Xiang Yu, Xu-Li Yang, You-Fang Zhao, Yan Qin, Jing Yan and Ya-Fang Chen*

The crystal structure of (1*E*,2*E*)-2-methyl-4-((7-oxo-7*H*-furo[3,2-*g*]chromen-9-yl)oxy)but-2-enal *O*-isonicotinoyl oxime–trichloromethane (3/1), C₆₇H₄₉Cl₃N₆O₁₈

$\beta = 77.885(3)^\circ$, $\gamma = 72.021(3)^\circ$, $V = 3075.20(19) \text{ \AA}^3$, $Z = 2$, $R_{\text{gt}}(F) = 0.0658$, $wR_{\text{ref}}(F^2) = 0.1558$, $T = 291.2(3) \text{ K}$.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.29 \times 0.27 \times 0.26 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.23 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω -scans
θ_{max} , completeness:	28.4° , $>90\%$ (up to 25.34° , $>99\%$)
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	72823, 13901, 0.055
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 8889
$N(\text{param})_{\text{refined}}$:	850
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

Source of material

In a representative experiment 9-((3-methylbut-2-en-1-yl)oxy)-7*H*-furo[3,2-*g*]chromen-7-one (1.0 mmol) reacted with SeO₂ (1.1 mmol) in dioxane (10 mL) at 80 °C to afford the aldehyde. A mixture of the aldehyde (1.0 mmol) hydroxylamine hydrochloride (2.0 mmol) in pyridine (0.5 mL) and absolute ethanol (10 mL) was stirred at 60 °C, and then reacted with isonicotinic acid, *N,N'*-dicyclohexylcarbodiimide and 4-dimethylaminopyridine in dry dichloromethane at room temperature. Suitable colorless block crystals of this compound were then obtained after recrystallization from trichloromethane.

Experimental details

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. Hydrogen atoms were treated using a riding model.

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Abstract

C₆₇H₄₉Cl₃N₆O₁₈, triclinic, $P\bar{1}$ (no. 2), $a = 12.3281(4) \text{ \AA}$, $b = 15.8887(5) \text{ \AA}$, $c = 16.9037(6) \text{ \AA}$, $\alpha = 89.015(2)^\circ$,

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cl1	0.58491(10)	0.19058(7)	0.66204(7)	0.1149(4)
Cl2	0.56603(14)	0.25465(12)	0.50636(8)	0.1525(6)
Cl3	0.66470(8)	0.33636(6)	0.60846(7)	0.0946(3)
C67	0.5610(3)	0.2840(2)	0.60546(19)	0.0692(8)
H67	0.4834	0.3251	0.6285	0.083*
O1	0.80945(18)	0.61618(13)	−0.03009(11)	0.0665(5)
O2	0.71619(15)	0.62087(11)	−0.12853(10)	0.0473(4)
O3	0.50045(17)	0.63883(12)	−0.33759(11)	0.0615(5)
O4	0.66282(16)	0.70181(11)	−0.25797(10)	0.0530(5)
O5	0.83821(16)	1.03289(11)	−0.45831(10)	0.0530(5)
O6	0.8420(2)	1.06928(15)	−0.58807(13)	0.0831(7)
N1	0.77585(19)	0.97342(13)	−0.47212(13)	0.0513(5)
N2	1.0134(2)	1.27540(16)	−0.47343(16)	0.0678(7)
C1	0.7470(2)	0.58604(18)	−0.05852(16)	0.0531(7)
C2	0.7027(3)	0.5149(2)	−0.02702(17)	0.0650(8)
H2	0.7214	0.4899	0.0204	0.078*
C3	0.6360(2)	0.48429(18)	−0.06392(17)	0.0592(7)
H3	0.6106	0.4377	−0.0424	0.071*
C4	0.6029(2)	0.52196(16)	−0.13596(15)	0.0461(6)
C5	0.5301(2)	0.49407(16)	−0.17468(16)	0.0505(6)
H5	0.5021	0.4480	−0.1551	0.061*
C6	0.5003(2)	0.53587(16)	−0.24257(15)	0.0464(6)
C7	0.4257(3)	0.52903(18)	−0.29548(18)	0.0603(7)
H7	0.3834	0.4893	−0.2926	0.072*
C8	0.4292(3)	0.5904(2)	−0.34900(19)	0.0708(9)
H8	0.3878	0.5999	−0.3901	0.085*
C9	0.5442(2)	0.60502(16)	−0.27120(14)	0.0439(6)
C10	0.6178(2)	0.63475(15)	−0.23527(14)	0.0401(5)
C11	0.6451(2)	0.59129(15)	−0.16618(14)	0.0409(5)
C12	0.6443(2)	0.74217(16)	−0.33208(14)	0.0453(6)
H12A	0.5614	0.7692	−0.3293	0.054*
H12B	0.6755	0.6980	−0.3768	0.054*
C13	0.7047(2)	0.81056(15)	−0.34447(14)	0.0435(6)
H13	0.7419	0.8197	−0.3044	0.052*
C14	0.7092(2)	0.85944(16)	−0.40872(14)	0.0441(6)
C15	0.6556(3)	0.8510(2)	−0.47887(18)	0.0726(9)
H15A	0.6265	0.8012	−0.4724	0.109*
H15B	0.7136	0.8423	−0.5284	0.109*
H15C	0.5925	0.9040	−0.4807	0.109*
C16	0.7704(2)	0.92503(16)	−0.41205(15)	0.0457(6)
H16	0.8055	0.9314	−0.3699	0.055*
C17	0.8605(2)	1.08034(17)	−0.52348(17)	0.0513(6)
C18	0.9978(3)	1.21611(19)	−0.42001(19)	0.0661(8)
H18	1.0215	1.2181	−0.3715	0.079*
C19	0.9484(2)	1.15128(17)	−0.43168(17)	0.0552(7)
H19	0.9388	1.1113	−0.3920	0.066*
C20	0.9141(2)	1.14754(15)	−0.50331(15)	0.0460(6)
C21	0.9301(2)	1.20863(16)	−0.56068(17)	0.0536(7)
H21	0.9081	1.2077	−0.6099	0.064*
C22	0.9797(3)	1.27081(18)	−0.5426(2)	0.0654(8)
H22	0.9902	1.3119	−0.5809	0.078*
O7	1.14311(17)	0.68087(13)	0.73170(13)	0.0676(6)
O8	1.04284(15)	0.58895(11)	0.72730(10)	0.0494(4)
O9	0.82594(18)	0.38705(12)	0.71507(11)	0.0613(5)
O10	0.91144(17)	0.53924(11)	0.64773(10)	0.0578(5)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O11	0.75199(16)	0.69168(12)	0.29413(10)	0.0561(5)
O12	0.6475(2)	0.64219(15)	0.22222(13)	0.0814(7)
N3	0.7337(2)	0.62642(15)	0.35079(13)	0.0557(6)
N4	0.7390(3)	0.89209(17)	0.06676(16)	0.0785(8)
C23	1.1107(2)	0.62244(18)	0.76430(17)	0.0545(7)
C24	1.1360(3)	0.5836(2)	0.83891(19)	0.0669(8)
H24	1.1791	0.6061	0.8670	0.080*
C25	1.0998(3)	0.51666(19)	0.86866(18)	0.0621(8)
H25	1.1190	0.4930	0.9164	0.074*
C26	1.0318(2)	0.48042(16)	0.82877(15)	0.0449(6)
C27	0.9934(2)	0.40974(17)	0.85734(15)	0.0501(6)
H27	1.0128	0.3824	0.9038	0.060*
C28	0.9259(2)	0.38121(16)	0.81516(15)	0.0472(6)
C29	0.8692(3)	0.31373(18)	0.82556(17)	0.0596(7)
H29	0.8722	0.2731	0.8660	0.072*
C30	0.8122(3)	0.32081(19)	0.76647(18)	0.0686(9)
H30	0.7674	0.2848	0.7601	0.082*
C31	0.8970(2)	0.42357(16)	0.74570(14)	0.0445(6)
C32	0.9348(2)	0.49381(15)	0.71436(14)	0.0426(6)
C33	1.0035(2)	0.52015(15)	0.75797(14)	0.0409(5)
C34	0.8543(2)	0.50722(16)	0.59578(14)	0.0454(6)
H34A	0.7759	0.5104	0.6241	0.055*
H34B	0.8971	0.4460	0.5780	0.055*
C35	0.8498(2)	0.56442(15)	0.52490(14)	0.0448(6)
H35	0.8921	0.6043	0.5199	0.054*
C36	0.7905(2)	0.56272(15)	0.46874(14)	0.0437(6)
C37	0.7187(3)	0.5020(2)	0.4682(2)	0.0835(11)
H37A	0.7213	0.4672	0.5151	0.125*
H37B	0.7495	0.4635	0.4202	0.125*
H37C	0.6393	0.5364	0.4689	0.125*
C38	0.7942(2)	0.62329(16)	0.40303(15)	0.0468(6)
H38	0.8413	0.6596	0.3997	0.056*
C39	0.7002(3)	0.69116(18)	0.23070(16)	0.0553(7)
C40	0.7920(3)	0.8786(2)	0.12809(19)	0.0755(9)
H40	0.8372	0.9142	0.1345	0.091*
C41	0.7843(3)	0.81378(19)	0.18402(18)	0.0659(8)
H41	0.8233	0.8063	0.2264	0.079*
C42	0.7172(2)	0.76149(17)	0.17439(15)	0.0518(7)
C43	0.6626(3)	0.77372(19)	0.10986(16)	0.0629(8)
H43	0.6181	0.7383	0.1013	0.075*
C44	0.6758(3)	0.8398(2)	0.05848(18)	0.0770(10)
H44	0.6381	0.8483	0.0154	0.092*
O13	0.45977(19)	−0.29029(13)	1.01382(12)	0.0712(6)
O14	0.46331(15)	−0.19020(11)	0.92236(10)	0.0497(4)
O15	0.47470(18)	0.02719(12)	0.72234(11)	0.0646(6)
O16	0.54589(17)	−0.06116(12)	0.87266(11)	0.0628(5)
O17	0.91324(16)	0.11265(11)	1.02146(10)	0.0552(5)
O18	0.95084(19)	0.24122(14)	0.98983(12)	0.0734(6)
N5	0.8575(2)	0.12372(15)	0.95355(13)	0.0589(6)
N6	1.1182(2)	0.14509(17)	1.23502(14)	0.0657(7)
C45	0.4250(2)	−0.25836(17)	0.95524(17)	0.0523(6)
C46	0.3474(2)	−0.28541(17)	0.91512(17)	0.0553(7)
H46	0.3189	−0.3309	0.9359	0.066*
C47	0.3155(2)	−0.24710(16)	0.84939(16)	0.0503(6)
H47	0.2663	−0.2669	0.8249	0.060*
C48	0.3555(2)	−0.17574(15)	0.81569(15)	0.0437(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C49	0.3237(2)	−0.13378(17)	0.74784(15)	0.0486(6)
H49	0.2741	−0.1510	0.7216	0.058*
C50	0.3672(2)	−0.06549(16)	0.71983(15)	0.0474(6)
C51	0.3558(3)	−0.00791(19)	0.65342(17)	0.0643(8)
H51	0.3119	−0.0074	0.6148	0.077*
C52	0.4199(3)	0.0439(2)	0.65813(19)	0.0754(10)
H52	0.4272	0.0874	0.6217	0.090*
C53	0.4414(2)	−0.04088(16)	0.76065(15)	0.0468(6)
C54	0.4755(2)	−0.08075(16)	0.82914(15)	0.0451(6)
C55	0.4300(2)	−0.14905(15)	0.85507(14)	0.0420(6)
C56	0.6003(2)	0.00362(16)	0.84509(16)	0.0496(6)
H56A	0.5417	0.0596	0.8409	0.059*
H56B	0.6508	−0.0148	0.7920	0.059*
C57	0.6697(2)	0.01388(16)	0.90423(15)	0.0467(6)
H57	0.6677	−0.0198	0.9497	0.056*
C58	0.7338(2)	0.06748(16)	0.89651(15)	0.0495(6)
C59	0.7428(4)	0.1261(2)	0.8267(2)	0.0965(13)
H59A	0.7212	0.1034	0.7821	0.145*
H59B	0.8216	0.1274	0.8105	0.145*
H59C	0.6913	0.1849	0.8428	0.145*
C60	0.7978(2)	0.07097(16)	0.95867(15)	0.0468(6)
H60	0.7950	0.0345	1.0022	0.056*
C61	0.9570(2)	0.17911(18)	1.03140(16)	0.0505(6)
C62	1.0453(3)	0.1013(2)	1.22677(16)	0.0615(7)
H62	1.0293	0.0636	1.2671	0.074*
C63	0.9916(2)	0.10800(18)	1.16218(16)	0.0533(7)
H63	0.9420	0.0750	1.1588	0.064*
C64	1.0130(2)	0.16479(17)	1.10254(15)	0.0467(6)
C65	1.0869(2)	0.21269(18)	1.11085(16)	0.0540(7)
H65	1.1016	0.2527	1.0725	0.065*
C66	1.1382(3)	0.2002(2)	1.17675(18)	0.0621(8)
H66	1.1893	0.2315	1.1811	0.074*

Discussion

Coumarins are natural products with diverse bioactivities such as anti-inflammatory [5], anti-tumor [6], and antimicrobial activity [7]. In order to discover more coumarin derivatives with potential bioactivities, here we prepared a coumarin-based derivative by introduction of pyridine into the coumarin.

There are three crystallographically independent molecules ($3(C_{22}H_{16}N_2O_6)$) and one solvent molecule ($CHCl_3$)

in the asymmetric unit, and all bond lengths are in normal ranges and they are similar with the known compound [8, 9]. In one of independent molecule ($C_{22}H_{16}N_2O_6$), the typical bond length of C—C double bond (atoms C14—C16) was 1.326(3) Å, and C—N double bond (atoms N1—C16) with 1.266(3) Å. In addition, both of the configuration of the C—C double bond and the configuration C—N double bond are in *E* configuration.

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