Synthetic, Infrared, ^{1}H and ^{13}C NMR Spectral Studies on Potassium Salts of N-Chloroarylsulphonamides

K. Jyothi and B. Thimme Gowda

Department of Post-Graduate Studies and Research in Chemistry, Mangalore University, Mangalagangotri – 574 199, Mangalore, India

Reprint requests to Prof. B. T. G. Fax: 91 824 2287 367; E-mail: gowdabt@yahoo.com

Z. Naturforsch. **59a,** 64 – 68 (2004); received January 7, 2003

Several N-chloroarylsulphonamides of the configuration, 4-X-$C_6H_4SO_2(K)NCl \cdot xH_2O$ (where X = H, CH_3, C_2H_5, F, Cl or Br) and *i*-X, *j*-$YC_6H_3SO_2(K)NCl \cdot xH_2O$ (where *i*-X, *j*-Y = 2,3-$(CH_3)_2$; 2,4-$(CH_3)_2$; 2,5-$(CH_3)_2$; 2-CH_3,4-Cl; 2-CH_3,5-Cl; 3-CH_3,4-Cl; 2,4-Cl_2 or 3,4-Cl_2) are prepared, characterised, and their infrared spectra in the solid state and NMR spectra in solution are measured and correlated. Comparison of the infrared spectra of the potassium salts of N-chloro-arylsulphonamides with the corresponding arylsulphonamides shows that the strong absorptions in the range 947 – 933 cm^{-1} are due to N-Cl stretching vibrations. The effect of ring substitution on the N-Cl frequencies is non-uniform. The frequencies in the ranges 1404 – 1370 cm^{-1} and 1149 – 1125 cm^{-1} are respectively assigned to S=O asymmetric and symmetric vibrations. The effect of substitution in the phenyl ring in terms of electron withdrawing and electron donating groups is non-systematic. Empirical correlations relating the chemical shifts to the structures are considered. The chemical shifts of aromatic protons and carbons in all the N-chloroarylsulphonamides have been calculated by adding substituent contributions to the shift of benzene, as per the principle of substituent addition. Considering the approximation made, the agreement between the calculated and experimental chemical shifts is reasonably good.

Key words: Synthetic, Infrared, ^{1}H and ^{13}C NMR Spectra; N-Chloroarylsulphonamides.

1. Introduction

The amide moiety is an important constituent of many biologically significant compounds [1, 2]. Sulphonamides are of fundamental chemical interest as they show distinct physical, chemical and biological properties. Many sulphonamides and their N-chloro compounds exhibit pharmacological activity, which has further stimulated recent interest in their chemistry. Further, many sulphonamides and their N-chloro compounds exhibit fungicidal and herbicidal activities because of their oxidising action in aqueous, partial aqueous and non-aqueous media [3 – 12]. Therefore an understanding of the formation, properties and reactions of sulphonamides is central to future development in such areas as medical and redox chemistry. A great deal of work on the spectroscopic aspects of amides needs to be done for correlating frequencies with the chemical bond parameters. Thus we are interested in the spectroscopic studies of amides in their crystalline state [13 – 23].

We have recently reported spectroscopic studies on a number of arylsulphonamides and their sodium salts of N-chloro compounds. We report now the infrared and NMR spectra of potassium salts of N-chloroarylsulphonamides of the configuration, 4-X-$C_6H_4SO_2(K)NCl \cdot xH_2O$ (where X = H, CH_3, C_2H_5, F, Cl or Br) and *i*-X, *j*-$YC_6H_3SO_2(K)NCl \cdot xH_2O$ (where *i*-X, *j*-Y = 2,3-$(CH_3)_2$, 2,4-$(CH_3)_2$, 2,5-$(CH_3)_2$, 2-CH_3,4-Cl, 2-CH_3,5-Cl, 3-CH_3,4-Cl, 2,4-Cl_2 or 3,4-Cl_2).

2. Experimental

2.1. Preparation of Potassium Salts of N-Chloroarylsulphonamides

The mono and di-substituted arylsulphonamides, were prepared by the chlorosulphonation of the corresponding substituted benzenes with chlorosulphonic acid and by subsequent conversion of the resulting arylsulphonylchlorides to amides by boiling the former with concentrated ammonium hydroxide by the procedures reported in [21 – 25]. The obtained arylsulphonamides were recrystallised to constant melting points from dilute ethanol and dried at 105 °C (Ta-

Sl. No.	Arylsulphonamides	m. p. (°C) obs (lit.)	Potassium salts of N-(chloro)-arylsulphonamides	m. p. (°C)
1	$C_6H_5SO_2NH_2$	150 (149 – 152)	$C_6H_5SO_2(K)NCl{\cdot}xH_2O$	140 – 142
2	$4\text{-}CH_3C_6H_4SO_2NH_2$	135 (135 – 137)	$4\text{-}CH_3C_6H_4SO_2(K)NCl{\cdot}3H_2O$	175 – 177
3	$4\text{-}C_2H_5C_6H_4SO_2NH_2$	99 – 101	$4\text{-}C_2H_5C_6H_4SO_2(K)NCl{\cdot}xH_2O$	179 – 180
4	$4\text{-}FC_6H_4SO_2NH_2$	125 (124 – 125)	$4\text{-}FC_6H_4SO_2(K)NCl{\cdot}xH_2O$	170
5	$4\text{-}ClC_6H_4SO_2NH_2$	143 (142 – 143)	$4\text{-}ClC_6H_4SO_2(K)NCl{\cdot}xH_2O$	160 – 161
6	$4\text{-}BrC_6H_4SO_2NH_2$	162 (163)	$4\text{-}BrC_6H_4SO_2(K)NCl{\cdot}xH_2O$	165
7	$2,3\text{-}(CH_3)_2C_6H_3SO_2NH_2$	138 – 140	$2,3\text{-}(CH_3)_2C_6H_3SO_2(K)NCl{\cdot}xH_2O$	125
8	$2,4\text{-}(CH_3)_2C_6H_3SO_2NH_2$	140 – 142	$2,4\text{-}(CH_3)_2C_6H_3SO_2(K)NCl{\cdot}xH_2O$	155
9	$2,5\text{-}(CH_3)_2C_6H_3SO_2NH_2$	149 – 151	$2,5\text{-}(CH_3)_2C_6H_3SO_2(K)NCl{\cdot}xH_2O$	185
10	$2\text{-}CH_3,4\text{-}ClC_6H_3SO_2NH_2$	180 – 182 (184 – 185)	$2\text{-}CH_3,4\text{-}ClC_6H_3SO_2(K)NCl{\cdot}xH_2O$	170
11	$2\text{-}CH_3,5\text{-}ClC_6H_3SO_2NH_2$	139 – 141 (142 – 143)	$2\text{-}CH_3,5\text{-}ClC_6H_3SO_2(K)NCl{\cdot}xH_2O$	168
12	$3\text{-}CH_3,4\text{-}ClC_6H_3SO_2NH_2$	132 – 134 (126)	$3\text{-}CH_3,4\text{-}ClC_6H_3SO_2(K)NCl{\cdot}xH_2O$	182
13	$2,4\text{-}Cl_2C_6H_3SO_2NH_2$	178 – 180 (179 – 180)	$2,4\text{-}Cl_2C_6H_3SO_2(K)NCl{\cdot}xH_2O$	205
14	$3,4\text{-}Cl_2C_6H_3SO_2NH_2$	141 – 143 (134 – 135)	$3,4\text{-}Cl_2C_6H_3SO_2(K)NCl{\cdot}xH_2O$	152

Table 1. Melting points of potassium salts of N-(chloro)-arylsulphonamides.

Table 2. Infrared absorption frequencies (cm^{-1}) of potassium salts of N-(chloro)-*p*-substituted benzenesulphonamides.

Assignment	$4\text{-}X\text{-}C_6H_4SO_2(K)NCl.xH_2O$, X =					
	H	CH_3	C_2H_5	F	Cl	Br
C-H (Ar sym str)	3058.6 w	3058.6 w	3037.3 w	3066.3 w	3074.9 w	3073.0 w
C-H (alk str)	–	2922.6 w	2967.9 m 2931.3 m 2871.5 w	–	–	–
combination bands	1963.2 w 1892.8 w 1735.6 w	1923.7 w	1916.9 w	1900.5 w 1769.4 w	1907.3 w	1906.3 w 1784.8 w
C=C (Ar in-plane str)	1646.9 m 1560.1 w 1443.5 m	1627.6 m 1596.8 m 1491.7 w	1635.3 m 1597.7 m 1493.6 w	1649.8 m 1595.8 s 1494.6 s	1653.7 m 1587.1 w 1477.2 m	1650.8 m 1576.5 m 1472.4 m
S=O (asym str)	1383.7 w	1395.3 w	1403.9 s	1403.0 w	1395.3 m	1385.6 s
(sym str)	1142.6 s	1130.1 s	1143.6 s	1143.6 s	1143.6 s	1139.7 s
C-X (str)	–	–	–	1243.9 s	1036.6 m	566.0 s
C-H (Ar in-plane bend)	1091.5 s 1021.1 w	1090.6 s 1018.2 w	1094.4 s 1018.2 w	1090.6 s 1011.5 w	1090.6 s 1014.4 w	1087.7 s 1011.5 m
N-Cl (str)	**934.3 s**	**945.0 s**	**937.2 s**	**935.3 s**	**935.3 s**	**933.4 s**
S-N (sym str)	856.2 w	849.5 w	827.3 s	832.1 s	825.4 w	821.5 s
C-S (str)	762.7 m	813.8 s	784.9 w	–	754.0 s	740.5 s
C-H (Ar out of-plane bend)	753.1 m 723.2 m	687.5 s	693.3 s	695.2 s	704.9 s 683.6 s	702.9 s 678.8 s
C=C (Ar out of-plane bend)	467.7 m	459.9 w	456.1 w	459.9 w	481.2 m	419.4 w

s = strong, m = medium and w = weak.

ble 1). To get potassium salts of N-chloro-arylsulphonamides, the arylsulphonamides were N-chlorinated:

Pure chlorine gas was bubbled through clear aqueous solutions of substituted benzenesulphonamides in 5M KOH at 70 °C for about 1 hr. The precipitated potassium salts of N-chloroarylsulphonamides were filtered, washed, dried and recrystallised from water. The purity of all the reagents was checked by determining the melting points (Table 1) and by estimating iodometrically the amounts of active chlorine present in them. All other reagents employed in the preparation and purification of reagents were of analytical grade.

2.2. Infrared Absorption Frequency Measurements

Infrared absorption frequency measurements were made on a JASCO-430 (Japan) FT/IR spectrometer. The resolution was set to 2 cm^{-1}, and the scanning range was from 400 to 4000 cm^{-1}. The spectra were measured in the solid state as pressed KBr pellets (13 mm).

2.3. NMR Measurements

The proton and carbon-13 NMR spectra of the compounds were measured on a BRUKER Ac 300F, 300 MHz FT-NMR spectrometer in $CDCl_3$ and DMSO with tetramethylsilane (Me_4Si) as internal standard. The spectral frequency (SF) was kept at 300.134 MHz, sweep width (SW) at 6024.096, pulse width (PW) at 8.0, relaxation delay (RD) of 1.0 (sec), acquisition time (AQ) was 1.360 (sec), receiver gain (RG) 10, decoupling power (DP) was 63L CPD, filter to suppress noise (LB) 0.0, reference value (SR) was set at 4125.36 ppm for H_2O internally. For ^{13}C NMR spectra, the spectral frequency (SF) was kept at 75.469 MHz, sweep width (SW) at 22727.273, pulse width (PW) at 5.0, relaxation delay (RD) of 1.0 (sec), acquisition time (AQ) was 0.360 (sec), receiver gain (RG) 400, decoupling power (DP) was 14H CPD, filter to suppress noise (LB) 6.0, reference value (SR) was set at 701.89 ppm for DMSO at 39.5 ppm externally.

3. Results and Discussion

3.1. Infrared Spectra

The infrared absorption frequencies of the fourteen potassium salts of N-chloroarylsulphonamides are listed in Tables 2 and 3.

Table 3. Infrared absorption frequencies (cm^{-1}) of potassium salts of N-(chloro)-di-substituted benzenesulphonamides.

	i-X, j-Y-$C_6H_3SO_2(K)NCl.xH_2O$, i-X, j-Y =							
Assignment	2,3-$(CH_3)_2$	2,4-$(CH_3)_2$	2,5-$(CH_3)_2$	2-CH_3, 4-Cl	2-CH_3, 5-Cl	3-CH_3, 4-Cl	2,4-Cl_2	3,4-Cl_2
C-H		–	3062.4 w	3106.8 w	3099.1 w	–	3094.2 s	3083.6 w
(Ar sym str)	3020.0 w		3020.0 w					
C-H	2972.7 w	2976.6 w	2981.4 w	2962.1 w	2977.6 w	2959.2 w	–	–
(alk str)	2941.9 w	2940.9 w	2927.4 w	2921.6 w	2929.3 w	2923.6 w		
Combination			1862.9 w	–	–		–	1901.5 w
bands	1772.3 w	1768.4 w	1807.0 w			1773.2 w		1770.3 w
C=C	1634.4 m	1631.5 m	1627.6 m	1627.6 s	1629.6 m	1636.3 m	1636.3 s	1637.3 m
(Ar in-plane	1559.2 w	1588.1 m		1598.7 s	1560.1 w	1569.8 w	1574.6 s	1564.0 w
str)	1491.7 w	1468.5 m	1486.9 m	1498.4 m	1465.6 s	1469.5 m	1457.0 m	1454.1 s
	1449.2 m		1454.1 w	1438.6 w		1444.4 m	1443.5 m	
S=O (asym str)	1386.6 w	1383.7 m	1386.6 w	1400.1 s	1382.7 m	1384.6 s	1369.2 s	1369.5 s
(sym str)	1129.1 s	1133.0 s	1126.2 s	1133.9 s	1149.4 s	1125.3 s	1140.7 s	1149.4 s
C-H (Ar in-plane bend)	1115.6 s	1100.2 s	1064.5 m	1101.2 m	1128.2 s	1090.6 s	1100.2 s	1128.2 s
C-X (str)	–	–	–	1058.7 m	1060.7 s	1039.4 s	1042.3 s	1097.3 s
N-Cl (str)	**944.0 s**	**936.3 s**	**939.2 s**	**933.4 s**	**941.1 s**	**946.9 s**	**944.0 s**	**943.0 s**
S-N (sym str)	879.4 m	879.4 s	906.4 w	877.5 w	900.6 m	886.1 m	867.8 s	887.1 m
C-S (str)	820.6 m	818.6 m	813.8 m	817.7 m	813.8 m	821.5 m	815.7 s	819.6 s
C-H		710.6 m	703.9 m	709.7 m	717.4 m	743.4 w		
(Ar out of-plane bend)	689.4 s	679.8 m	675.0 m	682.7 m	696.2 m	711.6 s	686.5 s	688.5 s
C=C	456.1 w	462.8 m	462.8 w	462.8 m	451.3 w	477.3 w	492.7 s	472.5 w
(Ar out of-plane bend)								

s = strong, m = medium and w = weak.

Table 4. The observed and calculated ^1H chemical shift values (δ, ppm) of potassium salts of N-(chloro)-*p*-substituted benzenesulphonamides ($4\text{-}XC_6H_4SO_2(K)NCl{\cdot}xH_2O$).

	δ (in ppm)				
X	H_o (2,6)		H_m (3,5)		Alkyl H
	Obs.	Calc.	Obs.	Calc.	
H	7.83 d	–	7.56 d	–	–
CH_3	7.65 d	7.73	7.33 d	7.41	2.34 s
C_2H_5	7.65 d	7.73	7.27 d	7.41	2.53 t 1.07 t
F	7.83 m	7.85	7.25 m	7.86	–
Cl	7.72 m	7.83	7.50 m	7.56	–
Br	7.63 d	7.83	7.61 s	7.56	–

s = singlet, d = doublet, t = triplet, and m = multiplet.

Table 5. The observed and calculated ^1H chemical shift values (δ, ppm) of potassium salts of N-(chloro)-di-substituted-benzenesulphonamides (i-X,j-$YC_6H_3SO_2(K)NCl{\cdot}xH_2O$).

	δ (in ppm)									
i-X, j-Y	2-H	3-H		4-H		5-H		6-H		Alkyl H
		Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	
2,3-$(CH_3)_2$	–	–	–	7.28	7.02	7.56	7.36	7.80	7.63	2.62 2.24
2,4-$(CH_3)_2$	–	7.78	7.26	–	–	7.85	7.31	8.35	7.63	3.09 2.83
2-CH_3, 4-Cl	–	7.56	7.41	–	–	7.69	7.46	8.55	7.73	2.82
2,4-Cl_2	–	7.86	7.56	–	–	8.02	7.56	8.39	7.83	–

Table 6. Shifts in the position of benzene protons (δ7.27) caused by the substituents.

Substituent	Ortho	Meta	Para
-CH_3, -R	−0.15	−0.10	−0.10
-COOH, -COOR	+0.80	+0.15	+0.20
-CN	+0.30	+0.30	+0.30
-$CONH_2$	+0.50	+0.20	+0.20
-COR	+0.60	+0.30	+0.30
-SR	+0.10	−0.10	−0.20
-NH_2, -NHR	−0.80	−0.15	−0.40
-$N(CH_3)_2$	−0.50	−0.20	−0.50
-I	+0.30	−0.20	−0.10
-CHO	+0.70	+0.20	+0.40
-Br	0.00	0.00	0.00
-NHCOR	+0.40	−0.20	−0.30
-Cl	0.00	0.00	0.00
-F	+0.30	+0.02	+0.22
-NH_3^+	+0.40	+0.20	+0.20
-OR	−0.20	−0.20	−0.20
-OH	−0.40	−0.40	−0.40
-OCOR	+0.20	−0.10	−0.20
-NO_2	+1.00	+0.30	+0.40
-SO_3H, -SO_2NH_2	+0.40	+0.10	+0.10

The general assignments of the important frequencies to various modes are also indicated in the tables. Assignment of various bands in different compounds has been dealt with, in detail in [26, 27]. The ranges of group absorptions have been assigned based on many compounds in which the groups occur. The precise frequency or wavelength at which a specific group absorbs is dependent on its environment within the molecule and on its physical state.

Comparison of the infrared spectra of the potassium salts of N-chloro-arylsulphonamides with the corresponding arylsulphonamides revealed that the strong absorptions in the range, 947 – 933 cm^{-1} are due to

X	C_1		$C_{2,6}$		$C_{3,5}$		C_4		Alkyl C
	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	
H	141.4	–	126.5	–	130.5	–	133.7	–	–
CH_3	136.2	138.5	126.5	126.4	130.9	131.2	144.8	143.0	22.0
C_2H_5	136.5	138.8	129.2	126.5	132.4	130.0	150.8	149.3	29.6 16.0
F	137.6	136.9	131.2	127.4	117.4	116.2	167.6	168.8	–
Cl	139.5	139.4	130.1	127.5	130.5	130.7	140.0	140.1	–
Br	140.6	140.4	127.6	128.7	133.5	133.9	130.2	128.3	–

δ (in ppm)

Table 7. The observed and calculated ^{13}C chemical shift values (δ, ppm) of potassium salts of N-(chloro)-*p*-substituted-benzenesulphonamides ($4\text{-}XC_6H_4SO_2(K)NCl\cdot xH_2O$).

i-X, *j*-Y	C_1		C_2		C_3		C_4		C_5		C_6		Alkyl C
	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	
2,3-$(CH_3)_2$	139.7	142.0	131.9	136.5	139.7	140.5	130.0	134.3	126.7	127.5	125.4	123.5	20.9
2,4-$(CH_3)_2$	138.7	139.2	133.4	135.7	132.6	131.9	140.7	142.9	127.2	128.3	127.2	126.3	21.0
2-CH_3, 4-Cl	141.5	140.1	133.8	136.8	133.2	131.4	138.8	140.0	129.5	127.8	127.6	127.4	21.6
2,4-Cl_2	138.8	139.6	134.4	133.9	133.3	130.9	139.6	141.1	129.1	128.7	129.1	128.5	–

δ (in ppm)

Table 8. The observed and calculated ^{13}C chemical shift values (δ, ppm) of potassium salts of N-(chloro)-di-substituted-benzenesulphonamides (*i*-X, *j*-$YC_6H_3SO_2(K)NCl\cdot xH_2O$).

N-Cl stretching vibrations. The effect of ring substitution on the N-Cl frequencies is non-uniform. The frequencies in the ranges 1404 – 1370 cm^{-1} and 1149 – 1125 cm^{-1} are, respectively, assigned to S=O asymmetric and symmetric modes of vibrations. This agrees with the assignments of bands in N-substituted-arylsulphonamides [28]. Similarly, IR bands in the ranges 906 – 822 cm^{-1} and 822 – 741 cm^{-1} are assigned to S-N and C-S modes of vibrations, respectively. The other frequencies are assigned to various other vibrations of the ring (Tables 2 and 3). In general, the discussions are similar to other organic aromatic compounds.

The effect of substitution in the phenyl ring in terms of electron-withdrawing and electron-donating groups could not be generalised, as the effect is non-systematic.

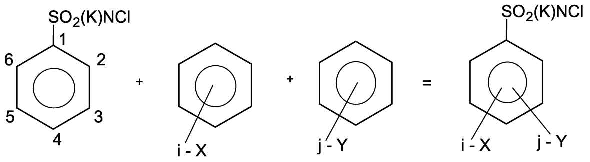
3.2. ^{1}H and ^{13}C NMR Spectra

The ^1H chemical shifts of the potassium salts of mono- and di-substituted N-chloro-arylsulphonamides are given in Tables 4 and 5.

Table 9. Incremental shifts of the aromatic carbon atoms of monosubstituted benzenes (ppm from benzene at 128.5 ppm, + downfield, − upfield).

Substituent	C-1 (Attachment)	C-2	C-3	c-4	C of substituent (ppm from TMS)
H	0.0	0.0	0.0	0.0	–
CH_3	+9.3	+0.7	−0.1	−2.9	21.3
CH_2CH_3	+15.6	−0.5	0.0	−2.6	29.2 (CH_2), 15.8 (CH_3)
$CH(CH_3)_2$	+20.1	−2.0	0.0	−2.5	34.4 (CH), 24.1 (CH_3)
C_6H_5	+12.1	−1.8	−0.1	−1.6	–
OH	+26.6	−12.7	+1.6	−7.3	–
OCH_3	+31.4	−14.4	+1.0	−7.7	54.1
COOH	+2.9	+1.3	+0.4	+4.3	168.0
NH_2	+19.2	−12.4	+1.3	−9.5	–
NO_2	+19.6	−5.3	+0.9	+6.0	–
F	+35.1	−14.3	+0.9	−4.5	–
Cl	+6.4	+0.2	+1.0	−2.0	–
Br	−5.4	+3.4	+2.2	−1.0	–
I	−32.2	+9.9	+2.6	−7.3	–
SO_2NH_2	+ 15.3	−2.9	+0.4	+3.3	–

Since the chemical shift depends on the electron density around the nucleus or associated with the atom to which it is bonded, empirical correlations relating the chemical shifts to the structures have been considered. Thus the chemical shifts of aromatic protons in all the N-chloroarylsulphonamides have been calculated by adding substituent contributions [26, 27] (Table 6) to the shift of benzene (7.27 ppm), as per the principle of substituent addition.



The incremental shifts of the aromatic protons (ppm from that of benzene proton value of 7.27) for different substituents are given in Table 6 and are used in the calculations. The values of shifts in aromatic protons due to -$SO_2(K)NCl\cdot xH_2O$ were calculated comparing the values of potassium salt of N-chlorobenzenesulphonamide (7.826, 7.564) with that of the benzene proton value of 7.27 ppm. The values are $H_o(2,6) = +0.556$ and $H_m(3,4,5) = +0.294$.

The calculated chemical shifts for different protons compared with the experimental values are listed in Tables 4 and 5. Considering the approximation made, the agreement between the calculated and experimental chemical shifts is reasonably good.

^{13}C chemical shifts are tabulated in Tables 7 and 8. The chemical shifts of aromatic carbons in all the N-chloroarylsulphonamides have also been calculated by adding the substituent contributions (Table 9) to the shift of benzene (128.5 ppm), similar to the procedure employed for aromatic protons. The shifts in aromatic carbons due to -$SO_2(K)NCl\cdot H_2O$ were calculated comparing the values of potassium salts of N-chloroarylsulphonamide with that of benzene carbon-13 of 128.5 ppm [26, 29]. The values are ^{13}C-1 $= +12.89$, ^{13}C-4 $= +5.23$, ^{13}C-3 or 5 $= +2.03$ and ^{13}C-2 or 6 $= -1.99$.

The calculated chemical shifts for different aromatic carbons compared with the experimental values are listed in Tables 7 and 8. The agreement between the calculated and experimental chemical shifts is reasonably good.

The ^{1}H and ^{13}C chemical shifts are also compared with those of the corresponding arylsulphonamides and sodium salts of N-chloroarylsulphonamides. The effect of substitution in the phenyl ring in terms of electron-withdrawing and electron-donating groups could not be generalised as the effect is non-systematic.

Acknowledgement

The Authors are grateful to the Nuclear Power Corporation of India Limited, Government of India, Mumbai for a Research Grant and thank the Sophisticated Analytical Instrumentation Facility, Panjab University, Chandigarh-160014, India, for the NMR measurements.

[1] The Chemistry of Amides, ed., J. Jabicky; Interscience, London 1970.

[2] S. R. Sandler and W. Karo, Organic Functional Group Preparations; Academic Press, London 1972, Vol. 3.

[3] M. M. Campbell and G. Johnson, Chem. Rev. **78**, 65 (1978).

[4] B. T. Gowda and D. S. Mahadevappa, Talanta **30**, 359 (1983).

[5] B. T. Gowda and D. S. Mahadevappa, J. Chem. Soc., Perkin Trans. **2**, 323 (1983).

[6] B. T. Gowda, B. S. Sherigara, and D. S. Mahadevappa, Microchem. J. **34**, 103 (1986).

[7] B. T. Gowda and J. I. Bhat, Tetrahedron **43**, 2119 (1987).

[8] B. T. Gowda and R. V. Rao, J. Chem. Soc. Perkin Trans. **2**, 355 (1988).

[9] B. T. Gowda and B. S. Sherigara, Int. J. Chem. Kinet. **21**, 31 (1989).

[10] B. T. Gowda and P. Ramachandra, J. Chem. Soc., Perkin Trans. **2**, 1067 (1989).

[11] B. T. Gowda, J. D. D’Souza, and K. R. Bhat, J. Indian Chem. Soc. **78**, 412 (2002).

[12] B. T. Gowda and B. H. A. Kumar, Oxidn. Commun. **26**, 403 (2003).

[13] B. T. Gowda and Al. Weiss, Z. Naturforsch. **49a**, 695 (1994).

[14] B. T. Gowda, S. Dou, and Al. Weiss, Z. Naturforsch. **51a**, 627 (1996).

[15] B. T. Gowda, D. K. Bhat, H. Fuess, and Al. Weiss, Z. Naturforsch. **54a**, 261, 679 (1999).

[16] B. T. Gowda, H. Paulus, and H. Fuess, Z. Naturforsch. **55a**, 711, 791 (2000); **56**, 386 (2001).

[17] B. T. Gowda, B. H. A. Kumar, and H. Fuess, Z. Naturforsch. **55a**, 721 (2000).

[18] S. Wrobel, B. T. Gowda, and W. Haase, J. Chem. Phys. **106**, 5904 (1996).

[19] B. T. Gowda, I. Svoboda, and H. Fuess, Z. Naturforsch. **55a**, 779 (2000).

[20] S. Dou, H. Fuess, Al. Weiss, B. T. Gowda, and V. G. Krishnan, Z. Krist. **212**, 532 (1997).

[21] B. T. Gowda, K. Jyothi, and J. D. D’Souza, Z. Naturforsch. **57a**, 967 (2002).

[22] B. T. Gowda, J. D. D’Souza, and B. H. A. Kumar, Z. Naturforsch. **58a**, 51 (2003).

[23] B. T. Gowda, K. Jyothi, and N. Damodara, Z. Naturforsch. **58a**, 563 (2003).

[24] A. I. Vogel, Quantitative Organic Analysis; Longman, London 1958.

[25] R. B. Baxter and F. D. Chattway, J. Chem. Soc. **107**, 1814 (1915).

[26] R. M. Silverstein, G. C. Bassler, and T. C. Morrill, Spectrometric Identification of Organic Compounds; John Wiley and Sons, New York 1991.

[27] W. Kemp, Organic Spectroscopy; MacMillan, London 1996.

[28] M. Goldstein, M. A. Russell, and H. A. Willis, Spectrochim Acta **25A**, 1275 (1969).

[29] D. E. Ewing, Org. Magn. Reson. **12**, 499 (1979).