DETERMINATION OF MERCURY IN SOILS AND BIOLOGICAL MATRICES BY THE VANADIUM PENTOXIDE DIGESTION PROCEDURE

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The wet digestion procedure using catalytic amounts of vanadium pentoxide with sulfuric-nitric acid mixture developed in this laboratory for the analysis of mercury in cereals is extended to other biological substrates and soils. Investigations of the different parameters, such as temperature and concentration of vanadium pentoxide on the oxidation process indicate that 0.20 g of vanadium pentoxide was adequate to degrade 5.0 g of animal feed at 70-75°C. Studies have shown that excellent recoveries of mercury are obtained in spite of the presence of excess sulfur-containing amino acids and of certain metallic ions commonly present in soils. This digestion procedure is compared with that using potassium permanganate-sulfuric acid as oxidant to illustrate the significant advantages of this technique. It is found that less quantities of reagents and shorter duration of digestion are required for vanadium pentoxide oxidation than are necessary for potassium permanganate system. The probable role of vanadium pentoxide in the oxidation process is also discussed.

In our previous communications (Malaiyandi and Barrette 1970, 1972) a simple, wet oxidation procedure for the degradation of cereal grains using sulfuric-nitric acids in presence of catalytic amounts of vanadium pentoxide was described. Recently Deitz et al. (1973) reported the convenient use of this method of digestion for the analysis of mercury in soils, alfalfa, etc.

The ubiquitous nature of mercury in the ecosystem has resulted in the contamination of food and feed commodities. Because of the highly toxic nature of mercury and its compounds (Karolinska Institute 1969), there has been considerable interest in their detection and determination in foods and feeds at residue levels. This paper describes some minor modifications and successful application of the vanadium pentoxide sulfuric-nitric acid oxidation system to completely degrade animal feeds, meat and meat products, fish and fish products and soils for the determination of submicro quantities of mercury by the flameless atomic absorption technique. It also details a) the studies regarding the effect of temperature and concentration of vanadium pentoxide on the degradation process; and b) the influence of high levels of sulfur-containing amino acids, (degraded animal tissue) and trace elements which commonly occur in soils. Furthermore, data are presented to explain clearly the significance and advantages of the vanadium pentoxide digestion system.

This paper was presented at the 84th Annual Meeting of the Association of Official Analytical Chemists, held in Washington, D. C., Oct. 12-15 (1970).
oxidation system over the potassium permanganate-sulfuric acid degradation procedure of Barrett (1956) and of Uthe et al. (1970). Finally this communication discusses the probable role of pentavalent vanadium in the degradation of some biological constituents.

Materials and methods

The apparatus and most of the reagents used in these experiments, except for the following, were previously described (Malaiyandi and Barrette 1972).

Potassium permanganate: “Baker Analyzed” Reagent
L-(+)-Cysteine hydrochloride: “Baker” grade
L-(+)-Methionine hydrochloride: “Baker grade”
Cobaltous carbonate: (Baker and Adams) labelled to contain 45-50% cobalt.
Copper sulfate (anhydrous), ferrous sulfate crystals, lead nitrate, Magnesium sulfate (anhydrous), and zinc oxide (J. T. Baker Chemical Co.) are “Baker Analyzed” reagents.

Preparation of samples

Animal tissues. Beef or porcine liver and kidney were diced or ground in a Sorvall-Omnimixer; rat liver portions were analysed without pretreatment.

Animal feeds. A 500-g sample of feed was ground in a Sorvall-Omnimixer and tumbled for about five minutes using a Fisher-Kendall mixer and reground. The latter operations were repeated twice to ensure thorough mixing. Three 5-g portions of the sample were used for digestion.

Fish. The body portion of the fish with scales was ground well as described for “animal tissues” above, and kept frozen until the day of analysis. After grinding each sample of fish, the Omnimixer was thoroughly washed with water and rinsed three times with 20 ml of 5% aqueous sodium thiosulfate. Finally it was washed with distilled water, rinsed with redistilled acetone, and dried at 120°C.

Soils. The freeze-dried soil samples (Kennedy et al. 1971; Sivasankara Pillay et al. 1971) were ground with an agate pestle and mortar and screened through nylon sieve (350-400 mesh) to obtain about 25 g of finely powdered soil sample and thoroughly mixed as described for “animal feeds” above.

Digestion procedure

Animal tissues. About 0.1 to 0.12 g of vanadium pentoxide and a known wet weight of animal tissues (four to six g; rat liver weight ca one g) along with three glass beads were placed in the digestion flask. After assembling the apparatus as previously described (Malaiyandi and Barrette 1972), three ml of concentrated nitric acid was added from the