

NR 700.13 Sample Preservation and Analysis

SECTION . NR 700.13(1) is created to read:

(1) All sampling, preservation, extraction and analytical methods used for compliance with NR 700 to 750 shall be according to the requirements in s. NR 716.13.

SECTION . NR 700.13(1) is renumbered as NR 700.13(2) and is amended to read:

(4 2) USE OF GASOLINE RANGE ORGANICS/DIESEL RANGE ORGANICS ANALYSIS. Soil or groundwater analyses for gasoline range organics or diesel range organics conducted for screening purposes shall be completed in accordance with the "Modified GRO, Method for Determining Gasoline Range Organics" and the "Modified DRO, Method for Determining Diesel Range Organics". NOTE: Screening purposes include determining whether a release has occurred, estimates made during site investigations or environmental assessments or other activities conducted in compliance with NR 700 to NR 750 to estimate the degree and extent of contamination. The "Modified GRO, Method for Determining Gasoline Range Organics: (WI-PUBL-SW-141) and "Modified Diesel Range Organics" (WI-PUBL-SW-14)) are available online at <http://www.dnr.state.wi.us/org/aw/rr/archives/pubs/SW140.pdf>. This method is referenced in s. NR 149, Appendix III, List of Authoritative Sources.

NR 700.13(2) is amended and moved to NR 716.13(11)

NR 700.13(3) is amended and moved to NR 716.13(5)

NR 716.13 Sampling and Analysis Requirements

(1) Responsible parties shall use laboratory analyses of environmental media samples which are collected, handled and analyzed in compliance with subs. (2) to ~~(4 2 17)~~ to confirm the nature and extent and evaluate the impacts of contamination, if a field investigation is required under s. NR 716.11 (1).

SECTION . NR 716.13(4) and (5) are renumbered as NR 716.13(2) and (3):

(4 2) All chemical and physical analyses for which a certification or registration test category is available under ch. NR 149 shall be conducted by a laboratory certified under ch. NR 149 for that test category.

(5 3) Responsible parties may use non-laboratory methods of sample analysis, including field screening with a photoionization detector or flame ionization detector, analysis with a field gas chromatograph, geophysical or downhole probe surveying, non-certified mobile laboratory analysis, immunoassays and other appropriate methods, to supplement the information derived from laboratory analysis of samples. If non-laboratory methods are used at a location from which a laboratory sample is collected, responsible parties shall use separate samples for the non-laboratory and the laboratory analyses, unless the target compound is not subject to loss or alteration through sample handling.

SECTION . NR 716.13 (6) is renumbered as NR 716.13 (4) .

(6 4) All soil samples obtained during the field investigation for the purpose of defining the degree and extent of the contamination shall be discrete, not composite, samples, unless the department explicitly approves in advance composite sampling for a specific site situation.

SECTION . NR 700.13(3) is renumbered as s. NR 716.13(5) and is amended to read:

(3 5) ~~HOLDING TIMES.~~ Maximum holding times for soils shall be in accordance with the sampling method, sample storage container and analytical methods used. following table:

Analysis Method	Sample Storage	Maximum Holding Times from Date and Time of Collection			
		Solvent Addition	Shipping	Extraction	Analysis
GRO/VOC/PVOC	VOC-vial	Immediately	4 days	21 days	21 days
	Brass Tube	Within 2 hours	4 days	21 days	21 days
	EnCore™	Within 48 hours	40 hours	21 days	21 days
VOC/PVOC	NA	NA	NA	NA	28 days
Confirmation					
DRO	VOC-vial	72 hours	72 hours	47 days	47 days

~~NOTE: The "Modified GRO, Method for Determining Gasoline Range Organics" (WI-PUBL-SW-141) and "Modified Diesel Range Organics" (WI-PUBL-SW-140)) are available from the Department of Natural Resources, Emergency and Remedial Response Section, 101 S. Webster St., Madison, WI 53507.~~

SECTION . NR 716.13(11) is renumbered as NR 716.13(6) and amended to read:

(116) Responsible parties shall provide for the following quality control and quality assurance procedures, at a minimum, when collecting samples for laboratory analysis for a field investigation conducted under this chapter:

(a) Chain of custody, which shall be documented in a format specified by the department, from the time of sample collection to the receipt of the sample by the analytical laboratory.

Note: Copies of the chain-of-custody format may be obtained from Public Information Requests, Bureau of for Remediation and Redevelopment, 101 S. Webster Street, P.O. Box 7921, Madison, WI 53707.

(b) For soil samples, one temperature blank for every batch of samples that require cooling for preservation, unless samples are received by the laboratory on ice and a temperature of ~~no greater than 4~~ from freezing to 6° C is maintained until their receipt by the laboratory.

(c) For water samples:

1. One replicate sample for every 10 or less samples.

2. One field blank for every 10 or less samples.

3. One trip blank for each batch of samples that will be analyzed for volatile organic chemicals.

4. One temperature blank for every batch of samples that require cooling for preservation, unless samples are shipped on ice and a temperature of ~~no greater than 4~~ from freezing to 6°C is maintained until their receipt by the laboratory.

(d) Decontamination of all sampling instruments between each sampling event, unless dedicated or disposable sampling devices are used in a manner that prevents cross contamination or other unintended contamination of samples.

SECTION . NR 716.13(12) is renumbered as (7) and amended to read:

(127) Responsible parties shall ensure that the following items are documented during the field investigation and are made available to the department upon request:

(a) Procedures for sampling and all other routine activities associated with the site investigation.

(b) A log of all routine and non-routine maintenance and calibrations performed on all instruments used during the field investigation.

(c) Field notes describing in detail the sequence of activities that took place during the field investigation.

SECTION . NR 716.13(8) is created to read:

(8) The reporting limit for volatile organic compound analysis and petroleum volatile organic compound analysis shall be the detection limit for the analytical method used. (detection limit required to be reported in 716.15(2)(f)).

SECTION . NR 716.13(2) is renumbered as 716.13(9):

(29) Responsible parties shall ensure that drinking water samples are collected, handled and analyzed according to the procedures specified in ch. NR 809.

SECTION . NR 716.13 (10) is created to read:

(10) Responsible parties shall ensure that groundwater samples are collected, handled and analyzed according to the procedures specified in s. NR 140.16(1), unless the department approves the use of an alternative procedure. The department may approve the use of an alternative procedure from one of the authoritative sources listed in s. NR 149, Appendix III, an alternate test procedure approved by the U.S. EPA, or, if the department determines that an appropriate procedure is not available in one of the authoritative sources listed in s. NR 149, Appendix III, from another source.

SECTION . NR 700.13(2) is renumbered as NR 716.13(11) and amended to read:

(211) VOLATILE ORGANIC COMPOUNDS (a) Soil samples collected for analysis of volatile organic compounds for compliance with chs. NR 700 to 734~~6~~ shall be preserved in methanol immediately after collection unless the samples are stored in a device which insures sample integrity. Samples stored in a suitable device, including brass tubes and EnCore™ samplers, shall be preserved in methanol according to sub. (3). ~~The department may approve alternate storage devices on a case-by-case basis prior to use in the field. Samples shall be preserved and handled as specified in section~~

~~8 and Table 1 of the "Modified GRO, Method for Determining Gasoline Range Organics".~~ according to the analytical method used. Sampling techniques shall be used that minimize volatilization from the sample. Extraction techniques shall be according to the analytical method selected. Analytical methods used shall be suitable for the matrix, type of analyte, expected level of analyte, regulatory limit, and potential interferences in the samples to be tested.

NOTE: Suitable analytical, preservation and extraction methods are included in the List of Authoritative Sources, ch. NR 149, Appendix III.

~~(b) Methanol-preserved soil samples shall be extracted in the vial by shaking for 2 minutes and sonicating in an ultrasonic water bath for 20 minutes. After sonication, an aliquot not larger than 100 microliters shall be removed and introduced into a purge and trap system as in par. 7.3.3.2.4 of method 5030A in "Test Methods for Evaluating Solid Waste (SW-846)".~~

~~(c) Methanol-preserved soil samples shall be analyzed by gas chromatography or gas chromatography/mass spectrometry using capillary columns. Suitable analytical methods are found in SW-846.~~

Do we need anything specific to groundwater VOC analysis, or is (10) adequate? Should the soil sampling/analysis requirements be broader than for VOCs?

SECTION . NR 716.13 (3) is renumbered as (12) and amended to read:

~~(3)~~ **(12)** Responsible parties shall ensure that samples other than drinking water or groundwater are collected, handled and analyzed according to the procedures specified in "SW-846: Test Methods for Evaluating Solid Waste", November 1986, including December 1987 and November 1990 updates, add any updates?., published by the U.S. EPA, unless the department approves the use of an alternative procedure. The department may approve the use of an alternative procedure from one of the authoritative sources listed in s. NR 449.03-~~(5)~~ 149, Appendix III, an alternate test procedure approved by the U.S. EPA, or, if the department determines that an appropriate procedure is neither available from "SW-846: Test Methods for Evaluating Solid Waste" nor from one of the authoritative sources listed in s. NR 449.03-~~(5)~~ 149, Appendix III, from another source.

Note: Copies of "SW-846: Test Methods for Evaluating Solid Waste" may be accessed at the following web site: <http://www.epa.gov/epaoswer/hazwaste/test/main.htm>. ~~are available for inspection at the offices of the department of natural resources, the secretary of state, and the revisor of statutes. Copies may be obtained from the Government Printing Office, Room 190, Federal Building, 517 East Wisconsin Avenue, Milwaukee, WI 53202.~~

SECTION . NR 716.13(13) is created to read:

(13) Responsible parties shall sample and provide an analysis for the geochemical indicators and parameters, where natural attenuation is potentially a remedy or part of a remedy. These may include, but are not limited to dissolved oxygen, nitrate, dissolved manganese, total and ferrous iron, sulfate and methane, alkalinity, oxidation reduction potential, pH, temperature and conductivity.

SECTION . NR 716.13(7) through (10) are renumbered as (14) through (17), and NR 716.13(16) is amended to read:

(714) Responsible parties shall inspect monitoring wells installed for field investigations conducted under this chapter at least annually to determine whether they are providing a conduit to the subsurface, and shall take action to repair or abandon the well if necessary in accordance with ch. NR 141.

~~(8)~~ **(15)** Responsible parties shall measure and record to the nearest 0.01 foot the static water level elevation in each groundwater monitoring well prior to obtaining a groundwater sample from the well. The measurement point shall be the top of the well casing and shall be identified on the well itself if the top of the casing is not level.

~~(9)~~ **(16)** Where site investigation data or other information indicate it is appropriate, or when directed to do so by the department, responsible parties shall make a good faith effort to sample public or private water supply wells as part of a regular monitoring program or to determine the extent of groundwater contamination, or both. Responsible parties shall report all water supply well sampling results to the department and to the well owner within 10 days after receiving the sampling results. The report to the department shall include a preliminary analysis of the cause and significance of any contaminant concentrations observed in the samples and an identification of any substances that attain or exceed ch. NR 140 preventive action limits, as well as any other substances observed in the samples for which there are no ch. NR 140 groundwater quality standards. Private and public water supply wells to be sampled shall include:

(a) Those wells that are known or suspected to be affected by the groundwater contamination.

(b) Other wells that the department determines have the potential to be affected by the groundwater contamination.

~~(10)~~ **(17)** If the responsible parties are unable to sample a public or private well because the property owner refuses access, the responsible parties shall notify the department within 30 days of the refusal, and shall document in writing the efforts undertaken to gain access when requested by the department.