666. *Electrolytic Conductance of Solutions of Sulphur Dichloride, and its Behaviour towards Aluminium, Iron, and Antimony Chlorides*

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Sulphur dichloride behaves as a weak electrolyte in acetone solution; values for its molar conductance are given. The following series of solid 1 : 1 addition complexes have been prepared and characterised: $AlCl_3,SCl_2$; $FeCl_3,SCl_2$; $SbCl_5,SCl_2$. Their reactions with benzene give the following compounds, and hydrogen chloride is evolved: $AlCl_3,PhSCl$; $FeCl_3,PhSCl$; $SbCl_5,PhSCl$. Both series of compounds are considered to be sulphenium salts of the type $[RS]^+[MCl_{x+1}]^-$, (R = Cl or Ph).

Sulphur dichloride, SCl_2, behaves as an acid chloride towards most reagents;[1] sulphoxylic acid is believed to be formed when it is hydrolysed, and it undergoes exchange reactions with some salt-like compounds.[2] Whether this property is related to the formation of sulphenium ions by the dissociation of its S–Cl linkages is not known, and information about its behaviour towards Lewis acids is meagre.[3,4] Arenesulphenyl chlorides, on the other hand, have been shown to form cations, ArS^+, in acid solution,[5] which are thought to be responsible for many of their chemical reactions.[6] They also form complex salts with Lewis acids such as aluminium chloride, and antimony(III) chloride and fluoride,

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[1] M. Becke-Goehring, H. Stamm, and U. Feldmann, *Z. anorg. Chem.*, 1942, **250**, 56; H. Stamm and M. Becke-Goehring, *Ber.*, 1943, **76***B*, 737.

[2] F. Fehér and L. Z. Mayer, *Z. Naturforsch.*, 1956, **11**b, 605; F. Fehér, K. Naused, and H. Weber, *Z. anorg. Chem.*, 1957, **290**, 303.

[3] O. Ruff and H. Golla, *Z. anorg. Chem.*, 1924, **138**, 17.

[4] J. W. Mellor, " A Comprehensive Treatise on Inorganic and Theoretical Chemistry," Longmans, London, vol. X, pp. 643, 646.

[5] (*a*) N. Kharasch, C. M. Buess, and W. King, *J. Amer. Chem. Soc.*, 1953, **75**, 6035; (*b*) N. Kharasch and S. J. Assony, *ibid.*, p. 1081.

[6] N. Kharasch, S. J. Potempa, and H. L. Wehrmeister, *Chem. Rev.*, 1946, **39**, 298; A. J. Parker and N. Kharasch, *ibid.*, 1959, **59**, 583.

e.g., $ArS^+AlCl_4^-$ (Ar = 2,4-dinitrophenyl, or *p*-tolyl).[5,7] Recent observations in this laboratory have revealed that trichloromethanesulphenyl chloride, $CCl_3{\cdot}SCl$, which gave the sulphenium ion, CCl_3S^+, also behaves similarly towards antimony pentachloride.[8] It was thus of interest to investigate the ionic dissociation of sulphur dichloride, and to study its reaction with some metal chlorides of the Lewis-acid type, with a view to isolating the chlorosulphenium ion in the form of complex salts. Attempts were then made to convert the chlorosulphenium ion into the benzenesulphenium ion, which is not known.

Sulphur dichloride dissolves readily in acetone to form a weakly conducting solution. Measurements on a freshly prepared solution show that the conductance rises with the addition of sulphur dichloride; this is attributed to the contribution of the available ions by the ionisation of sulphur dichloride. The specific conductance for a 0·35M-solution is $2{\cdot}45 \times 10^{-3}$ ohm^{-1} cm.$^{-1}$ at 25° (cf. 2×10^{-8} ohm^{-1} cm.$^{-1}$ for the pure solvent). Solutions covering a range of concentrations were examined. When molar conductance is plotted against the square root of the molar concentration, the end-points corresponding to the higher dilutions fall nearly on a straight line which, on extrapolition to zero concentration, gives a value of 40 ohm^{-1} cm.^{2} mole^{-1} for the molar conductance at infinite dilution (Λ_m, ∞). Thus, the relatively high conductance of these solutions leads to the conclusion that, in acetone, sulphur dichlororide ionises with the probable occurrence of the solvation of the ions. By analogy with disulphur dichloride[9] and trichloromethanesulphenyl chloride,[8] the ionisation occurring in the solution may be of the overall form,

$$SCl_2 \rightleftharpoons ClS^+ + Cl^-$$

This is supported by the result of a conductometric titration of sulphur dichloride with ferric chloride in acetone solution (see below), and by the quantitative precipitation of one atom of chlorine with silver nitrate.[10]

Aluminium chloride reacts with sulphur dichloride at below room temperature (10—15°) to give the 1 : 1 complex $AlCl_3,SCl_2$ (I). This compound was earlier reported[3] to be formed as a result of the thermal decomposition of $AlCl_3,2S_2Cl_2$, but was not adequately characterised. The complex is stable *in vacuo* and is highly hygroscopic. It is decomposed by hydroxylic solvents (water, alcohol), and is insoluble in carbon tetrachloride, chloroform, cyclohexane, and nitrobenzene. It does, however, react with benzene to give a new addition complex, $AlCl_3,PhSCl$ (II) by the replacement of one chlorine atom. The complex, on further reaction with a mole of benzene, gives[11a] the diphenyl sulphide adduct, $AlCl_3,Ph_2S$ (III). The reaction takes place in the following steps:

$$AlCl_3 + SCl_2 \longrightarrow AlCl_3,SCl_2 \tag{1}$$

$$AlCl_3,SCl_2 + PhH \longrightarrow AlCl_3,PhSCl + HCl \tag{2}$$

$$AlCl_3.PhSCl + PhH \longrightarrow AlCl_3,Ph_2S + HCl \tag{3}$$

Both benzenesulphenyl chloride, PhSCl, and the diphenyl sulphide, Ph_2S, can be isolated from aluminium chloride by treatment with diethyl ether. Formation of the ethereal complex indicates that ether, is, compared with benzenesulphenyl chloride or diphenyl sulphide, a relatively stronger ligand to aluminium chloride. This method can be satisfactorily employed for the preparation of benzenesulphenyl chloride. The existing method for the synthesis of diphenyl sulphide from benzene and sulphur chloride in presence of aluminium chloride makes no mention of the intermediate.[11] The complex (II) was deeply

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[7] N. Kharasch and D. L. Chamberlain, *J. Amer. Chem. Soc.*, 1955, **77**, 1041; R. Swidler, Master Thesis, University of Southern California, 1950 (cited in ref. 5*a*).

[8] S. N. Nabi, S. Ahmad, and S. Ahmad, jun., *J.*, 1963, 2636.

[9] V. Gutmann and G. Schöber, *Monatsh.*, 1956, **87**, 792; H. Spandau and H. Hattwig, *Z. anorg. Chem.*, 1961, **311**, 32.

[10] M. S. Amin, M.Sc. Thesis, Dacca University, 1964.

[11] (*a*) J. Böeseken, *Rec. Trav. chim.*, 1905, **24**, 209; A. H. Blatt, *Org. Synth.*, Coll. Vol. II, 1943, p. 242; (*b*) C. A. Thomas, " Anhydrous Aluminium Chloride in Organic Chemistry," Reinhold, New York, 1941, pp. 163—166.

coloured, highly hygroscopic, and decomposed on exposure to the atmosphere. It was insoluble in carbon tetrachloride, but was somewhat soluble in ethanol to give a yellow solution, which was a conductor of electricity. It reacted with 98% sulphuric acid with the evolution of hydrogen chloride and formed a deep violet solution. These facts, and previous studies on the behaviour of aluminium chloride towards certain other sulphenyl chlorides,[5] suggest that benzenesulphenyl chloride also probably forms a complex with aluminium chloride, which may be formulated as $PhS^+ AlCl_4^-$. The development of colour in sulphuric acid has a bearing on the existence of the arenesulphenium cation, PhS^+. Kharasch *et al.*[5] observed a similar colour in sulphuric acid solution containing the 2,4-dinitrobenzenesulphenium ion; evidently the colour does not depend on the presence of the nitro-groups.

It has been reported that, although certain arenesulphenyl chlorides react with benzene under Friedel–Crafts conditions, extension of the reaction to benzenesulphenyl chloride itself is unsuccessful.[12] In the present study, however, we found that the benzenesulphenyl chloride–aluminium chloride adduct reacted smoothly with benzene. This was almost certainly due to the existence, under these conditions, of the benzenesulphenium ion, PhS^+, which makes it amenable to attack by bases such as benzene. It is, therefore, reasonable to suppose that the scission of an S–Cl linkage in sulphur dichloride by its interaction with electrophilic aluminium chloride, giving rise to the chlorosulphenium ion, ClS^+, is also a precondition for its initial reaction with benzene, equation (2). The behaviour of the complex (I) will then be salt-like, $ClS^+ AlCl_4^-$.

Ferric chloride also formed a 1 : 1 complex with sulphur dichloride, $FeCl_3,SCl_2$ (IV). When ferric chloride was titrated conductometrically with sulphur dichloride, or *vice versa*, both freshly dissolved in acetone, steady variation of conductance took place initially, but there was a sharp break in the conductivity curve (see Figure) at a molar ratio of 1 : 1, implying the formation of a complex salt $ClS^+ FeCl_4^-$ in solution. The compound was deep brown and highly hygroscopic. It reacted with benzene to give hydrogen chloride and the addition complex, $FeCl_3,PhSCl$ (V). This was highly hygroscopic and decomposed in air, was insoluble in non-polar solvents and, by analogy with the aluminium complex, may be formulated as a salt, $PhS^+ FeCl_4^-$. Further substitution of chlorine by treatment with excess of benzene was, however, not possible. The direct reaction of the complex (IV) with benzene in 1 : 2 proportion also led to compound (V) only. This seems anomalous compared with the behaviour of the aluminium compounds, but is consistent with the lower efficiency of ferric chloride as a Friedel–Crafts reagent. Lability of the chloro-complex intermediates is a factor in such Friedel–Crafts type of reactions, but the probable participation of the inner *d* orbital of iron in the formation of $[FeCl_4]^-$ will mean enhanced stability for the latter as compared to $[AlCl_4]^-$ or $[SbCl_6]^-$. Further stabilisation of $[FeCl_4]^-$ may be achieved by the organic sulphenium ion, as the ferrichlorides of organic bases are known to be formed with unusual ease, and are stable.[13] So, although the complex salt $ClS^+FeCl_4^-$ reacts with benzene, the complex salt $PhS^+ FeCl_4^-$ does not. It was not possible to effect a satisfactory separation of benzenesulphenyl chloride by treating the complex (V) with ether. This may be due to the solubility of the ethereal complex of ferric chloride in excess of the reactant, or there may be very little reaction, as happens with benzene.

Antimony pentachloride reacted with sulphur dichloride at 10° to give the 1 : 1 complex, $SbCl_5,SCl_2$ (VI). By analogy with the other members of this series, this compound also may be formulated as $ClS^+ SbCl_6^-$. Conductometric titration in acetone was, however, unsuccessful, since antimony pentachloride and the solvent formed a stable adduct, from which it was impossible to displace the acetone by sulphur dichloride or Cl^-. The complex salt (VI) was deep brownish-yellow and was stable *in vacuo*. It was highly

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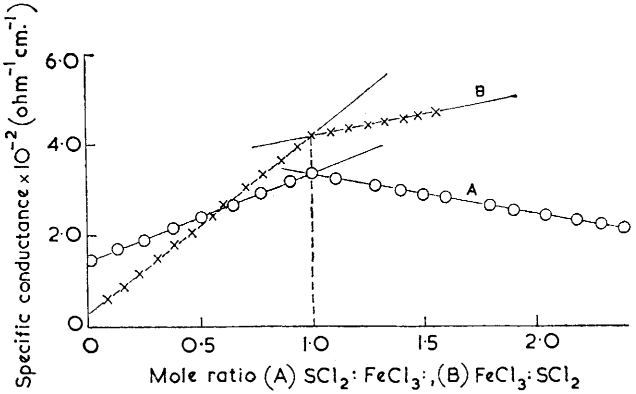
[12] C. M. Buess and N. Kharasch, *J. Amer. Chem. Soc.*, 1950, **72**, 3529; K. Fries and G. Schurmann, *Ber.*, 1919, **52**, 2170; H. Lecher, *ibid.*, 1925, **58**, 409.

[13] N. V. Sidgwick, "Chemical Elements and Their Compounds," Clarendon, Oxford, 1950, Vol. II, p. 1367.

hygroscopic and decomposed readily in contact with the atmosphere. It reacted readily with benzene to give the 1 : 1 adduct $SbCl_5,PhSCl$ (VII), which almost certainly has the sulphenium-salt structure $PhS^+ SbCl_6^-$. Its instability on exposure to the atmosphere and towards hydroxylic solvents is that of a typical antimony salt. Benzenesulphenyl chloride was extractable from this compound by treatment with ether. The complex reacted with further amounts of benzene to give hydrogen chloride, and a highly viscous liquid, which was not characterised, but is presumed to be a complex of antimony pentachloride with diphenyl sulphide.



Conductance titration in acetone solution of (A) ferric chloride with sulphur dichloride, (B) sulphur dichloride with ferric chloride

At higher temperatures, the reaction of antimony pentachloride and sulphur dichloride is more complicated, particularly in the presence of excess of the former. They react together at ordinary temperatures to give antimony trichloride and the addition complex, $SbCl_5,SCl_4$,

$$2SbCl_5 + SCl_2 \longrightarrow SbCl_3 + SbCl_5.SCl_4$$

Antimony pentachloride, thus, acts as a chlorinating agent towards sulphur dichloride in the first instance. Evidently, the complex salt $ClS^+ SbCl_6^-$ (VI) formed by the primary 1 : 1 reaction breaks up into antimony trichloride and sulphur tetrachloride; the latter is stabilised by complexing with a further mole of antimony pentachloride. In a related study on the reaction of antimony pentachloride and disulphur dichloride in sulphuryl chloride solution Partington[14] obtained the complex $SbCl_5,SCl_4$, but he does not state whether disulphur dichloride formed any adduct with antimony pentachloride. The complex $SbCl_5,SCl_4$ may well be $SCl_3^+ SbCl_6^-$, but conductometric evidence is lacking. Trichloromethanesulphenyl chloride forms complex salts with antimony pentachloride, but these decompose as the temperature is raised.[8]

From these studies it is therefore concluded that electrophilic attack by the above Lewis acids on polar sulphur dichloride, $\overset{\delta+}{S}\text{–}\overset{\delta-}{Cl_2}$, leads to the fission of an S–Cl linkage; and hence to the production of the complex salts. Generation of the chlorosulphenium, ClS^+, or benzenesulphenium ions, PhS^+, is a precondition for the reaction of the chlorides with benzene.

EXPERIMENTAL

Sulphur dichloride was prepared by the reaction of chlorine with boiling sulphur to form first disulphur dichloride which, on further chlorination at 15—18° in presence of active charcoal, gave sulphur dichloride. The fraction collected at 58—60° was redistilled. A freshly distilled sample was always used.

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[14] J. R. Partington, *J.*, 1929, 2573.

Reagent quality acetone (M. & B.) was further purified by treatment with silver nitrate and sodium hydroxide; it was then dried ($CaSO_4$) and distilled; it was redistilled before use. We found that in making up solutions, the sulphur dichloride should always be added to the acetone; the reverse process may lead to violent frothing because of the evolution of heat. Aluminium and ferric chlorides were purified by sublimation under reduced pressure, the former after admixture with aluminium powder. Other solvents and materials were purified and dried by standard procedures.

A precision Cambridge conductivity bridge type D1920 was used for conductance measurements. The cell was of the small dipping-electrode type, and the electrodes were of smooth platinum (cell constant 0·10 cm.$^{-1}$). The values of molar conductance obtained for sulphur dichloride are given in the Table.

Conductance of sulphur dichloride in freshly prepared acetone solution at 25°

C_m (mole l.$^{-1}$)	0·0025	0·005	0·010	0·021	0·043	0·087	0·175	0·350
10^4k (ohm^{-1} cm.$^{-1}$)	0·70	1·22	1·88	2·80	4·60	7·50	13·15	24·50
Λ_m (ohm^{-1} cm.^{2} mole^{-1}) ...	28·0	24·4	18·8	13·3	10·7	8·6	7·5	7·0

Reaction of Sulphur Dichloride with Aluminium Chloride.—Aluminium chloride (0·734 g., 5·5 mmoles) was treated with sulphur dichloride (0·566 g., 5·5 mmoles) in a trap connected to the vacuum system and kept at 10—15°. An immediate reaction was apparent by the formation of a yellow solid. When the volatile matter had been pumped off the product was identified as the 1 : 1 *complex*, $AlCl_3,SCl_2$ (1·26 g., 97%) (Found: Al, 12·0; Cl, 72·9; S, 13·5. $AlCl_5S$ requires Al, 11·4; Cl, 75·0; S, 13·5%). The product was analysed by fusion with potassium hydroxide, aluminium and sulphur were determined gravimetrically as aluminium oxide and barium sulphate, respectively, and chlorine was determined volumetrically by Volhard's method.

Reaction of the Complex $AlCl_3,SCl_2$ *with Benzene.*—The complex (16·45 g., 69·6 mmoles) contained in a trap at 10—15° was allowed to react very slowly with benzene (5·426 g., 69·6 mmoles). Hydrogen chloride (2·51 g., 99%) was immediately evolved, and a yellowish-green solid was formed, which was found to be the 1 : 1 *complex*, $AlCl_3,PhSCl$ (18·89 g., 98%) (Found: Al, 10·4; Cl, 50·7; S, 11·5; $C_6H_5AlCl_4S$ requires Al, 9·7; Cl, 51·0; S, 11·5%). The complex did not melt, but decomposed when heated.

Reaction of the Complex $AlCl_3,PhSCl$ *with Ether.*—The complex (2·632 g., 9·5 mmoles) was treated with dry ether (50 ml., in excess) at 15°. A white solid was formed and the liquid turned yellow. The solid product was isolated and identified as the 1 : 1 ether adduct, $AlCl_3,Et_2O$ (1·867 g., 94·8%), m. p. 36° (lit., 35°) (Found: Al, 13·2; Cl, 49·8. Calc. for $C_4H_{10}AlCl_3O$: Al, 13·0; Cl, 51·3%). The ethereal solution on fractionation gave a heavy yellow liquid (b. p., 150—151°), which was identified as the *benzenesulphenyl chloride*, PhSCl (1·168 g., 85·4%), b. p. 149° (Found: Cl, 23·5; S, 21·9%; C_6H_5ClS requires Cl, 24·5; S, 22·1%).

Reaction of the Complex $AlCl_3,PhSCl$ *with Benzene.*—The complex (5·829 g., 21·0 mmoles) contained in a trap was treated with benzene (1·635 g., 21·0 mmoles) at 15°, when hydrogen chloride (0·702 g., 94·6%) was evolved. After all volatile matter had been removed, the solid product was the 1 : 1 *complex* $AlCl_3,SPh_2$ (6·6 g., 98%) (Found: Al, 8·8; Cl, 32·2; S, 10·0; $C_{12}H_{10}AlCl_3S$ requires Al, 8·4; Cl, 33·3; S, 10·2%). This was soluble in ethanol, but reacted with ether (see below).

Reaction of the Complex $AlCl_3,Ph_2S$ *with Ether.*—The complex (1·491 g., 4·7 mmoles) was treated with dry ether (50 ml., in excess) at 15°. The white solid that formed was identified as the 1 : 1 ether adduct, $AlCl_3,Et_2O$, (0·865 g., 89·6%) (Found: Al, 12·8; Cl, 48·8; m. p., 35—36°; Calc. for $C_4H_{10}AlCl_3O$: Al, 13·0; Cl, 51·3%). The yellow ethereal solution was fractionally distilled and yielded diphenyl sulphide, Ph_2S, at 295—296° (lit., 296°) (0·824 g., 94·9%) (Found: S, 16·5. Calc. for $C_{12}H_{10}S$: S, 17·2%).

Reaction of Ferric Chloride with Sulphur Dichloride.—When ferric chloride (3·385 g., 20·9 mmoles) was heated with sulphur dichloride (2·15 g., 20·9 mmoles) in a trap at 15°, a brown solid was immediately formed. The volatile matter was pumped off and the residue was identified as the 1 : 1 *complex*, $FeCl_3,SCl_2$ (5·52 g., 99%). This was analysed by fusion with potassium hydroxide. (Found: Cl, 65·9; Fe, 21·5; S, 11·1. Cl_5FeS requires: Cl, 66·9; Fe, 21·1; S, 12·0%). The complex was decomposed by air, water, and ethanol, was insoluble in carbon tetrachloride and nitrobenzene, but was soluble in acetone, forming a conducting solution.

Conductometric Titration.—A solution (30 ml.) of ferric chloride (0·163 g.) in acetone was titrated conductometrically with an acetone solution of sulphur dichloride (0·13M). For the reverse titration, an equal volume of a solution of sulphur dichloride (0·132 g.) was titrated with 0·1M-ferric chloride solution under similar conditions.

Reaction of the Complex $FeCl_3,SCl_2$ *with Benzene.*—(i) *Reaction in equimolar ratio.* The complex (4·291 g., 16·2 mmoles) and benzene (1·262 g., 16·2 mmoles) reacted instantaneously at 15° to give hydrogen chloride (0·568 g., 98%) and a green solid identified as the 1 : 1 *complex*, $FeCl_3$,PhSCl, (4·88 g., 98·3%) (Found: Cl, 45·4; Fe, 18·4; S, 10·0. $C_6H_5Cl_4FeS$ requires Cl, 46·2; Fe, 18·2; S, 10·4%).

(ii) *Reaction in the molar ratio of* 1 : 2. The complex (3·51 g., 13·2 mmoles) and benzene (2·06 g., 26·4 mmoles) reacted vigorously at 15° with the evolution of hydrogen chloride (0·453 g., 12·4 mmoles, 93%). After the reaction was over, the product left in the reaction vessel contained a deep green solid and some unchanged benzene, which was removed. The solid was the 1 : 1 complex, $FeCl_3$,PhSCl (3·97 g., 97%) (Found: Cl, 45·3; Fe, 18·3; S, 10·2). When treated with ether the complex slowly went into solution and a slight turbidity appeared, but no precipitate settled out, even after prolonged centrifugation.

Reaction of Sulphur Dichloride with Antimony Pentachloride.—Antimony pentachloride (3·347 g., 11·2 mmoles) contained in a trap at 10—15° was allowed to react with sulphur dichloride (1·153 g., 11·2 mmoles). The mixture immediately changed into a yellowish-brown solid. After the volatile matter had been pumped off, the residue was identified as the 1 : 1 *complex* $SbCl_5,SCl_2$ (4·44 g., 99%). This was analysed by treatment with a 15% solution of potassium hydroxide. Antimony(V) was determined volumetrically by the iodimetric method, and chlorine and sulphur were determined as described above (Found: Cl, 60·2; S, 7·6; Sb^V, 30·6. Cl_7SSb requires Cl, 61·8; S, 7·9; Sb^V, 30·3%). The complex was decomposed by air, water, and ethanol, and was insoluble in carbon tetrachloride, chloroform, and cyclohexane.

Conductometric Titration.—When a solution (30 ml.) of antimony pentachloride (0·3 g.) in acetone was titrated conductometrically with an acetone solution of sulphur dichloride (0·1M) at 25°, the conductance decreased slowly without any sharp change.

Reaction of the Complex $SbCl_5,SCl_2$ *with Benzene.*—The complex (4·524 g., 11·3 mmoles) was treated with benzene (0·88 g., 11·3 mmoles) at 15°. Hydrogen chloride (0·391 g., 97%) was evolved immediately and the dark green solid that formed was identified as the 1 : 1 *complex* $SbCl_5$,PhSCl (4·93 g., 99%) (Found: Cl, 47·5; S, 6·9; Sb^V 27·2. $C_6H_5Cl_6SSb$ requires Cl, 48·0; S, 7·2; Sb^V, 27·5%). The complex, when treated further with an equimolar quantity of benzene gave hydrogen chloride and a viscous material, which was not characterised precisely.

Reaction of the Complex $SbCl_5$,PhSCl *with Ether.*—The complex (2·451 g., 5·5 mmoles) when treated with excess of dry ether (50 ml.) at 10—15°, reacted immediately to give a light yellow solid and a yellow solution. The solid was the *ether adduct*, $SbCl_5,Et_2O$, (1·98 g., 95%) (Found: Cl, 46·4; Sb^V, 33·2. $C_4H_{10}Cl_5OSb$ requires: Cl, 47·5; Sb^V, 32·6%). The ethereal solution on fractionation, gave a yellow liquid boiling at 150—151° (lit., 149°), which was benzenesulphenyl chloride, PhSCl (0·72 g., 90%) (Found: Cl, 24·2; S, 21·6. Calc. for C_6H_5ClS: Cl, 24·5; S, 22·1%).

Reaction of Sulphur Dichloride and Antimony Pentachloride in the Molar Ratio of 1 : 2.—Sulphur dichloride (0·52 g., 5 mmoles) and antimony pentachloride (3·00 g., 10 mmoles) reacted at 25° in a trap attached to the vacuum system; heat was liberated and the mixture solidified. The product was completely soluble in phosphorus oxychloride, partially soluble in sulphuryl chloride, and insoluble in solvents such as carbon tetrachloride, chloroform, and light petroleum. The reaction mixture was treated with sulphuryl chloride when a portion went into solution; the white crystalline solid that remained was identified as antimony trichloride (0·82 g., 92%). [Found: Cl, 45·8; Sb^{III} (volumetrically, potassium bromate method) 54·2. Calc. for $SbCl_3$: Cl, 46·5; Sb^{III}, 53·5%)]. The soluble component was crystallised under partial vacuum as needles and was the complex $SbCl_5,SCl_4$, (1·55 g., 85%). It was analysed by treatment with a 15% solution of potassium hydroxide. (Found: Cl, 66·8, 66·7; S, 7·3, 7·6; Sb^V, 24·4, 24·5. Cl_9SSb requires Cl, 67·5; S, 6·8; Sb^V, 25·7%).

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