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PRACTITIONER'S REPORT

Proficiency testing of animal nutrition laboratories

Gilberto Batista de Souza · Ana Rita A. Nogueira · Vitor R. Del-Santo · Cristina Maria C. Picchi · Edílson S. Guimarães · Waldomiro Barioni Jr

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Abstract The Embrapa Cattle-Southeast research unit conducts a program to compare the results provided by laboratories that perform animal feed analyses. Fifty-two laboratories, representing all the Brazilian regions, have participated in the program. The assays included are those normally carried out by animal nutrition laboratories on animal feed and mineral supplements, a total of 22 different analyses. Four rounds of the program are performed annually. Each package provided to the laboratories for testing contains three kinds of animal feed (three each of forage and commercial feed), along with three mineral supplements. For the evaluation of assigned values and standard deviation, the median and robust standard deviation of the participants' results are used. This paper reports the experience in coordinating the Brazilian interlaboratory comparison exercise.

Keywords Animal nutrition · Proficiency testing · Inter-laboratory comparisons

Introduction

Proficiency testing (PT) provides information so that a laboratory can detect unsatisfactory performance and is able to apply corrective or preventive actions. Participation in PT is highly recommended, according to the Brazilian Association of Technical Standards (ABNT NBR ISO/IEC 17025:2005), for quality assurance and to demonstrate the competence of laboratory staff to the accreditation bodies [1, 2]. The PT project for analytical laboratories use operational procedures described in the Brazilian technical rules [3] and in the harmonized international protocol for PT programs [2, 4].

In general, the PT scheme is organized considering the preparation and validation of testing materials and evaluation of their homogeneity and stability. Samples are distributed in accordance with a previously defined schedule. The participating laboratories perform the required analyses and send the results back to the provider. These results are submitted to statistical evaluation and a report is prepared in each round, including the main experimental information and a critical analysis of the laboratories' performance [3, 4]. Based on these criteria, and to provide confidence and credibility to the results attained by animal nutrition laboratories, the "Proficiency Testing for Animal Nutrition Laboratories" (PT-ANL) was implemented.

The PT-ANL evaluates the main analytical procedures that are carried out by animal nutrition laboratories: dry matter (DM), dry matter in vitro digestibility (DM-IVD), neutral detergent fiber (NDF), acid detergent fiber (ADF), crude protein (CP), ether extract (EE), lignin, ash, macro- and micro-nutrients—Ca, P, Mg, K, Cu, Fe, Mn, Zn, and Na, and nitrogen fractions—nonprotein nitrogen (NPN); neutral-detergent insoluble nitrogen (NDIN); acid-detergent insoluble nitrogen (ADIN); and borate–phosphate buffer-insoluble nitrogen (BPB-IN). Currently, a total of 52 laboratories from the public and private sectors participate in the PT-ANL. The laboratories represent universities (20%), governmental research centres (9%), laboratories of Embrapa research centres (29%), and private companies (42%), covering all regions of the country [5].

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G. B. de Souza · A. R. A. Nogueira (✉) · V. R. Del-Santo · C. M. C. Picchi · E. S. Guimarães · W. Barioni Jr
Embrapa Cattle-Southeast, Rodovia Washington Luiz, km 234, P.O. Box 339, São Carlos, SP 13560-970, Brazil
e-mail: anarita@cppse.embrapa.br

Materials and methods

Framework of proficiency testing

Samples

The study comprised nine different samples, consisting of three different materials in the categories of forage, feedstuff, and mineral supplements. Sugarcane (*Saccharum officinarum* L.), *Panicum maximum Jacq.* cv. Tanzânia and *Pennisetum purpureum Schum.* cv. Napier were the forage materials evaluated; fish meal, soybean meal, and field beans were the feedstuffs; and mineral supplements for sheep, for horses and for dairy cattle made up the last category. The forage samples were harvested from the Embrapa Cattle-Southeast research unit's field experiments. The feeds and mineral supplements were commercial products purchased from the local markets in São Carlos, São Paulo State, Brazil.

Operational scheme

Round definition, packaging, and labeling were performed by the PT provider. Each one of four rounds was composed of one sample of the three types of the evaluated materials. The round composition was determined at random as were the samples. The sample of sugarcane was repeated in the first and in the third round, soybean meal in the second and in the fourth round, and mineral supplement for sheep was repeated in the first and in the second round. The participants did not receive this particular information, and did not know which of the samples were repeated.

The samples were delivered in a single package. A total of 12 bottles of samples for each participant, individually numbered in an increasing order were sent in a padded box together with a covering letter, explaining the PT-ANL scheme and the schedule relating to submission of the measurement results, emphasizing the importance of storing the samples in a dry and dark place. The sample round distribution is depicted in Table 1.

Analyses of round 1 had to be carried out in the period April–May; round 2 from June to July; round 3 from August to September; and round 4 from October to November. Each laboratory received a code number and the instructions for sending its results *via* the internet. Besides the PT provider, only the participant laboratory knew its code number. Although there was some flexibility as to when the analyses were carried out there were fixed dates in each round for submitting the results to the provider. The same internet area was used for displaying the reports after each round so that laboratories could check their own performance.

Table 1 Round composition with identification of the materials used in the proficiency test of animal nutrition laboratories (PT-ANL)

Sample	Sample description
1	Sugarcane: *Saccharum officinarum* L.
2	Fish meal
3	Mineral supplement for sheep
4	*Panicum maximum Jacq.* cv. Tanzânia
5	Soybean meal
6	Mineral supplement for sheep
7	Sugarcane: *Saccharum officinarum* L.
8	Field beans
9	Mineral supplement for horses
10	*Pennisetum purpureum Schum.* cv. Napier
11	Soybean meal
12	Mineral supplement for cattle

Homogeneity, partitioning, and preservation of samples

After harvesting, 3,000 g of each sample were oven-dried at 65 °C for 48 h in a forced air oven and ground in a cutting mill fitted with 850 μ screen at the bottom of the cutting chamber. The mineral supplements were ground in a porcelain mortar.

Afterwards, each sample was divided into 100 subsamples of 30 g. To preserve the integrity of the samples, they were stored in a room at 10 °C and 25% relative humidity. The samples' homogeneity was evaluated according to the statistical procedures recommended by Lawn et al. [2], by the standards Brazilian technical rules (ABNT ISO/IEC Guide 43:1999) [3] and by Thompson et al. [4]. Ten randomly selected samples of each type of material—forage, feedstuff, and mineral supplement—were separately homogenized and from each two test portion were taken [2]. The forage and feedstuff samples were submitted to the crude protein analysis in a random order under repeatability conditions (in a single run). For the mineral supplement, sodium was the element used as control.

The averaged results were obtained for each material sample and the target standard deviation (σ) was computed using the Horwitz [4] equation, considering the analyte concentration. The homogeneity was evaluated by the Fisher test (F test), and by estimating sampling variance $\left(s_{\text{sam}}^2\right)$. In the former case, if F is less than the appropriate critical value for $p = 0.05$, then the material may be regard as sufficiently homogeneous. In the latter case, if the result of dividing the estimated sampling standard deviation (s_{sam}) by σ is lower than 0.30, the homogeneity was considered acceptable [2, 3].

Analytical determinations

- For each feed sample, the following analyses were carried out: dry matter (DM), ash, crude protein (CP), crude fiber (CF), ether extract (EE), and dry matter in vitro digestibility (DM-IVD) [6].
- Acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid-detergent lignin (LIGNIN) [7]. The NDF in the feedstuff was determined following previous treatment with urea and alpha-amylase enzyme [8].
- Nitrogen fractions composed of nonprotein nitrogen (NPN), neutral-detergent insoluble nitrogen (NDIN), acid-detergent insoluble nitrogen (ADIN), and borate–phosphate buffer insoluble nitrogen (BPB-IN) [9].
- Macronutrients (calcium, magnesium, phosphorus, potassium, and sodium), and micronutrients (copper, iron, manganese, and zinc). Phosphorus was determined by colorimetry, and the others were determined either by emission or by atomic absorption, after dry or wet sample digestion [6].

The participants are free to use independent methods of analysis in accordance with the applicable protocols and available equipment. Therefore, the analytical methods and the procedures can differ among the participants.

Statistical analysis

The laboratories' performances were computed for the three groups of analyses (A, B, and C). Table 2 shows the units to be used by the participants when submitting results, for forage and feedstuff materials, the results are after the correction for dry matter (DM). For the samples of mineral supplement material, only the B group analyses were considered.

The statistical model used was the one recommended by the Brazilian technical rules [3] and by International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [4]. The z score (Eq. 1) was adopted to evaluate the performance of each laboratory for each test.

$$z = (x_i - x_a)/\sigma_P \quad (1)$$

In this equation, x_i is the result obtained by the laboratory, x_a the assigned value, and σ_p the robust standard deviation. For each analyte, the median ($\hat{\mu}$) of all the results was chosen as the assigned value. The robust standard deviation was calculated from the median absolute difference (d_m) by calculating the difference (d_i) between the result reported by the laboratory and the median of the results from all participants ($x_i - \hat{\mu}$). The d_i values were arranged in order of magnitude without regard to the sign, and the median of these values found (d_m). The σ_p values were considered as d_m multiplied by 1.5, as proposed by the Analytical Methods Committee [10].

Table 2 Groups of analytes, acronyms and mass fraction level used in the proficiency test of animal nutrition laboratories (PT-ANL) for the forage and feedstuff materials evaluated

Analytes
Group A (g/100 g)
Dry matter (DM)
Acid detergent fiber (ADF)
Neutral detergent fiber (NDF)
Crude protein (CP)
Crude fibre (CF)
Ether extract (EE)
Ash
Group B (g/kg)
Calcium (Ca)
Magnesium (Mg)
Phosphorus (P)
Potassium (K)
Sodium (Na)
Copper (Cu)[a]
Iron (Fe)[a]
Zinc (Zn)[a]
Manganese (Mn)[a]
Group C (g/100 g)
Acid–detergent lignin (LIGNIN)
Dry matter in vitro digestibility (DM-IVD)
Nonprotein nitrogen (NPN)
Neutral-detergent insoluble nitrogen (NDIN)
Acid-detergent insoluble nitrogen (ADIN)
Borate–phosphate buffer-Insoluble Nitrogen (BPB-IN)

[a] mg/kg

Performance evaluation

The confidence interval was defined as follows: z score results in the limit $|z| \leq 2$ were considered satisfactory and did not receive a mark (*); z score results between the range $2 < |z| < 3$ were considered questionable and received one mark (*); and z score results above the limit $|z| \geq 3$ were considered unsatisfactory and received two marks (**).

Results and discussion

The results presented showed all samples were considered to be sufficiently homogeneous, as summarized in the Table 3. In all cases, the calculated F value is lower than

Table 3 Homogeneity data for the samples evaluated (details in the text)

Sample	Sample description	s_{sam}/σ[a]	$F_{9,10}$
1 and 7	Sugarcane (*Saccharum officinarum* L.)[b]	0.29	1.94
2	Fish meal[b]	0.24	1.28
3 and 6	Mineral supplement for sheep[c]	0.19	1.11
4	*Panicum maximum Jacq.* cv. Tanzânia[b]	0.26	1.27
5 and 11	Soybean meal[b]	0.27	1.77
8	Field beans[b]	0.23	1.63
9	Mineral supplement for horses[c]	0.29	1.66
10	*Pennisetum purpureum Schum.* cv. Napier[b]	0.28	1.29
12	Mineral supplement for dairy cattle[c]	0.26	1.94

[a] s_{sam} = sample standard deviation; σ = target standard deviation

[b] Crude protein was considered to the statistical homomogeneity evaluation

[c] Sodium was considered to the statistical homomogeneity evaluation

the critical value $F_{9,10} = 3.02$ at 95% confidence or, equivalently, the associated probability $p > 0.05$.

Also, $0.19 < s_{sam}/\sigma < 0.29$ (Table 3), confirming that the materials are sufficiently homogeneous for use in the proficiency test [2].

The median ($\hat{\mu}$) and the robust standard deviation (σ_p) used to define the *z score* are presented in Table 4. Samples 1 and 7; 5 and 11; and 3 and 6 (which were sent out twice), presented good agreement in the different rounds, independent of the analyte and the material, corroborating the acceptable homogeneity and stability of the evaluated materials.

In order to express the dispersion of the results among the laboratories, for each analyte and material, the relationship between the standard deviation and the average results − the coefficient of variation (*CV*) was considered. The *CV* values were calculated after the exclusion of outlier results, calculated by the Hampel test [11]. This procedure was adopted to avoid gross errors that could alter the normal data variability.

Table 5 shows the *CV* values obtained from the 52 PT participant laboratories for the determination of DM, CP, ADF, NDF, CF, DM-IVD, ash, and EE for the forage and feedstuff materials. The *CV* values calculated ranged from 1.3 to 32% and from 0.7 to 40% for forage and feedstuff materials, respectively. The analysis with the highest interlaboratory dispersion was EE, with *CV* values from 23 to 32% and from 7.2 to 40% for forage and feedstuff samples, respectively. In a previous PT scheme with animal feed analysis, Lanari et al. [12] also found higher *CV* values for EE and lignin determination, compared with other animal nutrition analyses.

Table 6 presents the *CV* values obtained from the 52 PT participant laboratories, for the analytical results of macro- and micro-nutrients for the forage, feedstuff, and mineral supplement materials. Values from 5.3 to 104% were observed. Sodium was the analyte with the highest *CV*—from 26 to 104% for forage and feedstuff materials. This was probably due to their low absolute values, probably close to the method's limit of detection. The same occurred with potassium, the reported *CV values range* from 43 to 59% for the mineral supplement material [13], although the LODs of the participants' used methods was not query. The lower consensus values presented in Table 4 confirm this observation.

The *CV* values also were considered when evaluating the results of the C group analyses (Table 7). Nitrogen fractions presented the highest *CV* values, as previously observed by Bovera et al. [14].

Laboratory performance

The laboratory performance was evaluated in accordance with the cited criteria for *z* scores and was calculated for the evaluated group of analyses presented in Table 2. The individual and the average medium performance values achieved by the participants during the four rounds of the PT scheme together with the *z* score values relating to each determination were included in the reports produced by the PT provider. This enabled the participants to check and to evaluate their performance and if required to improve their performance.

Considering the fractions of satisfactory *z* scores (%) of the evaluated analytes to determine the laboratories performance, we can conclude that:

1. Forage and feedstuff materials of group A (Table 2), 86% of the results were considered satisfactory ($|z| \leq 2$).
2. Forage and feedstuff materials of the group B (Table 2), approximately 81% of the results were satisfactory.
3. Forage and feedstuff materials of group C (Table 2) presented lower fraction of satisfactory *z score*, only 76%. This is in accordance with the highest interlaboratory variability (Table 7).
4. Mineral supplement material presented 79% of satisfactory *z* score fraction.

Conclusions

With the PT scheme developed, it was possible to draw up a profile of participants. The present study was undertaken to determine the main analytical problems in the evaluated procedures. The *CV* values showed that determination of ether extract, sodium and nitrogen fractions are the procedures that need to be improved. Because of the low

Table 4 Consensus values among the 52 laboratories

Sample		DM (g/100 g)	CP (g/100 g)	ADF (g/100 g)	NDF (g/100 g)	CF (g/100 g)	Ash (g/100 g)	EE (g/100 g)	Ca (g/kg)	Mg (g/kg)	P (g/kg)	K (g/kg)	Na (g/kg)	Cu (mg/kg)	Fe (mg/kg)	Zn (mg/kg)	Mn (mg/kg)	Lignin (g/100 g)	DM-IVD (g/100 g)	NPN (g/100 g)	NDIN (g/100 g)	ADIN (g/100 g)	BPB-IN (g/100 g)
1	$\hat{\mu}$	93	3.6	34	56	26	2.2	1.4	1.4	1.4	0.5	4.9	0.2	12	289	16	21	5.0	58	0.1	0.2	0.1	0.4
	σ_P	1.7	0.4	2.8	2.0	3.8	0.2	0.4	0.3	0.2	0.1	1.0	0.2	6.2	57	2.3	4.8	0.5	2.8	0.1	0.1	0.1	0.0
2	$\hat{\mu}$	95	65	13	39	1.1	19	11	57	2.9	29	3.4	5.9	14	823	102	28	6.8	48	0.9	4.5	1.6	9.3
	σ_P	1.1	2.1	4.7	10	0.8	0.3	0.7	7.7	0.4	3.3	0.4	1.5	2.5	134	14	6.7	4.1	21	0.2	0.1	0.9	1.0
3	$\hat{\mu}$								112	11	62	1.6	169	667	2856	3724	816						
	σ_P								20	1.5	6.4	0.8	27	117	465	713	101						
4	$\hat{\mu}$	93	15	39	68	30	11	2.1	6.6	4.9	2.2	29	0.3	9.0	110	22	63	3.9	61	0.6	1.1	0.2	1.6
	σ_P	1.0	0.6	1.5	3.0	2.6	0.4	0.5	0.5	0.6	0.3	1.9	0.2	3.3	17	7.1	11	0.5	2.1	0.1	0.3	0.1	0.1
5	$\hat{\mu}$	90	51	11	21	7.2	6.4	1.0	3.2	3.1	6.8	22	0.4	14	200	52	28	0.8	87	0.4	0.7	0.8	6.5
	σ_P	1.0	2.1	1.7	5.5	0.6	0.5	0.4	0.4	0.4	0.3	1.3	0.4	2.0	20	7.1	6.7	0.6	1.4	0.1	0.1	0.9	0.2
6	$\hat{\mu}$								118	10	60	1.1	170	602	2605	3890	818						
	σ_P								9.2	0.9	3.0	0.4	16	68	509	268	80						
7	$\hat{\mu}$	92	3.6	34	57	27	2.2	1.3	1.4	1.4	0.5	4.5	0.2	8.2	298	13	21	5.1	55	0.2	0.2	0.1	0.4
	σ_P	2.1	0.3	1.5	2.5	3.1	0.2	0.4	0.2	0.2	0.1	0.9	0.1	6.3	68.4	4.4	2.8	0.8	5.8	0.0	0.1	0.0	0.0
8	$\hat{\mu}$	90	23	10	36	4.6	4.2	1.4	1.7	1.9	4.3	15	0.2	11.1	82	36	20	1.4	87	0.5	1.1	0.5	1.7
	σ_P	0.7	1.0	1.1	10	0.4	0.3	0.5	0.3	0.2	0.2	2.4	0.2	1.9	25	5.5	5.5	1.1	4.2	0.4	0.9	0.2	0.0
9	$\hat{\mu}$								109	13	60	2.0	190	478	2118	5949	1112						
	σ_P								5.8	1.1	3.6	0.6	14	99	347	341	174						
10	$\hat{\mu}$	92	11	38	69	32	11	2.7	3.2	1.8	1.6	40	0.3	8.7	128	26	73	3.6	61	0.6	0.7	0.2	1.2
	σ_P	1.2	0.7	2.2	2.2	1.8	0.6	0.5	0.6	0.1	0.1	5.4	0.2	3.2	22	12	9.6	0.5	3.1	0.2	0.1	0.1	0.0
11	$\hat{\mu}$	91	51	11	21	7.5	6.3	0.9	3.1	3.1	6.5	23	0.4	14	200	54	30.1	1.1	88	0.4	1.2	0.3	7.4
	σ_P	0.9	1.9	1.5	4.2	0.6	0.4	0.3	0.4	0.2	0.4	2.3	0.1	1.8	40.9	6.2	4.0	0.2	2.3	0.2	0.9	0.2	0.1
12	$\hat{\mu}$								144	11	89	1.1	147	850	3532	5683	348						
	σ_P								11	0.8	6.4	0.5	15	72	472	374	75						

The sample numbers refer to the samples evaluated (described in Table 2)

$\hat{\mu}$: median; σ_P: robust standard deviation

Table 5 Coefficients of variation among the results presented by the 52 PT participating laboratories of group A analytes (described in Table 2)

Sample	DM	CP	ADF	NDF	CF	Ash	EE
Forage—*CV* (%)							
1	2.0	11	7.6	5.4	14	8.3	32
4	1.3	5.1	4.6	3.7	12	4.5	28
7	2.3	13	4.5	5.0	11	8.0	32
10	1.4	6.4	7.5	3.3	5.5	5.4	23
Feedstuff—*CV* (%)							
2	1.1	3.6	46	5.4	55	2.7	7.2
5	1.5	3.6	15	27	9.3	7.7	40
8	0.7	6.2	15	37	8.3	7.6	33
11	1.3	3.7	14	27	11	7.1	40

Table 6 Coefficients of variation among the results presented by the 52 PT participating laboratories of group B analytes (described in Table 2)

Sample	Ca	Mg	P	K	Na	Cu	Fe	Zn	Mn
Forage—*CV* (%)									
1	33	14	41	27	95	53	25	20	28
4	10	12	9.4	8.3	71	34	16	27	19
7	19	14	21	22	79	65	24	39	18
10	18	10	8.0	19	74	37	17	39	13
Feedstuff—*CV* (%)									
2	27	16	13	17	27	32	16	17	22
5	13	14	6.4	8.3	88	24	10	14	24
8	16	11	8.4	17	104	25	39	17	28
11	18	6.6	6.2	13	26	15	26	16	14
Mineral supplement—*CV* (%)									
3	16	13	9.9	51	24	17	21	18	12
6	10	11	6.7	59	14	14	20	7.1	9.8
9	5.3	8.5	5.8	43	7.2	21	16	10	23
12	8.0	7.5	7.2	48	12	12	13	6.6	21

Table 7 Coefficients of variation among the results presented by the 52 PT participating laboratories of group C analytes (described in Table 2)

Sample	Lignin	DM-IVD	Nitrogen fractions			
			NPN	NDIN	ADIN	BPB-IN
Forage—*CV* (%)						
1	18	8.6	53	50	52	5.7
4	16	7.7	16	23	46	6.3
7	19	11	43	47	46	1.9
10	24	5.0	43	24	79	1.7
Feedstuff—*CV* (%)						
2	43	44	49	2.9	66	11
5	59	2.2	53	16	130	2.8
8	70	4.8	80	69	40	2.1
11	46	2.8	53	76	82	0.8

sodium concentration, the presented results showed the highest *CV*, compared to the other inorganic chemical elements present in the mineral supplements. It was easy to work with the chosen statistical model and it was possible to identify meaningful differences in the participants' performance with the use of *z score*. It was also possible to infer the historical performance of each participant along the year, enabling the final results to serve as external quality controls and to identify possible errors.

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