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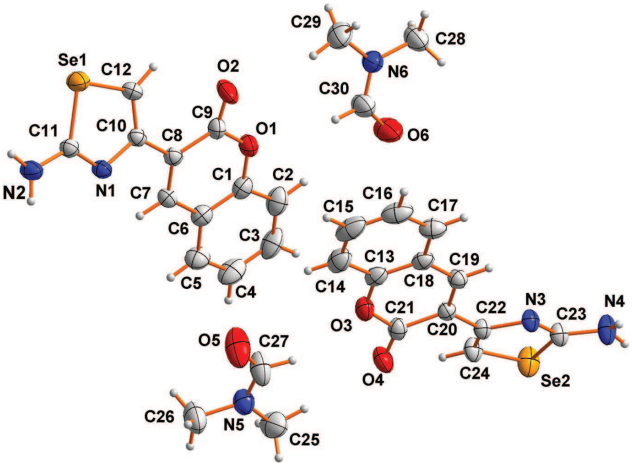
Crystal structure of 3-(2-amino-1,3-selenazol-4-yl)-2*H*-chromen-2-one – dimethylformamide (1/1), $C_{15}H_{15}N_3O_3Se$



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Abstract

$C_{15}H_{15}N_3O_3Se$, triclinic, $P\bar{1}$ (no. 2), $a = 9.5686(7)$ Å, $b = 12.3795(9)$ Å, $c = 14.7854(11)$ Å, $\alpha = 93.844(2)°$, $\beta = 106.344(2)°$, $\gamma = 110.816(2)°$, $V = 1543.4(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0489$, $wR_{ref}(F^2) = 0.1066$, $T = 300$ K.

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Table 1: Data collection and handling.

Crystal:	Dark red block
Size:	0.50 × 0.49 × 0.48 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	24.5 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	57°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	52857, 7728, 0.082
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4768
$N(param)_{refined}$:	417
Programs:	Bruker programs [1], SHELX [2]

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

3-(2-Bromo-acetyl)-chromen-2-one (0.05 mol) and selenourea (0.05 mol) were dissolved in 5 mL methanol-water (1 : 1) contaning 0.08 g NaF. The mixture was stirred at room temperature for 30 minutes. After the completion of the reaction, water (10 mL) was added to the reaction mixture and extracted with ethyl acetate. The organic layer was dried over anhydrous Na_2SO_4. The solvent was evaporated under reduced pressure and the resulting product was further purified by column chromatography (hexane:ethyl acetate, 7 : 3) [3, 4]. Single crystals suitable for X-ray structure analysis were obtained by dissolving the compound in methanol and DMF. Then, the solvents were concentrated using a rotary evaporator. The mixture was cooled to room temperature and allowed to rest overnight. The title compound formed reddish brown parallelepipeds in a yield of 50%.

Experimental details

The C—H atoms were constrained to an ideal geometry, with C—H distances of 0.93–0.96 Å. The U_{iso} values of the hydrogen

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Se1	0.68571(4)	0.85803(3)	0.33979(3)	0.04634(12)
Se2	−0.35364(4)	0.06206(3)	0.83165(3)	0.05157(13)
O1	0.1012(3)	0.44876(19)	0.35694(18)	0.0488(6)
O2	0.3386(3)	0.4889(2)	0.3510(2)	0.0732(9)
O3	0.2536(3)	0.47950(19)	0.85285(17)	0.0470(6)
O4	0.0094(3)	0.4379(2)	0.8474(2)	0.0567(7)
O5	0.4493(4)	0.8236(3)	0.8821(3)	0.0992(12)
O6	−0.0475(4)	0.1232(3)	0.4061(3)	0.0902(11)
N1	0.4604(3)	0.8586(2)	0.41660(18)	0.0353(6)
N2	0.6505(4)	1.0473(3)	0.4351(2)	0.0507(8)
N3	−0.0459(3)	0.0954(2)	0.90750(18)	0.0352(6)
N4	−0.2162(4)	−0.0862(3)	0.9236(3)	0.0527(8)
N5	0.2042(4)	0.8100(3)	0.8693(2)	0.0508(8)
N6	0.1661(3)	0.1283(3)	0.3681(2)	0.0486(7)
C1	−0.0168(4)	0.4795(3)	0.3700(2)	0.0420(8)
C2	−0.1565(5)	0.3905(4)	0.3643(3)	0.0583(10)
H2	−0.1697	0.3123	0.3513	0.070*
C3	−0.2765(5)	0.4186(4)	0.3780(3)	0.0657(12)
H3	−0.3710	0.3590	0.3754	0.079*
C4	−0.2577(5)	0.5349(4)	0.3955(3)	0.0643(11)
H4	−0.3402	0.5532	0.4037	0.077*
C5	−0.1186(4)	0.6229(4)	0.4009(3)	0.0520(9)
H5	−0.1069	0.7009	0.4132	0.062*
C6	0.0063(4)	0.5974(3)	0.3883(2)	0.0384(7)
C7	0.1553(4)	0.6830(3)	0.3915(2)	0.0353(7)
H7	0.1724	0.7622	0.4027	0.042*
C8	0.2717(4)	0.6537(3)	0.3789(2)	0.0329(7)
C9	0.2459(4)	0.5289(3)	0.3615(3)	0.0437(8)
C10	0.4216(4)	0.7410(3)	0.3776(2)	0.0319(7)
C11	0.5888(4)	0.9301(3)	0.4054(2)	0.0356(7)
C12	0.5199(4)	0.7204(3)	0.3351(2)	0.0389(8)
H12	0.5059	0.6462	0.3069	0.047*
C13	0.3873(4)	0.4553(3)	0.8703(2)	0.0418(8)
C14	0.5229(5)	0.5437(4)	0.8676(3)	0.0588(10)
H14	0.5226	0.6155	0.8527	0.071*
C15	0.6585(5)	0.5217(4)	0.8878(3)	0.0670(13)
H15	0.7516	0.5799	0.8869	0.080*
C16	0.6600(5)	0.4152(4)	0.9094(3)	0.0615(11)
H16	0.7534	0.4027	0.9233	0.074*
C17	0.5239(4)	0.3281(4)	0.9102(3)	0.0516(9)
H17	0.5249	0.2562	0.9240	0.062*
C18	0.3833(4)	0.3469(3)	0.8904(2)	0.0390(8)
C19	0.2353(4)	0.2617(3)	0.8891(2)	0.0364(7)
H19	0.2295	0.1872	0.8998	0.044*
C20	0.1040(4)	0.2845(3)	0.8732(2)	0.0324(7)
C21	0.1131(4)	0.4023(3)	0.8570(2)	0.0396(8)
C22	−0.0482(4)	0.1958(3)	0.8702(2)	0.0328(7)
C23	−0.1867(4)	0.0182(3)	0.8956(2)	0.0377(8)
C24	−0.1914(4)	0.1980(3)	0.8289(2)	0.0422(8)
H24	−0.2070	0.2603	0.8021	0.051*
C25	0.0468(5)	0.7364(4)	0.8673(3)	0.0719(13)
H25A	0.0377	0.6565	0.8660	0.108*
H25B	−0.0298	0.7406	0.8110	0.108*
H25C	0.0281	0.7633	0.9235	0.108*
C26	0.2389(6)	0.9318(4)	0.8623(4)	0.0859(16)
H26A	0.3338	0.9806	0.9132	0.129*
H26B	0.1526	0.9514	0.8673	0.129*
H26C	0.2534	0.9443	0.8016	0.129*
C27	0.3141(5)	0.7687(4)	0.8796(4)	0.0713(13)
H27	0.2869	0.6902	0.8855	0.086*
C28	0.1135(5)	0.0022(3)	0.3476(3)	0.0674(12)
H28A	0.0591	−0.0315	0.3908	0.101*
H28B	0.2032	−0.0186	0.3555	0.101*
H28C	0.0430	−0.0274	0.2828	0.101*
C29	0.3144(5)	0.2000(4)	0.3568(4)	0.0705(12)
H29A	0.3360	0.2816	0.3757	0.106*
H29B	0.3077	0.1848	0.2909	0.106*
H29C	0.3979	0.1813	0.3964	0.106*
C30	0.0803(5)	0.1763(4)	0.3969(3)	0.0680(12)
H30	0.1207	0.2580	0.4117	0.082*
H2A	0.616(4)	1.077(3)	0.474(2)	0.064(13)*
H4A	−0.138(4)	−0.093(4)	0.964(3)	0.088(16)*
H2B	0.736(3)	1.089(3)	0.425(3)	0.058(12)*
H4B	−0.309(2)	−0.129(3)	0.923(3)	0.064(13)*

atoms of the methyl groups were set to $1.5U_{eq}(C_{methyl})$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}(C)$. Nitrogen-bound hydrogen atoms were refined freely.

Comment

Organoselenium compounds have attracted great interest due to their potential application as anticancer, antioxidant, enzyme modulators, antitumors, antimicrobials, antihypertensive agents and cytokine inducers [3–5]. The title compound also constitutes a coumarin moiety [6], which is known to exhibit wide range of medical applications such as an inhibitor of Alzheimer's disease [7]. In continuation of our interest in the development of organoselenium compounds, herein we report the structure of a coumaryl 1,3-selenazole.

The asymmetric unit of the crystal structure contains two independent $C_{12}H_8N_2O_2Se$ molecules and two molecules of dimethylformamide (*cf*. the figure). All bond lengths in the molecule are within normal ranges. The C—Se lengths are in the range of 1.851(3)–1.891(3) Å. The coumarin ring and the 2-amino-1,3-selenazole ring are syn-periplanar to each other with a torsion angle of C(9)—C(8)—C(10)—C(12) to be 20.9(5)°. In addition, classical hydrogen bond exists between 3-(2-amino-1,3-selenazol-4-yl)-2*H*-chromen-2-one and DMF molecules.

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