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Crystal structure of [1-(4-carboxyphenyl)-4-oxo-1,4-dihydropyridazine-3-carboxylic acid]-(methylsulfinyl)methane, $C_{15}H_{16}N_2O_6S$

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Abstract

$C_{15}H_{16}N_2O_6S$, monoclinic, $P2_1/n$ (no. 14), a = 11.1325(3) Å, b = 6.2088(2) Å, c = 23.8080(6) Å, β = 99.266(2)°, V = 1,624.12(8) Å^3, Z = 4, $R_{gt}(F)$ = 0.0431, $wR_{ref}(F^2)$ = 0.1271, T = 293(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

Ethyl 1-(4-(ethoxycarbonyl)phenyl)-5-methyl-4-oxo-1,4-dihydropyridazine-3-carboxylate (0.5 mmol) and sodium hydroxide (0.5 mmol) were dissolved in 100 mL deionized water, then added to a 250 mL flask and heated at 333 K for 2 h and cooled to room temperature acidified by addition of 15 % HCl solution until pH = 2, the residue was thoroughly washed, carefully filtered, and subsequently dried, ultimately yielding the desired compound in the form of light yellow crystals.

2 Experimental details

Using Olex2,[2] the structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL[4] refinement package.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.11 × 0.10 × 0.05 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	2.09 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	67.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8,883, 2,874, 0.040
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,396
$N(param)_{refined}$:	222
Programs:	Bruker[1], Olex2[2], SHELX[3,4]

3 Comment

Pyridazine derivatives constitute a particularly significant class of biologically active heterocycles. These compounds have garnered considerable attention as ligands due to their remarkable structural and synthetic versatility, precise tunability, and selectivity towards transition metal atoms.[5] Their diverse therapeutic potential, encompassing anticancer,[6,7] antidiabetic,[8,9] and antibiotic activities,[10] has been extensively documented. Leveraging this understanding, we have achieved the successful synthesis of a novel pyridazine carboxyl derivative, achieved by incorporating a benzene ring adorned with carboxyl groups onto the pyridazine ring. Simultaneously, numerous syntheses of transition metal complexes utilizing this derivative have been documented in various studies.[11–16] However, it is noteworthy that the crystal structure of this pyridazine carboxylic derivative remains unreported thus far.

As shown in Figure 1, this crystal contains two molecules: 1-(4-carboxyphenyl)-4-oxo-1,4-dihydropyridazine-3-carboxylic acid and dimethyl sulfoxide. The title target molecule consists of a pyridazine ring and a benzene ring, there are also two carboxyl groups, a carbonyl group and a methyl group on these rings (C4, C6, C10 and C12), the dihedral angles between pyridazine ring and benzene ring is almost complete coplanar, the lengths of nitrogen-nitrogen bond (1.3396) has an intermediate value between typical N–N single (1.45) and N=N double (1.25) bonds, as well as carbon-nitrogen bond lengths, which indicates the delocalization of p-electrons in the molecule. Furthermore, the title molecule and solvent molecule form via stacking

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.76048 (5)	1.60720 (10)	0.63532 (3)	0.0647 (2)
O1	0.75417 (15)	−0.1886 (3)	0.28350 (7)	0.0660 (4)
O4	0.84380 (13)	1.1922 (3)	0.53246 (8)	0.0707 (5)
H4	0.831903	1.298642	0.551206	0.106*
O6	0.85163 (13)	1.5173 (3)	0.60028 (7)	0.0673 (5)
N1	0.76860 (14)	0.3610 (2)	0.37305 (6)	0.0416 (4)
N2	0.65978 (13)	0.2873 (3)	0.34829 (6)	0.0447 (4)
O3	0.44095 (15)	0.1443 (3)	0.29501 (8)	0.0829 (6)
O2	0.52639 (16)	−0.1382 (4)	0.26279 (9)	0.0908 (7)
H2	0.595412	−0.186745	0.264580	0.136*
O5	0.64328 (15)	1.1869 (4)	0.51509 (11)	0.0980 (7)
C1	0.76390 (16)	0.5524 (3)	0.40702 (7)	0.0411 (4)
C4	0.87441 (17)	0.2592 (3)	0.36670 (8)	0.0458 (4)
H4A	0.947929	0.320162	0.383288	0.055*
C6	0.87647 (18)	0.0735 (3)	0.33728 (8)	0.0460 (4)
C8	0.75008 (17)	0.9179 (3)	0.47296 (8)	0.0441 (4)
C10	0.76277 (18)	−0.0156 (3)	0.31102 (8)	0.0481 (5)
C12	0.65721 (18)	0.1105 (3)	0.31846 (8)	0.0473 (5)
C14	0.64754 (18)	0.8386 (3)	0.43882 (9)	0.0524 (5)
H14	0.573187	0.907504	0.438276	0.063*
C15	0.86704 (18)	0.6284 (3)	0.44145 (9)	0.0517 (5)
H15	0.940980	0.557272	0.442838	0.062*
C2	0.65401 (17)	0.6585 (3)	0.40552 (9)	0.0525 (5)
H2A	0.584682	0.608398	0.382102	0.063*
C9	0.73889 (18)	1.1112 (3)	0.50864 (9)	0.0512 (5)
C5	0.85922 (18)	0.8110 (4)	0.47375 (9)	0.0520 (5)
H5	0.928870	0.863103	0.496538	0.062*
C11	0.5306 (2)	0.0432 (4)	0.29110 (9)	0.0623 (6)
C3	0.9923 (2)	−0.0384 (4)	0.33024 (11)	0.0617 (6)
H3A	1.059852	0.033875	0.352600	0.093*
H3B	1.001781	−0.035621	0.290877	0.093*
H3C	0.989428	−0.185017	0.342723	0.093*
C13	0.6229 (2)	1.6484 (5)	0.58707 (11)	0.0706 (7)
H13A	0.632999	1.766644	0.562264	0.106*
H13B	0.558284	1.680443	0.608034	0.106*
H13C	0.603157	1.520439	0.564875	0.106*
C7	0.7122 (3)	1.3861 (6)	0.67315 (14)	0.0946 (10)
H7A	0.684606	1.272048	0.646999	0.142*
H7B	0.646784	1.430709	0.692308	0.142*
H7C	0.778947	1.335768	0.700622	0.142*

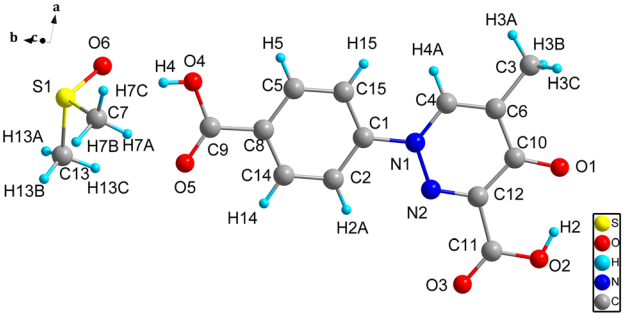

Figure 1: The asymmetric structural unit of title compound.

interactions and various hydrogen bonds a 3D supramolecular structure.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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