

Using Statistical Data From Magruder Program To Meet AOAC Collaborative Study Requirements

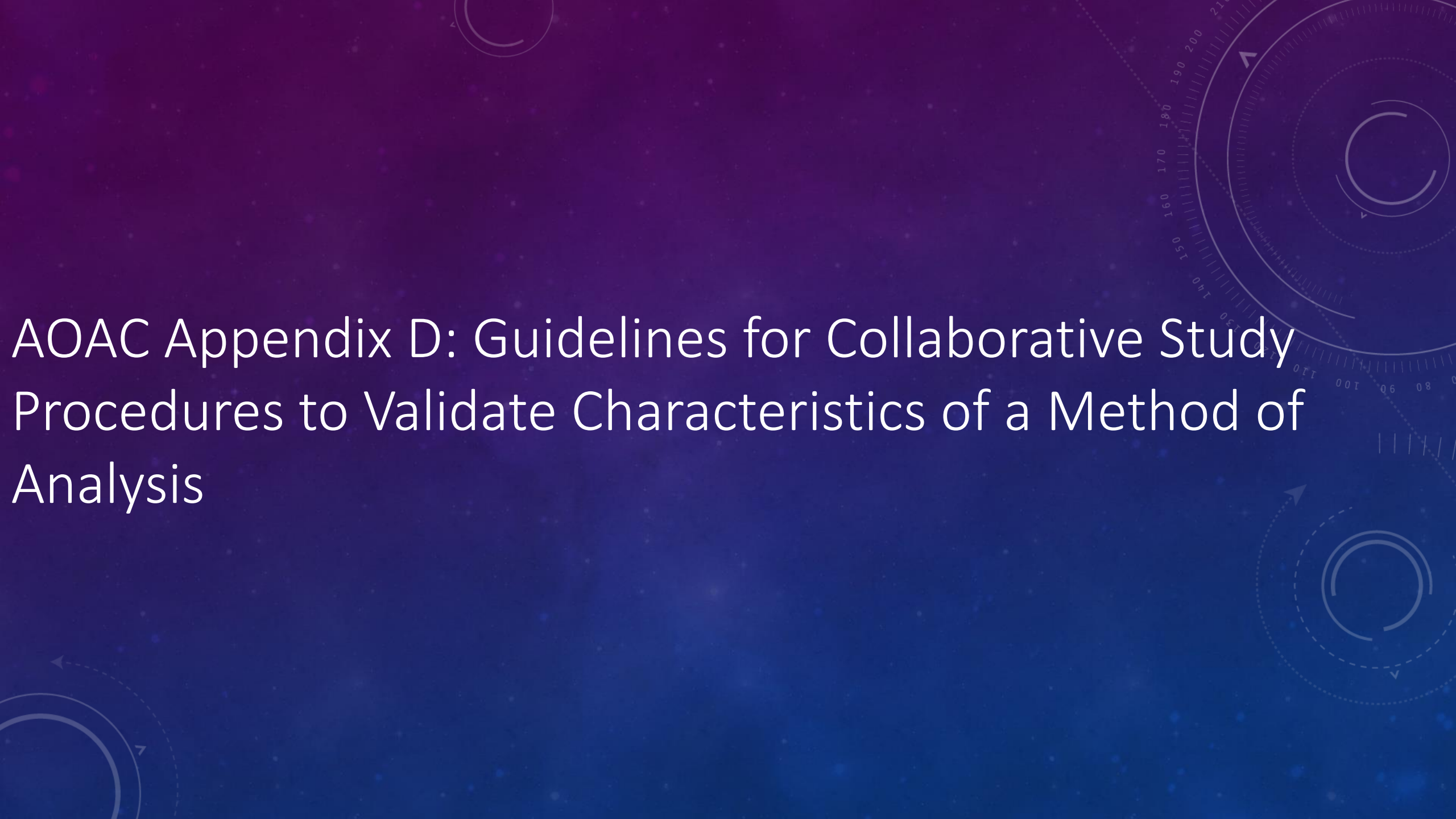
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Regulatory Services
College of Agriculture, Food and Environment

The background features a dark blue gradient with a subtle pattern of white stars and technical diagrams. On the right side, there are several circular gauges or dials with numerical scales (e.g., 140, 150, 160, 170, 180, 190, 200, 210) and arrows. Some of these diagrams are partially cut off by the edge of the frame. The overall aesthetic is clean and professional, typical of a technical or scientific presentation.

AOAC Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis

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http://www.aoac.org/aoac_prod_imis/AOAC_Docs/StandardsDevelopment/Collaborative_Study_Validation_Guidelines.pdf

Preliminary Work Includes

- Successfully completed the SLV & method is in First Action by AOAC-ERP
- Minimum Number of Materials
 - 5
- Minimum Number of Laboratories
 - 8 who report valid data
- Minimum Number of Replicates
 - Younden Pairs, 2
- Method Selection
 - Decided in the Methods Forum prior to SLV
- Optimization of Method Selected
 - Performed in SLV

What Makes up a Collaborative Study

- Preliminary Work (SLV, 1st Action Status)
- Design of Collaborative Study
- Preparation of Materials
- Submission of Test Samples
- Statistical Analysis
- Bias of Individual Results
- Precision
- HORRAT
- Frequency of False Positives/False Negatives
- Manuscript

If we are allowed to use data from Magruder

- Preliminary Work (SLV, 1st Action Status)
- ~~Design of Collaborative Study~~
- ~~Preparation of Materials~~
- ~~Submission of Test Samples~~
- Statistical Analysis
- Bias of Individual Results
- Precision
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Statistical Requirements from AOAC


- Review of raw data
- Determine if any outliers
- Bias of individual results
- Precision of results
- HORRAT
- False +/-
- Manuscript

Statistical Requirements from AOAC

- Review of raw data
 - We have that
- Determine if any outliers
- Bias of individual results
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Statistical Requirements from AOAC

- Review of raw data
 - We have that



STRIVING FOR EXCELLENCE IN ANALYSIS

Method Proficiency For All Labs (Lab Values)
Methods: 127

Sample # 171012
Labs Reporting: 100

TSP, 0-45-0
Issue Date : 12/31/2017

Method Code	Analyte Name and Method (Units)	Lab Code	Lab Data		Method Values			# Tests	Magruder CS Z Score	Threshold %RSD	Flag
			Value	Range	Rob Mean	Rob SD	R-bar				
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0177	12.49	0.1300	13.48	0.3932	0.2428	12	-2.88	4%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0117	13.29	0.1200	13.48	0.3932	0.2428	12	-0.47	1%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0233	13.37	0.4000	13.48	0.3932	0.2428	12	-0.27	0%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0034	13.40	0.3000	13.48	0.3932	0.2428	12	-0.19	0%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0136	13.45	0.1500	13.48	0.3932	0.2428	12	-0.08	0%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0086	13.54	0.5090	13.48	0.3932	0.2428	12	0.16	0%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0421	13.55	0.3000	13.48	0.3932	0.2428	12	0.19	0%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0494	13.71	0.5413	13.48	0.3932	0.2428	12	0.60	1%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0527	13.76	0.0300	13.48	0.3932	0.2428	12	0.72	1%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0023	13.87	0.2380	13.48	0.3932	0.2428	12	1.01	1%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0397	14.00	0.0666	13.48	0.3932	0.2428	12	1.32	2%	0
101.33	Acid Soluble Calcium, ICP, test portion 2006.... (13.5%)	0106	11.56*	1.880	13.48	0.3932	0.2428	12	-4.88	7%	1

Statistical Requirements from AOAC

- Review of raw data
- Determine if any outliers
 - Cochran Test
 - Grubbs Test
 - Can be determined not needed—even better!
- Bias of individual results
- Precision of results
- HORRAT
- False +/-
- Manuscript

Statistical Requirements from AOAC

- Review of raw data
- Determine if any outliers
- Bias of individual results
 - $\text{Bias} = \bar{X}_{\text{found}} - \bar{X}_{\text{assigned}}$

Analyte Group	Analyte (Units)	Lab Code	Lab Data		Method Values			# Tests	Magruder CS Z Score	Your Method	Flag
			Value	Range	Rob Mean	Rob SD	R-bar				
101	Acid Soluble Calcium (13.5%)	0354	12.92	0.2270	13.48	0.7251	0.2239	62	-0.77	101.30	0
101	Acid Soluble Calcium (13.5%)	0102	12.93	0.1468	13.48	0.7251	0.2239	62	-0.76	101.30	0
101	Acid Soluble Calcium (13.5%)	0042	12.97	0.8800	13.48	0.7251	0.2239	62	-0.70	101.99	0
101	Acid Soluble Calcium (13.5%)	0231	12.99	0.1400	13.48	0.7251	0.2239	62	-0.67	101.30	0
101	Acid Soluble Calcium (13.5%)	0157	13.00	0.4000	13.48	0.7251	0.2239	62	-0.66	101.30	0
101	Acid Soluble Calcium (13.5%)	0360	13.07	0.1000	13.48	0.7251	0.2239	62	-0.56	101.30	0
101	Acid Soluble Calcium (13.5%)	0444	13.09	0.0600	13.48	0.7251	0.2239	62	-0.53	101.30	0
101	Acid Soluble Calcium (13.5%)	0307	13.16	0.2240	13.48	0.7251	0.2239	62	-0.43	101.30	0
101	Acid Soluble Calcium (13.5%)	0255	13.22	0.5070	13.48	0.7251	0.2239	62	-0.36	101.30	0
101	Acid Soluble Calcium (13.5%)	0096	13.27	0.1600	13.48	0.7251	0.2239	62	-0.29	101.30	0
101	Acid Soluble Calcium (13.5%)	0117	13.29	0.1200	13.48	0.7251	0.2239	62	-0.26	101.33	0
101	Acid Soluble Calcium (13.5%)	0041	13.30	0.0000	13.48	0.7251	0.2239	62	-0.24	101.30	0

Statistical Requirements from AOAC

- Review of raw data
- Determine if any outliers
- Bias of individual results
- Precision of results
 - Repeatability—same lab different days, % RSD_r
 - Reproducibility—different labs, % RSD_R
- HORRAT
- False +/-
- Manuscript

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- Bias of individual results
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 - Repeatability—same lab different days, % RSD_r
 - Reproducibility—different labs, % RSD_R

Sample Number	Method Code	Analyte & Method	# Labs in Robust Calculations	Assigned Value Robust Mean	IA at Analyte Value	Robust sd	Robust % RSD	Method IA Ratio	Horwitz %RSD
170411	101.33	Acid Soluble Calcium, ICP, test portion 2006.03A-C... (%)	8	0.3435	0.2172	0.0262	7.62%	0.28	4.70%
171211	101.33	Acid Soluble Calcium, ICP, test portion 2006.03A-C... (1%	10	0.3531	0.2177	0.0238	6.75%	0.26	4.68%
180711	101.33	Acid Soluble Calcium, ICP, 2017.02 (%)	9	0.9351	0.2468	0.0672	7.19%	0.63	4.04%
170211	101.33	Acid Soluble Calcium, ICP, test portion 2006.03A-C... (4%	9	4.515	0.4258	0.1552	3.44%	0.85	3.19%
190311	101.33	Acid Soluble Calcium, ICP, 2017.02 (6%)	14	7.838	0.5919	0.3008	3.84%	1.18	2.93%
171012	101.33	Acid Soluble Calcium, ICP, test portion 2006.03A-C... (13	12	13.48	0.8738	0.3932	2.92%	1.05	2.70%

Statistical Requirements from AOAC

- Review of raw data
- Determine if any outliers
- Bias of individual results
- Precision of results
- HORRAT
 - $PRSD_R\% = 2 * C^{-0.1505}$
 - $HORRAT = \frac{RSD_R\%}{PRSD_R\%}$
- Method is acceptable if HORRAT is between 0.5 to 2
- Sometimes justifications as to why the value is outside the range is OK

Statistical Requirements from AOAC

- Review of raw data
- Determine if any outliers
- Bias of individual results
- Precision of results
- HORRAT
- False +/-
 - Not considered outliers
 - Appendix D has statistical requirements, a bit complicated
 - When proportion of 0's > 30%, may be problematic
- Manuscript

Next Steps

- Email dialogue has been established with AOAC regarding accepting statistics from PTP data as an alternative to collaborative study
- Begun mining data from Magruder
- Got a lot of data to go through
- Confident statistics from Magruder meets the requirements
 - Except for blind samples, aka Younden
 - Except for replicates, more than 2

Questions?

- Thanks!