

**Detection by Thermoluminescence of an Irradiation Treatment of Five Species of Dehydrated Fruit and Vegetables Report on a CTCPA/AIFLD International Interlaboratory Study Edited by Eric Marchioni and Henry Delincée  
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SUMMARY

In this collaborative study, 9 laboratories in the European Union (3 from France, 3 from Germany, 1 from Great Britain, 1 from Italy and 1 from Finland) agreed to test the method for thermoluminescence (TL) detection of irradiated food from which silicate minerals

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can be isolated - European Standard EN 1788:1996, adopted by CEN, the European Committee for Standardization, 1996 - in a set of five species of dehydrated fruit and vegetables. As these foods are commonly used as ingredients by the food industry, they have to be of very high bacteriological quality in order to avoid any contamination of the food which they are added to. These kinds of food may be favourably treated with ionizing radiation to improve their hygienic qualities and to combat insect infestation and/or microbial contamination.

Each laboratory investigated samples of dehydrated carrots, leeks, onions, asparagus and apples. The samples were non-irradiated (reference samples) or irradiated at an average dose of  $8.4 \pm 0.8$  kGy. A set of three samples of each of the five species of dehydrated fruit and vegetables was sent to the participating laboratories one month (trial 1), six months (trial 2), twelve months (trial 3), fifteen months (trial 4) and twenty-four months (trial 5) after irradiation. During preparation of the samples to be analysed twelve months after irradiation, some labelling errors were unfortunately made. The 242 samples analysed at that time, therefore, are not considered in this report; the data, however, are contained in Annex C.

This report considers only four of the five sets of results (one, six, fifteen and twenty-four months after irradiation). From the 765 samples analysed, 73 were rejected, because not enough minerals had been isolated [thereby not fulfilling the requirement that Glow 2 should be higher than ten times the Minimum Detectable integrated TL-intensity Level (MDL), see the European Standard EN 1788:1996]. Among the 692 remaining samples (352 non-irradiated and 340 irradiated), a total of 625 (90%) were correctly identified as irradiated or non-irradiated. 59 samples were classified as in doubt. Only 8 samples (1%) apparently were not correctly identified. However, the glow curves revealed that these 8 wrong results were due to labelling errors. Regarding the samples classified as in doubt, it should be kept in mind that in practice analysis of these samples would be repeated until an unequivocal result is obtained. Due to lack of sample material in this interlaboratory test, repetitions were not possible. In practice, enough sample material is usually available, thus higher identification rates can be expected.

The present collaborative study has shown that identification of radiation treatment of the five dehydrated fruit and vegetables studied using TL analysis is possible. It has been proposed to the CEN/TC 275/WG8 to extend the applicability of the TL method as described in the European Standard EN 1788:1996, with some minor modifications, to dried fruit and vegetables.

## INTRODUCTION

The thermoluminescence method for the detection of irradiated food has been widely used in numerous studies in many kinds of foods [1-34]. It is based on the isolation of contaminating silicate minerals (mineral debris) from the food, as these minerals store energy during irradiation by trapping charge carriers in an excited state. By controlled heating of the isolated minerals, the charge carriers are stimulated to return to the ground state, thereby emitting some of the energy as light. This light is detected by a sensitive photon counter, and the light emission, which is dependent on the temperature, is recorded as a so-called glow curve. The first glow curve (Glow 1) will be compared with a second glow curve (Glow 2) obtained by a second thermoluminescence measurement of the isolated minerals following the exposure to a defined radiation dose. This normalization procedure allows for differences in mineral composition – e.g. varying amounts of feldspar or quartz – and/or in weight of the aliquots of isolated mineral grains. A comparison of size and shape of the two glow curves reveals, whether the sample from which the mineral grains were isolated has been irradiated or not. The TL glow ratio which is the ratio integrated TL intensities of Glow 1 to Glow 2 - thus the Glow 1 area divided by the Glow 2 area – evaluated over a defined temperature interval, is typically greater than 0.5 for irradiated and generally below 0.1 for non-irradiated samples. A prerequisite of the calculation of the TL glow ratio is that the area of Glow 2 – evaluated over the defined temperature interval – is 10 times higher than the Minimum Detectable integrated TL-intensity Level (MDL), see the European Standard EN 1788:1996 [1].

Up to 1996, the thermoluminescence (TL) method had been tested successfully in interlaboratory blind trials (which, however, were never conducted over the whole storage time of the products studied as the authors considered the signals by thermoluminescence to be stable for many years) in spices, aromatic herbs, their mixtures [35-37] and shrimps [38 and 39]. As a consequence, the standard EN 1788:1996 [ 'Detection of irradiated food from which silicate minerals can be isolated - Method by thermoluminescence' available in all member-states of the European Union – and, in addition, in Iceland, Norway and Switzerland, pertains only to herbs, spices, their mixtures and shrimps. This limitation is inconsistent with the statement that this protocol may be applied - after suitable modification - to a large variety of foods (from which a sufficient amount of silicate minerals can be isolated). Therefore, additional interlaboratory trials were initiated to study TL detection of potatoes [40], fresh fruit and vegetables [41], and shellfish [42].

As irradiation of dehydrated fruit and vegetables is a promising technique to combat insect infestation and/or microbial contamination, it would be desirable to check the correct labelling of the irradiated products. Thermoluminescence measurement could be a suitable procedure for this purpose.

The European industry in the dehydrated fruit and vegetable sector was ready to support an international comparison based on the study of five species of dehydrated fruit and vegetables for the validation - over the entire storage time (24 months) - employing the thermoluminescence method (TL) for the detection of irradiated food in order to extend the applicability of the TL method as described in the standard EN 1788:1996 to dried fruit and vegetables.

One of the food items studied, apples, carried only a very low amount of silicate minerals. In fact, 1

contaminating minerals which are present on all foodstuffs which have been exposed to wind and soil, adhere only to the skin of the fruit. The apple product is mechanically peeled prior to dehydration. Most of the silicates had therefore been removed, and the final dehydrated food was only little contaminated by mineral dust.

Apples were still included into this study as a potential problematic sample, demonstrating the applicability of the TL method to the detection of irradiation even in food, little contaminated by silicate minerals.

## MATERIALS AND METHODS

### Food samples

Food samples were provided by European companies producing dehydrated foods. A total of 11 samples of sliced carrots, 9 samples (6 sliced and 3 powdered) of onions, 2 samples (1 sliced and 1 flaked) of leek, 7 samples of powdered asparagus and 4 samples (3 sliced and 1 powdered) of apples were studied. Among the large number of samples examined, those were selected for the intercomparison study which contained a representative amount of silicate contamination.

The selected food samples were sent to the laboratory of Centre Technique de la Conservation des Produits Agricoles (CTCPA-France). Each of them was homogenised by gentle agitation. To avoid possible problems of recontamination or cross-contamination, the foods were divided into 100 g aliquots (200 g for apples), packaged and thermo-sealed before irradiation.

### Irradiation

To achieve the best absorbed dose distribution, samples were irradiated in tight carriers in 60x40x40 cm cardboard boxes. Each box was filled with a total of five layers of the different dehydrated foods studied. The layers were arranged perpendicularly to the gamma beam, food of higher apparent density (onions asparagus) close to the container walls, food of lower density (leek) in the container centre.

A total of 20 boxes were irradiated at an absorbed dose of about  $8.4 \pm 0.8$  kGy ( $D_{\max}=10.5$ ;  $D_{\min}=6.8$ ) from an 800 000 Ci industrial source of  $^{60}\text{Co}$  delivering a dose rate of about 1 kGy/h at room temperature (Gammir II, GRIFFITH-MEDIRIS, Fleurus, Belgium).

A total of 94 Far West Technology dosimeters (FWT 60-00) calibrated against the French national reference (alanine pellets, L.M.R.I., Gif sur Yvette, France) were used to measure the distribution of the absorbed dose within each irradiated box. 20 boxes containing the non-irradiated samples were stored in the CTCPA laboratories.

After irradiation and until the date of analysis, the samples were stored in the cardboard containers in two separate rooms (one for the non-irradiated, one for the irradiated samples) at room temperature.

### Participating laboratories

9 laboratories in the European Union agreed to participate in this intercomparison study (1 from Great Britain, 3 from Germany, 3 from France, 1 from Italy and 1 from Finland), see Table 1.

Table I: List of the participants and the TL readers used

Responsible person	Laboratory name	Address	TL reader
<a href="#">H. Delincée</a>	<a href="#">Federal Research Centre for Nutrition</a>	Karlsruhe (D)	ELSEC, Littlemore
U. Wagner	<a href="#">BgVV</a>	Berlin (D)	RISØ TL reader
J. Raffi	Larqua/CEA/Marseille University	Marseille (F)	Harshaw 4000
Y. Pelleau	DGCCRF (LIR)	Marseille (F)	Harshaw 4000
<a href="#">D. Sanderson</a>	<a href="#">SURRC</a>	Glasgow (UK)	SURRC TL reader
<a href="#">S. Pinnioja</a>	<a href="#">Laboratory of radiochemistry</a>	Helsinki (SF)	RISØ TL DA10
<a href="#">E. Marchioni</a>	<a href="#">Aérial</a>	Strasbourg (F)	Harshaw 3500
<a href="#">H. Hungerbühler</a>	<a href="#">TeLA GmbH</a>	Berlin (D)	Harshaw 3500
<a href="#">E. Anklam</a>	<a href="#">JRC/food and drug unit</a>	Ispra (I)	Isotope TLD 7100 Teledyne isotope(*)

(\*)no glow curves available

## Sample distribution and analysis

The protocol used for the TL measurements is contained in Annex D; it is practically identical to the European Standard EN 1788:1996.

A set of three samples of each species of dehydrated fruit and vegetables was sent to the participating laboratories one month (trial 1), six months (trial 2), twelve months (trial 3), fifteen months (trial 4) a twenty-four months (trial 5) after irradiation. Only two laboratories (lab. N°5 and 8) were requested to analyse samples during the fourth trial (15 months after irradiation). The samples were sent by two separate mails, each containing three samples of each five species. All experiments have been done in duplicate (with two separate samples having the same labelling), during two separate weeks, following the CEN procedure (EN-1788:1996), with some minor modifications concerning the weight of the sample to be analysed (50 g for each vegetable sample, 100 g for apples, see [Annex D](#)).

After the first trial (1 month after irradiation) it was decided to increase the weight of the apple samples to 200g. However, this caused problems during preparation of the samples, as a gel formed in the aqueous layer of the polytungstate solution during the density separation step to free the minerals from organic material. It was decided, therefore, to modify the protocol. The sample of preconcentrated isolated minerals during the first polytungstate fractionation may be left in a stoppered tube overnight at room temperature. The water layer may then be extracted the next day and the gel, now in the upper part of polytungstate fraction, could be easily removed.

The participating laboratories were asked to determine the Minimum Detectable integrated TL-intensity Level (MDL) following the CEN procedure (EN 1788:1996). Any analysis with a Glow 2 area (integral) below 10 times the MDL value was excluded from the study. In addition, the duplicate was classified as in doubt, even if the area of its Glow 2 was above 10 times the MDL. Moreover, if the results of the duplicates disagreed, they were classified as in doubt. The percentage of results (success, error or in doubt) are given against the total number of samples analysed minus the number of excluded ones.

One laboratory (N°9) had no  $^{60}\text{Co}$  source and did not carry through the irradiation step for normalization (Glow 2). However, the results of this laboratory may be considered as a valuable screening and are therefore also presented in this report. The results are not included in the statistical evaluation, however.

## RESULTS AND DISCUSSION

### First part: Selection of the food samples to be submitted to the trial

Among the 33 specimens of dehydrated fruit and vegetables examined during the first screening tests (in which only two French laboratories participated: Aérial and Larqua), only 2 samples (apple flakes and leek flakes) could not be analysed by the protocol proposed. In fact, these two samples absorbed all the

water added during the preconcentration step of the minerals. A thick slurry quickly formed prevented purification. This kind of sample flakes hence had to be excluded from application of this protocol.

In the other 31 samples, a significant TL signal was detectable, even when the quantity of recovered minerals was less than 0.1 mg (Table II to VI).

**Table**

**II:**

Screening tests for the determination of the contamination of carrot samples by silicate minerals. Extracted silicate minerals were irradiated at 1.0 kGy.

Sample	Quantity of minerals (mg)	
	recovered in 100 g of food sample	TL signal at 220°C (Glow 1)
N°1	0.9	present
N°2	1.3	present
N°3	0.6	present
N°4	4.9	present
N°5	2.7	present
N°6	3.3	present
N°7	0.2	present
N°8	0.1	present
N°9	0.06	present
N°10	0.04	present
N°11	0.04	present

**Table**

**III:**

Screening tests for the determination of the contamination of onion samples by silicate minerals. Extracted silicate minerals were irradiated at 1.0 kGy.

Sample	Quantity of minerals (mg)	
	recovered in 100 g of food sample	TL signal at 220°C (Glow 1)
N°1	2.3	present
N°2	2.9	present
N°3	16	present
N°4	3.1	present
N°5	0.6	present
N°6	0.6	present
N°7	11.8	present
N°8	23	present
N°9	0.8	present

**Table**

**IV:**

Screening tests for the determination of the contamination of apple samples by silicate minerals. Extracted silicate minerals were irradiated at 1.0 kGy.

Sample	Quantity of minerals (mg)	
	recovered in 100 g of food sample	TL signal at 220°C (Glow 1)
N°1	<0.1	present
N°2	<0.1	present
N°3	<0.1	present
N°4 (flakes)	ND	absent

ND: not determined

**Table**

**V:**

Screening tests for the determination of the contamination of asparagus samples by silicate minerals. Extracted silicate minerals were irradiated at 1.0 kGy.

Quantity of minerals (mg)

Sample	recovered in 100 g of food sample	TL signal at 220°C (Glow 1)
N°1	13.4	present
N°2	24.7	present
N°3	29.4	present
N°4	3.6	present
N°5	1.8	present
N°6	6.4	present
N°7	11.9	present

## Table

## VI:

Screening tests for the determination of the contamination of leek samples by silicate minerals. Extracted silicate minerals were irradiated at 1.0 kGy.

Sample	Quantity of minerals (mg) recovered in 100 g of food sample	TL signal at 220°C (Glow 1)
N°1	0.24	present
N°2 (flakes)	ND	absent

ND: not determined

In some food samples (N°3, 7 and 8, Table III and N°1, 2, 3 and 7, Table V), a large quantity of recovered minerals has been found. Microscopic examination has shown that the silicates had not been sufficiently cleaned by the procedure recommended in the protocol, and some adhering organic contaminants increased the weight. In this case, the weight of the isolated mineral fraction did not represent the real quantity of silicates present on the TL disc. In case of too high or too low an amount of recovered minerals, a microscopic evaluation may be helpful to determine the purity of the matter deposited on the TL disc. The samples chosen for this intercomparison should yield enough minerals for TL measurements.

The selected samples for the intercomparison were:

- Apples, *Poma mela*, variety Cortland, produced in 1994
- Leeks, *Allium porrum*, mixed varieties, produced in 1994
- Onions, *Allium cepa*, variety Stuttgarter, produced in 1994
- Carrots, *Daucus carota*, variety Carotan, produced in 1994
- Asparagus, *Asparagus officinalis*, date of production unknown.

Of these samples, 50 g were sufficient to obtain enough clean minerals to run at least two TL measurements; only of apples a minimum of 100 g was necessary for one TL measurement.

The large number of samples analysed confirmed that the CEN TL method for the detection of irradiated foods (EN 1788:1996) is applicable to a large variety of food samples of any industrial and geographic origin.

### Second part: Interlaboratory blind trial

Laboratory 9 had no access to a radiation source delivering 1 kGy for the purpose of normalization. The TL reader of this laboratory, furthermore, did not have the option to display any glow curves. All interpretations were based only on the visual inspection of TL counts shown on a little LED display.

This laboratory which had specialized neither in the detection of irradiated food nor in TL methods, obtained valuable results (Table VII) which however, were not included into the statistical evaluation. They have shown, however, that a simplified procedure (without normalization step) may be used as a

screening method even by non-specialized laboratories equipped with simple and cheap instruments.

Because of technical problems, this laboratory was not able to participate in the third trial (12 months after irradiation).

**Table VII:** Rate of correct identifications reported by laboratory 9

	<b>Apples</b>	<b>Asparagus</b>	<b>Carrots</b>	<b>Leeks</b>	<b>Onions</b>
Trial N°1	70%	100%	100%	100%	100%
Trial N°2	70%	100%	100%	100%	ND
Trial N°3	ND	ND	ND	ND	ND
Trial N°4	ND	ND	ND	ND	ND
Trial N°5	100%	100%	100%	100%	100%

ND: not determined

Laboratory 4 observed the glow curves on a small liquid crystal display of the TL reader and glow curves could not be stored for documentation. The curves, therefore, were not easy to interpret. Nor did this laboratory print the results or stored them on a disc. Thus, results could not be re-interpreted.

Documentation of glow curves is very helpful for the classification of the samples. In fact, a look at the glow curves is always a good means of determining whether a sample has been irradiated or not. The glow curves (Annex A) show that, for all species of fruit and vegetables studied, non-irradiated samples did not exhibit any TL peak at 220°C (Glow 1), whereas all the irradiated ones always exhibited a significant peak (Glow 1) at this temperature. The re-irradiation step (Glow 2) is useful to recognize false negative result (absence of peak in the Glow 1 curve at 220°C in an irradiated sample due to an insufficient quantity of purified silicates).

Tables X to XIV presented in Annex B summarize the results for all participating laboratories (except laboratory N°9) for each product and for each trial. Considering that trial 3 has to be excluded because of labelling errors, it is demonstrated that the rate of correct identification is always above 86 % (overall results). For controls (non-irradiated samples), the rate of correct identification is 87 - 93 % (89 - 96 % excluding the apple samples) whereas for irradiated samples correct decisions amount to 77 - 96 % (82 - 99 % excluding the apple samples). As for the apple samples the rate of correct identification increases (table VIII) after the first set of analysis (trial 1), it is obvious that the experience of the laboratories is a very important factor. More experience leads to better results, especially for samples containing low amounts of silicates.

It is also remarkable that only one 100 % correct identification has been found during the first trial (non-irradiated carrot samples). In the other trials, there were more 100 % correct identifications (three, six and five times, resp. during trials 2, 4 and 5). These better performances are no doubt due to an increasing experience of the participating laboratories.

Results obtained during the trial N°3 (twelve months after irradiation) are not presented in table VIII because of problems of mislabelling. The number of excluded samples (due to the MDL rejection criteria) is so high in case of apple samples for trial N°4 (fifteen months after irradiation) that the final number of reported results (five, one irradiated and four non-irradiated) is not sufficient to be considered for statistical evaluation. Therefore, the results presented in table VIII (apple samples, trial N°4) are not considered for this purpose.

**Table VIII:** Correct identification rates (in %) of samples analysed during trials N° 1, 2, 4 and 5.

	<b>Irradiated</b>	<b>Non-irradiated</b>	<b>Total</b>	<b>False negatives</b>	<b>False positives</b>
<b>Apples</b>					
trial N°1	40%	73%	60%	0	0
trial N°2	73%	83%	80%	0	0
trial N°4	0%	50%	40%	0	0
trial N°5	67%	93%	79%	0	0
<b>Asparagus</b>					
trial N°1	83%	96%	91%	0	0
trial N°2	100%	93%	96%	0	0
trial N°4	100%	100%	100%	0	0
trial N°5	100%	100%	100%	0	0
<b>Carrots</b>					
trial N°1	89%	100%	95%	0	0
trial N°2	96%	82%	90%	0	0
trial N°4	67%	67%	67%	0	0
trial N°5	92%	83%	88%	2	0
<b>Leeks</b>					
trial N°1	85%	96%	91%	0	0
trial N°2	100%	96%	98%	0	0
trial N°4	100%	100%	100%	0	0
trial N°5	100%	92%	96%	0	0
<b>Onions</b>					
trial N°1	70%	92%	82%	2	2
trial N°2	100%	83%	92%	0	2
trial N°4	100%	100%	100%	0	0
trial N°5	100%	100%	100%	0	0

The remaining 8 wrong results (4 false negatives and 4 false positives) observed in the case of onion and carrot samples are a consequence of labelling errors during the trial 1 (2 false negatives and 2 false positives with onion samples), trial 2 (2 false positives with onion samples) and trial 5 (2 false negatives with carrot samples). A check of the glow curves confirmed the results reported by the laboratories concerned.

**Table IX: Overall results of the interlaboratory test (trials 1, 2, 4 and 5)**

		<b>Irradiated</b>	<b>False negatives</b>	<b>Non-irradiated</b>	<b>False positives</b>
Total number of samples analysed:	765	374	4	391	4
Sample number after rejections:	692	340	4	352	4
Correctly classified	625	305	4	320	4
Number of samples in doubt	59	31		28	
Correct results (%):	90.3%	89.7%		90.9%	

The overall results presented in table IX with 90.3 % correct results confirmed that the method as described in the protocol EN 1788:1996 - with some minor modifications concerning the weight of sample to be analysed - should be considered to be applicable to dehydrated irradiated fruit and vegetables. No problems due to fading of the signals by thermoluminescence were reported by the laboratories participating in the 24 months' trial. Thus, TL signals are stable enough; the method may be used over the whole storage time (at least 24 months).

Even in the case of samples containing very low amounts of silicates, as apple samples, identification successful. If the quantities of contaminating silicates are very low, it is important to apply the TL limits for Glow 2, and to reject any sample with Glow 2 lower than 10 times the MDL.

In this interlaboratory test, analyses were carried out in duplicate, and the result classified as in doubt if one of the two duplicates had to be rejected due to lack of minerals. If the results of the duplicates disagreed, they were classified as in doubt, too. This classification led to a rather high number of samples in doubt, (59 out of a total of 692 (8.5 %)). In practice, analyses would be repeated in these cases using



more sample material. Thereby, an increased rate of successful classification could be expected.

## CONCLUSION

Aim of this study was to extend the field of application of the TL method as described in the European Standard EN 1788:1996 to dried fruit and vegetables. Five species of dehydrated fruit and vegetables were selected (apple, carrot, leek, asparagus and onion). The samples were non-irradiated (controls) or irradiated at an average dose of about 8 kGy.

Nine laboratories in the European Union received a set of three samples of each of the five species, one month, six months, twelve months, fifteen months and twenty-four months after irradiation.

The overall detection on the basis of results reported by the participants after applying the MDL rejection criteria as described in EN 1788:1996 is higher than 90 % for all samples including the apple samples. As in practice more sample material will be available than in the present interlaboratory trial, higher identification rates may be expected.

The overall result of this collaborative study has shown that radiation treatment of the five studied dehydrated fruit and vegetables may be identified during the whole shelf-life of the food investigated. It has been proposed, therefore, to extend the applicability of the TL method as described in the European Standard EN 1788:1996 to dried fruit and vegetables. The proposal has been approved by CEN/TC 275/WG8, and CEN/TC 275 has accepted the extension of the field of application of the method. The protocol has already been revised and will probably be adopted in the year 2000.

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## Annex D

### Protocol used by the participating laboratories

# MATERIALS AND METHODS

## 1 - MATERIALS

### 1.1. Chemicals

- Distilled or deionised H<sub>2</sub>O (if possible filtered through 0.45 µm)
- Polytungstate solution (Na<sub>6</sub>W<sub>12</sub>O<sub>39</sub>\*2H<sub>2</sub>O) ; adjusted with H<sub>2</sub>O to a density of 2.0 g.ml<sup>-1</sup>
- 1 M HCl
- 1 M NH<sub>4</sub>OH
- Acetone
- Silicone spray or carboxymethyl cellulose (0.2 % in water)
- Ethanol or methanol for cleaning the discs
- Nitrogen (purity 99.999 %) as inert gas (for flushing the TL measuring chamber)

### 1.2. Equipment

- Beaker, 1000 ml - PE jet bottles for H<sub>2</sub>O and acetone
- Nylon sieves (250 µm)
- Pasteur pipettes
- Centrifuge glass tubes, 50 ml and 15 ml
- Vortexer for centrifuge glass tubes
- Ultrasonic bath
- Centrifuge with swing-out rotor, at least 1000 g ( $g = 9.81 \text{ ms}^{-2}$ )
- Stainless steel discs (diameter 0.96 – 0.97 cm x ca. 0.5 mm)
- Laboratory oven (temperature needed : 50°C)
- TL reader with the possibility to store glow curves and to integrate in different temperature areas.

### 1.3. Food samples

5 species of dehydrated food are used :

- thin slices of onions
- thin slices of carrots
- asparagus powder
- thin slices of leeks
- small cubes of apples.

For each species of food, three coded samples and one irradiated reference will be dispatched to each participating laboratory. Each sample will be sent in duplicate in order to present enough material for independent analyses. The food samples are packed in two individual plastic bags of about 100 g for vegetables and 200 g for apples.

## 2 - METHODS

### Remarks

All the considerations concerning Good Laboratory Practice are considered as known and are not included in this text (stability of photomultiplier sensitivity, temperature calibration, etc...).

The minerals should be isolated as clean as possible, without any organic impurities. A microscopic (binocular) analysis can be useful in order to check the purity and the quantity of the minerals on the disc.

The samples should not be unnecessarily exposed to strong light, and should be stored in the dark.

A minimum of 0.2 mg of minerals should be isolated.

Don't use more than 3 mg of minerals in order to avoid saturation of the photomultiplier.

All equipment in contact with the samples should be carefully cleaned and stored under dust free conditions. Stainless steel discs must be cleaned carefully by washing them several times in a beaker with water and treatment with ultrasound. After washing them twice in acetone, they should be dried in a laboratory oven and stored under dust free conditions.

## **2. 1. Analysis of the food samples**

Each sample has to be examined in two independent analyses. It is suggested that the analyses be performed on two different days. Parallel to each analysis series a full process blank has to be performed. Isolation of minerals should be performed within four weeks after receipt of samples. Storage in the dark is allowed (in the original packaging for example) at room temperature.

## **2.2. Isolation of silicate minerals from food**

### **2.2.1. Preconcentration step of minerals**

Preconcentration of minerals by wet sieving is recommended for most samples using the following procedure:

Suspend 50 g of dehydrated sample (depending on the degree of mineral contamination) in a 1000 ml glass beaker with 700 ml water added. It is advisable to cut with scissors the bottom of the bag and leaving the material to flow down.

\* For dehydrated apples :

- Increase the weight up to 100 g by using a second beaker.
- Treat the sample in the beaker with ultrasound for about 15 min (to shake loose the adhering minerals).
- Leave the sample to decant for a few minutes and remove the floating fruits.

\* For sliced dried vegetables (onions, carrots and leeks) :

- Stir with a glass rod.
- Treat the sample in the beaker with ultrasound for about 15 min. Sieve the sample (onions, carrots, leeks and apples) in portions through a 250  $\mu\text{m}$  nylon mesh into a large beaker (e.g. 500 ml to 1000 ml), rinsing the minerals through with water each time e.g. using a strong jet of water from a wash bottle. Discard constituents retained by the sieve cloth. Use a fresh nylon sieve cloth for each sample.

\* For asparagus powder :

- Treat the sample in the beaker with ultrasound for about 15 min
- Stir slowly with a glass rod in order to maintain the organic fraction in suspension
- Vacuum extract the aqueous suspension - Reduce the volume to a few tens of millilitres
- Rinse the minerals with approximately 100 ml of water. The minerals are now in the large beaker (in the case of finely ground samples fairly large amounts of organic material will also have passed the sieve) and are allowed to settle for about 5 min.
- Decant most of the water from the large beaker together with as much organic material as possible, leaving the minerals in only a few tens of millilitres of water. If there are still fairly large amounts of

organic material left, add water to a depth of about 1 cm to 2 cm, swirl, wait for about 5 s to 10 s to let the minerals settle again and then decant again. Repeat this step until only small amounts of organic material are left together with the minerals.

- Transfer the mineral fraction to a centrifuge tube (50 ml), e.g. using a Pasteur pipette.
- Centrifuge for 1 min at 1000 g, alternatively allow sedimentation for 5 min.
- Decant off or vacuum extract the water, leaving the mineral fraction behind.

### 2.2.2. Density separation step to free the minerals from organic material

- To the mineral fraction in the centrifuge tube add 10 to 15 ml of  $2 \text{ g.ml}^{-1}$  sodium polytungstate solution. Shake vigorously (Vortex) and agitate in an ultrasonic bath for about 3 min
- Centrifuge for 2 min at 1000 g. Silicate minerals (density  $2.5 \text{ g.ml}^{-1}$  to  $2.7 \text{ g.ml}^{-1}$ ) will sediment whereas organic components will float.
- Fill up the tube with water to facilitate removal of the organic material. Extract the upper water layer and the organic material either by decantation or vacuum suction, leaving the minerals behind in the low polytungstate layer. If necessary, clean the tube side by wiping with a small moist tissue. If not all organic material is removed, fill the tube with water again and repeat extraction.
- Now extract the sodium polytungstate layer, being careful to leave the mineral fraction behind. If too much organic material is still present, again add sodium polytungstate solution and repeat the procedure with agitation in an ultrasonic bath if necessary.
- The sodium polytungstate solution may be collected and purified for re-use.
- Wash the minerals twice to remove the tungstate residues by filling the tube with water, allow the minerals to settle or centrifuge shortly at 1000 g and remove the water.
- To dissolve carbonates adhering to the silicate minerals, add 1 ml to 2 ml of 1 M hydrochloric acid solution, agitate, and leave for 10 min in the dark.
- Neutralise the acid using 1 M ammonium hydroxide solution, fill up the tube with water, allow the minerals to settle or centrifuge shortly. Remove the water and wash the mineral residue twice with water.
- To displace the residual water, add about 3 ml of acetone and agitate. If the acetone becomes turbid, remove it and add new acetone.

### **2.3. Fixing the minerals on discs for TL measurement**

- Carefully clean stainless steel discs e.g. by rinsing in water, ultrasonic agitation, several washings in acetone, a second ultrasonic treatment, drying in an oven, and storage under dust free conditions. (The cleaning procedure may be checked as described in Annex D1).
- Weigh the empty disc.
- Transfer the isolated minerals in acetone to a disc using a Pasteur pipette. After suction of the mineral suspension into the pipette, the minerals will immediately sediment to the outlet of the pipette and can then be easily transferred dropwise (allow the acetone to evaporate in between) in an adequate amount to the disc. The discs are subsequently stored overnight at  $50^{\circ}\text{C}$  in a laboratory oven.



- Weigh the disc and calculate the mass of minerals.
- As an alternative to dropping minerals on the disc, transfer the mineral suspension in acetone to one or a set of clean flat bottomed tubes each containing a clean stainless steel disc. Place these tubes upright in laboratory oven at 50°C overnight. The acetone will dry off, leaving a deposit of minerals adhering to the discs.
- The deposit of minerals may be fixed on the disc by using silicone spray or by layering a drop of 0.2 % carboxy methyl cellulose.
- Leave the discs overnight at 50°C.

## 2.4. Thermoluminescence (TL) measurements

### 2.4.1 General

- For comparison of different analyses, identical measuring conditions should be assured. Measure the background integrated TL intensity regularly and ensure that it remains at the same level. Clean the infra-red barrier filter and the heating plate (planchet) regularly with ethanol.
- To reduce spurious TL in the presence of oxygen, flush the TL heating chamber with nitrogen at a constant flow rate during each measurement.

### 2.4.2. Measurement conditions

- The following instrument settings have been found satisfactory, e.g.
  - . initial temperature : 70°C
  - . heating rate : 6°C/s
  - . final temperature : 350°C to 500°C the measuring chamber should be flushed by nitrogen.

### 2.4.3. Measurement of Glow 1

- Place the disc with the mineral deposit (as prepared in 2.3) on the heating plate of the TL reader, and glow it under the specified conditions (2.4.2.). Store the glow curve (Glow 1) obtained on a data medium.

### 2.4.4. Irradiation for the purpose of normalisation

- After measurement of Glow 1, irradiate the discs with the mineral deposit with a defined radiation dose of 1 kGy using a suitable radiation source.
- The applied radiation dose for normalization should be controlled by adequate dosimetry.
- The discs should be packed in a manner which protects them from loss of material, exposure to light or cross contamination. It is essential that the minerals on the discs to be irradiated and subsequently measured for Glow 2 are identical to the minerals measured during Glow 1. If significant loss of minerals occurs, the discs should be rejected. This may be checked by visual inspection of the packaging or by weighing of the discs.
- After irradiation of the discs, store them overnight at 50°C before recording Glow 2.

### 2.4.5 Measurement of Glow 2

- Measure Glow 2 under the same conditions as Glow 1 (2.4.3.).

#### 2.4.6. Estimation of MDL

- Measure full process blank levels in parallel with sample extractions using portions of the same stock solutions and following the procedure in all stages. Calculate the MDL according to Annex D1.

#### 2.4.7. TL limits for Glow 2

- Reject any sample with a Glow 2 lower than 10 times the MDL, evaluated over a stated temperature interval, and repeat the analysis using a larger amount of minerals. In that case no assessment can be made of whether irradiation treatment of the food product has occurred.

- If the TL of Glow 2 approximates the counting saturation limit, reject the sample and repeat the analysis using a smaller amount of minerals. Alternatively, a restrictive aperture or a neutral density filter to reduce count rate may be effective.

### 3 - Evaluation

Base the identification of irradiated foods by TL analysis on the value of the TL glow ratio, evaluated over a recommended temperature interval. In addition, shapes of glow curves offer support for identification.

The recommended temperature interval for evaluation of the TL glow ratio is in the range of 150°C to 250°C. The absolute temperature scale can be determined in the TL reader using calibrated thermocouples. A temperature interval comprising  $\pm 10$  K up to  $\pm 40$  K around the optimal temperature may be chosen.

Calculate the integral of Glow 1 and Glow 2 over the recommended temperature interval. Reject any sample with a value of Glow 2 integral 10 times below MDL in the chosen temperature interval.

Calculate the TL glow ratio over the chosen temperature interval (by dividing the integral of Glow 1 by that of Glow 2).

TL glow ratios from irradiated samples are typically greater than 0.5, whereas those from unirradiated samples are generally below 0.1.

If TL glow ratios between 0.1 and 0.5 are obtained, interpretation of the shape of the glow curves is needed to decide whether the samples has been irradiated or not. Usually, Glow 1 curves of irradiated foodstuffs exhibit a maximum between 150°C up to 250°C, whereas low level natural radioactivity causes TL signals in the deep traps above 300°C

### **Annex D1**

It should be ascertained that stainless steel discs, glasswares and reagents are free from particulate contamination. As a general control, a full process blank is carried through the procedure. Integrated TL intensity of the first glow of the blank plus three standard deviations defines the MDL.

As a further control, the cleanliness of the employed stainless steel discs might be checked by irradiation of the cleaned discs with e.g. a radiation dose of 1 kGy, and recording of integrated TL intensities. Discs with TL levels more than 3 standard deviations above the mean blank level indicate surface contamination.

If the discs are not completely clean, diverging results depending on the kind of contamination may be

obtained. (Most frequently, just unirradiated dust grains will cause contamination, leading to false negative results, since the normalization procedure contains a radiation step, by which the contaminating dust on the discs will be excited and contribute to Glow 2).

If the full process blank shows integrated TL intensities greater than three standard deviations above the mean clean disc blank level, contamination of glassware or reagents is indicated and needs to be checked and eliminated.

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