

Certified Reference Material

Material Characterization Documentation

Gold

High Temperature Calibration Standard Temperature and Enthalpy of Fusion

The Certified Reference Material provided with this documentation is certified by Engineering Analytics Laboratories, USA for use as a standard calibration material for thermal properties testing analyzers such as Differential Thermal Analyzer (DTA), Simultaneous Differential Thermogravimetric (SDT) Analyzers, Simultaneous Thermal Analyzer (STA), or similarly designed analyzers in accordance with ASTM E-Series documents, or similar standard, in which a temperature sensing device does not directly interact with the test sample. This Certified Reference Material is vacuum sealed in a Mylar bag along with a desiccant package.

The following documentation outlines the material properties, property determination techniques, storage, and expiration.

Material:	Gold	EA Labs Lot #:	20CRMAU01
CAS #:	7440-57-5	T _{melt} :	1,063.13 ± 0.64 °C
Purity:	99.999+%	ΔH:	68.60 ± 14.81 J/g
Form:	Wire	Stability:	Single Use Only

Certificate Issuer:

Engineering Analytics Laboratories (EA Labs)

Accredited to ISO 17034: 2016 RMP for DSC/TGA/DMA #122465 through PJLA, Inc.

Approving Officers:

Kenneth E. Bukowski

M.S. Chemical Engineer, Florida Institute of Technology

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Certificate Issue Date: _____

App. Officer

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1. Certified Temperature of Melting Point

Temperature: $1,063.13 \pm 0.64$ °C

Based on 1st Run Testing

a. Testing Apparatus

Certified for use in the calibration of Differential Thermal Analyzer (DTA), Simultaneous Differential Thermogravimetric (SDT) analyzers, Simultaneous Thermal Analyzer (STA), or similarly designed analyzers in accordance with ASTM E-Series documents, or similar standard, in which a temperature sensing device does not directly interact with the test sample.

Verification testing of the Gold supplied with this Certificate has been tested utilizing the following test equipment:

- i. PerkinElmer DTA7

Equipment was tested and verified for accuracy prior to testing Gold samples of varying masses and lengths shapes. See Testing Regimen for the test process (Section 1.b) and Results Tables (Section 1.c) for the measured temperature resulting in the above temperature and uncertainty.

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b. Test Regimen

Testing via a PerkinElmer DTA7 was used for Certification purposes. Testing was conducted on multiple samples off the full Lot 20CRMAU01 (see Section 5 for additional information) with each sample ranging in mass from low to high to simulate possible in-situ use. Samples were heated at 10 °C/min to 10 °C above the melting point, and cooled to 200 °C below the freezing point for cyclical testing. Samples were tested in 3 different physical states A) submerged in Alumina Oxide Powder, B) seated on a bed of Alumina Oxide Powder, and direct contact with the high-density Alumina Oxide Crucible. Due to the extreme temperature and limitation inherent in the DTA, samples of Gold with defined characteristics were used for comparison for accuracy determination. Samples showed excellent accuracy and precision of the Melting Temperature reported herein.

In-Situ testing was additionally utilized to ensure proper use and outcome for this Certified Reference Material as the equipment for expected use, per ASTM Precision and Bias data, which states an expected use uncertainty is ± 0.41 °C for a Single User and ± 0.48 °C for Intra-laboratory testing. Values are compared to known tests from National Institutes and Literature Sources with Novel Testing.

Varying masses of Gold were tested in the above listed apparatus which were calibrated with Aluminum and Gold and cross compared with OEM sourced Gold. Samples were both submerged in and set on a bed of alumina oxide powder to be melted three times in succession in an air environment. The on-set temperature of the melt is measured and reported. Due to the high value price of Gold, 3rd Party Laboratories were not utilized for this verification and testing process. See Results Tables in Section 1.c.

c. Results Tables

The following results were determined for Gold, Lot 20CRMAU01

Table 1: Compiled Melting Temperature Data

Metric	Value
Melting Point Average	1,063.13 °C
Uncertainty (k=2)	0.64 °C
Standard Deviation (1 σ)	0.59 °C

Comparative Results:

Ruspy et. al Research ⁱ T _{melt} :	± 0.5 °C
Preston-Thomas Research ⁱⁱ T _{melt} :	1,064.18 °C
PerkinElmer Assigned ^{iv} :	1,063.0 °C
ASTM E967.18 ^v Uncertainty:	± 0.48 °C

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Uncertainty values are calculated using the root of the sum of squares (RSS) method for Type A and/or B uncertainty calculations. The $k=2$ value represents 95% confidence that the value determined through testing on the devices listed in this document will achieve this range of values. Calculated values also include the inherent uncertainty value of the equipment used for testing. This uncertainty value represents a confidence in the average value of the melting point of Gold, and does not represent the Standard Deviation or Range of potential values possible across users, equipments, and cross-sectional area of the CRM.

2. Certified Enthalpy of Fusion

Enthalpy of Fusion (ΔH): 68.60 ± 14.81 J/g

Based on 1st Run Testing

a. Testing Apparatus

Certified for use in the calibration of Differential Thermal Analyzer (DTA), Simultaneous Differential Thermogravimetric (SDT) analyzers, Simultaneous Thermal Analyzer (STA), or similarly designed analyzers in accordance with ASTM E-Series documents, or similar standard, in which a temperature sensing device does not directly interact with the test sample.

Verification testing of the Gold supplied with this Certificate has been tested utilizing the following test equipment:

- i. PerkinElmer DTA7

Equipment was tested and verified for accuracy prior to testing Gold samples of varying masses and lengths. See Testing Regimen for the test process (Section 2.b) and Results Tables (Section 2.c) for the measured temperature resulting in the above temperature and uncertainty.

b. Test Regimen

Testing via a PerkinElmer DTA7 was used for Certification purposes. Testing was conducted on multiple samples off the full Lot 20CRMAU01 (see Section 5 for additional information) with each sample ranging in mass from low to high to simulate possible in-situ use. Samples were heated at 10 °C/min to 10 °C above the melting point, and cooled to 200 °C below the freezing point for cyclical testing. Samples were tested in 3 different physical states A) submerged in Alumina Oxide Powder, B) seated on a bed of Alumina Oxide Powder, and direct contact with the high-density Alumina Oxide Crucible. Due to the extreme temperature and limitation inherent in the DTA, samples of Gold with defined characteristics were used for comparison for accuracy determination. Samples of both the control Gold and EA Labs CRM Gold showed a high range in the Enthalpy of Fusion due to the gold fusing into the Alumina Oxide Powder while forming into a molten ball during the melt process.

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In-Situ testing was utilized to ensure proper use and outcome for this Certified Reference Material as the equipment for expected use, per ASTM Precision and Bias data, which states an expected use uncertainty is 4.2% J/g for a Single User and 8.2% J/g for Intra-laboratory testing. Values are compared to known tests from National Institutes and Literature Sources with Novel Testing.

Varying masses of Gold were tested in the above listed apparatus which were calibrated with Aluminum and Gold and cross compared with OEM sourced Gold. Samples were both submerged in and set on a bed of alumina oxide powder to be melted three times in succession in a air environment. The on-set temperature of the melt is measured and reported. Due to the high value price of Gold, 3rd Party Laboratories were not utilized for this verification and testing process. See Results Tables in Section 2.c.

c. Results Tables

The following results were determined for Gold, Lot 20CRMAU01

Table 2: Compiled ΔH Data

Metric	Value
ΔH Average	68.60 J/g
Uncertainty ($k=2$)	14.81 J/g
Standard Deviation (1 σ)	6.11 J/g

Comparative Results:

Ho, Ervin, <i>et. al.</i> Research ⁱⁱⁱ T_{melt} :	64.59 J/g
PerkinElmer Assigned ^{iv} :	63.7 J/g
ASTM E968 ^{vi} Uncertainty:	$\pm 8.2\%$ of J/g

Uncertainty values are calculated using the root of the sum of squares (RSS) method for Type A and/or B uncertainty calculations. The $k=2$ value represents 95% confidence that the value determined through testing on the devices listed in this document will achieve this range of values. Calculated values also include the inherent uncertainty value of the equipment used for testing. This uncertainty value represents a confidence in the average value of the Enthalpy of Fusion for Gold, and does not represent the Standard Deviation or Range of potential values possible across users, equipments, and cross-sectional area of the CRM.

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3. Typical DTA Melting Point Graph

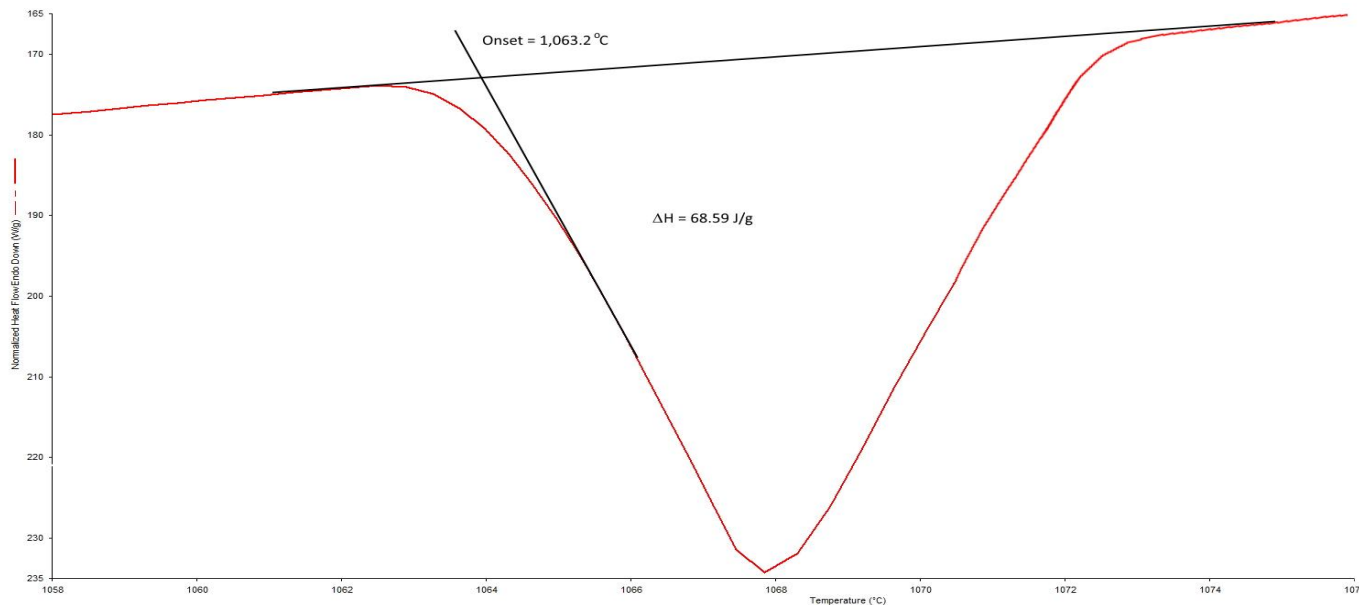


Figure 1: DTA Melting Point Graph of Gold, Lot 20CRMAU01

4. Expiration of Material

This material and material certificate expires 3 years from the manufacturing date printed on the signed Certificate of Analysis accompanying the material provided that the material is maintained in a desiccant (low humidity) box while being maintained in the issued glass scintillating vial. Storage in a reactive environment such as an oxygen or high humidity environment voids the signed / issued Certificate of Analysis. A clean working space and storage environment is required to ensure the maintenance of the purity of this material. Additional storage options are provided in Section 7.

5. Traceability

Original raw material has been purchased from ESPI Metals Lot Q18890A in May 2020. The following information is available for the 5N (99.999+%) purity of this material as purchased:

Table 3: Gold CRM Purity

Element	As Reported
Gold	99.999+%

Additional GD-MS testing was not conducted on the material due to the high cost nature of the material. Original test reporting by Vale, United Kingdom, is considered accurate and of high quality. Additional testing was not required.

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This Product is Made in the USA from processed materials imported from England and/or Germany. EA Labs has verified the source for raw material, processed materials, and final product generation. Manufacturing of the final product utilizing international materials is conducted in Florida, USA. The percentage of sale price remaining within the USA is ~98% with the remaining 2% paid to the US division of a non-USA, international company such that “all or virtually all” of this material and manufacturing process complies with FTC standards for a “Made in America” product.

6. Materials Handling

This CRM must be handled with powder free gloves to prevent any possible contamination which might affect the melting temperature of the material. Weighing of Gold samples must be conducted on single-use contaminant free surfaces to avoid cross-contaminations.

See the provided MSDS for additional details. In the form provided, this material is not toxic or hazardous to human health or the environment. In general, Gold can cause skin irritations and discolorations.

7. How to Use

For best use results:

- 7.1. Pre-heat the DTA, SDT, or STA to 700 °C or above and back to R.T. to pre-condition the unit prior to use.
- 7.2. Use 2 to 4 mg of EA Labs CRM Gold. Do NOT use a long section of Gold for testing as greater mass distance from center affects the values. Gold in this form will become a molten ball upon melting.
- 7.3. Do NOT cycle the CRM Gold sample with Alumina Oxide Powder through the Melt/Freeze phase prior to use for calibration as the powder will adhere to the metal and alter future cycle results.
- 7.4. Do NOT cycle the CRM Gold sample with Alumina Oxide Low Density Cups through the Melt/Freeze phase prior to use for calibration as the metal will fuse into the cup and alter future cycle results.
- 7.5. Analyze the resulting melt peak using the method prescribed in Section 8.
- 7.6. Run a Validation of your calibration post calibration activities using an alternate CRM Gold sample.
- 7.7. Suggested Temperature Range: 900 – 1,150 °C, post calibration.

In general, the valid calibrated use range for thermal analysis equipment due to limitations of single-point calibration is ± 150 °C. Beyond this range, temperature values of tested materials will be in error of 3+ °C, for testing of samples beyond this range, users are recommended to use an additional CRM for 2 or 3 point Temperature calibration.

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Clean all tools and work areas properly prior to removing the Certified Reference Material from storage to avoid any and all environmental contaminations. Due to high melting point of Gold, fusing and stage material can interfere with the melt or cause deviations when used successively.

Refer to the OEM calibration process steps to ensure the unit is set to factory default calibration table prior to the start of the calibration process. Ensure that the test method for calibration follows the expected use of the equipment, post calibration. Results are only valid for the Temperature Ramp Rate, Gas Type, Gas Flow Rate, and pan type / style used during the calibration process.

8. How to Analyze

Various OEM software will auto-analyze for the user. If auto-analysis is not available, the proper method is to graph the derivative of the curve over the Normalized Heat Flow graph, and measure the Peak Area with Point / Limit #1 placed on the Normalized Heat Flow graph matching to the onset of the derivate change in slope prior to the start of the melting peak. Point / Limit #2 should be placed on the Normalized Heat Flow graph matching with the point on the derivative cure where the curve levels off post melting peak. Utilizing this method will yield consistent results as provided in this documentation.

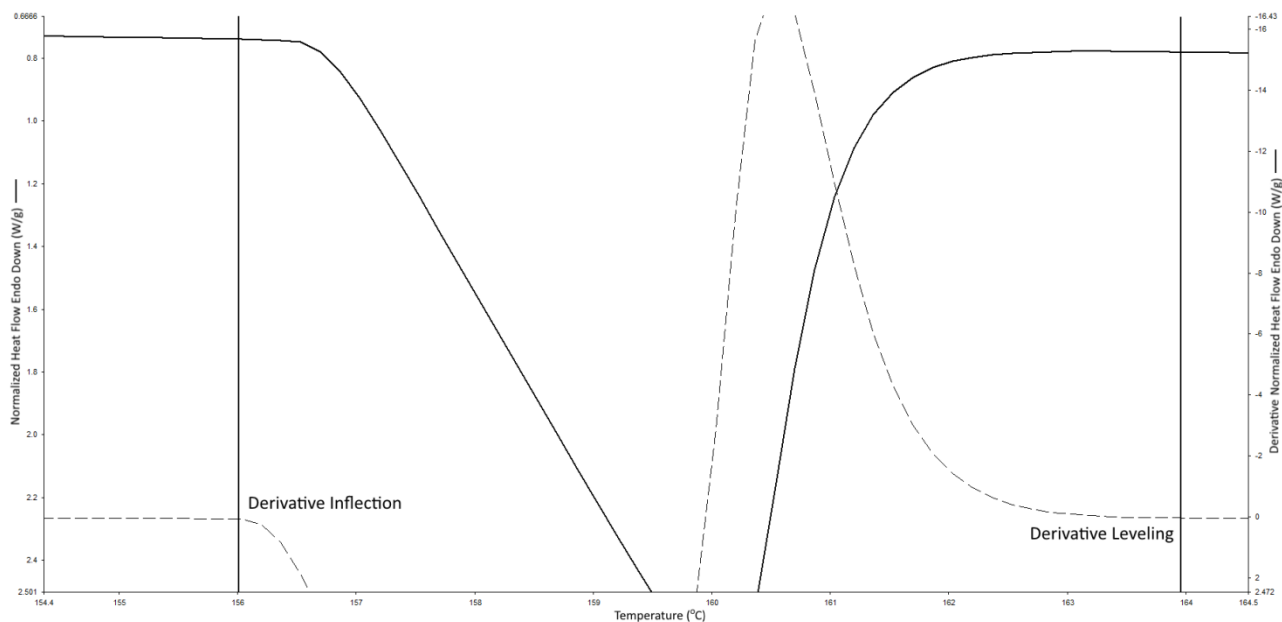


Figure 2: Analysis – DSC / DTA Melting Point

9. How to Store

This material is stable in general environments, and is shipped in a vacuum sealed Mylar bag. Store and maintain in accordance with industry standards for a clean and contaminate free working and storage environment.

Minimal Storage Conditions: Desiccant Box while sealed inside the original glass scintillation vial.

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Optimal Storage Conditions: Vacuum sealed container or under inert gas such as Nitrogen or Argon

10. Maintenance of Certification Documentation

This certificate expires with the expiration of the material and if the material is improperly stored. Engineering Analytics Laboratories is the sole authority to create, maintain, distribute, and re-issue certification documentation for EA Labs produced CRMs and RMs. Certification documentation is updated and maintained per EA Lab's SOP in accordance with applicable certifying agencies.

Document Identification: 5113.13.1 Gold CRM Rev. 1.0 06_2025

11. EA Labs Description

Engineering Analytics Laboratories is a USA based company currently accredited to ISO 17034: 2016 RMP for DSC/TGA/DMA and ISO/ICE 17025: 2017 DSC, TGA, and DMA Testing, #122465 through PJLA, Inc. EA Labs has 18+ years of experience testing the thermal properties of materials.

Mailing Address:

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Please direct any technical or scientific questions to KenBukowski@EngineeringAnalytics.us

Warranty claims against this manufacturer will cover only the material accompanying this paperwork and will require photographic proof of the storage environment, data results, and the return of the original material to EA Labs for analysis.

All material documentation and testing: Engineering Analytics Laboratories © 2025

12. References

1. Rusby, R.P. Hudson, M. Durieux, J.F. Schooley, P.P.M. Steur and C.A. Swenson, Metrologia, 28 (1991) 9
2. H. Preston-Thomas, The International Temperature Scale of 1990 (ITS-90), Metrologia, 27 (1990) 3.
3. David R. Lide, ed., CRC Handbook of Chemistry and Physics, International Version 2005 pg. 67
4. PerkinElmer – Gold Reference Material, as sold
5. ASTM E967.18, Section 12. Precision and Bias, 2017
6. ASTM E968.28, Section 13. Precision and Bias, 2014