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Paper, board and pulps — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium

Papiers, cartons et pâtes — Détermination de la teneur en magnésium, calcium, manganèse, fer, cuivre, sodium et potassium soluble dans l'acide

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12830 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This first edition of ISO 12830 cancels and replaces ISO 777:2005, ISO 778:2005, ISO 779:2005, ISO 1830:2005 and ISO 9668:1990.

Introduction

This International Standard combines the determination of the acid-soluble part of magnesium (Mg), calcium (Ca), manganese (Mn), iron (Fe), copper (Cu), sodium (Na) and potassium (K) into a single standard. The scope of this International Standard covers only the acid-soluble part of the elements.

ISO 17812 specifies the procedure for the determination of total magnesium, total calcium, total manganese, total iron and total copper by atomic absorption spectrometry or by plasma emission spectrometry.

Paper, board and pulps — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium

WARNING — The method specified in this International Standard involves the use of some hazardous chemicals and of gases that may form explosive mixtures with air. Care should be taken to ensure that the relevant precautions are observed.

1 Scope

This International Standard specifies the procedure for the determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium by atomic absorption spectrometry or by plasma emission spectrometry. The acid-soluble element comprises the acid-soluble part of the incineration residue, i.e. that part of the ignition residue obtained after incineration which is soluble in hydrochloric acid. In the case where the residue is completely soluble, the result obtained by the procedure specified in this International Standard is a measure of the total amount of each element in the sample.

This International Standard is applicable to all types of paper, board and pulps.

The limit of determination depends on the element and on the instrument used.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 186, *Paper and board — Sampling to determine average quality*

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 1762, *Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7213, *Pulps — Sampling for testing*

3 Principle

A test portion is incinerated at 525 °C and the residue is dissolved in 6 mol/l hydrochloric acid. The concentration of each element in the test solution is then determined by atomic absorption or plasma emission spectrometry, as specified in this International Standard.

4 Reagents and materials

4.1 General.

All chemicals shall be of reagent grade or better unless otherwise indicated. Water shall be distilled or deionized, of grade 2 in accordance with ISO 3696.

4.2 Hydrochloric acid (HCl), 6 mol/l. Dilute 500 ml of concentrated hydrochloric acid (specific gravity 1,19 g/ml) to 1 000 ml with water.

4.3 Nitric acid (HNO_3), concentrated (specific gravity 1,4 g/ml).

4.4 Lanthanum chloride ($LaCl_3$), solution, $\rho(\mathrm{La}) = 50$ g/l. In a 1 000 ml volumetric flask, dissolve 59 g of lanthanum oxide (La_2O_3), in small portions, in 200 ml of hydrochloric acid (4.2) and dilute to 1 000 ml with water.

NOTE This lanthanum solution is used to eliminate chemical interference when determining calcium and magnesium in an air/acetylene flame. The solution is not required when the nitrous oxide/acetylene flames or inductively coupled plasma technique (ICP technique) is used.

4.5 Cesium chloride (CsCl), solution $\rho(\mathrm{Cs}) = 50$ g/l. In a 1 000 ml volumetric flask, dissolve 63,5 g of ultra-pure cesium chloride (CsCl) in water and dilute to 1 000 ml with water.

NOTE This cesium solution is used to suppress ionization of sodium and potassium. It is also used to suppress ionization of calcium in a nitrous oxide/acetylene flame. The solution is not required when the air/acetylene flame or ICP technique is used.

4.6 Standard stock solutions of each element, commercially available certified atomic absorption or atomic emission standard solutions can be used. Standard stock solutions can also be prepared as follows:

4.6.1 Magnesium, 1 000 mg/l standard solution. Dissolve 1,000 g of magnesium metal ribbon in 100 ml of 1:4 nitric acid (4.3) and dilute to 1 000 ml with water.

4.6.2 Calcium, 1 000 mg/l standard solution. Dissolve 2,497 g of primary standard calcium carbonate ($CaCO_3$) in a minimum volume of 1:4 nitric acid (4.3) and dilute to 1 000 ml with water.

4.6.3 Manganese, 1 000 mg/l standard solution. Dissolve 1,000 g of manganese metal strip or wire in a minimum volume of 1:1 nitric acid (4.3) and dilute to 1 000 ml with water.

4.6.4 Iron, 1 000 mg/l standard solution. Dissolve 1,000 g of iron metal strip or wire in 20 ml of hydrochloric acid (4.2) and dilute to 1 000 ml with water.

4.6.5 Copper, 1 000 mg/l standard solution. Dissolve 1,000 g of copper metal strip or wire in a minimum volume of 1:1 nitric acid (4.3) and dilute to 1 000 ml with water.

4.6.6 Sodium, 1 000 mg/l standard solution. Ignite a portion of anhydrous sodium sulfate (Na_2SO_4) at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Dissolve 3,089 g of dried sodium sulfate in water and dilute to 1 000 ml with water. Store in a polyethylene bottle.

4.6.7 Potassium, 1 000 mg/l standard solution. Ignite a portion of anhydrous potassium sulfate (K_2SO_4) at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Dissolve 2,228 g of dried potassium sulfate in water and dilute to 1 000 ml with water. Store in a polyethylene bottle.

4.7 Acetylene gas and/or **nitrogen oxide gas**, of a grade suitable for atomic absorption spectrometry. Nitrous oxide is used only when measuring calcium.

WARNING — Acetylene gas forms explosive mixtures with air.

4.8 Carrier gas, appropriate gas for the plasma emission spectrometer. Argon is usually recommended as a carrier gas.

5 Apparatus and equipment

5.1 General.

Ordinary laboratory equipment is used. All glassware and plastic ware shall be rinsed with 0,1 mol/l hydrochloric acid prior to use.

5.2 Filter paper, ash free, particle retention 20 μm to 25 μm.

5.3 Crucibles, platinum or fused silica, of minimum capacity 50 ml.

5.4 Muffle furnace, capable of maintaining a temperature of 525 °C ± 25 °C.

5.5 Balance, of capacity 100 g, accurate to 0,1 mg.

5.6 Atomic absorption spectrometer (AAS), equipped with air/acetylene and nitrous oxide/acetylene burners and with hollow cathode lamps for Mg, Ca, Mn, Fe, Cu, Na and K.

NOTE Multi-element lamps can also be used.

5.7 Inductively coupled plasma/optical emission spectrometer (ICP/OES).

5.8 Disposable protective gloves.

6 Sampling and preparation of sample

If the analysis is being made to evaluate a lot of paper, board or pulp, the sample shall be selected in accordance with ISO 186 or ISO 7213, as relevant. If the analysis is made on another type of sample, report the source of the sample and, if possible, the sampling procedure. Select the specimens so that they are representative of the sample received. A sufficient amount of sample shall be collected to allow for at least duplicate determinations. Avoid cut edges, punched holes and other parts where metallic contamination may have occurred. Disposable protective gloves (5.8) shall be worn when handling samples to avoid contamination.

Prepare a test specimen by tearing at least 30 g of small pieces from various parts of the sample. This amount is sufficient for the duplicate determinations described in Clause 7.

Since iron tends to be non-homogeneous in the sample, it is recommended that a composite sample be used.

7 Procedure

7.1 General

Although dry ignition followed by acid treatment is described in this International Standard, other dissolution methods such as wet ignition or microwave digestion using various acid combinations can also be used, provided that the results have been validated.

WARNING — For samples with high silicon content, microwave digestion with nitric acid will give lower results for magnesium and for some other elements.

7.2 Incineration of the test portion

Carry out the procedure in duplicate.

Air-dry the specimen in the laboratory atmosphere until it reaches moisture equilibrium.

Determine the moisture content on a separate air-dried portion, as specified in ISO 638. Weigh this portion at the same time as the test portion used for incineration. For the determination of major elements, including magnesium, calcium, sodium and potassium, a 1 g to 2 g test portion (calculated as oven-dry) is recommended. For minor elements, including manganese, iron and copper, test portions of 5 g to 10 g are recommended. If trace levels of elements are needed, then use sample sizes that are larger than 10 g.

Carry out ashing of the test portion as described in ISO 1762.

NOTE In order to avoid flames, cover the dish with a lid. The lid should only cover the dish partially to avoid the occurrence of reducing conditions in the dish, in which case acid-insoluble compounds might be formed. Under reducing conditions, for example, copper might be lost due to the formation of a platinum alloy.

7.3 Dissolution of the residue

After ashing, allow the crucible to cool. To avoid splattering, carefully moisten the residue of ignition with water and add cautiously, in a fume hood, 5 ml of hydrochloric acid (4.2) to the crucible. Evaporate to dryness on a boiling water bath or hotplate or using an infrared lamp. Repeat this procedure.

NOTE For samples with high carbonate content, more than 10 ml of acid (2 × 5 ml) might be needed.

Add 5 ml of hydrochloric acid (4.2) to the residue. If some insoluble residue is visible, heat, without boiling, the crucible covered with a watch glass for a few minutes.

Using the filter paper (5.2), filter the content of the crucible into a 100 ml volumetric flask. To ensure that the transfer is complete, add another portion of 5 ml of hydrochloric acid (4.2) to the crucible and heat again if necessary. Filter this last portion of acid into the main portion in the volumetric flask with the aid of some water (5.1). If required for AAS, add 4 ml of lanthanum chloride solution (4.4) or 2 ml of cesium chloride solution (4.5) to the volumetric flask. Fill up to the mark with water and mix. This is the test solution.

8 Preparation of calibration solutions

It is important that the acid concentration and the lanthanum chloride/cesium chloride concentration in the calibration solutions are the same as in the test solution, since the acid and salt concentration affects the instrument signal.

Calibration solutions are unstable and should only be prepared on the day they are to be used and stored in plastic bottles. The standard stock solutions are less unstable and can be stored for several months.

Several elements may be combined in the same flask, if desired.

8.1 Atomic absorption spectrometry (AAS)

When atomic absorption spectrometry is used for the analysis, prepare at least three calibration solutions for each element in 100 ml volumetric flasks, each containing 10 ml of hydrochloric acid (4.2), by diluting the corresponding standard stock solutions (4.6). In addition, a blank solution similar to the calibration solutions, but containing no added element, shall be included.

When preparing the calcium and magnesium calibration solutions, 4 ml of the lanthanum chloride solution (4.4) shall also be added [ρ(La) will be 2 g/l] if an air/acetylene flame is used. When preparing the sodium, potassium and calcium calibration solutions, 2 ml of the cesium chloride solution (4.5) shall be added [ρ(Cs) will be 1 g/l] if an air/acetylene flame is used.

8.2 Plasma emission spectrometry (ICP/ES)

When plasma emission spectrometry is used, no more than two calibration solutions for each element are necessary, and no addition of lanthanum chloride or cesium chloride is required. Add 10 ml of hydrochloric acid (4.2) before diluting the corresponding standard stock solutions (4.6) to 100 ml. In addition, a blank solution similar to the calibration solutions, but containing no added element, shall be included.

9 Blank solution

A blank solution shall be prepared, omitting the test element, and containing the same amount of hydrochloric acid (4.2), as well as the same amount of lanthanum chloride (4.4) or cesium chloride (4.5), if required, as the calibration solutions.

10 Determination

For each element to be determined, optimize the conditions of the atomic absorption or plasma emission spectrometer, and operate the instrument as recommended by the manufacturer.

For atomic absorption, the recommended wavelengths are as follows:

- Magnesium: 285,2 nm
- Calcium: 422,7 nm
- Manganese: 279,5 nm
- Iron: 248,3 nm
- Copper: 324,8 nm
- Sodium: 589,0 nm
- Potassium: 766,5 nm

For plasma emission, the recommended emission lines are as follows:

- Magnesium: 279,55 nm (for low levels), 280,27 nm (for high levels)
- Calcium: 396,85 nm (for low levels), 317,93 nm (for high levels)
- Manganese: 257,61 nm
- Iron: 259,94 nm
- Copper: 324,75 nm
- Sodium: 589,00 nm
- Potassium: 766,50 nm

Carry out the measurement of the calibration solutions, the test solution and the blank solution. If the reading of the test solution is outside the range of the calibration curve, corrected for the blank, dilute with water to bring it within this range. All final dilutions of the test solution shall contain the same acid concentration (10 ml/100 ml) as the corresponding calibration solution, as well as lanthanum chloride/cesium chloride concentrations, if required.

NOTE If the test solution is used without dilution, then further addition of hydrochloric acid (4.2) is not necessary since the dilution already contains acid added after the ashing step.

Once the test solution is within the calibration range, determine the concentration of the element in the solution, by referring to the appropriate calibration curve.

In microprocessor-controlled spectrometers, the concentration is determined automatically and, consequently, plotting of calibration curves is not required.

11 Calculation

Calculate the concentration of each element in the test specimen from the following expression:

$$w_\mathrm{e} = \frac{f \rho_\mathrm{e} V}{m} \tag{1}$$

where

w_e is the acid-soluble content of the particular element in the sample, in milligrams per kilogram;

f is the dilution factor; if the test solution has not been diluted, then $f = 1$;

ρ_e is the concentration of the particular element in the test solution, corrected for the blank, as obtained from the calibration plot, in milligrams per litre;

V is the volume, in millilitres, of the original test solution (standard volume = 100 ml);

m is the mass of test portion used for ashing, on an oven-dry basis, in grams.

Calculate the mean to two significant figures for values below 10 mg/kg, and to three significant figures for values above 10 mg/kg.

NOTE Precision values are given in Annex A.

12 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the date and place of testing;

c) all information for complete identification of the sample;

d) the technique used for element determination (ICP/FAAS, flame atomic absorption spectrometry);

e) the result, expressed as indicated in Clause 11;

f) any departure from the procedure described in this International Standard or any other circumstances which may have affected the result.

Annex A
(informative)

Precision

A.1 General

In December 2009, an international round-robin was performed in which six laboratories from five different countries participated.

Six samples of different types of pulp, paper and board were tested. The samples were submitted to the participating laboratories for testing according to this International Standard. In some cases, the results were considered as outliers and were not included in the precision statement. Especially, the copper content was quite low in many cases, and some laboratories were not able to measure copper.

The calculations were made in accordance with ISO/TR 24498.

The repeatability and reproducibility limits reported are estimates of the maximum difference which should be expected in 19 out of 20 instances when comparing two test results for materials similar to those described under similar test conditions. These estimates may not be valid for different materials or different test conditions.

NOTE Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where $2{,}77 = 1{,}96\sqrt{2}$.

A.2 Repeatability

Table A.1 — Bleached softwood: Estimation of the repeatability of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_r mg/kg	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mg/kg
Magnesium (Mg)	6	213	6,7	3,3	19,3
Calcium (Ca)	6	38,5	2,4	6,1	6,5
Manganese (Mn)	6	0,32	0,05	16,9	0,2
Iron (Fe)	6	23,5	0,60	2,5	1,6
Copper (Cu)	5	0,37	0,04	12,3	0,1
Sodium (Na)	6	186	3,2	1,7	9,0
Potassium (K)	6	7,8	0,69	8,7	1,9

Table A.2 — Bleached hardwood: Estimation of the repeatability of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_r mg/kg	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mg/kg
Magnesium (Mg)	5	331	3,4	1,0	9,5
Calcium (Ca)	6	45,7	1,4	3,2	4,0
Manganese (Mn)	6	0,64	0,01	1,8	0,03
Iron (Fe)	6	32,6	0,70	2,2	1,9
Copper (Cu)	4	0,15	0,02	11,5	0,05
Sodium (Na)	6	281	5,2	1,8	14,3
Potassium (K)	6	8,0	0,26	3,2	0,7

Table A.3 — Chemo-thermo-mechanical pulp (CTMP): Estimation of the repeatability of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_r mg/kg	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mg/kg
Magnesium (Mg)	6	19,8	0,19	1,0	0,5
Calcium (Ca)	6	142	7,8	5,5	21,6
Manganese (Mn)	6	0,21	0,03	12,1	0,07
Iron (Fe)	5	1,1	0,13	11,6	0,4
Copper (Cu)	4	0,23	0,02	6,6	0,04
Sodium (Na)	6	1 070	14,5	1,4	40,3
Potassium (K)	5	6,8	0,23	3,5	0,6

Table A.4 — Uncoated paper: Estimation of the repeatability of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_r mg/kg	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mg/kg
Magnesium (Mg)	6	328	7,0	2,1	19,3
Calcium (Ca)	5	39 900	454	1,1	1 260
Manganese (Mn)	6	13,1	0,26	2,0	0,7
Iron (Fe)	6	77,2	1,2	1,5	3,2
Copper (Cu)	5	0,35	0,06	18,4	0,2
Sodium (Na)	6	1 370	37,9	2,8	105
Potassium (K)	5	23,0	0,80	3,5	2,2

Table A.5 — Coated paper: Estimation of the repeatability of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_r mg/kg	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mg/kg
Magnesium (Mg)	6	1 830	104	5,7	289
Calcium (Ca)	6	107 000	365	0,3	1 010
Manganese (Mn)	6	8,6	0,12	1,4	0,3
Iron (Fe)	6	267	10,3	3,9	28,6
Copper (Cu)	5	0,71	0,09	12,2	0,2
Sodium (Na)	6	1 560	54,4	3,5	151
Potassium (K)	6	42,7	1,3	3,0	3,5

Table A.6 — Board: Estimation of the repeatability of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_r mg/kg	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mg/kg
Magnesium (Mg)	6	234	4,1	1,7	11,3
Calcium (Ca)	6	19 800	471	2,4	1 300
Manganese (Mn)	6	10,1	0,27	2,6	0,7
Iron (Fe)	6	155	7,1	4,5	19,6
Copper (Cu)	5	1,0	0,15	15,1	0,4
Sodium (Na)	6	620	8,5	1,4	23,6
Potassium (K)	6	21,0	0,51	2,4	1,4

A.3 Reproducibility

Table A.7 — Bleached softwood: Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Magnesium (Mg)	6	213	44,8	21,0	124
Calcium (Ca)	6	38,5	9,7	25,3	26,9
Manganese (Mn)	6	0,32	0,06	17,2	0,15
Iron (Fe)	6	23,5	3,4	14,6	9,49
Copper (Cu)	5	0,37	0,36	97,6	0,99
Sodium (Na)	6	186	15,4	8,3	42,8
Potassium (K)	6	7,8	6,6	84,3	18,3

Table A.8 — Bleached hardwood: Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Magnesium (Mg)	5	331	78,6	23,7	218
Calcium (Ca)	6	45,7	11,0	24,1	27,0
Manganese (Mn)	6	0,64	0,08	12,7	0,23
Iron (Fe)	6	32,6	9,7	29,9	27,0
Copper (Cu)	4	0,15	0,13	84,0	0,36
Sodium (Na)	6	281	37,4	13,3	103
Potassium (K)	6	8,0	5,2	65,0	14,3

Table A.9 — Chemo-thermo-mechanical pulp (CTMP): Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Magnesium (Mg)	6	19,8	1,3	6,6	3,64
Calcium (Ca)	6	142	18,4	12,9	50,9
Manganese (Mn)	6	0,21	0,07	33,2	0,20
Iron (Fe)	5	1,1	0,40	35,3	1,12
Copper (Cu)	4	0,23	0,21	89,0	0,57
Sodium (Na)	6	1 070	118	11,1	328
Potassium (K)	5	6,8	2,4	35,2	6,59

Table A.10 — Uncoated paper: Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Magnesium (Mg)	6	328	38,6	11,8	107
Calcium (Ca)	5	39 900	3 450	8,6	9 550
Manganese (Mn)	6	13,1	2,9	22,1	8,0
Iron (Fe)	6	77,2	20,5	26,5	56,7
Copper (Cu)	5	0,35	0,17	49,0	0,5
Sodium (Na)	6	1 370	183	13,4	508
Potassium (K)	5	23,0	7,6	33,2	21,1

Table A.11 — Coated paper: Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Magnesium (Mg)	6	1 830	288	15,8	799
Calcium (Ca)	6	107 000	11 600	10,8	32 200
Manganese (Mn)	6	8,6	2,4	27,5	6,6
Iron (Fe)	6	267	102	38,1	282
Copper (Cu)	5	0,71	0,16	22,2	0,4
Sodium (Na)	6	1 560	286	18,3	791
Potassium (K)	6	42,7	29,4	68,7	81,3

Table A.12 — Board: Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Magnesium (Mg)	6	234	25,2	10,8	69,9
Calcium (Ca)	6	19 800	2 890	14,6	8 000
Manganese (Mn)	6	10,1	1,5	14,7	4,1
Iron (Fe)	6	155	32,5	20,9	90,1
Copper (Cu)	5	1,0	0,37	37,4	1,0
Sodium (Na)	6	620	139	22,4	385
Potassium (K)	6	21,0	12,1	57,6	33,5

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