Ganade, C.E., et al., 2022, Recurrent tectonic activity in northeastern Brazil during Pangea breakup: Constraints from U-Pb carbonate dating: Geology, v. 50, https://doi.org/10.1130/G50032.1

Supplementary information for

Recurrent tectonic activity in NE Brazil during Pangea breakup: constraints from U-Pb carbonate dating

Carlos E. Ganade[(1)]; Caue R. Cioffi[(1)]; João Pacífico Machado[(1)]; Tiago Miranda[(2)]; Leonardo B. Lopes[(1)]; Roberto Weinberg [(3)]; Maria Alcione Celestino[(2)]; Bruno Carvalho[(4)]; Marcel Guillong[(5)]; Nick M. W. Roberts[(6)]

[(1)]Geological Survey of Brazil, Rio de Janeiro, Brazil

[(2)]Federal University of Pernambuco, Recife, Brazil

[(3)]Monash University, Clayton, Australia

[(4)]CENPES-PETROBRAS, Rio de Janeiro, Brazil

[(5)]ETH, Zurich, Switzerland

[(6)]Geochronology and Tracers Facility, British Geological Survey, Nottingham, United Kingdom

U-Pb carbonate analytical method

U-Pb analyses were conducted by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) in two facilities with slightly distinct approaches. Sample AR-1 was analyzed at the Geochronology and Tracers Facility, British Geological Survey, UK, while the remaining samples (AR-2, RP-1, RP-2, JB-1, JB-2, JB-3 and JB-4) were analyzed at the Department of Earth Sciences, ETH Zürich, CH. The specific parameters and procedures of each laboratory are tabulated below. Further information, including single spot data, samples and reference materials information, and Tera-Wasserburg plots are available within the U-Pb data tables in the Supplementary Material.

Laboratory & Sample Preparation	
Laboratory name	Geochronology & Tracers Facility, NERC Isotope Geosciences Laboratory
Sample type/mineral	Carbonate
Sample preparation	Sample blocks mounted in epoxy resin
Imaging	Optical only
Laser ablation system	
Make, Model & type	ESI/New Wave Research, UP193UC
Ablation cell & volume	NWR TV2
Laser wavelength (nm)	193nm
Pulse width (ns)	4ns
Fluence (J.cm^{-2})	~7 J/cm^{-2}
Repetition rate (Hz)	10 Hz
Spot size (□m)	100 µm
Sampling mode / pattern	Static spot
Carrier gas	100% He, Ar make-up gas from DSN-100 combined using a Y-piece 50% along sample line.
Ablation duration (secs)	30 secs
Cell carrier gas flow (l/min)	0.7 l/min
ICP-MS Instrument	
Make, Model & type	Nu Instruments, Attom, SC-ICP-MS
Sample introduction	Ablation aerosol
RF power (W)	1300 W
Make-up gas flow	0.7 l/min Ar
Detection system	Single Mascom SEM

Masses measured	202, 204, 206, 207, 208, 232, 238
Integration time per peak (ms)	Dwell times of 200 µs to 1000 µs per peak, 100 sweeps per integration.
Total integration time per reading (secs)	0.35 sec (should represent the time resolution of the data)
Sensitvity / Efficiency (%, element)	~0.2% U
IC Dead time (ns)	15 ns
Data Processing	
Gas blank	30 second on-peak zero subtracted
Calibration strategy	NIST614 glass standard as primary reference material for drift and 207Pb/206Pb ratios (see Roberts et al., 2017); WC-1 carbonate reference material for matrix matching of 206Pb/238U (anchored to initial Pb composition of 0.85, see Roberts et al., 2017).
Reference Material info	Concentrations use WC1 (Roberts et al., 2017) 207Pb/206Pb: NIST614 (Woodhead and Hergt, 2001) 206Pb/238U: WC1 (Roberts et al., 2017) Validation with ASH15D (Nuriel et al., 2021)
Data processing package used / Correction for LIEF	In-house spreadsheet data processing after initial signal integration using Nu Instruments TRA software. No LIEF correction (mean of uncorrected ratios used). IsoplotR (Vermeesch, 2018) for isochrons, intercept ages, and initial Pb compositions.
Mass discrimination	Standard sample bracketing
Common-Pb correction, composition and uncertainty	None applied. Ages calculated from regressions used in Tera-Wasserburg plots.
Uncertainty level & propagation	Ages are quoted at 2σ absolute, propagation is by quadratic addition. Excess variance of reference material propagated into sample data. Systematic uncertainties include age uncertainty of reference material.
Quality control / Validation	ASH15D: 3.086 ± 0.087 Ma (n=30; MSWD = 1.6) (without systematic uncertainties propagated)

Laboratory & Sample Preparation	
Laboratory name	Department of Earth Sciences, ETH Zürich
Sample type/mineral	Carbonate
Sample preparation	Polished thick sections and chips mounted in epoxy
Imaging	CL8200 Mk5-2 Optical Cathodoluminescence System
Laser ablation system	
Make, Model & type	ASI (Resonetics) RESOlution S155
Ablation cell & volume	Laurin Technic, two-volume cell, effective volume ~1 cm3
Laser wavelength (nm)	193nm
Pulse width (ns)	25ns
Fluence (J.cm^{-2})	~1.8 J/cm^{-2}
Repetition rate (Hz)	5.0 (for 110 µm spot diameter) or 7.4 Hz (for 163 µm spot diameter)
Spot size (□m)	110 µm or 163 µm
Sampling mode / pattern	Static spot
Carrier gas	100% He in the cell, Ar make-up gas combined in cell above, ablation in funnel.
Ablation duration (secs)	40 secs
Cell carrier gas flow (l/min)	0.5 l/min
ICP-MS Instrument	
Make, Model & type	Thermo Element XR, sector-field single collector ICP-MS with high capacity interface pump
Sample introduction	Ablation aerosol
RF power (W)	1350-1550 W (optimized daily)
Make-up gas flow	0.90 - 1.05 l/min Ar (optimized daily), 2 ml/min N2
Detection system	Triple (pulse counting, analog, Faraday), cross-calibrated daily

	with 238U, fixed analogue counting factor (ACF) value, all isotopes usually in pulse-counting only (<5 Mcps)
Masses measured	202, 204, 206, 207, 208, 232, 235, 238
Integration time per peak (ms)	11 ms for all masses except 206, 207 (50 ms)
Total integration time per reading (secs)	0.174 sec
Sensitvity / Efficiency (%, element)	~1% U
IC Dead time (ns)	25 ns
Data Processing	
Gas blank	20 s
Calibration strategy	NIST614 glass standard as primary reference material for drift and 207Pb/206Pb ratios (see Roberts et al., 2017); WC-1 carbonate reference material for matrix matching of 206Pb/238U (anchored to initial Pb composition of 0.85, see Roberts et al., 2017); ASH-15D (Nuriel et al., 2021) and JT (Guillong et al., 2020) calcite validation reference materials for assessing accuracy and repeatability.
Reference Material info	NIST614 (concentration data of Jochum et al., 2011, Pb isotopes of Baker et al., 2004); WC-1 (Roberts et al., 2017); ASH-15-D (Nuriel et al., 2021); JT (Guillong et al., 2020) ; B6 (Pagel et al., 2018).
Data processing package used / Correction for LIEF	Iolite 2.5, VisualAge for integration, interval selection, and gas blank correction only. In-house spreadsheet data processing. IsoplotR (Vermeesch, 2018) for isochrons, intercept ages, and initial Pb compositions. No separate LIEF correction. 238U/206Pb corrected to primary. RM assumed to have similar LIEF as samples.
Mass discrimination	Normalized to reference material (sample-standard bracketing)
Common-Pb correction, composition and uncertainty	Not applied. Ages calculated by linear regressions in Tera-Wasserburg concordia plots.
Uncertainty level & propagation	Lower intercept ages are quoted at 2s absolute, propagation is by quadratic addition. Counting statistics uncertainties are propagated to the 207Pb/206Pb ratios, together with the

	uncertainty of the primary RM value and the uncertainty of repeated measurements. The uncertainty value for lower intercept isochron ages includes uncertainties from the primary RM and unsystematic uncertainties, estimated in this work to be 2.5%.
Quality control / Validation	ASH-15-D: Tera-Wasserburg intercept age: 2.86 ± 0.153 Ma (2s, MSWD = 2, n = 22 including internal and 2.5% total external uncertainty) without 230Th disequilibrium correction. JT: Tera-Wasserburg intercept age: 13.07 ± 0.49 Ma (2s, MSWD = 1.5, n = 22 including internal and 2.5% total external uncertainty) B6: Tera-Wasserburg intercept age: 40.7 ± 1.25 Ma (2s, MSWD = 1.5, n = 22 including internal and 2.5% total external uncertainty) Systematic uncertainty for propagation is 2.5 % (2s)

References

Baker, J., Peate, D., Waight, T., Meyzen, C., 2004. Pb isotopic analysis of standards and samples using a ^{207}Pb-^{204}Pb double spike and thallium to correct for mass bias with a double-focusing MC-ICP-MS. Chemical Geology 211, 275-303.

Guillong, M., Wotzlaw, J-F., Looser, N., Laurent, O., 2020. Evaluating the reliability of U-Pb laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) carbonate geochronology: matrix issues and a potential calcite validation reference material. Geochronology 2, 155-167.

Jochum, K.P., Weis, U., Stoll, B., Kuzmin, D., Yang, Q., Raczek, I., Jacob, D.E., Stracke, A., Birbaum, K., Frick, D.A., Günther, D., Enzweiler, J., 2011. Determination of reference values for NIST SRM 610-617 glasses following ISO guidelines. Geostandards and Geoanalytical Research 35, 397-429.

Nuriel, P., Wotzlaw, J-F., Ovtcharova, M., Vaks, A., Stremtan, C., Sala, M., Roberts, N.M.W., Kylander-Clark, A.R.C., 2021. The use of ASH-15 flowstone as matrix-matched reference material for laser-ablation U-Pb geochronology of calcite. Geochronology 3, 35-47.

Pagel, M., Bonifacie, M., Schneider, D., Gautheron, C., Brigaud, B., Calmels, D., Cros, A., Saint-Bezar, B., Landrein, P., Sutcliffe, C., Davis, D., Chaduteau, C., 2018. Improving paleohydrological and diagenetic reconstructions in calcite veins and breccia of a

sedimentary basin by combining Δ_{47} temperature, $\delta^{18}O_{water}$ and U-Pb age. Chemical Geology 481, 1-17.

Roberts, N.M.W., Rasbury, E.T., Parrish, R.R., Smith, C.J., Horstwood, M.S.A., Condon, D.J., 2017. A calcite reference material for LA-ICP-MS U-Pb geochronology. Geochemistry, Geophysics, Geosystems 18, 2807-2814.

Vermeesch, P., 2018. IsoplotR: A free and open toolbox for geochronology. Geoscience Frontiers 9, 1479-1493.

Woodhead, J.D., Hergt, J.M., 2001. Strontium, neodymium and lead analyses of NIST glass certified reference materials: SRM 610, 612, 614. Geostandards Newsletter 25, 261-266.