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1. Synopsis

1.1 Scope: This is a liquid chromatographic/mass spectrometry/mass spectrometry (LC/MS/MS) method for the determination of 45 polar pesticides and metabolites in bottled water and ground water. Twenty nine compounds are run with electrospray ionization in the positive mode and 16 analytes are run in negative mode. This method was validated using Ice Mountain® bottled water and ground water. The limit of detection (LOD) and limit of quantitation (LOQ) for each compound is listed in Section 12. The parent-daughter transitions for each compound are listed in Section 9.2. Most of the compounds are screened with 1 parent-daughter transition due to dwell time limitations and rerun with the confirmation transition if there is a detection in a sample.

1.2 Principle: A 200 mL sample is applied to a Supelco Envi-carb solid-phase cartridge using a Caliper Autotrace. The analytes are eluted with $MeCl_2$/MeOH and acidic $MeCl_2$/MeOH. The sample is evaporated and brought to volume with MeOH and HPLC grade water. The sample is filtered through a syringe filter and placed into autosampler vials for analysis by LC/MS/MS.

2. Responsibility

2.1 The laboratory director's responsibilities include ensuring that the laboratory is properly staffed and that supplies and equipment are available for proper analysis of samples.

2.2 The QA officer is responsible for reviewing all associated quality control associated with the analysis.

2.3 The supervisor is responsible for assigning samples to the appropriate analyst and ensuring that the analyst has supplies and equipment.

2.4 The analyst will keep the supervisor informed of any problems encountered during analysis that impacts the final result.

3. Safety and Environmental Considerations

3.1 Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method.

3.1.1 Use mechanical pipetting aides.

3.1.2 Avoid skin contact or inhalation.

4. Apparatus and Materials

4.1 Micromass Quattro™ LC (LC/MS/MS).

4.2 Waters 2690™ High Pressure Liquid Chromatograph (HPLC).

4.3 Zorbax RX-C8 column, 2.1 mm * 15 cm.

4.4 OPTI-SOLV® precolumn, 0.2 micron.

4.5 Caliper AutoTrace®, automated SPE station.

4.6 Caliper TurboVap II® evaporator.

4.7 Organomation® N-Evap, evaporator with water bath.

4.8 Ultrasonicator.

4.9 Vortex mixer.

4.10 Supelco Envi-carb SPE columns, 0.25g/6mL.

4.11 PTFE Acrodisc® syringe filter 13mm * 0.2 um.

4.12 Waters® Total Recovery vials, 12 * 32 mm with preslit screw caps.

4.13 Pipets, Pasteur, disposable, 9" length.

4.14 400 mL beakers.

4.15 Disposable 1 mL syringe.

4.16 1000 mL graduated cylinders.

4.17 15 mL volumetric Autotrace tubes, Caliper Life Science.

4.18 40 mL stemmed Turbovap® tubes, Dionex Corp.

5. **Reagents and Chemicals:**

5.1 Pesticide grade (nanograde, distilled in glass) solvents.
- 5.1.1 Methanol.
- 5.1.2 Pure Water, reagent.
- 5.1.3 Acetone.
- 5.1.4 Methylene chloride.

5.2 Extran® 300, Cleaning solution, EM Science.

5.3 Formic Acid, 88%.

5.4 Hydrochloric Acid.

5.5 80/20 $MeCl_2$/MeOH Elution Solution: 200 mL MeOH and bring to 1000 mL volume with methylene chloride.

5.6 Acidic 80/20 $MeCl_2$/MeOH Elution Solution: Add 0.4 mL HCL to an 80/20 solution prepared as above (section 5.5).

5.7 Stock standard solutions: Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

5.7.1 Prepare separate 1.00 mg/mL stock standard solutions of each compound by accurately weighing at least 0.0050 g of a certified standard of known purity into an amber vial with Teflon lined screw top cap. Calculate the amount of pure standard by multiplying the amount weighed in milligrams (mg) and the purity. Add an appropriate amount of pesticide quality methanol to obtain 1.00 mg/mL. Prepare hydroxy and deethyl deisopropyl in 0.1 mg/ml concentrations.

5.7.2 Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standards should be stable for one year if stored in the freezer and protected from light.

5.8 Intermediate mixed standards: The first mix (mix A) for each ionization mode (positive and negative) will be made in a 50 mL volumetric flask where each compound's concentration will be 10,000 times it's LOD or estimated LOD (standard LOD). For example, if an analyte's LOD is 1.0 ppb, it should have a concentration of 10,000 ppb or 10 ppm in mix A. Mix A will be brought to a 50 mL volume with methanol. Mix B is a 1 to 10 dilution of Mix A, and Mix C is a 1 to 10 dilution of Mix B. The dilution solvent for Mix B and C will be methanol and the final volume can either be 25 or 50 ML. See Sections 12.3 - 12.4 for intermediate Mix A standard concentrations.

5.9 Calibration standards: Calibration standards are made in 30% methanol and 70% HPLC grade water at concentrations that correspond to the analyte's LOD, LOQ, 3LOQ, 6LOQ and 10LOQ. The positive mode standards should be made fresh with each extraction batch. After adding the following volumes of MeOH and standards, bring to a final volume of 5 mL with HPLC grade water. The negative mode standards can be made up in a larger volume and stored for one month. The calibration curve is set up in relation to the LOQ level.

PDP Positive calibration standards:

Standard	Calib.level	Soln. added (mix - volume)	MeOH vol.
LOD	0.3	C – 50 uL	1450 uL
LOQ	1.0	C – 166 uL	1334 uL
3LOQ	3.0	C – 500 uL	1000 uL
6LOQ	6.0	B – 100 uL	1400 uL
10LOQ	10.0	B – 166 uL	1334 uL

PDP Negative calibration standards:

Standard	Calib.level	Soln. added (mix - volume)	MeOH vol.	Metazachlor (conc-volume)
LOD	0.3	C – 50 uL	1430 uL	0.1 – 20 uL
LOQ	1.0	C – 166 uL	1268 uL	0.1 – 66 uL
3LOQ	3.0	C – 500 uL	800 uL	0.1 – 200 uL
6LOQ	6.0	B – 100 uL	1392 uL	5.0 – 8 uL
10LOQ	10.0	B – 166 uL	1321 uL	5.0 – 13 uL

6. Interferences

6.1 Solvents, reagents, water, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. Due to the trace levels of analysis, all of these materials must be demonstrated to be free from interference's under the conditions of the analysis by analyzing a matrix and reagent blanks for each sample batch.

6.2 Sample preparation glassware **MUST** be washed, solvent rinsed and oven dried prior to being used for another sample.

7. Quality Control

7.1 Blanks: One reagent blank (HPLC grade bottled water) and one matrix blank (Evian® bottled water or ground water) will be analyzed with each sample batch.

7.2 Spikes: One matrix spike (Evian® bottled water or ground water) at 2 * LOQ will be run with each sample batch and the marker compounds recoveries reported with every batch. In addition to the required matrix spike, add a reagent spike to each batch due to some variability issues that have been detected with the matrix spike. Two times a year, the spike recoveries of every compound need to be reported. The spike recoveries should range between 50-150%. Spike recovery is an indicator on how accurately the method extracts and detects the analytes of interest.

7.2.1 ES Positive markers:
Hydroxy Atrazine
Imazamox
Disulfoton sulfone
Prometryn
Carbaryl
Metalaxyl
Bensulfuron methyl
Neburon

7.2.2 ES Negative markers:
Picloram
Acetochlor ESA
Acetochlor OXA
2,4-DB

7.3 Process control (surrogate spikes): Propoxur (ES positive analytes) and metazachlor ESA (ES negative analytes) are spiked at 5 * LOQ into every sample, blank and spike prior to extraction. Process control recovery is an indicator on how accurately the method extracts and detects the analytes in each matrix.

8. Procedures

8.1 Prepare Autotrace:

8.1.1 Check that all of the Autotrace solvent bottles are adequately filled. Prepare and fill solvent bottles as needed. See Autotrace method (Section 9.1) for a listing of the solvent bottles. Put dummy cartridges in all six positions and push down until the barrels snap into place and the green lights turn on.

8.1.2 Load the Purge Solvent Path disk to purge the solvent lines.

8.1.3 Load the Clean Sample Path disk to clean and purge the sample lines. Place all of the sample lines into a beaker of methanol for the first rinse. After the second instrument prompt, place all of the sample lines into a beaker of water. Clean and purge the sample lines immediately after each extraction batch is completed on the instrument.

8.2 Weigh 300 g of water into a 400 mL beaker. The Autotrace will use 200 mL of sample.

8.3 Add process control standards to all of the samples and QC. Add 40 uL of 100 ppb Metazachlor ESA and 100 uL of 150 ppb propoxur.

8.4 Add 100 uL of Neg-C standard and 100 uL of Pos-C standard to the spike samples. Use a glass syringe for the positive standard. A digital syringe can be used for the negative spikes.

8.5 Load the samples on the Autotrace making sure that the sample lines are fully submerged into the beakers and that the elution tubes are appropriately labeled with the corresponding sample number.

8.6 Load new Envi-carb™ columns into the holders and push the plungers down until the barrels snap into place and the green lights come on. Load the PDP method disk.

8.7 Transfer the elution solvent to 40 mL Turbovap® tubes. Rinse the Autotrace tubes with ~4 mL of acetone and transfer to the Turbovap™ tubes. Evaporate the samples on the Turbovap® using a 33˚ C bath and using an endpoint time of 1 minute past sensor and a nitrogen flow of 12 psi.

8.8 Rinse the Autotrace sample lines by following procedure 8.1.3.

8.9 When each sample is done on the Turbovap®, add ~1 mL of acetone and ~1 mL of methanol to the tube and vortex. Evaporate a second time. Samples should

be monitored on the first evaporation step to make sure that an emulsion hasn't been created in the tube. The sample will turn cloudy and the evaporation will be slowed or stopped if an emulsion develops. If an emulsion does develop, add additional acetone and vortex the sample.

8.10 Complete the evaporation process in the N-Evap with a bath temperature set at 38˚C. Evaporate the samples out of the bath but periodically dip the samples into the bath to remove condensation. Evaporate to ~100 uL and then add ~600 uL of acetone by rinsing down the sides of the tube and vortex the samples. Evaporate a second time to ~100 uL to assure complete removal of methylene chloride.

8.11 Add methanol to the 0.3 mL mark. Rinse the sides of the tubes as much as possible when adding the methanol.

8.12 Vortex the samples for 2 minutes by tipping the tubes so that the solvent approximately reaches the 10 mL mark. Continuously rotate the tubes so that the entire surface of the tubes (up to the 10 mL mark) is thoroughly rinsed with solvent.

8.13 Sonicate the samples for 3 minutes. Bring to a 1.0 mL final volume with HPLC grade water and vortex the samples again by tipping the tubes so that the maximum surface is rinsed. Sonicate the samples for an additional 3 minutes.

8.14 Filter the samples through PTFE syringe filters and dispense into autosampler vials.

8.15 Run the samples on the LC/MS/MS using the MDA PDP methods. Run the positive mode analysis before negative mode. Freeze the extracts if there will be a delay in analysis.

9. Instrument Conditions

9.1 Autotrace Method

Step	Procedure
1	Process 6 Samples using the following procedure:
2	Condition Column with 6.0 ml of 80/20 $MeCl_2$/MeOH into solvent waste.
3	Condition Column with 6.0 ml of Methanol into solvent waste.
4	Condition Column with 6.0 ml of Water into aqueous waste.
5	Load 200.0 ml of sample onto Column.
6	Rinse Column with 5.0 ml of Water into aqueous waste.
7	Rinse Column with 0.5 ml of Methanol into solvent waste.
8	Rinse Column with 10.0 ml of Air into solvent waste.
9	Soak and Collect 1.0 ml Fraction using Methanol.
10	Collect 6.0 ml Fraction into sample tube using 80/20 $MeCl_2$/MeOH.

11 Collect 6.0 ml Fraction into sample tube using acidic 80/20 $MeCl_2$/MeOH.

12 End.

Flow Rates

Cond Flow:	10.0 ml/min
Load Flow:	10.0 ml/min
Rinse Flow:	10.0 ml/min
Elute Flow:	4.0 ml/min
Cond Air Push:	10.0 ml/min
Rinse Air Push:	10.0 ml/min
Elute Air Push:	5.0 ml/min

SPE Parameters

Push Delay:	5 sec
Air Factor:	1.0
Autowash Vol.:	1.00 ml

Workstation Parameters

Max. Elution Vol.:	13.0 ml
Exhaust Fan On:	Yes
Beeper On:	Yes

Solvent Set:Standard

Solvent 1	Methanol
Solvent 2	Water
Solvent 3	Air (empty solvent bottle)
Solvent 4	80/20 $MeCl_2$/MeOH
Solvent 5	Acidic 80/20 $MeCl_2$/MeOH

9.2 LC/MS/MS Conditions

PDP Method Voltages
ES positive

Function 1: Time 0.0 – 6.2 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Deethyl deisopropyl Atrazine	3.2	146.05	68.1	30	21
		146.05	**104.1**	30	18

Function 2: Time 6.2 - 14.3 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
HydroxyAtrazine	7.8	198.17	86.09	34	23
		198.17	**156.20**	34	17
Imazapyr	11.9	262.03	149.08	32	25
		262.03	**217.17**	32	19

Function 3: Time 14.3 – 18.7 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Imazamethabenz acid	15.1	**275.14**	**86.12**	35	21
		275.14	161.16	35	23
Imazamox	16.0	306.05	193.08	36	26
		306.05	**246.09**	36	24
Imazapic	16.5	276.09	216.19	36	22
		276.09	**231.14**	36	20

Function 4: Time 18.33 – 22.5 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Imazamethabenz methyl	19.6	**289.06**	**144.08**	36	35
		289.06	229.18	36	20
Imazethapyr	19.6	290.03	177.10	36	27
		290.03	**230.10**	36	23
Propoxur	19.7	**210.24**	**111.13**	15	15
		210.24	168.2	15	8
Carbofuran	20.1	**222.16**	**123.19**	20	25
		222.16	165.17	20	14
Bromacil	19.8	261.09	188.03	20	30
		261.09	**205.05**	20	17
Nicosulfuron	20.1	**411.09**	**182.14**	29	22
		411.09	213.13	29	18
Triasulfuron	20.4	402.09	141.18	34	17
		402.09	**167.17**	34	19
Thifensulfuron methyl	20.7	387.92	141.10	28	20
		387.92	**167.18**	28	20
Carbaryl	20.7	202.14	117.14	15	24
		202.14	**145.15**	15	11

Function 5: Time 20.5 – 23.0 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Imazaquin	21.3	311.96	199.12	35	25
		311.96	**252.10**	35	25
Sulfometuron methyl	21.4	**365.11**	**150.19**	27	19
		365.11	199.07	27	24
Metsulfuron methyl	21.2	382.13	141.14	30	14
		382.13	**167.18**	30	15

Function 6: Time 21.8 – 24.3 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Disulfoton sulfone	22.6	**307.04**	**125.09**	20	18
		307.04	153.17	20	14
Diuron	22.6	233.16	46.09	30	14
		233.16	**72.11**	30	16

Function 7: Time 22.9 – 28.0 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Prometryn	23.4	**242.21**	**158.16**	35	25
		242.21	200.21	35	20
Metalaxyl	24.0	**280.16**	**220.21**	25	15
		280.16	248.25	25	10
Bensulfuron methyl	24.8	**411.05**	**149.14**	24	20
		411.05	182.14	24	19
Siduron	25.3	233.17	94.14	29	19
		233.17	**137.17**	29	17
Linuron	24.2	**249.02**	**160.05**	25	20
		249.02	182.11	25	16
Chlorimuron ethyl	26.1	**415.06**	**186.13**	30	18
		415.06	213.08	30	16

Function 8: Time 26.3 – 38.5 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Halosulfuron methyl	27.1	434.97	157.12	28	45
		434.97	**182.10**	28	14
Neburon	27.8	**275.07**	**88.17**	29	16
		275.07	114.18	29	15
Thiobencarb	29.4	258.07	100.16	24	13
		258.07	**125.11**	24	21

PDP Method Voltages
ES negative

Function 1: Time 0.0 – 8.8 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Clopyralid	4.6	**189.93**	**146.06**	14	8
Picloram	6.3	238.96	123.07	15	21
		238.96	**194.99**	15	12

Function 2: Time 8.8 – 14.0 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Propachlor OXA	11.2	206.07	118.16	9	21
		206.07	**134.19**	9	9
Metazachlor ESA	11.0	**321.85**	**120.86**	35	22
Flufenacet OXA	12.2	224.03	136.15	11	21
		224.03	**152.17**	11	11

Function 3: Time 14.0 – 16.6 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Dimethenamid ESA	15.4	319.85	79.87	40	31
		319.85	**120.86**	40	23

Function 4: Time 16.1 – 20.4 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Bentazon	17.1	**239.08**	**132.12**	22	25
		239.08	197.08	22	20
Dimethenamid OXA	17.2	269.92	166.01	18	17
		269.92	**198.01**	18	11
Alachlor ESA	17.8	**313.88**	**176.04**	35	21
		313.88	120.84	35	21
Acetochlor ESA	18.0	**313.88**	**162.05**	35	21
		313.88	120.84	35	21
Alachlor OXA	18.2	**263.95**	**160.06**	15	11
		263.95	192.10	15	6
Acetochlor OXA	18.5	263.95	128.97	17	44
		263.95	**146.06q**	17	10
Metolachlor ESA	18.7	**327.80**	**79.80**	45	28
		327.80	120.80	45	21
2,4-D	18.8	219.01	125.09	19	25
		219.01	**161.04**	19	15
MCPA	19.6	**199.05**	**141.11**	20	15
		199.05	155.11	20	10

Function 5: Time 19.2 – 35.0 min

Analyte	RT	Prnt(Da)	Dau(Da)	Cone(V)	Coll(eV)
Metolachlor OXA	20.8	277.90	174.03	10	17
		277.90	**206.03**	10	10
2,4-DB	23.0	246.97	125.09	15	29
		246.97	**161.03**	15	15
MCPB	23.3	**227.02**	**141.12**	14	15

* Quantififcation/Screening transistions in bold
* Many of the confirmation transistions are checked in the confirmation method due to dwell time restrictions.

9.3 LC Parameters

9.3.1 MDA PDP POS

Column – Zorbax™ RX-C8 (2.1 * 150 mm * 5 um)
Column Temperature – 40° C
Flow rate – 0.2 ml/min

Gradient

Time	0.1 % formic acid	Methanol
0.0	80%	20%
3.0	80%	20%
29.0	10%	90%
32.0	10%	90%
32.5	80%	20%

Run time – 38.5 minutes

9.3.2 MDA PDP NEG

Column – Zorbax™ RX-C8 (2.1 * 150 mm * 5 um)
Column Temperature – 60° C
Flow rate – 0.2 ml/min

Gradient

Time	0.1 % formic acid	Methanol
0.0	80%	20%
3.0	80%	20%
24.0	20%	80%
24.5	10%	90%
27.5	10%	90%
28.0	80%	20%

Run time – 35.0 minutes

10. Calculations

10.1 Determine the concentration of individual compounds according to the formula:

(LC/MS Conc.) * LOQ = ppt of analyte

WHERE:

LC/MS conc. = value instrument calculated from calibration table in relation to analyte LOQ level

LOQ = Analyte LOQ (ppt). See Section 12 for LOQ levels.

10.2 Report results in ppt without correction for recovery data.

11. Method References

11.1 Montana Department of Agriculture, “Determination of Polar Pesticides in Treated and Untreated Drinking Water Using Solid Phase Extraction and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry,”revision 1, December 2004.

11.2 Iowa Hygienic Lab, Vargo, John, “Determination of Acetochlor, Alachlor, Metolachlor, and Dimethenamid and their ESA and OXA Metabolites in Water by LC/MS/MS”.

11.3 Minnesota Department of Agriculture, "Chloroacetanilide and Chloroacetamide Metabolites in Water", revision 0, March 2002.

11.4 Minnesota Department of Agriculture, "Sulfonylurea, Imidazolinone and Sulfonamide Pesticides in Water", revision 0, March 1999.

12. **Tables**

12.1 PDP ES Positive LODs and LOQs

Compound	LOD(ppt)	LOQ(ppt)
Deethyl deisopropyl Atrazine	15.0	50.0
Hydroxy Atrazine	2.0	6.7
Imazapyr	2.5	8.3
Imazamethabenz acid	3.0	10.0
Imazamox	4.0	13.3
Imazapic	3.0	10.0
Imazamethabenz methyl	1.5	5.0
Imazethapyr	2.0	6.7
Propoxur	3.0	10.0
Bromacil	6.0	20.0
Nicosulfuron	8.0	26.6
Carbofuran	4.0	13.3
Triasulfuron	7.0	23.3
Thifensulfuron methyl	5.0	16.7
Carbaryl	7.5	25.0
Metsulfuron methyl	7.0	23.3
Imazaquin	5.0	16.7
Sulfometuron methyl	2.5	8.3
Diuron	4.0	13.3
Disulfoton sulfone	6.0	20.0
Prometryn	1.0	3.3
Metalaxyl	2.5	8.3
Linuron	6.0	20.0
Bensulfuron methyl	5.0	16.7
Siduron	2.0	6.7
Chlorimuron ethyl	6.0	20.0
Halosulfuron methyl	9.0	30.0
Neburon	3.0	10.0
Thiobencarb	2.5	8.3

12.2 PDP ES Negative LOD's and LOQ's

Compound	LOD(ppt)	LOQ(ppt)
Clopyralid	12.5	41.6
Picloram	12.5	41.6

Propachlor OXA	3.0	10.0
Metazachlor ESA	2.0	6.7
Flufenacet OXA	2.5	8.3
Dimethenamid ESA	2.0	6.7
Bentazon	0.3	0.8
Dimethenamid OXA	3.0	10.0
Alachlor ESA	12.5	41.6
Acetochlor ESA	9.0	30.0
Alachlor OXA	10.0	33.3
Acetochlor OXA	10.0	33.3
Metolachlor ESA	3.0	10.0
2,4-D	2.5	8.3
MCPA	1.5	5.0
Metolachlor OXA	3.0	10.0
2,4-DB	4.0	13.3
MCPB	3.0	10.0

12.3 PDP Positive Intermeditate Mix A concentrations, 50 mL volume

Compound	Standard LOD(ppb)	Stock Volume(mL)	Concentration ppm
DEDI Atrazine	3.0	15.0*	30.0
Hydroxy Atrazine	0.4	2.0*	4.0
Imazapyr	0.5	0.25	5.0
Imazamethabenz acid	0.6	0.30	6.0
Imazamox	0.8	0.40	8.0
Imazapic	0.6	0.30	6.0
Imazamethabenz methyl	0.3	0.15	3.0
Imazethapyr	0.4	0.20	4.0
Propoxur	0.6	0.30	6.0
Bromacil	1.2	0.60	12.0
Nicosulfuron	1.6	0.80	16.0
Carbofuran	0.8	0.40	8.0
Triasulfuron	1.4	0.70	14.0
Thifensulfuron methyl	1.0	0.50	10.0
Carbaryl	1.5	0.75	15.0
Metsulfuron methyl	1.4	0.70	14.0
Imazaquin	1.0	0.50	10.0
Sulfometuron methyl	0.5	0.25	5.0
Diuron	0.8	0.40	8.0
Disulfoton sulfone	1.2	0.60	12.0
Prometryn	0.2	0.10	2.0
Metalaxyl	0.5	0.25	5.0
Linuron	1.2	0.60	12.0
Bensulfuron methyl	1.0	0.50	10.0
Siduron	0.4	0.20	4.0

Chlorimuron ethyl	1.2	0.60	12.0
Halosulfuron methyl	1.8	0.90	18.0
Neburon	0.6	0.30	6.0
Thiobencarb	0.5	0.25	5.0

* - DEDI and Hydroxy Atrazine stock solutions are 100 ppm.

12.4 PDP Negative Intermeditate Mix A concentrations, 50 mL volume

Compound	Standard LOD(ppb)	Stock Volume(mL)	Concentration ppm
Clopyralid	2.5	1.25	25.0
Picloram	2.5	1.25	25.0
Propachlor OXA	0.6	0.30	6.0
Metazachlor ESA	0.4		
Flufenacet OXA	0.5	0.25	5.0
Dimethenamid ESA	0.4	0.20	4.0
Bentazon	0.05	0.025	0.5
Dimethenamid OXA	0.6	0.30	6.0
Alachlor ESA	2.5	1.25	25.0
Acetochlor ESA	1.8	0.90	18.0
Alachlor OXA	2.0	1.00	20.0
Acetochlor OXA	2.0	1.00	20.0
Metolachlor ESA	0.6	0.30	6.0
2,4-D	0.5	0.25	5.0
MCPA	0.3	0.15	3.0
Metolachlor OXA	0.6	0.30	6.0
2,4-DB	0.8	0.40	8.0
MCPB	0.6	0.30	6.0