Designation: D6689 – 01 (Reapproved 2006)

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

This standard is issued under the fixed designation D6689; 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 requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D6091 Practice for 99 %/95 % Interlaboratory Detection Estimate (IDE) for Analytical Methods with Negligible Calibration Error

D6512 Practice for Interlaboratory Quantitation Estimate

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

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

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods

STP 15D ASTM Manual on Presentation of Data and Control Chart Analysis, Prepared by Committee E11 on Statistical Methods

2.2 *Other Documents:*

ISO 17025 (previously ISO Guide 25) General Requirements for the Competence of Calibration and Testing Laboratories[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this Guide, refer to Terminology E135 and D1129.

3.2 *Defintions of Terms Specific to This Standard:*

3.2.1 *laboratory organization*—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 the same quality system.

NOTE 1—Key aspects of a quality system are covered in ISO 17025 and include documenting procedures, application of statistical control to measurement processes and participation in proficiency testing.

3.2.2 *maximum deviation*—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.

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

3.2.4 *workstation*—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 measurement quality objectives of the laboratory organization.

4. Significance and Use

4.1 Many analytical laboratories comply with accepted quality system requirements such as NELAC chapter 5 (see Note 2) 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 during the standardization process as part of the interlaboratory studies (ILS). Many laboratories participate in proficiency tests to confirm that they perform

[1] This guide is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

Current edition approved Aug. 15, 2006. Published August 2006. Originally approved in 2001. Last previous edition approved in 2001 as D6689 – 01. DOI: 10.1520/D6689-01R06.

[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, 25 West 43rd St., 4th Floor, New York, New York, 10036.

consistently over time. In both ILS and proficiency testing protocols, it is generally assumed that only one workstation is used to generate the data (see 6.5.1).

NOTE 2—NELAC chapter 5 allows the use of a Work Cell where multiple instruments/operators are treated as one unit: the performance of the Work Cell is tracked rather than each workstation independently. This guide is intended to go beyond the Work Cell to achieve the benefits of monitoring workstations independently.

4.2 Many laboratories have workloads and/or logistical requirements that dictate the use of multiple workstations. 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 then workstation variability. These problems can be minimized if the laboratory organization sets, complies with, and reports a unified set of measurement quality objectives throughout.

4.3 This guide can be used to harmonize calibration and control protocols for all workstations, thereby providing the same level of measurement traceability and control. 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 a prerequisite that all users of 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 gauging. In these areas, users will likely need to establish their own models for defining measurement quality objectives. 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 harmonized, but incorrectly calibrated multiple workstations are obvious and must be avoided.

5. Summary

5.1 Identify the Test Method and establish the required measurement 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 to be identical, so they will be statistically comparable.

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

6. Procedure

6.1 Identify the Test Method and establish the measurement quality objectives to be met throughout the laboratory organization.

6.1.1 Multi-element test methods can be handled concurrently, if all elements are measured using common technology, and 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 optical emission vacuum spectrometry. Workstations can be under manual or robotic control, as long as the estimated uncertainties are within the specified measurement quality objectives. Avoid handling multi-element test methods that concurrently use different measurement technologies. Their procedures and error evaluations are too diverse to be incorporated into one easy-to-manage package.

6.1.2 Set the measurement quality objectives for the use of the Test 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 allowable for any report value, at any concentration level, using the method of choice. An example of a possible method for establishing measurement quality objectives is given in Appendix X1.

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 parameters (personnel, equipment, etc.) 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.

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
			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
			3	0.02323	0.02584	0.02062	0.00087
	648	0.06268	1	0.06268	0.06721	0.05815	0.00151
			2	0.06198	0.06633	0.05763	0.00145
			3	0.06222	0.06576	0.05868	0.00118

E = Element determined
RM = Reference material used for SPC control
Assumed True Conc. = Concentration of E in the RM
WS = Work Station
Av. = Grand Mean from the SPC chart
UCL = Upper control limit from the SPC chart
LCL = Lower control limit from the SPC chart
Std. Dev. = Standard Deviation from the SPC chart {(UCL-LCL)/6}

6.2.3 Harmonize calibration protocols so that equivalent calibrants (i.e. same material source, same stock solutions) are used to cover the same calibration ranges for the same elements on all instruments (see Note 3). 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 accounted for. Verify the calibrations with certified reference materials not used in the calibration, when possible. Record the findings for each workstation.

NOTE 3—It is recommended that the same calibrants are used for each instrument, i.e. same material source, same stock solution, etc. when practical. Calibrations on all Workstations must be performed within a time period such that the stability of the calibration standards are not a concern, if applicable.

6.2.4 Use the same Statistical Process Control (SPC) materials and data collection practices on all workstations (see Note 4). Carry SPC materials through all procedural steps that contribute to the measurement uncertainty. Develop control charts in accordance with STP 15D, or equivalent. Do not develop control charts using SPC data from more than one instrument because this does not allow for adequate trend analysis of the instrument performance.

NOTE 4—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 workstation 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 measurement 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.