IAG proficiency test feed composition 2021

L.W.D. van Raamsdonk, C.P.A.F. Smits, B. Hedemann

WAGENINGEN
UNIVERSITY & RESEARCH

IAG proficiency test feed composition 2021

L.W.D. van Raamsdonk, C.P.A.F. Smits, B. Hedemann

Wageningen, October 2022

WFSR Report 2022.021

van Raamsdonk, L.W.D., C.P.A.F. Smits, B. Hedemann, 2022. *IAG proficiency test feed composition 2021.* Wageningen, Wageningen Food Safety Research, WFSR Report 2022.021. 32 pp.; 2 fig.; 5 tab.; 21 ref.

Project number: 1217388901
Project title: IAG ringtesten 2022
Project leader: L.W.D. van Raamsdonk

This report can be downloaded for free at https://doi.org/10.18174/577671 or at www.wur.eu/food-safety-research (under WFSR publications).

P.O. Box 230, 6700 AE Wageningen, The Netherlands, T +31 (0)317 48 02 56, E info.wfsr@wur.nl, www.wur.eu/food-safety-research. WFSR is part of Wageningen University & Research.

WFSR report 2022.021

Distribution list:
- International Association for Feeding stuff Analysis (IAG), Section Feeding stuff Microscopy (G. Frick, J. Vancutsem, R. Weiss, M. Zadravec, J. Dietz)
- All participants of the proficiency test
- European Union Reference Laboratory, Animal Proteins (CRA-W; G. Berben, P. Veys, V. Baeten)
- Joint Research Centre, Geel (IRMM-JRC; C. von Holst, A. Boix-Sanfeliu)
- European Fat Processing and Renderers Association (EFPRA; M. Alm, S. Woodgate)
- European Feed Manufacturers' Federation (FEFAC; A. Döring)
- European Former Foodstuff Processors Association (EFFPA; P. Featherstone, A. van den Brink)
- Ministry of Agriculture, Nature Management and Food Quality (LNV; L.A.M. Claassen, A.D. Wentzel)
- Food and Consumer Product Safety Authority (NVWA; K. Zwaagstra, M. van Brakel, S. Danneel)

Cover photo: equipment for cereal milling in Copenhagen castle mill.

Contents

Summary

A proficiency test was organized for the microscopic determination and semi-quantification of botanic ingredients in the formulation of an animal feed, in the framework of the annual proficiency tests of the IAG - International Association for Feeding stuff Analysis, Section Feeding stuff Microscopy. The organizer of the proficiency test was Wageningen Food Safety Research, The Netherlands. The aim of the proficiency study was to provide the participants information on the performance of the local implementation of the method for composition analysis of feed.

The current proficiency test was focusing on the control of the label declaration of an artificial composition of a cattle feed. Participants received a sample of the feed and were asked to check the correctness of the label information. Therefore, the label declaration was provided together with the sample. The formulation as declared by the label consisted of a wrong composition. Results should show the share of the different ingredients in percentages. Indicated shares were considered under- or overestimations when exceeding the limits of the IAG uncertainty interval model.

A total of 24 sets of results was returned.

Five out of 23 participants submitted correct quantitative results for the nine ingredients to be reported. Ignoring the general false negative reports for the presence of 5% milk powder (16 participants) an additional 10 participants had only one error in their quantitative estimations of the shares. Over 80% of all estimates of individual ingredients is correct within the limits of the IAG estimation model.

An evaluation of the results of four samples investigated for label control, either with a correct or a wrong declaration, extracted from the proficiency tests of 2017, 2019 and 2021 showed that several factors seem to influence the result of a label control. In those cases that an incorrect estimate was reported, this was generally an overestimation for ingredients having shares below 5%. Furthermore, overestimations are predominantly connected to incorrect label declarations exceeding twice the correct value, and underestimations are generally linked to incorrect label declarations less than half the correct share.

The current results show that a label declaration can be confirmed in a qualitative sense. In most cases a correct or at least useful indication of the quantities can be reached. This information is sufficient for monitoring purposes.

The analysis of composition in terms of ingredients is important for detecting economic fraud and for monitoring feed safety. Botanic composition analysis and label control of feed is regulated in Regulation (EC) 767/2009. This technique can support traceability (Regulation (EC) 178/2002), detection of fraud (Regulation (EU) 2017/625) and categorization (Regulation (EU) 1308/2013; Regulation (EU) 2016/1821). In a broader view, composition analysis in the entire food chain can improve the effect of monitoring actions. The legislation on food labelling (Regulation (EC) 1169/2011) obliges to provide more detailed information to customers on composition and related topics.

1 Introduction

In the framework of transparency and the demand for traceability of the source of feed ingredients, it is necessary to establish the formulation of a feed. The legal basis for this examination is the obligatory label declaration of feeds, regulated for years by EU legislation and currently part of Regulation (EC) 767/2009. The main objectives are the prevention of economic fraud and a sufficient monitoring of feed safety.

The analysis of composition of feeds by means of microscopic methods has a long history. It has been a major activity of the IAG section Microscopy from its existence in 1959 (www.iag-micro.org). In 1998 a protocol on the microscopic identification of ingredients in feed was established in German, and translations to English and French were decided to be prepared (http://www.iag-micro.org/files/39_wien98.pdf?10,12).

The method IAG-A2 is based on a procedure of sieving the sample and applying several embedding and staining methods. The different sieve fractions consist of a fine, mediate and coarse material. The presence of specific ingredients (e.g. starch, fibres seed hulls) deviates largely among the sieve fractions. Examinations are to be carried out with both a binocular microscope (up to 70 x magnification) and a compound microscope (100 – 400 x magnification; IAG, s.n.). At the final stages the share of the different ingredients are summed up over the different sieve fractions. The methods relies on identification of the ingredients supported by handbooks or reference material (IAG, s.n.). The identification of legal ingredients (Feed catalogue: Regulation (EC) 68/2013) is a complicated procedure.

Besides the availability of a protocol, the current practices are heavily based on the existing skills of the technicians. In the view of a process of improvement of monitoring programs, which was established for food in Regulation (EC) 1169/2011 (European Union, 2011). The maintenance and dissemination of these skills needs attention.

In this report the proficiency test for composition 2021 is presented, which was organised by Wageningen Food Safety Research (WFSR) in May 2021 on behalf of the IAG Section Feeding Stuff Microscopy.

2 Methods

2.1 Materials and procedure

The IAG proficiency test for botanic composition 2021 was chosen to be based on an artificially produced composition mimicking a cattle feed. The composition consisted of citruspulp (25%), wheat semolina (25%), soybeanmeal (15%), palmkernelmeal (15%), rapeseedmeal (10%), sugar beetpulp (8%), and a mineral mix (2%) consisting of monocalciumphosphate, bicalciumphosphate and calciumcarbonate in equal portions. The choice to produce a matrix from exclusively single ingredients was based on the situation of feed unintentionally contaminated with traces of ruminant material in the 2019 version of the IAG proficiency test (van Raamsdonk et al., 2019). The test was intended for label control.

The IAG proficiency test for botanic composition 2021 was combined with the IAG proficiency test for animal proteins (van Raamsdonk et al., 2021). Sample 2021-C was intended for composition analysis. The composition as originally declared was completed by adding 5% milk powder. The resulting shares of the ingredients are presented in Table 1. An incorrect label was distributed, which is also presented in Table 1.

Table 1 *intended composition and label declaration of sample 2021-C. The min-max range is calculated according to the IAG model for uncertainty limits; see paragraph 2.3.*

	correct formulation	range (MIN-MAX)	label
wheat	23.75%	13.75%-33.75%	25%
citruspulp	23.75%	13.75%-33.75%	25%
palmkernelfeed	14.25%	7.13%-21.38%	6%
soybeanmeal	14.25%	7.13%-21.38%	6%
rapeseedmeal	9.50%	4.50%-14.50%	10%
beetpulp	7.60%	2.60%-12.60%	8%
minerals	1.90%	0.00%-3.80%	2%
cornglutenfeed	0.00%	0.00%-2.00%	13%
milk powder	5.00%	0.00%-10.00%	
molasse	0.0%		5%

The microscopists of WFSR did not participate in the proficiency test.

2.2 Organization of the proficiency test

All IAG members, all NRLs, participants of former proficiency tests and a series of possibly interested laboratories were informed about the proficiency test for 2021. In all cases an invitation letter included in the IAG Newsletter 2020 and a participation form were distributed. Until the beginning of April 2021 a total of 26 participants for the microscopic composition analysis were listed. The samples with an accompanying letter (see Annex 1) were sent to all participants on Tuesday 25th of May 2021. On Friday May 28th an E-mail message was sent to all participants, together with a file containing a sheet with instructions and the electronic report forms, and the request to confirm the receipt of the package. The information sheet of the report form is shown in Annex 1, the form for the procedural survey is reproduced in Annex 2, the report form for the label control is presented in Annex 3, and the letter sent with the samples is reproduced in Annex 4. The participants were requested to submit the correct composition of the sample.

The closing date for reporting results was fixed at 24th of June. Several requests were received to extent the period for analysis. Results received after the date at which the evaluation of the results was started were ignored. The analysis of the results was carried out during Autumn 2021. A further evaluation of the results in the framework of the results of previous proficiency tests was carried out in Spring 2022 and the final report was written in May 2022.

The samples were intended to be analysed according to IAG method 2: "Method for the Identification and Estimation of Constituents in Animal Feedingstuff" (IAG, s.n.). Further instructions to the participants were enclosed in the box with samples, which are reproduced in Annex 4.

2.3 Analysis of results

The results are analysed according to the IAG scheme of uncertainty limits as approved during the 2006 meeting in Rostock and include in IAG method A2. These limits are presented in Table 2. The model is graphically presented in Figure 1. Shares of ingredients in the reported feed formulation outside the limits of the model were indicated as "wrong".

Table 2 *IAG model for uncertainty analysis of the composition of a compound feed.*

Actual amount in %	Accepted uncertainty limits
< 2%	"traces "
2.0 – 5.0%	+/- 100% relative
5.01 – 10.0%	+/- 5% absolute
10.01 – 20.0%	+/- 50% relative
– 50.0%	+/- 10% absolute
> 50%	+/- 20% relative

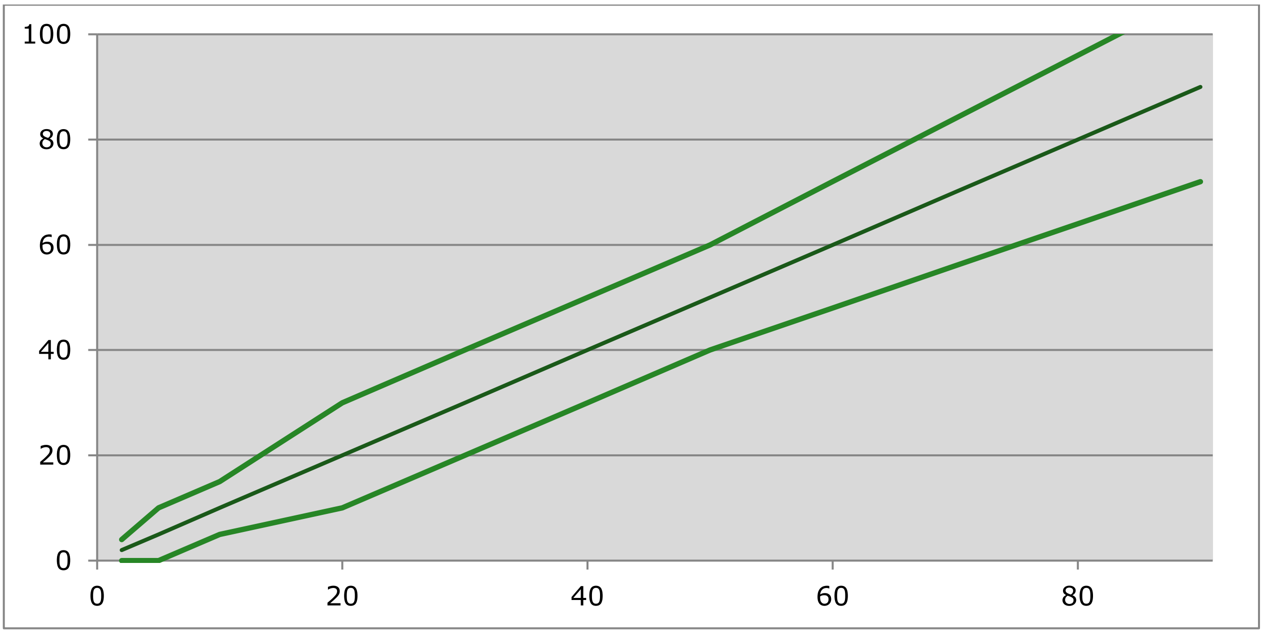

Figure 1 *IAG model for estimating uncertainty. X-axis: correct portion of ingredient in %, Y-axis: estimated portion of ingredient in %. Inner line: correct estimation, outer lines: limits for uncertainty interval at a given percentage.*

3 Results

Twenty-six samples were sent to all participants and results for composition analysis were returned by 24 participants. In all those cases that a participant submitted several versions of the report sheet the most recent version was used. Participant 20 submitted an incomplete report with three indicated ingredients. The missing occurrences (six ingredients) in this report were excluded from the calculations for underestimations.

The 24 participants, which successfully submitted their results, originated from 10 countries: 8 member states of the European Union, and two other countries. The list of participants is presented in Annex 5. Half of the participants originated from Germany (12).

The procedure for the analysis of the composition of a compound feed is described in IAG method A2 (IAG, s.n.). This method, familiar to most participants as members of IAG section Microscopy, was applied by 19 participants. Other applied methods include the VDLUFA III 30.7 method, AFNOR V18A method and internal laboratory procedures.

3.1 Composition

The results of the 24 participants are fully presented in Annex 6 and summarised in Table 3. The evaluations will be based on the pooled results per participants for the wheat products and for the corn products, since some participants did not discriminate between the specific types.

Table 3 *overview of the main ingredients of the analysed sample, the label declaration, the a-priori calculated uncertainty range, and the statistics of the results in terms of median and numbers of participants that under- or overestimated the share of the ingredients. N = 23.*

ingredient	label	correct range	median	# (%) under est.	# (%) over est.
wheat	25%	13.75-33.75%	27.2%	0	5 (22%)
citruspulp	25%	13.75-33.75%	20.0%	4 (17%)	0
palmkernelfeed	6%	7.13-21.38%	10.0%	6 (26%)	1 (4%)
soybeanmeal	6%	7.13-21.38%	8.7%	5 (22%)	0
rapeseedmeal	10%	4.5-14.5%	10.0%	2 (9%)	0
beetpulp	8%	2.6-12.6%	8.5%	2 (9%)	1 (4%)
minerals	2%	0-3.8%	2.0%	2 (9%)	0
cornglutenfeed	13%	0.0-2.0%	0.1%	0	10 (43%)
milk powder		0.0-10.0%	0.0%	16 (70%)	0
molasse	5%	0.0-2.0%			

There were major differences between the declared amount of palmkernel meal, soybean meal, cornglutenfeed, molasse and of milk powder with the actual amounts for these five ingredients. The reported shares of the ingredients with a lower limit in the IAG estimations model higher than zero (first six ingredients in Table 3) were within the limits of the uncertainty model in 88.9% of the total number of estimations (n=138). The correct estimations of all reports of the shares of nine ingredients is 75.8% (n=207). Some participants made their estimations ignoring the molasse. This ingredient is not evaluated. Milk powder was not reported by 16 participants. This is a major share in the number of underestimations. Ignoring this, the percentage of correct reports over the remaining eight ingredients is 83.6% (n=184). This is presented graphically in Figure 2. Five out of 23 participants delivered a composition with all estimations complying to the IAG model, which is 22%. Besides this, two participants made one error, eight made two errors and six participants made three errors. Here as well, the 16 false negative reports for milk presence are a major source for the number of errors made by the participants. Excluding this, still five participants submitted overviews for the composition with all estimates within the limits of the IAG model. The number of participants with one error increased to ten. The full profiles of participants' performances is presented in Table 4.

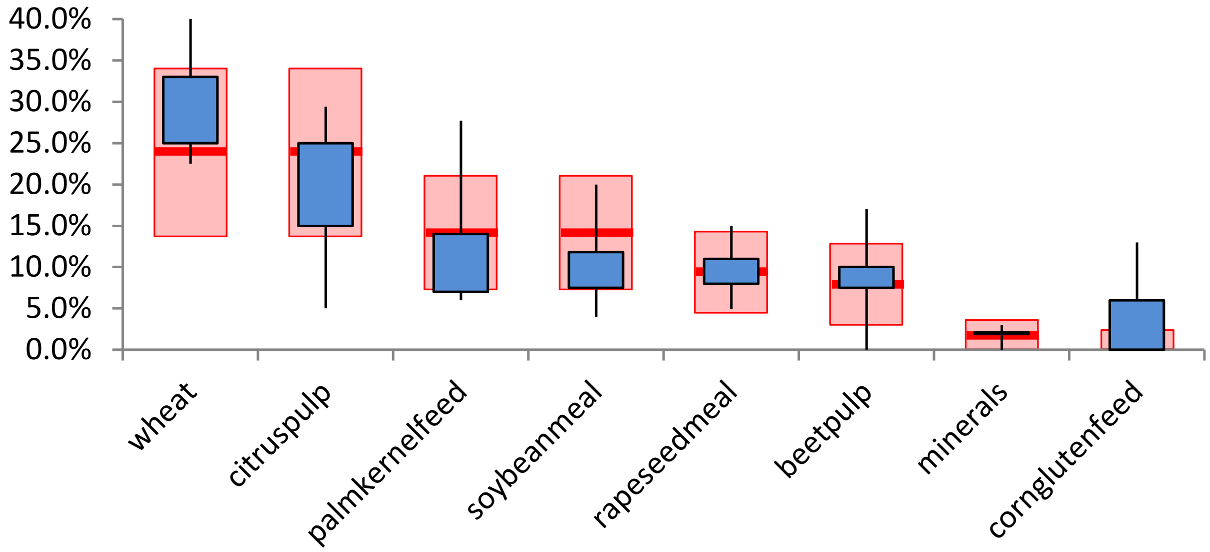

Figure 2 *the results of the IAG proficiency test composition 2021. Y-axis: share of ingredient. Blue bars: P_{25} – P_{75} percentile interval, vertical line: minimum – maximum range. Red background: upper and lower limit interval of the IAG uncertainty model, horizontal red line: share of the ingredient. N=23.*

Table 4 *overview of the number of participants making zero, one, two, etcetera errors in the estimations of nine ingredients, or eight ingredients excluding milk powder. N = 23.*

Number of errors / Number of participants	0	1	2	3	4	5
Including nine ingredients	5	2	8	1	6	1
Including eight ingredients, without milk powder	5	10	1	6	1	0

Considering the choice to use an artificial formulation, indications for the type of feed cannot be evaluated. Three participant judged the label declaration as incorrect.

4 Discussion

4.1 Justification for the establishment of composition

European legislation requires that feeds and feed materials are labelled according to a range of requirements, including composition. It has been stated that labelling serves enforcement, traceability and control purposes (Regulation (EC) 767/2009, pre-ambule 17).

There are several aspects in the monitoring of feed safety and security where the analysis of botanic composition or identity is a major tool for enforcement:

- Label control (Regulation (EC) 767/2009).
- Traceability (Regulation (EC) 178/2002).
- Detection of fraud (Regulation (EU) 2017/625 repealing Regulation (EC) 882/2004).
- Organisation of markets for agricultural products demanding tools to identify materials for proper categorization (Regulation (EU) 1308/2013; Regulation (EU) 2021/1832).[1]

In a declaration of composition feed materials should be mentioned in order of decreasing share, and additional information on composition should be available on request with uncertainty limits of +/- 15% (Regulation (EC) 767/2009, Article 17). It is not stated if this is a relative or absolute range. Annex IV of Regulation (EC) 767/2009 presents requirements for the labelling of basic parameters such as crude proteins, crude fibres, sugars, starch, oils and fats, minerals, moisture, crude ash and related parameters with a mix of absolute and relative ranges.

A most important aspect is the possibility to redirect the presence of a prohibited substance to one of the ingredients or fractions. Knowledge of the ingredients in a compound feed eases the traceability of prohibited substances. The fractionation of a sample in a sediment and a flotate can help to pinpoint the presence of contaminants and might improve their traceability. A multidisciplinary approach for evaluating incidences in the area of feed and food safety is a major achievement.

Economic fraud can be based on the replacement of an expensive ingredient by a cheaper one. Another aspect is the possibility that ingredients not fit for animal consumption, i.e. due to mould infestation, can be mixed in compound feeds.

In a broader view, composition analysis in the entire feed chain can improve the effect of monitoring actions. Likewise, legislation on food labelling (Regulation (EC) 1169/2011) obliges to provide more detailed information to customers on composition and related topics.

Strict quality assurance measures for monitoring feed and food products are demanded upon getting into force of Regulation (EU) 2017/625. Proficiency tests, as the current one on botanic composition, will gain in importance in the framework of the extended requirements.

4.2 Label control

In general, ingredients with a high share in the composition are commonly underestimated, and ingredients with a low share are usually overestimated. This applies to blind establishment of botanic composition of compound feed as well as for label control (van Raamsdonk et al., 2014; 2017). An exception was found in the results of the 2015 test. Total wheat (51.7%) was still slightly overestimated in the presence of 7.5%

[1] Regulation (EU) 2021/1832 is the most recent version of the 'Çustoms Handbook' listing all the import regulations. Imported lots and batches need to be correctly classified in order to find the applicable measures. This Regulation is renewed annually based on Council Regulation (EEC) No 2658/87.

bakery by-product, which was generally overlooked (van Raamsdonk et al., 2015). Another issue was the discrimination between citrus and beet pulp (van Raamsdonk et al., 2016, 2017).

The usual application of composition analysis of compound feeds in practice is label control, also because of the legal demands. Therefore, an evaluation will be presented of the past proficiency tests for composition analysis where a label declaration was available, either correct or incorrect. In general over 80% of the occurrences of an ingredient in a formulation appears to be correctly estimated within the limits of the IAG estimation model. The way this result is or should be retrieved will be discussed in the next section of this discussion. Here, an evaluation of the results will be presented in relation to the information of the provided label declaration.
The results of the proficiency tests of 2017 (two samples), 2019 (one sample) and 2021 (one sample, this report) will be included. The overview of these results is presented in Table 5.

Table 5 *Overview of results of composition analysis of compound feeds with correct (top) and incorrect (bottom) label declaration. For every set of results the correct share in the formulation, the label declaration, the number of participants (frequency) of which the report appeared to be under- or over-estimation is given. Colours of cells indicate: green: ingredient with a share of less than 5%; red: incorrect label declaration with at least double the correct share; blue: incorrect label declaration lower than half the correct share; yellow: frequency of participants exceeding 15%.*

	2017-correct label (n=20)				2019-correct label (n=22)			
	level	label	under	over	level	label	under	over
rapeseedmeal	5.0%	6.0%	0	2 (10%)	8.0%	8.0%	1 (5%)	0
grass and lucernemeal					0%	1.0%		7 (32%)
soybeanmeal	5.0%	6.0%	2 (10%)	1 (5%)	1.2%	1.2%		8 (36%)
corn total	27.5%	27.0%	2 (10%)	0	24.0%	24.0%	5 (23%)	0
milk powder								
palmkernelfeed					16.0%	16.0%	2 (9%)	2 (9%)
oat husks	2.5%	3%		8 (40%)				
coconutmeal					1.0%	1.0%		4 (18%)
wheat total	44.0%	44.0%	1 (5%)	2 (10%)	15.0%	15.0%	3 (14%)	0
citruspulp					10.0%	10.0%	2 (9%)	3 (14%)
beetpulp	0.0%			1 (5%)	13.0%	13.0%	0	1 (5%)
sunflower	12.5%	12%	0	0				
minerals	2.3%	2.0%	0	0	0.6%	<1%	0	0

	2017-wrong label (n=19)				2021-wrong label (n=23)			
	level	label	under	over	level	label	under	over
rapeseedmeal	5.0%	12.0%	1 (5%)	4 (16%)	9.50%	10.00%	0	1 (4%)
grass and lucernemeal								
soybeanmeal	5.0%	12.0%	2 (11%)	3 (16%)	14.25%	6.00%	5 (21%)	0
corn total	27.5%	8.0%	3 (16%)	0	0.00%	13.00%	0	10 (42%)
milk powder					5.00%		16 (67%)	0
palmkernelfeed					14.25%	6.00%	5 (21%)	1 (4%)
oat husks	2.5%			4 (21%)				
coconutmeal								
wheat total	44.0%	40.0%	2 (11%)	0	23.75%	25.00%	0	5 (21%)
citruspulp					23.75%	25.00%	4 (16%)	0
beetpulp	0.0%			1 (5%)	7.60%	8.00%	1 (4%)	1 (4%)
sunflower	12.5%	16.0%	1 (5%)	1 (5%)				
minerals	2.3%	3.0%	0	1 (5%)	1.90%	2.00%	1 (4%)	0

The different combinations of under- or over-estimations with correct or incorrect label declarations show different backgrounds:

- There are five occurrences of an ingredient below 5%, excluding minerals (Table 5, marked green). In all five cases there was an overestimation by more than 15% of the participants.
- In three cases the label declaration showed an excess of over 100% of the correct share (Table 5, marked red). This resulted in an overestimation by more than 15% of the participants in all three cases.
- In four cases the declared level was lower than 50% of the correct level (Table 5, marked blue). In all cases an underestimation was reported by more than 15% of the participants.
- Except for one case (2019: corn) and the mentioned ingredients with a share below 5%, all estimations were correctly reported by more than 85% of the participants in the presence of a correct label. All other under- and over-estimations were made in the presence of an incorrect label.
- There are some inconclusive results, not linked to wrong declarations for that ingredient. These are corn (sum of all types, 2019), wheat (2021) and citruspulp (2021) with more than 15% of the participants reporting incorrect results. It could be assumed that these ingredient shares are finally adjusted to get the final 100% composition. Corn (2019) was underestimated in the presence of overestimation of two ingredients with a share below 5%. Wheat and citrus show mutual excluding under- and over-estimations in the 2021 proficiency test (Appendix 6).
- The ingredients beetpulp and sunflowermeal were almost exclusively correctly estimated within the limits of the IAG model. This could be attributed to the clearly recognisable fragment of these ingredients.
- The estimations of the share of minerals, although usually far below 5%, are correct in most cases. This is due to the procedural consequence of an exact extraction by applying sedimentation. The use of a separation procedure based on specific density can also be applied to vegetal ingredients. Rapeseedmeal, soybeanmeal, corn and wheat show a specific density below 1.42 g/cm^3, whereas palmkernelfeed, citruspulp and beetpulp exceed that value (IAG Newsletter, 2020; van Raamsdonk et al., 2022).
- A special case is milk powder. This was included in the sample for this year at a level of 5%, but excluded in the label declaration. It is a not expected ingredient for a mainstream formulation and it might be overlooked for this reason. Another factor might be its difficult detection even if the technician is aware of the presence of milk powder. These options were discussed in the report on the proficiency test for animal proteins of 2021, where this sample was included for the detection of animal proteins. Options for a further evaluation of this underperformance have been proposed (van Raamsdonk et al., 2021).

With the exception of milk powder, the frequency of incorrect estimations is below 42% for all occurrences, with the highest frequencies for those ingredients with a share of less than 5%. The bias in incorrect label declarations is generally translated to the estimations: most underestimations are connected to lower declarations than actually present, and overestimations are mostly linked to exceeding label declarations. This could be due to the a-priori hypothesis that a label declaration is assumed to be correct unless results of examinations falsify this hypothesis. Also in declarations of dedicated feed errors can exist, which was demonstrated for the ruminant feed used in the proficiency test of 2019 (van Raamsdonk et al., 2019). A-priori information should not influence the inspection of a sample for composition. Blind examination followed by a comparison with the label declaration after the collection of all the results could solve this issue, but is at least evenly complicated. Starting the process of estimating the shares of ingredients in a compound feeds with the most abundant ingredients could help to avoid overestimation of the minor ingredients.

In a qualitative sense, the results in general show very good results. Only a few ingredients have been deliberately reported which were not listed in the label declaration and which were absent in the sample. These are beet pulp (Table 5) and apple pomace (2017), sunflower, peanet expeller and DDGS (2019), cichory pulp, oats and coconutmeal (2021), in all occasions by only one participant. These results show that the recognition of the ingredients can well be achieved in a qualitative way and that identification and quantification are two separate aspects.

4.3 Composition analysis

There are two aspects influencing the quality of the results of an analysis of botanic composition. The first one is the lack of supporting evidence for compensating possible under- and over-estimation. The sole visual analysis of the composition of a compound feed without complementary information lacks the possibility of confirmation and of adjustment, which might influence the precision of the final result. In the past assessment of the biological composition of a feed was supported and, if necessary, adjusted by proximate analysis, of which Weende analysis is the classical approach (German: Weender Analyse; https://de.wikipedia.org/wiki/Futtermittelanalytik). Weende analysis, originally developed in the 19th century (Henneberg and Stohmann, 1859) provides information on basic chemical parameters: moisture, contents of ash, fat, protein and crude fibres. Since extensive information is available on the parameters of individual ingredients, the initial visually estimated shares of the several biological ingredients can be confirmed or optimized using this detail data. Currently several approaches exist for proximate analysis, such as Cornell Net Carbohydrate and Protein System (CNCPS), Van Soest and Near-Infrared Reflectance Spectroscopy, although these systems do not aim at the same set of parameters (Bovera et al., 2003; Godoy et al., 2016).

The second aspect is the level of expertise of the technician. In specific situations a particular ingredient might be overlooked. Because of this situation, a knowledge system for identification of ingredients of compound feeds could assist in composition analysis. Such a system should be able to document individual identifications as well in order to store choices made in the process of identification.

A good process description consisting of necessary steps, sieve fractions, the necessary parameters to be established in every step and for every fraction, and the procedure for combining all data in a final conclusion, together with documentation on identifying feed ingredients could help to improve the method.

5 General conclusions and recommendations

5.1 Conclusions

Five out of 23 participants submitted correct quantitative results for the nine ingredients to be reported. Ignoring the general false negative reports for the presence of 5% milk powder (16 participants) an additional 10 participants had only one error in their quantitative estimations of the shares. Over 80% of all estimates of individual ingredients is correct within the limits of the IAG estimation model.

An evaluation of the results of four samples investigated for label control, either with a correct or a wrong declaration, extracted from the proficiency tests of 2017, 2019 and 2021 showed that several factors seem to influence the result of a label control. In those cases that an incorrect estimate was reported, this was generally an overestimation for ingredients having shares below 5%. Furthermore, overestimations are predominantly connected to incorrect label declarations exceeding twice the correct value, and underestimations are generally linked to incorrect label declarations less than half the correct share.

The current results show that a label declaration can be confirmed in a qualitative sense. In most cases a correct or at least useful indication of the quantities can be reached. This information is sufficient for monitoring purposes.

The current information on the capability of botanic composition analysis reveals that this technique is valuable as part of the enforcement of feed and food safety. Besides proper label control (Regulation (EC) 767/2009), composition analysis can support traceability (Regulation (EC) 178/2002), for detection of fraud (Regulation (EU) 2017/625) and for categorization (Regulation (EU) 1308/2013; Regulation (EU) 2016/1821).

The current lack of a complementary system for the analysis of chemical composition (ash, proteins, fat, dietary carbohydrates, fibres, etc.) could be a drawback for the overall approach of. Besides a proper method description and up-to-date descriptions of ingredients, well developed skills of technicians are vital for a good performance. An identification support system for the major feed ingredients would provide the necessary support for identification.

5.2 Recommendations

- A more detailed process description could help to optimise the method for establishing the composition of a compound feed.
- In the view of the need for proper means for identification, an expert system as tool for maintenance and dissemination of expertise may help to improve future performance.

Acknowledgements

The board of IAG section Feeding Stuff Microscopy (dr. G. Frick (Agroscope, Posieux), ir. J. Vancutsem (FAVV, Tervuren), R. Weiss (AGES, Vienna), J. Dietz (SGS, Hamburg), M. Zadravec (Croatian Veterinary Institute, Zagreb) supported this study as advisory board for communication with the scientists and laboratories working in this research field, and in the final report activities. Their contributions are greatly acknowledged. Thanks to colleagues M. van Iersel, A. Verschoor and G. van der Borg for the internal WFSR review procedure.

References

Bovera, F., M. Spanghero, G. Galassi, F. Masoero, A. Buccioni, 2003. Repeatability and reproducibility of the Cornell Net carbohydrate and Protein System analytical determinations. Ital. J. Anim. Sci. 2: 41-50.

European Commission, 2013. Commission Regulation (EU) No 68/2013 of 16 January 2013 on the Catalogue of feed materials. OJ L 29, 30.1.2013, p. 1–64.

European Commission, 2021. Commission Implementing Regulation (EU) 2021/1832 of 12 October 2021 amending Annex I to Council Regulation (EEC) No 2658/87 on the tariff and statistical nomenclature and on the Common Customs Tariff. OJ L 385, 29.10.2021, p. 1-1091.

European Union, 2002. Regulation (EC) No 178/2002 of the European Parliament and of the Council of 28 January 2002 laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety. OJ L 31, 1.2.2002, p. 1-24.

European Union, 2004. Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. OJ L 165, 30.4.2004, p. 1-141.

European Union, 2009. Regulation (EC) No 767/2009 of the European Parliament and of the Council of 13 July 2009 on the placing on the market and use of feed, amending European Parliament and Council Regulation (EC) No 1831/2003 and repealing Council Directive 79/373/EEC, Commission Directive 80/511/EEC, Council Directives 82/471/EEC, 83/228/EEC, 93/74/EEC, 93/113/EC and 96/25/EC and Commission Decision 2004/217/EC. OJ L 229, 1.9.2009, p. 1–28.

European Union, 2011. Regulation (EU) No 1169/2011 of the European Parliament and of the Council of 25 October 2011 on the provision of food information to consumers, amending Regulations (EC) No 1924/2006 and (EC) No 1925/2006 of the European Parliament and of the Council, and repealing Commission Directive 87/250/EEC, Council Directive 90/496/EEC, Commission Directive 1999/10/EC, Directive 2000/13/EC of the European Parliament and of the Council, Commission Directives 2002/67/EC and 2008/5/EC and Commission Regulation (EC) No 608/2004. OJ L 304, 22.11.2011, p. 18–63.

European Union, 2013. Regulation (EU) No 1308/2013 of the European Parliament and of the Council of 17 December 2013 establishing a common organisation of the markets in agricultural products and repealing Council Regulations (EEC) No 922/72, (EEC) No 234/79, (EC) No 1037/2001 and (EC) No 1234/2007. OJ L 347, 20.12.2013, p. 671–854.

European Union, 2017. Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017 on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products, amending Regulations (EC) No 999/2001, (EC) No 396/2005, (EC) No 1069/2009, (EC) No 1107/2009, (EU) No 1151/2012, (EU) No 652/2014, (EU) 2016/429 and (EU) 2016/2031 of the European Parliament and of the Council, Council Regulations (EC) No 1/2005 and (EC) No 1099/2009 and Council Directives 98/58/EC, 1999/74/EC, 2007/43/EC, 2008/119/EC and 2008/120/EC, and repealing Regulations (EC) No 854/2004 and (EC) No 882/2004 of the European Parliament and of the Council, Council Directives 89/608/EEC, 89/662/EEC, 90/425/EEC, 91/496/EEC, 96/23/EC, 96/93/EC and 97/78/EC and Council Decision 92/438/EEC (Official Controls Regulation)Text with EEA relevance. OJ L 95, 7.4.2017, p. 1–142.

Godoy, M.R.C. de, M. Hervera, K.S. Swanson, G.C. Fahey Jr., 2016. Innovations in Canine and Feline Nutrition: Technologies for Food and Nutrition Assessment. Annu. Rev. Anim. Biosci. 4: 311–333.

Henneberg W, Stohmann F. 1859. Ueber das erhaltungsfutter volljaehrigen rindviehs. J. Landwirtsch. 3: 485–551.

IAG, s.n. Method for the Identification and Estimation of Constituents in Animal Feedingstuff. IAG-Method A2. http://www.iag-micro.org/files/iag-a2_identification_estimation.pdf.

IAG, 2020. IAG Newsletter 2020. Distributed by WFSR.

Raamsdonk L.W.D. van, B. Hedemann, C.P.A.F. Smits, T.W. Prins, 2022. Specific density as parameter for separating feed ingredients. Biotechnologie, Agronomie, Société et Environnement, submitted.

Raamsdonk L.W.D. van, C.P.A.F. Smits, B. Hedemann, T.W. Prins, 2021. IAG proficiency test animal proteins 2021. Report 2021.019. WFSR, Wageningen, pp. 36.

Raamsdonk L.W.D. van, C.P.A.F. Smits, B. Hedemann, T.W. Prins, J.J.M. Vliege, 2019. IAG proficiency test animal proteins 2019. Report 2019.015. WFSR, Wageningen, pp. 36.
Raamsdonk L.W.D. van, C.P.A.F. Smits, J.J.M. Vliege, 2019. IAG proficiency test feed composition 2019. Report 2019.014. WFSR, Wageningen, pp. 26.
Raamsdonk, L.W.D. van, C.P.A.F. Smits, J.J.M. Vliege, V.G.Z. Pinckaers, 2017. IAG ring test feed composition 2017. Report 2017.011. RIKILT, Wageningen, pp. 32.
Raamsdonk, L.W.D. van, N. van de Rhee, V.G.Z. Pinckaers, J.J.M. Vliege, 2015. IAG ring test feed composition 2015. Report 2015.017. RIKILT, Wageningen, pp. 26.
Raamsdonk, L.W.D. van, N. van de Rhee, V.G.Z. Pinckaers, J.J.M. Vliege, 2016. IAG ring test feed composition 2016. Report 2016.014. RIKILT, Wageningen, pp. 26.
Raamsdonk, L.W.D. van, V.G.Z. Pinckaers, J.J.M. Vliege, 2014. IAG ring test feed composition 2014. Report 2014.010. RIKILT, Wageningen, pp. 23.

Annex 1 Basic instructions for the test procedure

	IAG proficiency test 2021 composition
	Instructions for the IAG proficiency test
1	You have received a box with an introduction letter and either one or four vials containing 50 grams of possibly contaminated animal feed. Please report the receipt of your package as soon as possible by E-mail to the address mentioned below. If subscribed, one of the four or the only one sample is meant for composition analysis. The number of the sample is mentioned on the letter enclosed in the box.
2	Analysis for composition is preferably carried out using method A2 of the IAG section Microscopy. Other methods, however, are allowed. Take care to homogenise the content of each vial before taking the amount for analysis.
	Link to IAG method A2
3	The results need to be reported as percentual estimations on the tab "Results". The organiser will apply the uncertainty intervals to your estimations as part of the evaluation. Reporting consists of the following steps:
3a	Please fill in the questionnaire on the page "Procedure". Most of the cells contain a drop-down list. These lists can be used to select an answer as follows. When clicking on a cell, the cursor changes into a hand. A second click will open the drop-down list. Your unique lab number is mentioned in the introduction letter. All the fields with a drop-down list have to be completed.
3b	Please enter your results in the fields at page "Results". Your unique lab number automatically shows up after you have entered it at the page Procedure. Enter yourself the unique label of the vial.
4	After completing the two forms "Procedure" and "Results", they have to be sent to the organisers in two ways:
4a	Save the 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 sent by E-mail as Excel file and as a scan (*.PDF) to leo.vanraamsdonk@wur.nl and to corina.smits@wur.nl.
4b	Results will be included in the final evaluation and report only if both forms are sent in by electronic mail, and after the proper receipt of the requested fee.
5	Direct any questions to leo.vanraamsdonk@wur.nl
6	Closing date is June 24th, 2021.
WFSR	Wageningen UR, the Netherlands

Annex 2 Report form for procedure details

Please complete at least all the pink cells with a drop down list that apply to your procedure	select your choice from a drop down list	type in your answer if necessary
IAG proficiency test 2021 composition		
Please select your unique lab number	**-- select --**	
Have you read the proficiency test instructions?	-- select --	
Which detection method do you use?	-- select --	

Annex 3 Report forms results

Please complete the necessary pink cells for showing your composition of the ingredients; add your sample number, the sediment amount and the final conclusion on feed type.	
IAG ring test 2021 composition	
lab number	
sample number	
	estimated %
Total:	0.0
Final conclusion on feed type:	-- select --
Comment if necessary	

Signature:

Date: 6-5-2019

Annex 4 Additional instructions

Test 2021-B: botanic composition of sample: [ ]
The sample with the number indicated here ↑ is meant for the analysis of the botanic composition. For this year the purpose of label control is chosen. Therefore, below you will find the label information for the declared composition. Take care to homogenise the content of the vial before taking the amount for analysis. This sample will be used for two purposes: detection of animal proteins if you have subscribed to this proficiency test, and analysis of botanic composition.

The report form contains a series of empty rows where the ingredients can be entered in order of their share (highest share on top), together with your estimation of the share in percent in the second column. Finally you are requested to indicate the correctness of the label (yes/no) and the assumed target of the feed (e.g. broiler feed, calve feed).

All results can be entered in the report form with “composition” in the name.

Label declaration	
Citruspulp	25%
Wheat	25%
Cornglutenfeed	13%
Rapeseedmeal	10%
Beet pulp	8%
Soybeanmeal	6%
Palmkernelmeal	6%
Minerals	2%
Molasse	5%

Annex 5 List of participants

Austrian Agency for Health and Food Safety-AGES	Austria
FLVVT	Belgium
Canadian Food Inspection Agency	Canada
Danish Veterinary and Food Administration	Denmark
Inovalys-Nantes	France
Laboratoire Départemental d’Analyse & de Recherche	France
BWZ der BFV	Germany
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 Germany GmbH	Germany
Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft, GB6-Labore Landwirtschaft / LUFA, FB62	Germany
National Food Chain Safety Office	Hungary
Ministero delle politiche agricole alimentari e forestali, Laboratorio di Modena	Italy
ForFarmers	Netherlands
Nutreco Nederland BV - Masterlab	Netherlands
Nutrilab BV	Netherlands
Agroscope (ALP), Swiss Research Station	Switzerland

Annex 6 Results composition analysis

					1	7	10	11	13	14	15	16	19	20	21	23
	correct	**range:**		**label**	**376**	**432**	**439**	**425**	**418**	**355**	**404**	**488**	**348**	**362**	**467**	**117**
wheat	23.75%	13.75%	33.75%	25%	26.0%	25.0%	27.1%	33.0%	32.0%	31.5%	38.8%	34.0%	39.0%		25.0%	25.0%
citruspulp	23.75%	13.75%	33.75%	25%	25.0%	25.0%	26.4%	27.0%	16.0%	11.0%	25.7%	18.0%	15.0%		20.0%	10.0%
palmkernelfeed	14.25%	7.13%	21.38%	6%	15.0%	10.0%	12.4%	9.0%	10.0%	17.0%	6.6%	12.0%	8.0%		15.0%	8.0%
soybeanmeal	14.25%	7.13%	21.38%	6%	10.0%	8.0%	8.3%	11.0%	12.0%	11.0%	6.8%	8.0%	10.0%	15.0%	10.0%	15.0%
rapeseedmeal	9.50%	4.50%	14.50%	10%	6.0%	8.0%	8.5%	10.0%	10.0%	13.0%	10.8%	11.0%	12.0%		8.0%	15.0%
beetpulp	7.60%	2.60%	12.60%	8%	8.0%	10.0%	8.5%		11.0%	11.0%	9.3%	8.0%	9.0%		8.0%	5.0%
minerals	1.90%	0.00%	3.80%	2%	2.0%	2.0%	2.5%		3.0%	1.5%	2.0%	2.0%	2.0%		2.0%	2.0%
cornglutenfeed	0.00%	0.00%	2.00%	13%	0.1%	2.0%	2.7%	5.0%	6.0%		0.0%		0.0%	0.0%		5.0%
milk powder	5.00%	0.00%	10.00%		present	3.0%	1.0%					7.0%		2.0%	7.0%	
molasse				5%	5.0%	5.0%			[5%]	4.0%			5.0%		5.0%	5.0%
probably minerals						2.0%										
minerals+beet+molasse+milk								5.0%								
ammoniumphosphaat																
cichory pulp																10.0%
traces barley, oat, rye						traces	2.6%									
oats																
sunflower						traces										
coconutmeal																
cotton fibres																
	100%			100%	97%	100%	100%	100%	100%	100%	100%	100%	100%	17%	100%	100%

					24	**27**	**29**	**32**	**33**	**35**	**37**	**38**	**40**	**42**	**44**	**45**
	correct	**range:**		**label**	**278**	**229**	**327**	**180**	**208**	**201**	**173**	**166**	**131**	**257**	**264**	**236**
wheat	23.75%	13.75%	33.75%	25%	35.7%	27.2%	25.0%	24.0%	25.0%	26.0%	25.0%	35.0%	22.5%	32.0%	32.0%	28.0%
citruspulp	23.75%	13.75%	33.75%	25%	29.4%	17.8%	25.0%	20.0%	25.0%	19.5%	25.0%	20.0%	22.5%	10.0%	5.0%	19.0%
palmkernelfeed	14.25%	7.13%	21.38%	6%	6.2%	27.7%	6.0%	14.0%	7.0%	13.9%	6.0%	9.0%	15.0%	14.0%	10.0%	9.0%
soybeanmeal	14.25%	7.13%	21.38%	6%	6.3%	11.8%	6.0%	16.0%	20.0%	8.3%	6.0%	8.0%	7.5%	8.0%	4.0%	9.0%
rapeseedmeal	9.50%	4.50%	14.50%	10%	10.7%	4.9%	10.0%	8.0%	8.0%	10.1%	10.0%	10.0%	12.5%	14.0%	10.0%	9.0%
beetpulp	7.60%	2.60%	12.60%	8%	9.9%	8.5%	9.0%	6.0%	8.0%	3.8%	8.0%	10.0%	7.5%	8.0%	17.0%	10.0%
minerals	1.90%	0.00%	3.80%	2%	1.8%	2.1%	3.0%	2.0%	2.0%	2.0%	2.0%	2.0%	3.0%	1.9%	1.2%	3.0%
cornglutenfeed	0.00%	0.00%	2.00%	13%	0.0%		12.0%	0.0%		10.1%	13.0%		5.0%	7.0%	13.0%	
milk powder	5.00%	0.00%	10.00%					10.0%								8.0%
molasse				5%			4.0%		5.0%	5.0%	5.0%	5.0%		5.0%	5.0%	5.0%
probably minerals																
minerals+beet+molasse+milk																
ammoniumphosphaat												1.0%				
cichory pulp																
traces barley, oat, rye																
oats															1.0%	
sunflower																
coconutmeal															1.8%	
cotton fibres										traces						
	100%			100%	100%	100%	100%	100%	100%	98.7%	100%	100%	95.5%	99.9%	100%	100%

Wageningen Food Safety Research
P.O. Box 230
6700 AE Wageningen
The Netherlands
T +31 (0)317 48 02 56
wur.eu/food-safety-research

WFSR Report 2022.021

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.

To explore the potential of nature to improve the quality of life

Wageningen Food Safety Research
P.O. Box 230
6700 AE Wageningen
The Netherlands
T +31 (0) 317 48 02 56
wur.eu/food-safety-research

WFSR report 2022.021

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.