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Standard Guide for Optimizing, Controlling and ~~Reporting~~Assessing Test Method Uncertainties from Multiple Workstations in the Same Laboratory Organization [1]

This standard is issued under the fixed designation E2093; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron

E415 Test Method for Atomic Emission Vacuum Spectrometric Analysis of Carbon and Low-Alloy Steel

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E2027 Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials

2.2 *ISO Standards:*[3]

~~ISO 17025~~ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories ~~ISO 9000Quality Management and Quality System Elements~~[3]

ISO 9000 Quality Management and Quality System Elements

2.3 *Other Standards:*

Measurement Systems Analysis Reference Manual[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology E135.

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.~~

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[1] This guide is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.22 on Laboratory Quality.

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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 (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.~~

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, www.ansi.org or from International Organization for Standardization (ISO) at www.iso.ch.

~~[4] Quality Systems Requirements, Chrysler Corporation, Ford Motor Company, and General Motors Corporation—available from AIAG, 26200 Lahser Rd., Suite 200, Southfield, MI 48034–7100.~~

[4] *Measurement Systems Analysis Reference Manual*, Copyright 1990, 1995, Chrysler Corporation, Ford Motor Company, and General Motors Corporation, available from AIAG, 26200 Lahser Rd., Suite 200, Southfield, MI 48034–7100, www.aiag.org.

~~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, and ISO 17025.~~requirements. When using standard test methods, their test results on the same sample should agree with those from other similar laboratories within the reproducibility estimates index (R) published in the standard. Reproducibility estimates are generated as part of the interlaboratory studies (ILS), of the type described in Practice E1601~~, during the standardization process.~~. Competent laboratories participate in proficiency tests, such as those conducted in accordance with Practice E2027, 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'~~Other stations are scattered throughout a facility (at-line laboratory format) and in some cases may even reside at different facilities. 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 ~~then~~than workstation variability. These problems can be minimized if the laboratory organization ~~sets, complies with, and reports a unified set~~ determines the overall uncertainty associated with results reported from multiple workstations and assesses the significance of ~~data quality objectives throughout.~~ the analytical uncertainty to the production process.

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~~ will have an established laboratory quality system. This system shall include the use of documented procedures, the application of statistical control of measurement processes, and participation in proficiency testing. ISO/IEC 17025 describes an excellent model for establishing this type of laboratory quality system.

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 gauging. 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.

4.8 This guide is not intended to provide specific guidance on development of statements of measurement uncertainty such as those required by ISO/IEC 17025. However, the statistical calculations generated using this guide may provide a useful estimate of one Type A uncertainty component used in the calculation of an expanded uncertainty.

4.9 This guide does not provide any guidance for determining the bias related to the use of multiple workstations in a laboratory organization.

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.

~~5.4Document items covered in 5.1-5.3~~

5.4 Perform statistical analysis of the data from the workstations to quantify variation within each workstation and assess acceptability of the variation of the pooled workstation data.

5.5 Document items covered in 5.1 – 5.4.

5.~~5~~6 Establish and document a laboratory organization-wide proficiency test policy that provides traceability to all workstations.

5.~~6~~7 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

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

6.1 *Test Method Identification and Establishment of the Data Quality Objectives*:

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 E415 that covers the analysis of plain carbon and low alloy steel by atomic 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 E350 because those methods cover many different measurement technologies.

~~6.1.2Set 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.~~

6.1.2 Set the data quality objectives for the application of the method throughout the laboratory organization, using customer requirements and other available data. Possible sources of other data may include production process data demonstrating the need for and values of specific analytical process control limits. At the conclusion of this effort, the laboratory organization will know the population standard deviation at specific concentrations. The laboratory can then use these data to draw conclusions about the acceptability of the data produced by the population of work stations.

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. Ensure that all interferences and matrix effects are addressed. ~~Verify the calibrations with certified reference materials not used in~~ It is reasonable to expect that similarly configured instruments will yield similar interference and matrix effect correction factors. Validate the ~~calibration, when possible.~~analytical method for each workstation. 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 Practice E1329, or equivalent practice.

NOTE 1—Generally, it is recommended that SPC concentrations be set about ⅓ from the top and ⅓ 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.

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

~~6.3.1Tabulate 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~~

6.3 Tabulate performance data for each workstation.

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

TABLE 1 Sample SPC Control Parameter Tabulation

E	RM	Assumed True Mass Fraction, %	WS	Mean	UCL	LCL	n	Pooled SD for WS 1-3	3 × Pooled SD for WS 1-3	6 × Pooled SD for WS 1-3	Standard Deviation
C	638	0.06014	1	0.05996	0.06764	0.05228	20				0.00256
			2	0.06040	0.06364	0.05716	20				0.00108
			3	0.06005	0.06308	0.05702	20	0.00171	0.00513	0.01026	0.00101
	648	0.25665	1	0.25212	0.27069	0.23355	20				0.00619
			2	0.25923	0.27402	0.24444	20				0.00493
			3	0.25861	0.27283	0.24439	20	0.00533	0.01599	0.03198	0.00474
Mn	638	0.29832	1	0.29620	0.30304	0.28936	20				0.00228
			2	0.29967	0.30567	0.29367	20				0.00200
			3	0.29908	0.30643	0.29173	20	0.00225	0.00675	0.0135	0.00245
	648	0.90328	1	0.90408	0.92088	0.88728	20				0.00564
			2	0.90408	0.92385	0.88431	20				0.00659
			3	0.90168	0.92664	0.87672	20	0.00694	0.02082	0.04164	0.00832
P	638	0.00563	1	0.00543	0.00600	0.00486	20				0.00019
			2	0.00575	0.00605	0.00545	20				0.00010
			3	0.00571	0.00601	0.00541	20	0.00014	0.00042	0.00084	0.00010
	648	0.03431	1	0.03413	0.03674	0.03152	20				0.00087
			2	0.03447	0.03702	0.03192	20				0.00085
			3	0.03434	0.03689	0.03179	20	0.00086	0.00258	0.00516	0.00085
S	638	0.01820	1	0.01702	0.02146	0.01258	20				0.00148
			2	0.01868	0.02153	0.01583	20				0.00095
			3	0.01891	0.02128	0.01654	20	0.00111	0.00333	0.00666	0.00079
	648	0.02424	1	0.02330	0.02771	0.01889	20				0.00147
			2	0.02475	0.02940	0.02010	20				0.00155
			3	0.02467	0.02884	0.02050	20	0.00147	0.00441	0.00882	0.00139
Si	638	0.01688	1	0.01565	0.01718	0.01412	20				0.00051
			2	0.01755	0.01863	0.01647	20				0.00036
			3	0.01743	0.01830	0.01656	20	0.0004	0.0012	0.0024	0.00029
	648	0.23283	1	0.22900	0.23911	0.21889	20				0.00337
			2	0.23240	0.24404	0.22076	20				0.00388
			3	0.23710	0.24619	0.22801	20	0.00344	0.01032	0.02064	0.00303
Cu	638	0.26588	1	0.26685	0.27555	0.25815	20				0.00290
			2	0.26569	0.27295	0.25843	20				0.00242
			3	0.26511	0.27276	0.25746	20	0.00263	0.00789	0.01578	0.00255
	648	0.10700	1	0.10654	0.11089	0.10219	20				0.00145
			2	0.10753	0.11086	0.10420	20				0.00111
			3	0.10694	0.13784	0.07604	20	0.00604	0.01812	0.03624	0.01030
Ni	638	0.69005	1	0.70014	0.72516	0.67512	20				0.00834
			2	0.68252	0.69440	0.67064	20				0.00396
			3	0.68750	0.71309	0.66191	20	0.00726	0.02178	0.04356	0.00853
	648	0.25063	1	0.25174	0.25906	0.24442	20				0.00244
			2	0.24891	0.25350	0.24432	20				0.00153
			3	0.25123	0.25927	0.24319	20	0.00227	0.00681	0.01362	0.00268
Cr	638	0.03746	1	0.03760	0.03886	0.03634	20				0.00042
			2	0.03745	0.03832	0.03658	20				0.00029
			3	0.03732	0.03813	0.03651	20	0.00033	0.00099	0.00198	0.00027
	648	0.23728	1	0.23190	0.23637	0.22743	20				0.00149
			2	0.24012	0.24414	0.23610	20				0.00134
			3	0.23982	0.24300	0.23664	20	0.00131	0.00393	0.00786	0.00106
Sn	638	0.00278	1	0.00255	0.00507	0.00003	20				0.00084
			2	0.00257	0.00296	0.00218	20				0.00013
			3	0.00322	0.00490	0.00154	20	0.00059	0.00177	0.00354	0.00056
	648	0.01424	1	0.01402	0.01600	0.01204	20				0.00066
			2	0.01412	0.01502	0.01322	20				0.00030
			3	0.01458	0.01668	0.01248	20	0.00058	0.00174	0.00348	0.00070
Mo	638	0.06346	1	0.06253	0.06604	0.05902	20				0.00117

TABLE 1 *Continued*

E	RM	Assumed True Mass Fraction, %	WS	Mean	UCL	LCL	*n*	Pooled SD for WS 1-3	3 × Pooled SD for WS 1-3	6 × Pooled SD for WS 1-3	Standard Deviation
			2	0.06398	0.06533	0.06263	20				0.00045
			3	0.06387	0.06621	0.06153	20	0.00085	0.00255	0.0051	0.00078
	648	0.08652	1	0.08539	0.08995	0.08083	20				0.00152
			2	0.08722	0.08941	0.08503	20				0.00073
			3	0.08696	0.09011	0.08381	20	0.00115	0.00345	0.0069	0.00105
V	638	0.02107	1	0.02076	0.02184	0.01968	20				0.00036
			2	0.02114	0.02219	0.02009	20				0.00035
			3	0.02132	0.02231	0.02033	20	0.00035	0.00105	0.0021	0.00033
	648	0.06937	1	0.06892	0.07123	0.06661	20				0.00077
			2	0.06949	0.07219	0.06679	20				0.00090
			3	0.06969	0.07233	0.06705	20	0.00085	0.00255	0.0051	0.00088
Ti	638	0.00224	1	0.00272	0.00296	0.00248	20				0.00008
			2	0.00200	0.00200	0.00200	20				0.00000
			3	0.00200	0.00200	0.00200	20	0.000046	0.00014	0.000276	0.00000
	648	0.04279	1	0.04285	0.04726	0.03844	20				0.00147
			2	0.04285	0.04684	0.03886	20				0.00133
			3	0.04268	0.04688	0.03848	20	0.0014	0.0042	0.0084	0.00140
Al	638	0.02346	1	0.02373	0.02964	0.01782	20				0.00197
			2	0.02343	0.02646	0.02040	20				0.00101
			3	0.02323	0.02584	0.02062	20	0.00137	0.00411	0.00822	0.00087
	648	0.06268	1	0.06268	0.06721	0.05815	20				0.00151
			2	0.06198	0.06633	0.05763	20				0.00145
			3	0.06222	0.06576	0.05868	20	0.00139	0.00417	0.00834	0.00118

Key:
E = Element determined.
RM = Reference material used for SPC control.
Assumed True Mass Fraction = Grand mean for all work stations.
WS = Work Station.
Mean = Grand Mean from the SPC chart.
Pooled SD = Pooled standard deviation for work station data at the assumed true concentration.
Standard Deviation = Standard deviation from the SPC chart.
Std. Dev. = Standard Deviation from the SPC chart {(UCL-LCL)/6}
Assumed True Mass Fraction = The grand mean for all of the means obtained for the work stations (see Note 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

TABLE Continued

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