

Supplementary material: Insights on the Permian tuff beds from the Saint-Affrique Basin (Massif Central, France): an integrated geochemical and geochronological study

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Samples		DEV21-1	PER21-3	PER21-4	CAN21-5	GAL21-6	GAL21-7	CAM20-3	LAT20-1
SiO ₂	wt%	74.04	70.80	77.85	72.51	75.48	73.07	75.73	42.99
Al_2O_3	wt%	14.45	15.60	12.43	14.61	11.96	10.73	10.19	11.28
Fe_2O_3	wt%	0.74	2.70	0.91	0.28	1.80	2.22	1.86	5.05
MnO	wt%	<l.d.< td=""><td><l.d.< td=""><td>0.015</td><td><l.d.< td=""><td>0.027</td><td>0.070</td><td>0.072</td><td>0.17</td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td>0.015</td><td><l.d.< td=""><td>0.027</td><td>0.070</td><td>0.072</td><td>0.17</td></l.d.<></td></l.d.<>	0.015	<l.d.< td=""><td>0.027</td><td>0.070</td><td>0.072</td><td>0.17</td></l.d.<>	0.027	0.070	0.072	0.17
MgO	wt%	0.28	0.53	0.21	0.14	3.17	2.77	1.09	6.05
CaO	wt%	0.08	0.04	0.10	<l.d.< td=""><td>0.28</td><td>2.61</td><td>2.38</td><td>10.33</td></l.d.<>	0.28	2.61	2.38	10.33
Na ₂ O	wt%	1.87	0.23	3.09	0.08	0.74	1.76	3.63	4.07
K ₂ O	wt%	6.48	5.61	3.85	10.20	0.62	0.77	0.59	1.47
TiO ₂	wt%	0.042	0.24	0.032	0.13	0.13	0.20	0.19	0.48
P_2O_5	wt%	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.22</td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.22</td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.22</td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.22</td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.22</td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td>0.22</td></l.d.<></td></l.d.<>	<l.d.< td=""><td>0.22</td></l.d.<>	0.22
LOI	wt%	2.50	4.69	2.04	1.73	5.89	5.66	3.66	17.15
As	ppm	4.86	2.81	2.84	1.25	5.20	5.07	1.76	4.67
Ba	ppm	150	223	92.0	134	352	2273	116	180
Be	ppm	4.01	3.30	2.62	0.87	1.73	1.60	1.17	1.37
Bi	ppm	0.35	0.18	0.95	0.70	0.18	0.24	0.37	0.29
Cd	ppm	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.14</td><td>0.12</td><td>0.12</td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.14</td><td>0.12</td><td>0.12</td></l.d.<></td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td><l.d.< td=""><td>0.14</td><td>0.12</td><td>0.12</td></l.d.<></td></l.d.<></td></l.d.<>	<l.d.< td=""><td><l.d.< td=""><td>0.14</td><td>0.12</td><td>0.12</td></l.d.<></td></l.d.<>	<l.d.< td=""><td>0.14</td><td>0.12</td><td>0.12</td></l.d.<>	0.14	0.12	0.12
Со	ppm	0.68	3.33	0.97	0.72	7.01	9.20	3.43	16.0
Cr	ppm	12.3	29.9	28.4	20.4	20.0	37.7	13.6	58.3
Cs	ppm	4.82	10.2	2.20	1.07	2.79	2.52	2.05	2.41
Cu	ppm	7.5	8.2	5.4	3.8	77.5	78.5	70.8	13.0
Ga	ppm	16.1	23.9	13.4	16.5	17.2	18.1	9.68	10.2
Ge	ppm	1.22	1.71	1.29	1.00	1.81	1.77	1.59	1.28
Hf	ppm	1.51	4.17	1.50	1.54	4.00	5.45	5.84	2.77
In	ppm	0.03	0.05	0.03	<l.d.< td=""><td>0.08</td><td>0.06</td><td>0.05</td><td>0.09</td></l.d.<>	0.08	0.06	0.05	0.09
Мо	ppm	0.50	<l.d.< td=""><td>1.64</td><td>0.99</td><td>0.68</td><td>1.63</td><td><l.d.< td=""><td><l.d.< td=""></l.d.<></td></l.d.<></td></l.d.<>	1.64	0.99	0.68	1.63	<l.d.< td=""><td><l.d.< td=""></l.d.<></td></l.d.<>	<l.d.< td=""></l.d.<>
Nb	ppm	4.98	23.7	7.37	8.28	13.3	11.4	9.79	8.91
Ni	ppm	2.9	7.7	3.3	<l.d.< td=""><td>4.7</td><td>7.7</td><td>5.9</td><td>19.2</td></l.d.<>	4.7	7.7	5.9	19.2
Pb	ppm	4.14	5.72	59.5	24.9	2.80	4.22	7.90	12.4
Rb	ppm	250	260	132	161	28.2	34.1	26.4	47.1
Sb	ppm	0.79	2.07	2.52	1.39	0.49	0.52	0.80	10.4
Sc	ppm	1.57	5.05	1.06	2.44	12.73	7.53	9.65	27.58
Sn	ppm	5.88	7.02	6.05	4.63	6.31	4.52	5.36	2.65
Sr	ppm	17.9	43.5	22.1	27.9	28.9	85.4	64.3	328
Ta	ppm	1.38	1.78	1.35	1.55	1.39	1.17	1.16	0.84
Th	ppm	6.09	11.9	6.24	7.88	15.3	12.9	13.2	17.2
U	ppm	2.04	3.31	2.33	2.30	3.72	3.85	3.23	2.16
V	ppm	6.4	25.3	4.2	12.2	10.9	20.0	14.7	78.3
W	ppm	0.86	4.20	1.63	1.48	0.95	1.84	0.82	1.70

Supplementary Table S1. Whole-rock geochemistry data from the Saint-Affrique Basin tuffs

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32.2

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ppm

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33.8

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Samples		DFV21-1	PFR21-3	PFR21-4	CAN21-5	GAI 21-6	GAI 21-7	CAM20-3	I AT20-1
72		14.0	20.4	22.0	<i d<="" td=""><td>177</td><td>150</td><td>62.5</td><td>42.7</td></i>	177	150	62.5	42.7
ZII	ppm	14.9	38.4	23.8	<l.d.< td=""><td>177</td><td>159</td><td>62.5</td><td>42.7</td></l.d.<>	177	159	62.5	42.7
Zr	ppm	29.8	111	33.0	38.2	93.3	179	186	165
La	ppm	5.33	24.8	12.8	27.1	22.8	28.2	23.8	26.6
Ce	ppm	8.95	45.6	25.2	34.4	56.0	62.2	56.6	54.8
Pr	ppm	0.986	4.64	2.98	5.61	7.47	7.78	7.25	6.65
Nd	ppm	3.60	14.8	11.1	20.2	30.6	30.5	29.5	26.5
Sm	ppm	0.845	2.31	2.05	3.91	7.87	6.71	7.23	6.38
Eu	ppm	0.249	0.400	0.371	0.745	0.405	0.856	0.545	1.38
Gd	ppm	0.710	1.52	1.26	2.66	6.63	5.86	6.11	5.95
Tb	ppm	0.125	0.218	0.148	0.324	1.13	0.948	0.957	0.969
Dy	ppm	0.723	1.21	0.633	1.52	7.28	5.85	5.75	5.96
Но	ppm	0.143	0.225	0.103	0.265	1.50	1.24	1.16	1.19
Er	ppm	0.365	0.604	0.238	0.645	4.14	3.35	3.22	3.20
Tm	ppm	0.0535	0.0948	0.0321	0.0897	0.648	0.517	0.499	0.483
Yb	ppm	0.343	0.657	0.198	0.556	4.28	3.41	3.19	3.10
Lu	ppm	0.047	0.099	0.028	0.080	0.623	0.509	0.470	0.468
Eu/Eu*		0.98	0.65	0.70	0.70	0.17	0.42	0.25	0.69
∑REE		22.47	97.15	57.17	98.04	151.38	157.88	146.25	143.56

Supplementary Table S1. (continued)

Supplementary Table S2. Operating conditions for the LA–ICP–MS equipment for the zircon U–Pb LA–ICP–MS dating

Laboratory and sample preparation	
Laboratory name	GeOHeLiS Analytical Platform, OSUR, Univ Rennes 1, France
Sample type/mineral	Zircon
Sample preparation	Crushed sample, grains mounted in epoxy puck
Imaging	CL: RELION CL instrument, Olympus Microscope BX51WI, Leica
	Color Camera DFC 420C
Laser ablation system	
Make, Model and type	ESI NWR193UC, Excimer
Ablation cell	ESI NWR TwoVol2
Laser wavelength	193 nm
Pulse width	<5 ns
Fluence	7.4 J/cm ²
Repetition rate	3 Hz
Spot size	30 μm
Sampling mode/pattern	Single spot

(continued on next page)

Supplementary Table S2. (continued)

Laboratory and sample preparation	
Carrier gas	100% He, Ar make-up gas and N2 (3 ml/mn) combined using in-house smoothing device
Background collection	20 s
Ablation duration	60 s
Wash-out delay	15 s
Carrier gas flow (He)	0.76 l/min
ICP-MS Instrument	
Make, Model and type	Agilent 7700×, Q-ICP-MS
Sample introduction	Via conventional tubing
RF power	1350 W
Sampler, skimmer cones	Ni
Extraction lenses	X type
Make-up gas flow (Ar)	0.75 l/min
Detection system	Single collector secondary electron multiplier
Data acquisition protocol	Time-resolved analysis
Scanning mode	Peak hopping, one point per peak
Detector mode	Pulse counting, dead time correction applied, and analog mode when signal intensity > $\sim 10^6$ cps
Masses measured	²⁰⁴ (Hg + Pb), ²⁰⁶ Pb, ²⁰⁷ Pb, ²⁰⁸ Pb, ²³² Th, ²³⁸ U
Integration time per peak	10–30 ms (²⁰⁷ Pb)
Sensitivity/Efficiency	23,000 cps/ppm Pb (50 μm, 10 Hz)
Data Processing	
Gas blank	20 s on-peak
Calibration strategy	GJ1 zircon standard used as primary reference material, Plešovice used as secondary reference material (quality control)
Common-Pb correction, composition and uncertainty	No common-Pb correction
Reference Material info	GJ1 [Jackson et al., 2004], Plešovice [Sláma et al., 2008]
Data processing package	Iolite [Paton et al., 2010]
Uncertainty level and propagation	Ages are quoted at 2 sigma absolute, propagation is by quadratic addition according to Horstwood et al. [2016]. Reproducibility and age uncertainty of reference material are propagated.
Quality control/validation	Plešovice: concordia age = 332 ± 4 Ma (N = 7; MSWD = 2.1)

Supplementary Table S3. LA-ICP-MS U-Pb data and LA-MC-ICP-MS Lu-Hf data for zircon extracted from tuff samples from the Saint-Affrique Basin. $f206_c\% = (^{207}\text{Pb}/^{206}\text{Pb}_m - ^{207}\text{Pb}/^{206}\text{Pb}^*)/(^{207}\text{Pb}/^{206}\text{Pb}_C - ^{207}\text{Pb}/^{206}\text{Pb}^*) \times 100.^{207}\text{Pb}/^{206}\text{Pb}_m$ is the measured ratio; $^{207}\text{Pb}/^{206}\text{Pb}_C$ is the common Pb ratio calculated at the age of the grain following the Pb evolution model of Stacey and Kramers [1975]

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P	TDM (Ma)				1298	1263	1259	1287		1256	697T		1229				1283		1257	1266		1269 13			1580						1493	1444	1462	1506	1479	0.014	1560		1549	1549								1498	
Correcte	eHfi CHUR				-0.3	0.2	0.3	-0.1		0.3	7'0-		0.8				-0.1		0.3	0.0	4	0.1 0.2			-4.8						-3.4	30	-2.9	-3.6	P C-	ţ	-4.5		43	, 4 W								-3.5	
Age	.76Hf/177Hfi				0.28260	0.28262	0.28262	0.28260		0.28262	N-2826U		0.28263				0.28261		0.28262	0 28261		0.28261 0.00001			0.28247						0.28251	1 2025	0.28253	0.28251	0 28254	104040	0.28248		0.28249	0.28249								0.28251	-
-	± 2s 1				0.00003	0.00003	0.0003	0.00003		0.00004	5000010		0.00002				00003		0.00003	10003	-				0.00003						0.00004	20000	0.0003	0.00003	00003		0.00002		00002	0.00003								0.00004	
я	Hf/177Hf				0.28261 (0.28262 (0.28262 (0.28261 (0.28262 (19787.0		0.28263 (0.28261 0		0.28262 (1 28262		1.28262			0.28248 (0.28253 (93565	0.28253 0	0.28252 (1 28256	00000	0.28249 (0.28250 0	0.28250 0								0.28253 (
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	176Yb				0.0	0.0	0.0	0.0		3.9			0.0				0.0		0.0	Ċ	5	anne 0.0 se 0.0			0.0						0.0	Ċ	0.0	0.0	00	5	0.0		0.0	0.0								0.0	
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	207Pb/235	322	315	313 308	281	279	285	278	280	281	647	281	277	283	285	156	284	278	282	282	}		444	286	278	284	2/2	447	464	286	288	459 205	278	290	324 288	449	275	302	282	283	295 205	305	948	319	2661	281	505 780	275	
nt Ages	J 2se (abs)	11	13	51 E	13	= :	1 1	11	Ξ	= :	3 5	1 3	Π	11	= :	= ~	r II	Π	=	3 2	1		15	1	11	12	= :	11	17	=	=	8 5	1 1	11	= =	11	11	12	9 =	1 1	=	12	5 12	12	85	11 1	n =	: =	
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	2se (abs)	8	55	56 60	8	8 8	8 5	51	49	88	% F	22	99	09	13 t	8 6	6	S	8	2 6	5		11	5	50	63	5	ŧ 4	44	42	6	42	6 6	45	41	1 69	44	71	9 4	6 6	51	З ¥	; 4	121	26	40	41 41	43	
	07Pb/206Pb	753	359	308 217	291	323	320	285	288	291	125 170	278	248	311	366	1591	320	299	339	341			503	318	348	436	306	524	600	289	270	458	316	366	6/9 345	478	292	508	316	305	402	528	961	582	2646	296 4°C	309	298	
	Rho	0.80	0.91	0.90	0.91	0:00	06.0	0.92	0.92	10.0	16.0	0.92	06.0	0.89	0.92	10.0	0.87	0.91	0.91	0.90	100		0.95	0.95	0.98	0.92	0.96	56°0	96.0	0.95	0.95	0.95 0.06	0.95	0.95	26.0 26.0	0.95	0.96	0.91	96.0	0.95	0.91	16.0 0.96	0.95	0.63	0.97	0.96	0.95 0.95	0.96	
plot	2se%	4.4	4.0	4.0	4.1	4.1	4.0	4.0	3.9	0.4	0.4	4.0	4.2	4.0	4.0	1.4	14	3.9	4.1	0.4 0.4	!		40	4.0	4.2	4.5	0,4 6	6.0	4.1	4.0	3.9	6 C C	4.0	4.0	5 C C	5 6	4.0	4.6	104	6.6	4.0	1.4	14	4.3	3.9	9.5 7 A	1.4	4.0	
Wetherill	206Pb/238U	0.04422	0.05089	0.05100	0.04482	0.04368	0.04445	0.04462	0.04475	0.04480	0.04357	0.04415	0.04378	0.04417	0.04456	0.01269	0.04551	0.04469	0.04524	0.04450	00000		0.06733	0.04465	0.04301	0.04265	0.04389	4/640'0	0.07038	0.04538	0.04610	0.0/416	0.04393	0.04475	0.04438	0.07023	0.04310	0.04379	0.04406	0.04436	0.04439	0.04416 0.15698	0.15532	0.04476	0.51179	0.04390	0.04386 0.04386	0.04326	
Data foi	2se%	5.5	4.4	4.4	4.5	45	4 4	4.3	4.3	6 4 6 4	4.4	64	4.7	4.5	4.4	1.4	£ 4	4.3	4.5	4.4	2		6.4	4.2	4.3	4.8	47	4.2	4.2	4.2	4.1	4.2	42	4.2	1.4	4.2	4.2	5.0	14	4.1	4.4	4.4	1 64	6.8	4.0	4.1	0 F4	42	
	7Pb/235U	0.3734	0.3638	0.3550	0.3185	0.3166	0.3237	0.3144	0.3174	0.3190	0.3201	0.3185	0.3134	0.3210	0.3243	0.3005	0.3230	0.3144	0.3203	0.3204			0 5479	0.3251	0.3150	0.3233	0.3153	0.5525	0.5793	0.3255	0.3279	0.5716	0.3146	0.3302	0.3776 0.3276	0.5554	0.3109	0.3457	0.3204	0.3209	0.3369	0.3500	1.5430	0.3690	12.7440	0.3184	0.3499 0.3181	0.3107	
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	Th/U	0.18	0.16	0.15	0.18	0.21	0.16	0.19	0.20	0.20	0.20	0.18	0.17	0.14	0.18	0.10	0.16	0.19	0.20	0.19 0.19			0.18	0.17	0.22	0.20	0.16	0.03	0.32	0.21	0.28	0.04	0.21	0.27	0.51	0.20	0.23	0.47	0.00	0.32	0.62	0.31	0.06	0.20	0.51	0.21	0.22	0.20	
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	f206c	1.559	0.115	0.000	0.023	0.135	0.114	600'0	0.017	0.023	0.148	0.000	0.000	0.092	0.248	1.210	0.095	0:050	0.156	2/1/0 0 306			0.424	0.106	0.222	0.493	0.082	0.278	0.527	0.009	0.000	0.000	0.110	0.245	0.177	0.126	0.057	0.702	0.110	0.072	0.359	0.764	0.125	0.934	0.000	0.055	CT0.0	0.070	
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	5.6	5.7	5.7	5.7	5.6	5.7	5.8	5.7	5.6		5.7	5.9	5.8	5.7	5.8	5.7	5.7	5.6	5.8	5.7
	0.16802	0.09454	0.09665	0.09656	0.38381	0.09514	0.35806	0.05165	0.14108		0.05423	0.04183	0.04077	0.04388	0.04188	0.03898	0.04186	0.04193	0.04272	0.03633
	7.5	7.6	7.5	7.8	7.5	7.5	7.5	7.6	7.5		7.5	7.6	7.6	7.6	7.6	7.5	7.6	7.5	8.0	7.5
	1.7248	0.8411	0.8172	0.8220	6.8243	1.0015	6.2317	0.9291	2.2773		0.4057	0.2969	0.2960	0.3165	0.3059	0.3867	0.3019	0.3530	0.3427	0.3659
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	Zr1	Zr2	Zr3	Zr4	Zr5	Zr6	Zr8	Zr9	Zr10		Zr1	Zr2	Zr3	Zr4	Zr5	Zr6	Zr7	Zr8	Zr9	Zr10
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% conc = Percentage of concordance: if Age206Pb/238U < 1000 Ma concordance $\% = (Age^{206} Pb/^{238} U/Age^{207} Pb/^{235} U)^* 100$; else concordance % $= (Age^{207}Pb/^{235}U/Age^{207}Pb/^{206}Pb) * 100.$

Uncertainties on ages include secondary standard uncertainty propagation as proposed by Horstwood et al. [2016].

Supplementary Table S3. (continued)



Supplementary Figure S1. Wetherill Concordia diagrams displaying all the analyses obtained for samples Lat20-1 and Cam20-3.

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