filled with the insoluble sulphate and it becomes hard as stone. The soft, porous, cheaper grades of wood are thus rendered equal in point of durability and strength to the hardest varieties of timber. Wood prepared in this way is largely used in England in connection with public works and railways.

[TO BE CONTINUED.]

[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY U. S. DEPARTMENT OF AGRICULTURE, SENT BY H. W. WILEY, NO. 12.]

NOTE ON THE ESTIMATION OF IRON AND ALUMINA IN PHOSPHATES.[1]

BY K. P. McELROY.

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IN 1888 Dr. Crampton, then First Assistant Chemist of the United States Department of Agriculture, was engaged in the analysis of a lot of baking-powders and did me the honor to entrust to me a portion of the analytical work. Among other things I had to determine the alumina in a number of alum phosphate powders and met with the usual difficulties. By the method I was instructed to use, I found it impossible to get results that were concordant. I therefore systematically went through all the known methods for separating alumina from phosphoric acid and finally decided to use the one accredited to Mr. T. R. Ogilvie in Crookes' Select Methods (page 538, edition of 1886). Briefly stated the method consists in neutralizing the filtrate from the phosphomolybdate precipitate with ammonia, redissolving the precipitate of mixed sesquioxides thus formed, in nitric acid, once more precipitating, filtering, burning, and weighing. The process gave very good duplicate results, and a trial with a known amount of alumina in the form of alum mixed with sodium phosphate gave results sufficiently near theory. This, together with the fact of its having appeared in a work so standard as that of Crookes, seemed sufficient credentials for the method, and I placidly went ahead with its use. When Dr. Crampton prepared his manuscript (which I did not see prior to publication) for his bulletin on baking-powders (Bulletin 13, Part Five, Division of Chemistry, U. S. Department of Agriculture) he gave me credit for the minor modifications I had made

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[1] Read at the Boston Meeting, December 27, 1894.

in the method, such as getting the alumina into solution, making up to volume, and the like. As to the main part of the method, the precipitation of alumina by ammonia in presence of molybdate solution, he had as little intention of accrediting it to me as I had of claiming it. He simply said (page 595) that the method "was adapted to the powders by Mr. K. P. McElroy."

Several years later Mr. W. H. Krug of this laboratory was engaged in making an exhaustive examination of a number of phosphate samples and had considerable trouble in determining iron and aluminum. In this connection he tried a number of the current methods on synthetic solutions and got results not agreeing with each other or with theory. At this juncture I suggested that he try the molybdate method. He used it on some phosphates which he had previously examined by the Glaser, Stutzer, etc., methods and obtained much better results. Subsequently he published a paper (*J. Anal. Appl. Chem.*, *1891*, **5**, 671) detailing his work and incidentally he courteously acknowledged my suggestion by saying of the molybdate method that it "was first used in this laboratory by Mr. K. P. McElroy for the estimation of alumina in phosphatic baking-powders." I had also suggested to Mr. Krug some modifications differing in minor detail from those of Ogilvie, by which it would be possible to determine calcium and magnesium in the same portion after precipitation of the iron and aluminum, and these Mr. Krug also published giving me full credit.

For some time since I have known that various chemists had fallen into the habit of calling this method for ferric oxide alumina, the "McElroy method" or "Krug method." Neither of us has taken the trouble to correct this mistake except orally since Crookes' Select Methods is a book of nearly as frequent occurrence in chemical laboratories as Fresenius' Quantitative Analysis, and publishing such a denial of authorship seemed nearly as ridiculous as it would be for one of us to rush into print to announce that he was not the originator of the method for the estimation of sulphuric acid as barium sulphate. Recently, however, a Northern chemist wrote a letter to the Department of Agriculture, designating the method by my name and announcing that in the case of a certain phosphate rock the method had failed to give results concordant with those of other chemists using other methods. I had always known that the molybdate method did not give results agreeing very closely with those of other methods, but as I had also known that most of these other methods gave incorrect results, I had not laid the fact up against the molybdate method. In view of this specific complaint, however, I have tried the method on known solutions and am astonished to find that it really is liable to lead to grave error.

In this work I tried several samples of the molybdate solutions prepared according to the directions of the Association of Official Agricultural Chemists, which happened to be in the laboratory and found that they gave varying results. The worst results were obtained with a solution made up about six months ago, from which a heavy crystallization has taken place, leaving the residual liquid so weak that it requires over 100 cc. to precipitate the amount of phosphoric acid supposed to be thrown down by fifty cc. of a fresh solution. In two beakers I placed portions of ferrous sulphate representing each 56.4 mgms. of ferric oxide, added a solution of sodium phosphate representing 100 mgms. of phosphorus pentoxide to each, brought into solution in weak nitric acid, oxidized with bromine water, and then precipitated with the molybdate. I washed the precipitate with weak nitric acid and neutralized the combined filtrate and washings with ammonia. The resultant precipitate was dissolved in a solution of ammonium nitrate and nitric acid, filtered, and again thrown down. This precipitate was collected on a filter, burnt, and weighed. The amount recovered was respectively 56.9 and 57.3 mgms., showing a small plus error. In a duplicate set of experiments calcium nitrate equivalent to 400 mgms. of calcium oxide was added to each of the initial solutions. The amount of precipitate recovered in one case was 62.3 mgms. and in another 63.1 mgms., showing a large plus error. During the first precipitation a heavy white crystalline deposit of calcium molybdate occurred. The weighed precipitate was found to contain both lime and molybdic acid, showing that the same phenomenon had occurred in the second precipitation, though it was not visible to the eye.

In each of two beakers were next placed 35.6 mgms. of alumina in the form of ammonia alum, together with 100 mgms. of phosphorus pentoxide in the form of sodium phosphate and the mixture treated as were those containing iron. The two precipitates of aluminum oxide finally obtained, weighed respectively 28.9 and 29.3 mgms., showing a large minus error. Another set containing 400 mgms. of lime was run through. The same precipitation of calcium molybdate was found to occur. The weights of alumina obtained were but 19.8 and 20.6 mgms. respectively, showing a much larger minus error.

From the above surprisingly bad results it is shown conclusively that the molybdate method for iron oxide and alumina, at least in this form is not at all trustworthy. Yet I have myself at times, and so have others, obtained good results by its use where these results were checked against those made by some of the standard methods used in delicate work.

SOME PRACTICAL POINTS IN THE MANUFACTURE OF NITROGLYCEROL.[1]

BY J. E. BLOMÉN, PH.D.

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FROM the very beginning of the commercial manufacture of nitroglycerol the aim of the inventors and manufacturers has been to eliminate the dangers of the operation to the highest possible extent and they have been so successful in this that very few, if any of the many accidents on record can be traced back to the apparatus. The problem is simple enough as the reaction is easily controlled. The indications of approaching danger are clear and the means to avoid it are easily kept at hand. From the old wooden boxes worked by hand with a paddle-wheel to the present elaborate apparatus, the principle is the same—to secure an even and low temperature through the mass of the mixture of acids and nitroglycerol.

It has long been considered that the safest way of stirring is with compressed air, but this can hardly be taken as an axiom. The water, which is formed during the reaction, is of the same temperature as the mixture and does not tend to elevate the temperature, but the moisture brought in with the air certainly does

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[1] Read at the Boston Meeting, December 27, 1894.