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Serine as an Index of Orange Content of Soft Drinks

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The natural content of serine in oranges is examined as a means of estimating the quantity of whole fruit or juice in orange preparations. The method gives results which are very close to those obtained by established methods.

Difficulty is experienced in obtaining a reasonably accurate assessment of the juice or fruit content of orange beverages due to the wide natural variation in constituents such as P, K, N etc., which are commonly used as analytical factors. Furthermore these constituents exist in different proportions in the peel, pith and juice of the fruit thus adding a further complication in the case of comminuted beverages. Still further complication is introduced by the presence of legitimate additives containing some of these elements, such as potassium preservative or phosphate sequestrant in beverages.

It is clear that the determination of a single above-mentioned constituent will not necessarily give a reasonably accurate assessment of juice or fruit content and experience has shown that it is necessary to determine a number of constituents and to consider these in relation to published average data in order to obtain a more reliable result.

Examination of scientific literature dealing with the constituents of orange disclosed the fact that serine is present in the juice, vesicles, carpellary membrane, albedo and flavedo in relatively high proportion to other free amino acids[1,2,3,4,5], and in the mature fruit it is present in more or less similar proportion in the juice, pith and peel. The latter observation appeared worthy of further investigation, particularly as comminuted fruit used in industry is so variable in the relative proportion of juice, pith and peel.

Amino acids are extractable from the tissues of component parts of the whole orange by hot aqueous 80 per cent. solution of alcohol[1], and can be absorbed on a cation exchange resin[6] and eluted with a suitable solvent. Filtered juices can be passed directly through the column. Of the known

amino acids in orange juice and tissues only serine, with a terminal ($-CH_2OH$) group, reacts with periodic acid to yield formaldehyde which can be estimated by a suitable method[7].

Experimental

REAGENTS

1. *Sodium sulphite:* 1 per cent. w/v aqueous solution.
2. *Chromotropic acid:* 0·4 g of the sodium salt dissolved in 4 ml water and 96 ml of 13 M sulphuric acid added (*freshly prepared*).
3. *Periodic acid:* 0·23 g of potassium periodate in 100 ml 0·15 M sulphuric acid.
4. *Stannous Chloride:* 6·0 g of stannous chloride in 100 ml of dilute hydrochloric acid (1 : 9 v/v).
5. *Alcohol:* 80 per cent. w/w of the *British Pharmacopoeia*, 1963.
6. *Phosphate buffer:* pH 8 (approx.) of the *British Pharmacopoeia*, 1963.
7. *Phenol red indicator:* of the *British Pharmacopoeia*, 1963.
8. *Ammonium hydroxide:* Normal solution.
9. *Sulphuric acid:* 13 M, 9 M and 0·15 M solutions.
10. *Formaldehyde:* Standardised solution of Analytical Reagent quality.

COLUMN PREPARATION

A 50-ml burette plugged with cotton wool for about half an inch above the tap is suitable for supporting the column, which should consist of B.D.H. Amberlite Resin 1R-120 (H) analytical grade to an approximate depth of 22 ml, covered with N HCl. Wash free from hydrochloric acid using about 100 ml of water.

NB: After use, the column can be re-generated by washing it free from ammonia and pouring through it, 100 ml of N HCl.

Method

Accurately weigh (or measure) a suitable quantity of product expected to yield about 1 g of juice or of any or all component parts of a whole orange.

For convenience the following quantities of sample are recommended:—

Juices (straight and concentrated)—dilute to one-quarter natural strength and accurately weigh about 4 g.

Comminuted orange bases —accurately weigh about 5 g, dilute to 50 ml and take 10 ml.

Orange drink, for dilution —10 ml.

* This buffer has pH ~ 6·5.

Orange drink, ready to drink —50 ml.
Orange squash —dilute five-fold and take 20 ml.
Orange crush —20 ml.

Filter the sample through a 9-cm, No. 4 Whatman paper (using additional water if necessary) and wash the residue and paper with three successive portions each of 10 ml of hot 80 per cent. alcohol.

Evaporate the combined filtrate to about 20 ml, cool, and pass through the prepared cationic exchange column, rejecting the effluent. Wash the column with distilled water until free from carbohydrates (check with Tollens reagent).

Elute the column with a normal solution of ammonia, collecting about 150 ml at a flow rate of one drop per second, and evaporate the solution on a water bath to a volume of 3 to 5 ml.

* Add 3 ml of periodate reagent and sufficient phosphate buffer to render the liquid neutral to phenol-red. Allow the reaction to proceed for not less than 45 minutes.

Add 2 ml of stannous chloride solution and transfer the mixture to a 50-ml distillation flask with sufficient aqueous rinsings to yield a volume of about 15 ml.

Distil approximately 10 ml into a 25-ml volumetric flask containing 2 ml of sulphite solution. Add 10 ml of distilled water to the residue in the distillation flask and distil a further 10 ml into the same volumetric flask. Make up to mark with distilled water, and mix well.

SPECTROPHOTOMETRIC DETERMINATION

Transfer 1 ml of distillate, together with 5 ml of distilled water, to a test tube graduated at 20 ml, and add 10 ml of chromotropic acid reagent. Run a blank simultaneously.

Place the tubes in a boiling water bath for exactly 30 minutes, cool, and make up to volume with 9 M H_2SO_4. Measure the extinction in a 4-cm cell at 565 mμ.

CALIBRATION OF THE INSTRUMENT

Prepare standards containing 0, 2 and 5 μg of formaldehyde respectively and treat each as described above, under SPECTROPHOTOMETRIC DETERMINATION.

1 μg of formaldehyde $\equiv$ 3·496 μg of serine.

Analytical Results

Samples from reliable sources were examined and compared with samples comminuted or expressed in the laboratory. Results are shown in the accompanying Tables.

* Add 3 ml of periodate reagent + sufficient N/2 NaOH to render the liquid neutral to phenol red. Then add 3 ml phosphate buffer solution (pH 8). Allow the reaction .....

TABLE I

THE SERINE CONTENT OF COMPONENT PARTS OF FRESH ORANGES

Country of Origin	*Microgrammes per gramme*				
	Flavedo	Albedo	Entire peel	Filtered juice	Pulp
Australia	203	192	—	195	207
South Africa	223	192	—	227	254
Spain	192	218	—	196	194
"	—	—	209	183	204
"	—	—	205	176	200
"	—	—	206	186	201
"	—	—	216	194	200
Israel	183	221	—	263	201
" (small)	—	—	216	169	192
" "	—	—	192	181	200
" "	—	—	180	192	196
" "	—	—	198	192	200
" (large)	—	—	215	207	196
" "	—	—	216	231	201
" "	—	—	212	221	200
" "	—	—	212	218	196
Minimum	183	192	180	169	192
Maximum	223	221	216	263	254
Average	200	206	206	202	203
Number of samples	4	4	12	16	16

TABLE II

SERINE CONTENT OF LABORATORY COMMINUTED WHOLE ORANGES

Country of Origin	*Microgrammes per gramme*
Cyprus	180
South Africa	229
" "	240
Spain	162
"	199
"	198
"	203
"	210
"	205
Israel	225
" (small)	198
" "	194
" "	201
" "	191
" (large)	225
" "	230
" "	235
" "	210

	Microgrammes per gramme
Minimum	162
Maximum	240
Average	208
Number of samples ..	18

TABLE III

SERINE CONTENT OF COMMERCIALLY COMMINUTED ORANGES

(These products contained 97–102 per cent. of orange when calculated from orthodox determinations of ash, alkalinity, potassium, phosphate, etc.)

Country of Origin	*Microgrammes per gramme*
Israel	196
"	196
"	189
Spain	182
"	166
South Africa	210
" "	216
Unknown	182
"	195
"	182
"	195

	Microgrammes per gramme
Minimum	166
Maximum	216
Average	192
Number of Samples	11

TABLE IV

THE SERINE CONTENT OF COMMERCIAL, CONCENTRATED ORANGE JUICES

Spanish Orange Juices		Israeli Orange Juices	
Degree of concentration	Serine content (after dilution) *μg per g*	Degree of concentration	Serine content (after dilution) *μg per g*
4 × 1	166*	4 × 1	198*
4 × 1	164	4 × 1	186*
4 × 1	184	4 × 1	182*
6 × 1	148*	4 × 1	201
6 × 1	166*	4 × 1	196*
6 × 1	166*	6 × 1	186*
6 × 1	186*	6 × 1	190*
6 × 1	173*	6 × 1	186*
6 × 1	162*	6 × 1	185*
6 × 1	166*	6 × 1	203*
6 × 1	166*	6 × 1	192*
6 × 1	167*	6 × 1	203*
6 × 1	177*	6 × 1	194*
6 × 1	180*	6 × 1	209*
6 × 1	186*	6 × 1	188*
6 × 1	172*	6 × 1	176*
6 × 1	192*	6 × 1	202*
6 × 1	168	6 × 1	182
6 × 1	170	6 × 1	188
6 × 1	152	6 × 1	176
6 × 1	170		
6 × 1	158		
6 × 1	166		

* Confirmed by analysis for mineral constituents.

Serine was determined on some commercial samples of orange juice. All samples were of the concentrated type, the strengths being from 4 to 6 times that of normal orange juice. The reputed concentration of all samples was checked by determination of the refractive index. Results marked with an asterisk in Tables IV and IVa were confirmed by orthodox analyses for mineral content, basing the calculation of the fruit juice present on the following factors as average for the natural juice:

Potash (K_2O)	0·2 per cent.
Phosphate (P_2O_5)	0·036 per cent.

The figures for the serine content of the samples have been calculated for the purposes of comparison, on the juice when diluted to normal strength. Table V compares the corresponding maximum, minimum and average figures for serine content.

TABLE IVa

THE SERINE CONTENT OF COMMERCIAL, CONCENTRATED ORANGE JUICES

Country of origin	Degree of concentration	Serine content (after dilution) *μg per g*
Sicily	6 × 1	166*
"	5½ × 1	166*
Greece	6 × 1	203*
Unknown	6 × 1	177*
British Honduras	6 × 1	233*
" "	6 × 1	212*
" "	6 × 1	232*
" "	6 × 1	220*

* Confirmed by analysis for mineral constituents.

TABLE V

COMPARATIVE RESULTS FOR THE SERINE CONTENT OF ORANGE JUICES

Country of origin		Serine content (*microgrammes per gramme*) Laboratory prepared	Commercial samples
Australia	minimum	—	—
	maximum	—	—
	average	195	—
Spain	minimum	176	148
	maximum	196	192
	average	187	171
Israel	minimum	169	176
	maximum	263	209
	average	208	191
South Africa	minimum	—	—
	maximum	—	—
	average	227	—
Various	minimum	—	166
	maximum	—	232
	average	—	200
Average of determinations		202	184

TABLE VI

THE SERINE CONTENT OF ORANGE PREPARATIONS—SUMMARY

Sample	Number of samples	Serine content *microgrammes per gramme* minimum	maximum	average
Juice .. .. laboratory expressed	16	169	263	202
Juice .. .. commercially prepared	51	148	232	184
Entire fruit .. .. laboratory expressed	18	162	240	208
Comminuted .. .. commercially prepared	11	166	216	192
All preparations	96	148	263	192

The overall average figure (192) has been applied to analyses of soft drinks and the orange values compared with those obtained by orthodox mineral assay methods.

TABLE VII

ESTIMATION OF FRUIT CONTENT OF SOFT DRINKS BY SERINE CONTENT

Class of Drink	Serine content *μg per ml*	Estimated fruit content *per cent. w/v*	Estimated fruit content determined by mineral analysis *per cent. w/v*
For consumption without dilution:			
Orange drink (A)	7·8	4·1	4·0
" " (B)	8·2	4·3	4·8
" " (C)	8·3	4·3	4·8
For consumption after dilution:			
Orange drink (Q)	21·0	11·0	10·9
" " (R)	22·4	11·7	12·0
" " (S)	23·0	12·0	12·3
" " (T)	27·0	14·1	14·5
" " (U)	21·0	11·0	10·6

Dehydrated Orange Products

	Serine content *μg per g*	Orange content *per cent. w/w*	Mineral assay orange content *per cent. w/w*
Orange Juice Drink	148	74	65
Orange Juice Powder	868	445	425
Sweetened Orange Juice Powder ..	764	354	351

Note: The accepted specific gravity of orange juice is 1·05, and to obtain the percentage of orange juice in orange drinks (volume in volume), the percentage weight in volume found must be divided by this figure.

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Analytical Chemists in India

Notice was given in this Journal (*J. Assoc. Publ. Analysts*, 1965, **3** (2), 72), of the formation in India of a society of Public Analysts of India.

It has been notified that the name of this society has been amended (to avoid confusion with the former name of the Society for Analytical Chemistry) to:

The Society of Public Analysts and Other Analytical Chemists of India

The declared objects of the Society are:—

"To assist in upholding and maintaining the character and position of Public Analysts and other Analysts and of the Profession of Analytical Chemistry and to protect and advance the interests of Public Analysts and other Analysts and to promote co-operation between them. To take into consideration questions affecting the said profession, and to take any steps that may be considered advisable for advancing or protecting its interests, including the presentation of petitions to Parliament, or to other Public Bodies, and the promotion of the opposition to, or support of Bills in Parliament. To endeavour to obtain advantage for Public Analysts and other Analysts and to procure the exclusion from the Association of any persons guilty of such conduct as may be deemed to have rendered them unfit to membership thereof. To encourage, assist and extend the knowledge and study of Analytical Chemistry by the holding of periodical meetings, lectures, discussions, etc., and to encourage, assist and extend the knowledge and study of all questions relating to the composition of articles of food, drugs, and commercial products generally, and thereby to promote, or assist to promote, the efficiency and proper administration of the law.

"Every applicant for membership of the Association shall be not less than 25 years of age and shall hold or have held the appointment of Public Analyst, Deputy Public Analyst, Official Analyst, or shall hold a professional qualification and appointment which, in the opinion of the Council, is not lower than that prescribed for Public Analysts or Official Analysts. Every application for membership shall be placed before the Council and the Council shall have the power in their absolute discretion to accept, suspend or reject any application".

Administration of a Public Analyst's Laboratory

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The value of reconsidering objectively the organisation of a laboratory which is continuously developing is described, with practical applications, by a professional Organisation and Methods Officer. Advantages of a punched card system over the older methods of office and laboratory administration are detailed.

PART 1

THE POTENTIAL VALUE OF ORGANISATION AND METHODS

It is fashionable at the present time to call upon the computer to solve administrative problems in offices. Although it is an expensive instrument, it offers a fantastic calculation speed and a clearly definable level of reliability, enabling firms to do away with whole clerical sequences and save many thousands of pounds on junior staff salaries. It also invites administrators in senior positions to take the opportunity to define their problems in quasi-mathematical terms and computerise them, thereby speeding up the whole decision-making function in which they are involved. It is questionable whether Public Analysts could benefit greatly from such competent electronic assistance, and without investing the large capital sums involved, much could be gained by allowing the simpler and more traditional methods of the Organisation and Methods analyst an opportunity to prove their value.

O & M has accumulated a certain mystique in its few years of practice, and its practitioners have acquired a certain amount of expertise. But it is really a rather phlegmatic and routine area of work. The experienced O & M Officer develops a skill in the rational approach to administrative problems. Often the only difference between him and the manager he is assisting is a certain objectivity, and time to spare for persistent inquiry and basic thought. In some jobs he will have a few facts and figures about typing or copying operations up his sleeve. In others he will have to rely solely on his capacity for thought and imagination.

If a Public Analyst, feeling his office and laboratory would benefit from an O & M Study, requested a detailed investigation, the investigator would probably approach the job in the following way:—

(i) An overall look at the organisation of staff, the aims of the enterprise, and the work flow, would be taken.

(ii) An examination in detail of operating methods and documents would be made to see if a relatively minor change in them could produce a disproportionately large saving in time or in direct expense.

(iii) A written report would be submitted with recommendations of changed methods and re-organisation to ensure that the most economic and efficient means were used to achieve the overall aims of the enterprise.

The documents and procedures in the office of a laboratory have normally developed over the years without review and consequently contain inefficiencies which the Analyst does not have time or incentive to uncover.

Figure 1 illustrates the sort of situation which is not at all unusual in such an office. It will be seen at once that there are no less than twelve separate documents or ledgers which must be filled in as a sample passes through the laboratory and the invoice is prepared. Further investigation would reveal that no less than five people are required by the system to enter details on one or more of the documents concerned.

Without pursuing the point further, it is probable that the situation illustrated is not efficient because it inclines too far towards a "belt and braces" clerical philosophy. Although numerous accounts ledgers and sample books should, in theory, act against clerical errors and omissions it is clearly best to try and achieve a compromise between "belt and braces" and carelessness. It should be possible to simplify the number and design of the documents and books with no danger either to efficiency or to security. Ideally one document only should provide a proper control and record of a sample's progress. Alternatively, punched-cards or wall charts might be used. A punched-card application is described in the second part of this article.

Outside the office in the laboratory, a parallel situation can and usually does occur. There is a constant danger that chronic waste of movement will set in. At the laboratory visited by the author it was noticed that someone seemed to be on the move all the time, and frequently two or more assistants. This implied the expenditure of at least one whole salary on walking, the cost of which might be reckoned at £1,000 a year or £10,000 in a ten-year period.

Observation of a test was made which had some interesting results. The test was for the butter-fat content in a sample of sweets, and it broke down into nine stages (shown in Table I), located in various parts of the laboratory.

The observation shows that the laboratory assistant walked at least 140 paces during the test. But since there were incidental journeys (e.g. to fetch cotton-wool from the bench during stage (3), in order to filter the washed fat before distillation), the real walking distance was two or three times greater than the theoretical distance—perhaps 300 paces in all.

Among the various possible ways of minimising "walking expenses" the following suggestions, many of which will already be known, are proposed:

(i) laboratories should be organised on flow systems with full attention to work study principles. Instruments should be located optionally —i.e. at the centre of gravity of their biggest users;

(ii) untidiness should be avoided at all times, as this encourages the displacement of articles which sometimes results in prolonged searches;

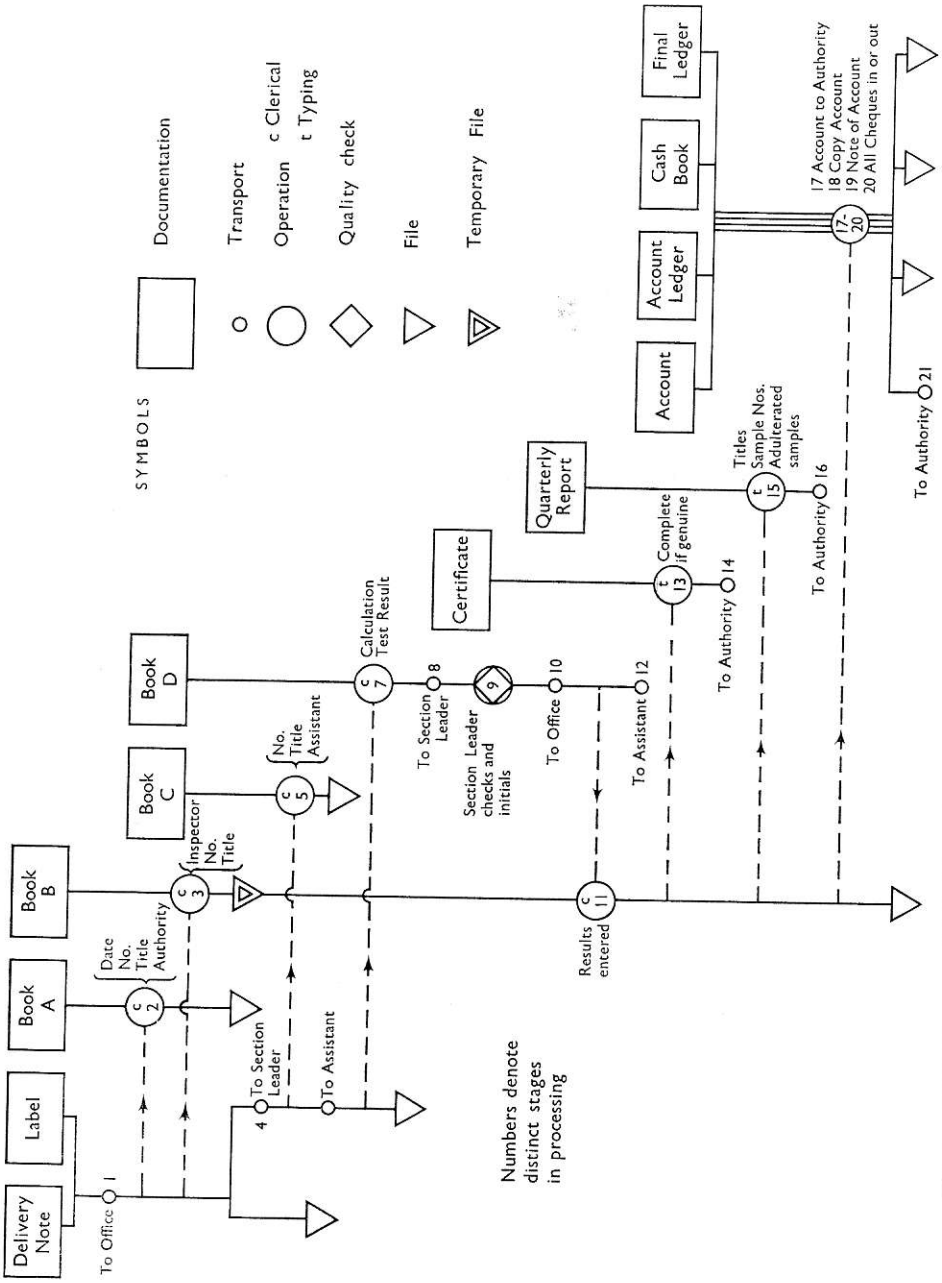

Fig. 1. Flow diagram of Administrative and Analytical stages in the traditional processes of a laboratory.

(iii) benches should be designed in circles or squares so that assistants may be surrounded by their instruments, all at an arm's length;

(iv) chutes, movable belts and trolleys should be installed to move chemicals and instruments about the laboratory;

(v) movable instruments should be given wheels so that they can be wheeled about in trolley fashion;

(vi) as much use as possible should be made of multiple instrumentation, so that one assistant on one visit can attend to several concurrent tests;

(vii) samples should only be tested when a large number of the same category have accumulated;

(viii) where long benches are used, they should be arranged radially in the laboratory with the most-used instruments in the centre.

The above observations suggest that all available effort should be made in two directions. Firstly, the ledgers and documentation involved in the office of a Public Analyst's laboratory should be carefully examined in the light of fundamental principles and then simplified as far as possible. Secondly, very serious consideration should always be given to laboratory layout. Generally speaking, machines and instruments, though they require capital outlay at the very beginning, are not as expensive as staff. A laboratory which employs only six staff is committed to an expenditure of about £60,000 over a ten-year period, the largest single item of expenditure. Every effort possible should surely be made to reduce such a large sum.

TABLE I

DISTANCES TRAVELLED DURING ROUTINE ANALYTICAL OPERATIONS

Stage	Description	Location and Paces from Work Bench
1.	Take sample of 3 sweets, crush them and weigh the beaker, beaker with sample and calculate weight	Balance, 7
2.	Heat with acetic acid	Steambath, 4
3.	Extract fat with mixed ether, wash free from acetic acid and distil	Inflammable, 18
4.	Add acetone to dry	Inflammable Store, 12
5.	Heat in oven at 100° C	Oven, 7
6.	Weigh to constant weight	Balance, 7 (× 2 at least)
7.	Micro-Reichert preparation	At Bench
8.	Distil	Stills, 8
9.	Titrate and calculate result	At Bench
	Total:	70 paces

The value of O & M in attempting to find solutions to these kinds of administration problems has been demonstrated consistently in many of the larger industrial corporations and in government departments. It is not

simply a question of the use of specialist techniques which admittedly are available to the O & M investigator, but of having the time available to devote to careful, critical examination of all the aspects of the problem. All too often, the Public Analyst, under heavy pressure to complete his day-to-day schedule of work, can only devote marginal time to problems which really need more attention before they can be solved properly.

PART 2

A RECORD SYSTEM FOR THE P.A. LABORATORY

In all Public Analyst laboratories there is an inherent need for systematically compiling records of samples received for analysis, and the results of the analytical work carried out upon them. In addition, a procedure whereby smooth operation of the final processing and reporting can be ensured is an obvious necessity. These processes may be considered as basic requirements, but a Public Analyst will also have responsibilities in other directions, as for example in the production of monthly, quarterly or annual reports for his Local Authority. These in turn involve the arrangement often of large numbers of samples into various different categories: Alphabetical; Formal/informal; Satisfactory/unsatisfactory; Foods/drugs; Complaint samples, and so on.

When treating special cases, the Analyst finds it invaluable to be able to refer to previous analyses of similar foods or drugs; he may be asked to provide information on a class of foodstuff which is being specially investigated elsewhere, e.g. Soft Drinks, Canned Meat Products; or he may be requested to assist in a national survey, be it on the subject of drugs, pesticides, etc., which involves collating results of a year's work or more in the appropriate section of the field.

Efforts to produce all the information required in each particular case with the minimum of delay and inconvenience to the administration of the laboratory have often resulted in the compilation of voluminous records—or, alternatively, if they are compact, such records defy simple sorting of all the required data.

From the above remarks, it can be readily deduced that there are two basic features which must be borne in mind when creating a suitable system of office and laboratory records, namely

(1) All possible information must be recorded.

(2) A highly efficient filing system must be devised for extracting the information.

The compactness of records governs the space they will occupy. Thus if any one sample is entered when received, the results of its analysis added to the entry and it is then finally processed and reported, the minimum requirements of (1) above are fulfilled. For future reference, one must be able to abstract the details under a dozen or more different headings, e.g.

(*a*) The date sample was submitted (month, quarter, etc.).

(*b*) Authority submitting the sample.

(*c*) Serial number.

(*d*) Category of food, drug, fertiliser, etc.

(*e*) Alphabetical classification.

(*f*) Any one of the individual analytical determinations performed on the sample.

This sorting can be carried out either by looking through all records for a shorter or longer period, known to include the required entry, or by duplication of the entries under various headings so that the collected information is immediately available in the form required.

Both methods are time-consuming and the latter causes rapid accumulation of record books or papers, most of which are never referred to.

Time is frequently lost by unnecessary duplication of effort, in that the laboratory assistants use work-books or sheets from which their results are later transferred to office record books. Means of obviating such labour have been demonstrated in the past by designing "sample-sheets" or cards on which both laboratory results and calculations can be recorded and the sheets or cards can then be used for the administrative process of reporting and finally filed for reference. Such systems have served their intended purposes, but fail in that they can be filed or segregated in only a few different ways, e.g. alphabetically, by serial number, or by Authority.

It is proposed that the advantages of all systems can be incorporated into a comprehensive plan which is ideal for use in a Public Analyst's Laboratory. The method is to use a punched card system in which the reverse face of the card is used for laboratory results and calculations, and the front for recording the final figures employed in the report, and for other significant details.

The punched card consists of a plain or ruled rectangular card which has about 100 holes punched round its perimeter as shown in Figure 2. Many standard types of these cards are available and others can be designed for specific purposes. There are also various systems of punching, but it has been found that the straightforward type illustrated here will fully perform the functions required by the present proposals, and as it is of standard design, it has the important advantage of being the cheapest available.*

At various stages in the processing of cards and samples, information is accumulated on the card and coded by clipping the appropriate holes. The well-known methods of arranging punched cards can then be applied after their eventual filing, allowing alphabetical, numerical or individual selection. The advantages of this ready means of producing an ordered array is obvious when monthly, quarterly or annual reports are required.

* The Copeland Chatterson Co. Ltd., London and Stroud (Glos.).

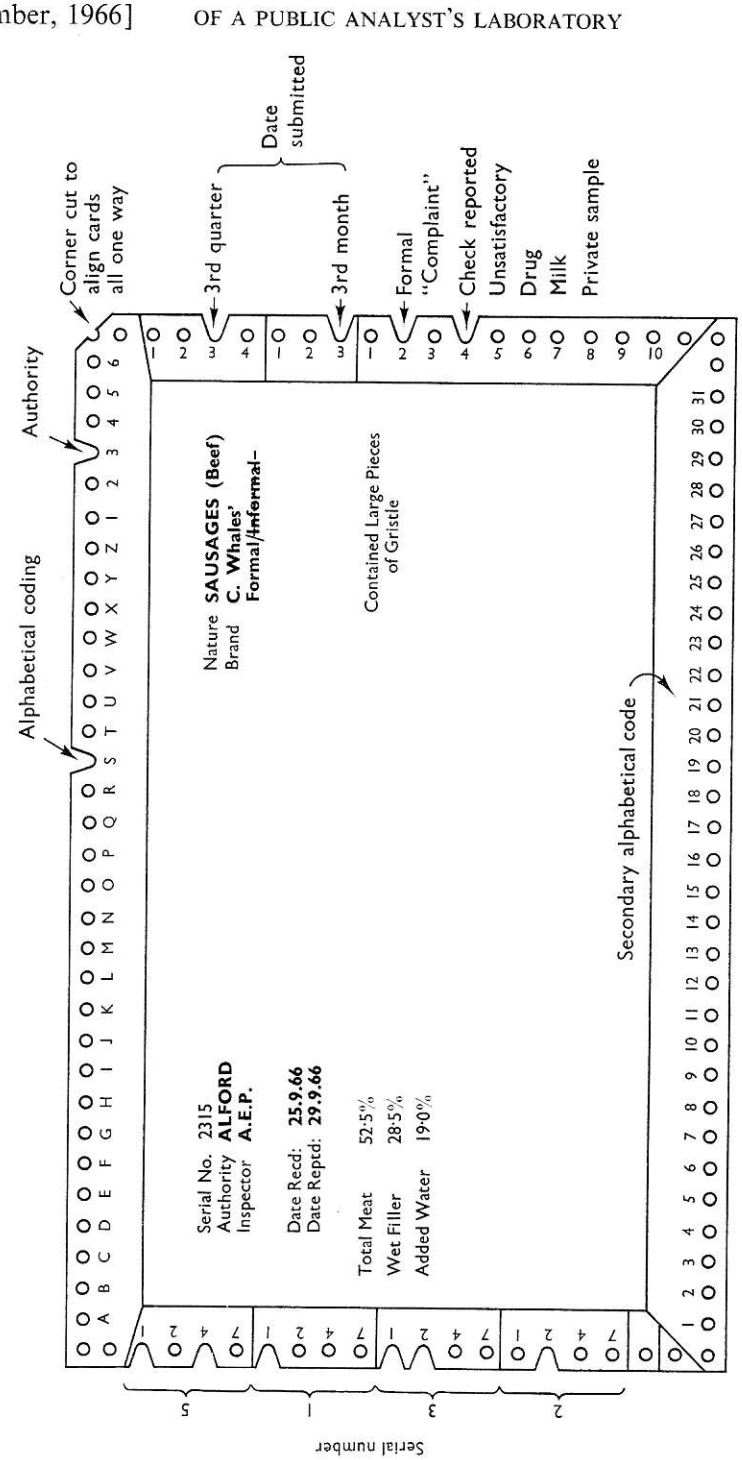

Fig. 2. Punched-card System for a Food and Drugs Laboratory.

It is useful to store the completed cards in drawers or cabinets to include preliminary filing of the normal type, so eliminating one step in the needle sorting process. A further step can be obviated by using different coloured cards for one section of the system, e.g. the Authority submitting the sample.

The following method of operating the system has been applied with most promising results.

Efficient administration of the system requires careful preparation of the cards. For each sample, one card is allotted, and the date, name and category of sample, brand name, type (formal or informal, complaint, etc.), date of receipt, Authority and other basic information are written or typed on the card. This information is translated into the appropriate hole coding and corresponding holes are clipped. At this stage, labels are examined for possible offences. Cards and samples are stored until needed for analysis with appropriate attention to the possibility of deterioration of perishable foods. A diary recording dates of receipt of samples is indispensable.

The analysis is made by laboratory staff who perform tests according to headings which are also added to cards when they are prepared. Weights, titrations, readings and other results, as well as calculations, are recorded on the rear of the card.

Completed cards are returned to the office, working is checked, and significant results are transferred to the front of the card, which then carries this and all the systematic information relating to the sample. The card is used for compiling a report, which it can accompany for the Public Analyst's use when he signs the certificate. The card having been inspected, the last action before filing it is to clip the hole allotted to indicate that it has been fully processed by all concerned, unless, for example, other holes are used later to indicate that relevant invoices have been sent or accounts paid.

A "Flow diagram" of the treatment of a sample is shown in Figure 3.

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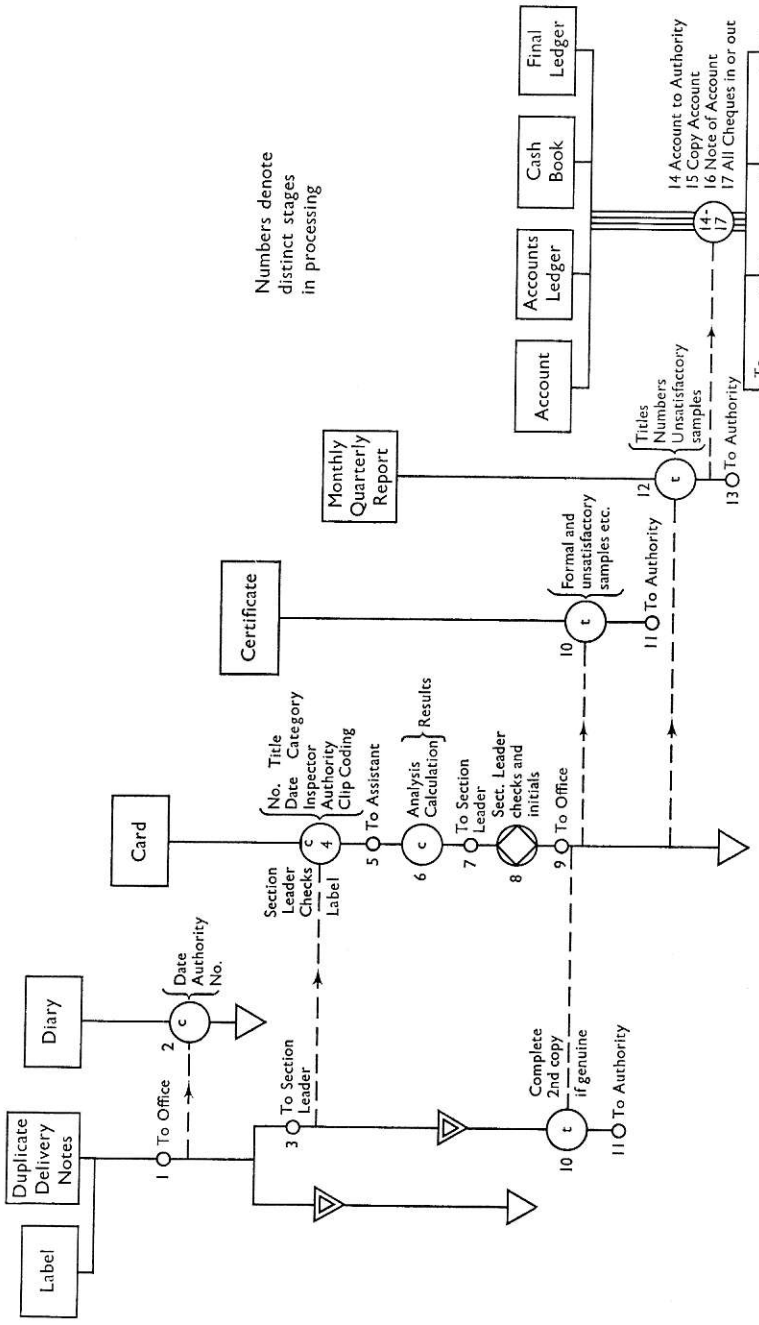

Fig. 3. The procedure shown in this diagram is considerably simpler than that in Figure 1. The punched card scheme eliminates two books and three "operations". It also simplifies alphabetical listing and sorting of test details. Symbols (see Fig. 1).

The Composition of Minced Beef

by D. PEARSON

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Compositional data are quoted for samples of minced beef purchased from retail butchers' shops and supermarkets.

There do not appear to be any composition figures quoted in the literature for minced beef as sold in retail establishments. The analytical data quoted in Table I summarise the results obtained on 46 samples of minced beef purchased from various butchers and supermarkets between 1957 and 1965. The figures cover the basic composition and two selected spoilage values.

Methods employed in the investigation

The methods employed for the determination of the water, fat and nitrogen contents were essentially those recommended for the examination of meat products by the Society of Public Analysts and other Analytical Chemists[1] (now the Society for Analytical Chemistry).

The total volatile nitrogen (TVN) was determined by macro-distillation from magnesium oxide.[2] For the estimation of the free fatty acids the fat was extracted by mechanical maceration with chloroform. The macerate was filtered, the filtrate washed with water and re-filtered. An aliquot of the final solution was mixed with an equal volume of neutralised alcohol and titrated with decinormal alkali.[3]

Discussion

The minimum, maximum and mean values for the samples examined are given in Table I. Only two samples contained fat in excess of 30 per cent., which is the maximum applied in certain parts of the U.S.A.

The mean nitrogen calculated on the fat-free material was 4·08 per cent., which is higher than the average factor of 3·55 recommended for beef by the Society for Analytical Chemistry.[4] This S.A.C. figure corresponds with fat-free beef of the following composition:

Protein = $3{\cdot}55 \times 6{\cdot}25 = 22{\cdot}2$ per cent.

Assuming the ash = 1·3 per cent., then

Water = $100 - (1{\cdot}3 + 22{\cdot}2) = 76{\cdot}5$ per cent.

This figure for the water in the fat-free material is higher than those obtained with any of the samples of minced beef examined (maximum 76·3 per cent.). These higher nitrogen and lower water contents correspond therefore with a certain degree of drying out before sale.

The TVN figure, which rises with increasing protein breakdown, did not exceed 19·3 mg of nitrogen per 100 g for any of the minced beef samples included for the compilation of Table I. All these meats were assessed organoleptically as having an acceptable odour.

TABLE I

ANALYTICAL DATA OBTAINED FROM "FRESH"* SAMPLES OF MINCED BEEF

Determination	Percentages - Min.	Max. (46 samples)	Mean
Fat	18·1	38·2	24·4
Water	46·3	60·2	55·3
Water in fat-free material	70·1	76·3	73·2
Protein (N × 6·25)	15·0	22·8	19·3
Protein in fat-free material	22·7	28·7	25·5
Nitrogen in fat-free material	3·63	4·58	4·08
†Total volatile nitrogen (mg of N per 100 g)	10·6	19·3	15·5
†Free Fatty Acids (as oleic acid in extracted fat)	0·38	1·74	0·97

* All samples were judged organoleptically as having an acceptable odour.
† Determinations covering 44 samples.

From the results of experiments on stored minced beef, it is apparent that the amounts of TVN and the FFA both increase progressively as the meat deteriorates. By correlating the objective chemical figures with the subjective odour 'scores' it was considered that, on the average, beef is adjudged as spoiled when the TVN reaches a value of 25 mg of nitrogen per 100 g, and the FFA exceeds 2·5 per cent. (calculated as oleic acid in the extracted fat). Fat hydrolysis appears to affect the odour at a later stage than the TVN, so that in the small number of instances where proteolytic breakdown is minimal, organoleptically noticeable deterioration tends to occur at a higher FFA level than in the majority of samples where both values increase concomitantly. As the predominating type of spoilage varies according to the sample under consideration, any attempt to adopt critical limits, based on chemical figures would have to specify values which reflect changes in both the lean and fatty tissue. The author suggests that the dual, maximum acceptability limits of 20 mg of total volatile nitrogen per 100 g, and 1·8 per cent. of free fatty acids, should be applied on a trial basis for minced beef. Further work is proceeding in an attempt to assess the possibility of applying practical limits of this type.

References

1. Society of Public Analysts, *Analyst*, 1952, **77**, 543.
2. Pearson, D., "Chemical Analysis of Foods", Fifth Edition, Churchill, London, 1962, p. 309.
3. Pearson, D., *J. Assoc. Publ. Analysts*, 1965, **3**, 76.
4. Society for Analytical Chemistry, *Analyst*, 1963, **88**, 422.

Analytical Notes

by G. S. MEADOWS

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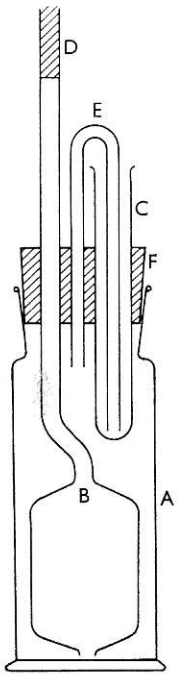
1. A SULPHUR DIOXIDE ABSORBER FOR USE IN THE MONIER–WILLIAMS METHOD

In the Monier–Williams method[1] for determining sulphur dioxide in food, the sulphur dioxide is absorbed by passing it through hydrogen peroxide solution in a conical flask followed by two Peligot tubes. Since this method was introduced several modifications have been suggested[2,3,4].

The absorption system shown in Figure I has been found to be very satisfactory and ideally suited to routine determinations being efficient, easy to manipulate, robust and compact. At the end of the distillation period, the solution in the secondary trap will automatically siphon over into the primary trap.



A = Gas jar—capacity 150 ml, overall height 15 cm, external diameter 5 cm, with tapered unground socket.
B = Bulb—capacity 40 ml.
C = Test tube—10 cm long, 1·5 cm internal diameter.
D = Connection to upper end of condenser.
E = Glass tubing.
F = No. 37 rubber bung.

Fig. 1. Sulphur Dioxide Absorber

Method of Use

Neutralise 30 ml of 3 per cent. hydrogen peroxide solution to bromophenol blue using 0·1 N sodium hydroxide solution. Add 25 ml of neutral solution to the gas jar, 5 ml to the test tube and connect to the condenser. At the end

of the normal distillation time, turn off the heat and the flow of carbon dioxide; the solution in the test tube will quickly siphon into the gas jar. Disconnect from the condenser at D. Wash the bulb inside and out whilst withdrawing it from the gas jar and titrate the peroxide solution in the normal way.

The Author thanks Mr. P. Glasgow for assistance in assembling and testing this apparatus.

References

1. Monier-Williams, G. W., *Analyst*, 1927, **52**, 343, 415.
2. Henville, D., *Analyst*, 1929, **54**, 228.
3. Shipton, J., *Food Pres. Quart. (Australia)*, 1954, **14**, 54.
4. *J. Ass. Off. J., Agric. Chem.*, 1966, **49**, 235.

2. A SIMPLE FILL/EMPTY SYSTEM FOR WATER BATHS

In connection with the British Standard method[1] for determining the density of milk, a siphon arrangement was sought for filling and emptying the water bath used for heating milk to 40°C and then cooling it to 20°C. As one could not be found in the laboratory catalogues, the type given in Figure 2 was tried, and proved to be successful. It consists of a length of rigid plastic tubing ($\frac{1}{4}$-inch internal diameter), which has been cut out near its mid-point to give a $\frac{3}{4}$-inch by $\frac{1}{4}$-inch hole, and then bent with the aid of heat so as to fit closely over the edge of the bath, and extend to the bottom. The tubes to the water supply and to waste are outside the bath.

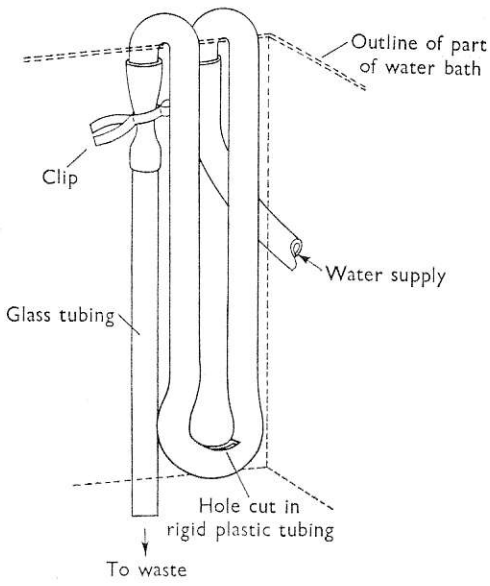


Fig. 2. A fill-and-empty system for water baths.

To fill the bath the clip must be in the closed position. To empty the bath, the clip is opened and a short sharp burst is given on the water supply. This primes the tube and the water is siphoned over.

The siphon is easy to construct and is ideal for using with an ordinary, rectangular, plastic pan or similar receptacle, thus avoiding the necessity of having a special bath with an outlet at the bottom and a separate filling device.

Reference

1. British Standard 734 (Part 2), 1959, "Density Hydrometers for use in Milk". British Standards Institution, Park St., London, W.1.

Drug Testing under the Food and Drugs Act during 1964-5

The report of the Drugs Sub-Committee of the Association of Public Analysts

A survey of the sampling and analysis of drugs submitted under the Food and Drugs Act, 1955, has been supervised by the Council of the Association of Public Analysts. It covers the period from 1st April, 1964, to 31st March, 1965, and is a summary of the work of 38 laboratories, representative of those in England and Wales.

During the 12 months under review, 6772 samples of drugs were examined, and of these, 306 (4·5 per cent.) were reported as unsatisfactory. The classes of "adulteration" were as follows:—

Labelling Irregularities	75
Deterioration on Storage	97
Misleading Claims	13
Compositional Irregularities	116
Unsuitable Containers	5
	306

ERRATUM

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Line 17: *For* "far apart" *read* "close".

Book Reviews

OILS, FATS AND FATTY FOODS. BY K. A. WILLIAMS, B.SC., PH.D., F.R.I.C. Fourth Edition. Pp. vi + 488. London: J. & A. Churchill, Ltd., 1966. Price £5.

The fourth edition of "Bolton and Revis" follows closely the plan of the third edition but contains added material made necessary by the wealth of new techniques and additional knowledge which has appeared during the intervening sixteen years.

The type has been changed and this has resulted in the condensation of more information into fewer pages. The chapter headings of the third edition have been retained (presumably there is no sinister significance in the change from Roman to Arabic numerals) and two extra appendices have been added—one providing a list of some of the numerous official standards and the other indicating new sources of oils and fats.

New legislation has made necessary some changes in the section on added colouring matters and has also resulted in the inclusion of short sections on anti-oxidants and emulsifiers and stabilisers in Chapter 2. Reference is made to the A.P.A. monographs on the first two subjects.

Chapter 3 on General Analytical Methods has a considerable amount of extra material made necessary by the introduction of the newer physical techniques; brief accounts of the application of infra-red absorption spectra, nuclear magnetic resonance, mass spectrophotometry and counter-current fractionation have been included and column, paper, thin-layer- and gas-liquid chromatography have been covered in 12 pages.

Among other new additions to the text is a method for the extraction of aflatoxin from ground-nuts, included in the chapter on Feeding Stuffs.

As in previous editions much ground has been covered and the author has had to preserve a balance between keeping abreast of new development and confining the book to manageable proportions. In general, this seems to have been admirably achieved although there are one or two omissions which are worth recording. For instance, under the determination of hydroxyl value some reference might have been made to the phthalation method and to the more recent stearic anhydride process of Sully. Milk is dealt with quite fully, and the determination of the non-fatty components and bacteriological tests are included, but no reference is made to the determination of penicillin.

Some mention of the semi-micro modification of the Reichert process, using one-fifth of the standard quantities, might perhaps have been made. This gives excellent results and is invaluable when the amount of sample is small.

Some eyebrows might be raised at the statement on page 448 under "Ice Cream", that "Practically, the analysis is often restricted to the determination of fat and total solids, . . .". Most analysts would include a determination of lactose as a measure of the non-fatty milk solids present.

However, these are minor criticisms and the book is certain to find its way into the library of most food analysts. It is well printed and produced and seems to be remarkably free from misprints. References to the original literature are numerous and, as in previous editions, are incorporated in the text instead of being collected at the end of each chapter.

S. J. BUSH.

LABORATORY HANDBOOK FOR OIL AND FAT CHEMISTS. BY L. V. COCKS AND C. VAN REDE Pp. xxiv + 419. Academic Press, London, 1966. Price £4 10s.

This book consists of seven sections dealing with Oilseeds, Nuts, Cakes, Meals and Animal Feeding Stuffs: Oils, Fats, Fatty Acids and Fatty Alcohols: Glycerol: Analyses in Connection with Processing: Fat Products: Specialist Methods and Techniques: and Analytical Characteristics and Composition of the more common Oils and Fats. The principal authors and the fifteen other collaborators who have contributed to the section on "Specialist Methods and Techniques" are, or have been, connected with oil and fat

analysis within the Unilever organisation: in fact, the book is based on current methods in use in the Unilever laboratories. Since such methods will inevitably have been chosen for their reliability and utility, both the inclusions and the omissions are of special interest.

Although this book covers substantially the same field as that covered by Williams, the emphasis is somewhat different: it is intended primarily as a laboratory handbook rather than as a general textbook. Nevertheless a little more information on the comparative merits of methods and a fuller discussion of the interpretation of results would have added to its usefulness. Adequate consideration has been given to the very important subjects of sampling and preparation and the arrangement is logical and easy to follow.

Section 1 contains descriptions of the determination of nitrogen and protein by the Kjeldahl method and one encounters the statement that the absorption of the ammonia in 4 per cent. boric acid has been found by some users to give unreliable results. Apparently, this criticism does not apply to the semi-micro method, for which the use of boric acid is standard, but it is a little disappointing that this alleged discrepancy has not been further investigated.

No reference is made to the occurrence or determination of aflatoxin in groundnut meal.

The chapter on Chemical Characteristics contains descriptions of most of the usual determinations and also some newer material, including paper chromatography of fatty acids and methods for the detection and determination of a number of antioxidants. Under "Iodine Value" only the popular and well-tried Wijs method and the Hofmann and Green modification are described. Sully's stearic anhydride process for determination of hydroxyl value is referred to but not described in detail and the reader is referred elsewhere for a description of the determination of thiocyanogen value. This seems a pity in a practical handbook and it might well have been preferable to include some newer and less known methods such as that of Sully rather than those which, like the acid value and iodine value, can be readily found elsewhere. The headings under each determination in this section of the book, e.g. Explanatory Note, Principle, etc., do not always fulfil the promise inherent in the title: this applies especially to the heading "Principle".

Micro- and semi-micro methods are becoming increasingly popular and, of course, many of the newer physical methods described are truly micro-methods. The semi-micro Kjeldahl process is now an accepted method, but semi-micro modifications of other classical methods such as, for example, the Reichert process and Thom's microscope slide method for iodine value receive no mention.

The section on Fat Products contains much useful information for the food chemist including methods for the detection of colouring matter and a spectrophotometric method for the determination of benzoic and sorbic acids in fatty products. The section on specialist techniques deals with many of the newer physical methods and also contains chapters on such varied subjects as the assessment of variability in routine analysis, the direct determination of trace metals without ashing and accelerated stability tests. These newer methods are given considerably more attention than in the book by Williams reviewed above.

It is a pity that the book is marred in places by loose or obscure English and by the use of inelegant expressions such as "weigh off" and "distil off". An example of obscure English is the definition of unsaponifiable matter on p. 123—

> "By the content of unsaponifiable matter is understood the percentage calculated on the original fat (Note 1) of natural unsaponifiable matter present in the fat (sterols and hydrocarbons) and organic unsaponifiable matter not volatile at 100° C. (mineral oils) which are generally absent from fats".

This book contains much material that is useful and not readily accessible elsewhere and the section on specialist methods and techniques is particularly interesting. Most analysts will have something to learn from this section alone. References to the original literature appear to be adequate and are given as footnotes at the base of each page. S. J. BUSH.