

Good Practice Guide on Compliance with the Legislation on Irradiation of Food Ingredients particularly for Food Supplements



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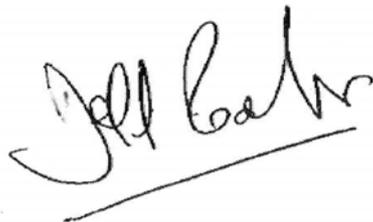
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FOREWORD

While food irradiation is considered a safe and effective processing technique, legislative controls are in place to ensure it is used appropriately and consumers are given the information they need to make an informed choice. I am delighted that the food supplements industry has recognised how important it is that irradiated ingredients in their products are declared, and is acting on this.

This guide will be an important reference document for both the industry and the enforcers. It clearly sets out the responsibilities of food business operators to produce safe food and will help protect consumers from the risks associated with irradiated food ingredients and food fraud.

The guide has been produced with the input of the Food Irradiation Stakeholder Group, which comprises stakeholders interested in the technical and regulatory aspects of food irradiation in the food supplements industry, who wish to share information, promote better understanding and build consensus. We hope it will help businesses build on existing good practice, undertake the correct testing for irradiated food ingredients and comply with relevant legislation.

A handwritten signature in black ink, appearing to read "Jeff Rooker", with a horizontal line underneath it.

Jeff Rooker
Chair, Food Standards Agency
May 2010

1. INTRODUCTION

Although there has been legislation in the United Kingdom for over forty years controlling the irradiation of food and food ingredients, there have been continuing problems with illegally irradiated products entering the market. A significant proportion of these products have been found to be food supplements.^{1, 2, 3}

Developments in the detection of irradiated foods and ingredients have resulted in testing becoming more readily available and have provided tools for official surveillance and enforcement. Following a number of meetings between the supplement industry and the United Kingdom Regulators it was agreed that the interested parties would work together to produce a Good Practice Guide on compliance with the legislation on the irradiation of food ingredients, particularly those used in food supplements.

2 BACKGROUND

The preservation of foods by ionising radiation was first investigated in the early 1950's and by 1972 research into food irradiation was being carried out in 55 countries.

The first laws on food irradiation in the United Kingdom came into force in 1967. These were replaced in 1990 by 'The Food (Control of Irradiation) Regulations (S.I.1990/2490)'. The 1990 regulations permitted seven categories of food to be irradiated with maximum doses of ionising radiation assigned to each category. The regulations also stated that irradiation of food could only be carried out if a current irradiation licence was held and the treatment was carried out according to the conditions of that licence. Imported food treated by ionising irradiation was prohibited unless the treatment and documentation met a number of stringent conditions.

In 1999 the European Union (EU) finally adopted two directives on the irradiation of food, which brought in Community-wide legislation. The United Kingdom regulations were amended in 2000 to incorporate the requirements of the EU legislation.

On 31st July 2009 new regulations came into force as The Food Irradiation (England) Regulations 2009 (SI 2009/1584). Parallel legislation also came into force in Scotland, Wales and Northern Ireland. These Regulations clarify that it is the European Commission which has the role of approving food irradiation facilities in non-EU countries and introduce a general update of the 1990 regulations, including the removal of inspection and approval fees for licensed food irradiation facilities. In October 2010, the Food Irradiation (England) (Amendment) Regulations 2010 will come into force which will update the lists of approved food irradiation facilities

2.1 Issues and Problems Relating to the Irradiation of Foods

Whilst legislation on the control of irradiation of foods has been in force in the UK for a very long time, there have been a large number of issues relating to non-compliance with the regulations in force. For many years a number of problems have been related to the illegal irradiation of ingredients in products, particularly food supplements, originating from non-EU countries. The evidence appears to indicate that it is individual ingredients

that have been irradiated rather than the final product. Irradiated ingredients that have been detected are predominantly botanical in origin (herbs) but other ingredients such as talc, yeast and beeswax have also been found to be irradiated. The supplements and products with irradiated ingredients appear to have had the irradiated components treated in unauthorised plants in countries outside the EU.

The legislation allows the use of irradiation for certain named herbs and spices. However, all the requirements of the legislation have to be met, including labelling on the containers of the herb or spice, and any products containing it, that the ingredient has been irradiated. In theory, a food supplement containing a herb or spice on the permitted list, which has been irradiated in accordance with the requirements of the legislation, could be legally placed on the market provided that the label of the product contained the appropriate statement.

One of the earlier problems with irradiated ingredients was the inability to rapidly test for possible irradiation. As a consequence, companies knowingly supplying irradiated materials believed themselves to be immune from investigation and their customers had no cheap and easy way of checking. The situation changed dramatically with the commercial availability of instruments for the detection of irradiated food by photostimulated luminescence (PSL). This technique provided a rapid, non-destructive screening of foods and ingredients and in the early days of its use facilitated the detection of a number of irradiated herbs, spices and food supplements. Whilst the PSL method has some limitations, it is now widely used by both industry and enforcement authorities as an initial screening method for irradiation.

In addition to the commercial aspects of irradiation there has for a long time been an antipathy amongst retailers and the general population in parts of Europe and the UK towards the irradiation of foods as a preservation method. The negative public opinion is attributed to several factors. These include negative associations with radioactivity and the nuclear industry, the misplaced belief that irradiation renders the food radioactive, concern about possible loss of nutritional value and concerns surrounding the potential to “recover” foods that would otherwise be unacceptable on microbiological grounds. The combination of negative public opinion and the reluctance of major retailers to offer irradiated foods, has halted the progress of irradiation in many European countries, including the UK.

A further aspect, which is particularly relevant to the food supplement and health food industries, is the requirement to label legally irradiated foods as having been irradiated or treated with ionising radiation. In view of the negative public opinion on irradiation the labelling of foods as having been irradiated has not generally been considered a viable option in the UK.

2.2 Responsibilities of a Manufacturer

A supplier, manufacturer or importer of food products has a number of legal obligations, many of which are contained in the Food Safety Act 1990 as amended and legislation made under provisions therein.

The Food Safety Act and food irradiation legislation contains 'strict liability' law, this means that there is no requirement to show the 'guilty mind' (*mens rea*) of the person or business accused. The concept was introduced to allow action to be taken in situations such as those where a person or business may be negligent in allowing the circumstances of an offence to arise but may have no actual knowledge of the occurrence prior to the event.

Under strict liability law a defence is generally open to the accused if they can show that they have applied *all due diligence* and *have taken all reasonable care* to ensure an offence would not be committed.

In relation to most of the offences relating to food irradiation it is also necessary to show:

- That the offence was due to an act or default of another person outside their control, or to reliance on information supplied by such a person. and
- That all such checks have been carried out on the food as were reasonable in all the circumstances, or that it was reasonable in all the circumstances for them to rely on checks carried out by the supplier of the food. and
- The defendant would also have to prove that they did not know, and could not reasonably have been expected to know, at the time of the commission of the alleged offence that their act or omission would amount to an offence.

From the above it can be seen that every supplier, manufacturer or importer of foods that could possibly be irradiated must be able to demonstrate that they have applied all due diligence and taken all reasonable care to ensure that unauthorised irradiated products do not enter the market. To achieve this, a detailed knowledge of the product or ingredient is required together with evidence that all reasonable enquiries have been made to each of the suppliers and reliable responses received on the status of each ingredient with regard to irradiation. Legislation is already in force regarding traceability and documentation of traceability and these should be adhered to by all food operators in the UK. In theory, if importers and suppliers of products and ingredients carried out the basic requirements of due diligence and traceability, illegally irradiated products should not be entering the UK market.

The purpose of this Good Practice Guide is to bring together in one document all the information needed by a manufacturer, supplier or distributor to ensure compliance with the legislation. The document not only covers the legal obligations but also provides advice on due diligence, sampling and testing.

3. LEGISLATION

3.1 Introduction

The purpose of this section is to summarise the laws relating to the irradiation of food and food ingredients and indicate where further guidance and information is available.

The following information is intended for food businesses that manufacture and supply food supplements and their ingredients within the United Kingdom. As described below, the laws relating to food irradiation are not fully harmonized across Europe. While this section contains some narrative on the legal position across the European Union, it is the responsibility of any food business to make sure that they comply with the legislation in place in any country they supply to or operate in.

The information in this section is provided for guidance only. Ultimately, it would be for a court to determine if there has been a breach of the law.

3.2 Specific Food Irradiation Legislation

Food irradiation is controlled in England by The Food Irradiation (England) Regulations 2009 (SI 2009/1584), which were made under the Food Safety Act 1990 – similar legislation applies in Scotland⁴, Wales⁵ and Northern Ireland⁶ and will be referred to collectively in this guide as the Food Irradiation Regulations 2009. The Food Irradiation Regulations 2009 update and consolidate previous Regulations.

The sale (and import, storage or transport for the purpose of sale) of irradiated food or food containing irradiated ingredients contrary to the provisions of the Food Irradiation Regulations 2009 is an offence under regulation 10 these Regulations. For this reason, appropriate action should be taken to remove any foods from sale which fail to comply with these Regulations.

The Food Irradiation Regulations 2009 (and relevant sections of the Food Labelling Regulations 1996, as amended) implement two European Directives on food irradiation, Directive 1999/2/EC and Directive 1999/3/EC`.

Directive 1999/2/EC establishes a harmonised framework of controls on food irradiation within the EU. It controls the sale and import of irradiated food and requires that food can only be irradiated in approved food irradiation facilities. It also requires that all irradiated food, and any foods containing irradiated ingredients, must be appropriately labelled.

Directive 1999/3/EC implements an initial positive list of foods which may be irradiated and traded across the EU.

Links to the original texts of the legislation mentioned above are available through the Food Standards Agency website:

http://www.food.gov.uk/foodindustry/imports/imports_advice/irradiated

3.3 Exemptions

The Food Irradiation Regulations 2009 exempt a number of practices from the controls of the regulations. These include the use of scanning inspection devices (for example at ports) and the irradiation of food prepared under medical supervision for patients requiring sterile diets.

Another important exemption is that the Food Irradiation Regulations 2009 are made under the Food Safety Act and as such only cover food and do not cover products

classified as medicines. Many medicines and medicinal ingredients are routinely irradiated and these can sometimes find their way into the food supply chain where they may not be permitted. The classification of a product as either a food supplement or medicinal product is not always clear; the final decision lies with the Medicines and Healthcare products Regulatory Agency (MHRA). Guidance on the definition of medicines is provided in 'A Guide to What is a Medicinal Product' which is available on the MHRA website:

<http://www.mhra.gov.uk/home/groups/comms-ic/documents/publication/con007544.pdf>

3.4 Permitted food categories

The Food Irradiation Regulations 2009 lists seven categories of food which may be irradiated and sold in the UK. The seven categories, and the maximum overall dose to which they may be treated (in kilogray (kGy)), are:

- (i) fruit, 2 kGy;
- (ii) vegetables, 1 kGy;
- (iii) cereals, 1 kGy;
- (iv) bulbs and tubers, 0.2 kGy;
- (v) dried aromatic herbs, spices and vegetable seasonings, 10 kGy;
- (vi) fish and shellfish, 3 kGy; and
- (vii) poultry, 7 kGy,

Although the framework for irradiated foods is harmonised across the EU, the categories of food which may be treated and sold is not. Directive 1999/3/EC introduced an **initial** positive list of foods which may be irradiated across the EU. This contains only a single category, dried aromatic herbs, spices and vegetable seasonings. It has always been the intention to produce a final positive list, but for a number of reasons this has yet to happen.

As a temporary measure, until the positive list is finalised, Directive 1999/2/EC allows member states to maintain their existing authorisations of foods they permit to be irradiated and maintain existing national restrictions or bans on trade in irradiated foodstuffs which are not included in the initial positive list. Several member states only permit the single category on the initial positive list. Other member states maintain additional categories, such as the six additional categories in the UK, but these vary between different member states. Examples of irradiated foods which are permitted in some other member states but are not permitted in the UK are frozen frogs legs, gum arabic, dehydrated blood, egg whites and casein.

Further information regarding the irradiation of foods in the European Union is available on the European Commission website:

http://ec.europa.eu/food/food/biosafety/irradiation/comm_legisl_en.htm

3.5 Importing irradiated food and ingredients into the UK

In the UK, local authorities and port health authorities are responsible for controlling imports of irradiated foods, including appropriate testing. Certain foods that have been irradiated may be imported into the UK, as long as they comply with certain rules. Any irradiated food or food ingredients which have not been imported in accordance with the Food Irradiation Regulations 2009 cannot be sold in the UK.

Guidance on the import of irradiated foods from both EU and non-EU countries is available on the Food Standards Agency website:

http://www.food.gov.uk/foodindustry/imports/imports_advice/irradiated

3.6 Storage and transport of irradiated food and ingredients

The Food Irradiation Regulations specify the documentation which must accompany irradiated food and food containing irradiated ingredients being stored and transported to enable traceability back to the irradiation treatment. The documentation must include an indication that the food has been irradiated, the name and address or reference number of the approved food irradiation facility, the batch number and the date of treatment.

3.7 Labelling of irradiated food and irradiated food ingredients

The description of irradiated foods in the supply chain but not yet ready for sale to the consumer is regulated by The Food Irradiation Regulations 2009 in so far as it must be accompanied by documentation which describes product using the words 'irradiated' or 'treated with ionising irradiation'.

The labelling of irradiated foods on sale to consumers is covered by The Food Labelling Regulations 1996 as amended. This specifies that irradiated food must be labelled with the words "irradiated" or "treated with ionising radiation". When irradiated food is not pre-packed and is sold for immediate consumption (for example, in restaurants) it must be marked or labelled on a menu, notice or ticket that the consumer can see when choosing the food.

Any food containing an irradiated ingredient must contain the words "irradiated" or "treated with ionising radiation" next to the ingredient's name in the list of ingredients. This applies to any quantity, no matter how small, of irradiated ingredient and still applies even if the ingredient would otherwise be exempt from inclusion on the list of ingredients.

Further Information can be found in the 'Food Labelling Regulations Guidance Notes', which is available along with other food labelling guidance on the Food Standards Agency website: <http://www.food.gov.uk/foodindustry/guidancenotes/labelregsguidance/>

3.8 General Food Law

The Food Safety Act 1990 (as amended) provides the framework for all food legislation in Great Britain – similar legislation applies in Northern Ireland, its provisions include:

- Offences of rendering food unsafe, selling food not of the nature, substance or quality demanded by the purchaser to the purchaser's prejudice and of misdescribing foodstuffs;
- The enforcement of food safety legislation including powers for authorised officers to enter premises and sample foodstuffs within the supply chain.

- A 'due diligence' defence (see Section 4 of this guide) and provision for persons to be prosecuted when they cause others to commit offences.

Further information can be found in 'The Food Safety Act 1990 – A Guide for Food Businesses', which is available with other guidance on general food law on the Food Standards Agency website: <http://www.food.gov.uk/foodindustry/regulation/foodlaw/>

The General Food Regulations 2004 (as amended) which apply in Great Britain, provide for the enforcement of certain provisions of Regulation (EC) 178/2002, which is EC legislation on general food safety and is directly applicable in the UK. Similar legislation applies in Northern Ireland. The following areas are of particular relevance to illegally irradiated products:

- Foods must comply with the provisions of Article 14 of 178/2002. Article 14 requires that foods must not be placed on the market if unsafe. Where food is found to be in breach of specific legislation governing its safety, it can be *presumed* to be either injurious to health or unfit for human consumption and thus 'unsafe' for the purposes of Article 14. Therefore the acts which constitute a failure to comply with The Food Irradiation Regulations 2009 may also constitute an offence under regulation 4 of the General Food Regulations.
- Foods must comply with the provisions of Article 16 of 178/2002. Article 16 requires that foods must not be presented in any way which may mislead consumers – failure to declare the presence of an irradiated ingredient may also constitute an offence under regulation 4 of the General Food Regulations.

It should be noted that while the above articles of Regulations 178/2002 may apply to illegally irradiated products, the primary consideration is that it is an offence to sell foods failing to comply with the Food Irradiation Regulations. It is for this reason that appropriate action should be taken to withdraw these foods from sale (see Section 3.2).

Further Information, including the application of Article 14 of 178/2002, can be found in 'Guidance Notes for Food Business Operators on Food Safety, Traceability, Product Withdrawal and Recall', which is available along with other guidance on general food law on the Food Standards Agency website: <http://www.food.gov.uk/foodindustry/regulation/foodlaw/>

The Consumer Protection From Unfair Trading Regulations 2008 (SI 2008/1277) – implement the EC 'Unfair Commercial Practices Directive' 2005/29. These Regulations apply to supply of goods, including foods, to consumers. The main provisions relevant to irradiated foods are those which prohibit misleading actions and misleading omissions which may prevent a consumer from making 'an informed transactional decision'. Failure to disclose an irradiated product may be such an omission.

The Business Protection from Misleading Marketing Regulations 2008 (SI2008/1276) – contain similar provisions to those found in the Consumer Protection From Unfair Trading Regulations 2008 but in this case relates to supplies of goods and services from trade to trade and therefore covering the supply chain.

3.9 Responsibility of a food business to ensure due diligence

See Section 4 of this guide.

Further information can be found in 'The Food Safety Act 1990 – A Guide for Food Businesses', which is available with other guidance on general food law on the Food Standards Agency website: <http://www.food.gov.uk/foodindustry/regulation/foodlaw/>

3.10 Responsibility of a food business on discovering an illegal product

A food business operator must withdraw food from the market where it considers or has reason to believe that a food which it has imported, produced, processed, manufactures or distributed does not comply with food safety requirements.

Food business operators must also notify the competent authorities (their local authority and the Food Standards Agency), and collaborate with these authorities on the action they should take.

Further Information can be found in the 'Guidance Notes for Food Business Operators on Food Safety, Traceability, Product Withdrawal and Recall', which is available along with other guidance on general food law on the Food Standards Agency website: <http://www.food.gov.uk/foodindustry/regulation/foodlaw/>

4. DUE DILIGENCE ACTIONS

4.1 Purchase specifications

The requirement for compliance with food irradiation legislation and the purchaser's policy on the use of irradiated ingredients should be included on the company's general purchasing specification and on every purchase order. Specifications should include a requirement that any changes made to the products or supply chain be notified to the company before they take place.

4.2 Supplier questionnaires

The understanding of and compliance with food irradiation legislation and, where relevant, the non-irradiation status of materials & bulk/finished products should be included on product questionnaires which are sent to their suppliers for completion before purchase.

4.3 Supplier audit

Major suppliers of materials or bulk/finished product should be audited for Good Manufacturing Practice of the substances and also investigation of potential risks of non-compliant irradiation by the supplier or its own suppliers. Where relevant, the supplier audits should include a check of the supplier's purchase documentation and/or the

supplier's sampling & analysis regime to confirm that the ingredients that they purchase & use have not been irradiated.

4.4 Certificate of Conformity/Analysis

All Certificates of Conformity or Certificates of Analysis that should be received for all purchases of relevant materials & bulk/finished product should make a declaration that the materials, ingredients or products have, or have not, been subjected to irradiation or ionising radiation and, where they have been, state the irradiation dose and identify the facility and its registration number the batch number and date of treatment.

4.5 Sampling Regime

A sampling regime should be established reflecting confidence in suppliers based on audit results & history of test results over time. See Section 5 on Sampling.

4.6 Response to Supplier on Intermediate or Positive Irradiation results

If a material or bulk/finished product declared as not irradiated results in an 'Intermediate' or 'Positive' irradiation test result when tested by PSL, TL or another standard test method, then the supplier should be contacted to explain the result and to be involved in any follow-up actions necessary.

PSL screening in its simplest form does not include sample preparation steps to enhance sensitivity, or calibration procedures to verify sensitivity. It may fail to detect the presence of irradiated components in dilute mixtures, or where mineral contents are low. Hence it is advisable to submit a proportion (for example 5 - 10% depending on product type and associated risk) of samples achieving negative PSL results for testing by another standard method such as TL. For further guidance see Section 6.

If TL analysis of a multi-ingredient finished product or intermediate mixture indicates the presence of irradiated material then the retained samples of the component ingredients should be analysed to identify the source. Companies should maintain their awareness of the existence of potentially irradiated materials on the market by regular updates from sources such as: Competent Authorities, Industry mailings and weekly EC RASFFs.

5. SAMPLING

5.1 Materials to be sampled

In general, materials most likely to have been irradiated are those that may suffer from microbiological contamination and are often naturally derived, although there are exceptions.

Ideally, every batch of material, bulk product & finished product should be sampled and submitted for a screening test such as PSL. Records should be kept of all results by substance and tests on materials and product with consistently negative results could be

reduced to an appropriate lower frequency, which can be substantiated based on the assessed risk.

If a substance is sourced from a new or different supplier, then the testing should recommence at the 100% level. It is recommended that all batches of botanical source material should continue to be tested by PSL at the 100% level whatever the results.

5.2 Frequency of sampling

The frequency of testing should be reassessed at intervals following review of previous results or when changes take place to supplier, process, specification or other material factor.

5.3 Size of sample & sampling plan to obtain Representative samples

At the time of publication (September 2010), a statistical basis for making firm recommendations for sampling materials for detection of irradiated materials was not available, although there is legislation covering the sampling of chemical contaminants and mycotoxins. Directive 2001/22⁷ (as amended by Directive 2005/4)⁸ on the official control of heavy metals & 3-MCPD in foodstuffs, Regulation (EC) 333/2007⁹ on the official control of levels of lead, cadmium, mercury, inorganic tin, 3-MCPD & BaP in foods give similar Official Procedures for the Sampling and Sample Preparation prior to analysis and Regulation (EC) 401/2006¹⁰ on mycotoxin sampling & testing (see References). However the incremental sample size and total aggregate sample size recommended may not be directly applicable to food irradiation and may be unnecessarily costly. Simpler sampling schemes may be adequate.

The sample regime ideally should be based on a risk assessment which should take into account the type of material to be tested, source, batch size and number of containers. Consideration should be given to the risk of failing to detect irradiated components and the number of samples to be taken.

For general guidance it is suggested that typically a sample of 50g but preferably greater than 100g should be prepared by aggregating $\sqrt{n} + 1$ incremental samples from a batch delivered in n containers.

Although this is a typical sampling-plan it may not be appropriate for samples with low mineral content or samples taken from heterogeneous bulk. These would require larger aggregate or multiple individual samples to be taken.

5.4 Homogenising samples

For a finished product or a bulk finished product in which the ingredients are distributed homogeneously or an ingredient used in quantities equating to multiples of containers an aggregate sample may be applicable. However, for products in which an ingredient is not dispersed homogeneously or an ingredient lot received in multiple containers which is used in small quantities, individual container samples may be more appropriate. Where appropriate, the aggregate sample may be prepared by combining the incremental samples and thoroughly mixing in an adequately sized container taking care not to 'demix' the material by so doing.

The sample will normally be homogenised by the laboratory prior to analysis.

5.5 Handling, storage and transport

Samples are combined in an opaque container with a close fitting opaque closure, both having good moisture barrier properties and stored in a cool, dry place at <25°C.

5.6 Avoiding signal bleaching or contamination

To avoid signal bleaching, the sampling should be carried out in low-light or special light conditions and the sample should not be exposed to light subsequently before testing.

At all stages the sample should be obtained and handled by persons trained in food hygiene, Good Manufacturing Practice & sampling techniques to ensure absence of contamination of the sample.

5.7 Traceability of samples

As required by Regulation (EC) 178/2002 on General Food Law (General Food Regulations SI 2990/2004), each business operator in the supply chain within the EU is responsible for keeping records of the supplier and customer for each batch of material & product. It is good practice to obtain information on the supply chain in third countries for raw materials and products imported into the EU and to ensure there are records of ingredient batch information held within manufacturing batch records when products are produced from a number of different materials.

6. TESTING & METHODOLOGY

6.1 Detection methods:

The two most frequently used methods for testing for the irradiation of food supplements and/or their ingredients are Photostimulated Luminescence (PSL – EN 13751:2009) & Thermoluminescence (TL – EN 1788:2001).^{11, 12, 13} However for certain materials, it may be more appropriate to use other CEN (European Committee for Standardisation) methods for detection of irradiation e.g. by ESR spectroscopy (EN 1787:2000)¹⁵ for products containing cellulose or by GC-Mass spectrometry (EN1785:2003) for products containing fats.¹⁸

More information on appropriate methodology and the published CEN Standard Test Methods are given in the Annex.

6.2 Photostimulated Luminescence (PSL)

This is a rapid method (taking 15 – 60 seconds) that is reliable for single substances (eg herbal ingredients) that are homogeneous and contain a significant level of mineral particulate matter. The test is quick to carry out as there is no sample preparation stage. It is less reliable for multiple, mixed ingredients (product matrices) or substances that contain insignificant levels of mineral particulates. Positive or Intermediate results should be confirmed by TL. Although PSL is promoted as a screening method, the results suffer from 'false negatives' and it is advisable to carry out a level of TL testing on a proportion

of samples giving negative results (for example 5-10% depending on the product type and associated risks).

6.3 Calibrated PSL

This is similar to the simple PSL test except that following initial photostimulation, the material is subjected to a standardised, low level irradiation (1 kilogray [kGy]) before a repeat PSL test. This process indicates materials that may have insignificant levels of mineral particulate indicated by the low level of emission from the repeated photostimulation following controlled irradiation and gives more reliable results based on a comparison of emissions before and after re-irradiation.

6.4 ThermoLuminescence (TL)

This is a more sophisticated method requiring sample preparation, more sophisticated equipment and experienced interpretation of the results. Although tests on mixtures of materials are more reliable due to the benefits of sample preparation, there are reported variations in interpretation of results between laboratories on samples from the same source. There are a limited number of laboratories in the world offering this technique.

6.5 Alternative & Combined methodology

Most companies will use a combination of detection methods as part of their due-diligence actions in preventing the use of irradiated ingredients in their products. Many of the larger ingredient suppliers and food supplement manufacturing companies have purchased their own PSL equipment (available from SUERC) to carry out screening in their own laboratories. Any Positive or Intermediate results are checked by subsequent TL testing in an appropriate commercial laboratory with a proportion of 'negative' results (e.g. 5-10% depending on the product type and associated risks) also being checked.

Ten CEN standardised test methods used in the detection of irradiation of foods are included with links to the methodology in Annex I.

ANNEX I - Summary of Detection methods

There are ten tests that have been standardised by CEN (European Committee for Standardisation) of these thermoluminescence and photostimulated luminescence techniques are the most widely used. More specific tests include analysis for alkyl cyclobutanones and electron spin resonance analysis.

Drafting Guidelines are attached to the PQ Reference Guide

1. EN 1784:2003 Detection of irradiated food containing fat - Gas chromatographic analysis of hydrocarbons¹⁷

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the identification of irradiation treatment of food which contains fat. It is based on the gas chromatographic (GC) detection of radiation-induced hydrocarbons (HC). The method has been successfully tested in inter laboratory tests on raw chicken, pork and beef as well as on camembert, avocado, papaya and mango.

During irradiation, chemical bonds are broken in primary and secondary reactions. In the fatty acid moieties of triglycerides breaks occur mainly in the α and β positions with respect to the carbonyl groups resulting in the respective C_{n-1}) and the $C_{n-2;1}$ HC). To predict these chief radiolytic products, the fatty acid composition of samples has to be known.

Saturated HC are frequently present both as contaminants and as naturally occurring compounds in food. Therefore, they are not used in isolation for identification of an irradiated sample. Detection of irradiated raw meat and Camembert has been validated for doses of about 0.5 kGy and above covering the majority of commercial applications. Detection of irradiated fresh avocado, papaya and mango has been validated for doses of approximately 0.3 kGy and above. The concentration of HC derived from fatty acids which are of low concentration in the particular fat will be low and might be below the detection limit in the case of low radiation doses. Particularly in fruit the applied doses might be lower than the doses used in the interlaboratory test. Detection limit is not influenced by usual applied commercial storage time.

2. EN 1785:2003 Detection of irradiated food containing fat - Gas chromatographic / mass spectrometric analysis of 2-alkylcyclobutanones¹⁸

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the identification of irradiation treatment of food containing fat. It is based on the mass spectrometric (MS) detection of radiation-induced 2-alkylcyclobutanones after gas chromatographic (GC) separation. The method has been successfully tested in inter laboratory trials on raw chicken, pork, liquid whole egg, salmon and Camembert. Other studies demonstrate that the method is applicable to a wide range of foodstuffs.

During irradiation, the acyl-oxygen bond in triglycerides is cleaved and this reaction results in the formation of 2-alkylcyclobutanones containing the same number of carbon atoms as the parent fatty acid and the alkyl group is located in ring position 2. Thus, if the fatty acid composition is known, the 2-alkylcyclobutanones formed can be predicted.

Detection of irradiated raw chicken has been validated for doses of approximately 0.5 kGy and above. The detection of irradiated liquid whole egg, raw pork, salmon and Camembert has been validated for doses of approximately 1 kGy and above. Validation at these doses covers the majority of commercial applications.

3. EN 1786:1996 Detection of irradiated food containing bone - Method by ESR spectroscopy¹⁴

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the detection of meat containing bone and fish containing bone which have been treated with ionizing radiation, by analysing the electron spin resonance (ESR) spectrum, also called electron paramagnetic resonance (EPR) spectrum, of the bones.

ESR spectroscopy detects paramagnetic centres (e.g. radicals). An intense external magnetic field produces a difference between the energy levels of the electron spins $m_s = +\frac{1}{2}$ and $m_s = -\frac{1}{2}$, leading to resonance absorption of an applied microwave beam in the spectrometer. ESR spectra are conventionally displayed as the first derivative of the absorption with respect to the applied magnetic field.

Detection of irradiated bone samples is typically possible above a dose of approximately 0.5 kGy, covering the majority of commercial applications.

Detection limits and stability are influenced by the degrees of mineralization and crystallinity of hydroxyapatite in the sample.

In general, the bones of larger animals and species are highly mineralized with low minimum detectable doses. However, variations within individual animals and species have been noted.

In case of meat bones the results of this detection method are not significantly influenced by heating of the sample (e.g. boiling in water). Detection of irradiation treatment is not significantly influenced by storage times of up to 12 months. For poorly mineralized fish bones it has been noted that non radiation induced signals are strongly enhanced if the temperatures recommended (6.1) for drying are exceeded and may interfere with the radiation specific signals.

4. EN 1787:2000 Detection of irradiated food containing cellulose by ESR spectroscopy¹⁵

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the detection of foods containing cellulose which have been treated with ionizing radiation, by analysing the electron spin

resonance (ESR) spectrum, also called electron paramagnetic resonance (EPR) spectrum, of the food.

ESR spectroscopy detects paramagnetic centres (e.g. radicals). They are either due to irradiation or to other compounds present. An intense external magnetic field produces a difference between the energy levels of the electron spins $m_s = +\frac{1}{2}$ and $m_s = -\frac{1}{2}$, leading to resonance absorption of an applied microwave beam in the spectrometer. ESR spectra are conventionally displayed as the first derivative of the absorption with respect to the applied magnetic field.

Detection limits and stability are influenced by the crystalline cellulose content and the moisture content of the samples. Positive identification of the cellulose radicals is evidence of irradiation but the absence of this signal does not constitute evidence that the sample is unirradiated.

5. EN 1788:2001 Detection of irradiated food from which silicate minerals can be isolated - Method by thermoluminescence¹²

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the detection of irradiation treatment of food and/or food ingredients by thermoluminescence analysis of contaminating silicate minerals. This method is applicable to those foodstuffs from which a sufficient amount of silicate minerals can be isolated.

Silicate minerals contaminating foodstuffs store energy by charge trapping processes as a result of exposure to ionizing radiation. Releasing such energy, by controlled heating of isolated silicate minerals, gives rise to measurable TL glow curves.

This method of TL analysis can, in principle, be applied to detect irradiation of any food from which silicate minerals can be isolated. Detection limits and stability of the method depend on the quantities and types of minerals recovered from individual samples, and the glow temperature intervals selected for analysis. Minerals from unirradiated samples show a residual geologically derived TL signal with maximum intensity at glow curve temperatures above 300 °C, and minor components in the 200 °C to 300 °C region which can influence detection limits. The stability of TL signals is strongly influenced by glow curve temperature and is greater for higher temperatures. For temperatures from 200 °C to 250 °C TL signals are stable for many years.

6. EN 13708:2001 Detection of irradiated food containing crystalline sugar by ESR spectroscopy¹⁶

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the detection of foods containing crystalline sugars which have been treated with ionizing radiation, by analysing the electron spin resonance (ESR) spectrum, also called electron paramagnetic resonance (EPR) spectrum, of the food.

ESR spectroscopy detects paramagnetic centres (e.g. radicals). They are either due to irradiation or to other compounds present. An intense external magnetic field produces a difference between the energy levels of the electron spins $ms = + \frac{1}{2}$ and $ms = - \frac{1}{2}$, leading to resonance absorption of an applied microwave beam in the spectrometer. ESR spectra are conventionally displayed as the first derivative of the absorption with respect to the applied magnetic field.

While the general formation processes of radiation-induced radicals are known, identification of the specific radicals responsible for individual signals has not yet been achieved.

7. EN 13783:2001 Detection of irradiated food using Direct Epifluorescent Filter Technique/Aerobic Plate Count (DEFT/APC) - Screening method¹⁹

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a microbiological screening method for the detection of irradiation treatment of herbs and spices, using the combined direct epifluorescent filter technique (DEFT) and aerobic plate count (APC). The DEFT/APC technique is not radiation specific, therefore, it is recommended to confirm positive results using a standardised method (e.g. EN 1788, EN 13751) to specifically prove an irradiation treatment of the suspected food.

The method is based on the comparison of the APC with the count obtained using DEFT. The APC gives the number of viable microorganisms in the sample after a possible irradiation and the DEFT count indicates the total number of microorganisms, including non-viable cells, present in the sample. The difference between the DEFT count and the APC count in spices treated with doses of 5 kGy to 10 kGy is generally about or above 3 to 4 log units. Similar differences between DEFT and APC counts can be induced by other treatments of the foods (e. g. heat), leading to death of microorganisms, thus positive results should be confirmed.

A limitation of the method is encountered when there are too few microbes in the sample ($APC < 10^3$ cfu/g). If fumigation or heat treatment has been used for decontamination, the DEFT/APC difference of counts can be similar to the difference of counts obtained after irradiation. However, the use of fumigation can be detected.

8. EN 13784:2001 DNA comet assay for the detection of irradiated foodstuffs - Screening method²¹

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a screening method for foods which contain DNA. It is based on micro-gel electrophoresis of single cells or nuclei to detect DNA fragmentation presumptive to irradiation treatment. The DNA Comet Assay is not radiation specific, therefore, it is recommended to confirm positive results using a standardized method to specifically prove an irradiation treatment of the respective food, e.g. EN 1784, EN 1785, EN 1786, EN 1787, EN 1788, EN13708, and EN 13751.

DNA fragmentation can be caused by various chemical or physical treatments including ionizing radiation. When food containing DNA is treated by ionizing radiation, modification of these large molecules occurs including fragmentation either by single- or double-strand breaks. This fragmentation can be studied by microgel electrophoresis of single cells or nuclei. These are embedded in agarose on microscope slides, lysed for disruption of membranes using a detergent and electrophoresed at a set voltage. DNA fragments will stretch or migrate out of the cells forming a tail in the direction of the anode giving the damaged cells the appearance of a comet. This comet assay to measure DNA damage can be carried out under various conditions. Both alkaline and neutral protocols exist. In general, under alkaline conditions both DNA single- and double-strand breaks and alkali-labile sites are measured, whereas under neutral conditions only DNA double-strand breaks are observed. However, using neutral conditions single-strand breaks also exert an influence on the comet appearance, due to relaxation of supercoiled DNA in the nucleus. Irradiated cells will show an increased extension of the DNA from the nucleus towards the anode thus considerably longer comets (more fragmentation) than unirradiated cells. Unirradiated cells will appear nearly circular or with only slight tails.

9. EN 13751:2009 Detection of irradiated food using photostimulated luminescence¹³

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This European Standard specifies a method for the detection of irradiated foods using photostimulated luminescence (PSL). The technique described here comprises an initial measurement of PSL intensity which may be used for screening purposes, and a calibration method to determine the PSL sensitivity to assist classification. It is necessary to confirm a positive screening result using calibrated PSL or another standardised or validated method (e.g. EN 1784 to EN 1788).

Mineral debris, typically silicates or bioinorganic materials such as calcite which originate from shells or exoskeletons, or hydroxyapatite from bones or teeth, can be found on most foods. These materials store energy in charge carriers trapped at structural, interstitial or impurity sites, when exposed to ionising radiation. Excitation spectroscopy has shown that optical stimulation of minerals releases charge carriers. It has subsequently been shown that the same spectra can be obtained from whole herb and spice samples and other foods using photostimulation. PSL measurements do not destroy the sample, therefore whole samples, or other mixtures of organic and inorganic material, can be measured repeatedly. PSL signals, however, decrease if the same sample is measured repeatedly.

10. Microbiological screening for irradiated food using LAL/GNB procedures²⁰

http://ec.europa.eu/food/food/biosafety/irradiation/anal_methods_en.htm

This document specifies a microbiological screening method comprising two procedures, which are carried out in parallel. It permits the identification of an unusual microbiological profile in poultry meat. The presence of a large excess population of dead micro-organisms can under certain circumstances be presumptive of irradiation

treatment, which means, that the results of the procedure of the determination of endotoxin concentration in the test

sample using the *Limulus* amoebocyte lysate (LAL) test and of the procedure of the enumeration of total Gram negative bacteria (GNB) in the test sample are not radiation specific. Therefore, it is recommended that a positive result be confirmed using a standardized reference method for the detection of irradiated food, e.g. EN 1784, EN 1785 or EN 1786.

The method determines the number of viable Gram negative bacteria present in the test sample and the concentration of bacterial endotoxin present on the surfaces of Gram negative bacteria as lipopolysaccharides (LPS) serving as a measure for the estimation of the amount of total Gram negative bacteria, both viable and dead. If the difference is high, it is assumed that the sample has been treated by a method of preservation, possibly by treatment with ionising radiation.

This method can give only an indication of a possible treatment by ionising radiation. A high amount of dead microorganisms in comparison to the viable fraction can be due to several other reasons. It is therefore necessary to confirm a possible treatment by ionising radiation by a standardized reference method for the detection of irradiated foods. The method is of particular use to routine microbiological laboratories, which may be involved in the examination of foods.

ANNEX II - An overview of EN detection methods for irradiated foods

Whereas the FAO/WHO Codex Alimentarius Committee in 1979 noted that “Despite the many investigations designed to detect physical, chemical and biological changes in foods subjected to ionising energy, no satisfactory method for identifying food as having been irradiated has so far been developed” this was no longer the case by the 1990’s. Partly as a result of the research programme initiated in the UK by the Ministry of Agriculture Fisheries and Foods, and also as a result of coordination activities in Europe which drew attention to work on analytical detection methods for irradiated food, a series of physical, chemical and biological detection methods have been validated and standardised. At the time of writing there is still no single test that is universally applicable to all food matrices or food ingredient mixtures. However the methods that are available cover a wide range of the food products which routinely undergo radiation treatment.

Of the physical methods, the thermoluminescence (TL) method was the first to undergo international validation, becoming a UK standard method in 1992¹¹ and a European Standard in 1996¹². The **TL** method depends on energy storage in structural lattice defects in mineral systems associated with foods when they are exposed to ionising radiation. During laboratory analysis the minerals are separated and heated in a TL reader. The heat releases trapped charge carriers arising from prior radiation exposure, which leads to emission of light that can be detected using photomultiplier tubes. The EN1788 method specifies procedures for mineral separation, conducting calibrated TL analysis and evaluation of data. The method was originally developed for dried products includes herbs, spices and seasoning mixtures. It has also been validated for seafood and fruits and vegetables, and is widely applied to a diverse set of samples. Food irradiated at doses from 100 Gy up to and above 10 kGy can be detected. In blended mixtures detection limits vary depending on sample mineral loads in the components, but 1-10% levels of irradiated ingredients are routinely detected. Related to TL is the photostimulated luminescence (**PSL**) method, developed in the UK and standardized in 2002 and updated in 2009 (EN13751)¹³. This uses light rather than heat to release luminescence induced by ionising radiation, and is normally used in a rapid screening mode where samples are tested instrumentally with little or no sample preparation. PSL screening is routinely conducted in food production and testing facilities in most food producing countries. Samples are classified into three screening bands (“Negative”, “Intermediate” and “Positive”) which are used to direct subsequent investigation .

Electron Spin Resonance provides another physical method, which has resulted in three European Standard methods. The ESR phenomenon arises as a result of the small energy differences taken up by unpaired electrons when placed in a magnetic field, which results in resonant microwave absorption at frequencies which correspond to rapid transitions between spin-up and spin-down states. The relationship between resonant energy and the applied magnetic field depends on the specific coordination of species with unpaired electrons, which is exploited in ESR spectrometers to reveal structural information about the coordination of such centres in materials. Irradiation produces many radicals, which by definition contain unpaired electrons, the majority of which are highly reactive and thus unstable species. However in certain dry matrices they can be preserved in relatively stable form, resulting in detectable radiation induced changes to those products. International standard methods for ESR detection of

irradiated foods have been developed for well mineralized bone (EN1786)¹⁴, for cellulose (EN1787)¹⁵ and for crystalline sugars (EN13708)¹⁶. In bone the radiation induced signal is identified as a trapped CO_2^- radical immobilized in the hydroxyapatite matrix of bone. Providing the bone is well mineralized it can be detected at doses above 0.5 kGy. It has been validated for chicken, beef and fish bones. The detection limits and stability of the radiation induced cellulose radical are influenced by the degree of crystallinity and moisture, and the standard notes that while positive identification of the radical can be considered as evidence of irradiation, the lack of signal does not constitute evidence that the sample is unirradiated. The cellulose ESR method has been successfully validated for pistachio nuts (>2 kGy), dried paprika (>5 kGy), and strawberries (>1-5 kGy). The specific radicals responsible for the ESR signals in irradiated crystalline sugars have not been identified, and the signals are known to depend on the degree of crystallinity of the sugar, and to be adversely affected by moisture. The method has been validated for dried figs, mangos, papayas and raisins.

The two chemical methods both apply to foods containing fat. The Gas-Chromatography Hydrocarbon (GC-HC) method (EN1784)¹⁷ depends on identifying specific fragmentation patterns associated with radiolysis of fatty acids in foods. The fats are first separated from the food by melting or solvent extraction, and the HC fractions obtained by adsorption chromatography prior to separation and detection by gas-chromatography. The HC spectra are complex, but the positions of triglyceride breaks relative to the carbonyl groups of irradiated parent fatty acids follow a predictable scheme. To identify a food as irradiated the HC spectra are interpreted relative to the parent fatty acid profiles associated with the specific food-type. The method has been validated for meat (chicken, pork, beef) cheese (Camembert) and fruits (avocado, papaya and mango). The other chemical method based on the radiolysis of triglycerides is the alkyl-cyclobutanone method (EN1785)¹⁸. Alkyl-cyclobutanones are molecules based on a 4-atom carbon ring with a hydrocarbon side-chain. They are formed following breaks in the acyl-oxygen bond within a triglyceride during irradiation, and can be detected using gas chromatography mass-spectrometry (GC-MS) using samples prepared by solvent extraction and column chromatography. Two alkyl-cyclobutanone have been synthesized for use as GCMS standards: 2-dodecyl-cyclobutanone (DCB), derived from palmitic acid, and 2-tetradecyl-cyclobutanone (TCB), derived from stearic acid. These have been validated for detection of irradiated chicken, pork and liquid whole egg irradiated at doses of 3 kGy and 5 kGy.

The biological methods are considered as screening methods rather than reference methods, since the effects which are utilized are not radiation specific. However they may be of considerable value in that they provide indirect evidence of irradiation, which can also under some circumstances indicate the extent to which the product has been retained in fresh condition. Two of the biological methods exploit a similar principle. The DEFT/APC method (EN13783)¹⁹ compares the results of direct epifluorescence detection of material associated with bacterial cells, which can be dead or alive, with the results of an aerobic plate count which responds to the live cells. Similarly the LAL/GNB method (EN14569)²⁰ uses the limulus amoebocyte lysate clotting reaction to estimate the amount of gram-negative bacterial endotoxin within a sample (ie resulting from both dead and live cells), and compared it with a gram-negative plate count which responds to the viable cells only. In both of these methods a significant excess of dead bacterial material in comparison with live cells may be an indicator of bactericidal treatment, of

which radiation treatment may circumstantially be a cause. The DEFT/APC method has been validated for herbs and spices irradiated and doses of 5-10 kGy. The LAL/GNB method was validated for chicken meat irradiated at a 5 kGy dose. Despite lack of specificity both of these methods offer the possibility of making an inference about the microbiological condition of irradiated product prior to treatment, and therefore could be important in investigating the circumstances of irradiation. The final biological method is the DNA comet assay (EN13784)²¹ which is a simple method for detecting fragmentation of the cell nucleus and DNA using microelectrophoresis. Again the method is not radiation specific, since DNA degradation can also occur as a result of thermal, enzymatic or bacterial spoilage, but it has been established that radiation gives rise to DNA fragmentation which can be readily detected using the Comet assay. The method has been validated for pork, chicken at doses of 1-5 kGy, and for almonds, figs, lentils, linseed, pepper, sesame seeds, soy beans and sunflower seeds irradiated at doses from 0.2-5 kGy.

The European Commission periodically publish reports with an indication of the numbers of samples examined by member states in surveys of undeclared irradiated foods on the market. Table 1 below summarises the numbers of analyses conducted, the number of undeclared irradiated samples detected, and the numbers of member states using each of the standard methods. From this it can be seen that the level of detection activity for enforcement purposes has remained relatively steady over this period, with some 5000-7000 results reported. Irradiated samples are being detected at significant rates in all years. It is also notable that while the physical methods, and in particular PSL and TL remain the most widely applied, there is increasing uptake of chemical and biological methods in recent years; mostly associated with new member states.

			Method (No. of Member states reporting)						
	Samples	Irradiated	PSL EN13751	TL EN1788	ESR EN1786 EN1787 EN13708	GC HC EN1784	Cyclo- butanone EN1785	DEFT /APC EN13783	DNA Comet EN13784
2001	6651	97	5	5	1	2			
2002	4894	137	4	5	2	1	1		
2003	5470	193	6	7	2	1	1	1	
2004	6890	279	8	12	3	1	3	1	
2005	6724	281	8	12	3	2	1	2	2
2006	6134	203	8	12	3	2	1	1	1
2007	6176	203	9	15	4	3	2	2	1

Table 1. EU Member State Reports to the Commission of Testing methods used in Official Testing between 2001 and 2007 for all Food Categories

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Further Reading:

'Detection Methods for Irradiated Foods: Current Status' Edited by: Cecil H. McMurray, Eileen M. Stewart, Richard Gray and Jack Pearce Published by: The Royal Society of Chemistry Year: 1996 ISBN: 0-85404-770-0

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