PESTICIDE RESIDUES

Modified Schöniger Combustion for Determination of Residues of Arsenic, Bromide, Chloride, Manganese, and Nickel in Pesticide-Treated Plant Material

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Use of the 5-liter modified Schöniger combustion flask has been extended to the combustion of potatoes, cherries, onions, cabbage, and oats for the determination of residues of arsenic, bromide, chloride, manganese, and nickel.

THE COMBUSTION of plant material has been carried out in a 5-liter flask (*5*) prior to the determination of residues of arsenic, bromide, chloride, manganese, and nickel.

Arsenic may be determined in potato following sodium arsenite herbicide applications by flask combustion of 10 grams of oven-dried potato tissue and absorption of arsenic in 100 ml. of 1*N* hydrochloric acid. After the flask has been rinsed twice with 25 ml. of acid, arsenic is determined (*6*) in 50 ml. of the absorbing solution; acid is omitted from the molybdate solution and the concentration of dilute stannous chloride is doubled. Recoveries of 2.5 and 5 p.p.m. of arsenic as sodium acid arsenate ranged from 81.6 to 103.2%.

Inorganic bromide in cherries, possibly resulting from Nemagon soil treatments, was determined by the following procedure.

Burn 10 grams of oven-dried cherries in the flask, absorbing hydrogen bromide in 100 ml. of water. Rinse twice with 25 ml. of water and separate bromide from chloride by ion exchange chromatography (*7*) [55 ml. of 0.6*N* sodium nitrate will remove chloride from the resin bed (12 mm. in inside diameter and 16 cm. high)]. Use 30 ml. of 1.8*N* sodium nitrate for elution of bromide and collect six 5-ml. fractions. Determine bromide in each fraction (*4*) by adding 10 ml. of water and 5 ml. of the color reagents to each and measuring the absorbance in a 2-cm. cell. Calculate bromide in the sample by subtracting the absorbance of the first fraction from each of the next four and adding the differences.

Recoveries of 5 and 10 p.p.m. of bromide as sodium bromide were 80.5, 65.8, 116.9, and 104.3 and 87.9, 86.8, 80.3, 95.9, and 133.7%, respectively.

Inorganic chloride in potatoes, resulting from heavy applications of DD to soils, was determined after flask combustion of 10 grams of vacuum-dried potato tissue and absorption of hydrogen chloride in 100 ml. of water. After the flask had been rinsed twice with 10 ml. of water, chloride was determined (*2*); the entire absorbing solution was taken for analysis. Recoveries of 2 mg. of chloride as sodium chloride were 103 and 98%.

Manzate [manganese ethylenebis-(dithiocarbamate)] was determined as manganese in cabbage and onions after combustion of 10 to 15 grams of the oven-dried vegetable and absorption of gases in 100 ml. of 6*N* hydrochloric acid. After rinsing twice with 25 ml. of acid, manganese was determined (*3*), using the entire solution and making absorbance measurements in a 10-cm. cell. Recoveries of 25 γ of manganese as manganese sulfate from cabbage and onions were 96.0, 94.0, 85.6, and 84.0 and 115.2, 101.2, 94.0, and 91.6%, respectively.

Rohm and Haas 0-3818B, a nickel-containing fungicide, was determined as nickel in oats following combustion of 1 gram of whole grain. Absorption of gases in 100 ml. of 0.1*N* hydrochloric acid and rinsing with 100 ml. of the acid and 150 ml. of water were followed by determination of nickel in the combined solutions (*7*). Recoveries of 20 γ of nickel as nickel nitrate were 89.5, 71.0, and 74.5%.

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BUTTER ADULTERATION

Detection of Hydrogenated Fats in Butter Fat by Measurement of cis-trans Conjugated Unsaturation

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A PERENNIAL PROBLEM to food-law enforcement agencies has been the detection of butter adulteration. As long as there exists a price differential between butter fat and possible adulterant fats, and until methods are available for the detection of all such adulterants, this problem will undoubtedly continue.

Methods for the detection of butter adulteration have been surveyed recently (*4*). Many of the methods mentioned lose their effectiveness for the detection of low levels of adulteration when the natural wide variation of pure butter fat is taken into account. Other methods are long and cumbersome and are unsuitable for the analysis of a large number of samples. One method developed in this laboratory (*12*) used the high tocopherol content of most vegetable