

Microwave synthesis, crystal structure and spectroscopic investigations of 2-([(2*E*)-(2-chlorobenzylidene) hydrazine] carbonyl} benzenesulfonamide and 2-([(2*E*)-2-[4-(dimethylamino) benzylidene] hydrazine} carbonyl} benzenesulfonamide

G Thiyagarajan^a, Ashutosh Pandey^{*a}, Peter Mayer^b & A Thamaraichelvan^c

^aDepartment of Chemistry, Motilal Nehru National Institute of Technology, Allahabad 211 004, India

^bDepartment of Chemistry and Biochemistry, University of Munich, Butenandtstr. 5-13, 81377 Munich, Germany

^cDepartment of Chemistry, Thiagarajar College, Madurai 625 020, India

E-mail: apandey70@yahoo.com

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The compounds 2-([(2*E*)-(2-chlorobenzylidene) hydrazine] carbonyl} benzenesulfonamide **5a** and 2-([(2*E*)-2-[4-(dimethylamino)benzylidene] hydrazine} carbonyl} benzenesulfonamide **5b** have been synthesized by microwave heating and characterized by NMR, FT-IR and single crystal X-ray crystallography. They have been obtained in higher yields in lesser reaction times through microwave irradiation. (C₁₄H₁₂ClN₃O₃S, **5a**): MW = 337.782, triclinic, *P*₁, *a* = 7.8804(7) Å, *b* = 11.5285(11) Å, *c* = 18.1620 (19) Å, α = 89.452(7)°, β = 76.847(8)°, γ = 75.999(8)°, *V* = 1557.2(3) Å³, *Z* = 4; (C₁₆H₂₀N₄O₄S, **5b**): MW = 364.421, Orthorhombic, *P*2₁2₁2₁, *a* = 16.5693(2) Å, *b* = 7.0856(3) Å, *c* = 37.8187(15) Å, α = 90°, β = 90°, γ = 90°, *V* = 1760.36(12) Å³, *Z* = 4. The delocalizations as well as the presence of the intra-molecular hydrogen bonding in **5a** and **5b** lead to the planarity of the benzene rings. There are π - π stacking interactions among the adjacent aromatic systems in both the compounds. It is also anticipated that the synchronized effect of both benzenesulfonamide and hydrazone groups in **5a** and **5b** should make them potential candidates for biological applications.

Keywords: Hydrazine, benzenesulfonamide, crystallography, microwave synthesis, condensation

Saccharin (Hsac = C₇H₅SO₃N, O-sulfobenzamide or 1,2-benzothiazol-3(2*H*)-one-1,1-dioxide) was discovered more than 100 years ago and is one of the best known and most widely used artificial sweeteners¹. Saccharin is a poly functional ligand with three hydrogen bond acceptors (one carbonyl and two sulfonyl O atoms) and one hydrogen bond donor (imino group NH). The imino hydrogen of saccharin is acidic with *pK_a* of 2.2. Besides, it has been used as an acid in the pharmaceutical industry^{2,3} and the crystal structures, and physical properties of several active pharmaceutical ingredient saccharinates have been reported recently⁴⁻⁶.

Furthermore, the syntheses and structures of Schiff bases have attracted much attention in biology and chemistry due to their physical properties and practical applications^{7,8}. It was found that the properties of these compounds are directly related to the presence of the intramolecular hydrogen bond and the conjugative interactions in the molecules^{9,10}. Furthermore, the sulfonamide derivatives constitute an important class of drugs¹¹ with a number of biological activities such as antibacterial¹²,

hypoglycemic¹³, diuretic¹⁴, anticarbonic anhydrase¹⁵, antithyroidal¹⁶ and anticancer¹⁷. Hydrazone derivatives which contain highly reactive azomethine group are very useful in designing of new drugs^{18,19}. By virtue of Nitrogen-Nitrogen-Oxygen (NNO) donor atoms they have also been introduced to coordination chemistry²⁰⁻²². It is expected that the synchronized effect of both benzenesulfonamide and hydrazone groups in a certain stoichiometry should have strong and manifold effect on the biological applications of the resulting products. Therefore, a detailed study on the preparation and structures of such compounds seems important for understanding their biological activities and coordination capabilities.

Chemists have synthesized a number of compounds by means of conventional heating methods²³. These reactions generally take longer time, consume greater energy and hence increase the cost of production. Among the available alternatives that may accelerate these synthetic processes, green microwave (MW) technology with power and temperature optimization is a better option²⁴ which has resulted in dramatic decline in reaction times *i.e.* from days and hours to minutes and seconds²⁵.

In the present study, we have synthesized 2-([(2*E*)-(2-chlorobenzylidene) hydrazine] carbonyl) benzenesulfonamide **5a** and 2-([(2*E*)-2-[4-(dimethylamino)benzylidene] hydrazine} carbonyl) benzenesulfonamide **5b** by reacting saccharin **1**, and hydrazine hydrate **2** with 2-chloro benzaldehyde **4a** and 4-dimethylamino benzaldehyde **4b** respectively through MW heating (**Scheme I**). The resulting products have been characterized by NMR and FT-IR spectral techniques and single crystal X-ray crystallography.

Results and Discussion

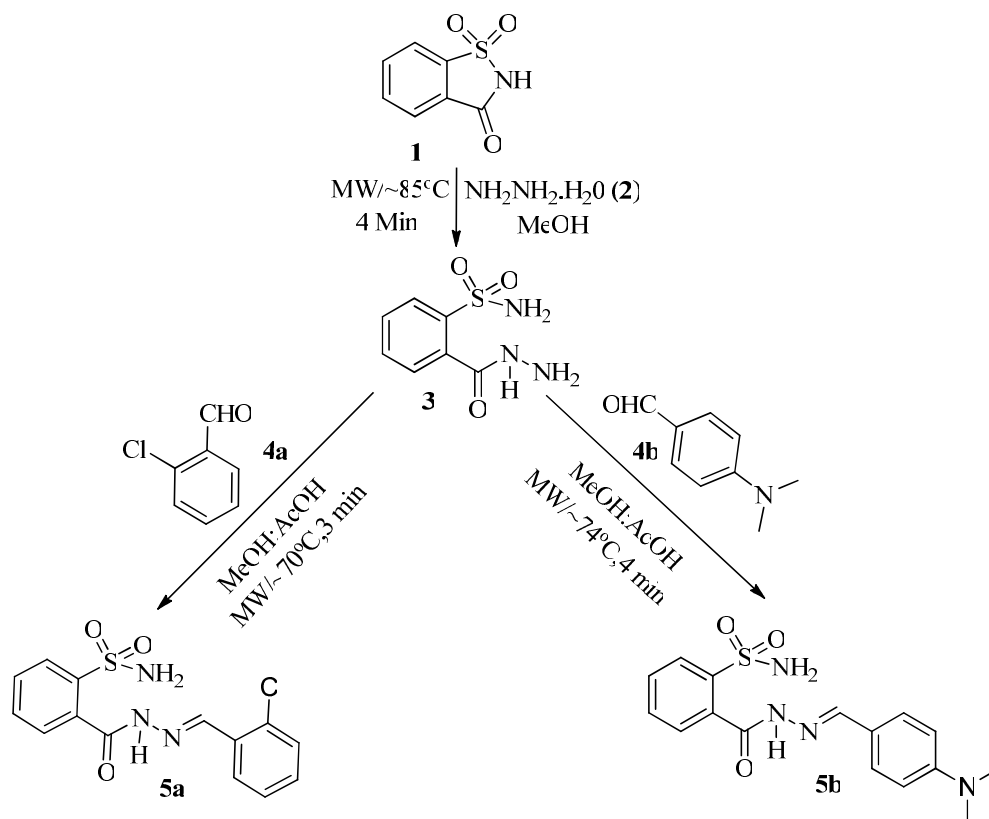
Synthesis

In the present study, compound **3** and target compounds (**5a** and **5b**, **Scheme I**) have been synthesized by microwave heating which involves two types of mechanistic pathways. As a consequence of an addition-elimination reaction, saccharin **1** reacts with hydrazine **2** to form 2-hydrazinocarbonyl-

benzenesulfonamide **3** by ring opening. Aromatic aldehydes **4a** and **4b** react with **3** to form the corresponding products through ‘aldehyde-amine’ coupling. Notably, we have synthesized compound **3** by new synthetic approach which is greener, easier and economical in comparison to earlier synthetic methods¹¹. Similarly, compounds **5a** and **5b** were synthesized in high yields, in shorter reaction times (within 3-4 min) without any further requirement of complex purification processes like column chromatography, *etc.* Therefore, the synthetic methodology adopted in the present work is much greener and economic for obtaining high purity products.

NMR spectral study of **3**, **5a** and **5b**

The appearance of four aromatic signals in ¹H NMR spectrum of compound **3** at values between δ 7.59-7.95 confirms its proposed molecular structure (as shown in **Scheme I**). In ¹³C NMR spectrum of



Saccharin **1**; Hydrazine hydrate **2**; 2-Hydrazinocarbonyl-benzenesulfonamide **3**; 2-Chloro benzaldehyde **4a**; 4-Dimethylaminobenzaldehyde **4b**;
2-([(2*E*)-(2-chlorobenzylidene) hydrazine] carbonyl) benzenesulfonamide **5a**;
2-([(2*E*)-2-[4-(dimethylamino)benzylidene] hydrazine} carbonyl) benzenesulfonamide **5b**.

Scheme I — Synthesis of target compounds **5a** and **5b**

compound **3**, six carbon signals at values between δ 141.2-126.9 correspond to the aromatic carbons and one peak due to the C=O group at δ 167.5 further substantiate its proposed structure. Furthermore, the aromatic protons of **3** have also been confirmed by DEPT spectra by the four carbon signals appearing at δ 131.7, 129.9, 129.1 and 126.9.

In ^1H NMR of compound **5a**, the peak at δ 12.24 corresponds to the occurrence of NH proton. Similarly, the proton signal at δ 11.78 confirms the existence of NH proton which is hydrogen bonded with a water molecule. An exchangeable proton signal appearing at δ 8.73 and 8.45 in **5a** was due to the presence of azomethene (N=CH) group. Similarly, the peak at δ 8.16 showed occurrence of azomethene group in **5b**. The two exchangeable protons at δ 7.01, 6.90 in **5a** and δ 7.03, 6.85 in **5b** has appeared as two singlets due to the presence of S-NH₂ group (Figure 1). Similar peaks owing to the S-NH₂ protons at δ 7.09 and 7.02 as two singlets have also been observed in compound **3**. The peaks corresponding to the two methyl groups in **5b** have appeared at δ 2.95 as singlet by integrating as six protons. Compound **5a** showed aromatic signal in the δ 8.14 to 7.38 range as multiplet by integrating eight protons. Similarly in the product **5b**, the peaks at δ 7.99-7.93 (2 protons), 7.71-7.48 (6 protons) and 6.76-6.71 (2 protons) confirmed its aromatic structure.

In **5a** and **5b**, the carbon signals due to the presence of N=CH group have been observed at δ values 144.4 and 149.2 respectively. The aromatic carbons could be readily distinguished from the other

carbons due to their characteristic peaks in the region of δ 110-140. The carbon signals of C=O group in **5a** and **5b** have appeared at δ 164.6 and 164 respectively. In compound **5b**, the peaks corresponding to the two methyl groups were visible at δ 39.9. All hydrogens of the aromatic rings in compounds **5a** and **5b** were further confirmed by peaks at δ values between 150 and 110 through DEPT spectra.

FT-IR spectra

FT-IR spectra of compounds **5a** and **5b** showed absorption bands at 1662 and 1654 cm^{-1} due to the presence of C=O groups while the bands observed at 1555.9 and 1526.8 cm^{-1} appropriated to C=N linkages respectively. The peaks at 3341 and 3359 cm^{-1} corresponded to the NH and NH₂ groups. The absorptions in the 3056 and 2991 cm^{-1} range were due to the CH stretching of the aromatic rings and the peaks due to O=S=O stretching appeared at 1166 and 1168 cm^{-1} .

Single crystal X-ray analysis

Single crystals of **5a** and **5b** suitable for X-ray diffraction study were obtained by slow evaporation of the respective compounds in methanol. For all the compounds, crystal data and structure refinements are given in Table I while Table II shows their selected bond lengths and angles. ORTEP diagrams for compounds **5a** and **5b** are shown in Figures 2 and 3. In **5a** the asymmetric unit has one molecule, while in **5b** it contains one benzenesulfonamide molecule and one water molecule. Molecular packing diagrams for

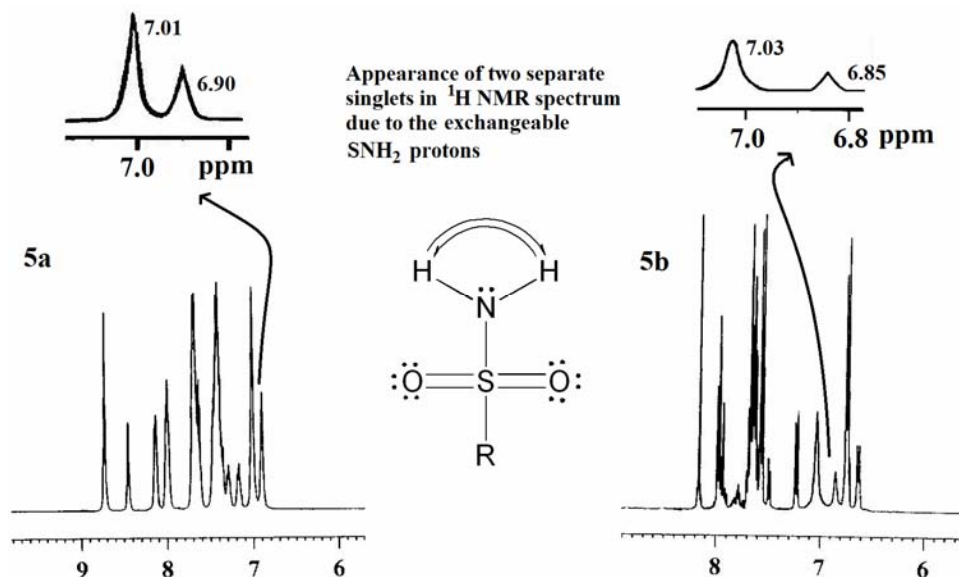


Figure 1 — Exchangeable protons of SNH₂ in **5a** and **5b**

Table I — Crystal data and structure refinement details for **5a** and **5b**

Compd	5a	5b
Net formula	C ₁₄ H ₁₂ ClN ₃ O ₃ S	C ₁₆ H ₂₀ N ₄ O ₄ S
Mol. weight (<i>M_r</i> /g mol ⁻¹)	337.782	364.421
Crystal size/mm	0.44 × 0.32 × 0.20	0.40 × 0.21 × 0.14
Temperature (K)	173(2)	173(2)
Radiation	MoK α	MoK α
Crystal System	triclinic	Orthorhombic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 2 ₁ 2 ₁
Unit cell dimensions	7.8804(7)	6.5693(2)
<i>a</i> (Å)	11.5285(11)	7.0856(3)
<i>b</i> (Å)	18.1620(19)	37.8187(15)
<i>c</i> (Å)	76.847(8)	90
α /°	89.452(7)	90
β /°	75.999(8)	90
γ /°	1557.2(3)	1760.36(12)
Volume (Å ³)	4	4
<i>D</i> (calc) [g/cm ³]	1.4408(3)	1.375(9)
μ (mm ⁻¹)	0.394	0.213
Absorption correction	multiscan	multiscan
Transmission factor range	0.53750/1.00000	0.97444/1.00000
Refls. Measured	8740	9764
<i>R</i> _{int}	0.0445	0.0263
Mean $\sigma(I)/I$	0.0678	0.0304
θ Range for data collection	4.25/26.37	4.23/26.37
Observed refls	4824	3401
Refls in refinement	6282	3573
Parameters	415	243
Restraints	6	5
<i>R</i> (<i>F</i> _{obs})	0.0579	0.0308
<i>R</i> _w (<i>F</i> ²)	0.1659	0.0752
<i>S</i>	1.030	1.075
Shift/error _{max}	0.001	0.001
Max electron density/e Å ⁻³	0.511	0.204
Min electron density/e Å ⁻³	-0.788	-0.239

5a and **5b** are given in **Figures 4** and **5** respectively. The crystal system of **5a** is triclinic whereas **5b** occurred in orthorhombic form.

The observed bond length of C7-O3 (**5a** = 1.232 Å, **5b** = 1.228 Å) falls in the range of double bond distance²⁶. The N3-C8 bond length of ~1.28 Å also confirmed it as a double bond. The hydrazone group displays a *trans* configuration about the C=N double bond in both compounds. In **5a** and **5b**, the C7-N2 bond length of 1.347 Å and the N2-N3 bond length of 1.387 Å in the hydrazone moiety are relatively short, suggesting some degree of delocalization in the system. The observed N2-N3-C8 bond angle in **5a** (113.3°) is less than the corresponding angle in **5b** (115.65°). This may be an outcome of the respective positions of the functional groups *i.e.* 2-chloro in **5a** and 4-dimethylamino in **5b**. The delocalization of the molecule as well as the presence of the intramolecular

N2-H2...O1 hydrogen bond in **5a** and **5b** leads to the planarity of the benzene in hydrazinecarbonyl benzenesulfonamide molecule.

In the crystal structure of **5b** the hydrazone groups are linked by the water molecules (**Figure 2**) through intermolecular O-H...O and O-H...N hydrogen bonds. Additionally, there are π - π stacking interactions²⁷ among the adjacent aromatic systems in both **5a** and **5b**. Other structural parameters associated with the benzenesulfonamide groups in both the molecules are normal.

Experimental Section

For all the syntheses, the used solvents were of reagent grade and dried by standard procedures²⁸. Saccharin **1**, hydrazine hydrate **2**, **4a** and **4b** were obtained from Sigma-Aldrich chemicals. MW irradiations were carried out in modified domestic

Samsung microwave oven, Model No. GE83HDT (2450 MHz, 850 W). The temperature during MW reaction was recorded by using an infrared thermometer. The synthesized products were frequently checked by thin layer chromatography

(TLC) on silica gel GF-254 using hexane/ethyl acetate as eluents and the spots were visualized by short exposure to iodine vapour, UV light and KMnO_4 solution. FT-IR spectra were recorded as KBr pellets on Nicolet 6700 spectrophotometer within 400-4000 cm^{-1} . NMR spectra were recorded in $\text{DMSO}-d_6:\text{CCl}_4$ (2:3) with TMS as internal standard on a Bruker AVANCE-DRX 400 MHz NMR spectrometer.

Table II — Selected bond length and bond angle for **5a** and **5b**

Atoms	5a	5b
Bond length (Å)		
N2-N3	1.387(3)	1.3886(19)
N3-C8	1.284(3)	1.277(2)
S1-N1	1.604(3)	1.617(14)
C1-C6	1.398(3)	1.408(2)
O3-C7	1.232(3)	1.228(2)
C10-C11	1.379(5)	1.370(2)
C11-C12	1.365(6)	1.412(2)
C3-C4	1.368(5)	1.381(3)
S1-O1	1.442(2)	1.427(12)
S1-O2	1.430(2)	1.4359(13)
C6-C7	1.496(3)	1.505(2)
C8-C9	1.466(3)	1.460(2)
N2-C7	1.348(3)	1.346(2)
Bond angle (°)		
C2-C3-C4	119.6(3)	119.99(18)
N2-C7-O3	123.3(2)	124.06(15)
N2-N3-C8	113.3(2)	115.65(14)
C7-N2-N3	119.4(2)	118.37(13)
O1-S1-C1	105.81(11)	107.1(1)
O2-S1-C1	107.22(10)	106.8(1)
C1-C6-C7	123.1(2)	123.0(2)
C11-C12-C13	120.1(3)	120.1(2)
O1-S1-O2	118.34(12)	119.21(8)
O1-S1-N1	107.81(11)	107.72(8)
O2-S1-N1	106.93(13)	106.62(7)

Synthesis of 2-hydrazinecarbonyl-benzenesulfonamide **3**

In a dry conical flask, saccharin (1 g, 5.46 mmol) and hydrazine hydrate (1.36 g, 27.3 mmol) were added and mixed with methanol (2 mL). The reaction mixture was then subjected to microwave irradiation at 300W/ $\sim 85^\circ\text{C}$ for 4 min to give a waxy yellow liquid. This liquid upon washing with 3 mL of 2-propanol: ethanol (7:3) solvent mixture yielded a white crystalline solid which was filtered and dried in vacuum. Crystallization of this product from methanol gave compound **3**. Yield: 1.12g, 95.4%. (When we employed less than 5 eq. of hydrazine hydrate, only 75% yield of the product was obtained. However, use of 5 eq. or more resulted in about 95% yield of the compound). White crystalline solid, m.p. $179-81^\circ\text{C}$. ^1H NMR (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 9.87 (s, 1H, NH), 7.95 (m, 1H, Ar-H), 7.65 (m, 2H, Ar-H), 7.59 (m, 1H, Ar-H), 7.09, 7.02 (ss, 2H, SNH_2), 3.87 (d, 2H, $J=2.8$, NNH_2); ^{13}C NMR (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 167.5(C=O), 141.2, 133.21, 131.7, 129.9, 129.1 and 126.9 (Ar-C); DEPT (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 131.7, 129.9, 129.1, 126.9 (Ar-CH); FT-IR (KBr): 3341.3 (NH, NH_2), 3068.7 (Ar-CH), 1662.8 (C=O), 1170.1 cm^{-1} (O=S=O).

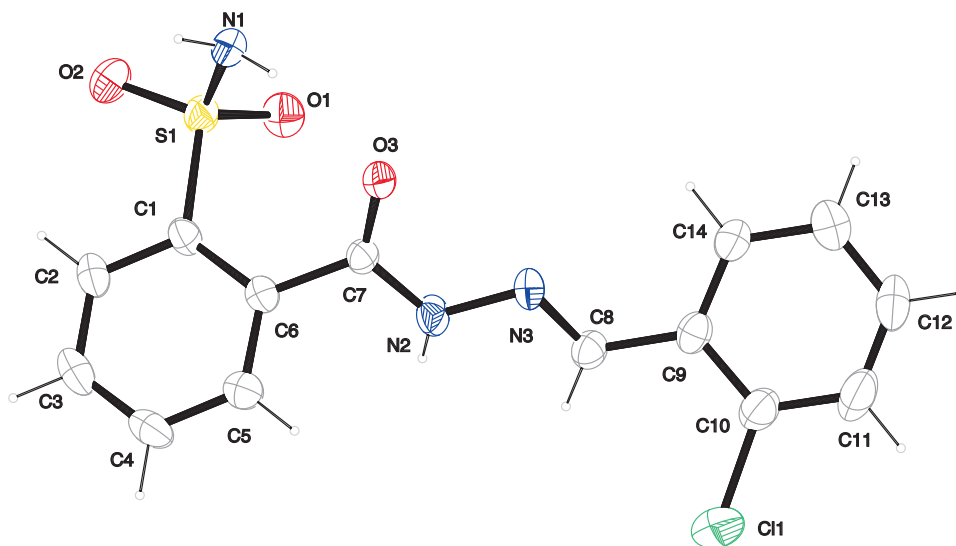


Figure 2 — ORTEP diagram for **5a**

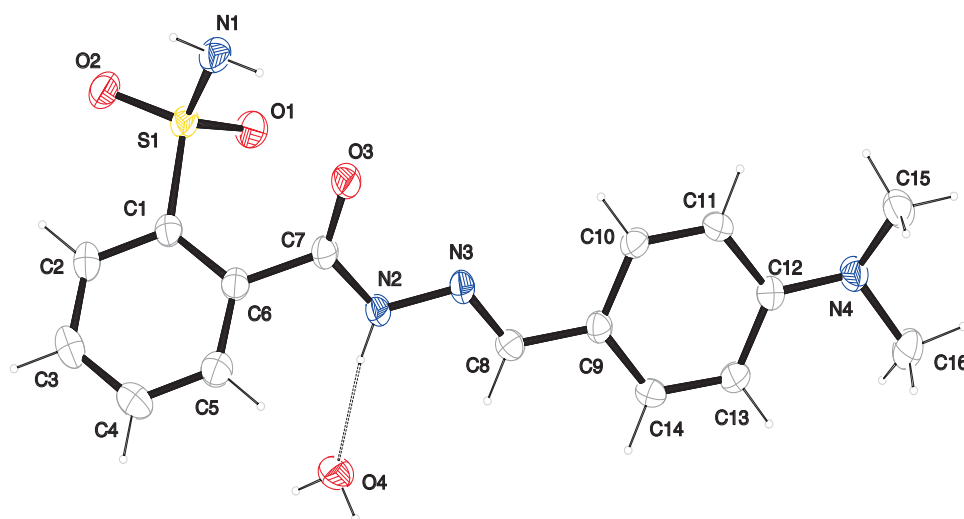


Figure 3 — ORTEP diagram of **5b** shows existence of intermolecular H-bonding with water molecule

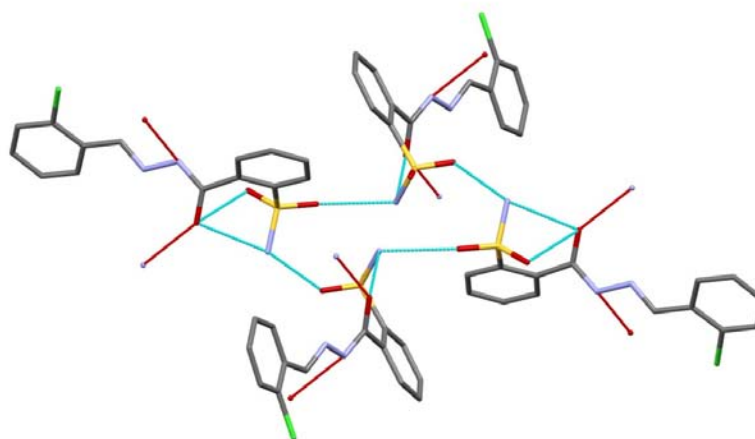


Figure 4 — Molecular packing in **5a**

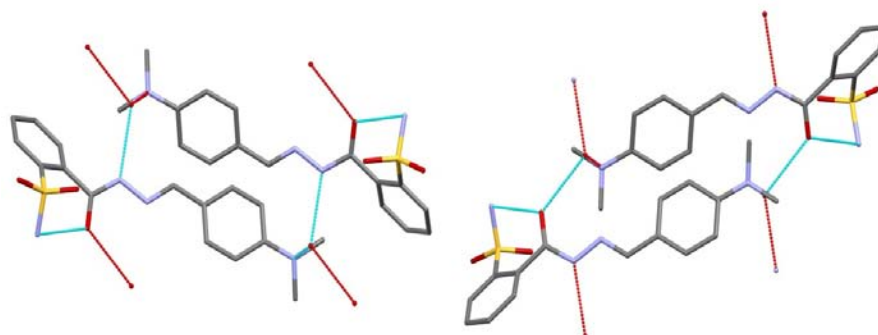


Figure 5 — Molecular packing view of **5b**

Synthesis of **5a**

To a dry conical flask, 2-hydrazinecarbonyl benzenesulfonamide **3** (0.5 g, 23.2 mmol) and *o*-chlorobenzaldehyde **4a** (0.39 g, 27.9 mmol) were mixed in 1 mL of methanol:acetic acid (9:1) mixture (under the mild acidic condition, yield of the product

has increased). The reaction mixtures were then subjected to microwave irradiation at 180W/~70°C for 3 min leading to the crude product which was cooled to RT and poured into 10 mL of ice cold water. The resulting solids were filtered, dried in vacuum and purified by crystallization in methanol.

White crystalline solid, yield: 96.3%, m.p. 185–87°C. ^1H NMR (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 12.24–12.22 (s, 1H, NH), 8.73–8.45 (s, 1H, N=CH), 8.14–7.38 (m, 6H, Ar-H), 7.01 and 6.90 (ss, 2H, S-NH₂), 6.81–6.66 (m, 2H, Ar-H); ^{13}C NMR (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 164.6 (C=O), 144.4 (N=CH), 141.6, 133.5, 133.1, 131.6, 131.1, 130.3, 129.5, 129.1, 128.9, 127.2, 127.0, 126.6; DEPT (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 144.4, 140.4, 131.6, 131.1, 130.3, 129.5, 128.9, 127.2, 127.0; FT-IR (KBr): 3332.1 (NH, NH₂), 3056.3 (Ar-CH), 1657.4 (C=O), 1555.9 (N=CH), 1166.1 cm^{-1} (O=S=O).

Synthesis of 5b

In a dry conical flask, 2-hydrazinecarbonyl benzenesulfonamide **3** (0.5 g, 23.2 mmol) with 4-methylamino benzaldehyde **4b** (0.41 g, 27.9 mmol) were mixed in methanol:acetic acid (9:1) mixture (1 mL) and then exposed to microwave irradiation at 180W/74°C for 4 min leading to the crude product which was cooled to RT and poured into 10 mL of ice cold water. The resulting solids were filtered, dried in vacuum and purified by crystallization in methanol. Orange crystalline solid, yield: 95.2%, m.p. 191–93°C. ^1H NMR (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 11.78–11.76 (s, 1H, NH), 8.16 (s, 1H, N=CH), 7.99–7.93 (m, 2H, Ar-H), 7.71–7.48 (m, 6H, Ar-H), 7.03–6.85 (ss, 2H, S-NH₂), 6.76–6.71 (m, 2H, Ar-H), 2.95 (s, 6H, N(CH₃)₂); ^{13}C NMR (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 164.0 (C=O), 149.2 (N=CH), 141.5, 133.7, 131.7, 130.2, 129.0, 128.9, 128.0, 127.1, 111.8, 39.9; DEPT (400 MHz, $\text{DMSO}-d_6:\text{CCl}_4$): δ 149.2, 131.7, 130.02, 129.0, 128.9, 128.0, 127.1, 111.8 (Ar-CH), 39.97 [N(CH₃)₂]; FT-IR (KBr): 3359.7 (NH, NH₂), 2991.6 (Ar-CH), 1654.2 (C=O), 1526.8 (N=CH), 1168.9 cm^{-1} (O=S=O).

Crystallography

The data collections were performed on an Oxford Diffraction Xcalibur 3 diffractometer (MoK α radiation, graphite monochromator) at a temperature of 173 K. The structures were solved by direct methods with SIR97 (Ref 29) and refined by full-matrix least-squares methods on F^2 with SHELXL-97 (Ref 30). All non-hydrogen atoms were refined anisotropically. For **5a** and **5b**, the C-bound hydrogen atoms were added geometrically and treated as riding on their parent atoms. The N-bound hydrogen atoms for compound **5a** and **5b** were refined to a fixed distance of 0.86(1) Å with $U(\text{H}) = 1.2 U(\text{N})$. Furthermore, the O-bound hydrogen atoms for crystal **5b** were refined to a fixed

distance of 0.82(1) Å with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{N/H})$. Multi-scan absorption corrections were performed with SCALE 3 ABSPACK³¹ for **5a** and **5b**.

Conclusion

Hydrazine carbonyl benzenesulfonamide derivatives **5a** and **5b** have been synthesized under MW irradiation, in short reaction times with small amount of solvents in reasonable yields. The obtained products have been characterized by various spectral techniques. The molecular structures of both the compounds have been determined by single crystal X-ray diffraction. The intermolecular H-bonding was observed in crystal **5b** with water molecule.

Acknowledgement

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Supplementary information

The crystallographic information file (CIF) data of **5a** (CCDC863708), **5b** (CCDC863709) can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223336033).

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