

From a brief mathematical consideration of the results obtained, the conclusion has been drawn that further progress in this field must be preceded by fundamental advances on the theoretical side of the subject. The experimental work has, at present, reached as far as it can go alone in showing that all simple solutions of the problem hitherto proposed are certainly insufficient.

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[CONTRIBUTION FROM THE DENVER FOOD AND DRUG INSPECTION STATION, BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

A COLOR TEST FOR OXALIC ACID.¹

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Recently Sacher² suggested a color test involving the use of a dilute solution of a manganous salt. This is treated with sodium or potassium hydroxide solution and allowed to stand till the manganous hydroxide is partially oxidized by the air. On adding an aqueous solution of oxalic acid, drop by drop, the precipitate dissolves and a red coloration is produced. Reducing substances, also excess of oxalic or any other acid, prevent the color formation.

Dorronsoro and Fernandez³ have observed a color reaction while working with pinene derived from Spanish oil of turpentine. When pinene was oxidized, a substance, supposed to be nopic acid, was produced which gave a *green* coloration with resorcinol and sulfuric acid. This substance was later identified as oxalic acid.⁴

The writer has confirmed this reaction with oxalic acid and has further developed it for use as a general test for oxalic acid. Under certain conditions described below, a *blue* color is obtained instead of green.

While resorcinol forms colorations with a great number of organic compounds, the colors are mostly red, pink or violet. When nitrates or nitrites are dissolved in conc. sulfuric acid and a crystal of resorcinol is added, a deep blue color is produced. If a little water is added, however, the color becomes brown to red. The writer is unaware of any other compound which gives a *blue* color *under the conditions described below*, although it is quite probable that such compounds exist. Some substances, such as carotin, xanthophyll, some terpenes, etc., give a *blue* color with sulfuric acid alone. It is best, therefore, in many cases to re-

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² Sacher, *C. A.*, **9**, 2043 (1915).

³ Dorronsoro and Fernandez, *Ibid.*, **9**, 2522 (1915).

⁴ Details of this reaction are not given in the abstract. The original article (*Anales. soc. españ. fis. quim.*, **II**, 441-3 (1913)) was not available.

move the oxalic acid by precipitation as the calcium salt and to apply the test to the precipitate.

No *blue* color was obtained with some of the commoner natural acids, such as formic, acetic, lactic, tartaric, citric, malic, succinic and benzoic acids, neither did these acids interfere with the test for oxalic acid. Although tartaric acid gives a brilliant red color with resorcinol and sulfuric acid, it did not interfere with the *blue* oxalic color (until heated) in a mixture of 10 mg. of oxalic acid, 100 mg. of tartaric acid and 2 cc. of water.

Color Test for Oxalic Acid.—A few crystals of resorcinol are added to about 5 cc. of the unknown solution in a test-tube, and the mixture is warmed slightly to dissolve the resorcinol. It is then cooled and 5 cc. of conc. sulfuric acid is carefully and slowly poured in along the side of the tube so as to form a layer. A blue ring will be formed at the junction of the 2 layers, if oxalic acid is present. The color is best seen if held to the light in front of a sheet of white paper. Care must be taken that the mixture does not warm up appreciably. If the blue color does not appear in a few minutes, the mixture is shaken thoroughly, and, after cooling somewhat, 5 cc. more of sulfuric acid is added. Should the color still fail to appear, the mixed contents of the tube should be gently warmed over a flame (not boiled) when an indigo blue color will diffuse throughout the liquid. If the mixture be cooled with ice-water, the color will disappear only to reappear again on heating. If the mixture be boiled a few minutes, the color will turn a deep dark *green*, which will become a light yellow-green on cooling. If to the cold yellow-green solution an equal volume of sulfuric acid be added so as to form 2 layers, the *blue* color will again appear. It is believed that all these reactions taken together are characteristic of oxalic acid alone.

This test may be made sensitive to one milligram if the dry unknown substance be warmed with 2 drops of a 10% aqueous resorcinol solution and the sulfuric acid added drop by drop. The blue color then appears immediately. For very dilute solutions of oxalic acid or its salts, it is best to evaporate to a concentration of about 10%.

If interfering substances are present the oxalic acid may be precipitated in ammoniacal solution as the calcium salt, washed with water, and the test applied directly to an aqueous suspension of the salt.