European Journal of Chemistry

View Journal Online
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Crystal structure and Hirshfeld surface analysis of *N*-(2-(*N*-methylsulfamoyl)phenyl)formamide: Degradation product of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide

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RESEARCH ARTICLE

10.5155/eurjchem.10.3.189-194.1903

Received: 03 June 2019
Received in revised form: 04 July 2019
Accepted: 05 July 2019
Published online: 30 September 2019
Printed: 30 September 2019

KEYWORDS

Hydrolysis
Formamide
Crystal structure
Hirshfeld surface
Benzothiadiazine
Hydrogen bonding

ABSTRACT

The hydrolysis of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide (2) during crystallization under humidity (85 %) conditions, lead to *N*-(2-(*N*-methylsulfamoyl)phenyl)formamide as second step hydrolysis product, identified in the proposed degradation mechanism. Crystal of *N*-(2-(*N*-methylsulfamoyl)phenyl)formamide $C_8H_{10}N_2O_3S$ (4), was obtained and characterized. The molecular structure determination was carried out with MoKα X-ray and data measured at 100 K. The compound 4 crystallizes in triclinic $P\bar{1}$ space group with unit cell parameters a = 4.8465(4) Å, b = 8.1942(9) Å, c = 11.8686(13) Å, α = 77.080(4)°, β = 82.069(4)°, γ = 80.648(4)°, V = 450.76 (8) Å^3 and Z = 2. The crystal structure is stabilized by intramolecular N-H···O and intermolecular C-H···O and N-H···O hydrogen bonds that extended as infinite 1D chain along [100]. Stabilization is also ensured by oxygen-π stacking interaction between the aromatic ring and oxygen of the sulfonamide group. The analysis of intermolecular interactions through the mapping of d_{norm} and shape-index revel that the most significant contributions to the Hirshfeld surface 40.6 and 33.9% are from H···H and O···H contacts, respectively.

Cite this: *Eur. J. Chem.* **2019**, *10(3)*, 189-194 **Journal website**: www.eurjchem.com

1. Introduction

Thiazide derivatives in general and benzothiadiazine 1,1-dioxides particularly are described as possessing numerous pharmacological activities [1-8]. Hydrochlorothiazide (HCTZ or 6-chloro-3,4-dihydro-2*H*-1,2,4-benzothiadiazine-7-sulfonamide 1,1-dioxide) is a well-known benzothiadiazine used as diuretic drug for the treatment of hypertension [9]. Despite the importance of these heterocycles as promising drugs and the report of various molecular structures obtained by X-ray diffraction analysis [10-12], studies have shown that benzothiadiazine 1,1-dioxides hydrolyze slowly in aqueous media. In the case of HCTZ, studies reveal that hydrolysis led to degradation product identified as 4-amino-6-chlorobenzene-1,3-disulfonamide and formaldehyde [13-16]. Although various by-products are later identified during thiazide hydrolysis [17,18], isolation and full characterization of all degradation products were not reported at all. Qun Xu have proposed a plausible hydrolysis pathway of benzothiadiazine under thermal and humidity conditions showing various intermediates [19]. In general, 2-aminobenzenesulfonamide derivatives are recognized as a main isolated and characterized product of benzothiadiazine 1,1-dioxides decomposition.

Our group is continuously interested in the synthesis, characterization and biological evaluation of benzoisothiazoles [20] and benzothiadiazine 1,1-dioxides [21,22]. In view to develop a new family of these interesting compounds, 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide was prepared as precursor. The crystallization of this latter under humidity conditions leads to mixture of by-products and monocrystal that was analyzed. In this paper, we are reporting the structural characterization by single-crystal X-ray diffraction analysis of *N*-(2-(*N*-methylsulfamoyl)phenyl)formamide (**4**) as hydrolysis uncommon by-product of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide. Furthermore, the intermolecular interactions were examined using Hirshfeld surface analysis.

http://dx.doi.org/10.5155/eurjchem.10.3.189-194.1903

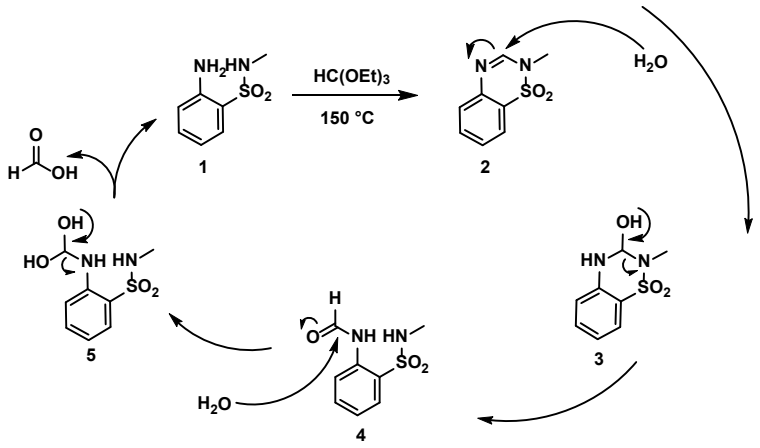

Figure 1. Formation and proposal hydrolysis pathway of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide **2** to yield *N*-(2-(*N*-methylsulfamoyl)phenyl) formamide (**4**).

2. Experimental

2.1. Crystal growth

Crystal of compound **4** was obtained by dissolving appropriate amounts (30 mg) of compound **2** in a mixture of acetone and dichloromethane. Solvent was added to ensure completely solubilization of the starting compound and the solution was transferred to 1 cm diameter tube without cap and keep in cold room at 6 °C. After solvent evaporation, monocrystals suitable for X-ray diffraction analysis were isolated and analysed.

2.2. X-ray crystallography

For the crystal structure determination, the data were collected by applying the omega and phi scans method on a Bruker D8 Venture PHOTON III-14 diffractometer using INCOATEC multilayer mirror monochromated with MoKα radiation (λ = 0.71073 Å) from a microfocus sealed tube source at 100 K with detector resolution of 7.3910 pixels/mm. Computing data and reduction were made with the APEX3 [23]. The structure was solved using SHELXT [24] and finally refined by full-matrix least-squares based on F^2 by SHELXL [25]. An empirical absorption correction was applied using the SADABS program. Software used to molecular graphics: ORTEP for Windows [26]. Software used to prepare material for publication: WinGX publication routines [26] and Mercury [27].

2.3. Refinement

All non-hydrogen atoms were refined anisotropically and the hydrogen atom positions were included in the model on the basis of Fourier difference electron density maps. All CH and aromatic CH hydrogen (C-H = 0.95 Å) atoms were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C). The methyl hydrogen (C-H = 0.98 Å) atoms were refined as a rigid group with torsional freedom [U_{iso}(H) = 1.5 U_{eq}(C)] and the hydrogens H1N and H2N atom as a free atom.

3. Results and discussion

3.1. Description of crystal structure of compound 4

The crystal of the title compound was obtained from slow degradation of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide (**2**) during crystallization. The synthesis of compound **2** was accomplished using the procedure; we have already reported in [4,28]. Indeed, the 2-amino-*N*-methylbenzene sulfonamide was heated in triethyl orthoformiate to reflux for 30 min. After cooling on an ice bath, the benzothiadiazine (**2**) was collected by filtration, washed with diethyl ether and dried. An appropriate amount (30 mg) of this product was dissolved in a mixture of acetone and dichloromethane. The amount of solvent used was sufficient to completely solubilize the compound and the solution was transferred to 1 cm diameter tube without cap and kept at 6 °C until the solvent evaporation was completed and formation of crystals. Monocrystal suitable for X-ray diffraction analysis were obtained and analyzed. Although studies report mainly 2-aminobenzenesulfonamides as the most stable and recurrent degradation product of benzothiadiazines, our present results have shown that the molecular structure correspond to *N*-(2-(*N*-methylsulfamoyl)phenyl)formamide (**4**) (Figure 1). Working without a cap allows solvent evaporation but also leads to solvent wetness and therefore favors the hydrolysis of the starting benzothiadiazine. The synthesis and the proposed pathway for the degradation of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide during crystallization under humidity conditions are shown in Figure 1. Based on the described benzothiadiazines hydrolysis studies, we propose here a mechanism for the formation of product **4** [19,29].

The compound **4** crystallizes in triclinic $P\bar{1}$ space group. All relevant crystal data are reported in Table 1. The molecular structure of compound **4** is shown in Figure 2. The S-O (~1.43 Å) and C-S (1.77 Å) bonds length are typical for that observed in sulfonamides and benzothiadiazines [30]. For the amide function, the carbonyl bond C1-O (1.2291(17) Å) influence electronically the N2-C1 bond length that becomes shorter than that observed for N1-C8 in the sulfonamide with a value of 1.3471(17) and 1.4163(17) Å, respectively (Table 2) [31,32]. The dihedral angles between the aromatic ring and the plane formed by the atoms C7-C2-N2 (1.45(7)°) and the one formed by C2-C7-S1 (5.50(6)°) revealed slightly deviation of aniline (N2-C2) and sulfone (C7-S1) bonds from the aromatic ring (C2---C7) plane. One intramolecular short hydrogen interactions N2-H2N···O1 (Figure 2) was observed resulting from the proximity of H2N and O1 atoms 2.21 (2) Å (Table 3) with N2-H2N and S1-O1 bonds orientation in the same direction.

The cell unit presents two molecules around the inversion center, so that the aromatic rings are confronted in parallel with a dihedral angle of 0.00(7)° (Figure 3).

Table 1. Crystal data of compound **4**.

Parameters	Compound 4
Chemical formula	$C_8H_{10}N_2O_3S$
Mw (g)	214.24
Crystal system	Triclinic
Space group	*P*-1
Temperature (K)	100
a (Å)	4.8465(4)
b (Å)	8.1942(9)
c (Å)	11.8686(13)
α (°)	77.080(4)
β (°)	82.069(4)
γ (°)	80.648(4)
V (Å³)	450.76(8)
Z	2
Radiation type	Mo-Kα radiation (λ = 0.71073 Å)
μ (mm⁻¹)	0.34
Crystal size (mm)	0.33 × 0.09 × 0.05
T_{min}, Tmax	0.911, 0.953
(sin θ/λ)max (Å⁻¹)	0.667
Measured/independent/observed [I>2σ(I)] reflections	21965/2245/2053
R_{int}	0.040
R[*F*2 > 2σ(*F*2)], *wR*(*F*2), *S*	0.029, 0.073, 1.06
$\Delta\rho_{max}/\Delta\rho_{min}$ (e.Å⁻³)	0.35, -0.45

Table 2. Selected geometric parameters (Å, °) for compound **4**.

Bonds	Length (Å)	Bonds	Angle (°)
C2-N2	1.4163(17)	C8-N1-S1	121.40(10)
C7-S1	1.7721(13)	C1-N2-C2	125.73(12)
C8-N1	1.4675(18)	O2-S1-O1	120.04(6)
C1-O3	1.2291(17)	O2-S1-N1	107.58(6)
C1-N2	1.3471(17)	O1-S1-N1	105.57(6)
N1-S1	1.6044(12)	N1-S1-C7	109.22(6)
O1-S1	1.4399(10)	C8-N1-S1-C7	77.02(12)
O2-S1	1.4339(10)	O3-C1-N2-C2	3.0(2)
C1-H1	0.95	C2-C7-S1-N1	72.31(12)
C2-C7	1.4018(18)	N2-C2-C7-S1	6.70(17)
N1-H1N	0.84(2)	C8-N1-S1-C7	77.02(12)
N2-H2N	0.85(2)	O3-C1-N2-C2	3.0(2)

Table 3. Hydrogen-bond geometry (Å, °)

D-H···*A*	*D*-H	H···*A*	*D*···*A*	*D*-H···*A*
N2-H2*N*···O1	0.85(2)	2.21(2)	2.7967(15)	127.0(17)
C1-H1···O1 [i]	0.95	2.4	3.2860(17)	155
N1-H1*N*···O3 [ii]	0.84(2)	2.07(2)	2.8988(16)	168(2)
N2-H2*N*···O3 [iii]	0.85(2)	2.57(2)	3.2832(16)	143.2(17)

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*+1, *y*, *z*.

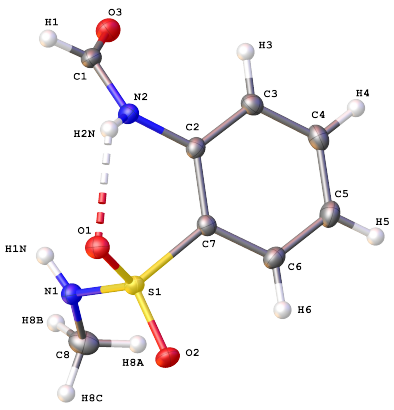

Figure 2. Molecular structure and intramolecular hydrogen bond of compound **4** with thermal ellipsoids drawn at the 50% probability level.

Analysis of intermolecular interactions of the compound **4** shows that the crystal packing is stabilized by intermolecular N-H···O and C-H···O hydrogen bonds that extended as infinite 1D chain along [100] (Figure 3 and Table 3).

3.2. Hirshfeld surface analysis

The intermolecular interactions were evaluated using Hirshfeld surface analysis. Molecular Hirshfeld surfaces of compound **4** were calculated using a standard (high) surface resolution and with the three-dimensional d_{norm} surfaces mapped over a fixed colour scale -0.5418 (red) to 1.0214 Å (blue) with the program CrystalExplorer 17 [33].

The Hirshfeld surface of the title compound mapped with shape-index (-1.0 to 1.0 Å) was also investigated (Figure 4). On the shape-index surface of compound **4**, convex blue regions represent hydrogen-donor groups and concave red regions represent hydrogen-acceptor groups [34]. The oxygen atom act as an acceptor while all the hydrogen implicated in hydrogen bonds act as donor group.

Table 4. Intramolecular S-O1→Cg (C2→C7 ring) interactions in compounds **4**.

Interactions	X···Cg	X-Perp	Gamma	Y-X···Cg	Y···Cg
O1 → Cg1 [1+*x*, *y*, *z*]	3.0284(12)	-2.920	15.38	109.98(5)	3.7716(8)

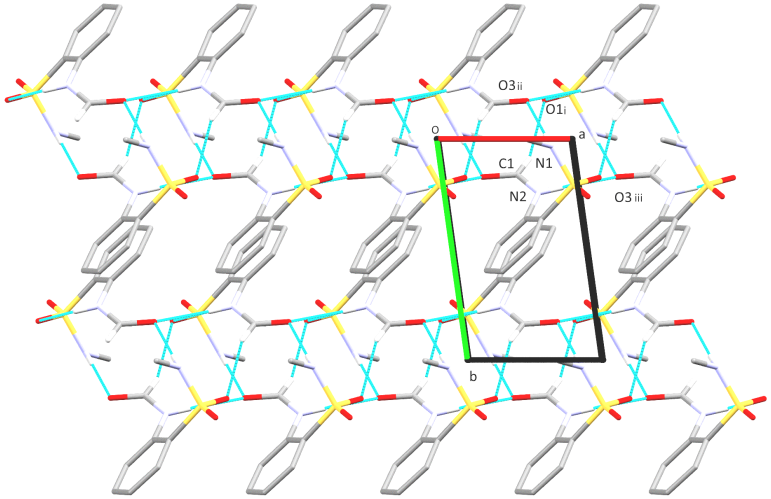

Figure 3. Infinite 1D chain along [100] by intermolecular hydrogen bonds observed in compound **4**. Non-bonded hydrogen atoms are omitted for clarity. Symmetry codes: (i) −*x*+2, −*y*, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*+1, *y*, *z*.

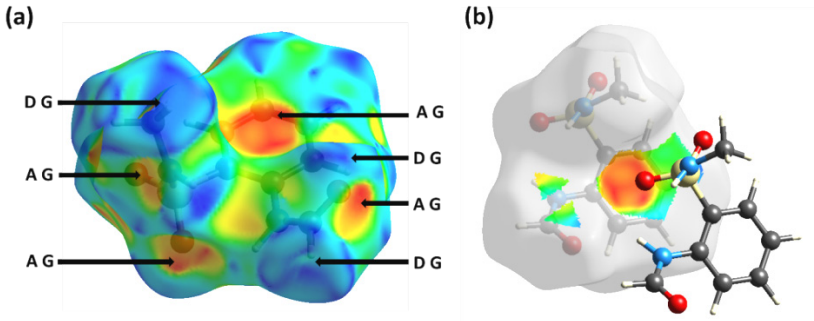

Figure 4. (a) Hirshfeld surfaces of compound **4** mapped with shape-index (DG: Donor Group, AG: Acceptor Group). (b) O···C /C···O contacts O1···Cg (1+*x*, *y*, *z*).

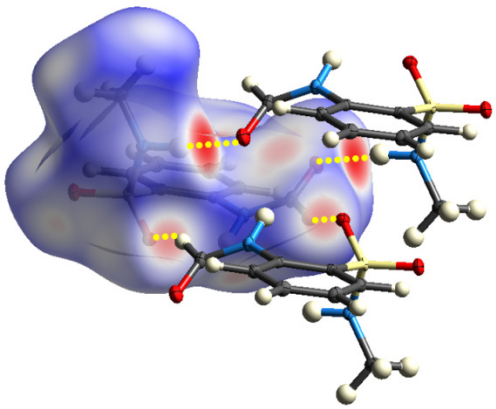

Figure 5. View of the three-dimensional Hirshfeld surface of the title compound plotted over d_{norm} in the range -0.5418 (red) to 1.0214 Å (blue), highlighting N-H···O and C-H···O hydrogen bonds by dashed yellow lines.

This analysis revelated the existence of an oxygen-π stacking interaction (C···O/O···C) between aromatic ring and O1 oxygen of sulfonamide group (Figure 4b). This interaction shows a distance between O1 and aromatic ring centroid (O1-Cg) of 3.0284(12) Å, an perpendicular distance of O1 respect to the aromatic ring plane of -2.920 Å, and an angle S-O-Cg of 109.98(5) ° (Table 4).

The analysis of intermolecular interactions through the mapping of d_{norm} is accomplished by considering the contact distances *di* and *de* from the Hirshfeld surface to the nearest atom inside and outside, respectively [35]. In compound **4**, the surface mapped over d_{norm} highlights four well defined red spots showing distances shorter than the sum of the Van der Waals radii (Figure 5).

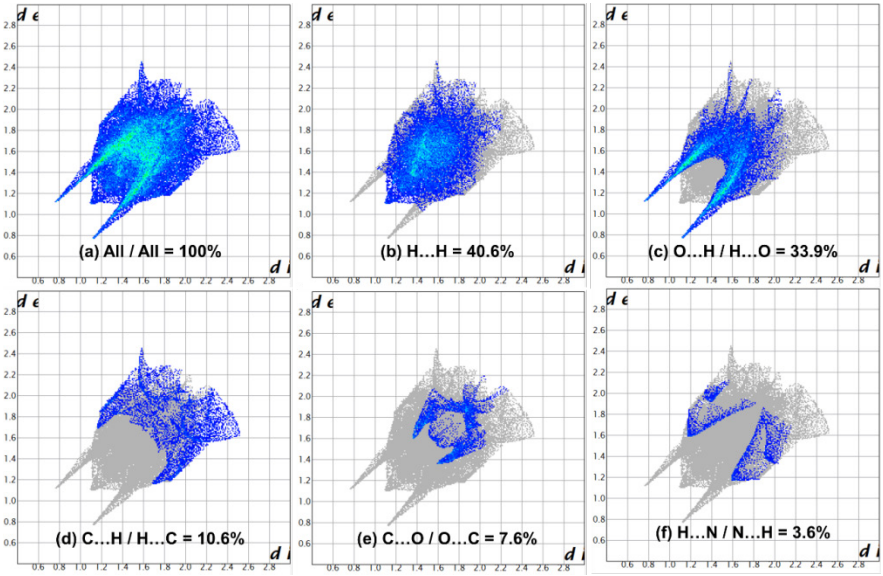

Figure 6. The Hirshfeld surface representations with the function d_{norm} plotted onto the surface for (a) all interactions, and (b) H···H, (c) O···H/H···O, (d) C···H/C···H, (e) C···O/O···C and (f) N···H/N···H interactions.

These dominant interactions correspond to intermolecular N-H···O and C-H···O hydrogen bonds and stacking interactions between the surface and the neighboring environment. The mapping also shows white spots with distances equal to the sum of the Van der Waals radii and blue regions with distances longer than the sum of the van der Waals radii [34,36]. The surfaces are transparent to allow visualization of the molecule.

We also analyzed the two-dimensional fingerprint (FP) of compound **4** plotted in Figure 6 highlighting particular close contacts of atom pairs and the contributions from different contacts. All interactions are presented in Figure 6a. The most significant contributions to the Hirshfeld surface (40.6 and 33.9%) are from H···H and O···H contacts (Figure 6b and 6c). These two interactions add to 74.5% of the intermolecular contacts of the Hirshfeld surface area. Others contributions correspond to C···H/C···H (10.6%), C···O/O···C (7.6%), N···H/N···H (3.6%), C···C (2.9%) and the remaining less-important interactions are inferior to 1%.

4. Conclusion

The hydrolysis of benzothiadiazine derivatives lead generally to isolated 2-aminobenzenesulfonamide, although substituted sulfamoylphenylformamide structures are described to be form during the first steps. In this study, we have determined the molecular structure of *N*-(2-(*N*-methyl sulfamoyl)phenyl)formamide obtained from slow degradation of 2-methyl-2*H*-1,2,4-benzothiadiazine 1,1-dioxide during crystallization using single crystal X-ray diffraction analysis. The molecular structure of compound **4** is stabilized by intra and intermolecular hydrogen bonds forming 1D chain along [100]. The analysis of intermolecular interactions through the mapping of Hirshfeld surface and two-dimensional fingerprint are presented and highlighted close contact of hydrogen bonds as well as acceptor and donor groups.

Acknowledgements

The authors thank the Université de Liège for a research grant.

Supporting information

CCDC-1915966 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
Author contributions: All authors contributed equally to this work.
Ethical approval: All ethical guidelines have been adhered.
Sample availability: Samples of the compounds are available from the author.

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