2nd Edition

Laboratory Handbook of Methods of Food Analysis

R. Lees MRSH AIFST

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R. Lees
M.R.S.H., A.I.F.S.T.

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Preface to the Second Edition

In this second edition of *Laboratory Handbook of Methods of Food Analysis* I have taken the opportunity to include a number of additional methods of food analysis which have been requested by factory control chemists. All the methods given in the first edition have been reviewed and, where appropriate, revised in accord with modern control laboratory practice. An extensive index has been prepared which will supplement the commodity cross index appearing in Section II of the book.

The aims of this book have not changed. They are: (1) to bring together in one book those methods of analysis which are most useful to the food factory control analyst, (2) to present these methods in a readable and readily understandable form, (3) to indicate, where appropriate, such information which will aid the task of the analyst.

The book was not prepared to lie on a library shelf but as a working aid for use at the laboratory bench. The revisions that have been made to the first edition have been carried out to further this aim.

May, 1971 R. Lees

Introduction

THIS book has been prepared with the working chemist in mind. The methods detailed in Section III have been chosen because they give good repeatable results and are suitable for use in a factory laboratory. Some of these methods may not be completely applicable for research establishments whose needs differ in certain fundamental aspects to those of a busy control laboratory. Within certain limits, time is not of prime importance to a research organization where the major requirement is absolute accuracy. The methods described in the text give results which can be repeated in duplicate and will agree with those found by other analysts. It matters little that a result is 0·1 per cent off absolute if the standards for that particular line has been based on the chosen method. For the majority of methods described, the research and control procedure is precisely the same as that given. Only when extensive analytical work is involved has a simpler method been substituted.

Equally it has not been the intention to fill the book with long descriptions of the theory behind an analytical technique. This type of material rightly belongs to a text book on that particular aspect of science. This book is entitled *A Handbook of Laboratory Methods of Food Analysis* and it has been written within this framework. The introductory chapters have been prepared as a practical discussion of problems likely to be encountered during factory control analysis. These chapters include material which would have become repetitive if included under each method heading.

Section II has been compiled to provide an extensive cross-reference to the analytical methods appropriate to the particular food commodities. Most of the published books on food analysis are written in chapters which consider each type of product in turn. This method gives rise to considerable repetition because certain analytical

techniques are basic and can be used for many different types of foodstuffs. Section II is an attempt to overcome this unwanted duplication. This system has considerable advantages in that it permits the methods section to be listed alphabetically, makes for easier reference, and enables comparison to be made between different approaches to the analysis of food products. Finally I have attempted to avoid the use of specialized terminology and to use an economy of words in order to achieve a readability suitable for the busy working chemist.

R. LEES

Contents

Fig

List of Illustrations

List of Tables

SECTION I

Notes on General Laboratory Methods used in Food Analysis

Sampling 1

To avoid errors arising which are unrelated to the efficiency and accuracy of the analyst, it is essential to try to ensure that adequate sampling procedures are used. This is often beyond the control of the chemist but the procedure can be applied once the bulk sample has been received in the laboratory.

Powdery or Granular Materials should be sampled by the technique of quartering which is carried out as follows:

Tip the powder or granules out on to a large paper sheet and mix with a spatula. Draw a cross over the heaped pile of material. Remove two diagonally opposite segments and return them to the package. Remix with the spatula and again draw a cross over the heap of powder. Remove the two opposite segments and return them to the original package. Continue this procedure until a sample of about 250 grams remains. Transfer to a sample jar and tightly stopper. Grind the granules where necessary before transferring to the sample jar.

Meat and Meat Products should be sampled by separating the meat from the bone, from the skin or from the crust. The meat or meat mixture should then be run through a meat chopper to reduce to a fine mince. Transfer to a sample jar and store at a low temperature.

Semi-Solid and Mixed Liquid/Solid Phase Materials such as cheese and chocolate should be coarsely grated. The grated materials should then be sampled by the technique of quartering as described for powders.

Semi-Viscous Pastes such as milk pudding and *Liquids Containing Solids* such as fruit squashes should be rendered thoroughly uniform in a high-speed blender. The sample should then be immediately bottled.

Sample Containers

Glass or polythene jars should be used to hold the food samples. These containers should be thoroughly cleaned before use. Particular attention should be paid to the lid of the container. Water can be held by the ridges in the lid and give rise to erroneous results during analysis. Polythene containers suffer the disadvantage that they are difficult to label.

Labelling and Work Sheets

Once the sample has been taken it should be immediately labelled and recorded. The following is the minimum amount of information which is needed to identify the sample:

consecutive sample number
sample type
batch or product number
date and time sampled
date received in the laboratory
date analysed
analyst
supplier of raw material
special features (if any).

Brief details of all samples received should be recorded consecutively on their receipt in the laboratory. This job can be carried out by the most junior member of the staff or by clerical or secretarial help where this is provided. The information on the label should be repeated on the work sheet or in the work book. Analysts should as a matter of course record all sample details, weighings, calculations and conclusions in a permanent form for future reference. The trend is to use stiff-backed books for ease and cheapness, but a major difficulty is that they are awkward to index, cumbersome to store and it is necessary to retain or throw away all the analyses. No selectivity is possible, neither can a rapid search be made over previous results to check a particular supplier or the repeatability of an analytical technique. The use of printed or duplicated work sheets therefore has considerable advantages which outweigh their additional costs. An example of a completed work sheet for a sample of suet is shown in Fig. 1a and b.

Accuracy

Despite intensive training, it is a sad fact that many chemists are poor mathematicians. For this reason it is advisable to have all calculations checked by a fellow worker to prevent stupid errors from occurring and tarnishing the reputation of the laboratory. The initial strangeness from

introducing this procedure soon disappears and the early antagonism from the staff is forgotten. The system has a secondary advantage in that it induces neatness, always a virtue when carrying out scientific work. Using work sheets has a considerable advantage again over recording in a work book as it causes less disruption in working.

REPORT SHEET

Sample number	7/162	Sample	suet
Date received	18/4/71	Batch/product number	18/6/B
Date analysed	19/4/71	Raw material supplier	—
Date completed	21/4/71	Special features	None
Analyst	J.P.V.		
Calculation checked by	Y.M.L.		

Moisture, per cent w./w.	0·8
Fat, per cent w./w.	83·1
Added filler, per cent w./w.	16·1

Action to be taken	Filler rather on high side. Suggest further checks to be made on this line.

FIG. 1*a*—Front of a Completed Laboratory Sheet

All determinations should be carried out in duplicate. No matter how much care is taken, unfortunate errors do occasionally occur during analysis. A sudden jolt to the dessicator and part of the ash is lost, a faulty recording is made of the weights on the pan and so on. These accidents, while the cause is not known, become apparent if determinations are carried out in duplicate. When single determinations are carried out and the summation of analysis does not equal 100·0 the young analyst may be tempted to use the infamous 'Cooks Factor'; a factor which is not considered either in this book or by any reputable worker. When incorrect totals are obtained they are either inherent as a fault of the method, the interpretation of the analyst, or the presence of another component.

Considerable wastage in effort occurs when analysts try to get results agreeing to the second decimal place. For all analysis of major components, the results are only

required to the first decimal place in control laboratories. Closer results are usually meaningless when translated back into batch weights. The inability to accept this fact leads to further time wastage in weighing to degrees of accuracy which are of no significance in calculating the results.

In carrying out an acidity determination on boiled sweets it is sufficient to know that 20·2 grams of sweets were taken and when dissolved required a titre of 10·2 ml. 0·1 normal sodium hydroxide for neutralization. Any closer definition of the weight of sample and of the titre would neither improve the result, nor increase its significance industrially. In this context it is interesting to consider the work sheet reproduced in Fig. 1b (p. 7). Here too the analyst need not have weighed to the third decimal place in view of the type of report that was presented.

A further source of time wastage arises from the continual attempt by analysts to weigh the quantity stated in the working method. Thus if the method specifies 5 grams, the analyst laboriously weighs 5·000 grams in order to simplify the calculation. This action can add as much as five minutes to weighing time. Considerably less time is taken to divide, for example, 5·122, on a slide rule or by using logarithm tables to achieve the same result.

If the food sample is completely water soluble, further time can be saved by preparing a master solution (20 per cent w./v.) and carrying out subsequent determinations on aliquots taken by pipette.

Abbreviations Useful When Writing Report Sheets

The number of abbreviations used in this book have been kept to the minimum and are those which can reasonably be used in practice. These are:

gm. —gram(s)
ml. —millilitre
cm —centimetre
mm. —millimetre
per cent w./v. —weight of component dissolved in 100·0 ml. of solution
per cent v./v. —volume of component in 100·0 ml. of solution
per cent w./w.—weight of component in 100·0 g. of sample

A further useful piece of scientific shorthand used in text is that which indicates the dilution of a solution. The method of writing this is to indicate first the volume that has been taken and then, after a stroke, the final diluted

volume. Thus 25/250 would indicate that 25 ml. of solution had been diluted to 250 ml.

Moisture

Dish + sample	37·755 g.	Dish + sample	41.182 g.
Dish 14	32·647 g.	Dish 8	36·154 g.
Sample	5·108 g.	Sample	5·028 g.

After drying

Dish + sample	37·725 g.	Dish + sample	41·152 g.
	37·717 g.		41·144 g.
	37·715 g.		41·141 g.
Dish + sample (original weighing)	37·755 g.	Dish + sample (original weighing)	41·182 g.
Weight loss	0·040 g.	Weight loss	0·039 g.

$$\% \text{ moisture} = \frac{0{\cdot}040}{5{\cdot}108} \times 100 = 0{\cdot}8 \qquad \% \text{ moisture} = \frac{0{\cdot}039}{5{\cdot}028} \times 100 = 0{\cdot}8$$

Average moisture 0·8

Added Filler

Bottle + filter paper + suet	28·702 g.	Bottle + filter paper + suet	27·950 g.
Weighing bottle + filter paper	18·684 g.	Weighing bottle + filter paper	17·870 g.
Suet	10·018 g.	Suet	10·080 g.

After extraction

Bottle + filter paper + filler	20·292 g.	Bottle + filter paper + filler	19·482 g.
Bottle + filter paper	18·684 g.	Bottle + filter paper	17·870 g.
Filler	1·608 g.	Filler	1·612 g.

$$\% \text{ filler} = \frac{1{\cdot}608}{10{\cdot}018} \times 100 \qquad \% \text{ filler} = \frac{1{\cdot}612}{10{\cdot}080} \times 100$$

Average filler % = 16·1

Fat

$$\% \text{ Fat} = 100{\cdot}0 - (16{\cdot}1 + 0{\cdot}8) = 83{\cdot}1$$

FIG. 1*b*—Reverse of a completed Laboratory Sheet