

## Measurement of Surface Shrinkage by Immersion in Boiling Water

## 1 Introduction

This method is intended to distinguish between leathers which shrink considerably when heated in the wet state to temperatures near the boiling point of water and leathers which shrink little, or not at all, under these conditions. It can be used for any type of leather (see 5.1 and 5.2).

## 2 Apparatus

The following apparatus is required:

- (1) A circular, flat bottomed, glass dish of diameter between 75 mm and 100 mm and of volume greater than 350 ml. Two glass rods about 2.5 mm in diameter and 100 mm long, each bent at the middle so that it lies in a plane with an angle of about  $60^\circ$  between its arms. A circular, flat brass plate D of diameter  $65 \pm 5$  mm and of weight  $100 \pm 5$  g.
- (2) A desiccator or other glass vessel which can be evacuated and which is large enough to contain the glass dish. A pump capable of reducing the pressure in the desiccator to a pressure of less than 20 mm of mercury within 120 seconds from the time the pump is switched on. A scale marked in mm and a stop watch or clock for measuring intervals of time.
- (3) A pressure vessel made of aluminium or aluminium alloy in which water can be boiled at a pressure exceeding atmospheric pressure. The lid of the vessel is such that it can be quickly removed or replaced, and a thermometer graduated in degrees centigrade passes through the lid near one side; when the lid is on, the bulb of the thermometer extends to within 20 mm of the bottom of the vessel. A spring loaded, adjustable release valve in the lid can be used to maintain the temperature of water boiling in the vessel at  $102.0 \pm 0.3^\circ \text{C}$  if the water is heated on a gas ring to which the gas flow is also suitably adjusted; the gas ring is such that, when the gas is turned on full, 1000 ml of water in the vessel are heated from  $98^\circ \text{C}$  to  $100^\circ \text{C}$  in less than 60 seconds.
- (4) A specimen holder which prevents the sample from curling up while it is being heated in boiling water. The holder (Fig 1) has a brass plate A of diameter about 100 mm and thickness about 3 mm standing on legs formed by brass screws which raise it about 10 mm. The specimen B (shaded) rests on A and a bent copper wire of diameter about 1 mm rests on the specimen. (For clarity, the wire is drawn thicker than 1 mm in the plan of Fig 1.) On the wire rests another similar brass plate A', which carries a bridge formed by two brass screws and a brass rod. When the holder has been assembled with the specimen in position, a brass nut is screwed on to each of the three vertical screws attached to the lower plate, so that the assembly can be lifted from boiling water by a bent wire hooked under the bridge, but the holes in the upper plate have sufficient clearance on the three screws

to permit the upper plate to slide freely on them. The nuts on these screws are screwed on sufficiently to prevent the upper plate from being lifted off, but leave it free to rise at least 5 mm, so that the load constraining the specimen to remain flat during its heating is merely the weight of the upper plate and bridge. To allow boiling water free access to the specimen, holes of 10 mm diameter are bored as shown in both the upper and lower plates. The weight of the upper plate and bridge is  $250 \pm 20$  g.

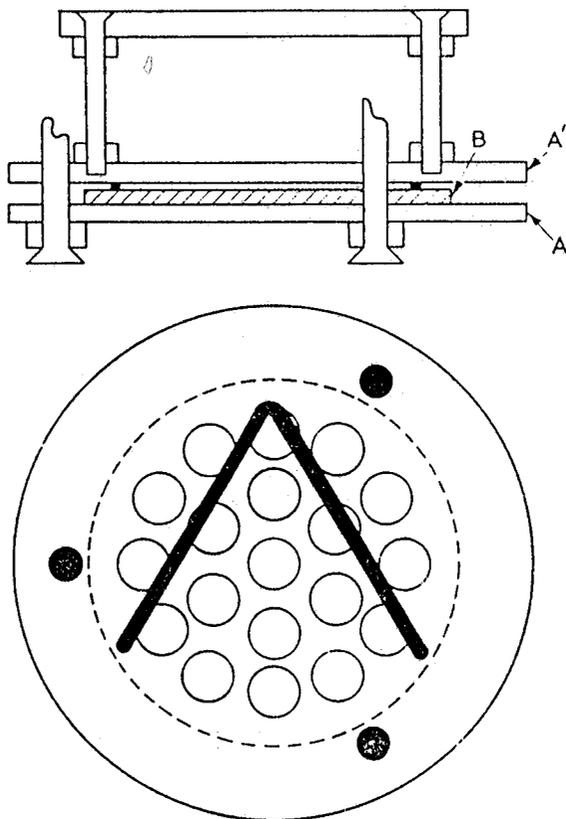


Fig 1 Part elevation and plan of plate A

- (5) Two circular brass plates Q and R of diameter about 100 mm and thickness about 3 mm. The specimen is placed horizontally between these while it is cooling and they prevent it curling during this time. The upper of these plates is loaded with a beaker containing sufficient water to make the total weight of this plate, beaker and water  $250 \pm 20$  g.

### 3 Preparation of the specimen

Cut the specimen with a steel press knife the inner wall of which is a right circular cylinder of 70 mm diameter. Mark on the grain surface two diameters at right angles; mark on the flesh side two diameters which are at right

angles to one another and which lie approximately mid-way between the pair marked on the grain side.

#### 4 Procedure

- 4.1 Place one of the bent glass rods on the bottom of the glass dish, place the specimen on the rod, the second rod on the specimen, and the brass plate D on the rod. Add  $200 \pm 5$  ml of distilled water to the dish, transfer it to the desiccator, and evacuate the desiccator for  $180 \pm 10$  seconds. Allow air to enter the desiccator to restore atmospheric pressure and force water into the specimen (see 5.3 and 5.4).
- 4.2 Put into the pressure vessel  $1000 \pm 20$  ml of distilled water and with the lid on, but not screwed down, heat the water to the boiling point. Then turn the gas down, so that the water continues to boil gently without much escape of steam.
- 4.3 Sixty minutes after beginning evacuation (and 57 minutes after restoring the pressure to atmospheric), remove the specimen from its dish of water and blot its surfaces gently with blotting paper to remove surplus water. Lay the specimen on a flat surface, taking care not to extend it, and measure its four marked diameters to the nearest 0.1 mm.
- 4.4 Turn the gas ring on full; quickly set up the specimen in the specimen holder as described in 2(4); immediately transfer the holder and specimen to the pressure vessel, noting the time when it enters the water; screw down the lid and allow the temperature to rise to  $102.0^\circ\text{C}$ ; by adjusting the flow of gas and the release valve of the pressure vessel, maintain the temperature at  $102.0 \pm 0.3^\circ\text{C}$  with a slow escape of steam through the release valve (see 5.5 and 5.6).
- 4.5 After the specimen has been in the pressure vessel for  $15 \pm 0.1$  minutes, transfer the vessel to a sink where a rapid stream of cold tap water plays on the lid to cool it, and after one or two seconds, depress the lid to allow two or three litres of tap water to enter the vessel; remove the specimen and holder from the vessel and the specimen from the holder; place the specimen between two horizontal brass plates Q and R (see 2(5)), and allow it to cool for five minutes.
- 4.6 After the specimen has cooled for  $5 \pm 0.5$  minutes, remove it, blot its surfaces gently and again measure its four marked diameters as in 4.3.
- 4.7 Calculate the sum  $S_0$  of the four marked diameters before heating and their sum  $S_1$  after heating. Calculate the mean percentage decrease in diameter  $P$  from the formula (see 5.7).

$$P = 100 \left( \frac{S_0 - S_1}{S_0} \right)$$

## 5 Notes

- 5.1 The boil test is a test of the resistance of leathers to wet heat and is not a test of quality in any other respect.
- 5.2 The method is devised chiefly for measurements on tanned leather that has been dried, rather than for tannery control of leather that is still in the wet state. For the control of wet leathers during tannage simpler methods will often suffice.
- 5.3 The object of reducing the pressure and restoring it again is to remove most of the air from the specimen and to force water into it, so that all its fibres become wetted. Mere immersion without pressure changes is insufficient to wet some leathers.
- 5.4 The percentage shrinkages of some leathers in the boil test depend to a marked extent on pH value, and to obtain consistent results, fixed volumes of distilled water must be used for wetting them and to boil them in.
- 5.5 A domestic pressure cooker (modified) is a suitable pressure vessel for the test. Although the volume of water in the pressure vessel is unlikely to be of critical importance, excessive losses as steam must be avoided by following the method described in 4.2 and 4.4.
- 5.6 The temperature of boiling water depends upon atmospheric pressure. To maintain the temperature of the boiling water at 100.0 °C requires more elaborate apparatus, and 102.0 °C is used in the method because this is a temperature (near 100 °C) which is easily maintained.
- 5.7 Specimens which are initially circular and which shrink considerably in the boil test are sometimes far from circular after shrinkage. For such specimens, the formula for  $P$  gives only a rough approximation of the mean percentage linear shrinkage. For such specimens, however, the exact amount of shrinkage is seldom (if ever) of interest. The simple formula given is accurate enough for specimens whose shrinkage is small (less than 5% to 10%) and should be used for all specimens.