



Standard Test Method for On-line Determination of Sodium in Water¹

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1. Scope*

1.1 This test method covers the on-line determination of trace amounts of sodium in water using an ion-selective electrode.

1.2 This test method is based on on-line application of the sodium ion electrode as reported in the technical literature (1-3).² It is generally applicable over the range of 0.01 to 10 000 $\mu\text{g/L}$.

1.3 The analyst should be aware that adequate collaborative data for precision and bias statements as required by Practice D2777 are not provided. See Section 16 for details.

1.4 The values stated in SI units are to be regarded as standard. The inch-pound units given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements see Section 6.

2. Referenced Documents

2.1 ASTM Standards:³

D1066 Practice for Sampling Steam

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3864 Guide for On-Line Monitoring Systems for Water Analysis

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

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² The boldface numbers in parentheses refer to the list of references at the end of this test method.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D3370 Practices for Sampling Water from Closed Conduits
D5540 Practice for Flow Control and Temperature Control
for On-Line Water Sampling and Analysis

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.

4. Significance and Use

4.1 Sodium is a pervasive contaminant and it is the first cation to break through deionization equipment. This test method allows measurement of micrograms per litre (parts per billion) concentrations of sodium in water to monitor low-sodium water sources for indications of contamination or proper operation. Applications include monitoring makeup systems, condensers, condensate polishers, feedwater, boilerwater, and steam.

4.2 This test method is more sensitive and selective than conductivity measurements on high purity samples.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.⁴ In many instances, reagent grade chemicals contain higher levels of sodium contamination than are compatible with this test method. It must be ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I. In addition, the sodium or potassium content shall not exceed 10 $\mu\text{g/L}$ (10 ppb) or 1 % of the lowest concentration to be determined, whichever is lower.

5.2.1 Single-distilled water passed through a mixed bed deionizing unit composed of strong cation and anion resins can

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

*A Summary of Changes section appears at the end of this standard

produce an effluent containing less than 1.0 µg/L (1.0 ppb) of sodium. If such water is stored in a closed alkali metal-free container, such as one made of polyethylene, TFE-fluorocarbon, or stainless steel, subsequent increases in conductivity, usually due to absorption of carbon dioxide, will not invalidate its use for this purpose.

6. Hazards

6.1 pH adjusting reagents are strongly alkaline and volatile. Use normal eye and skin protection when handling ammonia, ammonium hydroxide, dimethylamine, diisopropylamine, monoethylamine, or morpholine. Extra care is needed in handling the gas-permeable tubing exposed to reagents used with the passive diffusion reagent delivery system. Keep reagents in the open wherever possible and take necessary precautions to keep them from the respiratory tract in event of a spill or leak. Under certain conditions these reagents can produce an explosive mixture with air. OSHA standards must be followed.

7. Sampling

7.1 Sample the water for on-line sodium ion measurements in a flowing stream in accordance with Practice [D1066](#), Guide [D3864](#), Practice [D3370](#) and [D5540](#) as applicable.

7.2 Regulate the pressure of samples within the instrument manufacturer's requirements.

7.3 Regulate the temperature of samples that must be condensed, or cooled, or both, to a level between 15 and 40°C (59 and 104°F) or within manufacturer's requirements. For highest accuracy, bring the sample temperature close to the temperature of the standards during calibration.

7.4 When sample system plumbing has been newly installed, or has not been carrying process stream water for some time, or has been open to the atmosphere, it may take 24 h of purging to bring the sodium content at the receiving end down to the same level as the sample point, especially when the process stream is less than 1.0 µg/L (1.0 ppb). In the case of lines that are very dirty or have been subject to biological fouling, pumping a 25 % solution of nitric acid is effective for plastic and stainless lines. About 30 line-volumes of acid should be pumped through slowly, followed by the fastest practical purge of process water in the amount of 300 volumes. When using an acid-cleaning procedure, confine the acid to the dirty part of the system. Under no circumstances should the acid enter the measuring instrument.

7.5 Adjust the sample flow in accordance with the manufacturer's recommendation.

7.6 Where speed of response is not critical, sequential sampling of multiple streams may be effected with 3-way solenoid valves for sample selection. The 3-way valves allow samples not being measured to continue flowing (to drain) and to be current when they are selected. Automatic selection should include an adjustable timing device for typical sampling times near 10 min per point. It is not advisable to sequence sample streams of significantly different concentrations using sequential sampling.

8. Summary of Test Method

8.1 Sodium ion electrodes provide consistent logarithmic response over many orders of magnitude of concentration using the same principles as pH electrodes but with different ion selectivity. The electrode signal has a slope of approximately 59 mV/decade change in sodium ion concentration at 25°C (77°F).

8.2 This test method includes provision for the addition of pH adjusting reagent to suppress hydrogen ion concentration and assure accurate electrode response to sodium.

8.3 This test method is particularly adaptable to high purity water and is relatively free of interferences (1). The overall operating cost of this system is considerably less than that of on-line flame photometry, and it is more sensitive than electrical conductivity.

8.4 The repeatability of this test method is ±5 % of the reading.

9. Interferences

9.1 The sodium ion electrode, like all potentiometric electrode measuring systems, is responsive to changes in ion activity and not true concentration changes (that is, the response is to changes in concentration multiplied by an activity coefficient). However, as concentrations approach infinite dilution, activity coefficients approach unity and ion concentration and active ion concentration become very nearly equal.

9.2 The activity coefficient of sodium ion will vary with changes in the total ionic strength of the solution. Therefore, it is important to maintain either a low or constant ionic strength. A constant flow of pH adjusting reagent generally establishes a consistent ionic strength.

9.3 The sodium content of pH adjusting reagent, if delivered directly to the sample, must not be significant compared with the lowest concentration being measured. Any air contacting the sample must be sodium-free.

9.4 The sodium ion electrode is responsive to certain other monovalent cations. Interference by silver, lithium, hydrogen, potassium, ammonium, and other ions must be considered. The selectivity to interfering ions varies by electrode manufacturer. In the low-solids water to which this test method applies, silver and lithium ions are usually absent. Potassium ion, often contributed to the sample by the reference electrode, must be carried downstream away from the sodium ion electrode. Ammonium ion, present in many power plant samples, generally does not interfere with measurements greater than 1 µg/L (1 ppb). Measurements below 1 µg/L use a stronger base reagent that suppresses the ionization of ammonia.

9.5 Elevation of pH so that hydrogen ion concentration is 3 to 4 orders of magnitude lower than that for sodium generally makes the electrode response independent of variations in hydrogen ion concentration of the original sample. Any of the reagents mentioned is satisfactory to increase the pH to a level such that the electrode is essentially insensitive to hydrogen ion, within sodium ranges specified by the manufacturer. The

apparatus for sodium measurement may include pH measurement of the conditioned sample to assure absence of this interference.

9.6 The sodium ion electrode is not subject to interference from color, turbidity, colloidal matter, oxidants, and reductants.

10. Apparatus

10.1 *Measuring Instrument*—Use commercially available potentiometric specific ion monitors that have expanded-scale operation with adjustable ranges calibrated directly in sodium ion concentration units of micrograms per litre (parts per billion). Electrical output signals must be isolated from ground and from electrode input and may be scaled for logarithmic, linear, or bilinear ranges.

10.2 *Sodium Ion Electrode*—Use a commercially available sodium-sensitive electrode (sodium ion electrode). Because electrode selectivities vary among manufacturers, care must be taken that the electrode, reagent and sample conditions are compatible (see 9.4 and 9.5).

10.3 *Reference Electrode*—Use a reference electrode compatible with the measuring electrode.

10.3.1 When the sodium ion electrode has a silver-silver chloride internal half cell, the reference electrode should be silver-silver chloride. When the sodium ion electrode has a calomel internal half cell, the reference electrode should be calomel. Dissimilar reference electrodes may be used provided adequate compensation is made electronically to correct for the difference between the measuring electrode and the reference electrode. If the reference electrode filling solution is an interference in the measurement of sodium, then the reference electrode must be downstream from the measuring electrode (see 9.4 and 10.3.2).

10.3.2 If calomel electrodes are used, refer to Test Methods **D1293**. The electrolyte used in reference electrodes and all maintenance shall conform to the manufacturer's recommendations. With flowing junction reference electrodes to ensure the desired slow outward flow of electrolyte, the solution pressure inside the junction shall be kept somewhat higher than that outside the junction.

10.4 *Temperature Compensation*—Use an automatic temperature compensator in accordance with the manufacturer's recommendation.

10.5 *Flow Chamber*—For best results install the electrodes in a flow chamber and take the measurement on a flowing stream. Use a flow chamber as recommended by the manufacturer. If otherwise, design the flow chamber to minimize interference from the reference electrode and construct the chamber of inert materials such as plastic or stainless steel.

10.5.1 If a plastic is used, cast or machine from a solid block. Gasket electrodes to prevent in-leakage of air. Protection of electrodes shall be in accordance with the manufacturer's recommendations. Connections to the flow chamber must be *solution* or *earth* grounded. No glass or copper is permissible in flow chamber construction.

10.6 *Analyzer Fluidics*—Unattended automatic calibration may be provided using relays and valves for accurately adding

sodium standard solution with timing and sequencing controlled by the measuring instrument.

11. Reagents and Materials

11.1 *pH Adjusting Reagents*:

11.1.1 *Ammonia Gas*—Commercial anhydrous grade ammonia (NH₃) having a minimum purity of 99.9 %. Gas is absorbed directly by the sample.

11.1.2 *Ammonium Hydroxide*—Commercial ammonia solution, approximately 29 % NH₃ in water. Vapor diffuses into the sample through gas-permeable tubing.

11.1.3 *Diisopropylamine*—Commercial grade liquid is vaporized and transported by an inert carrier gas to the sample stream, or vapor diffuses into the sample through gas-permeable tubing.

11.1.4 *Dimethylamine Gas*—Commercial grade having a minimum purity of 99 %. Gas is absorbed directly by the sample.

11.1.5 *Monoethylamine*—Commercial grade vapor diffuses into the sample through gas-permeable tubing.

11.2 *Sodium Chloride Stock Solution* (1.00 mL = 0.100 mg Na)—Dissolve in water 0.2542 g of sodium chloride (NaCl), dried to constant weight at 105°C in water, and dilute to 1 L in a thoroughly cleaned polyethylene flask. Store sodium stock in a polyethylene or equally alkali metal-free container. Prepare standards of lesser concentrations by dilutions of the stock solution.

12. Calibration and Standardization

12.1 All procedures described in this section are subject to zero and calibration drift. Determine the frequency of calibration checks required to obtain the desired accuracy based on experience. Whenever a major shift in sodium concentration is noted, a calibration check is advisable.

12.2 Low sodium standards are subject to contamination from many sources. Calibrate using standards above 100 µg/L (100 ppb) to reduce the effects of contamination. To avoid most contamination connect the standard container to the measuring equipment so that the standard can be introduced without exposing it to nonfiltered air. Use manual or automatic valves to introduce the standard for calibration checks. Some analyzers use a known addition method that employs higher level sodium standards.

12.3 Run the sample and pH adjusting reagent through the flow chamber for at least 12 h before attempting measurements or calibration (24 h when the equipment is new) to purge sodium contamination from all parts of the sampling and measuring equipment, especially valves. In systems where serious damage to the measuring electrode may occur if the sample flow is interrupted, provide means to automatically discontinue the pH adjusting reagent flow.

12.4 *Sodium Ion Measurement with pH Adjustment by Addition of Anhydrous Ammonia*:

12.4.1 In this procedure, pH adjustment is by on-line addition of ammonia to the continuously flowing liquid sample. The pH level is maintained relatively constant at pH 11.0 to 11.1. Provide a reasonably stable sample flow rate,

sufficient to attain reasonable response time, but not so great as to unduly consume ammonia. Follow manufacturer's flow rate recommendations.

12.4.2 Pipe the standard sodium solution into the system to prevent contamination during calibration.

12.4.3 Turn on the instrument and allow it to warm up in accordance with the manufacturer's instructions.

12.4.4 Connect the analyzer to a water source expected to have sodium ion concentration below 10 µg/L (10 ppb). This may be demineralizer effluent or high quality process water. Start the water flow. Maintain the temperature of the sample below 40°C (104°F).

12.4.5 Allow the water to flow until a stable reading is obtained. For a new instrument this may be 24 h or more. It is extremely important that all new equipment and sample lines be thoroughly flushed to remove any residual sodium (see 12.3).

12.4.6 After a stable reading is obtained, calibrate the instrument according to the manufacturer's procedure.

12.5 *Sodium Ion Measurement with pH Adjustment by Passive Diffusion of Monoethylamine, Ammonium Hydroxide, or Diisopropylamine, and Calibration by Double Known Addition (DKA):*

12.5.1 In this procedure, pH adjustment is accomplished by passive diffusion, wherein the sample passes through a gas permeable tubing coil present in a reagent bottle containing monoethylamine, diisopropylamine, or ammonium hydroxide, which diffuses through the tubing wall and redissolves, raising the sample pH to about 11. Sample flow rate must be sufficient to attain reasonable response time. Manufacturer's sample flow rate recommendations must be followed.

12.5.2 Install the instrument according to manufacturer's instructions, allowing for adequate flushing of the fluidic system for stabilization before initial calibration and use.

12.5.3 Calibrate the instrument using a double known addition technique in which sample concentration and electrode slope are determined simultaneously. To calibrate the instrument using DKA, follow manufacturer's instructions for ensuring calibration concentration increments and flow cell volume are properly programmed into the monitor.

12.5.4 Follow manufacturer's instructions for pipetting two increments of standards into the calibration port of the flow cell or this may be accomplished automatically with valves built into the apparatus.

12.6 *Sodium Ion Measurement with pH Adjustment by Addition of Diisopropylamine or Dimethylamine:*

12.6.1 In this procedure, pH adjustment is made by on-line addition of amine to the continuously flowing liquid sample. The pH level will be relatively constant at pH 11.0 to 10.5. Maintain sample flow rate sufficient to attain reasonable response time. Follow the manufacturer's amine and sample flow rate recommendations.

12.6.2 Pipe the standard sodium solution into the system to prevent contamination during calibration.

12.6.3 Turn the instrument on and make adjustments in accordance with the manufacturer's instructions.

12.6.4 Start the water flow. Keep the temperature of the sample below 40°C (104°F).

12.6.5 Allow the water to flow until a stable reading is obtained. Flush sample lines thoroughly to remove any residual sodium (see 12.3).

12.6.6 Calibrate either by direct adjustment to standard solutions made up by dilution of the sodium chloride stock solution (see 11.2) or by the *known increment* principle, a technique that depends on the unique logarithmic response characteristic of the sodium ion electrode (3). Follow manufacturer's procedures.

12.6.7 Discontinue the flow of calibration solution and return the instrument to the analysis of process water.

12.7 *Electrode Treatment*—Condition and maintain sodium ion electrodes and those that have been stored dry as recommended by the manufacturer. If the assembly is for intermittent use, keep it filled with water and the electrodes immersed between measurements.

12.8 *Flow Chamber Treatment*—On a new electrode chamber or one that has been inoperative for a prolonged period, flushing of the system with sample or with demineralized water of the highest purity attainable is recommended before measurements are made. Flushing for 24 h or more may be necessary to remove traces of sodium from newly installed equipment and tubing (see 7.4).

12.9 *Standard Reservoir*—Make provisions for introduction of a sodium standard. Provision can be made to permit draining of the flow chamber before the standard is introduced. Stainless steel or plastic valves are recommended. The standard storage reservoir must be of material that does not contribute sodium. Glass is not permissible. Either polyethylene bottles or bags, acid-washed and rinsed with water, are recommended for storage. Vent or pressurize the container in such a manner as to prevent contamination of the standard.

13. Procedure

13.1 After calibration and standardization as instructed in 12.4, 12.5, or 12.6, maintain reagent addition, if used, and direct the sample flow to the electrodes. Record data continuously or manually until the run is completed.

14. Response Time

14.1 The response time of the system is determined by two independent factors: the length of time required for a unit volume of new solution to flow through the system and completely rinse out the previous solution, and the response time of the sensing electrodes themselves. The use of filters will greatly increase the time of response depending on the size and type. Upscale response time is generally much faster than downscale.

15. Quality Control

15.1 Instrument and sensor calibration must be performed according to the manufacturer's instructions.

15.2 Establish a calibration frequency based on experience with the measurement system's drift rate in the specific process and the allowable tolerance of the process, both of which are unique.

15.3 After two-point calibration, the slope (span) and E_0 factors computed by the instrument must fall within acceptable ranges as recommended by the manufacturer.

15.4 Grab sample or offline calibration is difficult to achieve successfully. Proper precautions must be taken, which include efforts to collect the sample without introducing contamination and using a laboratory system for calibration that meets or exceeds the accuracy requirements of the monitoring system.

15.5 A method for dynamic dilution verification may be useful for verifying accuracy of the monitoring system at the low-levels of interest.

NOTE 1—Continued exposure to low-levels of sodium will cause the electrode's response to degrade. Recondition the electrode periodically when it is seen that the response time falls off during calibration and does not meet the manufacturer's specifications.

16. Precision and Bias

16.1 Neither precision nor bias data can be obtained for this test method from a collaborative study because this test method is an on-line determination. No suitable means has been found of performing a collaborative study to meet the requirements of Practice D2777. This inability to obtain precision and bias data for on-line determinations is recognized and stated in the scope of Practice D2777.

16.2 Statistical data on sodium measurements employing passive diffusion and double known addition techniques in power plants has been obtained as part of EPRI project RP2712-3 (4).

17. Keywords

17.1 on-line measurement; ion-selective electrode; specific ion

ANNEXES

(Mandatory Information)

A1. CONSTRUCTION OF pH ADJUSTMENT SYSTEMS

A1.1 Anhydrous Ammonia

A1.1.1 A system for anhydrous ammonia addition to the sample shall be constructed as follows: Use a commercially available ammonia regulator on the ammonia cylinder. Immediately downstream of the regulator install a filter (5- μ m stainless steel filter element). Downstream of the filter install a check valve and a needle valve. A flow meter or capillary shall be used between the needle valve and point of addition. A capillary can be used where ammonia enters the mixing tee. Stainless steel or plastic fittings and tubing should be used throughout. The standard solution connection and drain or bypass valve shall be installed in the sample line upstream of the ammonia addition point.

A1.2 Dimethylamine

A1.2.1 Sample water having a temperature of 15 to 40°C (59 to 104°F) passes through a regulator and block valve. Sample flow rate is indicated and controlled by a flow meter-needle valve combination. Gaseous dimethylamine from a cylinder is regulated and supplied to a mixing (absorber) column where sample pH is elevated to 11.0 to 11.5. The conditioned sample enters a baffled flow chamber where an

electrode system consisting of a sodium-ion electrode, reference electrode, and an automatic temperature compensator produce a voltage output proportional to the logarithm of sample sodium ion activity. The signal is displayed on an indicating monitor. Voltage and current outputs from the monitor are used for analog recording or data acquisition.

A1.3 Diisopropylamine

A1.3.1 The system for diisopropylamine addition is similar to that for dimethylamine addition described in A1.2. Diisopropylamine liquid is contained in a 5-gal reagent vessel. A gas-vapor transport technique using dry nitrogen carrier gas conveys the diisopropylamine to the mixer (absorber) column where sample pH is adjusted to 11.0 to 11.5.

A1.4 Ammonium Hydroxide, Diisopropylamine, or Monoethylamine Solution

A1.4.1 The reagent solution is held in a container with a length of gas-permeable rubber tubing carrying the sample through it.

A2. ELECTRODE RESPONSE

A2.1 Faulty Electrode Response

A2.1.1 The sodium ion measuring assembly should be calibrated periodically with two reference standards to check the linearity of response of the electrode combination at different sodium ion concentrations and to detect a faulty sensing or reference electrode. A faulty sensing electrode is indicated by failure to obtain a correct value of sodium ion concentration on a second standard after the meter has been standardized with the first one. Special care must be taken with regard to handling and contamination of reference standards and pH adjustment of standards as described in 5.1, 5.2, 9.3, 9.4, and Section 12.

A2.1.2 Contamination or inadequate pH levels can also result in insufficient response between two calibration standards and unfounded suspicion of measuring electrode failure.

A2.2 Impaired Electrode Response

A2.2.1 The sodium ion responsive properties of the glass sensing electrode may be impaired by substances such as oily materials and precipitates. Normally such substances will not be present in appreciable quantities in low-solids water except possibly during startup of new equipment or in cycling power plants.

A2.3 Electrode Cleaning Techniques

A2.3.1 When an electrode is suspected of being coated by oily substances or insoluble precipitates, or both, its usefulness can normally be restored by appropriate cleaning procedures. Oily matter can generally be removed by solvents such as

alcohol (7 parts ethanol, 3 parts water). Precipitates or insoluble inorganic coatings can normally be removed by immersing the bottom third of the electrode in hydrochloric acid (1 part concentrated HCl (sp gr 1.19), 9 parts water). After either solvent or acid cleaning, the electrode should be rinsed thoroughly with a flowing stream of Type I or other high-purity water. Following this rinsing, the electrode should be installed in the electrode chamber and flushed at least 2 h with pH adjusted water or until a stable reading is obtained. It should then be rechecked with two known reference standards.

A2.3.2 If faulty response is still obtained after replacement of a suitably conditioned sodium electrode, as indicated by failure to give satisfactory readings with two known sodium standard solutions, the reference electrode, measuring instrument, and temperature compensator (if automatic type) should be examined for maloperation. These systems occasionally need service and should always be thoroughly checked if replacement of the sodium electrode does not improve measurements.

A2.4 Temperature Compensation

A2.4.1 The sodium ion electrode and reference electrode pair shall be operated only on samples within the temperature range recommended by the manufacturer. An automatic temperature compensator as supplied by the manufacturer for use with the sodium-sensitive electrode shall be installed in the flow chamber along with the sodium sensitive electrode and reference electrode.

REFERENCES

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- (4) Rice, J. K., Sopocy, D. M., Dooley, R. B., "Quantification of Continuous Instrument Error," International Conference on Measuring Waterborne Trace Substances, Electric Power Research Institute and National Institute of Standards & Technology, Baltimore, August 1990.

SUMMARY OF CHANGES

Committee D19 has identified the location of selected changes to this standard since the last issue (D2791 – 93(2001)) that may impact the use of this standard.

(1) Test Method B on flame photometry has been withdrawn.

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