

Handbook H

Page	CIPAC No.	Active	Formulations	Extraction	Filtration	Technique	Column	Column type	Mobile Phase	I.S.	Fit for purpose	Comments
5	338	Acephate	TC/SL	DCM	no	GC-FID	SP 2401 or equivalent	Packed Column	Nitrogen	Diisobutyl Phthalate	?	Use of chlorinated solvent. GC packed column - obsolete
10	215	Aldicarb	TC/GR	DCM	no	i.r.	n/a	n/a	n/a	n/a	?	Using the carbamate peak at 1740cm ⁻¹ with solvent compensation. Use of chlorinated solvent - obsolete IR method
14	454	α-Cypermethrin	TC/WP/EC/SC/UL	THF/5% Citric Acid	no	GC-FID	DB-1	Capillary column	He	Diethyl Phthalate	✓	Method uses He as make up gas, given supply problems suggest change to N2
22	133	Ametryn	TC/WP	CHCl ₃	yes	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dieldrin	?	Use of Chloroform not ideal. GC packed column method - obsolete
26	240	Asulam	TC/SL/SG	Acetonitrile	no	HPLC-UV - 270nm	Sperisorb 5 ODS	RP	Dihydrogen Phosphate buffer / ACN	4-Methoxybenzyl alcohol	✓	
33	91	Atrazine	TC/WP/WG	CHCl ₃	yes	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dieldrin	?	GC packed column - obsolete
38	501	Benfuracarb	TC/GR/EC	Acetonitrile	no	HPLC-UV - 280nm	Zorbax C ₁₈	RP		Nonaphenone	✓	
43	482	β-Cyfluthrin	TC/EC/SC	Tetra ButylMethyl Ether (TBME) / n-Heptane	no	HPLC-UV - 235nm	LiChrospher Si 60	NP	n-Heptane / TBME	none	✓	
52	203	Bioallethrin	TC/SL/EC	Acetone	no	GC-FID	OV-1	Packed Column	N ₂	Diisobutyl Phthalate	?	GC packed column - obsolete
57	87.407	Bromoxynil Heptanoate	TC/EC	Acetone	no	GC-FID	OV-101	Packed Column	N ₂	Diphenyl Phthalate	?	GC packed column - obsolete
61	263	Carbendazim	TC/WP/WG	Aqueous HCl	no	HPLC- 282nm	Partisil 10 ODS	RP	MeOH / Aqueous H ₂ SO ₄	none	✓	
67	88	Chlorfenvinphos	TC/WP/EC/GR/DP/X	Acetone	no	GC-FID	OV 225	Packed Column	N ₂	Di-n-Butyl Phthalate	?	E and Z isomers quantitated separately and summed to give the overall content. GC packed column - obsolete
76	111	Chloridazon	WG									
77	143	Chlormequat Chloride	TK/SL	Water	no	HPLC-Cond Det	Zorbax SCX	Cation exchange	Oxalic acid dihydrate / Ethylene Diamine / H ₂ O / Acetone	none	?	Could be a candidate for renewal with RP or HILIC type column
81	119	Chlorbenzilate	TC/WP	Acetone	no	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dibenzyl succinate	?	GC packed column - obsolete
85	120	Chloropropylate	TC/WP/EC	Acetone	no	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dibenzyl succinate	?	GC packed column - obsolete
89	391	Chlorsulfuron	TC/WG	Acetonitrile	yes	HPLC-UV	Zorbax SC C ₁₈	RP	ACN / H ₂ O pH = 3	Phenyl Sulfone	✓	
96	44 + 34	Copper compounds + Mancozeb	WP	HCl	no	Titration	n/a	n/a	n/a	n/a	?	copper -CIPAC E p.42- electrogravimetry or volumetric method works very well. The iodometric titration method for mancozeb is time consuming but this method is the only one which CIPAC has.
100	44 + 61	Copper compounds + Maneb	WP	HCl	no	Titration	n/a	n/a	n/a	n/a	?	copper -CIPAC E p.42- electrogravimetry or volumetric method works very well. The iodometric titration method for maneb is the same as for mancozeb
100	44 + 25	Copper compounds + Zineb	WP	HCl	no	Titration	n/a	n/a	n/a	n/a	?	copper -CIPAC E p.42- electrogravimetry or volumetric method works very well. The iodometric titration method for maneb is the same as for mancozeb
101	44 + 31	Copper compounds + Ziram	WP	HCl	no	Titration	n/a	n/a	n/a	n/a	?	copper -CIPAC E p.42- electrogravimetry or volumetric method works very well. The iodometric titration method for ziram is the same as for mancozeb
102	214	Cycloate	TC/EC/GR	CS ₂ /CHCl ₃ /MeOH	no	GC-FID	SE 30 or OV 1	Packed Column	N ₂	Pebulate	?	Poor choice of solvent. GC packed column - obsolete
106	385	Cyfluthrin	TC/WP/EC/EW	TBME / n-Heptane	no	HPLC-UV - 235nm	LiChrospher Si 60	NP	n-Heptane / TBME	none	✓	
122	15	Diazinon	TC/WP/EC/SL/GR	Acetone	yes	GC-FID	10% DC 200	Packed Column	N ₂ or He	Aldrin	?	GC packed column - obsolete
127	85	Dicamba	TC	CS ₂	no	i.r.	n/a	n/a	n/a	n/a	?	obsolete IR method, CIPAC K, p.32 HPLC method works well
		Dicamba + Dimethylamine (DMA)	SL	Acetone	no	i.r.	n/a	n/a	n/a	n/a	?	obsolete IR method , CIPAC K, p.32 HPLC method works well

		Dicamba + MCPA DMA	SL	HCl/CHCl ₃ /Acetone	no	i.r.	n/a	n/a	n/a	n/a	?	Use of Chloroform. obsolete IR method , CIPAC K, p.32 HPLC method works well
		Dicamba + 2,4-D DMA	SL	Water	no	i.r.	n/a	n/a	n/a	n/a	?	obsolete IR method , CIPAC K, p.32 HPLC method works well
135	11	Dichlorvos	TC/EC/SL/HN	Acetone	no	GC-FID	OV-225	Packed Column	N2	Diethyl Pimelate	?	GC packed column - obsolete
141	339	Diflufenuron	TK/WP	Dioxane/Acetonitrile	no	HPLC-UV - 254nm	Zorbax TM ₁₀₀ -C ₈ or Spherisorb RP ODS or Bondapak C ₁₈	Packed Column	ACN/H ₂ O/Dioxane	Linuron	√	
147	462	Diflufenican	TC/SC	Acetonitrile	no	HPLC-UV - 280nm	Spherisorb ODS	RP	H ₂ O/ACN/MeOH	Fluoranthrene	√	
153	59	Dimethoate	TC/SL/EC	Acetone	no	GC-FID	Chromosorb G	Packed Column	N2	Di-n-Butyl Phthalate	?	GC packed column - obsolete
161	55	Diquat salt - free bipyridyl	SL	CHCl ₃ /HCl/MeOH	no	GC-FID	Sodium Dodecylbenzene sulphate	Packed Column	N2	5,6-Benzoquinone	?	Chloroform not an ideal solvent. GC packed column - obsolete
	55 + 56	Diquat + Paraquat - free bipyridyl	SL/SG	CHCl ₃ /HCl/MeOH	no	GC-FID	Sodium Dodecylbenzene sulphate	Packed Column	N2	5,6-Benzoquinone	?	Chloroform not an ideal solvent. GC packed column - obsolete
165	373	Ethephon	TK/SL	Water	no	Titration	n/a	n/a	n/a	n/a	?	Non specific method - obsolete
171	435	Flusilazole	TC/WG/EC/EW	Acetonitrile	no	GC-FID	HP-17 or equivalent	Capillary column	He	Benzophenone	?	
178	160	Formothion	TC/EC	Toluene/Acetic Anhydride	no	GC-FID	Chromosorb W-HP	Packed Column	N2	Ethion	?	GC packed column - obsolete
182	284	Glyphosate	SG	Potassium Dihydrogen Phosphate/H ₂ O/MeOH/Phosphoric acid	no	HPLC-UV - 195	Anion exchange resin	IEC	Potassium Dihydrogen Phosphate/H ₂ O/MeOH/Phosphoric acid	none	√	
185	582	Imidacloprid	TC/WP/SC/GR/XX	Acetonitrile/Methanol	no	HPLC-UV	Octadecyl silane bonded silica gel	RP	ACN/H ₂ O	Propiophenone	√	
194	34	Mancozeb	TC/WP/WG/SC/DP	Water	no	EDTA Titration	n/a	n/a	n/a	n/a	?	the same iodometric titration method as for maneb CIPAC E p.116
199	264	Methomyl	TC/WP/SL/UL	Methanol/Acetonitrile	no	HPLC-UV - 254nm	Zorbax ODS	RP	ACN/H ₂ O	Benzamide	√	
204	441	Metsulfuron-methyl	TC/WG	Acetonitrile	no	HPLC-UV - 254nm	Zorbax SD C ₈	RP	ACN/H ₂ O	Phenyl Sulfone	√	
212	45	Mevinphos	TC/EC	Methanol	no	HPLC-UV - 245nm	Lichrosorb RP ₁₈	RP	H ₂ O/MeOH	none	√	
216	56	Paraquat salt - free 4,4-bipyridyl	SL	CHCl ₃ /HCl/MeOH	no	GC-FID	Sodium Dodecylbenzene sulphate	Packed Column	N ₂	5,6-Benzoquinone	?	GC packed column - obsolete
217	231	Pirimicarb	TC/WP/WG/EC/FU	CHCl ₃	no	GC-FID	SE-30	Packed Column	N ₂	n-Nonadecane	√	GC packed column - obsolete
222	461	Profenophos	TC/EC	Acetone	no	GC-FID	OV-210	Packed Column	N ₂	Di-(2-ethylhexyl) adipate	?	GC packed column - obsolete
		4,4-Bromo-2-chlorophenol	Impurity in TC	DCM	no	GC-FID	OV1701	Capillary column	H ₂	Lindane	√	
228	93	Prometryn	TC/WP	CHCl ₃	no	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dieldrin	?	GC packed column - obsolete
232	92	Propazine	TC/WP	CHCl ₃	no	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dieldrin	?	GC packed column - obsolete
236	177	Propineb	TC/WP/DP	Water	no	EDTA Titration	n/a	n/a	n/a	n/a	?	the same iodometric titration method as for maneb CIPAC E p.116
239	32 + 33	Pyrethrum + Piperonyl Butoxide	TK/AK	Propan-2-ol	no	GC-FID	Durabond-1	Capillary column	He	Hexadecane + Octadecane	√	
244	493	Quinclorac	TC/WP/WG/SC	THF/H ₂ SO ₄	yes	HPLC-UV-238nm	Spherisorb ODS	RP	THF/H ₂ SO ₄ /H ₂ O	NONE	√	
250	22	Simazine	TC/WP/GR	DMF	yes	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Di-(2-ethylhexyl) Phthalate	?	Poor choice of solvent. GC packed column - obsolete
255	198	Sulfotep	TC/FD	1,2-Dichloroethane	no	GC-FID	SE 52 on Chromosorb G-AW DMCS	Packed Column	He	Dimethyl Phthalate	?	Chlorinated solvent. GC packed column - obsolete
261	494	Tebuconazole	TC/WG/EW/SC/FS/ES	Acetone	no	GC-FID	Fused silica coated with methyl silicone	Capillary column	He	Dicyclohexyl Phthalate	√	
269	459	Terbufos	TC/GR/XX	Methanol	no	GC-FID	DB-1	Packed or Capillary column	N ₂	Dipropyl Phthalate	√	
275	212	Terbutryn	TC/WP	CHCl ₃	no	GC-FID	Carbowax 20M	Packed Column	N ₂ or He	Dieldrin	?	Chlorinated solvent. GC packed column - obsolete
279	543	Thiodicarb	TC/WP/WG/SC	DCM/Methanol	no	HPLC-UV - 254nm	LiChrospher C ₈ ODS	RP	H ₂ O/MeOH	Dimethyl Phthalate	√	
285	115	Thiometon	TC/EC	CS ₂	no	i.r.	n/a	n/a	n/a	n/a	?	Obsolete IR method
288	353	Triazophos	TK/EC	Toluene/isoctane/Water	no	HPLC-UV - 254nm	HIBAR	NP	Iso-octane/Dioxane/Water	Monolinuron	√	
292	183	Trifluralin	TC/EC/GR	Acetone	no	GC-FID	5% dc 200 on Chromosorb W-HP	Packed Column	N ₂	Diisobutyl Phthalate	?	GC packed column - obsolete
299	360	Triforine	EC	Methanol	no	HPLC-UV - 220nm	LiChrosorb RP-8	RP	ACN/H ₂ O	none	√	

Miscellaneous Techniques

Page	Comments
302 MT 18 Standard waters	MT 18.5 - temperature 105°C for drying calcium chloride is not enough. Anhydrous calcium chloride is obtained after drying at higher temperature than 200°C. Magnesium chloride hexahydrate is hygroscopic.
304 MT 178 Attrition resistance of granules	This method, as published, was initially applicable to all Granular Formulations. It was later supplemented by MT 178.2 which is titled in such a way as to be specifically applicable to Dispersible Granules. It would appear that the original method (MT 178) would then only be applicable to Granules for Dry Application while the revised version (MT 178.2) should be applicable to other Granular Formulations.
307 MT 179 Degree of dissolution and solution stability	a. Change the Reagent from Standard Water C (MT 18.1.3) to Standard Water D (MT 18.1.4). b. Change the time intervals for Residue Calculation from 5 minutes and 18 hours to 5 minutes and 24 hours. This change would be consistent with a recent change made to CIPAC MT 41 (Dilution Stability of Aqueous Solutions) in which the revised version (MT 41.1) increased the standing time for recording any separated material from 18 to 24 hours.
310 MT 180 Dispersion stability of suspo-emulsions	Originally published for use with SE Formulations. The Scope was later extended to other Formulation types. The procedure as written requires testing at a 2% concentration. This is exactly the concentration tested in the collaborative trial many years ago. Note that the results of this test depend upon a visual assessment, and even at the 2% concentration, sediment and cream formation can be very difficult to see. The recommendation made by the PRAPEr Expert Group in 2007, advising that this test should be conducted at high and low concentrations recommended on the label is simply not practical. For example, imagine trying to assess the sediment in an OD Formulation containing 10% insoluble solids at a concentration of less than 1%. Recommendation: Conduct the test at a 2% concentration. In cases where the maximum use rate is greater than 2%, conduct the test at the maximum recommended concentration.
314 MT 181 Solubility in organic solvents	instead of 25°C is more convenient temperature 30±5°C Recommendation: Conduct the test at a 2% concentration. In cases where the maximum use rate is greater than 2%, conduct the test at the maximum recommended concentration.