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The Retention of Antibiotic Residues in Milk Following Dry Cow Therapy

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The results of a study into the retention of antibiotic residues in the bovine udder following dry cow therapy are presented. Milk samples were tested for the presence of antibiotics and the results are discussed.

The Milk Marketing Board's conditions of sale stipulate that a producer shall not deliver any milk which contains antibiotic residues.

If mastitis arises during lactation it may require treatment with an appropriate antibiotic. However, the use of antibiotics in dry cow medication has now been shown to achieve control of herd infections at calving. There is little published information about the extent of the retention of antibiotics in the bovine udder following dry cow therapy[1].

It seems unfair to some enforcement authorities and members of the farming community that some producers may be penalised for supplying milk containing antibiotics even though the manufacturer's instructions for the use of the medication have been strictly followed.

Outline of Survey Procedure

The study was made possible through appropriate liaison between the Dyfed Trading Standards Department, the Public Analyst, the Microbiological Department of the Agricultural Development and Advisory Service (ADAS)/ Welsh Office Agricultural Department and the Milk Marketing Board. The antibiotic preparations studied involved Ampicillin, Cloxacillin, Penicillin and Streptomycin, all formulated in slow release bases. All cows were treated at the beginning of the drying-off period in accordance with the manufacturer's recommended time limits, and the milk was withheld for four days after calving. Cows receiving medication for other purposes (e.g. mastitis) were excluded from the survey.

Five farms were selected, each of which kept excellent records of milk yields and details of treatment, etc. All were within a short distance of the Public Analyst's laboratory in Carmarthen. The herds involved were milked twice daily morning and evening.

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Morning samples were taken daily from newly calved cows in each herd throughout the four day period after calving, starting with the colostrum. These samples from the individual cows were taken after milking into sterile containers. The samples were conveyed immediately in an ice-box to the laboratory. If antibiotic was detected in the final sample of the four day period further morning samples were taken daily until two consecutive negative results were obtained.

The samples were tested for antibiotics by the Delvotest and Intertest procedures. Samples giving a positive response for antibiotic residues were further tested quantitatively by the Microbiological Department of ADAS/ Welsh Office Agricultural Department. Some samples giving positive antibiotic residues were examined by the laboratory of the Government Chemist to identify specifically the antibiotics present[2].

Delvotest Procedure

The Delvotest P standard diffusion test[3] for milk involves adding 0·1 ml of milk to a glass ampoule containing a suspension of *Bacillus stearothermophilus* var *calidolactis* spores in 2 ml of 0·9 per cent. solidified agar. A nutrient tablet containing the pH indicator bromocresol purple is placed on top of the agar and the ampoule incubated in a water bath at 64°C for 2½ h. In the absence of antibiotics or inhibitory substances bacterial growth occurs, producing acid which is detected by the indicator changing from blue to yellow. If antibiotics are present they inhibit bacterial growth and no colour change occurs.

The Delvotest is sensitive down to 0·003 International Units (IU) of penicillin per ml of milk.

Before normal milk testing, the sample was flash heated to 95°C and immediately cooled to destroy any naturally occurring inhibitory substances. Some colostrum samples however clotted on heating, and the Delvotest procedure had to be modified, 0·1 ml of colostrum being added to the agar, and allowed to stand for 15 min at room temperature so that any antibiotic present in the colostrum could diffuse into the agar. The colostrum was removed by a pasteur pipette and replaced by 0·1 ml of sterile distilled water. The nutrient indicator tablet was added, followed by incubation and reading of the sample as for normal milk. The sensitivity of this modified Delvotest was the same as for normal milk viz. 0·003 IU penicillin per ml.

Intertest Procedure

Intertest accuspheres (supplied by Intervet Laboratories, Cambridge) contain a freeze-dried culture of a strain of *Streptococcus thermophilus*, nutrients and bromocresol purple indicator. Naturally occurring substances in the milk can inhibit the test organism, and the milk sample is flash-heated to 95°C and cooled prior to the test. The contents of the accusphere are mixed with the heat treated milk sample and incubated in a water bath at 45°C for 4 h. Provided no antibiotic or other inhibitory substances are present, acid, which is detected by the change of indicator colour from blue to yellow, is produced. Antibiotics inhibit bacteriological growth and acid production to a degree depending on the antibiotic and its concentration. The colour produced is matched against a chart

supplied with the Intertest. The Intertest procedure used in this project was sensitive to 0·02 IU of penicillin per ml of milk.

Procedure for Quantitative Antibiotic Determination

The range of antibiotic concentrations in the samples was determined using a dilution method in conjunction with the Intertest procedure.

Initially, successive tenfold dilutions of the sample (e.g. 10^{-1} to 10^{-4}) were prepared in nonsterile, antibiotic free, 10 per cent. reconstituted skim milk. Each set of dilutions and the undiluted sample were flash heated to 95°C and tested by the Intertest procedure.

The antibiotic-positive dilution containing the least amount of sample was prepared again using skim milk. This diluted sample was further diluted according to the dilution series in the Quantification Table, (Table I) and tested by the Intertest procedure.

Antibiotic levels were reported in terms of IU of penicillin per ml.

TABLE I
QUANTIFICATION TABLE—INTERTEST DILUTION PROCEDURE

Diluted sample *ml*	Skim milk *ml*	Penicillin in diluted sample *IU/ml*
10·00	0·00	>0·02
5·00	5·00	>0·04
2·86	7·14	>0·07
2·00	8·00	>0·10
1·50	8·50	>0·13
1·25	8·75	>0·16
1·00	9·00	>0·20

TABLE II

DATA RELATING LENGTH OF DRY PERIOD TO NUMBER OF DAYS BEFORE MILK WAS CLEAR OF DETECTABLE ANTIBIOTIC RESIDUES ACCORDING TO THE DELVOTEST (I.E. LESS THAN 0·003 IU PENICILLIN PER ML OF MILK)

Dry period *days*	Number of cows clear in: 4 days	5 days	6 days	7 days	8 days	9 days	10 days	11 days	12 days	13 days	14 days	Total number of cows
0–28	1											1
29–35	1										1*	1
36–42					1							1
43–49	1		1									2
50–56	5	2										7
57–63	14		1									15
64–70	7											7
71–77	3											3
78–84	2	2										4
85–91	3											3
92+	1											1
Total	38	4	2		1						1	46
Per cent.	82·4	8·8	4·4		2·2						2·2	100

* Cow calved prematurely.

Results

Results in Table II relate length of dry period to the number of days before the milk was clear of detectable amounts of antibiotic residues, pertaining to the sensitivity level of the Delvotest procedure (i.e. 0·003 IU of penicillin per ml).

According to the Intertest, of the forty-six cows included in the study, samples from only one contained 0·02 IU or more of penicillin or equivalent per ml of milk after the 7th milking (i.e. the fourth day after calving). It was recorded that the cow in question had calved prematurely seven days before the expected date. Results are tabulated in Table III.

TABLE III

APPROXIMATE LEVELS OF ANTIBIOTICS IN TERMS OF PENICILLIN FOR SAMPLES TAKEN FROM COW HAVING 0·02 IU PER ML AND ABOVE AFTER THE 7TH MILKING

Milking	Days after calving	Approx. levels of antibiotics in terms of penicillin *IU/ml*
1st (Colostrum)	1	0·20–0·40
3rd	2	0·07–0·10
5th	3	0·02–0·04
7th	4	0·04–0·07
9th	5	0·02–0·04
11th	6	0·02–0·04
13th	7	0·02–0·04
15th	8	0·04–0·07
17th	9	0·01–0·02
19th	10	0·01–0·02
21st	11	0·02–0·04
23rd	12	0·02–0·04
25th	13	0·01–0·02

Antibiotics in selected samples were identified by electrophoresis as being the same as those used in the original dry cow therapy preparations.

Conclusions

An examination of the data accumulated in this study shows that the antibiotic was nearly always removed from the bovine udder within the first four days of lactation. It was shown that if the dry cow antibiotic preparations used were administered within the recommended limits before normal calving, the levels of antibiotic detected in the milk five days and more after calving were less than 0·02 IU penicillin or equivalent per ml of milk. However, the results (Table III) for the one cow that calved prematurely indicate that, in some situations, detectable levels of antibiotic can be excreted in the milk for a prolonged period. In accordance with recommended practice for such circumstance, milk samples should be taken at regular intervals after premature calving and the milk discarded until antibiotics can no longer be detected.

It should also be noted that the extended period of low level antibiotic excretion after premature calving is similar to that shown by lactating cows that have been treated for mastitis with slow release dry cow antibiotics instead of quick release preparations.

Samples tested before the 9th milking showed that milk from 9 (20 per cent.) of the 45 cows that calved normally contained antibiotic levels above 0·02 IU of penicillin per ml during the first four days after calving. The highest levels recorded were 0·20–0·40 IU/ml. This indicates that for cows under dry cow antibiotic therapy, consideration should be given to preventing any residual antibiotic contaminated milk from entering the bulk tank during the four days after calving. Other work has shown that antibiotic-containing milk residues left in recorder jars after milking can subsequently contaminate the farm bulk tank consignment when flushed through by the next cow's milk[4].

It can be deduced from the results of this study that, for the preparations used, the antibiotic residues retained in the bovine udder and detectable in the milk after the 4th day of lactation would be less than 0·02 IU/ml, provided the administration of the dry cow therapy has been carried out according to the manufacturer's instructions.

However, if the acceptability level for antibiotic residues in milk is lower than 0·02 IU/ml, as occurs for example in Holland (0·01 IU/ml) and Eire (0·003 IU/ml), the incidence of failure will be greater. Table II shows that after the 4 day withholding period three (7 per cent.) of the cows that calved normally had antibiotic levels between 0·003 and 0·02 IU/ml on the 5th day after calving.

References

1. Smith, A., Westgarth, D. R., *et al.*, *Vet. Rec.*, **81**, 504–510.
2. Smither, R., and Vaughan, D. R., *J. Appl. Bact.*, 1978, **44**, 421–429.
3. "Delvotest, P. Standard diffusion test for the determination of antibiotic residues in milk". Gist Brocades NV, Industrial Enzymes Division, P.O. Box 1, Delft, Netherlands.
4. Bigg, N. R. and Hacking, A., "Antibiotics in Milk. First Welsh Agricultural Research and Development Conference", HMSO, London, 1984.

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Prediction of the Energy Value of Compound Feeds

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Collaborative research programmes in the UK over the period 1979–1983 have studied the relationships between the digestible energy (DE), or metabolisable energy (ME), value of ruminant, pig and poultry compound feeds and their chemical composition. As well as accurate animal measurements of DE or ME, attention was paid to within and between laboratory variation for each chemical parameter measured. Over 200 regression equations were found capable of predicting DE or ME with a precision of better than ± 0.5 MJ/kg, dry matter from one or more chemical determinations on the compound feed.

A joint Working Party of the Agricultural Development and Advisory Service (ADAS), the Council of the Scottish Agricultural Colleges (COSAC) and the United Kingdom Agricultural Supply Trade Association (UKASTA) members concerned with advice to livestock farmers has considered the research reports and recommended equations suitable for use in three different situations:

(a) voluntary routine use by ADAS, COSAC and UKASTA advisers
(b) possible future incorporation into legislation on compound feeds
(c) reference purposes

The Working Party's findings have been published in full, and this Technical Note presents a brief summary of the research programmes and the Working Party's recommendations for equations suitable for use in each of the three categories listed above. Implementation of the Report is at present under discussion between representatives from the Ministry of Agriculture, Fisheries and Food (MAFF) NFU, UKASTA, Local Authorities Co-ordinating Body on Trading Standards (LACOTS) and SAC.

This note is based on the Report of a joint Working Party on this subject, which was circulated in December, 1984, and has now been published[1]. The Working Party was set up in 1981, with representatives from the United Kingdom Agricultural Supply Trade Association, (UKASTA), the Agricultural Development and Advisory Service, (ADAS) of the Ministry of Agriculture, Fisheries and Food, (MAFF) and the Council of the Scottish Agricultural Colleges, (COSAC). This was to consider the Rowett Research Institute Feedingstuffs Evaluation Unit Report No. 3 on ruminant compound feeds[2]. Since then there have been studies published on poultry compound feeds[3,4], and pig compound feeds[5].

The terms of reference of the Working Party were: (1) To consider the published reports and to assess their implications for Feed Manufacturers and

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advisers generally. (2) To select, and test further if necessary, methods of predicting the metabolisable (and digestible) energy of compound feeds which are capable of routine use, economy of resource input and adequate accuracy of prediction for: (a) voluntary routine use by ADAS, COSAC, and UKASTA; (b) possible future incorporation into legislation on energy declaration for compound feeds; (c) reference purposes.

Energy Units For Animal Feeds

In the UK, the Megajoule, (MJ), is now the agreed metric unit for energy values, (1 MJ = 4·184 Mcal). Metabolisable energy (ME) is used for ruminant feeds, apparent metabolisable energy (AME) is used for poultry feeds, and digestible energy (DE) for pig feeds. All these units have a common theoretical basis, arising from metabolism or digestibility trials, in which energy consumed and energy excreted are measured over a period of many days.
Metabolisable energy for ruminants is defined as:

Gross energy, (GE) of feed eaten—(GE of faeces + GE of urine + GE of methane) and the unit used is MJ/kg of dry matter (DM).

In the case of poultry, there is no methane production and the faeces and urine cannot be separated. Thus ME has a slightly different meaning in the case of poultry and the unit used is MJ/kg *as fed*.

The unit used for pig feeds is the DE as MJ/kg *as fed*. DE omits urine energy as well as methane from the calculation, although it is known that pigs do produce small amounts of methane in the lower gut.

The technical terms, abbreviations and analytical methods used are shown in Table I.

Statistical Aspects of the Problem

An important part of the Working Party's studies were concerned with establishing the within laboratory variation or repeatability, r, and the between laboratory variation or reproducibility, R, of all the analytical methods which have been used in the three reports. A statistical technique to include the reproducibility, R, in the consideration of the prediction errors of the equations was available, based on a modification of the calculation of residual standard deviation, assigned the symbol, S''[2,3,4].

Criteria for Selection of Equations

Equations were then selected primarily on the basis of those with the lowest S'' value. Goodness of fit and possible bias at the extremes were also considered. Additionally the number of additional parameters above those already currently determined were to be minimised. Speed and cost of the determinations were assigned different weightings for each of the three classes of intended use specified in the Working Party's terms of reference.

TABLE I
GLOSSARY OF TERMS USED, ABBREVIATIONS AND ANALYTICAL METHODS

Chemical analyses	Abbreviation	Analytical Method
Acid detergent fibre	ADF	Goering & Van Soest, 1970[6]
Cellulase digestibility	NCD	Dowman & Collins, 1977, 1982[7,8]
Crude fibre	CF	Feedingstuffs (Sampling & Analysis) Regulations, 1982[9]
Crude Protein	CP	Feedingstuffs (Sampling & Analysis) Regulations, 1982[9]
Ether extract	EE	Feedingstuffs (Sampling & Analysis) Regulations, 1982[9]
Modified acid detergent fibre	MADF	Clancy & Wilson, 1966[10]
Neutral detergent fibre	NDF	Wainman *et al.*, 1981[12]
Starch	STA	EC Regulation 72/119/EEC, 1972[11]
Sugar	SUG	AOAC 10th Edition, 1965, Methods 29.039 & 43.012[12]
Total ash	TA	Feedingstuffs (Sampling & Analysis) Regulations, 1982[9]
Unsaturated: saturated fatty acid ratio	USR	
Energy terms		
Apparent metabolisable energy	AME	
Digestible energy	DE	
Gross energy	GE	
Megajoule, unit of energy	MJ	
Metabolisable energy	ME	

Recommended Prediction Equations for the Energy Value of Compound Feeds

Equations intended for prospective legal purposes were limited to those parameters required by the current UK Feedingstuffs (Sampling and Analysis) Regulations[9], i.e. crude protein, crude fibre, ether extract and total ash. To these were added starch and sugar determinations by the EEC official methods[11].

For voluntary use, a wider choice of method was possible, provided the methods were rugged, rapid and cheap to carry out. Neutral detergent fibre for pig feeds and cellulase digestibility for ruminant feeds are examples. For reference purposes, no such constraints should be applied, and the most accurate equation should be recommended, even if the necessary work is expensive and slow to carry out.

The Working Party's recommendations for the three areas of intended use are given below (Tables II–IV). The equation numbers are those assigned in the relevant research reports, except those with the prefix U, which were derived by UKASTA from the original database. The suffix "R" indicates a recalculated equation for poultry feeds, following the discovery of a minor error in one of the AME values in the poultry database.

Whilst these equations were agreed between the three parties represented on the Working Party, the implementation of the proposals is under discussion by a joint MAFF/UKASTA/National Farmers Union/Local Authorities Co-ordinating Body on Trading Standards (LACOTS) Working Party. Reservations have been expressed about the ruminant equations and additional work is in progress.

TABLE II

EQUATIONS FOR THE PREDICTION OF THE ME* OF RUMINANT COMPOUND FEEDS

(a) *For voluntary use:* Equations U1 or U3.

$$ME = 11{\cdot}78 + 0{\cdot}0654CP\% + 0{\cdot}0665EE\%^2 - 0{\cdot}0414EE\% \times CF\% - 0{\cdot}118TA\%$$

$S'' = 0{\cdot}36MJ$ (U1)

or

$$ME = 13{\cdot}83 - 0{\cdot}488EE\% + 0{\cdot}0394EE\% \times CP\% - 0{\cdot}0085MADF\% \times CP\% - 0{\cdot}138TA\%$$

$S'' = 0{\cdot}33MJ$ (U3)

(b) *For legislation*: Equation U1 as above.

(c) *For reference purposes*: Equation U2.

$$ME = 11{\cdot}56 - 2{\cdot}375EE\% + 0{\cdot}030EE\%^2 + 0{\cdot}030EE\% \times NCD\% - 0{\cdot}034TA\%$$

$S'' = 0{\cdot}32MJ$ (U2)

*For explanation of abbreviations see Table I. The equation numbers in parentheses relate to the original research. S'' represents the residual standard deviation.

TABLE III

EQUATIONS FOR THE PREDICTION OF THE DE* OF PIG COMPOUND FEEDS

(a) *For voluntary use*: Equations 1d or 22d.

$$DE = 17{\cdot}49 + 0{\cdot}157EE\% + 0{\cdot}070\ CP\% - 0{\cdot}325TA\% - 0{\cdot}149NDF\%$$

$S'' = 0{\cdot}44MJ$ (1d)

or

$$DE = 17{\cdot}95 + 0{\cdot}01EE\%^2 + 0{\cdot}069CP\% - 0{\cdot}305TA\% - 0{\cdot}151NDF\%$$

$S'' = 0.43MJ$ (22d)

(b) *For legislation*: Equations 16d or CF.

$$DE = 5{\cdot}98 + 0{\cdot}188EE\% + 0{\cdot}181CP\% + 0{\cdot}115STA\%$$

$S'' = 0.49MJ$ (16d)

or

$$DE = 17{\cdot}38 + 0{\cdot}114EE\% + 0{\cdot}105CP\% - 0{\cdot}402TA\% - 0{\cdot}317CF\%$$

$S'' = 0.59MJ$ (CF)

(c) *For reference purposes*: Equations 1d or 22d.

*For explanation of abbreviations see Table I. The equation numbers in parentheses relate to the original research. S'' represents the residual standard deviation.

RUMINANT COMPOUND FEEDS

The chemical specifications of the 24 compound feeds studied were as follows, to cover the range of chemical composition found in practice for this class of animal:

(1) Ether extract, (EE), either 2–4 or 5–7 per cent. in dry matter, (DM)
(2) Crude protein, (CP), either 12–14·9, 15–17·9 or 18–21 per cent. in DM
(3) Crude fibre, (CF), either 4–6 or 8–12 per cent. in DM
(4) ME values were to cover the range 9 to 14 MJ/kg of DM.

The original Rowett Report[2] did not consider the use of quadratic or product terms. Further work on the original database by the UKASTA members of the Working Party, resulted in equations of increased precision, particularly at the extremes of crude fibre and ether extract content. The recommended equations, *on a dry matter basis*, are given in Table II.

TABLE IV.

EQUATIONS FOR THE PREDICTION OF THE AME* OF POULTRY COMPOUND FEEDS

(a) *For voluntary use*:	Equations 32R, 74R or 77R.	
AME = 5·39 + 0·113CP% + 0·281EE% + 0·113STA% − 0·136CF%	$S'' = 0{\cdot}36$MJ	(32R)
or		
AME = 5.39 + 0.103CP% + 0·282EE% + 0·114STA% − 0·062NDF% + 0·095USR%	$S'' = 0{\cdot}34$MJ	(74R)
or		
AME = 0·345EE% + 0·165CP% + 0·172STA% + 0·158SUG%	$S'' = 0{\cdot}43$MJ	(77R)
(b) *For legislation*:	Equations 32R or 77R as above.	
(c) *For reference purposes*:	Equations 32R, 74R or 77R as above.	

*For explanation of abbreviations see Table I. The equation numbers in parentheses relate to the original research. S'' represents the residual standard deviation.

PIG COMPOUND FEEDS

The chemical specifications of the 36 compound feeds studied were as follows, to cover the range of chemical compositions found in practice for this class of animal:

(1) Ether extract, (EE), to be 2, 4 or 8 per cent. in the air dry feed
(2) Crude protein, (CP), to be either 14 or 20 per cent. in the air dry feed
(3) Crude fibre, (CF), to be 2·5, 5 or 10 per cent. in the air dry feed
(4) Starch content to be either <40 or >40 per cent. in the air dry feed.

The recommended equations, *all on a dry matter basis*, are given in Table III.

POULTRY COMPOUND FEEDS

The chemical specifications of the 32 compound feeds studied were as follow, to cover the range of chemical compositions found in practice for poultry feeds:

(1) Ether extract (EE), to be 2, 4, 8 or 16 per cent. in the air dry feed
(2) Crude protein, (CP), to be either 12 or 25 per cent. in the air dry feed
(3) ME values to be 9, 11, 13 or 15 MJ/kg of air dry feed.

The recommended equations, *all on a dry matter basis*, are given in Table IV.

Use of Recommended Equations

The application of this series of prediction equations to a particular set of analyses of a sample of compound feed, should only be made if the analyses lie within the range of the original data set, described above. Particularly in the case of ruminant feeds, manufacturers now market feeds with both higher ether extract and crude fibre contents than those set when the Rowett study was started in 1979. Extrapolation beyond the data is always risky, but in the case of fitted equations with square and product terms, it is doubly so.

References

1. Alderman, G., in "Recent Advances in Animal Nutrition", W. Haresign, (Ed.), Butterworths, London, 1985, pp. 3–52.
2. Wainman, F. W., Dewey, P. J. S., and Boyne, A. W., "Feedingstuffs Evaluation Unit, Rowett Research Institute, Third Report 1981". Department of Agriculture and Fisheries for Scotland, Edinburgh, 1985.
3. Fisher, C., "Energy values of poultry compound feeds. Occ. Pub. No. 2". Poultry Research Centre, Rosslin, Midlothian, 1982.
4. Fisher, C., "Report on a Ring Test to evaluate the proposed EEC equation for the declaration of ME values, poultry. Occ. Pub. No. 3". Poultry Research Centre, Roslin, Midlothian, 1983.
5. Morgan, C. A., Whittemore, C. T., Phillips, P., and Crooks, P., "The Energy Value of Compound Foods for Pigs", Edinburgh School of Agriculture, 1984.
6. Goering, H. H. and Van Soest, P. J., "U.S. Department of Agriculture Handbook No. 379", Washington, D.C. 1970.
7. Dowman, M. J., and Collins, F. C., *J. Sci. Fd. Agric.*, 1977, **28**, 1071.
8. Dowman, M. J., and Collins, F. C., *J. Sci. Fd. Agric.*, 1982, **33**, 689.
9. "The Feedingstuffs (Sampling and Analysis) Regulations (1982)", S.I. 1982 No. 1144, HMSO, London 1982.
10. Clancy, M. J., and Wilson, R. K., "Proc. Xth, Int. Grassld. Congr., Helsinki", 1966, p. 455.
11. EC Regulation 72/199/EEC, *OJ N. L123 p. 6*, 25th September, 1972.
12. "Official Methods of Analysis of the Association of Official Agricultural Chemists", 10th Edition, AOAC, Washington D.C., 1965.

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Chemical Characterisation of Mechanically Recovered Meats

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Forty samples of various types of mechanically recovered meats (MRM) were analysed for their chemical composition including moisture, fat, protein, ash, bone content, fatty acids, sterols, pH, hydroxyproline and trace element content. The MRM samples examined varied greatly in their chemical composition, and often differed significantly from equivalent hand-deboned meats. The significance of the data presented is considered.

The final figure for total UK meat production in 1984 is expected to be about 3·83 million tonnes carcase weight, comprising 60 per cent. of red meat (beef, veal, pork and lamb) and 40 per cent. of fowl (poultry, broiler and turkey)[1]. Mechanical recovery of meat following hand deboning operations gives an additional carcase yield of up to 4 per cent. for red meat and 15 per cent. for fowl, making obvious the attraction of such processes for the meat product manufacturer. In 1982 the value of potentially separable mechanically recovered meat from red-meat carcases alone was being put at around £9M[2]. Mechanically recovered meat is already being incorporated in traditional comminuted meat products such as sausages, pies and burgers, as well as soups, infant foods and special diets for the disabled. This usage is likely to increase until the economic recovery of MRM from UK produced bones has been maximised (at the moment the importation of bones for processing is prohibited).

Most of the analytical data on MRM composition have been published in the USA[3] where continuous auger-type machines, such as the Beehive, which process pre-ground bone material, are used rather than the press-type Protecon and Hydrau machines favoured in the UK which can handle batches of quite large intact bones[4]. Other major differences between the two recovery processes have been considered in some detail elsewhere[4,5].

Relatively little information is available concerning the composition of MRMs produced in the UK[5,6] or other EEC countries[7,8]. In this study a total of 40 samples of mechanically recovered meat were examined. Of these, 29 (numbered 1–29) were obtained through Trading Standards Officers or directly from meat product manufacturers. The remaining 11 (numbered 30–40) came from a major specialist supplier of MRM to the UK meat industry. The amount of background information available varied according to the source of the samples, although in most instances sufficient detail was available to identify the origin of the bones and the type of processing machinery employed. At the commencement of the study only samples 30–40 could be guaranteed as

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"authentic", that is, containing no foreign species nor "added water". However, the authors had no reason to suspect that any of the other samples collected were not produced to a similar standard.

Each sample of MRM was analysed for proximate composition, calcium, magnesium and iron contents. Samples numbered 30–40 whose histories were known more exactly (see Table 1) were subjected to additional analyses for pH, hydroxyproline, fatty acid isomers, sterols, bone content, copper, manganese, zinc, sodium, potassium and phosphorus. Microscopical examinations were carried out on each type of MRM.

Experimental

Ash, moisture, fat and L(-)hydroxyproline were determined in duplicate according to BS : 4401 Parts 1, 3, 4 and 11 respectively[9].

Total nitrogen was determined in duplicate by Kjeldahl distillation using Tecator Kjeltec system 1003 (manual titration)[10a,11] or model 1030 with automatic titration[12]. Nitrogen recoveries obtained with both systems were checked regularly against a secondary standard wheatflour and periodically with ammonium sulphate and glycine.

pH values were measured in duplicate on each of two sample portions according to BS : 4401 Part 9 using a glass electrode and a pH meter with automatic temperature correction facility at a temperature of 24–25°C[9].

Bone content was determined gravimetrically following suspension and comminution of the sample in carbon tetrachloride[13].

Calcium, copper, iron, magnesium, manganese and zinc were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) using an ARL 137 ICPQ system[14].

Sodium and potassium were analysed by atomic absorption spectrometry (AAS) using a Perkin Elmer AA spectrophotometer model 403[15].

Phosphorus was determined colorimetrically using a Technicon Auto-Analyser system[10b,16]. All inorganic constituent determinations were checked against a standard reference material (NBS liver 1577)[17].

Fatty acid isomers were analysed as their methyl esters by capillary column gas chromatography on a 50 m glass WCOT column coated with Reoplex 400 at a temperature of 175°C[10c,18].

Sterols were determined as their silyl ether derivatives by capillary column gas chromatography on a 25 m glass WCOT column coated with OV17 at a temperature of 250°C using betulin as internal standard[10d,19].

Microscopical examinations were carried out on 10 μm thick sections cut using a rotary retracting microtome in a cryostat cabinet at −20°C from small blocks of MRM previously frozen in liquid nitrogen. Sections taken from each sample were stained with toluidine blue to demonstrate general morphology[20], and with Carazzi's haematoxylin[21], sirius red F3BA[22], and alizarin red S[21] to demonstrate haemapoietic bone marrow, collagenous connective tissues and bone fragments respectively.

Results

PRODUCTION OF MRM SAMPLES 30–40

Table I shows the operating parameters for the Protecon MRS 30 and MRS 60 machines used to produce MRM samples 30–40. In general the conditions represent an empirically determined compromise intended to maximise yield while avoiding a significant rise in temperature and minimising inclusions of excess fat, fragments of bone, connective tissue and bone marrow. There are three main variables; the pressure applied, the time of pressing and the stroke length, the latter being a measure of the amount of material pressed at any one time. For example, higher pressures and longer pressing times than normal are used for the recovery of duck MRM from neck bones because of the toughness of the neck tissues whereas lower pressures are used for recoveries of veal and cooked chicken MRM because of the softness of the associated bones and connective tissues. Recovery of MRM from hard boned carcases such as pork and venison are carried out using longer stroke lengths which help regulate any temperature rise. The operating conditions shown in Table I are used to give a premium product suitable for resale as a raw ingredient and may not be typical of "end-of-line" recovery operations.

TABLE I
PRODUCTION CHARACTERISTICS OF MECHANICALLY RECOVERED MEAT SAMPLES 30–40

Sample No. and type	Age and type of bones	Pressing time *s*	Stroke length*	Pressure *atm*
30 Turkey	5 months, bodies	4·5	2·5	240
31 Chicken	10–12 weeks, frames	4·5	2·5	240
32 Chicken	10–12 weeks, backs	4·5	2·5	240
33 Chicken, cooked	10–12 weeks, bodies	4·5	2·5	210
34 Duck	10 weeks, necks	7·5	3·5	250
35 Pork	5 months, chines, neck and breast	4·5	4·5	210
36 Lamb	3–4 months, ribs and necks	4·5	2·5	230
37 Lamb, semi-lean	3–4 months, belly end of ribs	4·5	2·5	230
38 Beef	18–24 months, forequarter	4·5	2·5	230
39 Veal, heavy	3 months, forequarter	4·5	2·5	200
40 Venison, wild	forequarter	4·5	4·5	225

* Machine setting.

PROXIMATE ANALYSES AND HYDROXYPROLINE CONTENTS

For the 40 samples of MRM analysed the mean figure for "total analysis", i.e. moisture + ash + fat + protein (Nx6·25), was 99·5 per cent. (s = 1·8 per cent.), Tables II and III. As would be expected, fat and moisture contents were inversely related, with their sum consistently totalling between 80 and 90 per cent. of the total sample weight (mean = 84·3 per cent., s = 2·8 per cent.).

Fat contents of the MRM samples examined varied between 5 and 50 per cent. with beef MRM having the highest average fat content at 33·6 per cent. (s = 8·4 per cent., n = 11) and turkey MRM the lowest at 6·5 per cent. (s = 2·3 per cent., n = 13). Pork and chicken MRM were intermediate with average fat contents of 19·8 per cent. (s = 6·7 per cent., n = 5) and 17·5 per cent. (s = 8·5 per cent., n = 5) respectively. Chicken MRM samples showed the widest range of fat contents

TABLE II

ANALYTICAL COMPOSITION OF MECHANICALLY RECOVERED MEAT SAMPLES 1–29

Machine	Bones	Moisture	Fat	Ash	N	N × 6·25	Metals Ca	Metals Mg	Metals Fe	DFM*	Total† meat
		per cent.					*mg/kg*			*per cent.*	*per cent.*
Pork											
1 Protecon	ribs	63·1	19·1	1·1	2·61	16·3	784	144	12	75·7	94·8
2 Hydrau	no hock	61·1	22·3	1·0	2·50	15·6	440	75	10	72·5	94·8
3 Protecon	no marrow	56·0	29·9	0·9	2·22	13·9	697	155	46	64·3	94·2
4 Protecon	shoulder	67·2	13·4	1·1	2·48	15·5	794	152	38	71·9	85·3
Beef											
5 Hydrau	no marrow	54·0	37·6	0·9	1·92	12·0	756	59	26	54·1	91·7
6 Protecon	no marrow	51·5	32·2	1·1	2·15	13·4	1248	79	20	60·6	92·8
7 Protecon	no marrow	56·2	32·5	1·1	2·13	13·3	1462	149	44	60·0	92·5
8 Protecon	no marrow	59·6	23·4	1·3	2·25	14·0	1860	140	99	63·4	86·8
9 Beehive	no marrow	46·7	37·8	2·8	1·82	11·4	7966	201	83	51·3	89·1
10 Beehive	no marrow	39·6	48·8	2·6	1·52	9·5	7718	205	44	42·8	91·6
11 Beehive	no marrow	44·5	38·6	2·8	1·86	11·6	7942	202	71	52·4	91·0
12 Beehive	no marrow	46·4	40·5	2·6	1·86	11·6	7337	198	78	52·4	92·9
13 Protecon	?	51·5	33·0	1·0	2·13	13·3	1887	160	27	60·0	93·0
14 Protecon	?	60·7	21·1	1·1	2·54	15·9	1876	168	54	71·5	92·6
Chicken											
15 Protecon	backs	60·4	23·4	0·7	2·27	14·2	214	132	13	63·1	86·4
16 Protecon	necks	79·8	7·1	0·7	2·04	12·8	291	149	33	56·7	63·8
17 Protecon	necks	76·0	11·3	0·7	2·21	13·8	839	161	36	61·4	72·7
Turkey											
18 Protecon	necks	81·7	4·5	0·5	1·98	12·4	144	127	11	56·6	61·0
19 Protecon	necks	79·0	5·2	0·4	2·52	15·7	166	134	15	72·0	77·2
20 Protecon	all	79·2	6·7	0·6	2·03	12·7	188	152	18	55·6	62·3
21 Protecon	all	76·6	8·4	0·7	2·24	14·0	160	156	17	61·4	69·7
22 Protecon	all	78·7	6·0	0·7	2·26	14·1	278	168	20	61·8	67·8
23 Protecon	all	78·3	5·4	0·6	2·14	13·4	188	146	16	58·7	64·1
24 Protecon	all	77·6	5·4	0·7	2·51	15·7	158	172	17	68·8	74·1
25 Protecon	all	79·1	6·6	0·7	2·18	13·6	180	157	17	59·7	66·4
26 Protecon	all	77·7	4·1	0·7	2·29	14·3	172	165	20	62·7	66·8
27 Protecon	all	79·8	6·2	0·6	2·21	13·8	165	153	17	60·5	66·7
28 Protecon	all	72·6	5·6	0·7	2·34	14·6	178	162	14	64·0	69·6
29 Protecon	all	79·3	6·8	0·6	2·19	13·7	202	153	18	60·1	66·8

* "DFM" is an abbreviation for de-fatted meat. This is calculated from the equation DFM = (100 × Nitrogen)/Nf where Nf is the appropriate "nitrogen factor" for the sample; this is a pre-determined value for the nitrogen content of the sample-type expressed on a fat-free basis. In Tables II, III and VIII the nitrogen factors used to convert analytical nitrogen into raw defatted meat were as follows: Pork samples 1–4 and 35, 3·45; Beef samples 5–14 and 38, 3·55; Chicken samples 15–17 and 31–33, 3·60; Turkey (necks) samples 18–19, 3·50; Turkey (all bones) samples 20–30, 3·65; Duck sample 34, 3·31; Lamb samples 36 and 37, 3·59; Veal sample 39, 3·35 and Venison sample 40, 3·59. References: 10e,24,25.

† Total meat = de-fatted meat + fat.

TABLE III

PROXIMATE COMPOSITION OF MECHANICALLY RECOVERED MEAT SAMPLES 30–40

Sample	Moisture	Fat	Ash	N	N × 6·25	DFM*	Total† meat
	per cent.					*per cent.*	*per cent.*
30 Turkey	70·3	13·1	0·7	2·23	13·9	61·1	74·2
31 Chicken (frames)	65·1	17·8	0·8	2·56	16·0	71·1	88·9
32 Chicken (bodies)	57·1	27·9	0·7	2·24	14·0	62·2	90·1
33 Chicken (cooked)	47·9	27·7	0·6	3·67	22·9	101·9	129·6
34 Duck	76·0	5·5	0·9	2·66	16·6	80·4	85·9
35 Pork	69·3	14·3	1·3	2·38	14·9	69·0	83·2
36 Lamb	63·8	20·1	1·0	2·58	16·1	71·7	91·8
37 Lamb "semi-lean"	40·9	46·5	0·5	1·73	10·8	48·1	94·6
38 Beef	59·9	23·6	1·3	2·37	14·8	66·8	90·4
39 Veal	61·5	23·2	1·0	2·45	15·3	73·1	96·3
40 Venison	69·0	13·6	0·9	2·55	15·9	71·2	84·8

*† See Table II.

TABLE IV

MINERAL COMPOSITION OF MECHANICALLY RECOVERED MEAT SAMPLES 30–40

Sample	Mineral composition *mg/kg*								
	Ca	Cu	Fe	Mg	Mn	Zn	Na	K	P
30 Turkey	411	0·60	25·1	126	0·18	22·7	635	1541	1254
31 Chicken frames	383	0·31	22·0	173	0·24	12·6	864	2403	1665
32 Chicken bodies	243	0·38	16·4	146	0·24	11·8	791	2073	1451
33 Chicken cooked	340	0·25	25·9	127	0·15	47·7	914	1701	1142
34 Duck	275	0·35	21·4	164	0·17	34·4	1050	2562	1614
35 Pork	1739	1·10	56·5	173	0·11	21·6	1337	2469	2296
36 Lamb	721	0·66	42·1	167	0·11	34·1	1032	2506	1949
37 Lamb "semi-lean"	235	0·00	15·7	110	0·03	21·4	604	1781	1087
38 Beef	1061	0·37	118	143	0·07	26·6	1349	1837	2268
39 Veal	1112	0·68	34·2	146	0·05	24·7	1217	2046	1843
40 Venison	1290	0·82	60·0	163	0·20	41·1	803	1738	1852

TABLE V

pH, BONE, HYDROXYPROLINE, AND CHOLESTEROL IN MECHANICALLY RECOVERED MEAT SAMPLES 30–40

Sample	pH	Bone content	Hydroxy proline	Cholesterol content
		per cent.		*mg/100g*
30 Turkey	6·50	0·11	0·06	124
31 Chicken (frames)	6·40	0·17	0·13	164
32 Chicken (bodies)	6·35	0·04	0·24	165
33 Chicken (cooked)	6·40	0·26	0·84	300
34 Duck	6·60	0·02	0·18	142
35 Pork	6·70	0·60	0·08	209
36 Lamb	6·25	0·12	0·12	257
37 Lamb "semi-lean"	6·30	0·04	0·12	96
38 Beef	6·65	0·48	0·11	252
39 Veal	6·30	0·17	0·16	184
40 Venison	6·40	0·33	0·14	169

varying by a factor of four from 7·1 per cent. up to 27·9 per cent. reflecting the proportion of skin, and its associated adipose layer, attached to the carcases used for processing[23].

Fat-free moisture contents of red meat MRM (78 per cent.) and poultry MRM containing no added water (79 per cent.) were slightly higher (2–4 per cent.) than for equivalent samples of lean hand deboned meats (HDM)[24,25].

Ash figures obtained from the analysis of red meat MRM were generally higher than those found in comparable HDM[24]. This is best explained by the inclusion of small particles of bone produced by bone-sawing operations during hand deboning of the carcase (Protecon MRM) and/or by subsequent bone crushing during retrieval of the MRM (Beehive MRM). Ash contents varied from sample to sample and from species to species and, for red meat MRM at least, appeared to be dependent on the type of machinery used for the processing. Beef MRM produced on Beehive deboners had significantly higher ash contents than samples produced on either Protecon or Hydrau machines although without knowledge of the operating conditions employed it is difficult to assess whether this is a function of the machine itself or the mode of its operation. Poultry meat MRM generally contained similar or only slightly higher amounts of ash than HDM[25].

Hydroxyproline (Table V) and nitrogen contents of the MRM samples examined were both lower than would have been expected for hand deboned meats[25,26,27]. Accordingly, all the MRM samples tested (with one exception) had significantly low apparent fat-free meat contents when calculated using appropriate nitrogen factors (Nf)[10e,25]. There are three possible reasons for this. Firstly, because of their high tensile strength some connective tissues are witheld during extrusion of the MRM through the machine die. MRM samples are therefore deficient in connective tissues and their collagen contents, as measured by hydroxyproline determination, are significantly lower than comparable HDM[25]. Since connective tissues are higher in nitrogen content than muscle, the result is a lower overall figure for determined total nitrogen. The exception to this is shown in the results for cooked chicken MRM (sample 33) which is high in both nitrogen (even allowing for its reduced moisture content) and connective tissue (three to five times higher than for the comparable uncooked MRM samples). Here, because the connective tissue has been softened during cooking it has been extruded more easily along with the other MRM material, increasing the total nitrogen and hence the apparent defatted meat content of the sample.

Secondly, a minor contribution to the lower nitrogen figures may be obtained from low-nitrogen materials derived from the bone marrow although no direct evidence of this was sought.

Thirdly, the additional 2–4 per cent. moisture present in the MRM samples would also reduce the fat-free nitrogen (Nf) content, although by too small an amount to account fully for the extremely low "total meat" contents of some of the poultry samples. These results suggest the presence of extraneous water added either in the form of ice as a coolant during the MRM extraction process or more likely as water used for carcase-cooling. Study of Tables II and III shows

that most of the turkey MRM samples (18–29) also contained about 8–10 per cent. of extraneous water.

The presence of additional "natural" moisture, connective tissue abstraction and/or marrow inclusion had a significant effect upon the apparent meat contents of the MRM samples examined. Apparent total meat contents of the beef MRM samples examined were very consistent at an average of 91·3 per cent. with a low spread of results (s = 1·9 per cent., n = 11) even when samples produced on different types of machine were considered. To give a total apparent meat content of 100 for beef MRM the appropriate Nf factor would have to be 3·09 compared to the figure of 3·55 for HDM[10e]. Similar results were obtained for pork MRM which had an average apparent total meat content of 90·5 per cent. (s = 5·7 per cent., n = 5) with an Nf factor of 3·04 compared to 3·45 for hand deboned meat[10e], although here fewer samples were examined and those that were showed greater variation. If the effects of added water are ignored, appropriate Nf factors would be for chicken MRM approximately 3·1 and for turkey MRM around 2·6.

The similarity between the fat-free nitrogen contents of beef, pork and chicken MRM (samples 15, 31 and 32) at 3·09, 3·04 and 3·09 respectively implies that it is variations in the connective tissues, either qualitative, quantitative or both, that are responsible for the differences observed between the Nf figures of HDM from different species and that the nitrogen content of the sarcoplasmic proteins are fairly similar. Based upon the results in Table III for single samples of other species, appropriate Nf factors for MRM would be; venison 3·0, veal 3·2, lamb 3·2, and duck 2·8.

PH DETERMINATIONS

pH determinations made upon MRM samples 30–40 are reported in Table V to the nearest 0·05 pH unit. All of the results lie in a narrow band between pH 6·25 (lamb MRM) and pH 6·70 (pork MRM) and are comparable with results reported previously which range from pH 6·0–7·0[3]. These values are higher than those for corresponding HDM which typically lie between pH 5·4 and pH 6·0, but can rise to pH 6·5 in tissues with low initial glycogen levels[8].

The reason for this difference in pH between hand deboned and mechanically recovered meat is not entirely clear. It has been suggested that meat in close proximity to the bone, which will be present in higher concentrations in MRM, is of a naturally higher pH because of the lower amounts of lactic acid it produces during glycolysis[8]. Alternatively, the higher pH of MRM has been attributed to the presence of bone marrow which has a pH of 6·8–7·4 and is expressed during the recovery procedure[3,26]. The increased level of pigmentation suggests that red marrow was present in some of the samples of MRM and that the higher pH may partially be attributed to this. However, if bone marrow content were the only factor in determining the pH then the pork MRM sample (35) would contain over 50 per cent. by weight of bone marrow[28]; however, microscopical examination showed that this was not so. A third possibility is that the high pressures involved in MRM recovery cause deamination of proteinaceous components thereby releasing ammonia and causing the pH to rise.

BONE AND INORGANIC CONSTITUENTS

Red meat MRM contained more bone than poultry MRM as might be expected from the contrasting treatments undergone by the carcases prior to MRM recovery and the difference in the hardness of the bones. The product-moment correlation coefficient between ash and bone contents for MRM samples 30–40 was not as high as might be expected ($r = 0{\cdot}754$, $n = 11$) whereas agreement between ash and phosphorus ($r = 0{\cdot}969$) and ash and calcium ($r = 0{\cdot}839$) was considerably better.

This suggests that the method used for the determination of bone content was not satisfactory and that a more reliable figure for bone content might be obtained indirectly from the ash figure. Of the inorganic constituents determined (Tables II and IV) only Ca, Fe and Na were consistently higher and K was consistently lower than the levels found in comparable hand deboned meats. Calcium was 2 to 5 times higher than would be expected in a conventional cut, and was almost certainly derived from small bone particles. It has been suggested that bone content can be estimated in poultry MRM by the equation[23]:

$$\text{bone content per cent.} = 6{\cdot}25 \times (\text{Ca per cent.} - 0{\cdot}015)$$

A factor to convert P to bone content would also merit further consideration.

Iron contents were on average two to three times higher than in HDM reflecting the increased levels of blood or blood forming (haemapoietic) tissue in the samples. Alteration in the ratio of blood to cellular fluids was also the likely cause of the observed reductions in the ratios of potassium to sodium compared to equivalent HDM[24]. Magnesium, manganese, zinc, copper and phosphorus contents were similar to levels found in hand deboned meats[24,25].

FATTY ACID ISOMERS

Fatty acid isomer profiles of MRM samples 30–40 are shown in Tables VI and VII. Allowing for some natural variation these results are not significantly different from fatty acid profiles of comparable HDM[25]. This suggests that any marrow lipid present was either similar in composition to tissue lipid or present in insignificant amounts. The single exception is the cooked chicken MRM sample (33) which was significantly lower in polyenoic fatty acids than both hand deboned chicken and the two raw chicken MRM samples (31, 32). This may be attributed to the cooking process undergone by the birds prior to mechanical recovery, which could have three effects; firstly, to introduce non-chicken fat (possibly beef fat used for roasting); secondly, to extract more of the bone marrow lipid; and thirdly, to damage the more unsaturated acids thereby altering the profile.

STEROLS

In the samples examined cholesterol was the only sterol detected; no trace was found of any methyl sterols, dimethyl sterols nor uvaols. Cholesterol contents of MRM samples 30–40 are given in Table V. The reported results are between 30 and 300 per cent. higher than published figures for HDM, which typically range from 50 to 110 mg/100 g for raw samples and up to double that for cooked

TABLE VI

FATTY ACID ISOMER CONTENT OF AVIAN MECHANICALLY RECOVERED MEAT SAMPLES

Fatty acid*	Turkey *per cent.*	Chicken (frames) *per cent.*	Chicken (bodies) *per cent.*	Chicken (cooked) *per cent.*	Duck *per cent.*
14 : 0br iso					
14 : 0	1·4	1·4	0·8	1·7	0·9
14 : 1ω7					
14 : 1ω5	0·2	0·2	0·2	1·0	0·1
15 : 0br iso				0·2	
15 : 0br ante				0·2	
15 : 0	0·4	0·2	0·1	0·4	0·2
15 : 1ω8					0·1
16 : 0br iso	1·0		0·3		
16 : 0	27·2	22·8	23·7	21·0	24·0
16 : 1ω9	0·4	0·6	0·4	0·3	0·4
16 : 1ω7	3·5	4·5	5·1	5·6	3·0
16 : 1ω5				0·3	
16 : 2ω6					
17 : 0br iso				0·4	
17 : 0br ante			0·1	0·8	
17 : 0	0·3	0·3	0·2	0·8	0·2
17 : 1ω8	0·2	0·2	0·2	1·2	0·3
18 : 0br iso	0·5			0·2	
18 : 0	9·2	7·1	6·6	9·3	8·4
18 : 1ω9	33·7	36·5	36·9	45·8	40·4
18 : 1ω7	1·9	2·6	2·4	3·7	2·0
18 : 1ω5		0·2	0·1	0·5	
18 : 1ω3			0·1	0·8	
18 : 2 ttω?				1·4	
18 : 2ω6	17·0	18·8	18·4	1·4	15·5
18 : 2conj.ω7		0·2	0·1	1·3	0·2
18 : 3ω6			0·1	0·1	
18 : 3ω3	1·2	2·0	1·6	0·6	1·4
18 : 4ω6					
18 : 4ω3			0·1		
20 : 0	0·2	0·2	0·1	0·2	0·1
20 : 1ω11	0·3	0·3	0·1	0·2	0·2
20 : 1ω9	1·1	0·8	0·6	0·3	0·5
20 : 2ω6		0·2	0·2		0·1
20 : 3ω6		0·3	0·1	0·1	
20 : 4ω6	0·3	0·6	0·4	0·2	1·9
20 : 5ω3			0·4		
22 : 5ω6			0·2		
22 : 6ω3			0·4		

* Fatty acids are named according to standard procedure. The number of carbon atoms in the chain is followed by a colon and then by the number of double bonds in the molecule. The omega (ω) indicates that the carbon atoms are numbered from the methyl terminal group and the numbers following the ω sign indicate the position of the first carbon atom in each double-bonded pair. All double bonds are *cis* configured unless indicated by "t" (for *trans*) and the abbreviation "conj" shows that the double bonds are conjugated rather than the more usual methylene-interrupted. Saturated, branched-chain fatty acids are indicated by the abbreviation "br" followed by "iso" or "ante" (for anteiso) which indicate the position of the branching within the molecule.

samples[24,29,30]. These differences may be partially explained in terms of the mechanical recovery process during which relatively cholesterol-free connective tissues are abstracted, tending to enrich the remaining MRM tissue[31]. However, enrichment alone cannot account for the higher cholesterol levels of samples 30–40, which are more likely to be caused by the inclusion of some bone marrow constituents and differences in the analytical methodology used.

TABLE VII

FATTY ACID ISOMER CONTENT OF NON-AVIAN MECHANICALLY RECOVERED MEAT SAMPLES

Fatty acid*	Pork *per cent.*	Lamb *per cent.*	Lamb (semi-lean) *per cent.*	Beef *per cent.*	Veal *per cent.*	Venison *per cent.*
14 : 0br iso		0·1		0·1		0·4
14 : 0	1·2	5·3	4·7	3·2	4·0	6·1
14 : 1ω7			0·1			
14 : 1ω5		0·2	0·1	0·8	0·6	1·3
15 : 0br iso		0·2	0·3	0·4	0·2	0·5
15 : 0br ante		0·3	0·3	0·5	0·2	0·8
15 : 0	0·3	0·7	0·7	0·8	0·3	1·2
15 : 1ω8						0·2
16 : 0br iso		0·3		0·3	0·2	0·4
16 : 0	22·4	21·8	21·4	25·3	21·0	27·3
16 : 1ω9	0·5	0·9	0·4	0·5	0·5	0·5
16 : 1ω7	1·8	1·6	1·5	3·1	3·0	4·8
16 : 1ω5			0·1	0·2	0·2	0·3
16 : 2ω6				0·6		0·2
17 : 0br iso		0·6	0·5	0·6	0·5	0·5
17 : 0br ante		0·7	0·7	0·8	0·8	0·6
17 : 0	0·5	1·2	1·5	1·1	1·1	1·1
17 : 1ω8	0·4	0·7	0·5	0·8	0·9	0·4
18 : 0br iso		0·2		0·2	0·2	0·1
18 : 0	16·2	17·8	21·6	16·8	16·9	25·5
18 : 1ω9	36·9	30·8	27·8	33·2	40·6	16·7
18 : 1ω7	3·4	6·7	7·3	3·9	3·2	4·6
18 : 1ω5		0·9	1·5	0·9	0·5	0·6
18 : 1ω3	0·3	0·4	0·6		0·3	
18 : 2 tt		2·0	2·5	1·7	1·0	0·7
18 : 2ω6	10·2	1·7	1·5	2·0	1·9	1·7
18 : 2 conj.ω7	0·2	2·3	2·0	0·7	0·7	0·8
18 : 3ω6			0·2	0·2		0·4
18 : 3ω3	0·7	1·3	1·5	1·0	0·5	0·8
18 : 4ω6	0·3					
18 : 4ω3		0·1	0·1			
20 : 0	0·4	0·2	0·1	0·1	0·1	0·5
20 : 1ω11					0·1	
20 : 1ω9	1·8	0·6	0·5	0·2	0·3	0·5
20 : 2ω6	0·7					
20 : 3ω6	0·2					
20 : 4ω6	1·2	0·4			0·2	0·5
20 : 5ω3	0·4					
22 : 5ω6						
22 : 6ω3						

* See footnote to Table VI.

MICROSCOPY

Studies on the histochemical characterisation of MRM are continuing and incomplete[21]. However, there are several microscopical features of MRM that readily set it apart from equivalent, comminuted, HDM.

Staining the samples with toluidine blue and sirius red F3BA showed the muscle tissue to be extensively damaged while retaining sufficient of its microstructure to be identified as being of animal origin. The major difference from comminuted HDM was that the sarcolemma was often indistinct or absent and many of the muscle cell nuclei were no longer visible. In addition, few of the muscle fibres remained attached together, and many were irregular in shape and had lost all semblance of the familiar cross-striations. A certain amount of amorphous material was also visible. Take-up of the blue basic dyestuff by MRM appeared to be similar to that of HDM.

Sections of red meat MRM stained with alizarin red S contained variable numbers of very small bone particles, many less than 1 mm in diameter, which would appear to be diagnostic[7,21]. Such particles were not seen to the same extent in poultry MRM, leading to the conclusion that they were probably derived from the "bone-dust" adhering to bone and meat surfaces following sawing up of the red meat carcase.

To date, staining of samples with Carazzi's haematoxylin has failed to demonstrate identifiable signs of haemapoietic tissue in MRM although work on red marrow samples has shown that this is the technique most likely to do so[21].

Discussion

Mechanically recovered meat (MRM) is the term used in the UK to describe what the United States Department of Agriculture (USDA) defines as "the finely comminuted product resulting from the mechanical separation and removal of most of the bone from attached skeletal muscle of livestock carcasses and parts of carcasses"[32]. The fact that the UK term has received general acceptance is in contrast to the position in the USA where the USDA has been pressured by both manufacturing and consumer interest groups into making several changes of name. In 1976 the term "Mechanically Deboned Meat, MDM"[33] was adopted only to be changed in 1977 to "Tissue From Ground Bone, TFGB"[34], following a court ruling that MDM was not meat as traditionally defined because of its bone particle content which must be regarded as an addition made during processing. In 1978 TFGB was rejected as misleading, because it implied that the product was made wholly from parts of bone, and was replaced by "Mechanically Processed (Species) Product, MS(S)P"[35].

However, although the UK is fortunate in having a single and undisputed name for the product this does not make MRM any less variable in its composition. Even from the data presented here, the term MRM obviously does not describe a single chemically-definable product. Its composition is subject to variation not only from the differences occurring naturally between and within animal species but also from differences in machine type and operating practice. Some types of MRM, for example chicken and turkey, are more consistent in composition than others such as beef, where different samples might derive

from different parts of the carcase or even different breeds of cattle. From Tables I and III it can be seen that lamb MRM pressed from ribs and neck bones (sample 36) is very different in composition to lamb MRM produced under the same conditions from belly end of ribs (37). Add to this the fact that the fat content of poultry MRM can be raised by increasing the amount of skin processed with the carcase and it becomes obvious that, within certain limitations, MRM can be tailored to suit any particular market or end product.

Primary factors affecting MRM composition appear to be the animal species, the part of the animal used and its condition (for example whether or not it is trimmed of fat, cooked or frozen) as well as the type of machine used and the conditions under which it is operated (Table VIII). Because of its variable composition MRM presents several problems for the analyst wishing to detect or quantify its inclusion in a meat product.

(i) A single, simple chemical index of the presence of MRM will be difficult to find. The likelihood of finding an accurate and reliable quantitative index would appear to be even more remote.

(ii) MRM does have certain characteristics that may aid in its qualitative detection but often several of these would need to be considered together. MRM is more deeply coloured, can be expected to have a higher pH, can contain more bone, can have higher levels of cholesterol, Ca, and Fe, and can have a lower K/Na ratio and lower contents of connective tissue material and nitrogen than corresponding hand deboned meats.

(iii) Of the chemical parameters available for MRM detection total haeme pigment (as indicated by iron content) or the ratio of haemoglobin/myoglobin would probably be the best.

(iv) The microstructure of MRM is distinctive and quite different from comminuted HDM. Small particles of "bone dust" can be seen in red meat but not in all samples of poultry MRM while signs of haemapoietic marrow should be detectable in some types of MRM. However, when incorporated in comminuted meat products together with traditional ingredients, MRM would be much more difficult to identify. Also, the absence of bone particles would not prove the absence of poultry MRM.

(v) Current regulations controlling the meat content of foods make no reference to MRM[36]. The meat product manufacturer might therefore assume, unless advised otherwise by his supplier, that MRM can be used to replace hand deboned meat in product formulations on an equivalent weight basis. However, because of the lower fat-free nitrogen contents of MRM, this could result in a product containing less than the legal requirement for meat content as calculated following chemical analysis[10f].

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TABLE VIII

MAJOR SPECIES OF MECHANICALLY RECOVERED MEAT: PROXIMATE ANALYSES ARRANGED ACCORDING TO MACHINE USED FOR MEAT REMOVAL

Species/ machine		Moisture *per cent.*	Ash *per cent.*	Nitrogen *per cent.*	Nx6·25 *per cent.*	Fat *per cent.*	Total analysis *per cent.*	Total meat* *per cent.*
Beef								
Protecon	i	51·5	1·12	2·15	13·4	32·2	98·2	92·8
	ii	56·2	1·11	2·13	13·3	32·5	103·1	92·5
	iii	59·6	1·25	2·25	14·0	23·4	98·3	86·8
	iv	51·5	0·95	2·13	13·3	33·0	98·8	93·0
	v	60·7	1·14	2·54	15·9	21·1	98·8	92·6
	vi	59·9	1·30	2·37	14·8	23·6	99·6	90·4
Beehive	vii	46·7	2·78	1·82	11·4	37·8	98·7	89·1
	viii	39·6	2·55	1·52	9·5	48·8	100·4	91·6
	ix	44·5	2·76	1·86	11·6	38·6	97·5	91·0
	x	46·4	2·55	1·86	11·6	40·5	101·1	92·9
Hydrau	xi	54·0	0·89	1·92	12·0	37·6	104·5	91·7
Mean ($n = 11$)		51·9	1·67	2·05	12·8	33·6	99·9	91·3
Pork								
Protecon	i	63·1	1·14	2·61	16·3	19·1	99·6	94·8
	ii	56·0	0·90	2·22	13·9	29·9	99·8	94·2
	iii	67·2	1·08	2·48	15·5	13·4	97·2	85·3
	iv	69·3	1·32	2·38	14·9	14·3	99·8	83·3
Hydrau	v	61·1	0·96	2·50	15·6	22·3	100·0	94·8
Mean ($n = 5$)		63·3	1·08	2·44	15·2	19·8	99·3	90·5
Chicken								
Protecon	i	60·4	0·74	2·27	14·2	23·4	98·7	86·5
	ii	79·8	0·66	2·04	12·8	7·1	100·4	63·8
	iii	76·0	0·74	2·21	13·8	11·3	101·8	72·7
	iv	65·1	0·84	2·56	16·0	17·8	99·7	88·9
	v	57·1	0·74	2·24	14·0	27·9	99·7	90·1
Mean ($n = 5$)		67·7	0·74	2·26	14·2	17·5	100·1	80·3
Turkey								
Protecon	i	81·7	0·53	1·98	12·4	4·5	99·1	61·0
	ii	79·0	0·43	2·52	15·7	5·2	100·3	77·2
	iii	79·2	0·64	2·03	12·7	6·7	99·2	62·3
	iv	76·6	0·70	2·24	14·0	8·4	99·7	69·7
	v	78·7	0·70	2·26	14·1	6·0	99·5	67·8
	vi	78·3	0·58	2·14	13·4	5·4	97·7	64·1
	vii	77·6	0·70	2·51	15·7	5·4	99·4	74·1
	viii	79·1	0·74	2·18	13·6	6·6	100·0	66·4
	ix	77·7	0·71	2·29	14·3	4·1	96·8	66·8
	x	79·8	0·63	2·21	13·8	6·2	100·4	66·7
	xi	72·6	0·66	2·34	14·6	5·6	93·5	69·6
	xii	79·3	0·61	2·19	13·7	6·8	100·4	66·8
	xiii	70·3	0·67	2·23	13·9	13·1	98·0	74·2
Mean ($n = 13$)		77·7	0·64	2·24	14·0	6·5	98·8	67·6

* See footnote to Table II.

References

1. Anon., *Meat Industry,* 1984, **57**, (1) 13.
2. Anon., *Processing,* 1982, March, p. 49.
3. Field, R. A., *Advances in Food Research,* 1981, **27**, 23.
4. Newman, P. B., *Food,* 1983 July, p. 26.
5. Newman, P. B., *Meat Science,* 1981, **5**, 171.
6. Evans, G. G., Oliphant, G., and Ranken, M. D., "The Composition and Analysis of Mechanically Recovered Meat". Research Report No. 296, January 1979, Leatherhead Food R.A.
7. Bijker, P. G. H., Koolmees, P. A., and Logtestijn, J. G., *Meat Science,* 1983, **9**, 257.
8. Zwingmann, W., *Fleischwirtsch,* 1980, **60**, 99.
9. BS4401 "British Standard Methods of test for Meat and Meat Products: Part 1. Determination of ash (ISO 936–78). Part 3. Determination of moisture content (ISO 1442–1973). Part 4. Determination of total fat content (ISO 1443–1973). Part 9. Determination of pH (ISO 2917–1974). Part 11. Determination of L(−)-hydroxyproline content (ISO 3496–1978)". British Standards Institution, London.
10. Egan, H., Kirk, R. S., and Sawyer, R. (1981) "Pearson's Chemical Analysis of Foods. 8th edition, Churchill Livingstone, Edinburgh, 1981. (a) pages 16–19; (b) page 30; (c) pages 525–527; (d) pages 530–531; (e) page 398; (f) page 397.
11. Mindell, B., *Lab. Equip. Dig.,* 1975, **13**, 57.
12. Hjarlmarsson, S., and Mindel, B., *In Focus,* 1981, No. 8, p. 9.
13. Kamm, L., and Coffin, D. E., *Can. Inst. Food Tech.,* 1968, **1**, 83.
14. LGC NATLAS Documents FAN42/85/300 and FAN42/85/302, Laboratory of the Government Chemist, London, 1985.
15. LGC NATLAS Documents FAN42/85/300 and FAN42/85/301, Laboratory of the Government Chemist, London, 1985.
16. Horwitz, W. (Ed.), Method 3.062, in "Official Methods of Analysis". 11th edition, AOAC, Washington, D.C., 1970, p. 44.
17. Gladney, E. S., *Anal. Chim. Acta,* 1980, **118**, 385.
18. Meech, M. V., *Anal. Proc.,* 1982, **19**, (11) 516.
19. EEC Regulation 1058/77 OJ No. L 128 (24.5.77), p. 6.
20. Flint, F. O., and Meech, M. V., *Analyst,* 1978, **103**, 252.
21. Flint, F. O., (Personal communication), and Aston, S. P., MSc Thesis, Procter Department of Food Science, The University of Leeds, 1983.
22. Flint, F. O., and Firth, B. M., *Analyst,* 1983, **108**, 757.
23. Satterlee, L. D., Froning, G. W., and Janky, D. M., *J. Food Sci.,* 1971, **36**, 979.
24. Paul, A. A., and Southgate, D., "The Composition of Foods". 4th edition, 1978, HMSO. London.
25. LGC unpublished data.
26. Field, R. A., Kruggel, W. G., and Riley, M. L., *J. Animal Sci.,* 1976, **43**, (4) 755–762.
27. Dransfield, E., *et al., Meat Science,* 1983, **8**, 79–92.
28. Field, R. A., and Arasu, P., *J. Food Sci.,* 1981, **46**, 1622.
29. Watt, B. K., and Merrill, A. L., "Composition of Foods. Agriculture Handbook No. 8", USDA, 1975, Washington, D.C.
30. Feeley, R. M., Criner, P. E., and Watt, B. K., *J. Amer. Diet. Assoc.,* 1972, **61**, 134.
31. Kunsman, J. E., Collins, M. A., Field, R. A., and Miller, G. J., *J. Food Sci.,* 1981, **46**, 1785.
32. FSIS, USDA. *Federal Register,* 1982, **47**, 28214.
33. FSIS, USDA. *Federal Register,* 1976, **41**, 17560.
34. FSIS, USDA. *Federal Register,* 1977, **42**, 54437.
35. FSIS, USDA. *Federal Register,* 1978, **43**, 26416.
36. "The Meat Products and Spreadable Fish Products Regulations 1984", S.I. 1984, No. 1566. HMSO, London, 1984.

J. Assoc. Publ. Analysts, 1986, **24**, 27–39

Association of Public Analysts Survey of Pesticide Residues in Food, 1984

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This report summarises the activities of Public Analysts during 1984 in the investigation of foods for residues of pesticide treatments. Detectable residues of pesticides were found in a wide range of foods, although only a small percentage exceeded maximum residue limits.

The examination of foods for residues of pesticides, fungicides and other treatments continued in a number of Public Analyst Laboratories during 1984. The scope of the work was extended to cover a wider range of foods. Greater emphasis was placed on cereal products and meat products and due to the limitation of resources this resulted in a fall in the number of samples of fruit and vegetables examined.

Methods of analysis were standardised between the participating laboratories within the limitations of available equipment and expertise. The methods of analysis were those of Sissons, Telling and Usher[1] or The Canadian Pesticide Manual[2].

Residues above reporting limits were confirmed by gas chromatographic techniques using two columns of different polarity and in some cases by chemical treatment prior to chromatography.

Reporting Limits

In order to improve uniformity in the reporting of information, the laboratories were requested to work to reporting limits set at specific levels for different classes of pesticides. The capabilities of modern instrumentation can enable the detection of extremely low levels of many of these chemicals, but the lower the level of presence then the more expensive and time consuming are the procedures required to ensure positive identification of the residue. There is considerable merit in using specified levels of presence as "reporting limits" which are readily achieved by all laboratories, and provided the set level is below the Maximum Residue Limit (MRL) by a factor of at least 10, then there should be no danger of missing residues which could be of significance. The "maximum residue limit" is that concentration of a pesticide residue in or on a food commodity resulting from the use of the pesticide, according to good agricultural practice, directly or indirectly, for the production and/or protection of the commodity concerned.

The reporting limits (mg/kg) set for the various types of determination are given in Table I.

0004–5780/86/010027 + 13 $02.00/0

TABLE I

REPORTING LIMITS FOR DETERMINATIONS

	Flour and cereals	Fruit and vegetables	Meat (on fat basis)	Eggs
Organochlorine	0·01	0·01	0·01	0·05
Organophosphorus	0·02	0·02	0·10	0·05
Biphenyl		0·10		
2-Hydroxybiphenyl (0 Phenyl phenol)		0·10		
Ethylene Dibromide		0·10		
Polychlorinated Biphenyls			1·0	
Thiabendazole		0·10		

Sampling

Samples were purchased from normal retail outlets by Trading Standards Officers or Environmental Health Officers. Where it was possible to confirm the country of origin this was noted.

Findings

The results of the analyses are shown in Tables II to V: Table II, flour and cereal products; Table III, fruit; Table IV, meat and meat products; Table V, vegetables. The following facts are evident from the detail given in the tables which are appended to this report.

FLOUR AND CEREAL PRODUCTS

One hundred and seven products were examined and 32 (29·9 per cent.) were found to contain residues at or above the adopted reporting limits. Two of the products (1·8 per cent.) contained residues above the Codex Alimentarius Residue Limits. These were both samples of flour containing DDT at levels slightly above the MRL of 0·1 mg/kg.

An unexpected feature of the results, which probably needs further investigation, is that 17 out of 18 (94 per cent.) bread samples contained detectable residues, whereas only 8 out of 24 (33·3 per cent.) of the flour samples were found to contain pesticide residue levels above the reporting limit.

FRUIT

Fewer fruit samples were tested in 1984 compared with 1983[3] due to the increased attention given to other products.

Seventy four fruits were examined, and 10 (13·5 per cent.) contained levels of pesticide residue at or above the adopted reporting limit. This compares with 10·2 per cent. of 305 samples in 1983. Only one fruit, a sample of limes from an unknown source, contained an amount which slightly exceeded the Maximum Residue Limit. The limes contained 0·07 mg/kg of aldrin, whereas the Codex MRL is 0·05 mg/kg.

TABLE II

PESTICIDE RESIDUES IN FLOUR AND CEREALS: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	*Results of analysis of samples* Number with no detectable residues	Residues above reporting limits	Residues above MRL* (Codex)	Determinations *No.* *Type*	Details of residues found at/or above reporting limits *mg/kg*
Bran	3	3			3 Pyrethroids	
Bread (granary)	6		6		6 Organophosphorus	Fenitrothion 0·05: 0·05: 0·06: 0·07: 0·11: 0·15.
Bread (wholemeal)	12	1	11		12 Organophosphorus	Pyrimiphos-Methyl: Malathion: Fenitrothion 0·03 0·19 0·08 0·02 0·12 0·05 0·03 0·01 0·03 0·02 0·18 0·01 0·05 0·05 0·1 0·29
Flour	9	7	2		7 Organochlorines	γ-HCH DDT 0·012 0·015 0·014 0·062
					2 Organophosphorus	
Flour (bread)	1	1			1 Organophosphorus	
Flour (brown)	1	1			1 Organophosphorus	
Flour (pastry)	1		1		1 Organochlorines	DDT 0·08
Flour (soft)	1		1		1 Organochlorines	γ-HCH 0·01
Flour (stone ground)	1	1			1 Organophosphorus	
Flour (untreated)	1			1	1 Organochlorines	DDT 0·12
Flour (wholemeal)	8	4	3	1	2 Organochlorines	γ-HCH DDT Pyrimiphos-Methyl 0·019 0·043 0·007 0·114
					6 Organophosphorus	0·029 0·023
Flour (85 per cent. wheatmeal)	1		1		1 Organophosphorus	Pyrimiphos-Methyl 0·01 Chlorpyriphos-Methyl 0·02

Continued overleaf

TABLE II continued

PESTICIDE RESIDUES IN FLOUR AND CEREALS: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	*Results of analysis of samples* Number with no detectable residues	Residues above reporting limits	Residues above MRL* (Codex)	Determinations *No.*	*Type*	Details of residues found at/or above reporting limits *mg/kg*
Puffed wheat	6	6			6	Organophosphorus	
Shredded wheat	18	15	3		18	Organophosphorus	Malathion 0·02
							Pyrimiphos-Methyl 0·05
							0·023
Shreddies	2	2			2	Organophosphorus	
Shreddies (malted)	7	6	1		7	Organophosphorus	Pyrimiphos-Methyl 0·04
Sugar puffs	2	2			2	Organophosphorus	
Weetabix	16	16			16	Organophosphorus	
Wheat bran	1	1			1	Pyrethroids	
Wheatflakes	9	8	1		9	Organophosphorus	Pyrimiphos-Methyl 0·02
Wholewheat cereal	1	1			1	Organophosphorus	
Total	107	75	30	2			

* The maximum residue limit (MRL) is the maximum concentration consistent with the use of pesticide according to good agricultural practice.

TABLE III

PESTICIDE RESIDUES IN FRUIT: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	Country of origin (where known)	*Results of analysis of samples* Number with no detectable residues	Residue levels above reporting limits	Residues above MRL* (EEC & Codex)	Determinations *No.*	*Type*	Details of residues found at/or above reporting limits *mg/kg*
Apples	4		4			4	Organochlorines	
Apricots	1		1			1	Organochlorines	
						1	Organophosphorus	
Bananas	2			2		2	Thiabendazole	0·83: 0·75
Cherries	1		1			1	Organochlorines	
Clementines	1		1			1	2-Hydroxybiphenyl	
						1	Biphenyl	
Gooseberries	19	U.K.	18	1		19	DDT & isomers	*pp'*DDE 0·005; *pp'*DDT 0·005; Total DDT 0·01
Grapefruit	1	S. Africa	1			1	Organochlorines	
						1	Organophosphorus	
						1	Ethylene dibromide	
Lemons	1	Spain	1			2	Organochlorines	
	2		1	1		2	Organophosphorus	
						2	Ethylene dibromide	
						1	2-Hydroxybiphenyl	4·1
						1	Biphenyl	
Limes	1				1	1	Organochlorines	Aldrin 0·07
						1	Organophosphorus	
						1	Ethylene dibromide	
Mango	3	Mexico	3			5	Organochlorines	
	1	Kenya	1			5	Organophosphorus	
	1		1			5	Ethylene dibromide	
Melons	2		2			2	Organochlorines	
						2	Organophosphorus	
Nectarines	1		1			1	Organochlorines	
						1	Organophosphorus	

Continued overleaf

TABLE III Continued

PESTICIDE RESIDUES IN FRUIT: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	Country of origin (where known)	*Results of analysis of samples* Number with no detectable residues	Residue levels above reporting limits	Residues above MRL* (EEC & Codex)	Determinations *No.*	*Type*	Details of residues found at/or above reporting limits *mg/kg*
Oranges	1	S. Africa	1			1	Organochlorines	
	3	Spain		3		1	Organophosphorus	
	1			1		1	Ethylene dibromide	
						4	2-Hydroxybiphenyl	0·20: 0·65: 0·20: 7·60:
						4	Biphenyl	0·20: 0·20: 0·20: 0·10:
Papayas	1	Brazil	1			3	Organochlorines	
	2		2			3	Organophosphorus	
						3	Ethylene dibromide	
Plums	1		1			1	Organochlorines	
						1	Organophosphorus	
Satsumas	1	Brazil	1			1	Organochlorines	
						1	Organophosphorus	
						1	Ethylene dibromide	
								Total
Strawberries	21	U.K.	20	1		21	DDT & isomers	*pp'*DDE *pp'*DDT *pp'*DDT DDT
	2		2			2	Organochlorines	0·002 0·004 0·03 0·04
Totals	74		64	9	1			

* The maximum residue limit (MRL) is the maximum concentration consistent with the use of pesticide according to good agricultural practice.

TABLE IV

PESTICIDE RESIDUES IN MEAT AND MEAT PRODUCTS: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	Country of origin (where known)	*Results of analysis of samples* Number with no detectable residues	Residues above reporting limits	Residues above MRL*	Determinations *No.* *Type*	Details of residues found at/or above reporting limit Results expressed as mg/kg on fat basis (except Eggs on whole basis)
Beef	8	U.K.	8			10 Organochlorines	
(steak etc.)	1	Australia	1			6 Organophosphorus	
	1	New Zealand	1				
Beef	3	England	2	1		3 Organochlorines	γ-HCH 0·06
(brisket)						1 Organphosphorus	
Beef	1	U.K.		1		1 Organochlorines	*pp'*DDE 0·08
(rump)						1 Organophosphorus	
Beef	11	England	9	2		11 Organochlorines	*pp'*DDT: 0·05 γ-HCH 0·018
(shin)						9 Organophosphorus	
Beef	2	U.K.	1	1		2 Organochlorines	*pp'*DDE 0·02
(shoulder)						2 Organophosphorus	
Beef	6	England	5	1		6 Organochlorines	γ-HCH 0·049
(stewing)						4 Organophosphorus	
Chicken	1	Denmark	1			40 Organochlorines	*pp'*DDE: *pp*DDT: γ-HCH Dieldrin PCB'S
	33	U.K.	29	4			0·01 0·08 0·01
	2	Holland	2				7
	4		1	3			0·007 0·08 0·056 0·08 0·02 0·03
Eggs	31	U.K.	31			37 Organophosphorus 31 Organochlorines 19 Organophosphorus	

Continued overleaf

Table IV Continued

PESTICIDE RESIDUES IN MEAT AND MEAT PRODUCTS: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	Country of origin (where known)	*Results of analysis of samples* Number with no detectable residues	Residues above reporting limits	Residues above MRL*	Determinations *No.* *Type*	Details of residues found at/or above reporting limit Results expressed as mg/kg on fat basis (except Eggs on whole basis)
Lamb	3	New Zealand	3			19 Organochlorines	
							γ-HCH
	16	England	6	10			0·25
							1·37
							0·02
							1·0
							0·3
							0·4
							0·4
							0·7
							1·5
							0·6
						3 Organophosphorus	
Lamb (chops)	7	New Zealand	4	3		38 Organochlorines	DDE 0·33: 0·02: g-HCH 0·02
							γ-HCH: HCB: *pp'*DDT: *pp'*DDE: Dieldrin
	31	U.K.	13	17	1		3·57
							0·463 0·035
							0·039
							0·022
							0·342
							1·22 0·02
							0·066
							0·064
							0·04 0·04
							0·19
							0·01 0·01
							0·02
							0·13
							0·025
							0·07
							0·07
							0·02 0·03
							0·03

Table IV Continued

PESTICIDE RESIDUES IN MEAT AND MEAT PRODUCTS: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Product	No. of samples	Country of origin (where known)	*Results of analysis of samples* Number with no detectable residues	Residues above reporting limits	Residues above MRL*	Determinations *No.* *Type*	Details of residues found at/or above reporting limit Results expressed as mg/kg on fat basis (except Eggs on whole basis)
						24 Organophosphorus	One unidentified peak
Pork	2	U.K.	2			2 Organochlorines	
						1 Organophosphorus	
Pork (chops)	7	Denmark	7			35 Organochlorines	HCB *pp'*DDE *pp'*DDT γ-HCH α-HCH Hepta-chlor Epoxide
	26	U.K.	18	8			0·017 0·004 0·022
							0·047
							0·09
							0·01
							0·047
							0·02 0·028
							0·02
							0·02
	2		1	1			0·01
						23 Organophosphorus	
Pork (sliced)	1	China		1		1 Organochlorines	β-HCH 0·18: DDT 0·118
Pork (luncheon)	1	China		1		1 Organochlorines	α-HCH 0·19: β-HCH 0·35: DDT 0·56
Turkey	9	U.K.	7	2	1	10 Organochlorines	γ-HCH *pp'*DDT *pp'*DDE Dieldrin
							0·25 0·13 0·01
							0·01 0·01
	1			1			0·05 0·14 0·07 0·03
						7 Organophosphorus	
Veal	11	U.K.	7	4		12 Organochlorines	α-HCH γ-HCH HCB
	1		1				0·13
							0·02
							0·042 0·015
							0·062
						11 Organophosphorus	
Totals	222		160	61	1		

*The maximum residue limit (MRL) is the maximum concentration consistent with the use of pesticide according to good agricultural practice.

TABLE V

PESTICIDE RESIDUES IN VEGETABLES: DETAILS OF PRODUCTS TESTED AND POSITIVE RESULTS

Products	No. of samples	Country of origin (where known)	*Results of analysis samples* Number with no detectable residues	Residue levels above reporting limits	Residues above MRL* (EEC)	Determination *No.* *Type*	Details of residues found at/or above reporting limits *mg/kg*				
							*pp'*DDE	*op'*DDT	*pp'*DDT	TDE	Total
Bean curd	1	China	1			1 Organochlorines					
Bean threads	1	China	1			1 Organochlorines					
Brussels sprouts	25	U.K.	15	8	2	25 DDT & isomers		0·02	0·09	0·01	0·12
									0·02		0·02
									0·02		0·02
									0·01		0·01
								0·034			0·034
								0·01			0·01
											0·05
											0·05
									0·03		0·03
Cabbage	49	U.K.	40	8	1	49 DDT & isomers					
							0·02	0·04	0·04		0·1
							0·008		0·036	0.05	0·094
								0·058			0·058
								0·036			0·036
								0·026			0·026
								0·01			0·01
									0·02		0·02
									0·01		0·01
									0·01		0·01
Lettuce	21	U.K.	20	1		21 DDT & isomers	0·02		0·06		0·08
	1		1			1 Organochlorines					
Mushrooms	19	U.K.	18	1		19 DDT & isomers	0·003	0·009	0·007		0·019
Pepper (red)	1		1			1 Organochlorines					
						1 Organophosphorus					
Sweetcorn	1		1			1 Organochlorines					
						1 Organophosphorus					
Tamarind extract	1	China	1			1 Organochlorines					
Tomatoes	1		1			1 Organochlorines					
Totals	121		100	18	3						

*The maximum residue limit (MRL) is the maximum concentration consistent with the use of pesticide according to good agricultural practice.

MEAT AND MEAT PRODUCTS

Two hundred and twenty two samples were submitted for examination. The results are summarised in Table VI.

TABLE VI

SUMMARY OF RESULTS ON MEAT PRODUCTS TESTED

	No. of samples	Samples above reporting limit		Samples above MRL*	
		No.	*per cent.*	*No.*	*per cent.*
Beef	33	6	18·2	0	
Chicken	40	7	17·5	0	
Eggs	31	0	0	0	
Lamb	57	31	54·4	1	1·7
Pork	39	11	28·2	0	
Turkey	10	3	30	0	
Veal	12	4	33·3	0	
Total	222	62	27·9	1	0·5

*The maximum residue limit (MRL) is the maximum concentration consistent with the use of pesticide according to good agricultural practice.

Although examinations were carried out for organophosphorus compounds and organochlorine compounds, no residue of organophosphorus could be found in the meat samples except in one sample of lamb.

Several samples contained more than one residual organochlorine chemical; for instance, one chicken from Holland contained traces of *pp'*DDE, *pp'*DDT and Dieldrin and one sample of turkey from an unknown source contained gamma HCH; *pp'*DDT; *pp'*DDE and Dieldrin.

Only one sample was found to exceed the EEC or Codex Maximum Residue Limits and this was a sample of lamb of UK origin which contained 3·57 mg of gamma HCH (Lindane) per kilogram of fat, whereas the Codex MRL is 2 mg/kg.

The risk of obtaining residues in animal flesh as a result of dipping sheep in water treated with organochlorine compounds has been recognised, and appropriate action has been taken. The usage of organochlorine chemicals is now not officially recognised, and product licences have been withdrawn. Less persistent chemicals such as organophosphorus insecticides are now incorporated into the dips.

VEGETABLES

There was a drop in the number of vegetable products examined, from 178 in 1983 to 121 in 1984. The number of samples which were found to contain residues of pesticides was 21 (17·3 per cent.) compared with 37 (20·8 per cent.) in 1983. All the residues detected were DDT and its isomers.

Three of the positive results were at or above the EC maximum levels of 0·1 mg/kg, although they were within the Codex Alimentarius Maximum Residue Limit of 1·0 mg/kg.

Summary

Table VII gives summary of the sampling and analytical involvements.

TABLE VII

SUMMARY OF PRODUCTS TESTED AND RESULTS OBTAINED

Products	No. of samples	Samples above reporting limits		Samples above MRL's*		Determinations	
		No.	*per cent.*	*No.*	*per cent.*	*No.*	*Type*
Flour and cereal products	107	32	29·9	2	1·9	5 91 4	Organochlorines Organophosphorus Synthetic pyrethroids
Fruit	74	10	13·5	1	1·4	6 40 14 6 25 19 2	Biphenyl DDT and its isomers Ethylene dibromide Hydroxy biphenyl Organochlorines Organophosphorus Thiabendazole
Meat and meat products	222	62	27·9	1	0·5	222 147 1	Organochlorines Organophosphorus Polychlorinated biphenyl
Vegetables	121	21	17·3	3	2·5	114 6 2	DDT and its isomers Organochlorines Organophosphorus
1984 Foods Total	524	125	23·8	7	1·3	705	determination
1983 Foods Total	615	81	13·2	7	1·1		

* The maximum residue limit (MRL) is the maximum concentration consistent with the use of pesticide according to good agricultural practice.

The increased percentage of samples found to contain residues of chemicals was primarily due to more emphasis being placed on flour products and meat products during this year.

The number of samples found to contain levels of residues above the maximum recommended limits remained at approximately 1 per cent. of the foods examined. This finding is reasonably satisfactory and indicates that, in general, good agricultural practice is being maintained in relation to the application of pesticide treatments.

The co-operation of the Public Analysts in the following areas or practices in the above work is gratefully acknowledged: Avon, Derbyshire, Dr. B. Dyer & Partners, Hereford & Worcester, Kent, Lancashire, London, Manchester, Mid Glamorgan, Moir and Palgrave, Staffordshire, Strathclyde, Dr A. Voelcker & Sons.

References

1. Sissons, D. J., Telling, G. M., and Usher, C. D., *J. Chromatog.* 1968, **33**, 435.
2. "Manual on Analytical Methods for Pesticide Residues in Foods". Health Protection Branch, Health and Welfare Canada, Ottawa, 1985.
3. "Survey of Pesticide Residues in Food, 1983". *J. Assoc. Publ. Analysts,* 1984, **22**, 51–57.

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A Note on the Role of Occupational Exposure Limits*

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The paper seeks to address the general question of what is meant by Occupational Exposure Limits and how they may be applied to the control of toxic substances in the workplace. Consideration is given to the way in which the limits are now being developed, as well as to the approach to assessment of compliance with them.

With the recent development of general interest in, and awareness of, occupational health matters, it is not surprising that many people responsible for providing a consultant service on chemical matters, such as public analysts, should be turning to occupational hygiene as a natural extension to their business or professional interests. In some cases, this provides an opportunity for the better utilisation of specialised, and expensive, laboratory facilities, but it does, of course, bring with it new responsibilities and problems. Some of the responsibilities have a direct legal implication since an adviser can be called to account for the advice given in assisting other persons to meet obligations under the Health & Safety at Work Act. The analytical aspects of this work may be relatively straightforward to consultant analysts. The real problems are in the assessment of the results in the context of work routines and the design of control systems.

What are Occupational Exposure Limits?

Under the Health and Safety Work Act, an employer has to do everything that is reasonably practicable to protect the health of his employees from risks due to their exposure to hazardous substances at work. When exposure is discussed, more often than not what is really meant is the inhalation of toxic substances in dust, fume or vapour form. There may, of course, be other routes such as ingestion or absorption through the intact skin. In the case of inhalation, it was practice in the UK for the twenty years up to 1980 to refer to the Threshold Limit Values set by the American Conference of the Governmental Industrial Hygienists (ACGIH) when assessing the acceptability of levels of airborne contaminant. The ACGIH limits are regarded as levels of exposure to which most people may be exposed day after day, for the duration of their working life, without adverse health effects. Although this worked well for many years, problems began to emerge as more and more of the ACGIH limits were

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considered inappropriate for the UK situation. Because of copyright, the Health and Safety Executive (HSE) had to adopt the total list without amendment in publishing its own guidance note, Guidance Note EH15, which was last published in 1980.

It was therefore decided that the Health and Safety Commission and Executive should "go it alone" and produce a separate UK list. The home reader should now therefore refer instead to the new HSE Guidance Note EH/40/85: Occupational Exposure Limits 1985. This document will be revised and republished annually. The HSE will use these limits as one means of judging occupational exposures against the requirements of the Health and Safety at Work Act and, of course, it is expected too that employers will have regard to these limits in their monitoring of exposure in the workplace. Public analysts, as consultants, will need to know the up-to-date limits applying to any work done by them in occupational hygiene and also, of course, to assess the exposure of staff employed in their own laboratories.

Occupational Exposure Limits for the UK

Exposure Limits published in EH40/85 apply to all workplaces, not just factories. Some have long standing in Regulations, as in the limits for chromium and coal dust; others have a legal standing based on Approved Codes of Practice, such as lead and asbestos, or in EC Directives which are in the course of implementation in the UK, for instance the Directive relating to vinyl chloride monomer (VCM). The majority find their application under the general duties imposed by the HSW Act and other statutory provisions.

There are now two main categories of limit value:

CONTROL LIMITS

These are limits which are contained in Regulations, Approved Codes of Practice, in European Community Directives, or which have been adopted by the Health and Safety Commission (HSC). They are limits which have been judged after detailed consideration of the available scientific and medical evidence to be "reasonably practicable" for the whole spectrum of work activities in Great Britain. These exposure limits are known specifically as *Control Limits* and should not normally be exceeded.

RECOMMENDED LIMITS

These are limits recommended by the HSE on advice from the HSC's Advisory Committee on Toxic Substances. Recommended Limits are considered to represent good practice and realistic criteria for the control of exposure, plant design, engineering controls, and if necessary, the selection and use of personal protective equipment.

Both types of limit relate to personal exposure (except for cotton dust and VCM) not to background levels. The main distinction between the two types is in the depth to which the scientific and medical evidence has been evaluated. Control Limits may be expected to be enforced more stringently by enforcing authorities but for practical purposes, careful attention should be given to

compliance with *all* exposure limits. Indeed, they should be looked upon as maximum acceptable limits, not the ultimate aim to be achieved. Personal protective equipment is invariably an unwelcome additional burden on an operator and it is difficult to ensure that it is properly and regularly used. It provides a last resort when control at source to below the limit is not reasonably practicable, as in the case of short-term maintenance operations where exposure may otherwise be high.

The list of substances for which exposure limits have been defined is relatively small compared with the number in commercial use. Where none exists, there is an additional duty on the person in control of the activity to determine an appropriate level and to ensure control so that risks to health are minimised. The requirements under Section 6 of the Health and Safety at Work Act are important in this context.

Examination of EH40/85 will show that most substances have been given two limits, one based on an 8 h time weighted average and a further 10 min short-term limit. The latter is to take account of the effects of short-term excursions which inevitably occur with changing work patterns and process variations, or in some cases, to deal with additional acute effects such as irritancy. Both limits should be satisfied. Personal protection will be needed for some operations if a short-term limit is exceeded, even if the 8 h limit is comfortably met without it. The skin notation given to certain substances in EH40/84 provides an indication that the substance concerned may pose an absorption risk even if in contact with the intact skin. Special precautions are necessary in addition to avoidance of airborne contamination and inhalation.

Arrangements for Setting Occupational Exposure Limits

As was mentioned earlier, it is only for the last 4 years that the Health and Safety Commission and Executive have been setting their own limits. In fact, it is not quite as simple or as arbitrary as that may sound. Under the provisions of the Health and Safety at Work Act, the Health and Safety Commission has appointed a tripartite committee, known as the Advisory Committee on Toxic Substances, which it uses to obtain advice on a number of matters concerning toxic substances. This Committee makes recommendations for Control and Recommended Limits. The Committee works to an agreed programme and when a substance is to be considered for the recommendation of a limit, it is normal for Health and Safety Executive staff to prepare detailed submissions on the toxicology of the material concerned, which is provided by the Medical Division of the Health and Safety Executive, and on the scale and extent of the use of the substance, together with information on the levels of exposure which occur in the workplace and the controls which can reasonably be applied as compiled by the Occupational Hygiene Section. These facts together form the basis of a review paper which the Advisory Committee discusses and as a result, recommends the limit which it considers should be applied. The Committee has an HSE Chairman, but its members are appointed after nomination by the Trade Union Congress and Confederation of British Industry, or are present as independent experts on the basis of the specialist expertise which they can provide. The Committee can therefore fairly be regarded as independent and

representative of employer and employee interests. Its recommendations are put to the Health and Safety Commission for adoption in the case of Control Limits or are put to the Health and Safety Executive for acceptance in the case of Recommended Limits. Limits do not take effect until they have been promulgated and, in this respect, the annual publication of Guidance Note EH40 is of major importance, as is the newly introduced "Toxic Substances Bulletin"[2].

A prerequisite for the complete assessment of health risks at work is a knowledge of the effects of the materials concerned, the route of entry to the body, and the nature of the dose and effect. Unfortunately, data are often inadequate for all those to be defined adequately and the occupational exposure limit has to be set on the best available evidence at the time. New evidence may uncover previously unsuspected hazards, as the VCM story shows. This factor must be borne in mind when applying any exposure limit to a safety assessment. In addition, of course, the Health and Safety at Work Act places a legal duty on the person in control of an activity to ensure, so far as is reasonably practicable, that risks to health are minimised. This point is further discussed in Guidance Note EH18—"Toxic Substances: A Precautionary Policy"[3].

Assessing Compliance with Exposure Limits

When carrying out an assessment of exposure in the workplace it is vital that a structured approach is used if the assessment is to be valid and cost-effective. It goes without saying that analytical methods used for quantitative assessment must have sufficient precision and sensitivity for the levels of contaminant that are likely to arise, and certainly to have sufficient sensitivity to detect levels significantly below the occupational exposure limit. Air sampling is expensive and requires specialised services; there is little point in sampling while visible signs of dust leakage and poor working practices are there for all to see. These must be dealt with first. A suitable programme of assessment might be as follows:

PRELIMINARY DATA COLLECTION

First, materials and processes in use are identified, by whom used, how often, and for what purpose. This will provide the basic data for deciding what contaminants might enter the atmosphere directly from handling processes or indirectly from process leaks and fumes. The assessment must include possible effects of a process on an adjacent work area. This preliminary diagnostic work requires an intimate knowledge of work routines, and is often best performed by management and workers in the area(s) concerned, rather than by safety staff or external consultants. At this stage, it is likely that any gross deficiencies will be identified, for example, major dust leaks. These should be dealt with without further delay.

EVALUATION

The significance of the established data must now be evaluated to decide where there may be significant exposures which require further study. Minor matters can be dismissed from further study at this stage. Other matters that are amenable to better control will again emerge and can be dealt with, leaving a

smaller residual element to go forward for atmospheric monitoring to check compliance.

EXPOSURE ASSESSMENT

This too should be planned to avoid unnecessary attention to minor matters, and to direct the attention to areas where further controls need to be instituted and their effectiveness confirmed. A pilot survey of a range of key areas/jobs will often help to order priorities for a full and thorough sampling exercise. The pilot study will again identify action that can be taken in advance of a full survey of personal exposures. Expensive and time-consuming monitoring is usefully directed in this way.

What is often less well appreciated is that exposure levels may vary widely between one operator and another doing nominally the same job, and from shift to shift, and from week to week. Within-shift variations are particularly large in some instances, as with periodic spray application of solvent based materials. The sampling strategy must identify these variations to ensure a valid assessment and compliance with short-term limits. It may help to group workers according to the area or job undertaken, but even so, it may be necessary to sample at least 50 per cent. of those exposed where there is borderline compliance, or where work is particularly variable, such as, for example, the sorting of scrap lead, or hand-sanding operations.

There is further guidance on sampling strategies in a number of Guidance Notes in the Environmental Hygiene (EH) series, notably in the recent EH42 on strategies[4].

Maintenance and Ongoing Assessments

The first thorough assessment can be used to set the standards against which future monitoring to ensure continued compliance can be assessed. It may, for example, be possible to define engineering control parameters, such as extraction air flows, that can be used as a marker to reduce the need for extensive atmospheric sampling. However, it is unwise to place too much reliance on such surrogate measures as they are insensitive to changing operator work patterns. Their advantage lies in their being cheap and easy to perform on a more frequent basis, perhaps using technician level staff.

The ongoing assessments should:

(i) ensure that defined standards are maintained and exposure limits complied with;
(ii) check that personal protective equipment is properly maintained and used, and seek to improve its wearer acceptability where necessary;
(iii) identify changes in work procedures or processes that may signal the need for early corrective action;
(iv) pursue more exacting controls to reduce further the exposure to the lowest level that is reasonably practicable. The exposure limit represents a milestone in this respect but is not in itself the end to be achieved. Although Control Limits take account of what is reasonably practicable, this is only for the processes most difficult to control. Most can be improved.

If it is to be effective, the control regime outlined above needs the active participation and co-operation of all those involved with activities in the workplace. It is not a function solely for the safety specialist or consultant analyst. Many of the techniques used for exposure assessment lack some of the precision of pure chemical analysis. At times, this can be a drawback but more commonly problems arise from incorrect interpretation of the results in the context of the working methods and of the remedial action that needs to be taken to reduce exposure.

References

1. "Guidance Note EH40/85: Occupational Exposure Limits 1985", HMSO, London.
2. "Toxic Substances Bulletin: Health and Safety Executive," St Hugh's House, Bootle.
3. "Guidance Note EH18: Toxic Substances—A Precautionary Policy", HMSO, London.
4. "Guidance Note EH42: Monitoring Strategies for Toxic Substances", HMSO, London.

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Book Reviews

CHEMISTRY OF SULPHUR DIOXIDE IN FOODS. BY B. L. WĘDZICHA. Elsevier Applied Science Publishers. 1984. 365 pp. Price: £38·00

A modest but comprehensive title for this 322 page book which with 42 additional pages of detailed references, plus a practical and well cross-referenced index might in a more commercial area be titled with justification "The key to everything you ever wanted to know about Sulphur Dioxide".

Following an introductory chapter on the properties and reactions of sulphur dioxide the remainder is divided into six main chapters. Some 25 per cent of the book is devoted to all aspects of analysis from classical wet chemistry to automated systems, with constructive appraisal, performance comparisons, and applications, including the identification and quantification of the reaction products formed with other food components.

There follow chapters on the use of sulphur (IV) oxoanions, and the likely mechanisms involved, in the control of non-enzymic browning in food and the inhibition of enzyme systems which is well known. However, reference is also made to enzyme systems which are unaffected, those whose activity is enhanced and some which oxidise or reduce the additive itself.

Further chapters deal with the use of sulphur dioxide to prevent microbial spoilage and the factors influencing its performance, including reactions with microbial metabolites such as aflatoxin and the antagonistic and synergistic effects which occur with some combinations of additives.

A chapter on the uses of sulphur dioxide as a food additive adds information concerning interactions between sulphur (IV) oxoanions and other components in food systems such as tin plate, flavourings, colours and dough mixes to the applications already covered.

Finally, the toxicology of sulphur (IV) is reviewed to round off a comprehensive book which will serve as a valuable initial source of reference to all those associated with the study and practice of food science with ample guidance for any further reading required.

A. J. HARRISON

ANALYSES OF HAZARDOUS SUBSTANCES IN BIOLOGICAL MATERIALS, Vol. 1. Edited by J. Angerer and K. H. Schaller. VCH, Weinheim. 1985. Price DM 90 ($36). 222 pp.

This book is described as of interest to analytical chemists in industry, government agencies and university, safety guards, toxicologists, and specialists in forensic and industrial medicine. Being of German origin and translated in the

U.S.A. it is easy to see why the inclusion of public analysts in the list of users has been over-looked.

Also, in the preface Dr Henschler, the Chairman of the Commission for the investigation of Health Hazards of Chemical Compounds in the Work Area, states that the Editors have always had to confirm the postulate of the Commission and in case of doubt to select the more demanding method.

After preliminary remarks which include instructions as to the collection of specimens, there is a very short collection of the terms used, with definitions, and also a list of the symbols used.

The main body of the book follows with specification-type monographs dealing with detection of sixteen different substances often encountered in working conditions. Most the methods deal with carcinogenic substances and their metabolites whilst toxic metals like cadmium, cobalt and lead are not over-looked, and carbon monoxide is detected by Gas Chromatography using a neat chemical technique. Most of the methods depend on HPLC, but various forms of gas chromatography and atomic absorption spectroscopy are not overlooked where suitable. Thallium, however, is determined by inverse voltammetry.

The book is well printed, free from typographical errors, opens easily for laboratory use and can be thoroughly recommended, especially as at this moderate price it could be in all laboratories where work is undertaken to ensure compliance with the Health and Safety Rules and Regulations.

The main "biological fluid" examined is urine, hence the quantity of sample and it's acquisition are not difficult to ensure. However, it is advised that many substances, including carbon monoxide, must be sought for in blood.

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