

3241 measured reflections
3024 independent reflections
1499 reflections with
 $I > \sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 27.50^\circ$

Refinement

Refinement on F^2
 $R = 0.052$
 $wR = 0.034$
 $S = 1.66$
1499 reflections
220 parameters
All H atoms refined
 $w = \sigma_F^{-2}$
 $(\Delta/\sigma)_{\text{max}} < 0.01$
 $\Delta\rho_{\text{max}} = 0.21 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{Å}^{-3}$

6 standard reflections
every 150 reflections
intensity variation: $\pm 3.0\%$
(average maximum
relative intensity)

Extinction correction:
Zachariasen (1963, 1968)
Extinction coefficient:
 $4.7(12) \times 10^{-7}$
Scattering factors from
Stewart, Davidson &
Simpson (1965) for H and
Cromer & Waber (1974)
for C and O atoms

Table 3. Selected geometric parameters (Å, °) for (II)

O1—C11	1.333 (3)	O3—C12	1.422 (4)
O1—C14	1.450 (3)	O4—C8	1.363 (3)
O2—C11	1.199 (3)	O4—C13	1.421 (4)
O3—C5	1.385 (3)	C1—C11	1.506 (3)
C11—O1—C14	116.3 (2)	O3—C5—C10	114.5 (2)
C5—O3—C12	117.1 (2)	O4—C8—C7	125.6 (3)
C8—O4—C13	117.8 (2)	O4—C8—C9	114.3 (2)
C2—C1—C11	115.6 (2)	O1—C11—O2	123.9 (2)
C9—C1—C11	124.2 (2)	O1—C11—C1	111.1 (2)
O3—C5—C6	124.7 (2)	O2—C11—C1	124.7 (3)

Scan widths were $(1.00 + 0.35 \tan \theta)^\circ$ in ω for (I) and $(1.40 + 0.35 \tan \theta)^\circ$ in ω for (II), with background/scan time-ratios of 0.5. The data were corrected for Lorentz and polarization effects. The Laue group assignment, systematic absences and intensity statistics were consistent with centrosymmetry indicated space group *Pnma* (No. 62) for (I) and *P2₁/a* (No. 14) for (II), and since the refinements proceeded well they were adopted. Fourier difference methods were used to locate the H-atom positions. Full-matrix least-squares refinement was performed. In (I), the maximum effect of extinction was 22.2% of F_o for 002. The maximum peak in the final difference map occurred roughly halfway between C1 and C11; the minimum peak occurred near the center of the C1—C4, C9, C10 ring. In (II), the maximum effect of extinction was 2.6% of F_o for 12 $\bar{1}$. The maximum peak in the final difference map occurred ~ 0.8 Å from C5 and C6; the minimum peak occurred near the center of the C1—C4, C9, C10 ring.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *TEXSAN*; molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

The authors thank Dr J. C. Gallucci for help of various sorts. The diffractometer was purchased with funds provided in part by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1029). Services for accessing these data are described at the back of the journal.

References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
 Ceccarelli, C., Jeffrey, G. A. & Taylor, R. J. (1981). *J. Mol. Struct.* **70**, 255–271.
 Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2A, pp. 149–150. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 Fitzgerald, L. J., Gallucci, J. C. & Gerkin, R. E. (1991). *Acta Cryst.* **B47**, 776–782.
 Fitzgerald, L. J. & Gerkin, R. E. (1993). *Acta Cryst.* **C49**, 1952–1958.
 Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502–510.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Molecular Structure Corporation (1989). *TEXSAN. TEXRAY Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.
 Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
 Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.
 Zachariasen, W. H. (1968). *Acta Cryst.* **A24**, 212–216.

Acta Cryst. (1997). **C53**, 1080–1082

Anthracene-1-carboxylic Acid

LAWRENCE J. FITZGERALD AND ROGER E. GERKIN

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210, USA. E-mail: gerkin@chemistry.ohio-state.edu

(Received 18 December 1996; accepted 1 April 1997)

Abstract

The title compound, C₁₅H₁₀O₂, was found to crystallize in the centrosymmetric space group *P2₁/n*. The hydrogen bonding is of the cyclic dimer type about a center of symmetry. The carboxyl O atoms are ordered as is the carboxyl H atom. The anthracene core, while less nearly planar, shows good agreement with the anthracene cores of anthracene-9-carboxylic acid and anthracene-1,6-dicarboxylic acid. The carboxyl group plane makes a dihedral angle of 12.4 (2)° with the best-fit anthracene core plane.

Comment

This study of anthracene-1-carboxylic acid, (I) (Fig. 1), was performed as one of a series on hydrogen bonding in aromatic carboxylic acids. It follows studies of the structures of anthracene-1,8-dicarboxylic acid and anthracene-9-carboxylic acid (Fitzgerald & Gerkin, 1996, 1997).

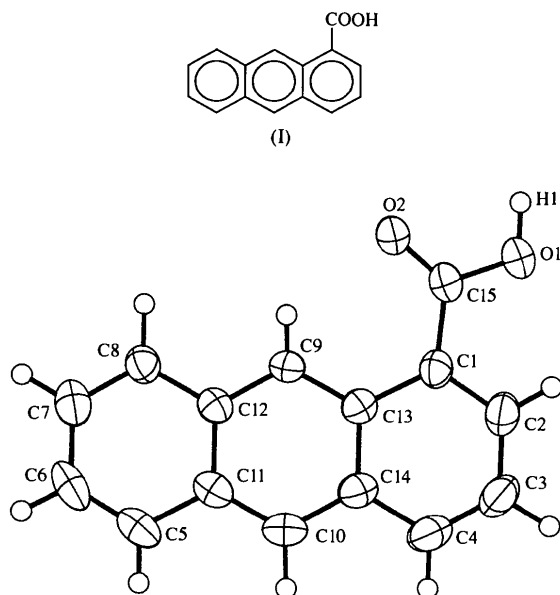


Fig. 1. ORTEP (Johnson, 1976) drawing of the anthracene-1-carboxylic acid molecule showing our numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for all atoms except H for which they have been set artificially small.

In the present structure, there is a single type of hydrogen bond which forms a cyclic dimer about a center of symmetry as shown in Fig. 2. Geometric details of the hydrogen bonding are given in Table 2.

The carboxyl H atom is ordered as are the carboxyl O atoms, for which the C—O distances are 1.315 (3) and 1.219 (3) Å, respectively. As in previous studies (Fitzgerald & Gerkin, 1993, 1996, 1997) and as shown in Fig. 1, the principal displacement tensor axes of the O atoms are nearly perpendicular to the carboxyl group plane.

The anthracene core exhibits moderate conformity to mirror symmetry with respect to a mirror through C9 and C10: for the eight pairs of distances which would be identical under this symmetry, the r.m.s. deviation within pairs is 0.009 Å, while the e.s.d. of individual distances is 0.003–0.004 Å. For the pseudo-mirror at right angles to this, the r.m.s. deviation within the analogous six pairs of distances is 0.011 Å. The average difference between the sixteen corresponding distances in the cores of the title acid and the 9-acid is 0.007 Å, and of the title acid and the 1,8-diacid is 0.008 Å, while the individual e.s.d.'s are 0.003–0.004, 0.002–0.003 and 0.007–0.009 Å, respectively.

Each ring of the molecular core is almost planar, the average deviations of the corresponding atoms from the best planes describing them being 0.014 Å for the C1—C4, C13, C14 ring, 0.003 Å for the C5—C8, C11, C12 ring and 0.014 Å for the central ring. The outer rings make dihedral angles of 3.5 (1) and 1.6 (1)°, respectively, with the central ring with the result that the whole core is less planar than in the 9-acid or the 1,8-diacid. [The average distance of the core atoms from the best-fit core plane is 0.035 Å for the title molecule, which may be compared with 0.020 Å for the 9-acid and 0.018 Å for the 1,8-diacid.] The dihedral angle between the plane of the carboxyl group and the best-fit core plane is 12.4 (2)°, substantially smaller than the analogous angle in the 9-acid [54.9 (1)°], but quite similar to those in the 1,8-diacid [13.9 (2) and 21.6 (2)°].

The molecular packing is shown in Fig. 2. The dihedral angle between the two orientations of the best-fit anthracene core planes is 56.03 (7)°. Excepting pairs of atoms involved directly in hydrogen bonds, the closest intermolecular approaches are between C5 and H4ⁱⁱ [symmetry code: (ii) $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z$] and fall short of the corresponding Bondi (1964) radius sum by only 0.09 Å.

The refined C—H distances ranged from 0.88 (2) to 1.04 (2) Å, with a mean value of 0.97 Å. All bond distances and angles are within normal ranges.

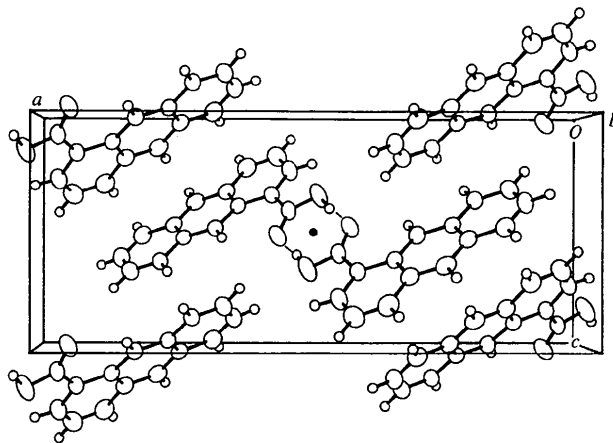


Fig. 2. ORTEP (Johnson, 1976) packing diagram of anthracene-1-carboxylic acid. Displacement ellipsoids are drawn at the 50% probability level for all atoms except H for which they have been set artificially small. The center of symmetry at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$ is shown as a filled circle, the hydrogen bonds as dashes.

Experimental

Anthracene-1-carboxylic acid from TCI America was dissolved at room temperature in ethanol. Slow evaporation of the solution at room temperature produced columns, one of which was mounted uncut to provide the experimental sample.

Crystal data

C₁₅H₁₀O₂
M_r = 222.24
 Monoclinic
*P*2₁/*n*
a = 20.527 (2) Å
b = 6.144 (2) Å
c = 8.604 (2) Å
 β = 90.08 (2)°
V = 1085.0 (4) Å³
Z = 4
D_x = 1.360 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 13.4–15.0°
 μ = 0.084 mm⁻¹
T = 296 K
 Clear column
 0.35 × 0.19 × 0.19 mm
 Yellow

Data collection

AFC-5S diffractometer
 ω scans
 Absorption correction: none
 2788 measured reflections
 2717 independent reflections
 1377 reflections with
 $I > \sigma(I)$
R_{int} = 0.028
 θ_{\max} = 27.5°

h = 0 → 26
 k = 0 → 7
 l = -11 → 11
 6 standard reflections
 every 150 reflections
 intensity variation: ±1.8%
 (average maximum
 relative intensity)

Refinement

Refinement on *F*²
R = 0.054
wR = 0.053
S = 1.56
 1377 reflections
 195 parameters
 All H atoms refined
 $w = \sigma_F^{-2}$
 $(\Delta/\sigma)_{\max} < 0.01$
 $\Delta\rho_{\max} = 0.21 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{Å}^{-3}$

Extinction correction:
 Zachariasen (1963, 1968)
 Extinction coefficient:
 2.7 (3) × 10⁻⁶
 Scattering factors from
 Stewart, Davidson &
 Simpson (1965) for H,
 Cromer & Waber (1974)
 for C and O atoms

Table 1. Selected geometric parameters (Å, °)

O1—C15	1.315 (3)	C1—C15	1.485 (4)
O2—C15	1.219 (3)		
C2—C1—C15	118.0 (2)	O1—C15—C1	114.4 (3)
C13—C1—C15	122.1 (2)	O2—C15—C1	124.5 (2)
O1—C15—O2	121.1 (3)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...O2 ⁱ	0.95 (4)	1.69 (4)	2.633 (3)	172 (3)

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

Scan widths were (1.40 + 0.35tan θ)° in ω with a background/scan time-ratio of 0.5. The data were corrected for Lorentz and polarization effects. The Laue group assignment, systematic absences and intensity statistics consistent with centrosymmetry indicated space group *P*2₁/*n* (No. 14) and since refinement proceeded well it was adopted. Moreover, axial photos were consistent with monoclinic but not with orthorhombic symmetry. Fourier difference methods were used to locate the initial H-atom positions. The maximum effect of extinction was 9.7% of *F_o* for 21 $\bar{2}$. The maximum positive residual peak was located near the midpoint of the C1—C13 bond, while the maximum negative peak was located near the center of the C5—C8, C11, C12 ring.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

Support provided to LJF by PPG Industries is gratefully acknowledged. We thank Dr J. C. Gallucci for help of various sorts. The diffractometer was purchased with funds provided in part by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1321). Services for accessing these data are described at the back of the journal.

References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
 Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71, 148. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 Fitzgerald, L. J. & Gerkin, R. E. (1993). *Acta Cryst.* **C49**, 1952–1958.
 Fitzgerald, L. J. & Gerkin, R. E. (1996). *Acta Cryst.* **C52**, 1838–1841.
 Fitzgerald, L. J. & Gerkin, R. E. (1997). *Acta Cryst.* **C53**, 71–73.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (1988). *MSCIAFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Molecular Structure Corporation (1989). *TEXSAN. TEXRAY Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.
 Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.
 Zachariasen, W. H. (1968). *Acta Cryst.* **A24**, 212–216.

Acta Cryst. (1997). **C53**, 1082–1084

An Estrone–Glucuronide Conjugate

C. MARK SMALES, LEONARD F. BLACKWELL, JOYCE M. WATERS AND ANTHONY K. BURRELL

Department of Chemistry, Massey University, Private Bag 11222, Palmerston North, New Zealand. E-mail: a.k.burrell@massey.ac.nz

(Received 14 October 1996; accepted 20 March 1997)

Abstract

The crystal structure of 2-*O*-[17-oxoestra-1,3,5(10)-trien-3-yl]- β -D-glucopyranosiduronic acid hydrate, C₂₄H₃₀O₈·