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Crystal structure of (*E*)-3-(2-(2-hydroxy-4-methoxystyryl)-3,3-dimethyl-3*H*-indol-1-ium-1-yl)propane-1-sulfonate monohydrate, C₂₂H₂₅NO₅S·H₂O

Table 1: Data collection and handling.

Crystal:	Orange block
Size:	0.25 × 0.24 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.19 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	23010, 3825, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3231
$N(\text{param})_{\text{refined}}$:	278
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7225(3)	−0.1537(2)	0.7324(2)	0.0725(7)
H1A	0.6275	−0.1901	0.7409	0.109*
H1B	0.6936	−0.0848	0.7796	0.109*
H1C	0.8031	−0.2195	0.7530	0.109*
C2	0.6965(3)	−0.00123(18)	0.57773(18)	0.0480(5)
C3	0.7698(3)	0.0479(2)	0.46841(18)	0.0525(5)
H3	0.8736	0.0098	0.4286	0.063*
C4	0.6885(2)	0.15175(18)	0.42058(16)	0.0461(5)
H4	0.7384	0.1842	0.3479	0.055*
C5	0.5307(2)	0.21173(17)	0.47805(15)	0.0388(4)
C6	0.4589(2)	0.15868(17)	0.58742(15)	0.0406(4)
C7	0.5430(2)	0.05369(18)	0.63697(16)	0.0453(5)
H7	0.4955	0.0208	0.7100	0.054*
C8	0.4470(2)	0.32551(17)	0.43207(15)	0.0401(4)
H8	0.3382	0.3523	0.4706	0.048*
C9	0.5142(2)	0.39758(18)	0.33651(15)	0.0413(4)
H9	0.6217	0.3677	0.2972	0.050*
C10	0.4380(2)	0.51299(16)	0.29053(14)	0.0353(4)
C11	0.2624(2)	0.58216(17)	0.32973(15)	0.0365(4)
C12	0.1468(2)	0.49850(19)	0.31730(19)	0.0512(5)
H12A	0.1833	0.4737	0.2426	0.077*
H12B	0.0386	0.5473	0.3323	0.077*
H12C	0.1464	0.4223	0.3692	0.077*
C13	0.2115(3)	0.6219(2)	0.44861(18)	0.0594(6)
H13A	0.2073	0.5458	0.5008	0.089*
H13B	0.1061	0.6753	0.4647	0.089*
H13C	0.2894	0.6694	0.4548	0.089*
C14	0.2672(2)	0.69969(16)	0.24546(15)	0.0363(4)
C15	0.1490(2)	0.80458(18)	0.23483(19)	0.0482(5)
H15	0.0454	0.8122	0.2863	0.058*

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Abstract

C₂₂H₂₅NO₅S·H₂O, triclinic, *P* $\bar{1}$ (no. 2), $a = 8.8121(4)$ Å, $b = 10.6053(4)$ Å, $c = 12.7365(5)$ Å, $\alpha = 80.943(2)^\circ$, $\beta = 70.890(2)^\circ$, $\gamma = 78.105(2)^\circ$, $V = 1095.33(8)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0378$, $wR_{\text{ref}}(F^2) = 0.0949$, $T = 302(2)$ K.

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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

A solution of 2,3,3-trimethyl-1-(3-sulfonatepropyl)-3*H*-indolium (100 mg, 0.36 mmol) in ethanol (15 mL) was

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C16	0.1881(3)	0.89807(18)	0.1460(2)	0.0565(6)
H16	0.1094	0.9690	0.1379	0.068*
C17	0.3409(3)	0.88812(18)	0.0697(2)	0.0543(5)
H17	0.3633	0.9522	0.0105	0.065*
C18	0.4625(2)	0.78443(17)	0.07894(17)	0.0443(5)
H18	0.5665	0.7772	0.0279	0.053*
C19	0.4201(2)	0.69263(15)	0.16833(15)	0.0350(4)
C20	0.6880(2)	0.53719(16)	0.13147(15)	0.0368(4)
H20A	0.7537	0.5012	0.1805	0.044*
H20B	0.7321	0.6115	0.0861	0.044*
C21	0.6962(2)	0.43617(18)	0.05646(16)	0.0424(4)
H21A	0.6441	0.4770	0.0006	0.051*
H21B	0.6348	0.3698	0.1011	0.051*
C22	0.8681(2)	0.37179(17)	−0.00242(15)	0.0382(4)
H22A	0.9334	0.4385	−0.0399	0.046*
H22B	0.8640	0.3223	−0.0590	0.046*
N1	0.51936(16)	0.57853(13)	0.19847(12)	0.0348(3)
O1	0.9789(2)	0.34651(14)	0.16596(13)	0.0665(5)
O2	0.86096(18)	0.17261(14)	0.14282(13)	0.0634(4)
O3	1.12221(16)	0.21072(13)	0.01802(12)	0.0543(4)
O4	0.30799(17)	0.21397(14)	0.64349(12)	0.0582(4)
H4A	0.2769	0.1724	0.7043	0.087*
O5	0.7866(2)	−0.10422(14)	0.61911(14)	0.0683(5)
O6	0.2126(2)	0.07566(15)	0.83634(12)	0.0618(4)
H6A	0.1768	0.0043	0.8508	0.093*
H6B	0.1740	0.1102	0.8975	0.093*
S1	0.96487(5)	0.26731(4)	0.08889(4)	0.03739(14)

added to a stirred ethanol solution (15 mL) of 2-hydroxy-4-methoxybenzaldehyde (60 mg, 0.39 mmol) in a three-necked flask. The reaction mixture was refluxed for 24 h, then the solution was cooled to room temperature. The orange precipitated residue was removed by filtration and then washed with ethanol and ether 3 times. Then orange solids (0.1 mmol) were dissolved in 20 mL ethanol. Single crystals suitable for X-ray diffraction were obtained from an ethanol by slow evaporation at room temperature.

Experimental details

Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the $d(C-H) = 0.97-0.99$ Å, $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ and $U_{iso}(H) = 1.5$ times $U_{eq}(O)$.

Comment

Over the past few years, so-called photoacids have been widely studied for their ability of controlling proton concentration and proton transfer processes using visible light, creating a large pH change or good reversibility. They have

been demonstrated to be used in photolithography, materials science, biological applications [5]. As valuable photoacids, metastable-state photoacids (mPAH) reversibly control proton transfer, which is involved in the mechanisms of many enzymes. Meanwhile, abnormal cellular pH is related to many diseases including cancer, cardiovascular diseases, and Alzheimer's disease, mPAHs have a lot of potential in the biomedical area [6].

In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one formula unit and one H₂O. The indole ring and the aryl moiety is connected by an ethendiyl fragment. This double bond presented "E" configuration, and the dihedral angle between the mean planes of the indole and the aryl moiety is 166.0°. The bond lengths and bond angles are in the normal ranges [7, 8]. Through hydrogen bonds: O4—H4A···O6, O6—H6A···O2, O6—H6B···O3, the target compound and water molecule extend into a 1D structure, which extends into a 3D structure through the hydrogen bonds: C1—H1C···O1, C7—H7···O6, C18—H18···O3, C20—H20A···O1, C20—H20B···O3.

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