A New Color Reaction of Sesame Oil

COLOR REACTIONS owing to characteristic compounds of sesame oil (*Sesamum indicum*) are extensively reviewed by Budowski and Markley (1) and by Budowski (2).

We have found an altogether new reaction which gives a characteristic color with sesame oil. When trichloroacetic acid is added to sesame oil, an initial blue color, which turns to green after a few hours, is noticed. The test is performed as follows. To 1 ml of 50% solution (v/v) of sesame oil in petroleum ether, 2 ml of chloroform solution of trichloroacetic acid (40% concentration, w/v) are added. After a few seconds, a faint blue color develops. The color increases in intensity and first becomes sky blue, then green after a few hours. Oils from brown as well as white seed give this test. Alkali refining and adsorption bleaching with earth and carbon accentuate the intensity of the response.

The reaction appears to be of the redox type. It takes place in the presence of peroxides or persalts. No blue color develops when hydroquinone is first added to the oil. If hydroquinone is added after the color has developed, the color is immediately discharged; upon addition of excess benzoyl or other peroxide, the blue color is revived.

The characteristic blue color with trichloroacetic acid may be attributed to the presence of sesamol and related compounds. Sesame oils which have been chromatographed on a column of alumina (eluted with n-hexane and with methanol:ether 975:25) do not respond to the test. Crude sesamol (Shulton Inc., Clifton, N.J.) added to petroleum ether along with a peroxide gives a blue color which later turns to green. Similarly sesamin, isolated from sesame oil by molecular distillation and low-temperature crystallization, also responds, although to a lesser extent. Sesamol, sesamin, and sesame oil are sensitive to the extent of 0.01, 0.2, and 20.0% by weight respectively in petroleum ether toward this test. For example, 0.0001 g of sesamol, 0.002 g of sesamin, and 0.2 g of sesame oil, added separately in 1 ml of petroleum ether, respond to the test.

Sesamol and sesamin dissolved in paraffin oil do not give a blue color with the trichloroacetic acid reagent alone. However, if benzoyl peroxide is added, a blue color appears immediately. Sesame oil, which invariably contains some peroxide because of autoxidation, responds positively when dissolved in paraffin oil. Piperonal does not give the test; but if piperonal is oxidized with peracetic acid, according to the Böeseken, Coden, and Kip method (3), the resulting sesamol acetate gives the blue color with trichloroacetic acid in the presence of benzoyl peroxide.

From these observations it may be postulated that the methylenedioxyphenoxy group characteristic of compounds which are present in sesame oil and trichloroacetic acid take part in a reaction in which peroxides might be involved through a free radical mechanism that results in the blue color.

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Gas Flowmeter from a Leak Detector and Potentiometer

CARRIER-GAS FLOW from a flame ionization detector (FID) is often troublesome to measure because the burner housings must be opened or partially dismantled. A Gow-Mac Leak Detector (GM-LD), formerly Gas Hound, can be operated as a specialized flowmeter that particularly applies to this measurement problem.

A 50K ten-turn potentiometer with locking duodial when wired in the meter line of the GM-LD allows its sensitivity to be attenuated so that, on the "low" range, large amounts of helium carrier-gas may be monitored against a background of air from the FID. Usually the measurement is made with no hydrogen flowing through the burners. However if the flames are adjusted to combust the hydrogen stoichiometrically, helium measurements may be made while the burners are operating. Results are best without hydrogen since no water vapor will be introduced into the GM-LD.

A typical GM-LD/flowmeter calibration procedure is as follows:

1) Hydrogen and helium to detector temporarily shut off.

2) Airstream through burner housing adjusted to 200 cc/min with the aid of a soap-film flowmeter.

3) Helium carrier turned on and adjusted to 70 cc/min (soap-film meter).

4) GM-LD used to monitor FID effluent from any convenient opening in the burner housings while 50K potentiometer is adjusted so that 70 cc/min helium in 200 cc/min air causes full-scale meter deflection. The potentiometer adjustment is locked and recorded so that it may be returned to this value without recalibration. Measurements should be made at the same opening in the FID housing so that readings are not influenced by variations in probe location.

5) Helium flow reduced, measured with the soap-film meter, and recorded;

6) GM-LD meter deflection at this lower helium flow recorded; and

7) Repeat steps 5 and 6.