

## The MAFF Programme on Food Authenticity

The term food authenticity is one which is not easily defined, but in the context of MAFF's programme it refers to food which has been misdescribed or not correctly labelled and is therefore not "of the nature or substance or quality demanded by the purchaser". Misdescription usually takes one or more of the following forms:

- Incorrectly labelled treatment;
- Mislabeled geographic/biological origin;
- Ingredient substitution in order to pass off food as a better quality product;
- Product extension in order to pass off food as a better quality product.

MAFF's programme on food authenticity can be divided into two parts:-

### R&D

For a number of years, MAFF has funded research projects to develop methods suitable for checking that food is correctly described, though it was only in 1991 that a food authenticity R&D programme was formally established and included in the MAFF R&D Requirements Document. Each year the Ministry spends over £1 million on food authenticity research.

### Surveillance

The MAFF's Working Party on Food Authenticity (WPFA) is one of the 11 working parties that monitor the UK's food supply to assess its safety and adequacy, and in this case whether foods have been correctly described and are not adulterated. The WPFA, which was established in 1992, co-ordinates the work on food authenticity and in particular oversees the national surveys.

It has developed a working system where authenticity issues are prioritised so that resources are available for the most important issues. It evaluates the methodology to be used for surveys to ensure that it is robust and any limitations quantified. It draws up sampling and analytical protocols for surveys to ensure these are carried out in a standardised manner. Where gaps are identified in methodology, these can be co-ordinated within the priorities of the R&D programme.

Surveys are carried out on a national basis to assess whether food sold in the UK is properly labelled. The findings of this work are made publicly available, through the MAFF/DH Food Safety Information Bulletin, and other publications. To date the WPFA has carried out six surveys to investigate:

- the species authenticity of coated white fish products;

- the authenticity of soluble coffee;
- the authenticity of dried durum wheat pasta;
- the authenticity of four single seed vegetable oils (corn, palm, sunflower and peanut);
- whether fresh meat and poultry has been previously frozen and thawed;
- the undeclared irradiation of foods (herbs and species, fruit and vegetables, raw poultry, shrimps and prawns and liquid egg).

A further three surveys are currently underway to investigate:

- the substitution of whisky brands sold through licensed premises (on-trade);
- the level of added water in cured meat products;
- adulteration of orange juice.

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## AUTHENTICITY OF SINGLE SEED VEGETABLE OILS

### - a survey of the UK market

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*The Ministry of Agriculture, Fisheries and Food's Working Party on Food Authenticity has carried out a survey to investigate the purity of four types of single seed vegetable oil (maize, sunflower, groundnut and palm) sold through retail and catering outlets. A total of 290 samples (79 maize oil, 110 sunflower oil, 82 groundnut oil and 19 palm oil) were collected from throughout the UK. All samples were screened on the basis of the fatty acid composition for the presence of oils other than that named on the product label. The  $^{13}\text{C}/^{12}\text{C}$  stable isotope ratio of maize oils was also determined and the slip melting point of palm oils was measured. The desmethylsterol composition and/or tocopherol composition of samples suspected of containing three percent or more of an undeclared oil (on the basis of the fatty acid composition) were determined in order to try and detect and quantify the presence of rapeseed and soyabean oil respectively. The bulk of the samples (81%) were found to contain less than three percent of an undeclared oil. Approximately 11% of the samples contained three to five percent of another oil which is higher than would be expected as a result of unavoidable mixing during processing. A further 7% of samples contained in excess of five percent of an undeclared oil which is suggestive of deliberate adulteration.*

## INTRODUCTION

Edible oils and fats can be obtained from a variety of animal and vegetable sources. The vegetable oils represent the largest and most diverse grouping and are the most important from a commercial point of view. Single seed vegetable oils tend to be more costly than blends (mixtures of two oils, usually rapeseed and soyabean) and within the single seed sector, certain products trade at a premium in comparison with others as the result of economic forces. Groundnut oil, for example, is significantly more expensive than rapeseed oil. The price

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difference between various products could provide some manufacturers with the financial incentive to adulterate more expensive oils with cheaper ones.

Although there is no specific legislation controlling the labelling of vegetable oils in the UK, the general provisions of the Food Safety Act 1990<sup>(1)</sup> and the Food Labelling Regulations 1984<sup>(2)</sup> (now superseded by the Food Labelling Regulations 1996<sup>(3)</sup>) make it an offence to misdescribe a product or present it in a misleading manner. The practice of presenting or labelling an adulterated single seed oil as a pure product prejudices the interests of both consumers and honest traders.

## BACKGROUND TO THE SURVEY

The potential adulteration of single seed vegetable oils was considered in July 1993 by MAFF's Working Party on Food Authenticity (WPFA). Information on the Working Party's structure, terms of reference and surveillance programme, together with details of its mechanism for considering authenticity issues are reported in *Food Surveillance Papers Numbers* 41, 45 and 49.<sup>(4,5,6)</sup> The vegetable oil issue was given high priority for surveillance. In assigning this priority the Working Party took into account evidence obtained from the industry's own oil monitoring programme which was established in 1988 by the National Edible Oils Distributors' Association following reports that certain oils were being traded at unrealistically competitive prices.<sup>(7)</sup>

The WPFA reviewed the analytical techniques available for determining the purity of vegetable oils. Different methods were objectively evaluated by comparing a number of essential parameters, namely: the limit of detection; limit of determination; relationship of analyte to adulterant; sensitivity; specificity and accuracy (trueness and precision). In addition, a number of other useful parameters, such as cost, availability of equipment, required training for analysts etc., were assessed. Details of the methods examined in the review are given at Appendix I. These included techniques for the determination of the fatty acid, *trans* fatty acid, triglyceride, sterol and the tocol (tocopherols and tocotrienols) composition of oils and for the measurement of the iodine value, stable carbon isotope ratio and slip melting point.

It was considered that all of the methods examined are well established for determining the purity of vegetable oils. With regard to the survey, it was decided that the fatty acid composition (FAC) of all samples should be determined as this would provide an important contribution to the assessment of the oil's purity. It was recognised, however, that analysis of other components may also be required in order to establish the authenticity of certain oils, the choice of which would depend on the suspected adulterant. In the case of maize, sunflower and groundnut oils, the most likely adulterants are rapeseed oil and to

a lesser extent, soyabean oil. Rapeseed and soyabean are major oilseed crops and the oils obtained from these sources are, therefore, readily available. The oils have similar physical properties to the premium products, including for example colour and fluidity, but trade at significantly lower prices.

The Working Party subsequently established a sub-group, the Vegetable Oil Sub-group, to devise and co-ordinate a surveillance exercise to investigate the purity of certain premium vegetable oils sold in the UK.

## SURVEY DESIGN

The samples chosen for the study were those that trade at a premium and are thus potential candidates for adulteration. These were maize (or corn) oil, sunflower (or sunflowerseed) oil, groundnut (or peanut) oil and palm oil.

Samples were collected during May to September 1994 by Trading Standards and Environmental Health Departments and by MAFF officials from retail and catering outlets throughout the UK.

The aim of the survey was to obtain a 'snapshot' of the authenticity of certain premium vegetable oils sold in the UK. The sampling strategy therefore concentrated on examining as many branded and own label products as possible but within the resources available could not be fully representative of the market. The samples were obtained from national, regional and local retailers, as well as manufacturers and independent bottlers and packers. The term 'retail outlet' was taken to include supermarkets, convenience stores, ethnic food shops, health food shops, discount centres and freezer centres. Catering samples were obtained from fast food outlets, restaurants, hotels, pubs, institutional caterers etc. and from wholesalers (including cash and carries). Samples taken for analysis were obtained from containers that had previously been unopened so that there was no risk of mixing with other oils in the catering establishment.

A total of 290 samples were submitted for analysis. Of these, 79 were labelled as maize or corn oil, 110 as sunflower or sunflowerseed oil, 82 as groundnut or peanut oil and 19 as palm oil.\*

Of the samples collected, 206 (71%) were obtained from retail outlets and 84 (29%) were destined for the catering sector. The samples included major and lesser known brands and retailer own-label products and were manufactured in the UK as well as a number of other European and non-European countries.

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\* These numbers are slightly different to those previously reported in the *Food Safety Information Bulletin*.<sup>(8)</sup> In preparing the final report one groundnut oil and one sunflower oil, both originally categorised as containing less than three percent of an undeclared oil on the basis of fatty acid composition, were found to have been hydrogenated. They are not included here since it would not be appropriate to evaluate them using purity criteria for unhydrogenated oils. In addition, data relating to one extra palm oil have been included.

The majority of the samples collected were fully processed products but a small proportion (20 samples or approximately seven percent) were described as unrefined or cold-pressed.

## **ANALYTICAL APPROACH AND METHODOLOGY**

### **Assessment of purity**

An oil may be shown to be adulterated or contaminated if it can be demonstrated that it contains either compounds not normally found in the pure product or compounds that are present at significantly different concentrations than are usually encountered.

The major constituents of edible oils are triacylglycerols (triglycerides) which are esters of glycerol and three fatty acids. Fatty acids are considered to be the most important aspect of an oil's composition as they greatly influence its physical and chemical properties. Further, the fatty acid composition (FAC) of a pure oil tends to be characteristic and, to a considerable extent, distinguishes it from other oils. Analysis of the FAC can, therefore, be used as a means of detecting the presence of undeclared oils.

Oils are complex mixtures and contain many components other than triglycerides. These include monoglycerides and diglycerides, free fatty acids, pigments, waxes, sterols and tocols. As with the fatty acid profile, the sterol and tocol composition of an oil tends to be characteristic and analysis of these compounds can similarly be used as a means of assessing purity.

The authenticity of the surveillance samples was thus assessed by comparing their chemical characteristics with those of the relevant pure vegetable oil. The database of purity criteria employed has been developed at Leatherhead Food Research Association (LFRA) during the past fifteen years as part of a project largely funded by MAFF.<sup>(9)</sup> Source materials were collected from different areas of the world over a number of harvest years. Oils were extracted from the oilseeds, kernels, etc. in the laboratory and were, therefore, guaranteed to be authentic. It was not, however, possible to obtain samples of palm oil in this way because the fruit is not suited for transportation. Palm oil samples of known authenticity were thus obtained through industry contacts and the Palm Oil Research Institute of Malaysia (PORIM).

The purity criteria have been established for crude oils rather than for the highly refined products that are sold at retail. While the refining process has little or no effect on the fatty acid composition or the stable carbon isotope ratio, some of the operations carried out, particularly deodorisation, reduce the concentrations of desmethylsterols and tocopherols in oils. It would, however, be extremely difficult to develop purity criteria for fully refined oils because of the wide

variation in processing conditions employed. Further, low-level unavoidable mixing of one product with another occurs during commercial processing making it difficult to obtain authentic samples.

The compositional data obtained for surveillance samples were compared to that for the relevant authentic oil (see Appendix II).

### First phase analysis

#### *Fatty acid composition*

During the first phase of the investigation, all samples were screened for the presence of undeclared oils on the basis of the FAC. This was determined using the technique described in British Standards BS 684: Part 2: Section 2.34 and BS 684: Part 2: Section 2.35<sup>(10,11)</sup> (these standards are dual numbered with ISO 5508<sup>(12)</sup> and ISO 5509<sup>(13)</sup>).

The full fatty acid profile of surveillance samples was compared with that of the relevant authentic oil. Particular attention was paid to the linolenic acid (C18:3) content of oils labelled as maize, sunflower or groundnut because the concentration of this fatty acid is much lower in these products than in the likely adulterant oils (see Table 1). For the purposes of the survey, samples containing linolenic acid at concentrations in excess of the maximum found for the relevant reference authentic oil were suspected of containing an oil other than that named on the product label. The percentage undeclared oil present was calculated according to the formula given at Appendix III.

**Table 1**

#### **Linolenic acid concentrations in crude vegetable oils** <sup>(14, 15,16)</sup>

Oil	Linolenic acid (% total fatty acids)	
	Range	Mean
Maize	0.7 - 1.4	1.0
Sunflower	nd - 0.1	na
Groundnut	nd - 0.1	na
Rapeseed	6.5 - 14.1	10.2
Soyabean	5.5 - 9.5	7.6

Notes: na = not applicable  
nd = not detected

*Iodine value*

The iodine value was calculated from the concentration of individual fatty acid methyl esters (as determined from the FAC analysis) using the American Oil Chemists Society method.<sup>(17)</sup>

*Stable carbon isotope ratio*

Approximately one percent of total carbon is found in the form of the naturally occurring stable isotope, <sup>13</sup>C. The absolute concentration of this isotope in plants is dependent on the photosynthetic pathway used for the fixation of carbon dioxide. In the majority of plants, this is accomplished using the Calvin cycle or C-3 pathway. Maize, however, is one of a small number of plants which employs the Hatch-Slack or C-4 pathway which is less discriminating against the <sup>13</sup>C isotope than the C-3 pathway. As a result, the ratio of the <sup>13</sup>C isotope to the more abundant <sup>12</sup>C isotope (<sup>13</sup>C/<sup>12</sup>C ratio) in maize is significantly different to that in the other oils of major commercial importance (see Table 2). The stable carbon isotope ratio (SCIR) can thus be used as a means of detecting the presence of C-3 vegetable oils in maize oil. Consequently, the <sup>13</sup>C/<sup>12</sup>C stable isotope ratios of surveillance samples described as maize or corn oil were also determined.

**Table 2**

**Stable carbon isotopic ratio ranges of commercial vegetable oils <sup>(18)</sup>**

Oil	Stable carbon isotope ratio	
	Range	Mean
Cottonseed	-27.40 to -28.28	-27.78
Groundnut	-26.48 to -28.69	-27.87
Palm olein	-29.51 to -29.84	-29.65
Palm kernel	-27.49 to -30.27	-29.47
Palm oil	-29.25 to -29.91	-29.64
Rapeseed	-27.47 to -29.40	-28.56
Safflower	-27.87 to -30.17	-28.94
Sesame	-25.38 to -29.28	-27.93
Sunflower	-27.94 to -29.76	-28.95
Soyabean	-29.67 to -30.55	-30.09
Cereal and virgin olive oils	-28.90 to -32.39	-30.79
Maize	-13.71 to -16.36	-14.95
All vegetable oils excluding maize	-25.38 to -32.39	-28.99

There are no known national or international standards for this measurement in relation to edible oils. Nevertheless, the method has been used routinely in many



laboratories throughout the world for several years and has been employed for establishing the purity of a range of other food commodities. The analytical procedure used is relatively straightforward and is described in Appendix I.

Samples with a SCIR outside the range for pure maize oils of -13.71 and -16.36 were suspected of containing undeclared oil. The percentage undeclared oil was established using the formula detailed at Appendix III.

#### *Slip melting point*

The slip melting point of palm oils, which are solid at room temperature, was measured as a means of detecting the presence of palm stearin using BS 684: Part 1: Section 1.3<sup>(19)</sup> which is equivalent to ISO 6321.<sup>(20)</sup>

#### **Second phase analysis**

Samples which were found not to comply with the purity criteria for the oil named on the product label were further analysed in the second phase of the study in order to try and establish the identity of the undeclared oil present.

As mentioned previously, the most likely adulterants of the premium oils included in this survey are rapeseed and soyabean oils. Rapeseed oil is characterised by a high concentration of brassicasterol and soyabean oil by a high concentration of  $\delta$ -tocopherol (see Table 3).

**Table 3**  
**Brassicasterol<sup>(14, 15)</sup> and  $\delta$ -tocopherol<sup>(15, 16)</sup> concentrations in crude vegetable oils**

Oil	Brassicasterol (mg/kg oil)		$\delta$ -Tocopherol (mg/kg oil)	
	Range	Mean	Range	Mean
Maize	nd - 30	6 <sup>a</sup>	23 - 75	54
Sunflower	nd - 10 <sup>a</sup>	na	nd - 7	0.5
Groundnut	nd - 3	0.1 <sup>a</sup>	nd - 22	9
Palm	nd	na	nd - 123	3
Rapeseed	511 - 1079	720	nd - 22	9
Soyabean	nd-10	4	154 - 932	425

Notes: <sup>a</sup> The data on the range in concentration of brassicasterol in sunflower oil and the mean brassicasterol contents of maize and groundnut oil were supplied by Leatherhead Food Research Association (personal communication).

na = not applicable

nd = not detected

Elevated levels of brassicasterol or  $\delta$ -tocopherol in oils described as maize, sunflower or groundnut are thus indicative of the presence of rapeseed and soyabean oil respectively. In view of this, the second phase of analysis involved determination of the desmethylsterol and tocol compositions of suspect samples. Desmethylsterol composition was determined according to British Standard method BS 684: Part 2: Section 2.38<sup>(21)</sup> (which is dual numbered with ISO 6799<sup>(22)</sup>) and tocol composition using the method described in ISO 9936 which is currently in draft form.<sup>(23)</sup>

The percentage undeclared oil present was calculated (see Appendix III) for those samples where the brassicasterol content and/or the  $\delta$ -tocopherol content was greater than the maximum found in the relevant authentic oil.

### **Authenticity limits applied**

Edible oil processing and refining is usually conducted on a very large scale and often one type of oil will pass through the system immediately after another. It is not realistic or economical to completely remove the residue of oil from pipelines, deodorisers etc. after each batch has been processed and a certain amount of mixing of one product with another will therefore occur. Consequently, it is generally accepted that contamination of one product with another is unavoidable in commercial vegetable oil operations. However, if good manufacturing practice is adhered to, it is unlikely that a product will contain more than one to two percent of the previous oil that has passed through the production line. Accordingly, for the purposes of the survey, oils found to contain less than three percent of an undeclared oil, within the limitations of the analytical methods employed, were not considered to be adulterated. Samples found to contain undeclared oils at levels of three to five percent, although not necessarily adulterated, were considered to contain higher levels than would be expected if good manufacturing practice was followed. More than five percent of another oil in a sample was considered to be suggestive of adulteration.

## **QUALITY CONTROL MEASURES**

Quality control and assurance measures were incorporated at each stage of the analytical protocol followed in order to ensure that the data produced were accurate and reliable.

### **Fatty acid analysis**

Each of the participating laboratories was supplied with four blind replicate samples of a maize oil which were coded such that they were indistinguishable from the surveillance samples. Further, duplicates of four commercially obtained reference samples, which were labelled as maize, sunflower, rapeseed and soyabean oil as appropriate, were supplied. Comparison of the data obtained for

the blind replicates with the commercial reference samples is made in Appendix IV and demonstrates that the laboratories were capable of determining the FAC of an oil and that there was significant agreement between the results.

In addition to analysis of the blind samples and the MAFF commercial reference samples, each laboratory incorporated its own quality control measures. These included analysis of certified reference material and/or in-house reference samples and duplicate analysis of a certain proportion of the surveillance samples.

### **Stable carbon isotope ratio analysis**

A similar procedure was used to ensure quality control of the SCIR measurements. To achieve this, the laboratory which conducted these analyses determined the  $^{13}\text{C}/^{12}\text{C}$  ratio of a maize oil reference sample which had previously been analysed by LFRA when establishing the purity criteria with which the surveillance samples were being compared.<sup>(18)</sup> The data obtained by each laboratory are given in Appendix IV. The results were very similar although the LFRA values were approximately one percent greater. Thus, to ensure that comparisons with the purity criteria were fully valid, a correction factor was applied to the SCIR data obtained for surveillance samples.

As with the FAC analysis, the SCIR of four blind replicate samples of a maize oil was determined. The results of these analyses, which are also given in Appendix IV, demonstrate that the technique being used was repeatable as well as reproducible.

### **Sterol and tocol analysis**

The laboratories which carried out desmethylsterol analysis were each sent six samples of an identical commercially obtained maize oil which were coded such that they were indistinguishable from the surveillance samples. The results obtained from each laboratory showed significant agreement (see Appendix IV). As with the other analyses, each of the participating laboratories also employed their own internal quality control measures.

The laboratory which conducted the tocol work was required to analyse the composition of three identical commercially obtained reference samples of maize oil. The results obtained are given in Appendix IV and were found to be repeatable.

## **RESULTS**

### **Maize oils**

Of the 79 maize samples submitted for analysis, 32 were found to be consistent with authentic maize oil in respect of both the fatty acid characteristics and the

SCIR. The remaining samples were considered to be impure on the basis of either one or other (30 samples) or both (17 samples) of these criteria. The concentration of undeclared oil present in these suspect samples was calculated on the basis of the individual analytical parameter. The results obtained are summarised in Table 4. When the results of both analyses were taken into consideration, 16 samples were believed to contain undeclared oil at a concentration of three to five percent. An additional 12 samples were suspected of containing more than five percent undeclared oil. Desmethylsterol and/or tocol analysis of these samples was undertaken to try and establish if rapeseed or soyabean oils were present.

**Table 4**  
**Concentration of undeclared oil present in maize samples**

% undeclared oil	Analytical parameter					
	Linolenic acid		SCIR		Linolenic acid and SCIR	
	No of samples	% of samples	No. of samples	% of samples	No of samples	% of samples
not detected	51	65	43	54	32	41
< 3	12	15	15	19	19	24
3 to 5	11	14	11	14	16	20
> 5	5	6	10	13	12	15
<b>Total</b>	<b>79</b>	<b>100</b>	<b>79</b>	<b>100</b>	<b>79</b>	<b>100</b>

The brassicasterol content of nine of the 16 samples containing three to five percent impurity suggested the presence of rapeseed oil. The concentrations of rapeseed oil in these samples was calculated to be between three and six percent. Three of the other samples in this group were also found to contain rapeseed oil but only at a concentration of around one percent. These were, however, samples of refined oils so the concentrations of rapeseed oil detected may have been underestimated as the sterol content of the oil blend is likely to have been reduced during processing. It is also possible that some other unidentified undeclared oil may have been present. The brassicasterol and  $\delta$ -tocopherol contents of the remaining four samples in this group were consistent with authentic maize oil. It was not, therefore, possible to identify the undeclared oils present in these products.

Rapeseed oil was also detected by desmethylsterol analysis in seven of the twelve maize oil samples found to contain more than five percent undeclared oil. In one case, it was established that the rapeseed oil content was approximately

27%. This finding confirmed the results obtained from the fatty acid screening test and the SCIR analysis which indicated that this product contained 24% and 23% undeclared oil respectively. Another sample was found to contain 82% rapeseed oil. This sample appeared to have characteristics more representative of rapeseed oil than maize oil as it was found to contain 89 and 78% undeclared oil on the basis of the linolenic acid content and SCIR respectively.

The five remaining samples found to contain in excess of five percent undeclared oil did not appear to contain either rapeseed or soyabean oil (as determined by tocopherol analysis). Consequently, the identity of the undeclared oils present was not established in these cases. Four of these samples were found to contain undeclared oil on the basis of the SCIR only. This is, however, a very robust technique and oils with values outside those normally encountered for authentic maize oils can justifiably be considered suspect. Repeat SCIR analysis of these samples was carried out and the results obtained were in excellent agreement with the initial findings. In view of this, these samples were strongly suspected of containing more than five percent undeclared oil.

### **Sunflower oils**

The maximum concentration of linolenic acid found in authentic sunflower oil is 0.1% of the total fatty acid content.<sup>(16)</sup> Of the 110 samples included in the study, 51 were found to contain 0.1% or less linolenic acid. The concentrations of undeclared oil present in the remaining samples which had a linolenic acid content of more than 0.1% were calculated and are detailed in Table 5.

Nine samples labelled as sunflower oil were found to contain from three to five percent undeclared oil. The brassicasterol content of seven of these samples suggested that rapeseed oil was present at concentrations in the range three to six percent. Rapeseed oil was detected in one additional sample in this group but at a concentration of less than one percent. However, the particular product in question was a refined oil so it is possible that the concentration of rapeseed oil may have been underestimated. Although the presence of soyabean oil in this sample was not detected by tocopherol analysis, it is possible that another unidentified undeclared oil had been added. The brassicasterol and  $\delta$ -tocopherol contents of the ninth sample in this category were consistent with authentic sunflower oil so it was not possible to establish the nature of the impurity in this instance.

**Table 5****Concentration of undeclared oil present in sunflower samples established from the linolenic acid content**

% undeclared oil	No. of samples	% of samples
not detected	51	46
< 3	47	43
3 to 5	9	8
> 5	3	3
<b>Total</b>	<b>110</b>	<b>100</b>

Three samples labelled as sunflower oil were found to contain more than five percent of an undeclared oil. The specific amounts of undeclared oil detected were 6, 23 and 57%. It was established from desmethylsterol analysis that the first two of these samples contained 6 and 41% rapeseed oil respectively. The third sample contained elevated levels of  $\delta$ -tocopherol and it was calculated that the sample had a soyabean oil content of 85%.

**Groundnut oils**

The maximum concentration of linolenic acid found in pure groundnut oil is 0.1% of the total fatty acid content.<sup>(16)</sup> Of the 82 samples included in the study, 17 were found to contain 0.1% or less linolenic acid and were thus consistent with the authentic named oil. The remaining 65 samples contained levels of linolenic acid in excess of 0.1% and were therefore suspected of containing an undeclared oil. The calculated levels of undeclared oil are shown in Table 6.

Seven samples were found to contain three to five percent undeclared oil. The concentration of brassicasterol in all of these samples was greater than the maximum of 3mg/kg found in authentic groundnut oil<sup>(14)</sup> and it was calculated that rapeseed oil was present at levels of between one to six percent. A further seven samples contained between 10 and 57% undeclared oil. Desmethylsterol analysis provided evidence that the identity of the undeclared oil in these samples was also rapeseed oil.

Table 6

**Concentration of undeclared oil present in groundnut samples established from the linolenic acid content**

% undeclared oil	No. of samples	% samples <sup>a</sup>
not detected	17	21
< 3	51	62
3 to 5	7	9
> 5	7	9
<b>Total</b>	<b>82</b>	<b>100</b>

Notes: <sup>a</sup> The percentage values for each of the individual categories of oil do not add up to the total because of rounding.

### **Palm oils**

The majority of the 19 samples tested had fatty acid compositions that were fully consistent with that of authentic palm oil. Some deviations from the FAC were detected in six samples in respect of the oleic and/or linoleic acid contents. These deviations, however, were of a minor nature and may have been a consequence of the difficulties in ensuring that the sample of palm oil taken was representative of the whole product. These difficulties arise because unlike the other oils included in the survey, palm oil is solid at ambient temperatures. These samples were not, therefore, suspected of containing undeclared oil.

One further sample exhibited a slip melting point of 27.7°C which is below the range established for authentic palm oils (32.7 - 39.6°C<sup>(24)</sup>). The reason for this is unclear especially as the fatty acid composition of the sample was not markedly different from that of pure palm oil. Thus again, this product was not suspected of containing any undeclared oil on the basis of the analyses performed.

### **Retail and catering samples**

The samples included in the survey were destined for either retail sale (71%) or for use in the catering sector (29%). The results from the analytical studies for samples from each of these sectors are summarised in Table 7.

## DISCUSSION

The majority of samples included in the survey (approximately 93%) were refined oils. The authenticity of all surveillance samples, however, was assessed against purity criteria for crude oils (see Appendix II). This approach has a bearing on the interpretation of the data from the analytical studies and is such that the degree of adulteration is likely to be underestimated. This is particularly relevant in the case of assessments made on the basis of the desmethylsterol and tocopherol composition of the surveillance samples as the concentration of these components is reduced during certain refining processes.

It is generally accepted that a certain amount of unavoidable mixing of one oil with another occurs during commercial scale refining of edible vegetable oils. Thus, for the purposes of this survey, samples found to contain less than three percent of a undeclared oil were considered unlikely in most cases to have been deliberately adulterated. The majority of the surveillance samples (81%) fell into this category and were thus correctly labelled with respect to the named oil (see Table 8 which presents the overall findings of the survey).

Approximately 11% of the samples contained three to five percent undeclared oil. Whilst these samples were not necessarily deliberately adulterated, the concentration of undeclared oil present suggests that good manufacturing practices had not been adhered to. The desmethylsterol composition of most of the samples in this group indicated that the likely identity of the undeclared oil was rapeseed oil which is less costly than the premium oils named on the product labels. The identity of the undeclared oil in some samples was not established. Although rapeseed or soyabean oil were not detected in these samples, their presence cannot be completely precluded as the oils in question were refined. It is also possible that these products contained other unidentified undeclared oil.

The finding that overall approximately seven percent of the samples tested (15% of maize oils, three percent of sunflower oils and eight percent of groundnut oils - see Table 8) contained more than five percent undeclared oil is of concern. Such levels are suggestive of deliberate adulteration and these oils are considered to have been misdescribed. Again, in the majority of cases, chemical analysis indicated that the identity of the adulterant was probably the cheaper rapeseed oil. Of these adulterated samples, 55 percent (four percent of the total samples) contained more than ten percent undeclared oil and were thus considered to have been grossly contaminated.

Examination of the data obtained from the analytical studies for samples from the different sectors of the vegetable oil market indicates that oils produced by unsatisfactory manufacturing procedures and impure oils are being presented for sale both in retail outlets and to catering establishments



**Table 7**  
**Concentration of undeclared oil present in retail and catering samples**

% undeclared oil	Retail samples		Catering samples	
	No. of samples	% of samples <sup>a</sup>	No. of samples	% of samples <sup>a</sup>
<b>Maize</b>				
< 3	43	70	8	44
3 - 5	12	20	4	22
> 5	6	10	6	33
<b>Total</b>	<b>61</b>	<b>100</b>	<b>18</b>	<b>100</b>
<b>Sunflower</b>				
< 3	77	90	21	88
3 - 5	7	8	2	8
> 5	2	2	1	4
<b>Total</b>	<b>86</b>	<b>100</b>	<b>24</b>	<b>100</b>
<b>Groundnut</b>				
< 3	50	85	18	78
3 - 5	5	8	2	9
> 5	4	7	3	13
<b>Total</b>	<b>59</b>	<b>100</b>	<b>23</b>	<b>100</b>
<b>Palm</b>				
< 3	-	-	19	100
3 - 5	-	-	-	-
> 5	-	-	-	-
<b>Total</b>	-	-	<b>19</b>	<b>100</b>
<b>All samples</b>				
< 3	170	83	66	79
3 - 5	24	12	8	10
> 5	12	6	10	12
<b>Total</b>	<b>206</b>	<b>100</b>	<b>84</b>	<b>100</b>

Notes: <sup>a</sup> The percentage values for each of the individual categories of oil may not add up to totals because of rounding.

**Table 8**

**Concentration of undeclared oil in surveillance samples**

% undeclared oil	Maize oils		Sunflower oils		Groundnut oils		Palm oils		All samples	
	No of samples	% of samples	No of samples	% of samples	No of samples	% of samples	No of samples	% of samples	No of samples	% of samples <sup>a</sup>
not detected	32	41	51	46	17	21	19	100	119	41
< 3	19	24	47	43	51	62	-	-	117	40
3 - 5	16	20	9	8	7	9	-	-	32	11
> 5 - 10	8	10	1	1	1	1	-	-	10	3
> 10	4	5	2	2	6	7	-	-	12	4
<b>Total</b>	<b>79</b>	<b>100</b>	<b>110</b>	<b>100</b>	<b>82</b>	<b>100</b>	<b>19</b>	<b>100</b>	<b>290</b>	<b>100</b>

Notes: <sup>a</sup> The percentage values for each of the individual categories of oil do not add up to the total because of rounding

## CONCLUSIONS

The analytical approach adopted for the surveillance exercise was effective in detecting the presence of undeclared oils. Examination of the fatty acid profile is well established as a means of authenticating vegetable oils and was a useful screening technique. For maize oil, measurement of the stable carbon isotope ratio provided a robust method for detecting undeclared oils and may be considered as a better first phase procedure. Analysis of additional parameters was required and was successful in identifying the adulterant in many of the suspect samples.

## FOLLOW-UP ACTION

A preliminary summary of the results of the survey has previously been made available to the public through the MAFF/Department of Health *Food Safety Information Bulletin*.<sup>(8)</sup> A full report of the survey was subsequently produced and is also available to any interested parties.<sup>(25)</sup> The individual Trading Standards and Environmental Health Departments which participated in the survey have been informed of the detailed results of the samples which they collected so that appropriate follow-up action can be initiated. In addition, manufacturers and retailers of products which were found to contain three percent or more undeclared oil were informed of the results for their samples so that manufacturing practices can be reviewed and improved where necessary.

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Mr J Turnbull	Association of Public Analysts
Mr A Wakelin	British Retail Consortium
The Late Mrs P Waring	National Federation of Consumer Groups

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## APPENDIX I

### METHODS WHICH MAY BE USED TO DETERMINE THE PURITY OF VEGETABLE OILS EVALUATED BY THE WORKING PARTY ON FOOD AUTHENTICITY

#### **Determination of the fatty acid composition**

##### *Basis of procedure*

The method involves the derivatisation of the oil with the production of fatty acid methyl esters (FAME) which are separated by capillary column gas-liquid chromatography (GLC) and quantified using flame ionisation detection (FID).

##### *Relevant standards*

BS 684: Part 2: Section 2.34 <sup>(1)</sup> and BS 684: Part 2: Section 2.35 <sup>(2)</sup>

ISO 5508 <sup>(3)</sup> and ISO 5509 <sup>(4)</sup>

IUPAC Method No 2.302 <sup>(5)</sup>

#### **Determination of the fatty acid composition at the 2-position of the triglyceride**

##### *Basis of procedure*

This determination involves neutralisation of the sample (removal of free fatty acids) by column chromatography followed by incubation of the oil with pancreatic lipase under carefully controlled conditions to yield 2-monoglycerides. The 2-monoglycerides are then separated by thin layer chromatography (TLC) and derivatised to produce FAME which are analysed using capillary column chromatography with FID.

##### *Relevant standards*

BS 684: Part 2: Section 2.39 <sup>(6)</sup>

ISO 6800 <sup>(7)</sup>

#### **Determination of tocopherols and tocotrienols (tocols)**

##### *Basis of procedure*

A solution of the oil in hexane or heptane is injected on to a normal phase high performance liquid chromatography (HPLC) system. The tocols are eluted using a solution of wet heptane and propan-2-ol. Detection is by fluorescence with excitation wavelengths of 280-290nm and emission wavelengths of 320-330nm.

##### *Relevant standards*

ISO 9936 (in draft) <sup>(8)</sup>

IUPAC method 2.432 <sup>(9)</sup>

## **Analysis of desmethylsterols**

### *Basis of procedure*

The oil is saponified and the sterols are extracted from the resulting soapstock. Separation of the sterols from the remainder of the unsaponifiable matter is achieved by TLC. The separated sterols are then derivatised (silylated) and determined by capillary column GLC with FID. An internal standard, such as betulin, is added to the sample of oil prior to saponification. This permits the calculation of the absolute concentration of individual desmethylsterols in the sample (mg/kg oil) and negates the need for recovery experiments.

### *Relevant standards*

BS 684: Part 2: Section 2.38 <sup>(10)</sup>

ISO 6799 <sup>(11)</sup>

## **Determination of iodine value (IV) by titration**

### *Basis of procedure*

The oil is dissolved in carbon tetrachloride and reacted with Wijs reagent. Potassium iodide solution and water are then added and the liberated iodine is titrated against sodium thiosulphate solution.

### *Relevant standards*

BS 684: Part 2: Section 2.13 <sup>(12)</sup>

ISO 3961 <sup>(13)</sup>

IUPAC Method No 2.205 <sup>(14)</sup>

Note - Unless it is deemed essential to accurately determine the IV by titration, estimation of the value should be made from fatty acid composition data. <sup>(15)</sup> This normally gives a value within one unit of that obtained by titration.

## **Stable isotope ratio analysis**

### *Basis of procedure*

The sample is burnt to form carbon dioxide which is purified by GLC and then analysed using mass spectrometry to determine the relative proportions of <sup>12</sup>C and <sup>13</sup>C. Isotopic compositions are presented as a ratio of the heavy isotope <sup>13</sup>C to <sup>12</sup>C measured as parts per thousand with respect to an international standard PDB (PeeDee Balamnite).

### *Relevant standards*

There are no standard protocols for the determination of the <sup>13</sup>C/<sup>12</sup>C isotope ratios of oils.

### **Determination of the triglyceride composition by carbon number using HPLC**

#### *Basis of procedure*

The oil is dissolved in a solution of chloroform and methanol and injected onto an HPLC system. Separation of the triglycerides is achieved by the reverse phase analytical column and analytes detected using a differential refractometer.

#### *Relevant standards*

This methodology has not been produced as a standard but has been used in oils and fats analysis for many years.

### **Determination of the triglyceride composition by carbon number using GLC**

#### *Basis of procedure*

After warming to completely liquefy the sample, the triglycerides are dissolved in chloroform. Separation of triglyceride groups having the same carbon number is then achieved by injecting the chloroform solution directly onto a packed or, more recently, a TAP (Triglyceride Analysis Profile) gas chromatograph column under temperature programmed conditions. FID is employed and identification of peaks is by reference to a standard triglyceride solution.

#### *Relevant standards*

IUPAC Method No 2.323 <sup>(16)</sup>

AOAC Method No 986.19 <sup>(17)</sup>

### **Determination of total *trans* fatty acids by infra-red spectroscopy**

#### *Basis of procedure*

Triglycerides or fatty acids are converted to the FAME for measurement of absorbance at 967/cm. The total isolated *trans* is calculated using a calibration curve of absorption versus *trans* unsaturation developed using a series of carbon disulphide (or bromoform) solutions containing different ratios of methyl elaidate and methyl oleate.

#### *Relevant standards*

IUPAC method 2.207 <sup>(18)</sup>



## Determination of total *trans* fatty acids by GLC

### *Basis of procedure*

Samples of oil are converted to FAME by the use of a standard procedure. After dilution with hexane or heptane, FAME are injected into a gas chromatograph and separated using a polar column. Each component peak is identified by comparison to a mixture of reference standards and the intensity of the peaks is used to quantify the amount of *trans* isomers present.

### *Relevant standards*

AOCS Official method Ce Ic-89<sup>(19)</sup>

## Measurement of slip melting point

### *Basis of procedure*

Fat is melted and then tempered at a particular temperature and time, the choice being dependent on the polymorphic nature of the fat. A prepared capillary tube containing a column of the fat is then immersed in a bath of water which is warmed at a specified rate until the melting point is reached. The slip melting point is the temperature at which the column of fat rises in an open capillary tube under the conditions laid down in relevant standards.

### *Relevant standards*

BS 684: Part 1: Section 1.3<sup>(20)</sup>

ISO 6321<sup>(21)</sup>

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## APPENDIX II

### PURITY CRITERIA FOR MAIZE, SUNFLOWER, GROUNDNUT AND PALM OILS

**Fatty acid composition,<sup>(1,2,3)</sup> iodine value,<sup>(1,2,3)</sup> slip melting point<sup>(4)</sup> and stable carbon isotope ratio<sup>(5)</sup> of authentic maize, sunflower, groundnut and palm oils**

Chemical parameter	Maize oil		Sunflower oil		Groundnut oil		Palm oil	
	Range	Mean	Range	Mean	Range	Mean	Range	Mean
Fatty acids (% total fatty acids)								
C12:0	nd - 0.3	0.1	nd	na	nd	na	nd - 0.2	0.1
C14:0	nd - 0.3	0.1	tr - 0.1	na	nd - 0.1	na	0.7 - 1.3	1.0
C16:0	9.2 - 16.5	11.8	5.6 - 7.6	6.5	8.3 - 14.0	11.4	40.1 - 46.3	44.8
C16:1	tr - 0.4	0.1	nd	na	nd - 0.1	na	nd - 0.3	tr
C18:0	tr - 3.3	2.2	2.7 - 6.5	4.5	1.9 - 4.4	3.3	4.0 - 6.5	4.8
C18:1	20.0 - 42.2	30.8	14.0 - 39.4	21.1	36.4 - 67.1	45.3	36.7 - 40.9	38.9
C18:2	39.4 - 65.6	52.8	48.3 - 74.0	66.2	14.0 - 43.0	32.5	9.4 - 12.1	10.6
C18:3	0.7 - 1.4	1.0	nd - 0.1	na	nd - 0.1	na	0.1 - 0.4	0.3
C20:0	0.3 - 0.7	0.5	0.2 - 0.4	0.3	1.1 - 1.7	1.4	0.1 - 0.7	0.3
C20:1	tr - 0.4	0.3	0.1 - 0.2	0.1	0.7 - 1.7	1.1	nd	na
C20:2	nd	na	nd	na	nd	na	nd	na
C22:0	nd - 0.5	0.2	0.5 - 1.3	0.8	2.1 - 4.4	3.3	nd	na
C22:1	nd	na	0.0 - 0.2	0.1	tr - 0.3	na	nd	na
C24:0	nd - 0.4	0.2	0.2 - 0.3	0.3	1.1 - 2.2	1.4	nd	na
C24:1	nd	na	nd	na	nd - 0.3	na	nd	na
Iodine value	107.4 - 134.9	120.6	117.8 - 140.8	133.0	85.5 - 107.1	96.1	50.3 - 55.0	52.5
Slip melting point (°C)	na	na	na	na	na	na	32.7 - 39.6	36.0
SCIR	-13.71 to -16.36	-14.95	-27.94 to -29.76	-28.95	-26.48 to -28.69	-27.87	-29.25 to -29.91	-29.64

Notes: na = not applicable  
 nd = not detected  
 tr = trace

## Desmethylsterol composition of authentic maize, sunflower, groundnut and palm oils <sup>(1,6)</sup>

Desmethylsterol (mg/kg oil)	Maize oil		Sunflower oil		Groundnut oil		Palm oil	
	Range	Mean <sup>a</sup>	Range	Mean	Range	Mean <sup>a</sup>	Range	Mean <sup>a</sup>
Cholesterol	20 - 100	51	7 - 44	17	nd - 40	22	12 - 27	18
Brassicasterol	nd - 30	6	nd -10 <sup>b</sup>	na	nd - 3	0.1	nd	na
Campesterol	1700 - 5300	2955	237 - 450	322	150 - 550	256	78 - 161	111
Stigmasterol	500 -1000	743	256 - 414	317	60 - 260	138	33 - 87	56
$\beta$ -sitosterol	5000 - 13000	8774	1489 - 2791	2029	520 - 1750	930	211 - 389	283
$\Delta$ -5-avenasterol	400 - 1800	786	nd - 219	118	80 - 360	189	nd - 15	9
$\Delta$ -7-stigmastenol	nd - 500	250	244 - 489	352	nd - 70	22	1 - 11	5
$\Delta$ -7-avenasterol	20 - 400	180	80 - 266	164	nd - 80	20	nd - 24	7

Notes: <sup>a</sup> The data included in this table on the mean concentrations of individual desmethylsterols in maize, groundnut and palm oil were provided by Leatherhead Food Research Association (personal communication).

<sup>b</sup> Leatherhead Food Research Association have updated the range for the brassicaterol content of pure sunflower oil since the original data were published (personal communication).

na = not applicable

nd = not detected

Tocol (tocopherol and tocotrienol) composition of authentic maize, sunflower, groundnut and palm oils <sup>(2,3)</sup>

Tocol (mg/kg oil)	Maize oil		Sunflower oil		Groundnut oil		Palm oil	
	Range	Mean	Range	Mean	Range	Mean	Range	Mean
α-Tocopherol	23 - 573	282	403 - 935	684	49 - 373	179	4 - 193	95
β-Tocopherol	nd - 356	54	nd - 45	19	nd - 41	8	nd - 234	8
γ-Tocopherol	268 - 2468	1033	nd - 34	8	88 - 389	205	nd - 526	27
δ-Tocopherol	23 - 75	54	nd - 7.0	0.5	nd - 22	9	nd - 123	3
α-Tocotrienol	nd - 239	49	nd	na	nd	na	4 - 336	136
β-Tocotrienol	nd - 52	8	nd	na	nd	na	nd	na
γ-Tocotrienol	nd - 450	161	nd	na	nd	na	14 - 710	302
δ-Tocotrienol	nd - 20	6	nd	na	nd	na	nd - 377	89
Total (including unknowns)	331 - 3716	32.6	447 - 1514	779	176 - 1291	444	141 - 1465	666

Notes: na = not applicable  
nd = not detected

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## APPENDIX III

## CALCULATIONS FOR THE DETERMINATION OF PERCENTAGE UNDECLARED OIL IN SURVEILLANCE SAMPLES

**Compositional parameters**

Calculations of the percentage undeclared oil were based on the concentration of a chemical marker (linolenic acid, brassicasterol or  $\delta$ -tocopherol) present in the surveillance samples.

The chemical marker originates both from the named oil and from the undeclared oil. Thus, in 100 parts of oil and given x percent marker in the undeclared oil:

$$\text{Marker conc} = \frac{(\text{conc. of marker in undeclared oil}) + (100-x)(\text{conc. of marker in pure named oil})}{100}$$

The equations were solved using the maximum concentration of marker found in the named oil (see Appendix II) as this represents the maximum potential contribution from this source. With regard to the concentration of marker in the undeclared oil, the mean value for the authentic product was employed as this represents the average likely contribution from this source to the total concentration of marker present.

The most likely adulterant of maize, sunflower and groundnut oils is rapeseed oil. In view of this, the percentage undeclared oil calculated from the linolenic acid content of surveillance samples was based on the assumption that rapeseed oil was present. The mean linolenic acid content of rapeseed oil is 10.2% of the total fatty acid content.<sup>(1)</sup>

Elevated levels of brassicasterol are indicative of the presence of rapeseed oil. Consequently, the percentage undeclared oil calculated using the analytical data obtained on the brassicasterol content of surveillance samples was based on the mean concentration of this sterol in rapeseed of 720mg/kg oil.<sup>(1)</sup>

Elevated levels of  $\delta$ -tocopherol are indicative of the presence of soyabean oil. Thus in this case, the percentage undeclared oil was calculated using the mean concentration of  $\delta$ -tocopherol in soyabean oil of 425mg/kg oil.<sup>(1)</sup>

**Stable carbon isotope ratio (SCIR)**

The SCIRs of authentic maize oils lie in the range -13.71 to -16.36.<sup>(2)</sup> Thus, for the purposes of the survey, any sample with a SCIR more negative than -16.36 was suspected of containing undeclared oil. The concentration of undeclared oil was established using the most negative SCIR observed for any vegetable oil (-32.39).<sup>(2)</sup>

Thus, if:  $x$  = maximum proportion of maize oil in the blend: and  
 $y$  = the minimum proportion of undeclared oil,  
 $x(-16.36) + y(-32.39) = \text{SCIR}$   
and  $x + y = 1$  or  $y = 1 - x$  or  $x = 1 - y$

The SCIR is derived experimentally and the simultaneous equations are solved.

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## APPENDIX IV

QUALITY CONTROL AND QUALITY ASSURANCE ANALYSIS  
UNDERTAKEN AS PART OF THE VEGETABLE OIL SURVEY

Control measures were undertaken by the laboratories participating in the survey. Typical results obtained are given below:

## 1 Fatty Acid Composition

*Fatty acid composition of 'blind' or 'unknown' replicates of a maize oil sample*

Table 1a shows the results of the analysis of four replicates of a maize oil sample which was obtained from a retail outlet. These samples were submitted 'blind' to each laboratory such that they were indistinguishable from surveillance samples. The range and mean are calculated from the replicate analyses reported by each laboratory. The results indicate that, within the limits of experimental error, there are no significant differences between laboratories.

Table 1a: Fatty Acid Composition of Retail Maize Oil Samples

Fatty acid	Percentage of total fatty acids							
	Laboratory 1		Laboratory 2		Laboratory 3		Laboratory 4	
	Range	Mean	Range	Mean	Range	Mean	Range	Mean
C12:0	nd	na	nd-tr	na	nd	na	nd	na
C14:0	tr	na	tr	na	nd-tr	na	tr	na
C16:0	8.6-9.7	9.2	9.2	9.2	9.2-9.6	9.5	9.2-9.3	9.2
C16:1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C18:0	1.7-1.8	1.8	1.8	1.8	1.9-2.1	2.0	1.8	1.8
C18:1	28.6-29.3	29.0	28.5-28.6	28.6	28.8-29.3	29.1	28.5-28.7	28.6
C18:2 n-6	56.7-57.8	57.1	56.9-57.1	57.0	55.7-56.4	56.1	56.2-56.5	56.4
C18:2 iso	nd	na	0.1-0.2	0.1	0.2-0.3	0.2	nd	na
C18:3 n-3c	1.7-1.8	1.7	1.7-1.8	1.8	1.8-1.9	1.8	1.9	1.9
C18:3 iso	0.1	0.1	0.1	0.1	0.1	0.1	nd	na
C20:0	0.4	0.4	0.4	0.4	0.4-0.5	0.4	0.4-0.5	0.4
C20:1	0.4	0.4	0.4	0.4	0.2-0.3	0.2	0.4-0.5	0.4
C22:0	tr-0.2	0.1	0.2	0.2	0.1-0.2	0.2	0.2	0.2
C22:1	tr	na	tr-0.1	0.1	nd-tr	na	nd	na
C24:0	tr	na	0.1	0.1	nd-0.1	0.1	nd	na
C24:1	nd	na	tr-0.1	tr	nd	na	nd	na

Notes: na = not applicable  
nd = not detected  
tr = trace

**Table 1b: Fatty acid composition of maize 'reference' oil sample**

Fatty acid	Percentage of total fatty acids							
	Laboratory 1		Laboratory 2		Laboratory 3		Laboratory 4	
	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2
C12:0	nd	nd	nd	nd	nd	nd	nd	nd
C14:0	tr	tr	tr	tr	nd	nd	0.1	tr
C16:0	8.8	9.2	9.2	9.2	9.8	9.8	9.2	9.2
C16:1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C18:0	1.8	1.7	1.8	1.8	2.0	2.1	1.8	1.8
C18:1	29.0	28.9	28.6	28.5	28.9	29.2	28.7	28.5
C18:2 n-6	57.4	57.4	57.0	56.9	56.4	56.1	55.8	56.2
C18:2 iso	nd	nd	0.2	0.2	0.1	0.1	nd	nd
C18:3 n-3c	1.7	1.7	1.8	1.8	1.9	1.8	2.0	1.9
C18:3 iso	0.1	0.1	0.1	0.1	nd	nd	nd	nd
C20:0	0.4	0.4	0.4	0.4	0.5	0.4	0.5	0.5
C20:1	0.4	0.4	0.4	0.4	0.2	0.3	0.4	0.4
C22:0	0.1	tr	0.2	0.2	0.1	0.1	0.2	0.2
C22:1	tr	tr	0.1	0.1	nd	nd	nd	nd
C24:0	tr	tr	0.1	0.1	nd	nd	0.1	0.1
C24:1	nd	nd	nd	0.1	nd	nd	nd	nd

**Table 1c: Fatty acid composition of rapeseed 'reference' oil sample**

Fatty acid	Percentage of total fatty acids							
	Laboratory 1		Laboratory 2		Laboratory 3		Laboratory 4	
	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2
C12:0	nd	nd	tr	tr	nd	nd	tr	tr
C14:0	tr	tr	tr	0.1	tr	tr	0.1	0.1
C16:0	4.8	4.4	4.6	4.7	4.9	4.9	4.5	4.6
C16:1	0.2	0.2	0.3	0.3	0.2	0.3	0.2	0.2
C18:0	1.5	1.5	1.5	1.5	1.7	1.7	1.5	1.5
C18:1	58.8	59.1	57.6	57.9	58.4	58.0	57.1	57.4
C18:2 n-6	21.2	21.4	21.8	21.4	21.8	21.8	21.7	21.7
C18:2 iso	nd	nd	0.1	0.1	nd	nd	nd	nd
C18:3 n-3c	9.9	9.9	10.2	10.3	10.4	10.4	10.7	10.6
C18:3 iso	0.8	0.8	0.8	0.8	0.3	0.3	nd	nd
C20:0	0.5	0.5	0.6	0.6	0.6	0.6	0.6	0.6
C20:1	1.4	1.3	1.3	1.3	1.1	1.1	1.4	1.4
C22:0	0.3	0.3	0.4	0.4	0.3	0.2	0.3	0.4
C22:1	0.3	0.2	0.2	0.3	0.2	0.2	nd	nd
C24:0	tr	tr	0.1	0.1	nd	nd	0.1	0.2
C24:1	tr	tr	0.2	0.3	nd	nd	nd	nd

Notes: nd = not detected  
tr = trace

**Table 1d: Fatty acid composition of soyabean 'reference' oil sample**

Fatty acid	Percentage of total fatty acids							
	Laboratory 1		Laboratory 2		Laboratory 3		Laboratory 4	
	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2
C12:0	nd	nd	nd	nd	nd	nd	nd	tr
C14:0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C16:0	10.5	10.6	10.6	10.6	10.9	11.0	10.6	10.6
C16:1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C18:0	3.3	3.4	3.3	3.3	3.7	3.6	3.4	3.4
C18:1	26.7	27.2	26.7	26.7	28.2	28.2	26.8	26.7
C18:2 n-6	52.1	51.5	51.6	51.5	49.8	49.6	50.7	50.8
C18:2 iso	nd	nd	0.1	0.1	0.2	0.2	nd	nd
C18:3 n-3c	5.5	5.3	5.5	5.5	5.5	5.7	5.8	5.8
C18:3 iso	0.6	0.6	0.5	0.5	0.2	0.2	nd	nd
C20:0	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
C20:1	0.4	0.3	0.3	0.3	0.2	0.2	0.3	0.3
C22:0	0.4	0.4	0.5	0.5	0.5	0.5	0.4	0.5
C22:1	tr	tr	nd	nd	nd	nd	nd	nd
C24:0	tr	tr	0.2	0.2	nd	nd	0.2	0.1
C24:1	nd	nd	nd	0.1	nd	nd	nd	nd

**Table 1e: Fatty acid composition of sunflowerseed 'reference' oil sample**

Fatty acid	Percentage of total fatty acids							
	Laboratory 1		Laboratory 2		Laboratory 3		Laboratory 4	
	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2	Dup. 1	Dup. 2
C12:0	nd	nd	nd	nd	nd	nd	nd	nd
C14:0	tr	tr	0.1	0.1	0.1	0.1	0.1	0.1
C16:0	5.6	5.9	6.0	6.2	6.4	6.5	6.1	6.1
C16:1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C18:0	4.0	4.0	3.9	4.0	4.3	4.3	4.1	4.0
C18:1	23.4	23.4	23.1	23.2	23.9	23.9	23.3	23.4
C18:2 n-6	65.7	65.2	64.3	64.3	63.0	62.9	63.8	63.5
C18:2 iso	nd	nd	0.5	0.5	0.8	0.8	nd	nd
C18:3 n-3c	0.2	0.2	0.2	0.2	0.2	0.3	0.3	0.3
C18:3 iso	tr	0.1	nd	nd	nd	nd	nd	nd
C20:0	0.3	0.3	0.3	0.3	0.3	0.3	0.1	0.1
C20:1	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
C22:0	0.5	0.6	0.6	0.7	0.7	0.7	0.7	0.6
C22:1	tr	tr	nd	nd	nd	nd	nd	nd
C24:0	tr	tr	0.2	0.2	nd	nd	0.2	0.2
C24:1	nd	nd	0.3	nd	nd	nd	nd	nd

Notes: nd = not detected

tr = trace

*Fatty acid composition of 'reference' oil samples*

The results of duplicate analysis of known 'reference' oils which were obtained from retail outlets are presented in Tables 1b to 1e. The results indicate that, within the limits of experimental error, there were no significant differences between laboratories.

*In-house quality control procedures*

In addition, each laboratory carried out its own in-house quality control procedures. These included the replicate analysis of in-house control material or of certified reference material. Each laboratory exhibited acceptable precision and agreement with certified values.

**2. Stable carbon isotope ratio analysis**

*Comparison of  $^{13}\text{C}/^{12}\text{C}$  stable isotopic results with Leatherhead Food RA data*

$^{13}\text{C}/^{12}\text{C}$  stable isotopic analysis was carried out on a reference sample which had previously been analysed by Leatherhead Food Research Association when establishing the purity criteria with which surveillance samples were being compared. There was a minor difference in the average value between the two laboratories as illustrated in Table 2a:

**Table 2a: SCIR of Reference Maize Oil Sample**

Laboratory	No of replicates	Maximum	Minimum	Mean	Standard deviation
LFRA	24	-16.02	-15.60	-15.84	0.07
Survey lab	32	-16.30	-15.65	-15.99	0.15

*SCIR of 'blind' or 'unknown' replicates of a maize oil sample*

The laboratory conducting the SCIR work also analysed four replicates of a maize oil sample obtained from a retail outlet. The samples were submitted to the laboratory 'blind' such that they were indistinguishable from the surveillance samples. The results obtained are detailed in Table 2b and indicate sufficient repeatability was achieved.

**Table 2b: SCIR of Retail Maize Oil Sample**

Replicate	SCIR
1	-17.1
2	-17.1
3	-17.1
4	-16.7

### 3. Desmethylsterol analysis

*Desmethylsterol composition of 'blind' or 'unknown' replicates of a maize oil sample*

The two laboratories reporting desmethylsterol results were each supplied with six identical samples of maize oil purchased at a local retail outlet. These oils were indistinguishable from the surveillance samples. The analysts were asked to determine the desmethylsterol composition and concentration of these oils at regular intervals during the analyses of the surveillance oils. The results of the individual analyses of these samples are shown in tables 3a and 3b.

**Table 3a**

#### **Desmethylsterol concentration in QA samples - Laboratory 1**

Desmethylsterol	Desmethylsterol content (mg/kg oil)					
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Cholesterol	22	16	20	16	23	23
Brassicasterol	4	4	4	5	5	7
Campesterol	1436	1440	1405	1148	1646	1711
Campestanol	84	64	63	68	141	117
Stigmasterol	486	481	470	383	574	593
$\Delta$ -7-Campesterol	19	21	19	6	22	15
$\Delta$ -5,23-Stigmastadienol	38	34	33	nd	24	52
Chlerosterol	23	20	18	nd	21	31
$\beta$ -Sitosterol	4375	4192	4269	3938	5149	5328
Sitostanol	232	232	225	238	330	305
$\Delta$ -5-Avenasterol	280	314	292	319	331	387
$\Delta$ -5,24-Stigmastadienol	21	25	24	40	23	42
$\Delta$ -7-Stigmastenol	44	41	49	49	33	28
$\Delta$ -7-Avenasterol	101	78	96	100	67	84
Total	7164	6961	6984	6308	8388	8722

Notes: nd = not detected

#### *In-house quality control procedures*

In addition, each laboratory carried out its own in-house quality control procedures. These were based on the replicate analysis of in-house control material. Acceptable results were obtained.

**Table 3b**  
**Desmethylsterol concentration in QA Samples -Laboratory 2**

Desmethylsterol	Desmethylsterol content (mg/kg oil)					
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Cholesterol	19	25	32	17	15	24
Brassicasterol	nd	nd	nd	nd	nd	nd
Campesterol	1241	1445	1302	1364	1226	1433
Campestanol	129	133	128	143	126	111
Stigmasterol	448	530	490	502	451	531
$\Delta$ -7-Campesterol	16	13	6	12	16	nd
$\Delta$ -5,23-Stigmastadienol	nd	nd	nd	nd	nd	nd
Chlerosterol	52	59	54	50	46	63
$\beta$ -Sitosterol	4025	4848	4366	4515	4060	4842
Sitostanol	263	253	265	311	269	253
$\Delta$ -5-Avenasterol	230	340	322	281	297	280
$\Delta$ -5,24-Stigmastadienol	58	15	7	46	20	50
$\Delta$ -7-Stigmastenol	31	34	33	31	33	29
$\Delta$ -7-Avenasterol	54	64	50	68	65	52
Total	6566	7759	7055	7340	6624	7668

#### 4. Tocopherol analysis

The laboratory conducting this work was asked to determine the tocopherol composition of three identical reference samples of maize oil (purchased from a retail outlet). The results are given in Table 4 and are acceptable for precision.

**Table 4**  
**Tocol Composition of QA sample.**

Tocol	Concentration of tocol (mg/kg oil)		
	Sample 1	Sample 2	Sample 3
$\alpha$ -Tocopherol	69	92	73
$\alpha$ -Tocotrienol	nd	nd	nd
$\beta$ -Tocopherol	10	16	14
$\beta$ -Tocotrienol	nd	nd	nd
$\gamma$ -Tocopherol	190	198	217
$\gamma$ -Tocotrienol	3	3	8
$\delta$ -Tocopherol	10	12	12
$\delta$ -Tocotrienol	nd	nd	nd
Total	282	321	323

Notes: nd = not detected

## THE FAT CONTENT OF MINCED BEEF

### Introduction

Until recently there were no statutory standards for the fat content of minced beef and similarly named foods. It was generally accepted by the courts that a fat content of 25 % was an allowable maximum for minced beef and that when the fat content exceeded this amount the sample was not of the quality demanded. During 1994/1995 the Association of Public Analysts reviewed data on the fat content of routine minced beef samples analysed for enforcement purposes since 1990 and compared the data with previous data. The findings of the review and a proposal of a guidelevel of 20% were published in 1995<sup>(1)</sup>. Subsequently, The Minced Meat and Meat Preparations (Hygiene) Regulations 1995 (the Regulations)<sup>(2)</sup> were issued. Later, Guidance Notes on the Enforcement of the Regulations<sup>(3)</sup> were also published. As a result, the review and its conclusions have been reconsidered. The review also included details of previously published work relating to minced beef which had been misquoted. The details of corrigenda, none of which effect the overall case for a guidelevel, are given.

### The Minced Meat and Meat Preparations (Hygiene) Regulations 1995

Schedule 11 of the Regulations lists certain compositional criteria for minced meats. In particular for 'lean minced' used in relation to meat of any permitted species a fat content of not more than 7% is set down and for 'minced pure' used in relation to meat of bovine animals (this includes beef) a fat content of not more than 20% is set down. Regulations 7(1)(0) and 7(2)(e) restrict these standards to the designations specified in the Regulations.

### The Guidance Notes

Paragraph 96 of these notes explains that "an occasional sample which exceeds the requirement for either fat content ... should not jeopardise the consignment. These should be regarded as an indication that the production process needs examining. The authorised officer should place emphasis on the overall daily or periodic averages and not individual re suits ".

Paragraph 97 explains that these compositional criteria only apply where the "exact wording (designations)" are used and "that the intention of the Directive was that the word 'pure' should indicate particular quality rather than production from a single species". There is therefore scope for great confusion and misunderstanding in the minds of all, i.e. consumers, producers and enforcement officials concerning the names and standards of fat content to be associated with these names. For example minced pure beef could be a different product from minced beef; lean minced beef could be a different product from lean ground beef.

### **Definition of Minced Beef**

The Regulations define "minced meat" but not "minced beef" except insofar as it is possible to deduce from the Regulations a definition for "minced pure beef". The definition of "minced meat" varies depending on whether the minced meat is for the market in Great Britain or for export to a relevant EEA State. As far as differentiating between "minced meats" and "meat preparations" the amount of added salt is also a factor. "Minced meat" may be prepared from meat "to which not more than 1 per cent salt has been added" whereas "meat preparations" may contain higher levels of added salt. The Food Labelling Regulations 1996 would require minced meat with added salt to be named so as to inform the purchaser of the true nature of the food and to enable the food to be distinguished from similar products with which it could be confused.

The generally accepted meaning of "beef" is meat from the skeletal muscle of an appropriate full-grown bovine animal i.e. it does not include meat such as heart. The Regulation makes this clear for minced beef produced for export to a relevant EEA State. The only general definition relating to minced beef that can be distilled from the Regulations and Guidance Notes is as follows:

Minced pure beef is the striated muscle (other than heart muscle), including the associated fatty tissues, from an appropriate bovine animal which has been minced into fragments or passed through a spiral screw mincer and has, on average, a fat content which does not exceed 20% and a collagen content in meat protein of not more than 15%.

The confusion arising from the interpretation of the word "pure" in paragraph 97 of the Guidance Notes points to the need for a means of controlling standards when the designations are other than "minced/pure beef". The standard dictionary definition of "pure" is "unmixed, unadulterated" and would indicate that the above definition of "minced pure beef" should be the definition for "minced beef" simpliciter. However, while the confusion exists the guideline which the Association of Public Analysts proposed is relevant.

### **APA Guidelevel**

The APA proposed a fat content guideline of 20% for all other designations of minced beef, other than those qualified as 'lean'. The argument for the guideline could not be based on strict statistical analysis of the natural variation of the raw material and normal fat levels in products, because much of the minced beef consumed today is manufactured to particular specifications. However, the review did show that an average fat content of just below 16% was currently provided on the market. A view was also expressed that a maximum positive variation of 25 % of the average was reasonable for a major



constituent, given that there is such a high degree of manufacturing control. This equates to 20% fat and coincidentally equals the regulatory standard for the fat content of minced pure beef.

### Analytical Data

The details of the corrigenda in the 1995 APA paper are:-

1. The data in Table II related to partially trimmed beef. There was no indication of this fact.
2. Tables IIIA and IIIB relating to "beef cuts-lean tissue" and "beef cuts - lean and intermuscular tissue" included columns headed "Standard Deviation" but which should have been headed "Standard Error".
3. The figures which followed Tables IV and V were graphical representations of the data in Table V and should have been appropriately referenced. The tables and figures are reproduced below as Table I and Table II and Chart 1 and Chart 2.

TABLE I - The fat content of enforcement samples of minced beef - Statistics

Reproduced from Table IV (J. Assoc. Publ. Analysts 1995, 31, 118)

	Up to 1989	1990 - 1994	All
Number of samples	508	1307	1815
Mean	16.47	15.73	15.93
Median	16.2	15.5	15.8
Sample SD	5.97	5.84	5.88
SKEWNESS	0.31	0.29	
Ftest	0.54		
S.E.	0.26	0.16	

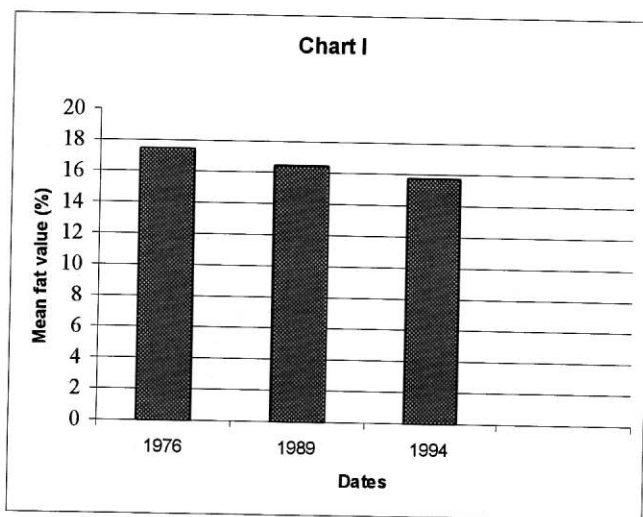
The Association of Public Analysts

TABLE II - The fat content of enforcement samples of minced beef -Rankings

Reproduced from Table V (J. Assoc. Publ. Analysts 1995, 31,119)

Pre 1990			Post 1990		
Range	Frequency	% of total	Range	Frequency	% of total
1	0	0	1	0	0
2	2	0.39	2	0	0
3	1	0.20	3	1	0.08
4	0	0	4	10	0.77
5	4	0.79	5	18	1.38
6	8	1.57	6	22	1.68
7	13	2.56	7	35	2.68
8	11	2.17	8	49	3.75
9	17	3.35	9	42	3.22
10	25	4.92	10	47	3.60
11	20	3.94	11	78	5.97
12	21	4.13	12	70	5.36
13	21	4.13	13	73	5.59
14	32	6.30	14	92	7.04
15	35	6.89	15	75	5.74
16	39	7.68	16	77	5.90
17	35	6.89	17	91	6.97
18	31	6.10	18	81	6.20
19	27	5.31	19	76	5.82
20	35	6.89	20	75	5.74
21	26	5.12	21	59	4.52
22	18	3.54	22	50	3.83
23	16	3.15	23	41	3.14
24	17	3.35	24	39	2.99
25	14	2.76	25	42	3.22
26	12	2.36	26	12	0.92
27	8	1.57	27	11	0.84
28	4	0.79	28	8	0.61
29	4	0.79	29	14	1.07
30	3	0.59	30	8	0.61
31	2	0.39	31	3	0.23
32	2	0.39	32	2	0.15
33	2	0.39	33	1	0.08
34	1	0.20	34	1	0.08
35	1	0.20	35	1	0.08
36	0	0	36	1	0.08
37	0	0	37	1	0.08
38	0	0	38	0	0
39	0	0	39	0	0
40	1	0.20	40	0	0
>40	0		>40	1	
	508	100		1306	100

The mean fat content of enforcement samples of minced beef  
(Table II)

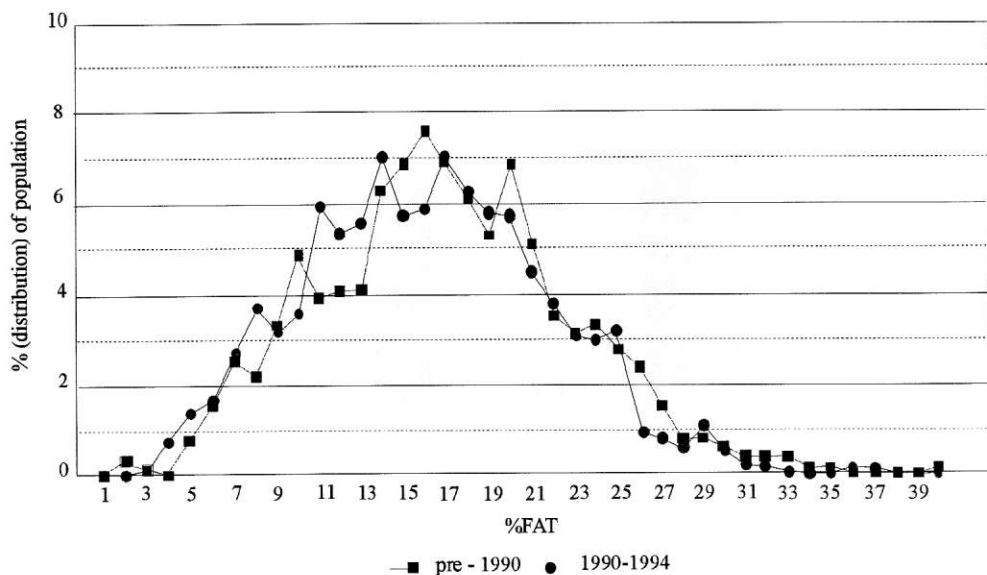


Date	1976	1989	1994
Number of samples	1324	508	1307
Mean Fat Values (%)	17.5	16.47	15.73

Chart 2

Comparative distribution curves (Table II - ranking).

FAT - distribution of minced meat fat contents  
Normalised to equivalent populations



### Conclusion

Where fat contents in excess of 20% are encountered such findings should trigger further investigation, which may include sampling and analysis, prior to the consideration of enforcement action. The absolute maximum of 25 % fat would remain for minced meats not covered by the Regulations.

### References

- (1) Fat in Minced Beef. A Review by the Association of Public Analysts. J. Assoc. Publ. Analysts 1995, 31,113-120.
- (2) The Minced Meat and Meat Preparations (Hygiene) Regulations 1995. Statutory Instrument 1995 No.3205. HMSO.
- (3) Guidance Notes on the Enforcement of the Minced Meat and Meat Preparations (Hygiene) Regulations 1995. Ministry of Agriculture, Fisheries and Food, Department of Health, Scottish Office, Welsh Office.

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Note (1) The MAFF Validated Methods  
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