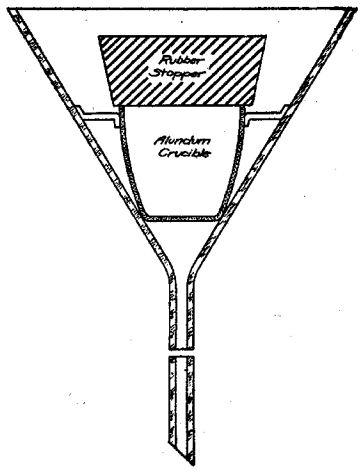
ble in the usual way. When all the soluble material had been removed, except the small amount in the upper rim, the crucible was filled about half full of water,



suction was applied and a moistened rubber stopper was quickly pressed down on top of the crucible. (The rubber stopper was always polished to a plane surface and washed free from any dust before using the first time.) As soon as the stopper was firmly held by the suction, water was poured on top of the holder (see figure) and around the stopper. The water drawn through the top rim easily completed the washing, as was indicated by the fact that no test for alkalinity could be obtained by addition of a drop of phenolphthalein to the edge of the crucible.

Continuous use of the crucible holder, since it was not designed for treatment in this manner, tended to crack the flange from the outer portion of the holder. A slight modification of the flange construction would perhaps lessen this difficulty.

RAPID DETERMINATION OF SMALL AMOUNTS OF COPPER BY THE IODIDE METHOD

By H. F. Bradley

PARK CITY, UTAH

Received April 5, 1920

The precipitate of cupric sulfide (containing not over 0.05 g. copper) is washed free from chlorides (complete removal of iron not being necessary), then moistened with a few drops of strong neutral zinc nitrate solution (free from chlorides), and ignited.

The residue of zinc oxide and cupric oxide is quickly dissolved by warming with 1 cc. of 1 : 2 hydrochloric acid. Neutralize with 5 per cent potassium hydroxide, acidify with acetic acid, add a little phosphate solution to prevent the action of iron,[1] then add 2.5 g. solid sodium iodide and titrate as usual.

SOME AMERICAN-MADE CHEMICAL REAGENTS

By W. D. Collins

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Received March 31, 1920

I—POTASSIUM FERRICYANIDE

A member of the AMERICAN CHEMICAL SOCIETY informed the Committee on Guaranteed Reagents that he had found potassium ferricyanide purchased a year or two ago wholly unsuited for use in making the gelatin test on tin plate. The salt made a turbid green solution.

Examination of two lots which had been in the Bureau of Chemistry for several years showed that they were of the same quality, but a single recrystallization from a filtered solution gave a perfectly satisfactory product.

Correspondence with manufacturers and dealers secured some analytical reports which indicated material of high purity. A sample furnished as representing the product now sold by one manufacturer made a perfectly clear 10 per cent solution with no traces of green color and no precipitate.

It therefore appears that no one need now accept potassium ferricyanide which will not make a 10 per cent solution without any precipitate or green color. Purification of poor material in stock is worth while in most cases.

II—METHYL ORANGE[1]

Similar complaint was made to the Committee of difficulty in procuring satisfactory methyl orange from American manufacturers. At the suggestion of the Committee, samples were purchased from four different sources. There was at hand for comparison one of a well-known foreign make. A sixth sample was obtained from a lot of methyl orange which was exhibited by a manufacturer at a meeting.

A few grams of the best appearing sample (Sample A) were repurified by precipitating the free acid, filtering, dissolving with sodium carbonate solution, and recrystallizing twice. This produced shining crystal leaflets of a beautiful orange-red color which were provisionally adopted as a standard.

The samples were judged by general appearance, odor, water-insoluble residue from one gram in 400 cc. of water, and indicator value. The results are given in the accompanying table.

COMPARISON OF SAMPLES OF METHYL ORANGE

SAMPLE	Appearance	Odor	Water-insol. Residue from 1 g.	Indicator Value	Notes
Standard	Orange-red crystals	None	0.0000	Good	
A	Orange-red crystals	Very faint	0.0008	Good	
B	Yellow-red powder	None	0.0294	Good	Foreign-made
C	Yellow-brown, lumpy[1]	Decided	0.0105	Good	
D	Brown, lumpy	Decided	0.0537	Fair	Dyestuff
E	Brown powder	Strong	0.1167	Good	Naphthol-like odor
F	Yellow-red powder	None	Slight	Good	

[1] A later sample from the manufacturer of C compared favorably with B or F.

The odor resembled aniline in three cases, and one sample smelled strongly like naphthol.

The two brown samples were not very pleasing in appearance. The manufacturer of one of these products stated in reply to criticism of his output that it was not intended for an indicator but was sold for use as a dyestuff.

A portion of Sample D was purified both by direct recrystallization and by precipitating the acid and subsequently neutralizing the free acid with soda. In each case a bronze-yellow crystalline powder was recovered which, in appearance, did not resemble the standard methyl orange. When used as an indicator its alkaline solution was bright yellow in color, but the acid solution was only pale red. Sample D did not give a clear solution when filtered.

Notwithstanding the poor quality of some of the samples obtained, the results show that it is entirely

[1] R. H. Deakin, *Chem. News*, **118**, 193.

[1] In collaboration with G. C. Spencer.