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Sodium 2-oxo-3-semicarbazono-2,3-dihydro-1*H*-indole-5-sulfonate dihydrate

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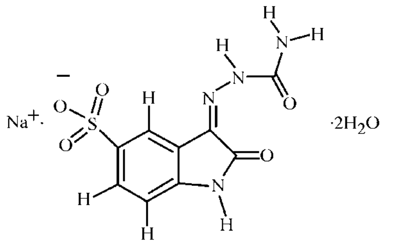
The title compound, $Na^+ \cdot C_9H_7N_4O_5S^- \cdot 2H_2O$, presents a *Z* configuration around the imine C=N bond and an *E* configuration around the $C(O)NH_2$ group, stabilized by two intramolecular hydrogen bonds. The packing is governed by ionic interactions between the Na^+ cation and the surrounding O atoms. The ionic unit, Na^+ and 2-oxo-3-semicarbazono-2,3-dihydro-1*H*-indole-5-sulfonate, forms layers extending in the *bc* plane. The layers are connected by hydrogen bonds involving the water molecules.

Comment

The biological properties of isatin derivatives are well known (Pandeya *et al.*, 1999, and reference therein). We have been working on these systems (Rodríguez-Argüelles *et al.*, 1999, 2004; Casas *et al.*, 2000; Belicchi Ferrari *et al.*, 2002; Bacchi *et al.*, 2005) and the main problem that these compounds present for their use in biological experiments is their poor solubility in water. To overcome this problem, we have turned our attention to sulfonated isatin. Against this background, we report here the synthesis and solid-state characterization of the sodium salt of 3-semicarbazonoisatin-5-sulfonate, (I).



(I)

In compound (I) (Fig. 1), the 3-semicarbazonoisatin moiety presents the same configuration as the corresponding non-sulfonated ligand reported previously (Pelosi *et al.*, 2005) and is stabilized by intramolecular hydrogen bonds, with N2—H···O2 = 2.903 (4) Å and N1—H···N3 = 2.586 (5) Å. Bond distances and angles of the organic moiety are comparable with those found in 3-semicarbazonoisatin. The six- and five-membered rings of the isatin moiety are less planar than those found in 3-semicarbazonoisatin (Pelosi *et al.*, 2005).

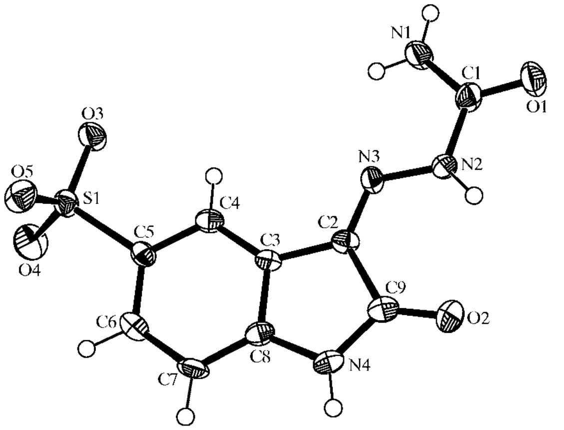


Figure 1
A view of the anionic moiety of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

The dihedral angle between the mean planes of the two rings is 3.9 (1)°, and that between the mean planes of the entire isatin and semicarbazide groups is 6.1 (1)°, similar to the value found in 3-semicarbazonoisatin (Pelosi *et al.*, 2005). The O—S distance range [1.450 (2)–1.454 (2) Å] agrees with the values reported in the literature for similar anions.

The Na^+ ion is surrounded in a slightly distorted octahedral fashion by six O atoms: O5 of the sulfonate group, O3 of a sulfonate group at symmetry position $(x, -y + \frac{1}{2}, z + \frac{1}{2})$, O1 of the semicarbazide group at symmetry position $(-x + 1, -y, -z)$, O2 of the isatin moiety at symmetry position $(-x + 1, y + \frac{1}{2}, -z + \frac{1}{2})$ and two water atoms, O6 and O7. The Na—O distance range [2.365 (3)–2.484 (3) Å] is slightly lower than the mean value of 2.593 (2) Å reported in the Cambridge Structural Database (Version 5.27 of November 2005, 6336 hits; Allen, 2002).

The packing is formed by sheets stacked along the *a* direction. Within the layers, the molecules are connected by an extended system of hydrogen bonds involving atoms N1 and N4, two O atoms of the sulfonate group (O3 and O5) and the O atom of the semicarbazide group (O1). These layers are connected by hydrogen bonds between atom O4 of a sulfonate moiety and water atom O7 at symmetry position $(x - 1, -y + \frac{1}{2}, z - \frac{1}{2})$.

Experimental

The title compound (m.p. >573 K, 37% yield) was obtained as a yellow solid by reacting sodium 5-sulfonatoisatin with neutral semicarbazide (1:1 molar ratio) following a standard procedure (Battaglia *et al.*, 1994), but using ethanol instead of methanol. Analysis found: C 31.4, H 3.2, N 16.5, S 8.9%; calculated for $C_9H_7N_4NaO_5S \cdot 2H_2O$:

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C 31.6, H 3.2, N 16.4, S 9.4%. Mass FAB (*m*-nitrobenzyl alcohol, *m*/*z*): 285 (21%, $M \pm 2H_2O - Na$), 199 (100%, $C_7H_5NO_4S$). The solid was dissolved in dimethyl sulfoxide and, after several days at room temperature, the solution afforded crystals of (I) suitable for X-ray diffraction studies.

Crystal data

$Na^+ \cdot C_9H_7N_4O_5S^- \cdot 2H_2O$
$M_r = 342.27$
Monoclinic, $P2_1/c$
$a = 7.379$ (3) Å
$b = 17.139$ (4) Å
$c = 10.788$ (3) Å
$\beta = 105.60$ (2)°
$V = 1314.1$ (7) Å^3
$Z = 4$
$D_x = 1.730$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.32$ mm^{-1}
$T = 291$ (2) K
Prism, yellow
0.5 × 0.4 × 0.3 mm

Data collection

Philips PW1100 diffractometer
$\theta/2\theta$ scans
4008 measured reflections
3838 independent reflections
1535 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.112$
$S = 0.90$
3838 reflections
227 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.227 (4)	O3—Na1$^{\rm iii}$	2.384 (3)
C1—N1	1.337 (4)	O4—S1	1.453 (2)
C1—N2	1.384 (4)	O5—S1	1.454 (2)
C2—N3	1.287 (4)	O5—Na1	2.353 (3)
C5—S1	1.782 (3)	O6—Na1	2.462 (3)
N2—N3	1.344 (4)	O7—Na1	2.441 (3)
O1—Na1$^{\rm i}$	2.365 (3)	Na1—O1$^{\rm i}$	2.365 (3)
O2—Na1$^{\rm ii}$	2.455 (3)	Na1—O3$^{\rm iv}$	2.384 (3)
O3—S1	1.450 (2)	Na1—O2$^{\rm v}$	2.455 (3)
O1—C1—N1	124.6 (4)	C2—N3—N2	120.0 (3)
O1—C1—N2	120.0 (3)	O4—S1—O3	112.22 (15)
N1—C1—N2	115.4 (3)	O4—S1—O5	112.20 (16)
N3—C2—C3	124.1 (3)	O3—S1—O5	112.57 (15)
N3—C2—C9	129.6 (3)	O4—S1—C5	107.79 (16)
C4—C5—S1	120.2 (3)	O3—S1—C5	106.38 (15)
C6—C5—S1	118.1 (3)	O5—S1—C5	105.12 (15)
N3—N2—C1	117.6 (3)		

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (v) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

All H atoms were located in a difference map and refined freely, except for those of the water molecules [C—H = 0.89 (3)–0.99 (3) Å].

Data collection: local program (Belletti *et al.*, 1988); cell refinement: local program; data reduction: local program; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *PLATON*(Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HJ3004). Services for accessing these data are described at the back of the journal.

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