



QUALITY ASSOCIATES INTERNATIONAL®, LTD

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QUALITY ASSURANCE IS THE BEST INSURANCE

QAI In Control Reference Materials™ (ICRM™) (REFERENCE MATERIALS FOR THE COAL AND COKE INDUSTRY)

Certified Reference Materials (CRMs) are available and should be used for calibration purposes in coal and coke testing laboratories. Once a method has been properly calibrated, the continued use of CRMs as control samples may not be a cost effective approach to quality control.

The use of secondary reference materials to monitor performance with respect to standard or contractual specifications can be an effective means of maintaining data reliability while reducing laboratory costs. Quality Associates International®, LTD (QAI) offers **In Control Reference Materials™ (ICRM™s)**, as reliable secondary reference materials **traceable to recognized CRMs**. Each QAI ICRM™ can be used not only to compliment the use of CRMs but also to monitor performance-based requirements in the coal and coke testing laboratory

ICRM™s are available for the following materials:

- Coal
- Coke
- Coal derived ashes

Each QAI ICRM™ is prepared on a rotary splitter. The values attached to each ICRM™ are developed through test programs run by QAI involving anywhere from 30 to over 100 laboratories. The same laboratories take part in QAI evaluations of candidate CRMs for agencies such as the National Institute for Standards and Technology (NIST) in the USA, the South African Bureau of Standards (SABS) and the US Geological Survey (USGS). This approach not only permits a direct comparison of the homogeneity of each QAI ICRM™ with recognized CRMs but also provides a means of comparing values generated by accepted industry standard test methods with those obtained using primary certification methods.

An example of an ICRM™ certificate for coal, as well as major, minor and trace appear on pages 2-5. These certificates provide explicit information with respect to the use, traceability and handling of a QAI ICRM™. Coal ICRM™s are priced in the range of \$1.00 to \$2.00 per gram and sold in bottles of 75 grams to 100 grams. Ash ICRM™s are priced in the range of \$5.00 to \$6.00 per gram and sold in bottles of 15 grams to 30 grams. Discounts are automatically applied to members of the CANSPEX™ and COKESPEX™ programs.

Contact QAI at louisjanke@renc.igs.net to obtain a full list of all available ICRM™s. IF QAI does not have an ICRM™ that suits a specific need every effort shall be made to find a suitable alternative source. In addition, the QAI web site provides links to recognized certified agencies that provide CRMs for coal and coke testing laboratories.



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Select ICRM™ 2001-4 hvAb Coal Kentucky Pond Creek

Parameter	Most Likely Value	99 % Confidence Interval Most Likely Value	Degrees of Freedom	1S Test Limits	2S Test Limits	3S Test Limits
Moisture wt%	2.14	0.04	72	0.08	0.16	0.24
Ash wt % db	8.10	0.01	62	0.05	0.09	0.14
Volatile wt % db	34.82	0.12	60	0.20	0.40	0.60
Btu/lb db	13691	9	74	27	53	80
Carbon wt % db	77.36	0.25	34	0.27	0.53	0.80
Hydrogen wt % db	4.98	0.05	30	0.07	0.13	0.20
Nitrogen wt % db	1.51	0.03	33	0.03	0.06	0.10
Total Sulfur wt % db	0.739	0.006	74	0.014	0.029	0.043
Pyritic Sulfur wt % db	0.171	0.012	9	0.012	0.023	0.035
Chlorine µg/g db	1898	63	27	115	229	344
Fluorine µg/g db	48	7	14	9	18	26
Mercury ng/g db	55	6	12	7	13	20
Selenium µg/g db	5.0	0.8	6	0.5	1.0	1.4
FSI	3.0	0.5	31	0.5	1.0	1.5



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QUALITY **A**SSURANCE IS THE BEST **I**NSURANCE

Select ICRM[™] 2001-4 hvAb Coal Kentucky Pond Creek

This sample is not to be construed or used as certified reference material as defined in ISO Guide 30. This sample can be employed as a reference material as defined in ISO Guide 30. This sample meets the traceability requirements outlined in ISO Guide 17025 *General requirements for the competence of testing and calibration laboratories*.

The **most likely value (MLV)** can be used for measurement control¹. In addition to the 99 % confidence interval of the MLV, test limits are provided. These are the **1 standard deviation (1S)**, **2 standard deviation (2S)** and **3 standard deviation (3S)** test limits for a **single calibration or control measurement**. The **test limits** not only **take into account the confidence interval** of the MLV, which includes **sample inhomogeneity** but also the **precision of a stable unbiased measurement process** employed to determine the calibration or control value.

A laboratory should **investigate and rectify calibration or control conditions** for any **value in excess** of the **3 standard deviation (3S)** test limits. A laboratory should implement **practices to verify the stability of calibration and control conditions** for any value **between 2 standard deviation (2S) test limits and 3 standard deviation (3S) test limits**. A **single calibration or control value** within **1 standard deviation (1S) and 2 standard deviation (2S) test limits can be considered acceptable**.

The following information concerning **traceability to recognized Certified Reference Materials (CRMs) or Reference Materials (RMs)** are included in this certificate for internal and external audit purposes.

- ICRM[™] 2001-4 was run through the same controlled proficiency test program (PTP) for the same test parameters as the following Certified Reference Material coals ISGS IBC 105, NIST1632c, NIST 2383b, NIST 2684b, NIST 2685b, NIST 2692b, SABS SARM 18.

1 The ICRM[™] sample bottle should be marked with a red line approximately $\frac{1}{4}$ of the height of the bottle from the bottom. When the level of the ICRM[™] sample reaches the red line, a new bottle of control sample should be tested as an unknown to verify the new control sample meets quality specifications stated on the control sample certificate. Prior to taking a test portion for analysis, mix the sample by rotating the bottle end over end for not less than 1 minute. As the sample in the bottle is depleted the residual moisture content can change. A change of 0.1 wt % in the residual moisture can affect the dry heating value by as much as 15 BTU/lb. Conduct measurements of the residual moisture content whenever the laboratory relative humidity changes by more than 10 %.



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QUALITY ASSURANCE IS THE BEST INSURANCE

Select ICRM™ 2000-1 Illinois 6 Laboratory Ash

Parameter	Most Likely Value	99 % Confidence Interval Most Likely Value	Degrees of Freedom	1S Test Limits	2S Test Limits	3S Test Limits
SiO ₂ wt%	43.74	0.55	32	0.58	1.15	1.73
Al ₂ O ₃ wt%	15.88	0.22	29	0.25	0.50	0.76
Fe ₂ O ₃ wt%	18.21	0.33	35	0.37	0.74	1.10
CaO wt%	6.32	0.10	33	0.09	0.17	0.26
MgO wt%	0.909	0.027	27	0.039	0.077	0.116
Na ₂ O wt%	0.859	0.023	38	0.033	0.065	0.098
K ₂ O wt%	1.78	0.03	37	0.04	0.07	0.11
P ₂ O ₅ wt%	0.156	0.020	31	0.018	0.035	0.053
TiO ₂ wt%	0.746	0.013	32	0.019	0.038	0.057
BaO wt%	0.043	0.008	10	0.010	0.020	0.031
SrO wt%	0.026	0.005	7	0.003	0.005	0.008
SO ₃ wt%	8.86	0.34	26	0.28	0.56	0.84
Arsenic µg/g	16	3	6	3	5	8
Beryllium µg/g	4.3	0.4	10	0.3	0.7	1.0
Cadmium µg/g	1.6	0.4	4	0.5	0.9	1.4
Cobalt µg/g	28	3	11	2	5	7
Chromium µg/g	122	7	13	6	13	19
Copper µg/g	63	6	11	6	12	19
Manganese µg/g	459	17	19	19	37	56
Nickel µg/g	90	7	14	7	13	20
Lead µg/g	50	7	10	4	7	11
Antimony µg/g	1.8	0.3	6	0.2	0.3	0.5
Vanadium µg/g	124	10	12	7	13	20
Zinc µg/g	588	32	17	30	60	90



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A laboratory should **investigate and rectify calibration or control conditions** for any **value in excess** of the **3 standard deviation (3S)** test limits. A laboratory should implement **practices to verify the stability of calibration and control conditions** for any value **between 2 standard deviation (2S) test limits and 3 standard deviation (3S) test limits**. A **single calibration or control value** within **1 standard deviation (1S)** and **2 standard deviation (2S)** test limits **can be considered acceptable**.

The following information concerning **traceability to recognized Certified Reference Materials (CRMs) or Reference Materials (RMs)** are included in this certificate for internal and external audit purposes.

- ICRM™ 2000-1 was run through the same controlled proficiency test program (PTP) for the same test parameters as the following Certified Reference Materials, CCRMP MRG-1, CCRMP WGB-1, CCRMP SY-4, NIST 2690, and NIST 1633b.

1 The ICRM™ sample bottle should be marked with a red line approximately $\frac{1}{4}$ of the height of the bottle from the bottom. When the level of the ICRM™ sample reaches the red line, a new bottle of control sample should be tested as an unknown to verify the new control sample meets quality specifications stated on the control sample certificate. Prior to taking a test portion for analysis, all samples should be mixed by rotating the bottle end over end for not less than 1 minute. According to the law of propagation of errors the **total of major and minor oxides** listed in the table above should fall **between 96.00 % and 99.30 %** based on the **2 standard deviation (2S)** test limits.