Standard Test Methods for Chemical Analysis of Sodium Chloride

This standard is issued under the fixed designation E 534; 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 (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analyses usually required for sodium chloride.

1.2 The analytical procedures appear in the following sections:

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1.3 The values stated in SI units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.

1.5 Review the current material safety data sheets (MSDS) for detailed information concerning toxicity, first-aid procedures, handling, and safety precautions.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

3. Significance and Use

3.1 Sodium chloride occurs in nature in almost unlimited quantities. It is a necessary article of diet as well as the source for production of many sodium compounds and chlorine. The methods listed in 1.2 provide procedures for analyzing sodium chloride to determine if it is suitable for its intended use.

4. Reagents

4.1 Purity of Reagents—Unless otherwise indicated, it is intended that all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

4.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean Type II or III reagent water conforming to Specification D 1193.

SAMPLE PREPARATION

5. Scope

5.1 This test method covers preparation of a sample that will be as representative as possible of the entire bulk quantity. The results of any analysis pertain only to the sample used.

6. Apparatus

6.1 Coarse Grinder.

6.2 High-Speed Blender.

6.3 Oven.

6.4 Riffle Sampler.

6.5 Scale.

7. Reagents

7.1 Hydrochloric Acid, Standard 1 N HCl—Prepare and standardize in accordance with Practice E 200.

8. Rock and Solar Salt Stock Solutions

8.1 Mix and split sample to 500 g, using the riffle sampler.

8.2 If sample appears wet, dry at 110°C for 2 h.

8.3 Grind the sample to −8 mesh in the coarse grinder.

8.4 Mix ground sample well and weigh out a 25.0-g representative portion for rock salt or 50.0 g for solar salt.

8.5 Place 200 mL of water in the high-speed blender and start at low speed.

8.6 Slowly add the salt sample to the high-speed blender and blend for 5 min.

8.7 Test for water insolubles as described in Sections 17-24.

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1 These test methods are under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and are under the direct responsibility of Subcommittee E15.02 on Product Standards.


3 Annual Book of ASTM Standards, Vol 15.05.

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 Pharmacopeial and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.
8.8 Save filtrate from water insolubles test and dilute in a volumetric flask to 1 L with water as a stock solution for subsequent analyses.

9.1 Mix and split the sample to 100 g for evaporated salt, or 200 g for purified evaporated salt.
9.2 Transfer to a 1-L volumetric flask.
9.3 Add 800 mL of water and allow the salt to dissolve.
9.4 Add 2 mL of concentrated HCl to dissolve any water insoluble calcium salts, particularly calcium carbonate.
9.5 Dilute to volume with water and use as a stock solution for subsequent analyses.

MOISTURE

10. Scope
10.1 This test method determines free moisture in the salt over a concentration range from 0.00 to 0.04 %. It does not determine occluded moisture trapped within the salt crystals. The procedure is based on weight loss after a sample is heated to volatilize moisture.

11. Apparatus
11.1 Analytical Balance.
11.2 Desiccator.
11.3 Oven.

12. Procedure, Rock and Solar Salt
12.1 Weigh 100 g of salt to the nearest 0.05 g into a previously dried and tared moisture dish.
12.2 Dry at 110°C for 2 h.
12.3 Cool in a desiccator and weigh.

13. Procedure, Evaporated and Purified Evaporated Salt
13.1 Weigh 20 g of salt to the nearest 0.001 g into a previously dried and weighed glass weighing bottle and cover.
13.2 Dry at 110°C for 2 h.
13.3 Cool in a desiccator, replace cover, and weigh.

14. Calculation
14.1 Calculate the percentage of moisture as follows:

\[
\text{moisture, weight} \% = \frac{A}{B} \times 100 \tag{1}
\]

where:
- \(A\) = loss of weight on drying, g, and
- \(B\) = weight of sample, g.

15. Report
15.1 Report the moisture content to the nearest 0.001 %.

16. Precision and Bias
16.1 The following criteria should be used in judging the acceptability of results (Note 1):
16.1.1 Repeatability (Single Analyst)—The standard deviation for a single determination has been estimated to be the percent absolute values shown in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is the percent absolute values in Table 1.
16.1.2 Laboratory precision (Within-Laboratory, Between-Days Variability)—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the percent absolute values shown in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the percent absolute values in Table 1.
16.1.3 Reproducibility (Multilaboratory)—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be percent absolute values shown in Table 1. The 95 % limit for the difference between two such averages is the percent absolute values in Table 1.

Note: 1—The preceding precision statements are based on an interlaboratory study performed around 1975 on five samples of sodium chloride containing covering the ranges of moisture in Table 1. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

16.2 Bias—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

WATER INSOLUBLES

17. Scope
17.1 This gravimetric method determines only the amount of insolubles present in sodium chloride which will not dissolve in water.

18. Apparatus
18.1 Analytical Balance.
18.2 Desiccator.
18.3 Magnetic Stirrer with Stirring Bar.
18.4 Parabolla Filter Funnel Assembly,\(^6\) 1000-mL, or its equivalent with 0.3-µm glass fiber filter disk.

\(^6\) Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: E15–1052.
\(^6\) Fisher Scientific No. 9-730-200 has been found satisfactory.
19. Reagents

19.1 Silver Nitrate, Standard Solution, 0.1 N AgNO₃— Prepare and standardize in accordance with Practice E 200.

20. Procedure, Rock and Solar Salts

20.1 Transfer a sample prepared in accordance with 8.1 to 8.6 to a 1-L Erlenmeyer flask, washing out the blender with 100 mL of water. Add 300 mL of water to give a total of 600 mL of water added.

20.2 Stir on a magnetic stirrer for 1 h. Adjust the stirrer speed to give maximum agitation without danger of losing any sample due to splashing. Place a beaker over top of the flask while stirring.

20.3 Filter the solution by vacuum through a previously dried (110°C for 1 h) and accurately weighed filter disk using the Parabella funnel. Transfer all insolubles to the paper and wash free of chlorides with water until the filtrate shows no turbidity when tested with 0.1 N AgNO₃ solution.

20.4 Dilute filtrate and washings to 1 L with water in volumetric flask.

20.5 Dry the filter disk at 110°C for 1 h.

20.6 Cool in a desiccator and weigh on an analytical balance.

20.7 Save the filtrate for subsequent analyses.

21. Procedure, Evaporated and Purified Evaporated Salts

21.1 Place a well mixed sample in a 2-L beaker. Use 100-g sample for evaporated or 200 g for purified evaporated salt.

21.2 Add 750 mL of water.

21.3 Mix with a mechanical stirrer until solution is complete.

21.4 Filter the solution by vacuum through a previously dried (110°C for 1 h) and accurately weighed filter disk using the Parabella funnel. Transfer all insolubles to the paper and wash free of chlorides with water until the filtrate shows no turbidity when tested with 0.1 N AgNO₃ solution.

21.5 Dry the filter disk at 110°C for 1 h.

21.6 Cool in a desiccator and weigh on an analytical balance.

21.7 Save the filtrate for subsequent analyses.

22. Calculation

22.1 Calculate the percentage of water insolubles as follows:

\[
\text{insolubles, weight %} = \frac{A}{B} \times 100
\]

where:

\[A = \text{increase in weight of filter disk, g, and}\]

\[B = \text{sample weight, g.}\]

23. Report

23.1 Report the percentage of water insolubles to the nearest percentage shown as follows:

<table>
<thead>
<tr>
<th>Range, %</th>
<th>Report to, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.002 to 0.005</td>
<td>0.01</td>
</tr>
<tr>
<td>0.01 to 0.04</td>
<td>0.01</td>
</tr>
<tr>
<td>0.15 to 0.35</td>
<td>0.01</td>
</tr>
</tbody>
</table>

24. Precision and Bias

24.1 The following criteria should be used in judging the acceptability of results (Note 2):

24.1.1 Repeatability (Single Analyst)—The coefficient of variation for a single determination has been estimated to be the percent relative values shown in Table 2 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is percent relative values shown in Table 2.

24.1.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 23.5 % relative at 57 df. The 95 % limit for the difference between two such averages is 66 % relative.

24.1.3 Reproducibility (Multilaboratory)—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be percent relative values shown in Table 2 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is percent relative values shown in Table 2.

Note 2—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the ranges of water insolubles in Table 2. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

24.2 Bias—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

25. Scope

25.1 This test method covers the EDTA titrimetric determination of calcium and magnesium and the EDTA titrimetric determination of calcium. The magnesium content is determined by difference.

26. Apparatus

26.1 Magnetic Stirrer with Stirring Bar.
27. Reagents

27.1 Erichrome Black T Indicator Solution, Hydroxy Naphthol Blue, or its equivalent.

27.2 Murexide (Ammonium Purpurate) Indicator Solution, or its equivalent.

27.3 EDTA Standard Solution (1 mL = 0.400 mg calcium)—Dissolve 4.0 g of disodium dihydrogen ethylene diaminetetraacetate (EDTA) in 1 L of water. Standardize this solution against a standard calcium solution prepared by dissolving 1,000 g of CaCO₃ and 2 mL of HCl in water and diluting to 1 L with water in a volumetric flask. Obtain an exact factor for the EDTA solution. This factor is equal to the milligrams of calcium equivalent to 1.00 mL of EDTA solution. See Practice E 200.

\[ \text{factor} = \frac{W}{V} \]  

where:

\[ W = \text{calcium in aliquot, mg, and} \]
\[ V = \text{EDTA solution required for titration, mL.} \]

27.4 Ammonium Chloride–Ammonium Hydroxide Solution—Add 67.5 g of ammonium chloride (NH₄Cl) to 570 mL of ammonium hydroxide (NH₄OH) contained in a 1-L volumetric flask. Reserve this solution for use as described in 27.6 and 27.7.

27.5 Potassium Cyanide Solution (50 g/L)—Dissolve 50 g of potassium cyanide (KCN) in water and dilute to 1 L with water. Store in a borosilicate glass bottle. (Warning—Potassium cyanide is extremely poisonous.)

27.6 Magnesium Sulfate Solution (2.5 g/L)—Dissolve 2.5 g of MgSO₄·7H₂O in water and dilute to volume with water in a 1-L volumetric flask. Determine the volume of EDTA solution equivalent to 50 mL of MgSO₄ solution as follows: Pipet 50 mL of MgSO₄ solution into a 400-mL beaker. Add 200 mL of water and 2 mL of NH₄Cl·NH₄OH solution (27.4). Add 1 mL of KCN solution and a sufficient amount of Eriochrome Black T Indicator solution or its equivalent. Titrate the solution with EDTA solution while stirring with a magnetic stirrer to the true blue end point. This gives the volume of EDTA solution equivalent to 50.0 mL of MgSO₄ solution.

27.7 Buffer Solution—Pipet 50 mL of MgSO₄ solution into the volumetric flask containing the remaining NH₄Cl·NH₄OH solution (27.4). Add the exact volume of EDTA solution equivalent to 50 mL of the MgSO₄ solution. Dilute to 1 L with water. Store the solution in a polyethylene bottle.

27.8 Potassium Hydroxide Solution (600 g/L)—Dissolve 150 g of potassium hydroxide (KOH) in 250 mL of water. Cool and store in a polyethylene bottle.

28. Procedure

28.1 Using Table 3 as a guide, pipet two aliquots of stock solution into 400-mL beakers to give a titer between 2 and 10 mL of standard EDTA solution. One aliquot is used to determine total calcium and magnesium and the other for calcium.

28.2 Dilute to 200 mL with water, if necessary, and place on magnetic stirrer.

28.3 Total Calcium and Magnesium:

28.3.1 Add 5 mL of buffer solution, 1 mL of KCN solution, and a sufficient amount of Erichrome Black T Indicator Solution or its equivalent.

28.3.2 Titrate with standard EDTA solution to a true blue color.

28.3.3 Record the milliliters used as Titration 1 (T₁).

28.4 Calcium Only:

28.4.1 Add 2 mL of KOH solution, 1 mL of KCN solution to the other aliquot in 28.1 and 28.2, and stir for about 2 min to precipitate magnesium.

28.4.2 Add a sufficient amount of murexide solution or an equivalent calcium indicator solution.

28.4.3 Titrate with standard EDTA solution to a true blue color.

28.4.4 Record the milliliters used as Titration 2 (T₂).  

28.4.5 Record the volume of EDTA solution used as Titration 2 (V₂) (3).

\[ V₂ = \text{calcium in aliquot, mg, and} \]

29. Calculation

29.1 Calculate the weight percent of calcium as follows:

\[ \text{Ca, weight \%} = \frac{(T₂ \times \text{factor}) \times (0.1)}{S} \]  

where:

\[ T₂ = \text{EDTA used to titrate calcium only, mL.} \]
\[ S = \text{weight of salt in aliquot, g.} \]

29.1.1 See 27.3 for factor.

29.2 Calculate the weight percent of magnesium as follows:

\[ \text{Mg, weight \%} = \frac{(T₁ - T₂ \times \text{factor}) \times (0.6064) \times (0.1)}{S} \]  

where:

\[ T₁ = \text{EDTA used to titrate total calcium and magnesium, mL.} \]
\[ S = \text{weight of salt in aliquot, g.} \]

30. Report

30.1 Report the percentage of calcium to the nearest 0.001 %. Report the percentage of magnesium to the nearest 0.001 %.

31. Precision and Bias

31.1 The following criteria should be used in judging the acceptability of calcium results (Note 3):

31.1.1 Repeatability (Single Analyst)—The coefficient of variation for a single determination has been estimated to be 3.56 % relative at 100 df. The 95 % limit for the difference between two such runs is 10 % relative.

31.1.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 6.34 % relative at 40
The 95% limit for the difference between two such averages is 18% relative.

31.1.3 Reproducibility (Multilaboratory)—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 9.82% relative at 7 df. The 95% limit for the difference between two such averages is 28% relative.

Note 3—The preceding precision statements are based on an interlaboratory study performed around 1975 on five samples of sodium chloride covering the range from 0.036 to 0.909% calcium. One analyst in each of eight laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

31.1.4 Bias—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

31.2 The following criteria should be used in judging the acceptability of magnesium results (Note 4):

31.2.1 Repeatability (Single Analyst)—The standard deviation for a single determination has been estimated to be percent absolute values shown in Table 4 at the indicated degrees of freedom. The 95% limit for the difference between two such runs is the percent absolute values shown in Table 4.

31.2.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the percent absolute values shown in Table 4 at the indicated degrees of freedom. The 95% limit for the difference between two such averages is the percent absolute values shown in Table 4.

31.2.3 Reproducibility (Multilaboratory)—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be the percent absolute values shown in Table 4 at the indicated degrees of freedom. The 95% limit for the difference between two such averages is the percent absolute values shown in Table 4.

Note 4—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the ranges of magnesium given in Table 4. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

31.2.4 Bias—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

32. Scope

32.1 This test method covers the gravimetric determination of the sulfate content of sodium chloride.

33. Apparatus

33.1 Gooch Asbestos Slurry.
33.2 Gooch Filtering Crucible and Holder.
33.3 Muffle Furnace.
33.4 Oven.

34. Reagents

34.1 Barium Chloride Solution (120 g BaCl₂ 2H₂O/L)—Dissolve 120 g of barium chloride dihydrate in about 750 mL of water, filter, and dilute to 1 L.7
34.2 Hydrochloric Acid Standard Solution 1 N HCl—Prepare and standardize in accordance with Practice E 200.
34.3 Methyl Orange Indicator Solution (1 g/L)—Dissolve 0.1 g of methyl orange in 100 mL of water and filter if necessary.7

35. Procedure

35.1 Using Table 5 as a guide, pipet the recommended aliquot of stock solution into a 400-mL beaker.
35.2 Dilute to 200 mL, add a few drops of methyl orange indicator solution and acidify with 1 mL of HCl (1 + 1) if necessary.
35.3 Heat solution gently to boiling and add 10 mL of BaCl₂ solution dropwise while stirring.
35.4 Digest on a hot plate below the boiling point for 30 min.
35.5 Cool overnight.
35.6 Filter through a tared Gooch crucible previously prepared with an asbestos mat and ignited in a muffle furnace at 800°C for 30 min. Transfer all the precipitate to the crucible with a rubber policeman. Wash with portions of hot water until washings are free of chlorides.
35.7 Dry the crucible at 110°C for 15 min, then ignite in a muffle furnace at 800°C for 30 min.
35.8 Cool in a desiccator and reweigh.

36. Calculation

36.1 Calculate percentage of sulfate as follows:

\[
\text{sulfate, weight \% = } \frac{A}{B} \times 0.4115 \times 100
\]

Note 4—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the ranges of magnesium given in Table 4. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

Note 3—The preceding precision statements are based on an interlaboratory study performed around 1975 on five samples of sodium chloride covering the range from 0.036 to 0.909% calcium. One analyst in each of eight laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

Note 4—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the ranges of magnesium given in Table 4. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.
where:
\[ A = \text{weight of precipitate, g, and} \]
\[ B = \text{weight of salt in aliquot, g.} \]

37. Report
37.1 Report the percentage of sulfate to the nearest 0.001 %.

38. Precision and Bias
38.1 The following criteria should be used in judging the acceptability of results (Note 5):

38.1.1 Repeatability (Single Analyst)—The coefficient of variation for a single determination has been estimated to be 7.14 % relative at 120 df. The 95 % limit for the difference between two such runs is 20 % relative.

38.1.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 8.03 % relative at 60 df. The 95 % limit for the difference between two such averages is 22 % relative.

38.1.3 Reproducibility (Multilaboratory)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst in different laboratories, has been estimated to be 8.01 % relative at 9 df. The 95 % limit for the difference between two such averages is 22 % relative.

NOTE 5—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the range from 0.016 to 2.030 % sulfate. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E 180 was used in developing these precision estimates.

38.1.4 Bias—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

39. Scope
39.1 Analyses should be reported on a dry basis. If analyses are on an as received sample, correction should be made by converting to a dry basis. Sodium chloride purity is determined by subtracting the total percentage of impurities from 100. Moisture should be reported as a separate value.

40. Procedure
40.1 Convert sulfate to calcium sulfate and the unused calcium to calcium chloride unless the sulfate in sample exceeds the quantity necessary to combine with the calcium. In this case, convert the calcium to calcium sulfate and the unused sulfate first to magnesium sulfate, and the remaining sulfate, if any, to sodium sulfate. Convert the unused magnesium to magnesium chloride.

40.2 Report rock and solar salt impurities to the second decimal place and salt purity, by difference, to the first decimal place.

40.3 Report evaporated salt impurities to the third decimal place and salt purity, by difference, to the second decimal place.

40.4 Report purified salt impurities to the fourth decimal place and salt purity, by difference, to the third decimal place.

41. Conversion Factors

\[
\begin{align*}
\text{BaSO}_4 &\times 0.4116 = \text{SO}_4 \\
\text{BaSO}_4 &\times 0.5832 = \text{CaSO}_4 \\
\text{BaSO}_4 &\times 0.6086 = \text{Na}_2\text{SO}_4 \\
\text{Ca} &\times 3.3967 = \text{CaSO}_4 \\
\text{Ca} &\times 0.6064 = \text{Mg} \\
\text{CaSO}_4 &\times 0.2944 = \text{Ca} \\
\text{CaSO}_4 &\times 0.8153 = \text{CaCl}_2 \\
\text{CaSO}_4 &\times 0.8842 = \text{MgSO}_4 \\
\text{CaSO}_4 &\times 1.0434 = \text{Na}_2\text{SO}_4 \\
\text{Mg} &\times 3.9173 = \text{MgCl}_2 \\
\text{MgCl}_2 &\times 1.4296 = \text{CaSO}_4 \\
\text{MgCl}_2 &\times 1.2641 = \text{MgSO}_4 \\
\text{MgSO}_4 &\times 1.4299 = \text{CaSO}_4 \\
\text{MgSO}_4 &\times 1.1311 = \text{MgCl}_2 \\
\text{MgSO}_4 &\times 1.1800 = \text{Na}_2\text{SO}_4 \\
\text{SO}_4 &\times 1.4173 = \text{CaSO}_4 \\
\end{align*}
\]

42. Keywords
42.1 calcium; evaporated salt; magnesium; moisture; purified salt; rock salt; sodium chloride; sulfate; water insolubles