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Ultramicrocolorimetric Argentimetry.

(Preliminary Report.)

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With 1 Figure.

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Chloride, bromide, iodide and sulphide in a phosphoric acid-ethyl acetate-chloroform solution react with the silver of the diphenylthiocarbazone-silver complex. The corresponding silver complexes are formed. Diphenylthiocarbazone is liberated and can be measured at 598 mμ. The reaction is extremely sensitive.

Reagents.

Ethyl acetate: 2 ml of 85% phosphoric acid were dissolved in 150 ml ethyl acetate.

Dithizonate: 10 mg of the yellow silver-diphenylthiocarbazone complex were dissolved in 100 ml chloroform. The solution was shaken several times with 0.1 N acetic acid.

Sodium chloride: 0.005 N solution in water.

Potassium bromide: 0.006 N solution in water.

Potassium iodide: 0.007 N solution in water.

Sodium sulphide: approximately 0.008 N solution in water. A crystal of sodium sulphide was dried with filter paper, weighed, dissolved in boiled-out water and diluted to volume.

Method.

Into 10-ml measuring flasks were pipetted 0.5, 10, 20 μl of the test solutions. 5 ml of ethyl acetate reagent were added followed by 2 ml dithizonate. After thorough shaking the volume was made up to the mark with ethyl acetate, and the colours were read at 598 mμ in a Beckman

model B spectrophotometer using 1-cm cuvettes and water as blank solution. The results are given in Fig. 1. The experiment with sulphide has to be considered as a qualitative test rather than a real deter-

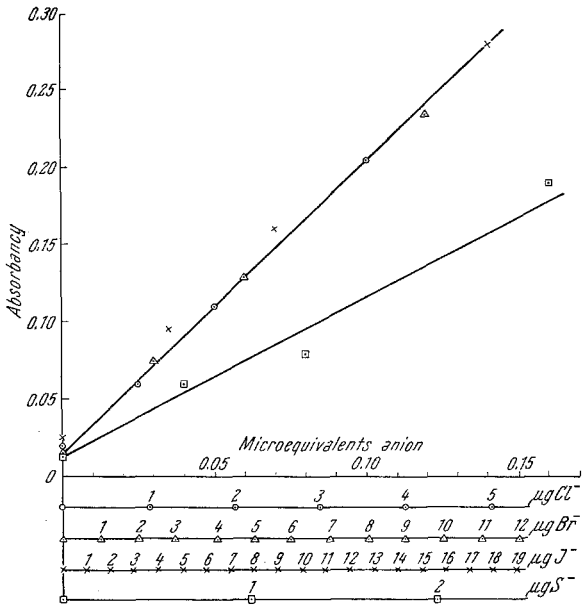

Fig. 1. Standard curves obtained with test solutions.

mination. The small blank is probably caused by impurities in the reagents. The silver dithizone complex has no absorption in this range. A more detailed paper will be published after a further investigation of the method.