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Presented at the Annual General Meeting of the Association of Public Analysts held in London on 10th–12th May, 1968 by the Honorary Secretary, Mr. F. A. Lyne.

The constant vigilance maintained by Public Analysts' laboratories to ensure the purity of our foods and drugs and to protect the public from environmental hazards such as pollution of our water supplies or of the air we breathe demands flexibility of approach and constantly changing emphasis. The gross and fraudulent adulteration of food, which was rife a century ago and which brought Public Analysts into existence, has almost entirely disappeared, to be replaced by more insidious dangers to our health and pockets. These subtler dangers require far greater knowledge and more complex and sophisticated equipment. The last decade has seen profound changes in the work of our laboratories.

In addition to the steady outflow of our routine work, the year 1967 was noteworthy for several developments of considerable importance and interest to the public.

Pesticides

The first year of the Pesticides Residues in Food Survey was completed in July, 1967. This survey, probably the first systematic national survey ever undertaken in any country to establish the degree of contamination of food by pesticidal chemicals, was commenced on 1st August, 1966, in collaboration with the Local Authorities Associations and the Ministry of Agriculture, Fisheries and Food.

In an exercise involving the co-operation of sampling officers of 48 counties and over 150 urban and municipal authorities with Public Analysts in 38 laboratories, some 2,500 samples of staple foodstuffs were submitted to a detailed analysis for pesticide residues.

An elaborate, statistically-designed programme was drawn up which ensured that each part of the country was covered, that the rate of sampling was related to the population, and that all common foods were sampled in proportion to their importance in the diet.

Public Analysts were required to use the newer instrumental techniques capable of detecting minute traces of pesticide far below the level which would

normally be considered objectionable. Such techniques are capable of detecting one part of certain insecticides in one thousand million parts of foodstuff. Otherwise expressed—if the sample submitted were magnified to the size of a not very large haystack and a trace of pesticide present then occupied the volume of a needle, its presence would be detected with certainty.

The results of the first year of the survey from 1st August, 1966 to 31st July, 1967, have now been collected and they show interesting features, though their detailed analysis has yet to be completed. Preliminary inspection suggests no cause for alarm, but neither must we fall into a state of complacency. The results of the two years' survey should indicate those fields to which particular attention should be paid. This should set the pattern for future work, always bearing in mind that the pattern may change with new applications or new pesticides.

Toys (Safety) Regulations

Analysis of pencil-type crayons during the previous year had shown that many crayons of foreign origin contained lead. The sucking and chewing of these by children could give rise to lead poisoning and there was some evidence that this could cause mental retardation. An agreement between manufacturers and the Ministry of Education imposed a voluntary limit of 250 parts per million of lead in pencils and crayons supplied to schools. This figure of 250 parts per million had previously been accepted as a reasonable standard by Public Analysts. The publication of the Toys (Safety) Regulations, 1967, which declared a maximum of 11,000 parts per million of lead during the initial period (October, 1967–October, 1968), to be reduced to one-half of this figure from October, 1968, caused surprise as it was thought that such a limit was far too high. However, a few months' experience has shown that a fair proportion of imported toys fail even this lenient standard. Under the same Regulations, cellulose nitrate (celluloid) is banned in the construction of toys (except table tennis balls) owing to fire risk.

From October 1968, a limit will be placed on other toxic metals in toys, *i.e.* barium, cadmium, antimony and arsenic.

Some toys have been found to be stuffed with unsuitable materials, *e.g.* imported teddy bears containing phenol formaldehyde resin waste. Not only was this in a powdery form which might choke a baby if it were inhaled, but traces of free formaldehyde were also present which could have a serious effect on an infant's lungs.

Apart from their presence in toys, toxic metals in trace amounts have also caused trouble in other commodities. A Canadian heart specialist showed that at least 25 deaths amongst beer drinkers in Quebec City alone, during 1966, were due to the presence in the beer of cobalt, which had been added as a "foam improver." Its use has now been discontinued in the U.S.A. and Canada. A survey in Public Analysts' laboratories in this country has shown that although a small proportion of beers contained cobalt, the amount did not

exceed 1 part per million and the addition has now been discontinued in view of the American findings. At the level found in the small proportion of samples tested in this country, even a daily intake of 30 pints would contribute far less than the therapeutic dose of cobalt. The consumer would have died of alcoholic poisoning before developing the heart complaint due to cobalt, but even this trace amount has now disappeared.

The presence of lead in drinking water, although not a new problem, is one which received renewed attention. Some waters, particularly soft, acid waters, tend to dissolve lead from lead pipes. The American standard which has been accepted for many years, decrees that potable water shall not contain more than 0·1 parts per million of lead, or not more than 0·3 parts per million of lead after remaining in contact with lead for 16 hours. It has now been suggested that the European standard shall be reduced to a maximum of 0·05 parts per million of lead, although it might be necessary to relax this very stringent standard in other parts of the world where the shortage of water supplies would make such a standard impracticable. Many Public Analysts' laboratories are examining a considerable number of samples of water with particular reference to their lead contents.

Nitrate in Baby Foods

An American biologist, Dr. Commoner, has for some years been warning the world that human life will eventually be unsupportable because of the accumulation of nitrates in the soil and in our lakes and rivers. This is partly due to the vast usage of artificial fertilisers to give rapid growth. Whether this long-term forecast is tenable is open to debate, but it has been known for many years that infants under three months cannot tolerate appreciable amounts of nitrate. In rural areas where households are dependant upon wells for their supplies it has become customary to check the nitrate content of the well water if it is likely to be consumed by infants.

It is known that vegetables can take up nitrates from the soil, and there is a possibility that canned vegetables used for infant feeding might contain dangerous amounts of nitrate. These foods are seldom given in quantity to infants under 5 or 6 months, so that the danger seems remote; but Public Analysts' laboratories are examining numbers of canned baby foods for the presence of nitrates.

It is also believed that nitrate could be reduced, in cans of food, to nitrite, which is toxic to human beings of all ages. This possibility is also being examined.

Blood-Alcohol Testing

The coming into force, last October, of the Road Safety Act, 1967, which laid down a statutory limit of 80 mg per 100 ml of alcohol in blood gave rise to an immense amount of publicity in the press and on radio and television. Every aspect of the matter was attacked from the validity of the test as a measure of fitness to drive, to the methods of sampling and analysis.

Any motorist from whom a specimen of blood is taken for analysis has a right to request that the specimen shall be divided, and that he should be given one portion so that he may have it analysed by a laboratory of his own choice. Each portion is placed in a small plastic capsule which should be secured in a sealed envelope by the police before being handed to the motorist. In conjunction with the Royal Institute of Chemistry a list of laboratories, to which the blood may be sent, has been published and circulated to motoring organisations and other interested bodies.

Experience has shown considerable defects in the system. A large proportion of capsules are received which are not in sealed envelopes and apparently never have been. In these cases it is impossible to certify that the specimen has not been tampered with. The design and material from which the capsules are made are also unsatisfactory as many specimens are received in a dried condition, or clotted to such an extent that they are incapable of analysis. It must be realised that although the police laboratories would usually receive the specimens within 3 days, the defendant's specimen may await the issue of a summons and may reach the independent laboratory weeks after the event. At least one case has been dismissed by magistrates on the grounds that the specimen supplied to the defendant could not be analysed and that he was therefore robbed of his only means of defence.

Unless steps are taken to resolve the difficulties, many more cases will be dismissed on these grounds. The capsules must be re-designed so that they can be sealed, the preservative and anti-coagulant must be adequate to keep the blood in good condition for several weeks. The material from which the capsules are made, and the closure, must be impervious to water vapour so that the specimen does not dry out. These ideal requirements may be difficult to achieve, but the position would be improved if the defendant could be given instructions with the specimen, indicating that he may submit it to an independant laboratory (the list would be supplied on request), and if he wished to do so, it should be done as soon as possible.

Medicines

For nearly a century, the control of the purity of drugs on sale to the public has been linked with the control of food under successive Food and Drugs Acts. It used to be alleged, with some justification, that Local Authorities and their Public Analysts were primarily interested in foods and that insufficient attention was paid to drugs. During recent years, however, there has been an upsurge in the sampling of drugs, particularly the more potent drugs, which are available on prescription only.

A Bill before parliament at present would virtually remove drugs from the Food and Drugs Act. We approve entirely of the intentions of the Bill, which will tighten the control of the manufacture of drugs and reduce the possibility of disasters such as those associated with thalidomide. But there is one aspect of the matter which does give this Association cause for alarm.

It is most important that any form of quality control at the point of sale or supply that may replace the present system, is exercised by an impartial authority, whether it be local or national, *i.e.* by Local Authorities or by the central government through the Ministry of Health. We hold that it is essential that this control should be independent of the pharmaceutical industry or pharmaceutical interests generally. It is a basic principle of good legislation that the control of an industry or of a commodity should not be placed in the hands of the industry itself or of a body having an interest therein. Any departure from this would be contrary to the public interest and to natural justice, and if it is ignored in the case of drugs one might as well place the control of the purity and genuineness of foods under the aegis of an association of food manufacturers or retailers. The controlling authority must be independent, accountable to the public and not merely impartial, but seen to be impartial.

In addition to the legislation or prospective legislation already referred to, eleven statutory instruments were published during 1967 which directly affect the work of Public Analysts.

These were:—

The Meat Pie and Sausage Roll Regulations, 1967

The Canned Meat Product Regulations, 1967

The Sausage and Other Meat Product Regulations, 1967

The Artificial Sweeteners in Food Regulations, 1967

The Solvents in Food Regulations, 1967

The Food (Control of Irradiation) Regulations, 1967

The Carcinogenic Substances Regulations, 1967

The Labelling of Food Regulations, 1967

The Coffee and Coffee Product Regulations, 1967

The Ice-Cream Regulations, 1967

The Margarine Regulations, 1967

The first three of these lay down minimum meat contents for various types of meat products and, insofar that they regularise and codify the position, are to be welcomed.

The Artificial Sweeteners in Food Regulations, besides permitting and regulating the composition of sweetening tablets containing cyclamates, permitted the use of cyclamates in foods. This was in line with the findings of an expert committee as published in the White Paper. The relaxation on the control of cyclamates caused considerable controversy in the press and in parliament. The committee intends to keep the matter under review for the next five years and if positive evidence is forthcoming which suggests that cyclamates should not be permitted, dispensation will be withdrawn.

The Labelling of Food Regulations have been drawn up in the light of experience of the 1953 Regulations and are based on the underlying principle that a prospective purchaser has a right to a clear indication of what is being sold. The rise of self-service supermarkets has made clear and unequivocal labelling essential if the public is to get a square deal.

A further step in this direction is to eliminate false claims in advertisements and in this context, draft Regulations to control such claims were also published in 1967. This Association submitted a memorandum on the draft putting forward constructive proposals for amendments.

We have also made official comment on compositional standards for cream. In this case, we have expressed the view that "clotted cream", which is a traditional product of our South Western counties, should be a superior article to "double cream". We contend that, in addition to being made by the traditional method of heating or "scalding", clotted cream should also have a fat content of not less than 60 per cent. compared with 48 per cent. in double or thick cream. We feel that, unless the higher standard is laid down in the Regulations, the accepted product may be debased by commercial pressure.

We have presented a memorandum to the Royal Commission on Local Government setting forth our views on the future role of the Public Analysts' service within the framework of the Local Authority structure which may emerge as a result of the Commission's deliberations. In common with all departments of local government, we await the findings of the Commission. Whatever scheme emerges there is no doubt that scientific control of the purity of food, water, etc., will be necessary and that the present tendency to widen the scope of protective legislation, as exemplified by the Toys (Safety) Regulations and other measures, will make laboratory facilities and scientific advice an indispensible part of the structure. It is almost certain that the changes which have taken place during the last decade will be followed by others of at least equal magnitude in the next few years.

Aids to the Identification of Meat in Meat Products

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An attempt is made to identify the origin of meat in meat products. In Part I the protein reactions are examined by means of the "precipitin" test while Part II is devoted to examination of the fat. The routine method, as described by Nickolls, for determining the source of bloodstains is adapted for the examination of meats. The method gives satisfactory results for raw meat but cannot be used for cooked meats. Some meat fat is normally present in all meat and meat products. Transesterification of this fat, followed by gas chromatography is suggested as a second method of identification of the meat.

PART I: USE OF THE PRECIPITIN TEST

Because of an increasing number of enquiries from the general public it appeared that there was a need to develop a method or methods for identifying the origin of meat in meat products. Willcox[1], in his paper on "The Biological Test for Blood", mentions the work done by Uhlenhuth in 1900–1901 on the precipitin test, and makes a further contribution. This paper, together with that of Roche Lynch[2], is mainly concerned with forensic applications, although Roche Lynch does mention the possibility of the use of the precipitin test in the examination of food. This aspect was investigated by the Meat Products Committee of the Association of Public Analysts[3] with little success.

Culliford[4] has described a more sensitive method for precipitin reactions which is also less critical with regard to clarity of extracts, requires less anti-sera and gives speedier results. This method is a form of gel-electrophoresis. It is hoped to adapt this for meat identification in the near future.

The practical details of the precipitin test are comprehensively described by Nickolls[5], together with its application to the identification of the source of blood-stains. The method described below is essentially that of Nickolls with a few slight modifications.

The availability of anti-sera in the laboratory prompted the authors to investigate their application to the recognition of different types of meat. As a preliminary step, authentic samples of pork, lamb, beef, poultry, horse, dog and cat were obtained for use as controls.

The method works satisfactorily for sausages, mince and raw meat generally, but fails with cooked meats.

Apparatus

1. Set of tubes, 5 cm long, prepared by sealing one end of tubing, 4 mm in diameter, which has been thoroughly cleaned in chromic acid.
2. Pasteur pipettes, prepared by drawing out lengths of glass tubing, 8 mm in diameter, into a capillary, and appending a rubber teat.
3. A "precipitin board" to hold the tubes as recommended by Nickolls[5].

Reagents

1. *Precipitating Sera:* Set supplied by Wellcome laboratories*. These are kept in the refrigerator until ready for use.
2. *Isotonic salt solution:* 0·9 per cent. solution of sodium chloride in distilled water.

Method

Wash approximately 1 g of the sample, first with petroleum ether, and then with diethyl ether. Extract the residue with 5 ml of isotonic saline, by mixing, and allowing the extraction to take place in the cold for approximately one hour. Decant the liquid and spin it in a centrifuge until the solution is clear. Transfer one drop of the clear solution by means of a Pasteur pipette to each of a number of tubes, each containing an anti-serum which has been previously clarified by use of a centrifuge. (A depth of 3 mm of anti-serum in the tube is normally adequate). Add the drop of test solution to form a layer with the anti-serum, taking care to avoid wetting the sides of the tube and avoiding the formation of air bubbles. Allow the tubes to stand for approximately half an hour at room temperature and then examine them against a black background. An opalescent precipitate, visible at the junction of the two liquids, indicates a positive reaction.

The results of a series of precipitin tests on uncooked and cooked meat products, compared with control specimens, are given in Table I.

TABLE I

REACTIONS OF SELECTED SAMPLES TO THE PRECIPITIN TEST

	Anti-serum						
	Cat	Cow	Dog	Hen	Horse	Pig	Sheep/goat
Control serum							
Pork	−	−	−	−	−	+	−
Lamb	−	−	−	−	−	−	+
Beef	−	+	−	−	−	−	−
Poultry	−	−	−	+	−	−	−
Horse	−	−	−	−	+	−	−
Dog	−	−	+	−	−	−	−
Cat	+	−	−	−	−	−	−
Uncooked meats							
Sausage No. 1	−	−	−	−	−	+	−
Mince No. 2	−	+	−	−	−	−	−
Mince No. 3	−	−	−	−	−	−	+
Sausage No. 4	−	−	−	−	−	+	−
Sausage No. 5	−	−	−	−	−	+	+
Cooked meats							
Lamb	−	−	−	−	−	−	−
Hamburgers	−	−	−	−	−	−	−
Beefburgers	−	−	−	−	−	−	−
Hot dog	−	−	−	−	−	−	−

The compositions of sample Nos. 1, 2, and 3 were later confirmed by the butcher supplying them. The absence of a reaction from the last four samples is consistent with heat treatment of the products.

* The Wellcome Research Laboratories, Beckenham, Kent.

PART II: INVESTIGATION OF FAT USING GLC.

Previous attempts to distinguish fats from different animals have concentrated on the recognition of horse fat in admixture with beef and pork. A method originated by Paschke[6] was a development of Rossmann's procedure involving the bromination of the characteristic linolenic acid in horse fat. Dalley[7] investigated the application of bromination to the estimation of horse fat in other animal fats and vegetable oils. Amounts of 5 to 10 per cent. of horse fat in the mixtures could be ascertained.

Dugan and Petheram[8] compared the hexabromide method with a spectrophotometric method of Mitchell, Kraybill, and Zscheile[9] and found the latter method to be more efficient. A procedure involving gas chromatography of fractions of the unsaponifiable matter (separated by column chromatography) was worked out by Cook and Sturgeon[10] for the chemical identification of meat.

Withington[11] has used transesterification and gas chromatography for the determination of butterfat in margarine. Since the separation of fatty acids of lower molecular weight were under consideration, the ethyl esters (which are less volatile) were prepared. In the present study, use of the higher volatility of the methyl esters, particularly when dealing with acids above C_{10}, appeared to be advisable. Hence transesterification was carried out in methyl alcohol.

After running controls on a number of authentic fats it proved an advantage to introduce an internal standard. The most suitable was found to be *n*-propyl palmitate, as it did not interfere with the chromatograms of methyl esters prepared from the edible fats being investigated.

Apparatus

1. Gas Chromatography: The Pye Panchromatograph, with flame ionisation detector and a 5-foot, glass column containing 10 per cent. polyethylene glycol adipate on 100/120 mesh celite at 190°C. Current range 3×10^{-9} amps. Nitrogen: 9 lbs/sq. in. Gas flow: Rotameter reading 1·0.
2. Recorder: Honeywell Brown. (Chart speed 6 inches per hour).

Reagents

1. *Sodium hydroxide soln.:* 0·6 per cent. solution in methanol.
2. *Diethyl ether:* of AR quality.
3. n-*Propyl palmitate:* of AR quality.

Method

Mix 0·5 g of the separated fat with 1 ml of methanolic sodium hydroxide solution in a dry test tube. Warm the mixture at 70°C for 5 min. with gentle agitation. Cool to room temperature, add 4 ml of ether followed by 2 ml of water. Shake the tube gently for one minute. Remove the ether layer, evaporate to dryness and weigh. Dissolve the methyl esters in diethyl ether

TABLE II

GAS CHROMATOGRAPHY OF METHYL ESTERS OF FATTY ACIDS: PEAK HEIGHTS RELATIVE TO *n*-PROPYL PALMITATE (= 100)

Type of fat	Figure	Myristic (5)	Palmitic (6)	Palmitoleic (7)	Stearic (8)	Oleic (9)	Linoleic (10)	Linolenic (11)	C_{20} Unsaturated (13)	C_{22} Unsaturated (15)
Pork	2	30	224	33	73	208	29	—	—	—
Lamb	3	42	144	18	166	130	—	8	—	—
Beef	4	40	140	18	162	127	—	10	—	—
Dog	5	50	142	176	27	193	23	—	—	—
Cat	6	88	190	29	66	166	20	—	—	—
Horse	7	97	223	40	26	114	24	49	—	—
Duck	8	14	208	34	34	234	38	—	—	—
Herring	9	94	143	59	7	115	—	—	19	19
Linseed	10	—	66	—	30	103	39	80	—	—
Cottonseed	11	21	180	—	14	92	104	—	—	—
Maize	12	—	105	—	17	140	223	—	—	—
Sausage (1)	13	30	206	27	71	198	46	—	—	—
Sausage (1) Cooked	14	25	199	24	68	197	47	—	—	—
Mince (2)	15	63	235	44	78	176	—	—	—	—
Mince (3)	16	126	192	29	65	176	—	—	—	—
Sausage (4)	17	34	197	30	69	194	31	—	—	—
Proprietary Cat Food	18	92	137	47	7	70	—	—	17	19
Beefburger	—	73	228	24	143	172	—	—	—	—
Hot Dog	—	43	249	41	79	253	40	—	—	—

with one fifth their weight of *n*-propyl palmitate. Dilute with diethyl ether to about 4 ml. Inject 1·0 μl of the ether solution on to the column and run the chromatogram for 45 minutes. If the presence of fish oils is suspected, run the chromatogram for 80 minutes, and for $2\frac{1}{2}$ hours if castor oil is present.

Results

Figure I is a composite chromatogram. The firm line is obtained from known esters. The dotted line, showing the position of methyl esters of the C_{20} unsaturated acids (peak 13) and of methyl esters of the C_{22} unsaturated acids (peak 15) is superimposed, using the chromatogram obtained with methyl esters obtained from herring oil. The (dotted) lines showing methyl arachidate (peak 12) and methyl behenate (peak 14) were obtained with authentic esters, and methyl ricinoleate (obtained from the esters of castor oil), is represented by peak 16. Figures 2 to 12 are chromatograms obtained from various known fats and oils, and numbers 13 to 18 were recorded from samples of the fat of meat products submitted to the laboratory. These have been mentioned in Part I in connection with the precipitin test. The proportions of each ester relative to *n*-propyl palmitate (peak M) were then calculated from the peak heights on the chromatograms giving the peak for *n*-propyl palmitate the arbitrary value of 100 (Table II).

Table III demonstrates ratios of various pairs of esters on the same basis and these were obtained from the figures given in Table II.

TABLE III

GAS CHROMATOGRAPHY OF METHYL ESTERS OF FATTY ACIDS: PEAK HEIGHT RATIOS

Type of fat	Figure	Palmitic / Myristic	Oleic / Stearic	Oleic / Myristic	Oleic / Palmitic
Pork	2	7·5	2·8	6·9	0·9
Lamb	3	3·4	0·8	3·1	0·9
Beef	4	3·5	0·8	3·2	0·9
Dog	5	2·8	7·1	3·9	1·4
Cat	6	2·2	2·5	1·9	0·9
Horse	7	2·3	4·4	1·2	0·5
Duck	8	14·8	6·9	16·7	1·1
Herring	9	1·5	16·4	1·2	0·8
Linseed	10	—	3·4	—	1·6
Cottonseed	11	8·6	6·6	4·4	0·5
Maize	12	—	8·2	—	1·3
Sausage (1)	13	6·9	2·8	6·6	1·0
Sausage Cooked (1) ..	14	8·0	2·9	7·9	1·0
Mince (2)	15	3·7	2·3	2·8	0·7
Mince (3)	16	1·5	2·7	1·4	0·9
Sausage (4)	17	5·8	2·8	5·7	1·0
Proprietary Cat Food ..	18	1·5	10·0	0·8	0·5
Beefburger	—	3·1	1·2	2·4	0·7
Hot dog	—	5·8	3·2	5·9	1·0

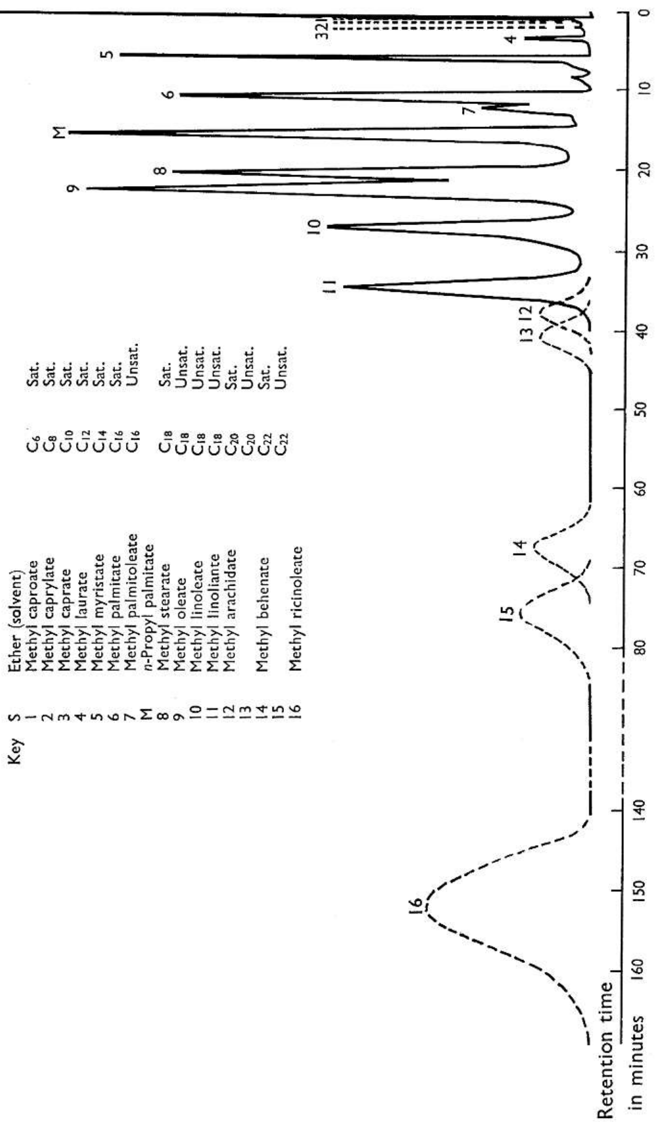

Fig. 1 Gas chromatography of the methyl esters of certain fatty acids

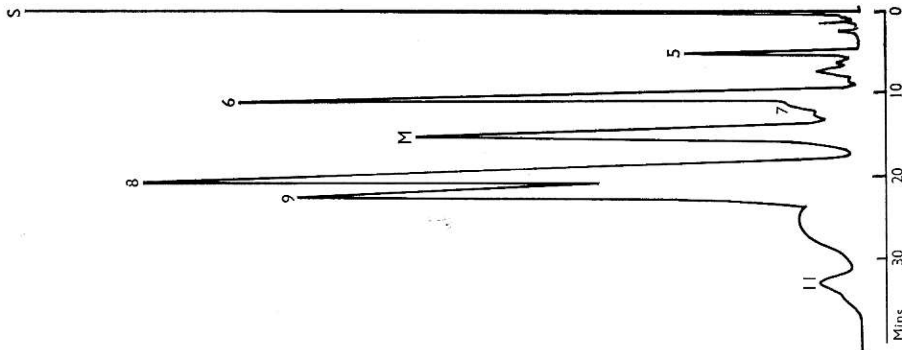

Fig. 4 Beef fat

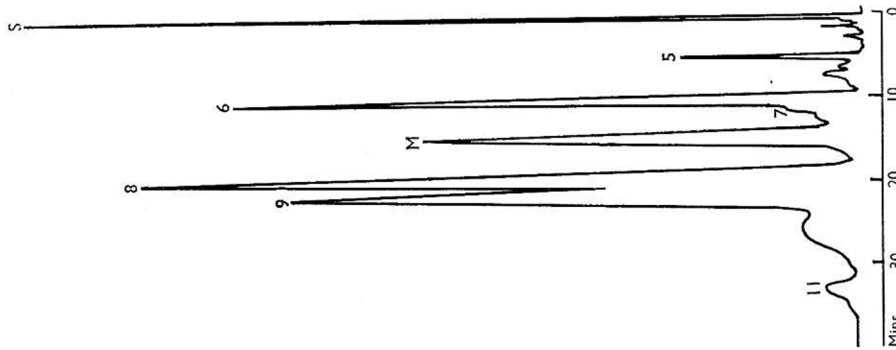

Fig. 3 Lamb fat

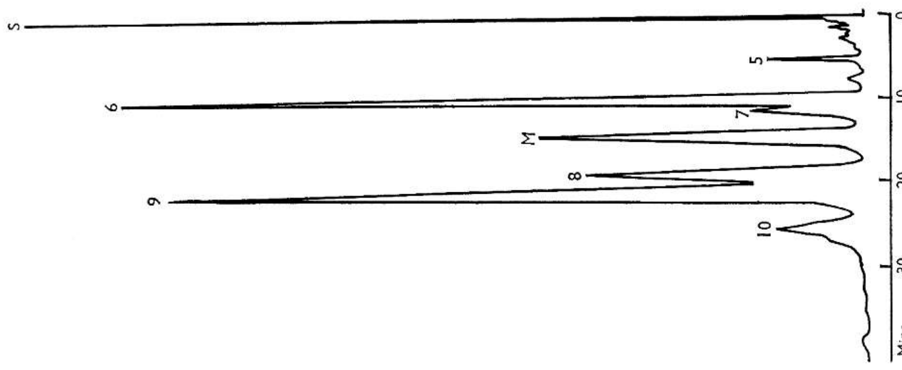

Fig. 2 Pork fat

Gas chromatography of the methyl esters of fatty acids

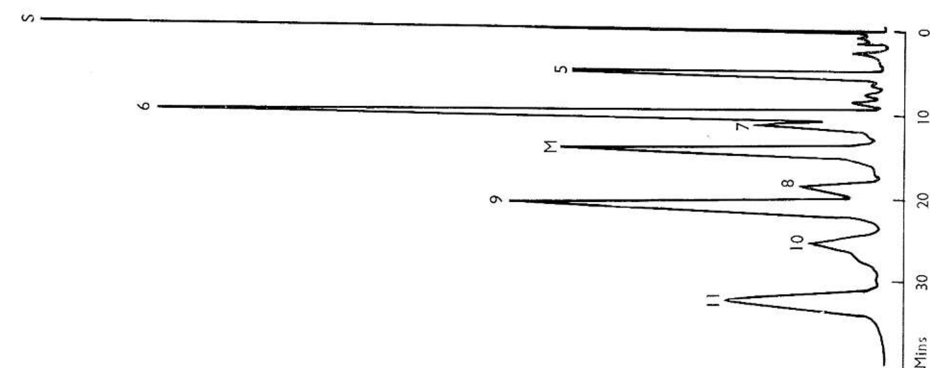

Fig. 7 Horse fat

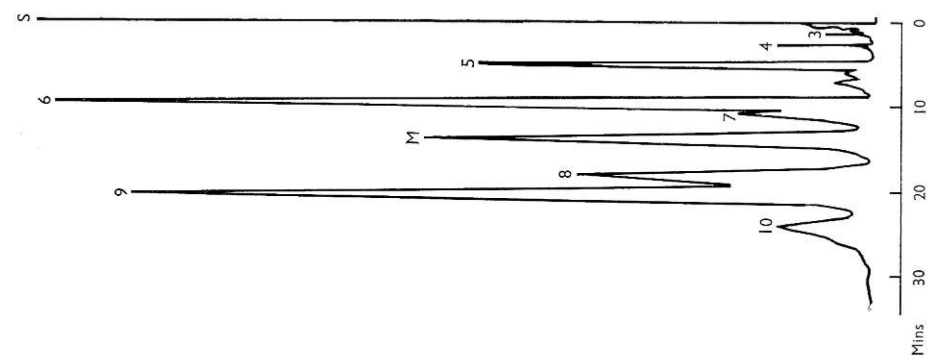

Fig. 6 Cat fat

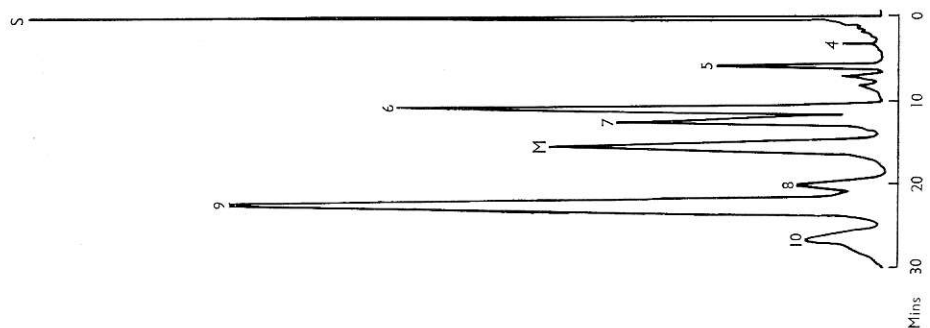

Fig. 5 Dog fat

Gas chromatography of the methyl esters of fatty acids

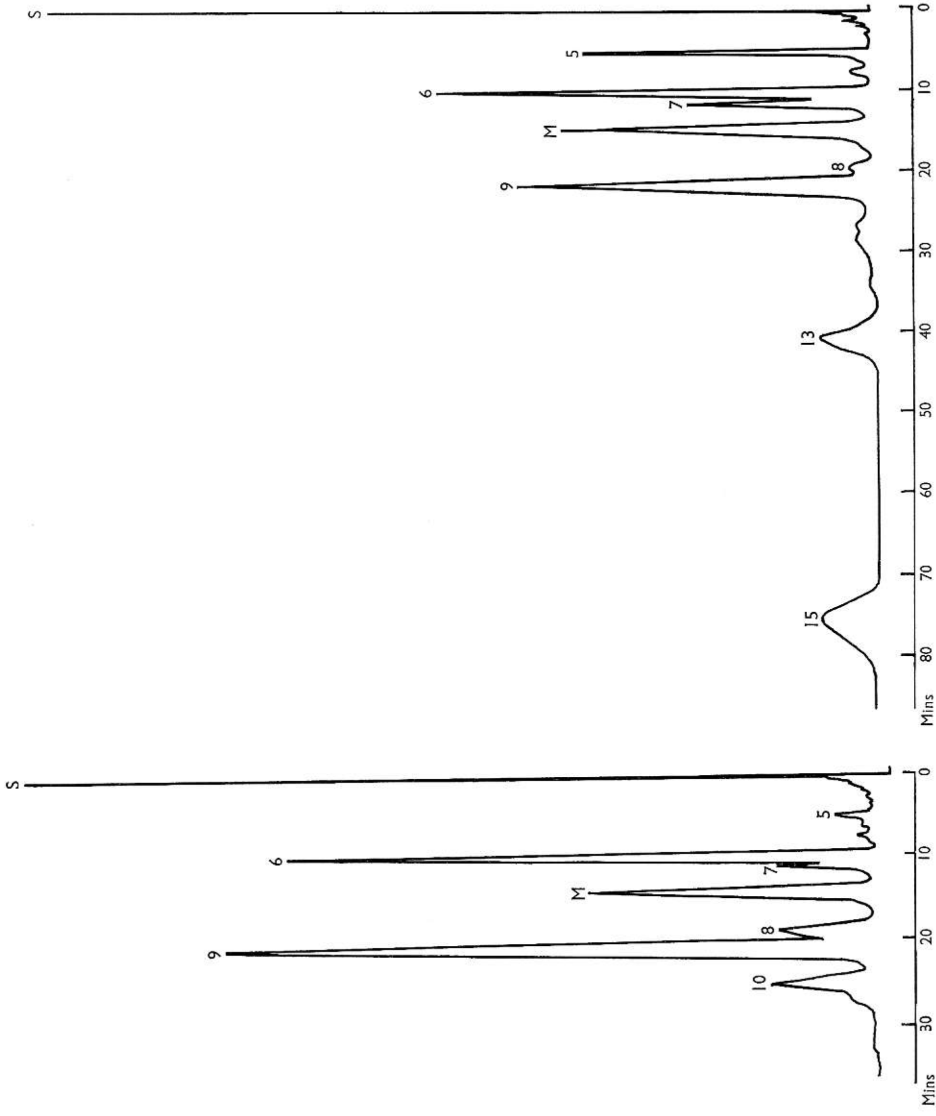

Fig. 8 Duck fat

Fig. 9 Herring oil

Gas chromatography of methyl esters of fatty acids

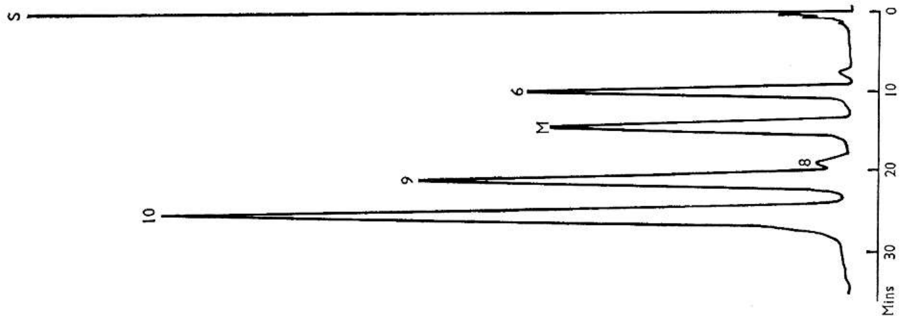

Fig. 12 Maize oil

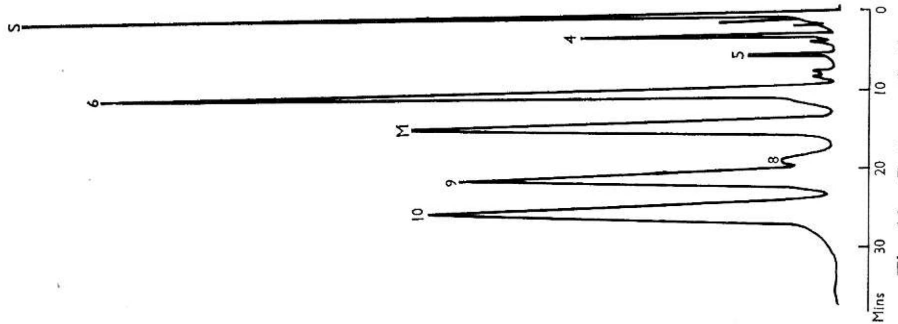

Fig. 11 Cottonseed oil

Gas chromatography of methyl esters of fatty acids

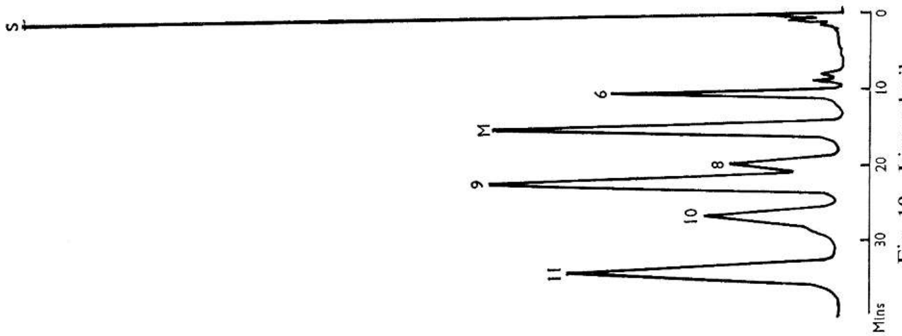

Fig. 10 Linseed oil

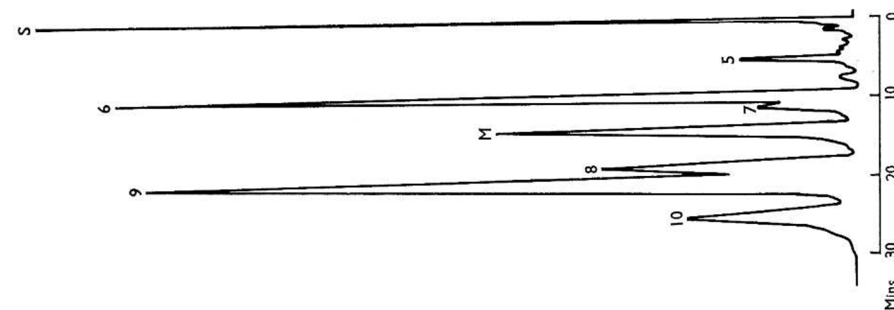

Fig. 13 Fat from uncooked pork sausage sample (1)

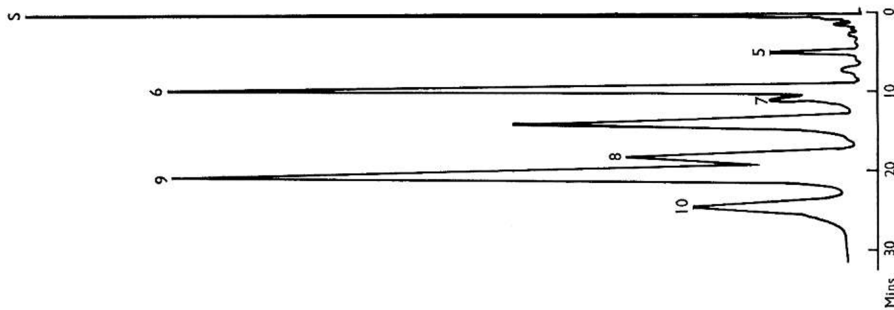

Fig. 14 Fat from cooked pork sausage sample (1)

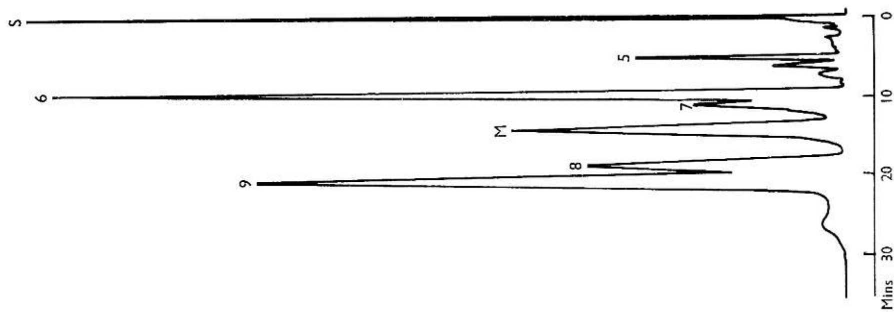

Fig. 15 Fat from minced meat sample (2)

Gas chromatography of methyl esters of fatty acids

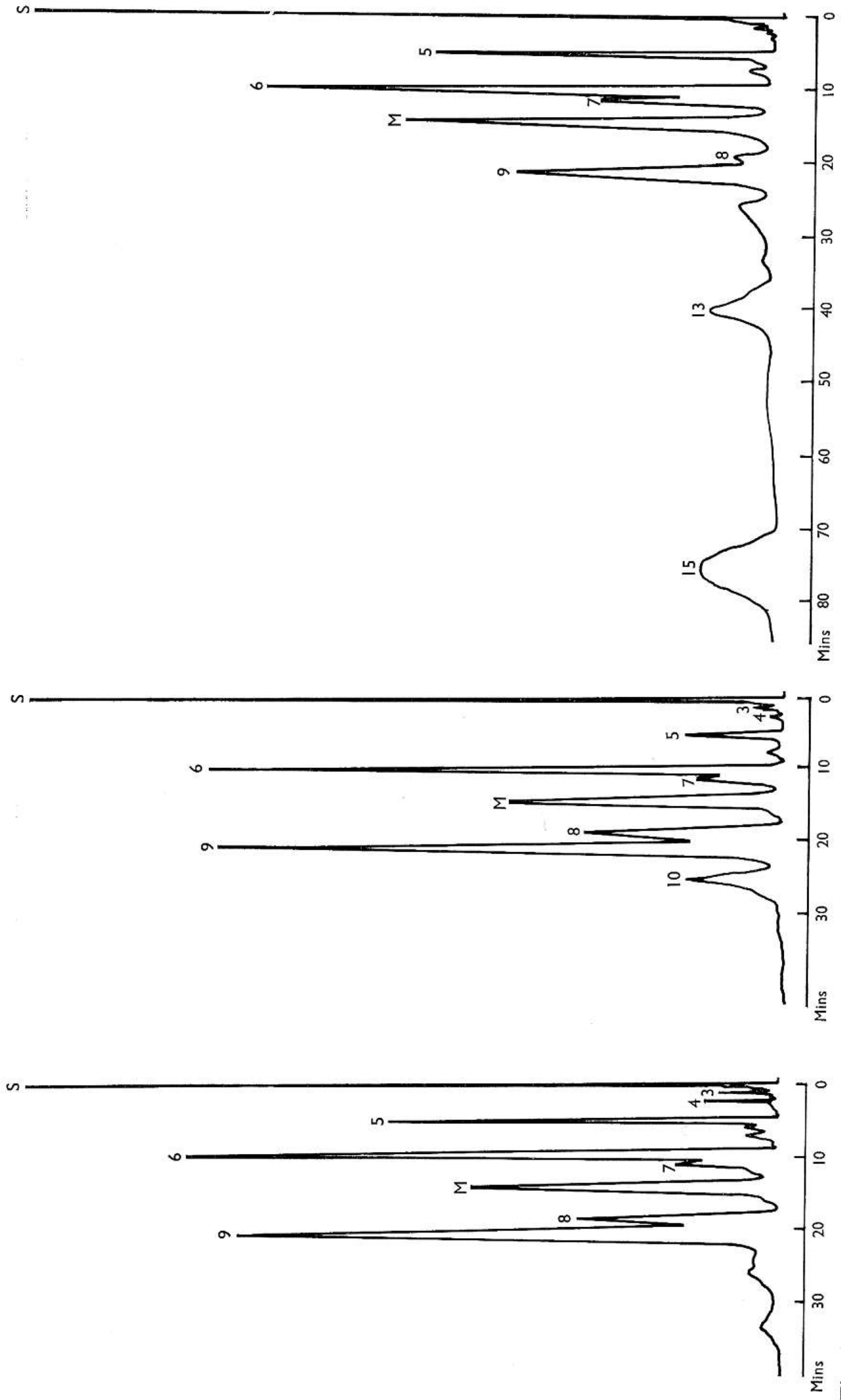

Fig. 16 Fat from minced meat sample (3)

Fig. 17 Fat from sausage sample (4)

Fig. 18 Fat from a proprietary cat food

Gas chromatography of methyl esters of fatty acids

Discussion

Examination of the Tables and chromatograms suggest the following conclusions.

(*a*) Palmitoleic acid occurs in most animal fats although its presence is not always pronounced in beef and lamb. It has not been observed in fat of vegetable origin.

(*b*) There is no significant difference between beef and lamb fats, as evidenced by the recorded results.

(*c*) Horse fat shows well-pronounced "linoleate" and "linolenate" peaks. The presence of significant amounts of these two compounds would provide evidence for the presence of horse fat.

(*d*) In pork fat, the palmitate/myristate and the oleate/myristate ratios normally exceed 6, compared with ratios of less than 4 for beef and lamb and more then 14 for duck.

(*e*) The "linoleate" peak is well pronounced in pork, but rather diffuse in beef and lamb.

(*f*) The fish oil (represented by herring oil) is the only type which shows the presence of C_{20} and C_{22} unsaturated acids.

Turning to the samples submitted, comparison of figures 13 and 14 and the relative ester ratios indicates that cooking has not invalidated this identification procedure. The chromatograms produced with samples Nos. 13, 14 and 17, and the figures derived from them, conclusively point to their containing pork. This confirms the results of the precipitin test and the stated identity of the meat. Mince No. 2 and Mince No. 3 show the characteristics of either beef or lamb. The precipitin test indicated that Mince No. 2 consisted of beef and Mince No. 3 of lamb. The butcher confirmed these identifications. Different methods of extraction of the fats from the meat product e.g. by direct extraction with ether after desiccation with sodium sulphate or by extraction by the Werner Schmidt method, have no effect on the chromatograms obtained with the resultant fat.

The figures point to the presence of beef or lamb, in the beefburger and the hot dog appears to comprise mainly pork. Neither of these samples gave indications of the presence of cat meat, dog meat, horse meat or of a cat food, as was at first suspected.

The authors thank Mr. B. Bisson, Miss K. Syvret and Mr. E. J. Walmesley for carrying out practical work; Mr. C. L. Gruchy (States Veterinary Surgeon), for supplying samples of meat from the horse, dog and cat; Mr. A. G. Averty, for supplying samples Nos. 1, 2 and 3; and Senator C. Le Marquand (President of the Finance Committee, States of Jersey), for permission to publish the paper.

References

1. Willcox, W. H., *Analyst*, 1928, **53**, 3.
2. Roche Lynch, G., *Analyst*, 1928, **53**, 5.
3. Association of Public Analysts, London 1958–59 (*Private Communication*).
4. Culliford, B. J., *Nature*, 1964, **201**, 1092.
5. Nickolls, L. C., "The Scientific Investigation of Crime," Butterworth, London, 1956, p. 175.
6. Paschke, B., *Z. Unters. Lebensm.*, 1938, **76**, 476.
7. Dalley, R. A., *Analyst*, 1950, **75**, 336.
8. Dugan, L. R. (Jr.), and Petheram, Marjory, *J. Ass. Off. Agric. Chem.*, 1952, **35**, 767.
9. Mitchell, J. H., Kraybill, H. R., and Zscheile, F. P., *Ind. Eng. Chem. Analyt.*, 1943, **15**, 1.
10. Cook, H. R., and Sturgeon, J. D., *J. Ass. Off. Agric. Chem.*, 1966, **49**, 877.
11. Withington, D. F., *Analyst*, 1967, **92**, 705.

A Tentative Specification for the Acceptability of Minced Beef Based on Chemical Methods

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[*This is a revised version of a paper which appeared in November* 1967, *in "Food Manufacture,"* (*London*).]

A tentative specification for minced beef suitable for application in routine control is presented, based principally on chemical determinations. The specification is designed to set standards for the fat content and freshness of the meat. The critical freshness acceptability limits included have been calculated from correlations between the chemical values (volatile nitrogen, free fatty acids of extracted fat and the extract-release volume) and the odour as assessed by a panel. Although the specification has been developed from work performed on stored samples of comminuted beef the author hopes that its publication will encourage manufacturers (*a*) to apply these three methods to deliveries of beef received and (*b*) to comment on the limits suggested.

There has been some renewed interest recently in the application of reasonably rapid methods for assessing the acceptability of meat using chemical techniques. The author has spent several years studying this problem and presents here a tentative specification which it is hoped will be considered and applied on a trial basis by laboratories where meat is being regularly examined.

Introduction

The microbiological, physical and biochemical changes which take place in meat have been studied by many workers. Most of the published work relates to studies of changes in individual samples of meat during storage rather than a consideration of the results as a more general picture. Further, apart from investigations related to measurements of the pH value, there has been little emphasis on the relationship between the results obtained from objective methods and acceptability of the meat for consumption.

Fresh meat, after dressing, contains few micro-organisms, but handling, transport, cutting and mincing may all contribute to their introduction. Although the original contaminating flora are diverse, it is usual for one organism to predominate eventually (depending on various factors such as pH, storage temperature and availability of nutrients, oxygen and water). The psychrophiles most commonly found on refrigerated meat are the obligate aerobes, the pseudomonads. Microbiological methods are less suitable for application to routine control than chemical tests in view of the time required for their completion, particularly if comminuted samples are under examination.

There is, however, no evidence that individual meat organisations have applied limiting values of freshness based on shorter chemical determinations. Work carried out by the author has been promising enough to recommend that certain methods could be used as the basis of a specification designed to assess the age and acceptability of beef.

Considerations in relation to method selection

Various samples of comminuted beef have been stored at 5°C (41°F) and examined at successive stages for chemical values and acceptability as judged from the odour. The analytical methods employed were considered mainly in relation to (*a*) agreement between replicates (*b*) consistency between different fresh samples, (*c*) whether there was a consistent increase or decrease in values as the meat deteriorated, (*d*) correlation with odour assessment and (*e*) reasonable rapidity and ease of performance. When considered from the point of view of such criteria, the most satisfactory methods were found by the author to be:

(i) the application of a modification of the Lucke and Geidel technique[1] for the determination of total volatile nitrogen (TVN) as a measure of the degree of breakdown of protein.

(ii) the free fatty acids (FFA) of the extracted fat, measuring fat hydrolysis.

(iii) the measurement of the extract-release volume (ERV), employing a modification of Jay's method[2].

Most samples of beef examined showed increases in the TVN and FFA, indicating that deterioration in quality is usually due to the concomitant actions of proteolytic and lipolytic enzymes. Correlation with subjective assessments indicated that such meats were acceptable provided they complied with the following maximum limits:

TVN 20 mg of nitrogen per 100 g of fat-free material.

FFA 1·8 per cent. (calculated as oleic acid on the extracted fat).

In a few instances one value advanced markedly during storage whilst the other showed only an insignificant increase. Where this occurred, the meat produced an extract-release volume filtrate of at least 20 ml.

It was considered feasible therefore that although it is preferable that beef should comply with the TVN and FFA limits above, individual meats complying with only one of these maxima is still acceptable provided that the ERV is at least 20 ml. These limits are therefore incorporated in Part I (4*b*, *c* and *d*) of the tentative specification. Other specification requirements should be self-explanatory. Requirement (1) of Part I relates to satisfactory appearance and odour and (2) to hygienic handling. A maximum fat content of 30 per cent. (4a) is also recommended in order to ensure the presence of a reasonable proportion of lean meat. The sampling and standardised analytical methods on which the criteria are based are detailed in Part II.

TABLE I

CHEMICAL CHARACTERISTICS OF BEEF (WHEN PURCHASED) AND ASSESSED AS FRESH

	Total volatile nitrogen *mg per 100 g of fat-free material*	Free fatty acid in the fat *as oleic acid per cent.*	Extract release volume *ml*
Minimum ..	10·9	0·24	14·1
Maximum ..	22·8	2·02	25·0
Mean	17·7	0·70	20·2

Beef comminuted at various retailers' premises or in the laboratory was used in the investigation mainly to reduce sampling errors in storage 'runs'. Although the mincing operation may introduce an additional source of contamination, it is felt that similar spoilage criteria at least could be applied to deliveries of meat received as a raw material for manufacturing purposes. As the TVN, FFA and ERV values have been found to change progressively as beef spoils, they are thought to be worthy of consideration as a reasonably rapid control method, even though the critical limits may require some adjustment in relation to specific requirements. Further, although no meat can be considered "safe" without an elaborate microbiological examination, it is hoped that the results obtained from the less time-consuming chemical methods would assist in presenting day-to-day comparisons of the degree of freshness.

MINCED BEEF

[*Note: This specification is designed to be used on a day-to-day basis in order to set limits of acceptability with respect to freshness and age of the beef. It must be realised however, that complete safety cannot be ensured without a lengthy microbiological examination.*]

Part I: Specification

1. *Description*

The minced beef shall be prepared from good quality meat. The product shall be comminuted to a reasonable extent so that small white pieces of fat are visible and evenly distributed throughout. The produce shall be clean, red and bright in colour and shall have a not unpleasant odour.

2. *Hygiene*

The conditions of production shall comply with the provisions of the Food Hygiene (General) Regulations, 1960.

3. *Statutory Requirements*

The product shall comply with all relevant statutory requirements or regulations relating to food currently in force in England and Wales.

4. *Detailed Requirements*

The minced beef shall comply with the following requirements:

(*a*) The fat content shall not be more than 30 per cent.

(*b*) The Total Volatile Nitrogen content shall not exceed 20 milligrammes expressed as nitrogen in 100 grammes of fat-free material.

(*c*) The Free Fatty Acid content shall not exceed 1·8 grammes when expressed as oleic acid per 100 grammes of the extracted fat.

Provided that, if the minced beef complies with the limit specified in (*b*) but not that in (*c*), or with the limit specified in (*c*) but not that in (*b*), it shall be deemed to have conformed with such specification requirements if it has an Extract Release Volume of at least 20 ml. [See (*d*), page 58].

5. *Sampling and Testing*

Sampling and testing will be in accordance with the methods described in Part II.

Part II: Sampling and Testing

1. *Sampling*

The minced beef will be mixed and prepared by the method recommended by the Society for Analytical Chemistry (formerly the Society of Public Analysts and Other Analytical Chemists) for the analysis of meat products[3].

2. *Testing*

(*a*) *Fat:* The prepared sample will be examined for fat content by the method recommended by the Society of Public Analysts and Other Analytical Chemists[3] for the analysis of meat products. An alternative method may be employed provided it has been shown to give similar results.

(*b*) *Total Volatile Nitrogen:* The prepared sample will be examined for total volatile nitrogen by the following method:

Apparatus

Distillation apparatus: As employed for the determination of nitrogen by the macro-Kjeldahl method, having a one-litre boiling flask and 500-ml receiving flask.

Reagents

All reagents shall be of recognised analytical purity.

1. *Magnesium oxide:* Powdered.

2. *Boric acid solution:* Dissolve 2 g of boric acid in distilled water and dilute to 100 ml.

3. *Screened methyl red indicator:* Dissolve 0·083 g of bromocresol green and 0·016 g of methyl red in 100 ml of industrial methylated spirit.

4. *Sulphuric acid:* 0·1 N solution.

Method

Place 10 ± 0·05 g of the prepared sample, 2 g of magnesium oxide and 300 ml of fresh tap water in the distilling flask. To the receiving flask add 25 ml of 2 per cent. boric acid solution and a few drops of screened methyl red indicator. Connect up the distillation apparatus with the receiver adapter dipping below the boric acid solution. Heat the distilling flask, supported on metal gauze, by means of a bunsen burner so that the liquid boils in 10 minutes. Using the same rate of heating, distil over the volatile nitrogen for 25 minutes. Wash down the condenser with distilled water into the receiving flask and titrate the contents with 0·1 N sulphuric acid (A ml). Carry out a blank test by the same procedure as described above, but omitting the sample (titration = B ml).

$$\text{Total Volatile Nitrogen (mg per 100 g)} = \frac{(A - B) \times 1400}{100 - F}$$

where F is the percentage of fat in the sample.

(*c*) *Free Fatty Acids of the Extracted Fat:* The prepared sample will be examined for free fatty acids of the extracted fat by the following method:

Apparatus

1. Mechanical blender, MSE Atomix Blender* or an equivalent high-speed mixing and cutting apparatus.
2. Separating funnels, calibrated, of 200 ml capacity.
3. Filter funnels, Pyrex brand, glass (10 cm diameter).

Reagents

All reagents shall be of recognised analytical purity.

1. *Chloroform:*
2. *Sodium sulphate (anhydrous):* Dry sodium sulphate at 100°C, cool and store in a desiccator.
3. *Industrial methylated spirit:* 95 per cent.
4. *Sodium hydroxide soln.:* 0·1 N solution.
5. *Phenolphthalein indicator:* 1·0 per cent. w/v solution of phenolphthalein in industrial methylated spirit.

Method

Macerate 50 to 100 g of prepared sample with 250 ml of chloroform for one minute in the mechanical blender, and filter immediately through a large fluted filter paper into a separating funnel. Collect about 80 ml of filtrate (A) and shake it with 15 ml of distilled water. Filter the

* Measuring and Scientific Equipment Ltd., Crawley, Sussex.

separated, lower layer through a filter paper containing 2 to 3 g of anhydrous sodium sulphate. Determine the concentration of this filtrate (B) by evaporating a known volume at 100°C.

To a 150-ml conical flask add 25 ml of industrial methylated spirit and 1 ml of phenolphthalein indicator, and just neutralise the mixture with 0·1 N sodium hydroxide solution. Add, by pipette, 25 ml of filtrate (B), and titrate with 0·1 N sodium hydroxide solution until the pink colour persists for 15 seconds (titration = V ml).

$$\text{Free Fatty Acid (per cent.)} = \frac{V \times 2{\cdot}845}{\text{Weight of extracted fat in 25 ml of filtrate (B)}}$$

(*d*) *Extract Release Volume:* The prepared sample will be examined for extract release volume by the following method:

Apparatus

1. MSE Homogeniser* with a 100 ml vortex beaker.
2. Filter funnels, Pyrex brand, glass, (10 cm diameter).
3. Filter papers, Whatman No. 1, (18·5 cm diameter).
4. Measuring cylinder, graduated (50 ml).

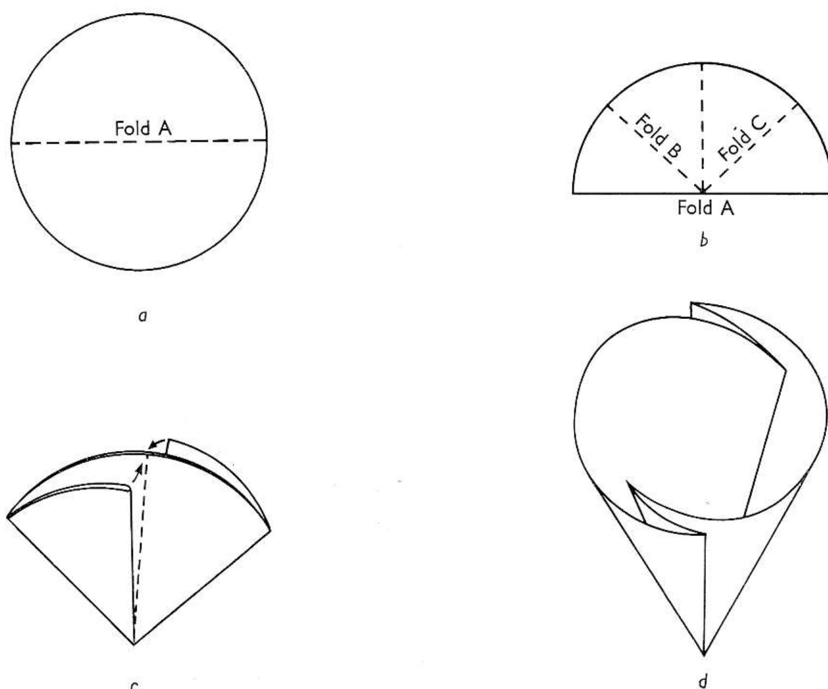

Fig. 1 Method of folding the filter paper for determination of the extract release volume

* Measuring and Scientific Equipment Ltd., Crawley, Sussex.

Reagent

All ingredients shall be of recognised analytical purity.

Extraction reagent—Mix 50 ml of 0·2 M potassium dihydrogen orthophosphate solution (KH_2PO_4) with 3·72 ml of 0·2 M sodium hydroxide solution and dilute with distilled water to 200 ml. Check that the reagent has a pH value of 5·6.

Method

Blend 15 ± 0·1 g of the prepared sample for 2 minutes with 60 ml of extraction reagent in the 100 ml vortex beaker of the homogeniser. Pour the homogenate immediately through a filter paper folded thrice so as to make eight sections (Fig. 1) and collect the filtrate at 21 ± 4°C in the measuring cylinder. The volume of filtrate (in ml) collected in 15 minutes after pouring the homogenate into the funnel shall be deemed the Extract Release Volume.

References

1. Lucke, F., and Geidel, W., *Lebensmitt. Untersuch.*, 1953, **70**, 441.
2. Jay, J. M., *Food Technol. (Champaign)*, 1964, **18** (10), 129.
3. Society for Analytical Chemistry (formerly, the Society of Public Analysts and Other Analytical Chemists), *Analyst*, 1952, **77**, 543.

A Survey of Fruits and Vegetables for Organo-chlorine and Organo-phosphorus Insecticides

by G. J. DICKES and P. V. NICHOLAS
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The incidence of organo-chlorine insecticides in English and imported fruits and vegetables, sampled during 1967, is given, together with that of organo-phosphorus insecticides in selected produce. Results of interest are discussed.

During 1967 fruits and vegetables were sampled and the organo-chlorine insecticide content was determined by GLC methods as in the 1966 survey[1]. In addition, during the period May to September, those crops most likely to be treated with organo-phosphorus insecticides were analysed for these compounds by a GLC method.

Apparatus

In addition to the apparatus previously listed[1], a gas chromatograph (Varian Aerograph) incorporating a caesium bromide thermionic detector and a modified thermionic detector [caesium bromide/alumina (2 : 1)] were used, with hydrogen and air.

Reagents

The following were used in addition to the reagents previously listed[1]:

1. *Chloroform:* of AR quality.
2. *Activated Carbon:* 40 mesh (British Carbo Norit).

Extraction

The method of extraction of organo-chlorine insecticides was that described previously in this Journal[1]. The organo-phosphorus extractions, based on a separation described by Laws and Webley[2], were carried out as follows:

1. Retain the final hexane solution after examination for organo-chlorine compounds. This contains the relatively non-polar organo-phosphorus compounds, including malathion, parathion, phenkapton, phorate, dichlorvos, disulfoton and azinphos methyl.
2. Extract the aqueous phase remaining after the hexane extraction with two separate portions of 10 ml of chloroform and carefully evaporate the combined extract on a water bath to 2 ml. Pass the 2 ml of extract through a chromatographic column containing 5 g of activated carbon followed by 50 ml of chloroform as developing solvent at a flow rate of 2 ml per minute. Carefully evaporate the eluate just to dryness in a rotary evaporator at room temperature, add 0·5 ml of acetone to dissolve

the residue, and make up to 5 ml with hexane (too high a concentration of acetone gives a large solvent peak on GLC analysis). Dry the extract over anhydrous sodium sulphate before injection on to the chromatograph.

This extract contains the relatively polar organo-phosphorus compounds including demeton-S-methyl, dimethoate, mevinphos and phosphamidon.

Procedure

1. ORGANO-CHLORINE RESIDUES

For organo-chlorine insecticide analysis, the column data, GLC and TLC techniques are the same as those used previously[1]. In addition, a TLC clean-up procedure, with hexane as mobile solvent, was used on lettuce extracts in order to differentiate γ-BHC (R_f Value = 0·1) and PCNB (the fungicide pentachloronitrobenzene) (R_f Value = 0·3). Table I of the previous publication[1] gives retention data, temperatures etc. to which should be added methoxychlor, which has a retention time relative to aldrin of 12·4 on 3 per cent. Apiezon L and of 10·4 on 5 per cent. QF-1 medium.

TABLE I

GLC CHARACTERISTICS OF ORGANO-PHOSPHORUS INSECTICIDES

Organo-phosphorus insecticide	Retention relative to phorate	Limit of detection *picogrammes* Commercial detector	Special detector
Dichlorvos	0·17	120	—
Mevinphos	0·35	130	—
Demeton-S-Methyl	0·77	450	110
Phorate	1·00	220	120
Dimethoate	1·10	1,200	140
Disulfoton	1·55	140	—
Phosphamidon	1·67	800	—
Malathion	2·20	500	300
Parathion	2·50	250	—
Phenkapton	11·3	2,000	—
Azinphos methyl	23·0	8,500	—

Column: 20 per cent. SE-30 on 60/80 mesh Chromosorb W.
Temperature: 190°C.
Special Detector: Made of Caesium bromide/alumina in the ratio of 2 : 1, in order to increase the sensitivity for dimethoate.
Limit of Detection: Recorded as 4 times the "noise-level".

2. ORGANO-PHOSPHORUS RESIDUES

Table I shows retention data and limits of detection of the three organo-phosphorus insecticides encountered, together with a further eight which might be found. When a positive GLC result is obtained, it is checked by using a clean-up procedure based on TLC, prior to re-examination by GLC. Details of mobile solvents used for the separation of organo-phosphorus compounds are given by Walker and Beroza[3]. Five μl of the hexane or the acetone/hexane extract are placed on the 20 per cent. SE-30 column in the chromatograph apparatus which incorporates the caesium bromide thermionic detector.

TABLE II

A SURVEY OF ORGANO-CHLORINE INSECTICIDES FOUND IN FRUIT AND VEGETABLES DURING 1967

Fruit or vegetable	Total number examined	Total DDT* Number contaminated	Total DDT* Range *parts per million*	γ-BHC Number contaminated	γ-BHC Range *parts per million*
Apple	88	35	0·0–2·8	50	0·0–0·11
Apple Beverage	38	0	—	1	0·025
Apricot	7	1	0·21	3	0·029–0·20
Asparagus	1	0	—	0	—
Beetroot	6	0	—	3	0·0–0·023
Bilberry	1	0	—	0	—
Blackcurrant	5	0	—	0	—
Broad Bean	11	0	—	3	below 0·002
Cabbage	9	0	—	0	—
Canned Vegetable/Fruit	43	0	—	3	0·0–0·10
Carrot	8	0	—	3	below 0·002
Cauliflower	7	0	—	3	0·0–0·012
Celery	7	0	—	1	0·040
Cherry	15	0	—	5	0·0–0·050
Coffee	31	0	—	6	0·0–0·020
Cucumber	11	0	—	6	0·0–0·034
Currant	37	6	0·0–0·70	2	0·002–0·26
Damson	1	0	—	0	—
Date	7	0	—	0	—
Dried Fruit (mixed)	2	0	—	0	—
Fig	1	0	—	0	—
Gooseberry	8	6	0·010–0·94	2	below 0·002
Grape	9	0	—	1	below 0·002
Grapefruit	44	1	below 0·010	12	0·0–0·056
Green Pepper	8	3	0·068–0·16	1	below 0·002
Leek	6	0	—	2	0·052–0·14
Lemon	8	0	—	2	below 0·002
Lettuce	84	2	0·20–5·3	27	0·0–5·9
Marrow	7	0	—	1	0·051
Melon	2	0	—	0	—
Mushroom	55	6	0·09–1·2	14	0·0–0·36
Onion	9	0	—	1	0·050
Orange	11	0	—	0	—
Parsnip	7	1	below 0·010	4	0·0–3·8
Pea	37	0	—	0	—
Peach	34	3	0·08–0·13	6	0·0–0·04
Pear	65	23	0·0–0·10	8	0·0–0·14
Pineapple	7	0	—	1	0·030
Plum	1	0	—	1	0·017
Pomegranate	4	0	—	0	—
Potato	42	0	—	2	0·0–0·076
Prune	6	1	0·46	0	—
Radish	7	0	—	2	0·0–0·027
Raisin	32	0	—	9	0·0–0·15
Raspberry	6	0	—	1	0·06
Rhubarb	7	0	—	1	below 0·002
Runner Bean	10	0	—	0	—
Spring Green	7	0	—	1	below 0·002
Spring Onion	7	0	—	0	—
Sprout	6	0	—	0	—
Strawberry	31	0	—	3	below 0·002
Sultana	35	5	0·19–0·61	7	0·0–0·76
Swede	9	0	—	4	0·0–0·04
Tangerine	3	0	—	0	—
Tea	36	0	—	1	0·01
Tomato	73	16	0·0–0·30	20	0·0–0·082
Watercress	36	6	0·0–0·45	17	0·0–0·042

* Total DDT includes *pp'*- and *op'*-DDT, plus *pp'*-DDE

Results and Discussion

A total of 1,095 samples has been analysed for organo-chlorine insecticides, and of these, 349 have been examined for organo-phosphorus residues. The results are given in Tables II and III. Crops were sampled in season and some fruits and vegetables, *viz*. apple, lettuce, peach, pear and tomato, were sampled at intervals throughout the year. It was considered more profitable to restrict the survey of organo-phosphorus insecticides to those crops sampled in the Summer months.

TABLE III

A SURVEY OF ORGANO-PHOSPHORUS INSECTICIDES IN FRUITS AND VEGETABLES DURING 1967

Fruit or Vegetable	Number examined	Number containing Organo-phosphorus residue
Apple	45	nil
Apricot	6	nil
Bilberry	1	nil
Broad Bean ..	9	nil
Blackcurrant ..	5	1 contained 0·95 p.p.m. of demeton-S-methyl
Celery	7	nil
Cherry	15	1 contained 2·36 p.p.m. of demeton-S-methyl
Currant	4	nil
Damson	1	nil
Gooseberry ..	8	nil
Grape	1	nil
Lettuce	50	1 contained 5·8 p.p.m. of malathion 1 contained 0·48 p.p.m. of dimethoate
Marrow	7	nil
Melon	2	nil
Mushroom ..	51	1 contained 0·25 p.p.m. of dimethoate
Pea	34	nil
Peach	30	nil
Pineapple ..	2	nil
Potato	6	nil
Radish	7	1 contained 0·072 p.p.m. of demeton-S-methyl
Raisin	1	nil
Raspberry ..	6	nil
Rhubarb ..	3	nil
Runner Bean ..	9	nil
Spinach	1	nil
Strawberry ..	31	1 contained 6·5 p.p.m. of malathion
Sultana	2	nil
Watercress ..	5	nil

As in the previous survey[1], apple and pear peels were taken for organo-chlorine insecticide analysis and only when a sample proved positive was the whole fruit examined. The whole fruit was used in the cases of the 45 apple samples retained for organo-phosphorus analysis.

In addition to the results listed in Table II, TDE, a breakdown product of DDT, was found in 3 samples of apples at concentrations of 0·14, 0·01 and 0·01 p.p.m. This substance was also found in a tomato sample at a concentration of 0·10 p.p.m. and three samples of pears contained 0·073, 0·010 and 0·009 p.p.m. An apple sample, thought to be from the U.S.A., contained 0·66

p.p.m. of methoxychlor (as well as 0·18 p.p.m. of DDT and 0·005 p.p.m. of γ-BHC).

The pattern of results on apple samples was the same as in the previous survey *i.e.*, almost half contained DDT and more than half contained γ-BHC. A quarter of the samples contained both insecticides. All except 3 of the samples with a DDT residue contained less than 0·1 p.p.m., the 3 having concentrations of 2·8, 0·62 and 0·33 p.p.m. Apart from a French apple sample of 0·11 p.p.m. of γ-BHC, all samples contained less than 0·01 p.p.m. of this substance. No apple samples examined contained an organo-phosphorus residue. At least 23 varieties of apples originating from at least 8 different countries were covered, but insufficient information was available to highlight one particular variety of fruit or country of origin as having a higher incidence of organo-chlorine insecticide residue than any other. Apple beverages, which included ciders, juices and soft drinks (with the exception of the juice containing 0·025 p.p.m. γ-BHC), were free of organo-chlorine insecticide.

In comparison with the previous survey, pears showed a drop in the incidence of DDT and γ-BHC. One third of the total contained DDT, 8 samples contained γ-BHC, and only two samples contained both insecticides.

The 20 per cent. incidence of DDT in tomatoes was the same as in the previous survey, but no examples of concentrations greater than 0·3 p.p.m. were observed; γ-BHC was found in approximately the same amounts as before but was not quite so common.

As in the previous survey, some relatively high results for γ-BHC were obtained from lettuce sampled in the period January to March. These were Dutch or English and 8 samples contained γ-BHC at concentrations greater than 1 p.p.m., the highest being a Dutch sample containing 5·9 p.p.m. (plus 0·20 pp.m. of DDT). Another Dutch sample contained 5·3 p.p.m. of DDT and 0·50 p.p.m. of γ-BHC. Lettuce has provided the highest DDT and γ-BHC-containing samples in the survey and two other samples contained 5·8 p.p.m. of malathion and 0·48 p.p.m. of dimethoate respectively.

Mushrooms were included in the survey because this food commodity appeared to have been by-passed in residue studies. Six out of the 55 samples contained DDT, one sample having 1·2 p.p.m. Fourteen samples contained γ-BHC and five contained both γ-BHC and DDT. One of these five in addition to 0·09 p.p.m. of DDT and 0·10 p.p.m. of γ-BHC, contained 0·25 p.p.m. of dimethoate and also 14·5 p.p.m. of the fungicidal dithiocarbamate, zineb[4].

Although only 8 samples of gooseberries were submitted, 6 of these contained DDT. Concentrations were 0·94, 0·91, 0·70, 0·55, 0·52 and 0·01 p.p.m. Of the 7 samples of apricots, one sample of South African origin contained 0·21 p.p.m. of DDT and one Hungarian sample and 2 Spanish samples contained 0·12, 0·20 and 0·029 p.p.m. of γ-BHC respectively. Three of the 34 peach samples contained DDT at levels of 0·13, 0·12 and 0·08 p.p.m.

As in the previous survey, some dried fruits inexplicably gave residues of DDT and γ-BHC. One prune sample contained 0·46 p.p.m. of DDT and 9

of the 32 raisin samples contained γ-BHC up to a maximum of 0·15 p.p.m. Six of the 37 samples of currants contained DDT. One sample, containing 0·36 p.p.m. of DDT also had a residue of 0·26 p.p.m. of γ-BHC. Five of 35 sultana samples, all Australian in origin, contained DDT at levels of 0·61, 0·41, 0·31, 0·26 and 0·19 p.p.m. Seven other Australian sultana samples contained γ-BHC, the two highest concentrations being 0·76 and 0·17 p.p.m.

The occurrence of DDT in 3 out of 8 samples of green peppers was surprising. The concentrations, in p.p.m. were 0·16 (Israel), 0·10 (South Africa) and 0·068 (Uganda).

Three of the 7 parsnips analysed contained 3·8, 0·11 and 0·022 p.p.m. of γ-BHC respectively. Watercress, as in the previous survey, showed a high incidence of γ-BHC although the amounts recorded were small. In contrast, however, DDT was found in watercress this time, albeit in minute trace amounts except for an English sample which contained 0·45 p.p.m.

The highest organo-phosphorus residue found was that of 6·5 p.p.m. of malathion in a sample of English strawberries. Three items, *viz.* blackcurrant, cherry and radish, contained demeton-S-methyl. The cherry sample, containing 2·36 p.p.m. was Italian. Assuming that this fruit was transported by sea, and allowing for marketing delay, this level of insecticide would have been much higher at harvest, since organo-phosphorus insecticides are generally metabolised rapidly.

The general conclusion of this second survey, which showed a sampling bias towards items which contained a high incidence of organo-chlorine insecticide as experienced from the 1966 results, suggests that residues higher than average are to be found in the following products: apples, apricots, blackcurrants, cherries, dried fruits, gooseberries, green peppers, lettuces, mushrooms, parsnips, peaches, radishes, strawberries, tomatoes and watercress.

Of the samples examined for organo-chlorine insecticide, 240 contained γ-BHC and 115 contained DDT. Of the samples containing γ-BHC, 148 had a concentration of less than 0·01 p.p.m. which is the limit of detection for DDT. Since this leaves 92 other samples all contaminated with γ-BHC compared with the 115 containing DDT, it is concluded that these two insecticides have approximately the same incidence.

The incidence of organo-phosphorus residues is not as high as that of organo-chlorine residues; however, in comparing the two groups, the relative limits of detection must be taken into account. Taking the limit of detection of organo-phosphorus insecticides to be 0·05 p.p.m. then 7 samples or 2 per cent. exceed this limit, whereas 114 (or 10 per cent.) of the samples contained organo-chlorine residues at or above the 0·05 p.p.m. level. The results of this survey suggest, therefore, that there is approximately a five times greater possibility of detecting organo-chlorine residues in a sample of fruit or vegetables than there is of finding organo-phosphorus above the 0·05 p.p.m. level.

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References

1. Dickes, G. J., and Nicholas, P. V., *J. Assoc. Publ. Analysts*, 1967, **5**, 52.
2. Laws, E. Q., and Webley, D. J., *Analyst*, 1961, **86**, 249.
3. Walker, K. C., and Beroza, M., *J. Ass. Off. Agric. Chem.*, 1963, **46**, 250.
4. Pagington, J. S., *J. Assoc. Publ. Analysts*, 1968, **6**, 27.

Daniel Donald Moir, M.Sc., F.R.I.C.

PRESIDENT 1962–64

By the sudden and quite unexpected death of Donald Moir, at his home, after a morning's gardening, on the 21st January, 1968, the Association of Public Analysts has lost one of its most valued members.

Moir was at school in Grays, Essex, and entered East London College, now Queen Mary's College, in 1924, obtaining first a B.Sc. in Chemistry and then an M.Sc. in 1927. After short periods with the Tottenham Gas Works and the Maypole Dairy Co., he joined, in the summer of 1929, the staff of the late Thomas Tickle, Public Analyst for the County of Devon, whose laboratory was in Exeter. Mr. R. E. Hedger, now on the editorial staff of "The Analyst", was engaged by Tickle at the same time, and he and Moir formed a friendship that was broken only by Moir's death. In a letter about those early days in Exeter, Hedger has this to say:

> "I worked in close association with him, sharing many problems in the analytical field of consulting work. I found him a most enthusiastic and industrious worker, and he soon developed very marked skills, showing much resource and inventiveness.
>
> "He was, in consequence, thought very highly of by Tickle and his colleagues, and was well liked by all. He was a quiet young man, and, while I would not say that he was of a shy disposition, he did not spend much time in conversation, but largely in preparing his own thoughts. In this last connection, I have rarely met anybody who could concentrate as he did; often, he would disregard remarks made to him when in process of working out some problem or other. There is no doubt that he could be very single-minded and pursue a matter until fully resolved to his satisfaction, and there was no question of abandoning it if it became difficult; a most admirable characteristic."

C. V. Reynolds, now Public Analyst for Devon and other Authorities and successor to Tickle in his practice, was also an assistant in the laboratory with Moir, and pays tribute to "his originality of mind and his ability to tackle the most difficult analytical problems with considerable success. . . . It was clear to those of us who knew Donald Moir in those days that he would achieve eminence in his profession."

At the end of 1934, Moir left Tickle's laboratory to become chief assistant to Edward Hinks, Public Analyst for the County of Surrey and other Authorities, replacing H. Amphlett Williams, who had moved on after obtaining a public appointment. He served under Hinks until the latter died in 1946, when Moir was naturally appointed as his successor. Amphlett Williams' laboratory was very close to Moir's, and they saw a good deal of each other. Amphlett Williams writes "Moir had a logical mind and if he loved to take the opposite side in a technical discussion his conclusions were almost invariably sound. The "almost" is used because I can remember just one instance in thirty-four years where we found ourselves in opposite camps!" Because of the proliferation of the Borough Authorities in the Surrey area, Moir soon found himself with greatly increased responsibilities; but he discharged his duties with great competence and conscientiousness and became a most esteemed member of his profession, both with Local Authorities and with his colleagues.

On a personal note, I can truthfully say that if ever I found myself faced with a difficult problem of interpretation of the law relating to some article of food or the best way to tackle some complex analysis, and felt that I wanted a second opinion, it was almost always to Donald Moir that I turned; ten minutes on the telephone was usually enough to settle the point or at least to clarify my mind, while, if occasionally a solution was not arrived at in that time, I knew that I need not be ashamed of admitting that I did not know the answer. Moreover, if the question was one on which Moir had already made up his mind, he could always produce clear and logical reasons for his view—though, like a true scientist, he was equally ready to listen to arguments for the opposite opinion and (on one occasion at least!) to change his mind.

Donald Moir was an active and enthusiastic member of the Association of Public Analysts, and he served it faithfully and well. At the 1955 Annual General Meeting he was elected as Honorary Secretary, in succession to Eric Voelcker, and he remained in that office until 1959. During that period he impressed all members of Council, not only by the quietly efficient way he carried out his duties, but also by the admirable letters he wrote on behalf of the Association to the Ministry and other official bodies. His command of English, coupled with his clarity of mind, resulted in a lucid literary style and a choice of the right word or phrase that could not have been bettered; he expressed the views of Council much more clearly than we had ever managed to express them ourselves! In 1959 he relinquished his office and was promptly elected as ordinary member of Council, only to be appointed as Vice-President in 1960 and as President from 1962 to 1964. He brought to that office a distinction and an achievement that we who served under him will not forget.

He will also be remembered for his kindness in acting as host to the Standards Committee, which has held meetings in his office since January 1960. He was Chairman of the Committee from the 1967 A.G.M. until his death.

It may surprise some who knew him only in later years to learn that as a young man, Donald was a very active walker and runner. He took up cross-country running with the Orion Harriers before he went to Exeter, winning their 10-mile handicap in 1928, and continued with the Exeter Harriers. He was a great walker also, knowing Dartmoor intimately and spending many holidays walking in the Highlands of Scotland, the Pyrenees, and elsewhere.

Donald married, in March 1934, Mary Hockaday, directly after he joined Hinks; there are two sons and a daughter. Those of us who have regularly attended the Annual General Meetings of the Association—and our wives perhaps even more—soon came to know Mrs. Moir, whose sincerity and friendliness have done much to promote the 'happy family' atmosphere that has become a characteristic of these meetings. The Association mourns, with her and her family, Donald Moir's death; they have lost a devoted husband and father, we an eminent and respected colleague whose services will long be remembered.

E. C. Wood.