

## SUMMARY.

A practical working outline for the microscopical identification of potassium chloride, potassium and sodium tartrate, potassium nitrate, sodium chloride and sodium nitrate has been devised. The identification is based on the fact that these substances have significant refractive indices which are important in identifying small quantities of material in practical analytical work.

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AN IMPROVED METHOD FOR THE ASSAY OF MERCURIAL OINTMENT U. S. P. AND BLUE OINTMENT U. S. P.\*

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The present official method for the assay of Mercurial Ointment and Blue Ointment, while accurate and reliable in itself, is not well suited for use in a Control Laboratory, where time is an important factor and where several lots may be held in process awaiting a chemist's report. At best, it is a time-consuming operation, and in the hands of one not thoroughly experienced with the method there is considerable chance for error. Chemists who are not well versed in the method frequently lose mercury in decanting the benzin and also have trouble collecting all the mercury in a globule while in the hydrochloric acid solution.

As this Laboratory has a large number of samples of both Mercurial and Blue Ointments to assay a modification of the official method was sought which would be more rapid and also require less experience for its satisfactory use.

The official assay at present reads as follows:

**Mercurial Ointment.**—Weigh 10 Gm. of Mercurial Ointment in a tared dish, melt it, then remove it from the fire and add 50 mils of warm purified petroleum benzin. Stir the mixture well, allow the mercury to settle completely, and decant the benzin. Wash the residue with successive portions of 10 mils each of warm purified petroleum benzin until it is entirely free from fatty matter, carefully retaining all of the separated mercury in the dish, and allowing all traces of the benzin to evaporate. Add to the residue 10 mils of diluted hydrochloric acid, heat it gently and stir with a glass rod until the mercury collects in a globule. Pour off the acid, warm the mercury with a little distilled water, dry the globule on bibulous paper, and weigh. The separated mercury weighs not less than 4.9 Gm. nor more than 5.1 Gm.

**Blue Ointment.**—Proceed as directed in the assay under Mercurial Ointment. The separated mercury weighs not less than 2.9 Gm. nor more than 3.1 Gm.

In examining this method for possible improvement purified petroleum benzin was not thought to be the ideal solvent. The prepared suet, and yellow wax used during hot weather in order to stiffen the ointment, are very slowly soluble in this solvent in the cold; and it is unwise to warm the mixture since the benzin begins to boil at 40 deg. C. with considerable bumping and spattering, often resulting in mechanical loss of mercury. Also considerable time is required for a complete separation of the residue containing the mercury and the solvent and in this Laboratory any saving of time in this assay is important.

After trying various solvents it was found that ether had a distinct advantage

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over petroleum benzin. The fats and wax are more readily soluble in the cold, and the separation takes place much more rapidly.

Among other solvents tried xylol showed promise of being of value as it could be warmed above the melting point of the fats and wax in the ointments without boiling or spattering, and thus effect complete solubility almost at once. However, when applied to the ointments without first removing some of the fats, the separation was as slow as when benzin was used, with no saving in time. When used, however, after most of the fats had been removed with ether, it was most effective in producing a practically fat-free mercury residue. Since the xylol cannot be removed from the mercury residue at ordinary temperatures, it was found necessary to remove the small amount remaining after decantation with a further washing of ether. If anhydrous ether has not been used, the xylol will sometimes cause a few drops of water to collect on the surface of the mercury. This can be readily removed by a single washing with ten or fifteen cc. of alcohol.

By using these solvents the treatment with dilute hydrochloric acid is rendered entirely unnecessary. After the last ether washing to remove the excess xylol is completed, the mercury residue is dried with a gentle current of air and a few strokes with a rubber-tipped stirring rod serve to collect all of the mercury in a globule, leaving behind a slight dark gray residue of mercury oleate. In case any of the mercury oleate adheres to the globule of pure mercury, it can be easily brushed off with a camel's hair balance brush.

We have, therefore, developed a modification of the U. S. P. method for the assay of Mercurial Ointment and Blue Ointment which is more rapid and equally accurate.

Its accuracy has been proved by numerous comparisons with the official method upon the same sample of ointment and upon ointments made up in the Laboratory containing a known amount of mercury. The time-saving is considerable, since a complete assay can be run in a little less than an hour and while ordinary quantitative accuracy is necessary there need not be such extreme attention to detail as is required by the official method.

Stated briefly, the directions for using this method, which we call the "Ether-Xylol Method" are as follows:

Weigh ten grams of the ointment into a 100-cc. tall form beaker and warm gently to soften the mass and make it more easily workable. Add about 50 cc. of ether, anhydrous ether preferred, and stir until the mixture is uniform with no lumps of ointment remaining. Allow the mercury to separate and decant the ether as closely as possible without losing any of the mercury. Repeat the washing with another 50 cc. of ether and decant as closely as possible. Now add about 50 cc. of xylol and heat on the steam-bath to about 80 deg. C. with stirring. Allow to stand at this temperature for five minutes, or until all of the mercury has separated, and decant the xylol as closely as possible. If any moisture is present in the mercury residue wash it out with a single washing of 10 or 15 cc. of alcohol. Finally wash again with 50 cc. of ether to remove the excess xylol and alcohol and dry the mercury residue in a stream of air. Collect the mercury in a globule by rubbing gently with a rubber tipped stirring rod. If any particles of Mercury Oleate adhere to the mercury globule, brush them off with a camel's hair balance brush. The separated mercury, if from Mercurial Ointment, shall weigh not less than 4.9 Gm. nor more than 5.1 Gm., and if from Blue Ointment shall weigh not less than 2.9 Gm. nor more than 3.1 Gm.

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