

ART. IV.—POTASSIC IODIDE AS A BLOWPIPE REAGENT.—By
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In the Transactions of the Royal Society of Canada for 1883 there is a paper by Prof. E. Haanel, Ph. D., "On the Applications of Hydriodic Acid as a Blowpipe Reagent." In this paper the author states how he was led to the use of iodide coatings in place of the ordinary characteristic oxides deposited on the charcoal, and his method of making and using the hydriodic acid, and also the little Plaster of Paris tablets. Many of the metals yield very characteristic coatings by this method, and the paper is illustrated by beautifully coloured diagrams of some of them.

While doing advanced blowpipe work under Prof. Kennedy at King's College, Windsor, he advised my following out Dr. Haanel's experiments. This I did, and easily got all his results. We found however one serious difficulty in the practical application of the method. Hydriodic acid is an unstable compound, the iodine tending to separate and come down as iodine crystals. These can, it is true, be re-dissolved by passing a stream of hydric sulphide through the acid for a little while, but this requires a laboratory and removes the reagent from a portable blowpipe outfit. Then again the acid must be prepared by the user.

These objections led Prof. Kennedy to suggest the use of a solution of potassic iodide and an acid. This I tried with most satisfactory results. The method of using the reagent is as follows: The assay being placed in a little hollow near one end of one of the plaster tablets, one or two drops of the solution of potassic iodide are deposited on and around it, and are absorbed by the tablet, then a drop or two of acid, preferably sulphuric, is added and the assay heated before the blowpipe. The potassic iodide is decomposed and vapours of iodine are given off. Some of the iodine unites with the assay to form an iodide of the metal in it. These iodides, many of them of brilliant hues, are deposited

on the tablet at a distance from the assay varying with their volatility. The potassic iodide solution I keep in a dropping bottle similarly to nitrate of cobalt. Acids are always present in a complete blowpipe cabinet. The keeping properties of the potassic iodide solution are proved by the fact that the solution now in the bottle, and with which some of the tablets before you were prepared, has been there since February, 1887, and still does its work perfectly.

When I came to renew these experiments in preparing this paper it occurred to me that it would be a great improvement if I could produce the same results with the dry crystals of Potassic Iodide. As this reagent will only produce the proper results in the presence of an acid it was necessary to find a dry acid to be used with it. As sulphuric acid appears to be the best to use with the solution of potassic iodide, it seemed best to try a crystalline sulphate. Copper and zinc sulphates would not do, as each of these metals gives a characteristic coating with Potassic Iodide. I therefore tried common sulphate of iron, green vitriol, and found that the best method was to powder both crystals and mix the powders well before adding to the assay. By this means the method becomes as simple and convenient as the use of carbonate of soda or any of the fluxes ordinarily in use in blowpipe work. Thus we have distinctive tests by a thoroughly practical method for some metals and a second or check on the ordinary tests for others. If one wishes to take a blowpipe outfit, for determining minerals, on a tour in the country, it is a great advantage not to have to carry acids. With regard to the plaster tablets, Dr. Haanel states that when blacked in a gas flame they are equal if not superior to charcoal for oxide coatings. If so, and if they can be made to take the place of charcoal for ordinary field work, it is an immense advantage, as they are easily prepared, clean to handle and occupy little space. I adopted Dr. Haanel's method of making them, which is to spread a coating of plaster of Paris about $\frac{1}{8}$ inch thick on a sheet of glass and just before it hardens to mark out the tablets with cuts nearly through to the glass. When dry they readily break apart. This method with dry potassic iodide has added seven

elements to the list of those that can be determined by a dry assay.

I have not specimens of some of the elements whose reactions are mentioned by Dr. Haanel, so could not test all his results with the dry crystals. The following are some of the results I have obtained. *Arsenic*.—The mineral used was orpiment, the common sulphide of arsenic. The characteristic reddish orange coating comes out very well with both the liquid and the dry potassic iodide. *Lead*.—The chrome yellow coating comes out very well with the dry reagents, and also with liquid potassic iodide and a drop of hydrochloric acid. *Tin*.—The coating is brownish-yellow, but is difficult to obtain, and fades rapidly so that a specimen tablet cannot be kept. With hydriodic acid the color comes out fairly well, but only poorly with potassic iodide solution either alone or with sulphuric acid and hardly at all with the dry crystals. Both metallic tin and the ore known as stream tin were tried.

The orange-red coating of *Antimony* is very easily got. A fragment of stibnite, the ordinary sulphide, giving a very decided re-action with potassic iodide solution alone or with an acid, and also with the dry crystals. *Bismuth* yields its chocolate-brown iodide readily, both with liquid and dry potassic iodide. *Gold*.—This metal gives a faint pink to purple coating, somewhat difficult to obtain, but coming out by either method. *Molybdenum* yields an ultramarine oxide coating with the liquid reagents, but I have not yet succeeded in producing it satisfactorily with the dry. A specimen of Molybdenite was the mineral used. *Tungsten* yields a faint greenish-blue coating which is an oxide. It is easily produced by potassic iodide with hydrochloric acid. *Silver*.—The iodide coating given by this metal is a canary yellow turning to greyish-yellow when cold. It fades away almost entirely ultimately. The coating comes out well with potassic iodide solution and nitric acid. It is also good with the dry reagents. *Mercury* yields a scarlet and yellow coating, the yellow very evanescent and changing to scarlet. The change may be hastened by breathing on the coating. The results with potassic iodide solution and sulphuric acid are good, also those with the

dry reagents. Grey fumes of mercury are often deposited at first but may be changed to yellow and red by touching them with the blowpipe flame. *Cobalt* gives a greenish-brown coating, the brown evanescent and passing into faint green. This fades out ultimately. Selenium, Tellurium, and Thallium yield characteristic coatings with hydriodic acid according to Dr. Haanel, but I have never had specimens of them to test for myself. Tungsten I tried when at College with potassic iodide solution, but had none to test with dry reagents now. Copper, cadmium and zinc all give white coatings, but the tablets have to be blackened to perceive them and then they can only be differentiated by some wet chemical test, so I have omitted them altogether.

After writing the above it occurred to me that as hydriodic acid is prepared in the laboratory from phosphoric iodide, possibly soda-ammonic phosphate, commonly called microcosmic salt, or salt of phosphorus, might serve to mix with the crystalline potassic iodide. Time did not allow of going over all the tests again with this substance, but those that were tried gave most satisfactory results.

This is a most decided improvement as microcosmic salt is always present in a blowpipe cabinet. The following are the results obtained with this reagent. Arsenic, lead and antimony yield good results. Cobalt is better than with ferric sulphate and gold is about the same.

This method with dry potassic iodide and a dry salt, I think I am safe in specifying microcosmic salt, has added certainly six and probably seven metals to the list of those that can be determined by a dry assay. I say probably seven, as I believe that practice in manipulation will bring out the result with tin, the only one I have failed with.

Thus a mineralogist fitted out with bottles of borax, carbonate of soda, microcosmic salt, and potassic iodide, with his lamp, blowpipe, a few plaster tablets, and possibly a few pieces of charcoal, can determine twenty-one metals, four non-metals; the silicates, nitrates, phosphates, chlorides and alkalies. The only two important substances not included are alumina and magnesia,

which require nitrate of cobalt. The test for alumina is hardly distinctive as a fusible silicate gives the same reaction; so that magnesia is the only one really omitted. The seven metals added by this method are tin, bismuth, gold, thallium, molybdenum, tungsten, and cadmium. Dry assay by either ordinary blowpipe methods or with this new iodide process will determine practically all ores of useful metals. One advantage of this new method is that sample tablets can be prepared and kept for comparison with results obtained from unknown specimens, as many of the colors are quite permanent. I have also tried barium nitrate with the dry potassic iodide, but it deflagrates too violently. I hope to be able to carry out some more extended experiments in this line and to communicate the results to this Institute if they are considered sufficiently valuable. In a later trial with tin and tin ore I believe I have succeeded in bringing out the proper reaction. As the color is somewhat the same as that of iodine alone on the tablet, I wish to compare my results with Dr. Haanel's before saying definitely that I have succeeded. Time did not permit of my doing this before reading this paper