

is too rapid, the proportion of the constituents of higher molecular weight which get into the charcoal is small and therefore little is held back; if the flow is too slow they will return by diffusing out of the charcoal into the whiskey. If the liquid remains in contact with the charcoal long enough, there will be nothing removed except the very small amount held by adsorption.

Third.—In the case of the furfural, I believe it is removed wholly by adsorption, because in every instance it is completely taken out by the charcoal. The same applies to the fatty and essential oils in solution.

Fourth.—I do not believe that oxidation plays any part in the process because whatever action absorbed oxygen may have in the pores of the charcoal, it would be entirely exhausted on the portion of the distillate which is used to wash the alkali out of the charcoal.

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NOTES.

The Determination of Antimony and Arsenic in Lead-antimony Alloys.
—In the March issue of the present volume of **THIS JOURNAL** (p. 378) the author published a paper on the above subject, and now wishes to call attention to a rather important modification which further experience has shown to be necessary.

While accurate results are obtained in many cases by using the method of dissolving described in this paper, there is always danger of losing arsenic by volatilizing, due, apparently, to the reduction of arsenic to arsenious chloride by the action of the undissolved portion. Some alloys are much more subject to this loss than others, the reason for which fact is not quite clear, as the behavior seems to be independent of the amount of arsenic present. By using a sufficiently large excess of nitric acid this loss can be avoided; but a better way seems to be to heat the alloy with hydrochloric acid alone until action ceases before adding the oxidizing agent. The lead is thus dissolved out, leaving the antimony and arsenic in very finely divided condition, and in no case have I been able to detect a trace of the latter two in solution. With the addition of nitric acid or potassium chlorate the antimony and arsenic then dissolve very readily upon shaking, without any further heating. I have not found any loss of arsenic in the subsequent boiling to get rid of free chlorine and nitrous gases.

The procedure recommended is as follows: Heat the finely divided alloy (it is a good plan to run the filings through a fairly fine sieve) with hydrochloric acid until action ceases. Remove the flask from the plate, add about 0.5 cc. of nitric acid and let it stand a few moments until the reddish color is obtained. Then shake the flask, when the antimony and arsenic will dissolve quickly and completely. Now place upon the plate

again, boil vigorously for five minutes or so, and proceed as in the original method.

There are several other points in the method which might be considered further. The time given for passing the air (one-half hour) applies to the case when there is no salt, such as stannous chloride, to be oxidized. This factor will also vary, naturally, with the rapidity of the stream, and will have to be determined for the special conditions of different operators.

In filtering off the arsenious sulphide, time in washing can be saved if the solution is not diluted further than is necessary in washing out the tubes, etc. This keeps more lead chloride in solution, and makes the washing less tedious, particularly if hot water is used throughout.

If the alloy is very high in antimony care must be taken that sufficient tartaric acid is used. The amount necessary has never been carefully determined, but an equivalent of 5 grams is sufficient for ordinary cases (less than 0.1 gram antimony).

If there is considerable excess of lead chloride, so that it crystallizes out to any extent in the antimony solution, it is necessary to use a larger excess of bicarbonate. In this case, also, the end point is not so sharp, although with practice it can readily be determined. The blue color should be permanent for a minute or so, at least.

In oxidizing the arsenious solution 10 cc. of hydrogen peroxide (3 per cent.) is sufficient for all ordinary cases, and the solution must be distinctly alkaline. If this is not the case, shown by separation of sulphur, add a few more drops of sodium hydroxide. It is a good plan also to wash down the sides of the flask before the boiling is finished, as sometimes a little hydrogen peroxide remains undecomposed, and reacts with the potassium iodide.

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Apparatus for Polarizing at 87°.—Analysts who have to polarize sugar solutions at 87° C. after inversion are familiar with the more or less inconvenient methods or arrangements for obtaining this temperature in the jacketed polariscope tube.

The writer uses the arrangement shown in Fig. 1 and has found it very satisfactory, both for occasional readings as well as for making a number of readings on the same day; it effects a great saving of time. As will be seen from the figure, the polariscope tube and contents are heated by means of an instantaneous water heater. The heater shown can be bought from any dealer in chemical apparatus. In Fig. 1, CW is the cold water supply, G the gas supply. The cold water flows into a copper-tube coil enclosed in HC under which there is a large gauze burner with a pilot light at P. By means of a combination valve, GWV, both the cold