71-1285. Tompsett, S. L. (Dept. of Clinical Chemistry, U. of Edinburgh, Royal Infirmary, Edinburg, Scotland). Paraquat poisoning. *Acta Pharmacol. Toxicol. 28(5): 346-58; 1970.* (23 references)

A colorimetric method for detecting and determining paraquat to a lower limit of 1 mcg/l in human urine, gastric aspirate, blood, dialyzates and tissues is described. The results obtained from applying the method in two fatal and four nonfatal cases of paraquat poisoning are given. When sodium hydrogen carbonate, followed by sodium dithionite, is added to paraquat-containing fluid, an intense blue color is immediately produced. For clinical purposes, urine and gastric aspirate were the most useful reaction substances. Both contained high concentrations of paraquat shortly after its ingestion and the reaction could be applied directly to either diluted liquid. Small quantities of paraquat continued to be excreted in the urine for long periods after ingestion. Using the test on urine several days after paraquat ingestion required concentration of the paraquat and removal of interfering substances on a cation-exchange resin column. In this instance, absorbance of the blue color was measured on a recording *spectrophotometer. With the ion-exchange column, 86 to 91%* recoveries were obtained after adding 50 to 100 mcg paraquat to 100 ml urine. Levels of paraquat in blood and peritoneal dialyzates and various tissues (taken postmortem) were low, when detected at all, and these substances were not, therefore, particularly useful in clinical tests. Preparations of the substances were applied to the ion-exchange column before colorimetric determination. A similar approach might be used to determine diquat, although the color evaluation would be more difficult. Diquat is recovered in the same manner and in the same fraction as paraquat from an ion-exchange column.

71-1286. Ishikawa, K.; Ishii, Y. (Animal Health and Toxicological Lab., Kumiai Chemical Industry Company, Limited, Kikupawa-cho, Agasa-gun, Shizuoka Prefecture, Japan). Colorimetric determination of 2-chlorophenyl N-methyl carbamate. *Agr. Biol. Chem. (Tokyo) 34(7): 1014-9; 1970.* (7 references)

A method for the colorimetric determination of 2-chlorophenyl N-methylcarbamate (CPMC) is described. The method involves hydrolyzing the technical grade material, emulsifiable concentrates of 20% CPMC and dusts containing 1.5 and 2.0% CPMC with a buffer solution made of a sodium barbiturate-sodium acetate solution, sodium chloride solution and hydrochloric acid (buffer adjusted to pH 8.0 with water). The reaction gave the corresponding 2-chlorophenol which was coupled with 4-nitrobenzenediazonium fluoroborate (NF) to produce a stable color with maximum absorption at 520 *mmu*. The determination procedures included passing aliquots of the test solutions on an alumina (Merck 1097) column with acetone solution. Solutions were then diluted with acetone and mixed and heated for 1 hr with the buffer solution. The solution was cooled in ice water, treated with NF and shaken. Absorbance of the solution was measured at 520 m*mu* using 1-cm cells and acetone as reference. CPMC contents were then calculated from the data obtained. The described analytical method can be applied to determine 1 to 5 ppm of CPMC and gives results which are sufficient for residue analysis. The analysis of CPMC residues will be described elsewhere in another report.

71-1287. Baum, G. (Res. and Development Lab., Corning Glass Works, Corning, N. Y. 14830). Determination of acetylcholinesterase by an organic substrate selective electrode. *Anal. Biochem. 39(1): 65-72; 1971.* (17 references)

A new electrochemical method is described for the determination of acetylcholinesterase activity. A liquid membrane electrode is used which has a high selectivity for acetylcholine over choline and various inorganic cations, thus permitting a continuous determination of the rate of change of substrate concentration in the presence of active enzyme. Enzyme activity ranging from 0.2 to 8 units was determined in 10 to 50 ml of saline buffered solutions. Consistent measurements of the rate of change in substrate concentration are obtained within 1 min of introduction of the enzyme. The rate of hydrolysis can be monitored up to about 40% hydrolysis before the interference due to choline concentration becomes significant. The electrochemical procedure was correlated with the spectrophotometric method of Hestrin. (Author abstract)

71-1288. Ahling, B.; Jensen, S. (Inst. of Analytical Chemistry, U. of Stockholm, S-104 05, Stockholm 50, Sweden). Reversed liquid-liquid partition in determination of polychlorinated biphenyl (PCB) and chlorinated pesticides in water. *Anal. Chem. 42(13): 1483-6; 1970.* (7 references)

A method for the extraction of chlorinated pesticides from water was developed based on a reversed liquid-liquid partition method. The column filter material was prepared with 10 g Carbowax 4000 monostearate and 30 g *n*-undecane dissolved in 100 ml acetone and applied to 100 g Chromosorb W, 60 to 80 mesh. Sufficient acetone was added to cover it and then all the acetone was evaporated off under slightly reduced pressure. Three g of the filter material was weighed into a filter column 30 cm long and 1 cm i.d. with a glass filter disk G 1 (100 to 120 *mu*) which was then fitted to the outflow of the water container with an all glass fitting. The lower end of the column was fitted to a water suction or vacuum pump giving an outflow of 65 to 130 ml water/min. Before filtering water with an estimated high particle content, 300 mg aluminum sulfate/l was added and allowed to sediment. The water was then decanted and filtered. The column was eluted with petroleum ether and 10 ml of the effluent was collected. One ml of this extract was mixed with 1 ml concentrated sulfuric acid, the mixture was centrifuged and then chilled in a mixture of carbon dioxide ice and acetone. After decantation of the petroleum ether a 10 ml portion was injected into the gas chromatograph (GLC) columns (one of 5% SE-96 and one of 8% QF-1 on Chromosorb W, 80 to 100 mesh). Air-dried 2-g aluminum sulfate sediment samples were extracted with 6 ml acetone followed by 5 ml petroleum ether and collected in 25 ml distilled water. This extract was then prepared for GLC as the samples above. When the extract of a 200-l sample is concentrated to 2 ml and 10 mcl is injected into the GLC set at 1/8 of highest capacity, 10

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ng/cu. m of lindane can be detected with this system. Verification at this detection level is difficult because a true blank in this concentration range is difficult to obtain. Analysis of tap water revealed 0.05, 0.07, 0.07, 0.22, 0.20 and 0.33 ng/kg water (ppt) of BHC, lindane, DDE, DDD, DDT and PCB, respectively. Waste water and sediment analyzed separately revealed that the water was almost free of residues as compared to the amount found in the aluminum sulfate sediment indicating the possible good effect if aluminum sulfate were used in sewage treatment procedures.

71-1289. Sadar, M. H.; Kuan, S. S.; Guilbault, G. G. (Dept. of Chemistry, Louisiana State U. in New Orleans, New Orleans, La. 70122). Trace analysis of pesticides using cholinesterase from human serum, rat liver, electric eel, bean leaf beetle and white fringe beetle. *Anal. Chem. 42(14): 1770-4; 1970.* (16 references)

A fluorometric method for determining organophosphorus and chlorinated pesticides is described. Electric eel cholinesterase (ChE) was made up in a stock solution of 0.092 mg/ml of protein in distilled water. Stock solutions of white fringe beetle, bean leaf beetle and rat ChE were prepared by the methods of Guilbault *et al.* (1970). Human serum from the blood bank was mixed with butanol according to the method of Morton (1950) to release the ChE from undesirable lipoprotein into true solution which was subsequently purified about 600 fold by chromatography on a Sephadex G-100 column. Pesticide solutions of various molarities were prepared in dioxane. Three ml of 0.1 M phosphate buffer (pH 7.0), 0.1 ml of 0.01 M N-methyl indoxyl acetate (prepared in methyl Cellosolve), 0.1 ml dioxane and 0.1 ml of enzyme solution were placed in a fluorometric cell and the increase in fluorescence with time was recorded using an Aminco Bowman spectrofluorometer (SPF). The procedure was repeated using 0.1 ml of the pesticide solution in place of the 0.1 ml dioxane. From the resultant data, the percentage inhibition was calculated and plotted against the concentration of the inhibitor present and from this plot, the I-50 values (concentration of pesticide to inhibit 50% of the enzyme activity) were found. The nonfluorescent ester used as a substrate, N-methyl indoxyl acetate, was cleaned by each of the enzymes to the highly fluorescent N-methyl indoxyl. It was found that electric eel ChE was generally least sensitive to inhibition. The I-50 values for this enzyme were 0.00030, 0.00010, 0.000067, 0.000070 and 0.00014 M/l from DDVP, aldrin, dieldrin, sevin and methoxychlor, respectively. Lindane and methyl parathion produced an I-50 only at concentrations greater than 0.001 M/l. The other compounds had very little or no inhibitory effect. Bean leaf beetle and white fringe beetle ChE were sensitive to all the compounds, but less sensitive than the rat liver or human serum enzymes, although the last two showed little or no sensitivity to aldrin, dieldrin, methoxychlor, mirex, heptachlor, lindane or DDT. I-50 values for human serum ChE produced by DDVP, paraoxon, parathion and methyl parathion were 0.0000010, 0.00000024, 0.00000150 and 0.000030 M/l, respectively. It was concluded that in addition to sensitivity, some specificity in the assay of pesticides using enzymic methods was achieved.

71-1290. Guilbault, G. G.; Sadar, M. H.; Kuan, S. S.; Casey, D. (Dept. of Chemistry, Louisiana State U. in New Orleans, New Orleans, La. 70122). Enzymatic methods of analysis. Trace analysis of various pesticides with insect cholinesterases. *Anal. Chim. Acta 52(1): 75-82; 1970.* (11 references)

The influence of various pesticides including carbamates, chlorinated hydrocarbons and organophosphorus compounds on partially purified cholinesterases from BHD resistant and NAIDM strains of the housefly, sugar boll weevil, fire ant and German cockroach is reported. The method used to purify the cholinesterase preparationswere the same as those of Guilbault *et al.* (1970). Solutions of the test pesticides of various concentrations (0.000000032 to 0.0000015 M) were prepared in dioxane. Fluorometric, colorimetric or titrimetric assays were used in determining the cholinesterase activity of the different preparations. Fluorometric analysis using the method of Guilbault *et al.* (1968 and 1969) and N-methylindoxyl acetate was employed to determine the inhibitory effect of the test pesticides on enzyme activity. The reaction mixtures generally had a total volume of 10 ml and contained 3 ml 0.1 M magnesium chloride, 1 ml 0.05 M *tris* buffer, 4 ml distilled water and 1 ml of enzyme solution diluted as desired. The mixture was treated with enough sodium hydroxide to adjust the pH to 7.4. The reaction was started by adding 1 ml of substrate (pH 7.4) to the mixture in the reaction vessel. The amount of hydroxide consumed versus time was recorded after a constant rate had been attained. The modified method of Simpson *et al.* (1964) was used to spot-check the enzymatic hydrolysis of acetylcholine. In other tests, acrylamide gel cast in glass tubes (Davis, 1964) was used to accomplish disc electrophoresis. The specific activity of the cholinesterase preparations ranged from 4 to 30. The cholinesterases from each insect showed different inhibition by the various pesticides tested. Evaluation of the experimental data led to the conclusion that in addition to the sensitivity, improved selectivity in the assay of pesticides by this enzymatic method was achieved.

71-1291. Ross, R. T.; Binns, F. J. (Perrine Primate Res. Branch, Div. of Pesticide Chemistry and Toxicology, Food and Drug Admin., U.S. Dept. of Health, Education and Welfare, Perrine, Fla. 33157). Correlation between 31-P n.m.r. chemical shifts and structures of some organophosphorus pesticides. *Anal. Chim. Acta 52(1): 139-41; 1970.* (13 references)

The 31-P nuclear magnetic resonance (NMR) spectra of a series of organophosphorus pesticides were examined. The chemical shift of 37 organophosphorus pesticidal compounds was measured and correlated with the structures of nine classes of compounds; viz., phosphates (I), phosphorothioates (II), phosphonothioates (III), phosphonates (IV), phosphorodithioates (V), phosphoramidothioates (VI), phosphoramidates (VII), phosphorotrithioates (VIII) and phosphites (IX). A Varian HA-100 NMR spectrometer was used to record the 40.5-MHz spectra. A capillary tube containing phosphorus oxide (external reference) was inserted into the 5 mm NMR sample tube containing solutions of the test pesticides. The solvents used were acetone, chloroform neat and benzene. Analytical standards of the organophosphorus pesticides were

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described. (Author abstract by permission of the American Chemical Society)

71-1310. Greve, P. A.; Wit, S. L. (Lab. for Toxicology, National Inst. of Public Health, Sterrenbos 1, Utrecht, The Netherlands). **Rapid identification method for endosulfan from Gle peak shifts under the influence of aykali [sic].** *J. Agr. Food Chem. 19(2): 372-4; 1971.* (15 references)

A simple method is described for the identification of endosulfan by observing the GLC peak shifts brought about by diluted alcoholic alkali. The two isomers of endosulfan yield upon this treatment one new product, the retention time of which is half the sum of the retention times of the original isomers. The method is valid for amounts down to 0.05 ng *alpha*- plus *beta*-endosulfan. The structure of the new compound could be derived from spectroscopical and chemical data. (Author abstract by permission of the American Chemical Society)

71-1311 Asai, R. I.; Gunther, F. A.; Westlake, W. E.; Iwata, Y. (Dept. of Entomology, U. of California, Riverside, Calif. 92502). **Differentiation of polychlorinated biphenyls from DDT by carbon-skeleton chromatography.** *J. Agr. Food Chem. 19(2): 396-8; 1971.* (6 references)

Application of carbon-skeleton chromatography to the qualitative differentiation between polychlorinated biphenyls (PCBs) and DDT is demonstrated. PCBs and biphenyl yield identical carbon-skeleton chromatograms that are strikingly different from that of DDT. Comparisons of the relative retention times of products yielded by the PCBs with known compounds, plus partial identification by their UV spectra, suggested that the products formed at 300 C catalyst temperature were cyclohexylbenzene and biphenyl and at 260 C, were cyclohexylbenzene and a small amount of bicyclohexyl. (Author abstract by permission of the American Chemical Society)

71-1312. Singmaster III, J. A. (Central Analytical Lab., U. of Puerto Rico, Rio Piedras, P. R. 00931). **A modification of the A.O.A.C. extraction procedures for parathion residues on tropical root crops.** *J. Agr. Univ. Puerto Rico 54(1): 189-91; 1970.* (1 reference)

The A.O.A.C. extraction procedures (1965) for parathion residues were modified to permit the extraction of such residues on the roots of yam, sweet potato, cassava and taniers. The solvent systems of alcohol or water and benzene recommended by A.O.A.C. did not serve their purpose because the free starch or mucilage-like substances found in the root samples emulsified the recommended solvents. Modified extraction procedures with benzene as the solvent and anhydrous sodium sulfate when necessary to break the emulsion by reducing the water in extract slurries were used. Chopped 150-g samples of cassava or tanier were placed in a blending jar and mixed with 300 ml of distilled benzene for 3 min. The mixture was transferred to a 500-ml centrifuge bottle and centrifuged for 5 min at about 1000 rpm. The extract was passed through a 1-in layer of anhydrous sodium sulfate in a funnel with a glass wool plug. Fifteen instead of 10 g of adsorbent mixture (24.169 (b), Association of Official Methods of Analysis, 10th ed., 1965) per 100 ml of extract were shaken for 5 min and filtered through rapid folded paper. The remainder of the procedure was as detailed at 24.172 in the "Official Methods of Analysis" (p. 404). The same procedure as described above was used for the extraction of sweet potato samples except that 20 g of the A.O.A.C. adsorbent mixture were used/100 g of extract. Chopped yam samples weighing 150 g were blended with 300 ml of distilled benzene for 2 min and mixed for 1 min more with 75 g of anhydrous sodium sulfate. The liquid was decanted into a 500 ml centrifuge bottle, treated with about 25 g of anhydrous sodium sulfate, shaken and then centrifuged for 5 min at 1000 to 2000 rpm. The yam extract could not be completely decolorized with the adsorbent mixture but the adsorbent treatment was sufficient to reduce interferences and make the rest of the procedure useful for the analysis of parathion residues. The supernatant was transferred to a weighed 500 ml Erlenmeyer flask to which 20 g of the A.O.A.C. adsorbent mixture/100 g of extract were added. The mixture was shaken for 5 min and filtered through rapid folded paper. Further study is needed to determine the correct proportion and time to add the sodium sulfate to obtain sufficient amounts of the extract. For yam and cassava samples fortified with 0.2 to 2 ppm parathion, recoveries ranged from 80 to 110%. Recoveries from sweet potato and tanier treated with 0.2 to 2 ppm parathion ranged from 65 to 89%. Analytical findings indicated that neither 2 nor 4 lb a.i. parathion/acre left parathion residue levels above 0.2 ppm in any of the four root crops at harvest. The described extraction procedures may also be applied to modify the official A.O.A.C. method for the analysis of other pesticides (DDT) in these crops.

71-1313. Singmaster III, J. A. (Central Analytical Lab., U. of Puerto Rico, Rio Piedras, P. R. 00931) **A simplified analytical method for determination of the soil adsorptivities of urea herbicides.** *J. Agr. Univ. Puerto Rico 54(1): 187-8, 1970.* (5 references)

A simplified analytical method for the determination of the adsorptivities of urea herbicides such as diuron in Puerto Rican soils is presented. The method involved equilibrating a known solution of herbicide with a soil sample. An air-dried soil sample weighing 50 g was shaken with 100 ml of a urea herbicide solution (Yuen and Hilton, 1962). The slurry was centrifuged at 1000 rpm for 5 min. A portion of the supernatant was filtered through a fine filter paper. Ten ml of the solution were placed in a 25-ml volumetric flask and hydrolyzed by adding 0.5 ml of concentrated sulfuric acid and heating in a boiling water bath for 4 hr. The flask was removed from the bath, cooled and treated with 2 ml of glacial acetic acid and 1 ml of 1% sodium nitrite solution. After 10 min, 1 ml of 10% sulfamic acid solution was added to the flask. After the flask was swirled several times during another 10 min period, 1 ml of 2% *N*(1-naphthyl)ethylenediamine dihydrochloride was added to the solution which was then diluted to 25 ml with water and shaken thoroughly. Absorption was read at 560 mμ after 15 min. Standard curves were made by carrying 10 ml of a urea herbicide solution through the

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71-1326. Sphon, J. A.; Damico, J. N. (Div. of Food Chemistry and Technology, Bureau of Science, Food and Drug Admin., Dept. of Health, Education and Welfare, Wash., D. C. 20204). The mass spectra of some chlorinated aromatic pesticidal compounds. *Org. Mass Spectrometry 3(1): 51-62; 1970.* (24 references)

The mass spectra of *o,p'*- and *p,p'*-DDT, -DDD and -DDE, perthane (1,1-dichloro-2,2-bis(*p*-ethylphenyl)ethane), methoxychlor (1,1,1-trichloro-2,2 bis(*p*-methoxyphenyl)-ethane), Dimite (bis(*p*-chlorophenyl)methyl carbinol), Etoxinol (1-ethoxymethyl-1,1 di(*p*-chlorophenyl)carbinol), chlorobenzilate (ethyl 4,4'-dichlorobenzilate), Kelthane (,1-bis(*p*-chlorophenyl)-2,2,2-trichloroethanol) and some of their isomers were investigated. The spectra were recorded with an Atlas CH4 mass spectrometer equipped with a probe inlet system, which was necessary to avoid thermal degradation in heated reservoirs. The operating parameters were: ion source regulation operated at 250 C, electron energy of 70 volts and magnetic scanning and accelerating potential of 3 kV. The temperatures of the samples during evaporation ranged from 20 C for methoxychlor and Dimite to 62 C for Kelthane. The metastable transitions determined by the defocusing technique were obtained with an AEI MS-9 mass spectrometer. Bar graphs of the data were obtained using a Calcomp plotter. The studies showed that the R' group is labile for all the compounds except the DDE isomers. The most prominent ion was a possible substituted phenyltropylium ion found at m/e 235 of the DDT and DDD isomers, at m/e 227 of the methoxychlor isomer and at m/e 223 of the perthane isomer. Metastable peaks indicated that the second most abundant ion (m/e 165) of the DDT and DDD isomers resulted from stepwise decomposition of the two R groups from the m/e 235 ion. The data indicated that this ion was of a fluorenyl ion structure. DDE isomers were distinguished by the significant differences in the percent total ion current of several chatacteristic peaks (m/e 316, 246, 210 and 176). The DDT isomers could also be distinguished by their mass spectra, but those of DDD could not. The principal ion of Kelthane was noted at m/e 251. This ion apparently fragmented again to an ion at m/e 139, which subsequently produced ions at m/e 111 and 104. Etoxinol and chlorobenzilate underwent similar decomposition. Dimite underwent dehydration of the molecular ion at m/e 248. This ion subsequently lost chlorine to yield abundant ions at m/e 213 and 178. Dimite also underwent fragmentation by cleavage between the aliphatic carbon and the ring to form the m/e 155 ion, which further decomposed to form the acetyl ion. Studies on the identity of the m/e 246 ion of DDE show that its structure is complex and that it probably comprises more than one structure.

71-1327. Richardson, A.; Robinson, J.; Crabtree, A. N.; Baldwin, M. K. (Tunstall Lab., Shell Res. Limited, Sittingbourne, Kent, England). Residues of polychlorobiphenyls in biological samples. *Pesticides Monit. J. 4(4): 169-76; 1971.* (21 references)

The presence of polychlorobiphenyls (polychlorinated biphenyls) in samples of aquatic origin has been demonstrated and confirmed by combined gas-liquid chromatography-mass spectroscopy. The relevance of this finding to the determination of organochlorine insecticides is discussed. (Author abstract by permission)

71-1328. Hlada, A. (Vyskumny Ustav Hygieny Prace a Chorob z Povolania, Bratislava, Czechoslovakia). The determination of *p*-nitro-*m*-cresol as a degradation product of fenitrothion in human urine. *Pracovni Lekar. 21(9): 405-8; 1969.* (18 references) (Slovak)

An analytical method for the determination of *p* nitro-*m*-cresol (NC) in the urine of persons exposed to fenitrothion was worked out. The method may serve as a sensitive test of exposure. NC is released from the conjugated form of hydrolysis and separated by extraction with acetonitrile from an alkaline medium and with a mixture of petroleum ether and ethyl ether from an acidic medium. It is separated from coextracts by thin-layer chromatography on silica gel and is determined spectrophotometrically as the yellow sodium nitrocresolate. The mean results of control determinations was 98 plus/minus 9.6%; the lower limit of detection was 2 mcg NC in 20 ml urine. (Author abstract edited)

71-1329. Renvall, S.; Akerblom, M. (National Inst. of Plant Protection, 171 07 Solna, Sweden). Determination of organophosphorus pesticide residues in fruits and vegetables on the Swedish market from 1964 to 1968. *Residue Rev. 34: 1-26; 1971.* (27 references)

Fruit and vegetables produced in or imported into Sweden were analyzed for residues of organophosphorus pesticides. The analytical methods, which included the cholinesterase inhibition test, gas and thin-layer chromatography are described. They permitted the identification of 25 organophosphorus pesticides. From July 1964 to December 1968, 2396 samples of different produce (apples, beans, broccoli, carrots, cauliflower, cherries, cucumbers, grapes, lettuce, peaches, pears, plums, potatoes, spinach, tomatoes and white cabbage) were collected. Half of the samples were collected from crops produced in Sweden. Cholinesterase inhibition, which could account for residues of pesticides, occurred in 11% of the samples and in 8% of these, the pesticides were actually identified. In 0.8% of the samples (0.7% of these were imported and 0.1% domestic), there were residues exceeding 0.5 mg/kg, a common tolerance level. A pesticide content of 0.1 to 0.5 mg/kg was obtained in 3.2% of the samples (3.1% imported and 0.1% domestic) and 0.01 to 0.1 mg/kg was found in 4% (3.0% imported and 1.0% domestic). The pesticides found were, in descending order of occurrence, parathion, ethion, parathion-methyl, diazinon, carbophenothion, azinphos-methyl, trichlorfonate, fenitrothion and malathion. Parathion and ethion, the most frequently occurring, were mainly found in imported apples and pears, while parathion was also often found in imported lettuce. (Author abstract by permisson)

71-1330. Reynolds, L. M. (Ontario Res. Foundation, Sheridan Park, Ontario, Canada). Pesticide residue analysis in the presence of polychlorobiphenyls (PCB's). *Residue Rev. 34: 27-57; 1971.* (61 references)

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Since 1966, PCBs have been detected in fish and wildlife, especially in Europe and North America. Contamination from industrial wastes via the water ways appears to be the main source of PCBs in aquatic species. The PCBs have many important industrial uses but are not utilized as pesticides. Due to their similarities in structure and properties to the organochlorine pesticides, the PCBs tend to interfere with accurate GLC-EC determination of organochlorine and many organophosphorus pesticides. In addition to their interference with pesticide residue analysis, the PCBs are themselves toxic so that their quantitation is desirable. A method for recognizing the presence of PCBs in samples, separating them from pesticides and determining the pesticide levels accurately is described. Simultaneously, an estimate of total PCB content is afforded. The residue data of some Canadian aquatic birds indicate that high levels of PCBs are found only in the presence of correspondingly high levels of the DDT pesticide group. On the other hand, high organochlorine pesticide levels are not necessarily accompanied by high PCB content. Further research on the toxicity and sublethal effects of PCB mixtures and their individual components is required. If particular components are shown to be more toxic than others, refining of the PCB quantitation methods will be necessary. (Author abstract by permission)

71-1331. Burke, J. A. (Div. of Pesticide Chemistry and Toxicology, Bureau of Foods and Pesticides, Food and Drug Admin., U.S. Dept. of Health, Education and Welfare, Wash., D. C. 20204). Development of the Food and Drug Administration's method of analysis for multiple residues of organochlorine pesticides in foods and feeds. *Residue Rev. 34: 59-90; 1971.* (103 references)

As discussed in the introduction, the most current and complete description of the Food and Drug Administration analytical method for multiple pesticide residues in fatty and nonfatty types of samples, and of supporting data, is given in the FDA "Pesticide Analytical Manual" (PAM) Vol. I, January 1968, July 1969, and June 1970 revisions. Revisions of the PAM to include new developments will be made once or twice a year. "Official Methods of Analysis" of the AOAC (11th ed., 1970) contains a detailed description of extraction, cleanup, GLC and TLC procedures recognized as official AOAC methodology. Improvements and additions to this methodology will be covered in reports of the General Referee for Chlorinated Pesticides, the General Referee for Collaborative Studies of Multiple-Residue Methods and the AOAC Subcommittee E to future meetings of the AOAC. The material presented, a review of the development of FDA's methodology for multiple residues of organochlorine pesticides and references to reports containing supporting or validating data bring together the results of the vast research effort which has gone into the development of this methodology. It shows the extent of refinement of the methodology and should serve as a source for information about the method: its scope, capabilities and limitations. No other residue analytical method has been developed to this extent. There will be a continual effort to refine and extend the FDA method, with changes appearing in the PAM Vol. I. Methodology included in PAM Vol. I, Chaper 2, Sections 211, 212, 231 and 232, Chapter 3 and Chapter 4, Sections 410, 411 and 412 is, in the author's opinion, the best available for the determination of multiple foods and feeds. (Author abstract by permission, edited)

71-1332. Mirkhaitov, T.; Ikramov, L. T. (Tashkent Pharmaceutical Inst., Tashkent, USSR). *New qualitative reactions for detecting phosalone and phthalophos. Sudebno-Med. Ekspertiza 13(1): 29-32, 1970.* (Russian)

Because of their forensic chemical and toxicological significance, new qualitative reactions for detecting phosalone (O,O-diethyl-S-(6-chlorobenzoxazolinyl-3-methyl) dithiophosphate) and phthalophos (O,O-dimethyl-N-phthalimidomethyl dithiophosphate) were developed. The interaction of the two insecticides with 180 reagents from various classes of chemicals was investigated. The results showed that phosalone gives characteristic products with the following reagents: iodine chloride, silver nitrate, cupric nitrate and Frochde's reagent. Phthalophos forms microcrystal residues with iodine chloride and mercuric iodide; in addition, it can be detected from its own microcrystals after recrystallization from alcohol solutions and with Frochde's reagent. Each of these reactions is briefly described and the microcrystals formed illustrated. The reactions can be applied to analyzing various concentrates and dusts manufactured for agricultural use.

71-1333. Goodrich, P. R.; Monke, E. J. (Dept of Agricultural Engineering, Purdue U., Lafayette, Ind. 46207). *Insecticide adsorption on activated carbon. Trans. ASAE [Am. Soc. Agr. Eng.] 13(1): 55-60; 1970.* (6 references)

The effectiveness of various sizes of granular activated carbon in carbon filters of different length in removing small amounts of dieldrin from water was determined. Two radiotracer techniques for the detection of low insecticide levels were also evaluated. Insecticide solutions were prepared by mixing 65 ng of carbon-14-labeled dieldrin with 1 L of deionized water, ½ of which was passed through activated carbon columns with lengths of 70, 140, 210 and 280 mm. The activated carbon used corresponded to U. S. standard sieves 6 to 8, 8 to 10, 10 to 12 and 12 to 14. The dieldrin in the filtered and unfiltered solutions was extracted with chloroform and evaporated on aluminum planchets after the extract had been treated with ethylene glycol to retard evaporation of the insecticide. The planchets containing 0.50-ml samples of the insecticide were dried slowly in a chemical hood. The samples were analyzed by radioactive counting by the RIDL system (Radiation Instrument Development Laboratory system), a low background automatic counting system or by the Beckman Low Beta Counting System. The functions of the latter system were internally programmed. The experimental findings showed that the granulated activated carbon removed up to 99% of the dieldrin from the water. Insecticide adsorption on the carbon was not significantly affected by the size of the granules or the length of the carbon filter. Recoveries of 98% were achieved with the radiotracer techniques used. The processes were much more simple than those of other analytical techniques such as gas chromatography and impurities in the samples did not interfere with the determinations.

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