XXXV.—*On the Detection and Estimation of Free Mineral Acids in various Commercial Products.*

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THE determination of free mineral acids under ordinary circumstances is a very simple matter when we are provided with a standard alkaline solution, and a few drops of litmus; but the increasing requirements of chemical industries render it imperative upon the chemist to discover and estimate with exactness these acids *in the presence of salts having an acid reaction, or it may be of other acids*. All persalts, and salts of zinc, copper, &c., it is well known, distinctly redden litmus paper,

* This number is approximate only : it was obtained by calculation from a shorter column.

although they may be basic in constitution, and standard alkali fails to distinguish sulphuric from acetic acid.

Of late years, the manufacture of sulphate of alumina from china clay and bauxite has assumed great importance, arising from the immense quantities used in paper-making, and as it is not a product capable of purification by crystallisation like alum, the quality of the article is in too great a degree dependent on the skill and conscientiousness of the manufacturer. The paper-maker is often too little of a chemist to decide which of several samples is the best, when, as is almost invariably the case, the whitest to the eye and strongest to the taste contains the most free acid and least alumina.

Our method is based on the fact that peracetate of iron, even in dilute solutions, has a distinct yellow colour, not perceptibly altered by acetic acid or solutions of persulphates, but instantly bleached by free sulphuric, hydrochloric, and nitric acids. The solution is made by dissolving 10 parts of iron alum and 8 parts of crystallised acetate of soda (both previously dried on blotting-paper) in 1,000 parts of acidulated cold water, made by mixing 920 parts of water and 80 parts of distilled acetic acid (25 per cent.).

Any person of ordinary intelligence, when provided with this solution, can in a few minutes readily determine the relative quantity of free acid in a cake by putting a piece about the size of a pea in a small white basin or cup containing two or three drops of cold water. Add one drop of the test-solution and shake. If the colour disappears add another, and so on until a slight yellow tint remains. In proportion to the number of drops bleached will be the acid character of the cake, and if that number be divided by five, the answer will approximately represent the percentage of free acid. For dropping the test-solution into the basin, we have found most suitable a small glass stoppered bottle, the stopper of which has a slit on each side, made by means of a file. This bottle, when inverted, allows the liquor to drop out without removing the stopper, as the air enters the upper slit, while the liquor drops out through the lower. The acetic acid is added to render the otherwise decomposable solution perfectly stable on keeping.

With most cakes in the market, bleaching of more than one drop will be instantaneous.

We have only met with one aluminous salt, excepting crystal alum, that does not affect the solution.

For this test it is not necessary to separate silica, present in most aluminous cakes.

When the material under examination is in the liquid form, a convenient quantity, say 200 grains, or, roughly, $\frac{1}{2}$ oz., is weighed out in a small beaker, and a standard solution of caustic soda run in from a burette, stirring after each addition, until two or three drops put in a

small white porcelain basin do not perfectly bleach one drop of the acetate solution.

Highly coloured mixtures are of course not well adapted for this test, but these do not occur in practice.

For accurate quantitative determinations the best procedure we have found to be as follows:—Dissolve 200 grains of the sample in the acetic acid water previously mentioned, triturating in a small mortar until all the soluble is extracted. Now add from a burette *strong* solution (made by dissolving 10 parts iron-alum and 8 parts of crystallised acetate of soda in about 50 parts of acidulated water) of the peracetate until the mixture has a decided yellow tint, showing that all the free acid has been neutralised. Dilute to 100 measures with the acetic acid water, and filter off a portion into a test-tube. In another test-tube put as many measures of the strong acetate of iron solution as have been used for the cake solution. Dilute to about 80 measures with acetic acid water, and drop in standard solution of sulphuric acid until the colour is the same as in the other test-tube. The bulk of the liquor must be so arranged that it equals that of the other, viz., 100 measures. The amount of sulphuric acid used is the measure of the free acid in the cake, and divided by 2 gives the percentage.

In this way the results are wonderfully concordant and remarkably expeditious, and we may say we have used it daily for the last two years. Even alum, which has always been understood to be a perfectly neutral salt of alumina, can be readily proved by it to contain small, though commercially insignificant, proportions of free acid, when large quantities are recrystallised, and the free acid is thus concentrated in the mother-liquor.

Other applications of this property of peracetate of iron may be suggested, such as the estimation of free acid in salt-cake, furnaced sulphate of potash, sulphate of ammonia, acetic acid, &c.