

Coal Certified Reference Material, Proximate Plus (Prox⁺)

Product No: AR1757

Lot No: 250506

Material and Intended Use

AR1757 is a coal certified reference material (CRM). The intended use of this CRM is for the verification and calibration of the methods of analysis below for the determination of the corresponding listed property values. This CRM can also be used to validate value assignment of in-house reference materials. A unit consists of one bottle containing 50g of powder. All reference materials should be verified as fit for purpose prior to use.

Reported Values

Certified values for AR1757 are given in Table 1. Certified values are reported as $x \pm U_{95\%}$, where $U_{95\%}$ is half the 95% coverage interval around the certified value. The expanded uncertainty is $U_{95\%} = k u_c$, with the combined standard uncertainty, u_c , multiplied by the expansion factor $k = 2$. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with approximately 95% confidence. The estimation of combined standard uncertainty (u_c) includes contributions from material heterogeneity, calibration, measurement, and other factors (1-2). Sampling and calculation of reported values for each measurand were performed using practices consistent with ISO 17034:2016 (3) and ISO 33405:2024 (4).

Table 1. Certified values for AR1757, Lot 250506.

| Property | Value | $U_{95\%}$ |
|--|-----------------|--------------|
| % Ash | 48.91 | 0.42 |
| % Volatile Matter | 12.61 | 0.48 |
| % Fixed Carbon | 38.43 | 0.65 |
| % Carbon | 39.83 | 0.63 |
| % Hydrogen | 1.82 | 0.15 |
| % Nitrogen | 0.40 | 0.07 |
| % Sulfur | 6.20 | 0.22 |
| % Chlorine | 0.0283 | 0.0053 |
| % Fluorine | 0.0201 | 0.0039 |
| Gross Calorific Value, BTU/lb (J/g) | 6217 (14461) | 349 (812) |

% Ash – Values indicate the amount of non-combustible matter in the material matrix as determined by combustion and gravimetry and are metrologically traceable to the International System of Units (SI) derived unit of mass fraction expressed as percent (%).

% Volatile Matter – Values indicate the amount of matter in the material matrix that is volatilized and released as gaseous substance, excluding moisture, when the material matrix is heated in the absence of air at 950°C +/- 20°C for 7 minutes. Values are metrologically traceable to the International System of Units (SI) derived unit of mass fraction expressed as percent (%).

% Fixed Carbon – Values indicate the amount of solid, combustible matter in the material matrix remaining after accounting for moisture, volatile matter, and ash. Values are metrologically traceable to the International System of Units (SI) derived unit of mass fraction expressed as percent (%).

% Carbon, Hydrogen, Nitrogen, Sulfur, Chlorine, Fluorine – Values indicate the amount of the element in the material matrix as determined using combustion and are metrologically traceable to the International System of Units (SI) derived unit of mass fraction expressed as percent (%).

Gross Calorific Value – Values indicate the heat produced by complete combustion of the material matrix at constant volume accounting for the condensation of all water formed during combustion. Values are metrologically traceable to the International System of Units (SI) derived unit of J/g and the Imperial Unit of BTU/lb.

Instructions for Use

This reference material should be dried to constant mass at 105°C before use. Bottles of powder should be kept sealed tight and stored in a cool, dry location.

For propagation of uncertainty, an estimate of the combined standard uncertainty can be obtained as $u_c = U_{95\%}(x)/k$, where $k = 2$ is the approximate coverage factor associated with the 95 % coverage level. The resulting value for u_c is at the level of one standard deviation, and it can be combined with a laboratory's standard uncertainty estimates for their own sources of error to calculate estimates of uncertainty for test results from methods with which this CRM was used. A laboratory uncertainty estimate that includes the uncertainty of the CRM value is the basis for a link of metrological traceability from the test result for a sample to the CRM value.

Minimum Sample Size

It is recommended that the following minimum sample specimen size be used for each given test method:

| Test Method | Minimum Specimen |
|---|------------------|
| Combustion/Gravimetry – % Ash | 1.0 g |
| Gravimetry – % Volatile Matter | 1.0 g |
| Combustion – % Carbon, Hydrogen, Nitrogen, Sulfur | 0.10 g |
| Combustion – % Chlorine | 0.01 g |
| Combustion – % Fluorine | 0.05 g |
| Combustion – Gross Calorific Value | 0.8 g |

Period of Validity

This certification is valid for 15 years from the initial certification date, within the measurement uncertainties specified, provided the CRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). Accordingly, periodic recalibration or recertification of this CRM is not required. The certification is nullified if the CRM is damaged, contaminated, or otherwise modified.

Homogeneity

This product was manufactured using pulverizing and blending to minimize heterogeneity. Samples were randomly selected using practices consistent with ISO 33405:2024. Homogeneity was evaluated by replicate analysis. Within- and between-sample variance was evaluated using Analysis of Variance (ANOVA).

Maintenance of the Reference Material Certificate

Alpha Resources will monitor this CRM throughout the period of its availability. If substantive technical changes occur that affect the value assignment, AR will notify the purchaser.

Users of this CRM should ensure the Reference Material Certificate in their possession is current. This can be accomplished by contacting Alpha Resources at the following: Telephone - (269) 465-5559; Email - info@alpharesources.com; or via the Internet - https://www.alpharesources.com.

Methods and References

- (1) JCGM 100:2008; Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed February 2025)
- (2) JCGM 101:2008; Evaluation of Measurement Data – Supplement 1 to the Guide for the Expression of Uncertainty in Measurement; Propagation Distributions Using a Monte Carlo Method; Joint Committee for guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008.pdf (accessed February 2025).
- (3) ISO 17034:2016 – General requirements for the competence of reference material producers.
- (4) ISO33405:2024 – Reference materials – Approaches for characterization and assessment of homogeneity and stability.



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