



Designation: D933 – 20

# Standard Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits<sup>1</sup>

This standard is issued under the fixed designation D933; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the manner in which the results of examination and analysis for constituents of deposits formed on systems using water as a heat transfer media or water formed deposits from any other purpose and how they are to be reported.

1.2 While various practices of reporting the analysis of water-formed deposits are in use, this practice is intended as a rational and comprehensive practice for general application. For use in specific industries or individual cases, molecular combinations may be useful and desirable.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[D887 Practices for Sampling Water-Formed Deposits](#)

[D1129 Terminology Relating to Water](#)

[E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology [D1129](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acid solubility in 1 to 1 acid, n*—concentration of a deposit or solid, in grams per litre that will go into solution in an acidic solvent divided by initial weight of the sample. Thus, results are reported as a percentage soluble or dissolved.

3.2.1.1 *Discussion*—For water borne deposits, these analyses are more qualitative or estimates of solubility of the deposit or solid (1 or more grams) in a solution of 1 to 1 acid. The acids most commonly used are either 1 to 1 hydrochloric acid (HCl (6 normal)) and nitric acid (HNO<sub>3</sub> (7.9 normal)) as the acidic solvent because they will normally dissolve most water formed deposits or scales. The sample can be weighed and placed in a soxhlet extractor with reflux and reweighed for calculation in % solubility. Note that inorganic carbonates will rapidly evolve and effervesce upon addition to the acid. Other inorganics will dissolve in the acid upon heat.

3.2.2 *loss on ignition (LOI), n*—the percent loss in weight of a material on being calcined at a temperature selected, for a time long enough, to achieve constant weight, expressed as a percent of the initial weight of the dry material (without free moisture).

3.2.2.1 *Discussion*—LOI's for deposits are normally run at 105°C for determination of moisture content. 450°C to 500°C for determination of organics in the sample. 900°C to 1000°C for determination of inorganic carbonates. The final calculation is for the remaining material, which is the ash content, which is the measure of the remaining inorganics in the sample. The sample can be weighed and stepped thru the process and reweighed after each temperature for calculation in % loss.

3.2.3 *non-polar solvent extractable solubility, n*—concentration of a deposit or solid, in grams per litre that will go into solution in a non-polar solvent divided by initial weight of the sample. Thus, results are reported as a percentage soluble or dissolved.

<sup>1</sup> This practice 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.3.1 *Discussion*—For water borne deposits, these analyses are more qualitative or estimates of solubility of the deposit or solid (1 or more grams) in a non-polar solvent. Ether, hexane and other solvents that will extract organic compounds from the deposit or solids can be used. Oils and greases and other organics can come from process contaminants and can bind the deposit together. The sample can be weighed and placed in a Soxhlet extractor with reflux and reweighed for calculation in % solubility.

#### 4. Significance and Use

4.1 The results are used to characterize the scale and water formed deposits formed in systems using water as a heat transfer fluid or deposits formed that has resulted from water being transported, stored or used for any other purpose. It is also used to evaluate the quality of water used in the system where the deposit has been found. Characterizing the scale/deposit will assist in the design of the water treatment protocols to avoid further scale or deposit buildups which can cause increased energy consumption or damage to the system due to corrosion or microbiological growth. The use of modern up-to-date chemical detection systems will increase the usefulness of the practice.

#### 5. History of Sample

5.1 Information regarding the source, background and history of the system shall be included in the report of the analysis. (Pictures are helpful to identify where the deposit was found and the conditions in the system.) This information should be as specified in Practices **D887**, as follows:

5.1.1 Name of individual or company supplying sample to the laboratory.

5.1.2 Geographic origin of sample (this helps in determining make-up water quality).

5.1.3 Date and time of sampling.

5.1.4 Number of sample/s.

5.1.5 Name and other designation of equipment from which sample or samples were removed.

5.1.6 Precise location from which sample was removed (for example, exactly what turbine blade, exactly what tube in a boiler or heat exchanger) or where in the heat exchanger or cooling tower it was found (for example, 6 o'clock position in a horizontal run of pipe right after feedwater tank). (Pictures again are helpful in this identification.)

5.1.7 *History of System*—Operational characteristics, when was the last time the system was inspected, and how long a period it took for the deposit to form.

5.1.8 Direction of flow in the areas where the deposit was found and surface temperature of that area, if known, of heat transfer equipment or zone where deposit was found.

5.1.9 Appearance and extent of deposit prior to removal. (Pictures again are helpful.)

5.1.10 Report the metallurgies involved with the system along with the metallurgy of area where the deposit was found. This is especially needed when the substrate from which the deposit was removed could influence the formation of the deposit or scale.

5.1.11 Type of deposit whether scale, sludge, biological deposit, or corrosion by-products as defined in Practices **D887**.

5.1.12 Exact method that was used in removing the sample and notes concerning any contamination that might have occurred during the process of removal by the tool used to remove the deposit (for example, if deposit removed by a wire brush, scraper, scalpel or pick, metallurgy of wire brush, scraper, scalpel or pick utilized should be recorded). Also, metallurgy of pipe or wall of pipe or surface where deposit is removed from should be recorded since the tools used and the metallurgy of the surface can lead to contaminants in the deposit (for example, nickel plated surface where deposit is removed from by stainless steel scraper – if nickel, molybdenum, or chromium is found in sample deposit, it could be from the removal action).

5.1.13 Operating temperature and pressure of liquid or vapor in the equipment that contained the deposit (water side) and also if in a heat exchanger or boiler also record the surface temperature of the liquid or vapor that the water comes indirect contact with.

5.1.14 Type and chemistry of the water treatment applied to the water that formed the deposit or to the water that furnished steam to the affected zone.

5.1.15 Make-up water analysis of water used to maintain water level in the system where deposit was formed and history of that water. (Please identify if any changes occurred in the make-up water since the last time the system was inspected, for example if system water was being maintained by municipal water switched to recycle water source.)

5.1.16 Water analysis of system water and any changes that have occurred since the last time the system was inspected (for example, water treatment company changed inhibitors utilized).

NOTE 1—If these water analyses are not available, they should be done as part of the deposit analyses.

5.1.17 An account of discrepancies in operating conditions that may have contributed to deposition (for example, water loss or starving, more or less load characteristics, water main break that caused poor water quality, loss of chemical feed, blowdown malfunctioned, etc.).

5.1.18 Results of field tests made on the sample or related equipment.

5.1.19 Any other pertinent information about the system that may or may not seem relevant including other equipment that could have impacted the water system (for example, exhaust fans near a cooling tower, sugar heat exchanger that has steam on one side and sugar on the other, etc.).

NOTE 2—Either can cause contamination of the water-based system.

5.1.20 Signature of sampler.

#### 6. Physical Characteristics

6.1 The report shall include a description of the physical characteristics of the sample, including any peculiarities that may be pertinent in its further examination. (For example, smell or odor, foaming when acid was added, etc.).

6.2 Characteristics such as the following may be recorded: color, form (scale, slimy, etc.), texture (oily, smooth, friable,

**TABLE 1 Proximate Analysis**

Solubility in Solvents	
Acid – soluble matter (soluble in 1 to 1 acid <sup>A</sup> )	%
Ether (solvent) - extractable matter (oil)	%
Water soluble matter	%
Loss on Ignition	
Moisture (at 105°C)	%
Loss on ignition at 450°C (organics)	%
Loss on ignition at 900°C (inorganic carbonates)	%
Ash % (900°C) (inorganics)	%

<sup>A</sup> One volume of deionized water to one volume of concentrated acid (see *Discussion* to term *acid solubility in 1 to 1 acid*, 3.2.1).

**TABLE 2 Constituents of Water-Formed Deposits to Be Reported**

Oxides: <sup>A</sup>	Acid Anhydrides: <sup>B</sup>
Iron oxide, <sup>C</sup> Fe <sub>2</sub> O <sub>3</sub>	Carbonate, CO <sub>2</sub>
Alumina, <sup>D</sup> Al <sub>2</sub> O <sub>3</sub>	Chloride, Cl
Calcium oxide, CaO	Nitrate, N <sub>2</sub> O <sub>3</sub>
Magnesium oxide, MgO	Sulfate, SO <sub>4</sub>
Sodium oxide, <sup>E</sup> Na <sub>2</sub> O	Sulfite, SO <sub>3</sub>
Potassium oxide, <sup>F</sup> K <sub>2</sub> O	Sulfide, <sup>F</sup> S
Copper oxide, CuO	Phosphate, PO <sub>4</sub>
Zinc oxide, ZnO	Silica, SiO <sub>2</sub>
Lead oxide, PbO	<i>Miscellaneous:</i>
Titanium oxide, TiO <sub>2</sub>	Ammonia (reported as NH <sub>3</sub> )
Nickel oxide, NiO	
Chromium oxide, Cr <sub>2</sub> O <sub>3</sub>	
Manganese Oxide MnO	
Barium Oxide	
Strontium Oxide	

Total %

Comments:

<sup>A</sup> Expression of analytical results in terms of oxides is an arbitrary procedure sanctioned by long usage and general convenience. Adherence to this convention does not imply that any of the oxides necessarily are present in the sample as such, although this may be the case with respect to such items as iron oxide and silica. A metal oxide shall be reported in the form listed in **Table 2** unless the form present has been otherwise identified. Any metal identified to be present in the sample in the metallic form shall be reported as such.

<sup>B</sup> Organic acid radicals of soaps shall be reported as the anhydride of oleic acid (C<sub>17</sub>H<sub>33</sub>O<sub>2</sub>), unless the specific acid has been identified.

<sup>C</sup> When iron oxides other than Fe<sub>2</sub>O<sub>3</sub> are known to be present (Fe<sub>3</sub>O<sub>4</sub> and FeO), but determination of the separate oxide forms is considered not to be essential, report as “Iron oxides as Fe<sub>2</sub>O<sub>3</sub>.”

<sup>D</sup> If alumina is not separated from iron oxide in the analytical procedure, report as “Iron and aluminum oxides as Fe<sub>2</sub>O<sub>3</sub>.”

<sup>E</sup> If sodium and potassium are not separated, report as “Sodium and potassium oxides as Na<sub>2</sub>O.”

<sup>F</sup> Elementary sulfur shall be reported as S.

gritty, etc.), hardness, magnetic properties (as determined by test with magnet), and structure (amorphous, crystalline, columnar crystals).

## 7. Completeness and Accuracy of Analysis

7.1 Reporting of the general physical analysis of the deposit shall first be reported. **Table 1** lists the most common Proximate Analysis to be reported but others can be reported.

7.2 The determinations to be reported in a complete analysis shall be those listed in **Table 2**. For many practical purposes, a less complete analysis will suffice. In other instances, the location or nature of the deposit or a knowledge of the composition of the equipment affected will suggest the need for determinations in addition to those listed.

NOTE 3—Other oxides may be present such as barium, strontium, and other metallurgical compounds. These should be tested for if knowledge in the background or history dictates these compounds may be present.

7.3 The completeness and accuracy of the analysis should be carefully appraised before reporting. In a complete analysis, the summation of all determinations under oxides, acid radicals, ether-extractable matter, carbonaceous matter, and combined water shall total 100 ± 2 %.

7.4 A summation in excess of 102 % indicates a positive error in one or more of the determinations (or the calculation of a metal or element as an oxide, or the calculation of a lower oxide as a higher oxide). The converse may be indicated when the summation is below 98 %, or it may be due to the presence of a substance not identified and not determined.

## 8. Reporting of Chemical Analysis

8.1 All data, the chemical properties table, shall be reported as percentage by weight of the dry sample. While the proximate analysis information shall be reported on the as-received basis.

8.2 For convenience in calculation, values for oxides, acid anhydrides, and combined water may also be expressed on a basis of chemical equivalents. Values for relative equivalents are obtained by dividing each value of percentage by weight by the equivalent weight of the respective oxide, acid radical, or water.

8.3 Quantitative determinations shall be reported to the nearest 0.1 % (8.4). When a quantitative determination is made and a negative result is obtained, it shall be reported as “less than 0.01 %” 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 the qualitative determination shows presence, and a quantitative determination shows absence, the item shall be reported as “less than 0.1” with a notation that this is a qualitative estimation.

8.4 In so far as applicable, Practice E29 shall be followed in expressing numerical results.

## 9. Reporting Results of X-ray Diffraction and Petrographic Examination

9.1 All constituents identified by X-ray diffraction or petrographic examination shall be reported according to the following rules:

9.1.1 *Silicon Compounds*—Report all compounds containing silicon, except the simple alkali silicates, by their trivial name and combined-oxide formula. Example: acmite, Na<sub>2</sub>O·Fe<sub>2</sub>O<sub>3</sub>·4SiO<sub>2</sub>.

9.1.2 *Polymorphous Compounds*—Report compounds having more than one crystal form by their trivial name and chemical formula. Examples: calcite, βCaCO<sub>3</sub> and aragonite, γCaCO<sub>3</sub>.