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The solubility of inorganic salts in ether

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THE SOLUBILITY OF INORGANIC SALTS IN WATER

by

CLARENCE BERNARD WEISS

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THESIS IS

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FORIWORD

The author wishes to express his sincere appreciation to Dr. C. J. Menree for the suggestion of this research and for his valuable assistance, and also to Dr. W. T. Schrenk and D. H. L. Dunlap, for valuable advice and suggestions given while this work was in progress.

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THE SOLUBILITY OF INORGANIC SALTS IN ETHER

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INTRODUCTION

The data on the solubility of inorganic salts in ether is scarce. Eder¹ made a study of this subject and found few salts whose solubility could be called high. He used both hydrated and dehydrated salts.

Neumann² mentions the great solubility of aluminum chloride in ether, but did no work to find its exact proportions. It was thought that perhaps with this particular salt, a solubility curve might be obtained that would be both instructive and interesting. Therefore the major portion of this work was spent in determining the solubility of aluminum chloride in ether.

Other salts whose solubilities were unknown were studied, but none were found whose solubility could equal that of aluminum chloride.

¹Eder Dingl., 221, 89

²Neumann B. 43, 314, 1910

PREPARATION AND PURIFICATION OF THE INORGANIC
SALTS, AND PURIFICATION OF ETHER

The aluminum chloride was prepared by passing dry hydrogen chloride gas over metallic aluminum. A simple method of evolving this gas was used. It was evolved by placing concentrated sulfuric acid in a large dropping funnel and regulating the flow into concentrated hydrochloric acid so that a steady stream of gas was obtained. This was passed through a concentrated sulfuric acid well, then through a calcium chloride drying tube, and an aluminum chloride drying tube. The aluminum was placed in a dried, long glass tube of proper size and length. The gas was allowed to pass through for about thirty minutes and the tube heated to drive out the last possible traces of moisture. On the end of this tube, a receiving vessel for the aluminum chloride formed, was placed. The aluminum was then heated to dull red heat and the resulting aluminum chloride passed part way the length of the tube. In this manner it was resublimed four times before ultimately reaching the receiver.

The other salts used were C. P. Analyzed which were recrystallized twice. The hydrated salts were prepared by placing the recrystallized salt in a desiccator in the presence of some of the dried salt. The dehydrated salts were prepared by placing

them in a vacuum desiccator at room temperature over phosphorus pentoxide for four weeks.

The ether was purified by washing twice with water, to remove any alcohol, and water soluble impurities present. It was then placed over calcium chloride for forty eight hours, distilled, placed over calcium chloride again for forty eight hours, redistilled on to sodium chips and left stand approximately one to two weeks. As the ether was needed it was distilled off. In this manner freshly dried and distilled ether was made use of.

APPARATUS

The apparatus and the method for determining the solubilities was very simple.

A DeKhotinsky Thermostat, the temperature which could be kept constant to within 0.1 degree, was used in obtaining equilibrium conditions in the solutions at constant temperatures. With the aid of ice, temperatures down to five degrees C. were easily reached. To get a sample at 100 degrees C., a ten liter tin container was used, under which was placed two Fischer burners. This did not hold the temperature exactly constant within the usual 0.1 degree, but varied about two degrees. The rest of the temperatures were taken in the thermostat.

The mechanical agitator or stirring device consisted of an electric motor, driving a 14 inch wheel held in horizontal position. To this was attached, several inches off center, a connecting arm which was similarly connected to a half inch rod, five feet long, held in a vertical position in the thermostat. To this rod were fastened the flasks containing the solutions which were to reach equilibrium.

When solubilities of salts other than aluminum chloride were run, the temperatures could not be advanced over 25 degrees C. on account of the volatility of the ether. In the case of the aluminum salt this was not so, as compounds were formed.

The solute and solvent in both cases were placed in Florence flasks. The two holed cork stoppers used, were washed in ether and dried, and then the outside coated with paraffin to exclude all moisture. A Soxhlet thimble was placed over the end of the outlet tube in the flask. The two outlets to the flask were closed with Bunsen valves. To secure a sample a slight pressure of dried air was applied to the flask.

PROCEDURE

The ether with the solute was placed in a 500 cc. Florence flask. This was placed in the thermostat, at a temperature somewhat higher than that desired, and gradually cooled down to the desired temperature. Samples were taken, in order to find the length of time at which equilibrium was reached. These conditions were obtained after two hours of agitation. In the case of aluminum chloride, one hour of agitation was sufficient to bring about equilibrium conditions. In the beginning, constant volume samples of known weight were taken, but this plan was later rejected and direct weighing was used.

When determining the solubility of anhydrous salts, the sample was taken in a weighing bottle, weighed, the ether evaporated, and then placed in an oven with the temperature ten degrees higher than the boiling point of ether, or about 45 degrees C. The weighing bottle was cooled in a desiccator and weighed.

In the case of the hydrated salts, the above procedure was followed up to the point of evaporation of the ether. Instead of evaporating the ether in the weighing bottle, it was transferred to a crucible, then evaporated, and placed in an electric muffle to

On account of the great deliquescence of the aluminum chloride, the method of taking the sample was altered slightly. The outlet tube of the flask projected into a bell jar, through which a stream of dry air was flowing. In this dry atmosphere, a sample was taken in a glass stoppered weighing bottle and the weight taken immediately. It was then placed in a large beaker containing about 600 cc. of distilled water and the stopper removed beneath the surface of the liquid. This was placed in a one liter volumetric flask and filled to the mark with distilled water. Ten cc. samples were taken and titrated for chlorine content by the Volhard method.

The weights used throughout the investigation were carefully calibrated, and the silver nitrate solutions used in titrations were standardized against pure sodium chloride.

TABULATED DATA

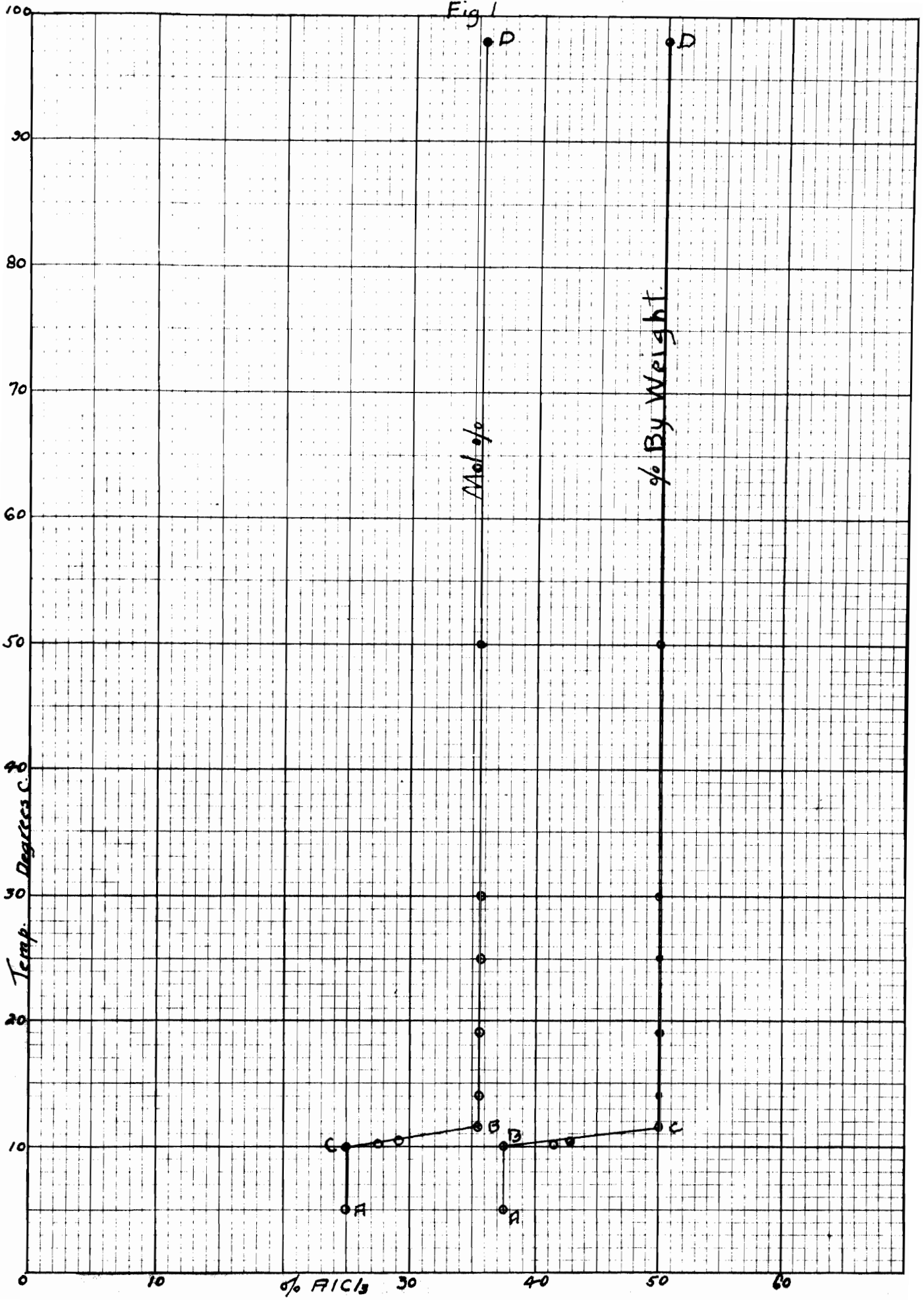
The solubility of salts in ether was calculated and recorded as grams salt per 100 grams solution. The temperature in all cases was 20 degrees C.

Salt	Solubility
KNO_3	insoluble
$NaCl$	insoluble
NH_4Cl	insoluble
NaI	Insoluble
$AgNO_3$	insoluble
CuI	insoluble
$CuBr$	insoluble
$HgNO_3$	decomposition
$Fe(NO_3)_3 \cdot 9H_2O$	decomposition
$CdBr_2$	0.004 grams
$LiNO_3$	0.005
$Ca(NO_3)_2$	0.083
$Zn(NO_3)_2 \cdot 4H_2O$	0.378
$Co(NO_3)_2 \cdot 4H_2O$	0.133

The solubility of aluminum chloride in ether was calculated and recorded as percent by weight, and as mol percent.

Temp. Degrees Cent.	% by weight	Mol %
5	37.52 37.45	24.9
10	37.45 37.55	24.9
10.1	41.50 41.45	28.2
10.5	42.82 42.80	29.2
11.5	50.00 50.00	35.6
14.	50.00 50.11	35.6
19	49.97 50.13	35.6
25	49.89 50.12	35.6
30	50.02 50.00	35.6
50	50.02 50.01	35.6
98	50.00 50.04	35.6

Fig 1



DISCUSSION

All of the salts with the exception of aluminum chloride showed only low solubilities. The effect of temperature on these salts was slight. This was proven by the fact that samples taken at ten degrees checked those taken at twenty-five. The difference of solubility of the dehydrated salts and the hydrated salts agrees with Mer's results. Water being soluble in ether up to two percent, is taken from the salt, and then the aqueous ether dissolves the salt to a larger extent. The decomposition of hydrated ferric nitrate upholds this view.

The solubility curve for ether and aluminum chloride is more complicated. When the ether is added to the aluminum chloride, heat is given off, and the resulting solution is dark brown, almost black. Both of these phenomena tend to point to compound formation. Another argument in this favor is that the reaction of this solution with water is not nearly so violent as that of free aluminum chloride and water. When this ether solution is added to water a small amount of tarry substance is formed. It has the odor of pine oils and is insoluble in water, but very soluble in ether, giving no tests for aluminum or chlorine. The amount of this product varies with the

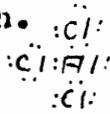
age of the solution. That is, the longer the ether has been in contact with the excess aluminum chloride, the the more the amount of this new compound or mixture. It was thought that this might be due to a trace of aldehydes, but a test on the purified ether proved it to be aldehyde free. Also tests were ran in order to determine whether the presence of aldehydes did affect the amount of this tarry substance produced. To small amounts of the aluminum chloride-ether solution, varying quantities of aldehyde were added, ranging from a trace in one case to an excess in another. In none of these tests was the amount of the tarry substance increased, which proved conclusively that aldehyde polymerization was not the cause of the formation of this product. Not enough is known of this seemingly ether-polymerization product to warrant further discussion.

An apparent maximum is at 37.5% by weight of aluminum chloride, corresponding to the formula of $\text{AlCl}_3 \cdot 3(\text{C}_2\text{H}_5)_2\text{O}$, for the compound freezing out along the line A-B in Fig. 2. The compound in equilibrium with the solution along line C-D was identified as solid AlCl_3 . The solid freezing out along the line B-C when exposed to the moisture of the air lost HCl by reacting with the water; it was characteristic to, and had physical appearances of AlCl_3 , instead of the etherate. The author was unable to isolate the solid

on account of the fineness of the crystals, and their decomposition when exposed to the air.

Above the maximum temperatures at which the determinations were made, the solutions evaporated with loss of aluminum chloride and ether.

The enormous solubility of aluminum chloride as compared to other salts, may in a way be explained by the peculiar electronic structure that is assigned to it. Aluminum needs two electrons to complete the stable outer layer, or shell of eight electrons. This may be seen in the sketch.



As the aluminum could get the stable number by sharing two electrons of one of the atoms, oxygen, or ether, it would be expected that an etherate of aluminum chloride could be formed. Lewis mentions the formation of a compound of ether and aluminum chloride.³ The data in this paper also evidences this.

It was also noticed that of all the salts, aluminum chloride had the lowest fusion point, but the decreasing solubility of the other salts does not follow the increase of temperature of fusion points.

³Valence & Structure of Atoms and Molecules, G.N. Lewis

SUMMARY

1. The solubilities of different inorganic salts were found.

2. A simple method for the preparation of pure aluminum chloride was devised.

3. A very peculiar curve for the solubility of aluminum chloride in ether was found.

4. Compound formation of aluminum chloride and ether is evident.

5. Small amounts of a viscous, carbonaceous substance are formed when the solution of aluminum chloride is added to water.

6. The large solubility of aluminum chloride might be explained by its electronic structure, and its low fusion point.

7. Much work is yet to be done on this problem, in that the solid that separated out between points B-C could not be identified. A method should be devised whereby the growth and purification of these crystals should be effected.

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