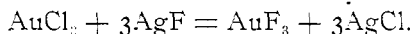
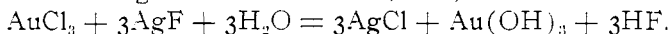


for the preparation of the fluoride would be that of double decomposition.

Silver fluoride and gold chloride, both being soluble salts, on being brought in contact in solution should yield theoretically:



The actual case is that when solutions of these two salts are brought in contact, gold hydroxide is quantitatively thrown out of solution along with silver chloride; thus,



The accuracy of this reaction has been carefully established in the laboratory.

If gold fluoride is even momentarily formed, it is immediately decomposed by water.

The method yet remaining for the preparation of a substance incapable of existence in presence of water would be the use of anhydrous solvents. A large number of organic solvents have been tried with this end in view, but no substance has been found which would dissolve both gold chloride and silver fluoride; either these salts are insoluble or are decomposed by the substances worked with. Among the solvents examined, mention may be made of the following: Alcohol, ether, carbon bisulphide, benzene, turpentine, pentane, hexane, chloroform, carbon tetrachloride, ethyl nitrate, nitrobenzene, ethyl acetate, ethyl propionate, and pyridine.

It thus appears that gold fluoride is incapable of existence not only in presence of water, but under the ordinary conditions met with in the laboratory and in nature.

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[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF CINCINNATI, NO. 60.]

### A METHOD FOR THE ESTIMATION OF CHLORIDES, BROMIDES AND IODIDES.

BY STANLEY BENEDICT AND J. F. SNELL.

Received September 3, 1903.

IN THE August number of this Journal we described a method for the detection of chlorides, bromides and iodides in presence of each other. The reagent used to liberate the iodine and bromine was potassium iodate, which sets free the iodine on acidifi-

cation with acetic and the bromine on acidification with dilute nitric acid. In the same paper we mentioned that the reaction with iodic acid had been employed by Bugarszky<sup>1</sup> for the quantitative separation of chlorides and bromides. He liberated the bromine with potassium biiodate and dilute sulphuric acid, and determined (1) the excess of biiodate and (2) the chlorine. This method is evidently inapplicable in presence of iodides. The purpose of the present paper is to describe a method of estimation of all three halogens when present together. It includes determinations of (1) total halogens, (2) iodine, and (3) chlorine, the bromine being estimated by difference.

The total halogens are determined by any of the ordinary methods, gravimetric or volumetric.

For the determination of the iodine, a suitable quantity of the substance (containing not over 0.5 gram iodine or 0.15 gram chlorine, if tenth-normal solutions are to be used) is dissolved in water and made up to about 50 cc. in a 100 cc. glass cylinder with close-fitting glass stopper. Neutral potassium iodate is added in about twice the quantity necessary to react with all the bromine and iodine believed to be present. The mixed solution is acidified with 4 or 5 cc. 5N (30 per cent.) acetic acid and shaken with 30-40 cc. carbon disulphide until all the liberated iodine has been taken up by the latter. The aqueous phase is now separated from the carbon disulphide phase by filtration through a wet filter, and the carbon disulphide is thoroughly washed with cold water on the filter. The filtrate and washings are reserved for the chlorine determination. The carbon disulphide solution is transferred to another beaker by puncturing the filter and is covered with 20-25 cc. 75 per cent. alcohol. Any carbon disulphide left adhering to the filter is rinsed down into the beaker with a portion of the 75 per cent. alcohol. The iodine is now titrated with sodium thio-sulphate with constant stirring. No starch indicator is necessary.

For the determination of the chlorine, the aqueous filtrate from the carbon disulphide is treated with 5 cc. 5N nitric acid (sp. gr. 1.18) to liberate the bromine and is boiled in a covered beaker until colorless. The excess of iodate is next destroyed by adding a quantity of potassium iodide slightly in excess of the amount necessary to react with it. The solution is again boiled until colorless, 2 or 3 cc.

<sup>1</sup> *Ztschr. anorg. Chem.*, 10, 387 (1895).

TABLE OF RESULTS.

	KI. Grams.	KBr. Gram.	NaCl. Gram.	KI. Grams.	KBr. Gram.	NaCl. Grams.	KI. Grams.	KBr. Gram.	NaCl. Gram.
Used.....	0.0798	0.0897	0.0893	0.0114	0.0640	0.0638	0.1140	0.1281	0.1277
Found .....	0.0796	(0.0896)	0.0896	0.0116	(0.0640)	0.0636	0.1138	(0.1285)	0.1275
Difference .....	-0.0002	....	+0.0003	+0.0002	....	-0.0002	-0.0002	....	-0.0002
“ per cent.	-0.25	....	+0.34	+1.75	....	-0.31	-0.18	....	0.16
Used.....	0.0456	0.0512	0.0510	0.1026	0.1152	0.1143	0.0228	0.2140	0.0255
Found .....	0.0457	(0.0511)	0.0510	0.1025	(0.1152)	0.1144	0.0230	(0.2141)	0.0252
Difference .....	+0.0001	....	0.0000	0.0001	....	+0.0001	+0.0002	....	0.0003
“ per cent.	+0.22	....	0.00	0.10	....	+0.09	+0.09	....	-0.12
Used.....	0.1710	0.0512	0.0127	0.0114	0.0128	0.0128	0.0114	0.0128	0.0127
Found .....	0.1704	(0.0507)	0.0128	0.0105	(0.0134)	0.0131	0.0116	(0.0125)	0.0128
Difference .....	-0.0006	....	+0.0001	-0.0009	....	+0.0003	+0.0002	....	+0.0001
“ per cent.	-0.35	....	+0.79	-7.89	....	+2.34	+1.75	....	+0.80
Used.....	0.0057	0.0064	0.0635	0.1368	0.0064	0.0063	0.1228	0.3815	0.1518
Found .....	0.0060	(0.0062)	0.0634	0.1369	(0.0066)	0.0060	0.1225	(0.3816)	0.1520
Difference .....	+0.0003	....	-0.0001	+0.0001	....	-0.0003	-0.0003	....	+0.0002
“ per cent.	+5.26	....	-0.16	+0.07	....	-4.76	-0.25	....	+0.13

more of the dilute nitric acid being added, if the color is not completely discharged after ten or fifteen minutes' boiling. A minute or two after the color has completely disappeared the solution is taken from the flame, cooled and neutralized with sodium carbonate. To secure exact neutralization, a little calcium carbonate may be added at first and then sodium carbonate solution until a precipitate just forms. The chlorine is then determined by titration with standard silver nitrate, using potassium chromate as indicator.

The results of twelve analyses of mixtures of potassium iodide, potassium bromide and sodium chloride are tabulated below. It will be observed that, with two exceptions, the differences between the taken and found quantities of potassium iodide and sodium chloride do not exceed 0.3 milligram.

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### ON CEROPTENE, A NEW ORGANIC COMPOUND.

BY W. C. BLASDALE.

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IN AN article published<sup>1</sup> in 1893 the writer briefly described an organic compound which had been obtained from the fronds of *Gymnogramme triangularis*, for which the name *ceroptene* was provisionally proposed. It was shown in that paper that this compound, together with others of unknown composition, was secreted by peculiar gland-tipped hairs, which were found in great abundance on the under surfaces of the fronds of the before-mentioned plant. The yellow secretion is produced in such abundance as to completely envelop and conceal the dorsal surfaces of the fully developed fronds, whence the popular name of the plant, the "golden-back" fern.

Secretions of a somewhat similar character are produced by other species of ferns, especially certain groups of species belonging to the genera *Gymnogramme*, *Cheilanthes* and *Notholoena*; possibly, also the secretions produced by certain species of *Primula* are of a like nature. Though none of these secretions appear to have been submitted to a chemical investigation, the opinion expressed or implied in most of the botanical treatises, which deal with these plants is that they consist of wax. Goep-

<sup>1</sup> *Erythea* (botanical journal published at Berkeley from 1893 to 1899), 1, 252.