[CONTRIBUTION FROM THE CHEMICAL ENGINEERING DEPARTMENT, THE OHIO STATE UNIVERSITY]

ZIRCONIUM. VI. USE OF DISPERSOIDS IN THE DETECTION OF TRACES OF POTASSIUM BY ZIRCONIUM SULFATE

BY RUFUS D. REED AND JAMES R. WITHROW

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The results of an investigation of the effect of the addition of a dispersoid of potassium zirconium sulfate in shortening the time required for a test for potassium by zirconium sulfate indicated that the time factor is materially reduced by adding the dispersoid. The sensitivity of zirconium sulfate for potassium is slightly greater in the presence of a dispersoid than in its absence.

The tardy formation of crystals in supersaturated solutions is commonly ascribed to lack of suitable nuclei. A frequently used method of overcoming such supersaturation has been to add a crystal of the desired substance. Where traces of constituents are sought, however, the addition of such a crystal would introduce a comparatively large amount of the product, hindering the detection of any minute quantities; hence the method is of little value in analytical chemistry. It would appear feasible that any supersaturation tendency in testing for traces of substances could be eradicated by introducing into the solution tested a drop of a liquid containing a very minute amount of the precipitate expected, in dispersoid state. If the solution tested contained no appreciable amounts of the substance to form that added in dispersoid form, then the dispersoid should not cause an increasing turbidity in the solution; but if there is any of the substance present, the turbidity should increase due to the nuclei growing and multiplying in number or size. To avoid adding appreciable quantities of solute, we have for some time in this Laboratory, added a controlled trace of the precipitate with the reagent, as has doubtless been done by others. We now find as far as information is available that Forbes[1] as early as 1911 in determining the solubility of silver chloride in chloride solutions overcame the supersaturation tendency "by adding, on the end of a stirring rod, quantities of precipitated silver chloride so minute as to produce no visible turbidity in 50 cc. of pure water." The method does not seem to have come into general use. This paper reports the application of the procedure or principle to the simple case of the detection of potassium by zirconium sulfate.

Experimental

Materials.—The zirconium sulfate solution (containing 11.31% of zirconium sulfate) and the potassium sulfate solutions used were those described in our previous work.[2]

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[1] Forbes, THIS JOURNAL, **33**, 1938 (1911).

[2] Reed and Withrow, *ibid.*, **50**, 2986 (1928).

Dispersoid.—The dispersoid of potassium zirconium sulfate was obtained from a mixture of dilute potassium sulfate solution and the zirconium sulfate reagent which had reacted to form a precipitate. Knowing the concentration of potassium in the original mixture of 2 cc. to be 2.4 mg. of potassium and assuming 30 drops to a cubic centimeter of solution, one drop of this mixture would add not over 0.04 mg. of potassium to the solutions tested.

Procedure.—The procedure was the same as that given under the general procedure in the previous work,[3] except that after the reaction mixture had stood for an hour at room temperature, a drop of a solution containing a suspension of potassium zirconium sulfate was added. After the completion of the test for potassium by zirconium sulfate and the dispersoid mixture, the solutions were filtered and the filtrates showed the presence of potassium when tested by sodium cobaltic nitrite after first throwing the zirconium into a complex ion with tartaric acid and sodium hydroxide according to the method previously reported.[4] The results appear in the table.

TABLE I

SUMMARY OF EFFECT OF DISPERSOID ON DETECTION OF POTASSIUM WITH ZIRCONIUM SULFATE

Expt.	K per sample, g.	Results with dispersoid added	Results with no dispersoid added
1	0.00119	Opal. 1 hr. r. temp.; small ppt. 0.5 hr. 0°	Opal. 1 hr. r. temp.; cloudy 0.5 hr. 0°
2	.00095	Opal. 1 hr. r. temp.; cloudy 0.5 hr. 0°; ppt. 1 hr. 0°	Opal. 1 hr. r. temp.; cloudy 0.5 hr. 0°; ppt. 1.25 hr. 0°
3	.00078	Cloudy 0.5 hr. 0°; small ppt. 2 hr. 0°	Opal. 0.5 hr. 0°; ppt. 3 hr. 0°
4	.00048	Opal. 0.5 hr. 0°; ppt. 3 hr. 0°	Very sl. cloudy 1.5 hr. 0° cloudy 5 hr. 0°
5	.00032	Slightly opal. 0.5 hr. 0°; ppt. 3 hr. 0°	Clear 5 hr. 0°
6	.00000	Slightly opal. 0.5 hr. 0°; unchanged 5 hr. 0°	Clear 5 hr. 0°

Total volume, 2 cc.; zirconium sulfate present, 0.1131 g.

Discussion.—These results were typical of about 80 experiments. Comparing the sensitivity with and without the dispersoid indicated that the addition of the dispersoid increased the sensitivity of zirconium sulfate for potassium from 0.48 mg. of potassium (0.05% elemental potassium or 0.006 molal potassium sulfate) to 0.32 mg. (0.03% elemental potassium or 0.004 molal potassium sulfate), Expts. 4 and 5.

Comparing the time needed for securing the test with a dispersoid present and without a dispersoid showed that the presence of the dispersoid

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[3] Reed and Withrow, THIS JOURNAL, 50, 1515 (1928).

[4] Reed and Withrow, *ibid.*, 51, 3238 (1929).

shortened the time in Expt. 2 with 0.95 mg. of potassium by 15 minutes, and in Expt. 4 with 0.48 mg. of potassium, two hours were saved.

In Expts. 4 and 5 containing 0.48 mg. and 0.3 mg. of potassium, respectively, the test for potassium in the filtrate (not removed by zirconium sulfate) was very faint and appeared only after allowing the test to stand for forty minutes.

Summary and Conclusions

1. "Seeding" by adding a solute dispersoid furnishes suitable nuclei for initiation of visible precipitation without introducing appreciable quantities of the material to be detected.

2. The addition of a drop of a dispersoid of the desired product hastens the detection of potassium by zirconium sulfate.

3. The sensitivity of zirconium sulfate reagent for potassium was increased through the presence of the dispersoid from 0.48 mg. of potassium to 0.32 mg. per 2 cc. of reaction mixture.

4. The zirconium sulfate reagent did not completely remove potassium from solution.

Columbus, Ohio

THE SORPTION OF ORGANIC VAPORS BY HIGHLY EVACUATED, ACTIVATED SUGAR CHARCOAL[1]

By James W. McBain, H. P. Lucas and P. F. Chapman

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It is of the greatest interest to determine in how far sorption data for a given specimen of charcoal may be made reproducible through improvements in the preliminary treatment, and then to ascertain in how far differently activated specimens of the same charcoal yield sorption data conforming to a definite type. In previous communications,[2] it has been increasingly shown that improvements in the preliminary evacuation of a given specimen of charcoal greatly affect not merely the position, but also the form of the sorption curve. This becomes especially noticeable as the cleansing of the charcoal through evacuation is made very thorough. It is found advisable to evacuate for a long period at as high a temperature as possible in a high vacuum, then to displace some of the impurities still sorbed on the charcoal by admitting to the charcoal some of the liquid to be investigated and repeating the thorough evacuation before commencing the experiment.

[1] These experiments were carried out at the University of Bristol, England, in 1925–1927 inclusive.

[2] A. M. Bakr and J. E. King, *J. Chem. Soc.*, **119**, 454 (1921); A. M. Bakr and J. W. McBain, This Journal, **46**, 2718 (1924); J. W. McBain, *Nature*, **117**, 550 (1926); J. W. McBain and G. T. Britton, This Journal, **52**, 2198 (1930); J. W. McBain, D. N. Jackman, A. M. Bakr and H. G. Smith, *J. Phys. Chem.*, **34** (1930).