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Crystal structure of (Z)-6-(dimethylamino)-3,3-bis(4-(dimethylamino)phenyl)-2-(2-(quinoxalin-2-ylmethylene)hydrazinyl)-2,3-dihydroinden-1-one, C₃₅H₃₅N₇O

Table 1: Data collection and handling.

Crystal:	Yellow cube
Size:	0.30 × 0.20 × 0.05 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.63 mm ⁻¹
Diffractometer, scan mode:	SuperNova, AtlasS2, ω
$\theta_{\max}$, completeness:	71.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10105, 5642, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4888
$N(\text{param})_{\text{refined}}$:	394
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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Abstract

C₃₅H₃₅N₇O, triclinic, $P\bar{1}$ (no. 2), $a = 10.2870(3)$ Å, $b = 11.9918(4)$ Å, $c = 13.8995(5)$ Å, $\alpha = 109.210(3)^\circ$, $\beta = 96.833(3)^\circ$, $\gamma = 107.594(3)^\circ$, $Z = 2$, $V = 1497.32(9)$ Å³, $R_{\text{gt}}(F) = 0.0401$, $wR_{\text{ref}}(F^2) = 0.1091$, $T = 110(1)$ K.

CCDC no.: 1915612

The asymmetric unit of the molecular 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.

Source of material

All the solvents and starting materials were commercially available and were used without further handling. The clear yellow crystals of the title compound was synthesized by two steps. **Synthesis of the intermediate crystal violet lactone hydrazide.** To a 250 mL flask-2-neck, crystal violet lactone (4 mmol, 1.66 g) was dissolved in a mixed solvent

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.22840(11)	0.82009(10)	0.23278(9)	0.0348(2)
N1	0.27066(11)	0.70328(11)	0.32885(9)	0.0242(2)
N2	0.37850(11)	0.67001(11)	0.29611(9)	0.0247(2)
N3	0.58578(12)	0.68684(11)	0.11347(9)	0.0266(2)
N4	0.67121(13)	0.52223(12)	0.19103(10)	0.0323(3)
N5	−0.20647(13)	0.87367(13)	0.40611(11)	0.0355(3)
N6	0.53696(13)	0.86631(12)	0.82556(10)	0.0320(3)
N7	0.00432(14)	0.10147(12)	0.19314(11)	0.0382(3)
C1	0.67407(14)	0.63007(13)	0.07083(11)	0.0269(3)
C2	0.72297(15)	0.65301(15)	−0.01439(11)	0.0326(3)
H2	0.7009	0.7111	−0.0374	0.039*
C3	0.80254(16)	0.58999(16)	−0.06298(12)	0.0374(4)
H3	0.8349	0.6061	−0.1185	0.045*
C4	0.83592(16)	0.50102(17)	−0.02972(12)	0.0392(4)
H4	0.8866	0.4560	−0.0655	0.047*
C5	0.79443(16)	0.48007(16)	0.05493(12)	0.0369(3)
H5	0.8198	0.4234	0.0781	0.044*
C6	0.71321(14)	0.54465(14)	0.10677(11)	0.0292(3)
C7	0.58788(14)	0.57816(14)	0.23093(11)	0.0289(3)
H7	0.5576	0.5651	0.2881	0.035*
C8	0.54125(13)	0.65818(13)	0.19083(10)	0.0255(3)
C9	0.43582(14)	0.70846(13)	0.22975(11)	0.0273(3)
H9	0.4113	0.7660	0.2073	0.033*
C10	0.19875(14)	0.76926(13)	0.29446(11)	0.0255(3)
C11	0.08425(13)	0.76643(13)	0.34900(10)	0.0250(3)
C12	−0.01085(14)	0.82655(13)	0.34499(11)	0.0268(3)
H12	−0.0101	0.8719	0.3021	0.032*
C13	−0.20005(16)	0.95518(15)	0.34822(13)	0.0352(3)
H13A	−0.1112	1.0246	0.3756	0.053*

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H13B	−0.2747	0.9875	0.3553	0.053*
H13C	−0.2100	0.9075	0.2754	0.053*
C14	−0.29188(16)	0.87889(16)	0.48167(13)	0.0372(3)
H14A	−0.3480	0.7943	0.4724	0.056*
H14B	−0.3522	0.9235	0.4713	0.056*
H14C	−0.2321	0.9224	0.5514	0.056*
C15	−0.10837(14)	0.81726(13)	0.40733(11)	0.0276(3)
C16	−0.10262(14)	0.74783(13)	0.47194(11)	0.0280(3)
H16	−0.1662	0.7413	0.5140	0.034*
C17	−0.00563(14)	0.68949(13)	0.47455(11)	0.0264(3)
H17	−0.0035	0.6454	0.5183	0.032*
C18	0.08834(13)	0.69764(12)	0.41102(10)	0.0238(3)
C19	0.20516(13)	0.64510(13)	0.40007(10)	0.0235(3)
C20	0.30630(13)	0.69771(13)	0.50769(10)	0.0239(3)
C21	0.40090(13)	0.82279(13)	0.55140(11)	0.0263(3)
H21	0.4126	0.8704	0.5099	0.032*
C22	0.47788(14)	0.87824(13)	0.65459(11)	0.0277(3)
H22	0.5410	0.9616	0.6808	0.033*
C23	0.58259(17)	1.00355(15)	0.87566(12)	0.0363(3)
H23A	0.6495	1.0420	0.8425	0.054*
H23B	0.6255	1.0306	0.9487	0.054*
H23C	0.5029	1.0287	0.8688	0.054*
C24	0.48921(19)	0.80011(16)	0.89284(12)	0.0423(4)
H24A	0.3948	0.7945	0.8956	0.063*
H24B	0.5497	0.8457	0.9623	0.063*
H24C	0.4916	0.7163	0.8651	0.063*
C25	0.46203(13)	0.81024(13)	0.72042(11)	0.0267(3)
C26	0.36864(14)	0.68379(14)	0.67624(11)	0.0282(3)
H26	0.3572	0.6354	0.7172	0.034*
C27	0.29310(14)	0.62986(13)	0.57232(11)	0.0270(3)
H27	0.2318	0.5458	0.5451	0.032*
C28	0.15119(13)	0.50068(13)	0.34596(10)	0.0237(3)
C29	0.24251(14)	0.43469(13)	0.34320(10)	0.0259(3)
H29	0.3371	0.4795	0.3769	0.031*
C30	0.19612(14)	0.30417(14)	0.29157(11)	0.0280(3)
H30	0.2600	0.2633	0.2904	0.034*
C31	0.05320(15)	0.23255(13)	0.24071(11)	0.0286(3)
C32	0.10360(18)	0.03867(15)	0.17626(14)	0.0418(4)
H32A	0.1622	0.0702	0.1350	0.063*
H32B	0.0539	−0.0510	0.1397	0.063*
H32C	0.1609	0.0549	0.2427	0.063*
C33	−0.13631(19)	0.03578(16)	0.12639(15)	0.0469(4)
H33A	−0.2027	0.0528	0.1667	0.070*
H33B	−0.1556	−0.0538	0.0981	0.070*
H33C	−0.1441	0.0646	0.0700	0.070*
C34	−0.03738(14)	0.29960(14)	0.24259(11)	0.0287(3)
H34	−0.1320	0.2555	0.2087	0.034*
C35	0.01083(14)	0.43017(13)	0.29380(10)	0.0261(3)
H35	−0.0522	0.4717	0.2933	0.031*

of dichloromethane (30 mL) and absolute ethanol (30 mL) to form a colorless solution. Hydrazine hydrate (7.0 mL, 85%, excess) was added dropwise under stirring at room temperature. The mixture was heated to reflux for 5 h

under stirring. After that, the solvent was concentrated to 15 mL under reduced pressure. And then, the solution was cooled at room temperature to yield a white precipitate. The resulting precipitate was filtered and washed several times with ethanol. The reaction afforded crystal violet lactone hydrazide white powder. Yield: *ca* 80%. **Synthesis of the title compound:** 2-quinoxalinecarbaldehyde crystal violet hydrazone. Crystal violet lactone hydrazide powder (1 mmol, 0.429 g) was dissolved in 10 mL dichloromethane. Ethanol solution 15 mL containing 2-quinoxalinecarbaldehyde (1 mmol, 0.158 g) was added to the above crystal violet lactone hydrazide solution. And then, the stirred mixture was refluxed for 8 h. The reaction solution changed from clear yellow clarified solution to grass green clarified solution. The solvent was concentrated under reduced pressure to yield a yellow precipitate. The precipitate was filtered and washed several times with ethanol to obtain yellow powder. Yield: *ca* 60%. The yellow powder (0.02 mmol, 11.38 mg) was dissolved in methanol/dichloromethane (1:1) (10 mL) to give a yellow solution to evaporate slowly at room temperature. After several days, clear yellow cube crystals appeared. Yield: *ca* 60%.

Experimental details

The H atoms were added geometrically using riding models and refined isotropically. Their *U*_{iso} values were set to 1.2*U*_{eq} of the parent C and N atoms, and 1.5*U*_{eq} of the parent O atoms.

Comment

Photochromic materials have been investigated for their diverse applications such as optical data storage [5], photo-controllable switches [6], molecular machines [7]. As a classic triarylmethane (TMP) organic dye, CVL (crystal violet lactones) is widely used as chromogenic reagent due to its intrinsic halochromic property [8]. CVL was also reported as an electrochromic material [9] and a photochromic material [10]. On the other hand, CVL derivatives is a good chelating ligand for metal ions. The addition of certain metal ions will lead to a spirolactam ring-open-reaction along with fluorescence or absorption spectra changes. Thus the title compound of 2-quinoxalinecarbaldehyde crystal violet hydrazone may be a fluorescent probe and chemosensors of certain metal ion, moreover form magnetic materials with certain transition metals or rare earth ions.

The asymmetric unit of the title compound consists of a ring-closed 2-quinoxalinecarbaldehyde crystal violet hydrazone molecule. The bond length of C9–N2 is 1.2869(18) Å, which shows the existence of schiff base C=N. The amide C=O bond distance is 1.2219(17) Å, indicative of the keto form of the amide. The hydrogen bond between adjacent

molecules is very weak. There are also no obvious π - π contacts.

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