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A Simple Procedure for the Determination of Arsenic and Tin in Food by Atomic Absorption Spectrophotometry

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A hydride generation technique, which merely utilises a boiling tube and a large bore tap, is presented as a reliable and simple method for the determination of levels of arsenic and tin in foods down to 0·1 p.p.m.

Many publications have appeared describing the determination of arsenic and tin by atomic absorption spectrophotometry with use of the hydride generation technique[1,2,3]. They all suffer from various disadvantages, e.g. in requiring specially made or expensive generation cells; manipulation of corrosive sample and reagent solutions in the vicinity of the instrument; cleaning of the cell between analyses, resulting in wastage of gases; adoption of a consistent technique for handling the pipette used in adding the reagent to obtain reproducible responses.

The method described is considered to have the novel aspects of being simple and rapid, requiring no elaborate glassware and needing minimal modifications to the instrumental fittings. It involves:

(a) the use of nitrogen and hydrogen in place of air and acetylene, respectively;

(b) the use of a simple boiling tube and a wide-bore tap costing about £6 as the generation cell;

(c) the addition of solid sodium borohydride reagent to the sample solution, the hydrides formed being swept into the flame by the excess hydrogen evolved; this obviates the need for complex by-pass systems for an auxiliary gas.

Procedure

HYDRIDE GENERATOR

This consists of a Pyrex boiling tube (25 × 150 mm), etched with a 20 ml mark, fitted with a rubber bung and a large PTFE stopcock† with 35-mm long side arms of 10-mm bore. The free side arm is connected to a polythene tube (180 mm × 3 mm i.d.) via a small rubber bung bored with a small hole. The polythene tube is connected to the A.A. instrument (Instrumentation Labora-

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† G. Springham & Co. Ltd, South Road, Templefields, Harlow, Essex; Cat. No. ISI/10/B.

J. Assoc. Publ. Analysts, 1978, **16**, 71–75

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tory Model 151) via another small rubber bung bored with a small hole, positioned in place of the normal nebuliser. The counterflow jet is turned away from the inlet hole in order to maintain the flame when the polythene tube is disconnected from the stopcock. A small screw clip, positioned in the interconnecting plastic tube of the instrument carrying the "oxidant", i.e. nitrogen, replaces the pin-hole constriction in the normal nebuliser.

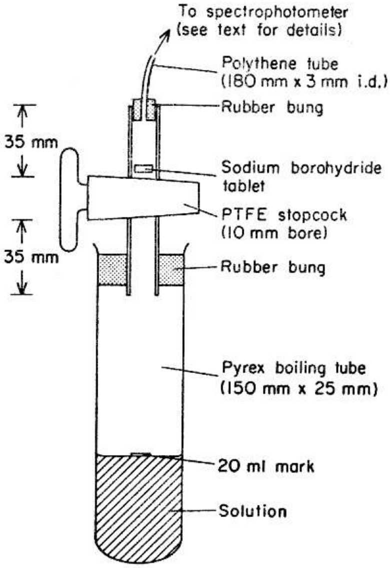

Fig. 1. The hydride generator.

The generator has also been successfully used with the Perkin-Elmer model 306 instrument by removing the oxidant tube from the nebuliser and blocking it off. The polythene tube of the generator is then simply pushed on to the oxidant inlet tube of the nebuliser chamber. The end of the sample tube is left open to the atmosphere.

INSTRUMENTAL PARAMETERS (FOR IL MODEL 151)

The maker's recommended settings are employed. Hollow-cathode lamps, background correction, peak height mode and 1/16 sec. response time are used. Wavelengths for arsenic and tin are 193·7 nm and 286·3 nm respectively. Flows of the nitrogen and hydrogen gases (used in place of the normally employed air and acetylene) are 6 and 9 ft^3 per hr. respectively.

PREPARATION OF SAMPLE SOLUTIONS

Food samples (2 to 5 g) are digested in 250 ml conical flasks with concentrated sulphuric acid (5 ml) and 50 per cent. (w/w) hydrogen peroxide solution using a hot plate[4]. The cooled clear digests are transferred quantitatively to 100 ml volumetric flasks with water and diluted to the calibration mark.

Tin. The blank, standard and sample solutions are prepared by pipetting 1-ml aliquots each of 5 per cent. sulphuric acid solution, 0·5 p.p.m. of tin in 5 per cent. sulphuric acid solution (prepared by diluting 1000 p.p.m. stock solution *ex* BDH), and the sample solution into three separate boiling tubes and diluting to the 20-ml marks with 0·5 per cent. sulphuric acid solution with shaking.

Arsenic. The diluted digest (20 ml) and concentrated hydrochloric acid (15 ml) are diluted to 50 ml with 5 per cent. sulphuric acid solution. The well mixed solution is poured into the boiling tube up to the 20-ml mark.

A blank and a standard are prepared by diluting 0 and 1 ml of 0·5 p.p.m. arsenic solution respectively to the 20 ml mark with a mixture of 35 ml of 5 per cent. sulphuric acid and 15 ml of concentrated hydrochloric acid.

The arsenic is then reduced from As^{V} to As^{III} by adding to the solutions in the boiling tubes 0·5 ml of iodide solution. (The iodide solution is prepared by dissolving 17 g of potassium iodide and 10 g of sodium ascorbate in water to a final volume of 100 ml. The solution should be stored in a refrigerator and prepared weekly.) At least 1 minute is allowed for the reduction.

HYDRIDE GENERATION PROCEDURE

The boiling tubes, held in a test-tube rack, should be sited in a well ventilated extraction area to remove the hydrogen evolved after they are replaced in the rack at the end of the analyses (see below).

The large rubber bung/tap is fitted to the first boiling tube. With the tap closed, a pellet of sodium borohydride (Aldrich Chemical Co. Ltd) is placed on the tap and the small rubber bung/plastic tube is secured to the free arm of the tap. The integrator button is pressed and the tap is opened to introduce the pellet to the solution, the boiling tube being shaken vigorously.

After a steady reading is obtained (within a few seconds), the boiling tube is removed from the large rubber bung and immediately placed back in the test tube rack; (hydrogen gas is still being evolved). The reading is recorded. The next boiling tube is fitted to the large rubber bung and the above operations are repeated.

About twelve boiling tubes can be dealt with before moisture accumulating in the tap prevents the smooth introduction of the pellet. This moisture can be quickly removed with a paper tissue.

Comments

The method is a simplification of the elaborate procedure described by Fiorino *et al.*[5] Unlike most hydride generation procedures described in the literature, the hydrides are swept into the flame by the hydrogen that is also evolved. This obviates the need for complicated apparatus for collecting the hydrides and for having by-pass gas systems for sweeping the latter into the flame.

For *tin*, the sensitivity of the method is very dependent on the acid strength of the solution. Linear dose-response relationships were obtained for 0 to 1 μg of tin in 0·5 per cent. and 5 per cent. sulphuric acid solutions, the sensitivity being about four times greater at the more dilute level. Using the procedure described, the 0·5 μg tin standard gives a typical absorbance of 0·300 with a blank value of under 0·010. This corresponds to a detection limit of about 0·2 p.p.m. of tin in food for a 5 g sample. (Sodium borohydride pellets supplied by BDH Chemicals Ltd gave an unacceptable high blank of about 0·070.)

For *arsenic*, a 60 per cent. response was obtained for As^{V} in 5 per cent. H_2SO_4 solution compared with the same amount of As^{III} and this was not altered on addition of iodide solution. Comparable results were obtained, however, in 5 per cent. H_2SO_4 and in 30 per cent. HCl solution but, contrary to the findings of Fiorino *et al.* (loc. cit.), only after addition of iodide solution. The concentration of sulphuric acid present is not critical, a similar response being obtained over the range of 3 per cent. to 5 per cent. Spurious results were sometimes obtained with use of KI solution, but these were eliminated when the mixed reagent of KI and sodium ascorbate was used as described by Siemer *et al.*[6] who also encountered the same problem; these authors attributed the interference to free iodine being present in the iodide solution. Using the procedures described, the 0·5 μg arsenic standard gives a typical absorbance of 0·320 with a blank value of under 0·020; (BDH sodium borohydride pellets gave a similar blank). This corresponds to a detection limit of about 0·1 p.p.m. of arsenic in food for a 5 g sample.

In routine operation, a 50-ml aliquot of the 100-ml digest is transferred to a 100-ml volumetric flask for the analysis of Pb, Fe, Cu and Cd by the APDC-MIBK procedure (further details may be obtained on request). A 1-ml aliquot is used for Sn and a 20-ml aliquot is treated with concentrated HCl for As, as described under Procedure. Direct aspiration of the remainder of the digest is undertaken for metals present in sufficiently high concentrations.

Hydrochloric acid extracts of canned foods prepared and analysed by direct aspiration in another laboratory were subjected to the present hydride generation technique. Table I gives the results obtained and illustrates the reliability of the hydride procedure.

TABLE I

TIN (P.P.M.) IN CANNED FOODS: COMPARISON OF THE RESULTS OBTAINED BY DIRECT ASPIRATION AND BY THE PRESENT HYDRIDE GENERATION PROCEDURES

Present method	Direct aspiration
180	186
370	393
270	213
25	20
85	55
130	112

Some samples of plant material, used in the hydride generation method described by Thompson and Thoresby,[3] were kindly supplied by the authors; these authors have demonstrated (loc. cit.) that their hydride generation procedure gave similar results to those obtained by the molybdenum-blue method but the latter was less accurate. Table II shows that the clean and rapid $H_2SO_4 + H_2O_2$ digestion procedure coupled with the present simple hydride generation technique give results which agree with those obtained by the above authors.

TABLE II

ARSENIC (P.P.M.) IN PLANT MATERIAL: COMPARISON OF RESULTS OBTAINED BY THE PRESENT HYDRIDE GENERATION PROCEDURE AND BY THAT DESCRIBED IN REFERENCE 3

Plant material	Present method	Reference 3
Herbage	7·1	6·4
Herbage	1·5	1·4
Herbage	0·69	0·79
Barley	0·095	0·093
Barley	0·010	0·012

In addition to the above comparative studies, recovery experiments were carried out on lemon and orange comminuted drinks spiked with 0·1 to 0·5 p.p.m. levels of Sn and As^{V}; average recoveries obtained were 99 per cent. and 98 per cent., respectively.

Thompson and Thomerson[1] have shown that the hydride generation procedure is also applicable to the determination of antimony, bismuth, germanium, selenium, tellurium and lead. Although they found that the conversion of lead into its hydride was poor compared with the other elements, the recent paper by Vijan and Wood[7] indicates the possibility of improving the sensitivity of this elegant technique sufficiently to permit its application to the determination of trace levels of lead in food.

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J. Assoc. Publ. Analysts, 1978, **16**, 77–79

Analytical Data for Various Iranian Vinegars

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One hundred and thirty-seven samples of various types of genuine vinegars from different parts of Iran have been analysed, to establish differential analytical figures for each type.

The vinegars much used in Iran are grape (wine), spirit, raisin and date vinegars, respectively. Determination of volatile substances has been suggested in order to differentiate the various types of vinegars[1,2,3,4]. In this study other analytical figures were also determined.

Materials and Methods

Vinegars from reliable sources were sampled, including laboratory-made as well as household preparations from different parts of the country.

The total and fixed acidity, specific gravity, total sugars, total solids, ash, phosphorus pentoxide and alkalinity of ash were determined according to the methods of the A.O.A.C.[5]; the iodine, oxidation, alkaline oxidation, and ester values by methods quoted by Pearson[6], and alcohol content according to that quoted by Lecoque[7]. The results of the analyses are shown in Table I.

Results and Discussion

With the exception of spirit vinegar, the respective analytical figures, especially those for the volatile substances of the various types of vinegars as shown in Table I, are very similar. Accordingly, differentiation of wine from raisin vinegar in relation to these values will not be possible. Apart, however, from organoleptic tests, which are very useful for distinguishing the various types of vinegar, the total solids and ash content of date vinegar are higher, while acidity, total reducing substances and phosphorus pentoxide contents are lower, than those of wine and raisin vinegar. The unripe wine vinegar, which is not often kept long enough for complete fermentation, is characterised by its low acidity. The spirit vinegar is marked by the lowest analytical figures of all except for acidity. The determination of oxidation, alkaline oxidation, ester and iodine values is very valuable for the differentiation of vinegars and non-brewed condiments. Of 61 components reported by Kohn in vinegar and investigated by gas chromatography and mass-spectrometry[8] the presence of diacetyl and acetyl methyl carbinol was not confirmed. As these substances contribute to

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TABLE I

ANALYTICAL FIGURES OF VARIOUS IRANIAN VINEGARS

Number of samples tested	Wine vinegar 66	Unripe grape vinegar 23	Raisin vinegar 22	Date vinegar 10	Spirit vinegar 16
Specific gravity at 25°C	1·011–1·033 (1·019)†	1·005–1·027 (1·009)	1·012–1·026 (1·016)	1·015–1·034 (1·025)	1·001–1·009 (1·005)
Total acidity as acetic acid, per cent.	3·21–7·59 (4·28)	0·9–2·94 (2·13)	3·51–5·82 (4·13)	1·81–4·9 (2·42)	4–5 (4·62)
Fixed acidity as acetic acid, per cent.	0·06–0·45 (0·28)	0·03–0·45 (0·27)	0·15–0·36 (0·21)	0·07–0·70 (0·33)	—
Total solids, per cent.	1·7–6·59 (3·35)	1·79–5·28 (3·16)	2·12–5·15 (3·13)	3·20–7·1 (5·56)	0·09–0·13 (0·1)
Total ash, per cent.	0·47–0·88 (0·5)	0·26–0·87 (0·51)	0·29–0·90 (0·45)	0·63–1·46 (0·86)	—
Alkalinity of ash (0·1 N/100 ml)	0·8–38 (21)	2–21·5 (14·5)	3·8–92 (16)	5–43 (32·5)	—
Alcohol, per cent. (v/v)	0·01–2·76 (0·09)	—	0·1–1·3 (0·15)	—	0·1–0·2 (0·13)
Total sugar, per cent.	0·03–0·70 (0·20)	0·09–0·82 (0·28)	0·13–0·45 (0·29)	—	—
Phosphoric acid as P_2O_5, per cent.	0·005–0·044 (0·031)	—	0·008–0·048 (0·026)	0·018–0·022 (0·020)	—
Iodine value	50–352 (184)	106–233 (159)	76–424 (198)	129–353 (191)	18–101 (86)
Ester value	17·2–127 (81)	25·5–152 (71·6)	27–86 (48·3)	19–64 (31·4)	12–52 (26)
Oxidation value	1414–2676 (2360)	2193–2636 (2370)	844–2824 (2461)	2170–2402 (2262)	720–1900 (1784)
Alkaline oxidation value	68–265 (109)	160–433 (207)	96–291 (186)	52–241 (139)	86–120 (108)

† Mean figures in parenthesis.

iodine and oxidation and alkaline oxidation values[1] of the vinegar, they may have been lost during the sample preparation treatment such as neutralisation, extraction or solvent evaporation.

The author wishes to thank Miss Zohre Ahmadi and Miss Mina Shoomer for their help in carrying out this investigation.

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J. Assoc. Publ. Analysts, 1978, **16**, 81–83

Paper Chromatographic Detection of Rapeseed Oil in Mustardseed Oil

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A paper chromatographic method has been developed that differentiates and distinguishes rapeseed oil in mustardseed oil. A hydrochloric acid extract of the oil is concentrated, paper chromatographed with a hydrochloric acid–water solvent system and visualised with ultra-violet light. The chromatographic behaviour permits the detection of the adulteration of mustard oil with as little as 5 per cent. of rapeseed oil.

Mustardseed oil (*Brassica nigra* and *Brassica juncea* or *Sinapsis alba*) is similar in composition to rapeseed oil (*Brassica campestris*, sub-species *napus* etc.) and is difficult to distinguish from it. It contains erucic acid, in much the same proportion as in rapeseed oil. Generally speaking, its appearance and behaviour in chemical tests is similar to that of the rapeseed oil. Although the smell of rapeseed oil is distinctly different from the pungent smell of mustard oil, nevertheless the presence of a maximum of 25 to 30 per cent. of rapeseed oil in the latter presents a problem for the analyst, the characteristic putrid smell of the rapeseed oil being, under the circumstances, completely masked by the pungency of the mustard oil. The saponification and iodine value determinations cannot establish the presence of one in the other except in extreme cases. Paper chromatography of the pigments extracted in hydrochloric acid affords a sensitive and reliable test for the detection of rapeseed oil in mustardseed oil.

Experimental

PAPER CHROMATOGRAPHY APPARATUS

1. Ultra-violet lamp.
2. Whatman chromatography paper.
3. Standard paper chromatographic jar.
4. Test tube (Corning make) ½ inch diameter.
5. Graduated 10-ml pipette.
6. Separating funnel, 100-ml capacity.
7. Beaker, 25-ml capacity.
8. Capillary tube.

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REAGENTS

1. *Analar hydrochloric acid.*
2. *Petroleum ether* (40–60°C range).
3. *Solvent system;* hydrochloric acid, specific gravity 1·17/water (40:60).

METHOD

Put 2 ml of sample oil in a test-tube and add 5 ml of concentrated HCl. Shake vigorously for 1 minute. Allow to stand for 10 minutes and shake again for another 1 minute. Transfer the mixture to a separating funnel and leave to separate. Run off the HCl fraction into a beaker; wash the separating funnel thoroughly and rinse with petroleum ether. Put the HCl fraction into the clean separating funnel and wash out the beaker with, first, 1 ml of concentrated HCl and then 5 ml of petroleum ether: add the washings to the HCl fraction in the separating funnel. Shake thoroughly with 5 ml of petroleum ether to remove any trace of oil present in the HCl fraction. Leave the petroleum ether fraction and finally transfer the acid extract to the beaker and concentrate it on a burner in a fume chamber. This concentrate should now measure 0·25 ml. The concentrate is now ready. Spot on the chromatographic paper and use HCl/water (40:60) as solvent. Allow it to run about 15 cm from the origin. Dry the paper and expose it under ultra-violet light. The running time is almost 30 minutes.

Results and Discussion

Experiments were performed with pure mustardseed oil and pure rapeseed oil. In the case of rapeseed oil the chromatographic behaviour of the imported variety was also observed. Levels of 5, 10, 20, 25, 30 and 50 per cent. of rapeseed in mustardseed oil were examined. It appears from Table I that the routine

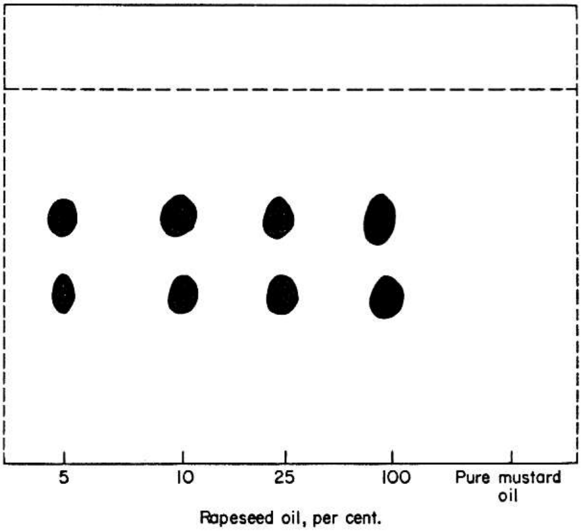

Fig. 1. Chromatograms of varying rapeseed oil levels added to mustardseed oil. In the case of pure mustard oil, no brilliant orange-red spots were observed.

TABLE I

Sample	Smell	Saponification value	Iodine value	R_F value of the orange spots observed with u.v. light
Pure mustard oil (20 samples examined)	Pungent smell characteristic of mustard oil	168–177	96–110	No such spots observed in case of mustard oil
Pure rapeseed oil (20 samples including imported variety examined)	Putrid smell characteristic of rapeseed oil	166–190	94–120	Two brilliant orange red spots observed with R_F values (1) 0·45; (2) 0·65
5 per cent. rapeseed oil	Smell of mustard oil	171–176	98–101	0·44 (1) 0·65 (2)
10 per cent. rapeseed oil	Smell of mustard oil	173–177	98–102	0·45 (1) 0·65 (2)
20 per cent. rapeseed oil	Faint old smell of mustard oil	174–177	99–105	0·46 (1) 0·65 (2)
25 per cent. rapeseed oil	Slight old smell of mustard oil	176–180	101–108	0·47 (1) 0·66 (2)
30 per cent. rapeseed oil	Old smell of mustard oil	177–181	103–109	0·45 (1) 0·66 (2)
50 per cent. rapeseed oil	Smell of rapeseed oil	180–182	106–112	0·48 (1) 0·67 (2)

saponification and iodine value determinations fail to establish the presence of even 25 to 30 per cent. of rape oil in mustard oil, whereas the chromatographic procedure can detect, with positive reliability, as low as 5 per cent. adulteration of mustard oil with rapeseed oil. The chromatographic method was tried with 10, 20 and 30 per cent. of HCl in water. It was found that the best separation of the two spots characteristic of rapeseed was obtained using HCl/water 40 : 60 as the solvent. It should be borne in mind that R_F values are influenced by factors such as the exact composition and age of the solvent, the quality of the paper and the room temperature; so a slight variation in the R_F value is not impossible. To compensate for these factors responsible for variation it is suggested that the R_F value with pure rapeseed oil be obtained under experimental conditions and this then compared with that of the sample.

The author thanks Mr P. K. Das, M.Sc. (London), D.I.C., Public Analyst to the Government of Assam, India for his valuable advice and constructive interest in this investigation.

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The Rapid Determination of Dextrose Equivalent of Glucose Syrups

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A rapid method is described for the determination of dextrose equivalent of glucose syrups. The method is based on the fact that osmotic pressure of glucose syrups is directly proportional to the D.E. of the syrup at constant refractive index. Calibration graphs have to be drawn initially but each determination thereafter takes only 2–3 minutes.

Glucose syrups or starch hydrolysates are conveniently characterised by the term Dextrose Equivalent, or D.E., which is the reducing sugar content expressed in terms of dextrose and calculated as a percentage of the total solids. It bears no relation to the actual glucose content of the product but is directly related to the total reducing power of all the sugars present towards Fehling's solution. D.E. is commonly measured by the Lane and Eynon titration based on the reduction of a copper salt[1] and, although an old method, this is still the standard by which others are judged. Many methods have been reported in the literature, mostly based on the gravimetric and volumetric reduction of copper[2] and one example which was based on the formation of a coloured complex of chelated copper[3].

The Lane and Eynon titration for reducing sugars is probably most commonly used and is also the method most likely to produce inaccurate results unless the method has been used extensively by the operator. Even with an experienced operator, the time needed for solution preparation and titration is seldom less than 15–20 minutes.

Recently, hydrogenated glucose syrups have been produced[4] and characterisation of these syrups is difficult since they possess no reducing ability and hence, in theory, no D.E. They can, however, be conveniently characterised by their "hypothetical D.E." which may be defined as "the D.E. of the glucose syrup possessing the same carbohydrate composition as the hydrogenated syrup". Obviously methods based on reducing capacity cannot measure this hypothetical D.E. and thus a method has been devised whereby the D.E. of any glucose syrup, whether hydrogenated or not, can be determined.

Materials

Glucose syrups were provided by Corn Products Ltd., Manchester, England, courtesy of Dr D. Howling.

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EQUIPMENT

Refractometer. Abbé 60 Refractometer, Bellingham and Stanley Ltd, London, accurate to 0·0001 units and using a light source of the same wavelength as the sodium D-line. All measurements were made at 20°C. Scale readings of the instrument were initially taken and converted to refractive index by reference to tables supplied with the instrument, but to simplify the method, scale readings alone were used.

Osmometer. Advanced Digimatic Osmometer, Model 3D, Advanced Instruments Inc., Needham Heights, Mass., U.S.A.

Experimental

The method is based on the experimental observation that the osmotic pressure of a glucose syrup before and after hydrogenation is not significantly different[4] and the fact that the osmotic pressure of a glucose syrup is directly proportional to the D.E. of that syrup.

METHOD

Standard Graphs

Make up approximately 20 per cent. w/v solutions of about ten glucose syrups (over the range 10–100 D.E.) and determine the refractive index (or scale reading) and osmotic pressure of each. Dilute each sample to approximately 18 per cent. w/v and redetermine refractive index and osmotic pressure. Repeat this procedure over the range 5–20 per cent. w/v solids for each sample.

Plot a graph of osmotic pressure *vs.* refractive index (or scale reading) for each syrup as shown in Figure 1.

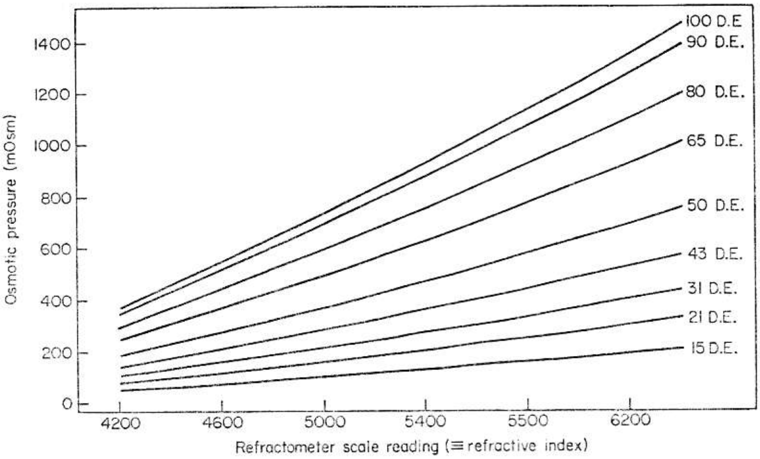

Fig. 1. Osmotic pressure *vs.* refractometer scale reading for each glucose syrup.

Extrapolate from this graph values of osmotic pressure at selected values of refractive index (or scale reading) and redraw the data as osmotic pressure *vs* D.E. as shown in Figure 2. (Best fit regression lines, x upon y, are determined

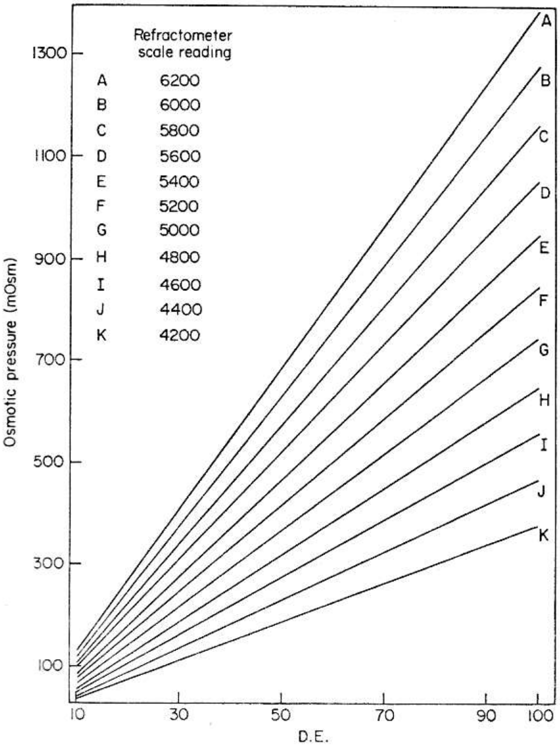

Fig. 2. Osmotic pressure *vs.* D. E. at each refractometer scale reading.

for each set of data and these alone are shown in the figure which is then used as a standard for further determinations.)

For an unknown sample, dilute the syrup or make up the solid to a suitable concentration such that the refractive index falls within the range used for the calibration graphs. Measure the refractive index (or scale reading) and osmotic pressure and read the D.E. from the standard graph.

Results and Discussions

A second graph is drawn (Figure 2) because although Figure 1 could be used as the standard graph there are differences between syrups which lead to inaccuracies. The distances between the curves, in Figure 1, for the syrups are not constant for a given number of D.E. units. The second graph straightens out these lines and although slight inaccuracies are therefore built into the system it compares favourably with the results obtained using the Lane and Eynon titration. Table I gives the results of D.E. measurement of a series of glucose syrups using the Lane and Eynon titration and the refractive index/osmotic pressure method.

Assuming the D.E. measurements by the Lane and Eynon method to be correct, it can be seen that the greatest inaccuracy overall is better than ±2 D.E. units.

TABLE I

DETERMINATION OF D.E. OF GLUCOSE SYRUPS

D.E. by Lane and Eynon titration	D.E. by osmotic pressure/refractive index method	
	1	2
15	15	15
21	21	22
31	29	29
39	38	38
40	38	39
50	50	50
65	66	67
79	80	78
90	92	91
100	98	99

Whilst this new method undoubtedly saves time, the equipment used is rather expensive and this cost would have to be offset by ensuring that the equipment was used to its fullest. In many laboratories, osmometers are used for other purposes so their use would merely be extended. The method could find use industrially or be adapted for automatic analysis on an industrial or laboratory scale.

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Survey of Fungicide (Thiocarbamate) Residues in Lettuce. A Report on Behalf of the Association of Public Analysts

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This Report gives the results of a survey carried out during the period 1 September, 1976–30 April, 1977.

It was planned by the Association of Public Analysts following the publication of the Report on a Survey of Pesticide Residues in lettuce covering the period 1 July, 1974 to 30 June, 1975 and at the request of the Ministry of Agriculture, Fisheries and Food.

This later survey has shown that during the period involved, 58·7 per cent. of lettuces contained significant amounts of residues of thiocarbamate fungicides.

The results of an earlier survey on lettuce, covering the period 1 July, 1974–30 June, 1975,[1] showed that 41·6 per cent. of the samples examined contained significant amounts of pesticide residues and that such residues were much more evident during the six months from October to March than at other times of the year.

This earlier survey showed, also, that, as a group, thiocarbamate fungicides were the most frequently occurring residues, being present in 29·9 per cent. of the samples. The amounts found were considered to be excessive in many instances, often in excess of 10,000 ng/g. One sample was found to contain 504,000 ng/g (0·05 per cent.).

Following the publication of the Report on this earlier survey,[1] the Ministry of Agriculture, Fisheries and Food modified their advice upon the use of thiocarbamate fungicides to the extent of increasing the period of time between the treatment and harvesting of lettuces from 7 to 14 days. The present survey was carried out to assess the effect of these revised instructions for use.

In order to effect some economy of effort it was agreed that the survey would be limited to thiocarbamate fungicides and cover a shorter period than in the earlier survey. The survey started one month before the 6 month period of heaviest use indicated by the earlier survey[1] and concluded one month after that period, a total of eight months.

Arrangements were made by the individual members of the Association with the Enforcement Officers of their Local Authorities for samples of lettuce to be included in the normal sampling programmes of the participating

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TABLE I

THIOCARBAMATE FUNGICIDE RESIDUES IN LETTUCE, 1 SEPTEMBER 1976–30 APRIL 1977

Zone 1 Samples examined 81 Samples containing thiocarbamate 42		Zone 2 Samples examined 55 Samples containing thiocarbamate 33		Zone 3 Samples examined 91 Samples containing thiocarbamate 54				Zone 4 Samples examined 97 Samples containing thiocarbamate 73			
Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin
560	c	1,260	c	560	c	19,500	a	560	c	18,100	c
560	c	1,260	a	610	c	23,900	a	700	c	19,500	c
700	c	1,670	c	630	c	25,100	a	980	c	19,500	c
700	c	2,300	a	700	a	28,700	a	1,400	c	20,400	c
840	c	3,490	a	960	a	60,000	a	1,600	c	20,500	c
840	c	3,630	c	1,440	c	68,400	a	2,400	c	20,900	c
840	c	3,770	a	1,450	b	73,900	a	2,510	c	20,900	c
1,720	c	4,740	a	1,450	a	97,700	a	2,650	c	22,300	a
1,810	a	5,290	c	1,700	a	116,000	a	2,790	c	23,700	c
2,230	a	5,310	c	1,700	a	158,000	a	2,800	c	29,000	c
2,790	a	7,560	c	1,760	c	307,000	a	3,500	c	32,100	c
3,770	c	8,370	b	1,870	a	307,000	a	3,800	c	36,300	c
5,160	a	8,790	a	1,900	a			3,800	c	39,800	c
5,580	c	9,770	a	1,950	a			4,190	c	40,000	c
6,560	c	9,770	a	2,060	a			4,200	c	50,000	c
7,950	a	9,900	a	2,090	a			4,300	c	52,900	c
8,790	c	10,200	a	2,090	a			4,800	c	53,700	c
9,070	c	10,500	a	2,090	c			4,800	c	54,900	c
9,900	c	11,300	a	2,120	a			5,000	c	56,000	c
10,200	c	15,300	a	2,290	c			5,100	c	59,000	c
10,200	c	15,700	c	2,340	a			5,580	c	59,100	c
10,200	a	18,100	a	2,370	a			5,580	c	60,000	c
10,300	b	19,500	a	2,960	a			5,580	a	85,200	c
10,500	c	23,900	a	3,140	c			6,200	c	110,000	c
11,000	a	25,100	a	3,530	b			6,500	c	130,000	c
11,900	c	28,700	a	3,910	a			7,800	c	140,000	c
12,000	c	60,000	a	4,520	c			8,200	c	162,000	c
12,700	a	68,400	a	4,740	a			8,200	c	180,000	c
14,000	a	73,900	a	5,290	c			8,370	c	190,000	c
14,100	c	97,700	a	5,310	c			8,370	a	220,000	c
15,100	c	116,000	a	7,560	c			9,770	c	230,000	c
17,900	a	158,000	a	8,370	b			9,800	c		
24,800	a	307,000	a	8,790	a			10,000	c		
27,900	c	307,000	a	9,770	a			12,000	c		
27,900	c			9,770	a			12,000	c		
30,700	a			9,900	a			12,800	c		
46,000	a			10,200	a			15,200	c		
50,200	a			10,500	a			16,700	c		
95,000	a			11,300	a			16,700	c		
174,000	c			15,300	a			16,700	a		
990,000	a			15,700	c			17,000	c		
2,230,000	a			18,100	a			17,800	c		

TABLE I (*continued*)

Zone 5 Samples examined 128 Samples containing thiocarbamate 79				Zone 6 Samples examined 127 Samples containing thiocarbamate 75				Zone 7 Samples examined 66 Sample containing thiocarbamate 23		Zone 8 Samples examined 8 Sample containing thiocarbamate 4	
Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin	Amount of thiocarbamate, *ng/g*	Origin
840	c	27,200	c	840	a	20,900	c	1,395	b	1,810	c
840	c	28,600	c	840	a	21,600	a	1,530	c	2,090	c
900	c	29,800	c	980	c	22,300	a	1,740	c	6,560	c
980	c	30,000	c	1,400	a	23,700	b	1,950	a	30,700	c
1,000	c	33,000	c	1,670	c	25,100	a	2,230	c		
1,400	c	33,500	c	1,740	c	30,700	c	2,930	a		
1,950	c	35,300	c	2,230	a	31,400	a	3,490	a		
2,000	c	36,300	c	2,440	c	34,200	a	6,840	c		
2,100	c	43,000	c	2,930	a	35,600	c	6,980	c		
2,650	c	44,000	c	3,350	c	39,100	a	8,370	c		
2,700	c	48,000	a	3,490	c	41,900	a	9,350	a		
2,900	c	52,000	c	3,770	a	44,100	a	9,770	a		
3,100	c	52,000	c	5,580	b	44,600	a	10,200	a		
3,350	c	54,000	c	6,280	c	46,000	a	12,800	c		
4,000	c	54,000	c	6,420	a	47,400	a	16,700	a		
4,600	c	54,400	c	6,980	a	58,600	a	20,900	a		
5,200	c	55,000	c	8,720	c	61,900	c	27,900	a		
5,300	c	58,000	a	8,860	c	63,500	c	41,900	a		
5,580	c	58,900	c	9,210	a	72,300	a	51,600	c		
5,580	c	63,500	c	9,210	a	83,700	b	53,000	a		
6,280	c	63,500	c	9,770	a	87,900	a	133,000	a		
6,420	c	69,800	c	9,770	c	97,700	a	195,000	a		
7,250	c	95,100	c	11,200	a	103,000	a	1,050,000	a		
7,400	c	96,300	c	11,900	c	123,000	a				
7,950	c	136,000	c	11,900	c	142,000	a				
8,090	c	141,000	c	11,900	c	153,000	c				
8,600	c	150,000	c	12,200	c	173,000	a				
11,700	c	150,000	c	13,300	c	306,000	a				
12,000	c	154,000	c	16,400	a	321,000	c				
12,400	c	159,000	c	16,600	a	328,000	b				
13,100	c	159,000	c	16,700	a	446,000	a				
13,500	c	167,000	c	17,600	c						
13,800	c	227,000	c	17,800	c						
14,000	c	249,000	c	18,000	a						
15,200	c	249,000	c	18,000	a						
16,300	c	403,000	c	18,100	a						
18,000	c			18,100	a						
18,000	c			18,300	a						
18,100	c			18,300	c						
18,300	c			18,500	a						
20,000	c			18,800	c						
20,900	c			19,500	a						
21,000	c			20,900	a						
26,000	c										

Authorities and the ready co-operation of the Enforcement Officers and their respective Authorities is gratefully acknowledged.

Organisation

The survey was organised on lines similar to those of the earlier survey on lettuce[1], which, in turn, was based on the previous surveys carried out by the Association of Public Analysts[2,3,4].

The participating authorities were as follows:

Zone	Authorities
1	London Boroughs of Barking, Croydon, Hammersmith, Harrow, Kingston, Kensington and Chelsea, Lambeth, Merton, Richmond, Sutton and Wandsworth.
2	Counties of Kent and Surrey.
3	Counties of Dorset, Dyfed, Mid-Glamorgan, Powys, Somerset, City of Cardiff, Borough of Vale of Glamorgan.
4	Counties of Durham, Humberside, Northumberland, South Yorkshire.
5	Counties of Cheshire, Clwyd, Gwynedd, Lancashire, Manchester, Merseyside.
6	Counties of Hereford and Worcester, Leicestershire, Staffordshire, West Midlands.
7	Counties of Cambridgeshire, Essex, Hertfordshire, Norfolk, Suffolk.
8	N. Ireland: Antrim, Armagh, Coleraine, Londonderry, Newtownabbey.

Sampling

The Sampling Officers were asked to give preference to lettuces grown in the United Kingdom.

Reporting Limit (Significance)

As in the previous survey[1] the reporting limit was set at 500 nanogrammes/gramme as ethylene bisdithiocarbamic acid, i.e. amounts of less than this quantity were regarded as not significant and reported as "nil".

Method of Preparation of Sample and Analysis

Before analysis, the root, if present, was removed and discarded together with any obviously inedible leaves.

The lettuces were not washed, unless there was contamination by soil, and then washing was kept to a minimum.

Thiocarbamates were determined by the procedure given in the Report of the Third Survey[4]. In this method the thiocarbamate was converted to carbon disulphide by acid hydrolysis, isolated by distillation and aborbed into a diethanolamine reagent containing copper. The resultant yellow colour was then related to standards prepared from carbon disulphide.

For the purpose of the Report, the results, in terms of CS_2, have been converted to ethylene bisdithiocarbamic acid on the basis that one molecule of ethylene bisdithiocarbamic acid yields two molecules of CS_2:

$$(CH_2\,NHCSSH)_2 \rightarrow 2\,CS_2.$$

Results

The amounts of pesticide found, Table I, are given as nanogrammes of ethylene bisdithiocarbamic acid per gramme of lettuce in its fresh natural state.

The origin of the samples in Table I is indicated by:

(a) United Kingdom;
(b) imported;
(c) origin unknown.

It should be noted that because of the variability of recovery rates for different fungicides of the thiocarbamate group, from less than 50 per cent. to approximately 100 per cent., none of the experimental findings have been corrected for recovery and all the results must, therefore, be regarded as minima. Laboratories were not asked to identify the particular members of the thiocarbamate group found, but there were reported 30 instances of the discovery of Thiram and 3 instances of the discovery of Maneb.

It is emphasised that the survey was confined to thiocarbamates and that a finding of "Nil" does not exclude the possibility that other residues may have been present. One laboratory did, however, look for other pesticides and found various amounts of BHC, up to 400 ng/g.

Table II summarises the results given in Table I.

TABLE II

ORIGIN OF LETTUCE

Country of origin	Number of samples	Samples containing thiocarbamate	Percentage of samples containing thiocarbamate
U.K.	239	144	60·3
Imported	28	11	39·3
Unknown	386	228	59·1
Total	653	383	58·7

Total number of samples examined = 653
Samples containing thiocarbamates above the reporting limit of 500 ng/g = 383

Laboratories were asked to record any information concerning the methods of growing, but in the event such information was available on only 40 samples. This information is summarised in Tables III and IV.

TABLE III

SOURCE OF LETTUCE EXAMINED IN THE SURVEY

	Number of samples	Thiocarbamates absent†	Thiocarbamates present
Grown under glass	31	13	20 (64·5 per cent. of total)
Grown in open	9	6	3 (33 per cent. of total)
Total	40	17	23

The method of growing 40 samples was known: 33 were grown in the United Kingdom and 7 imported.

†Thiocarbamates not detected, or less than the reporting level of 500 ng/g.

TABLE IV

THIOCARBAMATE RESIDUES FOUND IN LETTUCE UNDER KNOWN GROWTH CONDITIONS

	Origin	Thiocarbamates, *ng/g*
(a) Grown under glass	All samples originated in the U.K.	700
		1,810
		2,230
		2,790
		5,160
		8,370
		8,790
		11,000
		12,700
		14,000
		17,400
		17,900
		24,800
		50,200
		73,900
		94,900
		97,700
		307,000
		707,000
		990,000
(b) Grown in open	All samples originated in the U.K.	4,120
		7,950
		2,230,000

Conclusions

This survey shows that during the period 1 September, 1976 to 30 April, 1977, 58·7 per cent. of lettuces contained significant amounts of residues of thiocarbamate fungicides, compared with the 29·9 per cent. of lettuces reported in the survey carried out over the whole of the 12 months period July 1974 to June 1975 and the 44·8 per cent. of lettuces sampled over the period October 1974 to March 1975, the nearest comparable period.

The results suggest that such residues are most likely to be found on lettuces grown under glass, although it is noted that the highest amount found, 2,230,000 ng/g, was found on a lettuce grown in the open.

References

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Book Reviews

DEVELOPMENTS IN FOOD ANALYSIS, VOL. 1. TECHNIQUES. Edited by R. D. KING. London: Applied Science Publishers Ltd., 1978. Price £25. ISBN 0 85334 755 7.

This book, as its title indicates, traces developments, past and recent, in the techniques of food analysis. There are eleven authors, all from very good stables, and this is amply reflected in the quality of the text.

In such a modern book, the reader will expect a heavy emphasis on instrumental techniques and in this respect will not be disappointed to find chapters on HPLC, GC, ion-selective electrodes, automation and AA. Presumably future volumes will cover developments in other instrumental techniques. At the same time, the editor has blended in a few chapters on more classical methods of analysis, such as the determination of nitrogen and carbohydrates. This will undoubtedly provide some familiar and well-trodden ground for many of the more senior members of the analytical profession and might tempt them to inquire into the chapters dealing with the newer instrumental procedures.

The ten chapters are each prefaced by summaries of varying lengths and sprinkled liberally with references which will enable the reader to probe the various subjects more deeply.

The first chapter deals quite effectively with modern chemical methods for the estimation of vitamin A and its precursors, the B group, the D group, vitamins C, E and K and its authors introduce the subject by discussing the nomenclature and the latest position on the expression of results.

There follows a compact and useful chapter on the determination of nitrogen which begins with the Kjeldahl process, the choice of catalyst and the use of block digesters. Modifications to the chemistry of the digestion stage and automation are discussed. Various other alternative techniques are considered but analysts might be disappointed to find little discussion of the Tecator process.

Any reader who, as a student, had a morbid dread of thermodynamics might prefer to treat the chapter on the role of water present in foodstuffs like the curate's egg, and savour only those parts free from differential calculus. For those with the stomach to digest the whole text, it is a comprehensive and well presented dissertation, but stands out from other works in the book by being a little too academic.

The advantages of HPLC are ably demonstrated in the next chapter and many applications of this rapidly expanding technique are discussed. This chapter is followed by a parallel survey of the present trends in gas chromatography which includes the move to capillary columns, headspace analysis and the use of specific detectors. Computer interpretation of GC traces is briefly described.

A chapter on enzymatic methods of analysis provides a sharp contrast half way through the book, and traces the application of a large number of highly purified enzymes to detect and assay sugars, organic acids, amino acids, alcohols, sterols, triglycerides, nitrate and phosphate.

There follows a review of the application of ion-selective electrodes in the analysis of food with summaries of the more important methods. The tables and graphs shown in the text illustrate the usefulness of the technique and references are ample.

The philosophy of automation in the field of analysis is discussed in the eighth chapter together with its economic and scientific connotations. Its application at the Laboratory of the Government Chemist is described and illustrated by specific examples of its use. The chapter concludes with some of the more recent automatic methods of food analysis.

The chapter on carbohydrates analysis is a workmanlike summary of the preparation of food samples for analysis, physical and chemical methods of analysis and enzymatic procedures. The text brings the reader right up to date by leading him through paper chromatography, GC and TLC, to HPLC methods.

The volume finishes in a blaze of monochromatic light when the application of the technique of atomic absorption spectroscopy to food analysis is concisely dealt with. The construction of the spectrophotometer is discussed and the newer variations such as graphite furnaces are mentioned. The metals of particular significance and interest to the food analyst are listed.

The book itself is pleasantly set out, well but not gaudily illustrated with photographs, graphs, diagrams and tables. Inevitably the book will date and one cannot say that it is a "must" for the library shelf, but it does summarise the present state of development in food analysis techniques and provides the reader with a valuable platform for further enquiry.

D. J. TAYLOR

DETERMINATION AUTOMATIQUE DE LA DEMANDE CHIMIQUE EN OXYGEN DES EAUX.

This is a research publication of the Fondation Universitaire Luxembourgeoise published at Arlon in the Belgian Province of Luxembourg. The booklet is of 28 page length, quarto size and well printed on semi-glossy paper. Pierre Worzee, who designed the method described here, has also published works on other automatic methods for water analysis. He writes, of course, in French and the publication follows traditional lines, i.e. brief discussion of methods already published, proposed method, diagrams of equipment etc. and graphs showing comparison of methods. A few brief words give the advantages of the method described and there is a short bibliography; in turn this is followed by a "Table Des Matières". No price is quoted for the booklet.

It is claimed that the method can be applied to unfiltered liquids and chlorides have less effect than in other methods.

In brief, a small sample is oxidised with bichromate-sulphuric acid at 165°C using silver sulphate and mercuric sulphate as catalysts; the trivalent chromium following the reaction is measured at 600 mu.

On filtered liquids a precision of better than 3 per cent. is claimed and fewer reagents and manipulations are used, whilst the time of oxidation is also reduced.

This does appear to be a useful method; it could doubtless be introduced into routine analysis even if non-automated, and hence is worth a trial.

G. V. JAMES

SALMONELLA—THE FOOD POISONER. A Review by a Study Group of the British Association for the Advancement of Science. Price £1 from the Press Office, B.A.A.S., 23 Savile Row, London W1X 1AB.

This is a report of 49 pages which has been collated over three years by a study group including veterinary surgeons, a farmer, a former Public Health Laboratory Director, etc. It is claimed to have been written with a wide-ranging public in mind and hence is in layman's language, although with a scientific foundation.

Statistics record a high incidence of the disease in humans, it being one of a large group of diseases transmissible between animals and humans. According to figures quoted, the disease is on the increase and in turn this may be related to the use of intensive methods of livestock management, designed to produce more meat for human consumption at lower costs.

After discussing food poisoning in humans, animals, legislative control, etc. there is a short summary of constructive steps to be taken especially by [I presume] members of the public, in their education to handle meat and poultry products and avoid *Salmonella* infection.

The figures quoted in the tables are of interest as they apply only to England and Wales so we can only deduce that the disease does not occur in Scotland or that, if it does, the Scots wife already knows how to handle it. This, despite the penchant to haggis consumption which a layman might consider as being a suitable substrate for *Salmonella*.

The other thought might be that the Scots are protected by their national beverage – whisky.

The pamphlet is well produced and interesting but whether it is worth having in the library of a Public Analyst's Department is a moot point.

G. V. JAMES

POTENTIOMETRIC WATER ANALYSIS. By D. MIDGLEY and K. TORRANCE. Pp. 409. John Wiley & Sons, 1978. Price £16.50.

Books on the subject of water appear at an ever increasing rate, but this one is different. It is a treatise on a specialist subject and satisfies the need for the collection together in one volume of the mass of information on potentiometric techniques in analysis. The title does not do justice to the subject matter although it is difficult to think of a better one.

Nearly a third of the book is devoted to the theory, principles and equipment involved. Much space is given to electrodes, particularly ion-selective ones, and there appears a very useful appendix giving the names and addresses of manufacturers. Statistics is given its proper treatment without overdoing it and the whole balance is just right.

The major part of the book is devoted to analytical methods and each method is the subject of a critical appraisal of its usefulness and is compared with alternative techniques. This part of the book forms a very valuable source of reference and contains an adequate bibliography.

The whole book is well written in a very readable manner and is to be recommended to all analysts whether specialising in water analysis or not.

R. SINAR