AN INTERNATIONAL RING TRIAL TO ASSESS DIFFERENCES BETWEEN LABORATORIES IN FORAGE ANALYSIS: A NEW SLANT ON AN OLD PROBLEM

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Introduction

The quality of laboratory reference values has always been a major factor affecting NIR calibration accuracy. It is of particular relevance in the case of forage analysis, where several of the common quality measurements are "operationally defined" properties rather than specific constituents, and slight differences in methodology can produce different values for a given property. This issue is even more important where a central laboratory conducts reference analysis, derives calibrations and transfers them to other instruments in a network or where spectral databases and reference values from instruments from laboratories across the world are combined to produce "global" calibrations.

Materials and Methods

Seventy-four samples consisting of whole plant maize (both fermented and fresh) and grass/legume hay, silage and freshcut from Europe, North America and Australia collected during 2001. All samples were oven dried to be approximately 5% moisture. Samples were ground through a grinder with a 1 mm screen, then subdivided using a sample splitter to produced 8 sets of approximately 30 g samples. All subsamples were scanned by NIR and any set showing disuniformity was recombined and the splitting and scanning process was repeated until desired uniformity was achieved. The set included 10 blind duplicate samples. Participating laboratories were asked to run wet chemistry dry matter, protein, acid detergent fiber (ADF), and neutral detergent fiber (NDF) in duplicate on all samples.

Table 1. Mean Standard Deviation of Analysis of Blind Duplicates

Laboratory	DM	Protein	ADF	NDF	Ash
1	0.391	0.217	0.360	0.296	0.237
2	0.202	0.175	0.466	0.308	0.272
3	1.136	0.203	0.570	1.144	0.186
4	0.786	0.186	0.884	0.547	0.253
5	0.357	0.064	0.371	0.520	0.144
6	0.598	0.295	0.533	1.238	0.378

Table 2 Comparison of Protein Determination among Laboratories

	Laboratory						
Comparison	1	2	3	4	5	6	7
Slop Sig Diff from 1.0 at 0.01	TRUE	TRUE	FALSE	TRUE	TRUE	TRUE	TRUE
Bias Sig diff at 0.01	TRUE	TRUE	FALSE	TRUE	TRUE	TRUE	TRUE
Correlation	1.000	1.000	1.000	0.999	0.999	0.998	1.000
RMSED	0.324	0.278	0.198	0.304	0.490	0.579	1.033
SED	0.233	0.211	0.194	0.221	0.282	0.525	0.518
Mean	12.627	12.037	12.854	13.065	12.453	12.604	12.054
Bias	0.227	-0.034	-0.045	-0.211	0.401	0.250	0.897
Slope	1.028	0.975	0.996	0.986	1.030	0.951	1.078
Intercept	-0.131	0.146	0.003	-0.032	0.023	0.869	-0.044
Minimum	2.300	2.257	2.285	2.325	2.155	1.650	2.145
Maximum	29.350	30.797	30.055	30.195	29.210	30.600	28.055

Table 3 Comparison of Neutral Detergent Fiber Determination among Laboratories

	Laboratory						
Comparison	1	2	3	4	5	6	7
Slop Sig Diff from 1.0 at 0.01	TRUE	TRUE	TRUE	FALSE	TRUE	FALSE	FALSE
Bias Sig diff at 0.01	FALSE	TRUE	TRUE	TRUE	TRUE	TRUE	TRUE
Correlation	0.996	0.996	0.996	0.994	0.993	0.988	0.989
RMSED	1.095	1.073	1.750	2.436	1.543	1.816	6.957
SED	1.072	1.190	1.143	1.213	1.399	1.746	2.111
Mean	48.357	46.529	49.439	50.226	47.440	48.643	43.702
Bias	-0.249	1.580	-1.331	-2.117	0.669	-0.535	6.636
Slope	0.964	1.053	0.963	1.009	0.964	0.997	1.076
Intercept	1.496	-0.884	0.486	-2.592	2.356	0.565	3.299
Minimum	23.805	24.456	29.185	28.800	23.310	27.650	18.425
Maximum	79.895	76.242	81.570	81.130	79.430	79.650	71.400

Results

The results presented (table 1) illustrate how, when laboratories have no prior information as to the identity of samples and, therefore, the samples receive no preferential treatment, the "real" error in laboratories is larger than would be perceived from an analysis of replicate samples.

All the laboratories produced highly satisfactory results for Protein, NDF, ADF and Ash (tables 2 and 3). The results for Dry Matter were poor at sites where samples had been delayed in transit. Uptake of moisture in transit is a major problem when samples are moved worldwide. The differences seen between laboratories were clearly systematic, with both slope and bias differences being evident. Only with dry matter were there random differences between laboratories for the reasons mentioned previously.

The high quality of the results, combined with the large number of samples involved, produced one result that was not predictable in advance of the trial: the differences between laboratories (either slope or bias) were usually significant at a 1% level, even although the slope difference from 1.0 or the differences from 0.0 for bias were small

Recommendations

Allow 15 months for sample collection

The samples are first placed in medium grade airtight polythene bags that are then heat-sealed or have integral airtight zip tops.

Each individual sample is then placed inside another zip top polythene bag that then has a wax seal attached. The purpose of the wax seal is to identify if a particular bag has been opened.

All the samples are then placed within a single large plastic bag that is placed into a stout cardboard box for shipping.

Expect difficulties shipping biological samples across international