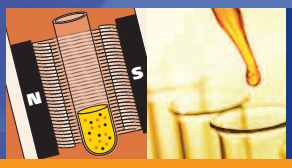
Application Note

Determination of Total Fat Content in Chocolate and other Cocoa Derivatives



NMR

Cocoa beans are processed to extract cocoa liquor or cocoa butter, a major ingredient of dark and milk chocolate. Cocoa beans are roasted, separated from their shell and cracked into nib. Nib is then ground to produce cocoa liquor or cocoa butter plus cocoa powder. As the quality of the beans can vary depending on the environmental conditions in the region where they were grown, it is important to quantify the fat content of the raw and intermediate materials to ensure consistency of the final product.

Method

The reference technique for measuring total fat content is traditional acid hydrolysis followed by soxhlet extraction in petroleum ether (Weibull-Stoldt method). This technique has the following shortcomings:

- The sample must be dried first; only a limited amount of representative sample (4-5g) is actually measured
- Sample drying, acid hydrolysis and solvent extraction are time-consuming, cumbersome procedures requiring expert laboratory skills. The many phases in the extraction process are prone to operator error, resulting in inter-laboratory variations
- The technique is destructive to the sample. As a result, the same sample cannot be measured more than once
- Soxhlet extraction uses harmful, flammable solvents with health and environmental hazards

In comparison, Nuclear Magnetic Resonance (NMR) offers an attractive alternative for the following reasons:

- The measurement is very rapid (less than 10 seconds) and the total experimental time to result is about 15-20 minutes (including sample conditioning at 50°C)
- It requires minimum sample preparation: ~8g of sample is placed inside a 23mm diameter vial
- It is environmentally friendly; it does not use any solvents and is non-hazardous for the operator
- The method calibration is very simple; it can be achieved by using a single sample of cocoa butter
- The technique is non-destructive; measurements can be repeated several times on the same sample
- The measurement precision is 0.1% fat or better

Calibration and Results

Calibrations were obtained by measuring various samples of dark and milk chocolate (Figure 1) and chocolate crumb (Figure 2), a mixture of cocoa, milk powder and sugar, and plotting the NMR data against the oil contents obtained by the International Office of Cocoa Chocolate and sugar Confectionery

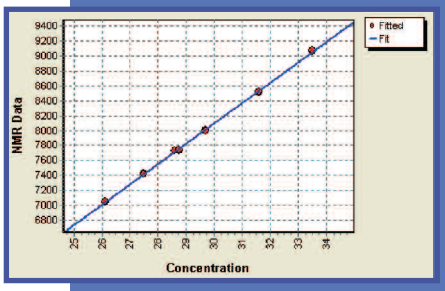


Figure 1. Correlation curve of Total Fat Content of Chocolate by NMR against soxhlet extraction (% total fat) using the results summarised in Table 1.

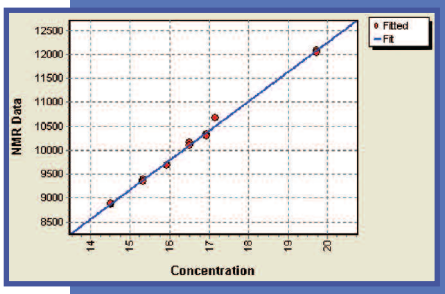
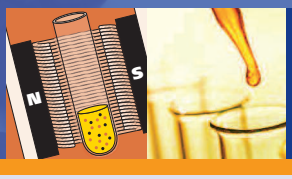


Figure 2. Correlation curve of Total Fat Content of Chocolate Crumb by NMR against soxhlet extraction (% total fat) using the results summarised in Table 2.

Benchtop NMR for Food & Agriculture

OXFORD INSTRUMENTS



NMR

(IOCCC) reference method (soxhlet extraction). The results for both chocolate and crumb show excellent correlations with R^2 values of 0.999 and 0.998 respectively. Reference values are compared against those calculated from the NMR calibration in Tables 1 and 2. The standard deviation over several measurements of the same sample is typically less than 0.1%. The standard error of estimate for these sets of samples provides an accuracy figure of 0.3% fat, although the accuracy can only be as good as the results of the reference technique against which it is being compared.

Table 1. A comparison of the oil contents of various samples of dark and milk chocolate obtained from the reference technique against those calculated from the NMR calibration.

Sample	Ref Value	NMR Calc.*	Diff.
1	29.7	29.7	0.0
2	28.6	28.7	0.1
3	33.5	33.6	-0.1
4	27.5	27.5	0.0
5	26.1	26.1	0.0
6	31.6	31.5	-0.1
7	28.8	28.6	-0.2

*Average of two subsamples

Table 2. A comparison of the oil contents of various samples of chocolate crumb obtained from the reference technique against those calculated from the NMR calibration.

Sample	Ref Value	NMR Calc.*	Diff.
1	14.5	14.5	0.0
2	15.3	15.3	0.0
3	15.9	15.8	-0.1
4	16.9	16.9	0.0
5	16.5	16.6	0.1
6	17.2	17.4	0.2
7	19.7	19.7	0.1

*Average of two subsamples

Recommended Instrument

The **MQC**-23 fitted with a 26mm diameter (21ml) probe is a suitable instrument for this application. The Fat in Chocolate package comprises:

- **MQC**-23 with a built-in computer operating the latest version of Microsoft® Windows® (no separate PC is required)
- **MultiQuant** software including **RI Calibration, RI Analysis,** and the **EasyCal** 'Fat in Chocolate' application
- Three setting up standards (SUSs) at 10, 25 and 40% oil content for calibration maintenance and quality control
- 26mm diameter sample vials
- PTFE sample holder
- PTFE sample packing tool
- Installation manual
- Method sheet

In addition to this package you will also require:

- A dry heater and aluminium block with holes for sample conditioning at 50°C
- A precision balance

The instrument offers multiple advantages over other instruments on the market:

- High signal sensitivity
- Small benchtop footprint
- Low maintenance
- Minimal sample preparation
- Low cost consumables
- A primary calibration can be produced using a single sample of 100% cocoa butter

References: 1. R.S. Kirk, R. Sawyer *Pearson's composition and analysis of foods,* Ch. 13, 469-529 (1991), 9th Ed., Longman Scientific & Technical, Harlow

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