

solution due to the action of the acid upon the organic base, or a decomposition product of the base. Seven fractionations, each one requiring a week, were carried out. The precipitates showed no divergence from the original, when in the form of neutral chloride solutions they were examined with the spectroscope. After the third precipitation, the filtrate became so turbid, either from decomposition products of the phenyl hydrazine, or through hydrolysis of such double compounds as are mentioned by Delafontaine, that the process was given up.

SUMMARY.

In endeavoring to prove the complexity of neodymium by fractional precipitation, in our hands the following methods failed: Fusion of the double nitrate (according to Berlin¹); precipitation by primary ammonium oxalate; solution in ammonium carbonate and precipitation by acetic acid; fractional precipitation by gaseous hydrochloric acid; precipitation by the organic bases— aniline, benzylamine, piperidine, and phenyl hydrazine.

The following methods are among those which will be tried in this laboratory: The use of sodium acetate and hydrogen peroxide, electrolysis, reduction by metallic magnesium, magnesium usta, treatment with mercury oxide and nitrate, copper oxide (method applied by Schutzenberger and Boudouard to cerium), dialysis and ammonium persulphate.

UNIVERSITY OF NORTH CAROLINA.

August 1, 1903.

[CONTRIBUTED FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF NORTH CAROLINA.]

CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS.

A GENERATOR FOR THE CONTINUOUS PREPARATION OF GASES ON A LARGE SCALE IN THE LABORATORY.

BY RESTON STEVENSON AND W. MCKIM MARRIOTTE.

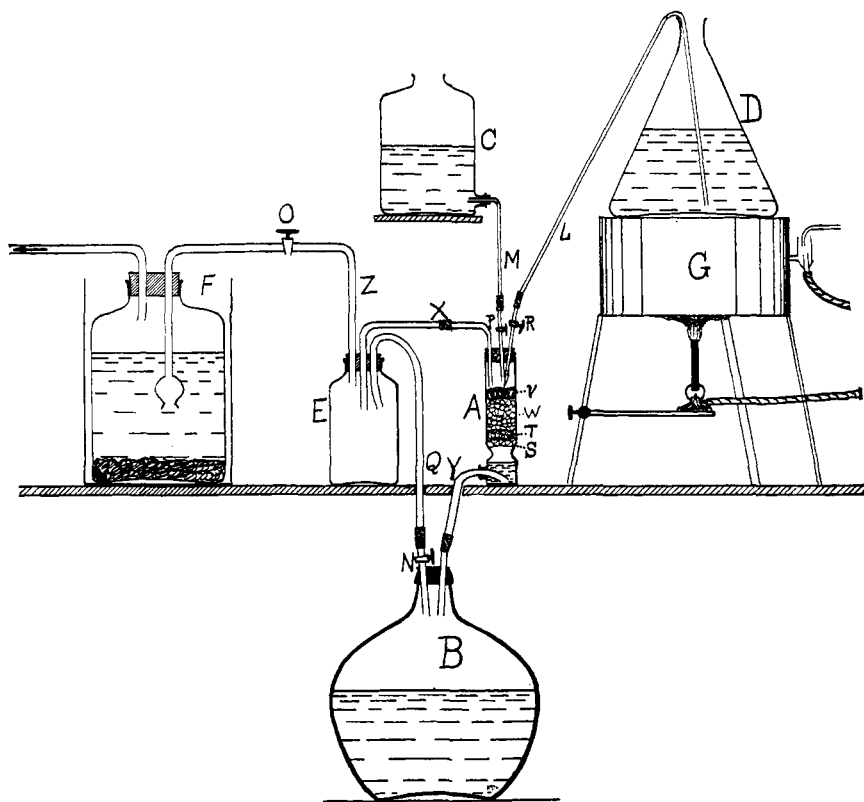
Received November 9, 1903.

A continuous stream of hydrochloric acid gas was required by one of us (S) in an investigation of the rare earths.² As the

¹ See preceding paper.

² Also modified by Dennis and Magee: This Journal, 16, 653.

amount of gas required was large and a steady flow necessary, the apparatus here described was devised, whereby commercial acids could be utilized with consequent economy. By variation of detail, the apparatus may be used for the generation of other gases. If desirable, the gases may be dried by insertion of suitable containers for the proper desiccating agents between *E* and *F* (see figure). The whole apparatus may be readily constructed of material in any laboratory. It was used for over a month, often night and day, requiring little or no attention, and gave perfect satisfaction.



The well-known method of generating gaseous hydrochloric acid by bringing its water solution in contact with strong sulphuric acid was found most convenient. Commercial hydrochloric acid was allowed to flow by gravity from the reservoir *C* through the

tube *M* into tower *A*. At the upper part of the constriction of *A* was placed broken cullet upon which rested glass wool. The tower was then nearly filled with glass beads, *W*, and this capped with glass wool, *V*. The tower was provided with a three-hole rubber stopper. Commercial sulphuric acid in the reservoir *D*, the bottom of which is on a level with the reservoir *C*, was siphoned through the tube *L* which projects through the stopper in *A* at an angle so that its end rests on the tube *M*, about 5 mm. from the end of the latter. These tubes are provided with stop-cocks *P* and *R* for regulating or discontinuing the flow of the acids. Allowing the sulphuric acid to flow down the tube carrying the hydrochloric acid this short distance was found to work most advantageously. The large Erlenmeyer flask *D* was placed upon a water-bath, *G*. By thus heating the commercial acid, a more rapid and larger yield of hydrochloric acid was obtained. As a mixture of acids passed through the glass wool and beads, an intimate mingling is brought about and the gas generated led through the exit tube *X* into the safety-flask *E*, from which the gas goes through *Z* into the vessel *F* in this particular case. To equalize pressure and to remove the partially spent acids, they were led from the bottom of the tower by gravity through the tube *Y* bent toward the bottom of the tower *A* into a large carboy *B*, resting on the floor. This carboy is provided with a two-holed rubber stopper through which passes the glass tube *Q* into a safety vessel *E*. By these means an equalization of pressure is had and the remaining gas generated in the waste acids obtained. Four stop-cocks, *N O P R*, were inserted for the regulation of the flow of acids or gas. The generation of the acid could be discontinued at any moment by closing them and disconnecting just beyond the stop-cock *O*. The safety-flask *E* served the dual purpose of equalizing pressure and collecting any back-flow of liquid from the precipitating vessel *F*, when such an accident occurred. Suitable rubber connections were made when necessary.

We wish to thank Professor Charles Baskerville for his interest and permission to publish the description of the apparatus.