



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 24131.1-2018

**Rubber, raw -- Determination of volatile matter content -- Part 1:
Hot-mill method and oven method**

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1.1 This part of GB/T 24131.1 specifies two methods for determining the volatile matter content of raw rubber by hot-mill method and oven method.

1.2 These methods are suitable for determining the volatile matter content of

the group-R rubbers as listed in GB/T 5576. The group-R rubber refers to a rubber containing an unsaturated carbon chain, such as natural rubber and a rubber which is at least partially synthesized from a diolefin. These methods are also applicable to the determination of other rubbers, but in this case, it must be verified that the change in mass is due only to the loss of actual volatile matter rather than rubber degradation.

1.3 The hot-mill method is neither suitable for natural rubber and synthetic

rubber that is difficult to handle on hot-mills, nor suitable for chip and powdered rubber.

1.4 Determination by these methods may not give the same result. Therefore, in the case of controversy, oven method A is a reference method.

Note. Refer to Appendix A for the test methods applicable to different types of rubber.

2 Normative references

The following documents are essential to the application of this document. For the dated

documents, only the versions with the dates indicated are applicable mass m_4). Before each weighing, it shall place the test portion in a desiccator to cool it to room temperature.

5.3.2 Hot-mill method B

5.3.2.1 Hot-mill Method B1

5.3.2.1.1 From the laboratory sample, weigh about 250 g of test portion, accurate to 0.1 g (mass m_5).

5.3.2.1.2 Adjust the roller's spacing to 0.30 mm $\pm$ 0.05 mm. Follow the lead strip method as used in GB/T 6038 to measure the roller's spacing. The surface temperature of the roller is kept at 105 °C $\pm$ 5 °C. Make the test portion pass through the roller for at least two times, accurate to 0.1 g. Then make the test portion pass through the roller for at least two times again; weigh it.

5.3.2.1.3 If the difference of mass of the test portion before and after passing through the roller is less than 0.1 g, the test portion is considered to be completely dry. Otherwise, continue to pass the test portion through the roller twice until the difference between the two consecutive weighing values is less than 0.1 g (final mass m_6).

5.3.2.2 Hot-mill method B2

5.3.2.2.1 From the laboratory sample, weigh about 250 g of test portion, accurate to 0.1 g (mass m_5).

5.3.2.2.2 Adjust the roller's spacing to 0.30 mm $\pm$ 0.05 mm. Follow the lead strip method as used in GB/T 6038 to measure the roller's spacing. The surface temperature of the roller is kept at 105 °C $\pm$ 5 °C. Make the test portion repeatedly pass through the open-mill for 4 min; weigh it, accurate to 0.1 g.

Then make the test portion pass through the open-mill for 2 min; weigh it, accurate to 0.1 g.

5.3.2.2.3 If the difference in mass between the end of 4 min and the end of 6 min is less than 0.1 g, calculate the volatile matter content. Otherwise, make the test portion pass through the open-mill for another 2 min, until the difference between the two consecutive weighing values is less than 0.1 g (final mass m_6).

Note. After the test, the test portion is preferably cooled in a desiccator.

5.4 Representation of result

5.4.1 Hot-mill method A The volatile matter content (w_1) is calculated by mass fraction (%) according to formula (1).

6.3.1.2 Synthetic rubber

6.3.1.2.1 According to the provisions of GB/T 15340, from the laboratory sample, weigh about 250 g of test sample; homogenize it according to Appendix B.

Weigh the mass of the test sample before and after the homogenization (the mass is m_7 and m_8 , respectively), accurate to 0.1 g. If necessary, from the homogenized test sample, cut the test portion for other chemical and physical tests.

6.3.1.2.2 From the homogenized test sample, weigh about 10 g of the test portion, accurate to 0.001 g (mass m_9).

6.3.1.2.3 Set the temperature of the open-mill's roller to $70\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. Make the test portion pass through the roller for two times to press it into a thin sheet which has a thickness of less than 2 mm.

6.3.1.2.4 If the test portion cannot be pressed into a thin sheet, from the homogenized test portion, weigh about 10 g of the test portion. Manually cut it into a rubber block which has a side length of 2 mm ~ 5 mm. Place it in a clean watch glass or aluminum dish which is convenient for weighing to weigh it, accurate to 0.001 g (mass m_9).

6.3.1.2.5 According to 6.3.1.1.4, dry the test portion and weigh it, accurate to 0.001 g (final mass m_{10}).

6.3.1.2.6 If the rubber is stuck to the roller and it is difficult to weigh the mass of the test portion before and after homogenization, take about 10 g of the test portion directly from the laboratory sample, manually cut it into a rubber block which has a side length of 2 mm ~ 5 mm. Place it in a clean watch glass or aluminum dish which is convenient for weighing to weigh it, accurate to 0.001 g (mass m_9). According to 6.3.1.1.4, dry the test portion and weigh it, accurate to 0.001 g (final mass m_{10}).

6.3.1.2.7 If the rubber is in powder form, it may randomly weigh about 10 g of test portion. Place it in a clean watch glass or aluminum dish which is convenient for weighing to weigh it, accurate to 0.001 g (mass m_9). Follow the requirements of 6.3.1.1.4 to dry the test portion and weigh it (final mass m_{10}), accurate to 0.001 g.

6.3.2 Oven method B

6.3.2.1 Take about 250 g of the test portion. Make it pass through the open-mill's roller which has a surface temperature of about $30\text{ }^{\circ}\text{C}$ and a roller spacing of $0.30\text{ mm} \pm 0.05\text{ mm}$ for two times, to roll it into thin sheet. From this sheet, randomly take two sets of about 50 g of test portion, accurate to 0.01 g (mass m_{11}). Place the test portion in an oven at a temperature of $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h, take it out, place it in a desiccator to cool it to room temperature, weigh it,

Appendix C (Informative) Precision

C.1 Summary According to the requirements of ISO/TR 9272:1986, finish the calculation of precision for repeatability and reproducibility. For the concepts and terminology of precision, see ISO/TR 9272.

C.2 ITP details carried out in 1984 C.2.1 At the end of 1984, the Malaysian Rubber Research Institute organized a precision according to the Interlaboratory Test Procedure (ITP). Two types of materials were sent to each laboratory and tested in two separate procedures in March and July, respectively.

- a) Mixed samples A and B of two rubbers;
- b) Two separate rubber samples A and B.

C.2.2 For the two mixed samples and the two separate samples above, the test results are the average of three independent test results.

C.2.3 Use the oven method A to determine the volatile matter content.

C.2.4 Follow the ITP requirements to carry out the type-1 precision test.

Repeatability and reproducibility tests were performed in a few days, where the mixed samples are tested by 14 laboratories, the separate samples were tested in 13 laboratories, respectively.

C.3 ITP details carried out in 2003 C.3.1 During the April and May 2003, according to the ITP requirements, seven laboratories participated in the hot-mill method B test, eight laboratories participated in the oven method B test.

C.3.2 According to hot-mill method B and oven method B respectively, test two kinds of raw rubbers. sample C (SBR 1500) and sample D (non-oil-filled BR).

C.3.3 According to the ITP requirements, the above-mentioned laboratory repeat the test on the two raw rubber samples. The results obtained are as shown in Table C.3 and Table C.4, respectively. The average value and precision estimation of oven method B and hot-mill method B are given