10   Soluble Salts[1]

J. D. RHOADES

U.S. Salinity Laboratory
Riverside, California

10-1   INTRODUCTION

The term soluble salts, as applied to soils, refers to major dissolved inorganic solutes. Soil salinity is described and characterized in terms of the concentrations (or, occasionally, the content) of soluble salts. The management and need for reclamation of saline soils are evaluated from measurements of such concentrations.

Ideally, it would be desirable to know the individual solute concentrations in the soil water over the entire range of field water contents and to obtain this information immediately in the field, without need for collection of soil samples or laboratory analyses. No practical methods are available at present to permit such determinations, although determinations of *total* salinity can be made in situ using electrical signals from various types of sensors (Rhoades, 1978, 1984). Such determinations are valuable for survey, monitoring, and irrigation and drainage needs, even though they give a measure of only total soluble salt concentration. Such methods are, in many cases, supplanting the need for more conventional analytical procedures.

Soluble salts in soils can be determined or estimated from measurements made (i) on aqueous extracts of soil samples, (ii) on samples of soil water itself obtained from the soil, (iii) in soil using buried porous salinity sensors that imbibe and equilibrate with soil water, and (iv) in soil (or soil pastes) using four-electrode probes or electromagnetic (EM) systems. The appropriate method of measuring soil salinity must be selected for the specific condition and purpose. If only a measure of total soluble electrolyte level is needed, salinity sensors, EM, or four-probe devices are recommended. To monitor soil water salinity as the soil dries between irrigations, the salinity sensor is recommended. When determination of a particular solute is needed, then either collection and extraction of soil samples or collection of water samples is required. Collection of water samples is more convenient for monitoring needs but is limited to relatively wet soil conditions. Soil sample extracts give relative comparisons only, since the soils are adjusted to unnaturally high water contents during extraction. A combination of the

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various methods minimizes need for sample collection and chemical analysis, especially when monitoring salinity changes with time and characterizing large field or project situations. For the latter, use of EM and four-probe techniques are recommended with supplemental use of the other methods as needed. Only methods for obtaining aqueous extracts of soil samples are dealt with in this section.

The following sections describe methods for determining soluble salts in water and soils using various methods and procedures for obtaining aqueous extracts of soil samples.

10-2 SATURATION EXTRACT AND OTHER AQUEOUS EXTRACTS

10-2.1 Principles

For some needs, knowing the composition of solutes in soil water at field water contents is desirable. However, present methods of obtaining soil water samples at usual field water contents are not practical for routine purposes. Thus, soil solution extracts often must be made at higher than normal water contents. Because the absolute and relative amounts of various solutes are influenced by the soil/water ratio at which the extract is made (Reitemeier, 1946), the ratio must be standardized to obtain results that can be applied and interpreted universally. Soil salinity is conventionally defined and measured on aqueous extracts of saturated soil pastes (U.S. Salinity Laboratory Staff, 1954). This soil/water ratio is used because it is the lowest reproducible ratio for which enough extract for analysis can be readily removed from the soil with pressure or vacuum and because it is often related in a predictable manner to field soil water contents. For these reasons, crop tolerance to salinity is often related to the electrical conductivity, or total electrolyte concentration, of the saturation extract (U.S. Salinity Laboratory, 1954).

Other extraction ratios, 1:1, 1:5, etc., are easier to use than that of saturation, but they are not so well related to field soil water contents. Errors from peptization, hydrolysis, cation exchange, and mineral dissolution also become greater for such extracts. As a compromise, Sonnevelt and van den Ende (1971) recommended a 1:2 volume extract. When relative changes rather than absolute solute concentrations are monitored, these wider extraction ratios may be used to advantage.

Once soil extract samples are obtained, laboratory chemical analyses are carried out to determine the electrical conductivity of the extract (σ_e) and the concentrations of major solutes (section 10-3).

10-2.2 Reagent

1. Sodium hexametaphosphate [$(NaPO_3)_6$] solutions, 0.1%: Dissolve 0.1 g of $(NaPO_3)_6$ in water, and dilute the solution to 100 ml.

10–2.3 Procedure

10–2.3.1 SATURATION EXTRACT

Weigh 200 to 400 g of air-dry soil of known water content into a plastic container having a snaptight lid. Weigh the container plus contents. Add distilled water to the soil with stirring until it is nearly saturated. Allow the mixture to stand covered for several hours to permit the soil to imbibe the water, and then add more water to achieve a uniformly saturated soil-water paste. At this point, which is generally reproducible to within ± 5%, the soil paste glistens as it reflects light, flows slightly when the container is tipped, slides freely and cleanly off a spatula, and consolidates easily by tapping or jarring the container after a trench is formed in the paste with the side of the spatula. After mixing, allow the sample to stand (preferably overnight, but at least 4 hours), and then recheck the criteria for saturation. Free water should not collect on the soil surface, nor should the paste stiffen markedly or lose its glisten. If the paste is too wet, add additional dry soil to the paste mixture. Upon attainment of saturation, reweigh the container plus contents. Record the increase in weight, which is the amount of water added. Calculate the saturation water percentage from the weight of oven-dry soil and the sum of the weights of water added and that initially present in the air-dry sample. After allowing the saturated soil paste to stand 4 or more hours, transfer it to a Büchner or Richards (1949) filter funnel fitted with highly retentive filter paper. Apply vacuum, and collect the filtrate in a test tube or bottle. If the initial filtrate is turbid, refilter or discard it. Terminate the filtration when air begins to pass through the filter. Add 1 drop of 0.1% $(NaPO_3)_6$ solution for each 25 ml of extract.

10–2.3.2 EXTRACTS AT SOIL/WATER RATIOS OF 1:1 AND 1:5

Weigh a sample of air-dry soil of appropriate size, and transfer it to a flask or bottle. Add the required amount of distilled water, stopper the container, and shake it in a mechanical shaker for 1 hour. If a mechanical shaker is not available, shake the container vigorously by hand for 1 min at least 4 times at 30-min intervals. Filter the suspension using highly retentive filter paper. (Discard or refilter the initial filtrate if it is turbid.) Add 0.1% $(NaPO_3)_6$ solution at the rate of 1 drop/25 ml of extract.

10–2.4 Comments

The weight of soil required will depend on the number and kind of determinations to be made on the extract, the analytical methods employed, and the salt content of the soil. In general, from one fourth to one third of the water in saturated soil pastes can be removed by vacuum filtration.

Soil samples should not be oven-dried before extracting for determination of soluble salts, because heating to 105°C converts at least a part of the gypsum $(CaSO_4 \cdot 2H_2O)$ to plaster of paris $(CaSO_4 \cdot 1/2H_2O)$. The latter hydrate has a higher solubility in water than does the former.

In determinations of extract water contents when a low ratio of soil to water is used, it is desirable to correct for the water content of the air-dry soil. For example, an air-dry sample containing 2% water on an oven-dry basis can be adjusted to a soil/water ratio of 1:1 by adding 98 ml of water to 102 g of air-dry soil. At soil/water ratios of 1:5 or greater, no correction is ordinarily made for water in the air-dry sample.

Special precautions should be taken in preparing a saturated soil paste with peat and muck soils or very fine or very coarse-textured soils. If possible, peat and muck soils should not be allowed to dry following collection because their saturation water content changes. Peat and muck, especially if coarse or woody, require an overnight imbibition period to obtain a definite endpoint for the saturation point. After the first wetting, pastes of these soils usually stiffen upon standing. Adding water and remixing then give a mixture that usually retains the characteristics of a saturated paste. With fine-textured soils, enough water should be added immediately, with a minimum of mixing, to bring the sample nearly to saturation. This minimizes the formation of clumps of soil during stirring, speeds the mixing process, and helps attain a more definite endpoint. Care should also be taken not to overwet coarse-textured soils. The presence of free water on the surface of the paste after standing is a very important indication of oversaturation in the case of coarse-textured soils. Even small amounts of free water can lead to appreciable errors in saturation paste water contents for these materials.

Sodium hexametaphosphate is added to the extract to prevent the precipitation of $CaCO_3$ from the extract upon standing. The quantity of $(NaPO_3)_6$ solution added increases the Na concentration ~0.5 ppm, or 0.02 meq/liter, which is inconsequential compared with the possible loss of $CaCO_3$. Alternatively, a subsample of the extract could be immediately diluted twofold and used for the Ca and alkalinity determinations.

Alternative methods of preparing the saturated soil paste have been described by Longenecker and Lyerly (1964), who proposed wetting the soil sample on a capillary saturation table, Beatty and Loveday (1974) and Loveday (1972), who recommended predetermining the amount of water at saturation on a separate soil sample using a capillary wetting technique, and Allison (1973), who recommended slowly adding soil to water (oversaturation method). Similar results are obtained with these methods. The choice of method is primarily one of personal preference.

Thymol can be added to the paste to minimize the effect of microbial activity on saturation extract composition during equilibration (Carlson et al., 1971).

The extracts should be stored at ~4°C until analyzed.

10-3 SOLUBLE CONSTITUENTS IN SOIL WATERS AND AQUEOUS EXTRACTS

The major solutes of interest found in soil waters and aqueous extracts of salt-affected soils are Ca^{2+}, Mg^{2+}, Na^+, K^+, CO_3^{2-}, HCO_3^-, SO_4^{2-}, Cl^-, NO_3^-, and H_3BO_3. There are many satisfactory analytical methods for de-

termining these solutes. These methods range from wholly automated to manual. The choice of which to use is usually determined by the number of samples being processed and the availability of analysts, automated equipment, or both. The methods described here are those in common use in laboratories having typical modern but not fully automated instrumentation. Methodology more suited to laboratories without such conveniences was given previously by Bower and Wilcox (1965).

10-3.1 Sequence of Analyses

Alkalinity and pH determinations should be made immediately on fresh extracts or on the solutions treated with hexametaphosphate. Next, electrical conductivity, σ, should be determined; it is a useful means of estimating total salt concentration (meq/liter $\cong 10\ \sigma$, in deci-Siemens per meter, dS/m). The cations can be determined in any sequence. After any three of the four major cations have been determined, the appropriate aliquot for the remaining cation can be estimated by deducting the sum of the three concentrations, in milliequivalents per liter, from $10\ \sigma$, in deci-Siemens per meter. Among the anions, NO_3^- and Cl^- determinations are normally made after alkalinity since they are simpler to measure than SO_4^{2-}. After that, SO_4^{2-} is determined; the appropriate aliquot is estimated from the difference between $(Ca^{2+} + Mg^{2+} + Na^+ + K^+)$ and (alkalinity $+ NO_3^-$ $+ Cl^-$). Finally, B is determined. Concentration of this solute is negligible compared with that of the major cations and anions, but it is still extremely important because of its pronounced toxicity even in small concentrations to many plants.

10-3.2 pH and Alkalinity

10-3.2.1 APPARATUS

1. Automatic potentiometric titrator.
2. Single probe combination pH electrode.

10-3.2.2 REAGENTS

1. Standard buffer solutions, pH 4.00 and 7.00.
2. Standard hydrochloric acid (HCl), approximately 0.0200N.

10-3.2.3 PROCEDURE

With electrode immersed in standard pH buffer, set the potentiometer to the pH (7.00) of the first buffer solution. Rinse electrode, and repeat calibration using the second pH buffer. Rinse electrode, immerse in 1 to 20 ml of sample solution (contained in a 50-ml plastic beaker along with a microsize, Teflon-coated magnetic stirring bar), and initiate the automatic titration operation using the full titration curve display mode. The initial potential recorded on the strip chart gives the sample pH. The volumes of titrant delivered to produce inflection points for CO_3^{2-} and HCO_3^- are ob-

tained from the titration curve (pH vs. volume of standard acid delivered from automatic burette).

10–3.2.4 CALCULATIONS

$$CO_3{}^{2-} \text{ in meq/liter} = 2P\,N\,1{,}000/\text{aliq}$$

where P is the number of milliliters of standard HCl of normality N to reach the $CO_3{}^{2-}$ inflection point (pH = 8.3), and aliquot is the sample volume in milliliters.

$$HCO_3{}^- \text{ in meq/liter} = (T - 2P)\,N\,1{,}000/\text{aliquot}$$

where T is the total number of milliliters of standard HCl of normality N to reach the $HCO_3{}^-$ inflection point (pH = 4.5), P is the number of milliliters of standard HCl required to reach the $CO_3{}^{2-}$ inflection point, and aliquot is the sample volume in milliliters. The blank is determined using $CO_3{}^{2-}$ free distilled water.

10–3.3   Electrical Conductivity

For this determination, use a direct readout from a temperature compensating conductivity meter.

10–3.3.1 APPARATUS

1. Conductivity meter.
2. Conductivity flow cell with automatic temperature compensation.
3. Vacuum line and suction flask.

10–3.3.2 REAGENT

1. Standard potassium chloride (KCl) solutions, 0.010 and 0.100N: For 0.010N solution (1.412 dS/m at 25°C) dissolve 0.7456 g of KCl in distilled water, and add water to make 1 liter at 25°C. For 0.100N solution (12.900 dS/m at 25°C), use 7.456 g of KCl.

10–3.3.3 PROCEDURE

Rinse and fill the conductivity cell with standard KCl solution. Adjust the conductivity meter to read the standard conductivity. Rinse and fill the cell with the soil extract or water sample, and read the σ, corrected to 25°C, directly from the digital display.

10–3.3.4 COMMENTS

Because of marked differences in the equivalent weights, equivalent conductivities, and proportions of major solutes in soil extracts and water samples, the relationships between σ and salt concentration or between σ

and osmotic pressure are only approximate. They are still quite useful, however. These relationships are:

1) Total cation (or anion concentration), meq/liter $\cong 10 \times \sigma$, in dS/m.
2) Salt concentration, mg/liter $\cong 640 \times \sigma$, in dS/m.
3) Osmotic pressure, bars at 25°C $\cong 0.39 \times \sigma$, in dS/m.

10–3.4 Soluble Cations

Determine Ca^{2+}, Mg^{2+}, Na^+, and K^+ using an atomic absorption spectrometer or by methods outlined in sections 13–3.3.2 and 13–3.3.4 for K, 13–4.3.3 and 13–4.3.5 for Na, and 14–3.1 for Ca and Mg. If large numbers of samples are to be routinely analyzed, it is extremely helpful to have this unit equipped for automatic sample transport, sequencing, siphoning, reading, and recording.

10–3.4.1 APPARATUS

1. Atomic absorption spectrometer.
2. Sampling and sequencing system.
3. Acetylene gas (C_2H_2), commercial grade.

10–3.4.2 REAGENTS

1. Suppressant solution for Ca^{2+} and Mg^{2+}: Add 29.0 g of lanthanum oxide (La_2O_3), 250 ml of conc hydrochloric acid (HCl), and enough distilled water to make up to 500-ml volume. Add sufficient to aliquot and diluent (distilled water) to give 10% (by volume) of this $LaCl_3$ solution in the final solution.
2. Suppressant solution for Na^+ and K^+: Add 6.358 g of lithium chloride (LiCl) and make to 1 liter in distilled water (0.15N). Add enough to aliquot, and diluent (distilled water) to give 10% (by volume) of this LiCl solution in the final solution.
3. Standard cation solutions: Ca^{2+} (0–0.4 meq/liter), Mg^{2+} (0–0.1 meq/liter), Na^+ (0–1.0 meq/liter), and K^+ (0–0.1 meq/liter).

10–3.4.3 PROCEDURE

Adjust the atomic absorption spectrometer controls and settings for the cation to be run as recommended by the manufacturer. Set the atomic absorption spectrometer readout to read the upper and lower standard solutions. Then initiate transport/readout system, which automatically positions a sequence of samples, siphons and aspirates the samples in the air-acetylene flame, and reads and records (as a digital printout) the concentration of the cation in the aspirated solution. Standard solutions are inserted into the sampling rack every 20 samples to ensure stability of instrument calibration during the automated run. Two-hundred samples can be processed per hour without attendance after the automatic sample processing system is initiated. Alternatively, the same sequence of operations can be performed by hand.

10-3.4.4 CALCULATIONS

Concentration of cations in original sample in meq/liter

= (atomic absorption spectrometer readout, meq/liter in aspirated sample)

× (Analytical dilution factor).

With Ca, the dilution factor must include the 1:1 predilution made at sampling time to prevent precipitation of $CaCO_3$ during storage, if appropriate.

10-3.5   Chloride

Determine Cl^- in solution by electrometric titration with silver or alternative procedures outlined in section 26-3. Silver ions are automatically generated coulometrically, and the endpoint is indicated amperometrically (elapsed time is indicated to the nearest 0.1 sec). Since the rate of Ag^+ generation is constant, the amount of Cl^- precipitated is proportional to time. The proportionality between duration of titration and concentration is established using standard Cl^- solutions and a blank.

10-3.5.1   APPARATUS

1. Automatic coulometric/amperometric Cl^- titrator (section 26-3.5).

10-3.5.2   REAGENT

1. Nitric acid–acetic acid/polyvinyl alcohol (PVA): Dissolve 1.8 g of powdered PVA in 100 ml of demineralized water with heating and stirring, cool to room temperature. Add 6.4 ml of conc nitric acid (HNO_3) and 100 ml of glacial acetic acid to a 1-liter volumetric flask containing 600 ml of demineralized water, and mix thoroughly. Add the cooled PVA solution to the nitric acid–acetic acid solution, mix, cool, and make to volume with demineralized water. Store this nitric acid–acetic acid/ PVA reagent in a tightly stoppered container at room temperature. This reagent is stable for at least 12 months.

10-3.5.3   PROCEDURE

Add 4 ml of nitric acid–acetic acid/PVA reagent together with a sample aliquot (< 3 ml) to a titration vial. Position vial in titrator, immersing electrode assembly into the solution. Zero the timer, and initiate automatic titration at low, medium, or high current setting. Note the titration times of blank, standards, and samples.

10-3.5.4   CALCULATION

Cl^- in meq/liter

= (K)(Titration time of sample minus titration time of blank)/aliquot

where K is a standardization factor and aliquot is sample size in ml.

K = (Volume of Cl⁻ standard in ml)(Conc of Cl⁻ standard in meq/liter)/

(Titration time of standard minus blank).

10–3.6 Nitrate

Determine NO_3^- concentration using a specific ion electrode (section 33–6) or alternative procedures (section 33–7). Its concentration is primarily used to allow for a check on cation/anion balance, because NO_3^- seldom is an appreciable fraction of the anions in saline waters. It is an important contaminant in return flows from many irrigated areas and an indicator of poor fertility management.

10–3.6.1 APPARATUS

1. Electrometer.
2. Specific NO_3^- selective electrode.
3. Calomel reference electrode.

10–3.6.2 REAGENT

1. Standard nitrate (NO_3^-) solutions: 0.01, 0.10, 1.00, 10.0, and 100 in meq/liter.

10–3.6.3 PROCEDURE

Prepare electrode according to manufacturers directions. Place electrodes in 5 to 10 ml of sample or standard solution, and record millivolt readings. Determine concentration of NO_3^- in sample by comparison with standard curve.

10–3.7 Sulfate

Determine SO_4^{2-} in samples using one of two methods, depending on amount of sample available and its estimated SO_4^{2-} concentration (SO_4^{2-} is estimated initially from the difference between the sums of cations and anions, excluding SO_4^{2-}). Use the turbidimetric method whenever a sample is expected to contain at least 5 meq/liter SO_4^{2-}. When the concentration is less, use the potentiometric titration method employing a Pb^{2+} selective electrode or methods outlined in section 28–3.

10–3.7.1 TURBIDIMETRIC METHOD

Sulfate is converted to a $BaSO_4$ suspension under controlled conditions. The resulting turbidity is determined by a spectrometer and compared with a curve prepared from standard SO_4^{2-} solutions (Standard Methods, 1980, p. 439–440).

10–3.7.1.1 Apparatus.

1. Absorption spectrometer.
2. Magnetic stirrer.

10–3.7.1.2 Reagents.

1. Barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) crystals, 20 to 30 mesh.
2. Conditioning reagent: Dissolve 75 g of sodium chloride (NaCl) in 275 ml of water in a 500-ml volumetric flask with magnetic stirring bar, add, with stirring, 30 ml of conc hydrochloric acid (HCl), 100 ml of absolute ethanol, and 50 ml of glycerol. Rinse glycerol into flask. Continue stirring until NaCl dissolves. Remove stirring bar and make to volume with distilled water.
3. Standard sulfate (SO_4^{2-}) solutions, 0 to 1 meq/liter.

10–3.7.1.3 Procedure.

Remove suspended material from sample, if present, by filtration or ultracentrifugation. Run a blank to correct for color interference and residual suspended material. Dilute sample to obtain sample of <1 meq SO_4^{2-}/liter. Transfer 100-ml aliquots of standards, blank, and diluted samples into 250-ml Erlenmeyer flasks. Add 5.00 ml of conditioning reagent to each flask with a 5-ml automatic pipetter. Introduce a clean magnetic stirring bar into the flask of the sample to be analyzed (read blank, standards and then samples in that order). Place flask on magnetic stirrer preset at constant speed (use fastest speed possible without splashing, but do not change speed once runs are begun). While stirring, add, all at once, 0.2 g of barium chloride crystals with a measuring spoon. Stir for exactly 60 sec, then remove flask from stirrer. Read maximum absorbance with spectrometer set at 340 nm after 1 to 3 min. Construct standard curve and determine concentration of SO_4^{2-} in final solution by comparison.

10–3.7.1.4 Calculations.

Concentration of SO_4^{2-} in sample, meq/liter = (Analytical dilution factor)

$$\times \text{ (Concentration of } SO_4^{2-} \text{ from standard curve).}$$

10–3.7.2 POTENTIOMETRIC METHOD

Where the concentration in the sample is <5 meq/liter, SO_4^{2-} is determined by titration with lead percolate, with the endpoint (presence of excess Pb^{2+}) detected potentiometrically using a Pb^{2+}-sensitive specific ion electrode (Goertzen & Oster, 1972).

10–3.7.2.1 Apparatus.

1. Semiautomated potentiometric titrator.
2. Lead-sensitive specific ion electrode.
3. Calomel reference electrode.

10–3.7.2.2 Reagents.

1. Methanol, ASC grade.
2. Lead perchlorate [$Pb(ClO_4)_2$], 0.002 and 0.005N.
3. Sodium perchlorate ($NaClO_4$), 0.100N.

4. Sodium hydroxide (NaOH), 6N.
5. Perchloric acid (HClO$_4$), 0.014N.

10-3.7.2.3 Procedure.

Transfer an aliquot containing about 0.004 meq of SO$_4^{2-}$ into a 50-ml beaker. Add 0.6 ml of NaClO$_4$ solution, and make to 5 ml with distilled water. Add 11 ml of methanol, and adjust the pH to 4.3 to 4.4 with dropwise additions of either HClO$_4$ or NaOH as needed using pH meter. Place the magnetic stirring bar in beaker, insert electrodes (the millivolt reading should be in the range -240 to -280), and titrate with standard Pb(ClO$_4$)$_2$ solution using the automatic titration apparatus. Determine the equivalence point from the inflection point of the titration curve.

10-3.7.2.4 Calculation.

$$SO_4^{2-} \text{ in meq/liter} = (V)\,(N)/\text{aliquot}$$

where V and N are volume in milliliters and normality of standard Pb(ClO$_4$)$_2$ used in titration to endpoint, respectively, and aliquot is the sample volume in ml.

10-3.7.2.5 Comments.

The Pb electrode is subject to poisoning. If the potential reading is $>$ -240 to -280 mV, the electrode should be polished and cleaned.

10-3.8   Boron

Concentration of B in samples is determined by formation of the colored boric acid–azomethine complex and spectrometry (John et al., 1975; section 25-5) or alternative methods outlined in Chapter 25.

10-3.8.1 APPARATUS

1. Automated spectrometric system: Use plastic containers. Do not use borosilicate glassware.

10-3.8.2 REAGENTS

1. Buffer masking solution: Dissolve 250 g of ammonium acetate (NH$_4$OAc) and 15 g of ethylenediaminetetraacetic acid disodium salt (EDTA disodium) in 400 ml of deionized distilled water, and slowly adding 125 ml of glacial acetic acid.
2. Azomethine-H reagent: Dissolve 0.45 g of azomethine-H in 100 ml of 1% L-ascorbic acid solution. Fresh reagent should be prepared each week and stored in a refrigerator.
3. Stock solution: Dissolve 0.1143 g of boric acid (H$_3$BO$_3$) in 1 liter of water to obtain a stock solution of 20 ppm of B. Prepare standards containing 0.5 to 4 ppm B by diluting stock solution with water.

10–3.8.3 PROCEDURE

Pipette 1 ml of blank, standard solution, or sample into a 15-ml poly-propylene tube, and then add 2 ml of buffer. Mix contents of tube with an electrical stirrer. Add 2 ml of azomethine-H reagent. Stir thoroughly, and allow to stand at room temperature for 30 min. Measure absorbance at 420 nm. Determine concentration of B in sample by comparison with standard curve (0–4 ppm).

10–3.8.4 CALCULATION

Boron in ppm

= (Concentration read from standard curve)(Analytical dilution factor).

10–3.9   Accuracy of Analyses

Examine the analytical results to detect gross errors by looking for ex-pected interrelationships among the constituent cations and anions. First check water analyses for chemical balance (the sum of the equivalent con-centration of cations in solution must equal the sum of the anions). Differ-ences of 3 to 4% are acceptable. Larger deviations indicate either a large error in one or more of the determinations or the presence of some unde-termined constituent, but a good balance is not conclusive evidence that each of the determinations is accurate nor that all constituents have been de-termined. Therefore, look for additional expected relations in the analyses. For solutions having σ values < 10 dS/m, check to see if the σ, in deci-Siemens per meter, multiplied by 10 is approximately equal to the total cation or anion concentration, in milliequivalents per liter. If CO_3^{2-} is pres-ent in titratable amounts, the pH of the extract must be ≥ 8.3. The HCO_3^- concentration of irrigation water seldom exceeds 10 meq/liter in the absence of CO_3^{2-} but may exceed 20 meq/liter in drainage waters having pH's of 6 to 7.5. Compare sample results from a sequence of soil depths for presence of a constituent deviating from profile trends. More extensive tests are de-scribed in the Standard Methods for the Examination of Waste and Waste-water (1980).

Some errors are practically unavoidable in analytical work. The analyst's skill and judgment play a large part in controlling the extent of such errors. The validity of the results must be evaluated after chemical analyses are completed. The analytical methods described yield results of moderate accuracy, probably with less error than the variation due to sam-pling.

10–4   LITERATURE CITED

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