

Table 401-a. Recovery of Chemicals Through Method 401 (E1-E2 + C1 + DL1)
(methanol extraction, cleanup with partitioning and charcoal/Celite column, HPLC with post-column derivatization and fluorescence detection)

Chemical	Recovery ¹	Rrt ²	ng ³	Notes
2,3,5-trimethacarb	C			
3,4,5-trimethacarb	C			
3-hydroxycarbofuran	C	0.6	10	
3-hydroxymethyl-2,5-dimethyl=phenyl methylcarbamate	P (70%)			
3-hydroxymethyl-4,5-dimethyl=phenyl methylcarbamate	C			
3-ketocarbofuran	V (67-110%)	0.85	11	
4-hydroxymethyl-3,5-dimethyl=phenyl methylcarbamate	C			
aldicarb	C	0.83	14	
aldicarb sulfoxide	P (50-60%)	0.33	9	
aldoxycarb	C	0.4	9	
bendiocarb	C	1	10	
bufencarb	C	1.44	19	Major peak is listed.
butocarboxim	C	0.75	15	
carbaryl	C	1.06	7	
carbofuran	C	1	10	
dioxacarb	C	0.67	15	
ethiofencarb	P (70-82%)	1.1	15	Breaks down to 2 peaks; other rrt 0.5.
fenobucarb	C	1.47	10	
isoprocarb	C	1.13	8	
methiocarb	C	1.26	10	
methiocarb sulfone	C	0.79	11	
methiocarb sulfoxide	C	0.64	12	
methomyl	C	0.46	10	32% recovery from peanuts.
metolcarb	C	0.85	10	
oxamyl	C	0.44	10	
promecarb	C	1.31	10	

¹ Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

² Retention time, relative to carbofuran, on C-8 column described in DL1.

³ ng that cause 50% full scale deflection detector response in DL1.

Table 401-a: Recovery Through Method 401 (E1-E2 + C1 + DL1)

Chemical	Recovery¹	Rrt²	ng³	Notes
propoxur	C	0.98	8	
thiodicarb	P (40-60%)	0.99	11	Recovery C if analytical breakdown product (methomyl) also measured.
XMC	C	1.06	10	

*Table 401-b: Recovery of Chemicals Through Method 401 (E1-E2 + C1 + DL2)
(methanol extraction, cleanup with partitioning and charcoal/Celite column, HPLC
with fluorescence detection)*

Chemical	Recovery¹	Rrt²	ng³	Notes⁴
carbaryl	C	1.06	3	Ex L 288, Em L 330.
carbofuran	C	1	90	Ex L 288, Em L 330.
CGA 161149	V (43-99%)	0.73	10	Ex L 288, Em L 330.
CGA 195654	S (15-132%)	0.57	300	Ex L 288, Em L 330.
dioxacarb	C	0.67	180	Ex L 265, Em L 294.
fluometuron	V (60-100%)	1.09	50	Ex L 288, Em L 330. Low level residues may be obscured by matrix interferences.
isoprocarb	C	1.13	370	Ex L 264, Em L 292.
naphthaleneacetamide	P (77%)	0.75	3	Ex L 288, Em L 320. For C rec., elute charcoal with additional 100 mL petr ether
napropamide	C	1.36	4	Ex L 288, Em L 330.
phosalone	C	1.7	90	Ex L 288, Em L 330.
phosalone oxygen analog	C	1.3	90	Ex L 288, Em L 330.
piperonyl butoxide	C	1.74	5	Ex 288, Em L 330.
propoxur	C	0.98	40	Ex L 276, Em L 300.

¹ Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

² Retention time, relative to carbofuran, on C-8 column described in DL1/DL2.

³ ng that cause 50% full scale deflection detector response in DL2.

⁴ Excitation (Ex) and emission (Em) wavelengths found optimum for the chemical.

Table 402-a: Recovery of Chemicals Through Method 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

(extraction from acidified mixture, GPC, methylation, and Florisil cleanup, determination by GLC)

Chemical	Recovery¹⁻³	Notes^{4,5}
2,3,5,6-tetrachloroterephthalic acid	E1: NR E2: NR	Methylated completely, but did not elute from GPC.
2,3,5-triiodobenzoic acid	E1: V (66-86%) E2: V (79-138%)	No ester reference standard.
2,3,6-TBA	E1: C E2: C	
2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	E1: NR E2: NR	Chemical did not methylate.
2,4,5-T	E1: P E2: P	79% mean recovery, 31% CV, n=270, nonfat and fat.
2,4-D	E1: P E2: P	72% mean recovery, 34% CV, n=186, nonfat and fat.
2,4-DB	E1: C E2: C	
2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	E1: NR E2: NR	Chemical did not methylate.
3,5,6-trichloro-2-pyridinol	NR	Some (<20%) recovered in 100mL ethyl ether.
3,5-dibromo-4-hydroxybenzoic acid	S (0-42%)	
3-carboxy-5-ethoxy-1,2,4-thiadiazole	NR	Methyl ester not eluted from Florisil column.
3-chlorosulfonamide acid	NR	Complete recovery from Florisil only, 14% from GPC.
3-methyl-4-nitrophenol		Methyl ether completely eluted from Florisil, but only 30% from GPC.

¹ Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

² Extraction module used during testing (*e.g.*, E1) is indicated with each result.

³ Florisil eluted with Eluant 1 and Eluant 2 only; chemicals eluted in ethyl ether (EE) are considered NR through basic method as normally performed.

⁴ Ester/ether elutes from Florisil with Eluant 2 unless otherwise noted.

⁵ When no reference material available for ester/ether, recoveries calculated against acid/phenol methylated per method.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery ¹⁻³	Notes ^{4,5}
4-chlorobenzoic acid	E1: S (27-66%) E2: S (2-76%)	Low temperature column needed to detect methyl ester.
4-CPA	E1: S (32-69%) E2: C	Chromatographs only on wide bore GLC. No ester reference standard.
6-chloropicolinic acid	NR	Methylates, but methyl ester does not elute from Florisil.
AC 263,222 ammonium salt	NR	Methyl ester not eluted from Florisil.
acifluorfen	E1: P (54-69%)	
aloxym-sodium	E1: NR E2: NR	Does not methylate.
arsanilic acid		Compound did not methylate under method conditions.
benazolin	E1: NR E2: NR	28-32% recovered if Florisil eluted with additional 100 mL EE. Complete recovery if Florisil eluted with additional 100 mL EE.
bifenox	E1: C E2: C	Parent is methyl ether.
bromacil	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
bromofenoxim	E1: P (57-86%) E2: C	No ether reference standard.
bromoxynil	E1: P (50-68%) E2: C	No ether reference standard.
chloramben	E1: S (40-43%) E2: P (49-59%)	
chloroxuron	E1: NR E2: NR	Does not methylate.
clofencet potassium salt	NR	Does not methylate.
cloprop	E1: P (50-66%) E2: C	Chromatographs only on wide bore GLC. No ester reference standard.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery ¹⁻³	Notes ^{4,5}
CP 106070	NR	Does not methylate.
CP 106077	NR	Some methylation, but does not elute from GPC.
CP 108064	E1: NR E2: NR	NR through method even in 100 mL EE; recovered from GPC only, Florisil only. Complete recovery if Florisil eluted with additional 100 mL EE.
CP 108669	NR	Some methylation, but does not elute from GPC.
CP 92429	NR	Does not methylate.
CP 95200	NR	Some methylation, but does not elute from GPC.
CP 97290	NR	Does not methylate.
cyclanilide	E1: C E2: V (45-67%)	
dicamba	E1: P (71-76%) E2: C	
dichlorprop	E1: C (80%) E2: C (72-104%)	No ester reference standard.
diclofop	E1: S (43-51%) E2: V (81-200%)	
dinoseb	E1: NR E2: NR	Does not methylate.
disul-Na	E1: NR E2: NR	Does not methylate; parent 50% recovered if Florisil eluted with 100 mL EE. Does not methylate; complete recovery of parent with 100 mL EE.
DNOC	E1: S (45-50%) E2: C	Nitrogen detector required. No ether reference standard.
dodine	E1: NR E2: NR	Does not methylate.
fenac	E1: C (74-92%) E2: C	No ester reference standard.
flumetsulam	E1: NR E2: NR	Methylated product not soluble in hexane.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery ¹⁻³	Notes ^{4,5}
fluroxypyr	E1: S (23-30%) E2: P (64-77%)	Two peaks result; 3-7% more eluted with 100 mL EE. No ester ref std. Two peaks result; complete recovery with 100 mL EE. No ester ref std.
haloxyfop	E2: P (54%)	Florisil elution with 100 mL EE not tested.
HOE-038182	E1: NR E2: S (30-41%)	Methylation was complete, but ester not recovered. Elution from Florisil only with eluant #2 + 100 mL EE.
HOE-099730	NR	Does not methylate.
imazamox	NR	Methyl ester not eluted from Florisil.
ioxynil	E1: C (80-87%) E2: C	No ether reference standard.
iprodione urea	NR	Methyl ether not eluted from Florisil.
MCPA	E1: C (78-89%) E2: C	
MCPB	E1: C (70-106%) E2: C	Chromatographs only on wide bore GLC. No ether reference standard.
mecoprop	E1: C (73-84%) E2: C	Wide bore GLC recommended. No ester reference standard.
PB-7	E1: NR E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
pentachlorophenol	E1: P E2: P	70% mean recovery, 31% CV, n=275, nonfat and fat.
picloram	E1: NR E2: NR	6-10% recovered if Florisil eluted with additional 100 mL EE. Complete recovery if Florisil eluted with additional 100 mL EE.
PPG-947	E1: P (49-78%)	Two peaks from methylation; only one seen by halogen detector.
pyrithiobac-sodium	E1: S (7-13%)	Additional 31-34% recovered if Florisil eluted with 100 ml EE.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery¹⁻³	Notes^{4,5}
RPA203328	NR	Small (0-34%) recovery in 100 mL ethyl ether.
silvex	E1: C E2: C	
triadimenol	E1: NR E2: NR	Methyl ether not eluted from Florisil.
triclopyr	E1: C E2: C	Recovery from fatty foods may be <50%.
vinclozolin metabolite B	E1: S (26-43%) E2: S (27-43%)	Methylated product is parent vinclozolin; 62% recovery through Florisil only.

Table 403-a: Recovery of Chemicals Through Method 403 (E1 + C1 + DL3 and DL4) (methanol extraction, cleanup by partitioning and Florisil chromatography, HPLC with post-column photolysis and derivatization, fluorescence detection)

Chemical	Recovery ¹	DL3: Methanol/ Water Mobile Phase		DL4: Acetonitrile/ Water Mobile Phase	
		Rrt ²	Notes ³	Rrt ²	Notes ³
chlorbromuron	C	1.16	LD 0.006, LQ 0.022	1.28	LD 0.003, LQ 0.011
chlorotoluron	C	0.87		0.91	
chloroxuron	C	1.25	LD 0.002, LQ 0.008	1.28	LD 0.001, LQ 0.003
diuron	C	1	LD 0.002, LQ 0.007	1	LD 0.001, LQ 0.003
fenuron	C	0.42		0.49	
fluometuron	C	0.87	LD 0.002, LQ 0.006	0.93	LD 0.001, LQ 0.003
isoproturon	C	0.96		1	
linuron	C	1.12	LD 0.004, LQ 0.014	1.23	LD 0.005, LQ 0.017
metobromuron	C	0.91	LD 0.004, LQ 0.015	1.04	LD 0.004, LQ 0.014
metoxuron	C	0.62		0.67	
monolinuron	C	0.91		0.99	
monuron	C	0.72		0.75	
neburon	C	1.34		1.43	
siduron	C	1.08		1.16	

¹ Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

² Retention time, relative to diuron, on the HPLC system described.

³ LD, limit of detection: concentration (ppm) of phenylurea found to cause response three times baseline noise; LQ, limit of quantitation: concentration (ppm) found to cause response 10 times baseline noise.

*Table 404-a: Recovery of Chemicals Through Method 404 (E1-E3 + DL5)
(methanol extraction, partitioning into methylene chloride, HPLC with ion pairing mobile phase and UV and fluorescence detection)*

Chemical	Recovery¹	Notes
allophanate	C	Determined by UV detector at 250 nm.
benomyl	C	Determined as MBC (carbendazim) by UV detector at 280 nm.
MBC ²	C	Determined by UV detector at 280 nm.
thiabendazole	C	Determined by UV detector at 280 nm; confirm, increase sensitivity with DL7.
thiophanate-methyl	C	Determined by UV at 280 nm; degrades in extract, must be determined quickly.

¹ Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

² Residue may result from use of: (1) benomyl, never found as a residue itself, because it is rapidly converted to MBC; (2) thiophanate-methyl, which degrades slowly to MBC; or (3) carbendazim, as MBC is called when used as a fungicide itself (not registered in the U.S.).

