



Designation: D 4025 – 97

Standard Practice for Reporting Results of Examination and Analysis of Deposits Formed from Water for Subsurface Injection¹

This standard is issued under the fixed designation D 4025; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the manner in which the various results of examination and analysis to determine the composition of deposits formed from water for subsurface injection are to be reported.

1.2 All analyses shall be made in accordance with the test methods of ASTM, unless otherwise specified.

NOTE 1—While reporting of inorganic constituents in water-formed deposits as specified in Practice D 933 is sufficient for certain industries, this practice provides for the reporting of organic and biological materials as well as inorganic constituents.

NOTE 2—Consistent with practices in industries where subsurface injection of water is practiced, reporting includes specifying of inorganic constituents as probable molecular combinations of the species for which analyses are performed.

1.3 *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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 887 Practices for Sampling Water-Formed Deposits²
- D 932 Test Method for Iron Bacteria in Water and Water-Formed Deposits²
- D 933 Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits²
- D 993 Test Methods for Sulfate-Reducing Bacteria in Water and Water-Formed Deposits³
- D 1129 Terminology Relating to Water⁴

¹ This practice is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Dec. 10, 1997. Published December 1998. Originally published as D 4025 – 81. Last previous edition D 4025 – 93.

² Annual Book of ASTM Standards, Vol 11.02.

³ Discontinued—See 1987 Annual Book of ASTM Standards, Vol 11.02.

⁴ Annual Book of ASTM Standards, Vol 11.01.

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁵

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, reference should be made to Terminology D 1129 and Practice D 887.

4. History of Sample

4.1 Information regarding the source and history of the sample shall be included in the report of the analysis. This information should be that supplied by the individual submitting the sample as follows:

- 4.1.1 Name of company supplying the sample,
- 4.1.2 Name of location of plant, facility, and well,
- 4.1.3 Date and time of sampling,
- 4.1.4 Number of sample,
- 4.1.5 Name and other designation of equipment from which sample was removed,
- 4.1.6 Precise location from which sample was removed,
- 4.1.7 Operating temperature and pressure of water or brine at location of deposit,
- 4.1.8 Type of treatment applied to the water that formed the deposit,
- 4.1.9 An account of system operating conditions that may have contributed to deposition (for example, filter channeling, chemical pump outage, or increased system temperature),
- 4.1.10 Appearance and extent of deposit prior to removal,
- 4.1.11 Exact method that was used in removing the sample and notes concerning any contamination that might have occurred during the process,
- 4.1.12 Specific methods used for preservation of sample prior to and subsequent to removal,
- 4.1.13 Results of field tests made on the sample or related equipment,
- 4.1.14 An analysis of the water from which the deposit was formed, and

⁵ Annual Book of ASTM Standards, Vol 14.02.

4.1.15 Identity of sampler.

5. Significance and Use

5.1 This practice sets down the manner in which data obtained from other test methods should be reported. This is done in an effort to standardize the report form used.

6. Physical Characteristics

6.1 The macroscopic characteristics of the sample, as ascertained by the analyst, including any peculiarities that may be pertinent to the results of the examination, shall be included in the report.

NOTE 3—Characteristics such as the following may be recorded: color, form (scaly, slimy, etc.), texture (oily, smooth, friable, gritty, etc.), magnetic properties (as determined by test with magnet), and apparent structure (amorphous, crystalline, columnar crystals).

NOTE 4—Any discrepancies between the physical characteristics observed by the analyst and those reported by the sampler should be noted.

6.2 Characteristics observed during microscopic examination shall be reported and include a description of the gross characteristics and homogeneity or heterogeneity of the sample.

6.3 Any distinct observable crystal morphology such as monoclinic, rhombic, etc., shall be reported.

NOTE 5—Specific note of the presence of any biological material in the sample should be made.

7. Determinations to Be Reported

7.1 The major headings for which determinations shall be reported in a complete analysis are as indicated in Table 1. Specific determinations which shall be reported under some of the major headings are listed in Tables 2-4. In certain instances, knowledge of the operating practices and water chemistry will have suggested the need to perform additional analyses or indicated the need for a less complete analysis; consequently, results from determinations may not be reported for each individual item.

8. Reporting of Biological Components

8.1 The results of examinations conducted to determine the presence of microorganisms in the sample, such as those delineated by Test Method D 932 and Practice D 993, shall be reported. The organisms present should be identified by phyla with the relative abundance noted.

NOTE 6—In the absence of quantitative measurements, the microbiological constituents may be grouped as major, minor, and trace. In categorizing the constituents, the qualitative nature of the examination and the subjective appraisal of its proportion must be considered.

TABLE 1 Major Headings for Determinations to Be Reported

Microbiological examination
Moisture and volatile material
Solvent-extractable components:
Fluorocarbon-extractables
Water-solubles
Acid-solubles
Solvent-insolubles
Loss at 900°C
Ash
Inorganic components

TABLE 2 Acid-Soluble Portion Ions to Be Reported

Inorganic materials:	
calcium	carbonate
magnesium	sulfide
barium	sulfate
iron	phosphate
sodium	hydroxide ^A
	oxide ^A
Organic materials	

^A These ions are determined by difference. The specific ion reported is determined by direct evidence or in absence of such evidence as hydroxide.

TABLE 3 Ash—Common Constituents to Be Reported

calcium	zinc
magnesium	sodium
barium	potassium
strontium	phosphate
iron	sulfate
aluminum	chloride
silicon	
manganese	

TABLE 4 Common Inorganic Components Found in Water-Formed Deposits and Their K_{sp} Values

Inorganic Material	K_{sp}	Temperature, °C	Reference
Al(OH) ₃	1.3×10^{-33}	18–25	A
BaCO ₃	2.6×10^{-9}	25	B
Ba ₃ (PO ₄) ₂	3.4×10^{-23}	18–25	A
BaSO ₄	1.1×10^{-10}	25	B
CaCO ₃	4.9×10^{-9}	25	B
Ca(OH) ₂	4.7×10^{-6}	25	B
Ca ₃ (PO ₄) ₂	2.1×10^{-33}	25	B
CaSO ₄	7.1×10^{-5}	25	B
FeCO ₃	3.1×10^{-11}	25	B
Fe ₂ O ₃	1.1×10^{-36}	18	B
Fe(OH) ₃	2.6×10^{-39}	25	B
FePO ₄ ·2H ₂ O	9.9×10^{-29}	25	B
SrCO ₃	5.6×10^{-10}	25	B
Sr ₃ (PO ₄) ₂	4.0×10^{-28}	18–25	A
SrSO ₄	3.4×10^{-7}	25	B
ZnCO ₃	1.2×10^{-10}	25	B
Zn(OH) ₂	6.8×10^{-17}	25	B
ZnS	2.2×10^{-25}	25	B
Na ₂ CO ₃			
NaCl			
Na ₂ SO ₄			
S			
SiO ₂			
Other siliceous material			

^ALange's Handbook of Chemistry, Dean, J. A., Ed., McGraw-Hill Book Co., 13th Edition, pp. 5–7 to 5–12.

^BHandbook of Chemistry and Physics, Weast, R. C., Ed., Chemical Rubber Co., 1991, pp. 8–39.

NOTE 7—The results of the examination should be qualified on the basis of any known or suspected effects related to sample handling prior to or during analysis likely to influence the validity.

9. Reporting of Chemical Analyses

9.1 The major headings under which chemical analysis determination shall be reported are moisture and volatile material, solvent-extractable components, loss at 900°C, ash, and inorganic components.

9.2 All data shall be reported as weight percent both on an as-received basis and on a dry weight basis, with the exception of water and volatile material, which shall be reported on an as-received basis only.

9.3 The loss at 105°C shall be reported as the moisture and volatile material content of the sample.

9.4 Reporting of solvent-extractable materials shall be divided into organic solvent extractables, water-solubles, acid-extractables, and solvent insolubles.

9.4.1 The organic solvent-extractable constituents shall be reported in terms of the generic nature of the extracted material. When the extractable portion of the deposit comprises greater than 10 % on a dry weight basis of the deposit, more specific identification of its composition is advised.

NOTE 8—A fluorocarbon refrigerant is the preferred organic solvent for such extractions. Where another solvent such as toluene, hexane, chloroform, or carbon tetrachloride is used, either in place of, or in addition to the fluorocarbon, the solvent used in and the results of such extractions shall be reported.

NOTE 9—The generic nature should identify the chemical class to which the material belongs, such as asphaltene, paraffin, amide, phosphate ester, etc.

NOTE 10—Elemental sulfur will normally be extracted in part by organic solvents. If sulfur is identified in the extract, specific notation of its presence shall be made.

9.4.2 The water-soluble portion of the sample shall be reported. If the water-soluble portion of the sample is in excess of 10 % on a dry weight basis, determination for sodium, Na⁺; potassium, K⁺; magnesium, Mg⁺²; calcium, Ca⁺²; sulfate, SO₄⁻²; and chloride, Cl⁻, shall also be reported.

9.4.3 Reporting of the acid-soluble portion shall indicate the total proportion of such components. Results of analyses of the acid-soluble portion for ions and radicals in Table 2 shall also be reported. Where the equivalents of cations exceed the equivalents of anions, the difference shall be reported as hydroxide or oxide ion. Where there exists evidence from petrographic examination, X-ray analysis, or other direct analyses that either is specifically present in the sample, such evidence shall be reflected in the reported acid-soluble components. Otherwise, the difference shall be reported as hydroxide ion.

NOTE 11—It is recommended practice to use 15 % hydrochloric acid (1 + 7) in the determination of the acid-soluble content. In some cases, however, the history of the sample may indicate a preference for use of another acid. In either case, the type of acid used and its concentration shall be noted.

NOTE 12—Where significant discrepancies exist between the total acid-soluble portion and the inorganic materials identified, due regard must be given to identification of the presence of acid-soluble organic constituents.

9.5 The loss at 900°C shall be reported. This determination is generally indicative of the decomposition of organic components; however, the determination will also include loss of combined water.

NOTE 13—Recognition should be made of the fact that the process of heating to 900°C results in the conversion of certain inorganic salts. For example, hydroxides, carbonates, and sulfides are converted to oxides.

9.6 The total ash shall be reported with the constituents listed in Table 3 specified.

9.7 The inorganic constituents identified in an analysis shall be reported both as their common ionic species and also in terms of probable molecular combinations listed in Table 4. For

convenience of calculation, the ionic species may also be reported as equivalents.

9.7.1 The probable mineral composition shall be determined by association of ions on an equivalent basis so as to form the compound having the lowest K_{sp} (based on stability constant of metal complexes) value. After complete association of one of the ions in the first compound is achieved, the association is continued in a manner to form the compound with the next lowest K_{sp} and so on until all ions are associated. K_{sp} values for common minerals are also listed in Table 4. Where evidence exists from petrographic examination, X-ray diffraction, or other direct analyses, such evidence shall override the probable combinations of ions above.

NOTE 14—The use of K_{sp} values in determining probable mineral composition of a deposit should be approached and the results of such considerations regarded with caution. Conditions under which deposition occurred may be such as to cause major changes in K_{sp} values and may then substantially alter the likelihood of a given mineral being present in a deposit.

10. Completeness and Accuracy of Analysis

10.1 The completeness and accuracy of the analysis should be carefully appraised before reporting.

10.1.1 The items to be included in a complete report are specified in Table 1. Any listed for which determinations were not obtained should be so indicated.

10.1.2 Each determination reported shall be accompanied by indication of the established precision for the methods used.

10.1.3 The summation of the determinations of moisture and volatile material, loss at 900°C, and ash shall total 100 ± 5 % on an as-received basis.

10.1.4 The inorganic composition developed should account for 100 ± 5 % of the ash content of the sample.

NOTE 15—In appraising the relationship between ash content and inorganic composition, conversion of certain salts during the ashing process as indicated in Note 15 must be considered.

10.1.5 Consideration should also be given to reconciling the determinations obtained for ions and radicals comprising the water-soluble components and acid-extractables with the inorganic composition reported.

10.1.6 Where the loss at 900°C is in excess of 15 % of the sample on a dry weight, the determinations for fluorocarbon-extractables and acid-soluble organics should be appraised. Where discrepancies exist between the loss at 900°C and the extracted organics, additional work to identify nonextracted organic materials in the sample is indicated.

10.2 Quantitative determinations shall be reported to significant figures consistent with the precision of the method used (Note 18). When a quantitative determination is made and a negative result is obtained, it shall be reported as “0.0” with a notation as to the amount of sample used and the method of determination. When a determination has been omitted, but the heading of the determination is carried in a tabulation with determinations of other samples, the absence of a determination for any other specific sample shall be indicated by an entry “no determination” or by a dash. The terms “nil,” “none,” and “trace” shall not be used. If a qualitative determination is conducted for a specific component and shows the component

present, and no quantitative determination is made, the item shall be reported as “present” with the notation that the determination was qualitative. Where a qualitative determination shows presence and a quantitative determination shows absence, the item shall be reported as less than the precision of test with the precision of the test being specifically noted, such as “less than 0.5 %.”

NOTE 16—In so far as applicable, Practice E 29 shall be followed in expressing numerical results.

11. Reporting Results of X-Ray Diffraction and Petrographic Examination

11.1 All constituents identified by X-ray diffraction or petrographic examination shall be reported in accordance with the following rules (Note 19):

11.1.1 *Silicon Compounds*—Report all compounds containing silicon, except the simple alkali silicates, by their trivial name and combined oxide formula. Example: acmite, $\text{Na}_2\text{O}\cdot\text{Fe}_2\text{O}_3\cdot 4\text{SiO}_2$.

11.1.2 *Polymorphous Compounds*—Report compounds having more than one crystal form by their trivial name and chemical formula. Examples: calcite, CaCO_3 , and aragonite, CaCO_3 .

11.1.3 *Multiple Salts*—Report multiple salts by their trivial name and combined-compound formula. Examples: burkeite, $\text{Na}_2\text{CO}_3\cdot 2\text{Na}_2\text{SO}_4$, and malachite $\text{CuCO}_3\cdot \text{Cu}(\text{OH})_2$.

11.1.4 *Other Compounds*—Report compounds not covered by the above rules by their chemical name and chemical formula. Example: sodium chloride, NaCl .

11.1.5 *Controversial Compounds*—Report compounds whose nomenclature is in controversy in accordance with the latest revision of the Powder Diffraction File,⁶ subject to conformance with rules (11.1.1-11.1.4).

NOTE 17—If a compound can be reported by several rules, the rule first listed that covers that compound shall prevail.

NOTE 18—Free elements shall be considered as compounds in applying the above rules.

NOTE 19—If the compound does not have a trivial name and the rule calls for a trivial name, the chemical name shall be used.

NOTE 20—The Greek letter and Roman numeral conventions already adopted in the literature shall be accepted as standard.

12. Reporting Results of Spectrochemical Analysis

12.1 Results of spectrochemical analysis shall be reported in terms of elements present.

NOTE 21—The elements may be grouped as major, minor, and trace constituents, with due regard for the variation in the sensitivity of the method for the various elements reported. A satisfactory grouping in terms of percentage of the element present is as follows:

	Amount of Element Present, %
Major	over 5
Minor	0.5 to 5
Trace	under 0.5

While the identification of elements is positive, quantitative measurements are only approximations.

13. Keywords

13.1 data reporting; deposits; subsurface injection; water injection

⁶ This card index file may be purchased from the Joint Committee on Powder Diffraction Standards, 1601 Park Lane, Swarthmore, PA 19081.

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