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

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**Coal direct liquefaction - Determination of softening point of
liquefaction residue - Ring and ball apparatus**

煤炭直接液化 液化残渣软化点的测定 环球法

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3 Terms and definitions

3 Some of the terms and definitions given in GB/T 23251 together with the following apply to this document.

3.1 coal direct liquefaction: The process in which coal undergoes a hydrogenation reaction with hydrogen under high pressure, high temperature and the action of a catalyst, and is thereby converted directly into a liquid fuel. The source given is GB/T 23251-2009, definition 3.8.

3.2 coal liquefaction residue: The solid mixture left after the heavy product of coal direct liquefaction has been separated from the liquid, mainly by vacuum distillation, and which includes heavy liquefaction oil, bituminous substances, unconverted organic matter of the coal, mineral matter and catalyst.

3.3 softening point of liquefaction residue: The temperature, expressed in degrees Celsius, at which a liquefaction residue specimen heated under specified conditions softens to a given consistency.

4 Principle of the method

4 Two horizontal discs of residue are placed in shouldered brass, or stainless steel, rings, and one steel ball is placed on each disc of residue. The assembly is heated in a heating medium at a set rate of temperature rise, and the temperature at which the softening specimen lets the two steel balls fall 25 mm and just touch the lower plate of the stand is the softening point of the liquefaction residue.

5 Reagents and materials

5.1 Glycerol: analytical grade.

5.2 Metal plate: a metal plate smooth on both faces, with dimensions of about 50 mm by 75 mm.

5.3 Scraper: made of metal, with a flat blade.

5.4 Crucible: porcelain, 25 mL.

6 Apparatus

6.1 Softening point determination apparatus: see Figure 1, whose key names the heater, the lower plate of the stand, the beaker, the shoulder ring support plate, the shoulder ring, the ball centring guide, the steel ball, the stand and the thermometer.

6.1.1 Electric furnace or electric hot plate: maximum power 1 kW, adjustable.

6.1.2 Glass beaker: diameter 105 mm, height 150 mm.

6.1.3 Shoulder ring support plate and stand: made of chromium-plated copper or of stainless steel, their shape and dimensions being given in Figure 2; the distance from the bottom of the shoulder ring support plate to the upper surface of the lower plate of the stand is 25 mm.

6.1.4 Shoulder rings: two, made of brass or stainless steel, their dimensions and specification being given in Figure 3.

6.1.5 Ball centring guide: made of brass or stainless steel, its shape and dimensions being given in Figure 4.

6.1.6 Steel balls: two, made of stainless steel, diameter 9.5 mm, mass 3.50 g plus or minus 0.05 g.

6.1.7 Mercury-in-glass thermometer: total immersion type, measuring range 50 degrees Celsius to 240 degrees Celsius, smallest scale division 1 degree Celsius.

7 Preparation of the analysis sample

7 The coal liquefaction residue sample is crushed to below 3 mm, about 100 g is divided out, and this is ground to below 0.4 mm to serve as the analysis sample, which is kept for use.

8 Test preparation

8.1 Melting the sample. About 15 g of analysis sample of below 0.4 mm is placed in a porcelain crucible and heated in a fume cupboard with an electric heater at 30 % of its heating power, stirring from time to time to prevent local overheating, until the specimen melts and can flow. The specimen is watched, and no yellow gas is to escape; if it does, the test is void. If the test is repeated, a fresh sample is melted in a clean crucible.

8.2 Preparation of the residue discs. The shoulder rings are placed on the metal plate and the melted sample is at once poured into them until it stands slightly above the upper edge of the rings; it is cooled to room temperature and the excess specimen is scraped off the shoulder rings with a preheated metal scraper, the face of the blade being kept flush with the face of the ring, so that each disc is full and level with the top of its ring.

9 Determination procedure

9.1 The softening point determination apparatus is assembled as in Figure 1 and placed in a fume cupboard. The shoulder rings holding the specimen are placed in the round holes of the ring support plate, the centring guide and the steel ball are fitted, and the stand is lowered into the beaker filled with glycerol, the glycerol having been preheated to 60 degrees Celsius, no part of the assembly being allowed to carry bubbles. The thermometer is then inserted so that the lower end of the mercury column is level with the bottom of the rings but does not touch the ring or the ring support plate.

9.2 Heat is applied from the bottom of the beaker so that the temperature rises at the constant rate of 5 degrees Celsius per minute plus or minus 0.5; if the rate of rise goes outside that limit, the test is void.

9.3 When a residue disc softens and sags and the steel ball just touches the lower plate of the stand, the temperature is read at once. The arithmetic mean of the softening temperatures of the two specimens is taken as the softening point of the sample.

10 Precision of the method

10.1 Repeatability limit. The absolute value of the difference between two independent test results obtained under repeatability conditions is not to be greater than 2 degrees Celsius at a confidence probability of 95 %.

10.2 Reproducibility limit. The absolute value of the difference between two independent test results obtained under reproducibility conditions is not to be greater than 3 degrees

Celsius at a confidence probability of 95 %.

11 Test report

11 The test report is to contain at least the following information: a) the identification of the sample; b) the standard applied; c) the test result; d) any departure from the standard; e) any abnormal phenomenon that occurred during the test; f) the date of the test.