

S1. Details of technical methods

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectral (EDS) analysis was done using the Tescan Vega II XMU machine at Carleton University. Polished thin sections of epoxy-impregnated samples were prepared by Vancouver Geotech Labs. Electron probe micro-analysis (EPMA) was done on the Cameca instrument at Carleton University Earth Sciences microbeam lab.

XRD was done at Actlabs, Activation Laboratories Ltd., Ancaster, Ontario. Packed powder samples were analysed on Panalytical X-Pert Pro diffractometer, with Cu X-ray source, at 40 kV, 40 mA, 4-80 ° 2θ range in 0.02 °2θ steps.

Chemical analyses were done on the ~20-63 μm fraction for 4 different samples of the Clay Hall material. Trace element composition was done by inductively coupled plasma mass spectrometry (ICPMS) at ActLabs (51 elements, the Ultratrace 4 package). The acid-digested samples were analyzed by Perkin Elmer Sciex ELAN 6000, 6100 or 9000 ICPMS. The detection limit for most elements is 0.1 ppm. Major species were analyzed at Actlabs by ICPMS on acid-digested lithium metaborate/tetraborate fused samples (the WRA-ICP 4B package). Detection limit is 0.01% for most species, and 0.001% for MnO and TiO₂.

The test for allophane, following Fieldes and Perrott (1966), exploits the alkaline reaction of allophane and NaF. Phenolphthalein indicator has no colour in pH <8.8 and a bright crimson in pH > 9.0. When ~10 mg of sediment is mixed with a few drops of ~1M NaF and a few drops of indicator (initially colourless) a change to crimson within 2 minutes indicates > 7% allophane, to pink 5-7% allophane, and no change indicates < 5% allophane. The samples must be tested first with sediment and indicator alone to be sure that the pink colour is not from natural alkalinity of carbonate-rich solutions.

Sr isotopic ratios were measured at the Carleton University Isotope Geochemistry and Geochronology Research Centre (IGCRC). Approximately 200 milligrams of the ~20-63 μm fraction was dissolved (using the sequence 50% HF-12M HNO₃, then 8M HNO₃ and finally 6M HCl). The

residue was taken up in 2.5M HCl for cation exchange (Dowex AG50-X8) elution. Total procedural blanks for Sr are < 250 picograms. Isotope ratios were measured on Thermo Finnigan Triton TIMS machine, run on a single Ta filament at 1380-1600°C. Isotope ratios were normalized to $^{86}\text{Sr}/^{88}\text{Sr} = 0.11940$ to correct for fractionation.

U-Th isotopic ratios of the tephra and speleothem were also measured at the Carleton University Isotope Geochemistry and Geochronology Research Centre (IGCRC). All chemical preparation was done in ultraclean conditions. Tephra was dissolved in a mixture of concentrated HNO_3 and HF over 48 hours. Speleothem samples were cleaned of any visible detrital particles, vugs, or cracks using a dentist's drill under binocular microscope; then ultrasonically cleaned, heated for 5 hours at 875 °C to remove organics, and dissolved in HNO_3 . U and Th were co-precipitated with iron hydroxide. Both speleothem and tephra samples were spiked with ^{233}U - ^{236}U - ^{229}Th tracer (calibrated by analysis of uraninite in secular equilibrium). U and Th were isolated on anion exchange columns (Dowex AG1-X 200-400 mesh) and measured on the Triton TIMS machine. Instrumental reproducibility is 0.06% for $^{234}\text{U}/^{238}\text{U}$ and 0.11% for $^{230}\text{Th}/^{234}\text{U}$. Speleothem samples were dated by standard U-Th disequilibrium techniques (e.g., Ivanovich et al., 1992, Chen et al., 1992), using half-lives from Cheng et al. (2000). Ages were adjusted for detrital contamination using the typical silicate activity ratio $^{230}\text{Th}/^{232}\text{Th}$ of 0.83 +/-0.42, derived from $^{232}\text{Th}/^{238}\text{U}$ activity ratio of 1.21 +/-0.6, $^{230}\text{Th}/^{238}\text{U}$ activity ratio of 1.0 +/-0.1, and $^{234}\text{U}/^{238}\text{U}$ activity ratio of 1.0 +/-0.1 (following Cruz et al., 2005).

Table S1. Individual results for EPMA on 11 shards

Sample	Analytical	Oxide weight %													
	Total %	SiO ₂	Al ₂ O ₃	TiO ₂	Cr ₂ O ₃	FeO	MgO	MnO	K ₂ O	CaO	Na ₂ O	BaO	P ₂ O ₅	SO ₃	Cl
CH-1	93.75	73.93	13.17	0.14	0.02	1.10	0.28	0.01	2.31	1.82	0.91	0.04	0.00	0.00	0.03
CH-2	95.59	75.66	11.95	0.17	0.00	1.16	0.23	0.10	2.34	1.83	2.01	0.07	0.05	0.02	0.01
CH-3	95.74	75.91	13.20	0.14	0.00	0.91	0.16	0.06	2.92	1.41	0.92	0.05	0.01	0.01	0.04
CH-4	93.17	73.29	12.40	0.12	0.00	1.05	0.23	0.09	2.81	1.71	1.34	0.06	0.03	0.00	0.04
CH-5	94.38	75.97	13.03	0.17	0.00	0.95	0.24	0.02	1.71	1.87	0.32	0.02	0.05	0.02	0.02
CH-6	93.26	72.66	12.85	0.13	0.00	1.03	0.23	0.06	2.86	1.67	1.63	0.03	0.06	0.00	0.04
CH-7	94.05	73.84	12.70	0.18	0.00	1.15	0.24	0.12	2.66	1.80	1.22	0.04	0.00	0.01	0.08
CH-8	94.54	73.98	13.14	0.14	0.00	1.09	0.23	0.08	2.57	1.68	1.57	0.04	0.00	0.00	0.02
CH-9	98.51	77.31	13.82	0.15	0.00	1.00	0.25	0.08	2.43	1.86	1.55	0.03	0.00	0.02	0.00
CH-10	90.72	70.59	13.86	0.14	0.00	1.05	0.25	0.11	2.14	1.66	0.76	0.04	0.01	0.02	0.07
CH-11	96.66	75.52	13.35	0.15	0.00	0.96	0.24	0.06	2.39	1.78	2.13	0.00	0.07	0.00	0.00
Average		74.42	13.04	0.15	0.00	1.04	0.23	0.07	2.47	1.74	1.31	0.04	0.03	0.01	0.03
St Dev		1.89	0.56	0.02	0.01	0.08	0.03	0.03	0.35	0.13	0.55	0.02	0.03	0.01	0.03

Appendix

Table A1. Trace elemental geochemistry of the Mulu tephra: ICPMS results, n = 2

Analyte	ppm	1 sigma	Analyte	ppm	1 sigma	Analyte	ppm	1 sigma
Li	21.40	1.84	Ga	16.00	1.27	Sm	3.20	0.00
Cd	< 0.1		As	0.60	0.42	Gd	2.90	0.28
V	5.50	0.71	Rb	54.10	0.85	Tb	0.40	0.00
Cr	13.60	1.84	Y	11.20	0.42	Dy	2.15	0.07
Mn	170.50	6.36	Sr	169.00	1.41	Cu	16.20	0.28
Hf	0.45	0.07	Zr	21.50	3.54	Ge	0.70	0.00
Ni	1.75	0.21	Nb	2.00	0.00	Tm	0.20	0.00
Er	1.05	0.07	Mo	0.45	0.07	Yb	0.90	0.00
Be	1.20	0.14	In	< 0.1		Lu	0.10	0.00
Ho	0.40	0.00	Sn	< 0.1		Ta	0.20	0.00
Ag	0.17	0.01	Sb	< 0.1		W	< 0.1	
Cs	3.80	0.14	Te	< 0.1		Re	0.00	0.00
Co	0.90	0.00	Ba	303.00	2.83	Tl	0.36	0.03
Eu	0.55	0.07	La	29.65	0.07	Pb	11.70	0.14
Bi	0.38	0.01	Ce	47.90	0.14	Th	16.35	0.92
Se	< 0.1		Pr	5.30	0.14	U	2.50	0.00
Zn	20.90	0.14	Nd	17.75	0.07			

References

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