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Synthesis, Spectroscopic And Crystal Structure Studies of N-(Aryl)-4-Methoxybenzenesulfonamides: (4-OCH_3) C_6H_4-SO_2NH-C_6H_4(I-X)

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ABSTRACT

*Synthesis of three N-(aryl)-4-methoxybenzenesulfonamides of the general formula, (4-OCH_3) C_6H_4-SO_2NH-C_6H_4(i-X) [where i-X = H (**1**), 4-OCH_3 (**2**) & 4-Cl (**3**)], by the reaction between 4-methoxybenzenesulfonyl chloride and different substituted anilines is described. IR, ^{1}H NMR, ^{13}C NMR and single crystal X-ray analysis were carried out to determine the molecular and the crystal structure of the three compounds. The dihedral angle between the two aromatic rings is 55.14(1)^o in **1**, 56.34(1)^o in **2** and 42.58(1)^o in **3**. The crystal structures of all the three compounds exhibit strong N-H…O hydrogen bonds running into C(4) chains. The crystal packing of **2** is further stabilized by two weak C-H…π interactions.The supramolecular architecture in **1** and **3** is one dimensional, whereas **2** exhibit's two dimensional architecture.*

Keywords: Sulfonamides; X-ray analysis; N-H…O hydrogen bonds; C-H…π interactions; weak interactions.

INTRODUCTION

Sulfonamide drugs were the first among the chemotherapeutic agents to be used for the cure and prevention of bacterial infection in human beings [1]. They play a vital role as key constituent in a number of biologically active molecules. Till date, sulfonamides have been known to exhibit a wide variety of biological activities such as antibacterial [2], insecticidal [3], antifungal [4], antihepatitis [5], antiinflamatory [6], antitumor [7], anticancer [8], anti-HIV [9] and antitubercular activities [10]. In recent years extensive research studies have been carried out on the synthesis and evaluation of pharmacological activities of molecules containing sulfonamide moiety for different activities, and have been reported to be important pharmacophores [11].

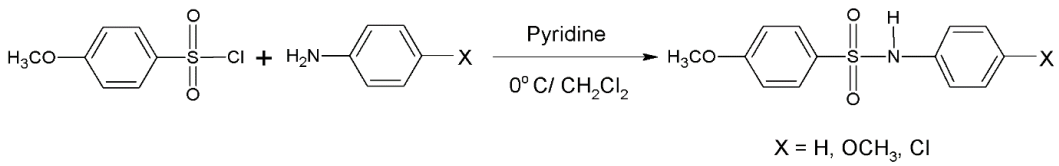
Keeping the above things in mind and in our continued efforts for understanding the structures of N-(aryl)-arylsulfonamides, we report herein the synthesis of N-(phenyl)-4-methoxybenzenesulfonamide (**1**), N-(4-

methoxyphenyl)-4-methoxybenzenesulfonamide (**2**) and N-(4-chlorophenyl)-4-methoxybenzene sulfon amide (**3**). Further, the formation of these compounds was established by recording their IR, ^{1}H NMR and ^{13}C NMR spectrum. The compounds were also subjected to single crystal X-ray diffraction studies to understand the substitution effect on the molecular structure and supramolecular architecture of the compounds in their solid state.

MATERIALS AND METHODS

Melting points were determined in open capillaries and are uncorrected. The molecular structures of the synthesized compounds were established using IR, ^{1}H NMR and ^{13}C NMR studies. Solid state FT-IR Spectra were recorded as KBr discs on Jasco FT-IR Spectrometer.^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ at 399.65 MHz and 100.50 MHz respectively on Bruker model avance II. All the chemical shifts were reported in parts per million (ppm) using tetramethylsilane (TMS) as an internal standard. Single Crystal X -Ray diffraction studies were carried out in Bruker Smart X2S diffractometer.

Synthesis: Aniline/4-methoxyaniline/4-chloroaniline (10 mmol) and excess pyridine were dissolved in dichloromethane (20 ml) and a solution of 4-methoxybenzenesulfonyl chloride (13 mmol) in dichloromethane (20 ml) was added drop wise with vigorous stirring at 273 K (**Scheme 1**). After 1 h, the reaction was quenched by addition of water and the oil was washed with dilute HCl. The organic layer separated was evaporated to give the crude product, which was recrystallized from n-hexane - dichloromethane (5:1).Yield: 78% for **1**, 81% for **2**& 75% for **3**.



Scheme 1

X-ray diffraction analysis: Colourless prisms of all the compounds were obtained from slow evaporation of the solutions of the respective compounds in aq. Ethanol (1:2). The data for all the three compounds were collected on a Bruker Smart X2S diffractometer using Cu Kα (λ = 1.54178) radiation. Image processing and data reduction were done using SAINT-Plus and XPREP [12]. The structure was solved by direct methods using SHELXS-97 [13]. The positions and anisotropic displacement parameters of all non-hydrogen atoms were included in the full-matrix least-square refinement using SHELXL97 [13] and the procedures were carried out for a few cycles until convergence was reached. The H atoms were placed at calculated positions in the riding model approximation (Aromatic C–H: 0.93 Å; Methoxy C-H: 0.96 Å) with U_{iso}(H) = 1.5Ueq(C) for methoxy H and U_{iso}(H) = 1.2Ueq(C) for all other H. The H atom of the NH group was located in a difference map and later refined freely. The non-hydrogen atoms were refined anisotropically. Molecular and packing diagrams were generated using ORTEP [14] and MERCURY [15].

Compound 1: Intensity data were collected up to a maximum of 64.87° for the compound in □-scan mode. A total of 8314 reflections were collected, resulting in 2091 independent reflections. The R factor after final convergence was 0.0366 and the maximum and minimum values of residual electron density were 0.31 and -0.32 eÅ^{-3}. The details of crystal data and structure refinement of **1** are given in **Table 1**.

Compound 2: Intensity data were collected up to a maximum of 64.66° for the compound in □-scan mode. A total of 10193 reflections were collected, resulting in 2348 independent reflections. The R factor after final convergence was 0.0521 and the maximum and minimum values of residual electron density were 0.29 and -0.22 eÅ^{-3}. The details of crystal data and structure refinement of **2** are given in **Table 1**.

Compound 3: Intensity data were collected up to a maximum of 64.53° for the compound in □-scan mode. A total of 5948 reflections were collected, resulting in 1899 independent reflections. The R factor

after final convergence was 0.0419 and the maximum and minimum values of residual electron density were 0.25 and -0.33 eÅ^{-3}. The details of crystal data and structure refinement of **3** are given in **Table 1**.

RESULTS AND DISCUSSION

All the three compounds were synthesized according to the **Scheme1** and characterized by IR, ^{1}H & ^{13}C NMR and single crystal X-ray diffraction studies.

Table 1. Crystal data and structure refinement for three compounds

Parameter	1	2	3
CCDC number	CCDC 1022758	CCDC 1022756	CCDC 1022757
Empirical formula	$C_{13}H_{13}NO_3S$	$C_{14}H_{15}O_4NS$	$C_{13}H_{12}O_3SClN$
Formula weight	263.30	293.33	297.75
Temperature/K	293(2)	293(2)	293(2)
Crystal system	orthorhombic	monoclinic	monoclinic
Space group	$P2_12_12_1$	$P2_1/c$	$P2_1$
a/Å	5.2049(4)	14.8640(12)	11.8514(8)
b/Å	14.5905(11)	5.2502(5)	5.0037(4)
c/Å	16.8621(12)	18.5252(14)	12.0364(9)
β/°	90.00	99.687(6)	104.736(4)
Volume/Å^3	1280.54(17)	1425.1(2)	690.29(9)
Z	4	4	2
ρ_{calc} mg/mm^3	1.366	1.367	1.432
Absorption coefficient	2.259	2.141	3.903
F(000)	552.0	616.0	308.0
Crystal size/mm^3	0.44 × 0.29 × 0.21	0.44 × 0.34 × 0.22	0.46 × 0.33 × 0.18
2Θ range for data collection	13.22 to 129.74°	9.68 to 129.32°	7.6 to 129.06°
Index ranges	- 6 ≤ h ≤ 5, - 17 ≤ k ≤ 16, - 19 ≤ l ≤ 19	-16 ≤ h ≤ 17, -4 ≤ k ≤ 6, -21 ≤ l ≤ 20	-13 ≤ h ≤ 13, -5 ≤ k ≤ 5, -14 ≤ l ≤ 13
Reflections collected	8314	10193	5948
Independent reflections	2091[R(int) = 0.0546]	2348 [Rint = 0.0911]	1899 [Rint = 0.0639]
Data/restraints/parameters	2091/ 0/ 165	2348/0/184	1899/1/174
Goodness-of-fit on F^2	0.737	1.050	1.016
Final R indexes [I>=2σ (I)]	R1 = 0.0366, wR2 = 0.0988	R1 = 0.0521, wR2 = 0.1340	R1 = 0.0419, wR2 = 0.1059
Final R indexes [all data]	R1 = 0.0412, wR2 = 0.1048	R1 = 0.0756, wR2 = 0.1507	R1 = 0.0488, wR2 = 0.1098
Largest diff. peak/hole / e Å^{-3}	0.31/ - 0.32	0.29/-0.22	0.25/-0.33

Spectral Analysis: Spectral data's of the synthesized compounds are in full agreement with its proposed structure. The details of the stretching frequencies, the signals of the respective protons (in ^{1}H NMR spectrum) and carbons (in ^{13}C NMR spectrum) of the title compound are verified on the basis of their chemical shifts, multiplicities, and coupling constants and are as follows.

N-(phenyl)-4-methoxybenzenesulfonamide (1): IR (νmax cm^{-1}): NH 3257.2 cm^{-1}, C-O 1267.0 and 1017.3 cm^{-1}, C-N 1092.5 cm^{-1}, S=O 1338.4 cm^{-1} (Asymmetric), 1160.9 cm^{-1} (Symmetric). ^{1}H-NMR (399.6 MHz, $CDCl_3$): δ 7.75 (d, *J* 9.9, 2H, Ar-H), 7.35 (s, 1H, NH), 7.23-7.19 (m, 2H, Ar-H), 7.10-7.05 (m, 3H, Ar-H), 6.87 (d, *J* 9.9, 2H, Ar-H), 3.82 (s, 3H, O-CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.85, 136.66, 130.10, 129.24, 129.24, 129.04, 129.04, 124.78, 120.97, 120.97, 113.99, 113.99, 55.29.

N-(4-methoxyphenyl)-4-methoxybenzensulfonamide (2): IR (νmax cm^{-1}): NH 3277.2 cm^{-1}, C-O 1249.6 and 1024.0 cm^{-1}, C-N 1092.5 cm^{-1}, S=O 1328.7 cm^{-1} (Asymmetric), 1158.0 cm^{-1} (Symmetric). ^{1}H-NMR (399.6 MHz, $CDCl_3$): δ 7.66 (d, *J* 9.9, 2H, Ar-H), 7.00 (d, *J* 9.9, 2H, Ar-H), 6.886 (d, *J* 9.1, 2H, Ar-H), 6.79 (s, 1H, NH), 6.77 (d, *J* 9.9, 2H, Ar-H), 3.82 (s, 3H, O-CH_3), 3.74 (s, 3H, O-CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.92, 157.77, 130.41, 129.41, 129.41, 129.02, 125.26, 125.26, 114.33, 114.33, 114.02, 114.02, 55.50, 55.34.

N-(4-chlorophenyl)-4-methoxybenzensulfonamide (3): IR (νmax cm^{-1}): NH 3245.6 cm^{-1}, C-O 1267.0 and 1020.2 cm^{-1}, C-N 1090.5 cm^{-1}, S=O 1331.6 cm^{-1} (Asymmetric), 1156.1 cm^{-1} (Symmetric). ^{1}H-NMR (399.6 MHz, $CDCl_3$): δ 7.74 (d, *J* 9.9, 2H, Ar-H), 7.37 (s, 1H, NH), 7.20 (d, *J* 9.5, 2H, Ar-H), 7.05 (d, *J* 8.7, 2H, Ar-H), 6.90 (d, *J* 9.1, 2H, Ar-H), 3.82 (s, 3H, O-CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.20, 135.27, 130.56, 129.89, 129.38, 129.38, 129.28, 129.28, 122.64, 122.64, 114.26, 114.26, 55.52.

Crystallography: The details of the crystal data and structure refinement of all the compounds are given in Table 1. The selected bond lengths and bond angles for compound **1**, **2** and **3** are respectively given in Table 2. Tables 3 gives the details of the respective hydrogen bonding and weak interactions in **1**, **2** and **3**. The ORTEP diagrams of molecules **1**, **2** and **3** with thermal ellipsoids drawn at 50% probability are respectively shown in Figures 1, 2 and 3. Further, the packing diagrams of **1, 2** and **3** are shown in Figures 4, 5and 6 respectively.

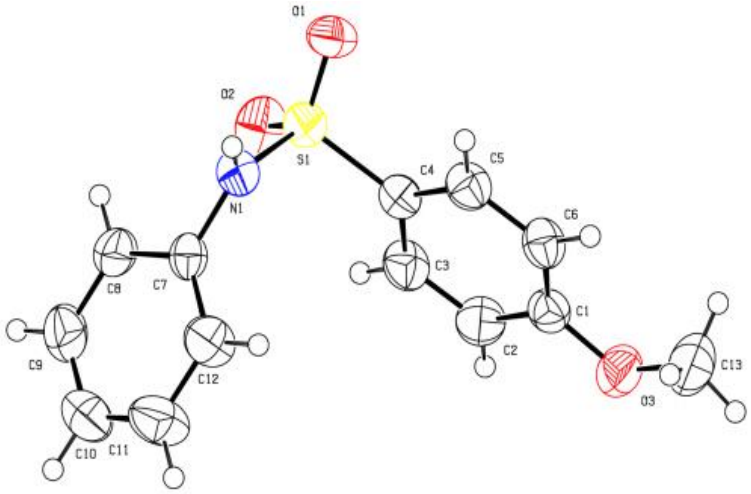


Figure 1: Molecular structure of 1, showing displacement ellipsoids drawn at the 50% Probability level.

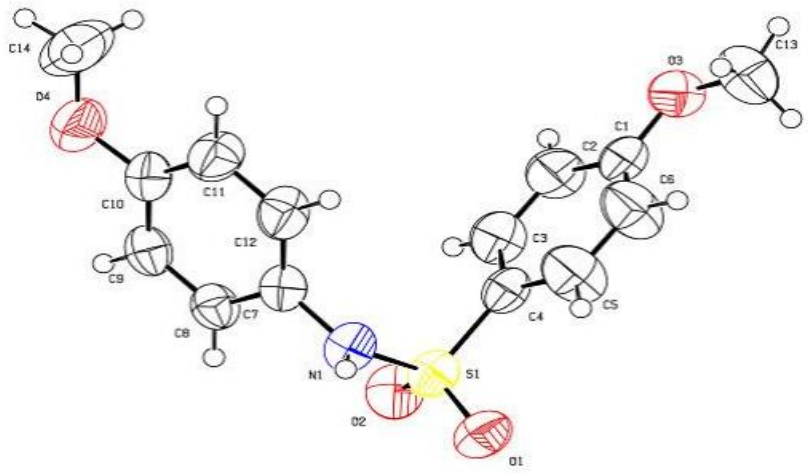


Figure 2: Molecular structure of 2, showing displacement ellipsoids drawn at the 50% Probability level.

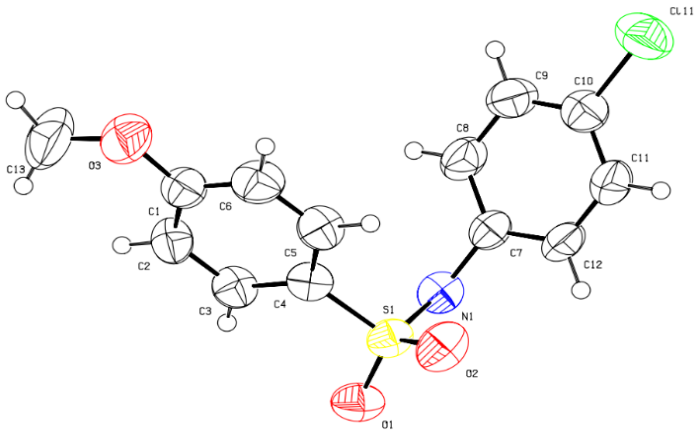


Figure 3: Molecular structure of 3, showing displacement ellipsoids drawn at the 50% Probability level.

Compound **1**, crystallizes in the orthorhombic crystal system and $P2_12_12_1$ space group with the unit cell parameters a = 5.2049(4) Å, b = 14.5905(11) Å, c = 16.8621(12) Å, Z = 4 and V = 1280.54(17) $Å^3$. The methoxy group is approximately coplanar with the attached benzene ring, the C13-O3-C1-C6 torsion being 0.77(1)°. The dihedral angle between the two benzene rings in **1** is 55.14(1)°. Also, the molecule is bent at S atom with the C4-S1-N1-C7 torsional angle being -72.87(1)°.

In contrast to this, compound **2** crystallizes in the monoclinic crystal system and $P2_1/c$ space group with unit cell parameters a = 14.8640(12) Å, b = 5.2502(5) Å, c = 18.5252(14) Å, β = 99.687(6)°, Z = 4 and V = 1425.1(2) $Å^3$. This shows that introducing a methoxy group into the para position of the aniline ring in **1**, significantly alters the crystal system, space group and unit cell parameters. The methoxy groups on the two benzene rings are approximately coplanar with their respectively attached benzene rings, the two torsions C13-O3-C1-C6 (Sulfonyl benzene ring) and C14-O4-C10-C11 (Aniline ring) being 0.18(1)° and -1.01(1)°. The dihedral angle between the two benzene rings in **2** is 56.34(1)°. Further, the molecule is bent at S atom with the C4-S1-N1-C7 torsional angle being 66.21(1)°.

Similar to **2**, compound **3** crystallizes in the monoclinic crystal system and $P2_1$ space group with unit cell parameters a = 11.8514(8) Å, b = 5.0037(4) Å, c = 12.0364(9) Å, β = 104.736(4)°, Z = 2 and V = 690.29(9) $Å^3$. Similar to **1** and **2**, the methoxy group in **3** is approximately coplanar with the attached benzene ring, the C13-O3-C1-C6 torsion being -4.94(1)°. However, in contrast to **1** and **2**, the dihedral angle between the two benzene rings in **3** is slightly less, it being 42.58(1)°. The molecule **3** is bent at S atom with the C4-S1-N1-C7 torsional angle being 72.54(1)°. From the above fact it is evident that introducing a substituent into the *para* position changes the crystal system from orthorhombic to monoclinic. However, such regularity is not observed in case of dihedral angles. Introduction of methoxy group has no significant effect on the dihedral angle, whereas, introducing chlorine slightly decreases the dihedral angle.

In the crystal structure of **1**, the molecules are linked by a single N-H…O (Sulfonyl) hydrogen bond (Table 3). Symmetry equivalent molecules are linked into C(4) chains [16] running parallel to [100] direction (Figure 4). Four chains pass through each unit cell but there are no direction-specific interactions between adjacent chains. Hence, the structure exhibits one dimensional architecture.

Table 2. Selected bond lengths (Å) and angles (°) for the three compounds

1				**2**				**3**			
Bond Lengths		**Bond Angles**		**Bond Lengths**		**Bond Angles**		**Bond Lengths**		**Bond Angles**	
S1-O2	1.423(2)	O2-S1-N1	106.89(13)	S1-O2	1.429(2)	O2-S1-N1	107.43(12)	S1-O2	1.423(3)	O2-S1-N1	107.05(17)
S1-O1	1.432(2)	O2-S1-C4	107.95(13)	S1-O1	1.429(2)	O2-S1-C4	107.55(13)	S1-O1	1.428(2)	O2-S1-C4	107.24(17)
S1-N1	1.624(2)	O1-S1-O2	120.23(14)	S1-N1	1.623(2)	O1-S1-O2	120.28(14)	S1-N1	1.629(3)	O1-S1-O2	120.09(17)
S1-C4	1.757(3)	O1-S1-N1	105.58(12)	S1-C4	1.749(3)	O1-S1-N1	104.87(13)	S1-C4	1.741(3)	O1-S1-N1	105.83(16)
N1-C7	1.433(3)	O1-S1-C4	107.84(12)	N1-C7	1.438(3)	O1-S1-C4	108.28(13)	N1-C7	1.437(4)	O1-S1-C4	108.60(15)
C1-O3	1.364(3)	N1-S1-C4	107.79(12)	C1-O3	1.350(3)	N1-S1-C4	107.87(13)	C1-O3	1.364(4)	N1-S1-C4	107.44 (15)
C13-O3	1.419(4)	C7-N1-S1	122.25(18)	C13-O3	1.428(4)	C7-N1-S1	121.60(18)	C13-O3	1.444(7)	C7-N1-S1	120.5(2)
		C8-C7-N1	120.0(2)			C8-C7-N1	120.9(2)			C8-C7-N1	120.2(2)
		C12-C7-N1	120.9(2)			C12-C7-N1	120.3(3)			C12-C7-N1	119.6(3)
		C3-C4-S1	120.6(2)			C3-C4-S1	122.3(2)			C3-C4-S1	120.2(2)
		C5-C4-S1	119.6(2)			C5-C4-S1	120.4(2)			C5-C4-S1	120.6(3)
		O3-C1-C2	115.4(3)			O3-C1-C2	116.3(3)			O3-C1-C2	124.5(3)
		O3-C1-C6	124.3(3)			O3-C1-C6	124.6(3)			O3-C1-C6	115.4(3)
		C1-O3-C13	118.2(2)			C1-O3-C13	118.7(3)			C1-O3-C13	118.1(3)

The supramolecular assembly of **2** is considerably more complex than that in **1**. In the crystal structure of **2**, the molecules are linked by a weak N-H…O (Sulfonyl) hydrogen bond (Table 3). Symmetry equivalent molecules are linked into C(4) chains [16]) running parallel to (010) direction (Figure 5). Similar to **1**, four chains pass through each unit cell. However, in this case there are two structure-directing weak C-H…π interactions (Table 3). Two adjacent chains within a unit cell are linked to one another through C9-H9…π (cg is the centroid of the aniline ring) interactions along (100) (Figure 5). Further, the C(4) chain in one unit cell is connected to the adjacent C(4) chain in the neighbouring unit cell by another structure directing C13-H13…π (cg is the centroid of the aniline ring) interactions along (100) (Figure 5). Thus, the supramolecular architecture in **2** is two dimensional.

Similar to **1**, the supramolecular architecture in **3** is influenced by a strong N-H..O (Sulfonyl) hydrogen bond. The hydrogen bond parameters and the symmetry are given in Table 3. The molecules in **3** are linked into C(4) chains [16] via N-H...O hydrogen bonds running parallel to (010) direction (Figure 6). Two chains pass through each unit cell, and similar to **1** there are no direction-specific interactions between the adjacent chains. Hence, **3** display one dimensional supramolecular architecture.

Table 3. Hydrogen-bond geometries observed in the three compounds (Å, ^{o})
(D: donor; A: acceptor; H: hydrogen).

Compound 1				
D-H…A	**D-H**	**H…A**	**D…A**	**D-H…A**
N1-HN1…O2^{i}	0.86(1)	2.29(1)	2.9575(1)	134(1)
Symmetry Code: (i) -1+x, y, z				
Compound 2				
Cg represents the centroid of the sulfonyl benzene ring.				
N1-HN1…O2^{i}	0.86(1)	2.47(1)	3.0385(1)	124(1)
C13-H13B-Cg^{ii}	0.96	2.88	3.7063	145
C9-H9-Cg^{iii}	0.93	2.96	3.6750	135
Symmetry Code: (i) x, 1+y, z; (ii) -x,1/2+y,1/2-z; (iii) 1-x,-1/2+y,1/2-z				
Compound 3				
N1-HN1…O2^{i}	0.86(1)	2.20(1)	2.8902(1)	138(1)
Symmetry Code: (i) x, -1+y, z				

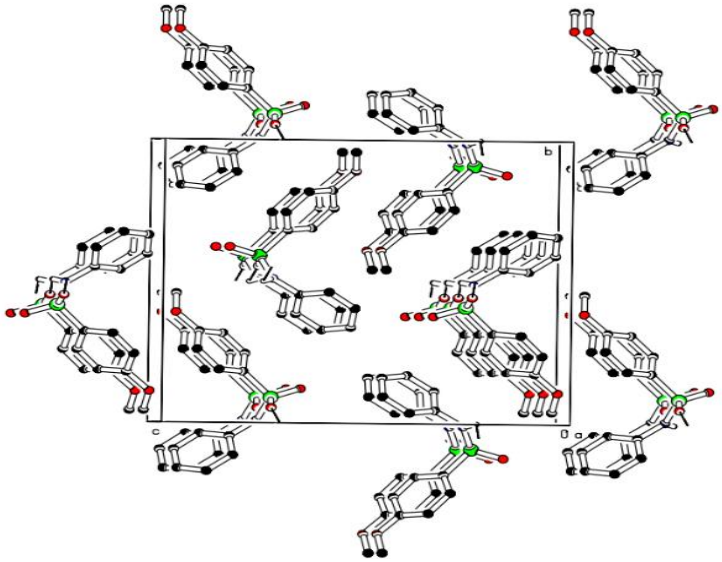


Figure 4: View of the molecular packing in 1, showing N–H...O hydrogen bonds running down *a* axis. H-atoms not involved in hydrogen bonding are omitted for clarity purpose.

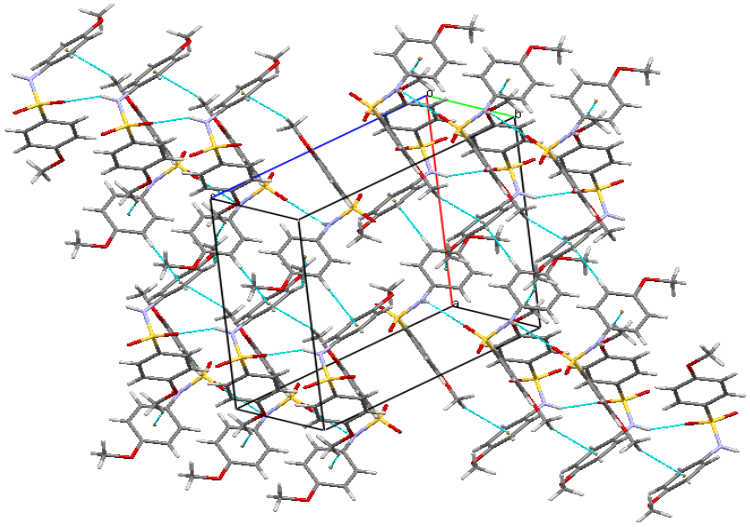


Figure 5: N-H…O hydrogen bonds and two C-H…π interactions leading to a two dimensional Supramolecular architecture in 2**.**

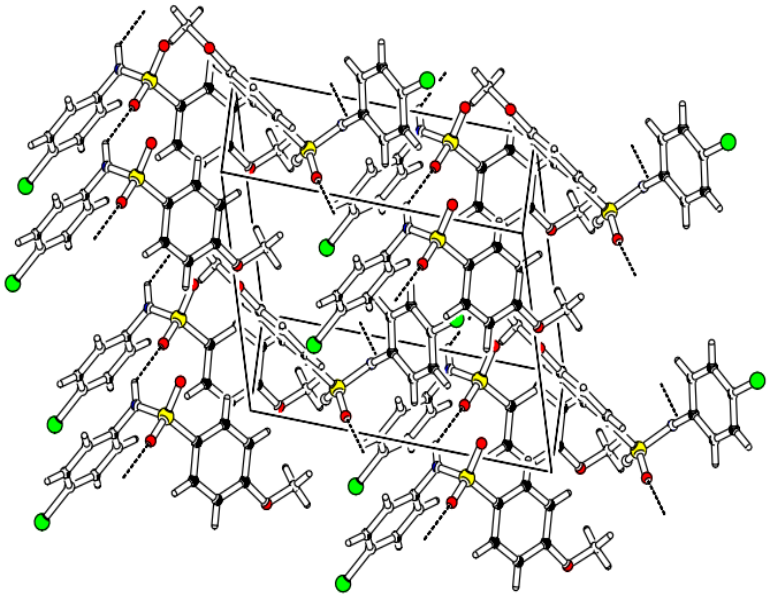


Figure 6: View of the molecular packing in3, displaying N–H...O hydrogen bonds along *c* axis.

APPLICATIONS

N-(Aryl)-aryl sulfonamides exhibits various biological activities and studying their molecular and crystal structure might give an insight to the mechanisms of their biological actions.

CONCLUSIONS

The present work describes the synthesis of three N-(aryl)-4-methoxybenzenesulfonamides and their characterization by IR, ^{1}H NMR and ^{13}C NMR techniques. Further, X-ray analysis was carried out to understand the effect of substituents on the molecular and crystal structure of the compounds. It is noteworthy that varying the substituents at the *para* position in the aniline ring has substantial effect on the molecular structure and supramolecular architecture of the compounds in the solid state.

Supplementary Materials: CCDC 1022756, 1022757 & 1022758 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk.

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