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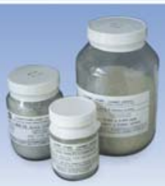
CCRMP
Canadian Certified Reference Materials Project



PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: March 2010 Version: March 2010

RTS-3a

Certified Reference Material for a Sulphide Ore Mill Tailings

Table 1 – RTS-3a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	11.1	0.6	1.0	0.4
Al non AD2[a]	%	5.12	0.08	0.14	0.07
As	µg/g	18.2	1.6	2.6	1.5
Ba non AD2[a]	µg/g	106	9	16	8
Bi	µg/g	31.3	1.6	1.7	1.0
Ca non AD2[a]	%	2.14	0.05	0.10	0.05
Cd	µg/g	9.21	0.30	0.85	0.40
Co	µg/g	143	3	12	4
Cu	%	0.2353	0.0046	0.0082	0.0030
Fe[b]	%	20.49	0.31	0.55	0.27
K non AD2[a]	%	0.460	0.014	0.027	0.013
Mg non AD2[a]	%	2.483	0.054	0.071	0.032
Mn non AD2[a]	%	0.1585	0.0035	0.0091	0.0045
Na non AD2[c]	%	0.684	0.017	0.011	0.006
Ni	µg/g	61.3	1.8	6.5	2.5

cont'd

Table 1 – RTS-3a Certified Values *cont'd*

Pb	µg/g	209	5	21	7
S$_{\text{non AD2}}$[a]	%	9.59	0.12	0.23	0.12
Si	%	18.28	0.15	0.30	0.20
Sr$_{\text{non AD2}}$[c]	µg/g	44.7	1.2	3.2	1.8
Zn	µg/g	2890	50	140	50

a digestion by two acids, usually hydrochloric and nitric acid, excluded as method outlier based on statistical tests (non AD2)
b digestions by one and two acids, usually hydrochloric and nitric acids, excluded as method outliers based on statistical tests, and only instrumental finishes included
c digestion by two acids, usually hydrochloric and nitric acids, excluded as statistical outliers (non AD2)

Table 2 – RTS-3a Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Au[d]	µg/g	0.561	0.108	0.052	0.060
Cr$_{\text{non AD2}}$[a]	µg/g	176	7	26	13
P$_{\text{non AD2}}$[a]	%	0.0446	0.0014	0.0046	0.0036
Sb$_{\text{non AD2}}$[a,e]	µg/g	2.83	0.12	0.22	0.23
Se	µg/g	44.8	1.5	8.9	5.7
Ti$_{\text{non AD2}}$[c]	%	0.351	0.008	0.052	0.027
Zr$_{\text{non AD2}}$[a]	µg/g	78	3	12	7

a digestion by two acids, usually hydrochloric and nitric acid, excluded as method outliers based on statistical tests (non AD2)
c digestion by two acids, usually hydrochloric and nitric acids, excluded as statistical outliers (non AD2)
d fire assay methods only
e statistical analysis of the data warrants classification as provisional despite only 6 sets of data for antimony

Table 3 – RTS-3a Informational Values

Analyte	Units	Mean	No. accepted sets/values	Analyte	Units	Mean	No. accepted sets/values
Al AD2	%	2.5	5/25	P AD2	%	0.036	4/20
C	%	0.04	8/40	Nb	µg/g	4	3/15
Ca AD2	%	0.8	4/20	Pd	µg/g	0.004	4/20
Ce	µg/g	30	4/20	Rb	µg/g	13	3/15
CO_2	%	0.04	3/15	S elemental	%	1.2	4/20
Cr AD2	µg/g	130	7/35	S sulphate	%	1.1	6/30
Cs	µg/g	0.4	3/15	S sulphide	%	8	4/20
Fe TITN	%	20.49	5/26	Sb AD2	µg/g	2	4/20
Ga	µg/g	30	5/25	Sc	µg/g	10	6/30
Hf	µg/g	2	4/20	Tb	µg/g	0.5	3/15
In	µg/g	1.6	4/20	Te	µg/g	2.0	5/25
K AD2	%	0.12	5/24	Th	µg/g	1.4	4/20
La	µg/g	10	4/20	Tl	µg/g	3	4/20
Li	µg/g	10	3/15	U	µg/g	0.3	5/25
LOI	%	10.6	3/15	V	µg/g	120	6/30
Mg AD2	%	1.4	4/20	W	µg/g	5	5/25
Mn AD2	%	0.07	5/25	Y	µg/g	10	5/25
Mo	µg/g	3	5/25				

AD2 ***digestion by two acids, usually hydrochloric and nitric acids***
LOI ***loss on ignition at 950°C***
TITN ***titration methods only***

Statistical parameters for iron by titration

- within-lab standard deviation = 0.02%
- between-labs standard deviation = 0.08%
- 95% confidence interval of mean = 0.05%

SOURCE

RTS-3a is a sulphide ore mill tailings obtained from an unoxidized zone of the Waite Amulet tailings, north of Noranda, QC, Canada. The raw material was donated by Xstrata Copper. The raw material for RTS-3a was obtained from the same source as its predecessor, RTS-3.

DESCRIPTION

The mineral species include: quartz (18.0%), pyrrhotite (16.6%),amphibole (15.1%), clinochlore (9.3%), K-feldspar (6.1%), vermiculite (6.0%), albite (5.8%), magnetite (5.7%), pyrite (5.0%), pyroxene (4.7%), cummingtonite (3.8%), muscovite (1.4%), gypsum (0.7%), ilmenite (0.5%), chalcopyrite and titanite (both at 0.3%), sphalerite and talc (both at 0.2%); and cassiterite, jarosite and tetrahedrite – tennantite (all three at 0.1%).

INTENDED USE

RTS-3a is suitable for the analysis of various elements at major, minor and trace levels in tailings. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE

RTS-3a should be used “as is”, without drying. The contents of the bottle should be thoroughly mixed before taking samples. The contents of the bottle should be exposed to air for the shortest time possible. Unused material should be stored under an inert gas in a desiccator, or in a new, heat-sealed laminated foil pouch. The values herein pertain to the material when produced. CANMET-MMSL is not responsible for changes occurring after shipment.

HANDLING INSTRUCTIONS

Normal safety precautions for handling fine particulate matter are suggested, such as the use of safety glasses, breathing protection, gloves and a laboratory coat.

METHOD OF PREPARATION

The raw material was dried at 32°C, crushed, ground and sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction was 77%. The product was blended, and then bottled in 100-gram units. Each bottle was purged with nitrogen and sealed in a laminated polyethylene - foil pouch.

HOMOGENEITY

The homogeneity of the stock was investigated using fifteen bottles chosen according to a stratified random sampling scheme. Three splits were analyzed from each bottle. Samples of 0.01g from each split were digested with hydrochloric and nitric acids using a microwave and analyzed for arsenic using inductively coupled plasma – mass spectrometry. Samples of 0.25g grams from each split were digested using four acids, hydrofluoric, hydrochloric, nitric and perchloric acids, and analyzed for cobalt, copper and zinc by inductively coupled plasma – optical emission spectrometry. Samples of 0.15 grams from each split were analyzed for sulphur using a combustion apparatus with infrared detection. Use of a smaller sub-sample than specified above will invalidate the use of the certified values and associated parameters.

A one–way analysis of variance technique (ANOVA)[1] was used to assess the homogeneity of these elements. No significant between-bottle variation was observed for arsenic, cobalt, copper, sulphur and zinc.

CERTIFIED VALUES

Twenty-five industrial, commercial and government laboratories participated in an interlaboratory measurement program using methods of their own choosing.

The methods involved multi acid digestions, microwave digestion, fusion, various types of fire assay pre-concentration (for gold), flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy, inductively coupled plasma – mass spectrometry and graphite furnace atomic absorption spectrometry. Fusion followed by X-ray fluorescence, as well as instrumental neutron activation analysis were used to determine several elements. Hydride generation atomic absorption spectrometry was used for the determination of arsenic, antimony, bismuth and tellurium. Photometry, titration and infrared spectrometry were used to determine carbon dioxide.

Iron was determined by multi acid digestions and various fusions followed by flame atomic absorption spectrometry, inductively coupled plasma – optical emission spectrometry, inductively coupled plasma – mass spectrometry and titration, as well as fusion and X-ray fluorescence. The mean of the sets for iron by instrumental finishes and titration is the same. Based on an F-test, the value for sets by titration was separated from the sets by instrumental finishes in order to report the better precision of the titration methods. The mean of the sets by instrumental finishes meets the criteria for certification. The mean of the 6 sets by titration is given an informational value.

Sulphur was determined by acid digestions, various classical separations, and followed by gravimetric finish, inductively coupled plasma – optical emission spectrometry, inductively coupled plasma – mass spectrometry, as well as combustion with infrared spectrometry. Elemental sulphur and sulphur in the form of sulphate and sulphide were determined by various leaches, calcination, various separations, combustion and the same finishes as sulphur, as well as high pressure liquid chromatography and titration.

ANOVA was used to calculate the consensus values and other statistical parameters from the interlaboratory measurement program. Values are deemed to be certified if derived from 10 or more sets of data that meet CCRMP statistical criterion regarding the agreement of the results. Twenty elements were certified (see Table 1). Many certified elements exclude digestion by two acids based on statistical tests.

Full details of all work, including the statistical analyses, the methods and the names of the participating laboratories are contained in the Certification Report. For more details on how to use reference material data to assess laboratory results, users are directed to ISO Guide 33:2000, pages 14-17, and the publication, “Assessment of laboratory proficiency using CCRMP reference materials”, at www.ccrmp.ca.

UNCERTIFIED VALUES

Six provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CCRMP statistical criterion regarding agreement; or alternatively, 10 or more sets of data, that do not fulfill the CCRMP statistical criteria required for certification. Additionally, the statistical analysis of the data warranted provisional status for the 6 sets of data for antimony by all methods except digestion by two acids. Informational values for 35 elements, shown in Table 3, were derived from the means of a minimum of 3 sets of data.

TRACEABILITY

The values quoted herein are based on the consensus values derived from the statistical analysis of the data from the interlaboratory measurement program, and the standards used by the individual laboratories. The report gives the available details.

CERTIFICATION HISTORY

RTS-3a is a new material.

PERIOD OF VALIDITY

The certified values are valid until March 31, 2032. The stability of the material will be monitored every two years for the duration of the inventory. Updates will be published on the CCRMP web site.

LEGAL NOTICE

CANMET-MMSL has prepared this reference material and statistically evaluated the analytical data of the interlaboratory measurement program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies CANMET-MMSL from and against all liability and costs arising out of the use of this material and information.

CERTIFYING OFFICERS

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FOR FURTHER INFORMATION

The Certification Report is available free of charge upon request to:

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REFERENCES

1. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960.