^{35}Cl NQR Spectra of N-(substitutedphenyl)-2,2-dichloroacetamides and Correlation of ^{35}Cl NQR γCl(ω) of Substituted N-Phenyl-Chloroacetamides $X_yC_6H_{5-y}NHCOR$ (X = Cl or CH_3, y = 1 or 2, R = CH_2Cl, $CHCl_2$ or CCl_3)

B. Thimme Gowda, B. H. Arun Kumar, and Hartmut Fuess[a]

Department of Studies in Chemistry, Mangalore University,
Mangalagangothri 574199, Mangalore, India
[a] Institute of Materials Science, Darmstadt University of Technology,
Petersenstrasse 23, D-64287 Darmstadt

Reprint requests to Prof. B. T. G.; Fax: 0091 824 742367; e-mail: gowdabt@yahoo.com

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To study the effect of electron donating or repelling group substitution in the phenyl ring on the γ(^{35}Cl NQR of Cl(ω)) of the dichloroacetyl group, several N-(methylsubstituted-phenyl)-2,2-dichloroacetamides have been synthesised, characterised and their ^{35}Cl NQR frequencies measured at 77 K. All the substituted amides, except N-(2,5-dimethylphenyl)-2,2-dichloro-acetamide, show two ω-C-Cl frequencies in the range of 37.009 - 38.014 MHz. N-(2,5-dimethylphenyl)-2,2-dichloroacetamide shows one ω-C-Cl NQR frequency at 37.50 MHz for the two chlorine atoms present. The two atoms may be crystallographically equivalent. The frequencies of all the methyl-substituted dichloroacetamides have been compared and correlated alongwith the corresponding chloro substituted-phenyl dichloroacetamides. The γ(^{35}Cl NQR) of Cl(ω) of all the N-(substituted-phenyl)-2,2-dichloroacetamides have been correlated with the NQR substituent parameters (κ_i), assuming additivity of the substituent effects. The frequencies are also correlated with Hammett σ. The effect of ring substitution on the average ^{35}Cl NQR Cl(ω) frequencies of the dichloroacetyl group is not substantial, but it affects the crystal structures of the substituted compounds. Using the κ_i values for various groups and ω-C-Cl NQR frequencies of N-(phenyl)-2,2-dichloroacetamide (37.195 and 37.596 MHz), γ(^{35}Cl NQR) of all the N-(methyl and chlorosubstitutedphenyl)-2,2-dichloroacetamides have been estimated. Similar calculations are extended to all the N-(methyl and chlorosubstitutedphenyl)-2-chloroacetamides and -2,2,2-trichloroacetamides. There is a reasonably good agreement between the computed and the experimental values for all the three groups of compounds. Further, γ(^{35}Cl NQR of Cl(ω)) of all the substituted N-phenyl-chloroacetamides represented by the general formula $X_yC_6H_{5-y}NHCOR$ (where X = Cl, or CH_3, y = 1 or 2 and R = CH_2Cl, $CHCl_2$ or CCl_3) are compared. The γ(^{35}Cl NQR of Cl(ω)) of the substituted N-(phenyl)-2,2-dichloroacetamides lie between the frequencies of the corresponding substituted N-(phenyl)-2-chloroacetamides and substituted N-(phenyl)-2,2,2-trichloroacetamides.

Key words: Nuclear Quadrupole Resonance; Aryl Dichloroacetamides.

The amide moiety is an important constituent of many biologically significant compounds [1 - 5]. Conjugation between nitrogen lone pair electrons and the carbonyl π-bond in it results in distinctive physical and chemical properties [1]. Therefore amides are of fundamental chemical interest and are central to future development in such areas as polypeptide and protein chemistry [2]. Many acetanilides exhibit fungicidal, herbicidal and pharmacological activities, which has further stimulated interest in their chemistry [3 - 5].

The nuclear quadrupole resonance (NQR) spectrum of a particular compound depends strongly on the chemical bonding of the atom in that compound [6 - 16]. The direct application of NQR studies is to provide evidences relating to the nature of the chemical bonding in the molecules or crystals. It finds numerous applications in the field of solid state chem-

Table 1. Elemental analysis of the N-(substitutedphenyl)-2,2-dichloroacetamides studied, $X_yC_6H_{5-y}NHCOCHCl_2$ (where X = H, CH_3, or Cl and y = 1, 2, or 3).

X_y	% Nitrogen Found	% Nitrogen Calcd.	% Carbon Found	% Carbon Calcd.	% Hydrogen Found	% Hydrogen Calcd.	m.p. (°C)
-H	6.94	6.86	47.11	47.09	3.43	3.46	116
2-CH_3	6.41	6.42	49.27	49.57	3.98	4.16	130
3-CH_3	6.40	6.42	49.55	49.57	3.94	4.16	99
4-CH_3	6.35	6.42	49.67	49.57	4.01	4.16	152
2,3-$(CH_3)_2$	6.03	6.03	51.75	51.75	4.78	4.78	166
2.4-$(CH_3)_2$	6.02	6.03	51.03	51.75	4.78	4.78	157
2,5-$(CH_3)_2$	5.90	6.03	49.93	51.75	4.87	4.78	150
2,6-$(CH_3)_2$	5.91	6.03	51.35	51.75	4.72	4.78	158
3,4-$(CH_3)_2$	5.98	6.03	51.55	51.75	4.68	4.78	168
3, 5-$(CH_3)_2$	6.00	6.03	51.32	51.75	4.65	4.78	128
2,3-Cl_2	5.11	5.08	34.96	35.04	1.67	1.99	136
2,4-Cl_2	4.95	5.08	35.22	35.04	1.72	1.99	131
2,5-Cl_2	4.95	5.08	35.14	35.04	1.72	1.99	146
2,6-Cl_2	5.09	5.08	35.39	35.04	1.92	1.99	172
3,4-Cl_2	5.03	5.08	35.82	35.04	2.11	1.99	142
3,5-Cl_2	5.23	5.08	35.05	35.04	2.04	1.99	139
2,4,6-Cl_3	4.58	3.68	21.21	25.41	1.47	1.05	187

istry and physics, biopolymers, crystal studies, medical, pharmacological chemistry and in the field of materials science. Thus NQR plays an important role in solving chemical and physical problems in industrial and academic research. A great deal of work on the spectroscopic aspects of a variety of compounds needs to be done for correlating frequencies of these compounds with their chemical bond parameters.

Pies et al. [9] have studied the ^{35}Cl NQR spectra of a number of N-(chlorophenyl)-2,2-dichloroacetamides. But there are no reports on the effects of electron donating or repelling group substitutions in the phenyl ring on the ^{35}Cl NQR Cl(ω) frequencies of the dichloroacetyl group, as chlorine is electron withdrawing. We have recently reported the effects of electron donating or repelling group substitutions in the phenyl ring on the ^{35}Cl NQR Cl(ω) frequencies of the 2-chloroacetyl [16] and 2,2,2-trichloroacetyl groups in the arylamides [15]. To complete our work aimed at correlating ^{35}Cl NQR frequencies of the substituted N-phenylchloroacetamides represented by the general formula $X_yC_6H_{5-y}NHCOR$ (where X = Cl or CH_3, y = 1 or 2 and R = CH_2Cl, $CHCl_2$ or CCl_3) with their chemical bond parameters, we have studied ^{35}Cl NQR spectra of several N-(methylsubstitued phenyl)-2,2-dichloroacetamides and hence intercorrelated the frequencies of all the substituted N-phenyl-2-chloro, 2,2-dichloro and 2,2,2-trichloro-acetamides and with their chemical bond parameters.

Materials and Methods

The substitutedphenyl dichloroacetamides were prepared from substituted anilines, dichloroacetic acid and phosphoryl chloride by a procedure similar to the ones described in [9, 15 - 17]. The commercial anilines (Sisco Research Laboratories, India) were purified by either double distillation or zone refining. Pure samples of the respective anilines (2-methyl aniline, 3-methyl aniline, 4-methyl aniline, 2,3-dimethyl aniline, 2,4-dimethyl aniline, 2,5-dimethyl aniline, 2,6-dimethyl aniline, 3,4-dimethyl aniline, 3,5-dimethyl aniline, 2,4,6-trimethyl aniline; 2-chloro aniline, 3-chloro aniline, 4-chloro aniline, 2,3-dichloro aniline, 2,4-dichloro aniline, 2,5-dichloro aniline, 2,6-dichloro aniline, 3,4-dichloro aniline, 3,5-dichloro aniline, and 2,4,6-trichloro aniline) were treated with mixtures of dichloroacetic acid (Aldrich, Germany) and phosphoryl chloride under constant stirring. The resulting mixtures were slowly warmed to expel HCl. Excess of phosphoryl chloride was hydrolysed by adding cold water dropwise under ice cold conditions. Produced HCl was removed by treating with 2M NaOH. The separated solids were filtered under suction, washed thoroughly with water and dried. The substitutedphenyl dichloroacetamides thus prepared were recrystallised from ethanol several times.

The purity of the compounds was checked by elemental analysis (C, H, and N) and by determining their melting points (Table 1). They were further characterised by recording their infrared spectra. All other reagents employed in the preparations and purification of reagents were of analytical grade.

^{35}Cl NQR Frequency Measurements

Polycrystalline samples of the title compounds were employed. The ^{35}Cl NQR of the N-(substituted phenyl)-2,2-dichloroacetamides were measured at 77 K. The spectra were registered by the continuous wave method with a superregenerative spectrometer. The temperature at the sample site was produced by a stream of temperature and flow regulated nitrogen gas or with a liquid nitrogen bath at 77 K. The temperatures at the sample site were measured by copper-constantan thermocouples to ± 1 K. The resonance frequencies were measured via a frequency counter to an accuracy of 5 kHz. The latter accuracy was determined by the line

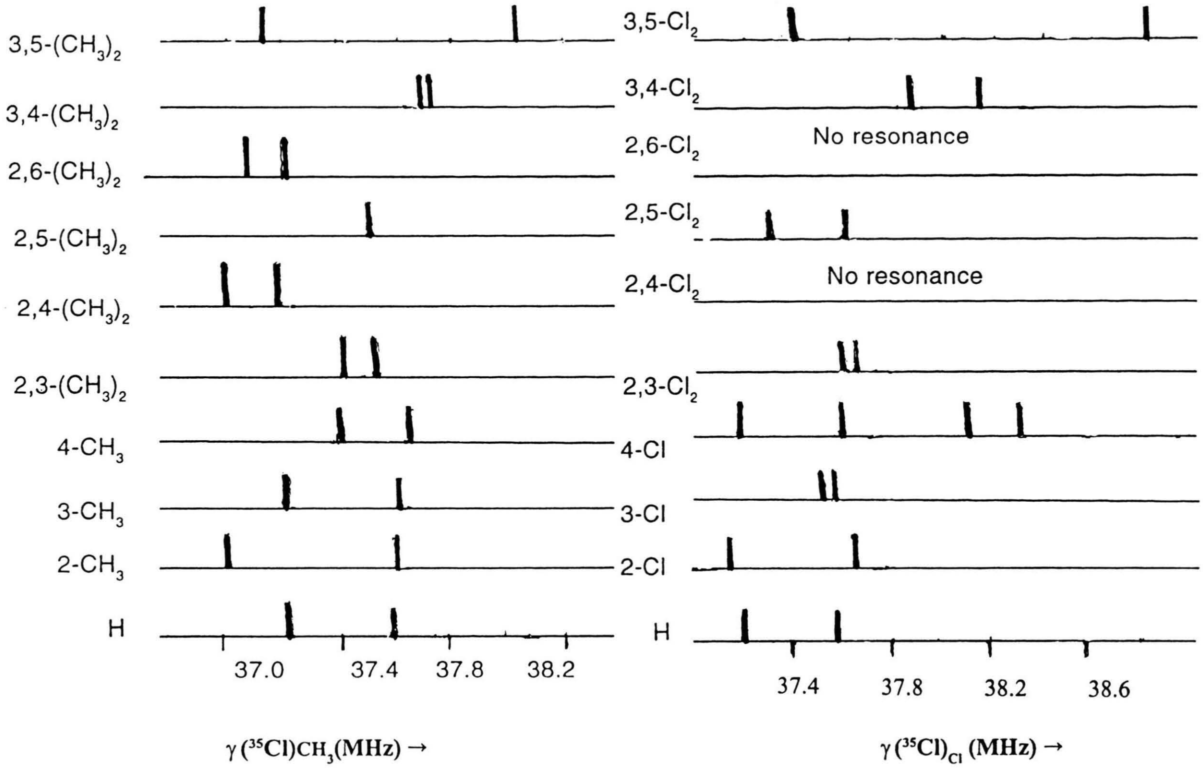

Fig. 1. Plot of $\gamma(^{35}\text{Cl NQR})_{\text{CH}_3 \text{ or Cl}}$ (MHz) versus substitution.

Table 2. ω C-Cl ^{35}Cl NQR frequencies of N-(methylphenyl)-2,2-dichloroacetamides, $X_yC_6H_{5-y}NHCOCHCl_2$ (Temp. 77 K, Assignment: ω).

X_y	γ (MHz)	S / N
-H	37.195, 37.596	25 to 30, 20 to 25
2-CH_3	37.009, 37.607	10 to 15, 15
3-CH_3	37.171, 37.623	10 to 15, 10 to 15
4-CH_3	37.385, 37.646	20, 5 to 10
2,3-$(CH_3)_2$	37.422, 37.509	15 to 20, 15 to 20
2.4-$(CH_3)_2$	37.063, 37.163	5 to 10, 5 to 10
2,5-$(CH_3)_2$	37.500	10 to 15
2,6-$(CH_3)_2$	37.082, 37.196	5 to 10, 5 to 10
3,4-$(CH_3)_2$	37.724, 37.771	20 to 25, 20 to 25
3,5-$(CH_3)_2$	37.121, 38.014	25 to 30, 20 to 25

width of the resonances, which was between 10 and 20 kHz.

The parent compound N-(phenyl)-2,2-dichloroacetamide was also prepared, characterised and its ^{35}Cl NQR frequencies were measured under identical conditions for comparison purpose. In fact all the corresponding N-(chlorophenyl)-2,2-dichloroacetamides were prepared, characterised and their ^{35}Cl NQR frequencies measured for comparison purposes.

Results and Discussion

The ^{35}Cl (ω) NQR frequencies of the parent and nine N-(methylsubstituted phenyl)-2,2-dichloroacetamides are shown in Table 2. There was no problem in assigning the frequencies, as there are only C-Cl ^{35}Cl (ω) NQR frequencies in all the N-(methylsubstituted phenyl)-2,2-dichloroacetamides. All the substituted amides, except N-(2,5-dimethylphenyl)-2,2-dichloroacetamide, show two ω-C-Cl frequencies in the range of 37.009 - 38.014 MHz. N-(2,5-dimethylphenyl)-2,2-dichloroacetamide showed one ω-C-Cl NQR frequency at 37.50 MHz for the two chlorine atoms present in it. The two atoms may be crystallographically equivalent.

^{35}Cl (ω) NQR spectra of all the N-(methylsubstituted phenyl)-2,2-dichloroacetamides have been compared with the corresponding chlorophenyl dichloroacetamides. The comparisons are schematic-

Table 3. NQR substituent parameters (κ) and Hammett σ values.

Group	κ	σ	Group	κ	σ	Group	κ	σ
o-CH_3	-0.392 ± 0.054	–	m-CH_3	-0.207 ± 0.161	-0.069	p-CH_3	-0.004 ± 0.126	-0.170
o-OCH_3	0.917 ± 0.172	–	p-OCH_3	0.122 ± 0.145	-0.268	o-OC_2H_5	0.793 ± 0.172	–
p-OC_2H_5	-0.027 ± 0.157	-0.250	o-OH	0.442 ± 0.101	–	m-OH	0.140 ± 0.140	-0.002 ± 0.106
p-OH	0.286 ± 0.143	-0.357 ± 0.104	o-COOH	1.704 ± 0.162	–	m-COOH	0.377 ± 0.153	0.355
p-COOH	0.409 ± 0.148	0.265 ± 0.126	o-Cl	1.206 ± 0.047	–	m-Cl	0.499 ± 0.035	0.373
p-Cl	0.329 ± 0.072	0.227	o-Br	0.975 ± 0.204	–	p-Br	0.312 ± 0.173	0.232
o-I	0.917 ± 0.196	–	p-I	0.199 ± 0.173	0.276	m-CF_3	0.611 ± 0.147	0.415
p-CF_3	0.740 ± 0.207	0.551	o-NH_2	-0.534 ± 0.119	–	m-NH_2	-0.103 ± 0.130	-0.161
p-NH_2	-0.119 ± 0.133	-0.660	o-NO_2	2.096 ± 0.087	–	m-NO_2	1.069 ± 0.093	0.710
p-NO_2	0.067 ± 0.125	0.778 / 1.270						

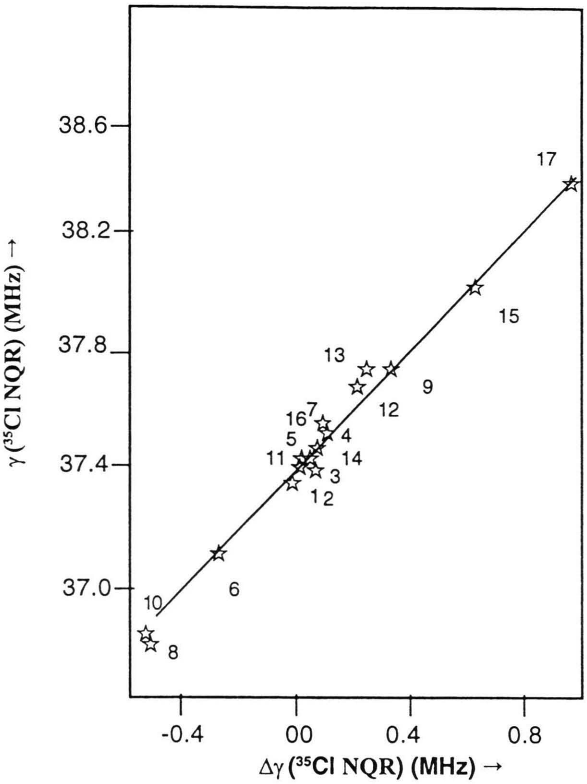

Fig. 2. Plot of $\gamma(^{35}Cl\ NQR)_{CH_3\ or\ Cl}$ (MHz) versus $\delta\gamma(^{35}Cl\ NQR)_{CH_3\ or\ Cl}$ (MHz). 1: H; 2: 2-CH_3; 3: 3-CH_3; 4: 4-CH_3; 5: 2,3-$(CH_3)_2$; 6: 2,4-$(CH_3)_2$; 7: 2,5-$(CH_3)_2$; 8: 2,6-$(CH_3)_2$; 9: 3,4-$(CH_3)_2$; 10: 3,5-$(CH_3)_2$; 11: 2-Cl; 12: 3-Cl; 13: 4-Cl; 14: 2,3-Cl_2; 15: 2,5-Cl_2; 16: 3,4-Cl_2; 17: 3,5-Cl_2.

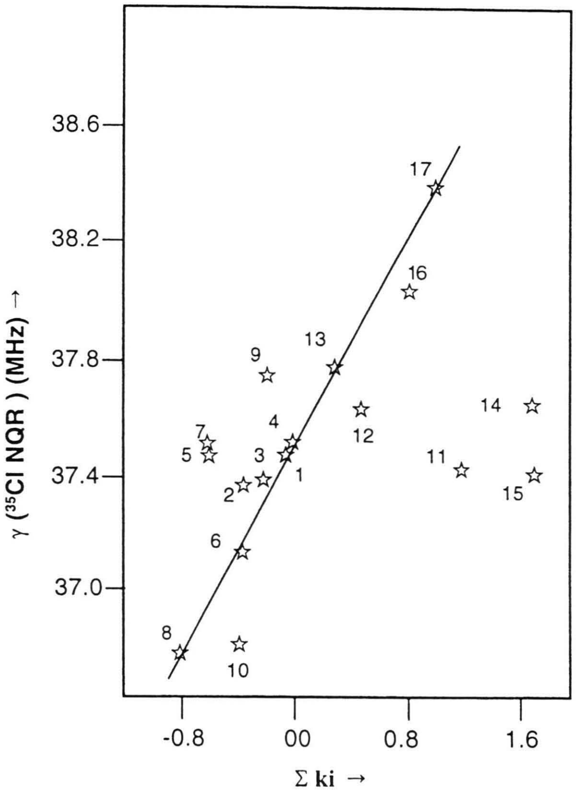

Fig. 3. Plot of $\gamma(^{35}Cl\ NQR)_{CH_3\ or\ Cl}$ (MHz) vs. Σk_i. 1: H; 2: 2-CH_3; 3: 3-CH_3; 4: 4-CH_3; 5: 2,3-$(CH_3)_2$; 6: 2,4-$(CH_3)_2$; 7: 2,5-$(CH_3)_2$; 8: 2,6-$(CH_3)_2$; 9: 3,4-$(CH_3)_2$; 10: 3,5-$(CH_3)_2$; 11: 2-Cl; 12: 3-Cl; 13: 4-Cl; 14: 2,3-Cl_2; 15: 2,5-Cl_2; 16: 3,4-Cl_2; 17: 3,5-Cl_2.

ally represented through line diagrams in Figure 1. Variations of the mean values of $\gamma(^{35}Cl\ (\omega)$ NQR) of the dichloroacetyl group with both the methyl and and chloro substituents in the phenyl ring are correlated with the difference between the frequencies ($\Delta\gamma$) of the substituted dichloroacetamide and the parent, N-(phenyl)-2,2-dichloroacetamide (Figure 2). The correlation is exceedingly good, although there was no systematic variation of the frequencies with the substituents in the phenyl ring. Generally the electron withdrawing groups in the ring increased the $\gamma(^{35}Cl$ NQR) of ω-C-Cl.

Table 4. Comparison of computed and experimental ω C-Cl ^{35}Cl NQR frequencies of N-(methylphenyl)-chloroacetamides, $X_yC_6H_{5-y}NHCOR$ ($R = CH_2Cl$, $CHCl_2$ or CCl_3).

	CH_2Cl, γ (MHz)		$CHCl_2$, γ (MHz)		CCl_3, γ (MHz)	
X_y	Found	Calcd.	Found	Calcd.	Found	Calcd.
-H	35.333	–	37.195	–	39.986	–
			37.596		39.452	
					39.428	
			37.396 (av)		39.622 (av)	
2-CH_3	36.026	34.941	37.009	36.803	39.242	39.230
			37.607	37.204		
			37.308 (av)	37.003 (av)		
3-CH_3	36.590		37.171	36.988	40.131	39.779
	35.760		37.623	37.389	39.453	39.245
					39.243	39.221
	36.178 (av)	35.126	37.397 (av)	37.189 (av)	39.609 (av)	39.415 (av)
4-CH_3	35.150	35.290	37.385	37.191	6 frequencies	39.990
			37.646	37.592	in the range	39.456
					39.023 to 40.127	39.432
			37.516 (av)	37.392 (av)	39.603 (av)	39.626 (av)
2,3-$(CH_3)_2$			37.422	36.592	6 frequencies	39.387
	No resonance		37.509	36.997	in the range	38.853
					38.968 to 40.254	38.829
			37.466 (av)	36.797 (av)	39.465 (av)	39.023 (av)
2,4-$(CH_3)_2$	35.835	34.937	37.063	36.596		
			37.163	36.997	No resonance	
			37.113 (av)	36.797 (av)		
2,5-$(CH_3)_2$			37.500	36.596	39.582	39.387
	No resonance		–	36.997	39.087	38.853
						38.829
			37.500	36.797 (av)	39.335 (av)	39.023 (av)
2,6-$(CH_3)_2$	35.723	34.549	37.082	36.799	39.713	39.202
			37.196	36.372	39.523	38.668
					39.072	38.644
			37.139 (av)	36.586 (av)	39.436 (av)	38.838 (av)
3,4-$(CH_3)_2$	-	-	37.724	36.596	-	-
			37.771	36.997		
			37.748 (av)	36.797 (av)		
3,5-$(CH_3)_2$	-	-	37.121	36.781	-	-
			38.014	37.182		
			37.568 (av)	36.981 (av)		

Biedenkapp and Weiss [8] have deduced NQR substituent parameters (κ_i) for various groups (Table 3) solely from $\gamma(^{35}$Cl NQR). Using these κ_i values listed in Table 3 and the ω-C-Cl NQR frequency of N-(phenyl)-2,2-dichloroacetamide frequencies (37.195 and 37.596 MHz), the ^{35}Cl NQR frequencies of all the N-(methyl and chlorosubstituted phenyl)-2,2-dichloroacetamides have been estimated. Similar calculations were extended to all the N-(methyl and chlorosubstitutedphenyl)-2-chloroacetamides and N-(methyl and chlorosubstituted phenyl)-2,2,2-trichloroacetamides. The computed ^{35}Cl NQR frequencies of all the three groups of compounds, along with the experimental frequencies are shown in Tables 4 and 5. As may be seen, there is a reasonably good agreement between the computed and the experimental values.

The $\gamma(^{35}$Cl NQR) values of Cl(ω) of all the N-(substituted phenyl)-2,2-dichloroacetamides have been correlated with κ_i (Fig. 3), assuming additivity of the substituent effects. In the light of the fact that the effect of substitution is not immediately next to the ω-C-Cl bond and it has to be transmitted through the peptide linkage -NHCO-, the deviations are understandable. The deviation is also not systematic. This is

Table 5. Comparison of computed and experimental ω C-Cl ^{35}Cl NQR frequencies of N-(chlorophenyl)-chloroacetamides, $X_yC_6H_{5-y}NHCOR$ (R = CH_2Cl, $CHCl_2$ or CCl_3).

X_y	CH_2Cl, γ (MHz) Found	Calcd.	$CHCl_2$, γ (MHz) Found	Calcd.	CCl_3, γ (MHz) Found	Calcd.
-H	35.333	–	37.195	–	39.986	–
			37.596		39.452	
					39.428	
			37.396 (av)		39.622 (av)	
2-Cl	36.278	36.539	37.682	38.401	40.239	41.192
			37.143	38.802	39.908	40.658
					39.225	40.634
			37.413 (av)	38.602 (av)	39.791 (av)	40.828 (av)
3-Cl	No resonance		37.675	37.694	39.604	40.485
			37.546	38.095	39.586	39.951
					39.521	39.927
			37.612 (av)	37.895 (av)	39.570 (av)	40.121 (av)
4-Cl	35.745	35.662	38.280	37.524	39.902	40.315
			38.089	37.925	39.562	39.791
			37.166, 37.610		39.392	39.757
			37.786 (av)	37.725 (av)	39.619 (av)	39.951 (av)
2,3-Cl_2	36.725	37.038	37.666	38.900	40.049	41.691
			37.636	39.301	39.649	41.157
			37.651 (av)	39.101 (av)	38.686	41.133
					39.461 (av)	41.327 (av)
2,4-Cl_2	No resonance		No resonance		39.983	41.521
					39.931	40.987
					39.488	40.963
					39.801 (av)	41.160 (av)
2,5-Cl_2	35.584	37.038	37.544	38.900	6 frequencies	41.691
			37.284	39.301	in the range	41.157
			37.414 (av)	39.101 (av)	38.680 to 40.470	41.133
					39.690 (av)	41.327 (av)
2,6-Cl_2	36.674	37.745	No resonance		39.732	42.398
					39.671	41.864
					39.322	41.840
					39.575 (av)	42.034 (av)
3,4-Cl_2	36.589	37.038	38.156	38.694	39.856	40.814
			37.856	38.424	39.556	40.280
			38.006 (av)	38.559 (av)	39.420	40.256
					39.611 (av)	40.450 (av)
3,5-Cl_2	No resonance		38.701	38.193	39.817	40.984
			37.441	38.594	39.550	40.450
			38.071 (av)	38.394 (av)	39.479	40.426
					39.615 (av)	40.620 (av)

due to the fact that the chemically equivalent chlorine atoms may exhibit different NQR frequencies due to crystal field effect [18, 19]. Further, the introduction of a steric parameter adds to the deviation due to steric inhibition of mesomerism [20]. Intramolecular hydrogen bonding may also partly contribute. The ^{35}Cl NQR frequencies of all the substituted phenyl 2,2-dichloroacetamides are also correlated with Hammett σ (Figure 4).

Finally, γ(^{35}Cl NQR) of all the N-(substituted phenyl)-mono-, di- and tri-chloroacetamides has been correlated through line diagrams (Figure 5). The ^{35}Cl NQR frequencies of phenyl ring substituted 2,2-dichloroacetamides lie between the frequencies of the corresponding phenyl ring substituted monochloroacetamides and 2,2,2-trichloroacetamides, but there is no regular trend, probably because of crystal field effects.

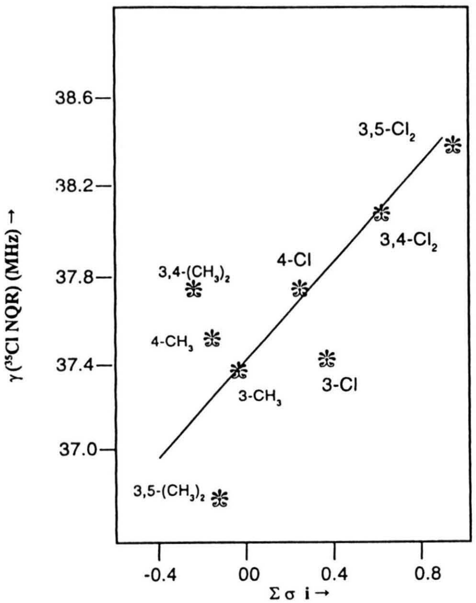

Fig. 4. Plot of $\gamma(^{35}\mathrm{Cl\ NQR})_{\mathrm{CH_3\ or\ Cl}}$ (MHz) vs. $\Sigma\sigma_i$.

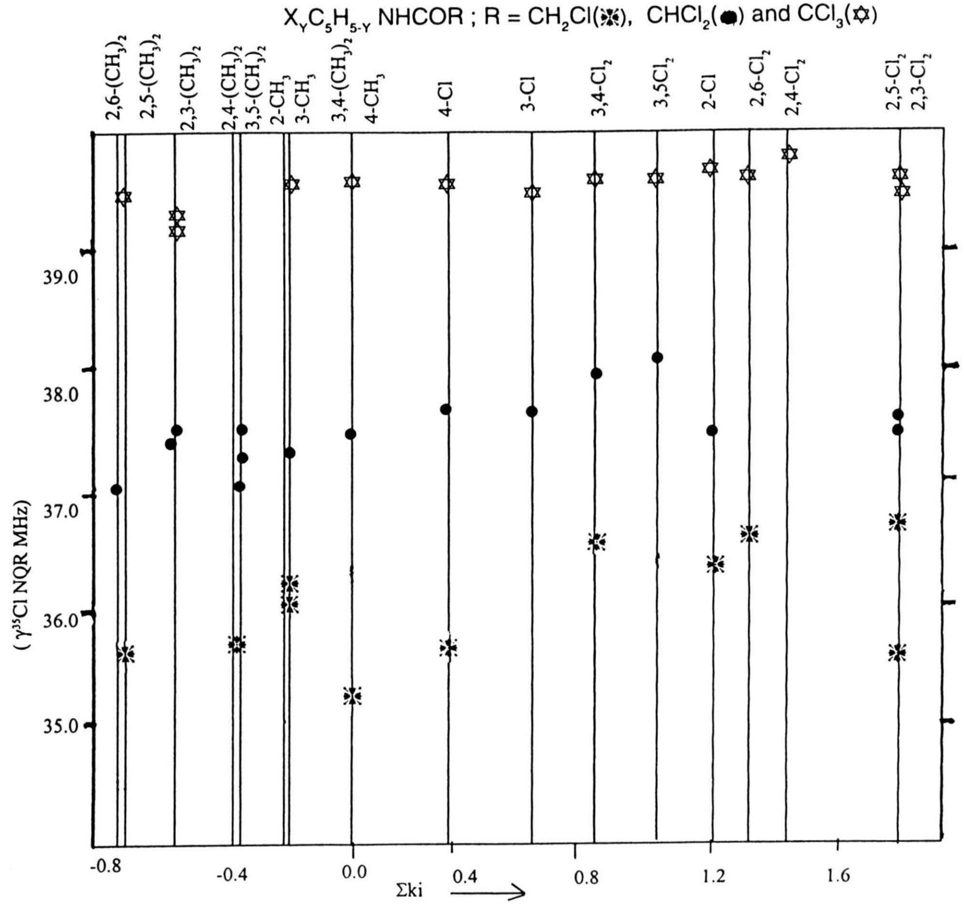

Fig. 5. Variation of $\gamma(^{35}\mathrm{Cl\ NQR})_{\mathrm{CH}_{3-y}\mathrm{Cl}_y\,(y=1\ \mathrm{to}\ 3)}$ with Σk_i.

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