ion by the fetuses prior to farrowing and by the piglets ingesting milk containing ^{36}Cl ion and normal constituents labeled with ^{14}C after farrowing.

In summary, the absence of dichlorvos and the known metabolites of dichlorvos, the presence of ^{36}Cl ion and normal constituents labeled with ^{14}C, and the presence of ^{14}C-labeled carbon dioxide suggest that the ^{14}C residues in the tissues of the sows and piglets are due to normal tissue constituents formed by degradation of the dichlorovinyl moiety in dichlorvos.

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COMMUNICATIONS

Identification of 2,4,6-Trichloroanisole in Several Essential Oils

The identification of 2,4,6-trichloroanisole (I) as a trace constituent of a number of essential oils (distilled Mexican lime, French petitgrain (Clementine), Bulgarian rose, and Spanish rue) is reported. Likely, although not firmly established, is its presence in French geranium, Italian lemon, and American peppermint (*Mentha piperita*). Circumstantial arguments indicate that I is more of fungal or microbial origin than a pesticide residue.

In the course of analytical work on various essential oils we repeatedly encountered fractions having a strong musty naphthalenic odor. After closer examination by gas and liquid chromatography, it became apparent that the same trace component was involved in concentrations between 0.0001 and 0.0009%. In all cases the characteristic odor was observed at the same retention time while smelling the exit gas eluting from a gas chromatograph operating under standardized conditions.

In the attempts to isolate the musty smelling component, we found that it eluted with the hydrocarbon fraction upon chromatographing the oils on silica gel with *n*-pentane as the eluting solvent (Kirchner and Miller, 1952). Further work showed that the compound eluted toward the end of the chromatography procedure together with aromatic compounds such as alkyl naphthalenes. In order not to introduce impurities contained in the silica gel as purchased, the column was washed thoroughly with (carefully purified) *n*-pentane prior to use.

Since apparently small quantities were involved, we did not succeed in preparing a sufficiently pure sample to obtain an acceptable infrared spectrum. Therefore, appropriate fractions were injected into a gas chromatograph coupled to a double-focusing mass spectrometer. The gas chromatograph was equipped with a stainless steel column packed with 10% SE-30 on 70–80 mesh Varaport 30. The mass spectrum of the compound in question indicated the presence of three chlorine atoms, with $M = 210$ for the isotope 35, and suggested a trichloroanisole as a possible structure. Comparing gas chromatographic and mass spectral data of the six isomeric trichloroanisoles showed that only 2,4,6-trichloroanisole and the unknown were identical in all respects.

The occurrence of traces of 2,4,6-trichloroanisole has been established in the essential oils of distilled Mexican lime, French petitgrain (Clementine), Bulgarian rose, and Spanish rue. Its presence in oil of French geranium, Italian lemon, and American peppermint (*Mentha piperita*) is presumed, based on mass spectral data, but has not been unequivocally demonstrated. In addition, direct gas chromatographic analysis of the oils of rose and petitgrain, using a Dohrmann cell, showed the presence of a halogen compound having the identical retention time as 2,4,6-trichloroanisole.

DISCUSSION

The origin of the trichloroanisole in these oils is of considerable interest. To our knowledge, no halogen compounds have been reported previously in essential oils and halogen metabolites are rare in higher plants. However, 1,4-dimethoxy-2-nitro-3,5,6-trichlorobenzene, 1,4-dimethoxy-2,3,5,6-tetrachlorobenzene, and 1-hydroxy-4-methoxytetrachlorobenzene have been discovered in fungi of the Fomes species (Butruille and Dominguez, 1972; Kavanagh *et al.*, 1952; Singh and Rangaswami, 1966). One can, therefore, ask whether the 2,4,6-trichloroanisole is produced by fungi that live on the plants or in the soil and are subsequently absorbed in the essential oil-bearing plants.

On the other hand, the use of polychlorophenols in agriculture as pesticides and in packaging materials as preservatives, and consequently the occurrence of metabolites derived from them, should be taken into account. Recently, 2,3,4,6-tetrachloroanisole has been identified in eggs and broiler chickens, apparently through absorption from litter shavings obtained from wood impregnated with polychlorophenols as antifungal agents (Engel *et al.*, 1966). These chlorophenols are reported to be converted into the corresponding anisoles through microbiological methylation in the litter (Curtis *et al.*, 1972). The same authors mention the extremely low odor thresholds of 2,3,6-trichloroanisole and 2,4,6-trichloroanisole in aqueous solution.

In connection with our discovery of 2,4,6-trichloroanisole, the use of 2,4,6-trichlorophenol (Dowicide 2S) as an antimicrobial agent should be mentioned. However, the difference in geographic location and subsequent agricultural practice, and the difference in plant family and in shipping containers used make it difficult to explain the external introduction of 2,4,6-trichloroanisole into the essential oil. Its absence in such oils as bergamot, cinnamon leaf, lavandin, and spearmint absolves practices used after the oils have left the original containers.

Examination of old essential oil samples antedating the era of wide use of chlorinated industrial chemicals may shed further light.

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Chlorodibenzo-*p*-dioxin Contamination of Two Commercially Available Pentachlorophenols

A comparison was made of chlorodibenzo-*p*-dioxin contamination in a technical and an analytical grade of commercially available pentachlorophenols. Hexa-, hepta-, and octachlorodibenzo-*p*-dioxin were found in both samples. The technical product was more contaminated than the analytical one by factors of 1400, 600, and 539 for the hexa-, hepta-, and octachlorodibenzo-*p*-dioxin, respectively. Tetrachlorodibenzo-*p*-dioxin was not found. Results were determined by gas chromatography and confirmed by infrared spectrophotometry and mass spectrometry. Mass spectral analysis indicated the presence of polychlorodibenzofurans and polychlorodiphenyl ethers. A distinction was made, using gas chromatography-mass spectrometry, between the chlorodibenzofurans observed as original constituents of the pentachlorophenol and those derived from the fragmentation of chlorodiphenyl ethers.

Recent research has shown that some chemicals receiving widespread use in the environment contain contaminants which are more toxic than the major compound (Goursaud *et al.*, 1972; Vos *et al.*, 1970; Woolson *et al.*, 1972). Studies should be conducted to determine the acute and chronic effects of the major compounds both with and without the toxic contaminants.

It is established that the conditions of manufacture for pentachlorophenol (PCP) can give rise to the toxic contaminants chlorodibenzo-*p*-dioxins (CDD's) (Higginbotham *et al.*, 1968; Jensen and Renberg, 1972). The toxicities of some of the compounds in this series have been reported (Higginbotham *et al.*, 1968; Rowe *et al.*, 1971; Williams *et al.*, 1972).

This paper reports an assessment of the levels of contamination of two commercially available PCP's: a technical product containing 86% PCP and a product of analytical grade containing 99+% PCP.

EXPERIMENTAL SECTION

The CDD's were extracted with hexane from an aqueous solution of the sodium pentachlorophenate and the extract was chromatographed on alumina columns according to the method of Firestone *et al.* (1972). Although four fractions were collected from alumina, only fractions III and IV were subjected to sulfuric acid cleanup (Firestone, 1971) and, subsequently, analyzed.

CDD concentrations in fractions III and IV were determined by gas chromatography (gc) coupled with electron capture (EC) ^{63}Ni, Coulson conductometric (CC), and flame ionization (FI) detectors. A Tracor 550 gas chromatograph was equipped with a ^{63}Ni EC detector and a 6 ft × ¼ in. o.d. glass column packed with 3% OV-1 on 80/100 mesh Supelcoport. The column temperature was 220°, and the nitrogen flow was 135 cm^3/min at 60 psi.

A Micro-Tek 2000 with a FI detector was used with a 6 ft × ¼ in. o.d. stainless steel column packed with 3% SE-30 on 100/120 mesh Chromosorb W, DMCS treated, AW. The column temperature was 220°. With a head pressure of 40 psi, the flow rates in cm^3/min are as follows: nitrogen, 80; oxygen, 31; air, 39; and hydrogen, 181.

A Micro-Tek 2000 was also used with a CC detector. A 6 ft × ¼ in. o.d. glass column was packed with 3% OV-1 on 70/80 mesh Chromosorb G, DMCS treated, AW. The column temperature was 220°. Helium flow was 65 cm^3/min at 40 psi.

Under these conditions, the limits of sensitivity for tetraCDD on EC, FI, and CC are 1.0, 10.0, and 2.0 ppb, respectively. Gas chromatograms were compared qualitatively and quantitatively to those of standard CDD solu-