

Greenpeace Research Laboratories Analytical Results 2015-06

Analysis of samples of solid waste generated and stored at a facility that processes rare earth metal ores in Inner Mongolia, China. July 2015

Introduction

Three samples of ore processing solid wastes were received from an independent journalist for analysis at the Greenpeace Research Laboratories on the 11th March 2015. According to documentation supplied, all samples were collected in 2014.

The samples were collected from adjacent locations in an area used to store solid waste slurry from the processing of ores for the extraction of rare earth metals, at a facility in Inner Mongolia, China. Two of the samples (NGP15001 & NGP15002) were collected from an area containing relatively dry solid and consisted of relatively coarse material, while NGP15003 was collected from an area close to the outfall of a pipe carrying slurry to the storage area and contained notably wetter slurry and consisted of relatively fine material,.

Details of the samples received are provided in Table 1a, together with GPS coordinates for the sample collection locations in Table 1b.

All samples were analysed quantitatively for the presence of a range of metals and metalloids.

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Sample code	Location	Sample type	Description
NGP15001	solid waste slurry storage area	Solid waste	Collected from relatively dry part of the storage area, adjacent to NGP15002 (coarse material)
NGP15002	solid waste slurry storage area	Solid waste	Collected from relatively dry part of the storage area, adjacent to NGP15001 (coarse material)
NGP15003	solid waste slurry storage area	Solid waste slurry	Collected from part of the storage area containing wetter slurry (fine material) close to outfall of a pipe carrying slurry to the storage area

Table 1a: details of samples received and analysed at the Greenpeace Research Laboratories

Sample code	N		E	
	degree (°)	minutes (') seconds (")	degree (°)	minutes (') seconds (")
NGP15001	40°	38'49.39"	109°	40'41.67"
NGP15002	40°	38'48.91"	109°	40'41.45"
NGP15003	40°	38'48.48"	109°	40'39.20"

Table 1b: GPS coordinated of sample collection locations

Materials and methods

Metal and metalloid concentrations were determined for all samples either by ICP atomic emission spectrometry (AES) following acid digestion at the Greenpeace Research laboratory, or by ICP mass spectrometry following lithium tetra/metaborate fusion extraction at an external laboratory. Both cases employed the use of appropriate certified reference materials and intra-laboratory standards.

Results and Discussion

The results for the samples are reported in Table 2. Where the concentration of a metal or metalloid was below the limit of quantification for the analytical method employed the result is presented as '<xx' where xx is the method limit of quantification for the individual metal or metalloid. Reported average concentrations for Earth's continental crust are also provided for comparison (Krauskopf & Bird 1994).

Sample code	NGP15001	NGP15002	NGP15003	Earth's continental crust, average*
Description	dark grey course material	dark grey course material	dark grey fine, clay like, material	-
Location	40°38'49.39"N 109°40'41.67"E	40°38'48.91"N 109°40'41.45"E	40°38'48.48"N 109°40'39.20"E	-
pH	8.13	8.19	7.90	-
concentrations (mg/kg dry weight)				
Antimony (Sb)	<20	<20	<20	0.2
Arsenic (As)	<5	<5	<5	1.8
Barium (Ba)	1330	1740	2475	425
Beryllium (Be)	4.6	3.5	6.8	2.8
Cadmium (Cd)	2.6	2.5	2.5	0.2
Cerium (Ce)	28383	27551	24005	60.0
Chromium (Cr)	10	14	16	100
Cobalt (Co)	30	28	9	25
Copper (Cu)	41	58	24	55
Dysprosium (Dy)	92	91	121	3.0
Erbium (Er)	43.6	42.3	53.5	2.8
Europium (Eu)	149	144	193	1.2
Gadolinium (Gd)	612	600	780	5
Germanium (Ge)	<40	<40	<40	1.5
Holmium (Ho)	11.5	11.3	15.0	1.2
Indium (In)	<10	<10	<10	0.1
Lanthanum (La)	14936	14913	20194	30.0
Lead (Pb)	556	472	290	13
Lutetium (Lu)	1.1	1.0	1.2	0.5
Manganese (mn)	7010	7310	6175	950
Mercury (Hg)	<20	<20	<20	0.08
Molybdenum (Mo)	47.0	39.9	72.4	1.5
Neodymium (Nd)	9917	9317	11649	28.0
Nickel (Ni)	27.7	27.7	12.9	75
Niobium (Nb)	302	137	76	20
Praseodymium (Pr)	3013	2851	3631	8.2
Samarium (Sm)	816	778	1004	6.0
Strontium	443	393	559	375
Tantalum (Ta)	9	9	8	2
Terbium (Tb)	40.3	39.9	51.8	0.9
Thulium (Tm)	1.7	1.7	2.1	0.5
Tungsten (W)	20.3	16.6	17.8	1.5
Vanadium (V)	26	25	36	135
Ytterbium (Yb)	8.8	8.8	10.6	3.4
Yttrium (Y)	139	126	188	33
Zinc (Zn)	449	530	375	70

Table 2: Concentrations of metals and metalloids (mg/kg dry weight) in the samples of solid waste; together with reported average concentrations for the Earth's continental crust (* Krauskopf & Bird 1994)

Extraction of the material that the containers used to store the samples were composed of indicated that there was the potential for traces of some elements to leach from the sample container plastic under moderately acidic conditions. The maximum amount of each element that could have leached

into a sample, however, is insignificant in relation to the concentrations present in the sample (<0.5% of sample value in all cases). In addition, all samples were slightly alkaline, which generally reduces the potential for elements to leaching from the sampling container into the sample.

For more information please contact:

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References

K.B. Krauskopf & D.K. Bird (1994) Introduction To Geochemistry, 3rd Edition.

Appendix 1: Details of methodologies

Analysis using acid digestion and ICP atomic emission spectrometry (AES) analysis

Preparation

A representative portion of each sample was air dried to constant weight, homogenised and ground to a powder using a pestle and mortar. Approximately 0.5 g of each dried and ground sample was digested with 2 ml concentrated hydrochloric acid and 8 ml concentrated nitric acid, firstly overnight at room temperature, then using microwave-assisted digestion with a CEM MARS Xpress system, with a temperature ramp to 180°C over 15 minutes followed by holding at 180°C for a further 15 minutes. Following cooling, digests were filtered and made up to 50ml with deionised water.

Analysis

Prepared sample digests were analysed by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) using a Varian MPX Simultaneous Spectrometer. Multi-element standards at concentrations of 0.5, 2.0 and 5.0 mg/l respectively, and matrix matched to the samples, were used for instrument calibration. Any sample exceeding the calibration range was diluted accordingly, in duplicate, and re-analysed.

Quality control

One of the samples were prepared for ICP analysis in duplicate and analysed to verify method reproducibility, along with a blank sample (10% v/v nitric acid in deionised water). To check the method efficiency, a certified reference material (CRM) was prepared in an identical manner; GBW07311, stream sediment certified by the China National Analysis Centre for Iron and Steel, Beijing, China.

Calibration of the ICP-AES was validated by the use of quality control standards of 0.4 mg/l and 4 mg/l respectively prepared in an identical manner but from different reagent stocks to the instrument calibration standards.

Further details of the methods employed can be provided on request.

Analysis using lithium tetra/metaborate fusion extraction and ICP mass spectrometry (MS)

The samples were subjected to a fusion process; 0.25 g of each sample was mixed with 1 g of lithium tetra/metaborate flux and then fused at 950°C for one hour. Each melt was then dissolved in 10% nitric acid made up to a final volume of 100 ml. An estuarine sediment certified reference material (CRM), BCR 667, was also subjected to the fusion procedure for QA purposes.

The analytes of interest in all samples were measured using a Thermo Scientific X Series 2 ICP-MS instrument. An independent check standard was also prepared and analysed as QA for the instrumental calibration. All balances, pipettes and instruments used were calibrated and operated according to the laboratories relevant standard operating procedures. Calibration standards were prepared with 2% nitric acid. An internal standard solution of rhodium, indium and iridium was used throughout.

pH

To determine the pH of each sample, 10 ml deionised water was added to a 10 g portion of air dried homogenised sample, the slurry was mixed well and allowed to stand to 10 minutes. The pH of the slurry was determined using a Hanna Instruments HI98129 pH meter calibrated using pH 4.01 and pH 7.01 Hanna buffer solutions. The pH meter and electrode was rinsed will with deionised water between samples.