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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 10742-2008

Replacing GB 10742-1989

Raw fiber material - Determination of pectin content

造纸原料果胶含量的测定

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1 Scope

GB/T 10742-2008 is the Chinese national standard on raw fiber material - determination of pectin content, in the field of paper technology. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 19 August 2008 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 May 2009. Classification: ICS 85.010, CCS Y30. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Standard specifies the determination methods of pectin content in raw fiber material. This Standard applies to the determination of pectin content in various raw fiber materials. This Standard provides two determination methods of pectin content, namely the gravimetric method and the carbazole colorimetric method. The two determination methods of are equal validity.

2 Normative references

The terms in the following documents become the terms of this Standard by reference to

this Standard. For dated references, all subsequent amendments (not including errata content) or revisions do not apply to this standard. However, parties to agreements that are based on this Standard are encouraged to study whether the latest versions of these documents can be used. For undated references, the latest edition applies to this Standard.

GB/T 2677.1, Fibrous raw material of sampling for analysis

GB/T 2677.2, Determination of moisture content in fibrous raw material

GB/T 6682, Basic requirements on exhibition logistics service (GB/T 6682-2008, ISO 3696.1987, MOD)

3 Method 1, Gravimetric method

3.1 Principle Use ammonium oxalate solution to extract the pectin in the raw material; then, add an ethanol solution containing hydrochloric acid, to separate the pectin from the extract; then, use a dilute ammonia solution to dissolve the obtained pectin; then, add sodium hydroxide to hydrolyze all the pectin into soluble pectinate; finally, use calcium chloride to precipitate calcium pectinate. Use the content of calcium pectinate to express the content of pectin.

3.2 Reagents Unless otherwise stated, use only reagents that are identified as analytical reagents in the analysis.

3.2.1 Water, GB/T 6682, grade 3.

3.2.2 Benzene-alcohol mixture. Measure 33 volumes of ethanol and 67 volumes of benzene and mix them. 3.2.3 1% ammonium oxalate solution. Weigh 5 g of anhydrous ammonium oxalate and dissolve it in water; then, add water to adjust the volume to 500 mL. 3.2.4 0.5% ammonium oxalate solution. Weigh 2.5g of anhydrous ammonium oxalate and dissolve it in water; then, add water to adjust the volume to 500 mL.

3.2.5 Ammonia water, NH3H2O, aqueous solution of ammonia, rho =

0.90 g/mL.

3.2.6 Ethanol solution containing hydrochloric acid. Measure 1 000 mL of ethanol; add 11 mL of hydrochloric acid (rho

1.19 g/ mL); mix well.

3.2.7 Ethanol washing solution containing hydrochloric acid. Measure 1 000 mL of ethanol, 11 mL of hydrochloric acid (rho

1.19 g/mL) and 250 mL of water; mix well. 3.2.8

0.1 mol/L sodium hydroxide solution. Weigh 4 g of sodium hydroxide and dissolve it in water; then, add water to fix the volume to 1 000 mL. 3.2.9 1 mol/L acetic acid solution.

Measure 29 mL of glacial acetic acid (99% ~ 100%); add water to adjust the volume to 500 mL. 3.2.10 1 mol/L calcium chloride solution. Weigh 110 g of anhydrous calcium chloride and dissolve it in water; add water to adjust the volume to 1000 mL.

3.3 Apparatus Commonly used apparatus in the laboratory and the following apparatus.

3.3.1 Electronic balance, sensitivity

0.001 g.

3.3.2 Soxhlet extractor. 3.3.3 500 mL conical flask with reflux condenser.

3.3.4 Thermostatically controlled hot plate.

3.3.5 Oven, adjustable at 105 °C ± 2 °C.

3.4 Test procedure

3.4.1 Sample collection and processing Follow the provisions of GB/T 2677.1.

3.4.2 Weighing of samples Accurately weigh 1 g ~ 3 g (accurate to

0.001

g) of the sample and prepare two parallel samples. If the pectin content of the sample is high, adjust the sample amount according to the test value, where the minimum shall not be less than 1 g. At the same time, weigh another sample and determine the moisture content according to GB/T 2677.2.

3.4.3 Purification of sample Use qualitative filter paper to wrap the sample; tie it with thread; put it into the Soxhlet extractor (3.3.2). Add 100 mL of benzene alcohol mixture (3.2.2); place it in a boiling water bath for extraction for 3 h to remove interfering impurities; take out the sample and spread it out to air dry.

Note. The test shall be carried out in a fume hood. The used benzene-alcohol mixture can be distilled and purified before reuse.

3.4.4 Extraction of pectin Transfer the air-dried sample (3.4.3) to a 500 mL conical flask (3.3.3); add 100 mL of 1% ammonium oxalate solution (3.2.3); install a reflux condenser; heat in a boiling water bath for 3 h. Filter out the extract by pouring; try to keep the residue in the conical flask; do not let it flow into the filter paper. Add 100 mL of 0.5% ammonium oxalate solution (3.2.4) to the conical flask; install a reflux condenser; put it back in a boiling water bath; heat for 2 h to fully extract the pectin. Use the filter paper used last time to filter out the extract. Use hot water to wash the residue and filter paper three times; combine the filtrate obtained twice and the washing liquid in a 500 mL beaker.

3.4.5 Precipitation of pectin Place the filtrate on a thermostatically controlled hot plate; place an asbestos net on it; control the temperature so that the filtrate does not boil;

evaporate and concentrate to 70 mL ~ 80 mL; cool it down and transfer it to a 100 mL volumetric flask; add water to the mark; shake it well. Transfer 25 mL of this solution to a 500 mL beaker; then slowly add 90 mL of ethanol solution containing hydrochloric acid (3.2.6) under constant stirring; let it stand overnight; filter. Use about 30 mL of ethanol washing solution containing hydrochloric acid (3.2.7) to wash the precipitated pectin substance three times.

3.4.6 Dissolution of pectin additional sample shall be tested. After excluding abnormal values, take the average of the three test values as the reported value.

4 Method 2, Carbazole colorimetric method

4.1 Principle Use ammonium oxalate solution to extract the pectin in the raw material; then, add an ethanol solution containing hydrochloric acid, to separate the pectin from the extract. Then, use a dilute ammonia solution to dissolve the obtained pectin, which is hydrolyzed in a strong acid to generate galacturonic acid, and reacts with carbazole to form a purple-red solution that can be used for colorimetric determination. Use the content of galacturonic acid to express the content of pectin.

4.2 Reagents Unless otherwise stated, use only reagents that are identified as analytical reagents in the analysis.

4.2.1 Water, GB/T 6682, grade 3. 4.2.2 1% ammonium oxalate solution. Weigh 5 g of anhydrous ammonium oxalate and dissolve it in water; then, add water to adjust the volume to 500 mL. 4.2.3 0.5% ammonium oxalate solution. Weigh 2.5g of anhydrous ammonium oxalate and dissolve it in water; then add water to adjust the volume to 500 mL.

4.2.4 Ammonia water, NH3H2O, aqueous solution of ammonia, rho =

0.90 g/mL.

4.2.5 Ethanol solution containing hydrochloric acid. Measure 1 000 mL of ethanol; add 11 mL of hydrochloric acid (rho =

1.19 g/mL); mix well.

4.2.6 Ethanol washing solution containing hydrochloric acid. Measure 1 000 mL of ethanol, 11 mL of hydrochloric acid (rho =

1.19 g/mL) and 250 mL of water; mix well. 4.2.7 1000 mg/L galacturonic acid standard solution. Weigh

0.1 g (accurate to 0.000 1

g) of galacturonic acid; dissolve it in water; add water to adjust the volume to 100 mL. 4.2.8 0.15% carbazole anhydrous ethanol. Weigh