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Project Summary

Evaluation of 10 Pesticide Methods

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Ten pesticide analysis methods were evaluated. The compounds listed in each method were analyzed in triplicate at two concentration levels in reagent water and POTW effluent. Each method was performed as written with only minor modifications. If a cleanup procedure was included in the analysis method, all analyses were performed with and without the cleanup step.

Resultant data reported included estimated detection limits (EDLs) in reagent water and recovery data from reagent water and POTW effluent for each compound. Suggestions for method improvements were included in the report where necessary.

This Project Summary was developed by EPA's Environmental Monitoring and Support Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Ten pesticide analysis methods were evaluated; a description of the methods and the compounds included in those methods are listed in Table 1. The compounds included in each method were analyzed in triplicate at two concentration levels in reagent water and POTW effluent. Each method was performed as written with only minor modifications as approved by the EPA Project Officer. If a cleanup procedure was included in the analysis method, all analyses were performed with and without the cleanup step.

Resultant data reported included estimated detection limits (EDLs) in reagent water and recovery data from reagent water and POTW effluent for each compound before and after the optional cleanup step. Suggestions for method improvements were offered where warranted.

Results

A summary of the EDL and recovery data obtained during the evaluation of the 10 pesticide methods is given in Table 2. Methods 641, 643, and 645 were found to yield acceptable results as written. Method 641.1 was not acceptable due to low recoveries of ethoxyquin, presumably due to adsorption of the ethoxyquin to particulate material prior to filtration of the sample for HPLC analysis. Method 642 was marginally acceptable; recoveries of biphenyl and o-phenylphenol were low, possibly due to the rigorous conditions needed to concentrate sample extracts. Method 632.1 was acceptable for two of the compounds, napropamide and propanil, but was not acceptable for the determination of carbaryl (carbaryl recoveries were low and dependent upon sample matrix and carbaryl concentration). Method 644 was not acceptable; picloram recoveries were low, presumably due to incomplete extraction of the picloram from the water matrix. The extraction and analysis portions of Method 614.1 were found to be acceptable for dioxathion, EPN, ethion, and terbufos. The silica gel cleanup procedure included in Method 614.1 was not acceptable; compound recoveries were decreased by 50 percent or more when the cleanup procedure was used. Method 646 was found to be acceptable for the determination of basalin and marginally acceptable for the determination of CDNB and dinocap. Method

Table 1. Pesticide Method Information

Method Number	Compound(s)	Extraction Method	Cleanup Method	Analysis Method
641	Thiabendazole	None	Filtration	HPLC-Fluorescence
641.1	Ethoxyquin	None	Filtration	HPLC-Fluorescence
642	Biphenyl	Separatory funnel	None	HPLC-UV
642	O-Phenylphenol	Separatory funnel	None	HPLC-UV
643	Bentazon	Separatory funnel	None	HPLC-UV
632.1	Carbaryl	Separatory funnel	None	HPLC-UV
632.1	Napropamide	Separatory funnel	None	HPLC-UV
632.1	Propanil	Separatory funnel	None	HPLC-UV
632.1	Vacor[a]	None	None	None
644	Picloram	Separatory funnel	Backextraction with base	HPLC-UV
614.1	Dioxathion	Separatory funnel	Silica gel	GC-NPD
614.1	EPN	Separatory funnel	Silica gel	GC-NPD
614.1	Ethion	Separatory funnel	Silica gel	GC-NPD
614.1	Terbufos	Separatory funnel	Silica gel	GC-NPD
645	Alachlor	Separatory funnel	Florisil	GC-NPD
645	Butachlor	Separatory funnel	Florisil	GC-NPD
645	Diphenamid	Separatory funnel	Florisil	GC-NPD
645	Lethane	Separatory funnel	Florisil	GC-NPD
645	Norflurazon	Separatory funnel	None	GC-NPD
645	Fluridone	Separatory funnel	None	GC-NPD
646	Basalin	Separatory funnel	Florisil	GC-ECD
646	CDNB	Separatory funnel	Florisil	GC-ECD
646	Dinocap	Separatory funnel	Florisil	GC-ECD
608.2	Chlorothalonil	Separatory funnel	Silica gel	GC-ECD
608.2	DCPA	Separatory funnel	Florisil	GC-ECD
608.2	Dichloran	Separatory funnel	Florisil	GC-ECD
608.2	Methoxychlor	Separatory funnel	Silica gel	GC-ECD
608.2	Permethrin	Separatory funnel	Silica gel	GC-ECD

[a]A standard of vacor could not be obtained; Method 5 was not evaluated for vacor.

608.2 was found to be acceptable for cis- and trans-permethrin, but was not acceptable for the determination of DCPA, methoxychlor, chlorothalonil, and dicloran. Recoveries of DCPA and methoxychlor were low and concentration and matrix dependent. Recoveries of chlorothalonil and dicloran could not be determined because of the presence of interferences.

Table 2. *Summary of Data from Evaluation of 10 Pesticide Analysis Methods*

Method Number	Compound	EDL, µg/L	Spike Level, µg/L		Recovery from Reagent Water, %[d]				Recovery from POTW Effluent[d]			
					Before Cleanup		After Cleanup		Before Cleanup		After Cleanup	
			Low	High	Low	High	Low	High	Low	High	Low	High
641	Thiabendazole	1.7	10	100	93 ± 3	81 ± 5	N.C.[a]	N.C.	96 ± 5	100 ± 2	N.C.	N.C.
641.1	Ethoxyquin	6.3	6.2	62	100 ± 32	82 ± 6	N.C.	N.C.	19 ± 14	58 ± 1	N.C.	N.C.
642	Biphenyl	0.04	2.5	25	74 ± 2	51 ± 11	N.C.	N.C.	56 ± 4	61 ± 1	N.C.	N.C.
642	O-Phenylphenol	0.01	5.0	50	73 ± 4	60 ± 5	N.C.	N.C.	69 ± 2	82 ± 15	N.C.	N.C.
643	Bentazon	1.1	10	100	92 ± 2	81 ± 10	N.C.	N.C.	94 ± 7	76 ± 2	N.C.	N.C.
632.1	Carbaryl	1.2	2.0	20	52 ± 10	83 ± 13	N.C.	N.C.	N.D.	28 ± 16	N.C.	N.C.
632.1	Napropamide	0.02	6.0	60	103 ± 2	102 ± 2	N.C.	N.C.	96 ± 7	94 ± 3	N.C.	N.C.
632.1	Propanil	0.3	0.2	2	79 ± 6	99 ± 3	N.C.	N.C.	85 ± 11	77 ± 7	N.C.	N.C.
644	Picloram	0.3	3.0	30	N.D.[b]	N.D.	N.D.	N.D.	52 ± 9	71 ± 0	58 ± 4	68 ± 3
614.1	Dioxathion	0.5	10	100	76 ± 10	78 ± 2	43 ± 7	58 ± 6	87 ± 15	91 ± 3	67 ± 6	74 ± 3
614.1	EPN	12	10	100	120 ± 6	120 ± 4	N.D.	65 ± 14	85 ± 2	110 ± 6	N.D.	62 ± 4
614.1	Ethion	0.3	10	100	120 ± 5	95 ± 2	54 ± 8	78 ± 11	94 ± 5	86 ± 5	59 ± 12	79 ± 3
614.1	Terbufos	0.02	10	100	90 ± 3	84 ± 1	N.D.	42 ± 13	94 ± 5	77 ± 3	57 ± 4	54 ± 8
645	Alachlor	0.2	10	100	96 ± 3	94 ± 2	96 ± 3	94 ± 3	109 ± 1	102 ± 1	105 ± 3	97 ± 3
645	Butachlor	0.2	10	100	96 ± 4	93 ± 1	95 ± 3	93 ± 2	103 ± 1	100 ± 1	104 ± 5	95 ± 2
645	Diphenamid	0.2	10	100	93 ± 6	94 ± 2	95 ± 2	97 ± 3	105 ± 1	103 ± 1	95 ± 3	94 ± 4
645	Lethane	0.1	10	100	93 ± 6	100 ± 1	97 ± 2	99 ± 4	120 ± 2	123 ± 1	108 ± 4	106 ± 3
645	Norflurazon	0.02	10	100	69 ± 10	92 ± 1	69 ± 6	60 ± 6	107 ± 1	108 ± 2	76 ± 11	65 ± 17
645	Fluridone	0.6	10	100	49 ± 16	81 ± 15	N.C.	N.C.	124 ± 6	111 ± 2	N.C.	N.C.
646	Basalin	0.0005	0.1	1	138 ± 12	113 ± 4	126 ± 8	109 ± 10	94 ± 6	113 ± 2	74 ± 3	108 ± 3
646	CDNB	0.0005	0.1	1	91 ± 8	69 ± 5	89 ± 6	71 ± 5	78 ± 2	71 ± 2	76 ± 4	70 ± 2
646	Dinocap	0.1	0.1	1	78 ± 7	77 ± 4	26 ± 3	72 ± 14	76 ± 40	72 ± 3	123 ± 53	80 ± 10
608.2	Chlorothalonil	0.001	0.02	0.2	I[c]	I	I	I	I	I	I	I
608.2	DCPA	0.003	0.02	0.2	128 ± 18	94 ± 11	73 ± 25	62 ± 5	N.D.	62 ± 5	71 ± 5	76 ± 2
608.2	Dicloran	0.002	0.01	0.1	I	I	I	I	I	I	I	I
608.2	Methoxychlor	0.4	0.1	1	57 ± 1	106 ± 12	37 ± 8	86 ± 12	47 ± 12	99 ± 2	40 ± 27	90 ± 14
608.2	Cis-permethrin	0.2	1.0	10	94 ± 9	91 ± 2	91 ± 6	128 ± 17	89 ± 8	85 ± 3	97 ± 14	98 ± 27
608.2	Trans-permethrin	0.2	1.0	10	111 ± 4	98 ± 3	83 ± 12	108 ± 13	85 ± 2	90 ± 2	41 ± 11	95 ± 29

[a]N.C. = No cleanup procedure included in this method.
[b]N.D. = Not detected.
[c]I = Presence of interferences precluded determination of compound in sample.
[d]Standard deviation is included.

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***Thomas A. Pressley** is the EPA Project Officer (see below).*
The complete report, entitled "Evaluation of 10 Pesticide Methods," (Order No. PB 85-238 608/AS; Cost: $14.50, subject to change) will be available only from:
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