

Evaluation Report

proficiency test

DLA ptRE02 (2021)

Detection of Food Irradiation:

Spices (ground) and Herbs (rubbed)

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Vertraulichkeit Confidentiality	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

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1. Introduction

The participation in proficiency testing schemes (PT) is an essential element of the quality-management-system of every laboratory testing food and feed, cosmetics and food contact materials. The implementation of proficiency tests enables the participating laboratories to prove their own analytical competence under realistic conditions. At the same time they receive valuable data regarding the verification and/or validation of the particular testing method [1, 5].

The purpose of DLA is to offer proficiency tests for selected parameters in concentrations with practical relevance.

Realisation and evaluation of the present proficiency test follows the technical requirements of DIN EN ISO/IEC 17043 (2010) and DIN ISO 13528:2009 / ISO 13528:2015 [2, 3].

2. Realisation

2.1 Test material

Four PT samples were provided for the qualitative verification of the irradiation. These are two different samples each of a spice mixture (ground) and a herb mixture (rubbed) made from commercially available products. Only one of the two samples was treated with ionizing radiation (range $0,5-10~\mathrm{kGy}$). For this purpose, a basic mixture was first prepared for the spice samples and the herb samples. After homogenization, an aliquot of the batches was treated in a gamma irradiation facility with a dose of $1~\mathrm{kGy}$.

After renewed homogenisation, the samples were portioned to approximately 40 g into metallised PET film bags.

Table 1 shows the composition of the PT samples.

<u>Table 1:</u> Composition of DLA samples

DLA Sample	Ingredients	Irradiation
Sample A	Herbs de Provence, herbal mixture Ingredients: rosemary, basil, marjoram	1 kGy
Sample B	Herbs de Provence, herbal mixture Ingredients: rosemary, basil, marjoram	-
Sample C	Curry, spice preparation Ingredients: Ground mustard, cumin, coriander, fenugreek, fennel, turmeric, table salt, cayenne pepper, celery seeds, onion, garlic	-
Sample D	Curry, spice preparation Ingredients: Ground mustard, cumin, coriander, fenugreek, fennel, turmeric, table salt, cayenne pepper, celery seeds, onion, garlic	1 kGy

Note: The metrological traceability of temperature, mass and volume during production of the PT samples is ensured by DAkkS calibrated reference materials.

2.1.1 Homogeneity

The qualitative detection of the irradiation of samples A and D was carried out using a duplicate determination by photostimulated luminescence (PSL). The deviations of the individual measured values from the mean were 5.5% and 14%. The homogeneity was thus considered sufficient for a qualitative proficiency test.

2.1.2 Stability

A water activity (a_W) of < 0,5 is an important factor to ensure the stability of dry or dried products during storage. Optimum conditions for storage is the a_W value range of 0,15-0,3. In this range the lowest possible degradation rate is to be expected [16].

The experience with various DLA test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_W value <0,5).

The a_W value of the PT samples was approx. 0,17 - 0,19 (20-21°C). The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

The portions of the test materials (sample A to D) were sent to every participating laboratory in the $43^{\rm rd}$ week of 2021. The testing method was optional. The tests should be finished at December $24^{\rm th}$ 2021 the latest.

With the cover letter along with the sample shipment the following information was given to participants:

There are two different samples each of a mixture of Spices (ground) and a mixture of herbs (rubbed). One of the two samples was treated with ionizing radiation (range 0.5--10~kGy). Suitable analytical detection methods are photostimulated luminescence (PSL) and thermoluminescence (TL).

Please note the attached information on the proficiency test. (see documentation, section 5.2 Information on the PT)

2.3 Submission of results

The participants submitted their results in standard forms, which have been sent by email. The results given as positive/negative were evaluated.

Queried and documented were the indicated results and details of the test methods and hints about the procedure.

In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

All 10 participants submitted at least one result.

3. Qualitative Evaluation

The evaluation of the present proficiency test was done exclusively qualitative.

In the results section, the results are presented separately according to the respective methods, photostimulated luminescence (PSL) and thermoluminescence (TL).

3.1 Agreement with consensus values from participants

The qualitative evaluation of the results of each participant was based on the agreement of the indicated results (positive or negative) with the **consensus values from all participants**. A consensus value is determined if $\geq 75\%$ positive or negative results are available for a parameter. The assessment will be in the form that the number of matching results followed by the number of samples for which a consensus value was obtained is indicated. Behind that the agreement is expressed as the percentage in parentheses.

3.2 Agreement with the irradiation of samples

The qualitative evaluation of the results of each participant was based on the agreement of the indicated results (positive or negative) with the irradiation of the four PT-samples.

The assessment will be in the form that the number of matching results followed by the number of samples is indicated. Behind that the agreement is expressed as the percentage in parentheses.

4. Results

All following tables are anonymized. With the delivering of the evaluation report the participants are informed about their individual evaluation number.

The participant results and evaluation are tabulated as follows:

Evaluation number	Sample A	Sample B	Sample C	Sample D	Qualitative Valuation	Qualitative Valuation	Method	Remarks
	pos/neg	pos/neg	pos/neg	pos/neg	Agreement with	Agreement with irradia-		

	Sample A	Sample B	Sample C	Sample D
Number positive				
Number negative				
Percent positive				
Percent negative				
Consensus value				
Irradiation				

4.1 Proficiency Test Photostimulated Luminescence (PSL)

Qualitative valuation of results

Evaluation number	Sample A	Sample B	Sample C	Sample D	Qualitative Valuation	Qualitative Valuation	Method	Remarks
	pos/neg	pos/neg	pos/neg	pos/neg	Agreement with consensus value	Agreement with irradia- tion of samples		
1								
2	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 2
3	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 2
4								
5	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 2
6	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 6
7	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 2
8	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 2
9								
10	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	PSL	Analysis: 2

	Sample A	Sample B	Sample C	Sample D
Number positive	7	0	0	7
Number negative	0	7	7	0
Percent positive	100	0	0	100
Percent negative	0	100	100	0
Consensus value	positive	negative	negative	positive
Irradiation	positive	negative	negative	positive

Methods:

TL = Thermoluminescence

PSL = Photostimulated Luminescence

Comment:

The consensus values of the results are in qualitative agreement with the irradiation of samples A and D. No false positive or false negative results were obtained.

Detailed information from the participants on the methods and measured values can be found in the documentation.

4.2 Proficiency Test Thermoluminescence (TL)

Qualitative valuation of results

Evaluation number	Sample A	Sample B	Sample Sample Qualitative Qualitative Valuation		-40.0	Method	Remarks	
	pos/neg	pos/neg	pos/neg	pos/neg	Agreement with consensus value	Agreement with irradia- tion of samples		
1	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1+2
2								
3	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1+2
4	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1+2
5	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1+2
6	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1+2
7	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1+2
8	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1
9	positive	negative	negative	positive	4/4 (100%)	4/4 (100%)	TL	Analysis 1
10	positive			positive	2/2 (100%)	2/2 (100%)	TL	Analysis 1+2

	Sample A	Sample B	Sample C	Sample D
Number positive	9	0	0	9
Number negative	0	8	8	0
Percent positive	100	0	0	100
Percent negative	0	100	100	0
Consensus value	positive	negative	negative	positive
Irradiation	positive	negative	negative	positive

Methods:

TL = Thermoluminescence

PSL = Photostimulated Luminescence

Comment:

The consensus values of the results are in qualitative agreement with the irradiation of samples A and D. No false positive or false negative results were obtained.

Detailed information from the participants on the methods and measured values can be found in the documentation.

5. Documentation

Note: Information given in German was translated by DLA to the best of our knowledge (without guarantee of correctness).

5.1 Details by the participants

Photostimulated Luminescence: Sample A

Photostimulated Luminescence (PSL)	Evaluation number	Unit	Date of Analysis	Final result (qualitative)	Final result (count)	Number of determinations		Analysis	Analysis (optional)	Analysis (optional)	Analysis (optional)	Analysis (optional)	Dose for calibration	Dark count
		count	day/month	postive / intermediate / negative		n = 1 to 6	1	2	3	4	5	6	kGy	
Sample A not irradiated (original PSL)	2	Signal	06. Dez	positive		2	25764	27118						
Sample A after irradiated (calibrated PSL)	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Sample A not irradiated (original PSL)	3	counts/60s	17.11.21	positive	positive	2	18066	15510						27
Sample A after irradiated (calibrated PSL)	3	counts/60s	18.11.21	positive		2	27383	28356					1	
Sample A not irradiated (original PSL)	5	Counts/20s	29.10.21	positive		1	10565	11767						4402
Sample A after irradiated (calibrated PSL)	5	Counts/20s	03.11.21	positive		1	11384	11830					1kGy	
Sample A not irradiated (original PSL)	6	counts/20sec	05.11.21	positive	6164	6	5202	5851	5892	6962	6133	6944		0
Sample A after irradiated (calibrated PSL)	6													
Sample A not irradiated (original PSL)	7	counts	16.11.	positive	40596	2	37930	43262						37
Sample A after irradiated (calibrated PSL)	7													
Sample A not irradiated (original PSL)	8	counts	08.11.21	positive	11621,5	2	12529	10714						-
Sample A after irradiated (calibrated PSL)	8													
Sample A not irradiated (original PSL)	10	?	13.12.21	Sample A = positive (value without test irraciation = positive)	12824	2	12870	12777						36
Sample A after irradiated (calibrated PSL)	10	?	15.12.21	Sample A = positive (value after test irradiation = positive)	12262	2	13901	10623					1	

Photostimulated Luminescence (PSL)	Evaluation number	Negative control (count)	Positive control (count)	Threshold T1	Threshold T2	detection	Method description, like in a analysis report / norm / literature		Irradiation source for calibration	Method accredited according to ISO/IEC 17025	Further remarks
						kGy	ASU L 00.00-82 / EN 13751 (2009)			yes / no	
Sample A not irradiated (original PSL)	2			500	1500			PSLfood	NA	no	
Sample A after irradiated (calibrated PSL)	2										
Sample A not irradiated (original PSL)	3	334 / 459	68513 / 574747	700	5000	1	DIN EN 13751:2009-11	SUERC PPSL Irradiated Food Screening System	3 Cs137 sources	yes	
Sample A after irradiated (calibrated PSL)	3										
Sample A not irradiated (original PSL)	5	Counts/20s	Counts/20s	700	1500		yes	PSLFood	Xray	yes	
Sample A after irradiated (calibrated PSL)	5										
Sample A not irradiated (original PSL)	6	153	121742	500	1000	0,5-1	ASU L 00.00-82 / EN 13751 (2009)	RadPro PSL Food		yes	
Sample A after irradiated (calibrated PSL)	6										
Sample A not irradiated (original PSL)	7	296	17949	700	5000		ASU L 00.00-82 / EN 13751 (2009)	PSL-Reader SURRC PPSL, Serial-No.: 0100		yes	
Sample A after irradiated (calibrated PSL)	7										
Sample A not irradiated (original PSL)	8	0	23804	700	5000	1	EN 13751	RadPro PSL food		yes	
Sample A after irradiated (calibrated PSL)	8										
Sample A not irradiated (original PSL)	10	225	281391	520	2000	depends on amounts of minerals of sample	ASU L 00.00-82 / EN 13751 (2009)	Surrc PPSL	X-ray	yes	60 Cycles / measurement
Sample A after irradiated (calibrated PSL)	10										

Photostimulated Luminescence: Sample B

Photostimulated Luminescence (PSL)	Evaluation number	Unit	Date of Analysis	Final result (qualitative)	Final result (count)	Number of determinations		Analysis	Analysis (optional)	Analysis (optional)	Analysis (optional)	Analysis (optional)	Dose for calibration	Dark count
		count	day/m onth	postive / intermediate / negative		n = 1 to 6	1	2	3	4	5	6	kGy	
Sample B not irradiated (original PSL)	2	Signal	06. Dez	negative		2	0	0						
Sample B after irradiated (calibrated PSL)	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Sample B not irradiated (original PSL)	3	counts/60s	17.11.21	negative	negative	2	183	265						
Sample B after irradiated (calibrated PSL)	3	counts/60s	18.11.21	positive		2	26794	18529					1	
Sample B not irradiated (original PSL)	5	Counts/20s	29.10.21	negative		1	52	0						
Sample B after irradiated (calibrated PSL)	5	Counts/20s	03.11.21	positive		1	13277	13225					1kGy	
Sample B not irradiated (original PSL)	6	counts/20sec	05.11.21	negative	0	6	0	0	0	0	0	0		
Sample B after irradiated (calibrated PSL)	6													
Sample B not irradiated (original PSL)	7	counts	16.11.	negative	346	2	298	395						
Sample B after irradiated (calibrated PSL)	7													
Sample B not irradiated (original PSL)	8	counts	08.11.21	negative	0	2	0	0						
Sample B after irradiated (calibrated PSL)	8													
Sample B not irradiated (original PSL)	10	?	13.12.21	Sample B = negative (value without test irraciation = negative)	297	2	202	391						
Sample B after irradiated (calibrated PSL)	10	?	15.12.21	Sample B = negative (value after test irradiation = positive)	17824	2	29540	6107					1	

Photostimulated Luminescence: Sample C

Photostimulated Luminescence (PSL)	Evaluation number	Unit	Date of Analysis	Final result (qualitative)	Final result (count)	Number of determinations		Analysis	Analysis (optional)	Analysis (optional)	Analysis (optional)	Analysis (optional)	Dose for calibration	Dark count
		count	day/month	postive / intermediate / negative		n = 1 to 6	1	2	3	4	5	6	kGy	
Sample C not irradiated (original PSL)	2	Signal	06. Dez	negative		2	0	0						
Sample C after irradiated (calibrated PSL)	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Sample C not irradiated (original PSL)	3	counts/60s	17.11.21	negative	negative	2	301	383						27
Sample C after irradiated (calibrated PSL)	3	counts/60s	18.11.21	positive		2	9447813	6100560					1	
Sample C not irradiated (original PSL)	5	Counts/20s	29.10.21	negative		1	0	0						547
Sample C after irradiated (calibrated PSL)	5	Counts/20s	03.11.21	positive		1	3459271	4309744					1kGy	
Sample C not irradiated (original PSL)	6	counts/20sec	05.11.21	negative	31	6	0	0	187	0	0	0		0
Sample C after irradiated (calibrated PSL)	6													
Sample C not irradiated (original PSL)	7	counts	16.11.	negative	344	2	369	320						37
Sample C after irradiated (calibrated PSL)	7													
Sample C not irradiated (original PSL)	8	counts	08.11.21	negative	0	2	0	0						-
Sample C after irradiated (calibrated PSL)	8													
Sample C not irradiated (original PSL)	10	?	13.12.21	Sample C = negative (value without test irraciation = negative)	235	2	196	274						36
Sample C after irradiated (calibrated PSL)	10	?	15.12.21	Sample C = negative (value after test irradiation = positive)	3690824	2	3086333	4295314					1	

Photostimulated Luminescence (PSL)	Evaluation number	Negative control (count)	Positive control (count)	Threshold T1			Method description, like in a analysis report / norm / literature	Apparatus (PSL)	Irradiation source for calibration	Method accredited according to ISO/IEC 17025	Further remarks
						kGy	ASU L 00.00-82 / EN 13751 (2009)			yes / no	
Sample C not irradiated (original PSL)	2			500	1500			PSLfood	N/A	no	
Sample C after irradiated (calibrated PSL)	2										
Sample C not irradiated (original PSL)	3	334 / 459	68513 / 574747	700	5000	1	DIN EN 13751:2009-11	SUERC PPSL Irradiated Food Screening System	3 Cs137 sources	yes	
Sample C after irradiated (calibrated PSL)	3										
Sample C not irradiated (original PSL)	5	Counts/20s	Counts/20s	700	1500		yes	PSLFood	Xray	yes	
Sample C after irradiated (calibrated PSL)	5										
Sample C not irradiated (original PSL)	6	153	121742	500	1000	0,5-1	ASUL 00.00-82 / EN 13751 (2009)	RadPro PSL Food		yes	
Sample C after irradiated (calibrated PSL)	6										
Sample C not irradiated (original PSL)	7	296	17949	700	5000		ASU L 00.00-82 / EN 13751 (2009)	PSL-Reader SURRC PPSL, Serial-No.: 0100		yes	
Sample C after irradiated (calibrated PSL)	7										
Sample C not irradiated (original PSL)	8	0	17661	700	5000	1	EN 13751	RadPro PSL food		yes	
Sample C after irradiated (calibrated PSL)	8										
Sample C not irradiated (original PSL)	10	225	281391	520	2000	depends on amounts of minerals of sample	ASU L 00.00-82 / EN 13751 (2009)	Surrc PPSL	X-ray	yes	60 Cycles / measurement
Sample C after irradiated (calibrated PSL)	10										

Photostimulated Luminescence: Sample D

Photostimulated Luminescence (PSL)	Evaluation number	Unit	Date of Analysis	Final result (qualitative)	Final result (count)	Number of determinatio		Analysis	Analysis (optional)	Analysis (optional)	Analysis (optional)	Analysis (optional)	Dose for calibration	Dark count
		count	day/m onth	postive / intermediate / negative		n = 1 to 6	1	2	3	4	5	6	kGy	
Sample D not irradiated (original PSL)	2	Signal	06. Dez	positive		2	1556343	1486678						
Sample D after irradiated (calibrated PSL)	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Sample D not irradiated (original PSL)	3	counts/60s	17.11.21	positive	positive	2	1456610	1649132						
Sample D after irradiated (calibrated PSL)	3	counts/60s	18.11.21	positive		2	5373187	3864934					1	
Sample D not irradiated (original PSL)	5	Counts/20s	29.10.21	positive		1	980420	919371						
Sample D after irradiated (calibrated PSL)	5	Counts/20s	03.11.21	positive		1	3814613	2989705					1kGy	
Sample D not irradiated (original PSL)	6	counts/20sec	05.11.21	positive	487635	6	477063	439213	530578	458649	485173	535135		
Sample D after irradiated (calibrated PSL)	6													
Sample D not irradiated (original PSL)	7	counts	16.11.	positive	5494097	2	3902246	7085948						
Sample D after irradiated (calibrated PSL)	7													
Sample D not irradiated (original PSL)	8	counts	08.11.21	positive	2216191,5	2	2454857	1977526						
Sample D after irradiated (calibrated PSL)	8													
Sample D not irradiated (original PSL)	10	?	13.12.21	Sample D = positive (value without test irraciation = positive)	2654482	2	2937217	2371746						
Sample D after irradiated (calibrated PSL)	10	?	15.12.21	Sample D = positive (value after test irradiation = positive)	5667140	2	5611996	5722284					1	

Thermoluminescence: Sample A

Thermoluminescence (TL)	Evaluation number	Date of Analysis	Final result (qualitative)	Tempera- ture peak Glow curve	(Glow curve 1)	TL 2 (Glow curve 2)	Units of TL1 and 2	TL Ratio	Dose for normalization	Limit of detection (MDL)	Initial temperature	Heat rate	Final temperature	Relevant temperature range
		day/month	postive / negative	°C					kGy		°C	°C/sec	°C	°C
Sample A (for TL) Analysis 1	1	05.11.21	positive	600	87,47	789	nC	0,1109	1		70	5	300	100-600
Sample A (for TL) Analysis 2	1	10.11.21	positive	600	164,8	1026	nC	0,1606	1		70	5	300	100-600
Sample A (for TL) Analysis 1	3	15.11.21	positive	175	176349	404768	Photons	0,4357	1	474	35	6K/sec	350	110-210
Sample A (for TL) Analysis 2	3	15.11.21	positive	175	779106	2416767	Photons	0,3224	1	474	35	6K/sec	350	110-210
Sample A (for TL) Analysis 1	4	03.11.21	positive	150-250	3311	491	nC	6,74338086	1,08	6 nC	70	6	300	150-250
Sample A (for TL) Analysis 2	4	23.11.21	positive	150-250	2776	2374	nC	1,16933446	1,01	6 nC	70	6	300	150-250
Sample A (for TL) Analysis 1	5	08.11.21	Positive	208°C	597,7	921,9	nC	0,648	1 kGy		70°C	6°C/sec	400	40-57°C
Sample A (for TL) Analysis 2	5	08.11.21	Positive	202°C	1481	2691	nC	0,55	1 kGy		70°C	6°C/sec	400	40-57°C
Sample A (for TL) Analysis 1	6	11.11.21	positive	175	14236613	16682003	counts	0,853	1	2000	60	6	400	173-233
Sample A (for TL) Analysis 2	6	11.11.21	positive	175	17169928	18322546	counts	0,937	1	2000	60	6	400	173-233
Sample A (for TL) Analysis 1	7	1820.11.	positive	199	6217122	19742730	counts	0,315	1	451	70	6	400	179-250
Sample A (for TL) Analysis 2	7	1820.11.	positive	278	6200053	22644434	counts	0,274	1	451	70	6	400	179-250
Sample A (for TL) Analysis 1	8	22.12.21	positive	240	553,86	1540,79	nC/mg	0,3595	1		70	6	350	70-350
Sample A (for TL) Analysis 2	8	22.12.21	positive	210	691,23	1314,71	nC/mg	0,5258	1		70	6	350	70-350
Sample A (for TL) Analysis 1	9	01.12.21	positive	172	304762	560333	Counts	0,544	1		70	6	400	155-215
Sample A (for TL) Analysis 2	9													
Sample A (for TL) Analysis 1	10	15.12.21	positive	240	15361578	9639385	?	1,59	1	22579	70	6	450	185 - 249
Sample A (for TL) Analysis 2	10	15.12.21	positive	200	11937283	12475094	?	0,957	1	22579	70	6	450	185 - 249

Thermoluminescence (TL)	Evaluation number	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Apparatus (TL)	Irradiation source for normalization	Method accredited according to ISO/IEC 17025	Further remarks
		ASU L 00.00-43 / EN 1788 (2002)	Sample weight / Method of isolation + amount of minerals			yes / no	
Sample A (for TL) Analysis 1	1	ASU L 00.00-43 2004-07 of official method collection according to §64 LFGB (German Food and Feed Code)	Sample wheight + method for isolation s. further remarks; amount of minerals: 20	Thermo Electron Corporation Harshaw TLD 3500	Gamma irradiation		Sample wheigt: complete sample for analysis 1 and 2; Method for isolation: according to applied method
Sample A (for TL) Analysis 2	1		amount of minerals: 20				
Sample A (for TL) Analysis 1	3	DIN EN 1788:2002-01	4,17 g sample wheight	Lexsygsmart (Freiberg Instruments)	3 Cs137 sources	yes	
Sample A (for TL) Analysis 2	3		9,37			yes	
Sample A (for TL) Analysis 1	4	EN 1788 (2002)	13,72g	Harshaw TLD 3500	Со	yes	
Sample A (for TL) Analysis 2	4		12			yes	
Sample A (for TL) Analysis 1	5	yes	10g/Tungsten;HCl/3-10 mg	HarshawTLD	Xray	yes	
Sample A (for TL) Analysis 2	5		10g/Tungsten;HCl/3-10 mg			yes	
Sample A (for TL) Analysis 1	6	ASU L 00.00-43 / EN 1788 (2002)	20g/ rinse out + physical separation	Risoe TL-DA-15	Sr90	yes	
Sample A (for TL) Analysis 2	6		20g/ rinse out + physical separation			yes	
Sample A (for TL) Analysis 1	7	ASUL 00.00-43 / EN 1788 (2002)	15 g sample wheight, 1,2 mg minerals	RISO DTU Denmark TL/OSL Reader Typ: DA-20, Serien- Nr.: 377-04/2014	Sr-90	yes	
Sample A (for TL) Analysis 2	7		15 g sample wheight, 2,0 mg minerals				
Sample A (for TL) Analysis 1	8	EN 1788	20 g sample wheight / 8.04 mg	Harshaw TLD 3500	Co-60	yes	
Sample A (for TL) Analysis 2	8		20 g SW / 14.93 mg			yes	
Sample A (for TL) Analysis 1	9		not determined	Lexyg Smart extended	X-ray radiation	yes	
Sample A (for TL) Analysis 2	9						
Sample A (for TL) Analysis 1	10	ASU L 00.00-43 / EN 1788 (2002)	10 g sample wheight / EN1788-8.2.1.1 / 3,2 mg minerals	TL/OSL-DA-20 from DTU Nutech	X-ray radiation	yes	TL-securing positive PSL-result
Sample A (for TL) Analysis 2	10		10 g sample wheight / EN1788-8.2.1.1 / 2,9 mg minerals			yes	TL-securing positive PSL-result

Thermoluminescence: Sample B

Thermoluminescence (TL)	Evaluation number	Date of Analysis	Final result (qualitative)	Tempera- ture peak Glow curve	(Glow curve 1)	TL 2 (Glow curve 2)	Units of TL1 and 2	TL Ratio	Dose for normalization	Limit of detection (MDL)	Initial temperature	Heat rate	Final temperature	Relevant temperature range
		day/month	postive / negative	°C					kGy		°C	°C/sec	°C	°C
Sample B (for TL) Analysis 1	1	05.11.21	negative	600	2,591	987	nC	0,0026	1		70	5	300	100-600
Sample B (for TL) Analysis 2	1	10.11.21	negative	600	1,85	988	nC	0,0019	1		70	5	300	100-600
Sample B (for TL) Analysis 1	3	15.11.21	negative	no Peak	668	537069	Photons	0,0012	1	474	35	6K/sec	350	110-210
Sample B (for TL) Analysis 2	3	15.11.21	negative	no Peak	1771	1023760	Photons	0,0017	1	474	35	6K/sec	350	110-210
Sample B (for TL) Analysis 1	4	03.11.21	negative	>250	43,75	175,6	nC	0,24914579	1,08	6 nC	70	6	300	150-250
Sample B (for TL) Analysis 2	4	23.11.21	negative	>250	23,48	2916	nC	0,00805213	1,01	6 nC	70	6	300	150-250
Sample B (for TL) Analysis 1	5	08.11.21	Negative	397°C	4,028	2180	nC	0,002	1 kGy		70°C	6°C/sec	400	40-57°C
Sample B (for TL) Analysis 2	5	08.11.21	Negative	397°C	4,713	2614	nC	0,002	1 kGy		70°C	6°C/sec	400	40-57°C
Sample B (for TL) Analysis 1	6	11.11.21	negative	345	11188	8991765	counts	0,001	1	2000	60	6	400	173-233
Sample B (for TL) Analysis 2	6	11.11.21	negative	360	4439	6780762	counts	0,001	1	2000	60	6	400	173-233
Sample B (for TL) Analysis 1	7	1820.11.	negative	332	1633	12991826	counts	0	1	451	70	6	400	179-250
Sample B (for TL) Analysis 2	7	1820.11	negative	305	2397	9407749	counts	0	1	451	70	6	400	179-250
Sample B (for TL) Analysis 1	8	22.12.21	negative	340	6,53	1263,9	nC/mg	0,0052	1		70	6	350	70-350
Sample B (for TL) Analysis 2	8	22.12.21	negative	350	13,66	5369,4	nC/mg	0,0025	1		70	6	350	70-350
Sample B (for TL) Analysis 1	9	01.12.21	negative	300	1358	1168022	Counts	0,001	1		70	6	400	155-215
Sample B (for TL) Analysis 2	9													
Sample B (for TL) Analysis 1	10													
Sample B (for TL) Analysis 2	10													

Thermoluminescence (TL)	Evaluation number	Method description, like in a analysis report / norm / literature	s Notes to sample preparation	Apparatus (TL)	Irradiation source for normalization	Method accredited according to ISO/IEC 17025	Further remarks
		ASU L 00.00-43 / EN 1788 (2002)	Sample weight / Method of isolation + amount of minerals			yes / no	
Sample B (for TL) Analysis 1	1		amount of minerals: 10				
Sample B (for TL) Analysis 2	1		amount of minerals: 20				
Sample B (for TL) Analysis 1	3		4,99			yes	
Sample B (for TL) Analysis 2	3		9,18			yes	
Sample B (for TL) Analysis 1	4		11,92g			yes	
Sample B (for TL) Analysis 2	4		9,66			yes	
Sample B (for TL) Analysis 1	5		10g/Tungsten;HCl/3-10 mg			yes	
Sample B (for TL) Analysis 2	5		10g/Tungsten;HCl/3-10 mg			yes	
Sample B (for TL) Analysis 1	6		20g/ rinse out + physical separation			yes	
Sample B (for TL) Analysis 2	6		20g/ rinse out + physical separation			yes	
Sample B (for TL) Analysis 1	7		15 g sample wheight, 1,5 mg minerals				
Sample B (for TL) Analysis 2	7		15 g sample wheight, 1,7 mg minerals				
Sample B (for TL) Analysis 1	8		20 g EW / 14.63 mg			yes	
Sample B (for TL) Analysis 2	8		20 g EW / 5.85 mg			yes	
Sample B (for TL) Analysis 1	9		not determined				
Sample B (for TL) Analysis 2	9						
Sample B (for TL) Analysis 1	10						calibrated PSL-result negative - no TL- securing necessary
Sample B (for TL) Analysis 2	10						calibrated PSL-result negative - no TL- securing necessary

Thermoluminescence: Sample C

Thermoluminescence (TL)	Evaluation number	Date of Analysis	Final result (qualitative)	Tempera- ture peak Glow curve	(Glow curve 1)		Units of TL1 and 2	TL Ratio	Dose for normalization	Limit of detection (MDL)	Initial temperature	Heat rate	Final temperature	Relevant temperature range
		day/month	postive / negative	°C					kGy		°C	°C/sec	°C	°C
Sample C (for TL) Analysis 1	1	05.11.21	negative	600	8,964	1264	nC	0,00709	1		70	5	300	100-600
Sample C (for TL) Analysis 2	1	05.11.21	negative	600	11,39	1356	nC	0,0084	1		70	5	300	100-600
Sample C (for TL) Analysis 1	3	15.11.21	negative	kein Peak	885	524634	Photons	0,0017	1	474	35	6K/sec	350	110-210
Sample C (for TL) Analysis 2	3	15.11.21	negative	kein Peak	765	879521	Photons	0,0009	1	474	35	6K/sec	350	110-210
Sample C (for TL) Analysis 1	4	03.11.21	negative	>250	40,24	3908	nC	0,01029683	1,08	6 nC	70	6	300	150-250
Sample C (for TL) Analysis 2	4	23.11.21	negative	>250	53,9	2578	nC	0,02090768	1,01	6 nC	70	6	300	150-250
Sample C (for TL) Analysis 1	5	08.11.21	Negative	397°C	2,638	738,5	nC	0,004	1 kGy		70°C	6°C/sec	400	40-57°C
Sample C (for TL) Analysis 2	5	08.11.21	Negative	397°C	4,21	1580	nC	0,003	1 kGy		70°C	6°C/sec	400	40-57°C
Sample C (for TL) Analysis 1	6	11.11.21	negative	385	28118	32016855	counts	0,001	1	2000	60	6	400	173-233
Sample C (for TL) Analysis 2	6	11.11.21	negative	340	6043	27403947	counts	0	1	2000	60	6	400	173-233
Sample C (for TL) Analysis 1	7	1820.11.	negative	281	7329	51124220	counts	0	1	451	70	6	400	179-250
Sample C (for TL) Analysis 2	7	1820.11.	negative	335	4246	29408905	counts	0	1	451	70	6	400	179-250
Sample C (for TL) Analysis 1	8	22.12.21	negative	310	37,93	376,31	nC/mg	0,1008	1		70	6	350	70-350
Sample C (for TL) Analysis 2	8													
Sample C (for TL) Analysis 1	9	01.12.21	negative	282	933	1063220	Counts	0,0009	1		70	6	400	155-215
Sample C (for TL) Analysis 2	9													
Sample C (for TL) Analysis 1	10													
Sample C (for TL) Analysis 2	10													

Thermoluminescence (TL)	Evaluation number	Method description, like in a analysi report / norm / literature	s Notes to sample preparation	Apparatus (TL)	Irradiation source for normalization	Method accredited according to ISO/IEC 17025	Further remarks
		ASU L 00.00-43 / EN 1788 (2002)	Sample weight / Method of isolation + amount or minerals	f		yes / no	
Sample C (for TL) Analysis 1	1	ASU L 00.00-43 2004-07 of official method collection according to §64 LFGB (German Food and Feed Code)	amount minerals: 15	Thermo Electron Corporation Harshaw TLD 3500	Gamma irradiation		Sample wheigt: complete sample for analysis 1 and 2; Method for isolation: according to applied method
Sample C (for TL) Analysis 2	1		amount minerals: 15				
Sample C (for TL) Analysis 1	3	DIN EN 1788:2002-01	6,34	Lexsygsmart (Freiberg Instruments)	3 Cs137 sources	yes	
Sample C (for TL) Analysis 2	3		10,15			yes	
Sample C (for TL) Analysis 1	4	EN 1788 (2002)	11,51g	Harshaw TLD 3500	Со	yes	
Sample C (for TL) Analysis 2	4		9,78			yes	
Sample C (for TL) Analysis 1	5	yes	10g/Tungsten;HCl/3-10 mg	HarshawTLD	Xray	yes	
Sample C (for TL) Analysis 2	5		10g/Tungsten;HCl/3-10 mg			yes	
Sample C (for TL) Analysis 1	6	ASU L 00.00-43 / EN 1788 (2002)	20g/ rinse out + physical separation	Risoe TL-DA-15	Sr90	yes	
Sample C (for TL) Analysis 2	6		20g/ rinse out + physical separation			yes	
Sample C (for TL) Analysis 1	7	ASU L 00.00-43 / EN 1788 (2002)	15 g sample wheight, 0,7 mg minerals	RISO DTU Denmark TL/OSL Reader Typ: DA-20, Serien- Nr.: 377-04/2014	Sr-90	yes	
Sample C (for TL) Analysis 2	7		15 g sample wheight, 0,4 mg minerals				
Sample C (for TL) Analysis 1	8	EN 1788	40 g SW / 15.02 mg	Harshaw TLD 3500	Co-60	yes	
Sample C (for TL) Analysis 2	8						
Sample C (for TL) Analysis 1	9		not determined				
Sample C (for TL) Analysis 2	9						
Sample C (for TL) Analysis 1	10	ASU L 00.00-43 / EN 1788 (2002)		TL/OSL-DA-20 von DTU Nutech	X-ray radiation		calibrated PSL-result negative - no TL-securing necessary
Sample C (for TL) Analysis 2	10						calibrated PSL-result negative - no TL- securing necessary

Thermoluminescence: Sample D

Thermoluminescence (TL)	Evaluation number	Date of Analysis	Final result (qualitative)	Tempera- ture peak Glow curve	(Glow curve 1)	TL 2 (Glow curve 2)	Units of TL1 and 2	TL Ratio	Dose for normalization	Limit of detection (MDL)	Initial temperature	Heat rate	Final temperature	Relevant temperature range
		day/month	postive / negative	°C					kGy		°C	°C/sec	°C	°C
Sample D (for TL) Analysis 1	1	05.11.21	positive	600	516	1893	nC	0,2726	1		70	5	300	100-600
Sample D (for TL) Analysis 2	1	05.11.21	positive	600	2484	2866	nC	0,8667	1		70	5	300	100-600
Sample D (for TL) Analysis 1	3	15.11.21	positive	180	247555	513755	Photons	0,4819	1	474	35	6K/sec	350	110-210
Sample D (for TL) Analysis 2	3	15.11.21	positive	180	283991	559075	Photons	0,508	1	474	35	6K/sec	350	110-210
Sample D (for TL) Analysis 1	4	03.11.21	positive	150-250	3854	897	nC	4,29654404	1,08	6 nC	70	6	300	150-250
Sample D (for TL) Analysis 2	4	23.11.21	positive	150-250	3675	4753	nC	0,77319588	1,01	6 nC	70	6	300	150-250
Sample D (for TL) Analysis 1	5	08.11.21	Positive	228°C	1140	1740	nC	0,655	1 kGy		70°C	6°C/sec	400	40-57°C
Sample D (for TL) Analysis 2	5	08.11.21	Positive	215°C	699,8	694	nC	1,008	1 kGy		70°C	6°C/sec	400	40-57°C
Sample D (for TL) Analysis 1	6	11.11.21	positive	185	12442677	21601992	counts	0,576	1	2000	60	6	400	173-233
Sample D (for TL) Analysis 2	6	11.11.21	positive	215	1382291	2066227	counts	0,669	1	2000	60	6	400	173-233
Sample D (for TL) Analysis 1	7	1820.11.	positive	209	18823665	73729424	counts	0,255	1	451	70	6	400	179-250
Sample D (for TL) Analysis 2	7	1820.11.	positive	241	5894958	25674251	counts	0,23	1	45	70	6	400	179-250
Sample D (for TL) Analysis 1	8	22.12.21	positive	220	207,28	299,84	nC/mg	0,6913	1		70	6	350	70-350
Sample D (for TL) Analysis 2	8													
Sample D (for TL) Analysis 1	9	01.12.21	positive	173	726842	1684353	Counts	0,432	1		70	6	400	155-215
Sample D (for TL) Analysis 2	9													
Sample D (for TL) Analysis 1	10	15.12.21	positive	225	46575959	4958337	?	9,39	1	22579	70	6	450	185 - 249
Sample D (for TL) Analysis 2	10	15.12.21	positive	210	27864887	77908854	?	0,358	1	22579	70	6	450	185 - 249

Thermoluminescence (TL)	Evaluation number	Method description, like in a analysi report / norm / literature	is Notes to sample preparation	Apparatus (TL)	Irradiation source for normalization	Method accredited according to ISO/IEC 17025	Further remarks
		ASU L 00.00-43 / EN 1788 (2002)	Sample weight / Method of isolation + amount of minerals			yes / no	
Sample D (for TL) Analysis 1	1		amount of minerals: 15				
Sample D (for TL) Analysis 2	1		amount of minerals: 10				
Sample D (for TL) Analysis 1	3		5,67			yes	
Sample D (for TL) Analysis 2	3		9,78			yes	
Sample D (for TL) Analysis 1	4		12,13g			yes	
Sample D (for TL) Analysis 2	4		13,98			yes	
Sample D (for TL) Analysis 1	5		10g/Tungsten;HCl/3-10 mg			yes	
Sample D (for TL) Analysis 2	5		10g/Tungsten;HCl/3-10 mg			yes	
Sample D (for TL) Analysis 1	6		20g/ rinse out + physical separation			yes	
Sample D (for TL) Analysis 2	6		20g/ rinse out + physical separation			yes	
Sample D (for TL) Analysis 1	7		15 g sample wheight, 0,6 mg minerals				
Sample D (for TL) Analysis 2	7		15 g sample wheight, 0,5 mg minerals				
Sample D (for TL) Analysis 1	8		40 g SW / 9.07 mg			yes	
Sample D (for TL) Analysis 2	8						
Sample D (for TL) Analysis 1	9		not determined				
Sample D (for TL) Analysis 2	9						
Sample D (for TL) Analysis 1	10		10 g sample wheight / EN1788-8.2.1.1 / 1,4 mg minerals			yes	TL-securing positive PSL-result
Sample D (for TL) Analysis 2	10		10 g sample wheight / EN1788-8.2.1.1 / 1,2 mg minerals			yes	TL-securing positive PSL-result

Thermoluminescence: Negative Control

Thermoluminescence (TL)	Evaluation number	Date of Analysis	Final result (qualitative)	Tempera- ture peak Glow curve 1	TL 1 (Glow curve 1)		Units of TL1 and 2	TL Ratio	Dose for normali-zation	Limit of detection (MDL)	Initial temperature	Heat rate	Final temperature	Relevant temperature range
		day/m onth	postive / negative	°C					kGy		°C	°C/sec	°C	°C
Negative control	1	05.11.21	negative	600	1,08		nC				70	5	300	100-600
Negative control	3	15.11.21	negative	no Peak	1320	294310	Photons	0,0045	1	474	35	6K/sec	350	110-210
Negative control	4	-	-	-	-	-	-	-	-	-	-	-	-	-
Negative control	5	08.11.21	Negative	397°C	0,2541	10,85	nC	0,0234	1 kGy		70°C	6°C/sec	400	40-57°C
Negative control	6	11.11.21	negative	kein Peak	480	902	counts	0,532	1	2000	60	6	400	173-233
Negative control	7	1820.11.		304	1787	20856530	counts	0	1	451	70	6	400	179-250
Negative control	8	22.12.21	negative	-	0,0574	404,28	nC/mg	0,0001	1		70	6	350	70-350
Negative control	9	01.12.21	negative	400	317	1554275	Counts	0,0002	1		70	6	400	155-215
Negative control	10	15.12.21				1181			1	22579	70	6	450	185 - 249

Thermoluminescence (TL)	Evaluation number	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Apparatus (TL)	Irradiation source for normalization	Method accredited according to ISO/IEC 17025	Further remarks
		ASU L 00.00-43 / EN 1788 (2002)	Sample weight / Method of isolation + amount of minerals			yes / no	
Negative control	1						
Negative control	3		0,63	Lexsygsmart (Freiberg Instruments)	3 Cs137 sources	yes	
Negative control	4	EN 1788 (2002)	-	Harshaw TLD 3500	Co	yes	none
Negative control	5	yes	10g/Tungsten;HCl/3-10 mg	HarshawTLD	Xray	yes	
Negative control	6	ASU L 00.00-43 / EN 1788 (2002)	blank	Risoe TL-DA-15	Sr90	yes	
Negative control	7	ASUL 00.00-43 / EN 1788 (2002)	12 g sample wheight, 0,9 mg minerals	RISO DTU Denmark TL/OSL Reader Typ: DA-20, Serial no.: 377-04/2014	Sr-90	yes	
Negative control	8	EN 1788	16.26 mg	Harshaw TLD 3500	Co-60	yes	
Negative control	9		not determined				
Negative control	10			TL/OSL-DA-20 von DTU Nutech	X-ray radiation		

Thermoluminescence: Positive Control

Thermoluminescence (TL)	Evaluation number	Date of Analysis	Final result (qualitative)	Tempera- ture peak Glow curve 1	(Glow curve 1)	1	Units of TL1 and 2	TL Ratio	Dose for normalization	Limit of detection (MDL)	Initial temperature	Heat rate	Final temperature	Relevant temperature range
		day/month	postive / negative	°C					kGy		°C	°C/sec	°C	°C
Positive control	1	05.11.21	positive	600	2626		nC				70	5	300	100-600
Positive control	3	15.11.21	positive	195	101158	448826	Photons	0,2254	1	474	35	6K/sec	350	110-210
Positive control	4	03/11/2021 & 23/11/2021	positive	150-250	8023/8316	9499/6984	nC	0,84/1,19	1,08/1,01	6 nC	70	6	300	150-250
Positive control	5	08.11.21	Positive	234°C	3893	4210	nC	0,9247	1 kGy		70°C	6°C/sec	400	40-57°C
Positive control	6	11.11.21	positive	215	6678857	3691753	counts	1,809	1	2000	60	6	400	173-233
Positive control	7	1820.11.		256	14101639	25797622	counts	0,547	1	451	70	6	400	179-250
Positive control	8	22.12.21	positive	270	369,64	574,62	nC/mg	0,6433	1		70	6	350	70-350
Positive control	9	01.12.21	positive	203	208955	2576577	Counts	0,081	1		70	6	400	155-215
Positive control	10	15.12.21				713572			0,1	22579	70	6	450	185 - 249

Thermoluminescence (TL)	Evaluation number	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Apparatus (TL)	Irradiation source for normalization	Method accredited according to ISO/IEC 17025	Further remarks
		ASU L 00.00-43 / EN 1788 (2002)	Sample weight / Method of isolation + amount of minerals			yes / no	
Positive control	1						
Positive control	3		0,64			yes	
Positive control	4		10,17/10,73			yes	1 irradiated curry (1kG +/-10%) in, each sample list
Positive control	5		10g/Tungsten;HCl/3-10 mg			yes	
Positive control	6		Sand 2kGy treated like sample form HCl			yes	
Positive control	7		12 g sample wheight, 0,8 mg minerals				
Positive control	8		8.86 mg			yes	
Positive control	9		not determined				
Positive control	10						

5.3 Information on the Proficiency Test (PT)

Before the PT the participants received the following information in the sample cover letter:

PT number	DLA ptRE02 - 2021				
PT name	Detection of Food Irradiation (Spices, ground + Herbs, rubbed) 2 different samples each (irradiated / non-irradiated)				
Sample matrix*	Samples A + B: Herbal mixture / Ingredients: rosemary, basil, marjoram Samples C + D: Curry powder / Ingredients: mustard, caraway seeds, coriander, fenugreek, fennel, turmeric, salt, cayenne pepper, celery seeds, onions, garlic				
Number of samples and sample amount	4 samples: 2 Samples of spice mixture 40 g each, 2 samples of herb mixture 40 g each.				
Storage	room temperature (dry and dark)				
Intentional use	Laboratory use only (quality control samples)				
Parameter	qualitative: irridiated / non-irradiated (positive / negative)				
Methods of analysis	Photon-stimulated luminescence (PSL) and thermoluminescence (TL).				
Notes to analysis	The analysis of PT samples should be performed like a routine laboratory analysis. In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.				
Result sheet	Separate information in the relevant result submission files for PSL and TL				
Units	qualitative: positive / negative (for measured values see result submission files)				
Number of significant digits	at least 2				
Further information	For information please specify: - see result submission files				
Result submission	The result submission file should be sent by e-mail to: pt@dla-lvu.de				
Last Deadline	the latest December 24th 2021				
Evaluation report	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.				

^{*} Control of mixture homogeneity and qualitative testings are carried out by DLA. Any testing of the content, homogeneity and stability of PT parameters is subcontracted by DLA.

6. Index of participant laboratories

Teilnehmer / Participant	Ort / Town	Land / Country
		BELGIUM
		AUSTRIA
		Germany

[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswerte-Berichts nicht angegeben.]

[The address data of the participants were deleted for publication of the evaluation report.]

7. Index of references

- 1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
- 2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment - General requirements for proficiency testing
- 3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
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