—C14	1.315 (4)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O2—H2···O5	0.98 (2)	1.56 (2)	2.520 (3)	166 (4)
O3—H3···O4 ⁱ	0.98 (8)	1.79 (7)	2.631 (4)	141 (8)
O4—H4···O3 ⁱ	0.98 (5)	1.69 (4)	2.631 (4)	161 (6)
N6—H6A···O1	0.88 (1)	2.06 (2)	2.910 (3)	162 (3)
N6—H6B···O5 ⁱⁱ	0.87 (2)	2.22 (2)	3.052 (3)	162 (3)

Symmetry codes: (i) 1 - x, 1 - y, 1 - z; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.

All the H atoms were located from difference syntheses and refined isotropically. The H atom of the O3—C11—O4 carboxylic acid group has two possible positions (H3 and H4) with site-occupation factors of 50% each. The H atoms were restrained with O—H = 0.98 Å and N—H = 0.87 Å (s.u. of 0.01 Å). The C—H distances are in the range 0.88 (4)–1.06 (4) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

The authors thank Mr Akihiro Kondo for preparing the title crystals.

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