



SERVICE CHANGE BULLETIN 624.1 – March 12, 2018

In 2017, the USEPA finalized an update to the approved test procedures for sampling and analyzing samples under the Clean Water Act. That ["Methods Update Rule \(MUR\)"](#) became effective on September 27, 2017. One of the changes in the MUR was the mandatory replacement of EPA method 624 with EPA method 624.1. While reviewing and implementing the new requirements of EPA 624.1, Paragon recognized that an adjusted sampling protocol was needed for three specific volatile organic compounds (VOCs). After further examining the regulations at 40CFR136 that incorporated the MUR, Paragon determined the desired sampling protocol.

Paragon SOP-N0172, "Sampling Wastewater and Other Water for Volatile Organic Compounds (VOCs) Analysis using USEPA Method 624.1" describes this new sampling protocol. In essence, special collections are necessary if the VOCs to be determined include the following three compounds:

- (a) Acrolein, (b) Acrylonitrile, &/or (c) 2-Chloroethylvinyl ether

Our prescribed practice for sampling analytes for EPA 624.1 analysis reflects what is necessary to maximize all holding times to 14 days. It also presumes that samples do not contain residual chlorine; arrangements with the laboratory must be made in advance if a sample will contain residual chlorine.

It is important to note that all 3 of the above compounds are included in the routine NPDES AMR compound list and in the IPP TTO compound list. The potential also exists to impact other sampling events, depending on their specific compound lists.

Lastly, due to the special collections, separate analyses are also required.

Selected excerpts from 40CFR136 are provided below for reference.

40 CFR 136.3 Identification of test procedures

Table II—Required Containers, Preservation Techniques, and Holding Times

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table IC—Organic Tests⁵			
13, 18-20, 22, 24, 25, 27,28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C, ¹⁸ 0.008% Na ₂ S ₂ O ₃ , ⁵ HCl to pH 2	14 days.
26. 2-Chloroethylvinyl ether	G, FP-lined septum	Cool, ≤6 °C, ¹⁸ 0.008% Na ₂ S ₂ O ₃ ⁵	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons (Benzene, Ethylbenzene, Toluene)	G, FP-lined septum	Cool, ≤6 °C, ¹⁸ 0.008% Na ₂ S ₂ O ₃ , ⁵ HCl to pH 2 ⁹	14 days. ⁹
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C, ¹⁸ 0.008% Na ₂ S ₂ O ₃ , pH to 4-5 ¹⁰	14 days. ¹⁰

¹“P” is for polyethylene; “FP” is fluoropolymer (polytetrafluoroethylene (PTFE); Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; “G” is glass; “PA” is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); “LDPE” is low density polyethylene.

²Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (e.g., using a 24-hour composite sample; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.24(f)(14)(iv) and (v) (volatile organics).

³When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirement of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory have data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional ATP Coordinator under §136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See §136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0-6 °C, with minimum head space.

⁵ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g., chlorine) for cyanide analyses. Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (20th and 21st editions) addresses dechlorination procedures for microbiological analyses.

⁸Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

⁹If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

¹⁰The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.