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Standard Guide for Optimizing, Controlling and 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 Analysis of Carbon and Low-Alloy Steel by Spark Atomic Emission Spectrometry

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/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories

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 *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. 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. 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.

[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, www.ansi.org or from International Organization for Standardization (ISO) at www.iso.ch.

[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.

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). 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 than workstation variability. These problems can be minimized if the laboratory organization determines the overall uncertainty associated with results reported from multiple workstations and assesses the significance of 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 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.

5.4 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.

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

5.7 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

6.1 *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.

6.1.2 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. It is reasonable to expect that similarly configured instruments will yield similar interference and matrix effect correction factors. Validate the 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.

6.3 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).

NOTE 2—The data in Table 1 were collected over an extended time period on two reference materials using three atomic emission spectrometers in a large, integrated steel mill. The data is typical of that produced in an ISO/IEC 17025 compliant laboratory prior to the availability of this guide.

NOTE 3—When all workstations are calibrated in accordance with 6.2.3 and all SPC charts are generated in accordance with 6.2.4, the grand means for each element/material combination should be sufficiently similar so as not to contribute significantly to the overall uncertainty of the method.

6.3.2 Calculate the pooled standard deviation for each element/SPC reference material for the data produced by the population of work stations. List the values in a manner similar to that shown in Table 1.

6.3.3 Calculate the 6 × Pooled SD value for each element/SPC reference material using the pooled SD calculated as per 6.4. List the values in a manner similar to that shown in Table 1.

6.3.3.1 High standard deviations for any item across all work stations may indicate a problem with the homogeneity of the SPC material (see Note 4).

NOTE 4—The standard deviations for carbon in RM 648 exceeded the expected precision on all three workstations by a small amount, suggesting a possible material problem.

6.3.3.2 High standard deviations for any element on any work station, especially if it shows on more than one SPC material, may indicate a precision problem with that channel on that instrument (see Note 5).

NOTE 5—Workstation 1 showed a high standard deviation for C, S, Sn, and A1 for RM 638. Since the precision on all other work stations were acceptable for these elements, the data suggest that Workstation 1 should be investigated for possible corrective action.

6.4 *Work Station Variability Assessment:*

6.4.1 One suggested approach for determining acceptability of the work variation is based on the approach to determining acceptable measurement system variation described in the Measurement Systems Analysis Reference Manual. This approach compares the measurement system variability observed to the specification range for the parameter being determined by the measurement system. A subjective rating of *acceptable*, *marginally acceptable*, or *unacceptable* is assigned using this comparison. For the purpose of this guide, the population of work stations is considered to be the measurement system.

6.4.1.1 Assign a value to the desired measurement quality objective for the element/mass fraction being determined by the work stations. For example, the user may select the specification range for the element being determined or a melt control limit for the element being determined as the measurement quality objective. Compare data for one of the SPC materials to this measurement quality objective. Choose data from the material with mass fractions falling in the range of the measurement quality objective. If multiple SPC materials have mass fractions falling within the range of the measurement quality objective, it is prudent to select data with the highest variability.

6.4.1.2 Make the comparison using the following formula:

$$(6 \times \textit{pooled SD})/(\textit{measurement quality objective}) \times 100 = \%\ \textit{error}\ (\textit{assigned to work stations}) \quad (1)$$

where a calculated % error of <10 % is considered *acceptable*, 10 % to 20 % is *marginally acceptable*, and >30 % is *unacceptable*.

6.4.1.3 For example, suppose a population of work stations is used to test carbon with a specification range of 0.10 to 0.30%. The laboratory may set its measurement quality objective as one of the three subjective ratings. The calculated 6 × pooled SD of 0.03198 for the SPC material RM648 and the

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
			2	0.06398	0.06533	0.06263	20				0.00045

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
			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).

specification range of 0.20 may be used to perform the acceptability calculation as follows:

$$(0.03198)/(0.20) \times 100 = 16\% \quad (2)$$

The population of instruments may be considered to be performing *marginally acceptable* for determining carbon in samples for this specification.

6.4.1.4 It should be noted that the 6 × pooled SD criteria is a stringent criteria in that it covers some 99.7 % of the values likely to be obtained by the workstations at a particular mass fraction. Some quality systems may be served acceptably if a 95% coverage factor is used. In this case the 4 × pooled SD criteria may be used instead.

6.4.2 Another suggested approach to determining acceptability of the work station population variation is based on a comparison of the work station population SD to "check analysis limits." "Check analysis limits" are taken from published material specification. "Check Analysis Limit" documents issued by organizations such as ASTM and the Society of Automotive Engineers (SAE), which state the allowable tolerance that an over check analysis by a customer may find an element out of the specification limit and still be accepted by the customer. For practical purposes, it is desirable for the 3 × pooled SD of the population of work stations to be less than the check analysis limit to minimize the probability that marginally acceptable material (as determined by a producer lab) would be found unacceptable during an over check analysis (as determined by a customer lab). This method of determining acceptability assumes that there is minimal analytical bias between labs. It must be noted that producer labs are not allowed to use "check analysis limits" for interpretation product conformance.

6.4.2.1 Obtain access to a document containing "check analysis limits."

6.4.2.2 Obtain access to material specifications for which the population of work stations is being used to produce analyses for determination of specification compliance.

6.4.2.3 Select an element/mass fraction for comparison against from the material specification. Determine the corresponding "check analysis limit" from the "check analysis limit" document. Compare the 3 × pooled SD values for the SPC materials to the selected "check analysis limit." The 3 × pooled SD values for the SPC limits should be less than the "check analysis limit."

6.5 Document items covered in 6.1 – 6.4.

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

6.6.1 Establish a laboratory policy for assigning incoming proficiency test samples to the work stations and demonstrating

traceability of results to all work stations based on the elements contained in this guide. That policy might call for proficiency test samples to be analyzed on a rotating basis among all workstations or selecting work stations on a random basis. Also, it must include provision for confirming the acceptability of proficiency test results and confirmation that all work stations were in statistical control at the time the proficiency test samples were analyzed.

6.7 Operate each workstation independently as defined 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.

7. Keywords

7.1 accreditation practice; proficiency testing; workstation