

# MAGNETISM AND MOLECULAR STRUCTURE OF SULPHUR COMPOUNDS

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IN previous communications<sup>1</sup> molecular structure of some of the selenium and tellurium compounds have been discussed from the magnetic standpoint, using Langevin equation and Slater's and Angus's methods for the calculation of  $\Sigma\bar{r}^2$ . Sulphur belongs to the same group of elements as selenium and tellurium and, like them, presents an interesting study on account of its variable valency. The study of the magnetic properties of sulphur compounds was initiated by Pascal<sup>2</sup> and was followed by Farquharson<sup>3</sup> who determined the susceptibilities of a series of these compounds with a view to test some of the modern theories of magnetism.

Recently Varadachari and Subramaniam<sup>4</sup> have measured and theoretically calculated the molecular susceptibilities of a number of sulphur compounds and they find that in some cases there is a wide divergence in their experimental and calculated values. Perhaps this is due to the fact that they used Kido's values for the constitutive constants. Nevgi<sup>5</sup> has also measured the magnetic susceptibilities of some of the sulphur compounds and has arrived at the conclusion that Pascal's susceptibility constants are in good agreement for sulphur in various valency states.

The present paper deals with the calculations of (i) the values of  $\Sigma\bar{r}^2$  according to Slater's and Angus's methods as well as with the slight changes mentioned in the previous papers (*loc. cit.*) and hence (ii) the ionic susceptibilities for sulphur in various valency states. These values, given in Table I, have been made use of in calculating theoretically the magnetic susceptibilities of several compounds of sulphur for which the experimental values are known. The symbols used in this paper are the same as given in the previous publications (*loc. cit.*).

TABLE I

*Diamagnetic Susceptibilities of S in Various Valency States Calculated from Various Methods*

Valency states	$\Sigma r^{-3}$ Calculated by Slater's method	$\chi \times 10^6$ Calculated by Slater's method	$\Sigma r^{-3}$ Calculated by modification in Slater's method	$\chi \times 10^6$ Calculated by modification in Slater's method using constant .790 instead of 0.807	$\Sigma r^{-3}$ Calculated by Angus's method	$\chi \times 10^6$ Calculated by Angus's method	$\Sigma r^{-3}$ Calculated by modification in Angus's method	$\chi \times 10^6$ Calculated by modification in Angus's method using constant 0.790 instead of 0.807
S divalent	15.05	-12.14	15.05	-11.890	13.65	-11.02	13.65	-10.78
S tetra-valent	7.10	- 5.732	7.10	- 5.61	6.98	- 5.636	6.98	- 5.517
S hexa-valent	1.73	- 1.397	1.73	- 1.367	1.61	- 1.302	1.61	- 1.275

#### Discussion of Results

(a) *Sulphur monochloride* ( $S_2Cl_2$ ).—Like selenium monochloride ( $Se_2Cl_2$ ) this compound also may have two different constitutions—the symmetrical Cl-S-S-Cl and the unsymmetrical  $S = S \begin{matrix} \text{Cl} \\ \diagdown \\ \text{Cl} \end{matrix}$ . Susceptibilities calculated for both these formulæ are given in Table II.

The values calculated by the author for the constitution  $S = S \begin{matrix} \text{Cl} \\ \diagdown \\ \text{Cl} \end{matrix}$  are in better agreement with those experimentally obtained by Varadachari and Subramaniam than those calculated by the author. The experimental values obtained by Nevgi do not agree with any of the calculated values.

The above remarks show that Slater's and Angus's values for ionic susceptibilities are more correct than Kido's values employed by Varadachari and Subramaniam. Further the above results point out that the constitution of the sulphur monochlorides is represented by the Formula II and not I. This structure is also supported by the results of Meyer (*Z. Anorg. Allm. Chem.*, 1931, **146**, 203) who studied the smekel-Raman effect of  $Me_2S_2$  and  $Et_2S_2$  and found that the structural formula of the monochloride is  $S = S \begin{matrix} \text{Cl} \\ \diagdown \\ \text{Cl} \end{matrix}$  (II) and not Cl-S-S-Cl (I). Matossi and Aderhold (*Z. physik*, 1931, **683**, 683) also arrived at the same constitution (II) from the measurement of

TABLE II

Constitution	Cl-S-S-Cl (I)	S = S $\begin{matrix} \diagup \text{Cl} \\ \diagdown \text{Cl} \end{matrix}$ (II)
( $\chi_a$ ) <sub>1</sub>	- 0.5088 × 10 <sup>-6</sup>	- 0.4613 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>1</sub>	- 68.68 × 10 <sup>-6</sup>	- 62.272 × 10 <sup>-6</sup>
( $\chi_a$ ) <sub>2</sub>	- 0.5051 × 10 <sup>-6</sup>	- 0.4585 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>2</sub>	- 68.18 × 10 <sup>-6</sup>	- 61.90 × 10 <sup>-6</sup>
( $\chi_a$ ) <sub>3</sub>	- 0.4922 × 10 <sup>-6</sup>	- 0.4523 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>3</sub>	- 66.44 × 10 <sup>-6</sup>	- 61.05 × 10 <sup>-6</sup>
( $\chi_a$ ) <sub>4</sub>	- 0.4887 × 10 <sup>-6</sup>	- 0.4496 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>4</sub>	- 65.96 × 10 <sup>-6</sup>	- 60.697 × 10 <sup>-6</sup>
( $\chi_a$ ) <sub>0</sub> <sub>1</sub>	..	- 0.461 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>0</sub> <sub>1</sub>	..	- 62.2 × 10 <sup>-6</sup>
( $\chi_a$ ) <sub>0</sub> <sub>2</sub>	..	- 0.4964 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>0</sub> <sub>2</sub>	..	- 67.0 × 10 <sup>-6</sup>
( $\chi_a$ ) <sub>0</sub> <sub>3</sub>	..	- 0.405 × 10 <sup>-6</sup>
( $\chi_m$ ) <sub>0</sub> <sub>3</sub>	..	- 54.7 × 10 <sup>-6</sup>

O<sub>1</sub> Refers to the values observed by Varadachari and Subramaniam (*loc. cit.*).

O<sub>2</sub> Refers to the values calculated by Varadachari and Subramaniam (*loc. cit.*).

O<sub>3</sub> Refers to the values observed by Nevgi (*loc. cit.*).

Raman spectra of various sulphur compounds including the monochloride. This formula is also expected from the analogy of the monochloride with thionyl chloride.

However, Spong (*J.C.S.*, 1934, 485) who investigated the Raman spectrum, parachor and dielectric constant of sulphur monochloride concluded that the monochloride consists of a mixture of the two forms S = S  $\begin{matrix} \diagup \text{Cl} \\ \diagdown \text{Cl} \end{matrix}$  and Cl-S-S-Cl. These results are not supported by the magnetic data.

In the case of selenium monochloride and monobromide (*loc. cit.*) the magnetic method definitely supports the constitution -Se-Se-, in which Se is divalent. Compounds of this type with tellurium are not known. It may therefore, be inferred that the affinity for halides decreases from Te to S in this group of elements and in S the union is of the most feeble kind.

(b) *Sulphur monoiodide* ( $S_2I_2$ ).—The existence of a compound between iodine and sulphur has been the subject of great controversy for a long time. It is only recently that sulphur mono-iodide has been prepared in a pure state (*cf.* Rao, *Proc. Ind. Acad. Sci.*, 1939, **10**, 423 ; Rao, *Ibid.*, 1940, **11**, 162). Susceptibilities calculated according to the plan followed in this paper for the two possible constitutions for the monoiodide along with the values obtained by Varadachari and Subramaniam are given in Table III.

TABLE III

Constitution	I-S-S-I (I)	$S=S \begin{matrix} \diagup I \\ \diagdown I \end{matrix}$ (II)
$(\chi_a)_1$	— $0.3633 \times 10^{-6}$	— $0.3430 \times 10^{-6}$
$(\chi_m)_1$	— $115.48 \times 10^{-6}$	— $109.072 \times 10^{-6}$
$(\chi_a)_2$	— $0.3616 \times 10^{-6}$	— $0.3418 \times 10^{-6}$
$(\chi_m)_2$	— $114.98 \times 10^{-6}$	— $108.7 \times 10^{-6}$
$(\chi_a)_3$	— $0.3560 \times 10^{-6}$	— $0.3393 \times 10^{-6}$
$(\chi_m)_3$	— $113.24 \times 10^{-6}$	— $107.856 \times 10^{-6}$
$(\chi_a)_4$	— $0.3546 \times 10^{-6}$	— $0.3381 \times 10^{-6}$
$(\chi_m)_4$	— $112.76 \times 10^{-6}$	— $107.497 \times 10^{-6}$
$(\chi_a)_{0_1}$		— $0.329 \times 10^{-6}$
$(\chi_m)_{0_1}$		— $104.6 \times 10^{-6}$
$(\chi_a)_{0_2}$		— $0.4064 \times 10^{-6}$
$(\chi_m)_{0_2}$		— $129.2 \times 10^{-6}$

$0_1$  refers to the values observed by Varadachari and Subramaniam.

$0_2$  refers to the values calculated by Varadachari and Subramaniam using Kido's values for

the constitution  $S=S \begin{matrix} \diagup I \\ \diagdown I \end{matrix}$

The agreement between the calculated and the observed results shows that the constitution of the iodide is represented by the formula II which is analogous to that of the monochloride.

Using Kido's values Varadachari and Subramaniam conclude that it is merely a mixture of sulphur and iodine. These authors refer to Mellor (*Inorganic Chemistry*, Vol. X, p. 653) in support of their conclusion but the same literature mentions definitely the formation of this compound (*cf.* also Caven Lander, *Inorganic Chemistry*, p. 281). This conclusion does not also seem to be satisfactory on account of the unreliability of Kido's constants and the isolation of the iodide (*loc. cit.*).

(c) *Sulphur dichloride* ( $SCl_2$ ).—Sulphur dichloride cannot have any other constitution excepting  $S \begin{matrix} \diagup Cl \\ \diagdown Cl \end{matrix}$ . It has been found in the case of  $TeCl_2$  and

$TeBr_2$  that the agreement between the theoretically calculated and the experimental values is not good and the divergence is assigned to the unstable nature of the compounds which makes it difficult to obtain the correct experimental values. Since sulphur dichloride is also an unstable compound, it is of interest to find if any disagreement exists in this case also. The theoretical values calculated according to Slater's and Angus's methods for this constitution are given in Table IV.

TABLE IV

Constitution	$S \begin{matrix} \diagup Cl \\ \diagdown Cl \end{matrix}$
$(X_a)_1$	— $0.5489 \times 10^{-6}$
$(X_m)_1$	— $56.54 \times 10^{-6}$
$(X_a)_2$	— $0.5466 \times 10^{-6}$
$(X_m)_2$	— $56.29 \times 10^{-6}$
$(X_a)_3$	— $0.5381 \times 10^{-6}$
$(X_m)_3$	— $55.42 \times 10^{-6}$
$(X_a)_4$	— $0.5358 \times 10^{-6}$
$(X_m)_4$	— $55.18 \times 10^{-6}$
$(X_a)_{0_1}$	— $0.467 \times 10^{-6}$
$(X_m)_{0_1}$	— $48.1 \times 10^{-6}$
$(X_a)_{0_2}$	— $0.478 \times 10^{-6}$
$(X_m)_{0_2}$	— $49.4 \times 10^{-6}$
$(X_a)_{0_3}$	— $0.478 \times 10^{-6}$
$(X_m)_{0_3}$	— $49.2 \times 10^{-6}$
$(X_a)_{0_4}$	— $0.4796 \times 10^{-6}$
$(X_m)_{0_4}$	— $49.4 \times 10^{-6}$

$0_1$  refers to the experimental values of Varadachari and Subramaniam.

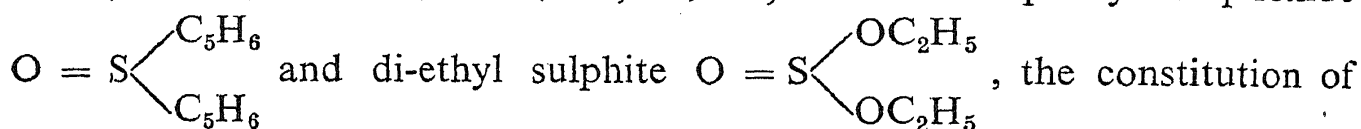
$0_2$  refers to the experimental values of Kido.

$0_3$  refers to the experimental values of Nevgi.

$0_4$  refers to the values calculated by Varadachari and Subramaniam.

The Table above shows that although the experimental values of all the workers agree well with each other the calculated values are rather high. This difference is, as mentioned above, due to the unstable nature of the compound.

(d) *Thionyl chloride* ( $SOCl_2$ ).—Judged by the formation of sulphonides (Stecker, *Ber.*, 1910, **43**, 1131; Grignard and Zorn, *Compt. rend.*, 1910, **150**, 1177; Oddo, *Gazetta*, 1911, **41**, 1, 11) such as diphenyl sulphoxide



thionyl chloride can only be represented as  $O = S \begin{cases} Cl \\ Cl \end{cases}$ . The calculations

of the magnetic susceptibilities were, however, made from the point of view of oxygen atom which, due to strong binding has been shown to exhibit a peculiar behaviour in the case of  $SeOCl_2$ . The values calculated with the dia- and paramagnetic constants of oxygen are given in Table V.

TABLE V

Constitution $O = S \begin{cases} Cl \\ Cl \end{cases}$	$\chi_a$ and $\chi_m$ obtained by using Pascal's value for $0 (-3.36 \times 10^{-6})$	$\chi_a$ and $\chi_m$ obtained by using the value of $0 (+1.8 \times 10^{-6})$
$(\chi_a)_1$	$-0.4494 \times 10^{-6}$	$-0.4061 \times 10^{-6}$
$(\chi_m)_1$	$-53.492 \times 10^{-6}$	$-48.332 \times 10^{-6}$
$(\chi_a)_2$	$-0.4485 \times 10^{-6}$	$-0.4052 \times 10^{-6}$
$(\chi_m)_2$	$-53.37 \times 10^{-6}$	$-48.21 \times 10^{-6}$
$(\chi_a)_3$	$-0.4487 \times 10^{-6}$	$-0.4053 \times 10^{-6}$
$(\chi_m)_3$	$-53.396 \times 10^{-6}$	$-48.23 \times 10^{-6}$
$(\chi_a)_4$	$-0.4477 \times 10^{-6}$	$-0.4043 \times 10^{-6}$
$(\chi_m)_4$	$-53.277 \times 10^{-6}$	$-48.117 \times 10^{-6}$
$(\chi_a)O_1$		$-0.372 \times 10^{-6}$
$(\chi_m)O_1$		$-44.3 \times 10^{-6}$
$(\chi_a)O_2$		$-0.4479 \times 10^{-6}$
$(\chi_m)O_2$		$-53.3 \times 10^{-6}$
$(\chi_a)O_3$		$-0.451 \times 10^{-6}$
$(\chi_m)O_3$		$-53.2 \times 10^{-6}$
$(\chi_a)O_4$		$-0.380 \times 10^{-6}$
$(\chi_m)O_4$		$-45.2 \times 10^{-6}$

$O_1$  refers to the values observed by Varadachari and Subramaniam.

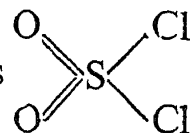
$O_2$  refers to the values calculated by Varadachari and Subramaniam.

$O_3$  refers to the values observed by Kido.

$O_4$  refers to the values observed by Nevgi.

The above Table clearly shows that the values calculated using Slater's and Angus's values and the paramagnetic value for O are in better agreement with the observed values of Varadachari and Subramaniam and Nevgi. Kido's values are, however, in good agreement with the values calculated from the diamagnetic values of O. It is not possible at this stage to decide whether O in  $\text{SOCl}_2$  is para- or dia-magnetic. However, if the analogy of this compound with  $\text{SeOCl}_2$  is taken into consideration it will appear that O is paramagnetic and Varadachari and Nevgi's experimental values are correct. The small differences in the calculated and experimental values can be attributed to the strong binding of oxygen atom as has been pointed out in the case of  $\text{SeOCl}_2$ .

(e) *Sulphuryl chloride* ( $\text{SO}_2\text{Cl}_2$ ).—On the basis of the physical and chemical properties of this substance its constitution has been accepted as



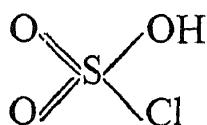
in which S is hexavalent. Values calculated for this constitution are given in Table VI.

Author's calculated values are in close agreement with those observed by Nevgi and Kido while those calculated by Varadachari and Subramaniam are rather high.

(f) *Chlorosulphonic acid* ( $\text{SO}_3\text{HCl}$ ).—Chlorosulphonic acid forms with aromatic hydrocarbons sulphonic acids of the structure  $\text{R-SO}_2\text{OH}$  and sul-

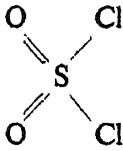
phones of the structure  $\begin{array}{c} \text{R} \\ \diagdown \\ \text{SO}_2 \\ \diagup \\ \text{R} \end{array}$ , where R represents an aromatic radical

such as phynyl ( $\text{C}_6\text{H}_5$ ) (*cf.* Muller, *Ber.*, 1873, **6**, 227; de Purgold, *Ber.*, 1873, **6**, 602; Claesson, *J. Pract. Chem.*, 1879, (ii) **19**, 231). From these and other reactions it is believed that in chlorosulphonic acid the chlorine atom is directly attached to sulphur and its constitution is represented



The compound  $\text{SO}_2\text{Cl}_2$  (discussed in the previous paragraphs) is obtained by replacing OH by Cl in  $\text{SO}_3\text{HCl}$ . The constitutions of the compounds  $\text{SO}_2\text{Cl}_2$  and  $\text{SO}_3\text{HCl}$  being the same, the difference between their molecular susceptibilities ought to be the difference in the susceptibilities of OH and Cl group which according to Pascal is 12.5. According to Nevgi's and

TABLE VI

Constitution	
$(X_a)_1$	- 0.3891 $\times 10^{-6}$
$(X_m)_1$	- 52.517 $\times 10^{-6}$
$(X_a)_2$	- 0.3888 $\times 10^{-6}$
$(X_m)_2$	- 52.487 $\times 10^{-6}$
$(X_a)_3$	- 0.3884 $\times 10^{-6}$
$(X_m)_3$	- 52.422 $\times 10^{-6}$
$(X_a)_4$	- 0.3820 $\times 10^{-6}$
$(X_m)_4$	- 52.365 $\times 10^{-6}$
$(X_a)O_1$	- 0.365 $\times 10^{-6}$
$(X_m)O_1$	- 49.3 $\times 10^{-6}$
$(X_a)O_2$	- 0.4193 $\times 10^{-6}$
$(X_m)O_2$	- 56.6 $\times 10^{-6}$
$(X_a)O_3$	- 0.397 $\times 10^{-6}$
$(X_m)O_3$	- 53.6 $\times 10^{-6}$
$(X_a)O_4$	- 0.402 $\times 10^{-6}$
$(X_m)O_4$	- 54.6 $\times 10^{-6}$

$O_1$  refers to the experimental values of Varadachari and Subramaniam.

$O_2$  refers to the calculated values of Varadachari and Subramaniam.

$O_3$  refers to the experimental value of Nevgi.

$O_4$  refers to the experimental value of Kido.

Kido's data this difference is 6.7 and 8.0, respectively. Values calculated according to Slater's and Angus's values are given in Table VII.

The values observed by Nevgi and Kido are much higher than the calculated values. Since in the case of sulphuryl chloride there is a very close agreement between the calculated and experimental values of Nevgi and Kido, they can be taken as correct. The difference between the molecular susceptibility of  $SO_2Cl_2$  (53.6) as observed by Nevgi and the molecular susceptibility of  $SO_3HCl$  (37.86) calculated above is 15.7. This is in close agreement with the value expected from Pascal's data (12.5).



TABLE VII

$(\chi_a)_1$	$- 0.3249 \times 10^{-6}$
$(\chi_m)_1$	$- 37.86 \times 10^{-6}$
$(\chi_a)_2$	$- 0.3246 \times 10^{-6}$
$(\chi_m)_2$	$- 37.287 \times 10^{-6}$
$(\chi_a)_3$	$- 0.3241 \times 10^{-6}$
$(\chi_m)_3$	$- 37.762 \times 10^{-6}$
$(\chi_a)_4$	$- 0.3239 \times 10^{-6}$
$(\chi_m)_4$	$- 37.735 \times 10^{-6}$
$(\chi_a)_{0_1}$	$- 0.402 \times 10^{-6}$
$(\chi_m)_{0_1}$	$- 46.9 \times 10^{-6}$
$(\chi_a)_{0_2}$	$- 0.400 \times 10^{-6}$
$(\chi_m)_{0_2}$	$- 46.6 \times 10^{-6}$

$0_1$  refers to the values observed by Nevgi.

$0_2$  refers to the values observed by Kido.

### Conclusion

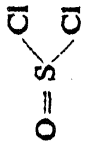
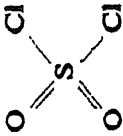
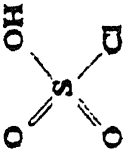
Table VIII gives at a glance the experimental and the calculated values for the constitution of the sulphur compounds found according to the plan employed in this investigation. Wide differences between the calculated and the observed values are observed only in the case of compounds (c) and (f). This may be due either to the want of purity of the substances used or to their instability.

In the case of sulphur compounds also as in the case of Se and Te compounds, the magnetic method seems to be of great value in fixing the correct molecular constitution and the magnetic susceptibilities calculated by Slater's and Angus's method seem to be in better agreement with the experimental ones.

Further work on compounds of this group is in progress.

TABLE VIII  
Sulphur Compounds

Compound	Constitution	$-\chi_a \times 10^6$ & $-\chi_m \times 10^6$ observed by other authors	$-\chi_a \times 10^6$ & $-\chi_m \times 10^6$ calculated by Slater's method	$-\chi_a \times 10^6$ & $-\chi_m \times 10^6$ calculated by the modi- fication of Slater's method	$-\chi_a \times 10^6$ & $-\chi_m \times 10^6$ calculated by Angus's method	$-\chi_a \times 10^6$ & $-\chi_m \times 10^6$ calculated by the modi- fication of Angus's method
$S_2Cl_2$	$\begin{array}{c} Cl \\ \diagdown \\ S = S \\ \diagup \\ Cl \end{array}$	$(\chi_a)$ 0.461 $(\chi_m)$ 62.2	0.4613 Varadachari and Subramaniam 62.272	0.4585 Varadachari and Subramaniam 61.90	0.4523 61.05	0.4496 60.697
$S_2I_2$	$\begin{array}{c} I \\ \diagdown \\ S = S \\ \diagup \\ I \end{array}$	$(\chi_a)$ 0.405 $(\chi_m)$ 64.7	Nevgi 0.3430 Varadachari and Subramaniam 109.072	0.3418 Varadachari and Subramaniam 108.7	0.3393 107.856	0.3381 107.497
$SCl_2$	$\begin{array}{c} Cl \\ \diagdown \\ S \\ \diagup \\ Cl \end{array}$	$(\chi_a)$ 0.467 $(\chi_m)$ 48.1	Varadachari and Subramaniam 0.5489 Kido 56.54	0.5466 56.29	0.5381 55.42	0.5358 55.18
		$(\chi_a)$ 0.478 $(\chi_m)$ 49.4				
		$(\chi_a)$ 0.478 $(\chi_m)$ 49.2	Nevgi			

$\text{SOCl}_2$		(X <sub>a</sub> ) 0.372 (X <sub>m</sub> ) 44.3	} Varadachari and Subramaniam 48.332	0.4061 Varadachari and Subramaniam 48.21	0.4053 48.23	0.4043 48.117
$\text{SO}_2\text{Cl}_2$		(X <sub>a</sub> ) 0.380 (X <sub>m</sub> ) 45.2	} Nevgi	0.3891 Varadachari and Subramaniam 52.517	0.3888 52.487	0.3239 37.735
$\text{SO}_2\text{HCl}$		(X <sub>a</sub> ) 0.397 (X <sub>m</sub> ) 53.6	} Kido	0.3249 Nevgi 37.86	0.402 46.9	0.400 46.6

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