until reaction is complete. Ash overnight at 450° C. in a muffle furnace. Dissolve the ash in 6 ml. of concentrated hydrochloric acid and sufficient water to give a final volume of 25 ml. Filter.

**Analytical Procedure.** Make analyses in duplicate.

Place a 3-ml. aliquot of sample or standard solution in each test tube, add 2 ml. of dipropylene glycol reagent, and mix thoroughly, then, at once, add 5 ml. of barium chloride reagent and again mix thoroughly.

Let mixture stand for 15 minutes to 1 hour and read transmittance at 720 mμ, using reagent blank as reference sample. Determine concentration of sulfur from standard curve.

If a smaller aliquot is used, enough zero sulfur standard should be added to make a volume with the aliquot of 3 ml.

Reading the transmittance at about 410 mμ increases sensitivity about two- or threefold.

### Discussion

**Comparison with Gravimetric Method.** Results obtained by the proposed method are in good agreement with those obtained by the official AOAC method (*3*), as shown in Table I.

Analysis of variance showed no significant differences due to method of chemical analysis. There was an interaction, barely significant at the 5% level, between sulfur level in the sample and the method of analysis. The proposed method gave slightly higher values than the official method on samples containing about 0.2% sulfur and slightly lower values on samples containing 0.4% sulfur. The method gave equally good results with oily material of the seed and the nonoily root material.

Use of Alcoholic Magnesium Nitrate Solution. Alcoholic magnesium nitrate was used instead of the aqueous solution described in the official AOAC method to give better penetration of oily materials such as soybean meal.

**Effect of Added Salts.** The presence of magnesium and chloride ions at the high levels contained in the ash solution increases the absorbance by a factor of about 1.8 following addition of barium to a sulfate solution. It was therefore necessary to include magnesium chloride reagent in the standard solutions.

**Table I. Results Obtained with Proposed Method and with AOAC Method**

| Sample No. | Material | Sulfur, % Proposed Method | | Official Method | | Mean Difference |
|---|---|---|---|---|---|---|
| 1 | Seed | 0.20 | 0.20 | 0.21 | 0.22 | −0.015 |
| 2 | Seed | 0.21 | 0.22 | 0.20 | 0.19 | +0.02 |
| 3 | Seed | 0.23 | 0.23 | 0.21 | 0.22 | +0.015 |
| 4 | Seed | 0.28 | 0.27 | 0.28 | 0.28 | −0.005 |
| 5 | Seed | 0.27 | 0.28 | 0.28 | 0.28 | −0.005 |
| 6 | Roots | 0.30 | 0.30 | 0.30 | 0.32 | −0.01 |
| 7 | Roots | 0.39 | 0.38 | 0.40 | 0.41 | −0.02 |
| 8 | Roots | 0.39 | 0.38 | 0.38 | 0.38 | +0.005 |

The increase in turbidity with the addition of magnesium chloride reagent was not due to traces of sulfate, as the same amount of reagent was added to each standard, nor was it due specifically to either magnesium ions, chloride ions, or pH as the addition of sodium chloride, hydrochloric acid, or nitric acid had similar effects. In agreement with Toennies and Bakay (*4*), the effect of added salts was found to be small at low sulfur concentrations.

The amount of salts in the ash solution originating from the sample itself was small compared to that which was added. Variations of salt concentration of the magnitude owing to differences in ash content of the samples were found to have no effect on turbidity. The addition of phosphate to the standards to give concentrations higher than would be found in the ash solutions had no effect.

In the presence of only moderate concentrations of salts it is possible to determine sulfate volumetrically. Asghar, Quayyum, and Rana (*1*) have recently published a rapid method in which sulfate is precipitated as barium sulfate and the excess barium is titrated with the disodium salt of (ethylenedinitrilo)-tetraacetic acid. However, the high concentration of magnesium necessary to prevent the loss of sulfur during ashing of plant materials would result in too high a blank for good accuracy with this method.

**Spectrophotometry.** Reading transmittance of 720 mμ gave satisfactory sensitivity over a wide range of sulfur concentrations, so that it was seldom necessary to change aliquot size. Greater sensitivity is possible at 410 mμ, but aliquot size must then be varied according to the expected percentage of sulfur. However, if the sample contains less than about 200 γ of sulfur, the greater sensitivity at the lower wave length is worth while.

Commercially available reagents were used except in the case of magnesium nitrate and no difficulty was experienced due to the trace of sulfur which the reagents may have contained. Deionized water prepared by passing distilled water through a column of Amberlite Monobed No. 3 resin proved very satisfactory.

### Literature Cited


(1) Asghar, A. G., Quayyum, M. A., Rana, G. M., *Soil Sci.* **83,** 239–41 (1957).
(2) Assoc. Offic. Agr. Chemists, Washington, D. C., "Official Methods of Analysis," 7th ed., p. 8, 1950.
(3) *Ibid.*, p. 104.
(4) Toennies, G., Bakay, B., *Anal. Chem.* **25,** 160–5 (1953).



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## Endrin Content of Milk and Body Tissues of Dairy Cows Receiving Endrin Daily in Their Diet—Correction

On page 520 [Kiigemagi, Ulo, Sprowls, R. G., Terriere, L. C., J. AGR. FOOD CHEM. **6,** 518 (1958)], in Table V, the heading of the fourth column should read "Ratio, Output/Intake."

## Enzymic Hydrolysis of Naringin in Grapefruit—Correction

On page 548 [J. AGR. FOOD CHEM. **6,** 546 (1958)], in the legend of Figure 5, the broken line should be the hydrolyzed sample and the solid line the original sample.

S. V. TING

**INSECT ATTRACTANTS**

# Esters of 6-Methyl-3-cyclohexene-1-carboxylic Acid as Attractants for the Mediterranean Fruit Fly


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ATTRACTANTS are useful in the control and eradication of insect pests. In combination with a toxicant, they may lure insects to their doom, or traps may be baited with them to determine the location and extent of insect infestation (*1*), so that control measures may be applied only when and where needed. Attractants are particularly helpful in detecting