

КРАТКИЕ СООБЩЕНИЯ

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4-HYDROXY-2H-1,2-BENZOTHAZINE-3-CARBOHYDRAZIDE 1,1-DIOXIDE-OXALOHYDRAZIDE (1:1):
X-RAY STRUCTURE AND DFT CALCULATIONSM.N. Arshad¹, O. Şahin², M. Zia-ur-Rehman³, I.U. Khan⁴,
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The title compound, 4-hydroxy-2H-1,2-benzothiazine-3-carbohydrazide 1,1-dioxide-oxalohydrazide (1:1), is determined using X-ray diffraction techniques and the molecular structure is also optimized at the B3LYP/6-31G(*d,p*) level using density functional theory (DFT). The asymmetric unit consists of four independent molecules. The oxalohydrazide molecules have the centre of symmetry at the mid-point of the central C—C bond. Each thiazine ring adopts a *half-chair* conformation. Intermolecular C—H...O, N—H...O and N—H...N hydrogen bonds produce $R_2^2(10)$, $R_2^2(13)$, $R_3^3(12)$ and $R_3^3(15)$ rings, which lead to one-dimensional polymeric chains. An extensive three-dimensional supramolecular network of N—H...N, N—H...O, C—H...O and O—H...O hydrogen bonds is responsible for crystal structure stabilization.

Keywords: X-ray diffraction analysis, benzothiazine, oxalohydrazide, DFT, Mulliken atomic charge, molecular electrostatic potential.

Synthesis and structural characterization of carbohydrazides and their derivatives is much focused in the recent literature due to their applications in biochemistry as well as in materials synthesis *via* complexation [1, 2]. They are well known for their anti-viral [3], anti-tuberculoses [4], anti-fungal [5], bacteriostatic [6], insecticidal [7] and anti-parasitic [8] activities. As part of a research program regarding the synthesis of new derivatives of benzothiazine 1,1-dioxides [9, 10], the synthesis and crystal structure of 4-hydroxy-2H-1,2-benzothiazine-3-carbohydrazide 1,1-dioxide-oxalohydrazide (1:1) is reported here.

The title compound was synthesized from commercially available saccharin, which was converted to methyl 4-hydroxy-2H-1,2-benzothiazine-3-carboxylate 1,1-dioxide by a reported procedure [11] and was subsequently subjected to hydrazinolysis in methanol along with equimolar dimethyl oxalate. It was interesting to note that dimethyl oxalate did not react with benzothiazine hydrazide under these reaction conditions, but reacted with hydrazine and co-crystallized with the product oxalohydrazide.

Experimental. Synthesis. A mixture of methyl 4-hydroxy-2H-1,2-benzothiazine-3-carboxylate 1,1-dioxide (2.00 g, 7.84 mmol), hydrazine hydrate (1.57 g, 31.36 mmol) and dimethyl oxalate (1.02 g, 39.2 mmol) was stirred in ethanol (30 ml) for half an hour. On completion of reaction, as indi-