

JIS

JAPANESE INDUSTRIAL STANDARD

Chemical Analysis of Limes

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JAPANESE INDUSTRIAL STANDARD

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Chemical Analysis of Limes

R9011-1981

1. Scope

This Japanese Industrial Standard provides the chemical analysis of industrial limes.

2. Analysis Items

The analysis items provided by this standard are as enumerated below.

- (1) Ignition loss (ig. loss)
- (2) Impurities (imp.)
- (3) Silicon dioxide + insoluble residue (SiO_2 + insol.)
- (4) Silicon dioxide (SiO_2)
- (5) Aluminium oxide (Al_2O_3)
- (6) Ferric oxide (Fe_2O_3)
- (7) Calcium oxide (CaO)
- (8) Magnesium oxide (MgO)
- (9) Sulfur trioxide (SO_3)
- (10) Total sulfur (S)
- (11) Carbon dioxide (CO_2)
- (12) Phosphorus Pentaoxide (P_2O_5)

3. Sample

Sample shall be prepared by taking a part of reduced sample which has been taken in accordance with 4.1 of JIS R 9001. When the sample is massive, it shall be pulverized to an appropriate fineness, and shall be reduced after it is mixed thoroughly. Then, the sample shall be pulverized so that all of the sample can pass the 149 μm mesh of standard wire sieve, and shall be mixed thoroughly to be furnished to the analyses.

Preparation of the analysis sample shall be carried out rapidly so that the sample will not absorb any moisture and carbon dioxide contained in the air, and the sample thus prepared shall be preserved in a hermetically sealed container.

4. Matters common to analysis

The matters common to analysis shall be as stipulated in JIS K 0050 and JIS K 0115, as a rule.

Applicable Standards : See page 22.

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5. Expression of Analysis Values

The analysis values, expressed in percentage, shall be rounded as follows:

- (1) For nine composition other than sulfur trioxide, total sulfur and phosphorus pentoxide, the values shall be rounded off to one place of decimals by the rule provided in JIS Z 8401.
- (2) For sulfur trioxide, total sulfur and phosphorus pentoxide, the values shall be rounded off to two places of decimals by the rule provided in JIS Z 8401.

6. Methods of Analysis

6.1 Determination of Ignition Loss The determination of ignition loss shall be made as follows:

- (1) Principle Weigh the loss at the time when igniting the sample in an electric furnace of 1000°C until it attains the constant mass.
- (2) Procedures Weigh approximately 1g of the sample into a platinum or porcelain crucible to the nearest 0.1 mg correctly, cover it loosely with a lid, increase temperature gradually by heating slowly, ignite in an electric furnace controlled to 1000 ± 50°C for 30 min, cool it in a desiccator, and weigh it rapidly.
Repeat this procedure every for 15 min, and obtain the loss of mass at the time when the sample attains constant mass.
- (3) Calculation Calculate the ignition loss by the following equation:

$$\text{ig. loss} = \frac{m_1}{m} \times 100$$

where ig. loss : ignition loss (%)
 m_1 : loss of mass (g)
 m : mass of sample (g)

6.2 Determination of Impurities The determination of impurities shall be made as follows:

- (1) Principle This method applies to the determination of an approximate amount of silicon dioxide + aluminium oxide + ferric oxide. Dissolve the sample with hydrochloric acid, add ammonia water to allow hydroxide to precipitate, filter, ignite and weigh.
- (2) Procedure Weigh a sample of approximately 1g into a beaker of 200 ml correctly to the nearest 0.1 mg, add a small amount of water to render it slurry like, add 20 ml of hydrochloric acid (1+1) and stir, and heat slightly to make the soluble matter to dissolve. Add warm water to make approximately 100 ml, add some drops of nitric acid, and boil for 2 to 3 min. Next, add one or two drops of methylred indicator, drip slowly ammonia water (1+1) while stirring the solution, until the solution turns yellow from red, and further add excessive one or two drops. As the next, boil it for about 1 min and stop heating, filter through a filter paper (Class 5 B 110 mm) immediately after precipitation settles, and rinse it about 8 times with warm solution of ammonium nitrate (2 % w/v) (1). Store the filtrate as it is, to furnish to the determination of calcium oxide.

Dry the precipitate in a crucible heat slowly until the filter paper

incinerates, ignite in an electric furnace controlled to $1000 \pm 50^\circ \text{C}$ for 1 h, cool it, and then weigh its mass.

Note (1) Add two or three drops of methylred indicator in the ammonium solution, and drip ammonia water (1+1) until the colour of the solution turns yellow from red and then use it. If its colour returns red when the solution is heated, further drip ammonia water, to turn it yellow, and then use it.

- (3) Calculation Calculate the content of the impurities by the following equation:

$$\text{imp.} = \frac{m_1}{m} \times 100$$

where imp. : content of impurities (%)
 m_1 : mass of precipitate (g)
 m : mass of sample (g)

6.3 Determination of Silicon Dioxide + Insoluble Residue The determination of silicon dioxide + insoluble residue shall be made as follows:

- (1) Principle Dissolve the sample with hydrochloric acid and perchloric acid, heat to make the silicon dioxide insoluble, filtrate, ignite, and then measure its mass.
- (2) Procedure Weigh approximately 1 g of the sample to the nearest 0.1 mg correctly into a beaker of 200 ml, add small amount of water to render it slurry-like, cover it with a watch glass, add 10 ml of hydrochloric acid (1 + 1), and stir to dissolve the soluble matter. Add 15 ml of perchloric acid (60%) in it, heat to evaporate on a sand bath or heat plate, and heat for about 10 min succeeding after perchloric acid gives out dense white fume. After cooling it for a while, add 5 ml of hydrochloric acid (1 + 1) and approximately 50 ml of warm water and stir, immediately after the precipitate almost settles, filter through a filter paper (Class 5 B, 110 mm), and rinse with warm water 10 to 12 times.
- Put the precipitate in a crucible to dry, heat slowly, and, after the filter paper incinerates with care not to generate any flame, ignite it in an electric furnace controlled at $1000 \pm 50^\circ \text{C}$ for 1 h. After cooling it, measure its mass.

- (3) Calculation Calculate the content of silicon dioxide + insoluble residue by the following equation:

$$\text{SiO}_2 + \text{insol} = \frac{m_1}{m} \times 100$$

where $\text{SiO}_2 + \text{insol}$: content of silicon dioxide + insoluble residue (%)
 m_1 : mass of precipitate (g)
 m : mass of sample (g)

6.4 Determination of Silicon Dioxide The determination of the silicon dioxide shall be made as follows:

- (1) Principle Dissolve the sample with hydrochloric acid and perchloric acid to render silicic acid insoluble, and then measure the mass after filtration and ignition. After treating with perchloric acid and hydrofluoric acid, measure the mass after ignition. Obtain the difference in mass before and after.

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- (2) Procedure Weigh approximately 1 g of the sample to the nearest 0.1 mg correctly in a beaker of 200 ml, add small amount of water to render slurry-like, cover with a watch glass, add 10 ml of hydrochloric acid (1 + 1), and stir to dissolve the soluble matter. Add 15 ml of perchloric acid (60%), heat to evaporate on a sand bath or heat plate, heat about 10 min succeedingly after perchloric acid gives out dense white fume. After cooling it for a while, add 5 ml of hydrochloric acid (1 + 1) and 50 ml of warm water, and stir. Immediately after precipitate settles, filter through a filter paper (Class 5 B, 110 mm), and rinse 10 to 12 times with warm water.

Receive the filtrate in a measuring flask of 250 ml, preserve as it is, and use in the procedures for aluminium oxide and ferric oxide.

Put the precipitate in a platinum crucible to dry it, heat slowly with care not to allow the precipitate to give off any flame until the filter paper incinerates. Next, ignite in an electric furnace controlled at $1000 \pm 50^\circ\text{C}$, and after cooling, measure the mass.

Next, moisten the precipitate in the platinum crucible with a small amount of water, add two or three drops of sulfuric acid (1+1) or perchloric acid (60%) and approximately 5 ml of hydrofluoric acid (46%) in it, heat to evaporate in a ventilated room. After white fume has expired to generate, ignite in the electric furnace controlled at $1000 \pm 50^\circ\text{C}$ for 10 min, and after cooling, measure the mass.

- (3) Calculation Calculate the content of silicon dioxide by the use of the following equation:

$$\text{SiO}_2 = \frac{m_1 - m_2}{m} \times 100$$

where SiO_2 : content of silicon dioxide (%)

m_1 : mass of precipitate which is obtained at first (g)

m_2 : mass of residue after treatment with hydrofluoric acid (g)

m : mass of sample (g)

6.5 Determination of Aluminium Oxide

6.5.1 EDTA Titration Method EDTA titration method shall be as prescribed in the following:

- (1) Principle Treat the precipitate of silicon dioxide with hydrofluoric acid, and fuse its residue with sodium disulfide to dissolve in the water. Add this solution to the filtrate from which the precipitate of silicon dioxide have been separated. Separate the total volume or a part which has been made constant volume, adjust its pH with ammonium acetate and hydrochloric acid, and titrate this with EDTA standard solution by the aid of the indicator of Cu-PAN. Determine the content of the aluminium oxide by subtracting an amount of EDTA standard solution which is equivalent to the content of the ferric oxide otherwise determined from this titration value.

(2) Reagents

- (a) M/100 EDTA standard solution Dissolve 3.8 g of ethylenediaminetetraacetic acid disodium salt in water to make 1 l, and store in a polyethylene bottle. This solution shall be subjected to standardization in accordance with JIS K 8006.
- (b) Cu-PAN solution Dissolve 2 g of Cu-PAN (a mixture of 1-(2-pyridilazo)-2-naphthol and ethylenediaminetetraacetic acid copper salt (Cu-EDTA) at mol

ratio 1 : 1) in 100 ml of isopropyl alcohol (50 % v/v %).

- (c) Ammonium acetate solution Add water to 250 g of ammonium acetate to make 1 l.

- (3) Procedure Put 1 to 2 g of sodium pyrosulfate in a platinum crucible to which the residue of hydrofluoric acid treatment has adhered in the procedure of silicon dioxide, cover the crucible with a lid, and heat it with weak flame to a degree so that the crucible presents dark red colour to melt the residue. After the crucible cools down, add approximately 10 ml of warm water, heat the crucible slightly enough to dissolve the solution add it to the filtrate contained in flask stored by the procedure of silicon dioxide, rinse the crucible once with 1 or 2 ml of hydrochloric acid (1 + 1), then wash thoroughly with warm water, and add this washing also into that, cool to room temperature, and carry out the following procedure by using this solution.

In this case, carry out determination using method (A) or (B) below according to the amount of content of aluminium oxide.

- (A) In the case where aluminium oxide is contained low and it is required to determine the aluminium oxide alone, use the total quantity of this solution (in this case, it is unnecessary to make the total quantity to 250 ml).
- (B) In the case where aluminium oxide is contained highly, add water in this total quantity of solution to the marked line, shake up to make a sample solution, and separate 100 ml of this solution with a pipette to furnish the test. Preserve the residual solution as it is to furnish to the determination of calcium oxide, magnesium oxide and ferric oxide.

Pour the solution (A) or (B) in a beaker of 300 ml, add 15 ml of ammonium acetate solution, and adjust its pH at 3.0 to 3.5 with hydrochloric acid (1+1) and ammonium acetate solution.

Add several drops of Cu-PAN solution, boil and titrate with EDTA standard solution while it is hot until its red colour fades away. If it returns red when boiling again, continue titration, and when boiling and titration is repeated if it comes non-returnable to red, even when boiling is continued for 30 sec. Consider the time as an end point.

- (4) Calculation Calculate the content of aluminium oxide by the use of the following equation:

In the case where the solution (A) is used,

$$\text{Al}_2\text{O}_3 = \left(\frac{v \times f}{m} - \frac{\text{Fe}_2\text{O}_3}{0.0007985 \times 100} \right) \times 0.0005098 \times 100$$

In the case where the solution (B) is used,

$$\text{Al}_2\text{O}_3 = \left(\frac{v \times f}{m} \times \frac{250}{100} - \frac{\text{Fe}_2\text{O}_3}{0.0007985 \times 100} \right) \times 0.0005098 \times 100$$

where Al_2O_3 : content of aluminium oxide (%)

v : consumption of EDTA standard solution (ml)

f : factor of EDTA standard solution

m : measured mass of sample taken in procedure for silicon dioxide (g)

Fe_2O_3 : content of ferric oxide separately obtained (%)

6.5.2 Gravimetric Method The gravimetric method shall be made as follows:

- (1) Principle Dissolve the residue which is obtained by treating the precipitate

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of silicon dioxide with the hydrofluoric acid with disodium sulfate, and dissolve in water. Then, add this solution to the filtrate from which the precipitate has been separated. Take its total volume or a part of the solution which has been made constant in volume, add ammonia water to make hydroxide to precipitate filtrate and ignite, and then measure the mass. Obtain the content of aluminium oxide by subtracting the content of ferric oxide which has been obtained separately.

- (2) Procedure Prepare a sample solution in accordance with the procedure of aluminium oxide (EDTA titration method).

Follow the procedure below by using this solution. In this case, use the method (A) or (B) below, according to the content of aluminium oxide.

- (A) In the case where aluminium oxide is contained low, and if it is required to determine aluminium oxide alone, use the total volume of this solution (in this case, it is unnecessary to make the total volume to 250 ml).
- (B) In the case where aluminium oxide is contained highly, add water in this total quantity of solution to the marked line, and shake up to make a sample solution, and separate 100 ml of this solution with a pipette to furnish to the test. Preserve the residual solution as it is to furnish to the determination of calcium oxide, magnesium oxide and ferric oxide.

Pour the solution (A) or (B) in a beaker of 200 ml, and boil for several minutes. Add 1 or 2 drops of methylred indicator, drip gradually ammonia water (1+1) while stirring the solution until its colour turns from red to yellow, add further one or two drops excessively. Succeedingly, boil for about 1 min, stop heating, and, after precipitation stops, filter with a filter paper (Class 5 A, 110 mm) waiting the precipitates to settle. Rinse about 8 times with warm solution of ammonium nitrate (2% w/v) (1). Preserve the filtrate as it is to furnish to the determination of calcium oxide.

Put the precipitate together with the filter paper in a platinum crucible or porcelain crucible to dry, and, after the filter paper incinerates by heating gradually ignite for 1 h in an electric furnace controlled at $1000 \pm 50^\circ \text{C}$. After cooling in a desiccator measure the mass.

- (3) Calculation Calculate the content of aluminium oxide by the use of the following equation (2):

In the case where the solution (A) is used,

$$\text{Al}_2\text{O}_3 = \frac{m_1}{m} \times 100 - \text{Fe}_2\text{O}_3$$

In the case where the solution (B) is used,

$$\text{Al}_2\text{O}_3 = \frac{m_1}{m} \times \frac{250}{100} \times 100 - \text{Fe}_2\text{O}_3$$

where Al_2O_3 : content of aluminium oxide (%)

m_1 : mass of precipitate (g)

m : measured mass of sample taken in the procedure for silicon dioxide (g)

Fe_2O_3 : content of ferric oxide separately obtained (%)

Note (2) In the case where the determined value of the phosphorus pentaoxide exceeds 0.1%, further subtract the content of phosphorus pentoxide from this value.