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1. Purpose:

To provide a listing of PDP identified required compounds for groundwater and the QC criteria that will be followed by the laboratory.

2. Scope:

This standard operating procedure (SOP) shall be followed by the Minnesota Department of Agriculture (MDA) laboratory analyzing groundwater samples for PDP program.

3. Outline of Procedure:

- 5.1 Compounds for Minnesota Groundwater Multi Residue Screen
- 5.2 Standards
- 5.3 General Method Validation Requirements
- 5.4 Marker Pesticides
- 5.5 Process Control Compounds
- 5.6 Establishment of Limits of Detection (LODs) and Limits of Quantitation (LOQs)
- 5.7 Verification of LODs and LOOs
- 5.8 LOD Check
- 5.9 Changing LODs
- 5.10 Determination of Method Range
- 5.11 Precision and Accuracy Data Collection
- 5.12 Method Evaluation Reporting
- 5.13 Method Validation Evaluation by MPO
- 5.14 Blanks and Spikes Required per Set and Continuing QC
- 5.15 Criteria for Method Validation and Continuing QC
- 5.16 Proficiency Testing Results Criteria
- 5.17 Measurement Uncertainty

4. References:

- Current PDP Program Plan
- PDP-QC SOP Chemical Compounds, PDP Commodity Grouping, Method Validation and Quality Control
 - Attachment 1 Method Evaluation Flowchart

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- Attachment 5 Method Evaluation Reporting Forms [Verification of LODs, LOD Check, Determination of Method Range, Precision and Accuracy Data Collection]
- Attachment 6 Process Control and Spike Recovery Acceptability Flowchart

5. Specific Procedures:

This SOP represents minimum PDP requirements and is presented as a general guideline. Each laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in that laboratory.

5.1 Compounds for Minnesota Groundwater Multi Residue Screen

Water Compound	Pest Code	Group	Priority
2,4,5-T	312	20	
2,4,5-TP	AJE	20	
2,4-D	26	20	1
2,4-DB	317	20	1
Acetamiprid	B80	1	
Acetochlor	807	1	2
Acetochlor ethanesulfonic acid (ESA)	ABN	20	2
Acetochlor oxanilic acid (OA)	ABO	20	2
Alachlor	227	1	2
Alachlor ethanesulfonic acid (ESA)	ABP	20	2
Alachlor oxanilic acid (OA)	ABQ	20	2
Aldicarb sulfone	168	14	1
Aldicarb sulfoxide	169	14	1
Atrazine	305	9	1

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Water Compound	Pest Code	Group	Priority
Azoxystrobin	B48	22	
Bensulfuron methyl	ABR	16	3
Bentazon	758	17	1
Boscalid	AEO	1	2
Bromacil	153	16	3
Carbaryl	102	14	1
Carbofuran	180	14	1
Chlorimuron ethyl	717	16	3
Chlorothalonil	164	2	3
Chlorpyrifos	160	11	1
Clomazone	719	17	3
Clopyralid	B46	20	2
Cyanazine	228	9	2
DCPA (Dacthal)	134	3	2
Desethyl atrazine	964	9	1
Desethyl-desisopropyl atrazine	784	9	1
Desisopropyl atrazine	785	9	1
Diazinon	24	11	1
Dicamba	155	20	
Dichlobenil	324	2	1
Dichloprop	A25	20	
Dimethenamid	ADD	1	2

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Water Compound	Pest Code	Group	Priority
Dimethenamid ethanesulfonic acid (ESA)	AFB	20	2
Dimethenamid oxanilic acid (OA)	AEY	20	2
Dimethoate	171	11	1
Disulfoton sulfone	216	11	1
Diuron	32	16	3
EPTC	200	14	2
Ethalfluralin	721	7	3
Flufenacet oxanilic acid (OA)	AEZ	20	2
Fluometuron	701	16	1
Fonofos	163	11	1
Halosulfuron methyl	AEH	16	3
Hexazinone	633	9	
Hydroxy atrazine	AED	9	2
Imazamethabenz acid	AGE	16	3
Imazamethabenz methyl	753	16	
Imazamox	ACA	16	3
Imazapic	ACZ	16	3
Imazapyr	ACB	16	3
Imazaquin	ACC	16	3
Imazethapyr	ACD	16	3
Imidacloprid	967	1	
Isoxaflutole	B15	17	

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Water Compound	Pest Code	Group	Priority
Isoxaflutole deg	AJD	17	
Linuron	129	16	3
Malathion	52	11	1
Malathion oxygen analog	208	11	1
MCPA	318	20	2
МСРВ	620	20	2
MCPP	A42	20	
Mesotrione	AJA		
Metalaxyl	607	1	3
Methidathion	197	11	1
Methomyl	159	14	1
Metolachlor	283	1	2
Metolachlor ethanesulfonic acid (ESA)	ACG	20	2
Metolachlor oxanilic acid (OA)	ACH	20	2
Metribuzin	181	9	2
Metribuzin DA	185	9	
Metsulfuron methyl	ACI	16	3
Myclobutanil	679	1	1
Neburon	61	16	3
Nicosulfuron	ACM	16	3
Norflurazon	596	17	
Oxamyl	537	14	1

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Water Compound	Pest Code	Group	Priority
Parathion methyl	57	11	1
Pendimethalin	230	7	3
Permethrin, cis	222	8	1
Permethrin, trans	223	8	1
Phorate	148	11	1
Phorate oxygen analog	928	11	1
Phorate sulfone	189	11	1
Phorate suloxide	190	11	1
Picloram	329	20	2
Prometon	942	9	2
Prometryn	249	9	2
Propachlor	675	1	2
Propachlor esa	AJC	20	
Propachlor oxanilic acid (OA)	AFA	20	2
Propanil	341	1	3
Propazine	333	9	2
Propiconazole	264	1	1
Propoxur	162	14	
Saflufenacil	AHZ	16	
Siduron	ACT	16	3
Simazine	149	9	2
Sulfometuron methyl	ACP	16	3

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Water Compound	Pest Code	Group	Priority
Tebuconazole	A58	1	1
Tebupirimphos	A59	11	1
Tebuthiuron	780	16	3
Tembotrione	AJB		
Terbufos	205	11	1
Tetraconazole	ADO	1	1
Thiamethoxam	B43	1	
Thifensulfuron methyl	AEQ	16	
Thiobencarb	726	14	2
Triallate	621	14	2
Triasulfuron	ADP	16	3
Triclopyr	731	20	1
Trifluralin	151	7	3
Triticonazole	ADR	1	1

5.2 Standards

The laboratory will follow the guidance listed in PDP-QC SOP "Chemical Compounds, Method Validation and Quality Control".

5.3 General Method Validation Requirements

The laboratory will follow the validation guidelines outlined in PDP-QC SOP (most current revision).

5.4 Marker Pesticides

The compounds that the MDA laboratory will use as marker compounds are:

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Atrazine	Saflufenacil
Metolachlor	Imidacloprid
Dimethenamid	Bromacil
Desethyatrazine	Alachlor ESA
Acetochlor	Metolachlor ESA and OSA
Hydroxyatrazine	2,4-D
Imazethapyr	MCPA

5.5 Process Control Compounds

The laboratory will use Metazachlor and Metazachlor ESA for the process controls.

5.6 Establishment of Limits of Detection (LODs) and Limits of Quantitation (LOQs)

The laboratory will follow the validation guidelines outlined in PDP-QC SOP (most current revision).

5.7 Verification of LODs and LOQs

The laboratory will follow the validation guidelines outlined in PDP-QC SOP (most current revision).

5.8 LOD Check

The laboratory will follow the validation guidelines outlined in PDP-QC SOP (most current revision).

5.9 Changing LODs

The laboratory will follow the validation guidelines outlined in PDP-QC SOP (most current revision).

5.10 Determination of Method Range

The laboratory will follow the validation guidelines outlined in PDP-QC SOP (most current revision).

5.11 Precision and Accuracy Data Collection

• PDP program guidelines require that laboratories meet 50 to 150% recovery for process controls and matrix spike recoveries. It has been agreed with MPO that the laboratory can also use 3 standard deviations from the mean.

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- If the laboratory is outside the 50 150 criteria, the laboratory looks at the 3 standard deviations and determines if there is need to re-inject the sample.
- The laboratory will use 3 standard deviations for determining acceptance criteria for recovery data.

5.12 Method Evaluation Reporting

The method and validation data is sent to MPO for their reviewing and approval.

5.13 Method Validation Evaluation by MPO

Method validation data is reviewed by MPO for adherence to criteria as outlined in PDP-QC SOP and a determination is made whether each compound shall be considered Validated, designated as a Marginal Performing Analyte or designated as Unvalidated. A Letter of Concurrence summarizing validation status is then issued to the laboratory.

5.14 Blanks and Spikes Required per Set and Continuing QC

The following QC will be run with each batch of samples.

- Method Blank
- Reagent Blank
- Matrix Spike the complete list of compounds is spiked with each sample batch.

5.15 Criteria for Method Validation and Continuing QC

The laboratory will follow the guidelines outlined in PDP-QC SOP (most current revision).

5.16 Proficiency Testing Results Criteria

The laboratory will analyze the proficiency tests (PT) provided by MPO.

5.17 Measurement Uncertainty

The Minnesota laboratory will follow the guidance for measurement uncertainty (MU) in AOAC ALACC Criteria most current to date.

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- Updated Outline of procedure (section 3)
- Updated References (section 4)
- Added Section 5 as Specific Procedures
- Updated the table containing the compounds to be looked for in water screening (section 5.1)

Original October 2006 Monitoring Programs Office

Established commodity specific testing profile for groundwater