

Data collection: CAD-4F software. Cell refinement: CAD-4F software. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: NA1258). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Bucourt, R. (1974). *Topics in Stereochemistry*, Vol. 8, edited by E. L. Eliel & N. L. Allinger, pp. 159–224. New York: Wiley Interscience.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Horton, D. & Liu, Q. (1996). American Chemical Society Meeting 212, Orlando, Florida, USA. August, 1996. Abstract CARB-024.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Spek, A. L. (1996). *PLATON96*. University of Utrecht, The Netherlands.

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9,10-Dihydro-9-oxo-10-acridineacetic Acid

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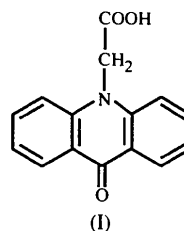
Abstract

In the crystal of the title compound, C₁₅H₁₁NO₃, the molecules form chains of strong acid-to-ketone hydrogen bonds, with an O···O distance of 2.585 (3) Å.

Comment

As a part of a study of the coordination properties of the 9,10-dihydro-9-oxo-10-acridineacetate ligand (CMA⁻), we have recorded the IR spectra and performed the X-ray structure determinations of a series of metal complexes with the CMA⁻ ligand (MCMA, M = Cu,

Zn, Ca, Pb; Miernik, Lis, Palus & Reedijk, 1993; Miernik & Lis, 1994, 1996) and also of the free acid, (I), which is presented here.



An ORTEPII (Johnson, 1976) view of the title molecule is presented in Fig. 1. The molecule exhibits a characteristic slightly bent shape; the dihedral angle between the planes determined by the atoms of the two halves of the macrocycle (*i.e.* N1, C7–C13 and N1, C3–C6, C13–C15) is 7.1 (1)°. The geometry of the acridone ring is in a good agreement with that of the CMA⁻ complexes; differences between average and individual values are statistically insignificant.

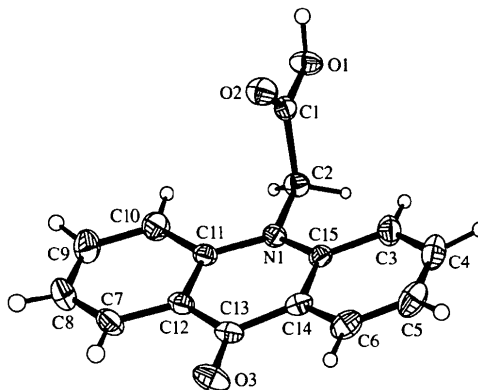


Fig. 1. An ORTEPII (Johnson, 1976) view of the CMAH molecule with the atomic labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and displacement parameters of the H atoms were artificially diminished by a factor of five for clarity.

A stereoscopic packing diagram is shown in Fig. 2. The CMAH molecules form ketone-to-acid intermolecular hydrogen bonds, which is relatively uncommon for keto carboxylic acids (Vanderhoff, Lalancette & Thompson, 1990; Lalancette, Vanderhoff & Thompson, 1991; Lalancette, Thompson & Vanderhoff, 1991; Lalancette, Coté & Thompson, 1996). The CMAH hydrogen chains are oriented parallel to the [011] and [01 $\bar{1}$] directions. The intermolecular ketone-to-acid distance O3···O1($\frac{1}{2} - x, -\frac{1}{2} + y, -\frac{1}{2} + z$) is 2.585 (3) Å, with O3···H1($\frac{1}{2} - x, -\frac{1}{2} + y, -\frac{1}{2} + z$) 1.69 (4) Å, which is characteristic of strong *syn* interactions; the hydrogen-bond angle O3···H1—O1 is 158 (3)°. The participation of both carboxylic O atoms (O1 and O2) in hydrogen bonding, and therefore the existence of typical carboxylic dimers in the solid state of CMAH, was excluded on the grounds of the solid-state (KBr) IR spectra; a very

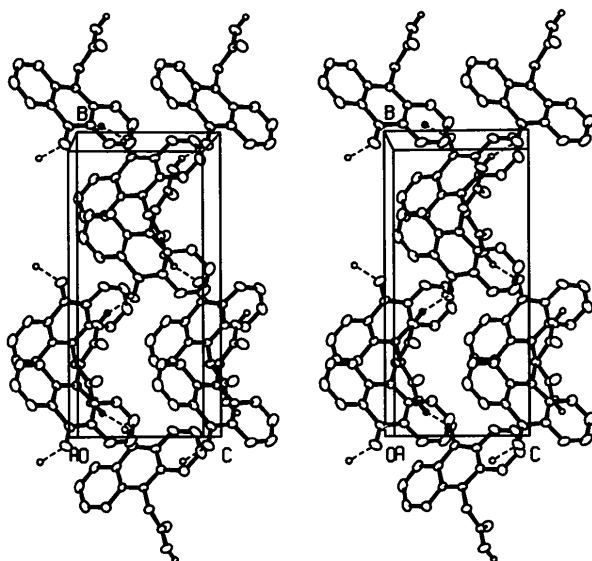


Fig. 2. A stereoscopic packing diagram displaying hydrogen bonding in a crystal lattice of the title compound. H atoms uninvolved in hydrogen bonds were omitted for clarity.

strong carboxyl absorption appears at 1737 cm^{-1} , a significantly higher value than observed for carboxylic acid dimers.

Experimental

The title compound was prepared according to a method described previously by Inglot, Młochowski, Szulc, Inglot & Albin (1985) and crystallized from ethanol.

Crystal data

$\text{C}_{15}\text{H}_{11}\text{NO}_3$
 $M_r = 253.25$
 Orthorhombic
 $Pna2_1$
 $a = 9.7700(10)\text{ \AA}$
 $b = 16.018(4)\text{ \AA}$
 $c = 7.575(2)\text{ \AA}$
 $V = 1185.5(4)\text{ \AA}^3$
 $Z = 4$
 $D_x = 1.419\text{ Mg m}^{-3}$
 D_m not measured

Data collection

Kuma KM-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 1864 measured reflections
 1864 independent reflections
 1478 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R(F) = 0.039$
 $wR(F^2) = 0.115$

Mo $K\alpha$ radiation
 $\lambda = 0.71073\text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 7.5\text{--}17.0^\circ$
 $\mu = 0.100\text{ mm}^{-1}$
 $T = 298(2)\text{ K}$
 Prism
 $0.81 \times 0.48 \times 0.33\text{ mm}$
 Pale yellow

$\theta_{\text{max}} = 30^\circ$
 $h = -13 \rightarrow 0$
 $k = 0 \rightarrow 22$
 $l = -10 \rightarrow 0$
 3 standard reflections every 50 reflections
 intensity decay: -1.2%

$S = 1.03$
 1864 reflections
 216 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.0682P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

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

C1—O1	1.317(2)	C6—C14	1.405(3)
C1—O2	1.196(2)	C14—C13	1.445(3)
C1—C2	1.515(3)	C13—O3	1.248(2)
C2—N1	1.458(2)	C13—C12	1.447(3)
N1—C11	1.389(2)	C12—C7	1.405(3)
N1—C15	1.385(2)	C12—C11	1.414(2)
C15—C3	1.415(3)	C7—C8	1.356(4)
C15—C14	1.414(2)	C8—C9	1.400(4)
C3—C4	1.380(4)	C9—C10	1.369(3)
C4—C5	1.384(4)	C10—C11	1.408(3)
C5—C6	1.369(5)		
O1—C1—O2	124.9(2)	O3—C13—C14	120.4(2)
O1—C1—C2	110.6(2)	O3—C13—C12	122.6(2)
O2—C1—C2	124.5(2)	C12—C13—C14	116.9(2)

The title structure was solved by direct methods. All non-H atoms were refined anisotropically. All H atoms were located from a difference Fourier map and refined without constraints and restraints. The absolute structure could not be determined because the anomalous scattering was insufficient. The origin was fixed by the method of Flack & Schwarzenbach (1988).

Data collection: *Kuma KM-4 Software* (Kuma Diffraction, 1989). Cell refinement: *Kuma KM-4 Software*. Data reduction: *Kuma KM-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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References

- Flack, H. D. & Schwarzenbach, D. (1988). *Acta Cryst.* **A44**, 499–506.
 Inglot, A. D., Młochowski, J., Szulc, Z., Inglot, O. & Albin, M. (1985). *Arch. Immun. Ther. Exp.* **33**, 275–285.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Kuma Diffraction (1989). *Kuma KM-4 Software*. Version 3.1. Kuma Diffraction, Wrocław, Poland.
 Lalancette, R. A., Coté, M. L. & Thompson, H. W. (1996). *Acta Cryst.* **C52**, 244–246.
 Lalancette, R. A., Thompson, H. W. & Vanderhoff, P. A. (1991). *Acta Cryst.* **C47**, 986–990.
 Lalancette, R. A., Vanderhoff, P. A. & Thompson, H. W. (1991). *Acta Cryst.* **C47**, 132–136.
 Miernik, D. & Lis, T. (1994). *J. Chem. Cryst.* **24**, 731–737.
 Miernik, D. & Lis, T. (1996). *Acta Cryst.* **C52**, 1171–1174.
 Miernik, D., Lis, T., Palus, J. & Reedijk, J. (1993). *Inorg. Chim. Acta*, **205**, 231–237.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Vanderhoff, P. A., Lalancette, R. A. & Thompson, H. W. (1990). *J. Org. Chem.* **55**, 1696–1698.