Method Troubleshooting

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10/15/2024

Overview

- Quality Control Plans
- Quality Control Samples
- Method troubleshooting
- Troubleshooting Method Example

Good Troubleshooting starts before the Issue arises

- Each method should have a quality control plan to indicate if the method has been performed correctly
- Quality plans can include blanks, standards, duplicate samples, spiked sample
- Run Blind/Rerun samples to check consistency
- Monitor equipment performance and maintain preventative maintenance
- Participate in Proficiency Testing
- Good quality control plans give confidence in results and identify when there are issues that need to be addressed

Quality Control Materials

Each batch should include a quality control standard

- Essential for tracking accuracy and precision
- The control standard should fall within a pre-defined range
  - Within the control range on the COA
  - Within a % recovery range
- Control results can be tracked to detect trends

Selecting appropriate quality control standards

- Ideally, use a control that has a known result that is a similar matrix and range as the samples
- Some Proficiency Testing Providers will sell past PT samples to be used as quality control standards

In-house controls

- If control sample is not available in an applicable matrix, an in-house control can be created
- The advantage is having a control that is a matrix match
- The disadvantage is there is no comparison to other laboratories to confirm the accuracy

Creating a DDGS In-House Standard

Composite a representative DDGS and thoroughly homogenize

- Consistency of the sample is very important

Grind the sample as fine as possible

- Samples that are ground consistently make the best standard
- For example: Grind the sample to less than 1 mm, thoroughly mix the sample then grind to less than 0.5 mm

After grinding, thoroughly homogenize the sample again and spilt into aliquots

- Depending on the stability of the sample, the aliquots can be placed in vacuum seal bags and frozen

Testing In-House Standard

Analyze the sample at least 30 times following the appropriate test method

- The 30 replicates should be split between different aliquots of the sample, different days, run by different analysts, and run on different instruments if applicable

Calculate the average and standard deviation of the 30-sample data set

- Analyze the data for outliers and remove if appropriate

The average can now be used as the known result on the sample

- Determine the acceptable range by either using the standard deviation range or a percent recovery range (e.g.,95-105%)

Method Troubleshooting– Where to start?

Define the problem - Clearly lay out the problem that needs to be solved

- Inconsistent Results
- Failed Quality Control Samples
- Failed Proficiency Results
- Results not aligning with other labs

What changed?

- Did you just make a change? Replaced a part? Opened a new reagent? Start there

Check for obvious problems

- Broken consumables/parts?
- Instrument Issues?

Method Review

Review the internal method and ensure the method is being followed

- Start at the beginning of the method and systematically check each step of the method
- Are there steps the have been inadvertently changed?
- Are all the analysts performing the method the same way?
- Is anything missing from the internal method?

Review applicable standard methods

- Ensure the internal method aligns with the standard methods
  - Standard methods can be from organizations like ASTM, AOAC, AOCS, etc
  - Standard methods can also be Application Notes or other publication
- Check for notes, tips, and footnotes that may have not been captured in the internal method

Materials and Equipment Check

Check the chemicals and resources used in the method

- Are any of the chemicals new or newly made? Maybe they are incorrect
- Have any chemicals gone bad or expired?
- Check the COA of the reagent and chemical

Check the equipment and instruments used in the method

- Are they all functioning correctly? When was the last time they were checked?
- Do not forget to check things like balances, thermometers, pH meters etc

Make a Plan

Make a list of all the things that were checked note the points that need to be checked

- Include on the list the things that were checked and do not appear to be causing the issue
- It is important to list the things that went “right”
- For example, “Balance calibration was checked before using the balance. The balance appears to be clean and in good condition and other methods were run using the scale and those methods had no issues”

For each potential issue point, plan how the issue will be checked or trialed

- Design the trials so that only one component is being checked at a time
- Ensure you have enough supplies and reagents to trial all the identified issues
  - This includes ensure there is enough sample material to test all the components

Record, Record, Record

Record the outcomes of the trials

- It is important to record as much information as possible
- Record what was being trialed, and what the outcome of the trials were
- It is important to keep good records on even “failed” trials
- It is also important to record when you find the issue as well, so the next time a problem arises you can see what has happened in the past

Continue to run trials and record the outcome until a resolution is found

Trial Records Example

Crude Fat Sieve Trial for Whole Corn and Ground Corn					
Sample #		Not Sieved	Sieved	# 18	# 40
4884	Whole Corn	2.9314	2.1503	2.2646	3.2762
		2.8919	3.1221	2.2905	3.2994
		3.0327	3.0023	2.6754	3.1193
CV		2.46%	19.21%	9.55%	3.03%
5165	Whole Corn	3.0120	2.6496	2.4719	3.8643
		2.8626	2.7551	2.2528	3.8002
		3.1714	2.5374	2.8463	3.6200
CV		5.12%	4.11%	11.89%	3.37%
5476	Whole Corn	2.8331	2.1063	2.2607	3.1972
		2.8932	2.8903	2.6538	3.2485
		2.7350	2.7631	2.7493	3.1545
CV		2.83%	16.27%	10.14%	1.47%
5696	Whole Corn	3.1949	2.0036	2.6139	3.7807
		2.9583	2.9207	2.3643	3.7452
		2.8619	2.8778	2.7617	3.6705
CV		5.70%	19.90%	7.78%	1.51%

Crude Fat Coffee Grinder vs Cyclotec Trial

7.14.2014

Purpose:

A new grinder was purchased, and the Crude Fat results are trending higher. A trial to compare the new style of grinder to the previous needs to be run. One DDGS sample was ground using each ginder and the sample was run for 6 replicates using each grind to compare the grinds

Findings:

The results from the new grinder are consistently higher than the results from the old grinder. The samples appear to be a finer grind using the new grinder. It was noted that the moisture was lower using the new grinder, so the higher Crude Fat maybe attributed to the difference is moisture. The results were calculated on a dry metter basis using the mositure results, and the results from the new grinder were still consistently higher.

	Coffee Grinder Crude Fat (%)	Cyclotec Crude Fat (%)	Difference Coffee Grinder - Cyclotec
14008576	4.5373	4.9127	0.3754
	4.5498	4.8731	0.3233
	4.5328	4.8953	0.3625
	4.5432	4.8807	0.3375
	4.5446	4.8641	0.3195
	4.5853	4.8754	0.2901
Average	4.5488	4.8836	
CV	0.41%	0.36%	

Some methods are so simple you don't have to worry about troubleshooting ....right?

Moisture Method Example

Troubleshooting the Method – Moisture Example

- Start at Sample Preparation
  - Does the method require grinding the sample?
  - What Grind size does the method Require?
  - What Style of Grinder is called for?
  - Is your grinder working properly?

Troubleshooting the Method – Moisture Example

- Sample Weighing
  - Are the pans properly prepared?
  - What is the correct sample size?
  - Is the correct size/ material moisture pan being used?
  - Are lids used on the sample?
  - When are the lids used on the sample?
  - Is the balance working properly?

Troubleshooting the Method – Moisture Example

- Oven
  - Is a thermometer being used to confirm the temperature of the oven?
  - Does the thermometer need to be checked?
  - Is the oven being overloaded with samples?
  - Are there a large amount of high moisture samples on the batch
  - If lids are used, are they being removed before putting the samples in the oven
  - Are the pans too close in the oven
- Time in the oven
  - How long does the method say to keep the samples in the oven?
  - Is the oven dedicated to one method, or is the oven being used for multiple methods at the same time

Troubleshooting the Method – Moisture Example

- Desiccant
  - Are the samples being cooled in a desiccator?
  - Is the desiccant fresh?
  - Is the desiccator being used for multiple methods at the same time
- Weighing Back the samples
  - Are the samples completely cool before being weighed back?
  - How long are the samples on the counter before being weighed?
- Check the calculation

Questions

Contact Us

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