AMMONIA AND ITS COMPOUNDS

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THEIR MANUFACTURE AND USES

BY

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TRANSLATED FROM THE FRENCH BY

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THIRTY-TWO ILLUSTRATIONS

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TRANSLATOR'S PREFACE.

AMMONIA and its Compounds are products of considerable use and value in the industrial arts, and of late their application to various purposes—manufacture of ice, agriculture, etc.—has increased very much, so that chemical manufacturers have been induced to pay more attention to methods and means of producing them in a comparatively pure condition on a large scale, and to seek new sources of raw material for their preparation. Under these circumstances, and seeing that there has been but little published in England about the manufacture of ammonia, the publication of the present translation of PROFESSOR CAMILLE VINCENT'S little treatise on Ammonia will not be without interest and value. The Author, who is Professor of Chemistry at the Paris Central School of Arts and Manufactures, describes very clearly the methods which are largely and successfully practised in France for the preparation of ammonia and ammonium salts from various waste and raw products. The description is materially enhanced in value by the many illustrations of the plant that is used in conjunction with these processes which accompany it.

A new Index has been compiled to facilitate reference.

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AMMONIA AND ITS COMPOUNDS.

CHAPTER I.

GENERAL CONSIDERATIONS.

§ 1. Various Sources of Ammoniacal Products.

Nitrogen, in the condition of an ammoniacal salt or of a nitrate, contributes to the nutrition of plants. Plants convey the nitrogen to the organs of animals to form compounds of a higher order of complexity. Exposed to the action of a high temperature, or simply allowed to undergo putrid fermentation, nitrogenous organic substances produce ammonia, which is easily recovered. The organs of plants and animals constitute, therefore, the natural source of ammoniacal products. The excrements of man and animals, which are rich in nitrogenous substances, also afford ammonia in abundance, both for agricultural and industrial requirements. Down to the end of the last (eighteenth) century there was employed in Europe only an unimportant amount of ammonia, which was obtained from India and Egypt in the form of carbonate or chlorhydrate. Crude ammonium chloride, or *sal ammoniac*, was obtained by burning the dung of animals, especially camels, fed upon saline vegetation. The poor people of the banks of the Nile were in the habit of gathering this dung and drying it in the sun, for the purpose of using it as fuel during the winter. The abundant soot obtained from these substances was collected and submitted to slow sublimation in glass flasks. Cakes of impure sal ammoniac, coloured grey by tarry products, were thus obtained, and were sent into commerce. This primitive manufacture is no longer in existence; more abundant sources of ammonia having been discovered, and simple and economical processes of preparing ammoniacal salts in a high state of purity having been brought into use. The fermentation of human dejections, the calcination of animal matters, and the distillation of coal, are the principal sources from which agriculture and manufactures at the present day derive the enormous quantity of ammoniacal compounds which they require.[1] A certain quantity is also extracted from the water of the Tuscan lagoons, and from the products of the distillation of the residual wash from beet-root spirit, but these sources are at present limited.

The increase in the consumption of ammonia has doubled its value in twenty years; especially of late, the manufacture of soda by the ammonia process has particularly contributed to raise the price. We learn, in fact, from an able work by Walter Weldon, on the *Soda Industry*, that by this process there is annually manufactured 163,000 tons of soda, consuming 9000 tons of ammonium sulphate. In spite of the great extension of this manufacture of soda, which cannot but still further increase, we may feel assured that the price of ammonia instead of rising will now diminish, thanks to the efforts which have recently been made to recover the enormous quantities which have hitherto been lost. Several years ago M. Carvès started in France, and more recently in England, coke ovens which, whilst yielding coke of good quality, allow of the recovery of the tar and ammonia. The ovens of this description at Tamaris, Terre-Noire, and Bessèges, produce in the aggregate about 300 tons of coke per day, with 6 tons of tar and 2 to 2½ tons of ammonium sulphate. This method of manufacture has not made the progress in France which might have been hoped, although it has given satisfactory results. Recently John Jameson of Newcastle-on-Tyne has worked out a process of condensation, applicable at a low cost to existing coke ovens. This new process has been working for a year at the factory of Mr. Hugh Lee Pattison at Pelling, and gives most satisfactory results. About 5·4 kilos. (11·9 lbs.) of ammonium sulphate is obtained from 1 ton

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[1] From documents published in 1882, there was produced in the United Kingdom 1,000,000 tons of ammoniacal gas liquor, resulting from the distillation of 9,000,000 tons of coal, and yielding about 25,000 tons of ammonium sulphate. The Paris Gas Company alone distilled 927,104 tons of coal in 1882.

of Northumberland coal, and there is reason to hope that this yield will be increased as the apparatus is improved. The yield of coke and its quality are in no way affected by this new process, which will soon be adopted by a large number of manufacturers.

Whilst England has been occupied in collecting ammonia from the coke ovens, Scotland has undertaken the condensation of ammonia from blast furnaces consuming coal. This idea was due to Mr. W. Ferrie; it has recently been applied to two of the six blast furnaces at Gartsherrie, and will shortly be applied to the remainder. The quantity of ammonia collected daily at Gartsherrie represents 900 kilos. (1980 lbs.) of ammonium sulphate, or a yield of 9 kilos. (19·8 lbs.) per ton of coal burnt. The decomposition of the coal takes place under these conditions at a temperature intermediate between that of the coke ovens and that of the gas retorts. The results obtained at Gartsherrie have induced other factories to endeavour to solve the same problem by different methods. It may therefore be hoped that the 120 Scotch blast furnaces which consume coal will be furnished in the near future with the apparatus for the condensation of ammonia. Each of these 120 blast furnaces consumes 50 tons of coal daily; the total yearly consumption is 2,190,000 tons, which would yield about 20,000 tons of ammonium sulphate, worth at least £400,000. This enormous quantity of ammonia barely represents one-tenth of that which might be obtained from the products of the English coke ovens, in which 20,000,000 tons of coal are annually converted; admitting a yield of 9 kilos. of ammonium sulphate per ton, already obtained at Gartsherrie, 180,000 tons of this product should be recovered. This brief account shows that the consumption of ammonia may be largely increased, perhaps many times multiplied, without any fear of a deficiency of this substance for technical purposes. It may further be added, that the combustion of coal is the most important source of ammonia which can be turned to account, at least in the majority of factories, as it already is in several of them.

Independently of these various sources of ammonia, attempts have for some time been made to produce this substance from atmospheric nitrogen. We will not here refer to the numerous and able researches which have been made on this subject, and the very interesting results which have already been obtained; but although this problem has not yet been solved from an industrial point of view, it may be anticipated that it will one day be solved, since patient and able investigators are attacking it vigorously. The production of ammonia would then be unlimited, to the great benefit of agriculture and manufactures.

§ 2. Human Urine as a Source of Ammonia.

The kidneys of the mammalia secrete a liquid, urine, which collects in the bladder, to be subsequently expelled from the body. This liquid is essentially a product of excretion, containing in solution mineral and organic matters, which the renal glands remove continuously from the blood. The chief substances which it contains are urea, common salt, and earthy phosphates. Urea is the final product of the oxidation of nitrogenous matters in the organism; these matters appear to decompose into substances free from nitrogen, such as glycogen, inosite, and cholesterin, and nitrogenous substances such as urea, creatinine, xanthine, and uric acid, which are found in all tissues, and are capable of transformation into urea by decomposition and oxidation. The urea is transferred to the blood, and continuously thrown out by the kidneys.

The urine secreted by a healthy man is a limpid yellow liquid, varying in density from 1·005 to 1·030, but more usually from 1·015 to 1·025; of bitter and saline taste, due principally to the common salt and urea, and with a peculiar slight odour. It has generally an acid reaction, but may be neutral or even alkaline. It contains all the mineral and organic substances which have been taken in excess of the bodily requirements, and are in consequence not assimilable, and all the products of the metamorphosis of tissue. It contains, besides urea and common salt, small quantities of uric and hippuric acids, creatinine, xanthine, colouring matters, and undetermined extractive substances, together with alkaline sulphates, alkaline and earthy phosphates, and, lastly, a trace of silica.

The following table gives the normal composition of the urine of an adult man weighing 65 kilos. (143 lbs.), taking a normal diet and moderate exercise:—

Substances.		Average daily quantity.	Average quantity per kilo.
Water		1238·07 grms.	952·36 grms.
Organic matters— Per day, 41·74 grms. Per kilo. 32·11 ,,	Urea	31·55 ,,	24·27 ,,
	Uric acid	0·52 ,,	0·40 ,,
	Hippuric acid	1·30 ,,	1·00 ,,
	Creatine and creatinine	1·30 ,,	1·00 ,,
	Xanthine	0·006 ,,	0·004 ,,
	Extractive and colouring matters	7·065 ,,	5·41 ,,
	Fatty acids Glucose Phenol Mucine	Traces.	Traces.
Mineral matters— Per day, 20·19 grms. Per kilo. 15·53 ,,	Sodium chloride	13·30 grms.	10·231 grms.
	Alkaline sulphates	4·03 ,,	3·10 ,,
	Calcium phosphates	0·408 ,,	0·313 ,,
	Magnesium ,,	0·591 ,,	0·455 ,,
	Alkaline ,,	1·86 ,,	1·431 ,,
	Silica, iron, ammonia Gases: Oxygen Nitrogen Carbonic acid	Traces.	Traces.
		1300·00 grms.	1000·00 grms.

These proportions are liable to considerable variations. Normal urine may contain 20 to 35 grms. of urea per litre; the quantity produced by a man varies from 22 to 37 grms. in twenty-four hours. The quantity of uric acid varies from $\frac{1}{30}$ to $\frac{1}{60}$ of the weight of the urea. Normal human urine is limpid when fresh; if left it often becomes slightly turbid on cooling, owing to the deposition of flocks of mucus and a trace of phosphates, in consequence of the loss of part of the carbonic acid. It then becomes acid, and its colour deepens, whilst uric acid and urates are deposited. It remains in this condition for some time; it then becomes neutral, and eventually strongly alkaline, whilst abundance of earthy phosphates, and oxalates, ammonio-magnesium phosphate, and ammonium urate separate. The urea has then become ammonium carbonate, by combining with the elements of water: $COH_4N_2 + 2H_2O = (NH_4)_2CO_3$.

If fresh urine, after boiling, is kept in a Pasteur flask to which all access of atmospheric dust is prevented, it may be kept unaltered for an indefinite time.

M. Van Tieghem has proved the constant presence in urine undergoing ammoniacal fermentation of a torula, consisting of minute globules connected into chains, which possesses the property of converting urea into ammonium carbonate. The rapid transformation of the urea is probably due to this organism. Since the

22 to 37 grms. of urea yielded daily by a man will produce 12·5 to 21 grms. of ammonia, it follows that the urine from a large town constitutes an important source of ammonia. It should, however, be noticed that a large part of the urine of towns is sent direct to the drains, either by the public urinals, or by movable close-stools which constantly discharge into the drains. For this reason urine is used to a very limited extent for the manufacture of ammonia. Paris is one of the towns where this utilisation is of most importance. The matters removed from the fosses are transferred to settling reservoirs, where the solid products are allowed to settle, for subsequent drying, whilst the supernatant liquid, which is known as "eau vanne," passes into other reservoirs to finish its fermentation. At the end of three or four weeks, these liquids, which consist essentially of putrefied urine, are used for the manufacture of ammoniacal products.

CHAPTER II.

EXTRACTION OF AMMONIACAL PRODUCTS FROM SEWAGE.

§ 1. Preliminary Treatment of Excreta in the Settling Tanks.

The excreta removed from the fosses, after the addition of metallic salts, iron sulphate, zinc chloride or sulphate, for the decomposition of the ammonium sulphydrate which they contain, are transferred to large covered tanks. In Paris there are daily extracted upwards of 2200 c.m. of excreta, 300 of which are obtained from the movable close-stools. The Lesage and the Parisian Sewage and Manure Companies convey these substances by night in cylindrical trucks, which are emptied into hermetically closed sheet-iron tank-barges, which are then towed to the works at Billancourt, Aubervilliers, and Maison-Alfort for the Lesage Company, or formerly to Nanterre, before the closing of the factory of the Parisian Company. Steam pumps lift the contents of the barges and discharge them by subterranean pipes into huge reservoirs excavated in the earth, covered with tile roofs, and made watertight by puddling with clay. Since the closing of the Nanterre works, the Parisian Company has been provisionally authorised to treat the sewage in the common sewers of Bondy. The sewage sent to the settling tanks of La Villette is discharged by cast-iron pipes into the tanks established at Bondy. More than half the sewage of Paris is treated by these two companies.

The average sewage sent to the settling tanks contains from 85 to 95 per cent. of liquid; left to itself, it soon deposits a black mud, and the more or less turbid *eau vanne* can be run off and allowed to ferment. The fresh sewage contains nitrogen in the state of uric acid, creatinine, albuminoid substances, and ill-defined compounds, and chiefly as urea. By fermentation, which takes place partly in the fosses, the urea is gradually converted into ammonium carbonate by fixing the elements of water; there are also produced ammonium sulphide, sulphate, and chloride, and ammonio-magnesium phosphate. At the same time a number of complex products are formed, notably compound ammonias, the presence of which, although in small proportion, renders the smell from the settling tanks very unpleasant. Neutral substances of disagreeable odour are also formed, which explains the difficulty of finding a simple and economical method for the complete deodorisation of sewage. Up to the present time it may be said that no satisfactory mode of destroying the emanations from sewage has been attained, except by agents such as chlorine, bromine, and nitrous gases, which decompose or greatly modify all odorous organic compounds; but these are costly.

The experiments made at the "Hôpital de la Pitié" by MM. Bapst and Girard on the use of nitrous sulphuric acid, have given very sharp and conclusive results; it is desirable that they should be repeated in practice.

It is always observable that sewage exposed to the air in the settling tanks, loses a notable proportion of its ammoniacal nitrogen, which passes off either as nitrogen or ammonium carbonate, or is converted into nitric acid by a special fermentation, as has recently been shown by the researches of Schlœsing and Muntz.

At the end of three or four weeks, the fermentation being completed, the liquid is passed to the apparatus for the extraction of the ammonia. The deposit in the tanks retains much liquid; this is dried and powdered. In many cases this mud is simply allowed to dry spontaneously, and requires several years, together with costly labour, whilst noxious affluvia are produced and a serious loss of nitrogen takes place. Thus, sludge, which after rapid drying would contain 2·5 to 3 per cent. of nitrogen, will retain at most 1·5 per cent. after exposure to the air for two years. The rapid desiccation of the sludge is therefore a matter of importance, and numerous attempts have been made to solve this problem in a manner which shall be both economical and conformable to public hygiene.

The Lencauchez Process.—In 1858, Lencauchez, a civil engineer of Paris, endeavoured to solve the problem of the rapid conversion of sewage into solid manure and ammonium sulphate. The following is a short account of the processes which he employed for dealing with the sewage accumulated in the immense settling tanks at Bondy. The plan first adopted had for its object—(1) to run off the liquid portion of the sewage; (2) to dredge up the sludge, dry it in stoves traversed by warm air mixed with the products of combustion from special furnaces, to obtain it in the form of powder (*poudrette*); (3) to distil the liquid portion alone, for the extraction of the volatile salts of ammonia; (4) to continue the distillation after adding lime to decompose the fixed salts of ammonia, and then run the residues into the drains. This process, whilst it allowed the extent of the tanks at Bondy to be reduced, did not satisfy hygienic requirements, either with regard to the effluvia or to the quality of the effluent liquids, which, whilst containing only a small amount of nitrogen, were rendered turbid by solid matters thrown up by the fermentation which took place in the tanks. About 1868, Lencauchez altered his process completely. He observed that the liquid portion of the sewage could only be run off conveniently when fermentation had ceased, since the bubbles of gas kept permanently in suspension those organic matters whose density was the same as that of the liquid, allowing only the heavy earthy matters to settle. He observed further, that boiling, which coagulates albuminous substances and destroys ferments, permits the rapid decantation of the liquid; and, lastly, that the action of lime, by destroying the ferments and saturating the acids, assists subsidence still further. If the sewage is distilled with an excess of milk of lime, lime salts are formed, which, mixed with the organic matters, form a deposit of greater density than the liquid, and from the absence of gases and ferments, and the effect of the excess of lime in preventing fresh fermentation, settling takes place rapidly. In 1867, Chevalet showed that after treatment with lime and distillation of the ammonia, the solid and liquid portions of the sewage could be separated without the loss of more than 1 or 2 per cent. of their nitrogen. This led Lencauchez to propose that the treatment of sewage should be commenced by distillation with lime. He took out a patent for this method on the 3rd March 1869, and a supplementary one on the 24th May 1870. The new method consisted—(1) in distilling the entire sewage in a vacuum with lime for the recovery of the ammonia; (2) separating the solid and liquid portions by settling; (3) drying the sludge. The following is a description of the process as submitted to a committee of visitation on the 8th June 1872:—

The sewage, on arriving at the establishment, is to be put through disintegrators to render it homogeneous. These are closed vessels through which a current of air is driven by a fan into a sulphuric acid tower for the fixation of the ammonia. The uncondensable gases are destroyed by passing through a furnace. The sewage issuing from the disintegrators passes through a sieve, and the coarser particles retained by the sieve are collected in waggons and burnt in special furnaces. The liquids flowing from the sieves are to be received in storage tanks of impermeable brickwork, vaulted over to prevent the escape of effluvia, and ventilated like the disintegrators. The treatment of the contents of these tanks will be examined in greater detail in § 2. The exhausted liquids, after boiling with lime, were to be raised by a pump into a transference apparatus, where they would part with most of their heat to fresh sewage. They would then flow into settling tanks, where they would be treated with a small quantity of aluminium sulphate (about 50 grs. per cubic metre), to assist the settling of the suspended matters and allow of the rapid decantation of the clear liquids, which would then be allowed to flow away. The sludge at the bottom of the tanks would then be passed through filter presses to obtain the solid matters in the form of cakes, which are to be dried and powdered, whilst the liquid returns to the tanks. The stoves for drying the press cakes are traversed by the products of combustion of the furnaces, to which enough air is added to reduce their temperature to 110° C., so as not to char the organic matter of the cakes. During the drying the excess of lime will be carbonated.

This brief sketch of the subject shows that M. Lencauchez proposed the following improvements in the treatment of sewage:—

(1) Distillation in vacuo, avoiding the formation of a large proportion of odoriferous matters, which form only at a high temperature; (2) treatment with lime, to remove ammoniacal products at once, and allow a ready separation of the solid and liquid matters by arresting fermentation and coagulating albuminous substances;

(3) the use of filter presses, to collect the solids in the form of cakes, and allow of rapid drying in a current of heated gas; (4) the use of closed and ventilated apparatus, to prevent the dispersal of the exhalations and allow the uncondensable gases to be destroyed by passing through furnaces. This ingenious process has, however, never been practically brought into use.

We shall not allude to all the attempts which have since been made to treat sewage with rapidity, as that would take us from our subject; we will only describe two processes, namely, that of Bilange, which is in use in Bondy, and that of Kuentz, which is chiefly employed at the Versailles works. The first of these processes permits the treatment of entire sewage, and allows the separation of practically the whole of the liquid portion, from which the ammoniacal products are then extracted; the second divides the sewage into clear effluent, and solid cakes containing assimilable phosphates.

The Bilange Process.—M. Bilange has sought to produce a manurial product of immediate utility, and to allow only inodorous and imputrescible matters to pass away into the air or the effluent liquids. His process is a satisfactory one from a hygienic standpoint, and he extracts from the sewage nearly the whole of the fertilising materials which it contains.

The treatment comprises three distinct operations—

1. Treatment by chemicals, which assist the settling of the solid matters, and the decantation of the liquid portion.
2. The conversion of the sludge into manure.
3. The distillation of the liquids for the recovery of the ammonia as sulphate.

As we shall see later on, the distillation and absorption of the vapours takes place in closed apparatus under reduced pressure.

The sewage discharged from the reservoirs of La Villette is delivered at Bondy directly into disintegrators, where it is mixed with definite proportions of chemicals, consisting chiefly of milk of lime, of density 1·16 to 1·20. Two disintegrators are employed, and they are capable of passing 100 c.m. of sewage per hour. From the disintegrators the mixture flows into a brickwork tank of 500 c.m. capacity, covered with a plank floor, with openings to allow access to the tank. The more lime there has been added, the more readily does settling take place. After about an hour the clear supernatant liquid can be removed by a pump. It has a pale amber colour, and contains free ammonia, the acids being in combination with lime, and it is free from most of the impurities of the *eaux vannes.* The reactions which have occurred in this treatment are easily understood; the carbonic acid has combined with the lime, and the insoluble calcium carbonate produced is precipitated; the other acids also combine with the lime, producing both soluble and insoluble calcium salts, the latter of which precipitate with the carbonate. Moreover, the albuminoid matters are coagulated by the lime and entangle the pulverulent precipitates. A coagulation of the whole has in fact taken place, and the black fetid matters which entered the disintegrators issue from the tanks in a short time in a clear and limpid condition, with only an odour of ammonia, and in a fit state for its subsequent extraction. When the clear liquor has been drawn off, it is replaced by an equal volume of the mixture delivered by the disintegrators, and this operation is repeated several times until the tank is full. The thick sludge is then further treated for the production of a solid manure and the recovery of the adherent ammoniacal liquor. The sludge is passed into working tanks holding about 70 c.m. and thence to filter presses. It is raised into a series of *monte-jus* similar to those used in sugar works; in these it is heated by a jet of steam, and is only passed through the filters when sufficiently hot. This preliminary heating is necessary for satisfactory filtration; it gives a more satisfactory texture to the deposit; without it, firm cakes cannot be produced, filtration is troublesome, and the effluent is turbid and blackish. After heating, on the contrary, the effluent from the presses is clear and yellowish, the cakes are hard, and are readily detached from the filter cloths. During the heating, ammoniacal vapours are evolved, which are drawn off from the *monte-jus* by a pipe and passed into a lead vat containing sulphuric acid, where they are converted into ammonium sulphate. The effluent from the presses being thus deprived of its ammonia, does not inconvenience the workmen. The cakes are the richer in nitrogen the less there has been employed of chemicals, and the richer the sewage in nitrogenous organic substances.

One hundred c.m. of crude sewage yield by this process 75 c.m. of clear effluent and 25 c.m. of sludge, yielding 6000 to 7000 kilos. of press cakes, containing 50 per cent. of moisture. The clear liquors drawn from the settling tanks, and those from the filter presses, are treated together for the manufacture of ammonium sulphate.

This process, as may be seen, gives the double advantage of a rapid treatment of the sewage and the avoidance of effluvium, whilst it yields liquors which are easily treated, and cakes which are dried without difficulty. But these cakes have a very low value on account of the small amount of fertilising material contained in them.

The Keuntz Process.—This process also avoids the stocking of the sewage in settling tanks, and allows its immediate separation into clear liquors and saleable press-cakes. The sewage is received in a large closed tank, with vertical partitions, where a first separation of solid and liquid takes place. The clear liquors from the top of the last compartments flow into a vat from which they pass into the ammonia stills after traversing the heaters. The thick deposits collected in the lower part of the first compartments are lifted by a *monte-jus* into a closed mixer, where they are treated with aluminium chloride, ferric chloride, and acid calcium phosphate to disinfect them, fit them for the filter press, and enrich them in fertilising materials. The reagent is prepared by treating a mixture of 15 parts of bauxite, 75 parts of natural phosphate of lime, and 3 parts of hydrated oxide of iron, with 110 parts of commercial hydrochloric acid, and 150 parts of water. Of the resulting solution, 50 kilos. are added for each cubic metre of the thick sludge. After settling for twenty-four hours, a further quantity of clear liquor, with only a faint odour, can be drawn from the surface, whilst the sediment is lifted by a *monte-jus* and forced by compressed air into a filter press. This process yields clear liquors and press-cakes which are readily dried. The cakes contain as dicalcic phosphate all the soluble phosphate added, which, together with the calcium carbonate, has been precipitated by the ammonium carbonate present. The iron has combined with the sulphuretted products, and the alumina has modified the albuminous and slimy matters, so that they no longer prevent filtration. The cakes thus obtained contain 3 to 3·5 per cent. of nitrogen and 10 to 12 per cent. of assimilable phosphoric acid. This process is at work at various places, especially at Versailles.

§ 2. Treatment of the Clear "Eaux Vannes."

1. *The Figuera Process.*—The oldest process for the extraction of the ammonia from the *eaux vannes* is due to Figuera, by whom it was worked at Bondy. It is based on the volatilisation of ammonium carbonate by steam. Figuera's apparatus consisted of an ordinary steam boiler of a capacity of 11 c.m., the steam from which could be passed in succession into two vertical wrought-iron cylinders, each holding 10 c.m., and containing the *eau vanne* to be treated. The steam, together with the volatile salts of ammonia which it carried off, was passed into a leaden condensing coil traversing a wooden tank of 25 c.m. capacity filled with fresh *eau vanne*, which, whilst condensing the steam, was itself warmed. The steam, with most of the ammonium carbonate, was thus condensed. The uncondensed gases were driven through sulphuric acid contained in a wooden vat lined with lead, where the ammonia became converted into sulphate. The steam boiler was fed with the exhausted liquor from the cylinders, which still contained a trace of ammonia, and the cylinders were refilled with the warmed *eau vanne* from the condensing vat. Each cylinder was furnished with a wrought-iron steam chest for the breaking down of the abundant froth carried off by the vapour, and fitted with a gauge-glass to regulate the progress of the operation. Each operation lasts about twelve hours: when finished, the contents of the steam boiler are run out, and the boiler is refilled from the cylinders. The latter are recharged with the *eau vanne* from the cooling vat, and a fresh operation is commenced. The condensed ammoniacal liquid, having been neutralised with sulphuric acid, yields a very weak solution of the sulphate, indicating scarcely 5° Beaumé, and requiring to be concentrated together with the liquor from the acid vat. This concentration consumes a considerable amount of fuel, which it has been sought to reduce by employing the weak liquors for feeding the steam generators, by which part of the *eau vanne* was treated. When concentrated to 24° Beaumé, it was drawn off from the generators, and the operation finished in open pans heated by direct fire. The sulphate deposited during the evaporation is fished out, drained, and dried: the acid mother liquors are returned to the absorption vats.

Figuera fitted up at Bondy eleven sets of apparatus, allowing of the daily treatment of 300 c.m. of *eau vanne*, and producing about 2500 kilos. of ammonium sulphate.

This process, which presented numerous imperfections, has long been abondoned, but it served as the starting-point for more economical processes, which we shall examine in succession.

2. *Apparatus of Margueritte and Sourdeval.*—Figuera's apparatus allowed only weak solutions of ammonium sulphate, contaminated with impurities carried over by the froth, to be obtained. It was therefore necessary to evaporate much water to obtain the crystalline salt. Moreover, liquors imperfectly exhausted had to be thrown away boiling hot, and lastly, the quantity of ammoniacal liquor treated was small in comparison with the size of the plant. An apparatus has been designed by Margueritte and Sourdeval which obviates most of these inconveniences, and which has been adopted by many large companies. They have adopted the principle of the distillation column, which has long been in use for the dephlegmation of alcohol.

Fig. 1 shows the whole of their apparatus, which consists essentially of two parts: (1) an ordinary distillation column, A, of wrought iron, 1·3 m. in diameter, and composed of twenty-two plates, each of which is furnished with ten inverted cups, through which the *eau vanne* descends, undergoing systematic exhaustion, and escapes through the syphon S; (2) of a cooler fed with the *eau vanne*, which cools and partly condenses the ammoniacal vapours. From the end of this cooler there flow only saturated solutions and vapours rich in ammoniacal products, which are received in sulphuric acid.

Fig. 2 shows in plan and section the details of the construction of the column.

The apparatus works in the following manner:—The *eau vanne* from the settling tanks is raised by a pump into a wrought-iron tank (R, Fig. 1) supported on strong wooden framework. It then flows continuously by a tap (not shown) into a funnel pipe, E, which conveys it to the lower part of the cylinder B. In this cylinder it rises gradually, acquiring heat from contact with the coils through which the current of vapour from the still passes, and then flows by the pipe F into the distillation column between the 18th and 19th plates. In the column it

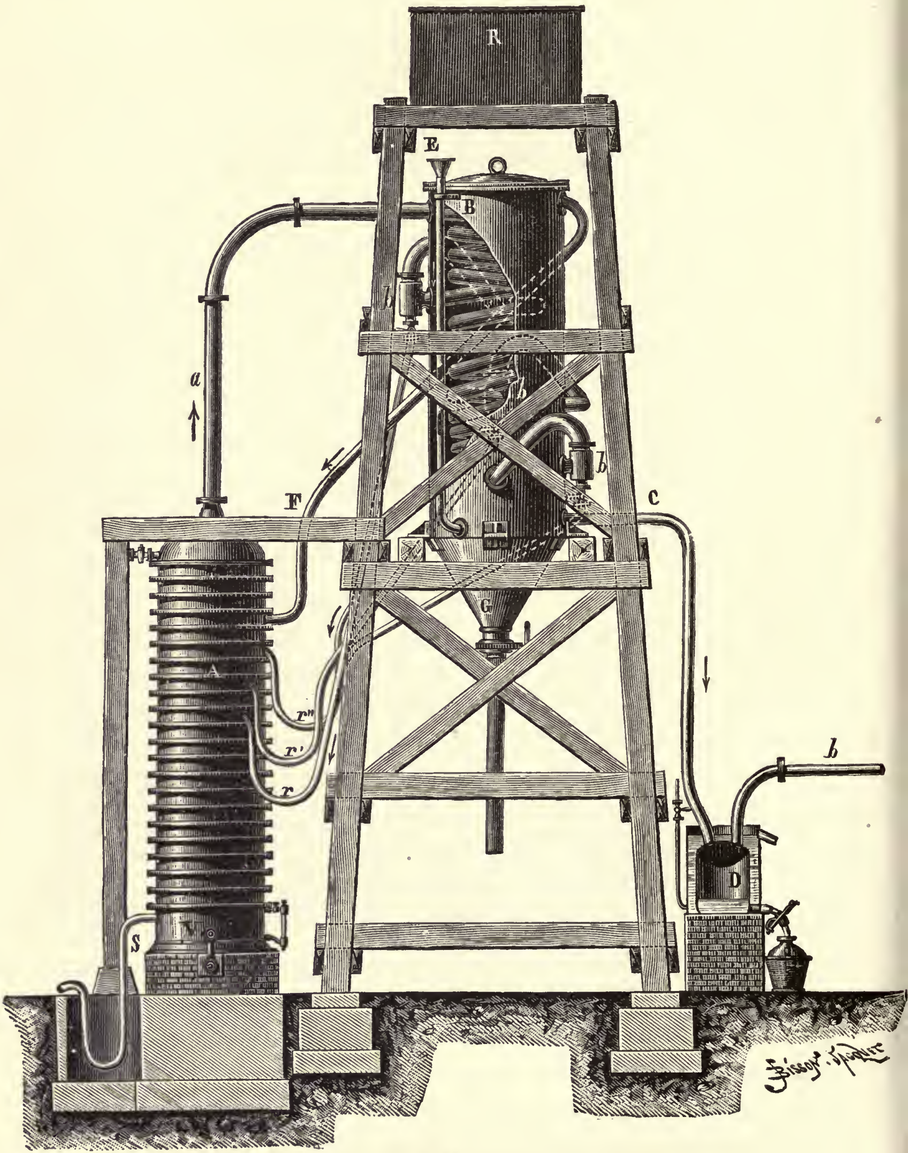
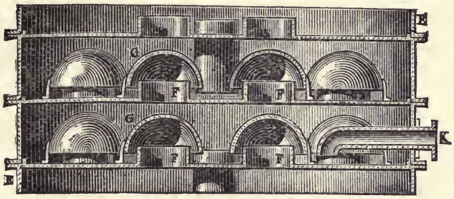


FIG. 1.—Apparatus of Margueritte and Sourdeval.

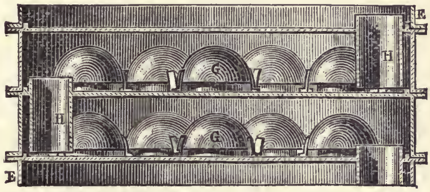
A, Cast-iron column, 1·3 m. in diameter.
B, Cast-iron cooler with back-flow pipes.
C, End of the condensing coil.
D, Leaden vessel for receiving the saturated solution of ammonium carbonate.
E, Pipe for the introduction of the *eau vanne.*
F, Feed-pipe for the column.
G, Conical part of the cooling cylinder for the reception of mud.
R, Reservoir of *eau vanne.*
S, Syphon for escape of exhausted liquor.
V, Steam inlet.
a, Pipe conveying the vapour to the coil.
b, b, b, Back-flow cups.
h, Escape pipe leading to acid vat.
r, r', r'', Back-flow pipes leading to column.

descends from plate to plate, and escapes through the syphon pipe S in a completely exhausted state.

Coupe suivant **AB.**



Coupe suivant **CD.**



Plan.

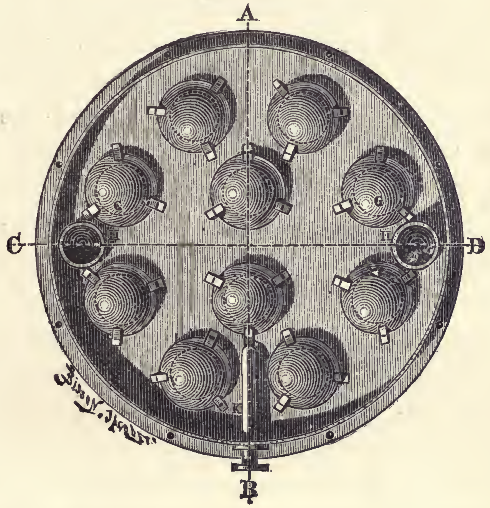


FIG. 2.—Details of construction of the column A of Fig. 1.

E, E,	Plates of the distillation column.
F, F,	Short pipes through which the vapour rises.
G, G,	Cups which force the vapours to bubble through the liquid on the plates.
H, H,	Down-flow pipes which limit the depth of liquid on the plate.
K,	Pipe for entry of *eau vanne*.

The steam, arriving from a boiler,[1] enters the column at V, and

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[1] A 50-horse power boiler is required.

keeps the liquor on all the plates in ebullition. The carbonate and sulphide of ammonium carried off by the steam, pass by the pipe *a* into the cooler B.

This cooler is constructed in four distinct parts, which are so arranged that the liquid condensed in the three upper portions is returned to the column, and that from the fourth is collected in D. This is a saturated solution of ammonium carbonate mixed with sulphide.

The uncondensed vapour escapes from the vessel D by a wide pipe, which conveys it into sulphuric acid of 53° (chamber acid), contained in a lead-lined wooden vat, with a cover, where the ammonia is retained in the state of sulphate. The cups *b*, *b*, *b*, and the pipes r, r', r'', allow the return to the column of the liquids condensed in the three upper divisions of the cooler, at such points that the returned liquids mix with products of approximately the same richness, so as to introduce no irregularity into the methodical extraction. The liquid collected in the vessel D shows a density of 16° Beaumé; it serves, as we shall see later, for the preparation of ammonium chloride, or of ammonia. It is drawn off into carboys by a caoutchouc pipe closed with a clamp.

The gases evolved during the absorption of the vapours by sulphuric acid were formerly conveyed by a wide wrought-iron pipe to the ashpits of the boiler fires, to diminish as far as possible the escape of noxious vapours; but the large proportion of carbonic acid and water vapour mixed with the offensive products prevented the complete combustion of the latter, and a very imperfect result was obtained. For some time the complete destruction of these offensive vapours has been successfully carried out by cooling them to remove water vapour, and driving them by a fan through a small cupola furnace fed with coke or cinders. The gases then escape from the cupola with no odour except one of sulphurous acid, and a great improvement from a hygienic point of view has thus been effected.

After working for several days, the muddy deposits which have collected in the conical part (G) of the cooler (B) are drawn out into waggons.

By the saturation of sulphuric acid of 53° there is obtained a solution of ammonium sulphate showing about 25°, which must be evaporated. This evaporation is performed in tanks by means of coils of thick lead pipe heated by steam. The salt is fished out as fast as it separates, thrown upon drainers, and dried on cast-iron plates heated by the waste heat of the steam boilers. The sulphate thus obtained is white and fine: it contains 20 to 21 per cent. of nitrogen. The exhausted liquors which issue from the column are boiling hot. They are conveyed through gutters covered with cast-iron plates, on which the sludge from the settling tanks is dried. They thus lose most of their heat before being allowed to enter the drains. The vapours from the drying of the sludge, consisting of water vapour and offensive gases, are drawn into a chimney placed at the end of the chambers, formed by covering the substances undergoing desiccation with loose wooden planking. In spite of these precautions, the offensive exhalations are far from being destroyed; the greater part escape from the chimney, and are distributed in different directions, according to the prevailing wind. It is mainly to these evaporations that most of the complaints which have occurred since the opening of the Nanterre works are to be attributed, and which have been the cause of the works being closed. An apparatus such as we have described, costing 30,000 francs, will treat 100 c.m. of ammoniacal liquor per twenty-four hours, and yield the equivalent of 9 to 10 kilos. of ammonium sulphate per cubic metre. The water from such an apparatus still contains, however, all the fixed salts of ammonia, representing 3 to 4 kilos. of ammonium sulphate per c.m. Many of these apparatus are at work round Paris, in the works of the Lesage Company,[1] and of the Urbaine Company. The Parisian Company de Vidanges et Engrais had put up ten of them in their works at Nanterre, which are now not employed.

3. *The Lair Apparatus.*—With the apparatus which we have just described, the ammonia existing in the fixed state in the *eaux vannes*, especially that in the form of phosphate, sulphate, and hydrochloride, is not extracted; there is obtained only the carbonate and sulphide which are carried over by the water vapour. The sulphate and hydrochloride result chiefly from the addition of metallic salts to the contents of the fosses, in order to disinfect

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[1] The Lesage Company treats 1300 to 1400 m. of sewage daily: 250 at Billancourt, 400 at Alfort, 600 at Aubervilliers, and the remainder at small establishments at Drancy and Nanterre.

them before emptying. This causes the loss of a notable quantity of ammonia, which it would be important to extract. Another defect in these appliances is that they discharge the exhausted liquors boiling hot, and recover only a very small part of their heat; these liquors, which are black and turbid, have to be passed through long channels or into tanks to allow them to deposit their suspended matters before letting them run away. Lastly, with the earlier forms, only weak solutions of ammonium sulphate are obtained, which it is necessary to evaporate to obtain the crystallised salt, an operation involving much expenditure of fuel. M. Lair has endeavoured to solve the difficult problem of extracting the whole of the ammonia from the *eaux vannes*, of running away the residual water clear and cold, and of obtaining at once in the neutralisation vats crystallised ammonium sulphate, which can be fished, drained, and dried immediately.

The arrangement which he has devised, and which was first set up at Bondy, is extremely ingenious, and has given excellent results. Although in practice it has shown some imperfections, it is still one of the best appliances at the present day for the treatment of clear *eaux vannes*.

Lair's apparatus, shown in Figs. 3 and 4, consists essentially of four distinct parts—

1. A distilling column of cast iron, A, 0·9 m. in diameter, and 5·5 m. high.
2. Two mud tanks, B, B, in which the turbid boiling liquor from A is clarified before passing through the heaters.
3. Two exchange heaters, C, C, for transferring heat from the exhausted liquor to fresh *eau vanne*.
4. Two wooden vats lined with lead (K, K, Fig. 3), for the absorption of the ammoniacal vapours from the column.

The column consists of twenty-five similar plates, each furnished with a single shallow inverted cup of cast iron, with notched edges. It is heated by steam, brought from a boiler by the pipe V, 20 mm. ($\frac{3}{4}$ in.) in diameter, and with an adjusting stop-cock.

A pump, P, driven by gearing, lifts the *eau vanne* from the settling tanks and passes it continuously through the heaters C, C, into the column A, by the pipe *e* (Figs. 3 and 4). This liquor, heated by passing through C, C, enters at the twentieth plate, then descends gradually from plate to plate in the column, parting with

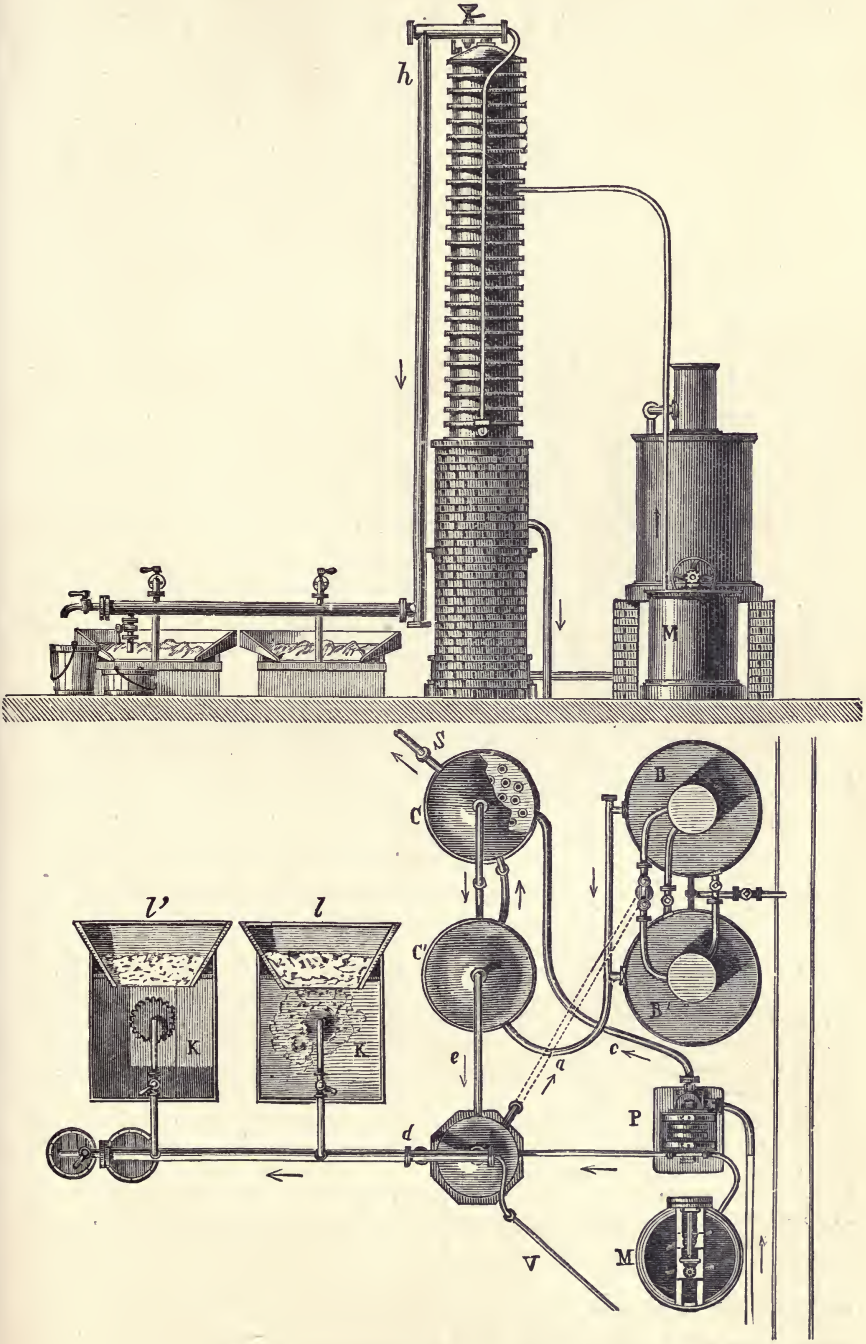

Fig. 3.—Plan and Elevation of Lair's Apparatus.

most of its volatile ammoniacal salts. By a pump driven by the same gearing as P, a quantity of milk of lime is injected through the pipe *d* into the column at a situation where the only ammonium salts left are the fixed sulphate, phosphate, and chloride, which would remain in the residuary liquors, and some ammonium

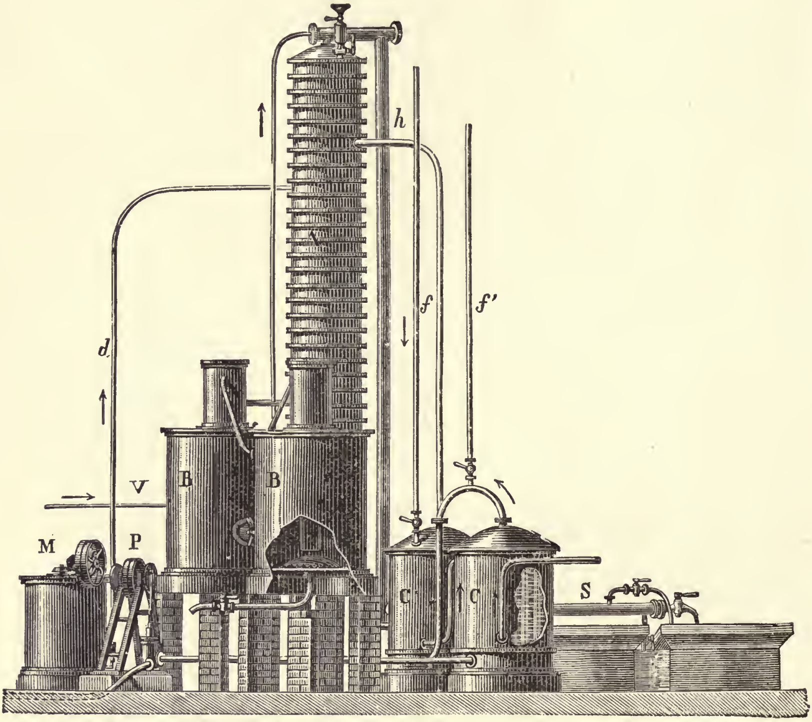

FIG. 4.—Elevation showing the Arrangement of the various parts of Lair's Apparatus.

A,	Cast-iron column, consisting of plates, each carrying a single inverted cup.
a,	Pipe conveying the exhausted hot liquor to the mud tanks, B, B.
B, B,	Mud tanks.
C, C,	Exchange heaters.
c,	Pipe conveying the *eau vanne* to the exchange heaters.
d,	Pipe for injecting milk of lime into the column.
e,	Pipe conveying the hot liquor to the column.
f, f',	Pipes for the escape of uncondensable gases.
h,	Pipe for conveying the vapours from the column to the acid vats, K, K.
K, K,	Wooden acid vats with lead linings.
l, l,	Sulphate of ammonia drainers.
M,	Churn for making milk of lime.
P,	Pump which lifts the *eau vanne* from the settling tanks, and conveys it to the exchange heaters C, C, by the pipe *c*.
S,	Exit pipe for the exhausted, clear, and cold liquor.
V,	Steam pipe for heating the column and the casing of the tube *h*.

sulphide, which, although less volatile than the carbonate, would be carried off, and would yield sulphuretted hydrogen, and even deposit sulphur in the acid vats. At the foot of the column the exhausted boiling liquor, rendered turbid by the lime and the insoluble lime salts in suspension, is conveyed by the pipe *a* first into the two mud tanks, B and B, where it clarifies in consequence of its slow rate of flow through these vessels, the diameter of which is 1·8 m., and height 2 m. The liquor reaches the first vessel, B, through a wide cylindrical tube reaching nearly to the bottom of the tank, as shown in Fig. 4; it then rises slowly, and flows over by a side pipe into the similar tube of the second vat. From the last it finally issues quite clear, and passes in succession through the heaters C and C. These heaters are fitted with vertical tubes, round which the hot exhausted liquors are made to circulate, whilst fresh cold liquor injected by the pump P, passes through the tubes in an opposite direction. On leaving the two heaters by S, the exhausted liquors are delivered clear and cold, and can be allowed to run away without inconvenience. By passing through the two heaters the fresh liquors are brought up to about 90 to 95°; they then enter the column at the twentieth plate, as was said above.

The matters in suspension in the liquors which issue from the column accumulate gradually in the mud tanks, and would in time fill them completely. Their level is ascertained from time to time by gauge-cocks, and they are removed as required into wrought-iron waggons through stop-cock pipes in the bottoms of the vats. An inverted cup of cast-iron, with serrated edge, placed over the opening, prevents the pipe from getting choked. After the cleaning out of a mud tank the course of the liquors from the column is changed, so that it becomes the last through which they pass. During the first heating of the *eau vanne* foul gases are evolved, which are allowed to escape by the pipes *f*, *f′* (Fig. 4). The vapours which escape from the column consist of steam, ammonium carbonate, free ammonia, and fetid, uncondensable gases. This mixture is passed into the sulphuric acid contained in one of the lead lined vats, where the ammonia is retained as sulphate, whilst the carbonic acid and the uncondensable gases escape. The pipe *h*, which carries these vapours to the vats, contains a steam tube, which prevents any obstruction from being caused by a deposit of ammonium carbonate. The water, saturated with ammonium carbonate, which is condensed in the pipe by the cooling action of the air, escapes into a receiver through a small tap not shown in the figure, and is returned to the apparatus. The sulphuric acid in the vat gradually becomes neutralised, and ammonium sulphate soon separates in small crystals. When neutralisation is complete the vapours are turned into the second vat, whilst the contents of the first cool and deposit a fresh quantity of the salt. The mixture is stirred from time to time to cause the formation of small crystals, and the salt is then removed to leaden drainers above the vats; it is finally dried on plates of wrought iron placed over the flues of the steam boiler. The mother liquor of the salt is mixed with a fresh quantity of sulphuric acid, and in its turn receives the gases from the column.

The milk of lime requires to be prepared with some care; it must be free from stones, which would choke the pipes, and should have as uniform a composition as possible. To obtain this result, a weighed quantity of good lime is slaked, sifted to separate stones, and mixed with a known amount of water in the churn M, the paddles of which keep the mixture stirred. It thus becomes easy to introduce an exact quantity of lime into the column by means of a pump throwing a known volume. M. Lair's apparatus will treat about 50 c.m. of *eau vanne* every twenty-four hours, extracting the ammonia of the fixed salts, and running off the exhausted liquors cool and clear.

The black sediment from the mud tanks is thrown into heaps, where it drains and dries readily, losing its colour by oxidation; it gives off no smell, and as it consists essentially of lime salts, and contains a small amount of fertilising agents, especially calcium phosphate, it is used as manure on the neighbouring fields.

On the occasion of a visit some years ago to the works of M. Lair at Bondy, there were four sets of the above apparatus in existence, capable of treating 200 c.m. of *eau vanne* per twenty-four hours. The liquors which were under treatment at that time contained only 2·5 grms. of ammoniacal nitrogen per litre; they yielded 11·4 kilos. of ammonium sulphate per cubic metre, and required the combustion of 1·8 kilo. of coal per kilo. of sulphate obtained. The liquors which were run off contained only 0·06 to 0·1 grm. of nitrogen per litre. Even with these unfavourable conditions, crystallised ammonium sulphate was obtained directly in the saturation vats, a result due to the slow speed of the operation, and to the presence of free ammonia in the mixture, which, being itself very volatile, assists in carrying off the ammonium carbonate.

This apparatus presents several important improvements on the preceding, both from the point of view of public health and that of the manufacture of ammonium sulphate. An objection to it is, however, that it treats a comparatively small quantity of the *eau vanne*, and that the column requires frequent cleaning to remove the deposits produced by the lime.

4. *The Apparatus of Sintier and Muhé.*—MM. Sintier and Muhé have made valuable modifications in certain portions of the preceding apparatus. These modifications produce no change in the course of the operations, but allow a more perfect result to be obtained. They consist essentially in the combination of the mud tank and the exchange heater into one vessel, and in a different method of using the lime intended for setting free the ammonia of the fixed salts. In Lair's apparatus the mud tank and the heater are distinct, a consequence of which is that the liquors from the column are much cooled by the surrounding air before they arrive at the heater; and further, the mud carries away heat which cannot be recovered. The combined mud tank and heater of Sintier and Muhé is intended to remedy these two defects. It consists of two tubular vessels (A and B, Fig. 5). The first, A, which is a heater resembling that of Lair, consists of a wrought-iron cylinder containing a group of tubes which connect the upper and lower parts. The second, B, consists of four portions—(1) The lower chamber under the plate, which carries the tubes; (2) a cylindrical vessel of wrought iron, supporting a cone, *b*, the apex of which is connected with a bent tube ending outside in a stop-cock, *s*, for drawing off the deposit; (3) a second cylindrical vessel resting on the base of the cone, and open above for the removal of the mud which collects round the tubes; (4) a small upper receiver, connected with the lower chamber by a group of 25 to 30 tubes. This receiver is fixed above the cylinder B, so as not to be in the way when cleaning the tubes. The *eaux vannes* delivered by the pump P pass through the tubes of the heater A, issue by the pipe *f*, and rise in the tubes of the vessel B, passing finally by the pipe C into the column. During this transit they acquire heat

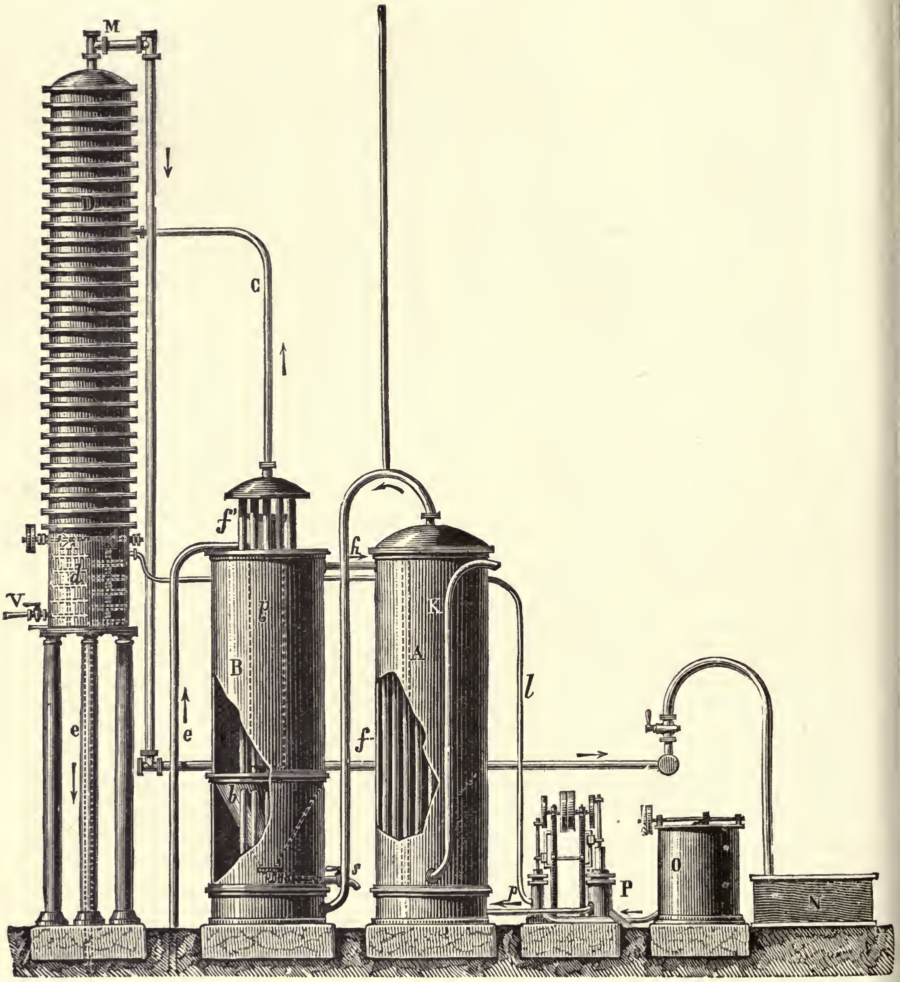

Fig. 5.—Apparatus of Sintier and Muhé.

A, B, Heaters.
b, Mud cone.
C, Tube by which the hot *eaux vannes* enter the column.
D, Column.
d, Lime chamber.
e, *e*, Syphon for conveying the liquors to the mud tank by the cylinder *p*.
f', Entrance to the cylinder *p* of the mud tank.
f, Pipe which conveys the liquor from A to B.
K, Exit for cold exhausted liquors.
l, Pipe for injecting milk of lime.
M, Pipe conveying the vapours from the column to the acid vat.
O, Lime mixer.
P, Pump for *eau vanne.*
p, Pump for lime.
N, Acid vat.
s, Exit for mud.
V, Entrance of steam to column.

from the exhausted liquors which travel in the opposite direction. These latter descend by the syphon-pipe *e*, *e*, enter the cylinder *p* at *f′*, flow over the cone *b*, enter the heater A at *h*, and escape by the pipe K completely cooled. The level at which the exhausted liquors stand in the heater B, is such that the cylindrical portion *d′* is always about four-fifths full. The quantity of liquid contained in this portion is equal to that on the plates. We have seen above that the introduction of lime into the column produces numerous obstructions, necessitating frequent cleaning.

MM. Sintier and Muhé have completely remedied this inconvenience by greatly augmenting the capacity of the first chamber (*d*) of the column, into which the steam is admitted by the stopcock V, and it is into this portion of the apparatus that the lime is injected by the pump *p*, which draws it from the mixer O. The lime is intimately mixed with the liquor by a stirrer, which is shown in dotted lines. The fixed ammoniacal salts are thus decomposed without fear of choking the apparatus. The vapours from the column are passed into sulphuric acid in the lead-lined vat.

5. *Bilange's Apparatus.*—As stated in Chap. II. § 1, M. Bilange has sought to solve the double problem of producing a manure of immediate utility to agriculture, and of not allowing the escape, either into the air or the waste liquors, of any but inodorous and imputrescible substances. We have described the first part of his process, that is to say, the separation of the crude *eaux vannes* and the production of filter-press cakes; it remains to describe the process of distilling the clear liquor for the extraction of the ammonia.

The distillation is performed at reduced pressure, so as to lower the boiling point of the liquid considerably, and thus diminish the effluvium which would still result from the treatment of these clarified liquors. The arrangement adopted strongly resembles that in use in sugar works for concentrating the juice. It consists of four vertical tubular drums, closed air-tight, and heated by steam from a boiler. In these vessels the ammoniacal liquors are raised to a temperature of only 60 to 65°, which is sufficient to cause them to part with the ammonia they contain, in consequence of diminished pressure, which is produced by a pump working during the whole operation. The ammoniacal vapours given off from the heaters pass into a closed chest connected with the air-pump. In this lead-lined chest the mother liquors of a previous operation are placed, and from time to time sulphuric acid is admitted. Samples of the liquor drawn from the chest show when the addition of more acid is needed. Only small quantities can be introduced at one time, lest the action should be too violent. The ammoniacal vapours, before reaching the sulphuric acid, are passed through a weak solution of ammonium sulphate in an intermediate vessel. This scrubber, which acts as a safety vessel, can, in case of repairs being needed, take the place of the neutralising vessel. When it is judged that sufficient salt has been formed, the addition of sulphuric acid is discontinued and the solution of sulphate is evaporated, steam being introduced into a coil lying on the bottom of the vessel, and connection being made with the vacuum chamber. Evaporation takes place very rapidly, and must not be pushed too far, lest the whole mass should solidify and need to be redissolved. When the liquor is sufficiently concentrated, it is allowed to run out into a crystalliser placed below. For this purpose the supply of steam to the coil is shut off, communication with the vacuum apparatus is closed, and air is allowed to enter; the hot liquor is then allowed to run out, and it crystallises on cooling. The salt is fished out as it crystallises, and is drained and dried. It is evident that this process avoids the production of the noxious vapours which result from the use of distillation columns, and which it has not been found possible to prevent on the large scale. In our opinion, the only objection to this process is that it requires a very costly plant, which must wear out very quickly. Nevertheless, a somewhat long experience at Bondy has proved the efficiency of this process, which satisfies public hygiene, and would prevent the accumulation of pasty matters.

6. *The Kuentz Process.*—M. Kuentz has suggested a special method of treatment of the *eaux vannes* to avoid the escape of noxious products, which of late years, and especially in Paris, has given rise to so many complaints. The vapours from the distillation columns are usually, as we have seen, passed into sulphuric acid of 53°, in which the ammonia is converted into sulphate, whilst the carbonic and hydrosulphuric acids, with the vapour of water and the offensive gases which accompany them, are sent into the chimney of the works, or better through a furnace. Under these conditions the offensive gases are diluted with a large volume

of carbonic acid arising from the decomposition of the ammonium carbonate, which prevents the complete destruction of these gases by heat and oxygen. After cooling, the gaseous mixture diffuses through the air, and the effluvia, wafted by the wind, reach the earth at a greater or less distance from the works.

To destroy these vapours, Kuentz has proposed to absorb the carbonic and sulphydric acids by lime, which would render the destruction of the odorous vapours, by passing them through a special coke furnace, easy and complete. It was pointed out by M. Girard that these vapours might be destroyed by passing them over glowing coke. But these fumes, in the condition in which they issue from the apparatus, being rich in carbonic acid, are incombustible, and are only destroyed with difficulty. Kuentz's process, whilst solving the problem completely, requires a large amount of lime. Kuentz has therefore proposed, as a means of rendering the operations more easy and complete, to acidify the vapours in closed vessels, and by double decomposition to retain most, if not all, of the carbonic acid; the uncondensable gases can then be passed through a furnace and completely destroyed. The following is the process for obtaining the ammonium sulphate:—Phosphate of lime is treated with dilute sulphuric acid to set free all the phosphoric acid; the phosphoric acid obtained is neutralised by strong ammoniacal liquors, prepared as we shall see further on. Ammonium phosphate is thus obtained, which is evaporated. The calcium sulphate, obtained in the treatment of the phosphate of lime by sulphuric acid, being treated with concentrated ammoniacal liquors, or by the gases from the column, yields ammonium sulphate, which remains in solution, and calcium carbonate, which precipitates or is separated by filtration. Operating thus, there is no disengagement of carbonic acid; the neutralisation and evaporation of the liquors takes place in closed vessels, and the uncondensable gases which issue from the apparatus are passed through scrubbers containing lime and oxide of iron, and then through a special furnace, where they are destroyed. This process is somewhat long, but is very reasonable.

7. *Process and Apparatus of Hennebutte and Vauréal.*—This process is worked by the Société anonyme des produits chimique du Sud-Ouest. It consists in treating the entire sewage with 2 to 3 thousandths of zinc sulphate and 5 thousandths

of aluminium sulphate, then allowing the mixture to settle, and running off the clear liquor, which is sent to the ammonia stills. The deposit is treated with a further quantity of the same chemicals, then forced into filter presses by compressed air. In this way very firm filter-press cakes are obtained, which are easily dried by simple exposure to air or in stoves, and clear filtrates, which are added to the former. By the use of the sulphates of zinc and alumina the liquors are desulphurised, and the alumina precipitate assists the separation of the solids. The dried cakes contain 3 to 4 per cent. of nitrogen and 3 per cent. of phosphoric acid. The apparatus devised by Hennebutte and Vauréal is shown in Fig. 6; it consists of three rectangular boilers, A, B, C, of cast iron, forming one chamber, and arranged so as to give a continuous fall. The boilers B and C contain vertical partitions *b* and *c*, which are fixed 0·1 m. from the bottom, so as to force the vapours from the lower boilers A and B to bubble through the contents of B and C, and keep them agitated. At the upper part of the apparatus there is a rectangular vat D, containing a condenser formed by an oval wrought-iron tube E, connected with the boiler C by a wide tube *e*. The pipe F, which is the outlet of this condenser, carries a valve *f*, which allows a partial vacuum to be maintained in the apparatus. This pipe is connected with a chest G, for the reception of the condensed liquids, or to pass them at pleasure into another chest H, which is heated by the boiler A, the top of which forms the bottom of H; a pipe with stop-cock allows H to be emptied into B. Lastly, the pipes *g* and *h* allow the ammoniacal gases to pass into a sulphuric acid vat, where they are retained as sulphate. The wrought-iron tank K is intended to receive the exhausted liquors issuing from the last boiler, and the tank L, which is fed with the hot liquors run off, contains a series of iron pipes through which the fresh *eau vanne* circulates, and where it becomes heated before entering the measuring vat M, and then the boiler C. The apparatus works in the following manner:—The crude *eau vanne*, arriving from an upper reservoir, fills the chamber D of the condenser; it then runs down by the pipe *d* into the system of tubes in the tank L containing the hot exhausted liquors, and becomes heated to about 80°. It then rises by the pipe *m* into the measuring vat M, which is connected by a wide stop-cock with the boiler C. Thence it passes into that boiler, where it reaches about 90°.

In this boiler it parts with most of its volatile ammonia, which is driven off by the current of mixed vapours arriving from the other boilers. The ammoniacal vapours pass into the condenser, where they are cooled, and by thus diminishing in volume produce a partial vacuum in the apparatus, which permits the liquid in the boiler C to boil at 90°. When the liquid in the lower boiler A is exhausted, it is run into the settling tank K, that from B takes its place in A, the liquid from C passes into B, and C receives a charge of fresh *eau vanne*. Slaked lime is introduced into A by a plug-hole N, which serves at the same time as a safety-valve. The proportion of lime added should be sufficient to decompose all the fixed salts existing naturally in the *eau vanne* or resulting from

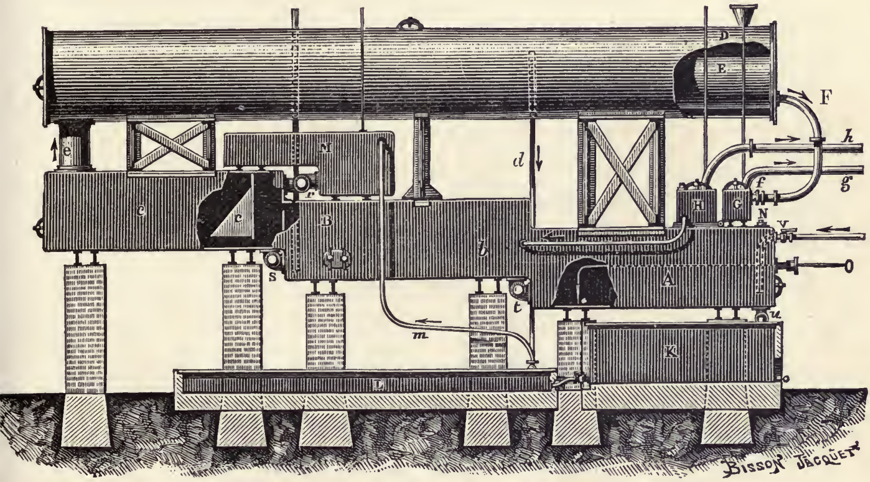

FIG. 6.—Apparatus of Hennebutte and Vauréal.

A, B, C, Rectangular wrought-iron boilers.
D, Wrought-iron tank which receives the crude *eau vanne* employed to cool the condenser E.
d, Pipe which conveys the *eau vanne* to the heater L.
E, Oval wrought-iron pipe, which serves as condenser.
e, Pipe by which the vapours reach the condenser.
F, Exit for the ammoniacal vapours and the concentrated solutions produced in the condenser E.
f, Valve.
G, Receiver for the concentrated solutions.
H, Chest for the distillation of the concentrated solutions.
g, *h*, Exit for the ammoniacal vapours to the acid vat.
K, Vat for the reception of the turbid exhausted liquors.
L, Heater.
M, Measuring vat.
m, Pipe by which the *eau vanne* reaches the measure.
N, Plug for introducing milk of lime.
O, Scraper for cleaning the bottom of the boiler A.
s, *t*, *u*, Stop-cocks for emptying the boilers C, B, A.
V, Steam-cock.

the sulphates of zinc and alumina introduced. When the boilers are thus filled, a new operation is commenced. The apparatus is heated by a steam jet introduced into A. The vapours from that boiler pass successively through the liquids contained in the two others, which thus become methodically deprived of their ammonia. The liquids condensed in the cooler are very rich in ammonia and ammonium carbonate; they are collected in the vessel G, then flow into H, whilst the ammoniacal vapours pass by the pipe *g* to the acid vat. The concentrated solution in H becomes heated by its contact with A, and gives up the greater part of its ammonia, which passes off by the pipe *h* to condense in the sulphuric acid. An apparatus of this kind has been fitted up at Villejuif, near Paris. Another is at work at Fribourg en Brisgau (Baden), and treats 25 c.m. of *eau vanne* per twenty-four hours, producing crystallised ammonium sulphate in the neutralising vat.

§ 3. TREATMENT OF ENTIRE SEWAGE.

1. *Chevalet's Apparatus.*—The preceding apparatus will treat only nearly clear *eau vanne.* Chevalet has devised an apparatus which permits the treatment of turbid liquors or even of whole sewage, without consuming more lime than is required by the fixed salts. This apparatus, shown in Fig. 7, consists of three cylindrical wrought-iron boilers, A, B, C, one above the other, through which the material to be extracted is passed in succession. Each of these boilers contain pipes *b*, *b*, which cause the gases from C to bubble through the liquors; and overflow pipes *p*, *p*, which maintain the level constant in the two upper boilers, and cause a continuous flow of the liquors into the lower one. The steam from a boiler introduced by the pipe V heats the liquor in C to boiling; the mixed vapours pass into B through the branched pipes *b*, becoming richer in ammonia, and finally reach the boiler A. These enriched vapours are then sent to the tank D, containing fresh liquor, which they heat whilst undergoing partial condensation. They then pass by the pipe E into sulphuric acid, where the ammonia is retained as sulphate, whilst the water vapour, carbonic acid, and noxious uncondensable gases are collected by the bell F. These products are then sent through the coil G, cooled by fresh liquor, where most of the water vapour condenses. The condensed liquid runs out by

the pipe *g*, whilst the gases are delivered by L below the bars of a furnace to be destroyed. Into the third boiler C a pump throws a quantity of milk of lime equal to about 6 to 7 kilos. of lime per cubic metre of *eau vanne*, which quantity is required to decompose the fixed salts. The ammonium sulphate solution is concentrated by a steam coil, the salt fished out, and drained on inclined plates. Once an hour the exhausted contents of the boiler C are drawn off by the valve K, those in B are drawn down by K′ into C, and those of A by K″ into B, and the upper boiler is then filled from D by opening the stop-cock H, which permits fresh liquor from

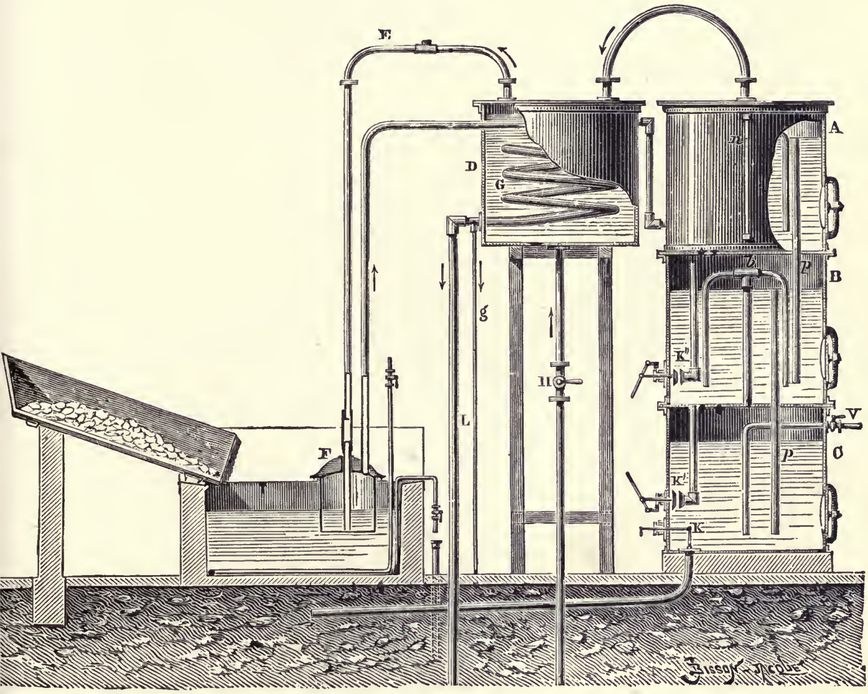

FIG. 7.—Chevalet's Apparatus.

A, B, C, Wrought-iron boilers, cased with wood to prevent external cooling.
D, Wrought-iron vat containing *eau vanne*, through which passes the coil G.
E, Pipe leading the ammoniacal vapours to the sulphuric acid vat.
F, Bell for collecting the uncondensable gases.
G, Condensing coil.
g, Exit pipe for the liquids condensed in G.
H, Entry for the *eau vanne*.
K, K,′ K,″ Valves for emptying the three boilers.
n, *n*, *n*, Water-level gauges.
p, *p*, Overflow pipes to regulate the level of the liquors in A and B.
L, Pipe conveying the uncondensed gases to a furnace.
M, Steam coil for concentrating the ammonium sulphate.

the overhead reservoir to rise into D. In this way the liquids which have been freed from their volatile salts are treated with lime to decompose the fixed salts, and are only run off after prolonged boiling, which exhausts them as completely as possible. The boiling residual liquors are then passed into a vessel, where they give up much of their heat to fresh liquors.

When very turbid *eaux vannes*, or entire sewage, is treated, the residuary liquors are passed through channels in which they deposit their suspended solids. Or, more simply, they may be passed through filter presses, yielding cakes, which when dry contain 5 to 6 per cent. of phosphoric acid, and 2 to 3 per cent. of nitrogen. Filtration is easy, the lime having modified the albuminous matters, and the drying of the cakes is very rapid. The filtrates are yellowish; they contain only salts of potash and soda, and a very small quantity of ammoniacal nitrogen, varying from 3 to 5 ten-thousandths. They may be allowed to enter the watercourses without inconvenience. A Chevalet apparatus, with boilers 0·8 m. in diameter and 0·8 m. high, will treat 4000 litres of *eau vanne* per twenty-four hours.

Large installations on this system have five or six superposed boilers, in some cases 3 m. in diameter and 0·75 m. high. The upper chamber serves for breaking the froth, and warms the liquor before it enters the first boiler.

Fig. 8 shows the arrangement of one of these large installations.

The *eau vanne* is raised into the tank A. It flows down into a heater B, immersed in boiling exhausted liquor; it then rises into the vat C, and passes by the pipe *c* into the froth chamber D, where it meets the vapours rising from the last boiler E. It then descends successively and intermittently through the boilers F, G, H, I, and J, undergoing methodical exhaustion. In the lowest of these it receives the charge of lime necessary for the decomposition of the fixed salts; it flows after exhaustion into the cistern K, from which it passes either through settling channels or into a filter press to separate the solid matter. The vapours from the top boiler are enriched in the heater D, and enter the acid vat under the bell L. The vapours which escape absorption rise through the coil M, where the water vapour is condensed, whilst the gases pass to a furnace.

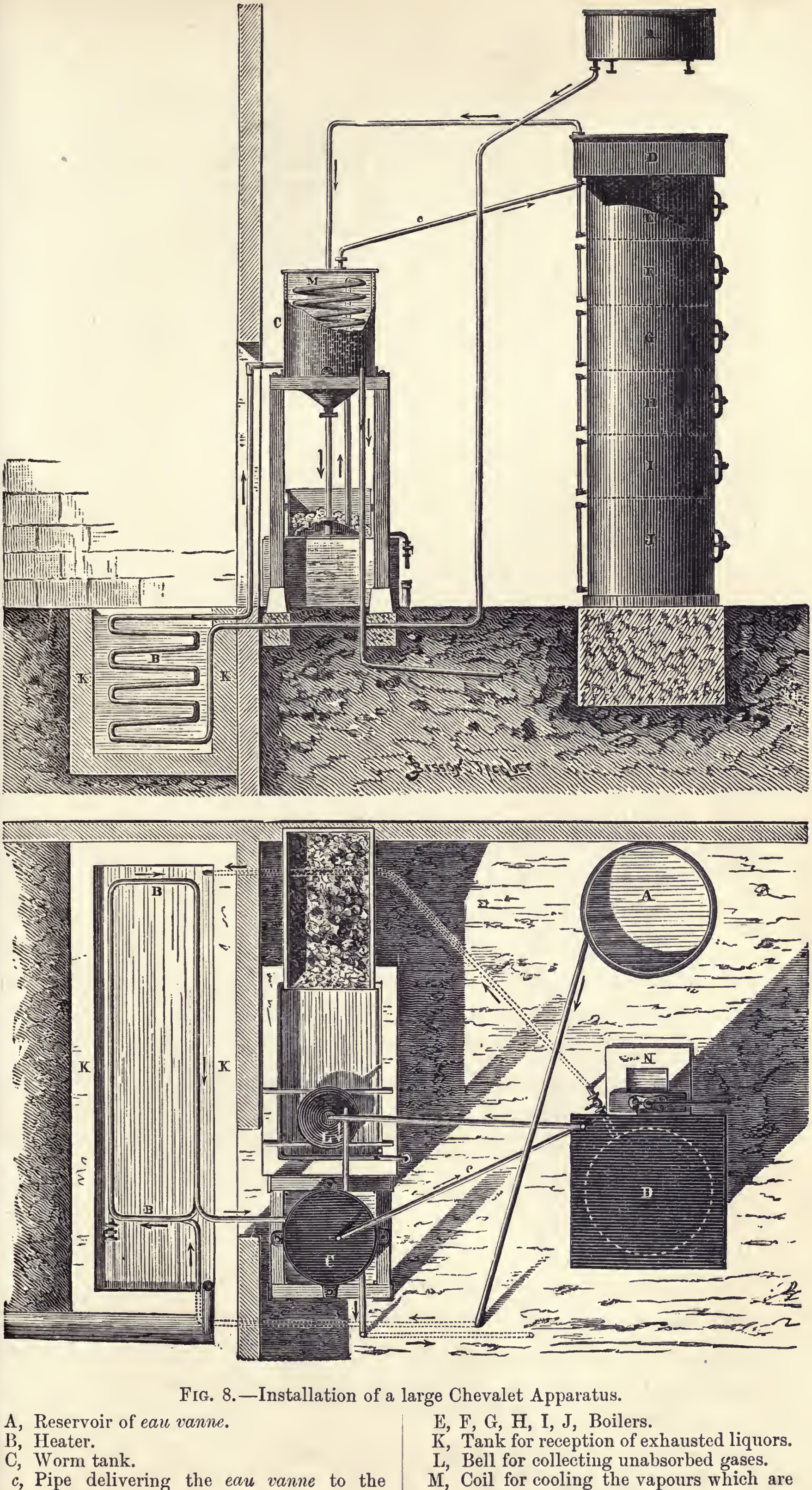

FIG. 8.—Installation of a large Chevalet Apparatus.

A, Reservoir of *eau vanne*.
B, Heater.
C, Worm tank.
c, Pipe delivering the *eau vanne* to the column.
D, Heater and froth separator.
E, F, G, H, I, J, Boilers.
K, Tank for reception of exhausted liquors.
L, Bell for collecting unabsorbed gases.
M, Coil for cooling the vapours which are not stopped by the acid.
N, Pump for milk of lime.

According to M. Chevalet, an apparatus on his system, which will treat 50 to 100 c.m. of *eau vanne* per day, will consume 25 to 30 kilos. of coal and 4 to 6 kilos. of lime per cubic metre treated, and 90 to 95 per cent. of the ammonia will be extracted. With smaller apparatus, treating 10 or 12 c.m. in twenty-four hours, the consumption of fuel will be 35 to 40 kilos., and the same proportion of lime.

Apparatus on this system, treating whole sewage, is at work in a large number of towns, such as Dijon, Havre, Lyons, Nice, Nanterre, Reims, and St. Quentin.

The Chevalet apparatus is equally applicable to the treatment of ammoniacal gas liquor.

2. *Paul Mallet's Apparatus.*—M. Paul Mallet in 1881 set up at the works of the Bordeaux Society de Vidanges et Engrais, at La Tresne, near Bordeaux, an apparatus for treating turbid *eaux vannes* or entire sewage, with the object of manufacturing ammonium sulphate.

The special feature of this apparatus is the constant mechanical agitation of the materials with lime during their passage through the column, after the removal of the volatile salts. Fig. 9 represents this ingenious apparatus, partly in elevation, partly in section. It consists essentially of five principal parts—

1. A tubular heater A.

3. A tubular analyser B.

3. A column C, built of cast-iron, 1·4 m. in diameter, on the Champonnois system, the upper part of which acts as an analyser, and as a dephlegmater beyond the point where the materials are introduced by the pipe *c*. This column is supported on a base plate D, with large openings, which rests on the mixing column E.

4. A column, 2 m. in diameter, constructed of plates, on each of which revolves a mechanical stirrer, keyed to a vertical axle *e*, driven by a pair of toothed wheels.

5. A double-bottomed mud tank F, on the Antony system, for running off the exhausted liquors. The muddy deposits are emptied into the waggons W, whilst the clarified boiling liquors pass into the heater A, and are drawn off by the pipe *s* after complete cooling.

The whole apparatus works as follows:—The sewage is lifted

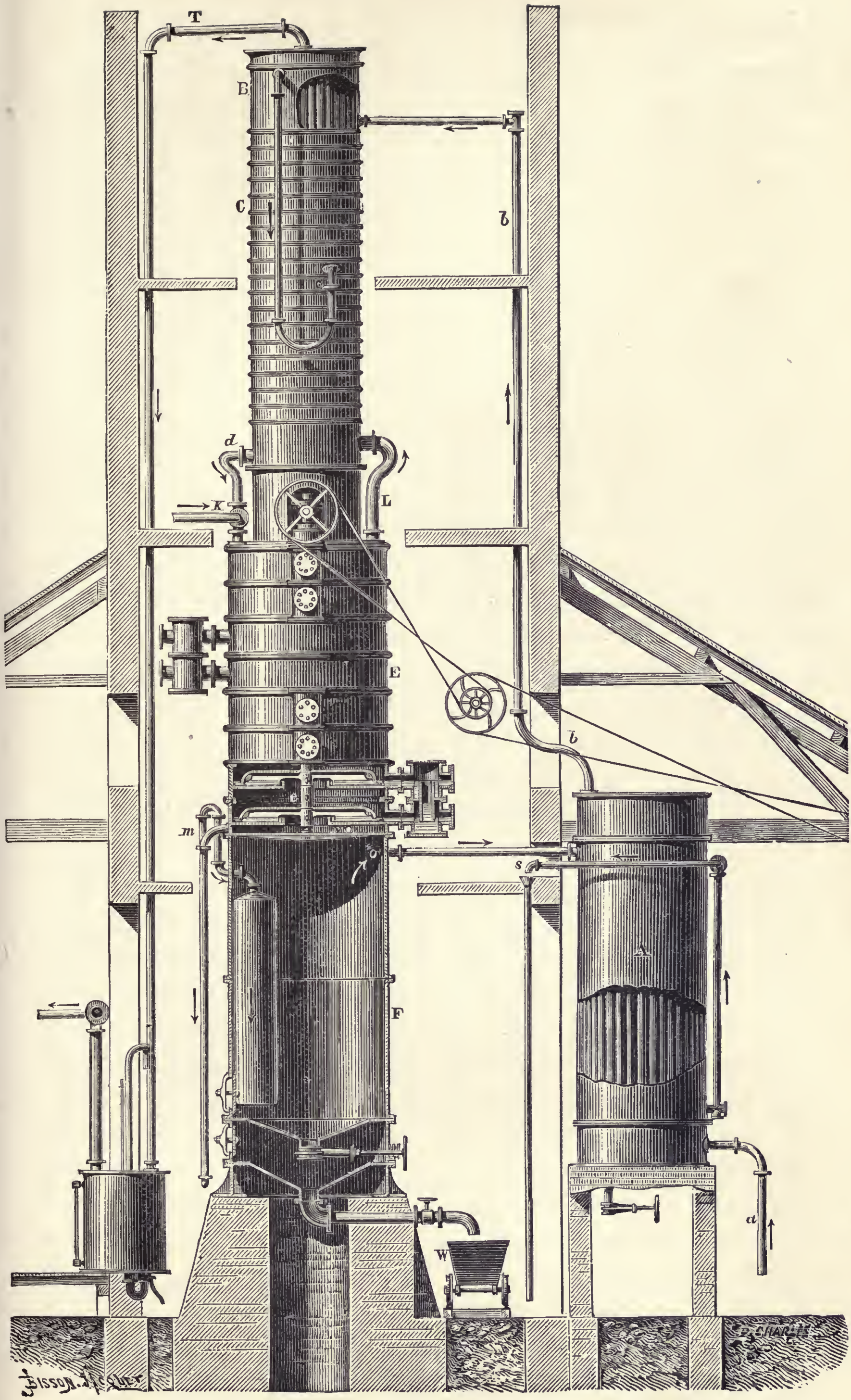

FIG. 9.—Mallet's Apparatus.

by pumps through the pipe *a* into the heater A, where it passes through the tubes, cooling the exhausted liquors which travel in the opposite direction before issuing by the pipe *s*. It then rises through the 4-inch pipe *b* into the analyser B, where it is still further heated by circulating round the tubes of the rectifier C. Issuing then from the analyser by the syphon *c*, it enters the column C at the tenth chamber, and descends regularly to the bottom, where it is delivered by the pipe *d* to the column E, furnished with the stirrers. During this passage the volatile salts of ammonia are carried off by the current of steam rising from the lower column E by the pipe L. The vapours pass through the rectifier C, where they are enriched with ammoniacal products, and then through the analyser, where they part with most of their water, and from which they pass by the pipe T to the sulphuric acid vat. The uncondensed gases are as usual sent to a furnace to be burnt. Before entering the column E by the pipe *d*, the materials which have lost their volatile ammoniacal salts are mixed with milk of lime, pumped in through the pipe K, to decompose the fixed salts; the proportion used being 8 kilos. of lime per cubic metre. This mixture descends from plate to plate as in an ordinary column, but is kept in a state of constant agitation by stirrers, working below the inverted cups, and driven by the shaft *e*. Lastly, the exhausted products, mixed with lime and lime salts, issue from the column E by the syphon *m*, and pass into the mud tank F through a wide descending cylinder. The solid matters settle rapidly, in consequence of the comparative quiescence of the mixture in the mud tank, which is 2 m. in diameter, and owing further to the coagulation of the albuminoid matters by the lime. The clear supernatant liquid flows over to the exchange heater A, where it gives up its heat to fresh sewage, and whence it issues, cooled and clear, by the pipe *s*. The sediments are removed from time to time by opening the valves in the double bottom. They may be put through filter presses to obtain cakes which can be easily dried, and clear filtrate, which is run away. The heating of the whole apparatus is performed by direct introduction of steam from a boiler by the pipe V, 1¼ ins. in diameter, which enters at the bottom of E.

This apparatus will treat 65 c.m. in twenty-four hours.

It does not require frequent cleaning, and exhausts the materials very completely.

The cleansing of the connections of the lime chambers is facilitated by the use of projecting pipes, with 4-way connectors, which can be cleaned by the simple removal of the caps.

Several sets of apparatus on this system have been working for some years, and have given excellent results.

3. *The Lencauchez Apparatus.*—This apparatus is intended to work at a pressure much lower than that of the atmosphere, and to yield either ammonium sulphate or chloride, or even liquid ammonia.

The sewage is prepared as described in Chap. II. § 1, according to Lencauchez' process. The apparatus consists essentially of two distinct parts: one in which the *eaux vannes* are exposed to the action of steam at a reduced pressure, to extract the dissolved gases and the volatile ammoniacal salts, which are then converted either into sulphate or chloride; the other, in which these liquors are subsequently heated with lime, to decompose the fixed ammoniacal salts, coagulate the albuminous matters, and facilitate their settling. We will briefly describe the various parts of this apparatus.

The first part, called the barometric chamber (Fig. 10), consists of a wrought-iron cylinder A, 2·2 m. in diameter and 3·4 m. high, fixed at the top of a vertical hollow cast-iron column B, about 10 m. high, ending below in a syphon *c*, for the escape of the exhausted liquors. This vessel is called the barometric chamber, because a somewhat considerable vacuum can be maintained in it by air-pumps, which continually remove the gases and vapours given off by the *eaux vannes.* These gases are passed into another vessel, where the ammonia is neutralised by sulphuric acid, whilst the exhausted liquors can escape freely at the lower part of the liquid column, which is supported by the pressure of the atmosphere. This chamber is divided into three compartments, each of which contains a small turbine T, the three running on the same vertical axle, and driven by an external pulley *p*. These turbines scatter the *eau vanne* in the form of spray, to facilitate the escape of its volatile constituents.

The *eau vanne*, arriving by the pipe *a*, flows into the first turbine, which throws it against the walls of the chamber. The liquid collected on the first horizontal diaphragm flows into the second turbine, where a second spraying takes place, and thence to the third. Finally, having parted with its gas and volatile ammoniacal salts, it flows freely away down the column B into the cistern C. The waste steam from the engines of the works entering at E, raises the *eau vanne* to ebullition, and is itself in

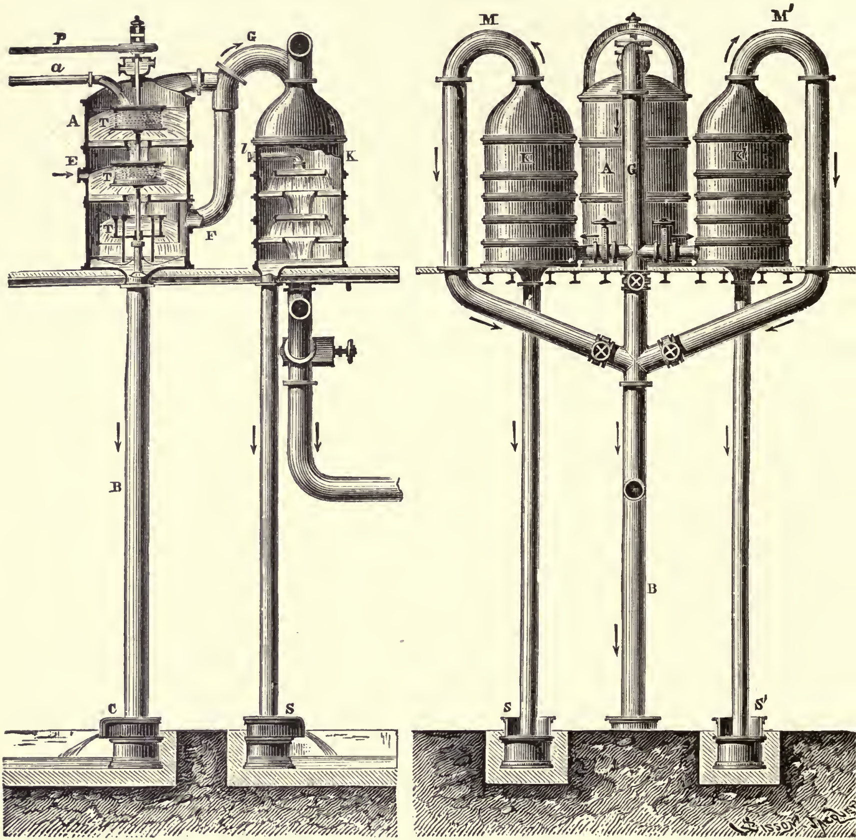

Fig. 10.—Lencauchez' Apparatus. The Barometric and Absorption Chambers.

A, Barometric chamber.
a, Entry for the *eau vanne*.
B, Cast-iron column supporting A, and serving for the escape of the exhausted liquor.
C, Syphon cup for the escape of the exhausted liquor.
E, Entry for steam.
F, G, Pipes conveying the vapours to one of the absorption vessels, and connected with the air-pumps.

K, K′, Absorption vessels.
l, Pipe for introducing acid.
M, M′, Pipes carrying the vapours and uncondensable gases to the air-pumps.
T, T, T, Turbines for spraying the liquids.
S, S, S, Syphon cups for drawing off the solutions of ammonium salts.

great part condensed. Lastly, the partial vacuum is maintained constant in the apparatus by the action of three air-pumps communicating with the barometric chamber. As may be seen, the gases and volatile ammoniacal products are extracted from the liquors in this apparatus—1st, by the rise of temperature produced by the waste steam of the engines; 2nd, by the action of a partial vacuum; 3rd, by the subdivision of the liquor into spray by the turbines. The vapours and gases issuing from the barometric chamber by the pipes F and G, pass through an absorption chamber constructed on the same principle as the preceding, but of smaller dimensions. As shown in the figure, two of these absorbers may be attached and worked alternately.

Each of the absorbers consists of a large cylinder K, 1·75 m. in diameter, furnished with a series of horizontal shelves, over which the acid flows in thin sheets, whilst the gaseous current travels in the opposite direction on its way to the air-pumps. The quantity of acid sent into the absorber, and the time of its contact with the gases, are such that all the ammonia is absorbed, and the acid completely neutralised. The solution of ammonium sulphate thus obtained flows over from the syphon cup S into an evaporation tank. The tube M or M′, according to the absorber which is at work, carries off the excess of steam with the carbonic and hydrosulphuric acids to the air-pumps, which deliver them into a cooled vessel, where the steam condenses, whilst the uncondensable gases are sent to a furnace.

The condensed vapour, still containing a little ammonia, which has escaped absorption, is sent into the lime chamber to be treated with the *eau vanne* from the barometric chamber.

The second part of the apparatus intended for the decomposition of the fixed salts is almost identical with that shown in Fig. 25, p. 83. It consists of a horizontal cylinder 3 m. in diameter and 6 m. long (Fig. 11), through the whole length of which runs a shaft with paddles, which is driven by machinery, to promote the intimate mixture of the liquors with the milk of lime. This shaft carries seven discs, which check the speed of the gaseous current.

The cylinder is divided into eight compartments by seven partitions, *c*, *c*, *c*, perpendicular to its axis and at equal distances apart, as shown in Fig. 11. The first, on the left, reaches to the middle of the cylinder, and each of the others is 0·15 m. shorter than the one preceding it. Into the second compartment there enters a pipe V, bringing waste steam from one or more engines; finally, above the last chamber there is a distillation column with wrought-iron plates. To avoid the choking which might result from materials being thrown up by the ebullition, the lower chambers of this column are not provided with inverted cups. The upper part of the column in Fig. 25 is connected with two return coils c, c', from which crude liquid ammonia flows into a reservoir. If the manufacture of the sulphate is aimed at, the coils being less cooled, the ammoniacal vapours from the last coil should be carried into a sulphuric acid vat. The *eau vanne* which has been deprived of its volatile ammonium compounds is introduced at *a* into the third chamber of the column; it flows down in thin layers and enters the cylinder F, where it becomes mixed with the milk of lime. The ammonia of the fixed salts, set free by the lime, escapes gradually in each chamber, and passes in succession from one to another, bubbling up through a column of *eau vanne* containing

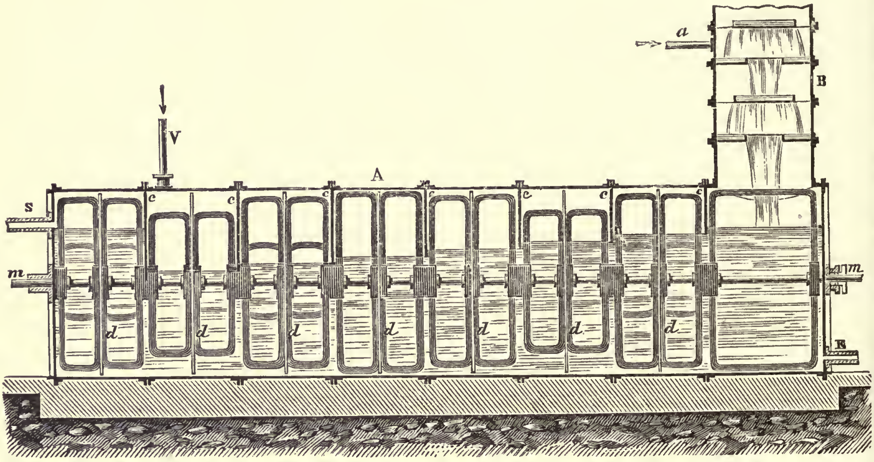

Fig. 11.—Lencauchez' Apparatus. Vertical Section of the Boiler for Decomposing the Fixed Salts.

A, Wrought-iron cylinder in which the fixed salts are decomposed by lime.
a, Entrance of the *eau vanne*.
B, Cascade column.
c, *c*, Fixed partitions of wrought-iron.
d, *d*, Wrought-iron discs fixed to a revolving shaft.
E, Entrance for the milk of lime.
m, *m*, Stirrer.
s, Syphon for the exit of the exhausted *eau vanne*.
V, Entry for steam.

lime, the height of which is limited by the height of the partitions. The ammoniacal vapours, mixed with steam, rise in the column, gradually getting richer, then pass through the coils, where they are deprived of most of their water, which flows back into the column. Ultimately the gaseous products issuing from the terminal coil are conveyed into sulphuric acid, where the ammonia is retained as sulphate.

The *eaux vannes*, which become gradually exhausted as they pass through the compartments of the apparatus in the opposite direction to the steam, escape at last through the syphon *s* into a large tank, where they deposit their suspended matters. The liquor takes about half an hour to pass through the apparatus, and is gradually and completely exhausted.

CHAPTER III.

EXTRACTION OF AMMONIA FROM GAS LIQUOR.

§ 1. Ammoniacal Liquor.

The distillation of coal in retorts for the manufacture of illuminating gas is, as mentioned above, one of the most important sources of ammonia. Coal, under these conditions, yields gas, condensable products, tar, ammoniacal liquor, and a solid residue, coke, which remains in the retorts. In gasworks a yield of 10 kilos. of ammonium sulphate, containing 21 per cent. of nitrogen per ton of coal, is considered exceptional; the French coals contain on the average 1 per cent. of nitrogen, the English as much as 2 per cent. The quantity of ammonia obtained is therefore far short of the nitrogen contained in the coal. Forster has recently shown that coke retains nitrogen, and, moreover, that during the distillation of coal, part of the nitrogen escapes in the free state. Forster's experiments were made with coal containing 1·73 per cent. of nitrogen, yielding 74·46 of coke and 25·54 of volatile matter. The quantity of nitrogen contained in the tar is known to be extremely small. The division of the nitrogen amongst the various products of the distillation was as follows :—

Nitrogen as ammonia . . .	0·251	or 14·50	per cent.
,, as cyanogen . . .	0·027	,, 1·56	,,
,, remaining in coke . .	0·842	,, 48·68	,,
,, not recovered, most of it being in the gas . .	0·610	,, 35·26	,,
	1·730	100·00	

The yield of 0·21 per cent. of the nitrogen in the coal, obtained as ammonia in the best works, is evidently very near the 0·251 obtained by Forster. The state of combination in which the nitrogen exists in the coke is unknown, but it is not unreasonable to suppose that it is as a polymerised cyanogen, that is to say, a nitride of carbon. The action of soda lime on the coke causes it to give up all its nitrogen. The condensed water, and that from the washing of the gas, contains nearly all the ammonia produced by the distillation of the coal, the tar, after the separation of the entangled ammoniacal liquor, retaining only a negligible proportion. The tar and ammoniacal liquors are arrested in coolers and condensers called coke towers. The working up of the tar cannot well be carried out on the small scale; it is therefore in most cases done in special establishments, where it is separated into such commercial products as naphtha, benzenes, heavy oils, naphthalene, anthracene, etc. The ammoniacal liquors, whose value is low, cannot bear cost of carriage; they are therefore always treated on the spot in simple and inexpensive apparatus. As they flow from the condensers they contain tar, and require to be left at rest in large cast-iron tanks or large brickwork cisterns for its separation, so that the ammoniacal liquor can be run off to be worked up. This decantation is of great importance for the manufacture of ammoniacal products, for liquors containing tar do not work well, and give off odorous carburetted vapours which cannot be condensed in the acid employed to absorb the ammonia; the result is an irregular treatment, and complaints from the neighbourhood. Some ten years ago the Parisian Gas Company provoked by this means very serious complaints from the inhabitants of the district surrounding the works at Vaugirard, where the separation of tar was imperfect. In consequence of these complaints, and following the advice of the Council of Public Hygiene and Salubrity of the Department of the Seine, the Parisian Gas Company was requested to take measures for the purification of the workshops at Vaugirard, and to put a stop to a state of things prejudicial to public health. To conform with the requirements of the administration, the Parisian Gas Company has set up settling tanks, which have proved completely satisfactory as regards health, as they now work up only clear ammoniacal liquors, free from tarry matter. The new arrangements adopted in the works at Vaugirard had for their object, as has been shown in the report of the Council of Public Hygiene, edited by M. Combes, Inspector-General of Mines—

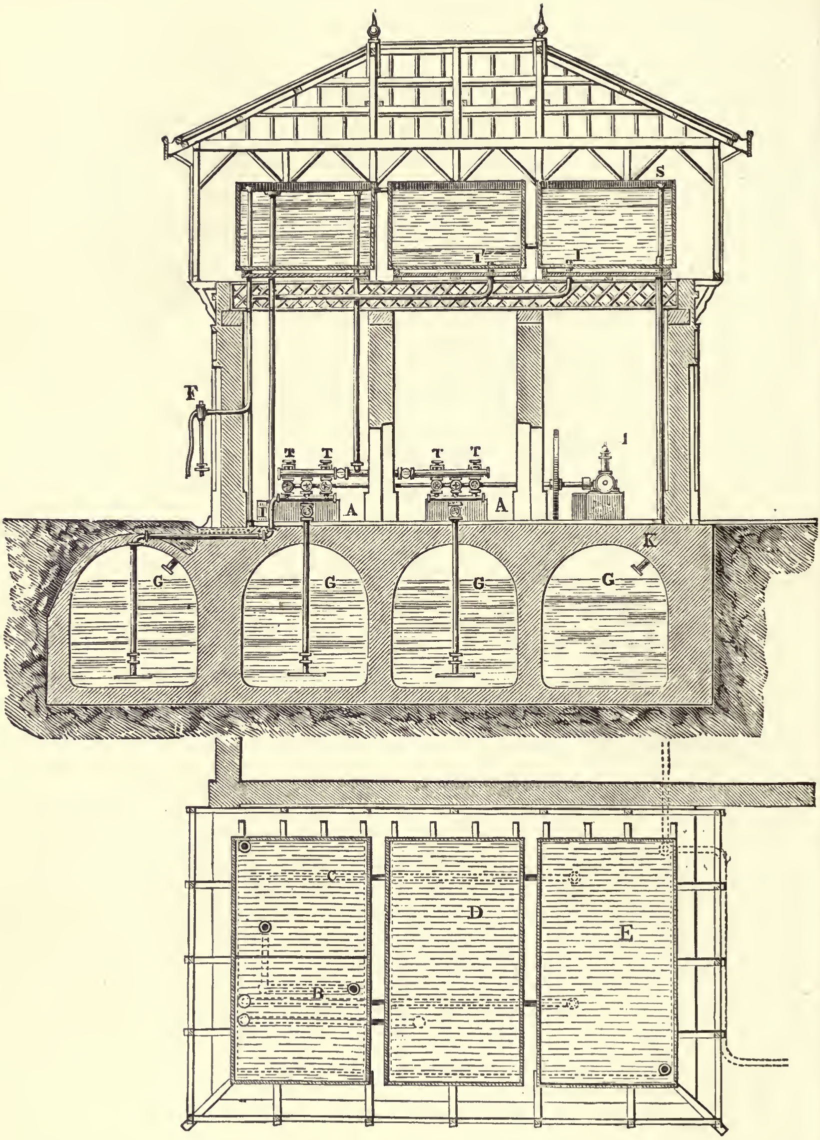

Fig. 12.—Plan and Vertical Section of the Reservoirs and Tanks for the Separation of the Ammoniacal Liquors at the Vaugirard Works.

A, A, Pumps for lifting the mixture of ammoniacal liquor and tar from the reservoirs G, G into the upper tanks.

B, C, D, E, Tanks, connected near the top, for the separation of the water from the tar.

F, Pipe and stop-cock for running off the tar.

G, G, Vaulted brickwork reservoirs for the reception of the mixture of ammoniacal liquor and tar.

K, Pipe for returning into G, G the tar which settles in the upper tanks.

M, Steam engine for driving the pumps.

S, Draw-off of the ammoniacal liquors for distillation.

T, T, Exit valves of the pumps.

1. To separate as perfectly as possible the tar from the ammoniacal liquors, before transferring these to the boilers for distillation with lime.

2. To prevent the escape of ammoniacal vapours when the lime is introduced and mixed with the liquid.

3. To prevent the evolution in the works of empyreumatic vapours from the sulphuric acid vats which receive the products of the distillation, by causing these vapours to pass into one of the chimney stacks of the works, the walls of which are always at a temperature higher than a dull red heat.

To promote the separation of the tar, the ammoniacal liquors, on leaving the coolers, are sent into large brickwork reservoirs, (G, G, Fig. 12), forming the basement of a special building, in the upper part of which are situated four large tanks, protected by a roof.

On the ground floor are the pumps A, A, which raise the mixture in the reservoirs G, G into the upper tanks. A steam engine M drives these pumps. The four tanks B, C, D, E communicate with one another at the tops; the liquor delivered by the pumps into the tank B passes in succession through C, D, and E; the greater part of the tar settles in B and C, a very small amount separating in the other tanks. The ammoniacal liquor, freed almost completely from tar, is drawn off by the pipe S from the surface of the tank E, into gauge tanks at the upper part of each set of stills. The tar which collects at the bottom of the first two tanks is drawn off by the tap F into wrought-iron cylinders, in which it is conveyed to the works at La Villette. This method of procedure yields with regularity clear ammoniacal liquor, which, being free from tar, is easily worked up.

§ 2. Manufacture of Ammonium Sulphate.

1. *A. Mallet's Apparatus.*—The nitrogenous substances in coal, being decomposed by heat during the manufacture of coal gas, yield cyanogen, free nitrogen, and carbonate, acetate, chloride, sulphide, and sulphocyanide of ammonia, the latter of which are condensed by cooling, assisted by the water vapour which accompanies them, and by that which is introduced into the scrubbers. These ammoniacal salts represent, according to Forster, scarcely

15 per cent, of the nitrogen contained in the coal, whilst about 50 per cent. remains in the coke. The complex, tarry liquid which is obtained from the condensing apparatus constitutes the so-called "gas liquor." We have seen that, prior to any other treatment, the crude liquids require to be left to settle, and to be drawn off with great care to free them as much as possible from the tar they contain.

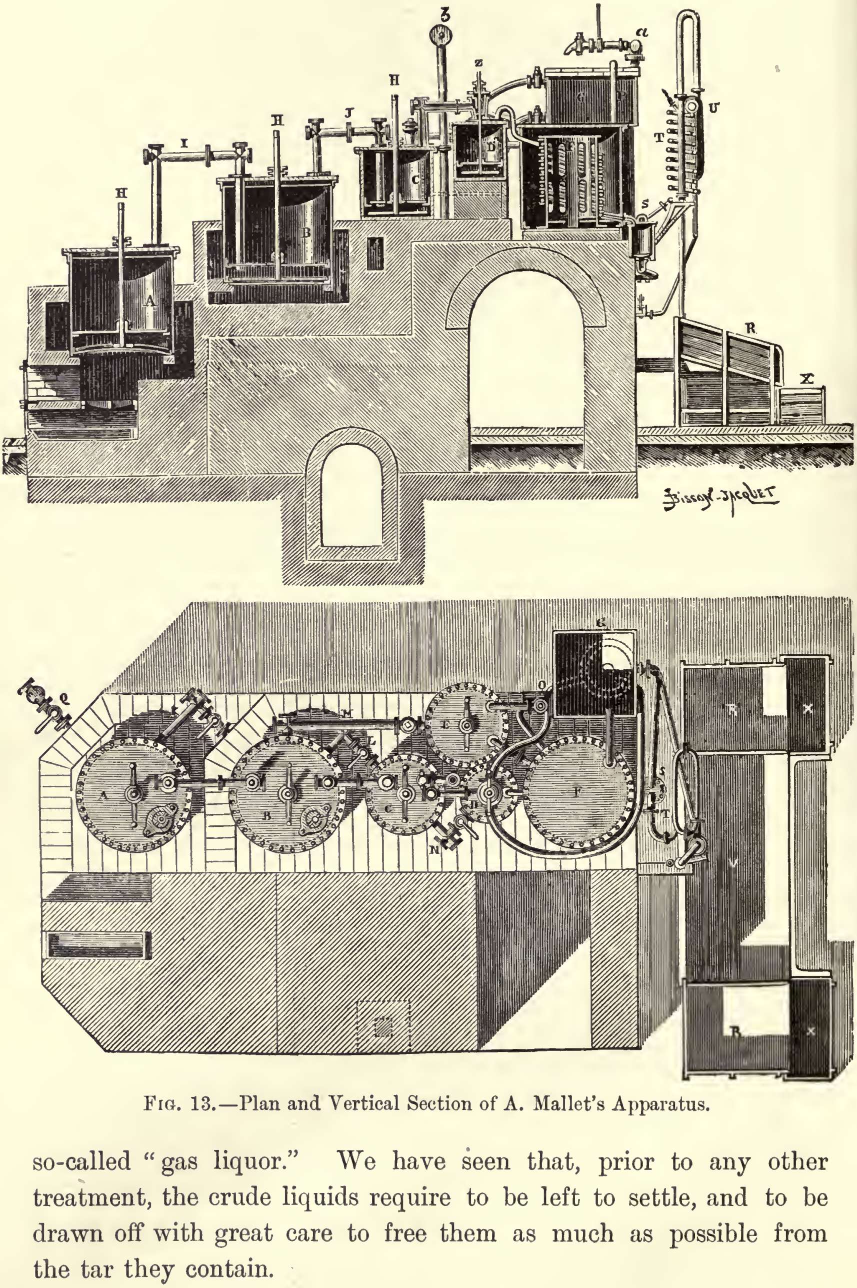

Fig. 13.—Plan and Vertical Section of A. Mallet's Apparatus.

The Parisian Gas Company has adopted for the treatment of its gas liquors, the apparatus devised in 1841 by A. Mallet. This apparatus, which at the present time is fitted up in the works of the company, consists, as is shown in Figs. 13 and 14, of

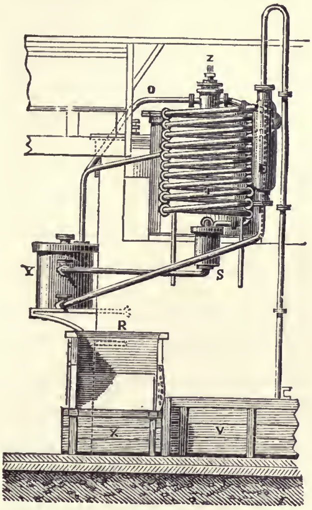

Fig. 14.—Elevation of the Saturation Tank.

Description of Figs. 13 and 14.

A, First boiler, heated directly by a fire.
B, Second boiler, heated by the waste heat of the fire.
C, Third boiler, heated by the steam from B.
D, Fourth boiler, which receives the condensed water from Y.
E, Lime boiler.
F, Charging tank, containing a condensing coil.
G, Tank which measures out the charge for one operation. It is connected with the charging tank by a stopcock at the bottom, and with the lime boiler by a tap at the top.
H, H, H, Stirrers.
I, J, Pipes connecting A with B, and B with C, for the conveyance of the ammoniacal vapours.
K, Pipe for emptying B into A.
L, Pipe for emptying C into B.
M, Pipe for emptying the lime boiler into B.
N, Pipe for emptying D into C.
O, Pipe for emptying the vessel Y into D.
P, Tube conveying the vapours from the tank F into G.
R, Sulphate drainer.
S, Vessel for receiving the liquids condensed by the coil in F, and connected with the vessel Y.
T, Coil, cooled by the atmosphere.
U, Safety vessel, to prevent water from S passing into the absorption vat.
V, Crystalliser.
X, Lead vat to receive the drainings from the salt.
Y, Vessel which receives the liquids condensed in the coils F and T.
Z, Three-way cock by which the air in Y can be compressed to empty that vessel into D.
a, Pipe by which the liquor for treatment arrives.
b, Pipe by which the ammoniacal vapours can be sent to a second absorber during the emptying of the salt.
c, Pipe for emptying the boiler A.

four wrought-iron boilers, A, B, C, D, of unequal capacity, furnished with stirrers, H, H. The two first hold about 2 c.m. The boiler A is heated directly by a coke fire; the second, B, by the waste heat of the fire; the third, C, is not set in the brickwork, and is heated simply by the steam from the boiler B; the fourth, D, serves as a scrubber for the vapours from the earlier ones. The covers of the three boilers A, B, C carry cast-iron pipes I, J, the first of which reaches nearly to the bottom of B, and the second nearly to the bottom of C. The four boilers communicate by the pipes L, M, N, furnished with stop-cocks, to allow the liquors in D to flow into C, those in C into B, and those in B into A. The boiler A has also a draw-off tap, by which its contents can be run to waste. The boiler C is connected with D by a three-way tap Z; lastly, the vessel D is in communication with a cooler F, consisting of cast-iron pipes immersed in cold ammoniacal liquor, which is renewed as the operation proceeds. The vessels S and Y receive the liquids condensed in the coil T; this coil, which is cooled by the surrounding air, is intended to stop the last portions of liquid carried off by the ammonia, which then passes into the sulphuric acid in the vat V. This vat V receives the product from two sets of apparatus. G is a measuring vessel, which receives the charge of ammoniacal liquor and which communicates with the tank F by a tap at the bottom, and with the lime boiler E by a tap *f*. The slaked lime for decomposing the fixed ammoniacal salts is introduced into the vessel E. Part of the ammoniacal liquor passes directly from the measuring vessel into the boiler E, and is there mixed with the lime by working the stirrer.

The following is the course of the operation:—The boilers A and B are charged with the crude liquor from G, which is first mixed with the requisite quantity of lime in E, and passed through B into A. The vessel C is half filled, and also the tank F, with the ammoniacal liquor. A fire is then lit under A, and the contents are stirred from time to time with the agitator H. The vapour produced passes into the boiler B, which it heats rapidly, and drives out the ammonia set at liberty by the lime. The vapours from the boilers A and B then pass into C and D, where they deposit part of the water vapour carried off by the ammonia; they then pass through the coil in the tank F, where further condensation takes place. The liquor in this tank becomes heated, gives off part of its ammonia, which passes into the measuring vessel G by the pipe T, and is there condensed. The impure products condensed in the coil F are collected in the vessel S, and then flow into Y. The gaseous current which has traversed the cooler F passes on to the coil T, which is cooled by the surrounding atmosphere, where its temperature is lowered to about 80°; the products condensed in this last coil also flow into the vessel Y. The ammonia must not be cooled too much before it is sent to the acid vat, as it is desirable to promote the evaporation of the liquor in that vat so as to obtain the sulphate directly in the crystalline state. The liquids collected in the vessel Y are returned to the vessel D, by opening the three-way tap, which produces a pressure on the surface of the liquor in Y, and the tap is closed again as soon as that vessel is empty. After working for about four hours, the liquid in A is exhausted; the fire is then drawn, and the contents run out into the drains. A is then refilled with the contents of B, and B receives the liquor from C, together with a fresh charge from E, mixed with lime. When D becomes too full, it is emptied into C. Before the end of one operation the charge in E is prepared for the next, by opening *f* and allowing the contents of G to enter the tank F at the bottom. As this tank is always full, the liquor entering below drives the hottest liquor from the surface over into E. The hot liquor in F is thus replaced by cold. The ammoniacal liquor is in this way exhausted as it travels towards A, and the vapours enriched as they pass in the opposite direction. Lastly, the cooling of the vapours for the removal of the greater part of the moisture which they carry over, being effected by crude ammoniacal liquor, a great economy of heat results, as only hot liquors are introduced into the boilers.

The absorption vats V (Figs. 13 and 14), which are about 3 m. long, 2 m. wide, and 0·7 m. high, and are lined with lead, are covered during the whole operation; the space under the cover communicates by a large pipe with an underground brickwork conduit, which conveys the gases to one of the chimneys of the works, where the temperature is above a dull red heat. This prevents the dispersal in the atmosphere of the volatile products which accompany the ammoniacal vapours, and which separate as the latter are absorbed. These products are the more abundant, the less perfectly the tar has been separated. When the sulphuric acid in the vat V is saturated, the ammoniacal vapours are sent to a second apparatus, and the salt is removed by shovels to the drainer R, of wood covered with lead, whence the liquid flows into a smaller vat X, measuring 1·2 × 0·5 × 0·4 m. The drained sulphate is then dried on cast-iron plates, heated by the waste heat of the boiler fires, and is then ready to be sent into the market.

Such an apparatus will take a charge of 12 hectolitres (264 gallons) of ammoniacal liquor, and six operations can take place in twenty-four hours. The quantity of fuel varies with the state of the apparatus, which requires frequent cleaning; when thoroughly clean, about 12 hectolitres (33 bushels) of coke will be required per twenty-four hours. The quantity of slaked lime, in sifted powder, is 1·3 hectolitres ($3\frac{1}{2}$ bushels) per charge. About 70 kilos. (154 lbs.) of dry ammonium sulphate are obtained from each cubic metre of liquor of 2°·5 Beaumé, or a total yield of 500 kilos. (half a ton) per twenty-four hours. By conducting the gas from the last coil into water instead of into sulphuric acid, caustic ammonia can be manufactured by this apparatus.

2. *The Modified Mallet Apparatus.*—The foregoing apparatus has, since its original construction, been modified by the inventor. In the new form the analyser consists of a group of tubes and a distillation column, which are more easily cleaned than the coils. Like the older pattern, the modified apparatus permits the manufacture of either caustic ammonia or the sulphate.

To manufacture sulphate, less lime is required than for making caustic, and the analysis of the vapours does not need to be so perfect for the former as for the latter. It is in fact sufficient if the mixture of ammonia and steam yields, with acid of 53° Bé. (specific gravity, 1·54), sulphate of ammonia, which crystallises in the absorption vats without evaporation. But for the manufacture of caustic ammonia the vapours must be passed through a vessel containing milk of lime, and must be thoroughly cooled and purified, as will be seen further on, before being dissolved in water. Mallet's modified apparatus, shown in Figs. 15 and 16, consists essentially —(1) of three wrought-iron boilers, C, D, E, heated by a fire; (2) a tubular heater A, of capacity equal to one of the boilers; (3) a tubular condenser or analyser cooled by a current of water, and intended to dry the ammoniacal vapours to any desired extent. The liquids from the condensation of the vapours flow down from plate to plate, giving up their ammonia, whilst the ascending vapours gradually become sufficiently dry for the manufacture of sulphate; (4) an absorption vat containing acid of 53° Bé. if sulphate is to be made, or an apparatus for further purifying the vapours, and an absorber, if caustic ammonia is required. Fig. 16 shows on a larger scale the arrangement of the pipes which connect the boiler C with the heater A.

To describe the mode of using the apparatus, we will assume it to be working normally, and that the liquor in the boiler E is exhausted. This liquor is then run off by the tap *g*, and is immediately replaced by the contents of D; the latter is in turn charged from C; the fresh hot liquor from A is run into C, and A is replenished with cold. The pipe *j*, which delivers the vapour in E directly to the heater, permits these transferences to be made rapidly, which is of importance as regards the endurance of the boiler, which is heated directly by fire. Similarly, the tap *h* allows the vapours from D to pass into B. By opening the plug L the necessary quantity of lime is added. After replenishing A the tap *j* is closed, that the vapours from D may pass through C. The vapours from E pass successively through D, C, and B, meeting in B the condensed liquid from the heater and the analyser. During this passage they become enriched at the expense of the liquors, and then become dried whilst passing through A and G. The liquor which surrounds the tubes in the heater is raised nearly to boiling point during one operation, and when it is run down into C it soon boils, giving up part of its volatile ammonia; the remainder, together with that from the fixed salts, being expelled in D and E. This method of work requires the consumption of a very small quantity of lime, since most of the volatile salts are expelled before the lime is added.

When the manufacture of caustic ammonia is desired, lime must be added to the boiler C in quantity sufficient to decompose all the ammoniacal salts of the original liquor.

This apparatus serves equally well for gas liquor and for *eau vanne*, and can be worked either by a special fire, or by waste heat, or by a jet of steam.

3. *P. Mallet's Apparatus.*—The apparatus described above is

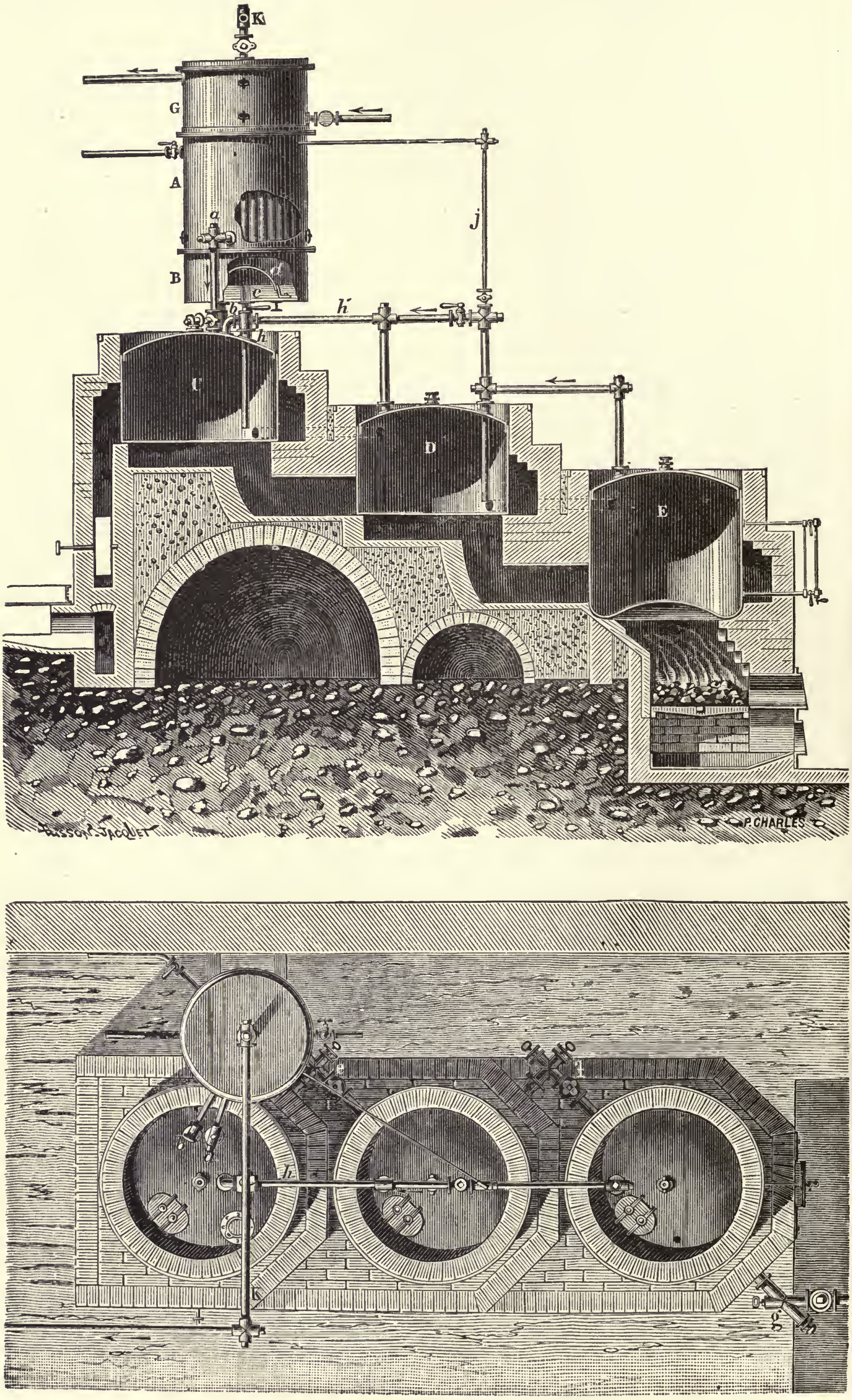

Fig. 15.—Mallet's Modified Apparatus—Plan and Section.

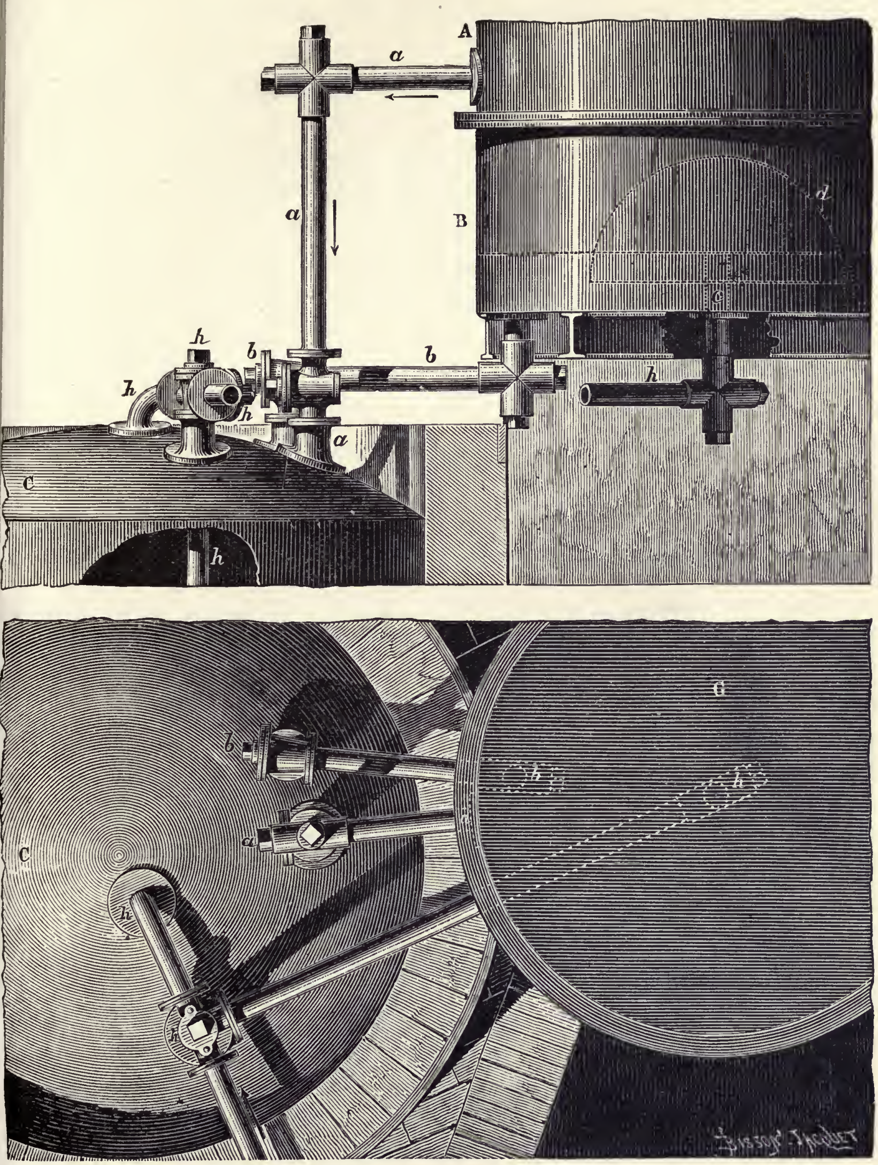

FIG. 16.—Details of Pipe System connecting the Last Boiler with the Heater.

Description of Figs. 15 and 16.

A, Heater containing a charge of gas liquor.
a, Tap for delivering this liquor into the boiler C.
B, Chamber which receives the liquid condensed in the pipes of A.
b, Tap by which this condensed liquor is drawn into C.
C, D, E, Three boilers of equal size, containing stirrers, through which the liquor descends in succession.
c, Pipe which carries the ammoniacal vapours into B.
e, *f*, Taps which place the boilers C, D, and E in communication.
g, Draw-off tap.
h, Tap which allows the vapours from D to pass direct to B.
G, Analyser, cooled by a current of water.
j, Pipe for exposing the contents of A to pressure of vapour from E.
K, Outlet for the ammoniacal vapour.
L, Lime plug.

easily worked, and exhausts the liquors thoroughly, but it is cumbersome and costly.

P. Mallet, in 1869, fitted up at the Colombes works, for the manufacture of ammonium sulphate, an apparatus which, though more simple, exhausts the liquors in a satisfactory manner. This apparatus, shown in Fig. 17, consists of an ordinary distillation column A, of cast-iron, formed by eleven analysing plates and four rectifiers, carrying a heater *a*, and of two boilers B and C, in which lime is used for the decomposition of the fixed salts.

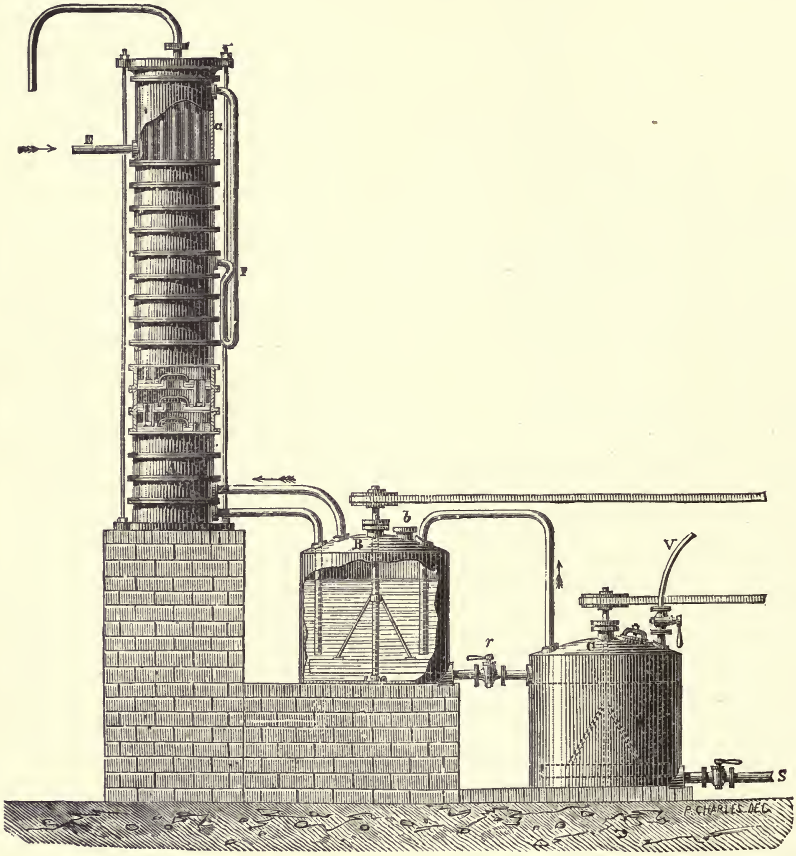

Fig. 17.—P. Mallet's Apparatus.

A, Column consisting of four rectifiers and eleven analysing plates.
a, Tubular analyser.
B and C, Wrought-iron boilers fitted with stirrers.
b, Plug for introdution of lime.
r, Tap for running the liquor from B into C.
S, Exit tap.
V, Steam supply.

The ammoniacal liquors entering at E are first heated by circulating round the tubes of the analyser *a*; they then enter the column by F, flow down from plate to plate into the boilers B and C. During their passage through the column they part with the volatile salts, and the fixed salts are subsequently decomposed by lime in the boilers. The operation is continuous in the column but intermittent in the boilers. This apparatus is inexpensive and gives good results, but occupies much space. It can be worked either by a special fire or by steam, as in the figure.

An apparatus of this description, with a column of fifteen plates 0·8 m. in diameter, and with two boilers of 1 c.m. capacity, can treat 20 c.m. of gas liquor in twenty-four hours.

Unless mixed with a certain quantity of lime, gas liquor very rapidly corrodes wrought-iron. It is therefore necessary to make the heaters of cast-iron, as in the apparatus just described, or else of lead.

When working up rich ammoniacal liquors, there is but little advantage in heating them by the vapours from the column or the waste liquors; on the other hand, fuel is always abundant in gasworks, and steam is often raised by the waste heat of the furnaces.

In 1873, P. Mallet fitted up at the Blois Gasworks an apparatus consisting essentially of a cast-iron distillation column, each plate of which carried a single inverted cup, keyed to a vertical revolving shaft. This ingenious arrangement allowed the milk of lime to be introduced into the column without any risk of obstructions. The same arrangement was introduced by Mallet, in 1881, at the Bordeaux works for the treatment of sewage.

4. *Chevalet's Apparatus.*—This apparatus, already described for the treatment of *eau vanne*, serves equally well for gas liquors. In this case, two to four, or even more boilers are used, according to the amount of liquor requiring treatment daily. Heat may be applied either by a fire under the boilers, or better by a jet of steam, or by thermo-syphon boilers placed in the flues of the retort furnaces. In the last case no fuel is expended, but the working depends on that of the retort furnaces. The work can, however, be made regular by putting the boilers in a special secondary flue,

through which all or part of the flue gases can be sent without interfering with the draught of the furnaces. In small sets of apparatus heated by direct fire, the consumption of coal or coke amounts to 40 kilos. (88 lbs.) per cubic metre of liquor treated; in apparatus of greater size, it may fall to 35 or 30 kilos. The use of waste heat constitutes therefore an important economy in the manufacture of ammonium sulphate.

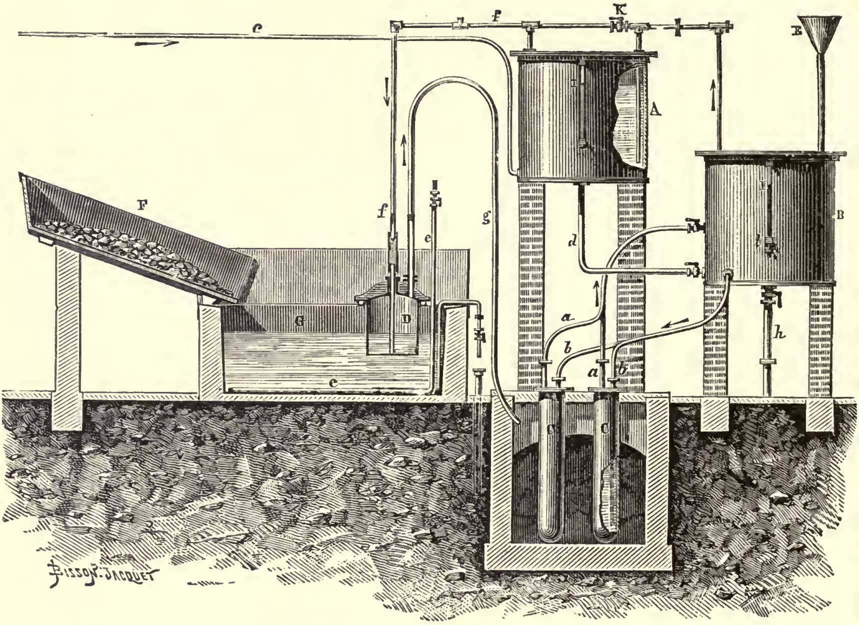

Fig. 18.—Chevalet's Apparatus, with thermo-syphons.

A and B, Wrought-iron boilers.
C, C, Heaters for the thermo-syphons.
D, Leaden bell which collects the gases and water vapour.
E, Lime funnel.
F, Sulphate drainer.
G, Acid vat.
K, Stop-cock which causes the vapours from B to bubble through the liquor in A.
a, *a*, Pipes conveying the hot water from the heaters to B.
b, *b*, Return pipes.
c, Pipe for introducing the ammoniacal liquor.
d, Pipe for emptying A into B.
e, Lead coil for concentrating the sulphate solution by steam.
f, *f*, Ammonia pipes.
g, Pipe conveying unabsorbed gases to flue.
h, Waste pipe for exhausted liquor.
I, I, Water levels.
l, Sampling cock.

Fig. 18 shows the general arrangement of a small Chevalet apparatus, heated by thermo-syphons. It consists of two wrought-iron boilers, A and B, placed at convenient levels for running the liquor from one to the other. The lower boiler B is connected by the iron pipes *a* and *b* to two heaters placed in the furnace flue. The pipes *a*, *a*, which leave the heaters at their upper part, are carried into the boiler B about half-way up, whilst *b*, *b*, which leave B at the bottom, are carried nearly to the bottoms of the heaters.

The two boilers are connected by the pipe *d* for running down the liquor, and by *f*, *f* for the passage of the vapours. When the stop-cock K is closed, the steam from B bubbles through the liquor in A, but passes direct to the sulphuric acid in G when K is opened. The boiler B is fitted with a funnel E for the addition of milk of lime, and a draw-off pipe *h*; A is supplied with ammoniacal liquor by the pipe *c*, and delivers the vapours through *f*. The uncondensed gases and steam from the acid vat are collected by the bell D and sent by the pipe *g* into the hot flue. At the bottom of the acid vat there is a lead pipe *e*, *e*, through which steam from a boiler is passed for concentrating the solution.

The working of the apparatus closely resembles that of the forms already described. Most of the volatile ammoniacal compounds are expelled whilst the liquor is in the boiler A; and when it has been let down into B, a charge of milk of lime equal to about 4 or 5 kilos. of lime per cubic metre of liquor is added, to decompose the fixed salts. This quantity of lime is not sufficient to produce a deposit, and there is no need for frequent cleansing. When working up liquors marking 2° or 3° Bé., as is generally the case, the boilers are emptied twice in twenty-four hours. When richer liquors are being treated, a longer time is required to exhaust them effectually. The residuary liquors contain very little insoluble matter in suspension, and can therefore be run away without clarifying. They contain a quantity of ammonia, which Chevalet estimates as not more than 125 to 250 grms. per cubic metre, corresponding to 500 to 1000 grms. of ammonium sulphate, which it does not pay to extract. Chevalet states that liquors of 3° Bé. yield 22 to 23 kilos. of ammonium sulphate per degree. The yield of ammonium sulphate per ton of coal varies from 4 to 9 kilos., according to the amount of washing to which the gas is submitted. Few works, however, obtain so large a yield; about 6 kilos. per ton. is a good average. Liquors of 5° to 6° Bé. yield 28 to 30 kilos. per degree and per cubic metre; it is therefore necessary, in purchasing gas liquor, to pay regard not merely to the density, but to the probable yield of ammonia. The theoretical quantity of sulphuric acid of

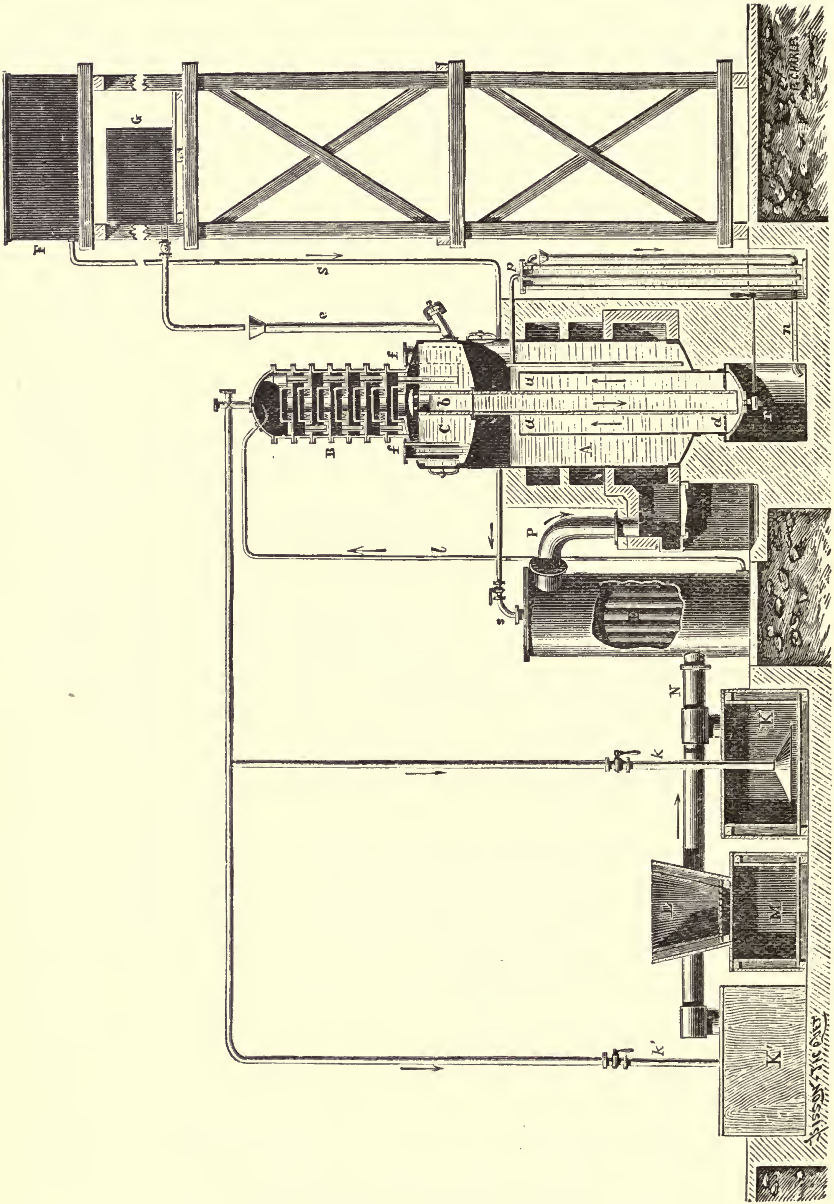

FIG. 19.—Grüneberg's Apparatus.

A, Wrought-iron boiler heated directly by furnace gases.

a, *a*, A wrought-iron cylinder, concentric with A.

B, Cast-iron column.

b, Pipe for the descent of the liquors to the boiler A.

C, Lime boiler.

d, Grating for retaining impurities.

E, Condenser through which the steam from the absorption vats circulates in the opposite direction to the flow of ammoniacal liquor from the reservoir F.

53°, or 54·69 per cent. of sulphuric anhydride for 100 kilos. of ammonium sulphate, is 110·8 kilos.; in practice it varies from 109 to 115. The dried ammonium sulphate contains from 20 to 21 per cent. of nitrogen. M. Chevalet has fitted up apparatus on this system at several gasworks; they permit the treatment at a low cost of considerable amounts of ammoniacal liquor which formerly had to be thrown away, as they would not pay for the erection of more expensive plant.

5. *Grüneberg's Apparatus.*—A very simple apparatus for the economical and thorough extraction of ammoniacal gas liquors has recently been patented by Dr. Grüneberg of Kalk, near Cologne. This apparatus serves for the manufacture of sulphate, or, with some modifications, that of concentrated ammonia. The sulphate apparatus is shown in section in Fig. 19.

It consists essentially of—

1. A vertical wrought-iron boiler, set in brickwork so as to be heated directly by a fire. This boiler contains a large cylinder *a*, *a*, with a grating at the bottom, below which there is a large mud tap. In the centre of this cylinder there is a tube *b*, which reaches nearly to the top of an upper chamber C, which serves as a lime boiler. In this chamber the vapour produced in the lower part bubbles up through the pipes *f*, *f*, keeping the lime in suspension and carrying off the ammoniacal gases.

2. A cast-iron distillation column B, with plates, in which the liquors are deprived of their volatile salts.

3. Acid vats K, K′, for the absorption of the ammoniacal vapour, and arranged so as to collect the uncondensed gases and the steam produced during the neutralisation, and convey them to a cooler E, through which the fresh liquors pass in the opposite direction, and finally to deliver the cooled gases into the furnace.

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e, Inlet for milk of lime.
F, Reservoir of ammoniacal liquor.
G, Lime vat.
f, *f*, Inverted cups.
K, K′, Acid vats, with covers, for collecting unabsorbed gases.
k, *k*′, Pipes conveying the ammoniacal gas.
L, Sulphate drainer.
l, Pipe which conveys hot liquor to the column.
M, Reservoir for the drainage from the sulphate.
N, Pipe which carries gas and steam from the vats to the condenser.
n, Exit for exhausted liquors.
P, Pipe which delivers the uncondensed gases to the furnace.
r, Tap for drawing out the lime mud.
s, *s*, Pipe for introduction of ammoniacal liquor.
p, Syphon pipe through which the exhausted liquors flow away.

4. A reservoir F, containing the liquors to be treated.

5. A lime vat for feeding the chamber C, through a pipe *e*, which is provided with a cleaning plug.

6. A syphon *p*, for the continuous outflow of the exhausted liquor by the pipe *n* and the drain *m*.

The liquor in the reservoir F is allowed to flow in a continuous stream by the pipe *s* into the cooler G, where it becomes heated whilst condensing the steam from the acid vats. It then rises by the pipe *l* to the top of the column B, through which it descends, parting gradually with its volatile ammoniacal salts. It then reaches the lime chamber C, where it is kept in agitation by the steam from A passing up through it by the pipes *f*, *f*. It then flows down the central tube *b*, carrying with it the lime, the coarser portions of which are caught under the grating *d*, and, rising in the concentric tube *a*, *a*, flows over into the boiler A, where it is exposed to the direct heat of the fire, which completes its exhaustion. It then flows away through the syphon *p*, which draws it from near the bottom of the boiler, and delivers it by the pipe *n* into a drain. The lime sediment is drawn off from time to time by the tap *r*; and the separation of the lime deposit from the liquor taking place in the cylinder *a*, which is not in contact with the fire, prevents the formation of incrustations and the consequent burning of the boiler plates. The liquors which flow away by the syphon *p* contain, according to Grüneberg, only 0·05 per cent. of free ammonia and 0·09 per cent. of combined ammonia. The rich ammoniacal vapours which issue from the column B are delivered by the pipes *k* and *k′* alternately to the absorption vats K and K′. So much heat is produced by the neutralisation, that much of the water contained in the acid is evaporated, and the sulphate produced crystallises in the absorption vat. The mixture of steam and uncondensed gases is sent through the cooler E; the steam condenses and flows away, whilst the gases are sent into the furnace and burnt. The sulphate from each vat is dried as soon as the acid is neutralised; the mother liquor is returned to the absorbing vat.

According to Grüneberg, about 50 kilos. (1 cwt.) of coke is burnt for each cubic metre of liquor treated.

Apparatus on this pattern has lately been set up in many localities: at Nice, at Tamaris for treating the ammoniacal liquors from coke ovens, at Valenciennes, Kalk, Frankfort, Hamburg, Göttingen, Goerlitz, Florence, and Palermo; and in 1884 it was established at Danzig, St. Petersburg, Moscow, and Barcelona. This apparatus works well on very varying scales; some of the installations treat only 5 to 10 c.m. of liquor in twenty-four hours, others are much larger.

Grüneberg has produced a modified form of this apparatus for making concentrated liquid ammonia. This will be described in Chap. III. § 3.

§ 3. CONCENTRATION OF AMMONIACAL LIQUOR.

1. *Solvay's Horizontal Still.*—The growing importance of the manufacture of soda by the ammonia process induced Messrs. Solvay some years ago to seek for the means of obtaining, in sufficient quantity and at a low price, the ammonia required by their works. It naturally occurred to them to utilise the crude liquors obtained in the washing of coal gas, which in many gasworks were not treated for the manufacture of ammoniacal salts. These liquors are, however, too weak to bear the cost of transport to any great distance; on the other hand, it would have been undesirable as well as expensive to convert them into solid ammonium salts, seeing that for use in the manufacture of soda it is free caustic ammonia that is needed. They were led therefore to devise means of converting them economically, at the place of production in the gasworks, into a crude product rich enough to bear the cost of carriage. With this object E. Solvay devised a form of continuous still, by which solutions containing 15 per cent. of ammonia could be obtained from ammoniacal gas liquors of 1°·5 to 3° Baumé. This apparatus, shown in Fig. 20, is composed of a long horizontal wrought-iron boiler A, part of which is heated by the furnace F. This boiler is divided into a number of compartments, *b*, *b*, by the partitions *c*, *c*. Each of these contains a vessel *t*, which is in communication with the following chamber by a short pipe, so that the liquid can pass from any compartment into the vessel *t* of the next one, whilst on the other hand the steam and gases produced in any chamber can bubble up through the liquor in the vessel *t* in the chamber which follows in the opposite direction.

The boiler being filled to the working level, which is regulated by the position of the overflow pipe S, the liquor, which has undergone preliminary heating in the vessel H, enters the first compartment of the boiler by the pipe G. The vapours which arrive from the second compartment by the duct D, drive part of this liquor out of the vessel *t* into the similar vessel in the next compartment, when the vapours from the next following one pass through it. The liquor thus gradually approaches the fireplace, whilst the vapours travel in the opposite direction, producing a regular extraction of the ammonia. The exhausted liquor finally flows away by the syphon S, whilst the strong ammoniacal vapours, issuing at the other end, pass through the coil in the tank H, being there cooled

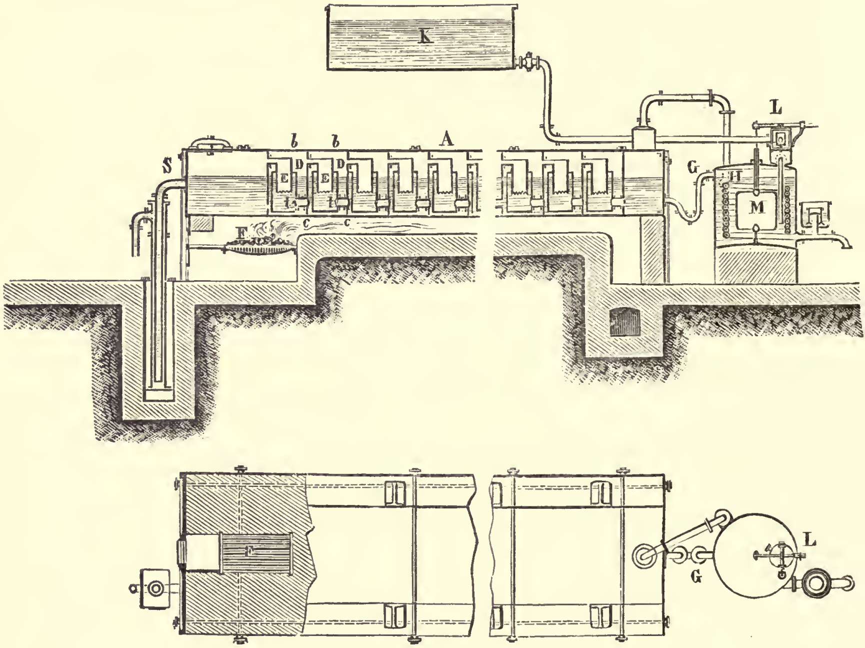

Fig. 20.—Solvay's Apparatus.

A, Boiler.
b, *b*, Compartments.
c, *c*, Partitions.
D, E, Ducts by which the vapours are passed from each compartment through the liquor in the vessels *t*, *t*.
F, Fireplace.
G, Pipe delivering ammoniacal liquor to the boiler.
H, Tank containing the condensation coil.
K, Reservoir of ammoniacal liquor for treatment.
L, Regulator for the flow of liquor.
M, Float which works the regulator.
t, *t*, Internal vessels, each communicating with the preceding compartment.
S, Syphon for escape of exhausted liquor.

and condensed whilst heating the fresh ammoniacal liquor. The rate of advance of the liquor through the apparatus depends on the relative areas of the plunging ducts E and the vessels *t*. The annular space between these should be proportioned to the quantity of liquor and the volume of vapour which it is desired to pass through, and it is to be observed that the advance of the liquid being produced by that of the vapour, the activity of the apparatus is proportional to the heating, and ceases entirely if the fire is extinguished. The steam which is condensed in the coil in the vessel H serves to heat the crude liquor before it enters the boiler. The liquor is passed into the vessel H through a regulator L, controlled by a float M. This float rises or falls as the temperature varies, in such a way that the more the apparatus is heated, and the more steam is produced, the larger is the quantity of liquor admitted. The ammonia gas which escapes from the solution condensed by the coil is arrested by a small acid scrubber, situated above the exit pipe. Solvay's apparatus has been constructed in sizes for treating 12, 18, 24, or 48 c.m. of liquor in twenty-four hours, producing ammonia solution of 12 to 17 per cent. strength. The consumption of fuel varies from 25 to 30 kilos. per c.m. of liquor treated. In dealing with very weak solutions, the heat from the exhausted liquors can be utilised to warm them.

An apparatus with fourteen compartments will treat either 18 c.m. of liquor of 1° to 1°·5 Bé., or 15 to 16 c.m. of 2°. The residual liquors contain at most 100 to 150 grms. of ammonia per cubic metre. To obtain solutions of 16 to 17 per cent. of ammonia, it is necessary to use liquor of 3° Bé.; weaker liquors will not yield more than 12 to 14 per cent. Gas liquor in which part of the ammonia exists as fixed salts, should be treated with lime after the removal of the volatile salts. With this object the liquors are passed through a vessel containing lime, situated between compartments 7 and 8, but it is impossible to add sufficient lime to decompose the fixed salts completely without risk of overheating the exposed plates of the boiler. The plates of the first compartments are rapidly destroyed by the sulphides in the liquors; they require to be renewed about every two years. A thorough cleansing must be given every three or four months to remove the tarry and calcareous incrustations, especially in the compartments immediately over the furnace. In the newest patterns the first five compartments are built of plate 10 mm. ($\frac{3}{8}$ in.) thick; the remainder are of cast-iron, which resists the action of the sulphides better. The arrangement for passing the vapours through the liquid has also been much simplified, as shown in Fig. 21. The common wall of two compartments, A and B, is pierced with two holes, through one of which, *v*, the vapours pass, whilst the other, *l*, brings the liquor in the opposite direction.

In Fig. 21 the part marked *b* is a wrought-iron box open at the top and covering the hole *v*; on the other side of the partition is a flattened box *d* of cast-iron, communicating by the bend E with the compartment C through the opening *l′*. From the positions of the hole *v*, and the tops of the boxes *b* and *d*, relatively to the level of the liquid in the compartments, it follows that the vapours passing from A into B traverse the boxes and throw up the liquor brought by the pipe E. The liquid flowing continuously from compartment C is thus thrown up in B, and the vapours travel in the opposite direction, as in the arrangement previously described. The construction of this new form of apparatus is simpler and less expensive than that of the older one.

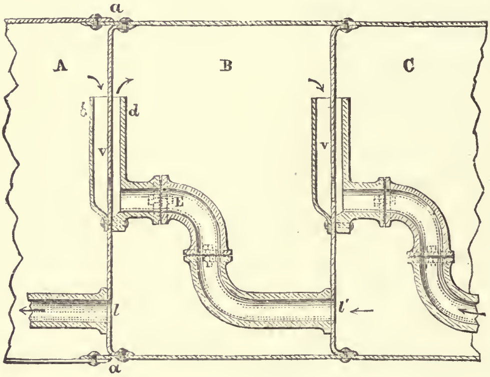

Fig. 21.—New Arrangement of the Boilers in Solvay's Apparatus.

Solvay's apparatus may be used for the manufacture of ammonium sulphate or chloride, by passing the vapours from the boiler into the respective acids, and in this case the tank H can still be used as a heater by passing the exhausted liquors from the syphon S through the coil.

2. *Kuentz's Apparatus.*—M. Henry Kuentz has invented and constructed a simple and inexpensive apparatus for the production of concentrated gas liquor. This apparatus, shown in Fig. 22, consists of two distinct parts, the first of which serves for the extraction, the second for the condensation, of the ammonia. The

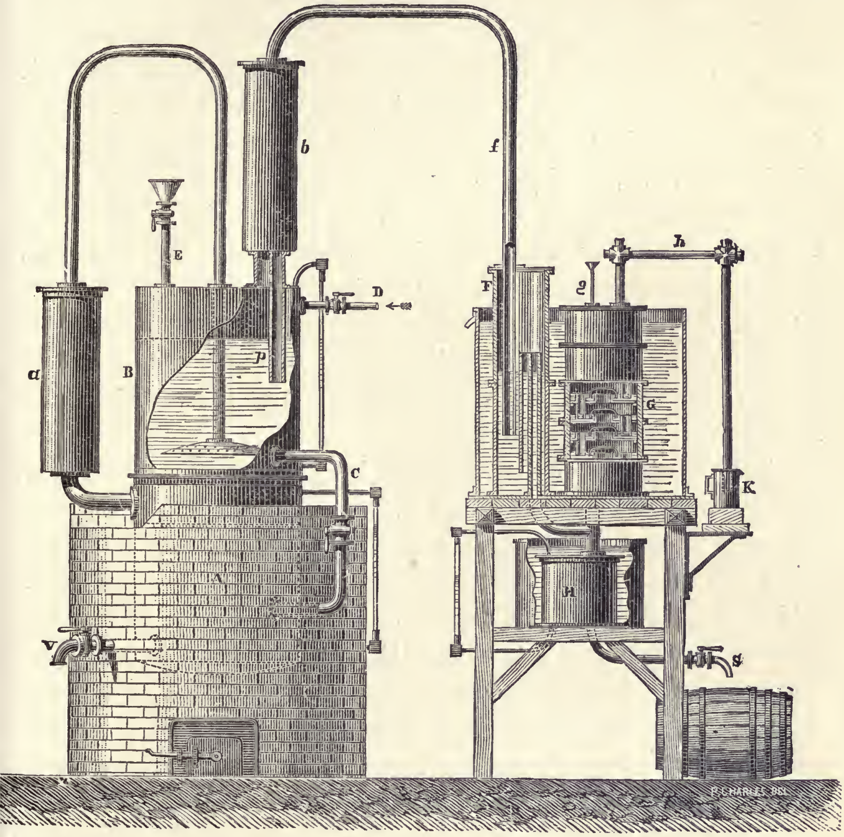

FIG. 22.—Kuentz's Apparatus.

A, Wrought-iron boiler, heated directly by a fire.
B, Upper boiler.
a, *b*, Froth separators.
C, Pipe for running down the contents of B into A.
D, Pipe for introduction of gas liquor.
F, Oval cast-iron pipe for the preliminary condensation of the steam.
f, Pipe conveying the vapours to the condenser.
G, Cast-iron scrubber in which condensation is completed.
g, Pipe for running water into the scrubber.
H, Cooled receiver for the concentrated solution.
h, Pipe for the escape of uncondensable gases.
p, Pipe for returning liquid from the froth separator *b*.
S, Tap for drawing off concentrated ammoniacal solution.

extraction apparatus consists of two wrought-iron boilers, A and B, of 750 to 1000 litres effective capacity, the lower of which is set in a furnace. The vapours given off by this boiler pass through a froth separator *a*, and then pass into the boiler B through a pipe which ends in a very shallow cone. Above the boiler B there is a second froth separator *b*, constructed, as shown in the figure, of a wide wrought-iron cylinder about a metre high, in which the froth, carried up by the gaseous current, breaks and flows back as liquid to the boiler by the sealed pipe *p*, whilst the vapour passes on by the upper pipe to the condensing apparatus. A pipe with stop-cock C, allows the liquor in B to be run down into A, and a waste-cock V draws off the exhausted contents of the boiler A. The gas liquor is brought to the boiler B by the pipe D; the funnel E is for the introduction of lime. The boilers are furnished with level gauges.

The second part of the apparatus, intended for the condensation, is constructed with an oval tube F, into which the vapours pass by the pipe *f*, and of a cast-iron scrubbing column G. These two parts are contained in a wrought-iron tank filled with cold water, which is kept constantly renewed. A small stream of water is allowed to flow into the column G by the pipe *g*, to assist the condensation. The uncondensed gases escape by the pipe *h*, and bubble up through water in a small vessel K, allowing the progress of the operation to be watched, or they may with advantage be passed through a lime purifier and then into a furnace.

The vessel H, which is surrounded by cold water, receives the concentrated ammonia solution from the condensers, and this is then drawn off by the tap S.

The course of an operation is as follows:—The exhausted liquor in the boiler A is drawn off after quenching the fire; the contents of B are run down into A, and a fresh charge is introduced into B. The milk of lime requsite for decomposing the fixed salts is added through the funnel E, and the boiler A is heated up. The steam from A, accompanied by a diminishing proportion of ammonia, passes through the froth separator, and rises through the liquor in B, which soon enters into ebullition, giving off the greater part of its ammonia; this becomes partially condensed in the water which accumulates at the bottom of the tube F. The ammonia solution thus produced flows over into the vessel H, whilst the unabsorbed gases rise through the scrubber C, meeting a stream of water from the funnel *g*, the quantity of which is regulated so as to secure complete condensation, and yield a solution of any desired strength. The uncondensable gases pass off by the pipe *h* through the vessel K. When the vessel H is filled to a fixed level, as shown by the gauge-glass, the operation is ended, and the liquors in the boilers are changed. Each operation lasts from 2½ to 4 hours, according to the quality of the fuel and the skill of the workmen. The concentrated solutions obtained contain 13 to 15 per cent. of ammonia.

Apparatus of this pattern is able to extract the ammonia from gas liquor very perfectly; it is inexpensive and easy to work, and has therefore been established in many places. There are upwards of ninety at work in gasworks, both in France and elsewhere. Kuentz's condenser attached to any of the stills intended for making ammonium sulphate, permits the production of concentrated ammonia liquor.

3. *Grüneberg's Apparatus.*—We have already described the apparatus devised by Grüneberg for manufacturing ammonium sulphate from gas liquor. In a modified form this apparatus is equally suitable for producing concentrated ammonia liquor. The arrangement for this purpose is shown in section in Fig. 23. At the top of the distillation column of the apparatus already described, there is attached a long pipe R, through which the ammoniacal vapours have to pass before reaching the condenser. This pipe is cooled by a water-jacket, to condense part of the water-vapour. The remainder is condensed in the coil D, producing a concentrated solution of ammonia, which flows first into the trapped tank E, and thence into a reservoir. The tube *o* conveys the uncondensable gases to a washing tank H, where they give up their last traces of ammonia, and the pipe *p* then conducts them to the grate of a fire. The crude gas liquor in the reservoir F flows in a constant stream through the covered tank containing the coil D, and after becoming warm passes over to the column by the curved pipe *l*, and passes through the still in the manner already described. This form of apparatus will economically extract comparatively large volumes of gas liquor, and yields solutions of 12° to 14°. It is free from the risk of burning out the plates, which is always present when tarry gas liquors are heated with lime, and it is

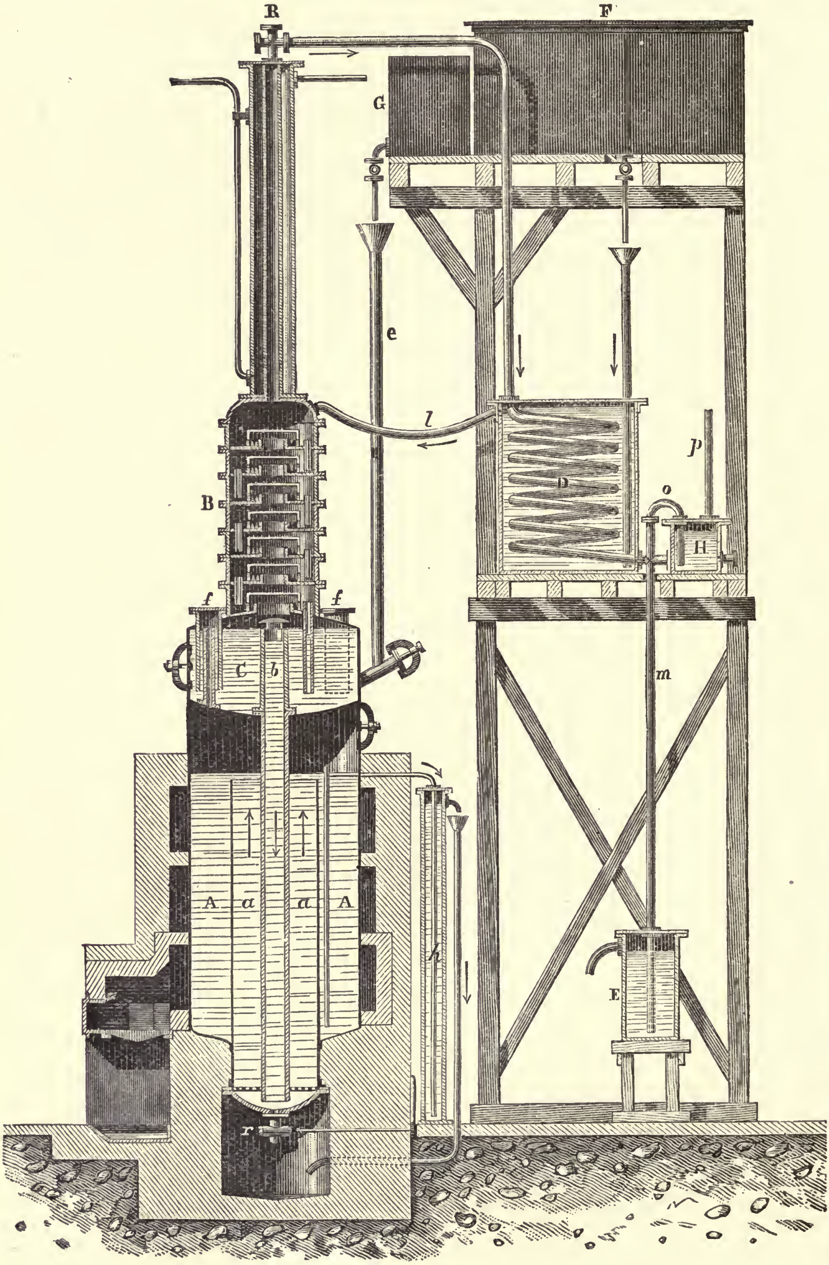

Fig. 23.—Grüneberg's Apparatus for the Manufacture of Concentrated Ammoniacal Liquor.

A, Wrought-iron boiler heated by the flame from a furnace.

a, *a*, Concentric cylinder in which the lime settles.

easily worked. A model with a boiler 1·5 m. in diameter and 3 m. high, will treat 10 c.m. of gas liquor in twenty-four hours.

B, Column.
b, Pipe through which the liquor descends.
C, Lime boiler.
D, Condenser for the ammoniacal vapours.
d, Lime strainer.
E, Receiver for the concentrated ammoniacal liquor.
e, Funnel for introducing milk of lime.
F, Reservoir of gas liquor.
f, *f*, Scrubbers.
G, Vat for milk of lime.
h, Syphon for running away exhausted liquor.
H, Washing vessel for the uncondensed gas.
l, Pipe for bringing the gas liquor to the column.
m, Downflow pipe for the concentrated liquor.
o, Pipe which carries the gas to the washing vessel.
p, Escape pipe for uncondensable gas.
R, Pipe, cooled by water-jacket, for conveying the ammoniacal vapour.
r, Draw-off for the lime mud.

CHAPTER IV.

MANUFACTURE OF AMMONIACAL PRODUCTS FROM BONES, NITROGENOUS WASTE, BEETROOT SPIRIT WASH (VINASSE), AND PEAT.

§ 1. Ammonia from Bones.

The bones from slaughter-houses or knackers' yards are employed for manufacturing either gelatine and phosphate of lime, or else bone charcoal and ammoniacal salts. Fresh bones, to which fragments of muscle still adhere, are soaked for twenty-four hours in hot water to allow them to be readily cleaned. When the soaking is finished, the liquid is run off, and the bones, freed from fleshy particles, are crushed between channelled rollers; they are then boiled to extract the fat. The boiling is carried out in the following manner:—The crushed bone is received in a wrought-iron basket, which can be transferred by a crane to a wooden vat containing water. This water can be rapidly boiled up by a steam coil lying on the floor of the vat. The boiling water extracts the fat from the bone, and causes it to rise to the surface. The fat is collected by a skimmer, strained through a sieve, and employed for soap-making. After two and a half hours' boiling with water the bones give up no more fat; the basket is then lifted out, and the contents submitted to copious washings. The bone is then thrown out in heaps, and allowed to dry spontaneously. When dry it is passed through mills, which reduce it to fragments of suitable size for making bone charcoal. These are freed by sifting from the fine powder which is employed for making phosphatic manure. The coarser fragments are then submitted to calcination, either in crucibles of clay or cast-iron, or in continuous furnaces. Calcination in crucibles does not allow the volatile products to be collected, as these burn in the heating chamber;

by using retorts the volatile products can be collected and the gases utilised for the partial heating of the retorts. A group of retorts, which are either of cast-iron, or better of fire-clay, and of cylindrical or oval section, are arranged vertically in a chamber which can be heated by one or two fires. Each retort is furnished at the top with a charging box, of cast-iron, situated outside the brickwork; this box has a wide opening in the bottom, which is closed by a luted plug, and one at the side for conveying the vapours and gases to the condensers. Each retort has also an opening at the bottom, which is closed during the distillation. The volatile products pass into a hydraulic main containing water, where a preliminary condensation takes place. A small stream of water is allowed to flow into the main to keep the level of the liquid constant, so that the delivery pipes may always dip to the same depth, and also to prevent the deposition of solid ammonium carbonate. The vapours which escape from the hydraulic main are carried first through a condenser heated by an exterior current of water, and then through a scrubber, where they meet a small stream of cold water, which frees them as perfectly as possible from the ammonia they contain. They are then passed into the retort fires and burnt. The liquors from the condenser, the hydraulic main, and the scrubbers, are collected in a wrought-iron vat, and serve for the manufacture of ammoniacal salts. The liquid from the condenser is of 14° to 15° Bé., that from the scrubber 1° or 2° only. On remaining at rest these liquids separate into two layers. An upper oily one, of very complex composition, is known as Dippel's oil; it contains a large number of compounds, especially bases of the pyridine series, of which at present no use has been made. These oils are either burnt in the furnaces or used for making illuminating gas. The lower aqueous layer is a solution of ammonium carbonate contaminated with tarry substances, and is employed for the manufacture of ammoniacal salts by distilling it and absorbing the ammonia by an acid. The salts obtained are deeply coloured by the small quantities of oily products which they contain. One hundred kilos. (2 cwt.) of bone, treated as described, yield 6 to 7 kilos. of ammonium sulphate, containing 18 to 19 per cent. of nitrogen and 1·7 to 2 kilos. of oil. A method formerly employed in France, and still occasionally used, for converting the carbonate of ammonia of the crude liquors into sulphate, consisted in filtering the liquor, after freeing from tar, through a layer of gypsum, when decomposition took place with formation of carbonate of lime and sulphate of ammonia, which merely required concentration.

For the manufacture of ammonium chloride from these liquors, see Chap. V.

§ 2. AMMONIA FROM NITROGENOUS WASTE.

Waste wool, hide, leather, horn, feathers, sponge, etc., containing 6 to 15 per cent. of nitrogen, is habitually worked up into manure. The decomposition of these substances in the soil is extremely slow, it is therefore advantageous to convert their nitrogen into ammonia. L'Hote's method consists in treating them with a 10 per cent. solution of caustic soda, which either dissolves them completely or brings about a complete disintegration. The mixture obtained is made into a paste with lime and introduced into a cast-iron retort. The retort is carefully heated to avoid decomposing the ammonia, and the vapours are passed into chamber sulphuric acid, in which they are condensed. Towards the end of the operation the retort should be raised to a red heat. The white pulverulent residue consists of carbonate of soda and lime, and when treated with water reproduces caustic soda, which is used over again. In this way the whole of the organic nitrogen in the original substance may be recovered as sulphate of ammonia. The sulphate obtained directly by this process is coloured, which however is no obstacle to its use in agriculture. Its purification, if necessary, would be easy.

§ 3. AMMONIA FROM BEETROOT (VINASSE).

The manufacture of ammoniacal salts at the same time as salts of the methylamines, by calcining the residue from the distillation of beetroot spirit, has been carried on for some years. A mixture is obtained of gaseous and condensable products, which on cooling yields a complex tarry liquid containing carbonate, cyanide, chloride, sulphide, etc., of ammonia and methylamines. When this mixture is neutralised with an acid and evaporated, the ammoniacal salts, which are less soluble than those of the methylamines, can be crystallised out. The mother liquor contains chiefly salts of trimethylamine and dimethylamine, and is used for the manufacture of methyl chloride.[1] Hydrochloride of trimethylamine is decomposed by heat. At about 285° C. the gaseous products evolved consist entirely of free trimethylamine and methyl chloride; whilst the solid residue contains hydrochloride of monomethylamine and unaltered hydrochloride of trimethylamine. The decomposition is represented by the equation $3\ N(CH_3)_3.\ HCl = 2N(CH_3)_3 + 2CH_3Cl + N(CH_3)H_2.\ HCl$. Above 305° the residue consists entirely of monomethylamine hydrochloride mixed with ammonium chloride. The gaseous products then contain much ammonia mixed with methyl chloride. Finally, at about 325°, the substance is wholly decomposed or sublimed. Above 305° the principal reaction may be represented by the equation $N(CH_3)H_2.\ HCl = CH_3Cl + NH_3$. The products of a dry distillation of trimethylamine hydrochloride are therefore methyl chloride, trimethylamine, and ammonia. The hydrochloride of dimethylamine behaves in a similar manner. The gaseous mixture, passed into commercial hydrochloric acid, deposits the alkaline products, ammonia and the methylamines, whilst the methyl chloride, washed with water, and collected in a gasholder, is liquefied by compression after being completely dried by sulphuric acid. Methyl chloride is now manufactured commercially by this method, and is used both in the preparation of coal tar colours and in freezing machines.

The chlorhydrates of ammonia and the methylamines are easily separated by crystallisation and centrifugal extraction, ammonium chloride being much less soluble than the other salts. The methylamine salts in the mother liquors are reworked for making methyl chloride. The sal ammoniac obtained by this process is rendered impure by chlorides of iron and lead from the boilers and evaporation coils; to purify it for use in Leclanché batteries, it is redissolved and treated with ammonium sulphide to precipitate the heavy metals. By recrystallisation and hydro-extraction a very pure product is obtained, which finds its application in telegraphy.

§ 4. AMMONIA FROM PEAT.

1. Peat is a highly important fuel, which at the present time is being formed by the decomposition of aquatic plants in marshy

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[1] C. Vincent, *Comptes rendus de l'Académie des sciences*, 21st May and 8th October 1877.

localities. Peat often contains the remains of trees which have undergone slow decomposition under water, and which have nourished the growth of plants which only live on decomposing lignite. These plants have grown and perished, and their remains have served as the soil of a new layer of vegetation. In this way a deposit of peat has been gradually formed, the thickness of which has continued to increase until it reached the surface of the water. Peat bogs have therefore had their origin either in a lake or pool, or even in an arm of the sea, which have in this way become gradually filled up. It is the opinion of some authorities that thirty to forty years are required to form a layer of peat 1 m. in thickness; others estimate that under the best conditions the production does not exceed 0·6 metre in a century. Both views are correct to this extent, that a century is required to form 0·6 m. of peat of good quality, whilst in thirty to forty years it is possible for a growth to form 1 m. thick of an imperfect peat which is not worth the trouble of working. It is of importance that the water of a peat bog should be renewed, for in stagnant water the decomposition of the vegetation would be complete, and peat would not be formed; but the water-current must be a slow one, and must not remove the débris on which the formation of peat partly depends.

There are found in Picardy two kinds of peat: one compact and foliated, which has been produced by the decomposition of wood or large plants; the other light, derived from the *Equisetaceæ* rushes, and mosses which have grown on the former. This spongy peat is sometimes rich in mineral matters, which have been introduced by floods. These mineral matters, crushed shells and stones, make the peat difficult to burn. Such peat (*tourbe à cendres*) is of very inferior value.

Peat when dug out from the bog can be worked up into a soft paste like clay, and moulded into bricks (*briquettes*); these, exposed to the air under sheds, rapidly lose most of their water and shrink considerably, sometimes to one-sixth of their original bulk. When, however, peat ceases to lose weight under these conditions, it is far from being dry; it retains a proportion of hygrometric moisture which is the more considerable the poorer the peat is in mineral matters. Thus the peat of first quality from the Somme retains 10 to 30 per cent. of water after long exposure to air, whilst under the same conditions an ashy peat will retain only 5 to 15 per cent. The proportion of moisture retained by peat depends also on its physical condition. Thus, when it has been well kneaded, then moulded, and dried for six months under a shed, peat of the first quality is compact, and contains only 10 to 14 per cent. of moisture, whilst bulky peat under the same conditions of exposure may contain 25 to 30 per cent. A satisfactory product should be hard and compact, have a density varying from 0·45 to 0·7, contain not more than 10 to 15 per cent. of moisture, and 5 to 15 per cent. of ash.

The peat as soon as it is dug out is treated as follows, according to Challeton's process:—It is first thoroughly disintegrated by a pulping machine, and then thrown upon an oscillating sieve, which allows the pulp to pass through, but keeps back the roots and coarse herbaceous fragments, which are utilised as fuel in the boiler fires of the establishment. The pulp falls into a wooden tub, where it is kept in suspension by a mechanical stirrer, whilst the sand, chalk, and fragments of shells are allowed to settle. This apparatus might be replaced by a machine such as is used for washing small coal.

The pulp is now pumped out of the tub into wooden frames 2·5 m. square and 0·6 m. deep, where the water drains away from it; or any other suitable form of filter may be used. When sufficiently drained to be plastic, the peat is moulded by hand and the bricks are dried under a shed. Good peat, like that of Essonnes or the Somme, which contains neither sand, shells, nor more than about 2 per cent. of extraneous mineral matters, can be dried without washing.

A much simpler and wholly mechanical method of treating peat has been established by Messrs. Bocquet and Bénard at Mareuil. Their process converts the original peat into a homogeneous paste, and yields hard, firm bricks of one quality only. Two different modes of procedure are adopted, according to circumstances: (1) If the peat bog is of sufficient size, the whole apparatus is put upon it on a floating dredge. The peat brought up by the dredge falls into a mill, where it is converted into a homogeneous pulp. This pulp is delivered into moulding trucks, where it is shaped into bricks, lightly pressed, and deposited on a drying floor. (2) If the bog is not capable of floating a boat, the peat is dug out by an excavator. In this case two parallel tram lines are laid down; one carries the excavator, the other the trucks for removing the peat to the factory. The pulp delivered by the mills is run into a tilt waggon, which carries it to the moulds, and the bricks are treated and dried as before. Challeton's process gives the purest product, but is expensive; by Bocquet and Bénard's process, on the contrary, the entire peat is pulped and made into bricks containing from 5 to 12 per cent. of mineral matter, and retaining 12 to 15 per cent. of water. When treated by Challeton's process, the Mareuil peat loses 50 per cent. in weight by the removal of the coarse portions, and retains only 3 per cent. of mineral matter, but its price is more than double. Machine pulping, by allowing greater compression than is obtainable by hand, reduces to one-half the number of bricks obtained from a given quantity of peat. Great economy is thus attained, and the bricks keep their shape, and their drying being slower they acquire greater density.

Kolb, by a series of reduction tests with litharge, has determined the calorific power of peat from various sources: he finds for good dry peat a calorific power of 3100 to 3500 calories The calorific power of oak-wood being 3500 calories, and that of the best coal about 6000 calories, it is obvious that peat is a very important industrial fuel. It may be utilised in four different ways—

(1) By burning it directly, after removal of its moisture by stoving; (2) by converting it by carbonisation into charcoal, which can be used in blast furnaces; (3) by converting it into producer gas, together with inferior fuel, which cannot be used in this way alone; (4) by using it alone in special gas producers.

By carbonisation in closed vessels, peat yields a hard charcoal, 60 per cent. of combustible gas, a tar particularly rich in phenols, paraffins, and acetic acid, and an ammoniacal liquor containing as much as 2 parts of ammonia per 100 of peat. These ammoniacal products can be collected even when the peat is converted into producer gas. As stated above, coal, when distilled, produces on the average 6 kilos. (13 lbs.) of ammonium sulphate per ton; *eau vanne*, 9 to 10 kilos. (20 to 22 lbs.) per cubic metre; whilst the best peat may yield 80 kilos. (176 lbs.) per ton; that of second quality, 68 kilos. (150 lbs.); and of third quality, about 40 kilos. (88 lbs.) per ton. Peat is therefore the richest raw material, and the most advantageous to work for ammonia, as will be seen below.

When peat is to be burnt under steam boilers or in metallurgical furnaces, it must first be deprived of its moisture, and when it is to be converted into charcoal this is equally desirable, so as to obtain the greatest yield. For drying, the peat must be exposed for thirty-six to forty-eight hours to a temperature which must not exceed 100° C., for even at 110° or 115°, according to Kolb, decomposition commences. Lencauchez has invented a brickwork stove which will hold 40 to 50 tons of peat, and which can be heated by waste heat from various sources. The description of this ingenious apparatus would take us too far from our subject; we therefore refer the reader to Lencauchez's treatise on Peat (Lacroix), from which we have drawn much information on this subject.

The well-dried peat can be burnt under boilers, in locomotives, or in blast furnaces; after carbonisation it is employed for domestic purposes, or instead of wood charcoal for metallurgy. The manufacture of charcoal from peat can be carried out in a modified Knab furnace, worked on the regenerator system. The vapours and gases given off are cooled in condensers resembling those of gasworks, to allow of the collection of the tar and ammoniacal liquor. The quantity of gas obtained is sufficient to keep up the heat; the gas producer is therefore only used to start the operation. Peat charcoal, when drawn from the furnace, must be received in extinguishing boxes, or it would continue to burn like wood charcoal. When the ammoniacal products are to be collected from peat employed in gas producers, as mentioned above, two cases arise: one in which the peat is mixed with inferior fuel, the other when it is used alone. In the former case the peat, used in the form of bricks, enables three to six times its weight of poor fuel, such as small coal, anthracite, etc., to be employed without choking, and since the ash of peat is very refractory it tends to hinder the formation of clinker, which would obstruct the lower part of the grate and necessitate frequent cleaning. The peat employed in this case need not be stoved, but may contain 15 to 20 per cent. of water. When, however, the fuel which is mixed with the peat yields very little ammonia, the peat should be dried. The gas is then used at once for burning. When, on the other hand, the added fuel yields abundance of ammonia, the peat should be used without drying, and the gases cooled in a condenser or scrubber, with the object of obtaining ammoniacal liquor from which to manufacture ammonium sulphate. To ascertain which of the two systems should be adopted, it is necessary to know how much ammonia the producer will yield daily with the mixture of fuel employed, and to calculate thence the net cost of the ammonium sulphate. For this purpose it is sufficient to estimate the percentage of nitrogen in the peat and in the added fuel, and to calculate the cost according to the composition of the charges. This method of using mixed fuel in a producer may be of great service in countries where, as in the south of France, Germany, and Austria, there exist considerable quantities of lignites and anthracites, which can be utilised by mixing with peat.

When the peat is used alone in the gas producer, it ought to be distilled without previous stoving, so as to facilitate the condensation of the ammoniacal products. In this case a special still worked in connection with a gas producer is employed when it is desired to work up 7 to 10 tons of peat in twenty-four hours; for still larger operations, a producer with a blast may be used, which will give six to ten times as much gas as an ordinary producer. The peat-still shown in Fig. 24, which we take from Lencauchez' work, is constructed to yield large quantities of rich gases. It consists of an ordinary gas producer A, above which are situated three cast-iron retorts, *b*, *b*, *b*, into which the peat is charged through the hoppers B, B, B. In these retorts the peat first becomes dried by the gaseous current, and is then distilled. The carbonised residue is pushed down into the gas producer by tools introduced through the working doors *c*, *c*, *c*. The gaseous products, mingled with water vapour and ammonia, are collected in a brickwork flue D, from which they pass to a condenser or a scrubber, in which they give up their water and ammonia.

Worked up in this way, peat is capable of competing advantageously with coal.

The table below shows the percentage of nitrogen in peat from various localities; of this nitrogen it may be assumed that nine-tenths is obtained as ammonia on distillation :—

Air-dried peat containing 20 per cent. of moisture.	Nitrogen per cent.
Peat from Mennecy	2·4
,, ,, Vulcaire, near Abbeville	2·09
,, ,, Tévin (Finisterre)	1·7
,, ,, Saumur	0·65
,, ,, Montoire (Loire Inferieure)	0·55

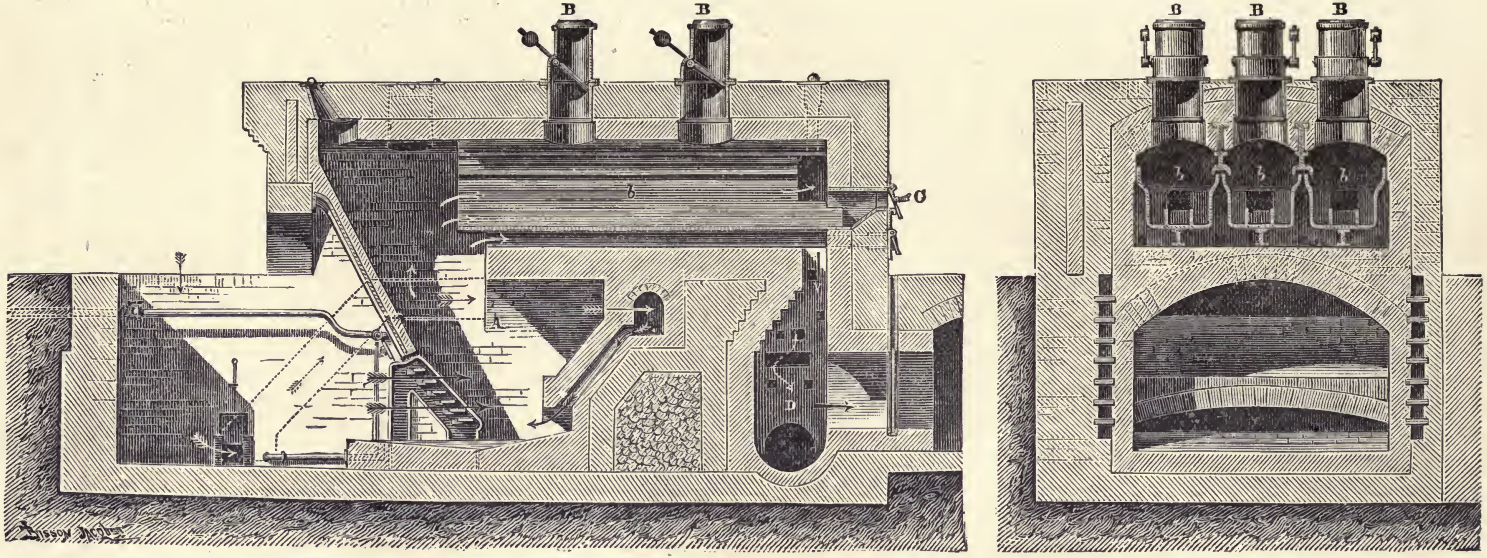

Fig. 24.—Peat Still.

A, Gas producer.
B, B, B, Hoppers for charging in the peat.
b, *b*, *b*, Cast-iron retorts in which the peat undergoes distillation before being pushed down into A.
C, C, C, Working doors.
D, Flue which conveys the products of distillation to a condenser, where they deposit the ammonia.

Assuming the selling price of sulphate of ammonia to be 40 frs. per 100 kilos. (£16, 5s. per ton), and 20 frs. as the cost of production, which may be taken as a maximum, it follows that the Mennecy peat, which yields 86 kilos. of sulphate, will leave a margin of 17 fr. 20 c. per ton of peat distilled, and of which the cost price varies from 17 to 18 frs. The sulphate produced therefore about pays for the peat, and the heating gas is obtained for nothing.

For the peat of Montoire, which is the poorest on the list, the margin on the sulphate would be 4 fr. 40 c., to be set against the cost of the peat. Taking 12 fr. per ton as the cost of this peat, the difference is 7 fr. 60 c.; and assuming that two tons of peat are required to give the same heating effect as a ton of coal, the cost as fuel would be 15 fr. 20 c., which is less than the cost of a ton of coal in most (French) industrial localities.

Treatment of the Ammoniacal Liquors.—The ammoniacal liquors from the distillation of peat are loaded with tar; they are collected in a brick cistern divided into two compartments, which are connected at the bottom. The mixture deposits most of its tar, which collects at the bottom of the first compartment, and flows into the second, whilst the clear ammoniacal liquor remains in the first tank, and is thence sent to the stills. The cisterns are covered to prevent loss of ammonia and avoid the production of a nuisance. By the side of these tanks there is another, in which the weaker liquors are collected which it is intended to return to the condensers to be enriched before proceeding to extract the ammonia. The apparatus for treating the clarified liquors is shown in Fig. 25; it consists essentially of four portions—(1) a dephlegmation column A, of cast-iron, for the extraction of the volatile salts; (2) a system of return condensers C, C′, for the enrichment of the ammoniacal vapours before absorbing them in sulphuric acid; (3) a horizontal cylinder B, with a mechanical stirrer, through which steam is passed, and into which the lime for decomposing the fixed salts is introduced; (4) an acid vat for absorbing the ammonia. The liquors to be treated are introduced at the bottom of the jacket of one of the condensers C; they become warmed, and pass in the same manner through the jacket of the second condenser C′, and are then delivered into the column A by the syphon *a*. They there give up their volatile salts and flow downwards in the column, whilst the vapour increases in richness as it rises, and after passing through the condensers C′ and C, reaches the acid vat. The liquor when it reaches the cylinder B, becomes mixed with enough lime to decompose the fixed salts completely. It gradually parts with its ammonia, and then flows away by the syphon S. The apparatus is heated by the waste steam from an engine, introduced at V, or by any other steam supply available.

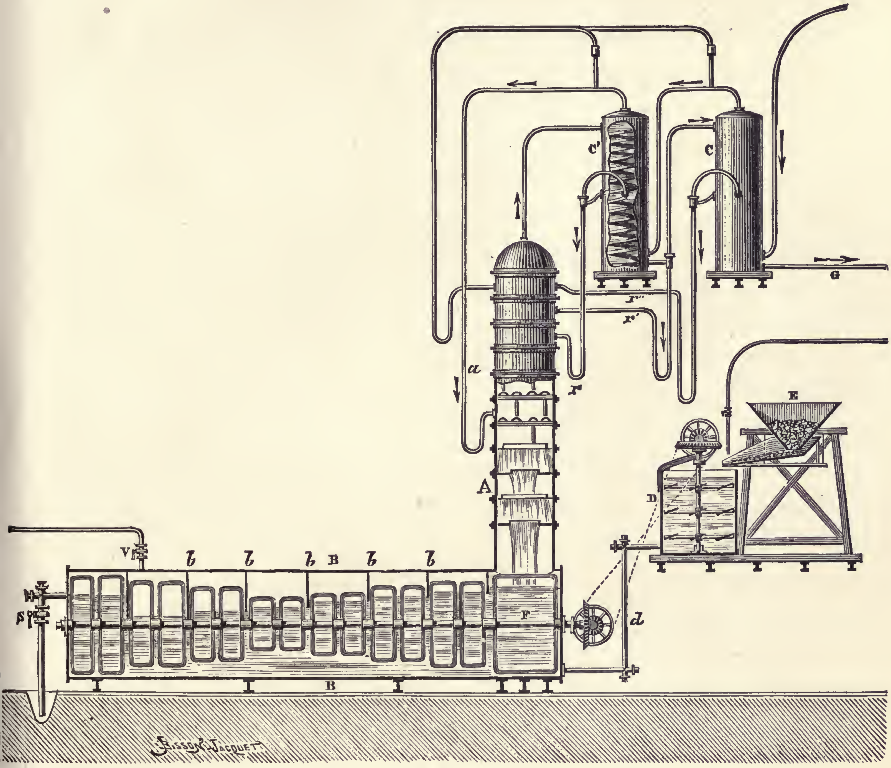

Fig. 25.—Apparatus for the Treatment of Ammoniacal Liquor.

A, Cast-iron distillation column for the extraction of the volatile ammoniacal compounds, and the enrichment of the vapour.
a, Syphon pipe by which the ammoniacal liquor enters the column.
B, Cylindrical boiler for decomposition of the fixed salts by lime, and the exhaustion of the liquors.
b, *b*, *b*, Wrought-iron partitions which compel the vapour to bubble through the liquid.
C, C′, Return condensers.
D, Lime mixer.
E, Lime hopper.
F, Mechanical stirrer.
G, Pipe conveying the ammoniacal vapour to the acid vat.
r, *r*′, *r*″, Return pipes for condensed water.
S, Escape pipe for exhausted liquor.
V, Steam supply.

The milk of lime is prepared in D, and is introduced into the boiler by the pipe *d*. The mechanical stirrer F, which runs through the whole length of the cylinder, keeps the liquor mixed and assists the removal of the ammonia. The cylinder B is provided with vertical partitions, *b*, *b*, which force the steam to pass through the liquid. The mixed vapours which issue from the column pass through the two condensers in succession, depositing the greater part of their water, which flows back to different parts of the column by the pipes *r*, *r'*, *r''*, whilst the ammonia passes onward to an acid vat. Care has to be taken that the temperature of the gas issuing from the last coil should not be too low, in order to avoid the deposition of ammonium carbonate, which would obstruct the pipes. A temperature of 25° to 39° C. is the most suitable.

CHAPTER V.

MANUFACTURE OF LIQUID AMMONIA, SAL AMMONIAC, PHOSPHATES OF AMMONIA, AND CARBONATE OF AMMONIA.

§ 1. Liquid Ammonia.

1. *Liquid Ammonia* is an aqueous solution of ammonia gas of greater or less strength and purity. It was formerly always manufactured by distilling sal ammoniac with lime; this process gave a very pure ammonia gas, which after washing was dissolved in water. The high price of sal ammoniac led to the substitution of the cheaper sulphate. The decomposition is effected in a wrought-iron boiler furnished with a mechanical stirrer, and heated by a fire. By using sulphate of ammonia obtained from urine, which is free from compound ammonias, a pure product is obtained, which is not the case with the sulphate from gas liquor. At the present time ammonia is only made from its salts in very special cases; it is usually obtained directly from gas liquor or fermented urine.

2. *Tarry Ammonia.*—This may be obtained directly from gas liquor by Mallet's apparatus, the gases being cooled completely before being dissolved in water. Such a product contains tarry matters and compound ammonias, which cause it to turn yellowish with age, especially if it is exposed to light. It is, however, possible to obtain colourless ammonia from gas liquor if the ammonia gas is more thoroughly washed before dissolving it in water.

3. *Colourless Liquid Ammonia.*—This may be manufactured by Mallet's apparatus (Fig. 15), slightly modified: between the heater A, and the analyser G, there is inserted a cast-iron distillation column F, of seven plates, and beyond the analyser there is a special purifier, which will be described further on. Such an

apparatus, with boilers 1·6 m. in diameter, and respectively 0·8, 1·1, and 1·35 m. high, is charged every three hours with 400 hectolitres of ammoniacal liquor. The method of work is in general that already described, with certain modifications. Thus, the boiler C is not in this case charged with gas liquor, but receives only the liquids condensed in the heater, column, and analyser. To these liquids, which are very rich in ammonia, there is added sufficient slaked lime to causticise the whole charge. The vapours given off from the boilers D and E bubble through this mixture, and thus become completely causticised. The crude gas liquor, heated in A, is delivered directly into D, where it becomes mixed with the lime from C. The vapours, after passing through the heater, rise through the column F, and come in contact with the very strong solutions condensed in G. This part of the apparatus, which is very thoroughly cooled by water, condenses almost the whole of the water vapour, and a large proportion of the foreign volatile substances. The passage of the vapours from the boiler D through the milk of lime in C causticises the volatile ammoniacal salts, which would otherwise contaminate the product. The ammonia gas issuing from the analyser is, however, not yet pure enough to yield a perfectly colourless liquid ammonia, but still contains small amounts of volatile ammoniacal salts, neutral substances, and compound ammonias. It is therefore passed through a further series of purifiers, shown in Fig. 26, before it is dissolved in water.

This apparatus consists of—(1) a cast-iron column A, 0·45 m. in diameter, containing fourteen chambers, and supplied with a strong solution of caustic soda by the funnel *a*; (2) a similar column B, fed with either a vegetable oil or a heavy paraffin; (3) a tubular saturator C, through the tubes of which is passed a current of the coldest water obtainable; (4) a safety vessel D, in which any gas which has escaped from the saturator may be condensed. The ammonia gas passing through the caustic soda, gives up nearly the whole of its impurities, and at the same time the last traces of the volatile ammonia salts are causticised, whilst the final passage through the oil removes the tarry matters and almost the whole of the volatile oily alkaloids. When the solution in C has reached the concentration desired, it is drawn out by the tap *h*, the water in D is run down, and any further water required is added by the funnel H, fresh water is run into D, and the operation is commenced anew. Liquid ammonia thus obtained is colourless, and does not darken on exposure to light; it nevertheless contains traces of impurities, which produce a rose or brown colour when it is mixed with nitric acid. Ammonia gas may also be purified by passing it through long columns filled with light wood charcoal to arrest empyreumatic products. The charcoal requires to be reburnt frequently to give satisfactory results.

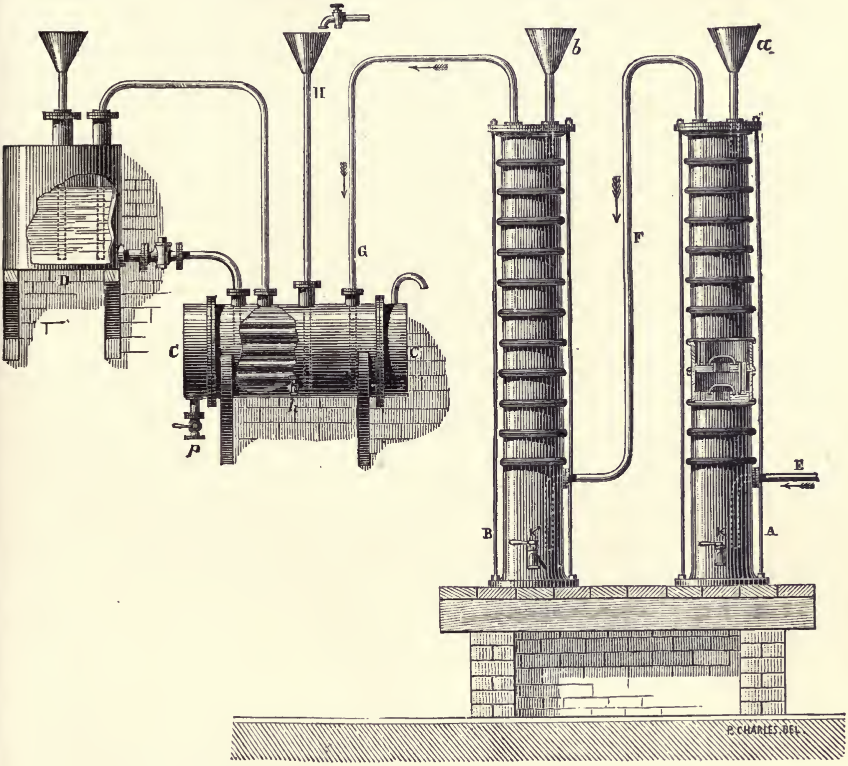

FIG. 26.—Mallet's Apparatus for Purifying and Dissolving Ammonia Gas.

A, Column of fourteen plates.
a, Funnel for adding soda.
B, Column of fourteen plates.
b, Funnel for adding oil.
C, C, Tubular saturator, cooled by water saturated at *p*.
D, Safety vessel.
E, Pipe by which the gas arrives at the column A.
F, Pipe conveying the gas to B.
G, Pipe conveying the gas to the saturator.
H, Safety funnel for running water into the saturator.
K, K′, Taps for emptying the columns A and B.

To obtain liquid ammonia which will give no coloration when mixed with nitric acid, it is necessary to start with sublimed sal ammoniac, or with sulphate made from urine, or the concentrated liquors or gases from the apparatus employed for working up *eaux vannes* may be employed directly. The Lesage Company manufactures a large quantity of very pure liquid ammonia from the crude solution of ammonium carbonate of 16° Bé., obtained from the apparatus of Margueritte and Sourdeval. Their apparatus consists of three parts—(1) a causticiser; (2) a purifying system; (3) a saturator. The causticiser is an upright wrought-iron cylinder A (Fig. 27), with a stirrer which can be worked by hand. At the lower part there is a system of closed steam coils, V, V′.

A wide pipe *t*, with a rubber sleeve closed by a pinch-clamp, allows the vessel to be emptied at the end of the operation. The causticiser is furnished with a pipe *a*, for introducing the solution of ammonium carbonate from the lead vat R, a pipe *b*, for the escape of the vapour, and an opening *o*, for adding lime. The purification system consists of—(1) an iron coil B, fixed in a tank through which a current of water flows. The steam is here condensed to water, which in the vessel C is heated by a steam coil V″, and returns by the pipe *c* to the causticiser; (2) a series of three leaden washing vessels, *l*, *l*′, *l*″, containing caustic soda lye of 26°, which can be run down from one vessel to the other.

The saturator is a wrought-iron cylinder, S, set in a wooden vat kept full of cold water, and containing the water to be saturated by the gas which enters by the tubes *e* and *f*. Any ammonia which escapes absorption passes on to the vessel *s*. The solution is drawn off by the pipes *g* and *h* into carboys. Several such absorbers are connected with the main pipe *e*, so that the work may be continuous.

An operation with this apparatus is conducted as follows:—Into the causticiser A there is introduced a quantity of milk of lime amply sufficient to decompose all the ammoniacal salts in a charge of about 250 litres of carbonate solution of 16° Bé., which is then gradually (during three hours) run into the lime. The decomposition proceeds rapidly, and after four hours, heating up may be commenced by sending steam first through one coil,

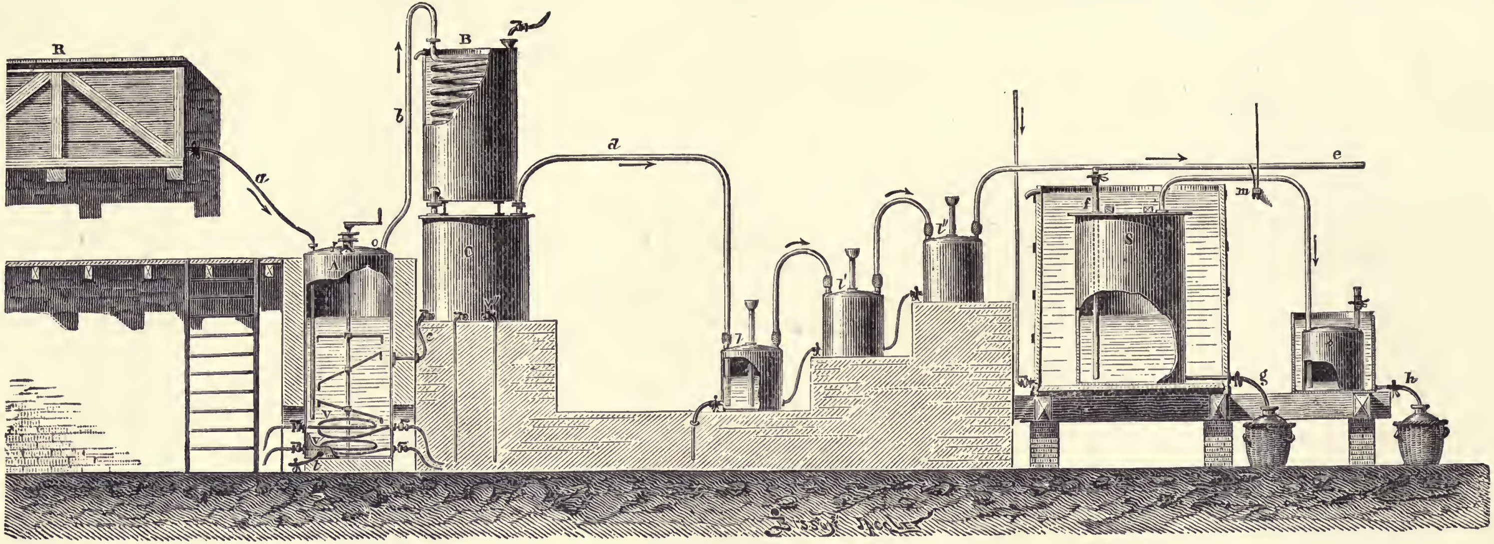

Fig. 27.—Apparatus of the Lesage Company for making Liquid Ammonia.

A,	Wrought-iron causticiser.	*g*, *h*,	Draw-off taps.
a,	Pipe for introducing ammonium carbonate.	*l*, *l′*, *l″*,	Caustic soda washing vessels.
B,	Cooler.	*m*,	Pressure gauge.
b,	Pipe for vapour.	*o*,	Opening for adding lime.
C,	Reservoir for the condensed liquor.	S,	Saturator.
c,	Return pipe.	*s*,	Safety vessel.
d,	Ammonia-gas pipe.	*t*,	Tap for drawing off causticiser.
e,	Pipe for conveying the gas to the saturators S.	R,	Reservoir of ammonium carbonate.
f,	Delivery pipe.	V, V′, V″,	Steam coils.

and then through both, to drive off the liberated ammonia. The vapours which escape contain ammonia, ammonium carbonate, and steam. The two latter are to a great extent removed by cooling in the coil B, and flow back to the causticiser, after a moderate heating in C, whilst the gaseous products issuing from C are still further purified from the last traces of carbonate in their passage through the three caustic soda washers, and finally reach the absorber S, where they are dissolved. When drawing off the solution, the pipe *f* is closed by a clip, and the ammonia gas sent to another similar absorber. When the charge in A is so far exhausted that no more gas passes through the soda vessels, the steam supply is stopped, and the contents of A are drawn off. They still contain some ammoniacal compounds, as it is not advisable to push the exhaustion of the liquors too far. They are therefore allowed to clarify, and are neutralised by hydrochloric acid, and evaporated, yielding sal ammoniac.

The liquid ammonia prepared by this process does not become coloured when exposed to light, or when mixed with nitric acid. It contains only the impurities of the water employed for absorbing the gas, and traces of ammonium carbonate. To obtain a perfectly pure product, it should be redistilled with a little slaked lime, and the gas condensed in distilled water. Pure liquid ammonia leaves no residue when evaporated on platinum foil, gives no precipitate with calcium salts, and, after acidifying with pure nitric acid, does not precipitate silver nitrate.

In our opinion the three caustic soda vessels of the apparatus here described might advantageously be replaced by a small cast-iron distillation column, through which the caustic soda lye would be allowed to flow.

Liquid ammonia is lower in density than water; it is sent out of several strengths according to the use for which it is intended. Most of it is sold at 22° Cartier (= specific gravity 0·918), a certain amount at 24° Cartier (0·904), and a small quantity at 28° (0·881).

Ammonia is employed in medicine as a rubefacient and revulsive, and to produce vesication. In small weak doses it is used as a corrective for alcoholic intoxication, and it is also employed in veterinary medicine as a corrective for flatulence.

In the arts it is used for cleansing woollen fabrics, for mixing with the scales of whiting for the manufacture of imitation pearls, for preparing ammoniacal cochineal liquor and archil. The liquid of density 0·881 is used in the Carré and Lindé freezing machines The greatest consumption, however, is in the manufacture of soda by the Solvay process, in which a solution of sodium chloride saturated with caustic ammonia is treated with carbonic acid gas.

Density at 14° of Aqueous Solution of Ammonia (Carius).

Ammonia per cent.	Density.	Ammonia per cent.	Density.	Ammonia per cent.	Density.
1	0·9959	13	0·9484	25	0·9106
2	0·9915	14	0·9449	26	0·9078
3	0·9873	15	0·9444	27	0·9052
4	0·9831	16	0·9380	28	0·9026
5	0·9790	17	0·9347	29	0·9001
6	0·9749	18	0·9314	30	0·8976
7	0·9709	19	0·9283	31	0·8953
8	0·9670	20	0·9251	32	0·8929
9	0·9631	21	0·9221	33	0·8907
10	0·9593	22	0·9191	34	0·8885
11	0·9556	23	0·9162	35	0·8864
12	0·9520	24	0·9133	36	0·8844

§ 2. AMMONIUM CHLORIDE (SAL AMMONIAC).

1. *Manufacture from fermented urine.*—A small amount of this salt is obtained by neutralising with hydrochloric acid the solution of ammonium carbonate obtained by Margueritte and Sourdeval's process for the treatment of *eau vanne.* This solution is worked up by the Lesage Company partly for the manufacture of ammonium chloride. The solution of ammonium carbonate is drawn out into carboys, which are then placed on a platform by the side of a large rectangular lead-lined vat, with a cover and draught pipe leading to a chimney. The solution is run down into the vat by syphons of glass or lead, whilst simultaneously, from another set of carboys, ordinary commercial hydrochloric acid of 18° to 20° Bé. is run down in such quantity as to keep the mixture neutral. Throughout the operation a mixture of carbonic acid and hydrogen sulphide gases is evolved, which is carried off by the chimney, whilst the temperature of the liquid rises, and unless care is taken ammonium carbonate would be lost by volatilisation. The mixture is left slightly acid at the end of the operation, and the solution is then

evaporated by passing steam through a coil of lead pipe lying at the bottom of the vat. During the evaporation the cover of the vat is removed, but temporary wooden flaps are laid on it in such positions that the steam is carried off by the chimney instead of escaping into the workshop. When sufficiently concentrated, the solution is run down into lead-lined crystallisers, where it cools slowly and deposits a perfectly white salt. In this operation, metal taps, which would be rapidly corroded by the solution, are replaced by rubber tubes closed by compression clips. To obtain the salt in small crystals, great attention must be paid to the degree of concentration of the solution, and the contents of the crystallisers must be stirred several times a day. When the crystallisation is completed, the mother liquor is returned to the neutralisation vat for a subsequent operation, and the salt is removed by wooden shovels, drained in baskets, and dried.

Kuentz has suggested the following method of utilising directly the ammonia gas from the distillation columns in which fermented urine is treated; native calcium phosphate is decomposed by hydrochloric acid so as to obtain acid calcium phosphate and calcium chloride, $Ca_3(PO_4)_2 + 4HCl = CaH_4(PO_4)_2 + 2CaCl_2$. This mixture is then treated in a closed vessel with the ammoniacal vapours from the still, which consist mainly of ammonium carbonate with a little ammonia, or it may be treated with the concentrated ammoniacal liquor. A precipitate is produced consisting of dicalcic phosphate and calcium carbonate, which is valuable as a fertiliser, whilst the whole of the chlorine is obtained as a solution of ammonium chloride, which is separated and crystallised. The uncondensable gases are passed through lime and then burnt in a coke fire.

As far as we are aware, this ingenious process has not been practically employed on the large scale.

2. *Manufacture from Gas Liquor.*—In England and Scotland much sal ammoniac is manufactured from tarry gas liquor, the liquor, freed as far as possible from tar by subsidence, being directly neutralised by hydrochloric acid and evaporated. The liquors are placed in large wooden vats furnished with mechanical stirrers, and ordinary commercial muriatic acid is gradually run in; the gaseous products, which in this case carry off some tarry matters, are passed through a fire in the usual manner. Much of the tar, which was held in solution by the ammonia, becomes insoluble when the alkali

is neutralised, and is separated from the liquid by prolonged subsidence. The clarified liquor, which is strongly coloured, is boiled down in cast-iron pans set directly over a fire. A further quantity of tar becomes insoluble as the concentration progresses, and is removed by skimmers. A small quantity of chalk or lime is added to the pans to neutralise free acid and prevent the boilers from being attacked. When concentrated to specific gravity 1·25, the solution is run down into crystallisers, in which it is stirred three or four times a day during the crystallisation to promote the formation of small crystals, which are more suitable than large ones for subliming. The crude ammonium chloride is obtained in granular crystals of a deep brown colour, due to the presence of tarry matter. It is partially purified by heating it on cast-iron plates, the volatile tarry substances and the water being driven off, whilst the remaining tarry matter becomes carbonised; at the same time the ammonium sulphate and thiosulphate, which the crude salt always contains, are decomposed. By careful working, and constantly stirring the mass, this operation can be conducted without appreciable loss of ammonium chloride. The greyish product is then sublimed, and yields commercial *sal ammoniac*, as a white mass, of fibrous structure and of a high degree of purity (see below, Chap. v. 5).

Kuentz has proposed to manufacture ammonium chloride in gas-works by treating the crude gas liquor directly with impure ferric chloride obtained from pyritic schist and common salt. A weak solution of ammonium chloride is thus obtained, together with a precipitate of ferric hydroxide mixed with a little sulphur, which may be used in the gas purifiers. The gas liquor is treated with the ferric chloride solution as long as any precipitate is produced, and the mixture is thrown upon a filter. The clear filtrate is evaporated by waste heat, and the precipitate, mixed with sawdust to render it porous, and gently dried, forms a very efficient purifying material. In this way, by the use of a very inexpensive material, the ammonia in the gas liquor can be completely converted into ammonium chloride without the evolution of any offensive gases. A process for the purification of coal gas has also been adopted in France by Mallet, by means of the residual liquor from the preparation of chlorine, which yields a precipitate of manganese carbonate and sulphide, with ferrous sulphide, and a solution of ammonium chloride. Many of the processes employed for working up gas liquor or fermented urine yield a product containing 14 to 18 per cent. of ammonia in the form of carbonate. Any of these liquors can be used for making ammonium chloride by the same process as that adopted in the case of the product from Margueritte's apparatus.

Kuentz has also suggested, as a means of avoiding the evolution of gas during the neutralisation of these solutions, to treat them in the cold with a solution of calcium chloride, and pass the mixture through a filter press. The filtrate, feebly acidified by hydrochloric acid, is then evaporated. This process would seem to be an economical one, if the almost valueless calcium chloride from the ammonia-soda process is employed.

The solutions of ammonium chloride are always evaporated in metal pans, either by direct fire, as in England, or by steam. If iron pans are employed, the metal is acted on, and a double chloride of iron and ammonium is produced, which volatilises when the salt is sublimed, and gives a coloured product. This inconvenience may be obviated by the addition of acid calcium phosphate, which forms non-volatile ferric phosphate, or by evaporating the solutions in leaden vessels.

The ammonium chloride employed for charging the Leclanché batteries used in telegraphy, must be free from lead. To prepare it for this purpose, it must be freed from heavy metals by treating the solution with ammonium sulphide, and filtering before it is left to crystallise.

3. *Ammonium Chloride from the distillation of Animal Matter.*—Laming has proposed to purify the distillate obtained in the manufacture of bone charcoal by means of fatty oils, and to treat the purified liquors with calcium chloride. A process has also been worked out by J. B. Divis, which yields a product pure enough to be used without subliming. The distillate is left at rest in wooden vessels for three or four days, and the tar which collects at the surface is skimmed off. The liquor is then gently heated and mixed with a strong solution of calcium chloride, in such quantity that the solution just ceases to effervesce with hydrochloride acid, excess being carefully avoided. The precipitate of calcium carbonate carries down the impurities, and after pressing can be used as a manure, whilst a solution of ammonium chloride of fair purity is obtained. The solution, which is slightly yellowish, is boiled up,

skimmed, and filtered hot through a mixture of coke and wood charcoal, supported by a layer of straw, in a wooden vat 1·25 to 1·5 m. high, and 0·45 to 0·6 m. in diameter. The filtered liquor is evaporated in a shallow pan, furnished with a cover and draught-pipe leading to a chimney to remove the offensive vapour. Fresh solution to run in as the evaporation proceeds, and when the surface becomes covered with a saline crust the evaporation is stopped, and the ammonium chloride which has salted out is removed to conical moulds resembling those used in sugar refining. The contents of these moulds are stirred to promote the formation of small crystals, and when crystallisation is complete the points of the moulds are pierced, the mother liquor allowed to drain away, and the crystals washed by percolation with a saturated solution of the salt, obtained by removing the upper portion of the crystalline mass to a depth of about an inch, and dissolving it in water. Two more washings are given with solutions of white salt; the contents of the moulds are then turned out, crushed with a wooden pestle, and allowed to dry. The mother liquor and the first washings are boiled down; later percolates are used for the first washings of fresh charges. The ammonium chloride obtained is of a high degree of purity, being free from calcium and iron, and having only a faint tarry odour.

4. *Manufacture from Ammonium Sulphate.*—From the year 1795 sal ammoniac and sodium sulphate were manufactured by Payen at Grenoble by decomposing common salt with a hot solution of ammonium sulphate of 21° Bé.; the sodium sulphate crystallises out, leaving ammonium chloride in solution. For the manufacture of sodium sulphate this process has long been displaced by more economical methods. The reaction has, however, been utilised in England more recently for the manufacture of ammonium chloride from gas liquor. The ammonia in the gas liquor is concentrated by distillation and neutralised by chamber sulphuric acid, and to the solution so obtained sodium chloride is added. On boiling down the mixture the less soluble sodium sulphate salts out, and is fished from the evaporating pans, drained, and washed, to free it from adhering ammonium chloride. When saline crusts begin to form on the surface of the liquid, the concentration is stopped; the liquid is left at rest to deposit the sodium sulphate, and the solution is run into lead-lined crystallisers. After cooling, the

mother liquor is run off, and the crystallised salt is dried and sublimed or purified by recrystallisation.

The following table shows the solubility of ammonium chloride in water at various temperatures:—

Temperature	Ammonium Chloride dissolved by 100 parts of Water.	Temperature.	Ammonium Chloride dissolved by 100 parts of Water.
5°	28·40	60°	55·04
10°	32·84	70°	59·48
20°	37·28	80°	63·92
30°	41·72	90°	68·36
40°	46·16	100°	72·80
50°	50·60	110°	77·24

5. *Sublimation.*—Ammonium chloride obtained by any of the above processes may be purified by sublimation. Originally obtained from Egypt and certain Asiatic localities, sal ammoniac was brought to Europe as early as the seventh century, in cakes with a fibrous fracture, more or less stained with empyreumatic substances. At the present day two modes of sublimation are in use. The French method yields small white or grey cakes, which resemble in appearance those brought formerly from Egypt, and for which there is a constant demand, whilst the English method produces larger and thicker crusts.

The dried salt, when heated, volatilises, and the vapour condenses on the cooler parts of the vessel, forming a white translucent layer. In this operation it is essential that the temperature should be constantly maintained at the point at which the sublimation proceeds slowly: at too high a temperature a considerable loss would be incurred, the cake would not possess the correct texture, and would be contaminated by empyreumatic products, resulting from tarry matters which were not destroyed by the roasting.

In England the sublimation of ammonium chloride obtained from gas liquor is carried out in large cast-iron subliming pans lined with fire-brick, and furnished with cast-iron covers, which are fixed down by keys, so as to make a tight joint capable of resisting the internal pressure developed during the sublimation. It is on the under side of these covers that the sal ammoniac is deposited, in a layer 4 to 6 in. thick. The pans used are from 3 to 9 ft. in diameter, and take charges of 1 to 9 tons each. The covers can be raised by a chain pulley affixed to the roof.

When a pan has been charged and covered, it is at first heated rapidly to the temperature of sublimation, after which the heat is regulated so that the operation proceeds slowly and uniformly. A small hole in the cover allows the escape of the air, with small quantities of gas and steam; it is closed by an iron plug as soon as fumes of sal ammoniac appear. To prevent the covers from cooling too rapidly, a layer of sand or ashes is placed upon them. The operation is not continued long enough to sublime the entire charge, as impurities accumulate in the last portion, and would, if volatilised, injure the colour of the product. When a sufficient quantity of this impure residue has accumulated from successive charges, it is removed and washed, and the solutions are added to those in the evaporating pans. A sublimation lasts about a week: when the pan has cooled sufficiently, the cover is raised, the crust is detached, and any portions coloured by contact with the iron are removed, and returned to subsequent operations.

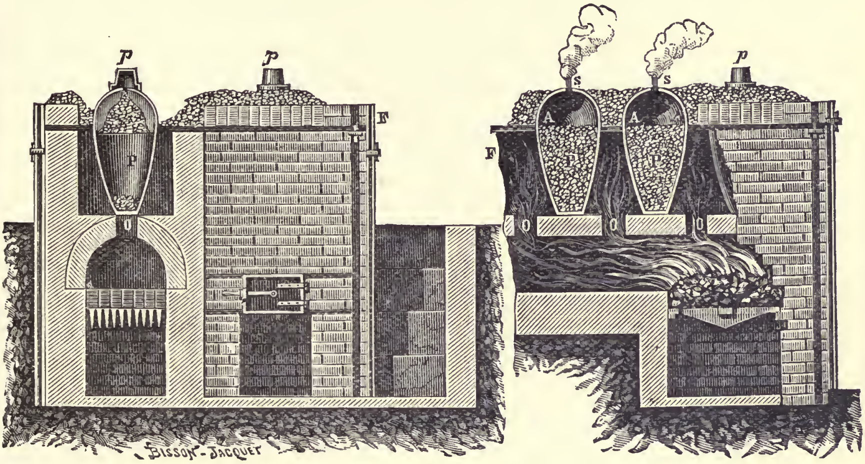

Fig. 28.—Furnace for the sublimation of sal ammoniac in pots.

In France the sublimation is always performed in earthenware pots 0·5 m. high and 0·3 to 0·35 m. in diameter. Fig. 28 shows the arrangement adopted. The pots, twenty in number, are arranged in two rows on the arch of a furnace, where they are exposed to the flames rising through the apertures O, O. The top of the furnace is formed by a cast-iron plate F, F, through holes in which the pots are introduced, and which confines the heat to the lower two-thirds of their height. On this plate lies a layer of sand, which can be heaped over the pots when required. The pots, first coated with a clay lute, are dried in the subliming house; they are then charged with crystallised ammonium chloride as dry as possible, set in the furnace and covered with sand. The heat is then raised slowly, to avoid cracking the pots. Steam is at first given off from the openings S, S, and when thick white vapours of sal ammoniac appear, an ordinary flower-pot *p* is inverted over each opening to condense them.

The operation is carried on as in the English process: the sal ammoniac is deposited in the upper parts of the pots at A, A, and in the flower-pots *p*, *p*; and gradually closes up the openings, so that towards the end of the operation, when the temperature has to be somewhat raised to give the product the requisite texture and translucency, an internal pressure is caused in the pots. To obtain cakes of sufficient weight, it is necessary to recharge the pots several times. The hole is therefore cleared out with a tool like a centrebit, and the whole operation is repeated as at first. After two or three charges the cakes will have reached the greatest attainable thickness: the pots are allowed to cool and are broken to get out the sublimed cake, which, if the original salt was free from iron and tarry matter, will be found to be perfectly white and of higher purity than that made in iron subliming pans. If it is desired to obtain a grey sublimate, which some consumers prefer to the white one, a little fat is thrown into the pot from time to time during the sublimation. In some works, glass vessels, which are cheaper than the earthenware pots, have been employed. The sublimation of sal ammoniac, especially by the French process, is a costly operation, whence the price of the sublimed salt is considerably higher than that of the crystallised salt. An attempt was made some years ago by the Lesage Company to replace the sublimed salt by a cheaper product, in the form of cakes 1 in. thick and 4 in. in diameter, obtained by strongly compressing the crystallised salt; but it did not meet with acceptance, and the manufacture was not continued.

§ 3. Ammonium Phosphates.

Phosphoric acid H_3PO_4, forms three compounds with ammonia, the normal or neutral phosphate $(NH_4)_3PO_4$, the diammonic, or intermediate phosphate $(NH_4)_2HPO_4$, and the monammonic, or acid phosphate $(NH_4)H_2PO_4$. The first of these salts is not manufactured commercially; the two others, especially the intermediate phosphate, are now commercial products. The acid phosphate has been manufactured for many years, but it was not until 1871 that Lagrange, having utilised this salt in sugar refining, found its preparation on the large scale necessary. The manufacture was taken up by Storck at Asnières, and the article is now sent into the market in a perfectly pure state. The acid ammonium phosphate is prepared by treating acid calcium phosphate with ammonia and evaporating and crystallising the filtered solution; the intermediate phosphate, by adding ammonia to a concentrated solution of the preceding.

We take the following details of the manufacturing processes from the account published by Lagrange:—

Acid calcium phosphate is prepared by treating finely powdered apatite or phosphorite with an equal weight of concentrated sulphuric acid in a cast-iron mixer with a mechanical stirrer. The mixture heats considerably and gives off gases rich in hydrofluoric acid, which are carried off by a chimney. After six hours, the pasty mass, consisting of calcium sulphate, acid calcium phosphate, phosphoric acid, and the excess of sulphuric acid, is submitted to systematic extraction with water. For this purpose it is divided between four vats of equal capacity, each of which is furnished with a steam pipe. The contents of the first vat are thinned with water, or the weak washings from a previous operation, and are boiled up by steam. The solution is separated from the insoluble matter by filtration through wicker baskets lined with filter cloth, and is passed in succession through the three other vats, boiling up in each of them. The residue on the filters is washed with boiling water and pressed. It consists almost entirely of calcium sulphate. The solution from the fourth vat, which marks 25° Bé., and contains free phosphoric acid, acid calcium phosphate, calcium sulphate, and a little sulphuric acid, is treated with a small excess of barium carbonate, which removes the sulphuric acid completely. The clarified solution, now containing nothing but acid calcium phosphate, free phosphoric acid, and a little acid barium phosphate, is mixed with a small excess of ammonia, when all the calcium is precipitated as tribasic phosphate, which after washing is returned to the apatite mixer. The filtered liquid is a solution of monammonic phosphate, which has now to be converted into the diammonic salt. If the operation has been carried out as described, this liquor will have a density of 20° Bé., and this strength is necessary for the precipitation of the diammonic salt, which is effected by running the solution into small crystallisers, simultaneously with liquid ammonia of 22° (specific gravity, 0·918), employing $1\frac{1}{2}$ equivalents of ammonia for 1 equivalent of ammonic phosphate. The diammonic phosphate crystallises out as the solutions mix. The crystallisers must be covered to avoid loss of ammonia from the elevation of temperature which ensues. After cooling, the crystals are drained and pressed, whilst the mother liquor is distilled to recover the ammonia, which is used over again. The pressed crystals consist of pure diammonic phosphate, which is employed in Lagrange's sugar-refining process.

§ 4. Ammonium Carbonates.

Three ammonium carbonates are believed to exist, the normal and acid salts, and the sesquicarbonate formed by the combination of the two former, but of these the normal salt has never been isolated, in consequence of the readiness with which it parts with its ammonia. It is therefore only known in solution, and the prismatic crystals which separate from an alcoholic solution of the sesquicarbonate, saturated with ammonia, are the simple sesquicarbonate. The bicarbonate or acid carbonate is obtained by saturating an aqueous solution of the sesquicarbonate with carbonic acid. When commercial ammonium sesquicarbonate is treated with small quantities of water or alcohol, neutral carbonate is dissolved out, and a residue of bicarbonate, which is insoluble in alcohol, is left. On exposure to air the sesquicarbonate loses ammonia and is converted into the acid carbonate; the latter slowly volatilises, giving off an ammoniacal odour. It crystallises from water in large orthorhombic prisms. Ammonium bicarbonate has been met with in crystalline masses, of yellowish white colour and density 1·45, in the guano deposits of Peru, Chili, and the western coast of Patagonia.

Ammonium Sesquicarbonate: preparation from Animal Matter.—Pure ammonium sesquicarbonate is obtained by dissolving the commercial carbonate in caustic ammonia and allowing the solution to crystallise: it separates in large orthorhombic prisms, which on exposure to air become opaque, and are converted into the bicarbonate. The commercial article (*sal volatile*) consists chiefly of the sesquicarbonate.

When animal matters, such as bone, horn, leather clippings, or muscular tissue, are submitted to dry distillation, the organic matter is decomposed and yields ammonium carbonate mixed with a little sulphide, water, and complex oily products. The quantity of ammonium carbonate thus obtained varies with the nature of the raw material, with the mode of distillation, the temperature employed, and the perfection of the condensation. Ammonium carbonate, which is the principal product, is partly deposited in the solid form in the coolers, if dry materials have been employed and water has not been added to assist the condensation. The product thus obtained, in crusts, stained yellow by the presence of tarry matter, was formerly known as *salt of hartshorn*, whilst the aqueous solution obtained at the same time was called *spirit of hartshorn.* The purification of these products is long and difficult. Sublimation is the only method applicable; it yields a product containing traces of empyreumatic substances, which is known in pharmacy as *ammoniacum carbonicum pyro-oleum.* This product may be completely freed from its impurities by mixing it with animal charcoal and resubliming. It is then white, translucent, and fibrous, has an ammoniacal odour and caustic taste.

These operations being costly, it is preferable to convert the impure carbonate, obtained as we have described in Chap. III., into sulphate, and from this obtain the pure carbonate at once.

Either ammonium chloride or sulphate, mixed with twice its weight of chalk, may be used. The mixture is heated in a cast-iron retort, connected with a leaden receiver cooled by water. The mixture is gradually heated to dull redness; ammonia, water vapour, and ammonium sesquicarbonate are evolved, and the last condenses as a white mass in the receiver. The addition of a little wood charcoal to the retort charge improves the colour of the product.

English Process.—In England, ammonium carbonate is manufactured from the crude chloride obtained by neutralising gas liquor with hydrochloric acid. The crystallised salt thus obtained is distilled with twice its weight of chalk in horizontal cast-iron retorts

2 m. long and 0·5 m. wide, similar to those in use for making coal gas. These retorts, of which three to five are set in the same furnace, are connected by large cast-iron pipes with two small leaden chambers, 2 m. high, $2\frac{1}{2}$ m. long, and 0·75 m. wide, arranged in series, and employed as condensers for the ammonium carbonate. Each of these chambers has one of its sides movable, for the removal of the product, and there is a small pipe which allows the escape of a little water vapour and a solution of ammonium carbonate, which is collected and returned to the process. The materials employed must be thoroughly dry, and during the whole of the distillation the mixture must be stirred by a rod passing through an opening in the door of the retort. As ammonium carbonate begins to sublime at about 50° C, the firing must be done with care and regularity. As soon as a charge is exhausted it is drawn from the retort and a fresh one introduced, and the sublimation proceeds for fourteen or fifteen days before the receivers are opened to remove the product. The condensed carbonate forms crusts, somewhat coloured by tarry matter, and rendered impure by small quantities of lime salts carried over by the current of gas. It is purified by a resublimation, conducted very slowly in cast-iron pots, 0·33 m. in diameter and 0·75 m. high, covered with leaden domes, and heated by the flue gases of the retort furnaces. These pots are sometimes replaced by large pans, 4 or 5 m. long, 0·75 m. wide, and 0·75 m. deep, covered with cast-iron plates luted on air-tight, and pierced with eight circular holes 0·33 m. in diameter, on which the leaden domes are adjusted. The crude salt from the chambers is placed in the tank with a certain quantity of water, and the leaden domes having been put in their places, the boiler is gently heated, the heat being controlled by observing a thermometer inserted into the dome the most distant from the fire. The operation lasts fourteen days, and the ammonium carbonate is then found in the domes in the form of white, translucent, fibrous crusts, which only require the removal of the surface which has been in contact with the metal. The liquid remaining in the pan receives a new charge of the crude salt, and a fresh sublimation is commenced.

Uses.—Pure ammonium carbonate is used in medicine, and as a baking powder in making pastry to give lightness to the paste. It may be used for the preparation of any other salt of ammonium, and in common with caustic ammonia it may be employed for the removal of grease from fabrics, and for developing the dyes obtained from lichens. Its chief use is, however, for the decomposition of sodium chloride in the manufacture of sodium carbonate.

CHAPTER VI.

RECOVERY OF AMMONIA FROM THE RESIDUAL LIQUORS OF THE MANUFACTURE OF SODA BY THE AMMONIA PROCESS.

§ 1. General Considerations.

The manufacture of soda by the ammonia process is based on the reaction which occurs between solutions of sodium chloride and ammonium bicarbonate. As shown by the equation $NaCl + NH_4HCO_3 = NaHCO_3 + NH_4Cl$, there is obtained a precipitate of sodium bicarbonate and a solution of ammonium chloride. The actual composition of the mother liquor from the precipitated bicarbonate is, however, much more complex. It actually contains ammonium chloride, ammonium carbonate, sodium bicarbonate, and the excess of sodium chloride added intentionally. It would be uneconomical to treat these mother liquors at once with lime for the recovery of the ammonia they contain, since lime would be wasted by combining with the carbonic acid of the ammonium carbonate and sodium bicarbonate, and the carbonic acid of these salts would also be lost.

If, however, these mother liquors are distilled, the ammonium carbonate and the carbonic acid of the sodium bicarbonate are both recovered; for, on heating, an inverse reaction between ammonium chloride and sodium bicarbonate takes place, with production of sodium chloride and ammonium bicarbonate: $NH_4Cl + NaHCO_3 = NaCl + NH_4HCO_3$. Since the mother liquors contain 6 per cent. of sodium bicarbonate, this recovery of carbonic acid is of considerable importance. After distillation, the mother liquor contains only chlorides of ammonium and sodium, and can now be treated with lime for the recovery of the ammonia. Both the free and carbonated ammonia are passed into condensers containing solution of sodium chloride. There is thus obtained an ammoniacal brine which is used for a fresh preparation of sodium bicarbonate. The residuary liquors, which contain only calcium and sodium chlorides, lime, and impurities, are run to waste.

§ 2. Apparatus of Schlœsing and Rolland.

This apparatus, which fulfils the conditions which we have pointed out for the treatment of the residual ammoniacal liquors, is shown in Fig. 29; it consists of five distinct parts—

1. A vessel called the *little still No.* 1, in which the liquor is deprived of its carbonic acid.
2. A similar vessel, called the *little still No.* 2, for the elimination of carbonic acid and ammonia.
3. The *lime mixer*, in which the liquors from 1 and 2 are mixed with lime.
4. The *large still*, in which the liquors are completely freed from ammonia.
5. The *condenser*, not shown in the figure, consisting of two concentric cylinders strongly cooled with water, in which the water vapour carried off by the ammonia gas from the large still is condensed and returned to the column by the pipe *t*.

Each part of this apparatus is heated independently by steam introduced at V_1, V_2, V_3. The gases which escape by the openings S_2, S_3, are passed separately into a tank containing sodium chloride solution, where they are condensed; those from S_1, which consist mainly of carbonic acid, are collected in a counterpoised gasholder.

The mother liquor is run into the apparatus in a constant stream by the syphon tube T_1. As it passes downward from one shelf to another, and meets the current of steam travelling in the opposite direction, it is heated to such a temperature (50° to 60° in the upper compartment) that it parts with carbonic acid containing only a small quantity of ammonia. It then passes by the syphon tube T_2 into the second still, where it meets with sufficient steam to maintain a temperature of ebullition in all the compartments.

When it reaches the bottom of the second still, the liquor is practically free from ammonium carbonate; it then flows by the syphon tube T_3 into the lime mixer. This is a wrought-iron horizontal cylinder containing a mechanical stirrer which revolves slowly. Slaked lime, introduced through K in a thick paste, is propelled into the vessel by the screw *h* revolving in a horizontal

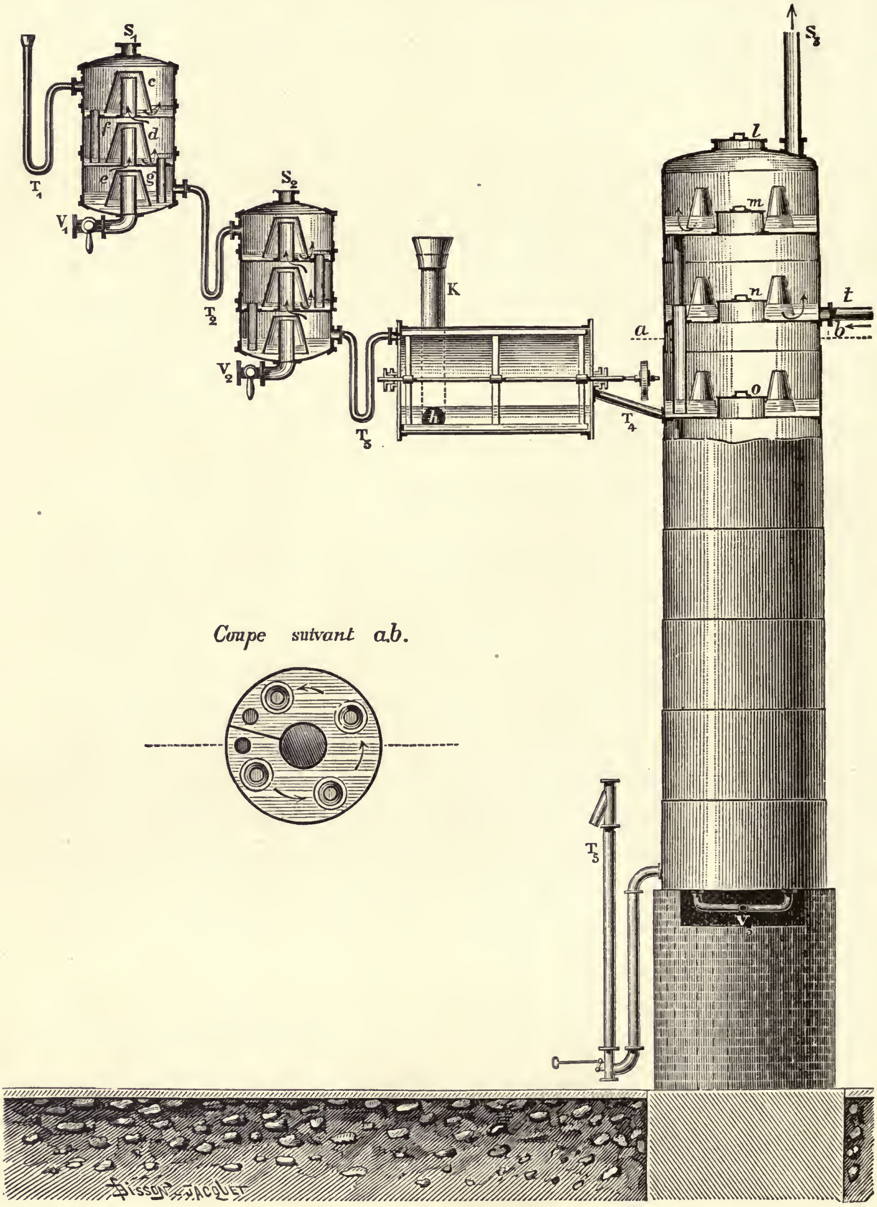

Fig. 29.—Apparatus of Schlœsing and Rolland.

cylinder at such speed as always to keep an excess of lime in the mixture. Samples are removed at intervals to insure the fulfilment of this condition. The paste of lime in the hopper K forms a sufficient obstacle to any loss of ammonia by that channel. The liquor, mixed with lime, passes then by the pipe T_4 into the large still, which is a wrought-iron column of eight compartments, constructed with a special view to its being readily cleaned out. The lime, which gradually settles on the plates would eventually obstruct the column altogether; and it is found necessary to clean it out at least once a week, for which purpose a workman enters through the manhole *l*, and through similar openings at *m*, *n*, *o*, etc., is able to pass through all the compartments. The liquor, when it has passed through the column, escapes by the pipe T_5, completely deprived of ammonia.

The apparatus just described would appear to fulfil the conditions required by the problem, in so far as it separates the operations of the simple distillation and the treatment with lime, whilst being continuous in its work; but it is nevertheless open to many objections. The adjustment of the three supplies of steam, V_1, V_2, V_3, is one difficulty: the separation of the functions of the two small stills Nos. 1 and 2 is by no means indispensable; it would be simpler to distil over the carbonic acid and the ammonia existing as carbonate at one operation. Further, the introduction of a paste of slaked lime into a boiling solution rich in ammonium chloride, produces a violent and irregular evolution of ammonia gas, which disturbs the steady progress of the operation. This difficulty has been recognised by Schlœsing and Rolland, who suggest the partial cooling of the lime cylinder by cold water. Grave inconvenience is also caused by the column becoming choked with the lime mud. Finally, the wrought-iron of which stills 1 and 2 are constructed, is rapidly corroded by the boiling solution of ammonium chloride: cast-iron would probably last longer.

§ 3. APPARATUS OF THE SOCIÉTÉ ANONYME DES PRODUITS CHIMIQUES DE L'EST.

This company, which has an alkali works in the suburbs of Nancy, has recently patented an apparatus which solves the same problem in a simpler manner, and avoids the inconveniences which

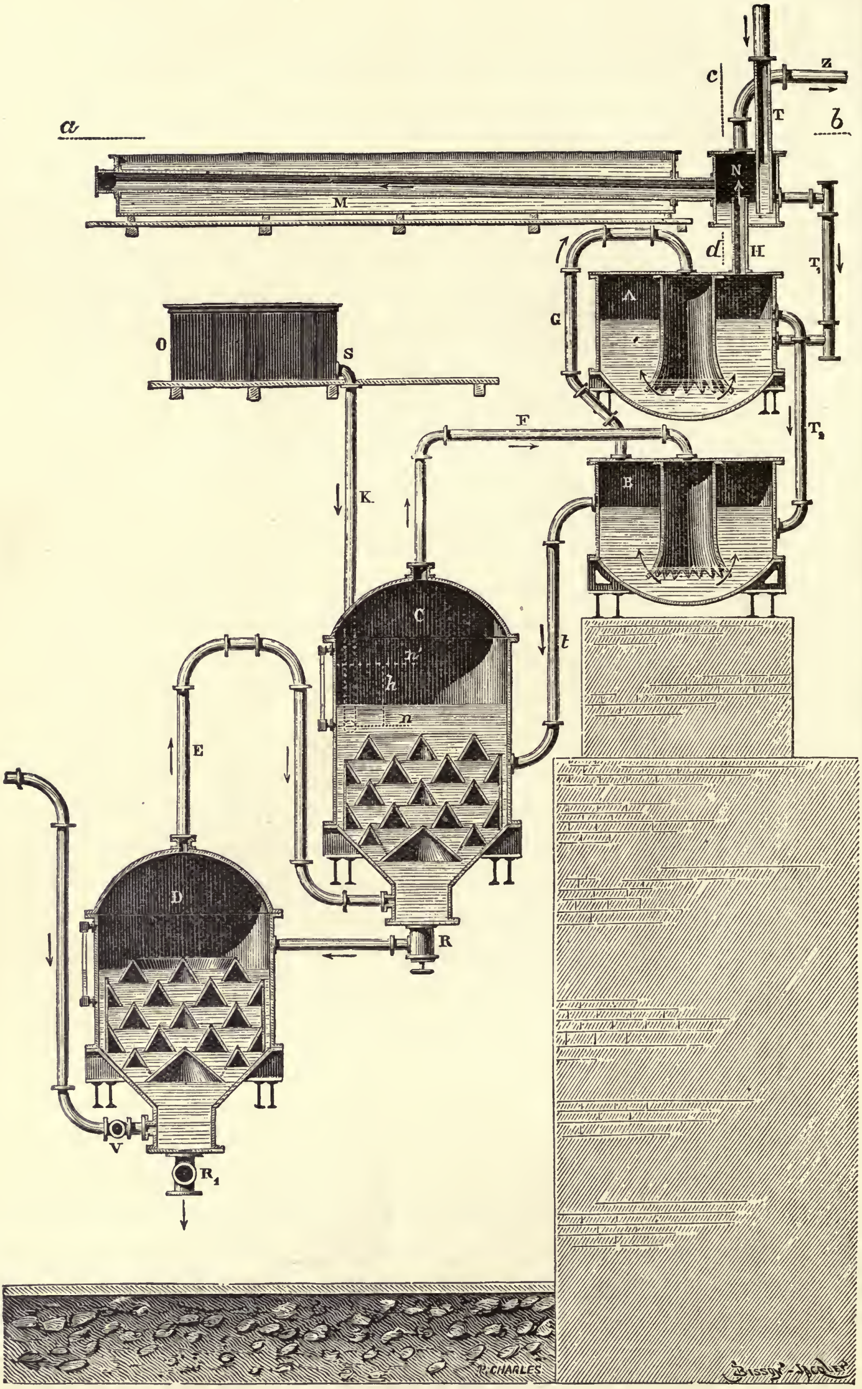

Fig. 30.—Apparatus of the Société Anonyme des Produits Chimiques.

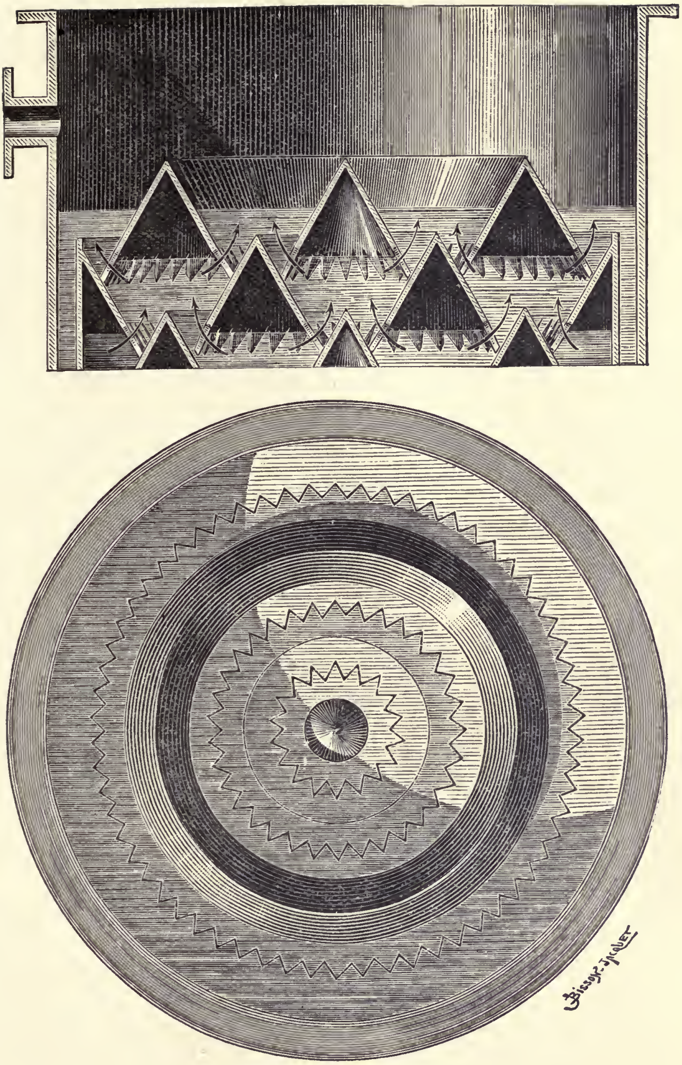

FIG. 31.—Cones piled up in the Boilers C and D.

Description of Figs. 30 and 31.

A, B, Cast-iron stills for driving off carbonic acid and ammonia.

C, D, Lime boilers, in which the liquors are treated after passing through A and B.

E, Pipe which conveys the steam from D to C.

F, Pipe which conveys the steam from C to B.

G, Pipe which conveys the steam from B to A.

H, Pipe which conveys the steam from A to the first compartment of the collector N.

K, Pipe for running down milk of lime from the vat O.

M, Separator, in which the water carried off by the ammonia gas condenses, and from which it flows into the collector N.

N, Collector, partitioned into compartments, from which the vapours pass successively into the tubes m, m, m of the separator, and from which the condensed water flows through T_1 into the boiler A.

n, n', Limits within which the level of the liquor in C is kept.

R, R_1, Stop-cocks by which the contents of C are run into D, and those of D are run away.

S, Lime vat tap.

T, Pipe for entrance of the mother liquor to be treated.

T_1, T_2, Pipes through which the liquors flow successively into A and B.

t, Pipe conveying liquor from B to C.

V, Steam-cock.

Z, Pipe conveying the ammonia and ammonium carbonate to the brine tanks.

we have pointed out. This apparatus, shown in Fig. 30, consists essentially of three parts—

1. The cast-iron stills A and B, the number of which may be increased to three, in which the liquors are deprived of their carbonic acid and ammonium carbonate. These boilers may be replaced by a column, also of cast-iron. 2. The lime stills C and D. 3. The separator M, with its collector N. The boilers C and D contain a number of hollow cast-iron cones, piled one on the other, the lower edges of which are deeply serrated, as shown in Fig. 31. The steam, which enters these vessels by V and E, displaces the liquid from the cones, and, escaping through the notches, bubbles up through the liquid, and is subjected to a very exhaustive scrubbing. This ingenious arrangement altogether prevents any choking by the lime or other insoluble matter, since these substances are very perfectly kept in suspension by the action of the steam, and when the contents of the stills are drawn off, the insoluble deposit readily falls from the surfaces of the cones, which have a slope of 60°. Actual incrustations, due to the calcium sulphate, which is always present in brine, only require to be removed at intervals of two or three months, for which purpose the dome-shaped tops of the stills are lifted off, the loose cones taken out, hammered and replaced; the whole operation being extremely simple and rapid.

The mother liquors from the soda carbonating vessels enter in a continuous stream by the pipe T, fed by a constant-level reservoir. From the chamber N they pass to the boiler A by the pipe T_1, and to B by T_2. When the partially exhausted liquor in C stands at the level n, a thick cream of lime is introduced by the pipe K, the quantity of lime introduced being more than sufficient to decompose all the ammonium chloride in a quantity of the mother liquor, which would occupy the space from n to n'. The lime can be added to a nearly exhausted solution without producing any violent or irregular evolution of ammonia gas, and as the boiler fills up from n to n', by the continuous arrival of fresh solution by the pipe t, the decomposition by the excess of lime takes place gradually. When the mixture arrives at the level n', it is drawn off to the level n into the boiler D, which has just previously been emptied of its exhausted contents. Steam is introduced into the apparatus at one place only by the stop-cock

V. It first expels the last traces of ammonia from the contents of the boiler D; the mixture of ammonia and steam then passes in succession through C, B, A, and N. At this last point the vapour consists almost entirely of ammonia and carbonic acid, with very little water; the latter is condensed whilst passing through the cooled pipes *m*, *m* of the separator M (Fig. 32), and flows back into the collector N. This vessel is provided with vertical partitions, which dip to a certain depth only into the liquid:

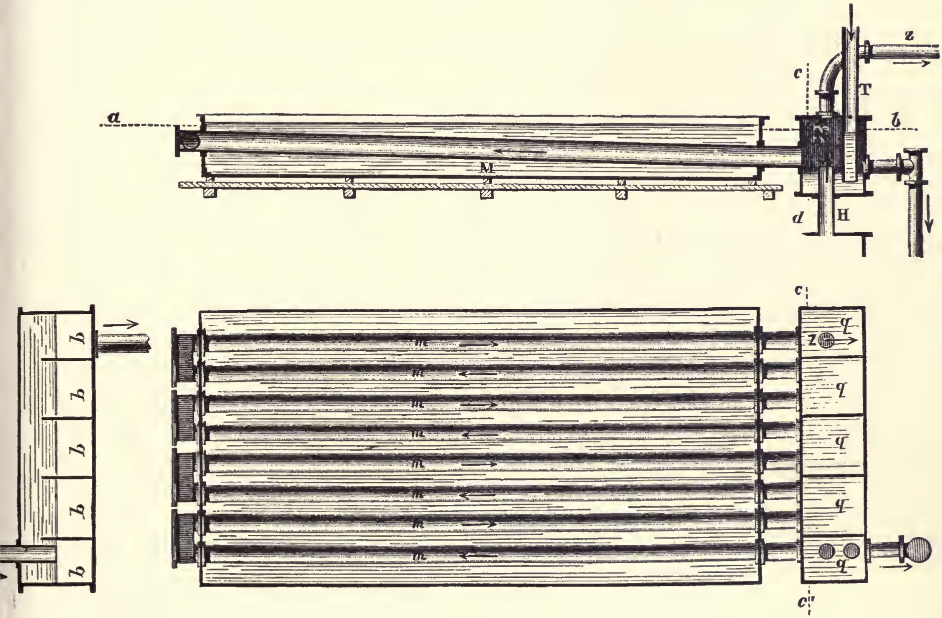

Fig. 32.—The Separator and Collector in Plan and Section.

H, Pipe through which the vapours enter the first compartment of the collector N.

m, *m*, Condensing tubes.

N, Collector which directs the gaseous current into the tubes of the separator, and receives the condensed liquid from them.

q, *q*, Compartments formed by vertical partitions dipping into the condensed liquid.

T, Pipe for entrance of the liquor to be treated.

Z, Exit for ammonia and ammonium carbonate.

they serve to direct the gaseous current into the pipes, whilst allowing the liquid to flow towards the end, where the pipe T_1 removes it. The ammonia and carbonic acid which issue from the last compartment of the collector are conveyed by the pipe Z to a brine reservoir.

Such an apparatus as the one we have described, with the boilers C and D 2 m. in diameter, and the former with a cylindrical portion 2 m. high, can exhaust in twenty-four hours 40 c.m. of mother liquor containing 9 per cent. of ammonia.

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