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Review

Thin-layer chromatographic methods for use in pesticide residue analysis

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ABSTRACT

A selective review is presented, focusing mainly on stationary phases, mobile phases, detectors and TLC techniques used for the detection, separation, determination and identification of pesticide residues in various environmental samples. The results from numerous papers are presented in tabular form.

CONTENTS

1. INTRODUCTION

TLC has grown rapidly in recent years and is now widely accepted as a rapid and efficient detection technique. TLC has replaced paper chromatography in pesticide residue analysis because of its higher resolution and shorter development time. It is lacking in the precise specificity of gas–liquid chromatography but it is more precise and sensitive than PC. Although most advances in pesticide analysis during the past few years have taken place in the field of GC and HPLC, TLC has retained its status as a valid and simple method for the qualitative and quantitative analysis of pesticide residues and their metabolites.

2. RESULTS

A number of books, reviews [1–88] and research papers have been published in this area. Several new coating materials have been discovered and tested for pesticides analysis. The older coating materials have been re-investigated using new solvents systems, especially mixed solvents. Many of the papers published on this subject originate from researchers in the less developed countries, probably because of the lack of more sophisticated instrumentation.

The results from numerous publications are presented in Tables 1 and 2.

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TABLE 1

ABBREVIATIONS

Stationary phases	
P1	Admixtures of barium sulphate and calcium sulphate
P2	Admixtures of silica gel and calcium sulphate
P3	Alumina G
P4	Aluminium oxide, Silica gel KCK and Silufol
P5	Aluminium oxide 60 F_{254} (Type E)
P6	Barium sulphate
P7	Calcium sulphate containing ammonium molybdate, aluminium oxide, calcium carbonate, copper sulphate, iron(III) chloride, magnesium sulphate, phthalic anhydride and zinc oxide
P8	Cellulose
P9	Chromarods-A
P10	C_8 and C_{18}
P11	C_{18} chemically bonded RP layer
P12	C_{18} SPE and Silica gel 60
P13	KC_{18} reversed-phase impregnated with Bratton–Marshall reagent
P14	Kieselgel 60, silica gel 60, Kieselguhr F_{254}, Aluminium oxide G, Polygram $SILUV_{254}$, Polygram cell 300 and Silufol
P15	Polyamide
P16	Polygram SILG UV_{254}
P17	Preadsorbed silica gel layers impregnated with silver nitrate
P18	Precoated plate CNF_{254S}
P19	RP-18-W layers
P20	Silica gel
P21	Silica gel G
P22	Silica gel–plaster of paris
P23	Silica gel LS/40m
P24	Silica gel impregnated with cresol (0.5%)
P25	Silica gel impregnated with metal ion or phenol
P26	Silica gel containing zinc acetate (1% w/w)
P27	Silica gel–Kieselguhr (2:3)
P28	Silica gel GF_{254}
P29	Silica gel impregnated with paraffin in hexane (5%)
P30	Silica gel impregnated with diethylene glycol (20%)
P31	Silica gel precoated with fluorescence indicator
P32	Silica gel, Merck No. 5721
P33	Silica gel impregnated with 2-diphenylacetyl-1,3-indandion-1-imine
P34	Silica gel SPF
P35	Silica gel 60
P36	Silica gel Sep-Pak or C_{18} preadsorbent TLC plate
P37	Silica gel F_{1500}
P38	Silica gel HF_{254}
P39	Silica acid-impregnated glass-fibre sheets
P40	Silufol
P41	Silufol UV_{254}
P42	Silver nitrate-impregnated alumina G
P43	Sorbfil HPTLC plates, silica gel with silicic acid
P44	Soil
P45	Whatman $LKC_{18}D$ chemically bonded reversed-phase plates
Mobile phases	
S1	Dichloromethane–heptane (2:9)
S2	Hexane–benzene (1:1) and hexane–diethyl ether (1:1)
S3	Acetonitrile–water (75:25)
S4	Hexane–chloroform (60:40) and hexane–benzene (45:55)
S5	Acetonitrile–water–ammonia (40:9:1) followed by hexane–diethyl ether (90:10)

TABLE 1 *(continued)*

Mobile phases	
S6	Acetonitrile–water–ammonia (40:9:1) followed by hexane–methanol–acetic acid (5:5:1)
S7	Chloroform–diethyl ether–hexane–toluene (293:257:250:200)
S8	Hexane–acetone (70:30) or benzene–chloroform (70:30)
S9	Chloroform–benzene (9:1) and chloroform–methanol (1:1)
S10	Hexane–acetone (4:1) and hexane–trichloromethane (1:1)
S11	Light petroleum–benzene–ethyl acetate (65:30:5)
S12	Ethyl acetate–chloroform (1:9)
S13	Hexane–acetone (1:1) followed by chloroform–acetone–methanol (1:1:1)
S14	Acetic acid
S15	Toluene–ethyl acetate (85:15)
S16	Hexane, acetone and ethyl acetate
S17	Hexane–acetone (2:1) or benzene–ethyl acetate (2:1)
S18	Hexane–diethyl ether (3:1)
S19	Benzene–chloroform–methanol (9:3:2) and chloroform–methanol (3:1)
S20	Water–acetonitrile (1:9) and water–methanol (25:75)
S21	Benzene, chloroform, carbon tetrachloride, distilled water, 1,4-dioxane and ethyl acetate
S22	Hexane–acetone (1:1) and diethyl ether–hexane–ethanol (77:20:3)
S23	Cyclohexane–benzene–acetic acid–liquid paraffin and cyclohexane–benzene–acetone
S24	Benzene–hexane–acetic acid (5:13:2) and hexane–acetic acid (7:3)
S25	Diethyl ether–toluene (1:3 or 2:1) and chloroform–nitromethane (2:1)
S26	Dichloromethane–acetone, toluene–acetone, chloroform–ethyl acetate and benzene–acetic acid (9:1)
S27	Toluene–acetone, ethyl acetate–chloroform and chloroform–acetone
S28	Hexane–butyl acetate
S29	Toluene–acetone (85:15)
S30	Chloroform–ethyl acetate or hexane–ethyl acetate
S31	Chloroform–acetone (95:5)
S32	Hexane–butyl acetate (60:60)
S33	Acetic acid–chloroform–isooctane (5:20:75) or acetic acid–chloroform (3:7)
S34	Butanol–formic acid–water (7:2:1)
S35	Hexane–diethyl ether (3:1) followed by hexane–acetone–acetic acid (35:25:0.05)
S36	Chloroform–methanol (49:1)
S37	Cyclohexane–acetone (10:1) and light petroleum–benzene–ethanol (65:30:5)
S38	Chloroform–acetone and hexane–acetic acid–diethyl ether
S39	Hexane–benzene (45:55)
S40	Carbon tetrachloride–light petroleum (60:40)
S41	Acetone–hexane (1:9)
S42	Hexane–methanol–diethyl ether (3:1:1)
S43	Hexane, hexane–benzene (1, 2, 3:1, 2, 3), benzene, hexane–benzene–acetonitrile (10:10:1, 10:10:2), chloroform–benzene (2:1, 9:1), chloroform, chloroform–acetone (9:1), chloroform–ethyl acetate–acetonitrile (9:1:1), dichloromethane–ethyl acetate (7:3), ethanol and acetone–water (1:1)
S44	Hexane–acetone (4:1)
S45	Hexane–chloroform (2:1, 4:1)
S46	Hexane–acetone (2:1)
S47	Hexane–chloroform (2:1) and hexane–acetone (3:1)
S48	Hexane–xylene–ethyl acetate–water (50:15:5:18)
S49	Hexane–acetone (3:1)
S50	Chloroform–light petroleum (1:2)
S51	Chloroform–acetone (68:32)
S52	Acetonitrile–water (80:20)
S53	Benzene–hexane (2:1), acetone–benzene (100:0.4), benzene and methanol
S54	Chloroform–acetone
S55	Hexane, heptane, chloroform, benzene, ethanol, ethyl acetate, carbon tetrachloride, diethyl ether, light petroleum, acetone and their mixtures

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TABLE 1 *(continued)*

Mobile phases	
S56	Chloroform
S57	Methanol, methanol–water (1:1, 9:1), water–formic acid–methanol (4:1:5), water–acetone (69:4), methanol–25% ammonia solution (30:1), acetone, methanol–diisopropyl ether (1:1, 1:4), hexane–acetone (9:3, 7:3), chloroform–acetone (9:1) and diethyl ether
S58	Acetone, acetonitrile, diethyl ether, ethyl acetate, acetone–hexane, diethyl ether–hexane, ethyl methyl ketone–light petroleum, acetone–acetonitrile–cyclohexane and acetone–cyclohexane–ethanol
S59	Hexane–ethyl acetate (13:7)
S60	Hexane–ethyl acetate–acetone (9:1:1)
S61	Benzene–ethyl acetate (9:1)
S62	Benzene, chloroform, carbon tetrachloride and distilled water
S63	Chloroform–methanol (1:1) and glacial acetic acid–methanol–benzene (1:1:3)
S64	Benzene–ethyl acetate (50:10)
S65	Benzene–ethylmethyl ketone (9:1)
S66	2-Propanol–ammonia–water (10:1:1)
S67	Cyclohexane–acetone (4:5)
S68	Dichloromethane–acetone–acetic acid (8:1:1), acetone–toluene–acetic acid (2:2:1), dichloromethane–toluene–acetic acid (2:2:1), toluene–benzene–acetic acid (2:2:1), dichloromethane–heptane–acetic acid (2:2:1), ethyl acetate–dichloromethane–acetic acid (8:1:1), ethyl acetate–acetic acid (49:1), acetone–acetic acid (97:3), dichloromethane–acetone–acetic acid (5:4:1), acetone–chloroform–heptane–acetic acid (3:3:3:1) and toluene–benzene–dichloromethane–acetic acid (3:3:3:1)
S69	Organic solvents, mixtures of water and acids
S70	Dichloromethane–methanol (99:1) and acetonitrile–water (85:15)–3% sodium chloride
S71	Chloroform–acetone (6:1)
S72	Dichloromethane
S73	Hexane impregnated with paraffin oil in light petroleum (8%), in acetonitrile–acetone–methanol–water (40:18:40:2), acetone, hexane–diethyl ether–acetone (7.5:2:0.5) or hexane–diethyl ether (8:2)
S74	Chloroform–diethyl ether (5:2) and hexane–acetone (20:1)
S75	Benzene–ethanol (9:1), chloroform–ethyl acetate (9:1), chloroform–ethyl acetate–acetone (17:2:1), light petroleum–acetone (7:3), chloroform–cyclohexane–ethyl acetate (15:2:3) and chloroform–ethyl acetate–acetone (13:5:3)
S76	Pentane–diethyl ether
S77	Methanol (25%)–potassium iodide–butanol (80:15:5)
S78	Carbon tetrachloride and hexane–acetone (95:5)
S79	Hexane–ethyl acetate (3:1)
S80	Dichloromethane–acetone–acetic acid (8:1:1), acetone–toluene–acetic acid (2:2:1), benzene–ethanol–acetic acid (6:3:1), acetone–light petroleum–acetic acid (2:2:1), acetone–methanol–acetic acid (6:3:1), acetone–diethyl ether–acetic acid (2:2:1), dichloromethane–toluene–acetic acid (2:2:1), acetone–chloroform–acetic acid (2:2:1), toluene–benzene–acetic acid (2:2:1), dichloromethane–heptane–acetic acid (2:2:1), dichloromethane, dichloromethane–acetone–benzene (2:1:1), acetone–benzene–acetic acid (5:4:1), dichloromethane–heptane–ethanol (2:2:1), chloroform–hexane–dichloromethane (1:1:1), chloroform, diethyl ether–toluene (1:1:1), hexane–heptane–ethanol (2:2:1), dichloromethane–light petroleum (2:3) and toluene–heptane–diethyl ether (1:1:1)
S81	Chloroform–methanol (1:1)
S82	Hexane–acetone (3:1), benzene–acetone (6.6:3.4), hexane–acetone (9:1) and hexane–acetone (4:1)
S83	Benzene–ethyl acetate–water (5:4:1), benzene–ethyl acetate–acetic acid (15:4:1) and toluene–ethyl acetate–acetic acid (25:15:2)
S84	Hexane–acetone (4:1) or hexane–diethyl ether (2:1), hexane–benzene (1:1) or hexane–toluene (1:1), benzene–methanol–acetone (7:1:2) or benzene–methanol–diethyl ether (7:1:2.5)
S85	Hexane–acetone (90:10), hexane–acetone (150:45), chloroform–nitromethane (100:100), chloroform–acetic acid (190:10) and benzene–hexane–acetic acid (50:100:20)
S86	Distilled water
S87	Hexane–dioxane–acetic acid (79:20:1), hexane–dioxane (80:20), hexane–dichloromethane (30:70) and chloroform–diethyl ether (80:20)

TABLE 1 *(continued)*

Detection	
D1	$SnCl_2$ in 50% HCl–aqueous fuchsin dye solution
D2	3-Methylbenzidine, N,N′-dimethylbenzidine, N,N′-tetramethylbenzidine and 3,5,3′,5′-tetramethylbenzidine in presence of sunlight
D3	Ammoniacal silver nitrate solution and densitometric scanning
D4	*o*-Tolidine reagent and densitometric scanning ranging from 300 to 900 ng
D5	Preadsorbed $AgNO_3$ and UV light
D6	1,4-Dihydroxybenzene
D7	Pyrolysis technique with IR laser and electron capture
D8	Iodine
D9	Cholinesterase
D10	KI_3 + KI solution
D11	Mass spectrometry
D12	Ammonium molybdate (15%) in HNO_3 (2:1)
D13	$PdCl_2$ or *p*-dimethylaminobenzaldehyde
D14	Copper(II) acetate in dilute HCl followed by KI
D15	*In situ* densitometry
D16	Acetylcholinesterase
D17	*p*-Dimethylaminobenzaldehyde–acetic acid (10:1)
D18	4-Amino-N,N′-dimethylaniline · 2HCl
D19	Swine liver hommogenate–alcoholic β-naphthol acetate
D20	Palladium(II) chloride–iodine
D21	KOH–*p*-nitrobenzenediazonium fluoroborate
D22	Dimethylaminobenzaldehyde, *p*-nitrophenyldiazomine or 2,6-dibromo-N-chloroquinoimine
D23	Iron(III) chloride (1%) in butanol and 2,4-dinitrophenylhydrazine (3%) in chloroform–methanol (3:1)
D24	$CuCl_2$ (1%, w/v) followed by metavanadate or potassium hexacyano–ferrate(III) (0.5%, w/v) in sodium hydroxide (0.5%, w/v)
D25	Diphenylamine
D26	Diazotized *p*-nitroaniline or diazotized *p*-aminoacetophenone
D27	Fluorescamine
D28	Ninhydrin
D29	Hill reaction inhibition detection technique
D30	*o*-Phthalaldehyde in 7 *M* sulphuric acid and ethanol (10%) and UV (350 nm)
D31	Bromophenol blue
D32	Silver nitrate and UV light
D33	UV light (365 nm) or densitometry
D34	Reflection–absorption photometry at 240 nm
D35	UV light and *o*-tolidine–Mitchells' reagent
D36	4,4′-Tetramethyldiaminodiphenylmethane
D37	Fluorimetry
D38	Photolysis, morin derivatization and fluorescence
D39	Dragendorff reagent
D40	Dithionite
D41	Microbioassay using *C. cucumerinum* spores
D42	Chloroplast homogenate and 2,6-dichloroindophenol, exposure to white neon light
D43	Aqueous K_2CO_3 (10%) and diazotized *o*-dianisidine solution or orthanilic acid or *o*-dianisidine or diazotized orthoanilic acid
D44	Aqueous sodium hydroxide (20%) followed by nickel aminine reagent [aqueous nickel chloride solution (5%, w/v)–ammonia (30%) (1:1)]
D45	3,5,3′,5′-Tetramethylbenzidine (0.2%)
D46	Differential-pulse polarography
D47	Gas chromatography with electron-capture detection

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TABLE 1 *(continued)*

Detection	
D48	$Hg(NO_3)_2$ + diphenylcarbazone, iodine, $HgNO_3$ and heating, acidic potassium permaganate
D49	Ammonia solution–water (1:4) followed by beef liver homogenate, indoxyl acetate, $K_3Fe(CN)_6$ and $K_4Fe(CN)_6$
D50	Magnesium chloride (5%) followed by N,2,6-trichlorobenzoquinonimine (0.3%)
D51	N,2,6-Trichlorobenzoquinonimine
D52	Triethanolamine exposed to mercury lamp, 254 nm
D53	Diazotization with sodium nitrite solution after thermal degradation with 4-aminoantipyrine in presence of ammonium persulphate (betanol), or *p*-dimethylaminobenzaldehyde (asulam, betanol), bromophenol blue reagent or *o*-toluidine after N-chlorination (eptane, tillam, yalan), Dragendorff reagent or sulphuric acid (roneet)
D54	UV quenching, NaOH, $AgNO_3$, *p*-nitrobenzenediazonium tetrafluoborate, fisetin and enzyme inhibition
D55	Rhodamine B–UV, *p*-dimethylaminobenzaldehyde, potassium permanganate, silver nitrate + bromophenol blue, pinacriptol yellow–UV, sodium fluoresceinate–UV, sodium fluoresceinate–UV (modified method), iodine vapour and iodine spray
D56	2,6-Dibromo-N-chlorobenzoquinonimine (0.5%) or N,2,6-trichlorobenzoquinonimine (Gibbs reagent) in acetic acid
D57	Diazotized *p*-nitroaniline
D58	*p*-Nitrophenyldiazonium chloride
D59	Methanolic potassium hydroxide (1%) followed by *p*-nitrobenzenediazonium tetrafluoroborate (0.1%) in acetone
D60	Sodium nitrite in 1 *M* hydrochloric acid (1%) followed by N-(1-naphthyl)ethylenediaminedihydrochloride (1%) in 2 *M* hydrochloric acid
D61	Ethanolic Fast Blue B (1%) followed by sodium hydroxide (20%), UV light (254 nm), 2,6-dibromoquinonechlorimide (0.5%) in dimethylformamide
D62	Autoradiography using Kodak X-ray film and analyte was eluted with aqueous ethanol (50%), 10 ml toluene–Triton X-100 (2:1) + 2,5-diphenyloxazole (4 g/l) and 1,4-bis(5-phenyloxazdyl-2) benzene (0.1 g/l), radioactivity measured using Philips PW 4540 liquid scintillation spectrometer
D63	3,5-Dichloro-*p*-benzoquinonechlorimine
D64	Fluorescence quenching or UV
D65	Scanning or autoradiography
D66	Silver nitrate–2-phenoxyethanol, 4-(4-nitrobenzyl)pyridine (NBP), 2,6-dibromobenzoquinone-N-chloroimine (DBBC) and its analogue
D67	Reflectance densitometry
D68	Flame ionization detection
D69	Spectrodensitometry
D70	UV light
D71	Phosphomolybdic acid
D72	GC
D73	Enzyme (mouse liver), then substrate containing β-naphthyl ethyl ester
D74	Palladium chloride or dibromo-4-chlorimide, 4-(4-nitrobenzyl)pyridine reagent
D75	Ammonium molybdate reagent
D76	Spectrophotometry
D77	Indoxyl acetate and human serum, exposed to 366-nm UV irradiation, fluorimetric scanning
D78	Zeiss ERI 65m spectrometer and integrator
D79	Enzyme (pig liver acetone powder), sprayed with 1-naphthylacetone solution followed by *p*-nitrobenzenediazoniumfluoroborate in acetone
D80	UV at 254 nm or 2,6-dichloroquinonechloroimide or indoplatinate reagent
D81	Inhibition of esterase, 1-thionaphthyl acetate and 2,2′-azo(1-naphthol-8-chloro-3,6-disulphonic acid) 4,4′-diphenyl disulphide, as post-chromatographic reagent
D82	Ammoniacal silver nitrate solution (0.5%) in aqueous acetone (1:3)
D83	*o*-Tolidine plug potassium iodide
D84	2-Methylthioacridone solution

TABLE 1 *(continued)*

Techniques, pesticides, etc.	
AMDTLC	Automated multiple development thin-layer chromatography
BPMC	2-*sec.*-Butylphenyl N-methylcarbamate (osbac)
C	Colorimetry
C_8	C_8 alkyl-bonded silica gel
C_{18}	C_{18} alkyl-bonded silica gel
2-D	Two-dimensional
2,4-D	2,4-Dichlorophenoxyacetic acid
DDD (mixed)	2,2-Bis(chlorophenyl)-1,1-dichloroethane and related compounds
o,p'-DDD (2,4'-DDD)	1-(*o*-Chlorophenyl)-1-(*p*-chlorophenyl)-2,2-dichloroethane
p,p'-DDD (4,4'-DDD)	2,2-Bis(*p*-chlorophenyl)-1,1-dichloroethane
o,p'-DDE (2,4'-DDE)	1-(*o*-Chlorophenyl)-1-(*p*-chlorophenyl)-2,2-dichloroethylene
p,p'-DDE	2,2-Bis(*p*-chlorophenyl)-1,1-dichloroethylene
DDT (mixed)	Dichlorodiphenyltrichloroethane (mixture of metabolites of *ca.* 80% *p,p'*- and 20% *o,p'*-)
o,p'-DDT (2,4-DDT)	1-(*o*-Chlorophenyl)-1-(*p*-chlorophenyl)-2,2,2-trichloroethane
p,p'-DDT (4,4'-DDT)	1,1-Bis(*p*-chlorophenyl)-2,2,2-trichloroethane
DNOC	2,6-Dinitro-*o*-cresol
DDP	Differential-pulse polarography
EI	Enzyme inhibition
EPTC	S-Ethyldipropylthiocarbamate
FID	Flame ionization detection
GC	Gas chromatography (gas–liquid chromatography)
α-HCH (α-BHC)	Hexachlorocyclohexane (α-isomer)
β-HCH	Hexachlorocyclohexane (β-isomer)
γ-HCH (lindane)	Hexachlorocyclohexane (γ-isomer)
HPLC	High-performance liquid chromatography
HPTLC	High-performance thin-layer chromatography
IAA	Indole-3-acetic acid
FC (IPC)	Isopropyl carbanilate
IPRPTLC	Ion-pair reversed-phase thin-layer chromatography
Llt	Lower limit of detection
MCPA	4-Chloro-2-methylphenoxyacetic acid (isooctyl ester)
MDTLC	Multiple-development thin-layer chromatography
MIPC	2-Isopropylphenyl-N-methylcarbamate (Isocarb)
MS	Mass spectrometry
β-NPA	β-Naphthaleneacetic acid
β-NPXA	β-Naphthoxyacetic acid
OC	Organochlorine
OP	Organophosphorus
OPTLC	Over-pressurized thin-layer chromatography
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PFB	Pentafluorobenzyl derivatives
ppb	Parts per billion (w/w)
ppm	Parts per million (w/w)
R_F	Migration distance relative to solvent front
RP	Reversed-phase
RTLC	Rod thin-layer chromatography
STLC	Sequential thin-layer chromatography
2,4,5-T	2,4,5-Trichlorophenoxyacetic acid
TCA	Trichloroacetic acid
TLC	Thin-layer chromatography
TGTLC	Temperature gradient (30–40°C) thin-layer chromatography
UV	Ultraviolet

TABLE 2

APPLICATION OF TLC TO PESTICIDE ANALYSIS

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Abate	Environmental water samples; RPTLC; determination	P45	S52	D50	89
Abate, actellic, anthionon, aphox, bromophos, chlorofos, cyanox, etaphos, heptrophos, malathion, parathion-methyl, phozalon, phthalophos, Rogor, ricid and trichlor-methaphos	HPTLC–EI, determination (Llt = 0.001–0.01 mg/)	P43	S82	D81	90
Abscisic acid	TLC; separation	P36	–	–	91
Abscisic acid	TLC; determination (Llt = 5 ng)	P28	S83	D5	92
Aearicides fungicides and insecticides	Fresh and processed apples; 2-DTLC; determination	P21	S37	D70/D31	93
Actellic and basudin(II)	Green, dry tobacco leaves; TLC–GC; determination (Llt = 0.5 μg, 0.1 mg/kg)	–	S44	D72	94
Acifluorfen, bifenox, chloroxuron, diphenoxuron, fluorodifeny, fomesafen, nitrofen and oxyfluorfen	Mixturex; STLC; separation	P32	S35	D69	95
Aldicarb, Baygon, bendiocarb, BPMC, carbaryl, carbofuran, MIPC, zineb and ziram	TLC; separation	P26	S64	–	96
Aldicarb, Baygon, carbaryl, carbofuran, lannate, mancozeb, thiram, zineb and ziram	*Post mortem* material; TLC; detection, separation (Llt = 0.5 μg)	P20	S65	D61	97
Aldicarb, carbaryl, diuron and propoxur	Fruits vegetables; HPTLC; detection	P20	S56	D54	98
Aldrin, α-BHC, dieldrin, heptachlor, heptachlor epoxide and methoxychlor	RPTLC; detection, determination	P11	S3	D4	99
Aminocarb and its major metabolites	TLC–EI, separation	P20	S22	D28	100
Amino acids, bile acids, hormones and pesticides	2-DTLC; detection	P18	–	–	101
Anilide, carbamate and urea herbicides	TLC; determination	P13	–	D15	102
Aphox	Air, water and soil; TLC–GC; determination (Llt = 0.5 μg, 0.5 mg/kg, 0.05 mg/m^3)	P40	S46	D32	103
Aromatic acid herbicides residues	Plant material; TLC; determination	P20	S24	D32	104
Arylcarbamates (aniline, asulam, bental, Carbyne, chlor-IFC, *m*-chloroaniline and IFC), phenylurea derivatives (3,4-dichloroaniline, diuron and dosanex), thiocarbamates (diptal, Eptam, roneet and yolan)	TLC–GLC; determination (Llt = 10 μg, 0.025 μg)	P4	S55	D53	105
Asulam and its degradation products	Soil; TLC; determination	P20	–	–	106
Asulam and its degradation products	HPTLC; determination (Llt = 2–200 ppb)	P20	–	D60	107
Asulam, sulphanilamide and sulphanilic acid	TLC; detection (Llt = 10 ng)	P28	S63	D27	108
Atrazine	Drinking water, ground water; TLC; determination (Llt = 20 ng)	P20	S29	D36	109

TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Atrazine, *p,p'*-DDD, *p,p'*-DDE, *o,p'*-DDT, *p,p'*-DDT, α-HCH, γ-HCH, metaphos, phosalone, phosphamide, prometryn and simazine	Environmental samples; 2-DTLC; GC; detection, determination	P40	S74	D72	110
Atrazine and simazine	Water and sewage; TLC; determination (Llt = 0.1 μg/dm^3)	P20	S31	–	111
Azinophos-ethyl, azinophos-methyl, coumaphos, diazinon, dimethoate, disulfoton, ethion, fenchlorphos, Malathion, oxydemeton-methyl, parathion, parathion-methyl and phorate	TLC; detection, separation, determination	P28	S43	D48	112
Azinophos-ethyl, diazinon, parathion-methyl and malathion	TLC; separation	P20	S14	D51	113
Bendiocarb	Workplace air; TLC; determination	P20	S17	D22	114
Benthiocarb, drepamon and yolan	Environmental samples; TLC; determination	–	–	–	115
Benzoate derivative of pentachlorophenol	TLC; detection	P37	–	D45	116
Benzoic acid, cinnamic acid, 2,4-D, IAA, β-NPA, β-NPXA, TCA and 2,4,5-T	STLC; separation	P7	S21	D31	117
Biphenyl residues, anilide carbamate and urea	Citrus; TLC; determination	P20	–	D15	118
Bolstar residues	Plants, soil and water; TLC–GC; detection, determination (Llt = 0.01 mg/kg)	P41	S47	–	119
4-Bromo-2,5-dichlorophenol, debromoleptophosoxon, leptophos, *o*-methylphenylphosphonothioate and phenylphosphoric acid	STLC; separation	P39	S5	D8	120
Bromophos residues	Peanut crops; TLC; detection	P20	S53	D52	121
Buturon, chlortoluron, diuron, fenuron, isoproturon, methabenzthiazuron, metonuron, monuron and neburon	TLC; separation	P16	S25	D15	122
Butylate, chlorfenvinphos, cycloate, *p,p'*-DDE, *p,p'*-DDT, dimethoate, EPTC, fenthion, lindane, mevinphos, molinate, parathion-methyl and trichlorphon	TLC; detection, determination	P14	S73	D66	123
Captafol, captan, Difolatan and folpet	Water, lettuce, apples; TLC; determination	–	D15	124	
Carbamates and OP residues	Fruits vegetables; TLC; detection, separation	–	–	–	125
Carbamates, phenylureas and triazines	Drinking water; HPTLC–AMD; determination	P12	–	D15	126
Carbaryl	TLC; detection (Llt = 1 μg)	P20	–	D24	127
Carbaryl residues	Apples; TLC; determination	P20	S61	–	128
Carbaryl	TLC, detection (Llt = 5 μg, 1 μg, respectively)	P21	–	D26	129
Carbaryl	Water; TLC; detection, determination (Llt = 20 μg, 40–200 μg, respectively)	P21	S62	D59	130

(Continued on p. 280)

TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Carbaryl and related compounds	TLC; detection, determination	P21	S21	D59	131
Carbaryl, dichlorvos and malathion	TLC–EI; determination	P21	–	D9	132
Carbendazin	TLC; separation, semi-quantitation	P21	–	–	133
Carbofuran	Fresh water, plant tissues, soil; TLC; separation	P20	–	D21	134
Carbofuran and quinalphos	Air; TLC; determination	P20	–	–	135
Carboxylic acid herbicides	TLC, 2-D TLC; separation	P7	S21	D31	136–138
Carboxylic acid herbicides	TLC; determination	P1	S69	D31	139, 140
Carboxylic acid herbicides	TLC; separation	P2	S21	D31	141
Carboxylic acid herbicides	TLC; separation	P40/P27	S23	–	142
Carboxylic acid plant growth regulators	TLC; separation	P6	S21	D31	143
Chlorbromuron, chloroxuron, linuron, methoxuron, metribuzine, prometryne, simazine, terbuthylazine and terbutryne	TLC; detection, determination	P40	S75	D42	144
Chlorocaragard, metazine and methoxy-caragard herbicides	Air; TLC; determination	P40	–	D72	145
Chlorocholine chloride (plant growth regulator)	Grain, grain products; TLC; determination (Llt = 0.1 mg/kg)	P8	S34	D39	146
Chlorophenols	Water;TLC; detection	P20/P40	–	–	147
Chlorophenoxy acid herbicides (2,4-D, 2,4,5-T) and triazines	TLC; determination	P17	S38	–	148
Chloropyrifos and its metabolites	TLC; determination	P20	–	D5	149
Chlorpropham	Onions; TLC; determination	–	–	–	150
Curacron or selecron residues	Environmental samples; TLC; determination (Llt = 1–2 μg)	P40	S46	–	151
Cyanox	Plant samples; TLC; determination	–	–	–	152
Diazinone, dimethoate, ethion, malathion, parathion and parathion-methyl	TLC; detection, determination (Llt = 0.03 μg)	P35	S8	D12	153
Diazinone, eptam, γ-HCH, lenacil, phen-medipham and phosphamide	TLC, 2-D TLC; identification, separation (Llt = 0.5–1 μg)	P41	S84	–	154
Dicarboximide fungicides, iprodion, procymi-done and vinclozolin	Drinking water; HPTLC; determination	P12/P35	–	D34	155
2,4-D, MCPA, 2,4,5-T and their PFB bromide derivatives	TLC; separation	P35/P5	S68	D63	156
DDT	TLC; detection	P20	–	D6	157
p,p'-DDT and dieldrin	Soil; TLC; determination	P3	–	–	158
DDD, DDE, DDT, β-HCH, lindane and methoxychlor	TLC; detection (Llt = 0.20, 0.20, 0.20, 0.20, 0.25 and 1.00 μg, respectively)	P35	S1	–	159
p,p'-DDE, *p,p'*-DDT, methoxychlor and parathion	Spiked samples; TLC; separation	P20	S78	–	160
Dichlorvos, dimethoate, malathion and phosphamidon	TLC; mobility	P44	S86	D82	161
Dicrotophos, ethion, fensulfothion, oxydeme-ton-methyl, phorate, phosmet, phospholan and trichlorfon	TLC; detection, separation	P20	–	D3	162
Dieldrin derivatives	TLC–GC; identification	P20	–	D72	163
Diflubenzuron urea herbicide	TLC; determination (Llt = 0.1 μg per spot, 2 ng/g)	P42	–	D3	164

TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Dimethoate, dimethoate oxygen analogue, dioxathion, disulfoton, fonogos, fonogos oxygen analogue and oxydemetonmethyl	STLC; separation	P20	–	–	165
DNOC	Urine; TLC; identification	P20	–	–	166
DNOC and dinoseb	Water; TLC–DPP; determination	P20	–	D46	167
Disulphoton, disulphoton oxygen analogue, fenthion, fenthion oxygen analogue, phorate and phorate oxygen analogue	TLC; detection, determination	P28	S49	D48	168
Disuphoton, monocrotophos and quinalphos	TLC; identification, determination (Llt = 10 μg)	–	–	D14	169
O-Ethyl-O-4-ntrophenyl phenylphosphonothioate and related compounds	STLC; separation	P39	S6	D8	170
Endrin	TLC; detection	P20	–	D1	171
Endosulphan	Biological materials in forensic toxicology; TLC; detection (Llt = 1 μg per spot)	P21	S44	D44	172
Fenitrothion	Water; TLC–EI; detection, determination	P21	S41	D79	173
Fenitrothion	TLC–EI; determination	–	–	D9	174
Fenitrothion and parathion	TLC; detection, determination	–	–	–	175
Fluchloralin, formothion, malathion and thiometon	TLC; mobility	P44	S86	D82	176
Flurecol	Formulations; TLC; detection, separation	P20	–	D15	177
Flucythriate insecticide residues	TLC; separation	P42	S39	D70	178
Flungicides	TLC; detection	P20	–	D41	179
Furadan and its metabolites	Biological materials (blood, urine); TLC; determination (Llt = 0.5 μg)	P40	–	D57	180
Furadan and its metabolites	Air; TLC; determination	P20	S59	D58	181
Gammacarbatox (carbaryl–lindane mixture)	Potatoes; TLC–EI; detection (Llt = 0.1 ppm, 1, pbb, respectively)	P20	S60	D25	182
Gardona (tetrachlorvinphos) residues	Apples; TLC; determination	P20	S50	D77	183
Gardona residues	Water, fish tissues; TLC; determination (Llt = 0.01 mg/l, 0.1 mg/kg)	P41	S54	–	184
Gibberelin A_3 and $A_4 + A_7$	Fermentation broths; HPTLC; determination	P35	S67	D15	185
Glyphosate and its metabolite (aminoethylphosphonic acid)	TLC; determination	–	–	–	186
Glyphosate and its metabolite (aminoethylphosphonic acid)	Water; TLC; determination	P8	–	–	187
Halogenated synthetic pyrethroid insecticide, *cis* and *trans* isomers of permethrin and cypermethrin from fenvalerate and decamethrin	Fruits, plants, soil and tomato; TLC; detection (Llt = 0.10 mg/l)	P40	S4	D5	188
Hazardous phenols	TLC; separation	P21	S36	D43	189
Herbicides	TLC; detection	P20	–	D29	190
Herbicides	TLC; separation	P31	–	D67	191

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TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Herbidice residues	TLC; identification	P20	–	D15	192
Herbicide residues	Agricultural crops, food, soil and water; TLC; determination (Llt = 1–10 μg/kg)	P20	–	D29	193
Herbicides	Sugar beet and sugar; TLC; determination	P20	–	–	194
Herbicides and related compounds	RTLC; determination	P9	S76	D68	195
Hexazinone metabolites	Rat-liver microcosmes, peanut seedlings, sugarcane; TLC–MS; determination	–	–	D11	196
Imidan and its degradation products, N-hydroxymethylphthalimide imidoxon, phthalic acid and phalmic acid	TLC; separation	P22	S9	D8	197
Indole-3-acetic acid	TLC; detection	P20	S66	D62	198
Indol-3-acetic acid	TLC; detection (Llt = 5–100 ng per spot)	P20	–	D30	199
Ioxynil residues	Animal tissues; TLC; determination (Llt = 0.1 ppm)	P29	–	–	200
Isouron and its metabolite	TLC; determination (Llt = 0.25–1.5 μg per spot)	P21	S71	D34	201
MCPA and its soil metabolites	TLC; detection, separation	P31	S80	D63	202
MCPA and its two metabolites	Soil, water; TLC; determination	P35	S72	D25	203
MCPA and terbacil	Apples; TLC–GC; determination	P20	–	D15/D47	204
Mecarbam and its degradation products	Crops; TLC–GC; determination	–	–	–	205
Mephosfolan, phosfolan and related compounds	TLC; separation	–	–	–	206
Methamidophos	Potato tubers and foliage; STLC; detection, separation	P40	S13	D19	207
Methamidophos	TLC–EI; detection, identification (Llt = 15 ng)	P20	S51	D49	208
Methanearsonic acid	Rice, soil; TLC; detection, separation	–	–	–	209
Methidathion and methoxychlor	Clinical samples; TLC; RPTLC; identification	P20	–	–	210
Methomyl	Serum and urine; RTLC; determination	P20	–	D68	211
Methomyl, parathion, parathion-methyl and sumithon	TLC; separation, determination	–	S12	D76	212
Methoxuron and its breakdown product (3-chloro-4-methoxyaniline)	Potato, soil and water; TLC; HPLC; determination (Llt = 0.02, 0.2, 0.001 mg/kg, respectively)	P45/P28	S70	D33	213
Methylnitrophos and its metabolites (fenitrooxon and *p*-nitrocresol)	Grain; TLC; detection, determination (Llt = 0.1, 0.1, 0.005 mg/kg, respectively)	P26	–	D17	214
Monocrotophos	TLC; detection	P21	S44	D10	215
OC	HPTLC; separation	P20	–	–	216
OC	TLC; detection (Llt = 0.20 μg)	P20	–	D2	217

TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
OC	TLC–GC; identification, determination	P20	–	–	218
OC	TLC; separation	P20	S2	D3	219
OC	TLC; detection	P20	–	D7	220
OC, petroleum distillates, PCBs, phenols and explosives	TLC; determination	P20	–	–	221
OC and OP	Animal tissues; TLC–EI; determination	–	–	D16	222, 223
OP and its metabolites	TLC–MS; detection, determination	P15	–	D11	224
OP residues	Dried fruits; TLC; determination, separation	P20	–	–	225
OP	HPTLC; determination	P35	S7	D74	226
OP	Vegetables and human blood; TLC; determination	P24	S48	–	227
OP (warfare agents)	2-D OPTLC; separation	P20	–	D15	228
OP	TLC; separation	P20/P3	S11	–	229
OP	Water; HPTLC; separation, identification	P20	–	D84	230
OP residues	TLC; detection (Llt = 100 ng)	–	–	D75	231
OP (sulphur-containing OP)	TLC; detection (Llt = 1 μg)	–	–	D10	232
OP residues	Vegetables and toxicological investigation; TLC; detection, separation	–	–	–	233, 234
OP	TLC; separation	P25	–	–	235
OP	Sewage sludge and drinking water; TLC–GC; R_F values	–	S16	–	236
Organotin compounds	HPTLC; determination	P20	–	–	237
Paraquat	Marijuana; TLC; determination	P20	–	D40	238
Paraquat	Plasma, urine; RTLC; determination	P10/P34	–	D68	239
Paraquat	Biological samples (blood, urine and tissue samples); TLC; determination	P38	S77	D39	240
Paraquat and related compounds	MDTLC; separation	–	–	–	241
Parathion	Crops; TLC–C; detection, determination	–	–	D18	242
Parathion and its metabolite (paraoxon)	Crops (rice); TLC–EI; detection, determination	P28	S42	D9	243, 244
Parathion residues and its metabolite (paraoxon)	Crops; TLC–EI; determination (Llt = 10^{-10} g)	P28	S79	D73	245
Pesticides (crop protection agents)	Drinking water, ground water; AMDTLC; STLC; detection, determination	–	–	–	246
Pesticides	Drinking water, ground water, surface water at trace level; AMDTLC; detection, determination	P35	–	–	247
Pesticides (acidic, basic, neutral)	TLC; 2-D TLC; detection, separation (Llt = 0.5–2 μg)	P21	S85	–	248

(Continued on p. 284)

TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Pesticides (predominantly fungicides and insecticides)	Standard solutions; HPTLC; detection, determination	–	–	–	249
Pesticides	Multi-residue analysis to confirm GLC results; HPTLC; detection, determination	P35/P19	–	–	250
Pesticides	TLC; detection	P33	–	D70	251
Phenoxy acid herbicides	IPRPTLC; detection, separation (Llt = 20–60 μg)	P7	S21	D31	252
Phenoxycarboxylic acid herbicides and organic acids	Drinking water; TLC; determination	P20	S69	D15	253, 254
Phenylcarbamate residues	Carrots, potatoes, wine; TLC; determination (Llt = 0.1 ppm)	–	–	–	255, 256
Phenyltin fungicides	TLC; detection, separation (Llt = 0.01 ppm)	P20	S33	D38	257
Phenylureas	TLC; determination	–	–	–	258
Phosmethylan and its major metabolite	Crops, meat, milk, soil, water; TLC; detection (Llt = 500 ng)	P20	S87	D13/D83	259
Propamocarb	Peppers; TLC; determination	P21	S57	D55	260
Pyramine herbicide residues	Soil; TLC; detection, separation (Llt = 0.02 ppm)	–	–	–	261
Pyrazophos	Plant products; TLC; separation, determination	–	–	D76	262
Radiolabelled pesticides	Soil surface; TLC; mobility	P20/P44	–	D65	263
Rodenticides and vitamins	TLC; separation (Llt = 1–8 μg)	P20	–	D71	264
Semicarbazone herbicides	TLC; RPTLC; separation	P20	S19/S20	D23	265
Tebuthiuron and related compounds	TLC; mobility	–	–	–	266
Terbufos and its four oxidative metabolites	TLC; separation	P35	S15	D20	267
Thiabendazole	Fruit, peel; TLC; determination	P20	–	D37	268
N,N′-Bis(1,3,4-thiadiazol-2-yl)methanediamine residues	Rice; TLC; determination	P20	–	–	269
Thiocarbamate herbicides	Foods; TLC; determination	P20	–	–	270
Thiocarbamate herbicides and their sulphoxide and silufon metabolites	TLC; detection	P40	S58	D56	271
Thiocarbamic acid	TLC; separation, detection (Llt = 0.01–0.02 mg/l)	P40	S18	D14	272
2-Thiouracil, 4(6)-methyl-2-thiouracil and 6(4)-propyl-2-thiouracil	Feed additives, biological materials; TLC; separation, identification	P28	S81	D80	273
Toxaphene	Bee honey; TLC; determination (Llt = 0–100 μg per spot)	P20	–	D78	274
Toxaphene	Surface water, standard solutions, water; TLC; determination (Llt = 3 μg)	P20	S40	D3	275
Triazine residues	Water; TLC; detection, determination (Llt = 10 ppb)	P20	–	D32	276
Triazines	TLC; separation	P20	S26	D35	277
Triazines	TLC; determination	P28	S32	D64	278
Triazine herbicides	TLC; separation	P40/P28	S27/S28	D70	279

TABLE 2 *(continued)*

Pesticide(s) studied	Sample; comments	Stationary phase	Mobile phase	Detection	Ref.
Triazine herbicides (degradation product)	TLC; separation	–	–	–	280
Triazine herbicides	TLC; R_F values	P30	–	–	281
Triazine herbicides	Milk; OPTLC; determination (Llt = 5 μg/kg, 10 μg/kg)	P20	S30	–	282
Triazine derivatives	RPTLC; detection, separation	P29	–	–	283
Triazine derivatives	TLC; determination	P10	–	–	284
Triazine and urea herbicides	Water; TLC; determination	P20	–	D29	285
Trichlorometaphos-3 residues	Grain; TLC; determination (Llt = 1–20 μg)	P20	S45	D3	286
Urea and carbamate herbicides	Drugs; TLC; separation	–	–	–	287
Urea herbicides	Drinking water; determination	P20/P11/P3	–	D64	288
Valexon residues	Grains, vegetables; 2-D TLC; determination	P23	S10	D14	289
Zineb	Foliage; TLC; determination	P20	–	–	290

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