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DEPARTMENT OF ECOLOGY

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TO: Carl Nuechterlein, David Knight, and Jim Jacobson, ERO

FROM: Art Johnson, EILS

SUBJECT: Pesticides Detected in the Walla Walla Drainage, April and June 1996

Summary

Pesticides were analyzed in water and sediment samples collected from six Walla Walla River tributaries and the lower main stem during April 22-23 and June 18-19, 1996 Water samples were analyzed for 162 pesticides, or breakdown products, that include most of the compounds currently or historically used in Washington Sediment analysis was limited to the eight chlorinated pesticides for which the Walla Walla River is listed as water quality limited (303d) Results showed relatively few instances where pesticides were a significant water quality concern

Twenty-two pesticides were detected at sub-parts per billion levels in the water samples Eighteen of these were herbicides, with 2,4,-D, bromacil, MCPA, and bromoxynil being found in 50 - 85% of the samples Although the detection frequency of herbicides appears high compared to other parts of the state, the concentrations were several orders of magnitude below levels considered toxic to aquatic life.

Four insecticides were detected, but each only once or twice Malathion exceeded EPA chronic water quality criteria once in Lower Mud Creek and once in Pine Creek The concentration in Pine Creek approached the LC-50 for some invertebrate species. Diazinon was detected at non-toxic levels in Yellowhawk and Mill creeks The historically-used insecticides aldrin and DDT exceeded state chronic standards once each in Yellowhawk and Dry Creek, respectively Aldrin is rarely reported in state waters and the concentration, 0.11 ug/L (ppb), was high compared to the 0.0019 ug/L standard

Low levels (generally less than 5 ug/Kg; ppb) of DDT, hexachlorobenzene, gamma BHC, and chlordane was detected in most of the sediment samples The limited data collected indicate Yellowhawk and Dry creeks may be the largest sources of these compounds to the Walla Walla main stem With the single exception for DDT mentioned above, none of these pesticides reached detectable levels in water.

Recommendations

1 Re-sample Yellowhawk Creek to verify aldrin contamination and, if found, investigate the source

2 Do more intensive water sampling for insecticides in Yellowhawk, Lower Mud, Dry, and Pine creeks to determine if these compounds are a chronic water quality problem Include sampling of Garrison Creek, pending final results and recommendations of the recent receiving water study by the Watershed Assessments Section (Rashin et al , in prep.)

Background

The Walla Walla River is on the 303d (water quality limited) list for DDT compounds, hexachlorobenzene, gamma BHC, chlordane, dieldrin, and heptachlor epoxide, due to their detection above EPA 10^{-6} human health criteria in fish fillets Only gamma BHC (lindane) continues to have a currently allowed use, and this is limited None of these pesticides is still in use The data for listing were collected in 1992-93 through Ecology's Washington State Pesticide Monitoring Program (WSPMP) (Davis and Johnson, 1994a; Davis et al , 1995) U S Fish and Wildlife Service reports show fish tissue concentrations of the herbicide DCPA (dacthal) have also been high historically (Schmitt et al , 1990) Trace amounts of DCPA and several other herbicides, including 2,4-D, bromacil, atrazine, and simazine, have been commonly detected in WSPMP water samples from the Walla Walla main stem (Davis, 1993; Davis and Johnson, 1994b)

Based on these findings, the Ecology "Needs Assessment" for the Lower Snake River Water Quality Management Area concluded that further pesticide monitoring should be done in the Walla Walla drainage (February 1996-draft) The Eastern Regional Office (ERO) subsequently requested that Environmental Investigations and Laboratory Services (EILS) obtain data on selected Walla Walla tributaries to determine pesticide levels and locations that may be contributing to the problem

Survey Description

Water and sediment samples were collected near the mouth of the Walla Walla River and at six of its major tributaries during the spring and early summer of 1996 (Figure 1) Sampling was done on April 22-23 (water) and June 18-19 (water and sediment) According to Larry Hooker of the Walla Walla Conservation District, April through June is the period of pesticide applications when most surface runoff occurs Specific sampling dates were selected so the chemical analysis could be done in conjunction with WSPMP statewide routine monitoring

Water samples were analyzed for 162 pesticides, or breakdown products, that include most of the compounds currently or historically used in Washington (Appendix A)

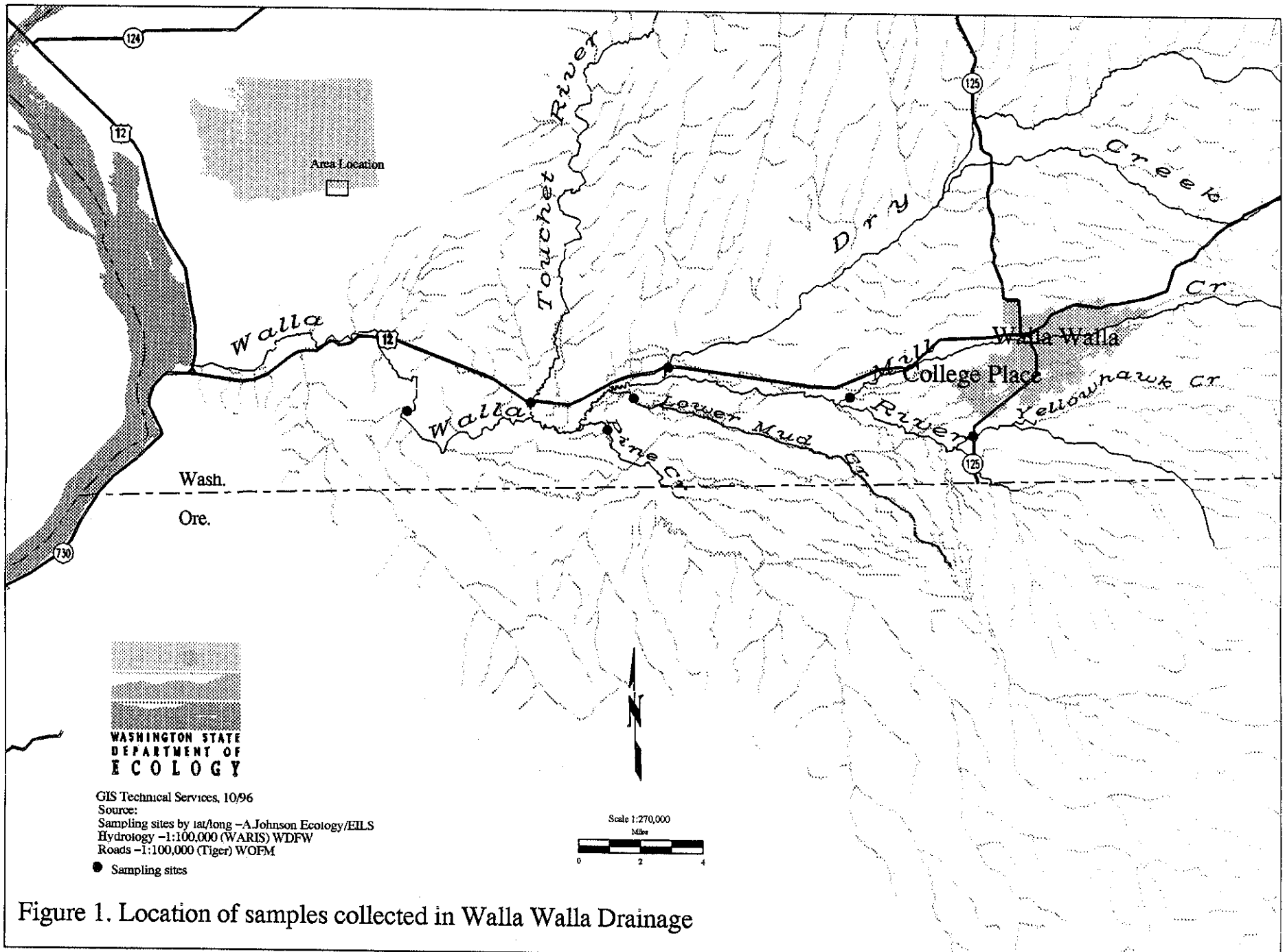

Figure 1. Location of samples collected in Walla Walla Drainage

Ancillary data were obtained on flow, temperature, conductivity, and total suspended solids. Sediment analysis was restricted to the chlorinated pesticides mentioned above as being elevated in fish tissue samples. Although these compounds were also analyzed in water, their affinity for particulates makes them easier to detect in the sediments. Total organic carbon (TOC) was determined for each sediment sample to use in comparing against TOC-based sediment criteria.

Methods

Sampling methods followed those used in the WSPMP (Davis, 1993). Briefly, water samples were collected as composites from quarter-point transects using USGS depth integrated samplers or by hand. Each sediment sample was a composite of multiple grabs taken with a stainless steel scoop and composited by stirring in stainless steel beakers. Equipment for sediment sampling was washed with Liquinox detergent and rinsed with deionized water and pesticide-grade acetone. Sample containers were glass with teflon lid liners, cleaned to EPA QA/QC specifications (EPA, 1990). All samples were placed on ice immediately on collection for transport to the Ecology/EPA Manchester Laboratory for analysis.

Water samples were analyzed as part of the WSPMP sample sets. Sediments were done separately using methods modified to improve detection limits for target compounds. Analytical procedures and data quality are described in case narratives prepared by Manchester (Appendix B). No significant problems were encountered in the analyses. Results for pentachlorophenol, detected in several of the water samples, were qualified as estimates because there was potential for cross-contamination at the laboratory.

Results and Discussion

Water

The results on water samples collected in April are summarized in Table 1, arranged by analytical group. Seventeen pesticides* were detected at one or more locations. Sixteen of these were herbicides and one an organophosphorus insecticide. Concentrations were less than 1 ug/L (part per billion) in all cases.

The largest number of herbicides were found in Dry Creek (13) and Yellowhawk Creek (10). Yellowhawk Creek also had the lone detection of an insecticide, diazinon. The fewest pesticides were in Mill Creek where only one herbicide was detected. The compounds seen most frequently were bromoxynil (all samples), MCPA and bromacil (6 of 7 samples), 2,4-D and diuron (5 of 7 samples). Diuron concentrations (0.13 - 0.56 ug/L) were an order of magnitude higher than most other pesticides.

*see Appendix C for trade names of pesticides detected

Table 1 Pesticides Detected in Water Samples Collected April 22-23, 1996 (ug/L)

Pesticide	Yellowhawk Creek	Mill Creek	Lower Mud Creek	Dry Creek	Pine Creek	Touchet River	Walla Walla River
			INSECTICIDES				
Organophosphorus							
diazinon	**0.006 J**	0 065 U	0 065 U	0 066 U	0 063 U	0 065 U	0 066 U
Organochlorine	nd	nd	nd	nd	nd	nd	nd
Carbamates	nd	nd	nd	nd	nd	nd	nd
Pyrethroids	nd	nd	nd	nd	nd	nd	nd
Propargite	0 16 U	0 16 U	0 16 U	0 16 U	0 16 U	0 16 U	0 16 U
			HERBICIDES				
Chlorophenoxy							
bromoxynil	**0.046**	**0.021 NJ**	**0.035 J**	**0.10**	**0.019 NJ**	**0.029 J**	**0.035 J**
MCPA	**0.063 J**	0 082 U	**0.041 J**	**0.049 J**	**0.024 NJ**	**0.044 J**	**0.032 J**
2,4-D	**0.11**	0 041 U	**0.057**	**0.047**	0 042 U	**0.019 J**	**0.033 J**
diclofop-methyl	**0.016 J**	0 061 U	0 061 U	**0.006 J**	0 063 U	0 060 U	0 060 U
pentachlorophenol	**0.017 NJ**	0 021 U	0 020 U	**0.011 NJ**	0 021 U	0 020 U	0 020 U
dicamba	0 040 U	0 041 U	0 041 U	**0.020 J**	0 042 U	0 040 U	0 040 U
DCPA	0 032 U	0 033 U	0 033 U	**0.014 J**	0 033 U	0 032 U	0 032 U
Nitrogen-containing							
bromacil	**0.007 J**	0 32 U	**0.012 J**	**0.021 J**	**0.019 J**	**0.068 J**	**0.049 J**
diuron	**0.50 J**	0 48 U	0 49 U	**0.56 J**	**0.20 NJ**	**0.13 NJ**	**0.16 NJ**
metribuzin	**0.048 J**	0 081 U	0 081 U	**0.049 J**	0 079 U	**0.008 J**	**0.013 J**
triallate	**0.058 J**	0 21 U	0 21 U	**0.033 J**	0 20 U	**0.007 J**	**0.008 J**
atrazine	0 080 U	0 081 U	**0.005 NJ**	**0.005 NJ**	**0.005 NJ**	0 081 U	0 082 U
pendimethalin	**0.006 J**	0 12 U	0 12 U	0 12 U	**0.015 J**	0 12 U	0 12 U
simazine	0 080 U	0 081 U	**0.016 J**	0 082 U	**0.006 NJ**	0 081 U	0 082 U
trifluralin	0 12 U	0 12 U	0 12 U	**0.009 J**	0 12 U	0 12 U	0 12 U
norflurazon	0 16 U	0 16 U	0 16 U	0 16 U	0 16 U	0 16 U	**0.011 NJ**
			ANCILLARY WQ DATA				
flow (cfs)	120	172	8	34	15	300est	1000
temperature (C)	10 4	11 7	14 6	11 2	11 5	12 5	11 5
conduct (umho/cm)	156	117	452	237	221	132	155
t. susp. solids (mg/L)	137	15	9	62	23	58	60

Note: Detected compounds highlighted in **bold**

J = The analyte was positively identified The associated numerical value is an estimate

NJ = There is evidence that the analyte is present The associated numerical value is an estimate

U = The analyte was not detected at or above the reported value (practical quantitation limit)

nd = No compounds detected in this chemical group

Pesticides were detected less frequently in the water samples collected in June (Table 2) Concentrations were again generally less than 1 ug/L. The fewer detections may be partly due to the drier conditions and that less irrigating was being done. Herbicide use is also heavier in the spring than summer

Insecticides were detected once each in Mill Creek (diazinon), Lower Mud and Pine creeks (malathion), Yellowhawk Creek (aldrin), and Dry Creek (DDT) Only diazinon and malathion are currently used The concentrations of malathion (0.18 and 0.48 ug/L) and aldrin (0.11 ug/L) were relatively high

Of the eight herbicides detected in June, 2,4-D, MCPA, and bromacil were again among the most common Bromoxynil and diuron, present in most of the April water samples, were not detectable

Table 3 compares the combined results for April and June to WSPMP statewide data. As shown in the table, the detection frequency for herbicides appears high in the Walla Walla drainage compared to other parts of the state. An exception is DCPA (dacthal) which, although reported at elevated levels in the past (see background), was detected only once. The few insecticides that were detected in Walla Walla tributaries are, similarly, occasionally reported in other waterbodies, with the exception of aldrin

The detection of aldrin in Yellowhawk Creek is unusual in that aldrin was banned in the 1970s and quickly degrades to dieldrin. Dieldrin was not detected in any of the Walla Walla samples including sediment (see below). As far as can be determined, the last report of aldrin in statewaters was in a sediment sample from the Yakima drainage (Spring/Snipes Creek) in 1985. Re-sampling showed no aldrin detectable (Johnson et al , 1986)

Aquatic life criteria for the 22 pesticides detected in Walla Walla water samples are shown in Table 4 Violations of criteria were limited to insecticides. The aldrin concentration measured in Yellowhawk Creek (0.11 ug/L) and the DDT concentration in Dry Creek (0.006 ug/L) exceed state standards for chronic exposure (24-hour average not to be exceeded) Aldrin is substantially above the 0 0019 ug/L standard. The concentrations of malathion found in Lower Mud Creek and Pine Creek (0 18 and 0.48 ug/L, respectively) substantially exceed EPA's chronic water quality criteria of 0.01 ug/L, with the Pine Creek value approaching the LC-50 for some aquatic invertebrates (Johnson and Finley, 1980). There are no state standards or EPA criteria for diazinon, but the concentrations found in Yellowhawk and Mill creeks are lower than the maximum recommended by the National Academy of Sciences

All herbicide concentrations seen in water are low compared to available criteria. No criteria or other guidelines could be found for MCPP, DCPA, bromacil, pendimethalin, or norflurazon. However, concentrations of these compounds were several orders of magnitude lower than levels shown to be toxic to fish or aquatic invertebrates (Seyler et al , 1994; EPA 1984, 1985, 1988a,b)

Table 2. Pesticides Detected in Water Samples Collected June 18-19, 1996 (ug/L)

Pesticide	Yellowhawk Creek	Mill Creek	Lower Mud Creek	Dry Creek	Pine Creek	Touchet River	Walla Walla River
			INSECTICIDES				
Organophosphorus							
malathion	0 067 U	0 065 U	**0.18**	0 067 U	**0.48**	0 069 U	0 070 U
diazinon	0 067 U	**0.008** NJ	0 067 U	0 067 U	0 069 U	0 069 U	0 070 U
Organochlorine							
aldrin	**0.11**	0 049 U	0 050 U	0 050 U	0 052 U	0 052 U	0 053 U
4,4'-DDT	0 050 U	0 049 U	0 050 U	**0.006** NJ	0 052 U	0 052 U	0 053 U
Carbamates	nd	nd	nd	nd	nd	nd	nd
Pyrethroids	nd	nd	nd	nd	nd	nd	nd
Propargite	0 17 U	0 16 U	0 17 U	0 17 U	0 17 U	0 17 U	0 18 U
			HERBICIDES				
Chlorophenoxy							
2,4-D	**0.014** J	**0.039**	**0.082**	**0.029** J	**0.069**	**0.040**	**0.064**
MCPA	0 083 U	0 084 U	**1.8**	**0.006** NJ	**0.012** NJ	0 081 U	**0.022** NJ
MCPP (mecoprop)	0 083 U	**0.028** NJ	0 088 U	0 084 U	0 086 U	0 081 U	0 085 U
dicamba	0 042 U	0 042 U	0 044 U	0 042 U	**0.006** NJ	0 040 U	0 042 U
Nitrogen-containing							
bromacil	0 34 U	**0.098** J	**0.018** J	**0.015** J	0 34 U	**0.019** J	**0.016** J
metolachlor	0 34 U	0 33 U	**0.10** J	0 34 U	0 34 U	0 34 U	0 35 U
atrazine	0 084 U	0 081 U	0 083 U	**0.014** J	0 086 U	0 086 U	0 088 U
simazine	0 084 U	0 081 U	0 084 U	**0.012** J	0 086 U	0 086 U	0 088 U
			ANCILLARY WQ DATA				
flow (cfs)	43	14	2	8	10	30 est	113
temperature (C)	14 2	20 8	20 2	18 2	17 2	17 2	15 9
conduct (umho/cm)	150	329	378	508	340	197	338
t. susp. solids (mg/L)	56	3	1	19	21	26	13

Note: Detected compounds highlighted in **bold**

J = The analyte was positively identified. The associated numerical value is an estimate

NJ = There is evidence that the analyte is present. The associated numerical value is an estimate.

U = The analyte was not detected at or above the reported value (practical quantitation limit)

nd = No compounds detected in this chemical group

Table 3. Frequency of Occurence of Pesticides Detected in Water Samples
(% of samples)

Pesticide	Walla Walla Drainage[1]	Rivers & Streams Statewide[2]
2,4-D	85	36
bromacil	78	19
MCPA	71	5
bromoxynil	50	6
diuron	36	1
metribuzin	28	5
triallate	28	4
atrazine	28	26
simazine	21	32
malathion	14	5
diazinon	14	20
diclofop-methyl	14	1
pentachlorophenol	14	19
dicamba	14	11
pendimethalin	14	0
aldrin	7	0
4,4'-DDT	7	8
MCPP (mecoprop)	7	11
DCPA	7	32
metolachlor	7	0
trifluralin	7	0
norflurazon	7	5

[1]Present survey (n = 14)

[2]Davis, D. 1996. Washington State Pesticide Monitoring Program: 1994 Surface Water Sampling Report. Wash. St. Dept. Ecology, Olympia. (n = 73)

Table 4 Water Quality Criteria for Pesticides Detected in the Walla Walla Drainage (ug/L)

	Washington Standards[1]		EPA Criteria[2]		Canadian Guidelines[3]	NAS Recommendation[4]
Pesticide	Acute	Chronic	Acute	Chronic	Max. Conc.	Max. Conc.
malathion				0 01		0 008
diazinon						0 009
aldrin	2 5	0 0019	3 0		0 004	0 01
4,4'-DDT	1 1	0 001	1 1	0 001	0 001	0 002
bromoxynil					5	
MCPA					2 6	
MCPP						
2,4-D					4	4
diclofop-methyl					6	
pentachlorophenol	5 5*	3 5*	5 5*	3 5*	0 5	
dicamba					10	200
dacthal						
bromacil						
diuron						1 6
metolachlor					8	
metribuzin					1	
triallate					0 24	
atrazine					2	
pendimethalin						
simazine					10	10
trifluralin					0 1	
norflurazon						

[1]Water Quality Standards for Surface Waters of the State of Washington (Chapter 173-201A WAC)

[2]EPA 1986 Quality Criteria for Water EPA 440/5-86-001 (and updates)

[3]CCREM 1987 (and updates) Canadian Water Quality Guidelines Ottawa

[4]National Academy of Sciences 1973 Water Quality Criteria, 1972 EPA-R3-73-033

*@ pH = 6 5

Sediment

The results on Walla Walla sediment samples are summarized in Table 5 Of the eight compounds analyzed, DDT, DDE, DDD, hexachlorobenzene, gamma BHC, and chlordane were detected in all or most of the sediment samples Dieldrin and heptachlor epoxide were not detected at detection limits of 3 7 - 5 3 ug/Kg and 1 2 - 1 8 ug/Kg, respectively

On a dry weight basis, concentrations were generally similar among sampling sites, ranging from 0 49 ug/Kg hexachlorobenzene to 6 6 ug/Kg DDE Because chlorinated pesticides are absorbed by carbon, normalizing the data to TOC in effect removes the influence this may have on apparent concentration differences between sampling sites When the data are normalized, as shown in the lower part of Table 5, Yellowhawk, Lower Mud, and Dry creeks have the highest concentrations Considering the small size of Mud Creek (2-8 cfs during this survey), the normalized data suggest that Yellowhawk and Dry Creek may be the largest sources of chlorinated pesticides to the Walla Walla mainstem

Table 6 compares these data to results of a similar analysis done by the U S Geological Survey on sediments from the Yakima River and its tributaries Both areas show frequent detection of DDT compounds, but with substantially higher concentrations occurring in the Yakima drainage The level of dieldrin in Yakima sediments also stands out Neither data set shows evidence of chlordane or heptachlor epoxide being significant sediment contaminants

Sediment quality criteria for the compounds detected in the Walla Walla sediments are summarized in Table 7 Both the EPA criteria and Canadian guidelines are for protection of benthic organisms, except EPA's DDT criterion is to prevent significant bioaccumulation in wildlife The EPA sediment criteria have not been formally adopted

The EPA no-unacceptable-effect criteria for DDT, gamma BHC, and chlordane are slightly exceeded in sediment samples from Yellowhawk Creek and/or Dry Creek The only compound that approaches a level of substantial concern is gamma BHC (lindane) in Dry Creek The concentration measure here was at Canada's lowest-effect-level (dry weight basis) and at half the severe-effect-level (normalized to TOC)

Other Walla Walla Data

During September 1996, EILS' Watershed Assessments Section conducted a receiving water study of Garrison Creek, which enters the Walla Walla between Yellowhawk Creek and Mill Creek (Rashin et al , in prep) The study was conducted to assist ERO in managing the discharge from the College Place wastewater treatment plant A variety of physical, chemical, and biological data were obtained, including analysis of pesticides in water, sediment, and fish

Table 5. Organochlorine Pesticides Detected in Sediment Samples Collected June 18-19, 1996

Pesticide	Yellowhawk Creek	Mill Creek	Lower Mud Creek	Dry Creek	Pine Creek	Touchet River	Walla Walla River
			DRY WEIGHT BASIS (ug/Kg, dry)				
4,4'-DDE	2.7	3.3	6.6	5.0	4.2	3.1	7.6
4,4'-DDT	3.0	1.8	1.2 J	1.1 U	1.9	0.69 J	1.2 J
4,4'-DDD	0.61 J	0.76 J	2.5 NJ	0.99 J	0.77 J	0.83 J	1.8
total DDT	6.3	5.9	10.3	6.0	6.9	4.6	10.6
hexachlorobenzene	0.49 J	0.45 J	0.89 J	3.7	1.2	1.4	2.0
gamma BHC	0.74 J	0.91 J	0.89 J	3.7	0.90 J	0.56 J	0.80 J
chlordane	1.7 NJ	3.5 J	18 U	3.0 J	1.5 NJ	14 U	3.0 NJ
t. organic carbon (%)	0.3	1.5	1.4	0.8	0.6	0.8	9.2
			ORGANIC CARBON NORMALIZED (mg/Kg TOC)				
4,4'-DDE	0.90	0.22	0.90	0.62	0.25	0.39	0.08
4,4'-DDT	1.0	0.12	0.17	nd	0.11	0.09	0.13
4,4'-DDD	0.20	0.05	0.35	0.12	0.05	0.10	0.02
total DDT	2.1	0.49	1.4	0.74	0.41	0.58	0.23
hexachlorobenzene	0.16	0.03	0.12	0.46	0.07	0.18	0.02
gamma BHC	0.25	0.06	0.12	0.46	0.05	0.07	0.01
chlordane	0.57	0.23	nd	0.38	0.09	nd	0.03

nd = Not detected

Table 6. Comparison with USGS Data[1] on Sediments in the Yakima River Drainage (ug/Kg, dry)

	Walla Walla Drainage (n=7)			Yakima Drainage (n=37)		
Pesticide	median	range	det. freq.	median	range	det. freq.
4,4'-DDE	4.2	2.7-7.6	100%	30	<0.1-1700	86%
4,4'-DDT	1.2	0.69-3.0	86%	8	<0.1-370	62%
4,4'-DDD	0.83	0.61-2.5	100%	13	<0.1-440	78%
hexachlorobenzene	1.2	0.45-3.7	100%	na	na	na
gamma BHC	0.9	0.56-3.7	100%	na	na	na
chlordane	1.7	1.5-3.5	71%	nd	<1-<10	0%
dieldrin	nd	<3.7-<5.3	0%	1.8	<0.1-47	59%
heptachlor epoxide	nd	<1.2-<1.8	0%	nd	<0.1-<10	0%

[1]Rinella, J.F. et al. 1992. USGS Open-File Report 92-644.
nd = Not detected.
na = Not analyzed

Table 7. Sediment Quality Criteria for Pesticides Detected in the Walla Walla Drainage

	EPA Criteria[1,2]	Canadian Guidelines[3]		
	No Unacceptable Effect	No Effect	Lowest Effect	Severe Effect
Pesticide	(mg/Kg TOC)	(ug/Kg, dry)	(ug/Kg, dry)	(mg/Kg TOC)
4,4'-DDT	0.8	--	8	71
total DDT	--	--	7	12
hexachlorobenzene	--	10	20	24
gamma BHC	0.2	0.2	3	1
chlordane	0.3	5	7	6

[1]EPA. 1988. Interim Sediment Quality Criteria Values for Nonpolar Hydrophobic Organic Compounds. EPA Rept. SCD No. 17, Washington D.C.

[2]EPA. 1990. Tables of criteria maximum concentrations, final chronic values, Koc values, and acute and chronic sediment quality criteria values. Memorandum from D. Hansen to D. Redford, ERL Narragansett, RI.

[3]Persaud, D. et al. 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario, Ontario Ministry of Environment and Energy, ISBN 0-7729-9248-7.

Preliminary results show detection of DDT, DDE, DDD, hexachlorobenzene, lindane, and several herbicides in water, with DDT compounds exceeding chronic water quality criteria. A sediment sample from the creek contained higher concentrations of DDT compounds than found in other tributaries analyzed for the present survey. Hexachlorobenzene was also detected in the sediments. As of this writing the fish samples collected in Garrison Creek have not been analyzed. The draft report on this study is expected in March 1997.

Conclusions

Results of this survey showed relatively few instances where pesticides were a significant water quality concern in the Walla Walla drainage. Herbicides appear to be chronic but low-level contaminants. Only one currently used pesticide, malathion, exceeded water quality criteria, approaching acutely toxic concentrations in one instance. Trace amounts of several first-generation chlorinated pesticides remain in the drainage. The detection of one of these, aldrin, is unusual given its short-term persistence.

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Appendices

Appendix A. Target Pesticides List for Surface Water

EPA Method 8085
(one extraction)

Chlorinated Pesticides
(one analysis)

Compound	Quantitation Limits* µg/L	Compound	Quantitation Limits µg/L
2,4'-DDT	0.05	chlordene-gamma	0.05
2,4'-DDE	0.05	dicofol (Kelthane)	0.50
2,4'-DDD	0.05	dieldrin	0.05
4,4'-DDT	0.05	endrin	0.05
4,4'-DDE	0.05	endrin aldehyde	0.05
4,4'-DDD	0.05	endrin ketone	0.05
DDMU	0.05	endosulfan I	0.05
aldrin	0.05	endosulfan II	0.05
BHC-alpha	0.05	endosulfan sulfate	0.05
BHC-beta	0.05	heptachlor	0.05
BHC-delta	0.05	heptachlor epoxide	0.05
BHC-gamma (Lindane)	0.05	methoxychlor	0.08
captafol	0.25	mirex	0.05
captan	0.15	nonachlor-cis	0.05
chlordane-alpha	0.05	nonachlor-trans	0.05
chlordane-gamma	0.05	oxychlordane	0.05
chlordene-alpha	0.05	toxaphene	1.00

Pyrethroid Pesticides
(one analysis)

Compound	Quantitation Limits µg/L	Compound	Quantitation Limits µg/L
cis-permethrin	0.17	phenothrin	0.17
fenvalerate	0.30	resmethrin	0.17

* - Quantitation limits are approximate and may change between samples.

Appendix A (cont.). Target Pesticides List for Surface Water

EPA Method 8085 (cont.)

Organophosphorus Pesticides
(one analysis)

Compound	Quantitation Limits μg/L	Compound	Quantitation Limits μg/L
abate (Temephos)	0.75	fenamiphos	0.13
azinphos-ethyl	0.13	fenitrothion	0.06
azinphos-methyl (Guthion)	0.16	fensulfothion	0.08
carbophenothion	0.08	fenthion	0.06
chlorpyrifos (Lorsban)	0.06	fonofos	0.05
chlorpyrifos-methyl	0.06	imidan (Phosmet)	0.09
coumaphos	0.09	malathion	0.06
DEF (Tribufos)	0.11	merphos	0.13
demeton-o	0.06	mevinphos	0.08
demeton-s	0.06	monocrotophos	0.58
diazinon	0.07	paraoxon-methyl	0.15
dichlorvos	0.07	parathion	0.06
diethyl fumarate	0.25	parathion-methyl	0.06
dimethoate	0.06	phorate	0.06
dioxathion	0.14	phosphamidan	0.19
disulfoton	0.05	propetamphos	0.16
EPN	0.08	ronnel	0.06
ethion	0.06	sulfotepp	0.05
ethoprop	0.07	sulprofos	0.06
		tetrachlorvinphos (Gardona)	0.17

Sulfur-Containing Pesticides
(one analysis)

Compound	Quantitation Limits μg/L
propargite	0.18

Appendix A (cont.). Target Pesticides List for Surface Water

EPA Method 8085 (cont.)

Nitrogen-Containing Pesticides
(one analysis)

Compound	Quantitation Limits µg/L	Compound	Quantitation Limits µg/L
Triazines			
ametryn	0.08	cycloate	0.13
atraton	0.25	di-allate (Avadex)	0.30
atrazine	0.08	EPTC (Eptam)	0.14
cyanazine	0.12	triallate (Fargo)	0.20
hexazinone	0.13	vernolate	0.14
metribuzin	0.08		
prometon	0.08	**Substituted Amides**	
prometryn	0.08	diphenamid	0.25
propazine	0.08	napropamide	0.25
simazine	0.08	pronamide	0.25
terbutryn	0.08		
		Uracils	
Anilines		bromacil	0.50
benfluralin (Benefin)	0.12	terbacil	0.25
ethalfluralin	0.12		
pendimethalin	0.12	**Ureas**	
profluralin	0.19	diuron	0.45
trifluralin	0.12	tebuthiuron	0.08
Anilides		**Miscellaneous**	
alachlor	0.20	carboxin	0.86
butachlor	0.29	chlorpropham	0.35
metolachlor	0.25	fenarimol	0.25
propachlor	0.17	fluridone	0.55
		metalaxyl	0.52
Cyano		MGK264	0.55
chlorothalonil	0.20	molinate	0.20
dichlobenil	0.10	norflurazon	0.13
		oxyfluorfen	0.25
Thiocarbamates		pebulate	0.20
butylate	0.13	triadimefon	0.22

Appendix A (cont.). Target Pesticides List for Surface Water

EPA Method 8085 (cont.)

Carbamates
(one analysis)

Compound	Quantitation Limits µg/L		
aldicarb	0.30	3-hydroxycarbofuran	0.12
aldicarb sulfone	0.12	methiocarb	0.30
aldicarb sulfoxide	0.12	methomyl	0.12
baygon (propoxur)	0.12	1-naphthol	0.12
carbaryl	0.12	oxamyl	0.12
carbofuran	0.12		

EPA Method 8085
(one extraction, one analysis)

Chlorinated Herbicides

Compound	Quantitation Limits µg/L	Compound	Quantitation Limits µg/L
2,4-D	0.04	chloramben	0.04
2,4-DB	0.05	DCPA (Dacthal)	0.03
2,4,5-TB	0.04	dalapon (DPA)	0.11
2,4,5,-TP	0.03	dicamba	0.04
2,4,5-trichlorophenol	0.02	dichlofop-methyl	0.06
2,4,6-trichlorophenol	0.02	dichlorprop	0.04
2,3,4,6-tetrachlorophenol	0.02	dinoseb	0.06
3,5-dichlorobenzoic acid	0.04	ioxynil	0.04
4-nitrophenol	0.07	MCPA	0.08
5-hydroxydicamba	0.04	MCPP	0.08
acifluorfen (Blazer)	0.16	pentachlorophenol	0.02
bentazon	0.06	picloram	0.04
bromoxynil	0.04	triclopyr (Garlon)	0.03

Appendix B

Manchester Environmental Laboratory

7411 Beach Dr E, Port Orchard Washington 98366

CASE NARRATIVE

June 28, 1996

Subject: WSPMP, weeks 16 & 17
Walla Walla, week 17

Samples: 96168060-66, 96178067-68 & 96178190-96

Officer(s): Dale Davis (for WSPMP)
Art Johnson (for Walla Walla)

By: Norman Olson
Bob Carrell
Organics Analysis Unit

PESTICIDE & HERBICIDE ANALYSIS

ANALYTICAL METHODS: (Draft EPA Method 8085; formerly modified 1618 & 1658) Separate water samples, one each for the neutrals and Acids, were extracted following Manchester Laboratory's standard operating procedure for the extraction of pesticides and herbicides. The samples for neutrals (NPest, ClPest, OPPest, SPest & Pyrethriods) analyses were extracted with methylene chloride and solvent exchanged to iso-octane. The samples for acid herbicide analysis were hydrolyzed at pH > 12, extracted with methylene chloride at pH < 2, solvent exchanged and methylated. The extracts were separately analyzed by capillary Gas Chromatography and Atomic Emission Detection (GC/AED). Confirmation of detected pesticides and herbicides was performed by Gas Chromatography and Ion-Trap mass spectrometry (GC/ITD) or comparisons of elemental ratios of heteroatoms to empirical formulas.

All analytes have a respective practical quantitation limit (PQL) that is higher than the corresponding method detection level (MDL). If a target analyte is detected and confirmed at a concentration below its PQL, the reported concentration is qualified as an estimate, ' J' qualifier. This procedure also applies to the method blanks.

NITROGEN-CONTAINING PESTICIDE ANALYSIS

BLANKS: No nitrogen-containing target compounds were detected in the laboratory blanks. Hence, the blanks demonstrate the system was free from this type of contamination.

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: 1,3-Dimethyl-2-nitrobenzene recoveries were acceptable, ranging from 62% to 100%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable ranging from 73% to 136%.

COMMENTS: The urea pesticide breakdown product, dichlorobenzene-isocyanate, was found in several samples. Most likely the parent pesticide is Diuron in all cases, but if Diuron itself wasn't also detected and confirmed in the sample, the reported value is qualified 'NJ'.

The non-target pesticide, Propiconazole (Tilt), was detected in sample 96178067. A estimate of quantitation based on elemental calibration is reported, the value being a combination of two isomers. A good mass-spectral match for identification was obtained along with the correct N:CL elemental ratio. No reference standard of this pesticide was analyzed for final confirmation, therefore, the 'NJ' qualifier was applied. See attached information on this compound.

Data is useable as qualified

ORGANOPHOSPHOROUS PESTICIDE ANALYSIS

BLANKS: No organophosphorous target compounds were detected in the laboratory blanks.

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: Triphenylphosphate recoveries were acceptable, ranging from 91% to 138%.

MATRIX SPIKING: No organophosphorous target compounds were spiked during this run.

COMMENTS: Data is useable as qualified

ORGANOCHLORINE PESTICIDE ANALYSIS

BLANKS: No organochlorine target compounds were detected in the laboratory blanks.

HOLDING TIMES: All samples were extracted within seven days of sampling

SURROGATES: Decachlorobiphenyl recoveries were acceptable, ranging from 48% to 92%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable ranging from 65% to 121%.

COMMENTS: The non-target organochlorine compound, Triclosan (a disinfectant) was detected in sample 96178195. A estimate of quantitation based on elemental calibration is reported. A good mass-spectral match for identification was obtained, but no reference standard of this compound was analyzed for final confirmation Therefore, the 'NJ' qualifier was applied. See attached information on this compound.

Data is useable as qualified.

SULFUR-CONTAINING AND PYRETHROID PESTICIDE ANALYSIS

BLANKS: None of these types of target analytes were detected in the laboratory blanks.

HOLDING TIMES: All samples were extracted within seven days of sampling

SURROGATES: There no designated surrogate compounds for these groups of targets. Recovery efficiencies of surrogates from other neutral pesticide groups should also apply to this group.

MATRIX SPIKING: The recoveries for the sulfur-containing pesticide Propargite were 103% & 108%. The pyrethroid pesticides were not spiked during this run.

COMMENTS: Data is useable as qualified.

ACID HERBICIDE ANALYSIS

BLANKS: No acid herbicide target compounds were detected in the laboratory blanks

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: 2,4,6-Tribromophenol recoveries were acceptable, ranging from 75% to 140%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable in LMX2 ranging from 39% to 128%, except Picloram at 18%. The recoveries of spiked analytes in LMX1 were quite variable and out of the ordinary. Although the precision for acid herbicides is usually less then the neutral analytes, LMX1 shows results we have not seen before. Analytes that historically perform adequately are in this case essentially not performing at all, while others are just fine. We can think of no explanation at this time. No qualifiers have been applied on this basis.

Dinoseb and Picloram are qualified 'UJ' throughout due to the poor precision these analytes have historically shown.

COMMENTS: Pentachlorophenol (PCP) was detected in several samples, but **not** in any of the method blanks However, all of the PCP detects are 'N' qualified for the following reasons:

1. Routinely the extraction apparatus is used for preparation of samples known to contain relatively large amounts of PCP.
2. Although steps are taken to reduce or eliminate cross contamination, the chance still exists.
3. This analyte has been detected in method blanks in the past.
4. The amount found in the these samples is not sufficiently large that that we can positively say contamination was not the source.

Data is useable as qualified

DATA QUALIFIER CODES:

U	-	The analyte was not detected at or above the reported value.
J	-	The analyte was positively identified The associated numerical value is an estimate.
UJ	-	The analyte was not detected at or above the reported estimated result.
REJ	-	The data are unusable for all purposes.
NAF	-	Not analyzed for.
N	-	For organic analytes there is evidence the analyte is present in this sample.

NJ - There is evidence that the analyte is present. The associated numerical result is an estimate.

E - This qualifier is used when the concentration of the associated value exceeds the known calibration range.

Manchester Environmental Laboratory

7411 Beach Dr E, Port Orchard Washington 98366

CASE NARRATIVE

August 26, 1996

Subject: WSPMP waters, weeks 24 & 25
Walla Walla waters, week 25

Samples: 96248020-26, 96258000-01 & 96258080,82,84,86,88,90,92

Officer(s): Dale Davis (for WSPMP)
Art Johnson (for Walla Walla)

By: Norman Olson
Bob Carrell
Organics Analysis Unit

PESTICIDE & HERBICIDE ANALYSIS

ANALYTICAL METHODS: (Draft EPA Method 8085; formerly modified 1618 & 1658) Separate water samples, one each for the neutrals and Acids, were extracted following Manchester Laboratory's standard operating procedure for the extraction of pesticides and herbicides. The samples for neutrals (NPest, ClPest, OPPest, SPest & Pyrethriods) analyses were extracted with methylene chloride and solvent exchanged to iso-octane. The samples for acid herbicide analysis were hydrolyzed at pH > 12, extracted with methylene chloride at pH < 2, solvent exchanged and methylated. The extracts were separately analyzed by capillary Gas Chromatography and Atomic Emission Detection (GC/AED). Confirmation of detected pesticides and herbicides was performed by Gas Chromatography and Ion-Trap mass spectrometry (GC/ITD) or comparisons of elemental ratios of heteroatoms to empirical formulas.

All analytes have a respective practical quantitation limit (PQL) that is higher than the corresponding method detection level (MDL). If a target analyte is detected and confirmed at a concentration below its PQL, the reported concentration is qualified as an estimate, ' J' qualifier. This procedure also applies to the method blanks.

NITROGEN-CONTAINING PESTICIDE ANALYSIS

BLANKS: No nitrogen-containing target compounds were detected in the laboratory blanks. Hence, the blanks demonstrate the system was free from this type of contamination.

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: 1,3-Dimethyl-2-nitrobenzene recoveries were acceptable, ranging from 73% to 105%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable ranging from 67% to 144%. The range of recoveries for all spiked targets other then Simazine was 67% to 115%. The higher recoveries for Simazine are most likely due to interference because it was not detected in the sample associated with the matrix spiking.

COMMENTS: The data is useable as qualified

ORGANOPHOSPHOROUS PESTICIDE ANALYSIS

BLANKS: No organophosphorous target compounds were detected in the laboratory blanks.

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: Triphenylphosphate recoveries were acceptable, ranging from 94% to 130%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable ranging from 96% to 138%.

COMMENTS: The data is useable as qualified

ORGANOCHLORINE PESTICIDE ANALYSIS

BLANKS: No organochlorine target compounds were detected in the laboratory blanks.

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: Decachlorobiphenyl recoveries were acceptable, ranging from 65% to 106%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable ranging from 71% to 92%. The recoveries for Kelthane were calculated from its breakdown product, 4,4'-dichlorobenzophenone.

COMMENTS: The data is useable as qualified.

SULFUR-CONTAINING AND PYRETHROID PESTICIDE ANALYSIS

BLANKS: None of these types of target analytes were detected in the laboratory blanks.

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: There no designated surrogate compounds for these groups of targets. Recovery efficiencies of surrogates from other neutral pesticide groups should also apply to this group.

MATRIX SPIKING: No matrix spiking was performed for these groups of targets during this run.

COMMENTS: The data is useable as qualified.

ACID HERBICIDE ANALYSIS

BLANKS: No acid herbicide target compounds were detected in the laboratory blanks

HOLDING TIMES: All samples were extracted within seven days of sampling.

SURROGATES: 2,4,6-Tribromophenol recoveries were acceptable, ranging from 67% to 137%.

MATRIX SPIKING: Recoveries of spiked analytes were acceptable ranging from 34% to 138%, except Picloram and Dinoseb, @18% & 23% and 23% & 67% respectively.

Dinoseb and Picloram are qualified 'UJ' throughout due to the poor precision these analytes have historically shown.

COMMENTS: The data is useable as qualified.

DATA QUALIFIER CODES:

U	-	The analyte was not detected at or above the reported value.
J	-	The analyte was positively identified. The associated numerical value is an estimate.
UJ	-	The analyte was not detected at or above the reported estimated result.
REJ	-	The data are unusable for all purposes.
NAF	-	Not analyzed for.
N	-	For organic analytes there is evidence the analyte is present in this sample.
NJ	-	There is evidence that the analyte is present. The associated numerical result is an estimate.
E	-	This qualifier is used when the concentration of the associated value exceeds the known calibration range.

Manchester Environmental Laboratory

7411 Beach Dr E, Port Orchard Washington 98366

CASE NARRATIVE

August 26, 1996

Subject: Walla Walla Sediments

Samples: 96258081,83,85,87,89,91 & 93

Officer(s): Art Johnson

By: Norman Olson
Bob Carrell
Organics Analysis Unit

CHLORINATED PESTICIDE ANALYSIS

ANALYTICAL METHODS: EPA SW-846 8081 was the analytical method used for the gas chromatographic analysis of all the target chlorinated pesticides except Dieldrin, for which EPA SW-846 Draft 8085 was used Confirmation of detected pesticides was performed by dual dissimilar column retention time comparisons The extraction method used was EPA SW-846 3540, which is a soxhlet extraction procedure Sample clean-up entailed collection of two florisil elution fractions, 6% & 50% The 6% fraction was then treated with concentrated sulfuric acid and analyzed by GC/ECD for all targets except Dieldrin The 50% fraction was analyzed by GC/AED for Dieldrin

All analytes have a respective practical quantitation limit (PQL) that is higher than the corresponding method detection level (MDL) If a target analyte is detected and confirmed at a concentration below its PQL, the reported concentration is qualified as an estimate, ' J' qualifier

All results are reported on a dry weight basis

BLANKS: No target compounds were detected in the laboratory blanks Hence, the blanks demonstrate the system was free from contamination

HOLDING TIMES: All samples were extracted within 14 days of sampling

SURROGATE(S): All surrogate recoveries were acceptable, ranging from 66% to 147%

MATRIX SPIKING: Recoveries of spiked analytes were acceptable, ranging from 79% to 146% Recoveries of all spiked analytes other than DDE ranged from 79% to 117% The precision of DDE results between the LMX samples is less then it is for the other analytes, this is most likely due to the higher native concentration of this compound being present in the sample

DUPLICATE: The duplicate analysis of sample 96258081 shows very good precision for all analytes

COMMENTS: The data is useable as qualified

DATA QUALIFIER CODES:

U	-	The analyte was not detected at or above the reported value.
J	-	The analyte was positively identified. The associated numerical value is an estimate
UJ	-	The analyte was not detected at or above the reported estimated result.
REJ	-	The data are unusable for all purposes
NAF	-	Not analyzed for.
N	-	For organic analytes there is evidence the analyte is present in this sample.
NJ	-	There is evidence that the analyte is present. The associated numerical result is an estimate.
E	-	This qualifier is used when the concentration of the associated value exceeds the known calibration range.

Appendix C. Names of Pesticides Detected in the Walla Walla Drainage

Common Name	Trade Names[1]
Insecticides	
malathion	Acimal, Malathion 50, For-Mal 50, Prentox, Rion, Sulmalathion
diazinon	Diazol, Gardentox, Spectracide
aldrin[2]	Altox, Aldrex, Aldrite
4,4'-DDT[2]	Anofex, Chlorophenothane, Pentachlorin
4,4'-DDE	DDT metabolite
4,4'-DDD[3]	Rothane, also a DDT metabolite
hexachlorobenzene[2]	No Bunt
gamma BHC	Lindane, Gammexane, Gexane, HCCH, Hexyclan, Soprocide
chlordane	Chlortox
Herbicides	
bromoxynil	Buctril
MCPA	Chiptox, Rhonox, Sword, Cheyenne/Tiller(w/fenoxaprop)
MCPP (mecoprop)	Mecopar, Mecopex, Turf Herbicide, Chipco Turf Clean/ Penomec(w2,4,-D), Crossbow(w/triclopyr)
2,4-D	Formula 40, Hi-Dep, Miller's Envy, Salvo, Savage, Weed-Rhap Campaign, Landmaster (w/glyphosate), Envert/Weedone(w/dichlorprop) Phenabam 801/ Weedmaster(w/dicamba), Trimec(w/MCPP&dicamba)
diclofop-methyl	Hoelon
pentachlorophenol	Pentacon, Penwar, GLAZD, Penta
dicamba I	Banvel, Clarity, Trooper, Vanquish, Banvel 720(w/2,4-D) Fallow Master(w/glyphosate), Marksman(w/atrazine)
DCPA	Dacthal
bromacil	Hyvar X/XL, Krovar I DF(w/diuron)
diuron	Direx, Karmex DF, Surefire(w/paraquat)
metolachlor	Dual, Pennant, Bicep(w/atrazine), Cycle(w/atrazine), Derby(w/simazine)
metribuzin	Lexone, Sencor
triallate	Far-Go
atrazine	AAtrex, Atrapril, Cheat Stop, Conquest/Extrazine (w/cyanazine), Laddok(w/bentazon), Lasso/ Lariat (w/alachlor) Ramrod(w/propachlor), Surpass 100(w/acetochlor)
pendimethalin	Pentagon, Prowl, Oriental Herbicide II(w/oxyfluorfen)
simazine	Aquazine, Princep, Amizine(w/amitrole), Pramitol 5 PS(w/prometon)
trifluralin	Treflan
norflurazon	Evital, Predict, Solicam, Zorial

Sources:1995 Pacific Northwest Week Control Handbook; 1996 Farm Chemicals Handbook

[1]Trade names are examples and not necessarily the source of residues detected in the present survey.

[2]Banned in the U.S. [3]Discontinued by manufacturer

Appendix D Locations of Water and Sediment Samples Analyzed for Pesticides

Site Name	Location	Lat. (N)	Long. (W)
Yellowhawk Creek	Old Milton Highway	46 01 35	118 23 97
Mill Creek	Whitman Mission Road	46 02 58	118 28 21
Lower Mud Creek	Barney Road	46 02 61	118 36 96
Dry Creek	Highway 12	46 03 35	118 35 40
Pine Creek	Sand Pit Road	46 01 70	118 38 01
Touchet River	Highway 12	46 02 50	118 41 01
Walla Walla River	Cummins Road Bridge	46 02 24	118 45 87