Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dimethylammonium 2-[(2-oxo-2*H*-chromen-7-yl)oxy]acetate

Feng-Xia Dong

State Key Laboratory of Supramolecular Structure and Materials, College of Chemistry, Jilin University, Changchun 130012, People's Republic of China
Correspondence e-mail: dongfx@jlu.edu.cn

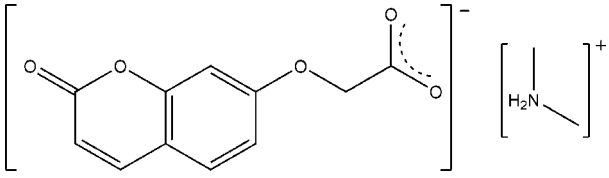
Received 1 April 2011; accepted 6 April 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.131; data-to-parameter ratio = 15.8.

In the title salt, $C_2H_8N^+\cdot C_{11}H_7O_5^-$, the acetate group is twisted out of the plane of the coumarin ring system with a C—O—C—C torsion angle of 76.3 (2)°. In the crystal, N—H···O hydrogen bonds link the cations and anions into chains propagating in [100].

Related literature

For the synthesis, see Matsuda *et al.* (2000).



Experimental

Crystal data

$C_2H_8N^+\cdot C_{11}H_7O_5^-$
M_r = 265.26
Triclinic, $P\overline{1}$
a = 6.714 (5) Å
b = 8.146 (7) Å
c = 12.767 (12) Å
α = 83.33 (4)°
β = 79.16 (3)°
γ = 67.78 (3)°
V = 634.1 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.44 × 0.22 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.955, T_{max} = 0.986
6310 measured reflections
2881 independent reflections
1878 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.131
S = 0.93
2881 reflections
182 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4	0.90 (1)	1.92 (1)	2.799 (2)	166 (2)
N1—H1B···O5^{i}	0.90 (1)	1.86 (1)	2.729 (3)	160 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The author thanks Jilin University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5144).

References

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Matsuda, T., Mizutani, M. & Arnold, S. C. (2000). *Macromolecules*, **33**, 795–800.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o1105 [doi:10.1107/S1600536811012761]

Dimethylammonium 2-[(2-oxo-2*H*-chromen-7-yl)oxy]acetate

Feng-Xia Dong

S1. Comment

Coumarin derivatives have been widely studied due to the applications in medicine and optical materials. In this paper, we report the synthesis and crystal structure of the title compound, which is a type of carboxyl modified coumarin derivative.

In the title compound, the acetate group twist outside the plane of coumarin group with a C7—O3—C10—C11 torsion angle of 76.3 (2). The hydrogen atom of carboxyl transfer to the dimethylamine molecule forming N—H···O hydrogen bonding interaction (Figure 1).

In the crystal structure of the title compound, the N—H···O hydrogen bonds bewteen coumarin anions and dimethylammonium cations link them to form a chain structure (Figure 2, Table 1).

S2. Experimental

A mixture of 7-hydroxycoumarin (0.16 g, 1.0 mmol), potassium carbonate (0.20 g, 1.4 mmol), ethyl bromoacetate (0.20 g, 1.2 mmol), and dry acetone (30 ml) was refluxed for 4 h while stirring in a N_2 atmosphere. After removal of salt by filtration, the resulting ester was recrystallized from ethanol. After then, the carboxylic acid derivatives was obtained through refluxing in sodium hydroxide solution and protonized with HCl. Mix the obtained carboxylic acid derivatives with dimethylamine with molar ratio of 1:1 in methanol, needle-like crystals of title compound were obtained after several days.

S3. Refinement

The reflection data (2 3 2) had been omit in the refinement. H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic C), C—H = 0.97Å (methylene C), and with U_{iso}(H) = 1.2Ueq(C) or C—H = 0.96 Å (methly C) and with U_{iso}(H) = 1.5Ueq(C). The N-bound H atoms were initially located in a difference Fourier map and they were refined with N—H=0.90 Å.

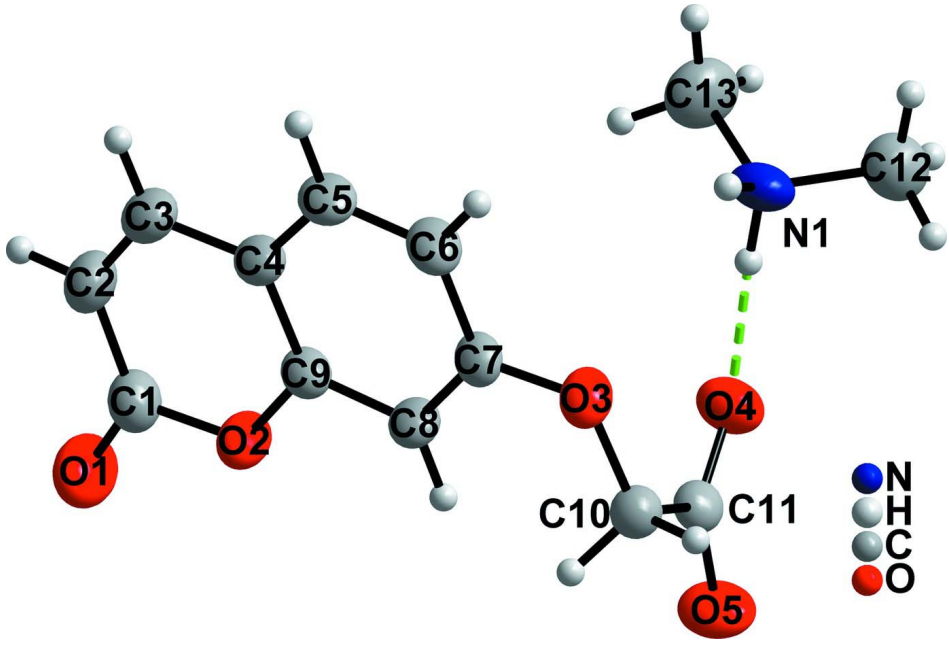


Figure 1

Molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level for non-H atoms. Dashed lines indicate the hydrogen-bonding interactions.

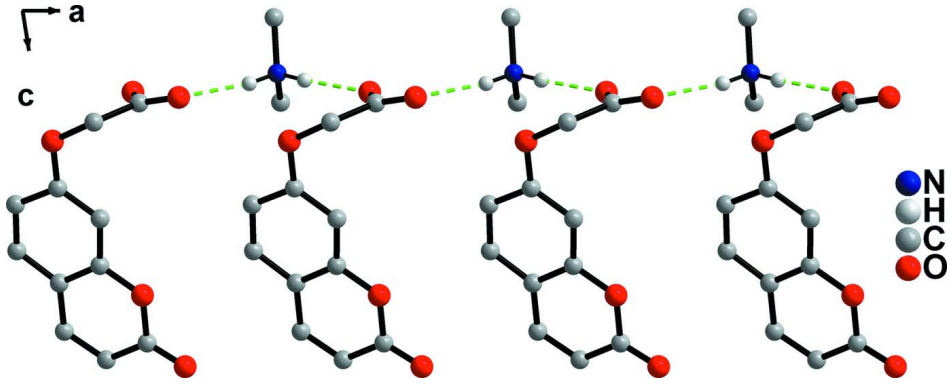


Figure 2

A partial packing view, showing the hydrogen-bonding chain structure. Dashed lines indicate the hydrogen bonds, no involving H atoms have been omitted for clarity.

Dimethylammonium 2-[(2-oxo-2*H*-chromen-7-yl)oxy]acetate

Crystal data

$C_2H_8N^+·C_{11}H_7O_5^-$
M_r = 265.26
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.714 (5) Å
b = 8.146 (7) Å
c = 12.767 (12) Å
α = 83.33 (4)°
β = 79.16 (3)°
γ = 67.78 (3)°
V = 634.1 (9) Å^3
Z = 2
F(000) = 280
D_x = 1.389 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4269 reflections
θ = 3.1–27.5°
μ = 0.11 mm^{-1}

T = 293 K
Block, colorless

0.44 × 0.22 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.955, T_{max} = 0.986

6310 measured reflections
2881 independent reflections
1878 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −8→8
k = −10→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.131
S = 0.93
2881 reflections
182 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 0.0078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.1702 (2)	0.71671 (17)	0.56112 (12)	0.0410 (3)
C1	0.4408 (3)	0.6603 (2)	1.12637 (14)	0.0474 (4)
C2	0.2575 (3)	0.6054 (3)	1.16133 (14)	0.0509 (4)
H2	0.2490	0.5412	1.2261	0.061*
C3	0.0990 (3)	0.6456 (2)	1.10186 (14)	0.0492 (4)
H3	−0.0200	0.6120	1.1270	0.059*
C4	0.1103 (2)	0.7390 (2)	1.00064 (12)	0.0390 (4)
C5	−0.0479 (2)	0.7886 (2)	0.93318 (14)	0.0469 (4)
H5	−0.1734	0.7628	0.9553	0.056*
C6	−0.0222 (2)	0.8737 (2)	0.83609 (13)	0.0441 (4)
H6	−0.1294	0.9053	0.7929	0.053*
C7	0.1668 (2)	0.9134 (2)	0.80145 (12)	0.0352 (3)
C8	0.3255 (2)	0.8682 (2)	0.86578 (12)	0.0377 (4)

H8	0.4510	0.8942	0.8435	0.045*
C9	0.2930 (2)	0.7836 (2)	0.96401 (12)	0.0366 (4)
C10	0.3639 (2)	1.0429 (2)	0.66355 (12)	0.0366 (4)
H10A	0.3951	1.0951	0.7201	0.044*
H10B	0.3299	1.1324	0.6057	0.044*
C11	0.5675 (2)	0.8869 (2)	0.62315 (12)	0.0340 (3)
C12	0.1952 (3)	0.7031 (3)	0.44443 (15)	0.0557 (5)
H12A	0.0662	0.6949	0.4271	0.084*
H12B	0.3187	0.5991	0.4223	0.084*
H12C	0.2168	0.8065	0.4081	0.084*
C13	0.1659 (3)	0.5537 (3)	0.62202 (18)	0.0646 (5)
H13A	0.2810	0.4530	0.5894	0.097*
H13B	0.0281	0.5430	0.6225	0.097*
H13C	0.1856	0.5584	0.6940	0.097*
O1	0.5856 (2)	0.6407 (2)	1.17600 (11)	0.0675 (4)
O2	0.45322 (16)	0.74415 (16)	1.02652 (9)	0.0459 (3)
O3	0.17667 (14)	0.99754 (15)	0.70317 (8)	0.0410 (3)
O4	0.54894 (16)	0.75011 (15)	0.60073 (10)	0.0472 (3)
O5	0.73879 (16)	0.91839 (17)	0.61151 (11)	0.0571 (4)
H1B	0.040 (2)	0.802 (2)	0.5816 (16)	0.067 (6)*
H1A	0.279 (2)	0.747 (3)	0.5738 (16)	0.068 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0340 (6)	0.0366 (7)	0.0567 (9)	−0.0137 (5)	−0.0116 (6)	−0.0086 (6)
C1	0.0462 (9)	0.0598 (11)	0.0368 (9)	−0.0195 (8)	−0.0072 (7)	−0.0023 (8)
C2	0.0554 (10)	0.0640 (11)	0.0366 (9)	−0.0294 (9)	−0.0012 (8)	0.0013 (8)
C3	0.0459 (9)	0.0642 (11)	0.0437 (10)	−0.0311 (8)	0.0046 (7)	−0.0071 (9)
C4	0.0322 (7)	0.0512 (9)	0.0375 (9)	−0.0207 (6)	0.0018 (6)	−0.0102 (7)
C5	0.0318 (7)	0.0694 (11)	0.0474 (10)	−0.0274 (7)	−0.0002 (7)	−0.0124 (9)
C6	0.0289 (7)	0.0650 (11)	0.0413 (9)	−0.0179 (7)	−0.0062 (6)	−0.0105 (8)
C7	0.0275 (7)	0.0447 (8)	0.0315 (8)	−0.0107 (6)	−0.0008 (6)	−0.0087 (7)
C8	0.0267 (7)	0.0534 (9)	0.0358 (8)	−0.0189 (6)	−0.0009 (6)	−0.0046 (7)
C9	0.0280 (7)	0.0492 (9)	0.0345 (8)	−0.0152 (6)	−0.0035 (6)	−0.0080 (7)
C10	0.0345 (7)	0.0388 (8)	0.0361 (8)	−0.0131 (6)	−0.0043 (6)	−0.0026 (7)
C11	0.0302 (7)	0.0415 (8)	0.0315 (8)	−0.0125 (6)	−0.0090 (6)	−0.0007 (6)
C12	0.0580 (10)	0.0542 (10)	0.0547 (11)	−0.0179 (8)	−0.0144 (9)	−0.0018 (9)
C13	0.0706 (12)	0.0565 (11)	0.0663 (13)	−0.0243 (10)	−0.0181 (10)	0.0147 (10)
O1	0.0595 (8)	0.1038 (11)	0.0491 (8)	−0.0383 (8)	−0.0243 (6)	0.0135 (8)
O2	0.0351 (5)	0.0709 (8)	0.0374 (6)	−0.0263 (5)	−0.0095 (5)	0.0047 (6)
O3	0.0282 (5)	0.0550 (7)	0.0365 (6)	−0.0115 (4)	−0.0052 (4)	−0.0019 (5)
O4	0.0392 (6)	0.0417 (6)	0.0615 (8)	−0.0118 (5)	−0.0088 (5)	−0.0145 (6)
O5	0.0301 (5)	0.0638 (8)	0.0809 (10)	−0.0189 (5)	−0.0076 (6)	−0.0134 (7)

Geometric parameters (Å, º)

N1—C13	1.466 (3)	C7—O3	1.361 (2)
N1—C12	1.480 (3)	C7—C8	1.381 (2)
N1—H1B	0.904 (9)	C8—C9	1.381 (2)
N1—H1A	0.899 (9)	C8—H8	0.9300
C1—O1	1.209 (2)	C9—O2	1.378 (2)
C1—O2	1.378 (2)	C10—O3	1.4287 (19)
C1—C2	1.441 (3)	C10—C11	1.524 (2)
C2—C3	1.341 (3)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.426 (3)	C11—O4	1.238 (2)
C3—H3	0.9300	C11—O5	1.2490 (19)
C4—C9	1.392 (2)	C12—H12A	0.9600
C4—C5	1.404 (2)	C12—H12B	0.9600
C5—C6	1.362 (3)	C12—H12C	0.9600
C5—H5	0.9300	C13—H13A	0.9600
C6—C7	1.406 (2)	C13—H13B	0.9600
C6—H6	0.9300	C13—H13C	0.9600
C13—N1—C12	112.95 (16)	C9—C8—H8	120.9
C13—N1—H1B	106.2 (13)	C7—C8—H8	120.9
C12—N1—H1B	107.5 (14)	O2—C9—C8	116.35 (13)
C13—N1—H1A	111.7 (14)	O2—C9—C4	120.33 (15)
C12—N1—H1A	107.7 (13)	C8—C9—C4	123.32 (14)
H1B—N1—H1A	110.8 (19)	O3—C10—C11	114.33 (13)
O1—C1—O2	116.32 (16)	O3—C10—H10A	108.7
O1—C1—C2	126.56 (18)	C11—C10—H10A	108.7
O2—C1—C2	117.12 (15)	O3—C10—H10B	108.7
C3—C2—C1	120.98 (18)	C11—C10—H10B	108.7
C3—C2—H2	119.5	H10A—C10—H10B	107.6
C1—C2—H2	119.5	O4—C11—O5	127.04 (13)
C2—C3—C4	120.98 (15)	O4—C11—C10	119.35 (13)
C2—C3—H3	119.5	O5—C11—C10	113.50 (14)
C4—C3—H3	119.5	N1—C12—H12A	109.5
C9—C4—C5	116.59 (16)	N1—C12—H12B	109.5
C9—C4—C3	118.16 (15)	H12A—C12—H12B	109.5
C5—C4—C3	125.24 (14)	N1—C12—H12C	109.5
C6—C5—C4	121.69 (14)	H12A—C12—H12C	109.5
C6—C5—H5	119.2	H12B—C12—H12C	109.5
C4—C5—H5	119.2	N1—C13—H13A	109.5
C5—C6—C7	119.80 (14)	N1—C13—H13B	109.5
C5—C6—H6	120.1	H13A—C13—H13B	109.5
C7—C6—H6	120.1	N1—C13—H13C	109.5
O3—C7—C8	124.62 (13)	H13A—C13—H13C	109.5
O3—C7—C6	115.05 (13)	H13B—C13—H13C	109.5
C8—C7—C6	120.33 (15)	C9—O2—C1	122.27 (13)
C9—C8—C7	118.24 (13)	C7—O3—C10	117.87 (11)

C7—O3—C10—C11	76.32 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4	0.90 (1)	1.92 (1)	2.799 (2)	166 (2)
N1—H1*B*···O5^{i}	0.90 (1)	1.86 (1)	2.729 (3)	160 (2)

Symmetry code: (i) *x*−1, *y*, *z*.