decarboxylation of herbicide acids have been useful to the analyst. Automated pesticide residue analysis, mass spectrometry and other approaches including nuclear magnetic resonance spectrometry, ultraviolet, visible, infrared and fluorescence spectrometry, as well as oscillographic polarography and isotope techniques are other procedures which are currently expected to be modified or replaced to enable the analyst to detect even smaller quantities of toxicants and their metabolites.

71-0876. Mestres, R.; Campo, M.; Tourte, J. (Laboratoire de Chimie Appliquee a l'Expertise, Faculte de Pharmacie, 34 Montpellier, France). Examination and determination of thiabendazole in citrus fruit: Whole fruit, juices and pulps. *Trav. Soc. Pharm. Montpellier 30(3): 193-202; 1970.* (12 references) (French)

A method for the identification and determination of thiabendazole (2-(4-thiazolyl) benzimidazole) in citrus fruit and their juices is presented. Thiabendazole is more commonly employed as an anthelmintic but has also been used as a fungicide. Fruits to be analyzed were quartered and a homogeneous pulp was prepared from the opposing quarter sections. Twenty-five g of fruit pulp were placed in a 1-L vessel with 2.5 ml of concentrated ammonia and 75 ml of ethyl acetate and mixed in a mixer at a moderate speed for 20 min. The extract mixture was filtered through Pyrex wool. The filtrate was extracted again with 50 ml of ethylacetate for 20 min. The combined ethyl acetate extract was treated twice with 20-ml portions of 1 N sodium hydroxide and agitated for 5 min. The organic phase was then washed twice with 5 ml of distilled water and reextracted three times, twice with 10-ml portions of 0.1 N hydrochloric acid (HCl) and once with 5 ml of 0.1 N HCl. The organic phase was then discarded. The aqueous phase was placed with 5 ml of 1 N sodium hydroxide, 5 ml of a sodium acetate and sodium chloride mixture and 25 ml of ethyl acetate in a 125-ml separatory funnel and shaken for 5 min. The phases were allowed to separate completely. The aqueous phase was thrown away. The ethyl acetate phase was washed with 10 ml of distilled water and treated with 10 ml of 0.1 N HCl saturated with ethyl acetate. The extract was then analyzed spectrophotometrically as detailed. The results were compared to those obtained for the reference solutions. Fruit pulp and juice were extracted with a mixture containing crystallized sodium sulfate, concentrated ammonia and ethyl acetate as detailed. Extracts were purified with the help of sodium hydroxide and a mixture of sodium acetate and sodium chloride and then analyzed spectrophotometrically. Recoveries for thiabendazole were in the order of 80 to 100%.

71-0877. Mestres, R.; Illes, S.; Cornet, R.; Gillet, M. (Laboratoire de Chimie Appliquee a l'Expertise, Faculte de Pharmacie, 34 Montpellier, France). Procedure for the preparation of purified petroleum ether for the analysis of pesticide residues. *Trav. Soc. Pharm. Montpellier 30(3): 219-25; 1970.* (5 references) (French)

A technique for the purification of petroleum ether from special G essence (Schell) for the analysis of pesticide residues is described. The method involved oxidation with perhydrol and treatment of the preparation with nascent hydrogen. The quality of the product is very good and permits the gas chromatographic analysis of pesticide residues with electron-capture detection to be carried out with a minimum of interference from unsaturated hydrocarbons. A modification of a method reported earlier (*Ann. Fals. Expert. Chim. 62(685): 75-85; 1969*; for an abstract see 70-0981) for the analysis of pesticide residues in natural waters is presented. Instead of the triple extraction of 500-ml water samples with 200, 200 and 100 ml of petroleum ether, three extractions with 200, 100 and 100 ml of a mixture containing equal volumes of petroleum ether (purified as described herein) and sulfuric ether are substituted. The ether phases are dried on 10 g of anhydrous sodium sulfate before being evaporated to about 10 ml prior to elution from a Florisil column. The modified method gives a better yield for polar compounds and permits the investigation of a wider range of compounds in addition to the organochlorine and organophosphate pesticides.

71-0878. Mestres, R.; Illes, S.; Chevallier, Ch. (Laboratoire de Chimie Appliquee a l'Expertise, Faculte de Pharmacie, 34 [illegible] France). DDT and [illegible] residues in pollen samples [illegible] *Trav. Soc. Pharm. Montpellier 30(3): 227-35; 1970.* (14 references) (French)

Traces of chlorinated plasticizers closely resembling DDE, aldrin and lindane were identified in pollen samples which had been stored in plastic sacks for several months. Two pollen samples from a lot believed to contain 0.45 mg/kg HCB, 0.25 mg/kg *gamma*-HCH, 0.65 mg/kg aldrin and 1.5 mg/kg DDE were submitted for verification analysis. Twelve g of pollen were pulverized with a mortar and then placed in a mixer with 100 ml of acetonitrile for 20 min. The extract was filtered through Pyrex wool, allowed to evaporate and treated with 40 ml of pure petroleum ether. Residues were eluted from a Florisil column. Gas chromatographic analysis was carried out on columns with QF-1 using equipment with a tritium electron-capture detector. Further analysis was carried out using gas chromatographic apparatus equipped with a Ni-63 electron-capture detector with a DC-200 column. In addition, residue samples were chromatographed a third time on a DC-200 column using apparatus equipped for microcoulometric detection. Evaluation of the data obtained led to the conclusion that the organochlorine residues found in the pollen samples originated from a nonpesticide source. Polyethylene sacks which were used to store pollen samples were considered as a possible source of the contaminant. A sample of the sacks was washed in water. Ten mg of the plastic film was then extracted with 100 ml of petroleum ether. The extract was gas-chromatographed using the above mentioned methods and further analyzed using IR spectrophotometry. Pollen samples were also subjected to IR spectrophotometric analysis. The analyses confirmed the presence of chlorinated biphenyls in the samples. The concentrations of chlorinated biphenyls found in the plastic bags ranged from 0.3 to 0.5 g/kg. The corresponding value for the pollen was about 6 mg/kg. The findings showed that food stored in plastic containers can become contaminated by the chlorinated

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plasticizers which are present at low concentrations in the plastic material. Scientists should be careful in interpreting the organochlorine pesticide residue levels in test samples, especially in those samples which have been stored in plastic containers.

71-0879. Mestres, R.; Chevallier, Ch. (Laboratoire de Chimie Appliquee a l'Expertise, Faculte de Pharmacie, 34 Montpellier, France). Determination and evaluation of organochlorine pesticide residues in blood. *Trav. Soc. Pharm. Montpellier 30(3): 237-41; 1970.* (3 references) (French)

Three methods for analyzing organochlorine insecticide residue levels in blood were evaluated. A method involving acetone extraction and elution on a silica gel column which was employed by Richardson *et al.* (1967) to determine levels of dieldrin in blood was used in the determination of zeidan in blood. Various foreign organochlorine compounds interfered with the analysis of pesticide residues. The interfering substances were found to be chlorinated plasticizers in the Merck silica gel. Analytical results were unsatisfactory even after washing the silica gel with acetone, sulfuric ether and methanol. Recoveries ranged from 30 to 50%. The second method of analysis was the one used by Paubionsky (1969) to determine pesticide residue levels in the blood of pesticide factory workers. One-ml blood samples were treated with 3 ml of petroleum ether. Residue samples were then analyzed by direct method using a Microtec chromatograph equipped with a Ni-63 electron-capture detector. Recoveries of 90% for dieldrin and about 85% for zeidan were obtained when this method was used to analyze blood samples containing 0.5 mg/l of either insecticide. The third method of analysis discussed was described by Grosby and Archer (1966). One ml of blood was mixed with 2 ml of an alcohol-potash mixture and heated for 15 min in a water bath at 75 to 80 C. The mixture was cooled under cold running water and treated with pentane or petroleum ether. The ether phase was then chromatographed with the aid of a tritium electron-capture detector. Zeidan (in the form of DDE) and dieldrin recoveries ranged from about 85 to 95%. For pesticide levels under 0.02 mg/l the results obtained with the two latter methods of analysis are almost impossible to verify by microcoulometry. Such verification can be easily obtained when pesticide residue levels are 0.05 mg/l or greater.

See also 71-0657, 71-0701, 71-0720

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