National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2556

Used Auto Catalyst
(Pellets)

(In Cooperation with the International Precious Metals Institute)

This Standard Reference Material (SRM) is intended for use in evaluating chemical and instrumental methods for the analysis of platinum group metals and lead. It is a recycled pellet auto catalyst in the form of fine powder (less than 74 μm (-200 mesh)). This SRM is issued in 70 g units.

Certified Values

Element	Concentration, mg/kg			Method
Pt	697.4	±	2.3	ID-ICPMS
Pd	326.0	±	1.6	ID-ICPMS
Rh	51.2	±	0.5	ICP-MS
Pb	6228	±	49	ID-ICPMS

The certified values for platinum, palladium, rhodium, and lead were determined on samples that were calcined for 2 h at 500 °C prior to analysis, and are based on results obtained using inductively coupled plasma mass spectrometry with isotope dilution quantitation for Pt, Pd, Pb, and internal standard quantitation for Rh. The stated uncertainties are 99% confidence intervals of the single method means.

NOTICE TO USERS

Stability: This material is considered to be stable, however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Please return the registration card to facilitate notification.

Use: SRM 2556 is hygroscopic, and contains some organic matter absorbed from automotive fuel. To assure a stable weighing form for analysis, the sample must be ignited for 2 h at 500 °C, and then stored in a desiccator until the analytical sample is weighed out. Homogeneity assessments indicate that analyses performed on samples of
0.1 g may be related to the certified value; samples of 5 g or larger are more typical for platinum group metal analyses.

The overall direction and coordination of the analyses for certification were under the chairmanship of P.J. Paulsen and J. Fassett of the NIST Inorganic Analytical Research Division and of J. Bozic of INCO Ltd, Copper Cliff, Ontario. The mass spectrometric analyses were performed by E.S. Beary and P.J. Paulsen of the NIST Inorganic Analytical Research Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.S. Kane.

Gaithersburg, MD 20899
August 24, 1993
(Revision of certificate dated 7-9-93)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

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Source and Preparation of Material: INCO Ltd, Copper Cliff, Ontario, CANADA collected the material for this SRM from used pellet type auto catalysts by reserving 2.3 kg (5 lb) samples from each of several batches of auto catalyst as it was being recycled. Each 2.3 kg (5 lb) sample was individually pulverized to less than 74 µm (-200 mesh) and blended, and several samples were pooled until more than 136 kg (300 lb) had been collected. The material was then shipped to NIST, where it was sieve-tested, blended, and bottled in 70 g units. Ninety-six randomly selected samples were withdrawn from the lot for homogeneity testing and certification analysis.

Analysis: Homogeneity testing for the certified platinum group elements and lead was performed by high-precision X-ray fluorescence analysis of 3.5 g and 6 g samples, and by inductively coupled plasma analysis preceded by fire assay of 6 g samples. No statistically significant material variability was observed for the elements evaluated. Certification analyses performed at NIST on 0.1 g samples also showed no evidence of sample heterogeneity.

Certified Values and Uncertainties: The certified values are based exclusively on isotope dilution mass spectrometry (Pt, Pd, Pb) or inductively coupled plasma mass spectrometry (Rh). Confirmatory analyses were performed by instrumental neutron activation at NIST and by X-ray fluorescence, inductively coupled plasma atomic emission and flame atomic absorption by cooperating laboratories.

Non-certified Values: The non-certified values, shown below in parentheses, are given to provide additional information on the overall matrix composition of the SRM. The values are non-certified because there was not intermethod agreement or because they were determined by only one analytical method.

Element	Concentration, Wt %*	Element	Concentration, mg/kg
Al	(40)	Ba	(100)
Ca	(0.1)	Zn	(600)
Ce	(1)	Zr	(300)
Fe	(0.8)		
La	(0.7)		
Si	(0.2)		

*Wt % = mg/kg x 10^{-4}

Methods/Techniques

Element	Methods
Pt	ID-ICPMS, INAA, ICP-AES, XRF
Pd	ID-ICPMS, INAA, ICP-AES, XRF
Rh	ICP-MS, ICP-AES, XRF
Pb	ID-ICPMS, ICP-AES, XRF
Al	XRF, INAA, ICP-MS
Ba	XRF, ICP-MS
Ca	XRF, ICP-MS
Ce	XRF, INAA, ICP-MS
Fe	XRF, INAA, ICP-MS
La	XRF, INAA, ICP-MS
Si	XRF, ICP-MS
Zn	XRF, INAA, ICP-MS
Zr	XRF

ID-ICPMS - Isotope dilution inductively coupled plasma mass spectrometry, Carius tube decomposition.
INAA - Instrumental neutron activation analysis.
ICP-MS - Inductively coupled plasma mass spectrometry, Carius tube decomposition (Rh), mixed acid digestion (all other elements).
XRF - Wavelength dispersive X-ray fluorescence.
ICP-AES - Inductively coupled plasma atomic emission spectrometry, mixed acid digestion or fire assay.

Contributing Laboratories:

NIST, Inorganic Analytical Research Division, E.S. Beary, D.A. Becker, K. Garrity, A.F. Marlow, J.A. Moody, P.J. Paulsen, P.A. Pella, R.L. Watters, Jr., and L.J. Wood.

INCO Ltd., Ontario Division, Copper Cliff, Ontario; J. Bozic, A. Glaab, and S. Maggs.

Ford Motor Co., Dearborn, MI; F.W. Kunz.

Engelhard Corp., Huntsville, AL; L. Edwards and L. Tannehill.

Johnson-Matthey, Wayne, PA; L.E. Brooks and G. Smith.

Johnson-Matthey, West Deptford, NJ; T.M. Piecara.

Accredited Laboratories Inc., Carteret, NJ; J. Anselmo and R. Hagen.

Handy and Harman, Fairfield, CT; J.B. Whitney and M.A. Worthington.

Addendum to the Certificates

for

SRM 2556, Used Auto Catalyst (Pellets)
SRM 2557, Used Auto Catalyst (Monolith)

NIST Standard Reference Materials (SRMs) are used extensively for the evaluation of analytical methods and the assessment of laboratory performance. NIST SRMs are certified at a relatively high degree of accuracy and the uncertainties of the certified values are small with respect to the intended use. This allows one to test for analytical measurement bias, to determine analytical measurement acceptability, and to establish statistical control of the analytical measurement process.

This addendum is to provide the users of these two automobile catalyst SRMs with information that can be used to compare NIST certified values with mean values obtained by the laboratories that participated in the round robin exercises. The laboratories are identified by number and their results reflect variabilities that can be expected among and between laboratories. In most cases, the laboratories' results are means of four to eight replicates reported without any assessment of systematic error. The overall interlaboratory means are reported along with the average standard deviation at one sigma (σ).

NIST certified values should be used for equipment calibration and/or method evaluation. The certified values are the "best" estimate of the true value, while the round robin results provide the user with some measure of the reproducibility that can be expected using more traditional techniques or methods.

Gaithersburg, MD 20899
August 24, 1993

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

Contributing Laboratory Data for SRM 2556 in mg/kg

Lab	Pt[a]	Pd[a]	Rh[b]	Pb[b]
1	768 ± 13**	402 ± 11**	52 ± 4	6188 ± 161
2	663 ± 22	304 ± 9	47 ± 2	
3	678 ± 14	314 ± 4	50 ± 2	
4	718 ± 33	315 ± 11	51 ± 2	
5	614 ± 3**	237 ± 3**	30 ± 2**	
6	684 ± 13	325 ± 4	50 ± 2	
7	644 ± 23**	298 ± 16**	52 ± 1	5690 ± 160
8	703 ± 12	345 ± 10	54 ± 2	7802 ± 225
9	690 ± 8	317 ± 3	51 ± 1	
10	654 ± 26	310 ± 31		
11			48.3 ± 1.9	6260 ± 130
12	701 ± 22	326 ± 3	51.8 ± 0.4	
13	680 ± 23	324 ± 4		
Mean	686	320	50.7	6485
(1σ)	20	12	2.0	914
NIST Cert. Value	697.4	326.0	51.2	6228
(99% CL)	2.3	1.6	0.5	49

Contributing Laboratory Data for SRM 2557 in mg/kg

Lab	Pt[a]	Pd[a]	Rh[b]	Pb[b]
1	1204 ± 13**	257 ± 7	143 ± 4	13440 ± 89
2	1131 ± 6	224 ± 2	140 ± 0	
3	1120 ± 16	219 ± 4	133 ± 4	
4	1131 ± 41	215 ± 8	127 ± 6	
5	1062 ± 8**	131 ± 2**	77 ± 1**	
6	1113 ± 33	237 ± 9	128 ± 9	
7	1054 ± 47**	226 ± 12	137 ± 1	13440 ± 74
8	1153 ± 14	241 ± 5	134 ± 3	16800 ± 180
9	1125 ± 9	226 ± 2	137 ± 1	
10	1095 ± 38	236 ± 8		
11			134 ± 4	13900 ± 300
12	1075 ± 16	227 ± 5	127 ± 1	
13	1131 ± 15	232 ± 2		
Mean	1119	231	134	14395
(1σ)	23	12	5	1618
NIST Cert. Value	1131	233.2	135.1	13931
(99% CL)	11	1.9 1.9	97	

[a] INAA, ICP, XRF
[b] ICP, XRF