

# COKESPEX Proficiency Manual Web Example

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# COKESPEX Proficiency Manual Web Example

## 0

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## 0 Introduction

COKESPEX™ Proficiency Manual Web Example is an Adobe Acrobat® document. In this format the document can be searched using key words such as calibration, sulfur, etc. The proficiency manual includes three sections.

1. Evaluating the Proficiency of Measurements
2. Maintaining a Proficient Quality System
3. Quality Assurance Information (QAI) Sheets

The COKESPEX™ Proficiency Manual Web Example manual does not include all of the information that appears in the full manual, which is updated in January of each year. The full manual provides laboratory staff, management and auditors with a comprehensive document that can be used to address key quality elements including technical competence, measurement uncertainty and training. As such the manual can serve as an essential component of an organization's commitment to quality and should be included in the appropriate quality records.

Further information on the development and revision of this manual can be obtained from,

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# COKESPEX Proficiency Manual Web Example

# 1

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## 1 Evaluating the Proficiency of Measurements

**This section describes the basic characteristics of measurement, the concept of most likely value estimation (MLV) as a basis for evaluating proficiency test data and the QualMark™ performance rating system which provides critical information concerning the uncertainty of measurements. This information allows laboratories to focus continuous improvement efforts where they are most beneficial.**

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## Evaluating the Proficiency of Measurements-1

### 1.1 Characteristics of Measurement

This manual employs quality control and assessment principles from the following publications.

**ISO guide 17025** *General requirements for the competence of testing and calibration laboratories*<sup>1</sup>

**ISO 5725-6** *Accuracy (trueness and precision) of measurement results- Part 6 Use in practice of Accuracy Values*<sup>1</sup>

**Use of Statistics to Develop and Evaluate Analytical Methods**<sup>2</sup>

1 Available from International Organization for Standardization (ISO), [www.iso.ch](http://www.iso.ch)

2 Available from Association of Official Analytical Chemists (AOAC), [www.aoac.org](http://www.aoac.org)

The **three principal components** of a **measurement** are (1) the **system** on which the measurement is made, (2) **the measuring instrument** and (3) the **operator**. In coke testing it is convenient to breakdown (1) the **system** on which the measurement is being made into the **sample** and the **laboratory environment**. A measurement process can be broken down into the following steps.

1. Preparing a laboratory analysis sample from a gross sample.
2. Taking a test sample from the laboratory analysis sample.
3. Treating the test sample physically and, or chemically to eliminate or minimize interferences.
4. Measuring some physical or chemical property of the treated test sample.
5. Developing a calibration curve to employ the measured property to estimate some desired characteristic of the sample.

### 1.2 Analysis of Proficiency Test Data (PTD)

All **measurement processes** exhibit **two fundamental characteristics**. One is **precision**, the **spread of results** generated by the measurement process. The second is **accuracy**, the **agreement of a result with the true value** of the property being measured. The **distinction** between **different measurement processes** calibrated to **same accuracy** is their **respective precision**.

Working from this premise, Quality Associates International Ltd. applies **most likely value (MLV) estimation** to proficiency test data to find the “value most likely to be correct” for a given parameter. **How does the MLV concept differ** from calculation of the **conventional average, weighted averages** based on precision or **robust estimation**?

**The conventional average** assumes all values are equally likely. This approach is not robust against unrealistically large precision, which reflects poor quality control or unrealistically small precision, which reflects unwarranted cleansing or rejection of results.

**Weighted averages** assign the highest weights to those values with the smallest precision. Although this approach is robust against unrealistically large precision it is not robust against unrealistically small precision, which reflects unwarranted cleansing or rejection of results. As a result this approach can be even less reliable than the conventional average as it can assign high weights to values with a very small precision that depart significantly from the central tendency of a distribution.

# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-2

**Robust estimation** chooses a value in the middle of a distribution based on the number of results reported and accepts any value within fixed limits of this value. Although this approach tends to eliminate results that depart significantly from the central tendency of a distribution, it fails to take into account the individual laboratory precision whether it is unrealistically large or unrealistically small.

**Most likely value (MLV) estimation** is an approach that can overcome the shortcomings of the above three methods. MLV starts with the assumption that each laboratory precision and average is equally likely and assigned a vote of 1.

Since **no conclusion** can be drawn concerning **accuracy without acceptable precision** the **first step in MLV involves evaluation of laboratory precision for the COKSEPEX™ proficiency sample. Individual laboratory results** are employed to determine a **calculated laboratory precision**. An **expanded precision** is established from the calculated laboratory precision. The expanded precision is determined employing the  $\alpha$  and  $1-\alpha$  ( $\alpha = 0.95$ ) F percentiles for four measurements (3 degrees of freedom). If at least one other **calculated laboratory precision** does not fall within the expanded precision the laboratory precision vote is changed to 0. This process is conducted iteratively until no more 0 precision votes are assigned. **This approach not only identifies any laboratory with an unrealistically high precision but also any laboratory with an unrealistically low precision.** Once this step is complete a **preliminary MLV COKESPEX™ proficiency sample precision is calculated** by pooling the precision of those laboratories that still have a vote of 1.

There are **two predominant reasons** why laboratories tend to report **unrealistically low precision**. One is **unwarranted rejection of cleansing of results**. This issue is addressed in section 2 of the manual. The second is **participants do not apply the significant figure requirement specified on proficiency report forms**. This number represents the number of measured significant figures to be reported. In the case of COKESPEX™CK0403, one participant reported values of 0.610, 0.610, 0.610 and 0.610 for sulfur. This calculates to a precision of 0.0. The laboratory instrument records showed values of 0.6054, 0.6065, 0.6142, and 0.6149. The lab computer had rounded these values to 0.61. The lab merely multiplied the rounded value back out to three figures after the decimal. That does not constitute 3 significant figures. The values from the instrument readings give 0.605, 0.607, 0.613, and 0.615. These are the correct results to report to 3 significant figures. The laboratory precision calculated from these results is 0.0006.

Laboratories that report **unrealistically low precision** are assigned a minimal acceptable precision calculated from those laboratories that pass the precision screening procedure described above.

# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-3

Using the same procedure as described above for the **preliminary MLV proficiency sample precision**, a **preliminary MLV COKESPEX™ Reference sample precision** is determined. The **preliminary MLV COKESPEX™ Reference sample precision** and the **preliminary MLV COKESPEX™ proficiency sample precision** are pooled to generate a **preliminary MLV combined COKESPEX™ precision**. The **combined precision is multiplied by the 99% t statistic** for the **number of laboratories** reporting results to give a **preliminary expanded MLV uncertainty**.

A laboratory gives **1 accuracy vote** to each **COKESPEX™ Reference sample result** that **agrees** with its **COKESPEX™ Reference result** within the expanded MLV uncertainty. Laboratories that failed the combined COKSEPEX™ precision step are not included in this comparison. In this way each laboratory **COKSEPEX™ Reference Sample Value** is assigned an **MLV accuracy score**. The **current COKSEPEX™ Reference Sample Value** is calculated from the **combined accuracy scores**.

Next each proficiency sample result reported laboratory by a laboratory is **corrected (Note1)** by comparing the **current COKSEPEX™ Reference Sample Value** with the **laboratory value** for the **COKSEPEX™ Reference Sample**.

The adjusted proficiency sample results are used to derive a **final MLV COKSEPEX™ proficiency sample precision employing the same process as described for the preliminary MLV COKSEPEX™ proficiency sample precision**. The **final MLV COKSEPEX™ proficiency precision is multiplied by the 99% t statistic** for the **number of laboratories** reporting results to give a **final expanded MLV uncertainty**.

In the concluding step a laboratory gives **1 accuracy vote** to each **adjusted proficiency sample value** that **agrees** with its **adjusted proficiency sample value** within the final expanded MLV uncertainty. Laboratories that failed the final precision step are not included in this comparison. In this way each laboratory adjusted proficiency value is assigned an **MLV accuracy score**. The **COKSEPEX™ Proficiency Sample Value** is calculated from the **combined accuracy scores**.

### NOTE 1:

**COKESPEX™** requires participants to analyze the **COKSEPEX™ Reference Sample** concurrent with the **COKSEPEX™ proficiency sample**. The **COKESPEX™ Reference Sample** serves as an **external standard** that can be used to **minimize differences** in test results **between laboratories**.

In the **past** COKSEPEX™ has evaluated **performance for laboratory results as reported (unadjusted)**. COKESPEX™ has also evaluated **performance for laboratory results to which a correction has been applied (adjusted)**, based on the **laboratory values submitted for the COKESPEX™ Reference Sample**. COKESPEX™ produced a table summarizing performance for the **unadjusted and adjusted** laboratory results.

**Beginning** with sample **CK0404**, COKESPEX™ will provide a **performance report for adjusted laboratory results only**. If a laboratory quality system does not sustain consistent **precision and accuracy for the COKSEPEX™ proficiency sample and the COKESPEX™ Reference Sample**, it is reasonable to assume the **laboratory quality system** is not **sufficiently stable** to produce reliable results for routine test samples.

# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-4

The table below compares conventional, robust and MLV estimation for volatile wt % dry basis in COKSEPEX™ CK0403. The MLV precision vote and accuracy score for each laboratory is shown. Values highlighted in black bold are those that fail the MLV precision test. The laboratories with averages of 0.544 and 0.570 failed the MLV precision test as having unrealistically low precision even though the laboratory averages are in excellent agreement with the MLV average. As expected the conventional precision is affected by values that depart significantly from the general trend. The robust precision is somewhat underestimated because although it includes the 0.241 value in the robust estimate it also includes the 0.014 and 0.012 values in the robust estimate. Notice the labs remaining after the precision exclusion all receive accuracy scores ranging from 12 to 14. This suggests the COKESPEX™ Reference Sample correction step does minimize difference between laboratories.

Approach	Precision	Average	
Conventional	0.093	0.568	
Robust	0.071	0.558	
MLV	0.081	0.576	
Adjusted Laboratory Average	Adjusted Laboratory Precision	MLV Precision Vote	MLV Accuracy Score
0.444	0.241	0	0
0.665	0.108	1	14
0.523	0.130	1	14
0.740	0.067	1	11
0.551	0.090	1	14
0.522	0.081	1	14
0.542	0.014	0	0
0.570	0.012	0	0
0.585	0.096	1	14
0.565	0.030	1	14
0.713	0.027	1	12
0.458	0.089	1	12
0.601	0.038	1	14
0.628	0.058	1	14
0.608	0.116	1	14
0.505	0.075	1	13
0.530	0.065	1	14
0.466	0.036	1	12

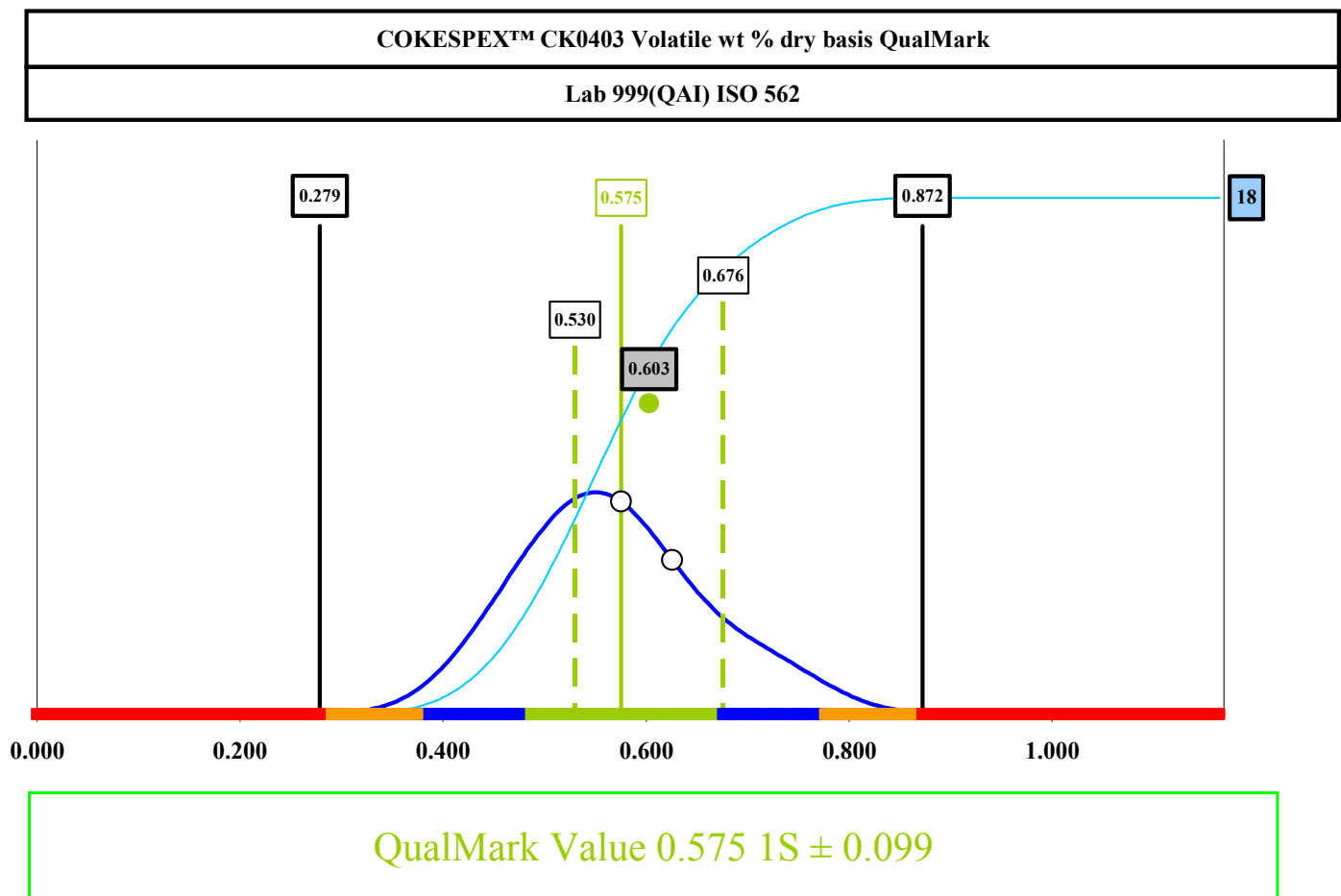
# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-5

### 1.3 QualMark™ Performance Rating System

Quality Associates International Ltd. has established the **QualMark™ performance rating system** for COKESPEX™. QualMark™ employs **MLV estimation** to create **laboratory performance graphs** as well as a **laboratory performance summary table**. The table and graphs provide **information on the uncertainty of measurements** based on component z score analysis of laboratory precision and accuracy. An example graph, summary table and explanation follow.

#### 1.3.1 QualMark™ Graph



# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-6

**MLV estimation** establishes a **QualMark™ value and an associated data distribution**. The distribution graphs are colour coded to provide detailed information with respect to laboratory accuracy, laboratory precision and overall uncertainty.

The **vertical green line on the data distribution identifies the QualMark™ value** with the value appearing in the **green box** immediately above the line.

**Two vertical black bars** define the distribution limits. The limit values appear immediately above the black bars. The area between the two vertical black bars is separated in to three regions, highlighted along the x-axis.

The **green region** represents values **within 1 standard deviation** of the QualMark™ value. This corresponds to values with a **z score of 1 or less**. The single standard deviation limits are listed with the QualMark™ value in the green box below the graph.

The **blue regions** represent values **greater than 1 standard deviation but within 2 standard deviations** of the QualMark™ value. This corresponds to values with a **z score of greater than 1 and less than or equal to 2**.

The **orange regions** represent values **greater than 2 standard deviations but within 3 standard deviations** of the QualMark™ value. This corresponds to values with a **z score of greater than 2 and less than or equal to 3**.

The **red regions** represent values **greater than 3 standard deviations from the QualMark™ value**.

**Adjusted laboratory results** are used to calculate a **laboratory average**. This appears as a **colour coded circle** identifying the **z score** of the laboratory average. The laboratory average appears in a gray box above the circle. In the example above the laboratory average of **0.603** falls within **1 standard deviation, which corresponds to a z score of 1**. Thus the lab average circle is **green**.

Adjusted laboratory results are used to calculate a **day 1 average** and a **day 2 average** shown as **white circles** on the graph. Each white circle is accompanied by a set of **horizontal bars** that represent the **within day precision** for the laboratory. The horizontal bars are either **green** or **red**. **Green** represents **acceptable** within day precision. **Red** represents **suspect** within day precision.

The **within day precision is combined with the difference between the within day averages** to calculate **overall laboratory precision or limits** which appear as **vertical dashed bars** on the graph. Using the QualMark™ single standard deviation limits a colour is assigned to the laboratory limits. In this case the upper and lower **laboratory limits are within 0.073 of the laboratory average**. Since the **lab limits are less than 0.099** they colour coded **green**.

A QualMark™ graph is provided for every parameter reported by a laboratory.

# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-7

### 1.3.2 QualMark™ Summary Table

The **QualMark™ summary table** for all parameters reported by lab 999 appears below. The table lists the parameters reported, the QualMark™ value and the QualMark™ single standard deviation limits. The lab value and lab limits colour coded to the appropriate z score also appear in the table. The table includes three additional columns **A**, **P** and **QS**. The **number** in the **A** column is colour coded to the z score of the lab accuracy and in the **P** column to the z score of the lab precision. The **QS** column indicates the status of the laboratory quality system at the time of the proficiency test.

Lab 999 CK0403 QualMark™								
Parameter	A	P	QS	QualMark Value	QualMark 1S Limits	Lab Value	Lab Limits	Lab Method
Moisture wt%	1	1	IC	0.249	0.029	0.222	0.015	ASTM D 3173
Ash wt % dry basis	1	1	IC	8.30	0.06	8.33	0.03	ASTM D 3174-00
Volatile wt % dry basis	1	1	IC	0.575	0.099	0.603	0.073	ISO 562
Total Sulfur wt % dry basis	1	2	IC	0.612	0.009	0.618	0.017	ASTM D 4239

The table below describes the possible status indicators.

QS Status	Description	A z score	P z score	Quality attributes and recommended action.
IC	In Control	≤3	≤3	Both upper and lower lab limits within QualMark™ distribution limits. No action required.
VA	Verify Accuracy.	≤3	≤3	Either upper or lower lab limit outside QualMark™ distribution limits. Possible results outside acceptable limits. Verify calibration conditions.
VP	Verify Precision	≤3	≤3	Either upper or lower lab limit outside QualMark™ distribution limits. Possible results outside acceptable limits. Verify stability of measurement-to-measurement test conditions.
SA	Suspect Accuracy	>3	≤3	Lab average outside distribution QualMark™ limits. Test calibration suspect.
SP	Suspect Precision	≤3	>3	Lab limits greater than QualMark™ distribution limits. Measurement-to-measurement test conditions unstable.
SAP	Suspect Accuracy and Precision	>3	>3	Lab average outside QualMark™ distribution limits. Lab limits greater than QualMark™ distribution limits. Measurements completely unreliable.

# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-8

**VA and SA are attributable to calibration conditions.** Standards may have deteriorated, may not include a significant interferant present in the test sample or may not include the concentration of the material under test. Instrument operational parameters including temperature profile, detector response or linear dynamic range may have shifted or be significantly different from those obtained in the majority of laboratories with an IC rating.

From the table it can be seen that **lab 999 volatile** has a z score **accuracy rating** of **1** and a **precision rating** of **1**. The lab limits of **0.530** and **0.676** as defined by the vertical dashed bars on the graph both fall within the **QualMark™ distribution limits** of **0.279** and **0.872** as defined by the vertical black bars. The lab 999 volatile measurement is assigned a **QS status of IC**. It is evident from the table the **lab 999 quality system** was **in control** at the time of the proficiency test **for all parameters**.

# COKESPEX Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-9

### 1.3.3 The Benefits of QualMark™

**QualMark™** gives an estimate of the overall uncertainty of a laboratory measurement process while **avoiding** the highly **misleading limitations of basing performance on the z score of the laboratory average only**. The **example below illustrates this point**.

The consensus (MLV) for sulfur in a proficiency test is 0.650 wt %.

The MLV standard deviation is 0.020 wt %.

The upper and lower distribution limits are 0.590 % and 0.710 % respectively.

Lab A has an average of 0.630 wt %, which calculates to a **z score for the lab A average of 1.0**.

Lab B has an average of 0.640 wt %, which calculates to a **z score for the lab B average of 0.5**.

Lab C has an average of 0.625 wt %, which calculates to a **z score for the Lab C average of 1.5**.

This comparison based on lab averages suggests lab B produces the most reliable sulfur measurements followed by lab A and finally lab C. Investigation of the individual laboratory results reveals the following.

Lab A has a precision of 0.05 %. The lab A precision z score is 2.5. The lower expected value for lab A is then 0.58 %. The upper expected value is 0.68 %. **The lower lab limit falls outside the distribution limits.**

**QualMark™ assigns a VP QS status to lab A.**

Lab B has a precision 0.07 %. The lab B precision z score is 3.5. The lower expected value for lab B is then 0.57 %. The upper expected value is 0.71 %. **The lab B limits are wider than the distribution limits.**

**QualMark™ assigns a SP QS status to lab B.**

Lab C has a precision of 0.02 %. The lab C precision z score is 1.0. The lower expected value for lab C is then 0.605 %. The upper expected value is 0.645 %. **Both the lab C average and limits fall within the distribution limits. QualMark™ assigns an IC QS status to lab C.**

This comparison, which constitutes a comprehensive assessment of lab averages and precision shows lab C produces the most reliable sulfur measurements followed by lab B and finally lab A. This is a complete reverse of the limited assessment based on averages only.

All **measurement processes** produce a **central value** with a **spread or distribution of results** around the central value. One would expect a **stable measurement process** to produce the **same central value** with the **same spread of results**. It makes sense a **certain number of results** would be **close to the central value** while the **remainder** of results would be **distributed all the way out to the extremes** of the spread or distribution. In other words, **despite the most intensive control efforts all distributions can and do produce results at the extremes**. **QualMark™** provides participants with **quality assessment information** that allows labs to **identify stable measurements** as well as **measurements that are approaching or exceeding the extremes**. **This information allows laboratories to focus continuous improvement efforts where they are most beneficial.**

# COKESPEX Proficiency Manual Web Example

## 2

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## 2 Maintaining a Proficient Quality System

There are two fundamental characteristics that distinguish a successful quality system. They are technical competence and organizational behaviour with respect to the quality. Without the latter the former is doomed to failure. An analogy familiar to most is that of the computer. Clearly these devices can process and manage information at an astonishing rate and in highly diverse ways. However, ultimately they are only as good as the information with which they are provided. Garbage in equals garbage out. Similarly if the staff and management of an organization are not committed to due diligence in monitoring and, where necessary, correcting the quality of a product or service then technical competence will degrade and ultimately the reputation of the organization will suffer. Quality behaviour should not be confused with attitude. One can have a good attitude about quality but may not be able to translate that attitude to acceptable quality behaviour unless they have the tools and information to do so. This section provides essential information on establishing and maintaining not only technical competence but also an organizational behaviour committed to quality.

# COKESPEX Proficiency Manual Web Example

## Maintaining a Proficient Quality System-1

### 2.1 ISO Guide 17025 and Proficiency

The primary **purpose of proficiency testing** is to provide participants with an **external, objective assessment of a laboratory competence**. One can think of **proficiency testing** as a **monitor** of the **stability of the laboratory quality system**. Obviously there is **not much use** taking part in a proficiency test program if **laboratory management and staff are not committed to establishing and maintaining a stable quality system**.

**ISO Guide 17025** *General requirements for the competence of testing and calibration laboratories* contains the **elements of good laboratory practice** that allow a facility to **establish and maintain a reputation for reliable test work**.

The Introduction to ISO Guide 17025 states, *This International Standard has been produced as the result of extensive experience in the implementation of ISO/IEC Guide 25 and EN 45001, both of which it now replaces. It contains all of the requirements that testing and calibration laboratories have to meet if they wish to demonstrate that they operate a quality system, are technically competent, and are able to generate technically valid results.*

### 2.2 Essential Elements of Good Laboratory Practice

#### 2.2.1 Validation of Laboratory Methods

Laboratories involved in a **proficiency testing (PT)** often use a **variety of standard, modified-standard and in-house methods**.

**ISO Guide 17025** includes the following **conditions** with respect to **test method selection and use**.

- The laboratory **shall use test and/or calibration methods, including methods for sampling**, which meet the **needs of the client** and which are **appropriate for the tests and/or calibrations it undertakes**. **Methods published in international, regional or national standards shall preferably be used**. The laboratory shall ensure that it **uses the latest valid edition of a standard** unless it is not appropriate or possible to do so. When necessary, the **standard shall be supplemented with additional details to ensure consistent application**.
- When it is necessary to use **methods not covered by standard methods**, these shall be subject to **agreement with the client** and shall include a clear specification of the client's requirements and the purpose of the test and/or calibration. The method developed shall have been **validated appropriately before use**.
- The laboratory **shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm** that the methods are **fit for the intended use**. The **validation** shall be as extensive as is necessary to **meet the needs of the given application** or field of application. The laboratory shall **record the results obtained**, the **procedure used** for the validation, and a statement as to whether the method is fit for the intended use.

# COKESPEX Proficiency Manual Web Example

## Maintaining a Proficient Quality System-2

In summary these conditions mean that **as long as a laboratory is using the most recent version of a standard method strictly within the defined scope** and conditions of the method there is **no need to validate the laboratory method**. **Otherwise the laboratory shall provide objective technical evidence** that demonstrates the **ability to consistently produce equivalent results** for a specific test **whether using a standard, modified standard or in-house procedure**.

**Laboratories do not need to validate methods of test published by standard writing organizations (SWOs)** because they are normally **validated through** a two-step process carried out by **qualified laboratories**. Most SWOs categorize a **qualified laboratory** as one with a **reputation for reliable test work (2.1)**. **In other words the laboratories must have experience with the use and application of standard methods as well as procedures for the validation of non standard methods**.

The process to validate a standard method proceeds as follows. Initially, a **ruggedness test** is performed by **one or two qualified laboratories to identify conditions that must be controlled to ensure** measurement results fit for the intended use can be obtained. Then an **Interlaboratory Study (ILS)** involving a **minimum of eight qualified laboratories** is conducted. These **qualified laboratories** carry out a series of measurements on representative samples. **Conditions identified in the ruggedness test are controlled while keeping the test equipment, operator(s), calibration and environment constant from measurement to measurement**. These are known as **repeatability conditions**. Results generated from an ILS under these constraints are used to derive a value known as the repeatability of the standard method of test. This **repeatability value represents the within laboratory precision** (spread of results) that can be expected **under repeatability conditions**.

Except in the case of a controlled ILS, it is **obvious that repeatability conditions do not prevail on a day-to-day basis in the laboratory**. Laboratory staff change, equipment is serviced, repaired or replaced, calibrations are updated and most certainly the environment does not remain constant over extended periods of time. All **SWOs make it clear that an on-going process employing control charts is essential to establish and monitor** the impact of changes in any of these factors on **within laboratory precision**.

# COKESPEX Proficiency Manual Web Example

## Maintaining a Proficient Quality System-3

### 2.2.2 Evaluating the Acceptability of Laboratory Results

Laboratory staff, management and clients must understand the constraints imposed when employing the precision values in standards to evaluate the acceptability of within and between laboratory results. The following points summarize these constraints.

- When using the **repeatability** values in standards to evaluate the acceptability of within laboratory results, conduct repeat analysis employing **repeatability conditions**. This means **each repeat measurement is performed** employing the **same operator, equipment, and calibration combination**. Conduct each measurement **in a period of time during which there is minimal change in the laboratory environment**.
- **Standards** also include, in most cases, a **wider precision value** known as **reproducibility**. This precision value **recognizes operator, equipment and calibration combinations as well as environmental conditions** are **most certainly different from laboratory to laboratory**. However, when **comparing results** under these conditions it is **essential test samples be taken from a representative portion of the same laboratory analysis sample**, because that is exactly how the reproducibility value was originally derived. In other words, **identical equipment must be employed to obtain and prepare the laboratory analysis sample**, which is to be used to determine acceptability of results from different laboratories. Another way of looking at this is the **reproducibility value in a standard cannot be used** to determine the acceptability of **results from different laboratories if those results are determined on samples obtained and prepared employing distinctly separate equipment which we shall call preparation system A and preparation system B**. The results can be compared if the laboratories exchange the samples. It is then **acceptable to compare, the different laboratory results for preparation system A or for preparation system B**.
- The **repeatability value** in a standard **can be used to determine the acceptability of results from different laboratories**. If this approach is to be used the **same sample limitations apply as** for the **reproducibility** case described above. **In addition** it is necessary to include a **blind certified reference material (CRM) or reference material (RM) traceable to a recognized CRM** in the evaluation. The CRM or RM can be used to **ascertain the impact of the different operator, equipment and calibration combinations as well as environmental conditions** on the results from the different laboratories.

### 2.2.3 Entry and Verification of Results

**Establish data entry, calculation and verification procedures. Review these procedures at least once a year.** The individual entering or calculating data **should not verify** data entry or calculations.

**Data verification** includes **analysis of calibration and/or control samples** concurrent with the laboratory analysis samples. **Examine calibration and control sample results for suspect results before reporting laboratory analysis sample results to clients.** Establish written procedures describing **action** to be taken when a **calibration or control result** does **not fall within validated laboratory limits**.

# **COKESPEX Proficiency Manual Web Example**

## **Maintaining a Proficient Quality System-4**

### **2.2.4 Reporting Results from Repeat Analysis**

**See Full Manual**

### **2.2.5 Calibration of Laboratory Instrumentation**

**See Full Manual**

### **2.2.6 Reference Materials**

**See Full Manual**

### **2.2.7 Extraction and preparation of test portions for analysis**

**See Full Manual**

### **2.2.8 Maintenance, Certification, and Calibration of Laboratory Balances**

**See Full Manual**

# COKESPEX Proficiency Manual Web Example

## 3

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### 3 Quality Assurance Information (QAI) Sheets

This section contains information on factors known to impact the reliability of measurements in the coke-testing laboratory. The information in these QAI sheets has been extracted from over sixty (60) ruggedness, method validation and certification studies. Such studies are conducted on a on-going basis by standard writing bodies<sup>1</sup>, agencies providing Certified Reference Materials (CRMs)<sup>2</sup> for coke testing as well as industrial and governmental research organizations<sup>3</sup>. The information can be used for training purposes, may prove useful in resolving quality excursions within the laboratory, as well as reconciling disputes between laboratories.

1 ASTM, BSI, DIN, GBC, ISO, SAA, SABS

2 BCR, NIST, SABS

3 CANMET, CSIRO, CCMRI, CONSOL R&D

# **COKESPEX Proficiency Manual Web Example**

## **3.1 QAI Sheet Moisture wt %**

**See Full Manual**

## **3.2 QAI Sheet Ash wt % dry basis**

**See Full Manual**

# COKESPEX Proficiency Manual Web Example

## Quality Assurance Information (QAI) Sheets-6

### 3.3 QAI Sheet Volatile Matter wt % dry basis

#### International, Regional and National standards

ASTM D 3175

ASTM D 5142

BS 1016

ISO 562

This QAI sheet describes factors that are known to affect the measurement of volatile matter in the analysis sample. The measurement characteristic(s) precision and/or accuracy affected by the factor appear in brackets.

#### 3.3.1 Final Soak Temperature (Accuracy)

Standards specify **final soak temperatures** that range from **890 °C to as high as 970 °C**. A **lower final soak temperature** will **always give a lower volatile matter** result if all other conditions of test are held constant and in control.

#### 3.3.2 Crucible Material and Geometry (Accuracy)

**Crucibles** made of **different materials** can **affect the rate of heat transfer to the sample**. For example the transfer of heat to a **sample in a platinum crucible** can be **more rapid than** that in a **quartz or nickel chromium** crucible. As a result the **decomposition and polymerization reactions** that occur during the volatile matter test are likely to be **different for each crucible type**. Several **studies** conducted throughout the 80s and more recently in the late 90s have **failed to provide a definitive method for reconciling differences** in volatile matter results **between crucible types**. It is **not possible to reliably predict how the crucible material affects the end result**.

The geometry of a crucible has also been shown to have an effect on volatile matter results.

# COKESPEX Proficiency Manual Web Example

## Quality Assurance Information (QAI) Sheets-7

### 3.3.3 Volatile Matter Heating Rate (Precision & Accuracy)

COKESPEX™ continues to detect **differences in volatile matter results from TGA analyzers versus those from tube furnaces.**

**Standards specify heating** the coke sample at a final soak temperature of **890 °C to 970 °C** for a total soak time of **7 minutes**. When **employing a TGA it necessary to match these specifications** as closely as possible **to obtain comparable results**. **Cool the crucibles used for the volatile matter determination in a dessicator. Do not under any circumstances employ a constant weight condition to terminate the test. Do not employ TGA sequences recommended for coal.**

**Do not calibrate a TGA analyzer** for the determination of coke volatile matter **with coal samples**. The primary reason is because the **volatile matter of coal** is well **outside the acceptable calibration range (see 2.2.5)**. If the **conditions of test** employed to determine the **volatile matter content of a coal standard are not known do not use the coal standard for calibration**. The conditions of test under which the volatile matter of coal, especially those that pop or spark is determined, can be significantly different from those specified for coke.

**A muffle furnace can also be used for the determination of volatile matter**. The volatile matter determination is restricted to a zone where the temperature is controlled to recover in 4 minutes to the final soak temperature. Once the furnace has recovered to the final soak temperature, samples are heated for an additional 7 minutes. The total time is 11 minutes.

A study carried out by British researchers in the 1950s demonstrated a **4-position square wire rack** provided the most **consistent recovery to the final soak point**. Place the **rack** such that it is **centered below the thermocouple**. Place a **crucible containing 1 g of sand** in any of the four **positions not employed for an actual determination**.

# **COKESPEX Proficiency Manual Web Example**

## **Quality Assurance Information (QAI) Sheets-8**

### **3.4 QAI Sheet Gross Calorific Value dry basis**

**See Full Manual**

### **3.6 QAI Sheet Total Sulfur wt % dry basis**

**See Full Manual**