THE MICROSCOPICAL IDENTIFICATION OF SOME SODIUM AND POTASSIUM SALTS.

BY GEO. L. KEENAN.

Some sodium and potassium salts in admixture are often difficult to identify chemically when two or more acid radicals are present. For example, in a chemical examination of a mixture of potassium chloride and sodium nitrate, it is a simple matter to identify nitrates, chlorides, sodium, and potassium, but these results do not furnish any information as to the combination of the sodium and potassium with the acid radicals that might be present. To overcome this difficulty it has seemed worth while to outline a method of procedure for the microscopical identification of five of these salts commonly met in food and drug analysis—sodium chloride (NaCl), potassium chloride (KCl), potassium nitrate (KNO_3), sodium nitrate ($NaNO_3$), and potassium and sodium tartrate (Rochelle salt, $KNaC_4H_4O_6.4H_2O$).

In describing the method it is taken for granted that the analyst has a microscope equipped with the lenses commonly furnished with such an instrument, a rotary stage, and a micro-polariscope. The most common form of the micro-polariscope consists of two nicol prisms, the analyzer and the polarizer. The polarizer is placed in the diaphragm carrier which swings out from under the condenser beneath the microscope stage. The analyzer is placed at some level above the object, often above the ocular, or it may consist of a combination ocular-analyzer in which case it replaces the ordinary ocular in the tube.

In addition to the microscope and micro-polariscope, five oily liquids of known refractive index are necessary. These may consist of single oils or of mixtures, the index of refraction of which must be determined by a refractometer and, if not of the desired value, must be brought to that point by mixing in small quantities of one or the other of the oils.

1. Refractive index 1.488 = Squibb's mineral oil.
2. Refractive index 1.495 = toluene or a mixture of mineral oil and clove oil.
3. Refractive index 1.505 = sandalwood oil or a mixture of clove oil and mineral oil.
4. Refractive index 1.545 = clove oil or a mixture of mineral oil and monochlornaphthalene.
5. Refractive index 1.585 = aniline or a mixture of mineral oil and monochlornaphthalene.

DIAGNOSTIC OPTICAL PROPERTIES.

Sodium chloride and potassium chloride crystallize in the isometric (cubic) system and therefore exhibit no change from light to dark when the microscope stage is rotated with nicols crossed (dark field). Each of these salts possesses but one refractive index which is significant in establishing the identity of the substance. The index of refraction characteristic of sodium chloride is 1.544 and that characteristic of potassium chloride is 1.490. Potassium nitrate, sodium nitrate, and potassium and sodium tartrate, on the other hand, exhibit changes from light to dark when the stage is rotated with the nicols crossed, *i. e.*, they are doubly refractive and also possess two or three significant refractive index values which make possible their identification. The identification of potassium nitrate is easily

accomplished by reason of the fact that a mean value of the two higher refractive indices, approximating 1.505, is shown so frequently that it becomes of diagnostic importance in practical analytical work. The intermediate index significant for potassium and sodium tartrate is 1.495 and the index value diagnostic for sodium nitrate is 1.587.

PROCEDURE.

(*a*) A small quantity of the crystalline material is crushed on a microscope slide, a drop of the liquid with $n = 1.505$ is added, and the cover-glass is applied. The preparation is transferred to the microscope stage and examined in the dark field formed by crossing the nicol prisms (analyzer and polarizer). If the crystal fragments exhibit no change from light to dark when the stage is rotated with nicols crossed, the substance in question must crystallize in the isometric system and may be either sodium chloride or potassium chloride. On the other hand, the fragments may become alternately dark and light when the stage is rotated with crossed nicols. In this instance sodium nitrate, potassium nitrate, or potassium and sodium tartrate may be sought.

(*b*) The preparation is next examined in ordinary polarized light. To do this replace the analyzer with the ocular, leave the polarizer in position, and slightly close the substage diaphragm. If the first examination with crossed nicols has shown that the substance is isometric, some indication as to whether it is sodium chloride or potassium chloride may be gained from the behavior of these fragments when the microscope tube is slightly raised. A band of light will pass from the liquid into the crystal fragment if the refractive index of the substance is higher than that of the liquid; or conversely, the band of light will pass from the crystal into the liquid if the liquid has a higher index of refraction than the crystalline material. This test will determine whether the substance has a refractive index value approaching that of either sodium chloride or potassium chloride. If the first examination with crossed nicols has shown that the substance is not isometric but the fragments become alternately light and dark when the stage is rotated, there is a possibility that the substance may be potassium nitrate, sodium nitrate, or potassium and sodium tartrate, confirmation of any of which can be made according to the following directions:

CONFIRMATION OF SODIUM CHLORIDE.

($n_D = 1.544$)

Since the preliminary tests already described have indicated that the substance examined was isometric and had an index of refraction higher than the liquid, 1.505, a preparation is made by crushing a small quantity of the material and mounting it in a drop of the liquid having a refractive index of 1.545.[1] Examining the preparation in ordinary light shows that the identity of the crystal fragments has been practically lost in the liquid. The definite outlines of the fragments have disappeared, but most of these fragments have yellow and bluish bands of light surrounding them, and nearly every one has its central portion characterized by dark bordered pockets of air. In other words, the phenomena described indicate that

[1] Isometric substances, not being doubly refractive, must be examined in liquids slightly higher or lower than the diagnostic index in order to be visible.

the refractive index of the substance, sodium chloride, has been approximately matched against that of the liquid and the substance definitely identified.

CONFIRMATION OF POTASSIUM CHLORIDE.

($n_D = 1.490$)

If examination of the unknown isometric substance in liquid with refractive index of 1.505 has shown that the substance has a refractive index decidedly lower than that of the liquid, *i. e.*, a band of light is seen to pass out from each fragment into the liquid as the tube is raised, then a fresh portion of the material should be crushed and mounted in a drop of the liquid having an index of refraction of 1.488. If the substance in question is potassium chloride, the identity of the fragments will be lost when mounted in this liquid. The location of the fragments is established only by the yellow and blue bands surrounding some of them and by the inclusion of air in the interior of the fragments.

CONFIRMATION OF POTASSIUM NITRATE.

($n_\alpha = 1.335$, $n_\beta = 1.505$, $n_\gamma = 1.506$)

It is a simple matter to identify the substance as potassium nitrate when it is mounted in the liquid with a refractive index of 1.505. On rotating the stage (using ordinary polarized light) the eye is attracted by the large number of fragments which completely lose their identity in some position in the field, the margins of the fragments disappearing as though substance had gone into solution. In other words, mean values of the refractive index approaching that of the liquid, 1.505, occur so frequently in the substance that no difficulty whatever is encountered in confirming the identity of the material as potassium nitrate. The fragments have then been approximately matched against the refractive index of the liquid, 1.505, which is diagnostic for this substance.

CONFIRMATION OF SODIUM NITRATE.

($n_\omega = 1.587$, $n_\epsilon = 1.336$)

The presence of sodium nitrate may be substantiated by mounting the material in a drop of the liquid having a refractive index of 1.585. The margins of most of the fragments practically disappear in some position in this liquid when the stage is rotated (using ordinary polarized light). This liquid is diagnostic for the substance and makes it possible to establish its presence without difficulty.

CONFIRMATION OF POTASSIUM AND SODIUM TARTRATE.

($n_\alpha = 1.492$, $n_\beta = 1.493$, $n_\gamma = 1.496$)

The presence of potassium and sodium tartrate is confirmed by mounting the crystalline material in a drop of liquid having an index of refraction of 1.495. This refractive index value occurs so frequently in the material that it is easily matched and therefore of value in establishing the identity of the substance.

TABLE OF DIAGNOSTIC REFRACTIVE INDICES.

Substance.	Diagnostic refractive index.
Potassium chloride	1.490
Potassium and sodium tartrate	1.493
Potassium nitrate	1.505
Sodium chloride	1.544
Sodium nitrate	1.587

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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