# Instrumentation for Thermopenetrometry of Fats

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## ABSTRACT

A new instrument for thermopenetrometry of fats was developed. A linear displacement transducer was modified by removing the spring actuated return mechanism on the moving shaft and attaching a cone to the lower end of the shaft. The cone dimensions were those specified in AOCS method Cc 16-60. A platform was fitted to the upper end of the shaft to accommodate the addition of selected weights. The total weight of the cone, shaft and platform assembly was 100 g. The displacement transducer was connected to a signal conditioner and a recorder. The temperature of the sample was programmed from 5 to 25 C by means of a Haake circulating water bath, and the penetration of the cone into the sample recorded continuously as the temperature was changed. The use of the instrument was demonstrated with butter and margarine, and the precision of the instrument proved to be satisfactory. The coefficient of variability did not exceed 7.0%.

## INTRODUCTION

Penetration measurements are useful for analyzing the consistency of plastic fats. A number of different methods have been described, including needle and cone penetrometers (1). The most commonly used procedure involves the cone penetrometer standardized by the American Oil Chemists' Society (Method Cc 16-60). In the AOCS method, measurements are made at one temperature only, and if data over a temperature range are required, repeated measurements must be performed. Kleinert (2) has drawn attention to thermopenetrometry, a technique whereby penetration data are obtained at increasing sample temperatures. For many plastic fats a temperature range of 5-25 C is of interest, as this range covers both refrigerator and room ambient temperatures. It was advantageous, therefore, to develop a technique which would make it possible to obtain penetration data as a function of temperature.

## EXPERIMENTAL

An instrument was constructed based on a displacement transducer for recording penetration depth in a sample subjected to a programmed temperature cycle. The transducer used was a Daytronic 102 B-120 linear displacement transducer modified by the removal of the spring actuated return mechanism on the moving shaft to eliminate this variable force from the measurement. A cone with dimensions specified in the AOCS method Cc 16-60 was fitted to the lower end of the shaft and a platform to hold additional weights fitted to the upper end. The weight of the cone, shaft and platform assembly, i.e. the minimum weight applied on the sample, was adjusted to 100 g by machining the components. The assembly was mounted on a stand (Fig. 1) which provided height adjustment and a mechanism to release the shaft at the start of a test.

The transducer was connected to a Daytronic 300D signal conditioner containing a model 70 differential transformer input module. The signal was recorded on a 10 mV strip chart recorder. The sample was contained in a brass sample cup (diameter 25 mm, height 25 mm), which in turn was placed in a brass circulating water bath controlled by a Haake F3 programmed thermostat.

Fat samples were placed in the sample cups using a stainless steel cylindrical core sampler. After insertion of the sample into the cup, the surface of the fat was levelled with a spatula. The sample cup was then placed into the circulating bath and equilibrated at the starting temperature of 4 C. Temperature of the sample was measured by inserting the probe of a digital thermometer into the sample.

The displacement transducer was calibrated for a full scale displacement of 25 mm using accurately machined metal blocks (± 0.002 mm) that were placed under the the cone. At the start of the measurement, the cone was positioned to just touch the sample surface the then released. The temperature was increased at a rate of about 0.5 C/min and the cone penetration recorded continuously.

Fats used in this study were butter and margarine obtained from local outlets and stored at 4 C.

## RESULTS AND DISCUSSION

Results of the calibration showed that the relationship between cone displacement and recorder readings was linear within ± 0.75 mm over the range 5-25 mm. Displacement readings were repeatable within ± 0.01 mm.

The raw penetration-temperature curves obtained with butter and margarine using a total weight on the sample of 200 g (i.e. 100 g shaft assembly + 100 g added on the platform) are presented in Fig. 2(A and B). These curves indicate, in addition to the penetration depth at each temperature, those temperatures at which the rate of softening increases. This is a complex relationship since the cone is tapered so that the penetration is a combined effect of depth of penetration and properties of the fat which are changing with temperature. The curves in Fig. 3(A and B) represent the same data that have been converted to a hardness index based on an interpretation technique previously described (3).

In this type of measurement, the cone rests on the sample continuously, in contrast with the AOCS method where the cone penetrates for 5 sec only. It was expected that the results would not be identical. A comparison was made of the results obtained with the thermopenetrometer method and the official AOCS method using a Precision penetrometer. Penetrometer values were converted to hardness by using the equation $H = M/P$, where: H = hardness, M = mass of cone assembly in g, P = depth of penetration in mm (3). Results are presented in Table I. Statistical evaluation of these results indicates that there was a significant ($P < 0.05$) difference at every temperature between the results obtained by the two methods for both products. At 5 and 10 C, the thermopenetrometer results for butter indicated lower hardness. At 15 and 20 C, the reverse was the case. With margarine the thermopenetrometer gave a hardness index which was lower only at 5 C. The difference in test methods, therefore, will cause small but significant differences in the results obtained by the two methods.

Thermopenetrometer results provide useful information on the effect of temperature on the physical properties of fats. Fig. 3 illustrates how hardness of the butter and margarine samples differed in their response to temperature change. The butter (A) was much harder than the margarine (B) at 5 C but decreased more rapidly in hardness with increasing temperature.

The precision of the thermopenetrometer test is evidenced from the results of six replicate determinations on butter and margarine (Table II). The coefficient of variability did not exceed 7.0%.

The penetrometer described in this paper provides a convenient and precise method for determining hardness of fats as a function of temperature.

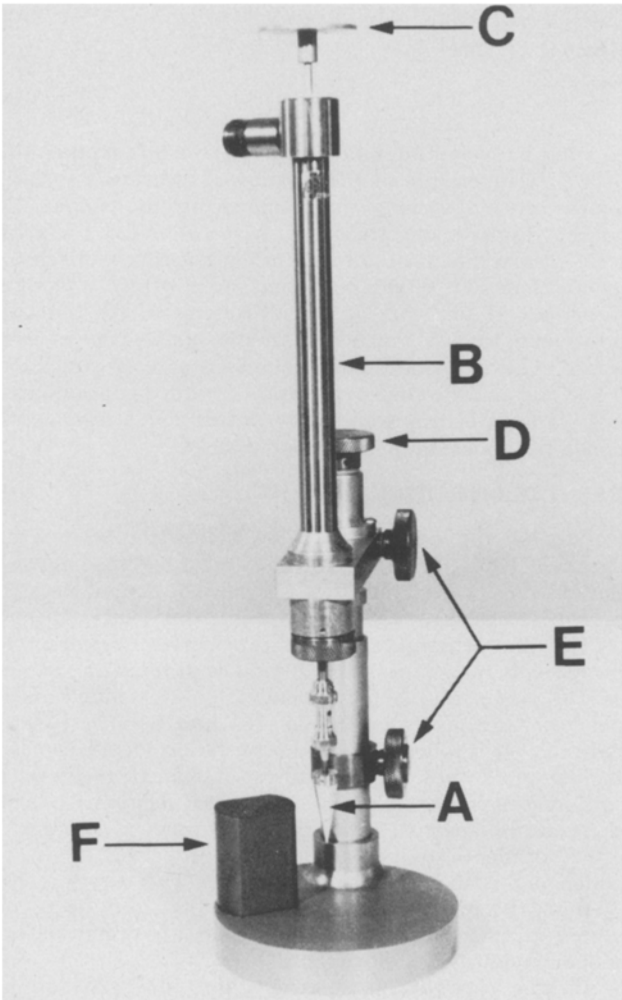


FIG. 1. Instrument for continuously monitoring depth of penetration in a sample increasing in temperature. A, penetrometer cone; B, displacement transducer; C, weight platform; D, fine height adjustment; E, coarse height adjustments, and F, calibration block.

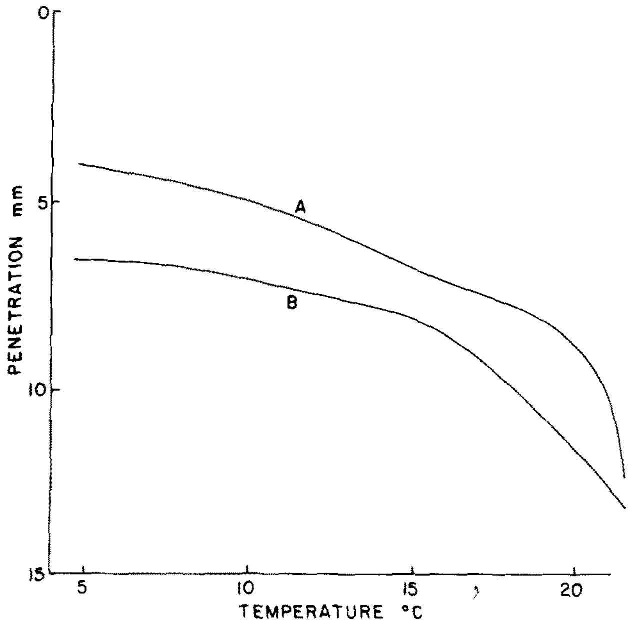


FIG. 2. Penetration-temperature recordings of butter (A) and margarine (B).

TABLE I

**Comparison of Hardness Values Determined by Thermopenetrometry and AOCS Method Cc 16-60**[1]

| | | Hardness (g/mm) at temp. | | | |
|---|---|---|---|---|---|
| Sample | Method | 5 C | 10 C | 15 C | 20 C |
| Butter | AOCS | 51.80 | 39.36 | 22.35 | 14.01 |
| | Thermo | 42.90 | 34.90 | 24.00 | 17.30 |
| Margarine | AOCS | 32.64 | 22.65 | 13.82 | 5.14 |
| | Thermo | 27.58 | 24.24 | 19.00 | 12.41 |

[1]Results are based on 6 reps – SD's and CV's are given in Table II.

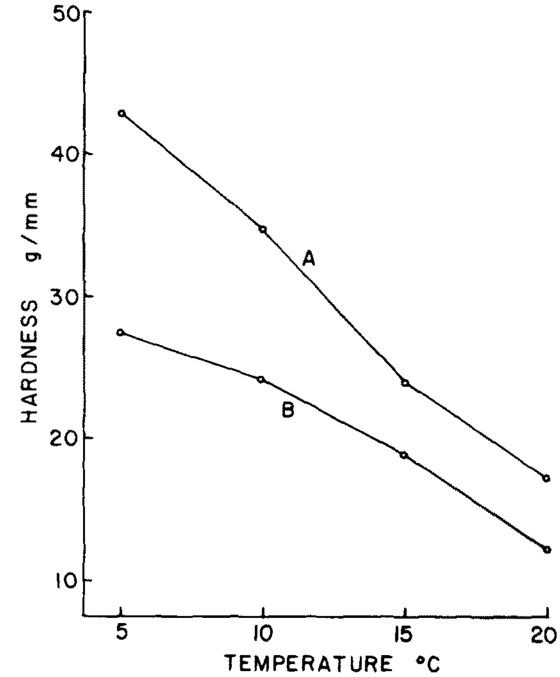


FIG. 3. Changes in hardness of butter (A) and margarine (B) with temperature.

TABLE II

**Precision of Thermopenetrometer Measurements on Butter and Margarine (6 Replicates of Each Sample)**

| Sample | Temp. C | Standard deviation | Coef. of variability % |
|---|---|---|---|
| Butter | 5 | 1.47 | 3.49 |
| | 10 | 2.37 | 7.00 |
| | 15 | 0.56 | 2.32 |
| | 20 | 1.02 | 6.24 |
| Margarine | 5 | 1.47 | 5.11 |
| | 10 | 1.68 | 6.50 |
| | 15 | 1.08 | 5.47 |
| | 20 | 0.53 | 4.36 |

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