

# CANSPEX™ Proficiency Manual Web Example

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# CANSPEX™ Proficiency Manual Web Example

## 0

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## 0 Introduction

CANSPEX™ 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 CANSPEX™ 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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# 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 coal 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.

# CANSPEX™ Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-2

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

**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**. 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 **final MLV precision is calculated** by combining the calculated 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 CANSPEX™ 2004-4, one participant reported values of 0.310, 0.310, 0.310 and 0.310 for sulfur. This calculates to a precision of 0.0. The laboratory instrument records showed values of 0.3054, 0.3065, 0.3142, and 0.3149. The lab computer had rounded these values to 0.31. 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 0.305, 0.307, 0.313, and 0.315. 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.

The **concluding step in MLV estimation involves evaluating laboratory values**. The **final MLV precision is multiplied by the 99% t statistic** for the **number of laboratories** reporting results to give an expanded MLV uncertainty. A laboratory gives 1 accuracy vote to each result that agrees with its average within the expanded MLV uncertainty. Laboratories that failed the precision step are not included in this comparison. In this way each laboratory average is assigned an **MLV accuracy score**.

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## Evaluating the Proficiency of Measurements-3

The table below compares conventional, robust and MLV estimation for manganese (Mn) in CANSPEX 2004-4 ash. 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. Those highlighted in gray received 0 accuracy votes. Notice the laboratories with averages of 326 and 334 failed the MLV precision test as having unrealistically low precision. As expected the conventional precision and average are affected by values that depart significantly from the general trend. Although the robust average agrees with the MLV average the robust precision is somewhat underestimated as it includes the values from the laboratories reporting 326 and 334.

Approach	Precision	Average	
Conventional	23	298	
Robust	5	303	
MLV	6	303	
Laboratory Average	Laboratory Precision	MLV Precision Vote	MLV Accuracy Score
211	18	1	2
334	3	1	9
250	18	1	3
314	4	1	12
300	3	1	10
400	10	1	0
317	5	1	12
309	5	1	11
353	3	1	5
315	3	1	12
302	6	1	10
354	39	0	0
221	26	0	0
235	103	0	0
310	2	1	12
256	14	1	4
223	2	1	4
313	3	1	12
296	3	1	11
332	9	1	10
359	12	1	2
235	5	1	5
289	5	1	7
276	4	1	4
326	2	0	0
290	7	1	7
334	0	0	0

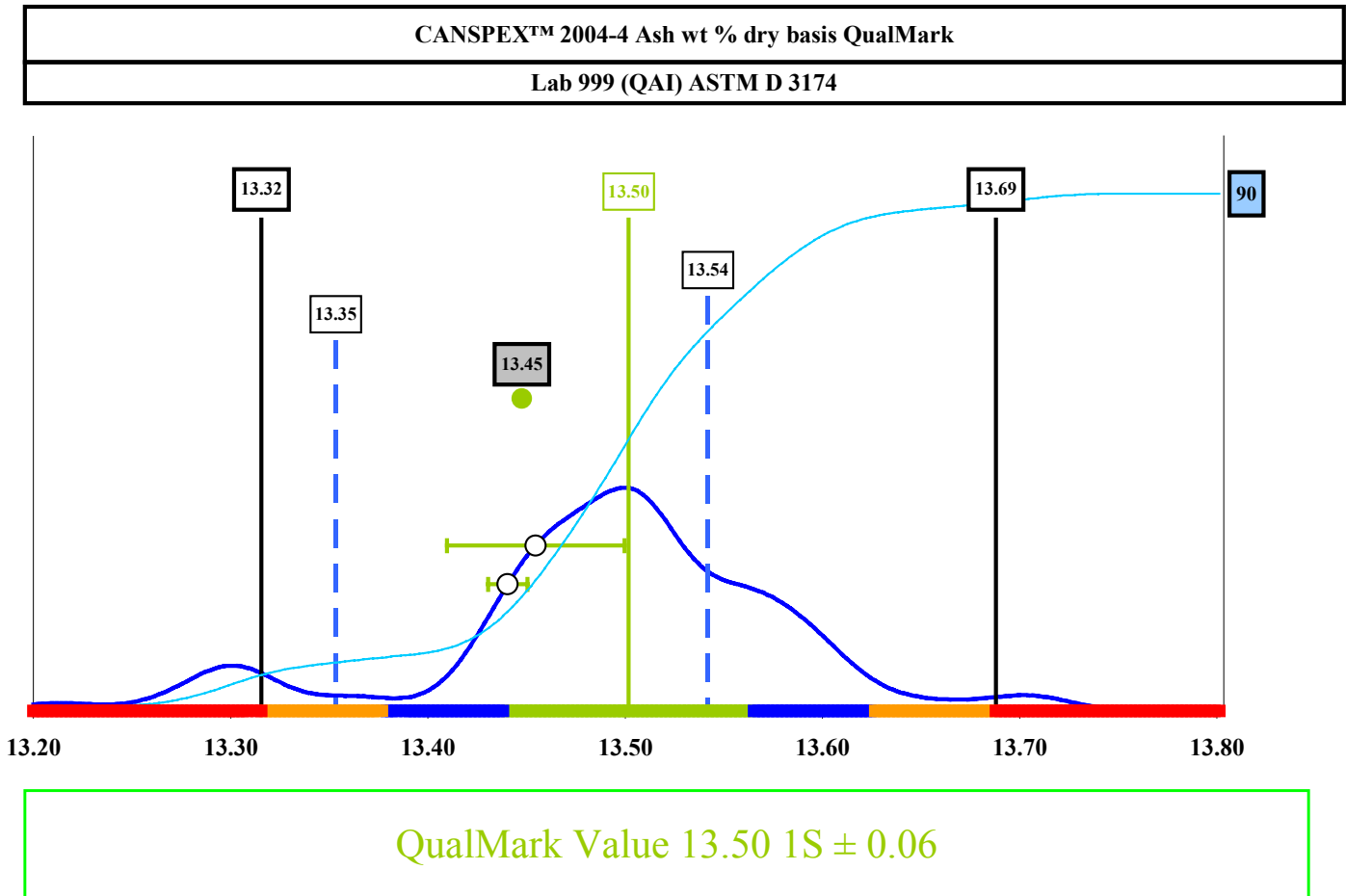
# CANSPEX™ Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-4

### 1.3 QualMark™ Performance Rating System

Quality Associates International Ltd. has established the **QualMark™ performance rating system** for CANSPEX™. 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



# CANSPEX™ Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-5

**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**.

**Individual 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 **13.45** falls within **1 standard deviation, which corresponds to a z score of 1**. Thus the lab average circle is **green**.

Individual 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 0.09 of the laboratory average**. Since the **lab limits** are **between 0.06 and 0.12** they colour coded **blue**.

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

# CANSPEX™ Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-6

### 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.

The table below describes the possible status indicators.

Lab 999 2004-4 QualMark™								
Parameter	A	P	QS	QualMark Value	QualMark 1S Limits	Lab Value	Lab Limits	Lab Method
Moisture wt%	2	2	IC	2.55	0.07	2.43	0.11	ASTM D 3173
Ash wt % dry basis	1	2	IC	13.50	0.06	13.45	0.09	ASTM D 3174
Btu/lb dry basis	1	1	IC	12513	23	12500	21	ISO 1928
Nitrogen wt % dry basis	1	4	SP	1.43	0.04	1.44	0.16	ASTM D 5373
Total Sulfur wt % dry basis	1	2	IC	0.312	0.009	0.314	0.010	ASTM D 4239

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.

# CANSPEX™ Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-7

**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 ash** has a z score **accuracy rating** of **1** and a **precision rating** of **2**. The lab limits of **13.35** and **13.54** as defined by the vertical dashed bars on the ash graph both fall within the **QualMark™ distribution limits** of **13.32** and **13.69** as defined by the vertical black bars. The lab 999 ash 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 except nitrogen**. The **SP** rating for nitrogen suggests one or more erratic conditions of test. This could be attributable to instrument leaks, depleted conversion catalyst, or erratic response of the nitrogen detection cell. The **CANSPEX™ Proficiency Manual** provides guidance on factors that affect test calibration and stability.

# CANSPEX™ Proficiency Manual Web Example

## Evaluating the Proficiency of Measurements-8

### 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 QAI proficiency test is 2.00 wt %.

The MLV standard deviation is 0.10 wt %.

The upper and lower distribution limits are 1.70 % and 2.30 % respectively.

Lab A has an average of 1.90 wt %, which calculates to a **z score for the lab A average of 1.0**.

Lab B has an average of 2.05 wt %, which calculates to a **z score for the lab B average of 0.5**.

Lab C has an average of 1.85 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 0.25 %. The lab A precision z score is 2.5. The lower expected value for lab A is then 1.65 %. The upper expected value is 2.15 %. **The lower lab limit falls outside the distribution limits.**

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

Lab B has a precision 0.35 %. The lab B precision z score is 3.5. The lower expected value for lab B is then 1.70 %. The upper expected value is 2.40 %. **The lab B limits are greater than the distribution limits.**

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

Lab C has a precision of 0.05 %. The lab C precision z score is 1.0. The lower expected value for lab C is then 1.80 %. The upper expected value is 1.90 %. **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.**

## 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.

# CANSPEX™ 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**.

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.*

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

### 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.

# CANSPEX™ 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**.

# CANSPEX™ 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**.

# **CANSPEX™ Proficiency Manual Web Example**

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

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

**See Full Mnaual**

### **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 Fulll Manual**

## 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 coal-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 coal 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 CCRMP, BCR, NIST, SABS, USGS

3 CANMET, CCME, CSIRO, CCMRI, CONSOL R&D, CRL, EPA, EPRI, USGS

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## Quality Assurance Information (QAI) Sheets-1

### 3.1 QAI Sheet Moisture wt %

#### International, Regional and National standards

AS 1038  
ASTM D3173  
ASTM D 5142  
BS 1016  
DIN 51718  
GB/T212  
ISO 589  
ISO 11722  
ISO 5068  
SABS 925

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

#### 3.1.1 Condition of test sample (Accuracy)

Laboratories normally employ **two alternatives for extracting a test portion** for analysis. **Procedure A** involves mixing the sample in the bottle and **extracting a test portion from the bottle** for analysis. **Procedure B** involves mixing the sample in the bottle, extracting sufficient material for the test, allowing the **material to equilibrate with the lab atmosphere** and then conducting analysis on **test portions from the equilibrated material**. The majority of laboratories employ procedure A. Samples treated by procedure B can lose or pick up moisture depending on the ambient humidity of the laboratory. As a result laboratories employing procedure B can produce residual moisture results that are either lower or higher than that produced by procedure A. For this reason as well as the obvious practical limitations of ensuring the moisture condition of test samples exchanged by laboratories does not change, comparing moisture results determined on the analysis sample from different laboratories has absolutely no technical validity.

#### 3.1.2 Type of Purging Atmosphere (Accuracy)

Studies have shown **nitrogen will produce higher moisture results than air** conditioned to the same requirements. Since the **residual moisture value** is employed to **calculate other coal parameters to a dry basis**, nitrogen as a purging atmosphere will tend to produce higher dry basis estimates for other parameters than air.

# CANSPEX™ Proficiency Manual Web Example

## Quality Assurance Information (QAI) Sheets-2

### 3.1.3 Dryness of Purging Atmosphere (Precision & Accuracy)

**Control the dryness** of the purging atmosphere **to the requirement specified in the standard**. Verify this dryness requirement is met by passing the drying atmosphere through a suitable desiccant and then through a tube containing magnesium perchlorate for a fixed period of time. Remove the tube after a fixed period of time and weigh. By knowing the flow rate the amount of moisture in the dried purging atmosphere in mg/L can be calculated.

### 3.1.4 Insufficient drying time (Precision & Accuracy)

**Lower rank coals** have the capacity to **hold significantly more moisture than higher rank coals**. A low rank coal may not lose all of this moisture by the end of a one-hour drying period. **Place low rank coal** samples in the drying apparatus for **additional half hour periods** until a **minimum change in mass** is reached. The term minimum change in mass is used because excessive drying can result in oxidation of low rank coals. Studies have shown that most low rank coals will achieve a minimum change in mass after 1.5 to 2 hours. The example below lists the weight of 1g of low rank coal after an initial one hour drying period and then after each additional half hour drying period. The minimum mass is indicated in bold.

1h: 0.8256g, **1.5h: 0.8149g**, 2h: 0.8156g, 2.5 h 0.8270g

This low rank coal has residual moisture content of 18.51 wt %.

### 3.1.5 Concurrent drying coals of significantly different rank (Precision)

If a low rank coal is dried in a batch with higher rank coals, the coals can exchange moisture in an unpredictable way. **Do not dry coals of significantly different rank together**.

### 3.1.6 Cooling and Weighing of Samples (Precision & Accuracy)

On the **same day as test samples are to be analyzed**, **heat empty capsules or crucibles** and lids including those used in instrumental analyzers under the **conditions at which the sample is to be dried**. **Cool the capsules and lids in a dessicator**.

Some **laboratories process a large number of samples** at a time **in one batch**. Before the **latter samples** are **weighed** they can **pick up moisture** from the dessicator environment particularly if the samples are not covered with a suitable lid. This occurs because **coal can act as a better desiccant than certain chemical agents**. **Cover the sample with a lid to minimize the pick up of moisture** from the **dessicator environment** as well as **other coal samples** in the dessicator. **Dry samples in batches of 6 to 8 further minimize these effects**. Once the **initial batch** is put **in the drying apparatus**, **another** is weighed out no less than **15 minutes later** and placed in the drying apparatus. Remove samples in each batch sequence from the drying apparatus at the required time interval cool and weigh.

# **CANSPEX™ Proficiency Manual Web Example**

## **Quality Assurance Information (QAI) Sheets-3**

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

**See Full Manual**

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

**See Full Manual**

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

**See Full Manual**

### **3.5 QAI Sheet Carbon, Hydrogen and Nitrogen wt % dry basis**

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

### **3.7 QAI Sheet Forms of Sulfur wt % dry basis**

**See Full Manual**

### **3.8 QAI Sheet Chlorine $\mu\text{g/g}$ dry basis**

**See Full Manual**

### **3.9 QAI Sheet Fluorine $\mu\text{g/g}$ dry basis**

**See Full Manual**

### **3.10 QAI Sheet Mercury $\text{ng/g}$ dry basis**

**See Full Manual**

### **3.11 QAI Sheet Selenium $\mu\text{g/g}$ dry basis**

**See Full Manual**

### **3.12 QAI Sheet Swelling Number**

**See Full Manual**

### **3.13 QAI Sheet Fusibility Properties of Ash (FPA)**

# **CANSPEX™ Proficiency Manual Web Example**

**See Full Manual**

## **3.14 QAI Sheet Composition of Ash Major and Minor Oxides and Trace Constituents**

**See Full Manual**

# **CANSPEX™ Proficiency Manual Web Example**