LABORATORY APPARATUS FOR RAPID EVAPORATION

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When it is considered that evaporation is a daily proceeding in general laboratory practice, the importance of reducing the time factor in this operation will be at once appreciated. Nevertheless, there seem to be comparatively few analytical chemical laboratories where provision is made for a rapid and effective system of evaporation. The authors have found the following extremely simple apparatus very useful and efficient for this purpose:

The air from the blast is first passed through a screw-capped brass cylinder, A, 3 cm. $\times$ 15 cm., packed with cotton, which filters out any scale or particles which might contaminate the residue during the operation; then, before passing to the blowers, it is conducted

FIG. 1

through a 2-meter coil of 0.6 cm. copper tubing which rests on the steam pipes in an ordinary steam bath and has its terminal, B, sufficiently elevated above the top level of the bath to enable rubber connections to be made. The individual blowers, which are supported over the respective holes in the steam bath, are provided with glass stopcocks, so that as many as are desired can be used simultaneously. The heavy bent wires C that support the blowers slide within hollow standards by means of which the height of the blowers above the bath can be adjusted as desired. Fig. 1 shows the arrangement of the apparatus.

When the steam is turned on, the air passing out at the orifices of the blowers is heated to approximately 60° C. but can be varied according to the volume of air passing. No doubt a higher temperature could be obtained by using a longer coil. The following tabulation shows how much evaporation may be expedited by the use of this apparatus when other conditions are constant:

TABLE I—RATE OF EVAPORATION OF SEVERAL SOLVENTS ON STEAM BATH, WITH AND WITHOUT BLAST¹

SOLVENT	Volume Cc.	Surface Sq. cm.	TIME IN MINUTES—		
			No Blast	Ordinary Blast	Hot Air Blast
Ether.....	50	20	7	5	3
Chloroform.....	50	20	23	11	7
Benzene.....	50	20	55	14	10
Alcohol, 95 per cent....	50	20	100	24	16
Water.....	50	20	175	100	64

¹ All determinations made in 100 cc. pyrex beakers.

The apparatus has been found especially useful for the rapid top-evaporation of solutions which otherwise are prone to decrepitate, such, for example, as strychnine in chloroform, and also for the rapid drying of wool fibers used in qualitative color analysis. Another advantage of the apparatus is that by insulating the beaker from the bath a rapid evaporation at a low temperature can be made of solution of materials which are apt to volatilize, polymerize, or decompose at higher temperatures. It is useful, for example, in the evaporation of aqueous solutions of glycerin, petroleum-ether solutions of volatile alkalis such as conine, ethereal solutions of volatile oils, chloroform solutions of salicylic acid, etc.

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A FUSION BOMB FOR SULFUR DETERMINATION IN COAL

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The determination of sulfur in coal by use of sodium peroxide as an oxidizing medium has met with favor wherever it has been tried. The results have been shown to be in close agreement with those obtained by the Eschka method, hence a reference only to tables already published is sufficient.¹

In this process the need of a suitable device for carrying out the combustion has been evident for some time. It is the purpose of this note to call attention to a simple piece of apparatus which has been found to operate satisfactorily in this connection.

A fusion cup, 2AC of the figure, has a cover, 7AC, which is held in place by a screw cap, 5AC. The fusion cup is removable and by having duplicate cups a number of samples may be made ready at the same time.

SULFUR BOMB

The charge consists of 0.5 g. of coal with 9 or 10 g. of sodium peroxide which, after being sealed within the holder, is thoroughly mixed by shaking. Ignition is effected by holding the bottom of the cup for a moment in the flame of a Meker burner or, still better, by having a jet of flame from a blast lamp strike the bottom of the fusion cup. This method avoids the use of a hot slug or fuse wire to be made red hot by an electric current. After ignition, which begins almost immediately, the cup is removed from the flame. Combustion is complete in less than half a minute. After cooling under the tap the cup is removed from the holder and placed on its side in a beaker of about 200 cc. capacity. Solution of the fusion is complete in a few minutes, when the cap may be removed, rinsed, and dried. It is then ready for another charge. The composition of the cup is such

¹ Preliminary Report of Joint Committee on Standard Methods of Coal Analysis, THIS JOURNAL, 5 (1913), 525; also *Ibid.*, 1 (1909), 689.

as to withstand the action of the fused alkali in excellent shape; no roughening or corrosion of the interior can be detected even after some hundreds of fusions have been performed.

The same apparatus has been used with much satisfaction in the determination of the sulfur in pyritic

material, in rubber and other organic combinations. It has a still wider application in the determination of carbonaceous matter in soils, and as a substitute for the Carius method of determining halogens in organic compounds.

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ADDRESSES AND CONTRIBUTED ARTICLES

THE PRESENT STATUS OF NITROGEN FIXATION^{1,2}

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INTRODUCTION

Fixed nitrogen in some form is an essential constituent of the food of all the higher animal and vegetable organisms. Fixed nitrogen in the form of potassium and sodium nitrates has been of prime importance in warfare since gunpowder came into general use. The ammonia resulting from the destructive distillation of coal has been recovered and used in the chemical industry for more than a century. Free nitrogen forms nearly eighty per cent of the air we breathe, but in the free form it can be utilized neither by the bodily mechanism nor in explosives or fertilizers. The chemist has known for many years how to convert this inert gas into other compounds in his laboratory, but it is only within the last twenty years that the fixation of nitrogen has been recognized as an industrial as well as a scientific problem, and only within the last five years that its importance has become generally recognized.

Sir William Crookes, in 1898, called attention to the diminishing supply of Chilean nitrate, and the need of replacing it with a synthetic product if the world was not to be confronted with possible starvation as a result of shortage of nitrogen fertilizers. But although this stimulated interest and may almost serve as a date for the commencement of industrial research on nitrogen fixation, it was ultimately war and not peace which caused the rapid development of the processes for fixation of atmospheric nitrogen. One of the proofs of Germany's cold-blooded calculation is found in the subsidized development of the nitrogen fixation industry. The sodium nitrate vitally necessary for explosives was found only in Chile, and its supply would almost certainly be cut off in a war with a first-class naval power. The German government did not declare war until it had the Haber, Ostwald, and cyanamide processes developed to the point where it knew it could become independent of Chilean supplies.

Almost all of the military explosives, whether used as propellants or as bursting charges, contain large percentages of the nitrate group. If this is to be supplied from sodium nitrate, there will be needed nearly two pounds of sodium nitrate for each pound of explosive, as shown somewhat more in detail in the following table:

EXPLOSIVE	NITRATE FACTOR
Smokeless powder.....	1.70
Trinitrotoluol.....	1.70
Picric acid.....	2.50
Miscellaneous high explosives.....	1.50

Ammonium nitrate, while not itself an explosive under ordinary conditions, becomes, when mixed with a portion of its weight of TNT, the very satisfactory high explosive amatol, important as a bursting charge for shells. Ammonium nitrate is the richest of all explosives in nitrogen.

¹ Published by permission of the Chief of Ordnance.

² Address delivered at the Chicago Meeting of the American Institute of Chemical Engineers, January 16, 1919.

SUMMARY OF FIXATION PROCESSES

It is the first step in nitrogen fixation which is the most difficult. The nitrogen molecule as it exists in the air is very inert and becomes active only at high temperatures or in the presence of some activating substance. The processes may be classified as follows:

I—THE ARC PROCESS for the direct combination of the nitrogen and oxygen of the air to form nitric oxide which subsequently by oxidation with air and combination with water forms nitric acid of approximately 35 per cent concentration. There are required about 10.5 h. p.-years electrical energy per ton of nitrogen fixed as nitric acid per annum.

II—THE CYANAMIDE PROCESS, involving:

(1) The production of calcium carbide through reaction between lime and coke in an electric furnace.

(2) The interaction of calcium carbide and pure nitrogen at a red heat to form calcium cyanamide.

(3) The decomposition of cyanamide by steam under pressure, to form ammonia.

(4) The oxidation of ammonia with air and combination with water to form dilute nitric acid of approximately 50 per cent concentration.

The power required by this process is approximately 2.5 h. p.-years per ton of nitrogen converted to nitric acid per annum.

III—NITRIDE PROCESSES. The best developed of these processes is that for making aluminum nitride from aluminum oxide, coke, and nitrogen heated to a temperature of perhaps 1800° C. in an electric furnace. This process has not been developed far enough to show its ultimate power requirements, but it is approximately in the same class as the cyanamide process. The aluminum nitride, after formation, may be decomposed with steam or dilute caustic solutions yielding ammonia and regenerating the alumina.

IV—THE DIRECT SYNTHETIC AMMONIA PROCESS, usually called the Haber process, wherein pure nitrogen and hydrogen are made to combine in the presence of a catalyst, at temperatures which in commercial work have usually approximated 500° to 600° C. and under a pressure of 100 atmospheres or higher. The ammonia made by this process is then oxidized with air and converted to nitric acid. Electrical energy is not necessary for this process and the total power requirements are only about 0.5 h. p.-year per ton of nitrogen fixed as nitric acid per annum.

V—THE CYANIDE PROCESS, wherein a mixture of sodium carbonate and coke with iron in small quantities is heated in a stream of pure nitrogen to a temperature of approximately 1000° C., resulting in the formation of sodium cyanide. This furnace product may be decomposed with steam, yielding ammonia. Power requirements for this process are of the same order as for the Haber process.

It will be seen that all of the above processes, except the arc process, yield ammonia as their initial product. The arc process requires the greatest expenditure of electrical power, the cyanamide and nitride processes rank next, and the direct synthetic ammonia and the cyanide processes require only small amounts of power. In fact, these two latter processes do not necessarily require any electrical power, it being possible to carry out all the heating reactions without the use of electrical energy, although electrical heating may in some cases be more economical. If nitric acid is desired, the ammonia produced by these processes may be oxidized to nitric oxide by air in the presence of a catalyst, usually platinum, working at 750° to 850° C. The nitric oxide resulting is oxidized by cooling, mixing with more air if necessary, and passing through towers, down which water or dilute nitric