A Test for Chlorate Residues

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Modern farming methods have brought into play the use of defoliants to knock the leaves off of plants before the use of harvesting machines to harvest the crops produced by the plants involved. The advent of the cotton-picking machine in particular has brought on a large increase in the use of defoliants. One of the most popular groups of these is the chlorate defoliants. These are sprayed on the cotton plants just before harvest in order that the leaves will fall off of the plants and make it easier for the machines to pick the cotton and produce a cleaner product free of chaff from cotton leaves and stems.

This laboratory has often been asked to test for spray drift of this type of defoliant on crops and adjacent fields when damage became apparent on the adjoining plantings.

The writer recalled using available chlorine as the oxidant in wet-ashing animal muscular tissue to test for thallium. The available chlorine was produced by adding $KClO_3$ and concentrated Hydrochloric Acid to the ground muscle tissue and heating in a porcelain casserole on the water bath. Available chlorine is produced according to the following reaction:

Bulletin of Environmental Contamination & Toxicology,
Vol. 2, No. 4, 1967, published by Springer-Verlag New York Inc.

$$KClO_3 + 6HCl \text{ — } KCl + 3H_2O + 3Cl_2$$

By combining this reaction with the ortho-Tolidine test for available chlorine in swimming pool or potable water a sensitive test was evolved.

Procedure:

Standard Solutions

1. Dissolve 1.151 grams of C. P. $KClO_3$ in 1 liter of distilled water. 1 ml will contain 1 mg. of $NaClO_3$ equivalent.

2. Dilute 100 ml of (1) to 1 liter with distilled water. 1 ml will contain 100 micrograms of $NaClO_3$ equivalent.

3. Dilute 100 ml of (2) to 1 liter with distilled water. 1 ml will contain 10 micrograms of $NaClO_3$ equivalent.

4. ortho-Tolidine reagent: Dissolve 100 mg of ortho-tolidine in 100 ml of 1 + 9 Hydrochloric Acid. Shake if necessary to effect complete solution. Filter through a Whatman #1 filter paper if necessary to remove undissolved particles.

Color standard development:

Add 2 ml of ortho-Tolidine reagent to each of six glass-stoppered 50 ml graduated cylinders. Add zero, 10, 20, 30, 40 and 50 micrograms of the 10 micrograms per ml chlorate standard to these cylinders and make up to the 25 ml mark with distilled water. Make to 50 ml mark with concentrated Hydrochloric Acid and mix by inverting several times. Color development is immediate and is read in a 1 cm cell in a spectrophotometer at 446 millimicrons. This laboratory has used a photo-electric colori-

meter with a Wratten #42 (blue) filter. This permitted use of a 4 cm cell which permitted reading of smaller quantities due to the longer cell path.

Note: For proper color development, it is important that the ortho-Tolidine reagent be added first to the cylinder in which the color development is to take place.

Preparation of Sample:

Cut alfalfa or Milo blades or vegetable leaves to be tested into about one-inch pieces and weigh, packing loosely, into a covered quart jar. Add distilled water to the jar, the amount depending on the nature and looseness of the sample. Roll for one-half hour on a jar-tumbling machine. Filter some of the liquid through a Whatman #1 filter paper and place not over 23 ml into each of two glass-stoppered 50 ml calibrated cylinders. Add 2 ml of ortho-Tolidine reagent to one of the cylinders and (if less than 23 ml of sample were used) make both cylinders up to 25 ml mark with distilled water. Make up to 50 ml with concentrated Hydrochloric Acid. The cylinder without the ortho-Tolidine reagent may be used for zeroing the instrument in case there was some color extracted from the material being tested.

This method has recently been used to indicate 10 parts per million of chlorate expressed as magnesium chlorate on milo foliage.

We recall a case which involved cattle deaths. The cattle had eaten foliage along a fence line which had been sprayed with a chlorate-type weed killer. The laboratory found 175 parts per

million of chlorates in a sample of stomach contents fluid which had been submitted.

Since this is a test for the chlorate ion only, the following factors may prove useful:

$KClO_3$ x .8654 = $NaClO_3$ equivalent

$KClO_3$ x .7802 = $Mg(ClO_3)2$ equivalent