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Standard Guide for Optimizing, Controlling and Reporting Test Method Uncertainties from Multiple Workstations in the Same Laboratory Organization¹

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1. Scope

1.1 This guide describes a protocol for optimizing, controlling, and reporting test method uncertainties from multiple workstations in the same laboratory organization. It does not apply when different test methods, dissimilar instruments, or different parts of the same laboratory organization function independently to validate or verify the accuracy of a specific analytical measurement.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E 350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron

E 415 Test Method for Optical Emission Vacuum Spectrometric Analysis of Carbon and Low-Alloy Steel

E 1329 Practice for Verification and the Use of Control Charts in Spectrochemical Analysis

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E 2027 Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials

2.2 ISO Standards:

ISO 17025 General Requirements for the Competence of

Calibration and Testing Laboratories³
ISO 9000 Quality Management and Quality System Elements³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology E 135.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *data quality objectives, n*—a model used by the laboratory organization to specify the maximum error associated with a report value, at a specified confidence level.

3.2.2 *laboratory organization, n*—a business entity that provides similar types of measurements from more than one workstation located in one or more laboratories, all of which operate under a unified quality system.

3.2.3 *maximum deviation, n*—the maximum error associated with a report value, at a specified confidence level, for a given concentration of a given element, determined by a specific method, throughout a laboratory organization.

3.2.4 *workstation, n*—a combination of people and equipment that executes a specific test method using a single specified measuring device to quantify one or more parameters, with each report value having an established estimated uncertainty that complies with the data quality objectives of the laboratory organization.

4. Significance and Use

4.1 Many competent analytical laboratories comply with accepted quality system requirements such as ISO 9000, QS9000,⁴ and ISO 17025. When using standard test methods, their test results on the same sample should agree with those from other similar laboratories within the reproducibility estimates (R2) published in the standard. Reproducibility estimates are generated as part of the interlaboratory studies (ILS), of the type described in Practice E 1601, during the

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁴ Quality Systems Requirements, Chrysler Corporation, Ford Motor Company, and General Motors Corporation—available from AIAG, 26200 Lahser Rd., Southfield, MI 48034.

standardization process. Competent laboratories participate in proficiency tests, such as those carried out according to Practice E 2027, to confirm that they perform consistently over time. In both ILS and proficiency testing protocols, it is generally assumed that only one work station is used to generate the data.

4.2 Many laboratories have workloads, or logistical requirements, or both, that dictate the use of multiple work stations. Some have multiple stations in the same area (central laboratory format). Others' stations are scattered throughout a facility (at-line laboratory format). Often, analysis reports do not identify the workstation used for the testing, even if workstations differ in their testing uncertainties. Problems can arise if clients mistakenly attribute variation in report values to process rather than workstation variability. These problems can be minimized if the laboratory organization sets, complies with, and reports a unified set of data quality objectives throughout.

4.3 This guide describes a protocol for efficiently optimizing and controlling variability in test results from different workstations used to perform the same test. It harmonizes calibration and control protocols, thereby providing the same level of measurement traceability and control to all workstations. It streamlines documentation and training requirements, thereby facilitating flexibility in personnel assignments. Finally, it offers an opportunity to claim traceability of proficiency test measurements to all included workstations, regardless on which workstation the proficiency test sample was tested. The potential benefits of utilizing this protocol increase with the number of workstations included in the laboratory organization.

4.4 This guide can be used to identify and quantify benefits derived from corrective actions relating to under-performing workstations. It also provides means to track improved performance after improvements have been made.

4.5 It is assumed that all who use this guide comply with ISO 17025, especially including the use of documented procedures, the application of statistical control of measurement processes, and participation in proficiency testing.

4.6 The general principles of this protocol can be adapted to other types of measurements, such as mechanical testing and on-line process control measurements, such as temperature and thickness gaging. In these areas, users may need to establish their own models for defining data quality objectives and proficiency testing may not be available or applicable.

4.7 It is especially important that users of this guide take responsibility for ensuring the accuracy of the measurements made by the workstations to be operated under this protocol. In addition to the checks mentioned in 6.2.3, laboratories are encouraged to use other techniques, including, but not limited to, analyzing some materials by independent methods, either within the same laboratory or in collaboration with other equally competent laboratories. The risks associated with generating large volumes of data from carefully synchronized, but incorrectly calibrated multiple workstations are obvious and must be avoided.

5. Summary

5.1 Identify the test method and establish the data quality objectives to be met throughout the laboratory organization.

5.2 Identify the workstations to be included in the protocol and harmonize their experimental procedures, calibrations, and control strategies so that all performance data from all workstations are directly statistically comparable.

5.3 Tabulate performance data for each workstation and ensure that each workstation complies with the laboratory organization's data quality objectives.

5.4 Document items covered in 5.1-5.3.

5.5 Establish and document a laboratory organization-wide proficiency test policy that provides traceability to all workstations.

5.6 Operate each workstation independently as described in its associated documentation. If any changes are made to any workstation or its performance levels, document the changes and ensure compliance with the laboratory organization's data quality objectives.

6. Procedure

6.1 Identify the test method and establish the data quality objectives to be met throughout the laboratory organization.

6.1.1 Multi-element test methods can be handled concurrently, provided that all elements are measured using common technology, and that the parameters that influence data quality are tabulated and evaluated for each element individually. An example is Test Method E 415 that covers the analysis of plain carbon and low alloy steel by optical emission vacuum spectrometry. Workstations can be under manual or robotic control, as long as the estimated uncertainties are within the specified data quality objectives. Avoid handling multi-element test methods concurrently that use different measurement technologies. Their procedures and error evaluations are too diverse to be incorporated into one easy-to-manage package. An example of test methods that should not be combined into one program is Test Methods E 350 because those methods cover many different measurement technologies.

6.1.2 Set the data quality objectives for the application of the method throughout the laboratory organization, using customer requirements and available performance data. At the conclusion of this effort, the laboratory organization will know the maximum deviation allowed in any report value, at any concentration level, using the method of choice. An example of a possible method for establishing data quality objectives is given in Annex A1.

6.2 Identify the workstations to be included in the protocol and harmonize their experimental procedures, calibrations, and control strategies so that all performance data from all workstations are directly statistically comparable.

6.2.1 For each workstation, list the personnel and equipment that significantly influence data quality. Each component of each workstation does not have to be identical, such as from the same manufacturer or model number; however, each workstation must perform the functions described in the test method.

6.2.2 Harmonize the experimental procedures associated with each workstation to ensure that all stations are capable of generating statistically comparable data that can be expected to fall within the maximum allowable limits for the laboratory

organization. Ideally, all workstations within the laboratory organization will have essentially the same experimental procedures.

6.2.3 Harmonize calibration protocols so that the same calibrants are used to cover the same calibration ranges for the same elements on all instruments. Avoid the use of different calibrants on different instruments that may lead to calibration biases and uncertainties that are larger than necessary. Make sure that all interferences and matrix effects are addressed. Verify the calibrations with certified reference materials not used in the calibration, when possible. Record the findings for each workstation.

6.2.4 Use the same SPC materials and data collection practices on all work stations (see Note 1). Carry SPC materials through all procedural steps that contribute to the measurement uncertainty. Develop control charts in accordance with E 1329, or equivalent practice.

NOTE 1—Generally, it is recommended that SPC concentrations be set about 1/3 from the top and 1/3 from the bottom of each calibration range. It is also recommended that single point, moving range charts be used so that calculated standard deviations reflect the normal variation in report values.

6.2.5 Collect at least 20 SPC data points from each work station to ensure that the workstations are under control and that the control limits are representative.

6.3 Tabulate performance data for each workstation and ensure that each workstation complies with the laboratory organization’s data quality objectives.

6.3.1 Tabulate the SPC data by parameter (element), Reference material, assumed true concentration, workstation, average, upper control limit, lower control limit, and standard deviation, as illustrated in Table 1 (see Notes 2 and 3).

TABLE 1 Sample SPC Control Parameter Tabulation

E	RM	Assumed True Conc.	WS	Av.	UCL	LCL	Std. Dev.	
C	638	0.06014	1	0.05996	0.06764	0.05228	0.00256	
			2	0.06040	0.06364	0.05716	0.00108	
			3	0.06005	0.06308	0.05702	0.00101	
	648	0.25665	1	0.25212	0.27069	0.23355	0.00619	
			2	0.25923	0.27402	0.24444	0.00493	
			3	0.25861	0.27283	0.24439	0.00474	
	Mn	638	0.29832	1	0.29620	0.30304	0.28936	0.00228
				2	0.29967	0.30567	0.29367	0.00200
				3	0.29908	0.30643	0.29173	0.00245
648		0.90328	1	0.90408	0.92088	0.88728	0.00564	
			2	0.90408	0.92385	0.88431	0.00659	
			3	0.90168	0.92664	0.87672	0.00832	
P	638	0.00563	1	0.00543	0.00600	0.00486	0.00019	
			2	0.00575	0.00605	0.00545	0.00010	
			3	0.00571	0.00601	0.00541	0.00010	
	648	0.03431	1	0.03413	0.03674	0.03152	0.00087	
			2	0.03447	0.03702	0.03192	0.00085	
			3	0.03434	0.03689	0.03179	0.00085	
S	638	0.01820	1	0.01702	0.02146	0.01258	0.00148	
			2	0.01868	0.02153	0.01583	0.00095	
			3	0.01891	0.02128	0.01654	0.00079	
	648	0.02424	1	0.02330	0.02771	0.01889	0.00147	

TABLE 1 Continued

E	RM	Assumed True Conc.	WS	Av.	UCL	LCL	Std. Dev.
Si	638	0.01688	2	0.02475	0.02940	0.02010	0.00155
			3	0.02467	0.02884	0.02050	0.00139
			1	0.01565	0.01718	0.01412	0.00051
	648	0.23283	2	0.01755	0.01863	0.01647	0.00036
			3	0.01743	0.01830	0.01656	0.00029
			1	0.22900	0.23911	0.21889	0.00337
Cu	638	0.26588	2	0.23240	0.24404	0.22076	0.00388
			3	0.23710	0.24619	0.22801	0.00303
			1	0.26685	0.27555	0.25815	0.00290
	648	0.10700	2	0.26569	0.27295	0.25843	0.00242
			3	0.26511	0.27276	0.25746	0.00255
			1	0.10654	0.11089	0.10219	0.00145
Ni	638	0.69005	2	0.10753	0.11086	0.10420	0.00111
			3	0.10694	0.13784	0.07604	0.01030
			1	0.70014	0.72516	0.67512	0.00834
	648	0.25063	2	0.68252	0.69440	0.67064	0.00396
			3	0.68750	0.71309	0.66191	0.00853
			1	0.25174	0.25906	0.24442	0.00244
Cr	638	0.03746	2	0.24891	0.25350	0.24432	0.00153
			3	0.25123	0.25927	0.24319	0.00268
			1	0.03760	0.03886	0.03634	0.00042
	648	0.23728	2	0.03745	0.03832	0.03658	0.00029
			3	0.03732	0.03813	0.03651	0.00027
			1	0.23190	0.23637	0.22743	0.00149
Sn	638	0.00278	2	0.24012	0.24414	0.23610	0.00134
			3	0.23982	0.24300	0.23664	0.00106
			1	0.00255	0.00507	0.00003	0.00084
	648	0.01424	2	0.00257	0.00296	0.00218	0.00013
			3	0.00322	0.00490	0.00154	0.00056
			1	0.01402	0.01600	0.01204	0.00066
Mo	638	0.06346	2	0.01412	0.01502	0.01322	0.00030
			3	0.01458	0.01668	0.01248	0.00070
			1	0.06253	0.06604	0.05902	0.00117
	648	0.08652	2	0.06398	0.06533	0.06263	0.00045
			3	0.06387	0.06621	0.06153	0.00078
			1	0.08539	0.08995	0.08083	0.00152
V	638	0.02107	2	0.08722	0.08941	0.08503	0.00073
			3	0.08696	0.09011	0.08381	0.00105
			1	0.02076	0.02184	0.01968	0.00036
	648	0.06937	2	0.02114	0.02219	0.02009	0.00035
			3	0.02132	0.02231	0.02033	0.00033
			1	0.06892	0.07123	0.06661	0.00077
Ti	638	0.00224	2	0.06949	0.07219	0.06679	0.00090
			3	0.06969	0.07233	0.06705	0.00088
			1	0.00272	0.00296	0.00248	0.00008
	648	0.04279	2	0.00200	0.00200	0.00200	0.00000
			3	0.00200	0.00200	0.00200	0.00000
			1	0.04285	0.04726	0.03844	0.00147
Al	638	0.02346	2	0.04285	0.04684	0.03886	0.00133
			3	0.04268	0.04688	0.03848	0.00140
			1	0.02373	0.02964	0.01782	0.00197
	648	0.06268	2	0.02343	0.02646	0.02040	0.00101
			3	0.02323	0.02584	0.02062	0.00087
			1	0.06268	0.06721	0.05815	0.00151
648	0.02424	2	0.06198	0.06633	0.05763	0.00145	