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1,2-Dihydrobenzocyclobutene-4-carboxylic acid

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Key indicators

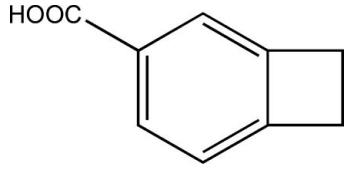
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.065
wR factor = 0.178
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the molecule of the title compound, $C_9H_8O_2$, the benzocyclobutene system is nearly planar. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules into dimers along the c axis.

Comment

Benzocyclobutene (BCB), a simple polycyclic aromatic hydrocarbon, is frequently used to create photosensitive polymers. BCB-based polymer dielectrics may be spun on or applied to various substrates for use in microelectromechanical systems (MEMS) and microelectronics processing. Applications include wafer bonding, optical interconnects, low-K dielectrics and even intracortical neural implants.



(I)

Benzocyclobutene-4-carboxylic acid is an important intermediate. It can be synthesized *via* a Grignard reaction in over 90% yield (Lloyd & Ongley, 1965). We report here the crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1)., the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The benzocyclobutene unit is nearly planar, with a puckering amplitude of Q_T = 0.051 (1) Å (Cremer & Pople, 1975).

As can be seen from the packing diagram (Fig. 2), intermolecular O—H···O hydrogen bonds [H1A···O2^{i} = 1.81Å, O1···O2^{i} = 2.63 (2) Å and O1—H1A···O2^{i} = 172°; symmetry code: (i) $-x$, 1 − y, $-z$] link the molecules into dimers. Dipole–dipole and van der Waals interactions are also effective in the molecular packing in the crystal structure.

Experimental

A solution of 4-benzocyclobutenylmagnesium bromide (1.0 g, 5.46 mmol) and Mg (0.132 g, 5.43 mmol) in Et_2O (20 ml) was poured on to powdered solid CO_2 (25.0 g). After warming to room temperature the mixture was acidified (HCl; 30 mmol) and the product extracted with Et_2O. The combined extracts (50 ml) were shaken with Na_2CO_3 (5%, 10 ml) three times, the aqueous extracts were acidified (HCl 10%, 10 ml) and the precipitated product was filtered off. It was recrystallized from EtOH (yield 0.744 g, 92%, m.p. 413–414 K).

Crystal data

$C_9H_8O_2$
M_r = 148.15
Monoclinic, $P2_1/n$
a = 7.233 (2) Å
b = 5.886 (3) Å
c = 17.682 (4) Å
β = 98.26 (2)°
V = 745.0 (6) Å^3
Z = 4
D_x = 1.321 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 (2) K
Block, colorless
0.30 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.973, T_{max} = 0.991
1558 measured reflections
1440 independent reflections
675 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.0°
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.178
S = 0.99
1440 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.029 (6)

H atoms were positioned geometrically, with O—H = 0.82 Å and C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,O)$, where x = 1.5 for hydroxyl H and x = 1.2 for all other H.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *PLATON* (Spek, 2003).

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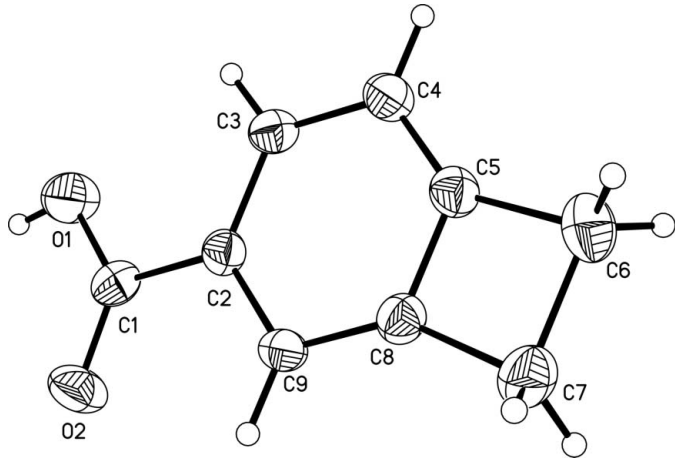

Figure 1
A view of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

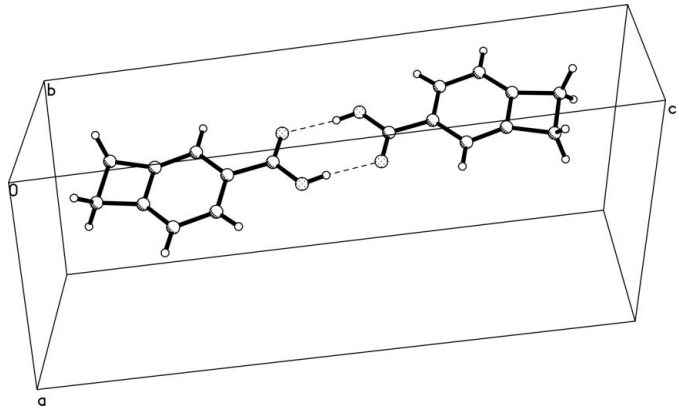

Figure 2
A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4.* University of Marburg, Germany.
Lloyd, J. B. F. & Ongley, P. A. (1965). *Tetrahedron*, **21**, 245–254.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97.* University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.