A Simple Colorimetric Test for Available Iron in Alkaline Soils

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Thioglycolic acid (SHCH₂COOH) neutralized with calcium carbonate is suggested as a reagent for the detection and estimation of available iron in neutral or alkaline soils. Its potentialities should be of interest to those research personnel conducting studies on the problems of iron availability, of phosphate fixation and of soil genesis.

MATERIALS AND EQUIPMENT

Few chemicals and few pieces of laboratory equipment are necessary for conducting the test. Three solutions are needed: (1)* thioglycolic acid diluted 1–50 with distilled water and freshly neutralized by the addition of an excess of calcium carbonate, (2) thioglycolic acid diluted 1–10 with distilled water but not neutralized, and (3) concentrated ammonium hydroxide. In addition to miscellaneous glassware the procedure requires the use of a soil grinder (or mortar and pestle), a laboratory balance, an accurate timing device, a suction filter setup, and a photoelectric colorimeter provided with a green (540 mμ) filter. Before any tests are run it would be well, of course, to prepare a calibrated curve, using a standard iron solution, for translating colorimetric readings obtained into measurements of iron concentration.

PROCEDURE

The following procedure is recommended tentatively. Thoroughly mix an air-dried soil sample, remove a small portion, and pulverize or grind it to destroy particle aggregation, then weigh out a 1-gram portion. Place this in a test tube or other suitable container. Add quickly 10 ml of 1–50 calcium thioglycolate solution (as from a small graduate) and stopper the test tube. Shake the tube and its contents vigorously for 2 or 3 seconds and then gently until 1 minute has elapsed from the instant of adding the reagent. Immediately filter off the resulting solution, utilizing moderate suction, so as to stop further extraction of the iron in the sample. Pipette a 5 ml aliquot of the filtrate into a 50 ml graduated cylinder. Add 1 ml of 1–10 thioglycolic acid, then 10 to 20 ml of distilled water. Next add 5 or 6 drops of concentrated ammonium hydroxide. Make up to volume with distilled water then shake the 50 ml of solution vigorously for 2 or 3 seconds in order to mix it thoroughly and to develop completely the purple-red color of the complex. Finally, using the colorimeter and a suitable absorption cell, obtain a reading of the color density of the red ferrous complex which represents the iron removed from the 1 gram sample of soil. Distilled water should be found satisfactory for use as the blank.

If the reading obtained is converted to parts per million and that figure then divided by 10, the result will represent grams of iron removed from the gram of soil in a 1-minute extraction period and will be a convenient measure of the availability (as herein defined) of the iron in the sample.

NATURE OF THE REAGENT

Thioglycolic acid will react with most insoluble iron compounds, will react readily with phosphate, fresh ferric hydroxide, etc., and slowly attack such relatively stable and insoluble compounds as haematite, limonite, and many salts such as ammonium, potassium, or calcium thioglycolate likewise will attack iron in its various forms and will do so in a neutral or alkaline medium. Thioglycolate, for instance, will function readily in the presence of free calcium carbonate. Indeed, few if any compounds commonly present in soils are likely to cause serious interference.

If a solution containing the resulting iron thioglycolate compound or complex is or is made weakly acid, it will be colorless. If it is or is made alkaline (pH 7.5 or over) a reddish-purple color will appear, although for awhile the color can be restored temporarily by shaking the solution in air.

The red color complex follows Beer's law and supposedly is adaptable for the detection and estimation of as one part iron in 10,000,000 parts of solution. It is insensitive to either excess of the reagent or to a surplus of the reagent is present. However, if the iron line or is made alkaline and little or no free hydroxide present, the color will develop and then will fade (often with the formation of ferric hydroxide) or will not develop at all—a gray-green precipitate of ferrous hydroxide (?) being formed instead. If a surplus of reagent is present the red color compound is stable for several minutes at least, perhaps for several hours. Nevertheless, on standing it eventually will fade, although for awhile the color can be restored temporarily by shaking the solution in air.

The red color complex follows Beer's law and is supposedly adaptable for the detection and estimation of as one part iron in 10,000,000 parts of solution. It is insensitive to either an excess of the reagent or to a wide variation in pH ranging from about 7 to 8. Furthermore it can be diluted within wide limits without affecting the relation of concentration to color intensity, a particularly useful fact for instances where the color density may be too great for an accurate reading on the colorimeter.

* Contribution from the Department of Agronomy, Florida Agricultural Experiment Station, Gainesville, Fla. Published with the approval of the Director of the Station. Certain preliminary studies relevant to this work were made by the author while associated with the Colo. Agr. Exp. Sta., 1940–43.

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