DEPARTMENT OF AGRICULTURE

www.mda.state.mn.us/incidentresponse

Fixed Based Commercial Laboratory Quality Assurance/Quality Control Plans and Requirements

Guidance Document 24

The Minnesota Department of Agriculture (MDA) requires that a current Quality Assurance/Quality Control (QA/QC) plan from a proposed commercial laboratory as well as specific analytical methods be pre-approved by the MDA for most agricultural chemical investigations. A QA/QC plan and analytical methods which were approved by the MDA and/or updated within the last two years are generally acceptable for fulfilling this requirement. Refer to Section D of this document for information required to be submitted or reported to the MDA. Please be aware that all laboratory data submitted to the MDA are subject to the reporting requirements listed below in item D.8.

A. Pesticides

The MDA maintains standard analytical lists for pesticides commonly required for agricultural chemical investigations in Guidance Document 26 Analytical Lists for Pesticide Incident Investigations. The MDA does not require specific methods for these analyses, therefore the proposed analytical methods also must be pre-approved. The minimum acceptable reporting limit will vary from compound to compound. Specific reporting limits for MDA standard list compounds in soil and water are attached to this document. The MDA laboratory staff will be available to discuss proposed analytical methods.

B. Nutrients – Nitrate/Nitrite-Nitrogen, Total Kjeldahl Nitrogen, Ammonia/Ammonium-Nitrogen

Nitrogen analyses required on agricultural chemical investigations for the compounds listed in Attachment 1 must have MDA approval for water and soil matrices. The laboratory will follow the requirements listed in Part D. of this document. Specific reporting requirements for MDA Standard list compounds are included in Attachment 1.

As a service to the public, the MDA will maintain and distribute a list of commercial laboratories (GD23 Pre-approved Commercial Laboratories: Fixed Base and Mobile) with MDA pre-approved QA/QC plans and analytical methods for standard MDA list compounds. **All information provided to the MDA will be public information unless clearly marked as a trade secret under Minn. Statutes, Section 13.37, Subd. 1(b).**

C. Additional Compounds

Additional compounds that could be requested on a site, including but not limited to arsenic, pentachlorophenol, polynuclear aromatic hydrocarbons (PAHs) and dioxins require the laboratory have ISO/IEC 17025:2005, National Environmental Laboratory Accreditation Program (NELAP), or Minnesota Department of Health Laboratory Accreditation. The laboratory will provide the current scope list and certificate for the accrediting authority to MDA.

With the exception of MDA List pesticides and nutrients described above, the accreditation must be for the same compounds of interest at the site. There may be some exceptions to this requirement that will be handled on a case by case basis. Again, the MDA does not require specific methods for these analyses, therefore proposed analytical methods for non-accredited compounds also must be pre-approved.

D. Commercial Laboratories

Commercial laboratories performing work for agricultural chemical investigations involving MDA pesticides and nutrient analyses must provide the information described below to the MDA prior to doing work on a site.

1) Quality Assurance Manual
2) Organizational Chart
3) Primary contacts including the project manager and QA manager/officer. Please provide names, phone numbers and email addresses.

4) Analytical Methods: The laboratory will provide the written methodologies employed by the lab. The minimum reporting limits that must be me on standard analytical list compounds are found in Tables 1, 2 and 3 of Attachment 1.

5) Method Detection Limit Studies: A method detection limit (MDL) study is required with each method and matrix submitted for approval. 40 CFR Part 136, Appendix B protocols will be followed. Include the initial MDL determination, and ongoing annual verification with the yearly submission. Include individual results with analysis dates, along with statistics used in the MDL calculation.

6) Proficiency Testing: The laboratory will participate in a proficiency testing (PT) program that includes the compounds listed in Attachment 1 at a minimum of once per year. Please refer to the A2LA website for approved providers (https://cabportal.touchstone.a2la.org/index.cfm?event=directory.index). The PT results must be submitted to the MDA on an annual basis. Compounds that fall outside the performance limits will require corrective action. Corrective actions will be forwarded to the MDA.

7) Quality Control Requirements: Laboratories participating in the MDA Pesticide Approval Program need to meet the QC outlined in *Guidance Document 29-Laboratory Quality Control and Data Policy Guidance (GD29).* Laboratories will not be required to fill out or submit the attachment that is included with GD29.

8) Reporting requirements for results submitted to the MDA: Please note that this information should be submitted for all compounds analyzed by the laboratory.

- Date of collection
- Date of extraction
- Date of analysis
- Results of analysis
- Internal Standards and Surrogate Recoveries as appropriate
- Method detection and reporting limits
- Assessment of data; include definitions of flags used in reporting
- Results of field quality control samples
- Results of quality control recoveries (spike recoveries, duplicate expressed as relative percent difference (RPD) or RSD
- Narrative to describe any problems with analyses. This includes but is not limited to homogeneity of samples received, raising reporting limits, matrix interferences (e.g., discussion as to issues with matrix spike or the decision to analyze the sample in duplicate or triplicate and forgo matrix spike because of high levels in sample) and other issues with analysis.
- Other information required by order, permit, etc.

Please direct any general questions or correspondence to: *Stuart Orlowski, Laboratory Coordinator, Pesticide Fertilizer Management Division at (651) 201-6148.*

Attachment 1

Reporting Limits (R.L.)

Table 1. MDA List 1 Pesticides			
Compound	**CAS #**	**Soil R.L. (mg/kg)**	**Water R.L. (ug/L)**
Acetochlor	34256-82-1	0.05	0.5
Alachlor	15972-60-8	0.05	0.5
Atrazine	1912-24-9	0.05	0.5
Chlorpyrifos	2921-88-2	0.05	0.5
Cyanazine	21725-46-2	0.05	0.2
Deethylatrazine	6190-65-4	0.05	0.5
Deisopropylatrazine	1007-28-9	0.05	0.5
Dimethenamid	876-68-8	0.05	0.5
EPTC	759-94-4	0.05	0.5
Ethalfluralin	55283-68-6	0.05	0.5
Fonofos	944-22-9	0.05	0.5
Metolachlor	51228-45-2	0.05	0.5
Metribuzin	21087-64-9	0.05	0.5
Pendimethalin	40318-45-4	0.05	0.5
Phorate	298-02-2	0.05	0.3
Propachlor	1918-16-7	0.05	0.5
Prometon	1610-18-0	0.05	0.5
Propazine	139-40-2	0.05	0.5
Simazine	122-34-9	0.05	0.5
Terbufos	13071-79-9	0.05	0.2
Triallate	2303-17-5	0.05	0.5
Trifluralin	1582-09-8	0.05	0.5

Table 2. MDA List 2 Pesticides			
Compound	**CAS #**	**Soil R.L. (mg/kg)**	**Water R.L. (ug/L)**
2,4,5-TP (Silvex)	93-72-1	0.1	0.5
2,4-D	94-75-7	0.1	0.5
2,4-DB	94-82-6	0.1	0.5
2,4,5-T	93-76-5	0.1	0.5
Bentazon	25057-89-0	0.1	0.5
Dicamba	1918-00-9	0.1	0.5
MCPA	94-74-6	0.1	0.3
Picloram	1918-02-1	0.1	0.5
Triclopyr	55335-06-3	0.1	0.5

Attachment 1

Reporting Limits (R.L.)

Table 3. MDA Nutrients			
Compound		**Soil R.L. (mg/kg)**	**Water R.L. (mg/L)**
Nitrate-Nitrogen	NO_3-N	10	NA
Nitrate/Nitrite-Nitrogen	NO_3/NO_2-N	NA	1.0
Total Kjeldahl Nitrogen	TKN	150	1.0
Ammonia/Ammonium-Nitrogen	NH_3/NH_4-N	20	0.02