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Synthesis and crystal structure of trans-tetraaquabis((1-((7-hydroxy-3-(4-methoxy-3-sulfonatophenyl)-4-oxo-4H-chromen-8-yl)methyl)piperidin-1-ium-4-carbonyl)oxy-κO)zinc(II) hexahydrate, C₄₆H₆₄N₂O₂₈S₂Zn

Abstract
C₄₆H₆₄N₂O₂₈S₂Zn, monoclinic, P₂₁/c (no. 14), a = 18.155(2) Å, b = 9.0872(11) Å, c = 16.373(2) Å, β = 108.095(13)°, V = 2567.6(6) Å³, Z = 2, Rgt(F) = 0.0712, wRref(F²) = 0.1295, T = 296.15 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.

1 Source of materials

All reagents and chemicals were purchased from commercial sources and used without further purification. The educt sodium 5-(8-((4-carboxypiperidin-1-yl)methyl)-7-hydroxy-4-oxo-4H-chromen-3-yl)-2-methoxybenzenesulfonate was synthesized via a Mannich reaction. Formaldehyde solution (5 mL, 37 %), sodium 5-(7-hydroxy-4-oxo-4H-chromen-3-yl)-2-methoxybenzenesulfonate (1.85 g, 0.005 mol), water (5 mL)
and piperidine-4-carboxylic acid (0.4844 g, 0.00375 mol) were added to ethanol (50 mL, 99%) and stirred for 0.5 h at 328 K. Then, hot water (20 mL) was added until a transparent solution was obtained. After 12 h reaction time, the mixture was filtered, and the residue was collected. Then the residue was dried at 383 K. Sodium 5-(6-((4-carboxypiperidin-1-yl)methyl)-7-hydroxy-4-oxo-4H-chromen-3-yl)-2-methoxybenzenesulfonate (0.6877 g) was obtained.

IR spectra (potassium bromide pellet) were recorded on a Nicolet 6700. IR (ν/cm⁻¹): 3387, 3079, 2992, 2747, 1733, 1627, 1608, 1494, 1444, 1390, 1368, 1294, 1266, 1215, 1175, 1127, 1094, 1070, 1035, 1015, 989, 957, 923, 903, 843, 829, 800, 720, 689, 620, 503, 581, 548, 489, 469, 447. Zinc nitrate hexahydrate (0.0297 g, 0.1 mmol) and sodium salt described before (0.0256 g, 0.05 mmol) were added to water (10 mL) and sonicated for 10 min. The mixture was heated for 18 h at 363 K. Colorless block crystals of the title complex were obtained. IR spectra (potassium bromide pellet) were recorded on a Nicolet 6700. IR (ν/cm⁻¹): 3431, 3023, 2763, 1628, 1604, 1573, 1494, 1450, 1417, 1373, 1361, 1326, 1291, 1266, 1215, 1171, 1096, 1056, 1025, 1004, 959, 926, 897, 824, 797, 780, 722, 637, 622, 550, 516.

### 2 Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{iso}$(H) set to 1.2 $U_{eq}$(C). The oxygen-bound and nitrogen-bound H atoms were located on a different Fourier map.
In recent years, a series of formononetin derivatives have been synthesized through different procedures to study their pharmacological activities. Some of formononetin derivatives possess various pharmacological activities such as antihypertensive activity and anticancer activity [5–7]. Our focus was on the design and synthesis of a novel series of formononetin derivatives, with the aim of investigating their potential as effective anticancer agents.

X-ray crystallographic analysis of the Zn(II) complex reveals a mononuclear structure. There is one half of a Zn(II) ion, one organic ligand, two coordinated water molecules and three uncoordinated water molecules in the asymmetric unit of the title structure (cf. the figure, the asymmetric unit is labeled). Zn1, located at an inversion center, is six-coordinated with a distorted octahedral geometry by four coordinated water and two O atoms from two monodentate coordinating ligands. The Zn–O bond distances are in the range of 2.025(2) and 2.196(2) Å. The bond distances and bond angles are in normal ranges [8–10]. The carboxylate groups adopt trans conformations. The pyridine groups adopt chair conformations. The nitrogen atom N1 is protonated. There exist various O–H⋯O and N–H⋯O hydrogen bonds forming a three-dimensional framework. It is obvious that the hydrogen bonds play important roles in the self-assembly and enhance stability of the resultant structure.

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