

## Technique Note

# Determination of cadmium in geological samples by aerosol dilution ICP-MS after inverse aqua regia extraction

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## Supplementary Information

1. **Table S1.** Spectral interferences on Cd isotopes.
2. **Table S2.** The Cd levels in 77 Chinese geological SRMs by the proposed method,  $\mu\text{g g}^{-1}$ .
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4. **Table S4.** The Ag levels in 8 geological SRMs by the proposed method,  $\mu\text{g g}^{-1}$ .
5. **Fig.S1.** Comparison of the SBR of three analysis method for Cd determination under the respective optimized conditions. Methods are: conventional standard mode ICP-QMS after complete digestion, aerosol dilution ICP-QMS after complete digestion, and aerosol dilution ICP-QMS after boiling inverse aqua regia extraction. The certified Cd concentration ( $1 \text{ ng mL}^{-1}$ ) and the matrix element concentrations ( $200 \text{ ng mL}^{-1}$  Mo and  $1000 \text{ ng mL}^{-1}$  Zr) were used to calculate the SBR values. 90% Zr was removed by the boiling inverse aqua regia extraction.
6. **Fig.S2.** Stability of Cd values obtained by aerosol dilution ICP-MS in SRM GSM-1. Mean  $\pm$  SD, the SD is the standard deviation for ten separate aliquots of the samples analyzed over a period of three months. The error bars in Figure were defined as the standard deviation for 3 replicates in single analysis.

**Table S1.** Spectral interferences on Cd isotopes

Analyte		Interference ions (Abundance, %)	
Isotope	Abundance (%)	Polyatomic	Isobaric
<sup>106</sup> Cd	1.25	<sup>89</sup> YOH (100)	<sup>106</sup> Pd (27.33)
		<sup>90</sup> ZrO (51.45)	
		<sup>91</sup> ZrOH (11.22)	
<sup>108</sup> Cd	0.89	<sup>92</sup> ZrO (17.15)	<sup>108</sup> Pd (26.46)
		<sup>92</sup> MoO (14.53)	
		<sup>93</sup> NbOH (100)	
<sup>110</sup> Cd	12.49	<sup>94</sup> MoO (9.15)	<sup>110</sup> Pd (11.72)
		<sup>94</sup> ZrO (17.38)	
		<sup>94</sup> ZrOH (17.38)	
<sup>111</sup> Cd	12.8	<sup>94</sup> MoOH (9.15)	/
		<sup>95</sup> MoO (15.84)	
		<sup>95</sup> MoOH (15.84)	
<sup>112</sup> Cd	24.13	<sup>96</sup> MoO (16.67)	<sup>112</sup> Sn (0.97)
		<sup>96</sup> ZrO (2.8)	
		<sup>96</sup> ZrOH (2.8)	
<sup>113</sup> Cd	12.22	<sup>96</sup> MoOH (16.67)	<sup>113</sup> In (4.29)
		<sup>97</sup> MoO (9.6)	
		<sup>97</sup> MoOH (9.6)	
<sup>114</sup> Cd	28.73	<sup>98</sup> MoO (24.39)	<sup>114</sup> Sn (0.66)
		<sup>98</sup> RuO (1.87)	
		<sup>99</sup> RuOH (12.76)	
<sup>116</sup> Cd	7.49	<sup>100</sup> RuO (12.6)	<sup>116</sup> Sn (14.54)
		<sup>100</sup> MoO (9.82)	

**Table S2.** The Cd levels in 77 Chinese geological SRMs by the proposed method,  $\mu\text{g g}^{-1}$ 

Geological SRMs	Description	Determined values (N=3)	Certified values	
Soil	GSS-1	Dark-brown earth	$4.10 \pm 0.10$	$4.30 \pm 0.40$
	GSS-2	Chestnut soil	$0.061 \pm 0.004$	$0.071 \pm 0.014$
	GSS-3	Yellow-brown earth	$0.054 \pm 0.001$	$0.060 \pm 0.009$
	GSS-4	Limy red earth	$0.321 \pm 0.003$	$0.350 \pm 0.060$
	GSS-5	Yellow-red earth	$0.460 \pm 0.020$	$0.450 \pm 0.060$
	GSS-6	Yellow-red earth	$0.134 \pm 0.007$	$0.130 \pm 0.030$
	GSS-7	Laterite(latosol)	$0.088 \pm 0.008$	$0.080 \pm 0.020$
	GSS-8	Loess	$0.115 \pm 0.008$	$0.130 \pm 0.020$
	GSS-9	Soil	$0.089 \pm 0.002$	$0.100 \pm 0.020$
	GSS-10	Farming soil	$0.099 \pm 0.008$	$0.105 \pm 0.013$
	GSS-11	Soil	$0.115 \pm 0.008$	$0.125 \pm 0.012$
	GSS-12	Soil	$0.140 \pm 0.007$	$0.150 \pm 0.020$
	GSS-13	Soil	$0.120 \pm 0.002$	$0.130 \pm 0.010$
	GSS-14	Soil	$0.200 \pm 0.010$	$0.200 \pm 0.020$
	GSS-15	Soil	$0.213 \pm 0.009$	$0.210 \pm 0.020$
	GSS-16	Sandy soil	$0.264 \pm 0.004$	$0.250 \pm 0.020$
	GSS-17	Saline-alkali soil	$0.055 \pm 0.002$	$0.058 \pm 0.011$
	GSS-18	Brown desert soil	$0.140 \pm 0.009$	$0.150 \pm 0.010$
	GSS-19	Saline-alkali soil	$0.099 \pm 0.008$	$0.108 \pm 0.009$
	GSS-20	Sierozem soil	$0.103 \pm 0.005$	$0.108 \pm 0.011$
	GSS-21	The Yellow Sea tidal flat soil	$0.132 \pm 0.002$	$0.139 \pm 0.008$
	GSS-22	Soil	$0.058 \pm 0.003$	$0.065 \pm 0.012$
	GSS-23	Tidal flat soil	$0.130 \pm 0.010$	$0.150 \pm 0.020$
	GSS-24	Soil	$0.102 \pm 0.004$	$0.106 \pm 0.007$
	GSS-25	Huai River soil	$0.167 \pm 0.008$	$0.175 \pm 0.010$
	GSS-26	Yangtze River soil	$0.130 \pm 0.005$	$0.140 \pm 0.010$
	GSS-27	Soil	$0.570 \pm 0.010$	$0.590 \pm 0.040$
	GSS-28	Soil	$0.520 \pm 0.030$	$0.520 \pm 0.030$
Sediment	GSD-1	Granite area sediment	$0.075 \pm 0.008$	$0.088 \pm 0.014$
	GSD-2	Stream sediment	$0.060 \pm 0.003$	$0.065 \pm 0.011$
	GSD-3	Porphyry copper deposit sediment	$0.090 \pm 0.010$	$0.100 \pm 0.020$
	GSD-4	Limestone ore district sediment	$0.192 \pm 0.003$	$0.190 \pm 0.020$
	GSD-5	Skarn mining area sediment	$0.840 \pm 0.030$	$0.820 \pm 0.050$
	GSD-6	Porphyry copper deposit sediment	$0.425 \pm 0.013$	$0.430 \pm 0.030$
	GSD-7	Stream sediment	$1.20 \pm 0.06$	$1.05 \pm 0.06$
	GSD-8	Acidic volcano rock area sediment	$0.074 \pm 0.003$	$0.081 \pm 0.012$
	GSD-9	Yangtze River sediment	$0.250 \pm 0.030$	$0.260 \pm 0.040$
	GSD-10	Carbonate area sediment	$1.160 \pm 0.045$	$1.12 \pm 0.08$
	GSD-11	Multi metal mining area sediment	$2.33 \pm 0.05$	$2.30 \pm 0.20$
	GSD-12	Stream sediment	$4.20 \pm 0.30$	$4.00 \pm 0.30$
	GSD-13	Quartz sand area sediment	$0.031 \pm 0.001$	$0.045 \pm 0.015$
	GSD-14	Sediment	$0.180 \pm 0.010$	$0.200 \pm 0.030$
	GSD-15	Multi metal mining area sediment	$0.325 \pm 0.008$	$0.340 \pm 0.020$
	GSD-16	Metamorphic rock area sediment	$0.084 \pm 0.010$	$0.093 \pm 0.009$
	GSD-17	Stream sediment	$4.50 \pm 0.30$	$4.30 \pm 0.50$
	GSD-18	Granite area sediment	$0.091 \pm 0.005$	$0.095 \pm 0.010$
	GSD-19	Stream sediment	$0.088 \pm 0.007$	$0.120 \pm 0.01$
	GSD-20	Copper nickel mine sediment	$0.216 \pm 0.005$	$0.220 \pm 0.010$
	GSD-21	Acidic volcano rock area sediment	$0.780 \pm 0.04$	$0.760 \pm 0.030$
	GSD-22	Copper ore district sediment	$0.164 \pm 0.002$	$0.165 \pm 0.010$
	GSD-23	Stream sediment	$4.69 \pm 0.07$	$4.80 \pm 0.50$
GSD-1a	Granite area sediment	$0.091 \pm 0.006$	$0.110 \pm 0.030$	

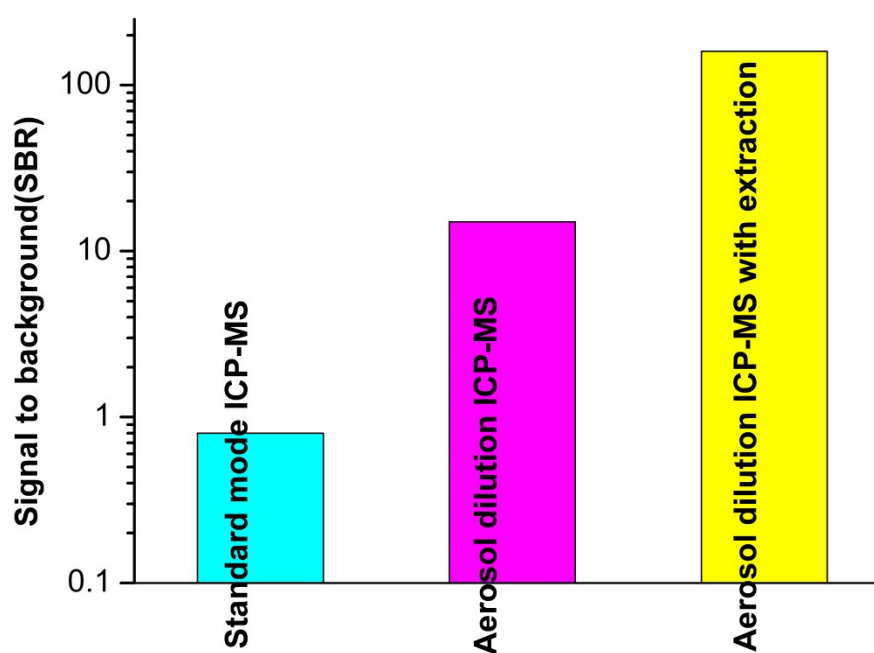
	GSD-2a	Granite area sediment	$0.102 \pm 0.004$	$0.108 \pm 0.009$
	GSD-3a	Copper nickel mine sediment	$0.493 \pm 0.005$	$0.500 \pm 0.060$
	GSD-4a	Limestone area sediment	$0.850 \pm 0.060$	$0.900 \pm 0.050$
	GSD-5a	Skarn mining area sediment	$1.36 \pm 0.08$	$1.37 \pm 0.10$
	GSD-7a	Lead zinc mine sediment	$5.20 \pm 0.20$	$5.60 \pm 0.60$
	GSD-8a	Acidic volcano rock area sediment	$0.150 \pm 0.010$	$0.160 \pm 0.010$
Rock	GSR-2	Andesite	$0.062 \pm 0.007$	$0.061 \pm 0.014$
	GSR-4	Quartz sandstone	$0.055 \pm 0.005$	$0.060 \pm 0.016$
	GSR-6	Argillaceous limestone	$0.062 \pm 0.030$	$0.070 \pm 0.020$
	GSR-8	Lujavritite	$0.660 \pm 0.030$	$0.610 \pm 0.080$
	GSR-10	Gabbro	$0.069 \pm 0.003$	$0.090 \pm 0.030$
	GSR-11	Rhyolite	$0.150 \pm 0.010$	$0.140 \pm 0.020$
	GSR-12	Dolomite	$0.067 \pm 0.003$	$0.070 \pm 0.020$
	GSR-16	Diabase	$0.370 \pm 0.010$	$0.390 \pm 0.080$
	GSR-17	Kimberlite	$0.570 \pm 0.050$	$0.460 \pm 0.200$
	GSR-22	Limestone	$0.121 \pm 0.011$	/
	GSR-23	Limestone	$0.770 \pm 0.040$	/
	GSR-24	Limestone	$0.063 \pm 0.006$	/
	GSR-27	Limestone	$0.550 \pm 0.050$	/
	GSR-28	Limestone	$0.510 \pm 0.030$	/
	GUI-1	Limestone	$0.042 \pm 0.005$	/
	GUI-1	Limestone	$0.160 \pm 0.017$	/
	DIAN-1	Limestone	$0.520 \pm 0.030$	/
	DIAN-2	Limestone	$0.366 \pm 0.027$	/
DIAN-3	Limestone	$0.209 \pm 0.015$	/	

**Table S3.** Closed pressurized digestion with a mixture of HF + HNO<sub>3</sub>

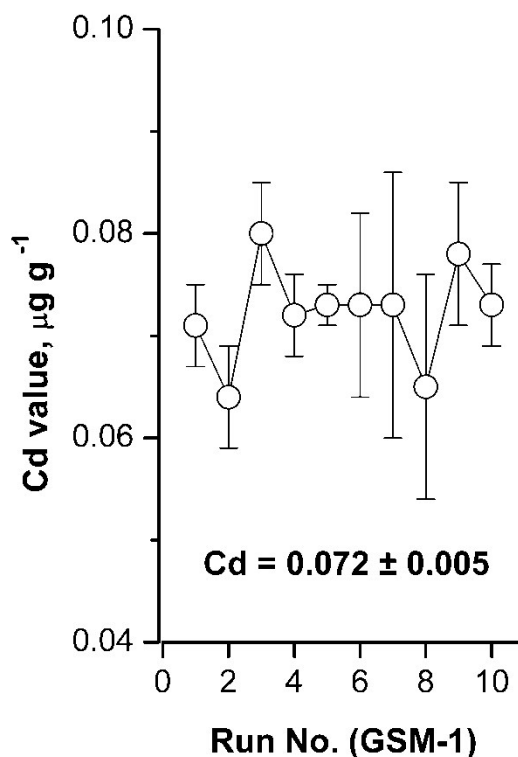
Step	Description
1	50 mg sample powder (< 200 mesh) was weighed into a Teflon bomb, moistened with a few drops of ultrapure water.
2	1.0 mL HNO <sub>3</sub> +1.0 ml HF were added. The sealed bomb was heated at 190 °C in oven for > 48 h.
3	Open the bomb and evaporate the solution at ~120 °C to dryness. This was followed by adding 1 ml HNO <sub>3</sub> and evaporating to the second round of dryness.
4	The resultant salt was re-dissolved by adding ~3 ml of 30% HNO <sub>3</sub> and resealed and heated in the bomb at 190 °C for >12 h.
5	The final solution was diluted to ~100 g with mixture of 2% HNO <sub>3</sub> for ICP-MS analysis.

**Table S4.** The Ag levels in 8 geological SRMs by the proposed method,  $\mu\text{g g}^{-1}$ 

Geological SRMs	Determined values	Certified values
	(N=3)	
GSS-1, Dark-brown earth	$0.365 \pm 0.004$	$0.35 \pm 0.05$
GSS-6, Yellow-red earth	$0.180 \pm 0.006$	$0.2 \pm 0.02$
GSS-7, Laterite (latosol)	$0.063 \pm 0.009$	$0.057 \pm 0.011$
GSD-3, Porphyry copper deposit sediment	$0.57 \pm 0.01$	$0.59 \pm 0.05$
GSD-6, Porphyry copper deposit sediment	$0.351 \pm 0.011$	$0.36 \pm 0.03$
GSR-2, Andesite	$0.059 \pm 0.003$	$0.071 \pm 0.009$
GSR-4, Quartz sandstone	$0.057 \pm 0.002$	$0.062 \pm 0.007$
BCR-2, basalt	$0.035 \pm 0.001$	$0.041 \pm 0.004$



**Fig.S1.** Comparison of the SBR of three analysis method for Cd determination under the respective optimized conditions. Methods are: conventional standard mode ICP-QMS after complete digestion, aerosol dilution ICP-QMS after complete digestion, and aerosol dilution ICP-QMS after boiling inverse aqua regia extraction. The certified Cd concentration ( $1 \text{ ng mL}^{-1}$ ) and the matrix element concentrations ( $200 \text{ ng mL}^{-1}$  Mo and  $1000 \text{ ng mL}^{-1}$  Zr) were used to calculate the SBR values. 90% Zr was removed by the boiling inverse aqua regia extraction.



**Fig.S2.** Stability of Cd values obtained by aerosol dilution ICP-MS in SRM GSM-1. Mean  $\pm$  SD, the SD is the standard deviation for ten separate aliquots of the samples analyzed over a period of three months. The error bars in Figure were defined as the standard deviation for 3 replicates in single analysis.