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Interim Stack Sampling Performance Protocol, Version 1.0

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*Note: This document is an electronic (scanned) version of the original paper report and formatting may vary somewhat from the original.

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Acknowledgements

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Preface

The intent of this document is to provide general guidance as to specific procedures and sampling performance (i.e., conformance with this protocol and applicable source test methods) that Manitoba Environment requires as part of any demonstration or compliance program. This document should be referenced as the "Manitoba Department of Environment Stack Sampling Performance Protocol, version 1.0."

The basic rationale behind developing this protocol is quite simple. A sufficient number of 'gray areas' exist for the application of source test methods so as to make implementation of source test methods arbitrary in many cases. This can lead to a tendering process where two or more companies have submitted proposals based on different interpretations of the required sampling program. Since few test programs are conducted under ideal "text book conditions", a unifying document is necessary to create a platform that will provide a greater opportunity for the collection of representative point source emission data. Following the guidance provided in this protocol will result in a higher probability for acceptance of source test data, especially when Departmental personnel are not in attendance to provide immediate direction. It should be noted that this is the Department of Environment's accepted stack sampling performance protocol, and as such its requirements should be met or exceeded to avoid rejection of sampling results. The Department also expects any proposed deviations from the protocol to be identified prior to sampling - otherwise the sampling will normally be rejected.

This protocol is intended for use not only by Manitoba Environment, but could be used as a guide by personnel from facilities who are drafting tenders for demonstration programs (compliance and developing technology performance, etc.) as required by the department. Applicants filing proposals for new developments under the Environment Act would also benefit from the use of this protocol when submitting source emission data that were obtained using these specifications.

1. Introduction.

This protocol is designed to give facility personnel, such as engineering, technical or environmental staff, and consultants, general guidance as to Manitoba Environment's requirements for formal air emission testing from point sources.

The focus of this document is on i) acceptable process conditions for conducting emission testing, ii) validity of stack sampling procedures, iii) deviations from promulgated or otherwise accepted methods, and iv) reporting requirements.

Since it is recognized that any emission sampling program can only indicate or reflect emissions under which the sampling occurred, it is imperative that acceptable process conditions be defined before a sampling program is initiated, and that actual process conditions are documented during the collection of integrated stack samples.

As this is a guidance document only, specific methodologies for source sampling of air contaminants will not be discussed. However, unless otherwise stated or agreed upon in advance, Manitoba Environment will expect all sampling to be conducted in accordance with the latest release (version) of applicable methods as promulgated by recognized agencies such as the United States Environmental Protection Agency (US EPA) and Environment Canada's Environmental Protection Service (EPS). Methods from sources other than EPS and US EPA will only be considered by the Department if the method has been validated according to procedures documented in US EPA Method301 (Field Validation of Pollutant Methods from Various Waste Media). Appendix 1 includes a listing of all the US EPA and EPS methods that the Department currently accepts.

The use of this protocol could also assist Manitoba Environment's client groups in preparing specifications for tendering and will also assist qualified environmental or engineering consultants in the preparation of *formal* stack sampling proposals.

In various places throughout this document the term "Environment Officer" is used to designate an employee of Manitoba Department of Environment.

For application to specific sampling projects, the reader is encouraged to contact Manitoba Environment directly to ascertain specific requirements for that project. More information can be obtained by phone at (204) 945-7100 or by fax at (204) 948-2420.

2. Call for Proposals.

Where a formal proposal is required to be submitted in response to an invitation to tender, the proposal should identify the work proposed to be undertaken to quantify emissions associated with specific source. If accepted by the proponent (facility owner) and the Department, the proposal will eventually serve to fulfill requirements for the Department.

The proposal should include a description of the facility, the process (including raw materials and finished product), process flow diagrams, and identification of all points of emission including emergency and by-pass vents. Include stack diagrams indicating the sample ports, nearest upstream and downstream flow disturbances, and dimensions. The number of sample points, sample time, and minimum sample volume must be indicated. In addition, for non-ideal sampling locations[1] , specify the proposed compensating measures.

The Department will expect that acceptable process conditions for sampling will be defined in the formal proposal, and that all sampling will be conducted under those conditions.

Each proposal should include: i) a statement of qualifications for their analytical, sampling and consulting services, ii) credentials (including source testing certification) for personnel who may be committed to the project, iii) project experience (field sampling), iv) testing methods and protocols that the company has recently demonstrated proficiency, v) sampling equipment, vi) quality assurance project planning, and vii) technical support and resources.

Other required information includes: the date or version number of the method, the status of the method (i.e., draft, provisional, promulgated, etc.), and the issuing agency or organization. A complete copy of each sampling method should be included in an appendix. Include a copy of each analytical method if the analytical procedures are not otherwise detailed in the sampling method.

Since Departmental approval will be for the use of standard methods or specific modifications to promulgated methods, *any planned deviations must be accepted or approved in advance of their occurrence,* as this will affect the acceptance of the test data to all those involved.

In order to assess proper sampling, sample preparation and analysis conditions, a quality assurance/quality control (QA/QC) program is required. Obviously the complexity of the methods involved will dictate the complexity of the QA/QC program.

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(1) Sampling locations are classified as "Ideal", "Non-Ideal", and "Non-conforming" locations. These are defined as sampling locations at eight (or greater) stack or duct diameters downstream from the nearest flow disturbance (bend, constriction, etc.) and two or more stack or duct diameters upstream of the nearest flow disturbance for ideal locations. Non-ideal locations are defined as sampling locations less than eight but greater than or equal to two stack or duct diameters upstream and less than two but greater than or equal to 0.5 stack or duct diameters downstream respectively of a flow disturbance. Sampling locations positioned in stacks or ducts of less than 0.3 m diameter or at less than the above noted distances and are defined as non-conforming locations (see Section 9).

3. Acceptability of Process Conditions.

For compliance programs and demonstration trials (such as may be submitted in support of an Environment Act Proposal), the normal production rate and process activity will have to be defined by the facility prior to delineating acceptable conditions for sampling. Information required for defining normal process activity includes feed rate, material composition, operating temperature and pressure, processing time, equipment on-line, etc. All sampling is to be conducted under process and emission control device conditions as defined as acceptable to the test program.

With regard to process interruptions during performance and emission testing of facilities, tests will still be considered for validation providing the following conditions are complied with:

i) Sampling is discontinued at the onset of a process interruption or major upset;

ii) The sampling probe is removed from the stack, sealed with pre-cleaned caps or other material, as stipulated in the appropriate method(s), and the sampling train is maintained at the temperatures required by appropriate method(s);

iii) Sampling does not commence before: a) a time period equivalent to that required to stabilize the process has elapsed since normal (feed rate and operating parameters) operation was resumed (unless otherwise authorized by an Environment Officer), and b) the sampling train integrity is validated by a prescribed method leak check; and

iv) A process log is maintained and the occurrence of process interruption(s) and major upset(s) are noted in that log;

For emission testing where personnel from Manitoba Environment are on-site to audit sampling procedures, the following condition will also apply:

v) The occurrence of the process interruption or major upset is reported to the Environment Officer from Manitoba Environment;

A decision will need to be made on a case by case basis to determine if other non-stack sampling (process, other waste streams, etc., if applicable) will have to be discontinued during process upsets. If other sampling is discontinued during process upsets, timing of the restart should be re-synchronized with the stack sampling schedule.

The following table represents a typical matrix for process upset(s) during performance and emission testing.

Table 1. Example Process Upset/Source Emission Sampling Matrix.

Time Period	Action
< 5 minutes	Continue sampling if specified process conditions are maintained at or near optimized settings
> 5 minutes to 10 minutes	Discontinue sampling, remove probe from the stack and cap when nozzle is cool enough, maintain sampling train temperatures. When process conditions are stabilized, resume processing for an equal amount of time to the upset time, insert probe to the correct point and resume sampling
> 10 minutes	Discontinue sampling, remove probe from the stack, when nozzle is cool enough perform leak check, then cap nozzle, and maintain sampling train temperatures. When process conditions are stabilized, resume processing for xx (TBA) minutes, perform a leak check at the highest vacuum attained during the test, insert probe to the correct point and resume sampling.

4. Auditing.

For those sampling programs designated for auditing by Manitoba Environment staff, scheduling of the preliminary survey and all compliance sampling must be coordinated with Manitoba Environment so that staff are available to audit the required portions of the program.

5. Unobserved Compliance Testing Programs.

For those compliance testing programs where Departmental staff are not in attendance, stack sampling must be conducted under the on-site direction of a team leader certified by an accepted source testing training organization such as that offered by CSP Environmental Consultants/University of Windsor Department of Civil and Environmental Engineering or equivalent. This training must include a lecture program detailing sampling basics, field training, and a written examination.

6. Glassware Inventory.

Sufficient cleaned glassware, complying with method specifications, must be available to replace glassware that is broken during sampling. All glassware used in sampling programs must meet or exceed applicable method specifications for design, cleanliness, and composition. Proofing data must be supplied in accordance with applicable method specifications. Substitution or any changes in the configuration of components will only be considered if written requests are received in advance of the sampling program.

Glassware from QA trains must not be substituted for broken glassware in compliance sampling trains.

7. Calibration Data.

Reports certifying the calibration of the console (dry gas meter γ and orifice, in combination), pitot tubes (complete with nozzle and thermocouple), stack thermocouple, and nozzle are required to be available for inspection during the sampling program.

8. Preliminary Survey.

A preliminary survey is required at any facility where either no sampling has been conducted at the proposed sampling locations or where changes in the process have occurred since the last sampling program. This survey must include US EPA Methods 1 to 4 (or equivalent), conducted under conditions equivalent to those specified for formal sampling. Cyclonic and reverse flow determination are mandatory.

9. Stack Sampling in Non-Conforming Locations.

For the reasons listed below, source samples obtained from non-conforming locations (as defined in footnote #1) will not usually be accepted.

EPS and US EPA stack sampling methods allow considerable latitude in the placement of sampling ports/points. Corrections are included in the methods to account for 'non-ideal' sampling locations. Placement of sampling ports within the disturbed upstream and downstream flow areas, as denoted in the approved methods, are considered to be non-conforming locations.

The intent of the various source testing methods is the collection of representative samples. This premise can be compromised in general terms via two different mechanisms (with respect to a source under investigation), external and internal biases. External bias includes actions of a sample crew (whether intentional or unintentional) that affect the results (e.g., non-isokinetic sampling, errors in sampling train preparation, sampling under abnormal process conditions, etc.). Internal biases include flow disturbances (cyclonic and reverse), and skewed distribution patterns (such as occurs in close proximity to flow disturbances). Since carrier gases and their entrained particulate matter are subject to various laws of physics, empirical research has indicated that sampling close to flow disturbances can result in biased samples.

The standard technique is to sample for an equal length of time from equal area quadrants across a traverse (isokinetic sampling for sources that include particulate matter), with the volume of sample collected varying in direct proportion to the volumetric flow rate for that quadrant. However, this will not account for a skewed distribution of particulate matter that occurs when stack gases. react to a flow disturbance [compression, velocity changes (venturi effects, stagnant areas)], and the particulate matter is redistributed temporarily by the effects of inertia.

Once the stack gas re-enters an undisturbed section of a stack, the distribution of particulate matter begins to 'normalize', and representative samples can be collected from those locations.

Since stack sampling normally consists of the collection of an integrated sample (spatially and temporally), it is impossible to determine if equal area sampling from non-conforming locations is in effect biasing the results.

10. Validity of Stack Sampling Procedures

For methods that require isokinetic sampling, Manitoba Environment requires isokinetic sampling to be based on actual flow rates (orifice) during sampling [with minor corrections to maintain the calculated differential pressure (ΔH)], and will not accept sampling based on a pre-calculated volume per sampling interval, where the flow rate is dramatically changed during the final portion of the interval to "optimize" isokinetic conditions (i.e., target volume sampling).

Criteria for the determination of valid sampling procedures are specified in individual test protocols (see Appendix 1 for a listing of test methods currently accepted by the Department).

The maximum leakage rate limit of 0.02 cfm at a vacuum of 15 inches of mercury (over a one minute test period) is only applicable to pre-test conditions. The actual leakage rate must not exceed 0.02 cfm or four percent of the estimated sampling rate (over a one minute test period), whichever is less. Although protocols now require the nozzle inlet to be plugged for leakage determination, consideration will be given to plugging glassware immediately after the probe where glass lined probes are utilized and an acceptable leakage rate cannot be maintained at the nozzle. In any case, initial leak checks will require plugging of the nozzle inlet to ensure nozzle and probe liner integrity.

For leak checks during port change-over or other temporary interruptions to sampling, the leakage rate can be determined at a vacuum equal or greater to the maximum reading observed during the test. This will also apply t9 the final leak check upon completion of sampling.

Broken glassware and or failures of other sampling train components during sampling, or prior to sample recovery, will normally invalidate an individual test (or pair of concurrent tests, if applicable).

Any other unforeseen excursions from the test protocols that occur during sampling will be considered on a case by case basis.

11. Maximum Process Interruption Period.

Subject to conditions listed in Section 3, sampling must be stopped when process condition(s) do not conform to designated acceptable conditions. The maximum cumulative interruption period for a single test is four hours unless otherwise specified in a test method, or varied by an Environment Officer in attendance.

12. Replicate Sampling.

Unless otherwise specified, three valid tests are required for each sampling procedure (method), Tests that are determined to be invalid are to be repeated under conditions that meet the specifications of the accepted proposal.

13. Concurrent Sampling.

Under certain circumstances, concurrent sampling may be required in order to ensure the integrity of the results, data interpretation, etc. Because of the potential problem regarding passing all leak checks for multiple sampling trains used for concurrent emission testing, unless otherwise specified, this requirement for concurrent sampling will be waived for one of the triplicate set of tests. On the first occasion that this situation occurs, only the test in which a sampling train that fails to pass the leakage rate requirements (see Section 10) will have to be repeated under identical process conditions. If the situation re-occurs, then both of the concurrent tests will have to be repeated.

14. Sample Recovery.

Disassembly of major components of the stack sampling train and subsequent recovery of samples must be conducted in such a manner so as not to lose sample or introduce contaminants into the sample, Sample probes must be maintained in a level position with a maximum of one opening exposed to atmosphere at anyone time, any suitable precautions taken against sample loss in areas subject to wind. Both ends of the probe and all openings of the balance of the train are to remain capped with suitable material whenever possible.

15. Analysis.

For those tests that require laboratory analysis of field samples, documentation supporting specific laboratory accreditation for appropriate procedures (as documented in applicable sampling methods) will be required as part of the sampling report.

16. Reporting Conditions.

Unless otherwise directed, air emission rates should be reported per dry standard cubic metre calculated at 25°C and 760 mm of mercury. For the calculation of particulate matter, samples recovered from both the front and rear halves (excluding the silica gel) of the train are. to be combined, and carbon dioxide should be corrected to 12% for processes involving combustion.

Except as noted in the preceding paragraph, all data must be reported in an uncorrected form, and corrected (with appropriate supporting rationale) for laboratory blank removal, recovery rate (efficiency of spike recovery), etc.

17. Submission of Sampling Report.

A report detailing all calibrations, preliminary sampling, compliance sampling, results, conclusions, QA/QC program data, and process data (during the sampling program) is required to be submitted within 60 days of the sampling.

18. Special Requirements.

As it is impossible to predict the special requirements that individual Regulations, Licences, and Orders stipulate, this protocol is subject to modification on a case by case basis. Note that requirements of Regulations, Licences, and Orders take precedence over the specifications contained in this protocol. As indicated in Section 1, the reader is encouraged to contact Manitoba Environment directly to ascertain specific requirements for that project.

Appendix 1: Accepted Methods.

Currently accepted US EPA Methods.

Method #	**Parameter**
EPA Method 1	Traverse Points
EP A Method 2	Velocity & Flowrate
EPA Method 3	Gas Molecular Weight
EPA Method 4	Gas Moisture
EPA Method 5 (A to H)	Particulate Matter
EP A Method 6	SO_2
.EPA Method 7	NO_x
EP A Method 8	SO_2 & H_2SO_4
EPA Method 9/Alternate Method 1	Opacity
EPA Method 10	CO
EP A Method 11	H_2S
EPA Method 12	Inorganic Lead
EPA Method 13A	Fluoride
EPA Method 14	Fluoride (Al Plants)
EP A Method 15	H_2S, COS, CS_2
EP A Method 16	TRS Compounds (semi-CEM)
EPA Method 17	PM (In-stack Filter)
EPA Method 18	Gaseous Organics
EPA Method 19	PM, SO_2, NO_x
EPA Method 20	$S0_2/NO_x$ Stationary Turbines
EPA Method 21	VOC leaks
EPA Method 22	Fugitive Emissions
EPA Method 23	PCDD/PCDF
EPA Method 25	VOC (MDL= 50 ppm)
EP A Method 25A	VOC(MDL= <50 ppm)
EPA Method 26	HCl, HBr, HF, Cl_2, BR_2
EPA Method 26A	as above (isokinetic)
EPA Method 29	Metals (various)
EPA Method 201	PM_{10} Exhaust Gas Recycle
EPA Method 201A	PM_{10} Constant Sampling Rate
EP A Method 202	Condensable PM
EPA Method 205	Verification of Gas Dilution Systems
EPA Method 301	Field Validation of Methods
EPA Method 0011 (40CFR266}	Aldehyde & ketone
EPA Method 0011A (40CFR266}	Analysis Method for 0011
EPA Method 0050	HCl, Cl_2
SW-846 Method 0010	SVOC
SW-846 Method 0020 (SASS)	Part. & SVOC
SW-846 Method 0030	VOCs POHCs
Hexavalent Chromium (EPA)	Cr^{+6}
Isocyanates (EPA/Radian)*	TDI, MDI, HDI, MI

*Pending official release by EPA. This method has been peer reviewed but not officially promulgated.

Currently accepted EPS (Environment Canada) Methods.

Method #	Parameter
EPS 1/RM/1	Gaseous HCl
EPS 1/RM/2	Selected SVOCs
EPS 1/RM/3	Analysis of PCDDs, PCDFs, and PCBs
EPS 1/RM/4	CO
EPS 1/RM/5	Hq
EPS 1/RM/6	TRS Compounds
EPS 1/RM/7	Pb (in particulate matter)
EPS 1/RM/8	PM, Traverse Points, Velocity, Molecular Weight, Moisture
EPS 1/RM/15	Gaseous Emissions from Fossil Fuel-fired Boilers
EPS 1/PG17	Protocols and Perf. Specifications for CEM