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2,2,2-Trifluoro-*N*-(4-methyl-2-oxo-2*H*-chromen-7-yl)acetamide

Hong-Da Li and Bing-Zhu Yin*

Key Laboratory of Natural Resources of Changbai Mountain & Functional Molecules (Yanbian University), Ministry of Eduction, Yanji 133002, People's Republic of China
Correspondence e-mail: zqcong@ybu.edu.cn

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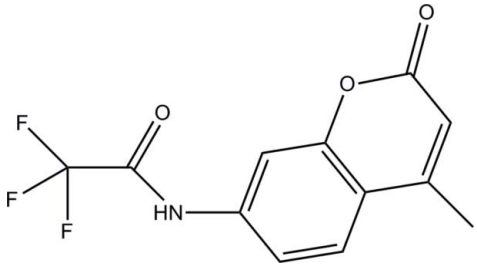
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.046; wR factor = 0.143; data-to-parameter ratio = 11.3.

In the title molecule, $C_{12}H_8F_3NO_3$, the trifluoromethyl group is rotationally disordered over three orientations in a 0.5:0.3:0.2 ratio. In the crystal, N—H···O hydrogen bonds link the molecules related by translation into chains along the c axis. The crystal packing exhibits π–π interactions between the pyran rings of neighboring molecules [centroid–centroid distance = 3.462 (4) Å] and short C···O contacts of 3.149 (4) Å.

Related literature

For applications of coumarin derivatives, see: Li *et al.* (2012). For potential applications of the title compound as a fluorescent probe for cyanide, see: Li *et al.* (2011).



Experimental

Crystal data

$C_{12}H_8F_3NO_3$ M_r = 271.19
Triclinic, $P\overline{1}$
a = 8.4897 (17) Å
b = 8.5777 (17) Å
c = 9.3677 (19) Å
α = 89.36 (3)°
β = 68.27 (3)°
γ = 65.12 (3)°
V = 566.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 293 K
0.33 × 0.25 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.954, T_{max} = 0.968
5577 measured reflections
2558 independent reflections
1660 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.143
S = 1.01
2558 reflections
227 parameters
84 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.05	2.8960 (19)	170

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5256).

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supplementary materials

Acta Cryst. (2012). E**68**, o1003 [doi:10.1107/S1600536812009634]

2,2,2-Trifluoro-*N*-(4-methyl-2-oxo-2*H*-chromen-7-yl)acetamide

Hong-Da Li and Bing-Zhu Yin

Comment

Coumarins, with the structure of benzopyrone, have many advantages including high fluorescence quantum yield, large Stokes shift, excellent light stability, and low toxicity. Therefore, coumarin derivatives have been used as fluorescent probes of pH, for detection of nitric oxide, nitroxide, and hydrogen peroxide so far. Moreover, coumarin derivatives have served as good chemosensors of anions including cyanide, fluoride, pyrophosphate, acetate, benzoate, and dihydrogenphosphate as well as various metal ions comprised of Hg (II), Cu(II), Zn(II), Ni(II), Ca(II), Pb(II), Mg(II), Fe(III), Al (III), Cr(III), and Ag(I) (Li *et al.*, 2012). Herein, we report the crystal structure of the title compound (I), a potential fluorescent probe for cyanide (Li *et al.*, 2011).

In (I) (Fig. 1), all bond lengths and angles are normal. Intermolecular N—H···O hydrogen bonds (Table 1) link the molecules related by translation along axis *c* into chains. The crystal packing exhibits $\pi\cdots\pi$ interactions between the pyran rings from the neighboring molecules [centroid-centroid distance of 3.462 (4) Å] and short C···O contacts of 3.149 (4) Å.

Experimental

A solution of 7-amino-4-methylcoumarin (100 mg, 0.57 mmol) and trifluoroacetic anhydride (360 mg, 1.70 mmol) was stirred in THF (5 ml) at room temperature for 1 h under N_2. Then the mixture was concentrated and the solid was recrystallized from THF to give the title compound (113.4 mg), yield 73.3%. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a mixture of tetrahydrofuran and petroleum (60–90 °C) at room temperature.

Refinement

C-bound H-atoms were placed in calculated positions (C—H 0.93 and 0.96 Å) and were included in the refinement in the riding model with U_{iso}(H) = 1.5 or 1.2 U_{eq}(C). N-bound atom H1 was placed in calculated position with N-H = 0.86 Å and refined with U_{iso}(H) = 1.2 U_{eq}(N). Trifluoromethyl group was treated as rotationally disordered over three orientations with the occupancies refined in the initial cycles, but in the final cycle they were fixed to 0.5, 0.3 and 0.2, respectively.

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 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* (Sheldrick, 2008).

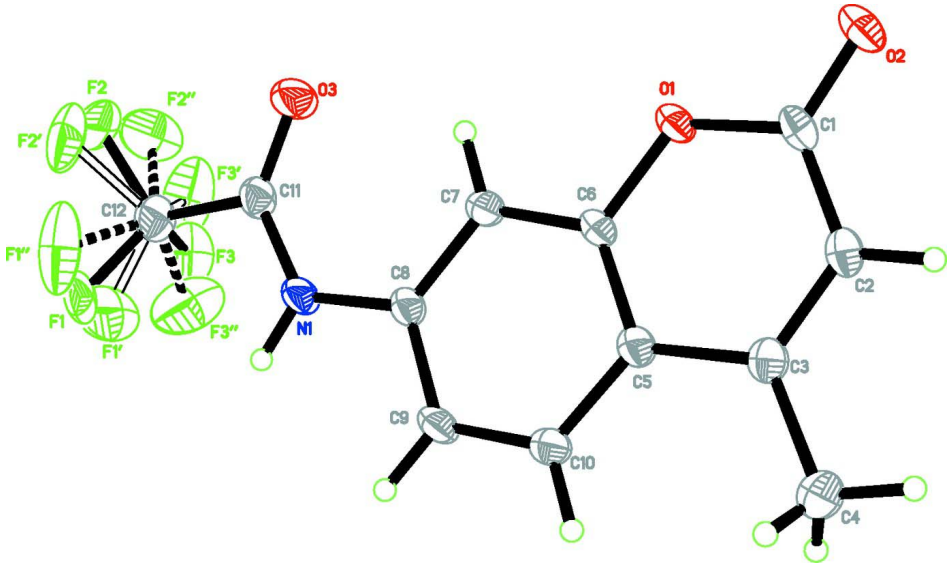

Figure 1

The molecular structure of (I) with the atomic numbering. Displacement ellipsoids are drawn at the 30% probalility level.

2,2,2-Trifluoro-*N*-(4-methyl-2-oxo-2*H*-chromen-7-yl)acetamide

Crystal data

$C_{12}H_8F_3NO_3$
$M_r = 271.19$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.4897\ (17)$ Å
$b = 8.5777\ (17)$ Å
$c = 9.3677\ (19)$ Å
$\alpha = 89.36\ (3)°$
$\beta = 68.27\ (3)°$
$\gamma = 65.12\ (3)°$
$V = 566.3\ (2)$ Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.590$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4341 reflections
$\theta = 3.3–27.7°$
$\mu = 0.15$ mm^{-1}
$T = 293$ K
Block, colourless
0.33 × 0.25 × 0.22 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)
$T_{min} = 0.954$, $T_{max} = 0.968$
5577 measured reflections
2558 independent reflections
1660 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -10\rightarrow11$
$k = -9\rightarrow11$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.143$
$S = 1.01$
2558 reflections
227 parameters
84 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0898P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.043$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Special details

Experimental. (See detailed section in the paper)

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}	Occ. (<1)
C1	0.2553 (3)	0.1720 (2)	0.69632 (18)	0.0411 (4)	
C2	0.2740 (3)	0.0012 (2)	0.66094 (19)	0.0428 (5)	
H2	0.2863	−0.0729	0.7336	0.051*	
C3	0.2746 (3)	−0.0569 (2)	0.52793 (19)	0.0378 (4)	
C4	0.2947 (4)	−0.2365 (3)	0.4954 (2)	0.0558 (6)	
H4A	0.3023	−0.2943	0.5827	0.084*	
H4B	0.4080	−0.3019	0.4038	0.084*	
H4C	0.1866	−0.2284	0.4789	0.084*	
C5	0.2568 (3)	0.0591 (2)	0.41442 (17)	0.0340 (4)	
C6	0.2416 (2)	0.2238 (2)	0.44841 (17)	0.0317 (4)	
C7	0.2285 (3)	0.3436 (2)	0.34783 (17)	0.0345 (4)	
H7	0.2167	0.4531	0.3753	0.041*	
C8	0.2336 (3)	0.2953 (2)	0.20462 (17)	0.0326 (4)	
C9	0.2482 (3)	0.1313 (2)	0.16628 (18)	0.0411 (5)	
H9	0.2504	0.1001	0.0706	0.049*	
C10	0.2595 (3)	0.0160 (2)	0.26924 (19)	0.0406 (4)	
H10	0.2690	−0.0927	0.2423	0.049*	
C11	0.2084 (3)	0.5682 (2)	0.10611 (19)	0.0367 (4)	
C12	0.2258 (3)	0.6488 (3)	−0.0436 (2)	0.0470 (5)	
F1	0.1414 (13)	0.6068 (13)	−0.1225 (11)	0.0525 (18)	0.50
F2	0.1399 (19)	0.8175 (10)	−0.0063 (12)	0.065 (3)	0.50
F3	0.4003 (12)	0.5937 (15)	−0.1389 (9)	0.068 (3)	0.50
F1′	0.215 (4)	0.570 (3)	−0.150 (3)	0.115 (8)	0.30
F2′	0.087 (3)	0.821 (3)	−0.016 (2)	0.078 (6)	0.30
F3′	0.383 (2)	0.669 (3)	−0.096 (3)	0.098 (6)	0.30
F1″	0.102 (3)	0.685 (4)	−0.094 (3)	0.108 (10)	0.20
F2″	0.273 (6)	0.776 (3)	−0.0344 (18)	0.090 (6)	0.20
F3″	0.394 (5)	0.524 (2)	−0.166 (2)	0.104 (9)	0.20
N1	0.2276 (2)	0.40564 (18)	0.09096 (15)	0.0368 (4)	
H1	0.2373	0.3638	0.0034	0.044*	
O1	0.2404 (2)	0.27853 (15)	0.58734 (12)	0.0395 (3)	
O2	0.2518 (3)	0.23117 (19)	0.81527 (14)	0.0599 (5)	

O3	0.1836 (3)	0.65505 (17)	0.22024 (15)	0.0554 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0547 (12)	0.0453 (10)	0.0236 (7)	−0.0192 (9)	−0.0200 (8)	0.0085 (6)
C2	0.0605 (13)	0.0417 (10)	0.0304 (8)	−0.0229 (9)	−0.0231 (8)	0.0157 (7)
C3	0.0504 (12)	0.0351 (9)	0.0319 (8)	−0.0214 (8)	−0.0184 (8)	0.0117 (7)
C4	0.0946 (19)	0.0414 (11)	0.0499 (10)	−0.0384 (12)	−0.0391 (12)	0.0206 (8)
C5	0.0453 (11)	0.0336 (8)	0.0280 (7)	−0.0194 (8)	−0.0179 (7)	0.0090 (6)
C6	0.0415 (10)	0.0343 (8)	0.0222 (7)	−0.0165 (7)	−0.0162 (7)	0.0043 (6)
C7	0.0512 (11)	0.0293 (8)	0.0292 (7)	−0.0202 (8)	−0.0200 (7)	0.0070 (6)
C8	0.0444 (10)	0.0330 (8)	0.0267 (7)	−0.0192 (7)	−0.0188 (7)	0.0096 (6)
C9	0.0690 (14)	0.0402 (9)	0.0301 (8)	−0.0298 (9)	−0.0302 (9)	0.0096 (7)
C10	0.0655 (13)	0.0327 (8)	0.0356 (8)	−0.0264 (9)	−0.0278 (9)	0.0088 (7)
C11	0.0477 (11)	0.0370 (9)	0.0359 (8)	−0.0234 (8)	−0.0227 (8)	0.0146 (7)
C12	0.0608 (15)	0.0455 (11)	0.0448 (10)	−0.0288 (10)	−0.0262 (11)	0.0207 (8)
F1	0.078 (5)	0.055 (4)	0.056 (3)	−0.038 (4)	−0.052 (3)	0.037 (3)
F2	0.102 (8)	0.032 (3)	0.066 (3)	−0.026 (4)	−0.043 (5)	0.028 (2)
F3	0.049 (3)	0.093 (8)	0.056 (4)	−0.035 (5)	−0.011 (3)	0.032 (5)
F1′	0.23 (2)	0.080 (10)	0.067 (9)	−0.075 (15)	−0.088 (15)	0.039 (7)
F2′	0.067 (7)	0.073 (8)	0.070 (6)	−0.007 (4)	−0.030 (4)	0.040 (5)
F3′	0.056 (7)	0.112 (15)	0.127 (15)	−0.047 (10)	−0.030 (9)	0.076 (11)
F1″	0.078 (10)	0.14 (2)	0.119 (15)	−0.043 (14)	−0.058 (10)	0.089 (16)
F2″	0.175 (18)	0.073 (11)	0.063 (7)	−0.083 (12)	−0.057 (12)	0.038 (7)
F3″	0.135 (16)	0.072 (9)	0.044 (5)	−0.035 (10)	0.012 (7)	0.007 (6)
N1	0.0603 (11)	0.0358 (7)	0.0268 (6)	−0.0268 (7)	−0.0245 (7)	0.0120 (5)
O1	0.0628 (9)	0.0376 (6)	0.0253 (5)	−0.0235 (6)	−0.0240 (6)	0.0071 (4)
O2	0.0991 (13)	0.0576 (9)	0.0330 (6)	−0.0343 (9)	−0.0380 (8)	0.0094 (6)
O3	0.0959 (13)	0.0392 (7)	0.0502 (8)	−0.0366 (8)	−0.0420 (8)	0.0136 (6)

Geometric parameters (Å, º)

C1—O2	1.215 (2)	C8—N1	1.417 (2)
C1—O1	1.367 (2)	C9—C10	1.373 (2)
C1—C2	1.434 (3)	C9—H9	0.9300
C2—C3	1.345 (2)	C10—H10	0.9300
C2—H2	0.9300	C11—O3	1.209 (2)
C3—C5	1.454 (2)	C11—N1	1.337 (2)
C3—C4	1.499 (2)	C11—C12	1.542 (2)
C4—H4A	0.9600	C12—F1″	1.23 (2)
C4—H4B	0.9600	C12—F1′	1.259 (19)
C4—H4C	0.9600	C12—F3	1.287 (8)
C5—C6	1.393 (2)	C12—F2	1.297 (8)
C5—C10	1.403 (2)	C12—F3′	1.327 (15)
C6—O1	1.3835 (17)	C12—F2″	1.327 (14)
C6—C7	1.384 (2)	C12—F1	1.343 (7)
C7—C8	1.388 (2)	C12—F2′	1.400 (18)
C7—H7	0.9300	C12—F3″	1.42 (2)
C8—C9	1.398 (2)	N1—H1	0.8600

O2—C1—O1	116.61 (16)	F1′—C12—F3′	111.2 (12)
O2—C1—C2	125.67 (16)	F3—C12—F3′	30.8 (9)
O1—C1—C2	117.72 (13)	F2—C12—F3′	84.4 (9)
C3—C2—C1	123.21 (15)	F1″—C12—F2″	114.2 (12)
C3—C2—H2	118.4	F1′—C12—F2″	134.9 (11)
C1—C2—H2	118.4	F3—C12—F2″	72.7 (13)
C2—C3—C5	118.12 (15)	F2—C12—F2″	42.9 (14)
C2—C3—C4	121.53 (15)	F3′—C12—F2″	42.7 (10)
C5—C3—C4	120.35 (14)	F1″—C12—F1	27.2 (16)
C3—C4—H4A	109.5	F1′—C12—F1	22.9 (14)
C3—C4—H4B	109.5	F3—C12—F1	105.9 (5)
H4A—C4—H4B	109.5	F2—C12—F1	106.3 (5)
C3—C4—H4C	109.5	F3′—C12—F1	129.8 (11)
H4A—C4—H4C	109.5	F2″—C12—F1	135.9 (10)
H4B—C4—H4C	109.5	F1″—C12—F2′	59.8 (14)
C6—C5—C10	116.77 (14)	F1′—C12—F2′	105.0 (11)
C6—C5—C3	118.39 (13)	F3—C12—F2′	123.9 (9)
C10—C5—C3	124.81 (14)	F2—C12—F2′	20.2 (10)
O1—C6—C7	115.01 (13)	F3′—C12—F2′	101.2 (11)
O1—C6—C5	121.47 (13)	F2″—C12—F2′	62.0 (14)
C7—C6—C5	123.52 (13)	F1—C12—F2′	87.0 (9)
C6—C7—C8	117.96 (14)	F1″—C12—F3″	104.1 (14)
C6—C7—H7	121.0	F1′—C12—F3″	59.8 (11)
C8—C7—H7	121.0	F3—C12—F3″	28.5 (10)
C7—C8—C9	120.26 (14)	F2—C12—F3″	135.5 (9)
C7—C8—N1	122.56 (14)	F3′—C12—F3″	59.3 (10)
C9—C8—N1	117.18 (12)	F2″—C12—F3″	100.8 (12)
C10—C9—C8	120.32 (13)	F1—C12—F3″	82.7 (15)
C10—C9—H9	119.8	F2′—C12—F3″	139.8 (12)
C8—C9—H9	119.8	F1″—C12—C11	120.0 (10)
C9—C10—C5	121.17 (14)	F1′—C12—C11	115.8 (9)
C9—C10—H10	119.4	F3—C12—C11	111.5 (4)
C5—C10—H10	119.4	F2—C12—C11	109.2 (4)
O3—C11—N1	127.88 (15)	F3′—C12—C11	109.6 (8)
O3—C11—C12	117.70 (15)	F2″—C12—C11	108.7 (5)
N1—C11—C12	114.40 (14)	F1—C12—C11	112.2 (4)
F1″—C12—F1′	47.4 (13)	F2′—C12—C11	113.0 (8)
F1″—C12—F3	119.8 (13)	F3″—C12—C11	106.8 (10)
F1′—C12—F3	84.0 (13)	C11—N1—C8	126.56 (12)
F1″—C12—F2	79.3 (15)	C11—N1—H1	116.7
F1′—C12—F2	122.1 (9)	C8—N1—H1	116.7
F3—C12—F2	111.5 (6)	C1—O1—C6	121.07 (13)
F1″—C12—F3′	130.4 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1···O2^{i}	0.86	2.05	2.8960 (19)	170

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