Proficiency test of detection of packaging material in bakery by-products 2021

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Cover photo: packaging material extracted from a test sample organised according to the type of material: metal (top), hard plastic (right), soft plastic (bottom) and paper/board (left)

Contents

Summary

A proficiency test (PT) was organised for the detection of packaging materials in bakery by-products intended to be used as feed ingredients. Two blind samples of a size of 250 grams at spike levels of 0 mg (blank) and 250 mg (0.1%) per sample were distributed to 30 participants, with the request to return the selected sample material investigated to the organiser. One participant informed the organiser of not being able to do the analysis in the requested time frame. The evaluation of the results was carried out according to the principles of the *Quality Guidelines for visual research*, in the framework of individual spiking of the samples. This specific procedure was chosen to avoid large inhomogeneity of a general batch as basis for the production of the PT samples, with the consequence that usual statistics such as Z-scores could not be applied or needed modified interpretations. Results of this PT have been compared with the two previous editions of the PT for packaging materials in bakery by-products in 2016 (n=23 participants) and 2019 (n=25), with spike levels of 0.02% and 0.1% (no blank).

The results of the current proficiency test for packaging material in bakery-by-products is comparable to those of the previous editions when evaluating the constant element: the sample spiked at a level of 0.1% (250 mg spike material in 250 g matrix). 20-24% of the participants reported a recovery level outside the interval of 66-134%. The process for verification of the materials as returned by the participants showed that more than 9% of that material still consisted of moisture and fat (n=11) and that matrix material in excess of 10% of the weight of the returned portions was found for both the blank sample (n=9) and the spiked sample (n=8). The final results showed four cases of amounts below the lower acceptable limit of 66% recovery. The results indicate that four factors need further attention: these are a proper implementation of a validated method (moisture, fat), a good recognition of matrix material for avoiding false positive results and a good recognition of packaging material for avoiding false negative results. An additional factor in reporting is the difference between the reported weight and the actual weight of the returned portion.

The chosen interval for acceptable recovery rates at a level of 0.1% (250 mg in 250 g matrix; 66-134%) is based on experiments with ergot sclerotia and weed seeds in a granular matrix such as cereals. This transfer to another type of matrix material seems reasonable, but proficiency testing is not a good source for derivation of such intervals. Further collection of data in the visual domain for adjusting the modified Horwitz equation is needed.

1 Introduction

An important factor in circular agriculture is the reuse of former food products (FFP). This term applies to all products produced with the intention of human consumption. Withdrawal from this principal use can occur after production (factory), during storage and transport, or in the retail stage. In contrast to materials which result from the processing of primary commodities (e.g. oil-seed hulls and kernels, cereal by-products, pulp, etc.), these FFPs have to be unpacked in a range of cases. Regulation (EC) 767/2009 Annex III mentions zero tolerance for the presence of remnants of packaging material in FFPs. In practice, relatively low action limits were installed in member states (van Raamsdonk et al., 2011).

Several methods for the detection of packaging material have been developed, with the scope of bakery by-products (van Raamsdonk et al., 2012) or compound feed containing bakery by-products (Amato et al., 2017). The RIKILT method for bakery by-products was accepted as IAG method during the Autumn meeting of the IAG section Feed Microscopy in 2015. Subsequent plans were made to organise a proficiency test (PT) for this and comparable methods. This PT was held in 2016 and results indicated that a training was recommended. Such a training was organised during the annual meeting of IAG section Feed Microscopy in Uppsala in 2017. Training samples were distributed in 2018 to every interested member of IAG section Feed Microscopy. A second PT was organised during Autumn 2019 (van Raamsdonk et al., 2020). A non-spiked sample was lacking in both PTs. It was concluded that results from a blank sample are needed for getting more information on the specificity of the method. Additionally, material selected by the participants should be returned to the organiser for checking the effectivity of the procedure by the participants and for verification of the balance between packaging material and matrix material. This report presents the results of the third PT of 2021.

2 Methods

2.1 Materials

Samples were based on bakery by-products from practice. These materials have been sent to WFSR in the past in the framework of the national monitoring program and the contamination was determined by removing all visible parts of packaging material from the matrix. The detection limit, as established by WFSR (van Raamsdonk et al., 2012) is 0.004%, which means that the remaining material should be below that threshold, which translates to 10 mg/250 g. Coarsely ground material of puffed rice wafers has been added at a level of 1%. A sufficient amount of matrix material was mixed for preparing a matrix for the samples of the PT. It was the intention to prepare a homogeneous batch of matrix material. However, due to unknown factors causing inhomogeneity, the amount of 10 mg packaging material per sample is an approximation.

This PT consisted of two samples of 250 gram each: one blank, which could contain up to 10 mg of remaining packaging material, and a sample spiked with 250 mg of packaging material (range 248.0-252.3 mg), which could contain up to 260 mg of packaging material. All samples were spiked on an individual basis. The fragments of packaging material are selected from samples from the last ten years, stored by WFSR. Portions were selected of the required amount, with attention to a balanced composition of paper (55%, 135.6-137.3 mg), soft plastic (17%, 42.3-44.0 mg), hard plastic (20%, 49.0-51.0 mg) and tin foil (8%, 20.2-22.1 mg). The amounts per sample are presented in Annex 6 and will be used for calculating the exact recovery.

With matrix and packaging material from practice, these samples can be considered to represent the practical situation.

Recovery is calculated as

$$R_{\%} = \frac{r_i}{s_i} * 100$$

with r_i as the amount of recovered material, and s_i as the amount of spiked material for every individual sample at each of the two spike levels. This parameter is derived from the percent difference $D_{\%}$, one of the possible parameters for evaluating results in proficiency testing (ISO 17043: 2010, Appendix B.3.1.3, paragraph b). The relationship can be described as $R_{\%} = D_{\%} + 100\%$. The results obtained from incomplete examination of the samples were excluded from the final evaluation. The recovery intervals applied are extracted from the Quality Guidelines for visual research.

2.2 Organization of the proficiency test

All IAG members, all NRLs, participants of former proficiency tests and a series of putative interested laboratories were informed about the proficiency test for 2021 by means of mailing using distribution lists. Until the beginning of April, a total of 30 participants for the proficiency test packaging material in bakery by-products were listed. The sets of two samples with an accompanying letter (see Annex 1) were sent to all participants on Thursday 21st of October 2021. The file with instructions and the report form were sent to the participants on Wednesday 27th of October (see Annex 3 and 4). The closing date for reporting results was Monday 22nd of November. The analysis of the results was carried out during Spring 2022. The report was distributed in draft to the Board of IAG section Feed microscopy and the participants on Monday the 23rd of May 2022.

A blank sample was examined by WFSR in Spring for verifying the expected level of packaging material in a non-spiked matrix. The amount of material selected appeared to be 30 mg, which would be a level of 0.012%.

2.3 Evaluation of results

Based on the conclusions of the previous proficiency tests for packaging material in bakery-by-products, the participants were requested to return the selected packaging material as selected by them. The availability of this material was deemed essential for a further evaluation of the sensitivity and specificity of the method, and the precision of the method application by the participants.

A specially designed verification procedure for evaluation by WFSR technicians was applied, based on the WFSR procedure. An important deviation was made for the defatting step. The method describes a defatting time of 10 minutes. In order to avoid overexposure of the plastic to TCE, which might cause dilution of a part of the plastic, a defatting step during only 5 minutes was applied in this verification procedure.

The procedure was applied as follows:
1. Receipt and administration of the reported results and of the two portions from each participant.
2. Materials were photographed.
3. The two portions per participant were weighed. The material selected from the spiked sample was processed as follows:
4. The material per portion was dried for 4 hrs at 60 °C, and weighed afterwards.
5. The material per portion was defatted in TCE during 5 minutes.
6. The material per portion was dried again for 4 hrs at 60 °C, and weighed afterwards.
7. The material per portion was classified according to the four categories, and matrix material was separated from the packaging material. Each of the five subportions was weighed.
8. The material per portion divided over the four subportions was photographed. This image could be compared to a similar picture made before sending the samples to the participants.

The drying and defatting steps (4-6) were skipped for the material selected from the blank samples in the assumption that this amount was limited or almost absent. The fragments of packaging materials were separated from the matrix material of the blank as one subportion.

This procedure resulted in the following data per portion of selected and returned material of the spiked sample:
a. Reported amount (step 1).
b. Returned amount as weighed by the organiser (step 3).
c. Amount of material after drying (step 4).
d. Final net amount of returned material (step 6).
e. Amount of matrix material, if any (step 7).
f. Total net amount of packaging material (step 7).
g. Amount per type of packaging material after processing (step 7).

The results for the blank samples comprise:
a. Reported amount (step 1).
b. Returned amount as weighed by the organiser (step 3).
c. Amount of matrix material, if any (step 7).
d. Total amount of packaging material (step 7).

The evaluation of the results will take the following steps:
I. Overview of the reported amounts of both sample types. This would be the normal final result of a proficiency test.
II. A comparison of result a. and b.
III. A comparison of result b. and d.
IV. A comparison of result d. and f.
V. Recovery per type of packaging material for the spiked sample (result).

The organisation and evaluation of the IAG proficiency test packaging material in bakery-by-products follows the Quality Guidelines for visual research in food and feed (van Raamsdonk et al., 2022).

3 Results

A total of 29 participants returned results. All participants achieved to submit the selected portions to the organiser before the end of the year. This situation allowed to perform a full evaluation of the followed procedure and the obtained results.
The full results are presented in the tables of Annex 5, 6 and 7.

3.1 Reported results

The results as reported by the participants are shown in Figure 1. Six participants (21%) reported an amount of packaging material in the blank exceeding the amount of 30 mg as found in the control sample (Figure 1 top). Seven participants (24%) reported amounts outside the interval of 66-134% recovery for the spiked sample (Figure 1 bottom).

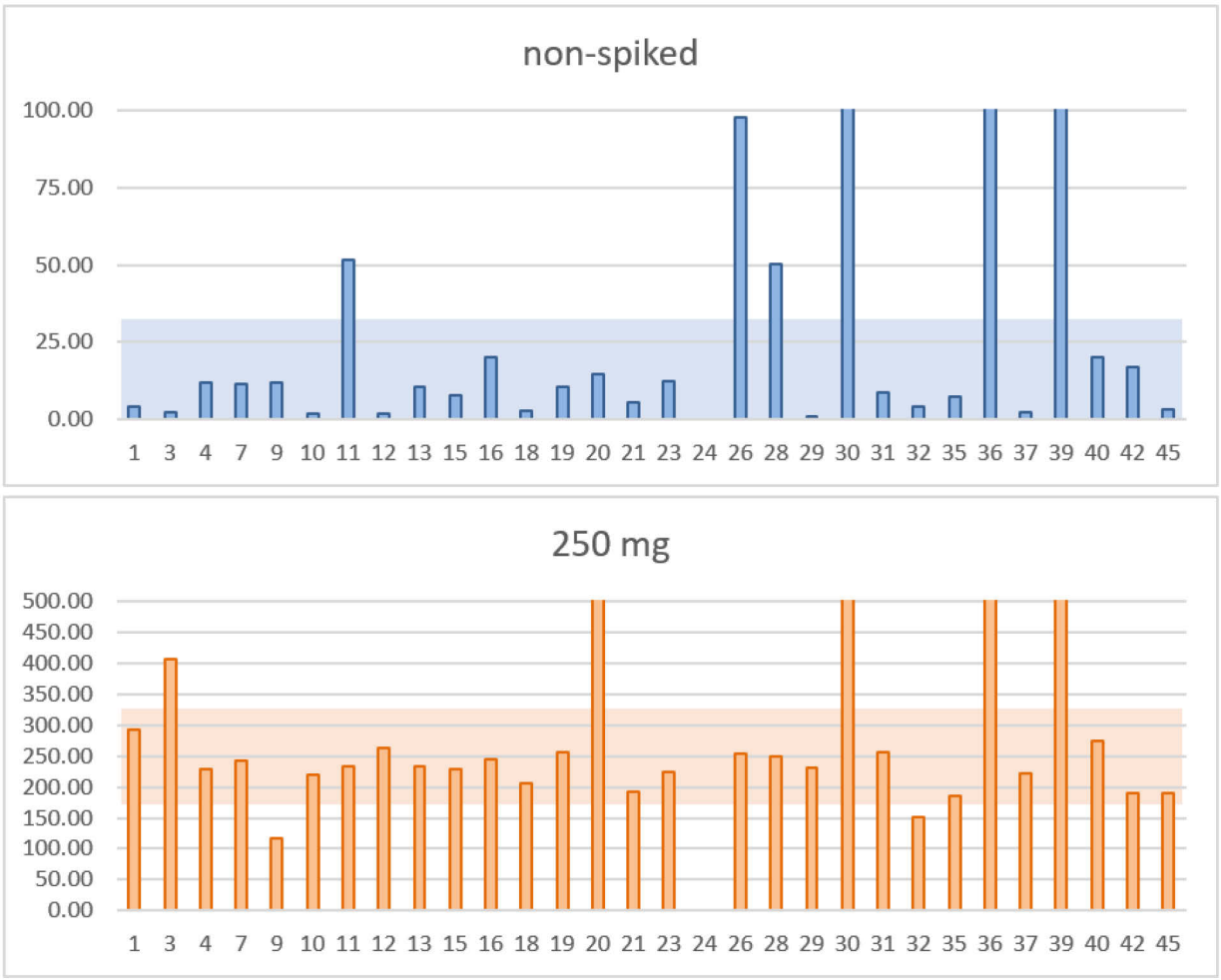

Figure 1 *Overview of the results reported by the participants for the blank sample (top) and for the sample spiked with 250 mg of packaging material (bottom). The shaded areas show the ranges of the accepted recoveries.*

The results as shown in Figure 1 would represent the final results of a typical proficiency test. However, several procedural errors can exist, which can be evaluated by re-examination of the returned material by the organiser. The next sections will present these evaluations.

3.2 Returned material

The first step in the further evaluation is the verification of the amount of returned material compared to the reported amount (Figure 2). A difference between these two values was observed for most participants. Deviations of the presented material exceeding 10% of the reported amount, either under- or over-presence, were found for 14 out of 29 participants for the blank (48%). Seven participants (24%) returned amounts deviating for more than 10% from the reported value for the spiked sample. These figures show a relation with the level of contamination: 10% of a low level is a lower absolute amount than 10% of 250 mg and is more easy to exceed. Considering differences resulting from calibration of balances and rounding off, a difference higher than 2 mg absolute could be a signal of another cause of a deviation. Frequencies of deviation of this threshold of 2 mg were 41% for the blank (12 out of 29) and 90% for the spiked sample (26 out of 29). Maximum differences were a reported amount of 10.4 mg and returned amount of 0.2 mg for the blank (participant 19) and a reported amount of 887.0 mg and returned amount of 211.9 mg for the spiked sample (participant 20). The reported amount of 887.0 could be a representation of a level (mg/kg).

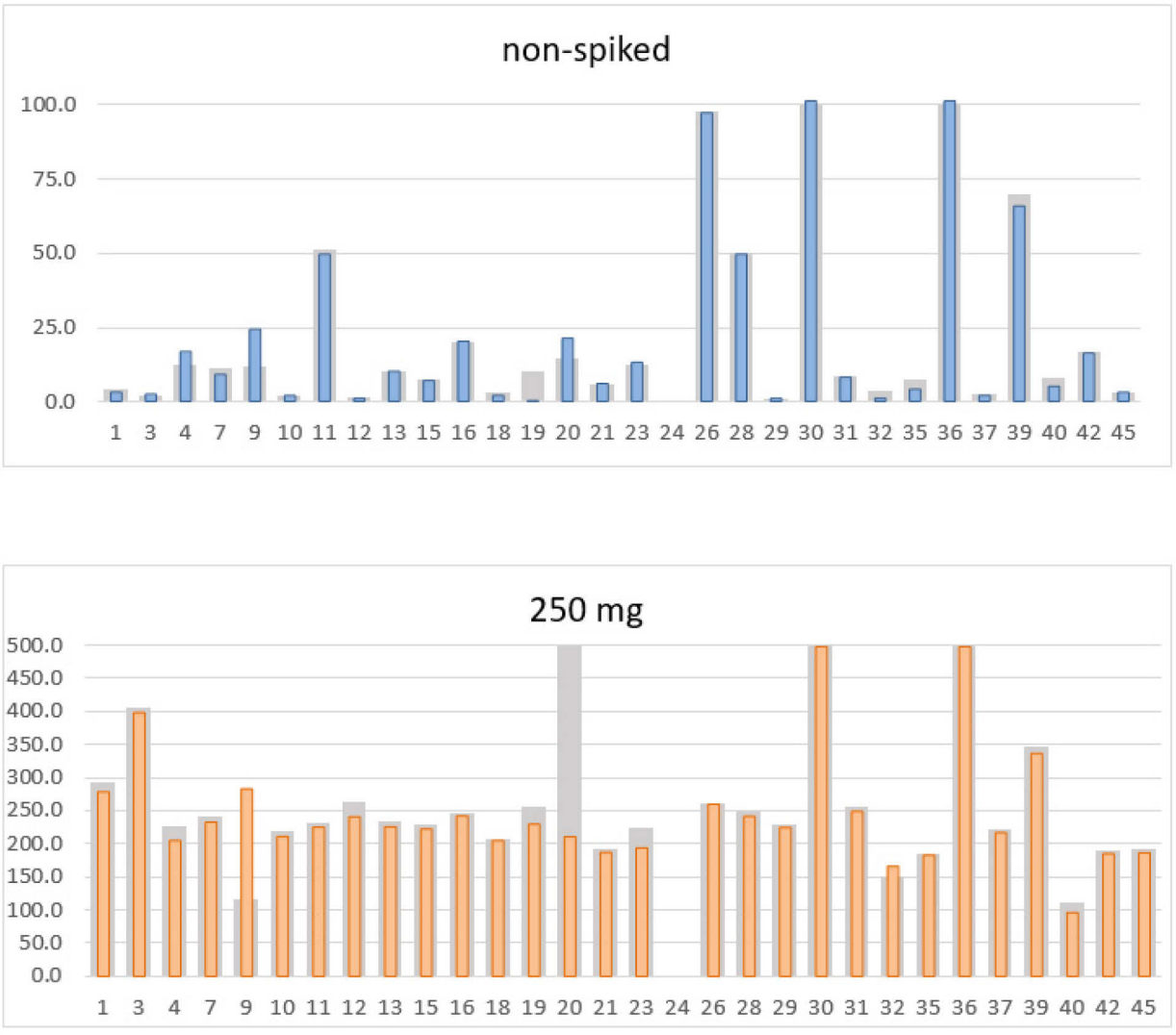

Figure 2 *Overview of the amounts returned to the organiser by the participants for the blank sample (top) and for the sample spiked with 250 mg of packaging material (bottom). The grey bars show the reported amounts for comparison.*

3.3 Remaining material after defatting and drying

All returned portions of the spiked sample have been dried and defatted. The intermediate value of drying and the final amount after defatting are presented in Annex ##. The amount of moisture removed in the first step was in most cases higher or much higher than the amount of fat removed in the subsequent step. In this section the final effect of processing will be presented. A simplified procedure for verification was applied to the portions selected from the blank. In addition, only the highest amounts of returned material from the blank (participant 11: 49.0 mg; participant 20: 21.0 mg) have been processed as well. This resulted in adjusted values of 47.0 mg and 18.0 mg, respectively.

The gain in optimal weight is shown in Figure 3. A removal of moisture and fat of 6% or lower of the returned portions of the spiked sample is achieved for 18 out of 29 participants (62%). The portions of the other 11 participants showed a remaining level of moisture and fat of 9% or more (participants 1, 9, 12, 13, 19, 23, 29, 31, 36, 39 and 40). The highest gain was a recovery of 30% of moisture and fat (participant 39).

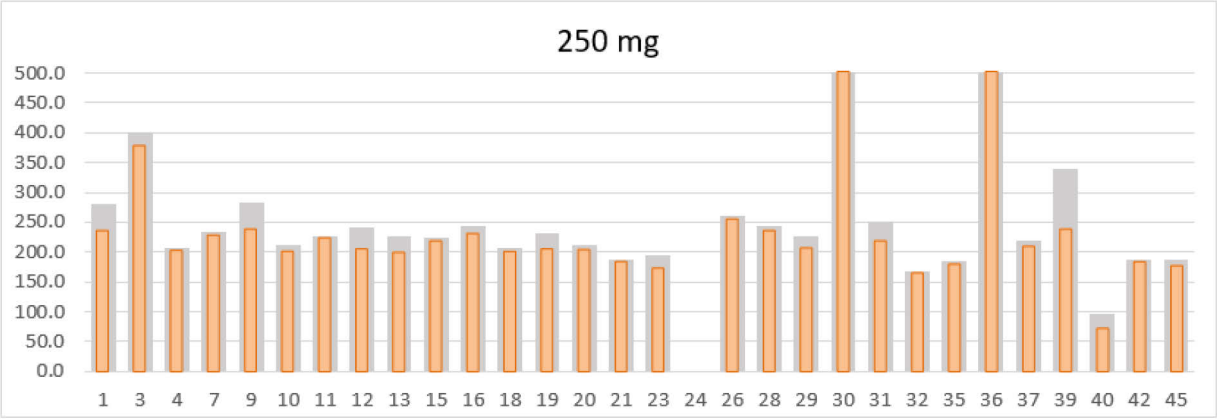

Figure 3 *Overview of the amounts after drying and defatting for the sample spiked with 250 mg of packaging material. The grey bars show the amounts returned to the organiser by the participants for comparison.*

Three methods have been applied for the defatting and drying of the raw material to find the final result. These are the RIKILT/WFSR method (18 participants), a VDLUFA method (n=2), own laboratory methods (n=8) and additionally a method reported to be a mix of elements of the WFSR and a VDLUFA method (n=1). The effect of these different procedures as applied by the participants has been verified by WFSR. The gain of 9% or higher in weight of the material of the mentioned 11 portions of the spiked sample was achieved after all four methods or combination thereof as applied by the participants. Of these excesses found, four out of 18 participants (22%) applied the RIKILT/WFSR method, five out of eight (63%) applied their own laboratory method, one out of two (50%) applied a VDLUFA method and one (100%) applied a combination of the WFSR and VDLUFA methods.

3.4 Segregation of matrix material

The final step in the evaluation is the separation of the matrix material, if any, from the packaging material in all portions and the classification of the material in the four types and used for spiking: paper and board, soft plastic, hard plastic and tin foil.

Several participants selected matrix material as target. These confused non-target materials consisted primarily of candy material (participants 3, 26, 28, 31, 35, 36, and 39), puffed rice (16, 28, 30 and 36) and plant material (9, 26 and 37). Six participants selected these materials from both the blank and the spiked sample.

The final net weight of packaging material in the returned portions for both samples is shown in Figure 4. A share of matrix material over 10% in the portions extracted from the blank was found for nine participants (9, 26, 28, 30, 31, 35, 36, 39, 45), illustrated by the grey bars exceeding the blue bars in Figure 4 (top). In some cases these high shares represent low amounts. The excess over the accepted range for the blank as retrieved from the portion of participant 11 consist correctly of packaging material, which points to an apparent inhomogeneity of the original batch of bakery by-products. A share of matrix material over 10% in the portions extracted from the spiked sample was found for eight participants (3, 26, 28, 30, 31, 36, 37 and 39; Figure 4 bottom).

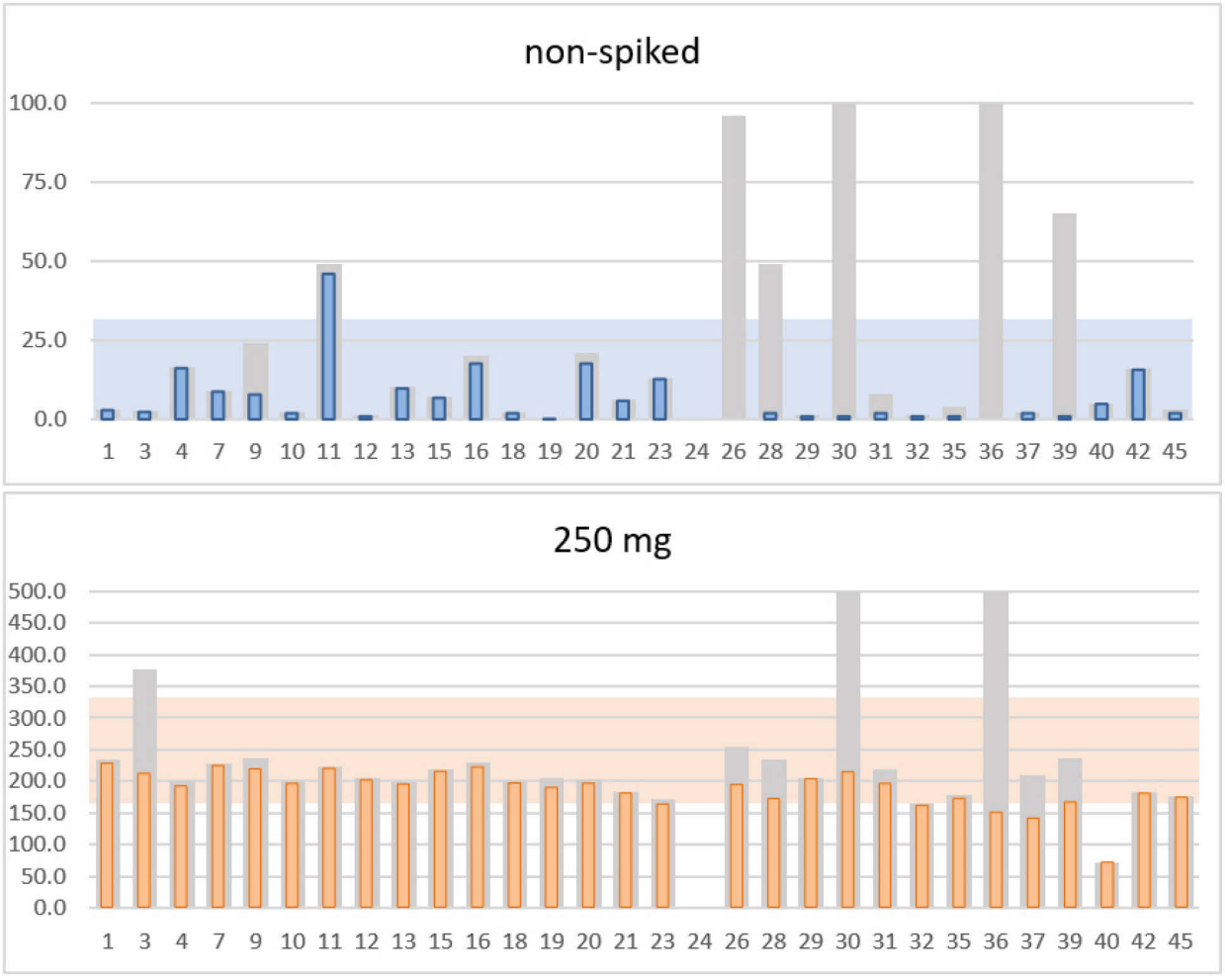

Figure 4 *Overview of the net amounts of packaging material as selected by the participants for the blank sample (top) and for the sample spiked with 250 mg of packaging material (bottom). The grey bars show the amounts after drying and defatting for comparison and before removal of non-target material. The excess of the grey bars represents the presence of matrix material. The shaded areas show the ranges of the accepted recoveries.*

3.5 Evaluation of recovery per type of packaging material

Every portion as selected from the spiked sample has been separated in the four types of packaging material as used for spiking (paper and board, soft plastic, hard plastic, tin foil). In some cases an amount of hard plastic was found exceeding the amount used for spiking in combination with an underrepresentation of soft plastic. In the assumption of extraction of a part of the softener in soft plastic material after TCE treatment, resulting in an excess of hard plastic, the two types of plastic were pooled for evaluation.

The results are shown in Figure 5. All relative amounts of the three types of packaging material are below 1. No extra material was retrieved, even in de situation that matrix of a sample could contain up to 30 mg of non-spiked packaging material. There is one exception: the portion of the spiked sample investigated by participant 19 appeared to contain 107% of the spiked amount of plastic after processing and after removal of the matrix material. When applying the same interval for acceptable recovery rates as for the entire portions (66-134%), differences in recovery between the three types can be observed. The best frequency of good recovery (higher than 66%) is found for plastic with 26 out of 29 good results. Lower frequencies have been found for paper and board (18 out 29) and for tin foil (20 out of 29).

Figure 5 *Overview of the net amounts of packaging material as selected by the participants for the blank sample (top) and for the sample spiked with 250 mg of packaging material (bottom). The grey bars show the amounts after drying and defatting for comparison and before removal of non-target material. The excess of the grey bars represents the presence of matrix material. The shaded areas show the ranges of the accepted recoveries.*

4 Discussion

The reported results for the sample spiked at 250 mg show five participants exceeding the upper limit for recovery (134%) and three participants with a recovery below the lower limit (66%). These results can be compared with the results of the previous two proficiency tests (van Raamsdonk et al., 2020; Table 1). The performance of the 2016 version has been recalculated retrospectively to the 66-134% recovery limits. Nevertheless, comparison is complicated for the different factors involved. For example, puffed rice was not included in the 2016 and 2019 versions.

Further focus on the performance through time reveals some interesting aspects. A total of 18 laboratories participated in all three editions with eight of them with recoveries for the 250 mg sample within limits in all three cases. A further nine laboratories participated in two editions of which 6 showed a performance within limits in both cases. Three participants in the current 2021 edition participated for the first time and showed good performance for at least the spiked sample.

The given brief overview does not insinuate a limited value for training. A good performance of a method for visual inspection is intertwined with a good and extensive expertise of the technician. A good understanding of the factors involved in the result of a method is a first step for increasing the performance of that method and for further training.

Table 1 *Overview of previous and current results for PTs of packaging material in bakery by-products. Number of participants (share of total number) showing a result outside the accepted range for recovery of 66-134%. For every PT the sample with a spike level of 250 mg (1000 mg/kg) has been used as basis.*

Year of PT:	2016 (n=23)	2019 (n=25)	2021 (n=29)	2021 (n=29), net recovery
Below lower limit	3 (13%)	0	2 (7%)	4 (14%)
Exceeding upper limit	2 (9%)	5 (20%)	5 (17%)	0

4.1 Application of the method

A method consist of several steps: preparation of the sample, selection and identification of the target material, and proper reporting. Although the reported results are the final product of a method application, it is the first parameter available for evaluation. Six participants reported levels exceeding 200 mg/kg (0.02%), which is clearly higher than the established level of detection for the method (Figure 1 top). Seven participants reported values for the spiked sample outside the interval for recovery (66-134%; Figure 1 bottom; Table 1). In most cases the amount of the returned material deviated from the reported result. This situation does not necessarily mean that result reported by the participants is incorrect; a part of the bias can be caused by the balance used by WFSR. Notwithstanding this, the message is twofold: (a) frequent calibration of balances in necessary, at least quarterly, and (b) comparison of results in interlaboratory studies needs a high level of harmonisation.

The need of drying and defatting has been disputed. ACAF (2013) referred to the RIKILT method in their brief method description and indicated drying as optional step, without mentioning defatting. Amato et al. (2017) mentioned defatting as optional step ("if necessary"), and described the subsequent drying step as using a hood for at least two hours. The current results show that the returned material of 11 participants contained more than 9% of moisture and/or fat. The material returned by participant 39 showed after processing that the reported result, exceeding the accepted interval with a recovery of 140%, could have been correct when properly processed (recovery 95%). The type of method appeared to be critical as well. The results of this proficiency test indicate the importance of drying and defatting and of the application of a carefully designed and tested method.

4.2 Selection of packaging material

The removal of moisture and fat from the selected packaging material is one aspect of optimisation of the sensitivity of the method. Another aspect is a careful selection and separation of target and non-target material. Overlooking target material in general will result in (too) low values for sensitivity and selection of non-target material will result in (too) low values for specificity. Both situations were found among the results of this proficiency test. Most notably candy fragments were selected as packaging material by seven participants. The presence of puffed rice resulted in major overestimations for two participants. Participants 26, 28, 30, 36 and 39 (5 out of 29, 17%) would have reported the blank as non-compliant because of the selection of non-target material (Figure 4 top), considering the zero tolerance as mentioned in the legislation.

Recognition of matrix material could be facilitated by several types of documentation. An image bank with representations of packaging material could support proper identification. Such an image bank should contain materials from different countries. The three editions of the proficiency test for packaging materials only contains spike material of Dutch origin for obvious reason. Chemical tests for suspicious particles could help identification. Examples are phloroglucinol staining for lignin, lugol staining for starch containing particles such as puffed rice (Figure 6), or chloralhydrate slides for identification of plant material (Feigl and Anger, 1958).

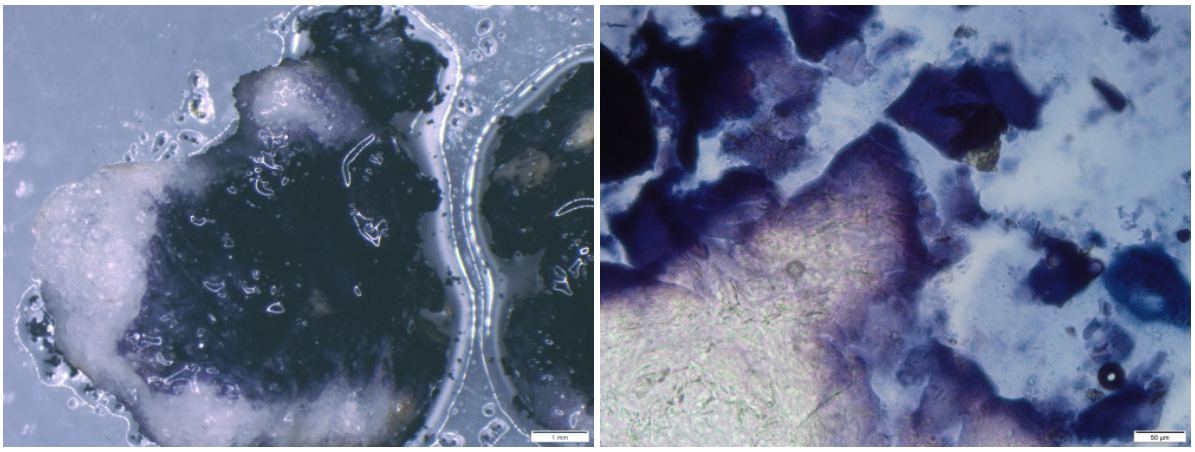

Figure 6 *Images of puffed rice stained with lugol. Left: magnification 6.3X, right: magnification 40x.*

In terms of net recovery, i.e. recovery of packaging material after removal of moisture, fat and non-target material, one participant could have reported the blank as non-compliant, due to unintentional presence of packaging material. Recoveries of target material in excess of the upper limit of 134% were obviously not found in the spiked sample, since all non-target material has been removed in the final step of the verification procedure for achieving the value for net recovery.

Four participants reported amounts below the lower limit of 66% for net recovery of packaging material (32, 36, 39, 40; 14%). Any correction was not possible in the frame of the verification process. However, the further analysis of the different fractions of the three types revealed that for paper/board (18 out 29) and for tin foil (20 out of 29) a better selection can be made.

4.3 Quality assurance

The chosen interval for acceptable recovery rates at a level of 0.1% (250 mg in 250 g matrix; 66-134%) is based on experiments with ergot sclerotia and weed seeds in a granular matrix such as cereals (van Raamsdonk et al., 2022). This transfer to another type of matrix material seems reasonable (Table 1), but proficiency testing is not a good source for derivation of such intervals. One compromising factor is the application of different protocols, another the diverse level of skills of the participants. The first factor was largely compensated by the verification procedure as applied in this proficiency test, but the effect of overlooking packaging material, resulting in underestimates, could not be compensated (Table 1, last column). Another factor is the limited number of datasets available. The derivation of the original Horwitz equation, used for calculating values for measurement uncertainty for chemical tests in the field of feed and food safety, was in 1990 based on a number of 3000 test results (Pocklington et al., 1990). Further collection of data in the visual domain is needed.

5 Conclusions and recommendations

5.1 Conclusions

The results of the current proficiency test for packaging material in bakery-by-products is comparable to those of the previous editions when evaluating the constant element: the sample spiked at a level of 0.1% (250 mg spike material in 250 g matrix). 20-24% of the participants reported a recovery level outside the interval of 66-134%. The process for verification of the materials as returned by the participants showed that more than 9% of that material still consisted of moisture and fat (n=11) and that matrix material in excess of 10% of the weight of the returned portions was found for both the blank sample (n=9) and the spiked sample (n=8). The final results showed four cases of amounts below the lower acceptable limit of 66%. The results indicate that four factors need further attention: these are a proper implementation of a validated method (moisture, fat), a good recognition of matrix material for avoiding false positive results and a good recognition of packaging material for avoiding false negative results. An additional element in reporting is the difference between the reported weight and the actual weight of the returned portion.

5.2 Recommendations

- Only well established and validated methods should be implemented and applied.
- Documentation on the visual classification of packaging material and matrix material (e.g. knowledge bank, image database, expert system) need to be developed.
- Differences between reported weight and actual weight of the selected portion should be evaluated as part of measurement uncertainty.

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Annex 1 Basic instructions for the test procedure

Instructions

Test 2021-C: packaging material in bakery products
The package consists of two bags with 250 gram of bakery products. The purpose of the proficiency test is to detect any particle of packaging material. Recommended procedure is the method developed by RIKILT: report 2012.007 (http://edepot.wur.nl/240039).

For each of the two samples the **weight** in mg of the total sample and of the total amount of selected particles of packaging materials needs to be reported on the report sheet.

Additionally, it is mandatory to return the selected portions of packaging material from both samples in a vial each, duly labelled and packed, to the organiser. In a situation that these portions are not received, your reported weights will not be included in the final report, since the information in the report form and the materials in the vials are inseparable.

The report file will be send to you by mail. The report files consists of:
- A tab with specific instructions.
- A tab with an inquiry for a specification of the procedure.
- A tab for entering the results.

All pink cells have to be filled.
After completing the two forms "Procedure" and "Results", they have to be saved as Excel file by using "Save as …", add your unique lab code to the end of name (replace the ## signs with your lab number). The forms have to be send to the organisers in two ways. Every form has to be sent by E-mail as Excel file and as a scan (preferably *.PDF). and send the two files to Corina.smits@wur.nl and to Leo.vanraamsdonk@wur.nl. Results will be included in the final analyses and report only if both forms are send in by electronic mail, **and if the material selected from both samples have been returned to WFSR**.

Closing date is Monday November 22nd, 2021, for returning the report forms. A mail confirming the sending of the vials should be sent not later than the same date, including the name of the carrier and the track and trace number of the package.

Annex 2 Report form for procedure details

IAG proficiency test 2021 packaging materials in bakery products		
Please select your unique lab number	**-- select --**	
Have you read the ring test instructions?	-- select --	
Which detection method do you use?	-- select --	
I confirm to have sent the material selected from the sample(s) to the organiser	-- select --	

Annex 3 Report form: results

IAG proficiency test 2021 packaging materials in bakery products

lab number		
sample number		
examined amount of sample (gram)		
	weight (mg)	weight (mg)
packaging materials		
Comment, if necessary		

Signature

date

Annex 4 List of participants

Austrian Agency for Health and Food Safety-AGES	Austria
FLVVT	Belgium
SGS Bulgaria	Bulgaria
Canadian Food Inspection Agency	Canada
Croatian Veterinary Institute	Croatia
Danish Veterinary and Food Administration	Denmark
S.C.L. Laboratoire de Rennes	France
CVUA-RRW	Germany
Futtermittelinstitut Stade (LAVES)	Germany
Landesbetrieb Hessisches Landeslabor, Landwirtschaft und Umwelt	Germany
Landeslabor Berlin-Brandenburg	Germany
LLFG Landesanstalt für Landwirtschaft	Germany
LTZ Augustenberg	Germany
LUFA Nord-West	Germany
LUFA Rostock	Germany
LUFA-Speyer	Germany
SGS Analytics Germany GmbH, Jena	Germany
SGS Germany GmbH	Germany
Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft, GB6-Labore Landwirtschaft / LUFA, FB62	Germany
Thüringer Landesanstalt für Landwirtschaft Jena	Germany
Department of Agriculture, Fisheries and Food, Backweston Agri Laboratories	Ireland
IZS PLV Torino – CreAA	Italy
Ministero delle politiche agricole alimentari e forestali, Laboratorio di Modena	Italy
CCL - Nutricontrol	Netherlands
ForFarmers	Netherlands
Nutrilab BV	Netherlands
TLR	Netherlands
University of Ljubljana, Veterinary Faculty, Natl. Veterinary Institute, Unit for Pathology of Animal Nutrition and Environmental Hygiene	Slovenia
National Veterinary Institute, SVA	Sweden
Agroscope (ALP), Swiss Research Station	Switzerland

Annex 5 Results: blank sample

Explanation:
Matrix: gradient from 0% (green) to 86% (red).
PM: packaging material. *: after drying and defatting.
Indices in brackets refer to chapter 2.3. Cell colouring of matrix material ranges from green (0%: correct absence) to red (100%: only matrix material selected).

part.	sample	reported (a) mg	returned (b) mg	matrix (e) mg		net PM (f) mg	Type of material
1		4.4	3.0	0.0	0.0%	3.0	
3		2.2	2.4	0.0	0.0%	2.4	
4		12.4	16.6	0.0	0.0%	16.6	
7		11.6	9.0	0.0	0.0%	9.0	
9		12.0	24.0	16.0	66.7%	8.0	Puffed rice, plant material
10		2.0	2.0	0.0	0.0%	2.0	
11		51.4	49.0	0.0	0.0%	47.0*	
12		1.8	1.0	0.0	0.0%	1.0	
13		10.6	10.0	0.0	0.0%	10.0	
15		7.8	7.0	0.0	0.0%	7.0	
16		20.0	20.0	2.0	10.0%	18.0	Plant material
18		3.0	2.0	0.0	0.0%	2.0	
19		10.4	0.2	0.0	0.0%	0.2	
20		14.9	21.0	0.0	0.0%	18.0*	
21		5.8	6.0	0.0	0.0%	6.0	
23		12.6	13.0	0.0	0.0%	13.0	
26		97.8	96.0	96.0	100.0%	0	Candy, plant material
28		50.2	49.0	47.0	95.9%	2.0	Candy, puffed rice
29		1.0	1.0	0.0	0.0%	1.0	
30		1391.0	1361.0	1360.0	99.9%	1.0	Puffed rice
31		9.0	8.0	6.0	75.0%	2.0	Candy
32		4.0	1.0	0.0	0.0%	1.0	
35		7.5	4.0	3.0	75.0%	1.0	Puffed rice
36		1124.0	964.0	964.0	100.0%	0.0	Candy, puffed rice
37		2.6	2.0	0.0	0.0%	2.0	
39		70.0	65.0	64.0	98.5%	1.0	Candy
40		8.0	5.0	0.0	0.0%	5.0	
42		16.8	16.0	0.0	0.0%	16.0	
45		3.2	3.0	1.0	33.3%	2.0	Puffed rice

Annex 6 Results: spiked sample (250 mg)

Explanation:
Reported: overestimations in red and underestimation in yellow compared to the interval 66-134%.
Effect of processing, loss: gradient from 0% (green) to 30% (red).
Matrix: gradient from 0% (green) to 86% (red).
PM: packaging material.
Indices in brackets refer to chapter 2.3.

part.	sample	spiked mg	reported (a) mg		returned (b) mg	dried (c) mg	effect processing defatted (d) mg	loss (c+d)	matrix (e) mg		net PM (f) mg	net recovery
1	*605*	249.0	292.0	117%	280.0	274.0	235.0	16%	4.0	1%	231.0	**92.8%**
3	*563*	248.0	406.4	164%	399.9	394.0	376.3	6%	161.7	40%	214.6	**86.5%**
4	*493*	248.5	227.7	92%	206.0	204.0	201.8	2%	7.5	4%	194.3	**78.2%**
7	*521*	248.9	240.5	97%	234.0	230.0	227.1	3%	0.0	0%	227.1	**91.2%**
9	*577*	247.9	116.0	47%	284.0	279.0	237.4	16%	15.5	5%	221.9	**89.5%**
10	*430*	247.2	219.0	89%	212.0	210.0	200.6	5%	2.1	1%	198.5	**80.3%**
11	*598*	249.0	232.6	93%	226.0	221.0	222.8	1%	0.0	0%	222.8	**89.5%**
12	*541*	247.3	262.3	106%	242.0	237.0	204.3	16%	0.0	0%	204.3	**82.6%**
13	*367*	246.8	234.3	95%	226.9	224.9	198.7	12%	1.0	0%	197.7	**80.1%**
15	*360*	245.8	230.1	94%	223.5	220.1	217.9	3%	0.0	0%	217.9	**88.6%**
16	*591*	248.5	246.0	99%	243.0	238.0	230.0	5%	5.0	2%	225.0	**90.5%**
18	*507*	250.7	206.0	82%	205.9	202.0	200.2	3%	1.0	0%	199.2	**79.5%**
19	*423*	248.8	256.6	103%	230.8	223.5	204.5	11%	12.5	5%	192.0	**77.2%**
20	*416*	247.3	887.0	359%	211.9	207.8	203.2	4%	4.3	2%	198.9	**80.4%**
21	*500*	247.4	192.7	78%	188.0	185.9	183.3	2%	0.0	0%	183.3	**74.1%**
23	*584*	249.8	224.5	90%	194.8	191.0	172.2	12%	6.6	3%	165.6	**66.3%**
26	*402*	248.0	260.8	105%	260.9	256.4	253.8	3%	56.8	22%	197.0	**79.4%**
28	*479*	247.4	248.6	100%	242.7	239.7	235.0	3%	60.6	25%	174.4	**70.5%**
29	*570*	247.8	230.0	93%	225.6	221.2	206.0	9%	0.0	0%	206.0	**83.1%**
30	*472*	247.1	2211.0	895%	2208.5	2141.4	2106.3	5%	1889.4	86%	216.9	**87.8%**
31	*409*	248.0	255.0	103%	250.5	247.1	217.8	13%	19.3	8%	198.5	**80.0%**
32	*556*	247.9	150.0	61%	166.9	163.9	164.3	2%	1.2	1%	163.1	**65.8%**
35	*465*	248.5	184.0	74%	184.0	181.9	179.3	3%	4.5	2%	174.8	**70.3%**
36	*549*	248.6	3014.0	1212%	1965.4	1855.1	1698.7	14%	1546.2	79%	152.5	**61.3%**
37	*542*	247.9	222.0	90%	218.0	214.5	208.9	4%	65.7	30%	143.2	**57.8%**
39	*458*	248.5	347.0	140%	338.4	327.5	237.1	30%	68.1	20%	169.0	**68.0%**
40	*451*	248.4	110.0	44%	96.4	93.4	72.3	25%	0.0	0%	72.3	**29.1%**
42	*535*	247.8	190.7	77%	186.1	182.9	182.8	2%	0.0	0%	182.8	**73.8%**
45	*444*	248.4	191.1	77%	187.7	183.1	176.3	6%	0.0	0%	176.3	**71.0%**

Annex 7 Results: spiked sample (250 mg), types of material

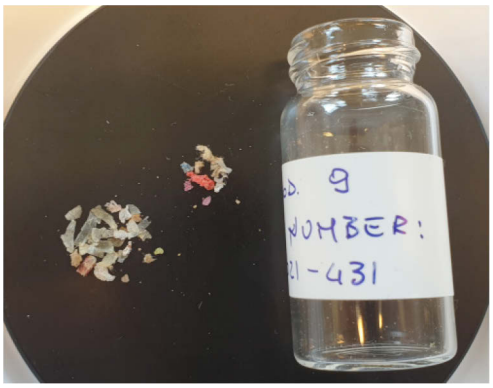
Explanation:
Spiked: amount (mg) of the three types of packaging material spiked in the samples per participant.
Returned: amount (mg) of the three type of packaging material returned by the participants.

part.	spiked			returned		
	paper/board	plastic	tin foil	paper/board	plastic	tin foil
1	136.0	92.0	21.0	124.0	90.0	17.0
3	136.0	93.0	21.0	105.3	92.9	16.4
4	135.7	92.7	22.1	87.0	89.9	17.4
7	136.3	93.3	21.4	122.3	91.6	13.2
9	135.6	93.1	20.5	120.2	92.5	9.2
10	135.6	92.2	20.2	96.7	89.6	12.2
11	137.3	92.7	21.0	121.0	81.8	20.0
12	136.9	92.5	20.8	107.3	81.8	15.2
13	136.4	92.2	20.5	104.6	79.3	13.8
15	136.3	91.8	20.8	128.0	77.0	12.9
16	136.8	92.9	21.2	129.0	78.0	18.0
18	136.8	94.1	21.4	93.0	86.6	19.6
19	136.4	93.3	21.4	72.0	100.0	20.0
20	136.3	92.9	20.6	98.6	83.7	16.6
21	136.7	92.4	21.2	80.9	90.0	12.4
23	136.7	94.1	21.1	91.1	61.5	13.0
26	136.7	92.9	20.7	100.0	81.0	16.0
28	136.2	92.7	20.9	76.3	82.1	16.0
29	135.7	92.7	20.6	113.0	75.7	17.3
30	135.8	93.4	20.3	116.1	81.3	19.5
31	136.0	93.1	21.4	106.3	77.3	14.9
32	136.5	92.0	21.2	71.3	78.4	13.4
35	136.0	93.8	21.4	69.5	89.0	16.3
36	135.8	92.9	21.8	55.0	84.3	13.2
37	136.9	92.5	21.2	38.1	83.6	21.5
39	136.8	92.7	20.8	81.4	70.2	17.4
40	136.8	92.1	20.9	41.1	23.1	8.1
42	135.9	93.2	20.7	83.0	85.5	14.3
45	136.9	93.1	21.2	100.1	56.7	19.5

Annex 8 Example images

Overview of spiked material (left) before sending and returned material after processing (net amounts; participant 1) tube number is independent from participant number.

Overview of spiked material (left) before sending and returned material after processing (net amounts); some particles from plant material and candy in the centre (participant 26) tube number is independent from participant number.



Packaging material (left) and matrix material (right) extracted from the blank (participant 9).

Matrix material, mostly puffed rice and a few candy particles, extracted from the blank (participant (36).

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WFSR Report 2022.009

The mission of Wageningen University & Research is "To explore the potential of nature to improve the quality of life". Under the banner Wageningen University & Research, Wageningen University and the specialised research institutes of the Wageningen Research Foundation have joined forces in contributing to finding solutions to important questions in the domain of healthy food and living environment. With its roughly 30 branches, 7,200 employees (6,400 fte) and 13,200 students and over 150,000 participants to WUR's Life Long Learning, Wageningen University & Research is one of the leading organisations in its domain. The unique Wageningen approach lies in its integrated approach to issues and the collaboration between different disciplines.

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