Sakurai, T. (1967). *RSLC-3. X-ray Analysis of Crystal Structures.* Tokyo: Syokabo.
Sheldrick, G. M. (1994). *SHELXL93. Program for Crystal Structure Refinement.* Univ. of Göttingen, Germany.

Acta Cryst. (1994). C**50**, 1952–1953

7-Hydroxy-4-methylcoumarin Monohydrate

JERRY P. JASINSKI AND RICHARD C. WOUDENBERG

Chemistry Department, Keene State College,
229 Main Street, Keene, New Hampshire 03431, USA

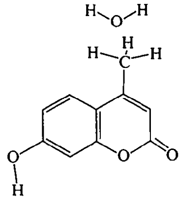
(*Received 5 October* 1992; *accepted 25 May* 1994)

Abstract

The crystal structure of the title compound, $C_{10}H_8O_3.H_2O$, is stabilized by three intermolecular contacts of the O—H···O type involving the water molecules of crystallization. The coumarin moiety is planar and the hydroxy group is located in the plane of the benzene ring.

Comment

The title compound, (I), a hydroxycoumarin laser dye, has been used in laser gain studies (Masilamani & Sivaram, 1982). It is also known as Coumarin 4 (Eastman Kodak Company, Rochester, NY, USA) and Umbelliferon 47.



(I)

An *ORTEP* view (Johnson, 1976) of the title molecule with atomic labeling is shown in Fig. 1. Bond lengths and angles in the coumarin moiety are normal (Gnanaguru, Ramasubbu, Venkatesan & Ramamurthy, 1985; Murthy, Ramamurthy & Venkatesan, 1988). The coumarin moiety is planar [χ^2(pyrone ring) = 62.2 and O13—C7—C8—C9 = 178.9 (2)°].

The packing of the molecules in the unit cell viewed near the *a* axis is shown in Fig. 2. The molecules are linked by three hydrogen bonds of the O—H···O type to water molecules of crystallization, two of the interactions involving the carbonyl O atom [O2···O11^{i} 2.790 (2), O2—H2*A* 0.83 (3), H2*A*···O11^{i} 1.97 (3) Å and O2—H2*A*···O11^{i} 172.5 (1)°; O2···O11^{ii} 2.873 (2), O2—H2*B* 0.88 (3), H2*B*···O11^{ii} 2.00 (3) Å and O2—H2*B*···O11^{ii} 169.5 (1)°; symmetry code: (i) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$; (ii) $x-\frac{1}{2}$, $\frac{1}{2}+y$, $\frac{1}{2}-z$] and one involving the hydroxy O atom [O13···O2^{iii} 2.655 (2), O13—H13 0.87 (2), H13···O2^{iii} 1.78 (2) Å and O13—H13···O2^{iii} 174.0 (1)°; symmetry code: (iii) $1-x$, $-y$, $1-z$].

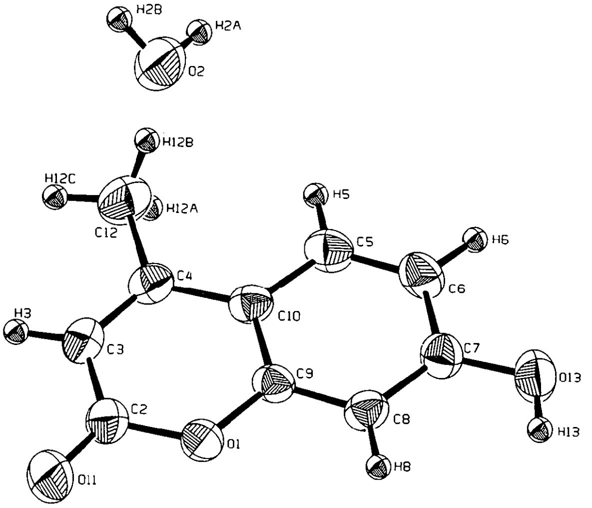


Fig. 1. *ORTEP* (Johnson, 1976) drawing (50% probability ellipsoids) of the title compound and the atomic numbering scheme.

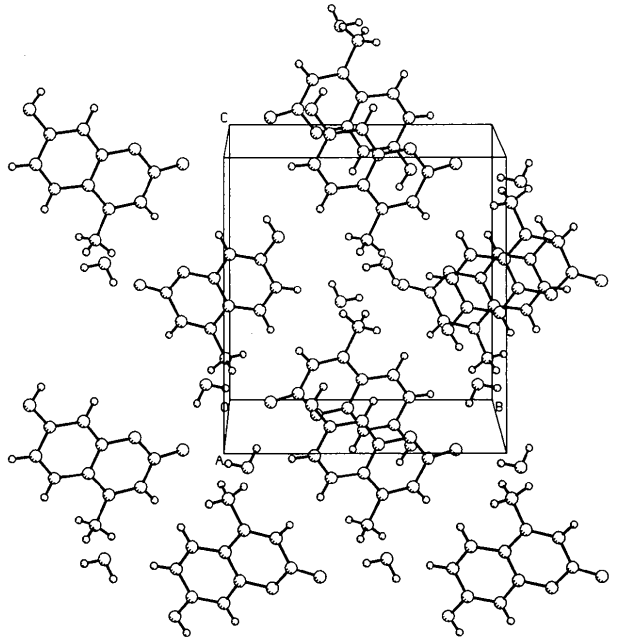


Fig. 2. Molecular packing of the title compound in the unit cell viewed close to the *a* axis.

Experimental

Crystals of the title compound (Exiton Chemical Company, Dayton, Ohio 45431, USA) were grown from acetonitrile by slow evaporation.

Crystal data

$C_{10}H_8O_3.H_2O$
$M_r = 194.19$
Monoclinic
$P2_1/n$
$a = 7.106$ (2) Å
$b = 11.335$ (2) Å
$c = 11.817$ (2) Å
$\beta = 105.30$ (1)°
$V = 918.1$ (3) Å^3
$Z = 4$
$D_x = 1.405$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 22.5\text{–}25.0°$
$\mu = 0.1023$ mm^{-1}
$T = 296$ K
Prism
1.00 × 0.90 × 0.30 mm
Colorless

Data collection

Rigaku AFC-6*S* diffractometer
ω scans with profile analysis
Absorption correction: empirical (ψ scan)
$T_{\min} = 0.95$, $T_{\max} = 1.00$
2395 measured reflections
2227 independent reflections
1356 observed reflections $[I > 3.0\sigma(I)]$

$R_{\text{int}} = 0.017$
$\theta_{\max} = 27.49°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 15$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 150 reflections intensity variation: −0.70%

Refinement

Refinement on F
$R = 0.039$
$wR = 0.045$
$S = 1.741$
1356 reflections
168 parameters
All H-atom parameters refined
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = 0.0624$

$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: Type 2 Gaussian isotropic (Zachariasen, 1963)
Extinction coefficient: 8.3852×10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
O1	0.8319 (2)	0.16979 (9)	0.50906 (10)	0.0425
O2	0.5273 (3)	0.0770 (2)	0.8603 (1)	0.0673
O11	0.9597 (3)	0.3396 (1)	0.5758 (1)	0.0663
O13	0.5438 (2)	−0.1888 (1)	0.3440 (1)	0.0538
C2	0.9316 (3)	0.2374 (2)	0.6005 (2)	0.0459
C3	0.9940 (3)	0.1842 (2)	0.7135 (2)	0.0448
C4	0.9563 (3)	0.0707 (2)	0.7328 (2)	0.0394
C5	0.7975 (3)	−0.1184 (2)	0.6422 (2)	0.0419
C6	0.6975 (3)	−0.1787 (2)	0.5453 (2)	0.0438
C7	0.6437 (3)	−0.1239 (2)	0.4363 (2)	0.0396
C8	0.6915 (3)	−0.0070 (2)	0.4257 (2)	0.0374
C9	0.7905 (3)	0.0529 (1)	0.5247 (2)	0.0343
C10	0.8483 (3)	0.0009 (1)	0.6355 (1)	0.0353
C12	1.0253 (4)	0.0161 (2)	0.8518 (2)	0.0564

Table 2. *Selected geometric parameters* (Å, °)

O1—C2	1.362 (2)	C4—C12	1.496 (3)
O1—C9	1.380 (2)	C5—C6	1.362 (2)
O11—C2	1.223 (2)	C5—C10	1.407 (2)
O13—C7	1.352 (2)	C6—C7	1.390 (2)
C2—C3	1.424 (3)	C7—C8	1.382 (2)
C3—C4	1.346 (2)	C8—C9	1.374 (2)
C4—C10	1.439 (2)	C9—C10	1.395 (2)
C2—O1—C9	121.3 (1)	O13—C7—C6	117.6 (2)
O1—C2—O11	115.3 (2)	O13—C7—C8	122.6 (2)
O1—C2—C3	118.2 (2)	C6—C7—C8	119.9 (2)
O11—C2—C3	126.5 (2)	C7—C8—C9	118.6 (2)
C2—C3—C4	122.5 (2)	O1—C9—C8	116.1 (1)
C3—C4—C10	118.6 (2)	O1—C9—C10	120.7 (1)
C3—C4—C12	121.6 (2)	C8—C9—C10	123.3 (1)
C10—C4—C12	119.8 (2)	C4—C10—C5	125.0 (2)
C6—C5—C10	121.3 (2)	C4—C10—C9	118.8 (2)
C5—C6—C7	120.7 (2)		

The weak reflections $[I < 10.0\sigma(I)]$ were rescanned (maximum of two rescans) and the counts accumulated to assure good counting statistics. Lp corrections were applied; no correction was made for decay. Plots of $\Sigma w(|F_o|-|F_c|)^2$ *versus* $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The enantiomorphs are indistinguishable using the X-ray data. In the weighting scheme, $\sigma^2(F_o^2) = [S^2(C + 4B) + (0.03F_o^2)]/\text{Lp}^2$, where S is the scan rate, C is the number of counts per scan and B is the sum of two background counts.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

The authors wish to acknowledge the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research, and also the National Science Foundation Research in Undergraduate Institutions Instrumentation Program (grant No. 8818307) for the creation of the New England Molecular Structure Center at Keene State College.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HH1047). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Gnanaguru, K., Ramasubbu, N., Venkatesan, K. & Ramamurthy, V. (1985). *J. Org. Chem.* **50**, 2337–2346.

Johnson, C. K. (1976). *ORTEP*II. ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Masilamani, V. & Sivaram, B. M. (1982). *J. Lumin.* **27**, 219–221.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. Version 2.1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Murthy, G. S., Ramamurthy, V. & Venkatesan, K. (1988). *Acta Cryst.* C**44**, 307–311.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.