dissolves at the cathode and is precipitated at the anode, two gram-atoms of lead being thus transferred for each Faraday equivalent. In more concentrated solutions the metallic character comes into evidence, and the conductivity is in part electrolytic and partly electronic.

All known chemical compounds may be grouped in the three classes: non-polar, polar and metallic; except in so far as the same compound may in part or at times fall under two of these groups.

POTASSIUM AMMONOMAGNESATE, Mg(NHK)$_2$.2NH$_3$.

By EDWARD CURTIS FRANKLIN.

Introduction.

It will be recalled that the writer and his collaborators have shown that just as zinc hydroxide and lead-hydroxide dissolve in aqueous solutions of the aquo base, potassium hydroxide, to form potassium zincate and potassium plumbite, respectively, so similarly zinc amide and lead imide are acted upon by liquid ammonia solutions of the ammono base, potassium amide, to form potassium ammonozincate,[1] $Zn(NHK)_2.2NH_3$, and potassium ammonoplumbite,[2] $PbNK.2^1/_2NH_3$; thus showing that the amphoteric properties of zinc and lead hydroxides are carried over into the corresponding derivatives of the ammonia system.[3]

It has been a matter of some surprise, however, to find that cadmium and copper, metals whose hydroxides are not known to exhibit amphoteric properties,[4] form respectively potassium ammonocadmiate,[5] $Cd(NHK)_2.2NH_3$, and potassium ammonocuprite,[6] $CuNK_2.4NH_3$, and that thallous nitride, the ammonia analog of the strong base forming thallous oxide, dissolves in liquid ammonia solutions of potassium amide to form potassium ammonothallite,[7] $TlNK_2.4NH_3$. All these compounds, excepting potassium ammonocadmiate, which is amorphous or at best microcrystallin, have been obtained beautifully crystallized and of sharply definit composition, while it will be recalled that, generally speaking, only indifferent success has attended the many efforts to obtain the corresponding aquo compounds from aqueous solution.[8]

[1] THIS JOURNAL, **29**, 656, 274 (1907).

[2] *J. Physic. Chem.*, **15**, 509 (1911).

[3] For a systematic exposition of the writer's ammonia system of acids, bases and salts, cf. *Am. Chem. J.*, **47**, 285 (1912).

[4] The fact is noted, however, that the preparation of a sodium cuprate of the formula $Cu(ONa)_2$, is recorded in the literature, *Z. anal. Chem.*, **9**, 463 (1870). Furthermore, it is well known that without exception metallic oxides are soluble in fused potassium hydroxide.

[5] Prepared by G. S. Bohart in the laboratory of the Leland Stanford Junior University. Experimental results not yet published.
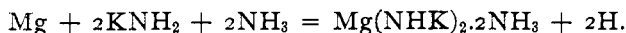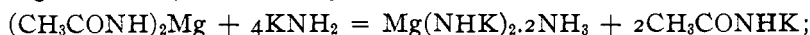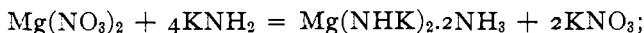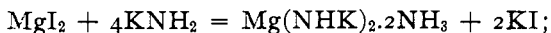
[6] THIS JOURNAL, **34**, 1501 (1912).

[7] *J. Physic. Chem.*, **16**, 682 (1912).

[8] Comey and Jackson, *Am. Chem. J.*, **11**, 145 (1894); Woods, *J. Chem. Soc.*, **97**, 878 (1910); Hantzsch, *Z. anorg. Chem.*, **30**, 289 (1902).

In view, therefore, of the demonstrated existence of the cadmium, copper and thallium compounds just mentioned, it becomes a matter of interest to determin whether the amides, imides or nitrides of other and more positive metals are capable of reacting with potassium amide to form similar potassium salts.

In this paper an account is given of successful attempts to prepare an ammonomagnesate of potassium, a compound in which the rather strongly positive metal, magnesium, plays a part in an ammono salt entirely similar to that played by zinc, lead, and aluminium, in the aquo compounds formed by the solution of the respective hydroxides of these metals in aqueous potassium hydroxide.

A compound of the composition represented by either of the formulas $Mg(NHK)_2.2NH_3$ or $Mg(NH_2)_2.2KNH_2$ has been obtained by the action of an excess of potassium amide in liquid ammonia solution on the iodide and the nitrate of magnesium, on magnesium acetamide and on metallic magnesium in accordance with the reactions represented by the equations:

$$MgI_2 + 4KNH_2 = Mg(NHK)_2.2NH_3 + 2KI;$$

$$Mg(NO_3)_2 + 4KNH_2 = Mg(NHK)_2.2NH_3 + 2KNO_3;$$

$$(CH_3CONH)_2Mg + 4KNH_2 = Mg(NHK)_2.2NH_3 + 2CH_3CONHK;$$

$$Mg + 2KNH_2 + 2NH_3 = Mg(NHK)_2.2NH_3 + 2H.$$

It is interesting to note that, of these reactions taking place in liquid ammonia, the first is between an ammono base and a halogen salt, the second and third between a base of the ammonia system, on the one hand, and a salt of the water system and the ammonia system, respectively, on the other. The fourth equation is interesting for the reason that it represents a reaction in liquid ammonia analogous to the familiar action of potassium hydroxide in water solution on metallic zinc. Fitzgerald[1] has shown that potassium ammonozincate is similarly formed by the action of a liquid ammonia solution of potassium amide on metallic zinc.

The reactions represented by the first three equations undoubtedly take place in two steps,[2] the first of which results in the formation of magnesium amide in accordance with the equation:

$$Mg(CH_3CONH)_2 + 2KNH_2 = Mg(NH_2)_2 + 2CH_3CONHK$$
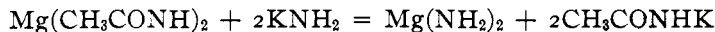
the second of which results in the formation of potassium ammonomagnesate by the interaction of the strong base, potassium amide, and the amphoteric magnesium amide as represented by the equation

$$Mg(NH_2)_2 + 2KNH_2 = Mg(NHK)_2.2NH_3.$$
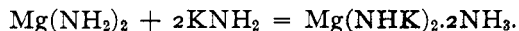
It should be said, however, that the isolation of magnesium amide has not yet been attempted.

[1] THIS JOURNAL, **29**, 660 (1907).

[2] Vide. The preparation of potassium ammonozincate, potassium ammonoplumbite, potassium ammonocuprite and potassium ammonothallite, *loc. cit.*

Anhydrous Magnesium Salts.

Since ammono salts and ammono bases generally[1] are incapable of existence in the presence of water—they are immediately and sometimes vigorously hydrolyzed[2]—it is obvious that the magnesium salts selected for use in the experiments to be described must be free from water. Several methods for preparing such salts have suggested themselves and are described herewith:

First. Upon the theory that magnesium should replace the silver in a liquid ammonia solution of silver nitrate, an attempt was made to pre-

[1] It is to be noted that a considerable number of salts of the stronger ammono acids, potassium phthalimide, for example, have been prepared from aqueous solution; and it is to be especially remembered that the ammono salts of mercury are capable of resisting the hydrolytic action of water, which explains the fact that many mercury derivatives of the acid amides are to be found described in the literature. Mercury so to speak, prefers the ammonia system, it is an azophile.
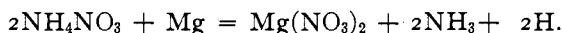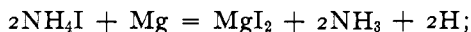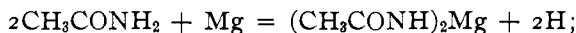
[2] It is interesting to note the contrast in the behavior of ammono acids and ammono bases toward water. In general, though not always, the ammono acids are hydrolyzed with extreme slowness while the ammono bases are vigorously hydrolyzed in the presence of water. As a result of the stability of the one class of substances in the presence of water and the instability of the other, we find that hundreds of ammono acids have been prepared and are recorded in the literature, whereas but few ammono bases are known, and of these the majority are the result of metathetic reactions carried on in water-free liquid ammonia solutions.

The explanation of this difference in behavior toward water seems fairly obvious in view of the following considerations:

First, the acid amides and imides are acids and as such must be assumed to ionize when dissolved in water or in ammonia, considering acetamide an extremely weak acid, for example, in accordance with the equations, $CH_3CONH_2 \rightleftarrows CH_3CONH' + H'$ and $CH_3CONH' = CH_3CON'' + H'$. Ionization in the manner indicated by the equation, $CH_3CONH_2 \rightleftarrows CH_3CO' + NH_2'$ probably takes place also but to so slight an extent that the hydrolysis of acetamide, as represented by the equation $CH_3CONH_2 + H_2O \rightleftarrows CH_3COOH + NH_3$, read from left to right, or the ammonolysis of acetic acid as represented by the same equation read from right to left, takes place at an appreciable speed only at elevated temperatures, or in the presence of some catalizing agent. The tendency, therefore, of aquo acids in the presence of liquid ammonia to ammonolyze to ammono acids, or of ammono acids in the presence of water to hydrolyze to aquo acids is generally very slight. It must be remembered, however, that certain ammono acids, such as silicon amide and boron amide (Lengfeld, *Am. Chem. J.*, **21**, 531 (1899)), for example, are immediately and completely hydrolyzed in the presence of water, a behavior which may be explained on the assumption that their ionization as bases is much more marked than is the case with the acid amides generally.

Second, the ammono bases, when dissolved in liquid ammonia, dissociate into metallic cations and amide anions as represented, for example, in the case of potassium amide, by the equation $KNH_2 \rightleftarrows K' + NH_2'$. Now since it has been shown by Franklin and Kraus (*Am. Chem. J.*, **23**, 285 (1900) and THIS JOURNAL, **27**, 101 (1905)) that the specific conductance of pure liquid ammonia certainly lies well below that of pure water it must be concluded that the ionization constant of ammonia is considerably less than that of water. It therefore follows that if water is added to a liquid ammonia solution of potassium amide the NH_2 ions will disappear and give place to OH ions,

pare anhydrous magnesium nitrate by bringing together clean magnesium ribbon and a liquid ammonia solution of silver nitrate. For some reason the metal was practically unattacked by the silver nitrate solution.

Second. Failing to accomplish the replacement of the silver by simple contact of metallic magnesium with silver nitrate solution, the desired reaction was brought about by passing a current of electricity through the solution between an anode of magnesium and a cathode of platinum. Metallic silver separated at the cathode and a deposit of difficultly solu ble crystals, presumably of magnesium nitrate, was formed in the cell. The explosion of the reaction tube brought the experiment to an end and it was not repeated for the reason that it was found more convenient to prepare the magnesium salts required by the method described in the succeeding paragraph.

Third. The writer has from time to time called attention to the fact that the acid amides, and the ammonium salts of halogen and of oxygen acids in solution in liquid ammonia, exhibit certain characteristically acid properties;[1] that in point of fact such solutions are to be looked npon as liquid

that is, potassium amide will be hydrolyzed to potassium hydroxide. The equilibrium constants for the equations $KNH_2 \rightleftharpoons K' + NH_2'$ and $KOH \rightleftharpoons K' + OH'$ are both high and therefore of no practical influence on the system. The dissociation constant of water, that is of the equation $H_2O \rightleftharpoons H' + OH'$, although very low, must be, as explained above, much higher than the constant for the equation $NH_3 \rightleftharpoons NH_2' + H'$ with the inevitable result that potassium amide and water react with each other to form potassium hydroxide and ammonia as represented by the scheme:

$$KNH_2 \rightleftharpoons K' + NH_2'$$
$$+ \qquad +$$
$$H_2O \rightleftharpoons OH' + H'$$
$$\updownarrow \qquad \updownarrow$$
$$KOH \qquad NH_3$$

It is interesting also to consider for a moment the behavior of salts of ammono acids toward water. It is a property of acids generally to ionize when dissolved in ionizing solvents and the ammono acids are no exception to this rule. While, on the one hand, many acid amides have long been recognized as having acid properties—aqueous solutions of potassium benzenesulfonnitramide, $C_6H_5SO_2NHNO_2$ for example, are stable in the presence of water—other amides on the other hand, acetamide for example, are possessed of such extremely weak acid properties that their salts are completely hydrolyzed in the presence of water and they are not generally recognized as acids. Ammonia, however, with its lower dissociation constant is not so powerful an ammonolyzing solvent as is water a hydrolyzing solvent, whence it follows that acid amides which are unrecognizable as acids in aqueous solution have been found to show distinct acid properties in ammonia solution (cf. *Am. Chem. J.*, **23**, 302 (1900); **28**, 83 (1902); **42**, 285 (1912), and THIS JOURNAL, **27**, 820 (1905)). In accordance with these considerations we find that many salts of the stronger ammono acids have been made in aqueous solutions whereas the salts of the weaker ammono acids can be made only in the absence of water, and most successfully, as the results of the writer and his collaborators have shown, by working in liquid ammonia solutions.

[1] Cf. *Am. Chem. J.*, **47**, 285 (1912).

ammonia solutions of ammono acids, halogen acids and oxygen acids, respectively. Among the properties of such solutions is their capability of attacking metallic magnesium with the evolution of hydrogen and the formation of the respective magnesium salts as represented by the equations:

$$2CH_3CONH_2 + Mg = (CH_3CONH)_2Mg + 2H;$$

$$2NH_4I + Mg = MgI_2 + 2NH_3 + 2H;$$

$$2NH_4NO_3 + Mg = Mg(NO_3)_2 + 2NH_3 + 2H.$$

The first of the above equations was established some years ago by Franklin and Stafford,[1] who obtained well crystallized specimens of magnesium acetamide, or magnesium ammonoacetate, of the composition represented by the formula $(CH_3CONH)_2Mg.4NH_3$. The salt is but slightly soluble in liquid ammonia.

That magnesium iodide and magnesium nitrate are formed in accordance with the second and third of the above equations by the action of liquid ammonia solutions of ammonium iodide and ammonium nitrate, respectively on metallic magnesium, is shown by the data recorded herewith.

Magnesium Nitrate Hexammonate, $Mg(NO_3)_2.6NH_3$.—When metallic magnesium is covered with a liquid ammonia solution of magnesium nitrate, evolution of hydrogen begins immediately and the metal gradually goes into solution. The salt formed is but slightly soluble in liquid ammonia and usually begins to crystallize from the solution before all of the metal has disappeared.

A specimen of the well crystallized product weighing 0.3511 g. was submitted to analysis. One-fourth of the specimen gave 0.0430 g. $MgSO_4$, another fourth gave 0.0360 g. NH_3, and 0.0120 g. NH_3, resulting from the reduction of the nitrate ion.

Calculated for $Mg(NO_3)_2.6NH_3$: Mg, 9.7; NO_3, 49.6; NH_3, 40.7.
Found: Mg, 9.8; NO_3, 49.9; NH_3, 41.0.

Ammonated Magnesium Iodide, $MgI_2.6NH_3$.—One-fourth of the specimen of the white, practically insoluble product of the action of liquid ammonia solution of ammonium iodide on metallic magnesium, dried *in vacuo*, at laboratory temperature and weighing 1.0974 g., was found to contain 0.0781 g. NH_3, another fourth gave 0.0893 g. $MgSO_4$, and a third-quarter gave 0.3366 g. AgI.

Calculated for $MgI_2.6NH_3$: Mg, 6.4; I, 66.9; NH_3, 26.8.
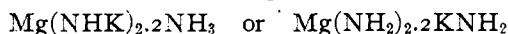Found: Mg, 6.6; I, 66.5; NH_3, 26.7.

Detailed accounts of the manner in which these water-free salts were prepared and used for the purpose of this investigation are given in the following pages.

[1] *Am. Chem. J.*, **28**, 83 (1902).

Potassium Ammonomagnesate.

Five specimens of a salt of the composition represented by the formula

$$Mg(NHK)_2.2NH_3 \quad or \quad Mg(NH_2)_2.2KNH_2$$

have been prepared in accordance with the methods described in general terms above.[1] The manipulative and analytical details are given herewith.

Preparation I.—In this experiment an old specimen of magnesium iodide made by the action of ammonium iodide on metallic magnesium, and which had remained for several years in contact with a liquid ammonia solution of ammonium iodide was used. The salt, which is practically insoluble,[2] was transferred[3] in suspension in liquid ammonia to one of the familiar

[1] P. 1456.

[2] Franklin and Kraus, *Am. Chem. J.*, **20**, 828 (1898), recorded magnesium iodide as being soluble. Their observations were made on the hydrated salt. The solubility of the hydrated salt has been confirmed.

[3] Since the method which has been used for transferring liquid ammonia solutions to these reaction tubes is not entirely obvious, it seems worth while to give here an outline
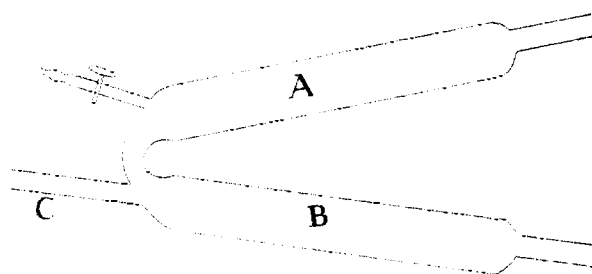
Fig. 1.

of the process. In this particular case the operations were carried on as follows: Into one leg, A, of the familiar apparatus provided with the tubulure C (Fig. 1), a quantity of metallic potassium for supplying the potassium amide required for the reaction is introduced and the ends of the two legs A and B closed before the blow pipe. Immersing leg A in a refrigerating bath, the desired quantity of liquid ammonia is distilled upon the metallic potassium, after which the apparatus is set aside until the metal is converted into potasssium amide. In the meantime the tube containing the solution which is to be mixed with the potassium amide solution is cooled in a bath of liquid ammonia, opened at the upper end and provided with a long, slim delivery tube by drawing out the open end of the tube before the blow pipe. Then with the two legs of the reaction tube immersed in liquid ammonia, the neck of tube D is opened and inserted through the tubulure C of the reaction tube as shown in Fig. 3. On turning the tube D carefully
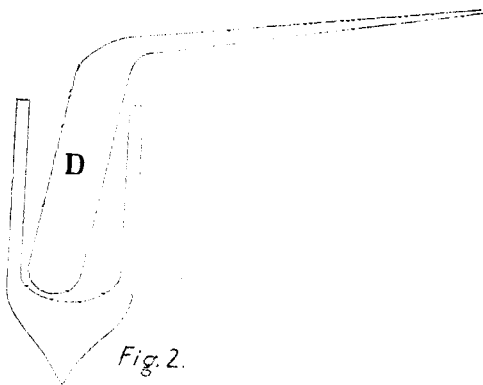

Fig. 2.

about the axis of its neck the contents will flow quietly into leg B of the reaction apparatus. The tube D is then withdrawn from the tubulure C, and the latter is closed

two-legged reaction tubes where it was mixed with an excess of potassium amide. The first portions of magnesium iodide dissolve in the potassium amide solution but their solution is followed after a short interval by the separation of minute, colorless crystals which fill the solution and give to the contents of the tube a peculiar nacreous luster. After standing for a time the precipitate was washed, hydrolyzed by means of water vapor, then dissolved in dilute sulfuric acid and removed from the reaction tube for analysis by methods already described.[1]

The preparation dried *in vacuo* at laboratory temperature weighed 0.4343 gram. One-fourth of the solution was evaporated to dryness and the residue was ignited with ammonium carbonate. The mixture of $MgSO_4$ and K_2SO_4 thus obtained weighed 0.1921 gram and was found to contain 0.1147 gram K_2SO_4. Another fourth gave 0.0350 gram N.

Preparation II.—A specimen of ammonoacetate of magnesium was before the blow pipe. Then after removing the Dewar tube baths and allowing the apparatus and its contents to warm up to laboratory temperature, the succeeding operations, which have already been described elsewhere, are proceeded with. In order to prevent all contamination with moisture the tube D is not opened until the

Fig. 3.
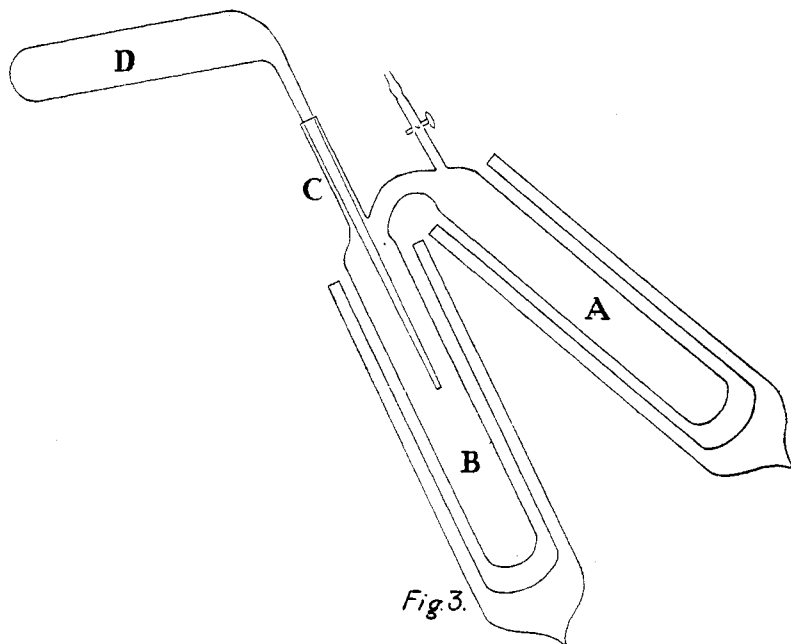
operator is sure that the pressure in the tube is slightly greater than outside; moreover, during the operations of transferring the solutions and sealing off the tubulure C a current of dry ammonia gas is kept passing through the apparatus, entering by way of the stopcock tube and escaping through the tubulure C. The apparatus ready for bringing together the magnesium iodide suspension and the solution of potassium iodide is shown in Fig. 4.

[1] *J. phys. Chem.*, **15**, 509 (1911).

prepared by introducing acetamide and metallic magnesium, respectively, into the two legs of the reaction tube, distilling a proper quantity of ammonia upon the acetamide, pouring the solution thus obtained upon the metallic magnesium and setting the apparatus aside until the evolution of hydrogen had ceased and a crop of well formed crystals had separated from the solution. The magnesium acetamide thus obtained as a mass of well formed crystals, but slightly soluble in liquid ammonia, was transferred from the excess of magnesium to the second leg of the reaction tube, the unused metal was removed from the apparatus and sufficient metallic potassium introduced to form a liberal excess of potassium amide. Upon pouring the solution of potassium amide upon the magnesium acetamide the crystals of the latter were observed to disintegrate, with the result that after a time the contents of the tube took on much of the appearance shown by the precipitate obtained in the preceding experiment.
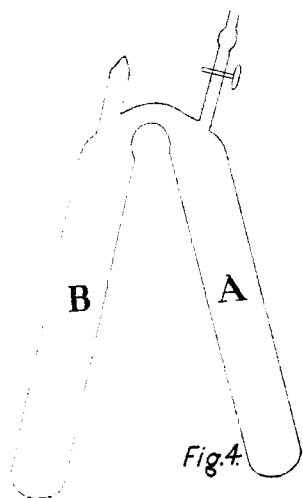
Fig. 4.

For analysis the specimen was hydrolyzed by the action of water vapor and then dissolved in dilute hydrochloric acid. In one-half of the solution the potassium and magnesium were separated and determined by the method of Berzelius with mercuric oxide, with the result that 0.0287 gram MgO and 0.0977 gram KCl were obtained. The other half of the solution gave 0.0363 gram N. The specimen weighed 0.2220 gram.

The high result for nitrogen and low result for potassium are probably due to the demonstrated presence of water in the acetamide used. It has been calculated that the analytical results given above would be given by a mixture of 0.07 mol. $Mg(OH)_2$ with 0.93 mol. $Mg(NHK)_2.2NH_3$.

Preparation III.—Magnesium iodide, prepared in one leg of the reaction tube by the action of ammonium iodide solution on metallic magnesium, was poured into an excess of potassium amide solution contained in the second leg.

The resulting product dried *in vacuo* at 20° weighed 0.2358 gram. One-half of the specimen gave 0.0284 gram MgO and 0.1052 gram KCl. One-fourth of the specimen gave 0.0189 gram N.

Preparation IV.—One attempt was made to prepare the same magnesium compound by treating the product of the action of ammonium nitrate solution on metallic magnesium with an excess of potassium amide. The specimen obtained was found to contain 19.3% magnesium, 29.7% nitrogen and 41.1% potassium, thus showing that although much contaminated by impurities the product found was in fact potassium ammonomagnesate.

Preparation V.—A fifth specimen of potassium ammonomagnesate was prepared by the action of a liquid ammonia solution of potassium amide on metallic magnesium. When potassium amide solution was poured upon the spiral of metallic magnesium, the solution immediately became intensely blue and at the same time an evolution of gas was observed. No test was made to prove the identity of this gas. It was assumed to be hydrogen. In the course of some hours the metal had completely disappeared and in its place was found a colorless, granular mass of difficultly soluble ammonomagnesate. After a few washings the salt was pure and ready for analysis.

One-half of the specimen which weighed 0.7415 gram dried *in vacuo* at laboratory temperature gave 0.0888 gram MgO and 0.3290 gram KCl. One-fifth gave 0.0500 gram N.

From the analytical data recorded above the percentage composition of four specimens of the salt has been calculated with the following results:

	Calculated for Mg(NHK)₂.2NH₃.	Found.			
		I.	II.	III.	V.
Mg	14.5	14.4	15.3	14.5	14.4
N	33.7	32.2	32.6	33.5	33.7
K	47.0	47.5	46.1	46.9	46.5
H	4.8	..	..	..	..

Obviously the action of potassium amide in excess on magnesium salts or upon metallic magnesium results in the formation of a magnesium compound which is strictly analogous to potassium ammonozincate, the first representative of this class of compounds to be prepared. It may be given either of the formulas Mg(NHK)₂.2NH₃ or Mg(NH₂)₂.2KNH₂.

Properties of Potassium Ammonomagnesate.

Potassium ammonomagnesate is obtained as a fine, crystallin powder which is but very slightly soluble in liquid ammonia. Liquid ammonia solutions of ammonium iodide, a halogen acid, of ammonium nitrate, an aquo acid, and of acetamide, an ammono acid convert it respectively into magnesium iodide, magnesium nitrate and magnesium ammonoacetate. It is rapidly hydrolyzed by the action of liquid water or water vapor to magnesium hydroxide, potassium hydroxide and ammonia. It has been heated to 100° without losing ammonia. It is not explosive.

The writer is under obligation to Mr. H. M. Miller for the major portion of the analytical work recorded in this paper.
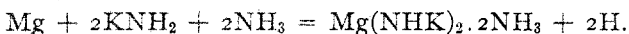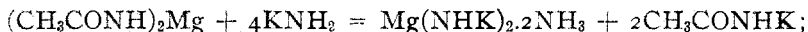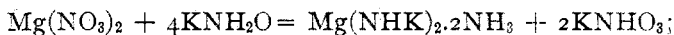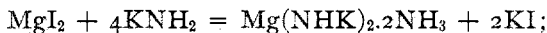
Summary.

In this paper are described the methods used for the preparation of potassium ammonomagnesate, a new salt belonging to the writer's ammonia system of acids, bases and salts.

The salt has been prepared by the action of potassium amide in liquid ammonia solution, first on a halogen salt of magnesium, second, on an aquo

salt of magnesium, third, on an ammono salt of magnesium, and fourth on metallic magnesium itself.

The reactions studied were represented by the equations:

$$MgI_2 + 4KNH_2 = Mg(NHK)_2.2NH_3 + 2KI;$$
$$Mg(NO_3)_2 + 4KNH_2O = Mg(NHK)_2.2NH_3 + 2KNHO_3;$$
$$(CH_3CONH)_2Mg + 4KNH_2 = Mg(NHK)_2.2NH_3 + 2CH_3CONHK;$$
$$Mg + 2KNH_2 + 2NH_3 = Mg(NHK)_2.2NH_3 + 2H.$$

The compound may be formulated either as a salt with ammonia of crystallization, as has been done above, or as a molecular compound of magnesium amide and potassium amide, $Mg(NH_2)_2.2KNH_2$.

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A STUDY OF THE ACTION OF SULFUR MONOCHLORIDE ON CERTAIN MINERALS.

BY HIRAM STANHOPE LUKENS.

Two fields in which sulfur monochloride is now proving a most helpful reagent are in the decomposition of minerals and in the preparation of anhydrous chlorides. Sulfur monochloride was first used for the decomposition of minerals by E. F. Smith,[1] in 1898, when it was employed with a number of naturally occurring sulfides and sulfarsenides. The minerals wolframite, columbite and scheelite were also decomposed. Anhydrous chlorides of arsenic, antimony and bismuth were prepared by Oddo and Serra[2] by the use of sulfur monochloride. Hall[3] repeated the decomposition of columbite, decomposed chromite and converted a number of metallic oxides to chlorides, notable among the latter being the preparation of the chlorides of columbium and tantalum. He also observed that the oxides of silicon and boron resisted the action of the reagent. Bourion[4] repeated the decomposition of the minerals scheelite and wolframite and prepared a number of anhydrous chlorides. Hicks,[5] working in this laboratory, employed sulfur monochloride in the decomposition of the minerals fergusonite, euxenite and samarskite and in the decomposition and analysis of aeschynite. By this means columbium, tantalum, titanium and tungsten were separated completely from silica and the rare earths.

Hicks had noticed that traces of silicates present in some of the minerals

[1] THIS JOURNAL, 20, 289 (1898).
[2] Gazz. chim. ital., [2] 29, 355 (1899).
[3] THIS JOURNAL, 26, 1243 (1904).
[4] Ann. chim. phys., 20, 547 (1910).
[5] THIS JOURNAL, 33, 1492 (1911).