

¹⁶Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.

¹⁷Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

¹⁸Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “≤ 6 °C” is used in place of the “4 °C” and “<4 °C” sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

¹⁹An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

²⁰To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

²¹Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

²²Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

²³For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB-EC) or 1681 (A-1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

²⁴The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (*i.e.*, that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (*i.e.*, within 15 minutes of collection).

[38 FR 28758, Oct. 16, 1973]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting § 136.3, see the List of CFR Sections Affected, which appears in the Finding Aids section of the printed volume and at www.govinfo.gov.

§ 136.4 Application for and approval of alternate test procedures for nationwide use.

(a) A written application for review of an alternate test procedure (alternate method) for nationwide use may be made by letter via email or by hard copy in triplicate to the National Alternate Test Procedure (ATP) Program Coordinator (National Coordinator), Office of Science and Technology (4303T), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave. NW., Washington, DC 20460. Any application for an ATP under this paragraph (a) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate test procedure is being requested.

(3) Provide a detailed description of the proposed alternate test procedure,

together with references to published or other studies confirming the general applicability of the alternate test procedure for the analysis of the pollutant(s) or parameter(s) in wastewater discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternative test procedure compared to the performance of the reference method.

(b) The National Coordinator may request additional information and analyses from the applicant in order to evaluate whether the alternate test procedure satisfies the applicable requirements of this part.

(c) *Approval for nationwide use.* (1) After a review of the application and any additional analyses requested from the applicant, the National Coordinator will notify the applicant, in writing, of whether the National Coordinator will recommend approval or disapproval of the alternate test procedure for nationwide use in CWA programs. If the application is not recommended for approval, the National Coordinator may specify what additional information might lead to a reconsideration of the application and notify the Regional Alternate Test

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Procedure Coordinators of the disapproval recommendation. Based on the National Coordinator's recommended disapproval of a proposed alternate test procedure and an assessment of any current approvals for limited uses for the unapproved method, the Regional ATP Coordinator may decide to withdraw approval of the method for limited use in the Region.

(2) Where the National Coordinator has recommended approval of an applicant's request for nationwide use of an alternate test procedure, the National Coordinator will notify the applicant. The National Coordinator will also notify the Regional ATP Coordinators that they may consider approval of this alternate test procedure for limited use in their Regions based on the information and data provided in the application until the alternate test procedure is approved by publication in a final rule in the FEDERAL REGISTER.

(3) EPA will propose to amend this part to include the alternate test procedure in §136.3. EPA shall make available for review all the factual bases for its proposal, including the method, any performance data submitted by the applicant and any available EPA analysis of those data.

(4) Following public comment, EPA shall publish in the FEDERAL REGISTER a final decision on whether to amend this part to include the alternate test procedure as an approved analytical method for nationwide use.

(5) Whenever the National Coordinator has recommended approval of an applicant's ATP request for nationwide use, any person may request an approval of the method for limited use under §136.5 from the EPA Region.

[77 FR 29809, May 18, 2012, as amended at 82 FR 40874, Aug. 28, 2017]

§ 136.5 Approval of alternate test procedures for limited use.

(a) Any person may request the Regional ATP Coordinator to approve the use of an alternate test procedure in the Region.

(b) When the request for the use of an alternate test procedure concerns use in a State with an NPDES permit program approved pursuant to section 402 of the Act, the requestor shall first submit an application for limited use

to the Director of the State agency having responsibility for issuance of NPDES permits within such State (*i.e.*, permitting authority). The Director will forward the application to the Regional ATP Coordinator with a recommendation for or against approval.

(c) Any application for approval of an alternate test procedure for limited use may be made by letter, email or by hard copy. The application shall include the following:

(1) Provide the name and address of the applicant and the applicable ID number of the existing or pending permit(s) and issuing agency for which use of the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate test procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Tables IA through IH of §136.3, or in the NPDES permit.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(5) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the reference method.

(d) *Approval for limited use.* (1) The Regional ATP Coordinator will review the application and notify the applicant and the appropriate State agency of approval or rejection of the use of the alternate test procedure. The approval may be restricted to use only with respect to a specific discharge or facility (and its laboratory) or, at the discretion of the Regional ATP Coordinator, to all dischargers or facilities (and their associated laboratories) specified in the approval for the Region. If the application is not approved, the Regional ATP Coordinator shall specify what additional information might lead to a reconsideration of the application.

(2) The Regional ATP Coordinator will forward a copy of every approval