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The Identification of Bulk Asbestos and Monitoring of Airborne Fibre. Part II: Monitoring

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Muter and Hackman, Kennington, London

The literature on monitoring of airborne asbestos fibre is reviewed. The method used and results over a period are described, and a discussion is given of some of the problems involved in fibre counting.

Literature Review

An excellent account of the monitoring and identification of airborne asbestos has been given by Beckett[1]. The most widely used monitoring method is the membrane filter method, which involves drawing a known volume of the air to be sampled through a membrane filter using an electric or hand-operated pump. The method has been well described in Technical Notes 1 and 2 published by the Asbestosis Research Council[2,3] and in Millipore literature[4].

In the ARC method[2], white, gridded membrane filters of 25 mm diameter, and 0·8 μm pore size are recommended. The sampling flow rate should not exceed 2 litres/min, and sample volumes from 20 litres to 2 litres or less are used, depending on the anticipated concentration of asbestos dust. Technical Note 2 distinguishes snap samples (few seconds to 30 seconds), 10-min samples (initial assessments), and 4-h sampling periods, to provide an indication of the level of airborne dust to which an employee has been exposed during his working day[3]. The ARC method[2] also recommends fixing the dust deposit on the membrane surface using Perspex solution or a cytological fixative, and finally mounting the filter with triacetin (glycerol triacetate) to clear it. The filter should be left for approximately 30 min before counting. All fibres >5 μm in length and $\leqslant 3$ μm in diameter, with an aspect ratio (length:breadth) of at least 3:1 are counted, using a magnification of $\times 500$ and phase contrast, until about 200 fibres or alternatively 100 fields have been counted.

The fibre concentration is then calculated from:

$$\text{Fibre concentration (F)} = \frac{D^2}{d^2} \times \frac{n}{N} \times \frac{1}{V} \text{ (fibres/ml)} \qquad (1)$$

where D = effective diameter of dust deposit on filter (mm)
d = diameter of the field of view, or of the graticule area employed (mm)

This paper together with Part I (*J. Assoc. Analysts*, 1981, **19**, 21–30) was delivered at the Annual Conference of the Association in London in May 1981.

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n=number of fibres counted
N=number of fields examined
V=volume of air sampled (ml)

In this expression, notice that $\frac{D^2}{d^2}$ is the number of microscope fields in the whole filter, and $\frac{n}{N}$ is the mean number of fibres per field.

Other liquids which can be used for clearing membrane filters include a 1:1 solution of dimethylphthalate and diethyl oxalate[4]; cyclohexanone; dioxan+5 per cent. water; cinnamaldehyde[1]; and immersion oil. It is also possible to use a penetrating oil[5].

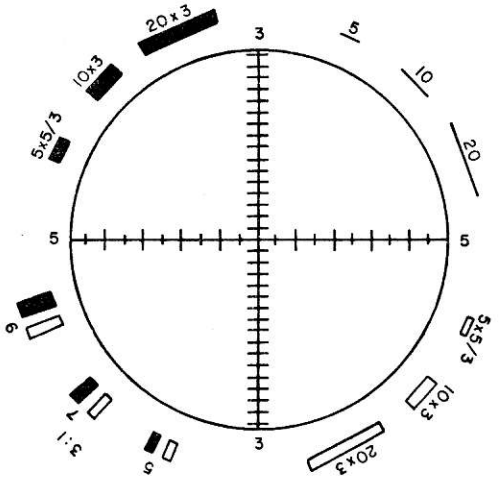

Fig. 1. "Walton and Beckett" eyepiece graticule for the evaluation of airborne fibre (Reproduced from *Ann. Occup. Hyg.*, 1977, **20**, 21 by permission of the British Occupational Hygiene Society).

Use of an eyepiece graticule, such as the Walton and Beckett graticule[6] (Figure 1) is very helpful in judging the lengths and diameters of fibres, but it is essential that these graticules be used with the optics for which they are designed. The veracity of measurements indicated by the graticule should be checked with a stage micrometer. Fibre recognition can sometimes be difficult, and the operator may encounter split fibres, fibres with particles attached, wedge-shaped fibres and many other difficult cases. An Australian guide[7] has given diagrams of different fibre types with instructions about counting them, but Beckett[1] points out that as yet no internationally agreed standards exist.

Several statistical studies have been made of the accuracy achievable in manual counting. Coefficients of variation of $\pm$20 to 40 per cent. are achievable within a single laboratory, but inter-laboratory agreements are much poorer[8]. The second interim report of the Simpson Committee[9] suggests that, even with good microscope equipment and techniques, fibre counting errors may be of the order of $\pm$50 per cent. To minimise such errors,

some authorities recommend that no more than about 10 samples should be manually counted by an operator in any one day. Although the lower limit of detectability may be of the order of 0·0005 fibres/ml for a 1-h sample, the ARC method gives only a rough estimate below 0·01 fibres/ml, and low results should be expressed as $<0{\cdot}01$ fibres/ml.

The Public Analyst who undertakes asbestos monitoring is generally faced with the exposed filter, which may be received in the original filter holder, or in a small round tin or plastics container. The method used in this laboratory, results obtained, and problems encountered will now be described.

Apparatus and Reagents

1. *Phase contrast microscope* with built-in illumination, and with $\times 10$ and $\times 40$ negative phase contrast objectives, condenser with appropriate phase plates, $\times 12{\cdot}5$ wide field eyepieces, one with *Walton and Beckett graticule*, and *focussing telescope* for phase ring alignment.
2. *Stage micrometer* for field diameter measurement.
3. *Glycerol triacetate* (*Triacetin*), membrane filtered if necessary.
4. 3×1 *inch microscope slides*, and 24×24 mm, No. $1\frac{1}{2}$ coverslips (solvent cleaned).
5. *Micropipette* delivering 100 μl.
6. *Fine pointed forceps.*
7. *Heating block* 0—100°C.

Method

Place one or two drops of triacetin (or deliver 100 μl) on to a clean microscope slide, and spread it out into a circle of the same diameter as the filter (25 mm). Remove the filter from the container using forceps and lay, dust side up, on top of the triacetin, with the filter grid lines parallel to the slide edges. It should be arranged that the amount of triacetin used just wets the whole of the filter. Cover with a square No. $1\frac{1}{2}$ coverslip and press down gently to remove air bubbles. Leave for 15-30 min to clear, or preferably place the slide on top of a heating block at about 60°C to clear within about 1 min. If the filter is not properly wetted, place a small drop of triacetin on the filter before the coverslip is added, but care is necessary, particularly with loosely attached deposits, in case the fibres are washed to the coverslip boundary.

First scan the filter using the low power, phase contrast objective ($\times 10$) to assess the fibre distribution. If the distribution is reasonably random, then using the high power, phase contrast objective ($\times 40$)* and a magnification of $\times 500$, count all fibres >5 μm in length, $\leqslant 3$ μm in diameter, and with length: breadth $\geqslant 3:1$ until 200 fibres or 100 fields have been counted. Calculate the fibres/ml from equation (1). It has been found that the normal setting of the substage condenser (to focus the field iris) does not always give the best phase contrast, but optimum contrast can be achieved by slight re-focussing of the condenser.

*Negative phase contrast shows the fibres much more clearly than positive phase contrast.

TABLE I

TABULAR RECORD OF PROGRESS OF A TYPICAL ASBESTOS FIBRE COUNT

				Total
Number of fields	5	5	5	100
Number of fibres	3	4	1	

The number of fibres in 100 fields can be added mentally while the count is made, or alternatively, if the count is high a tabular record can be filled in as the count proceeds, as shown in Table I. The number of fibres/five fields can usually be easily memorised, and then recorded as shown until 20×5 fields have been counted. In the case of heavily contaminated filters e.g. taken on the dirty side of the screen in asbestos removal, up to 80 or more fibres/field may be found; in this case the counts for one field at a time are tabulated until a representative count has been assembled.

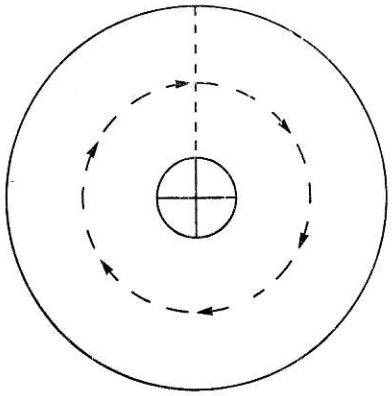

Fig. 2. Systematic sweep of a heavily contaminated field.

Where a large number of fibres/field are to be counted, and a Walton and Beckett type graticule is in use, it is helpful to sweep the outer annulus from 12 o'clock round to 12 o'clock, and finally to add the fibres in the centre circle (Figure 2). This prevents double counting of fibres, since the eye is guided round a narrow channel. Some workers count only fibres in the centre circle, but this necessitates a multiplying factor $\left(\frac{D^2}{d^2}\right)$ of 30,000 or more, compared with 2000 to 6000 where whole fields are counted. There are arguments in favour of both procedures. A practical compromise is provided by the following rules:

If the mean number of fibres/centre circle is:

(1) greater than 2, count sufficient centre circles to give 200 fibres or just over this figure;
(2) from 2 to $\frac{1}{2}$, count 100 centre circles;

(3) from $\frac{1}{2}$ to $\frac{1}{10}$, count 25 to 100 whole fields to give 200 fibres or just over this figure;
(4) less than $\frac{1}{10}$, count 100 whole fields.

If a filter has an uneven distribution, i.e. most fields with a low count, but a few fields with a very high count, an approximate figure for the fibre concentration can be calculated from:

$$\text{Fibre concentration (F)} = \left[\left(\frac{D^2}{d^2} - f\right) \times \frac{n}{N} + H\right] \times \frac{1}{V} \text{ (fibres/ml)} \quad (2)$$

where f = number of fields with very high counts
H = number of fibres in these high count fields
n = number of fibres in the low count fields
N = number of fields with low counts
V = volume of air sampled (ml).

However, the count should follow the velocity profile across the filters, and provided that only just sufficient triacetin to wet a filter is used, the fibres should not flow to the edges or to a particular region, unless the filter holder is leaking at some point in the periphery, in which case the count is meaningless. A speck of dust containing 100 or more fibres could alight on the filter, scattering the fibres over a minute localised area; in this case equation (2) would be applicable.

TABLE II

APPEARANCE GRADING OF AIR FILTERS

Grade	Dry appearance (Macro observation)	Amount of grit (Micro observation)
1	Cleanish	Few particles
2	Light grey/brown slightly dusty	Slightly gritty
3	Dirty/dusty	Gritty
4	Very dirty/ very dusty	Very gritty

To provide additional information, place the filter in one of four or more grades (Table II) according to its initial appearance in the container, or, more important, the number of grit particles seen on first examination through the microscope.

Results and Discussion

The results obtained on over 600 of the many filters received for counting have been analysed. Flow rates ranged from 2 litres/min to 3 litres/min, flow times from 10 min to 240 min, and total air volumes from 20–720 litres.

Some of the filters came from the "dirty" side of the screen surrounding a site during asbestos removal, and some from the "clean" side. The dirty filters usually lie in the range 0·2–10 fibres/ml, and the highest count recorded

here so far has been 7 fibres/ml; the clean side filters are generally much lower than this (<0·02 fibres/ml), indicating good working practices. Some authorities like to keep concentrations below 0·02 fibres/ml, since this represents $\frac{1}{10}$ of the limit recommended for crocidolite in the Simpson report.

Monitoring errors are made up of sampling variance, "between-fields-within-slides" variance, and operator variance. Data studied by Ogden[10] have shown that variability from the last two of these sources depends on the number of fibres counted, and that within a single laboratory the upper 95 per cent. confidence limits when 1,2,5,10,20,50 fibres counted was given approximately by multiplying the fibre concentration by factors of 4·5, 3·1, 2·2, 1·9, 1·6 and 1·5 respectively*. Above 50 fibres the counting accuracy did not greatly improve. This gives a rough idea of the confidence associated with counts in one particular laboratory, but does not take into account inter-laboratory variance.

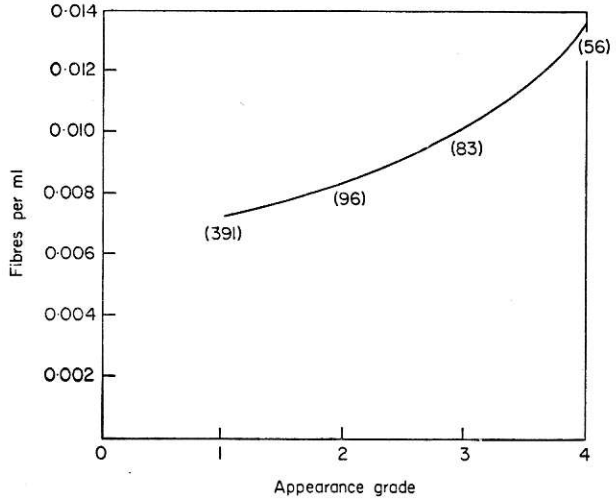

Fig. 3. Correlation between fibre concentration and grittiness.

Most insulation involves both fibre and a certain amount of filler, and in asbestos removal particulate matter as well as fibre becomes airborne. It would be expected, therefore, that a rough correlation might be found between the gritty appearance of a filter and the fibre count, and in Figure 3 the mean fibres/ml found for each filter grading is plotted against the filter grade; the numbers in parentheses indicate the number of filters on which each mean is based. There is an obvious trend, but the correlation depends on the proportions of fibre and filler, and caution is necessary as individual cases may show a little fibre and many grit particles and vice versa. In fact, in extreme cases, there have occurred dirty (i.e. gritty) filters with low fibre concentrations (<0·01 fibres/ml), and cleanish-looking filters with high fibre concentrations up to 7 fibres/ml.

*If the number of fibres counted is multiplied by the factor, there will be a 95 per cent. chance that the mean result for the count will lie at or below the derived figure.

A few problems have occurred in the Authors' experience which are worthy of mention. Air monitoring personnel do not generally fix the deposit on the filter before dispatching it to the analyst, despite the ARC recommendation[2], and some containers have been received with dust-coated membranes and an appreciable amount of loose dust in the container. If the filter is transported to the analyst, not in the filter holder but in a separate container, it is important that the filter be placed in the container with the dust side uppermost, and the use of gridded filters rather than plain filters is recommended, to help to ensure that this is done. "Fixing" is not invariably recommended, and in the absence of loose dust it may be better to rely on the electrostatic attraction between the fibres and the filter.

Occasionally membrane filters are encountered with "high background counts" i.e. which show many "fibre-like" bright streaks throughout the whole depth of the membrane, when viewed by negative phase contrast. These streaks are not in fact fibres, as membrane filters are mainly surface-retaining filters, and only the fibres on or near the uppermost surface of the filter should be counted. The cause of these "in-depth" streaks, which appear bright in negative phase contrast, is not known. A good practice here is to fine-focus downward from any speck of dust on the top of the coverslip to the next plane with particles or fibres in focus; this should be the top of the membrane filter, and this can be checked as further downward focussing reveals the internal granularity of the filter. The second advantage of gridded filters with coloured grid lines is their usefulness in locating and maintaining focus on the filter surface.

Sooner rather than later the analyst is bound to encounter filters that are difficult to assess because of branched/split fibres, entangled fibres, fibre-particle aggregates, and other doubtful cases (for example, wedge-shaped and

TABLE III

FIBRES ELIGIBLE FOR COUNTING*

(>5 μm length; $\leqslant 3$ μm diameter; $\geqslant 3:1$ aspect ratio)	
Count	Do not count
All "size-eligible" fibres where undecided whether asbestos or not	Obviously non-asbestos fibres
Single uniform fibres (Straight, curved and curly)	Crescent or boat-shaped fibres
	Wedge-shaped fibres
Irregular diameter fibres if $\leqslant 3$ μm diameter	Irregular diameter fibres if > 3 μm diameter anywhere
Bent fibres, hooked fibres	Flat, bladed fibres
Branched/split fibres (as one fibre)	Branched fibres > 3 μm diameter anywhere
Tangled fibres up to seven fibres (as seven)	Tangled fibres > 7 fibres
Tight fibre bundles up to seven fibres (as one)	Tight fibre bundles >7 fibres (Usually >3 μm diameter)
Fibre-particles if fibre eligible and length $>$ particle diameter (as one)	Fibre-particles if fibre length $<$ particle diameter
Crossed fibres (as two, three etc)	Right-angled fibres
	Sharp V-shaped fibres
	Dumb-bell shapes
	Cotton-type twisted fibres
	Spiral vessels
	Stellate hairs and obvious vegetable trichomes

*More than half the length must be within the field.

bladed fibres). The original ARC method[2] implies a *total fibre count* on all size-eligible fibres, but this can be misleading if the filter is mainly populated by vegetable fibres. A more critical assessment of what is seen on the filter can help in getting closer to the *asbestos* fibre concentration. To this end a preliminary attempt has been made to evolve some guidelines as to what should and should not be counted, and these are set out in Table III.

Depending on the local environment, material other than asbestos fibres that may be seen on membrane filters includes "grit" particles, plant, animal and synthetic fibres, insect scales, pollen grains, spores, crystals, moisture droplets etc.

Much work is now in progress on several pieces of equipment for automatic asbestos fibre counting. Such equipment is very useful where 100 or more filters per day are to be counted, but as yet such machines cannot compete with the visual cortex where the judgment of difficult cases, such as fibre-particles, entangled fibres etc. is concerned.

Our location and assessment of the literature on identification and monitoring of asbestos has been greatly guided by the two excellent publications by Michaels and Chissick[11] and McCrone[12]. The authors acknowledge helpful discussion with Dr T. L. Ogden and his permission to quote statistical data; also many useful discussions with clients.

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Rapid Differentiation Between Vinegars and Non-Brewed Condiments: Part I

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Two rapid methods of differentiating between vinegar and non-brewed condiment are proposed. The first method is based on the Ultra-Violet absorption of the distilled sample and can be used as a rapid sorting method, although the data need careful interpretation as some samples produce ambiguous results. The second method is based on the osmotic pressure or freezing point depression of the distilled neutralised sample and no anomalies were found, differentiation being straightforward.

Vinegar as defined by the Food Standards Committee[1] is "the liquid produced from a suitable raw material containing starch or sugar, or starch and sugar, by the process of double fermentation, alcoholic and acetous and which contains at least 4 per cent. w/v acetic acid". Starting materials include malted barley, apples, grapes, molasses, bananas, rice and prunes, the different materials being favoured in different parts of the world.

Dilute solutions of acetic acid, produced by chemical means rather than by fermentation, with or without added caramel, are sold under the name of "non-brewed condiment" in the United Kingdom.

The presence on the market of such a variety of vinegars and condiments necessitates objective methods which may be used in conjunction with organoleptic determinations to assess the nature and authenticity of samples.

Traditional methods based on the determination of Oxidation Value (OV), Alkaline Oxidation Value (AOV), Iodine Value (IV) and Ester Value (EV)[2–5] are time-consuming and dependent on the analyst for reproducible and meaningful results.

More recently methods have been reported based on fluorescence studies[6,7], polarography[8], $^{13}C/^{12}C$ ratios[9], ^{14}C and ^{3}H radioactivity[10–12] and gas liquid chromatography[13,14], to differentiate between vinegar and non-brewed condiment. Many of these methods are outside the scope of those laboratories that do not possess the sophisticated instruments needed for many of these analyses.

Materials

Twenty-one commercial samples labelled as vinegar and four non-brewed condiments were used in the ultra-violet (UV) absorption method, 31 commercial samples of vinegar and nine non-brewed samples in the osmotic pressure method and 16 commercial samples of vinegar and nine non-brewed condiments in the freezing point depression method. All commercial samples of

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vinegar were purchased from retailers in the Weybridge area and five of the non-brewed samples were made in our laboratory, their composition being given in Table I. The remaining four samples of non-brewed condiment were commercially produced and were kindly supplied by British Vinegars Ltd., London.

All reagents and chemicals were obtained from BDH Chemicals Ltd., Poole, Dorset, England.

TABLE I

COMPOSITION OF NON-BREWED CONDIMENTS

Sample No.	Per cent. w/v acetic acid	per cent. w/v citric acid	Caramel
32	5	0	—
33	4	2	—
34	5	0	+
35	4	2	+
36	4	0	—

Equipment

Spectrophotometers: Unicam SP 800 recording spectrophotometer and Unicam SP 500 fixed wavelength spectrophotometer, Pye Unicam, Cambridge, England or equivalent instrument.
Osmometer: Advanced Digimatic Osmometer, Model 3D, Advanced Instruments Inc., Needham Heights, Mass., U.S.A. or equivalent instrument.
Freezing Point Apparatus: The Hortvet Apparatus, as described in British Standard 3095: 1959.

Experimental

UV ABSORPTION

Initially, for rapidity it was envisaged that the whole sample would be placed in a spectrophotometer and a UV absorption spectrum obtained characteristic for each type of vinegar. Samples containing caramel, and the characteristic vinegar coloured samples, absorbed strongly in the UV region and a distillation step was therefore introduced prior to the UV determination to decolourise the samples. Absorption at approximately the wavelength of maximum absorption (λ_{max}) was also determined.

Method

Pipette exactly 13 millilitres of sample (to provide a slight excess) into a 150-ml round-bottomed flask, add anti-bumping granules and connect the flask to a simple distillation apparatus. Heat the flask using a bunsen burner and collect the first 10 millilitres of distillate in a 10-millilitre volumetric flask. Place a sample of the distillate in a quartz cuvette and, using distilled water as a blank, obtain the UV absorption spectrum of the sample with a scanning UV spectrophotometer over the range 450–250 nm. Wash the apparatus and repeat the procedure with the next sample. Determine accurately the absorption of the sample at 275 nm using a suitable instrument.

OSMOTIC PRESSURE

To avoid misleading results caused by dissolved solids in each sample, it was considered essential to introduce a distillation step prior to osmotic pressure measurements. Unfortunately, it proved impossible to use the distillate from the UV method, owing to the high concentration of acetic acid in it. With the acetic acid present, all the osmometer readings were very high and no difference was found between the samples.

Method

Pipette exactly 10 millilitres of sample into a 150-millilitre round-bottomed flask, add anti-bumping granules and a predetermined quantity of molar sodium hydroxide to neutralise the acetic acid in the sample (this is calculated from the acidity determination below). Connect the flask to a simple distillation apparatus, heat the flask using a bunsen burner and collect the first 10 millilitres of distillate in a 10-millilitre volumetric flask. Determine the osmotic pressure using a suitable instrument.

FREEZING POINT DEPRESSION

If no instrument is available to measure osmotic pressure, the freezing point depression of the distillate can be determined, using for example the Hortvet apparatus, since freezing point depression is directly related to osmotic pressure.

Method

Proceed as in the determination of osmotic pressure but use 35 millilitres of sample. Neutralise, distil and collect 35 millilitres of distillate for the determination.

ACIDITY

The acidity of each sample is determined by titrating a 10-millilitre aliquot with 0·5M sodium hydroxide using phenolphthalein indicator and calculating the acidity as acetic acid (per cent. w/v).

ALKALINE OXIDATION VALUE, OXIDATION VALUE, IODINE VALUE AND ESTER VALUE

These are determined as described by Pearson[15].

Results and Discussion

UV ABSORPTION

All results are given in Table II. Typical UV absorption spectra for vinegars and non-brewed condiments are shown in Figure 1. The maximum absorption point for all vinegars was between 270 and 285 nm and accurate determinations of absorption for all samples were carried out at the fixed wavelength of 275 nm. Acidity of all samples was determined to ensure that all samples conformed to legal requirements for acidity and these results are given in Table II. Generally it can be seen that the non-brewed samples have lower absorption values than the vinegar samples. It was initially thought possible that vinegars and non-

TABLE II

ACIDITY, ABSORBANCE, OSMOTIC PRESSURE AND FREEZING POINT DEPRESSION OF VINEGARS AND NON-BREWED CONDIMENTS

Sample type	Sample no.	Acidity *per cent.* w/v acetic acid	Absorbance (at 275 nm)	Osmotic pressure *milliosmoles/kg*	Freezing point depression °C
Malt vinegar	1	4·8	0·371	37	0·110
	2	5·2	0·176	20	ND
	3	5·0	0·335	19	0·088
	4	5·1	1·460	24	ND
	5	5·2	0·778	55	ND
	6	5·0	ND	19	ND
	7	5·0	ND	16	ND
	8	5·2	ND	38	0·140
Distilled malt vinegar	9	5·0	∞	30	0·082
	10	5·0	ND	29	ND
	11	5·0	ND	25	ND
White wine vinegar	12	6·2	0·519	16	ND
	13	6·4	0·890	31	0·035
	14	6·4	0·481	18	0·049
	15	6·4	ND	22	ND
	16	6·4	ND	35	ND
Red wine vinegar	17	6·4	0·281	34	0·056
	18	6·0	0·491	44	0·101
	19	6·0	∞	89	0·150
Cider vinegar	20	5·8	0·609	51	ND
	21	5·8	0·615	50	0·139

TABLE II (*continued*)

ACIDITY, ABSORBANCE, OSMOTIC PRESSURE AND FREEZING POINT DEPRESSION OF VINEGARS AND NON-BREWED CONDIMENTS

Sample type	Sample no.	Acidity *per cent.* w/v acetic acid	Absorbance (at 275 nm)	Osmotic pressure *milliosmoles/kg*	Freezing point depression °C
	22	5·3	1·590	23	0·129
	23	5·9	0·655	48	0·159
	24	5·1	0·745	28	0·031
	25*	5·7	0·855	52	0·154
	26	5·9	ND	31	ND
	27	5·3	ND	23	ND
	28	5·1	ND	22	ND
Spirit	29†	5·1	0·307	63	ND
	30	4·9	0·078	18	0·048
	31	5·0	0·069	21	0·051
Laboratory	32		0·068	1	0·0
produced	33	See	0·038	0	0·001
non-brewed	34	Table	0·248	0	0·0
condiment	35	I	0·156	2	0·001
	36		ND	0	0·0
Commercially	37	4·4	ND	2	0·005
produced	38	4·9	ND	1	0·001
non-brewed	39	4·2	ND	2	0·003
condiment	40	4·1	ND	1	0·0

*Tarragon flavoured; †garlic flavoured; ND=not determined.

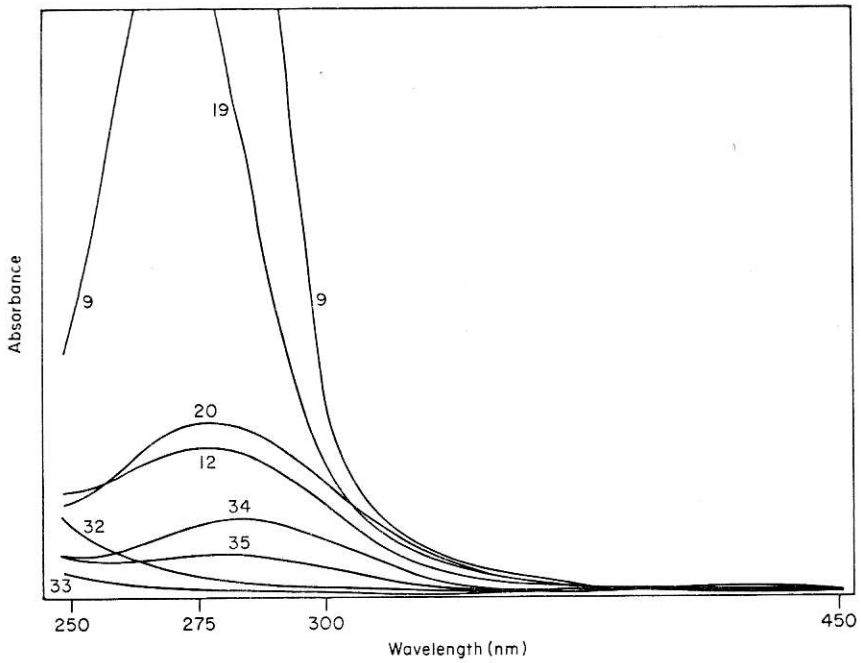

Fig. 1. Absorption spectra for vinegars and non-brewed condiments. 9, Distilled malt; 12, white wine; 19, red wine; 20, cider; 32–35, non-brewed samples.

brewed condiments would have completely different absorption spectra but in certain cases there is no difference. The non-brewed samples giving the high absorption readings (samples 34 and 35) differed only in their caramel content from other non-brewed samples examined by this technique (samples 32 and 33). We therefore concluded that some non-coloured components of the caramel distil over and cause these high values.

Comparing absorption spectra, there are four dubious vinegar samples (2, 17, 30, 31). The magnitude of the absorption readings depends on the concentrations of the minor components in the samples, e.g., aldehydes, acetates, alcohols and esters, but not acetic acid which does not absorb in the range 450–250 nm, so further analyses were carried out on the doubtful samples to confirm that they were genuine. Alkaline oxidation values, oxidation values, iodine values and ester values are given in Table III. These give a good indication of the concentration of the minor components and it can be seen that the results are

TABLE III

ANALYSIS OF SUSPECT SAMPLES OF VINEGAR

Sample	AOV*	OV*	IV*	EV*
2 — Malt	52	710	134	28
17 — Red wine	65	593	164	16
30 — Spirit	29	623	16	18
31 — Spirit	39	663	20	34

* See text.

within the ranges quoted in the literature for the particular samples[15]. We therefore conclude that although the UV absorption method could be used to sort rapidly vinegars from non-brewed condiments it is not an absolute method of differentiation and the results need careful interpretation. The method does compare favourably in our opinion with the formol titration which is an established rapid sorting test but which has the disadvantage that it does not differentiate between distilled vinegars and non-brewed samples, but merely between non-distilled vinegars and non-brewed samples.

OSMOTIC PRESSURE

The results are given in Table II and show that non-brewed samples give negligible osmometer readings (0–2) whilst all the vinegars give, as expected, substantially higher readings (16–89). It may be argued that 16 is not substantially higher than 2 but since there were no exceptions or dubious samples exposed by the method, we feel that it may have a use in this field. The osmotic pressure of the samples depends on the concentrations of the minor components present in the samples, since the acetic acid was effectively removed by neutralisation, as we had expected.

FREEZING POINT DEPRESSION

The results are given in Table II and show that non-brewed samples give negligible freezing point depressions (0–0·005°C) whilst all the vinegars give substantially higher readings (0·031–0·159°C). No exceptions were found.

Conclusions

The methods described above might usefully be applied in cases where rapid sorting of vinegar samples from artificial products is required. It was not possible to differentiate between different types of vinegar, e.g. cider from wine, using either method.

We are indebted to Mr E. Newman, British Vinegars Ltd., for the kind gifts of non-brewed condiment.

Mrs S. Welham is thanked for her help with the analytical work.

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Stability of Aqueous Solutions of Ascorbic Acid Prepared for Analysis by High Performance Liquid Chromatography

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The stability of aqueous solutions of ascorbic acid, prepared for analysis by high performance liquid chromatography, is improved by the addition of ethylene-diamine-tetra-acetic acid (EDTA), as the di-sodium salt.

During the determination of the ascorbic acid content of fruit juices by high performance liquid chromatography[1] it was found that the dilute solutions required for chromatography slowly lost ascorbic acid on standing. Ascorbic acid is known to be more stable in acid solution and a variety of acids have been examined to determine their ability to stabilise ascorbic acid in solution. Ponting[2] found that oxalic and metaphosphoric acids were the most effective in preventing loss of ascorbic acid (2·8 per cent. loss for each acid after 24 hours) at an ascorbic acid concentration of 32·2 to 33·2 μg/ml. In the presence of copper (6·3 μg/ml), oxalic acid was the more effective of the two acids (9·9 per cent. loss for oxalic acid, 14·4 per cent. loss for metaphosphoric acid). Citric and perchloric acids had some stabilising effect (24·1 per cent. and 14·5 per cent. loss respectively) in the absence of copper but in the presence of copper neither acid was effective (97·6 per cent. and 95·7 per cent. loss respectively). Under the same conditions, ascorbic acid solutions containing acetic acid lost 31·4 per cent. in the absence of copper and 99·5 per cent. in the presence of copper. Lamden[3] reported that metaphosphoric acid alone did not completely prevent the loss of ascorbic acid in the presence of copper and found that the addition of thiourea was effective in reducing this loss. Oxalic acid[2,4], metaphosphoric acid[5,6], perchloric acid[7,8] and metaphosphoric acid and acetic acid[7,9] have, nevertheless, been used to stabilise solutions of ascorbic acid but the use of these acids produces solutions with pH values of about 2 or below[2]; these solutions are unsuitable for analysis by high performance liquid chromatography using columns containing reverse phase packings. To avoid the use of strong acids, ethylene-diamine-tetra-acetic acid (EDTA), as the di-sodium salt, was examined as a reagent to stabilise dilute solutions of ascorbic acid.

Experimental

The apparatus, reagents and chromatographic procedure used were as described previously[1] with the addition of EDTA solution, 1 mg/ml in water, and copper sulphate solution, 40 μg Cu/ml in water. 1 ml of each of these solutions was added as required to the final solution prepared for chromatographic analysis, which was then diluted to 50 ml with water.

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Results and Discussion

Standard aqueous solutions of ascorbic acid were prepared[1] with and without EDTA; each solution also contained added copper, 0·8 μg Cu/ml. The solutions were allowed to stand at room temperature (22°C) and the ascorbic acid content of each solution was determined at intervals[1]. The results obtained are shown in Table I.

TABLE I

STABILITY OF ASCORBIC ACID IN AQUEOUS SOLUTION IN THE PRESENCE OF COPPER (0·8 μg/ml) WITH AND WITHOUT EDTA

Time	10 *min*	20 *min*	40 *min*	2 *hours*	3 *hours*	4 *hours*	24 *hours*
Without EDTA	99·5	95·9	90·0	67·9	58·4	47·0	nil
With EDTA*	100·0†	—	100·0	—	99·5	98·9	98·3

*20 μg/ml.
†Percentage of original concentration (40 μg/ml).

Solutions for analysis were also prepared from a sample of fresh orange juice. Solution A omitted citric acid and contained no added EDTA, solution B contained no added EDTA and solution C contained added EDTA; none contained added copper. These solutions were allowed to stand at room temperature (22°C) and the ascorbic content was determined at intervals[1]. The results obtained are shown in Table II.

TABLE II

STABILITY OF ASCORBIC ACID IN DILUTED ORANGE JUICE WITH AND WITHOUT EDTA

Time	0·5 *hours*	1·5 *hours*	2 *hours*	3 *hours*	5 *hours*	7 *hours*	24 *hours*
Solution A (a)	75·9*	63·3	47·6	39·3	22·2	11·7	nil
Solution B (b)	98·1	95·4	88·9	83·4	68·0	56·0	nil
Solution C (c)	100·0†	98·8	97·9	96·8	95·6	94·8	94·4

(a) No added citric acid or EDTA.
(b) Added citric acid, no added EDTA.
(c) Added citric acid and EDTA (20 μg/ml).
*Percentage of original concentration.
†Original concentration in undiluted orange juice=410 mg/kg.

Blank solutions, containing no ascorbic acid, were analysed periodically and the area corresponding to the internal standard (nicotinic acid) noted; no significant changes in the peak areas were found.

From the results in Table I it can be seen that EDTA is effective in preventing the copper-catalysed oxidation of ascorbic acid in aqueous solution. The results in Table II show that EDTA and citric acid (and to a lesser extent, citric acid alone) stabilise the ascorbic acid in dilute solutions of orange juice prepared for analysis by high performance liquid chromatography.

The main losses of ascorbic acid in processed orange juice are due to aerobic and anaerobic reactions of a non-enzymatic nature[10]. The presence of

EDTA does not prevent a small (*ca.* 5 per cent.) loss of ascorbic acid on standing for 24 hours and it is possible that this loss is due to the action of oxygen. It has been shown[11] that in canned orange juice there is an initial rapid loss of ascorbic acid due to the presence of free oxygen, followed by a much slower anaerobic loss of ascorbic acid. Solutions of oxalic acid with EDTA have been used[12,13] to stabilise extracts prepared for the colorimetric analysis of ascorbic acid.

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The Influence of Processing Techniques on the Final Quality of Tripe

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In New South Wales (N.S.W.), Australia, tripe for export is scalded but that produced for the local market is bleached by hydrogen peroxide or sodium peroxide. The historical background leading to the pH standards for tripe (6·5–7·5) is discussed. The chemical parameters of tripes from each of the three types of processing used in N.S.W. are tabulated and the colour, pH, percentage of protein and percentage of total solids of tripes from each process are compared. With increasing pH in the final product, the content of retained water increases giving a reduced content of protein in the final product. In view of these results, and the previously reported complete loss of thiamine with the alkaline peroxide treatment, the opinion is expressed that alkaline peroxide bleaching of tripes should be prohibited.

In New South Wales (N.S.W.), Australia, tripe is produced both for export and for local consumption. Tripe for export is cleaned, scalded and frozen, whereas tripe for local consumption is cleaned, scalded and bleached, and then chilled or frozen. Bleaching is carried out with hydrogen peroxide or sodium peroxide (i.e. alkaline peroxide). The steps in the three processes and the times required are shown in Table I.

TABLE I
STEPS IN THE THREE PROCESSES

Scalded	H_2O_2 treated	Na_2O_2 treated
Spray rinsed	Washed	Washed
Trimmed	Trimmed	Trimmed
Washed (agitated)	Hot agitated with alkaline powder	Scalded (1 hour)
Scalded, 10 minutes	Drained	Na_2O_2 soaked (48 hours)
Spin dried	Rinsed with water	Neutralisation with $CaCl_2+CO_2$ gas (24 hours)
Trimmed	H_2O_2 overnight soaked (initially 50–80°C)	
Cartoned		
Frozen	Drained	Drained
	Soaked in H_2O tank without agitation	Chilled
	Drained	
	Chilled/frozen	
Total Time Required		
Completed on day of slaughter. 30 to 45 minutes from paunch opening to cartoning.	24 hours for complete process	approximately 3 days

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Prior to 1980, there was no known published material comparing bleached and unbleached tripes other than in the 1935 Annual Report of the N.S.W. Director General of Public Health[1]. In this report E. S. Ogg compared the solids and pH of tripes, both bleached and unbleached, and concluded that the bleaching process resulted in a product with a decrease of approximately 50 per cent. of solid matter.

As a result of these experiments, a standard for tripe was introduced into the N.S.W. Pure Food Regulations which required the pH to be between 6·5 and 7·5, i.e. similar to that of the natural, unbleached product. The intention of the Regulation, according to the Annual Report of 1938, was to prevent the bleaching of tripe[2], but the processors turned their efforts to bleaching followed by adjustment of the pH to within the prescribed limits[3].

During the late 1930s housewives became accustomed to "white" tripe and adapted to cooking the tripe in at least two changes of water before adding milk and onions. By leaching out the alkali the milk did not curdle.

Tripe comes almost exclusively from cattle. Of the four compartments of the bovine stomach [rumen (paunch), reticulum (honey-comb), omasum (bible) and abomasum or true stomach][4], normally only the rumen and reticulum are used for food in N.S.W. The texture of the reticulum is similar to "honey-comb," while that of the rumen can be likened to "heavy towelling." Throughout the rumen there are a number of seams or joins and these are smooth and denser in texture.

Work was carried out to establish the chemical parameters of tripe that had been subjected to differing processing techniques. The pH, percentage of total solids and percentage of protein were determined on whole tripes that had been scalded, hydrogen peroxide bleached and sodium peroxide bleached.

Experimental

MATERIALS

The six scalded tripes were obtained direct from the abattoir and were collected on the same day immediately prior to packaging. Food inspectors from the N.S.W. Health Commission sampled the 12 hydrogen peroxide-bleached tripes and 24 sodium peroxide-bleached tripes (12 from each processor). One tripe from each processor was taken on a weekly basis. All the tripes were ready for sale.

METHODS

Each whole tripe was cut into three portions based on the different textures, namely the reticulum, rumen and seam of the rumen. Each portion was minced, homogenised and analysed separately. Nitrogen analyses were carried out using antimony-based catalyst in a Kjel–Foss automatic protein analyser[5]. All nitrogen determinations were made in triplicate. Total solids were determined in duplicate by the standard AOAC method[6]. The pH was determined on a slurry of one part of minced tripe to two parts of distilled water by weight. The slurry was shaken in a stoppered bottle every 5 minutes

TABLE II

DIFFERENCES IN pH, PROTEIN AND TOTAL SOLIDS CONTENT BETWEEN THE DIFFERENT AREAS OF TRIPES TREATED BY SCALDING AND BLEACHING

			(a) Scalded			(b) H_2O_2 bleached			(c) Na_2O_2 bleached	
Number of samples		Reticulum 6	Rumen 6	Rumen seam 6	Reticulum 12	Rumen 12	Rumen seam 12	Reticulum 24	Rumen 24	Rumen seam 24
Range	pH	6·9–7·2	7·2–7·4	7·1–7·2	7·6–8·2	7·7–8·1	7·6–8·25	6·7–10·2	6·85–10·2	6·85–10·1
mean	pH	7·1	7·3	7·15	7·9	7·9	7·9	8·5	8·3	8·3
S.D.	pH	0·1	0·1	0·05	0·15	0·1	0·2	1·0	0·9	1·0
Range	Protein	22·25–24·1	21·9–24·6	23·5–24·6	12·2–16·7	13·4–18·5	14·4–19·4	7·9–15·95	8·45–15·65	9·1–17·4
mean	Protein	23·4	22·6	24·2	14·8	15·8	16·6	11·95	12·0	13·2
S.D.	Protein	0·7	0·6	0·4	1·4	1·4	1·5	2·2	1·9	2·3
Range	Total solids	23·6–24·4	22·15–23·3	24·6–27·2	15·2–19·3	14·4–19·4	16·5–20·9	9·0–15·7	9·1–16·0	9·8–17·4
mean	Total solids	23·9	22·8	25·9	17·4	16·95	18·9	12·8	12·6	14·1
S.D.	Total solids	0·4	0·5	1·0	1·1	1·5	1·2	2·1	1·7	1·7

for 30 minutes after which the pH was determined electrometrically. The determination of the thiamine content has been described[7].

Colour photographs were taken of tripes from the three processes and the Methuen Handbook of Colour[8] was used to compare the colour of the three differently processed tripes in the colour prints.

Results and Discussion

The results obtained from different portions of the stomach treated by the three processes are shown in Table II. There are only small differences within the three areas of the stomach treated by the same process but for all processing techniques the protein content and solids content is marginally higher in the rumen seam. This is consistent with the denser texture of the seam. The greatest pH range within any one tripe is 0·65 units. From Table III, the

TABLE III

COMPARISON OF THE THREE DIFFERENT COOKING PROCESSES ON VARIATION OF COMPOSITION OF TRIPE

		Process (a) scalded	Process (b) H_2O_2 bleached	Process (c) Na_2O_2 bleached
	n	6	12	24
pH	Range	6·9–7·4	7·6–8·25	6·7–10·2
	Mean	7·2	7·9	8·3
	S.D.	0·1	0·2	1·0
Percentage protein	Range	21·9–24·6	12·2–19·4	7·9–17·5
	Mean	23·4	15·7	12·4
	S.D.	0·5	1·6	2·2
Percentage total solids	Range	22·1–27·2	14·4–20·9	9·0–17·4
	Mean	24·2	17·7	13·2
	S.D.	0·5	1·5	1·9

mean values show that with increasing pH there is a decrease in protein accompanied by a parallel decrease in total solids content. As the protein content on a dry basis is similar for each process the apparent loss of protein and solids in bleached tripe is actually due to an uptake of water. The protein content of alkaline peroxide bleached tripe is roughly half the protein content of scalded tripe.

The relationship between pH and protein content of sodium peroxide bleached tripe, processed in two different plants, is shown in Figure 1. A straight line regression fits the data, indicating that as the pH increased the protein decreased which can be attributed to a net increase in water content as the pH increases.

The colours of the tripes from the three processes, using the Methuen Handbook of Colour[8] for comparison, are as follows.

Scalded: ranged from page 2, A_2–A_3 to page 3, A_2–A_4.

Bleached: close to page 2 A_2. That is , all tripes are a yellowish-white but the scalded tripes are slightly yellower.

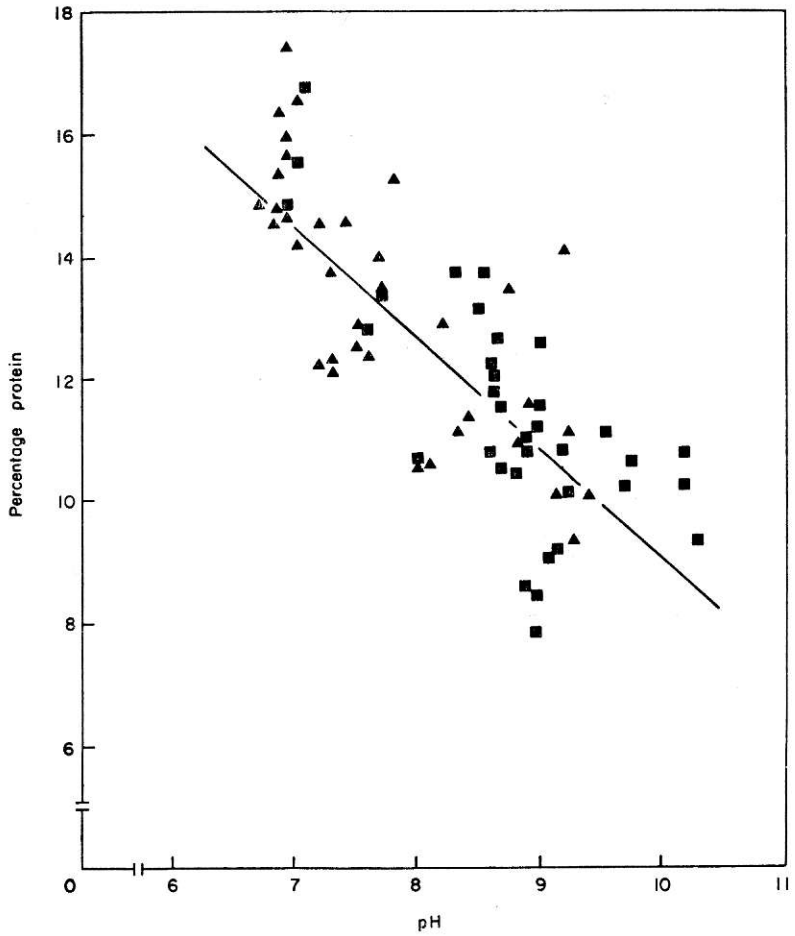

Fig. 1. Relationship between the pH and protein content of Na_2O_2 bleached tripes. (▲) Manufacture 1, process (c); (■) manufacturer 3, process (c); $y=27{\cdot}1-1{\cdot}8x$; $r=-0{\cdot}79$; $P<0{\cdot}001$.

As the moist surface of the tripes dried out, the colour of the tripes became a darker yellow.

Conclusion

The protein content of tripe is dependent upon the technique used in its processing. If hydrogen peroxide is used, the uptake of water is significant but less dramatic than with the alkaline peroxide treatment, from which the resulting tripes can be expected to contain roughly twice as much solid, by weight, as unbleached scalded tripes. It is also true to say that bleached tripe takes up more water than unbleached tripe.

These findings are in agreement with the 1935 Report of the Director General of Public Health,[1] where it was stated that "the process (using alkaline peroxide) results in 1 lb of tripe before treatment weighing 2 lb after treatment."

Skurray and Perryman[7] have shown that, using amino-acid analyses and rat bioassays, the nutritional value of the protein was not significantly decreased

by the sodium peroxide bleaching process, except that the thiamine was completely destroyed. The scalded tripes examined contained 140 ± 32 μg/kg thiamine. These studies have brought the authors to the conclusion that adequate cleaning produces a clean, aesthetically acceptable product in which the thiamine is retained and the protein content is approximately twice that of the product produced by alkaline bleaching with sodium peroxide. Consequently, the authors are of the opinion that alkaline bleaching of tripe should be prohibited.

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Thin Layer Chromatographic Detection of Argemone Oil and Rape Seed Oil in Mustard Seed Oil

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A quick TLC method has been developed for the separation and identification of both argemone oil and rape seed oil in mustard seed oil. The sample is dissolved in acetone, spotted on to a silica gel G TLC plate and developed with hexane-acetone solvent system. Argemone oil can be detected by exposing the plate under u.v. light, whereas rape seed oil is detected by spraying with Dragendorff's reagent followed by heating the plate to 110°C. This is a very simple, rapid and direct method.

Introduction

Argemone seeds (*Argemone mexicana*) and rape seeds (*Brassica campestris*, sub-species *napus*, etc.) resemble mustard seeds[1] (*Brassica nigra* and *Brassica juncea* or *Sinapsis alba*) in appearance. Therefore, mustard seed oil is often adulterated with rape seed oil and sometimes with argemone oil. Argemone oil contains the alkaloids sanguinarine and dihydrosanguinarine, which are physiologically active and highly toxic[2]. Rape seed oil on the other hand does not differ appreciably from mustard seed oil in respect of physical and chemical tests[3]. In this paper a simple TLC method is described for detecting both argemone oil and rape seed oil in mustard seed oil. The oil is dissolved in a suitable solvent, spotted on to a TLC plate and developed with a solvent system. The presence of argemone alkaloids, and hence the oil, is detected by exposing the plate under u.v. light[4]; rape seed oil is also detectable, if present, on the same plate by the method described in a previous publication[5]. This is a very simple, rapid and direct method and may be used as a screening test for argemone oil and rape seed oil in mustard seed oil.

Experimental

THIN LAYER CHROMATOGRAPHIC APPARATUS

1. Shandon TLC apparatus or the equivalent.
2. 21 cm × 10 cm TLC plate.
3. Standard jar.
4. Glass sprayer, capillary tubes, beakers, etc.
5. Desiccator, u.v. lamp, oven.

REAGENTS

1. *Silica gel G.*
2. *n-Hexane, A.R.*

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3. *Acetone, A.R.*
4. *Dragendorff's reagent.*[6]
   (a) 1·7 g of basic bismuth nitrate and 20 g of tartaric acid are dissolved in 80 ml of water.
   (b) 16 g of potassium iodide are dissolved in 40 ml of water.
   Stock solution. A 1:1 mixture of (a) and (b).
   Spray reagent. 5 ml of the stock solution are added to a solution of 10 g of tartaric acid in 50 ml of water.

METHOD

Dissolve about 0·5 ml of oil in a 20-ml beaker with about 2 ml of acetone and follow the TLC procedure previously described for the detection of rape seed oil[5], except that the solvent system is *n*-hexane/acetone (50:10). It is unnecessary to saturate the air in the tank; the time taken for the solvent to run 10 cm from the origin is only about 15 minutes, and the plate is allowed to dry at room temperature. Examine the plate under a u.v. lamp; argemone oil gives two fluorescent orange spots, due to the two alkaloids at R_f 0·19 and 0·60, whereas no such spots are given by either rape seed oil or mustard seed oil.

If it is desired to test for rape seed oil also, the plate can now be sprayed with Dragendorff's reagent as previously described. Using the *n*-hexane/acetone solvent, the rape seed oil gives two spots at R_f 0·53 (black) and 0·76 (grey) and mustard seed oil gives one spot at R_f 0·80 (grey).

Results and Discussion

Experiments were performed with argemone oil, refined rape seed oil (both imported and indigenous) and mustard seed oil. It was found that with approximately 5 per cent. adulteration of argemone oil in mustard seed oil, both the alkaloids could easily be detected under u.v. light. When the amount of argemone oil was further lowered, only the major alkaloid dihydrosanguinarine (R_f 0·60) could be detected. As little as 5 per cent. adulteration of rape seed oil in mustard seed oil could easily be detected by this method. Though various solvent systems were tried, it was found that the best separation of the spots of these three oils could be obtained with the solvent system used in this analysis. Although a paper chromatographic method[7], based on the extraction of pigment from raw rape seed oil, has been reported, this method even in conjunction with other routine physical and chemical examinations fails to establish the presence of *refined* rape seed oil in mustard seed oil, as the pigment is absent in refined rape seed oil. The spot and colour reactions of argemone oil on the other hand cannot establish the presence of rape seed oil. In fact, no method has so far been described for the detection of both argemone oil and rape seed oil by a single experiment. The present TLC method can detect with positive reliability and reproducibility both argemone oil and rape seed oil even when present in very low quantity in mustard seed oil. Moreover, this method is very quick. It should be borne in mind that R_f values are influenced by such factors as temperature, thickness of the layer, composition

of the solvent, etc., which may be compensated for by obtaining R_f values with pure oils under the same experimental conditions or by running controls alongside the samples under investigation. The total time of the experiment may further be shortened to about 15 minutes using microscope slides.

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Food Microscopy

(An Annotated Bibliography)

PART III D. DAIRY PRODUCTS

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This review covers three areas: cheese; fresh, frozen and whipped cream; ice cream. Milk and milk powder have already been covered in an earlier review (Part II E), dealing with major food ingredients.

An extraordinary range of cheeses can be produced from milk, though the sources of the milk may vary and microscopy has been applied to blue, Camembert, Cheddar, Cheshire, Cottage, Domiati, Italian, Kashkawal, Meshanger (soft), Norwegian whey, Parmesan, Rossiiskii, Suluguni, Swiss, Tilsit and white pickled cheeses, to spray-dried cheeses and cheese spreads. Aspects of cheese microscopy that have been studied include general elucidation of the microstructure, fat globule sizes, casein particles and micelles, lipoprotein granules and the location of lipoproteins, the formation and size distribution of crystalline material, "bloom" on processed cheese slices, mould growth and general bacteriology. The gradually increasing use of electron microscopy in these studies is evident.

In creams, fat globule size distribution, clustering and "free fat" have been studied, and the effects of homogenisation, pasteurisation and UHT treatment, and of refrigeration on the stability of the fat and protein phases investigated, both by light and electron microscopy. The milk fat globule membrane in ordinary fresh cream and in homogenised cream has also come under intensive scrutiny. The churning of cream has been studied microscopically, but most of the literature on this will be given later in Part III E on butter and margarine. Microscopic changes in the structure of frozen cream have been investigated, and destabilisation phenomena studied. The whipping properties of cream, and the structure and stability of the whipped product have been much examined microscopically, because of problems such as drainage and collapse. The role of ingredients in the formulation of whipped toppings has also been investigated by photomicrography.

A considerable amount of microscopy research has been done on ice cream. The literature covers many years of work, and involves the investigation of fat globule size in ice cream emulsions and the influence of homogenisation variables,

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the basic structure of ice cream including air cells, fat globules and clumps, ice crystals, casein micelles, the air-mix interface and the fat globule membrane. Also studied have been the principal defects in ice cream—shrinkage, buttery texture (fat clumps), coarse texture (iciness, sandiness), and poor melt-down properties. Comprehensive investigation by optical and electron microscopy has helped to elucidate the influence of all the product and processing variables on the properties of ice cream.

The following references are a selection taken from the vast amount of literature on this subject.

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Ivor Dembrey, 1904–1981

The former Additional Public Analyst for the City of Bristol and the County of Gloucestershire, Ivor Dembrey, B.Sc., M.Chem.A., C.Chem., F.R.S.C., died on 9 April 1981 in his 77th year.

A native of Bristol, he was educated at the Merchant Venturers School and gained his degree in Chemistry at the University of Bristol. At the same time, he became an Associate of the then Institute of Chemistry, now the Royal Society of Chemistry. His early professional career took him into industry with Capper Pass in Bedminster and later into a short research project at Long Ashton Research Station.

His real career commenced in 1934 when he was appointed as an Assistant Analyst to the late F. E. Needs, Public Analyst for Bristol from 1934 to 1946. During these early years he helped to develop the new laboratories at Canynge Hall at Clifton Down, but the Second World War prevented him from qualifying to be a Public Analyst in his own right until 1946. During the war years, he, like many other Air Raid Wardens, did much to defend the City of Bristol. In the late war years, he assisted in Gas Identification Training, and it was not surprising that he gave more voluntary service as a Scientific Intelligence Officer within Civil Defence during the late Forties and Fifties.

When the late Ernest Whittle was appointed Public Analyst in 1946, Ivor Dembrey was appointed his deputy, later becoming Additional Public Analyst when the laboratory was asked to include Gloucestershire, a post which he held until his retirement in 1969.

Since then, he took a very keen interest in the future of the laboratory, its emergence as the County of Avon Scientific Services Department and its recent move to Redcross Street in Bristol.

He was renowned for his knowledge and expertise as a professional scientist and despite his apparently gruff exterior, Ivor Dembrey was a very warm-hearted man, always ready to encourage his staff in the pursuit of analytical chemistry and to champion the underdog.

"I.D." as he was affectionately known, will be sadly missed by his family, his friends and his former colleagues.

D. J. Taylor