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CERTIFICATE OF ANALYSIS FOR CERTIFIED REFERENCE MATERIAL

AC18.13566

Description: Spent Automotive Catalyst (Monolith).

The material consists of the cordierite ceramic honeycomb monolith structure found inside automotive catalytic converters. This structure supports the washcoat (gamma-Al_2O_3, or alumina) and the catalytic materials, including platinum, palladium, and rhodium.

AC18.13566 is available as 70 g units packed into glass, wide-mouth jars.

Intended use: For evaluating total quantitative analytical methods for the chemical analysis of spent automotive catalysts.

Certified and indicative values derived from analytical methods of analysis are provided in Tables 1 and 2, respectively. Table 3 provides some indicative physical properties and Table 4 provides indicative mineralogy by semi-quantitative XRD analysis.

Approving officer: Management of the interlaboratory certification program by Craig Hamlyn (Technical Manager, OREAS).

Minimum sample size: To relate analytical determinations to the values in this certificate, a minimum dry sample mass of 1 g should be used for the analysis of Pt, Pd and Rh. For all other analytes, a minimum dry sample mass of 0.2 g can be used. Use of lower masses than those recommended may lead to higher variability.

Storage and period of validity: The certification of AC18.13566 remains valid, within the specified measurement uncertainties, until November 2039, provided the CRM is stored in a clean and cool dry place away from direct sunlight. This certification is nullified if the CRM is any way changed or contaminated.

Maintenance of Certified Values: OREAS will monitor this CRM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, OREAS will notify the purchaser (using the contact's email address on the Sales Order).

Table 1. Certified Values and their associated 95 % Expanded Uncertainty.

Constituent (wt.%)	Certified Value	95 % Expanded Uncertainty
Al	18.97	0.34
Ba	0.7852	0.0063
Ca	0.358	0.048
Ce	3.12	0.13
Cr	0.1312	0.0041
Fe	1.592	0.034
La	0.521	0.022
Mg	5.07	0.13
Nd	0.356	0.026
Ni	0.358	0.014
P	0.352	0.011
Pr	0.1492	0.0079
S	0.1813	0.0076
Si	17.59	0.64
Sr	0.188	0.017
Zn	0.197	0.034
Zr	3.42	0.19

Constituent (ppm)	Certified Value	95 % Expanded Uncertainty
Eu	0.562	0.060
Ho	0.2489	0.0080
Lu	0.109	0.011
Pd	**2034.6**	**8.8**
Pt	**512**	**13**
Rh	**238.9**	**2.1**
Sm	15.4	1.3
Th	6.18	0.39
Tm	0.187	0.020
V	59.5	7.7
W	38.0	3.0
Y	584	33
Yb	1.07	0.11

SI unit equivalents: ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95 % Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 2. Indicative Values for AC18.13566.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Ag	ppm	*3.88*	Gd	ppm	*101*	Rb	ppm	*15.8*
As	ppm	*48.7*	Ge	ppm	*0.79*	Re	ppm	*0.038*
Au	ppm	*< 3*	Hf	ppm	*833*	Ru	ppm	*0.055*
B	ppm	*90*	Hg	ppm	*0.20*	Sb	ppm	*4.46*
Be	ppm	*< 5*	In	ppm	*0.10*	Sc	ppm	*75*
Bi	ppm	*1.10*	Ir	ppm	*1.95*	Se	ppm	*4.98*
C	wt.%	*1.75*	K	wt.%	*0.280*	Sn	ppm	*7.16*
Cd	ppm	*9.23*	Li	ppm	*11.0*	Ta	ppm	*0.76*
Co	ppm	*19.3*	LOI^{1000}	wt.%	*1.76*	Tb	ppm	*1.60*
Cs	ppm	*7.20*	Mn	wt.%	*0.028*	Te	ppm	*< 1*
Cu	ppm	*235*	Mo	ppm	*128*	Ti	wt.%	*0.428*
Dy	ppm	*1.10*	Na	wt.%	*0.163*	Tl	ppm	*0.50*
Er	ppm	*0.69*	Nb	ppm	*14.4*	U	ppm	*1.29*
Ga	ppm	*21.9*	Pb	ppm	*86*			

SI unit equivalents: ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction). Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of OREAS' in-house CRM-specific LIMS.

Table 3. Physical properties of AC18.13566.

Bulk Density (kg/m³)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
805	0.58	N6	Medium Light Gray

‡The Munsell Rock Colour Chart helps geologists and archaeologists communicate with colour more effectively by cross-referencing ISCC-NBS colour names with unique Munsell alpha-numeric colour notations for rock colour samples.

Table 4. indicative mineralogy by semi-quantitative XRD analysis for AC18.13566.

Constituent	% (mass ratio)
Annite - biotite - phlogopite	4
Fe-Mg amphibole	1
Cordierite group	80
Sillimanite group	2
Moissanite	4
Quartz	3
Nahcolite and/or gotzenite	6

The results have been normalised to 100 %, and the values shown represent the relative proportion of the crystalline material in the sample. Totals greater or less than 100 % are due to rounding errors. Some amorphous material and monazite may be present in the samples. Nahcolite and/or Gotzenite" was reported based on poorly crystalline and overlapping patterns; both the identification and quantification carry significant uncertainty. "Steel and/or metallic iron" was reported based on a single peak, and the results carry high uncertainty. Minor amounts of "steel and/or metallic iron" may also be present.

Commutability: AC18.13566 is derived from spent automotive catalysts and is expected to behave similarly to routine 'process plant' samples in the relevant measurement process. Commutability should not present an issue for this CRM.

Instructions for handling, correct use and safety: Fine powders pose a risk to eyes and lungs. The use of safety glasses and dust masks are advised. Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [11]. After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. AC18.13566 is considered stable and contains a non-hygroscopic matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 3 in this certificate.

Certified values with their corresponding 95% expanded uncertainties are reported on a dry sample basis, determined in accordance with ISO/IEC Guide 98-3:2008 [4,14], and presented in Table 1. These values are metrologically traceable to the international measurement scale (SI) of mass with major elements expressed in % (mass ratio) and minor elements expressed in mg/kg. In line with popular use, data are expressed as the mass fraction in either weight percent (wt.%) or parts per million, 1 x 10^{-6} (ppm). They are the means of accepted laboratory means after outlier filtering and are the present best estimate of the true value.

Indicative values shown in Table 2 are metrologically traceable to the international measurement scale (SI) of mass and are expressed in % (mass ratio) or mg/kg. In line with popular use, data are expressed as the mass fraction in either weight percent (wt.%) or parts per million, 1 x 10^{-6} (ppm). Indicative values are present where interlaboratory consensus is insufficient to meet OREAS' criteria for certification. AC18.13566 was also tested by OREAS for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

Sample Preparation and Analysis: The spent automotive catalysts comprising AC18.13566 were derived from a blend of monolith material that was crushed and pulverised to a fine powder. The material was dried to constant mass, then subjected to multi-stage milling to achieve 100% passing 30 µm. Homogenisation was carried out using OREAS' proprietary processing technologies, and the final product was packaged into 70 g units in glass jars sealed with plastic lids.

For the round robin program, twelve 1.5 kg test units were systematically sampled at predetermined intervals during packaging and are considered representative of the entire prepared batch. Twenty-one commercial analytical laboratories participated in the program to certify the elements reported in Table 1. Each laboratory received a sample of the candidate reference material and was instructed to report results in triplicate. A control sample was also included to facilitate data evaluation and metrological traceability. Results generated by these quantitative analytical methods were pooled for certification purposes.

For the key commodities (palladium, platinum and rhodium), most data were obtained from trade/speciality laboratories using sodium peroxide fusion, lead collection fire assay, or nickel sulphide collection fire assay, followed by ICP-OES or AAS finish. One laboratory employed pressed powder pellet with XRF finish.

For the other analytes, a range of analytical methods were applied for characterisation, including various acid digestions and fusions, followed by ICP-OES, ICP-MS and AAS finishes.

The homogeneity of AC18.13566 was assessed using a nested Analysis of Variance (ANOVA) study. Twelve 10 g pulp samples were sent to the ANSTO laboratory for Ce, Cr, Hf, La, Pd and Pt analysis by INAA. These consisted of paired samples from the odd sampling intervals (representative of the prepared batch) that were randomised prior to assigning sample numbers. Duplicate samples enabled ANOVA comparison of within- and between-unit variances across the six pairs. The objective was to test whether variance between units was statistically different from variance within units, thereby assessing homogeneity of the batch.

The test parameters were:

- Null Hypothesis, H_0: Between-unit variance is no greater than within-unit variance (reject H_0 if p-value < 0.05);
- Alternative Hypothesis, H_1: Between-unit variance is greater than within-unit variance.

No significant p-values were observed across the six reported elements. The Null Hypothesis was therefore accepted, with no evidence of heterogeneity detected.

Document history:

Revision No.	Date	Changes applied
2	7th January, 2026	Removal of accreditation mark to reflect certification activities conducted outside the scope of accreditation.
1	30th October, 2025	Adding Table 4 indicative mineralogy by semi-quantitative XRD analysis
0	4th September, 2025	First publication.

References

[1] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.

[2] ISO 33401:2024-01. Reference materials – Contents of certificates, labels and accompanying documentation.

[3] ISO 33405:2024-05. Reference materials – Approaches for characterization and assessment of homogeneity and stability.

[4] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

[5] ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.
[6] ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.
[7] ISO 17025:2017, General requirements for the competence of testing and calibration laboratories.
[8] ISO 17034:2016. General requirements for the competence of reference material producers.
[9] Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), Minnesota (USA).
[10] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.
[11] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.
[12] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.
[13] Thompson, A.; Taylor, B.N. (2008); Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed 22 November 2024).
[14] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.

Participating laboratories

1. AH Knight, St Helens, Merseyside, UK
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3. ALS Inspection, Prescot, Merseyside, UK
4. American Assay Laboratories, Sparks, Nevada, USA
5. ANSTO, Lucas Heights, NSW, Australia
6. Heraeus Precious Metals GmbH & Co. KG, Hanau, Hesse, Germany
7. Inspectorate (BV), Shanghai, Bao Shan District, China
8. Inspectorate (BV), Witham, Essex, UK
9. Intertek, Cupang, Muntinlupa, Philippines
10. Intertek, Perth, WA, Australia
11. Intertek LSI, Rotterdam, Zuid-Holland, Netherlands
12. Ledoux & Company, Teaneck, New Jersey, USA
13. Lukasiewicz Research Network, Gliwice, Upper Silesia, Poland
14. MINTEK Analytical Services, Randburg, South Africa
15. MSALABS, Vancouver, BC, Canada
16. NSL Analytical Services, Cleveland, OH, USA
17. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
18. Rigaku Corporation, Osaka, Kansai region, Japan
19. Scrooby's Laboratory Service Pty Ltd, Benoni, Gauteng, South Africa
20. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
21. UIS Analytical Services, Centurion, South Africa