
**Plastics — Unsaturated-polyester resins —
Measurement of gel time at ambient
temperature**

*Plastiques — Résines de polyesters non saturés — Mesurage du temps de
gel à température ambiante*

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 2535 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This third edition cancels and replaces the second edition (ISO 2535:1997), which has been revised to include four variants of the original method, calibration procedures for the device used to determine the gel time (see clauses A.2 and B.2) and precision data.

Annexes A and B form a normative part of this International Standard.

Plastics — Unsaturated-polyester resins — Measurement of gel time at ambient temperature

1 Scope

This International Standard specifies methods of measuring, under defined conditions, the gel time at ambient temperature (i.e. within the range 18 °C to 30 °C) of unsaturated-polyester resins.

The referee temperature, if needed, is 25 °C.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 472:1999, *Plastics — Vocabulary*.

ISO 4619:1998, *Driers for paints and varnishes*.

ISO 15038:1999, *Plastics — Organic-perester crosslinking agents for unsaturated-polyester thermosetting materials — Determination of active-oxygen content*.

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 472 apply, together with the following term and definition.

3.1

gel time at ambient temperature

the time which elapses between the end of the addition of an initiator to a resin and the moment when the viscosity reaches 50 Pa·s

4 Principle

The time required for a defined formula (resin, accelerator, initiator) at ambient temperature (within the range 18 °C to 30 °C) to reach a viscosity of 50 Pa·s (the viscosity conventionally accepted as corresponding to the gel state) is measured.

Four methods are specified:

Method A1 uses an automatic-release device (see annex A) in which a glass rod rotates in a resin mixture contained in a test tube, and rotation of the glass rod is stopped automatically when a viscosity of 50 Pa·s is reached.

Method A2 also uses an automatic-release device, but the resin mixture is contained in a beaker.

Method B1 uses a continuous-measurement device (see annex B) in which a glass rod executes a horizontal oscillatory motion in a resin mixture contained in a test tube. The viscosity is measured continuously to obtain a plot of viscosity against time.

Method B2 also uses a continuous-measurement device, but the resin mixture is contained in a beaker.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Toluene.

5.2 Reference accelerator: cobalt octoate solution containing 0,6 % by mass of Co.

This solution may, for example, be prepared as follows:

Pipette 5 ml of a stock solution of cobalt octoate, containing 6 % by mass of Co, in an inert solvent (for example an aliphatic ester) into a volumetric flask fitted with a ground-glass stopper. Dilute to 50 ml with toluene (5.1) and mix well by shaking.

1 ml of this solution corresponds to 0,100 g of the stock solution of cobalt octoate containing 6 % by mass of Co.

If it is necessary to determine the Co content of any solutions, this shall be done in accordance with ISO 4619.

5.3 Reference initiator: 50 % by mass solution of methyl ethyl ketone peroxide in dimethyl phthalate, the solution assaying 9 % of active oxygen.

Check the active-oxygen content using the method specified in ISO 15038.

It is desirable to store the peroxide solution at low temperature (5 °C), e.g. in a refrigerator. In this case, the solution shall be maintained at room temperature for 24 h prior to use.

WARNING — Never mix the methyl ethyl ketone peroxide with the cobalt octoate solution, as an explosive mixture is formed. Mix each separately into the unsaturated-polyester resin.

6 Apparatus

6.1 Measurement vessel, capable of containing the quantity of mixture required for the determination:

- for methods A1 and B1, **borosilicate-glass test tube**, minimum length 150 mm, internal diameter 18 mm;
- for methods A2 and B2, **polypropylene beaker**, capacity 50 ml.

6.2 Viscosity-measurement device: any device which can be calibrated and is capable of measuring a viscosity of 50 Pa·s (± 2 Pa·s) with the least possible effect on the viscosimetric properties of the mixture; the shear rate shall be negligible.

NOTE Examples of suitable automatic-release and continuous-measurement devices are described in annexes A and B, respectively.

6.3 Water bath, thermostatically controlled to within $\pm 0,5$ °C of the test temperature (which will be within the range 18 °C to 30 °C) and protected from light.

6.4 Glass beaker, capacity 100 ml, for preparing the resin mixture.

6.5 Two graduated pipettes, capacity 1 ml, with 0,01 ml graduations, or **two precision microburettes,** of at least equivalent accuracy.

IMPORTANT — The pipettes (or microburettes) shall be clearly identified (accelerator, initiator) to avoid any possibility of confusion.

6.6 Balance, accurate to at least 0,01 g.

6.7 Spatula, stainless steel.

6.8 Stopwatch, accurate to 1 s.

6.9 Thermometer, capable of reading a temperature within the range 18 °C to 30 °C to $\pm 0,5$ °C.

7 Procedure

7.1 Carry out the determination at ambient temperature (i.e. in the range 18 °C to 30 °C). The procedure described in 7.2 to 7.9, has been written, for simplicity's sake, for a temperature of 25 °C (the referee temperature).

To obtain the best possible accuracy, the container and the reagents used during the determination shall be at a temperature close to the test temperature.

With pre-accelerated resins, omit 7.4.

7.2 Weigh 50 g \pm 0,1 g of the resin to be analysed into a beaker (6.4).

7.3 Place the beaker in the bath (6.3) until the resin reaches a temperature of 25 °C \pm 0,5 °C.

7.4 Using a pipette or microburette (see 6.5), add 0,5 ml of cobalt octoate solution (5.2). Mix with the spatula (6.7).

7.5 Using the second pipette or microburette (see 6.5), add 0,7 ml of methyl ethyl ketone peroxide solution (5.3).

7.6 Start the stopwatch (6.8) and mix with the spatula for 30 s.

7.7 Transfer the mixture to the measurement vessel (6.1) and place the viscosity-measuring device (6.2) in position in the manner described in the procedure given in annex A or annex B.

7.8 Record the time, in minutes and seconds, when the viscosity reaches 50 Pa·s.

7.9 Carry out a second determination under the same conditions. The two results shall not differ by more than 10 %. If they do, continue testing until this requirement is fulfilled for two consecutive measurements.

8 Expression of results

The gel time, at the test temperature (within the range 18 °C to 30 °C), is the arithmetic mean of the two test results, expressed in minutes and seconds.

9 Precision

Precision data were determined from a round-robin test organized in the year 2000 using method B2 and involving eight European laboratories. Two commercial unsaturated-polyester resins with different gel times were analysed, and the results processed in accordance with ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*. The data may be obtained from the ISO/TC 61/SC 12 Secretariat at ASTM.

The repeatability and reproducibility calculated from the round-robin data are given in Table 1.

Table 1 — Repeatability and reproducibility

Sample	Gel time (averaged) s	Repeatability s_r	Reproducibility s_R
UP resin A	667	17	34
UP resin B	2 758	73	196

where

s_r is the standard deviation within a laboratory for results obtained on identical material by one operator, in one laboratory, using the same equipment within a short interval of time, using the standardized test method;

s_R is the standard deviation between the laboratories for results obtained on comparable material by operators in different laboratories, using the standardized test method.

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all details necessary for the complete identification of the sample;
- c) the method used (A1, A2, B1 or B2) and details of the measurement device;
- d) the test temperature (in the range 18 °C to 30 °C);
- e) the gel time;
- f) the date of the determination.

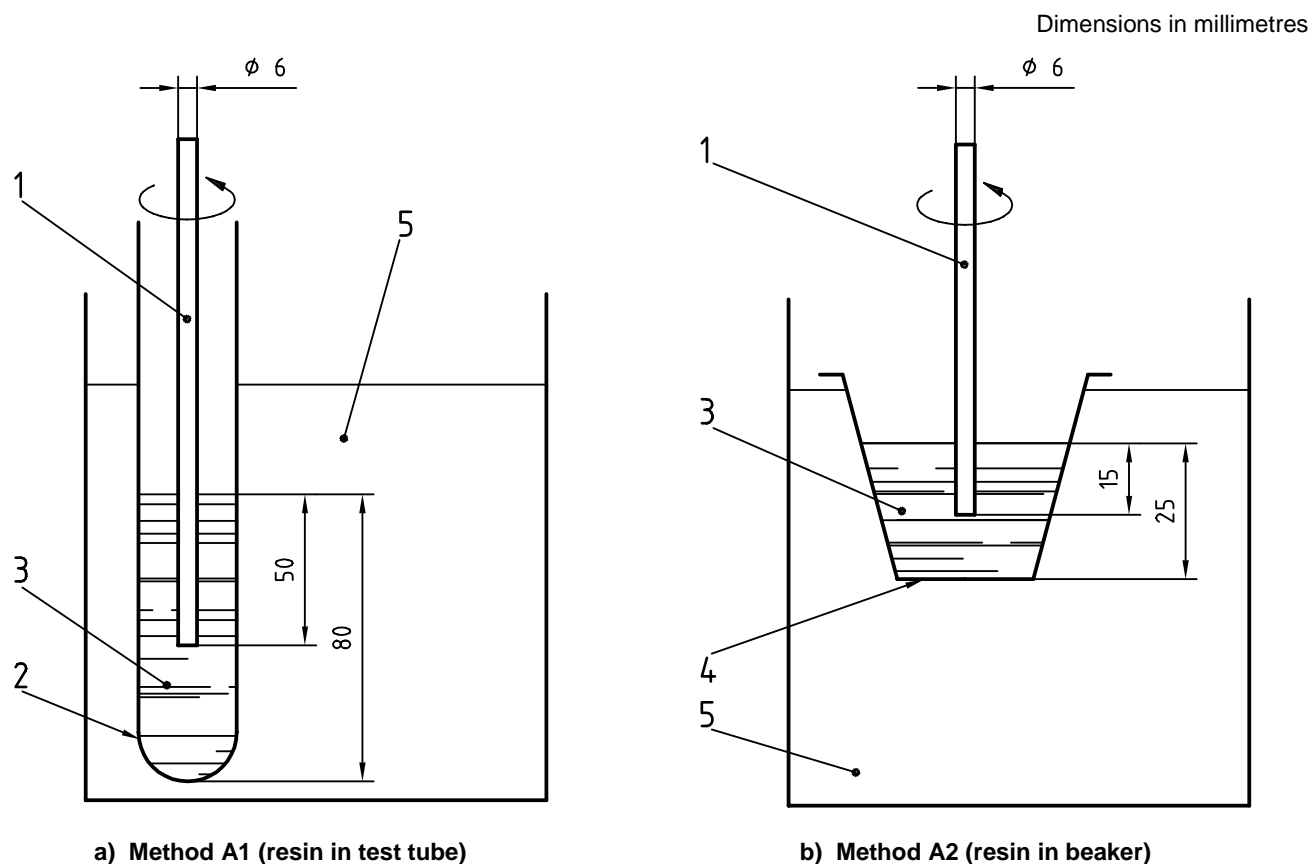
Annex A (normative)

Automatic-release apparatus

A.1 Basic principles

A glass rod, 6 mm in diameter and of adequate length, is inserted to a depth of 50 mm into a resin mixture contained in a test tube (method A1) or to a depth of 15 mm into a mixture contained in a beaker (method A2) (see Figure A.1).

The rod is rotated about its axis at very low speed (1 Hz to 2 Hz) by means of a steel torsion wire connected to an electric motor. When the torsion wire has twisted through an angle corresponding to a viscosity of 50 Pa·s (see calibration procedure described in clause A.2), the gel time determination is completed. An automatic release stops the electric motor and records the corresponding time.



Key

- 1 Glass rod (diameter 6 mm)
- 2 Borosilicate-glass test tube
- 3 Resin mixture
- 4 Polypropylene beaker
- 5 Thermostatically controlled water bath

Figure A.1 — Schematic drawing of automatic-release apparatus

A.2 Calibration procedure

A.2.1 Procure a standard oil with a viscosity of 50 Pa·s at a temperature x and 48 Pa·s at a temperature y (the exact temperatures will be given on the calibration certificate supplied with the oil), these viscosities being measured at a low shear rate.

NOTE The oil may be either a Cannon oil or a silicone oil, provided it is accompanied by a calibration certificate established in accordance with the relevant standards.

A.2.2 Introduce into the measurement vessel the quantity of oil necessary for the method being used (A1 or A2).

A.2.3 Insert the rod to the depth necessary (50 mm for method A1, 15 mm for method A2).

A.2.4 Place the whole assembly in the thermostatic bath maintained at temperature x and wait for the temperature to stabilize. Then place this assembly under the measurement apparatus.

A.2.5 Start the measurement apparatus and adjust the automatic-release mechanism to the threshold limit of the measurement system. Stop the measurement apparatus.

A.2.6 Increase the temperature to y and wait for the temperature to stabilize.

A.2.7 Start the measurement apparatus, and check that release does not occur at this temperature but measurement continues.

Temperatures x and y shall be situated between 20 °C and 50 °C in order to ensure that the calibration is carried out under the correct conditions.

EXAMPLE

Cannon oil, Ref. No. 600:	60 Pa·s at 25 °C
	50 Pa·s at 35 °C
	40 Pa·s at 50 °C

(The precise temperature and viscosity values will be indicated on the calibration certificate accompanying the oil.)

Annex B (normative)

Continuous-measurement apparatus

B.1 Basic principles

A glass rod, 6 mm in diameter and of adequate length, is inserted to a depth of 50 mm into a resin mixture contained in a test tube (method B1) or to a depth of 15 mm into a mixture contained in a beaker (method B2) (see Figure B.1).

The rod is driven to execute a low-frequency (< 2 Hz), low-amplitude (< 2 mm) horizontal oscillatory motion (neither rotational nor pendular).

The force of reaction of the mixture to the rod motion is measured using a sensor. When this force corresponds to a viscosity of 50 Pa·s (see calibration procedure described in clause B.2), the corresponding time is noted.

The calibration may then be stopped, or it may be continued in order to obtain the whole crosslinking (and therefore product reactivity) curve up to complete setting.

Insertion of a thermocouple into the measurement vessel [see Figure B.1 b)] allows the rate of heat evolution to be followed (see Figure B.2).

NOTE 1 Methods B1 and B2 are particularly suitable for testing to complete setting and allow work with different quantities of mixtures in order to evaluate the effect of the mass present on the reactivity and on the heat-evolution properties.

NOTE 2 In method B2, it is possible to replace the 6 mm diameter glass rod by a threaded metal rod fitted with a 16-mm-diameter bakelite ball in order to enhance the measurement sensitivity.

B.2 Calibration procedure

B.2.1 Procure a standard oil with a viscosity of 50 Pa·s at a temperature x , the viscosity being measured at a low shear rate.

NOTE The oil may be either a Cannon oil or a silicone oil, provided it is accompanied by a calibration certificate established in accordance with the relevant standards.

B.2.2 Introduce into the measurement vessel the quantity of oil necessary for the method being used (B1 or B2).

B.2.3 Insert the rod to the depth necessary (50 mm for method B1, 15 mm for method B2).

B.2.4 Place the whole assembly in the thermostatic bath maintained at temperature x and wait for the temperature to stabilize. Then place this assembly under the measurement apparatus.

B.2.5 Start the measurement apparatus and note the measured value of the force. This value corresponds to a viscosity of 50 Pa·s under the conditions of measurement.

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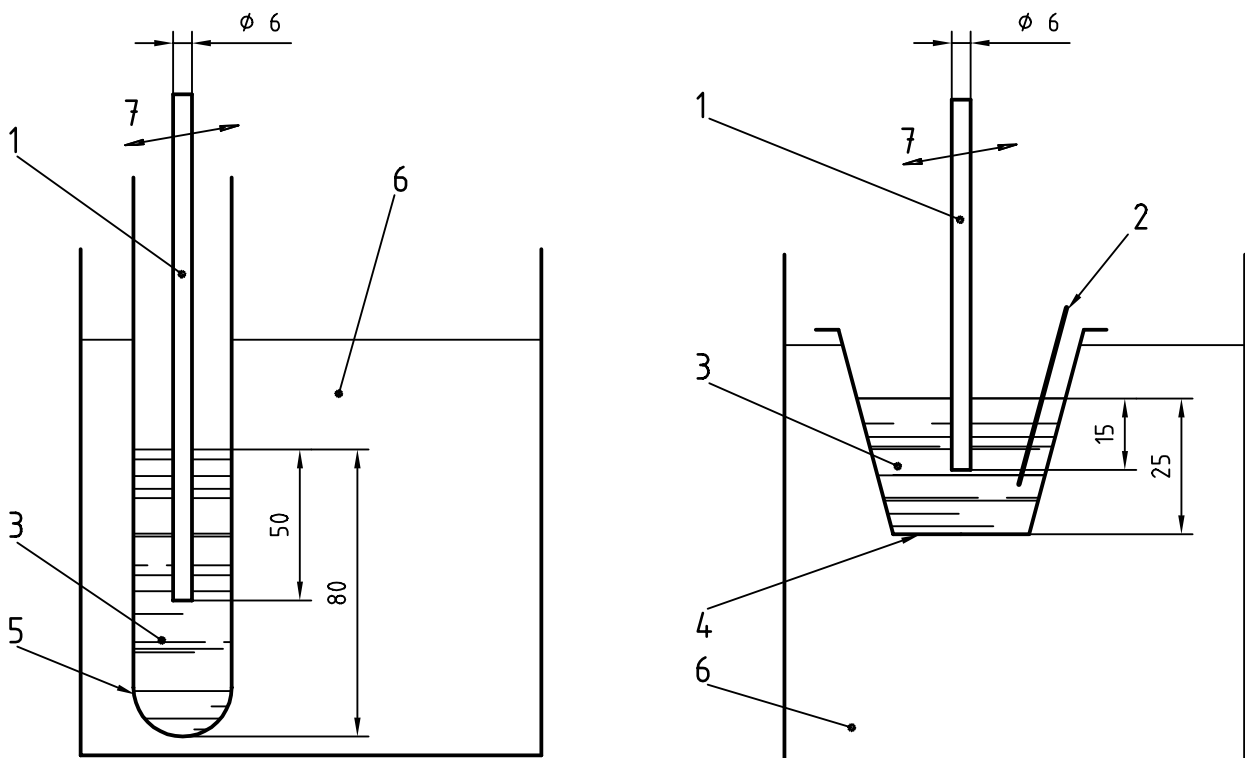
Temperature x shall be situated between 20 °C and 50 °C in order to ensure that the calibration is carried out under the correct conditions.

EXAMPLE

- Cannon oil, Ref. No. 600: 60 Pa·s at 25 °C
- 50 Pa·s at 35 °C
- 40 Pa·s at 50 °C

(The precise temperature and viscosity values will be indicated on the calibration certificate accompanying the oil.)

Dimensions in millimetres



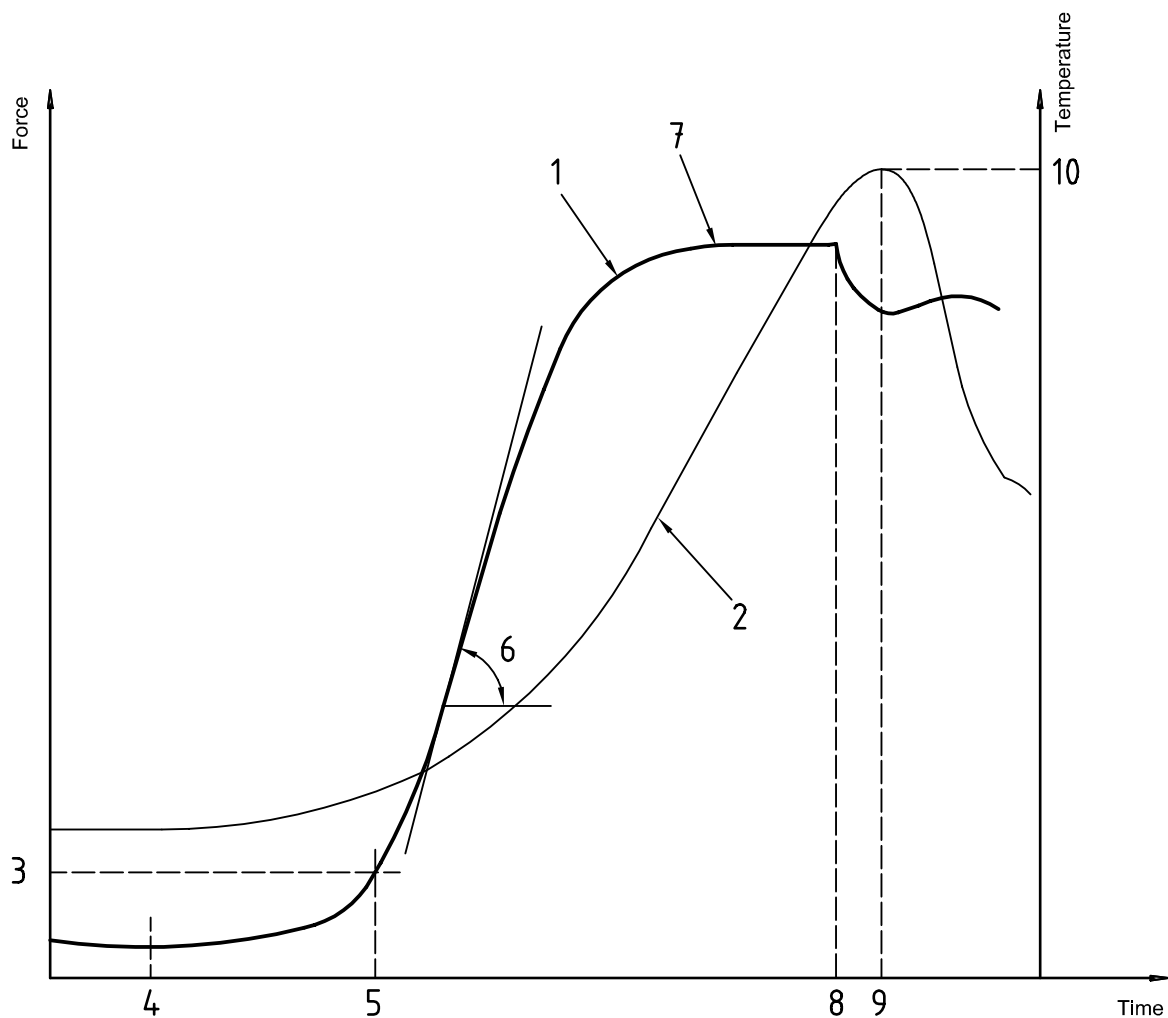
a) Method B1 (resin in test tube)

b) Method B2 (resin in beaker)

Key

- | | | | |
|---|---------------------------|---|---|
| 1 | Glass rod (diameter 6 mm) | 5 | Borosilicate-glass test tube |
| 2 | Thermocouple | 6 | Thermostatically controlled water bath |
| 3 | Resin mixture | 7 | Horizontal oscillatory motion (neither rotational nor pendular) |
| 4 | Polypropylene beaker | | |

Figure B.1 — Schematic drawing of a continuous-measurement apparatus

**Key**

- | | | | |
|---|--|----|---|
| 1 | Force-time plot | 6 | Gradient at point of inflection indicates reactivity of resin |
| 2 | Temperature-time plot | 7 | End of polymerization |
| 3 | Force corresponding to 50 Pa·s (calibration value) | 8 | Time to start of shrinkage |
| 4 | Start of gelation | 9 | Time to maximum heat evolution |
| 5 | Gel time | 10 | Point of maximum heat evolution |

Figure B.2 — Typical force-time and temperature-time plots obtained with methods B1 and B2

