1 **Integrating Zircon Trace Element Geochemistry and High-Precision**

2 **U-Pb Zircon Geochronology to Resolve Timing and Petrogenesis of**

3 **the late Ediacaran-Cambrian Wichita Igneous Province, Southern**

4 **Oklahoma Aulacogen, USA**

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6 **Supplementary Material**

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26 **Analytical Methods and Results**

27 *Sample preparation*

28   An abundant population of relatively small (approximately 100-200 μm in long

29 dimension), equant to prismatic zircon crystals was separated from each hand sample by

30 conventional density and magnetic methods. The entire zircon separate was placed in a muffle

31 furnace at 900°C for 60 hours in quartz beakers to anneal minor radiation damage; annealing

32 enhances cathodoluminescence (CL) emission (Nasdala et al., 2002), promotes more

33 reproducible interelement fractionation during laser ablation inductively coupled plasma mass

34 spectrometry (LA-ICPMS) (Allen and Campbell, 2012), and prepares the crystals for subsequent

35 chemical abrasion (Mattinson, 2005). Following annealing, individual grains were hand-picked

36 and mounted, polished and imaged by CL on a scanning electron microscope. From these

37 compiled images, the locations of spot analyses for LA-ICP-MS were selected.

38

39 *LA-ICPMS analysis*

40   LA-ICPMS analysis utilized an X-Series II quadrupole ICPMS and New Wave Research

41 UP-213 Nd:YAG UV (213 nm) laser ablation system. In-house analytical protocols, standard

42 materials, and data reduction software were used for acquisition and calibration of U-Pb dates

43 and a suite of high field strength elements (HFSE) and rare earth elements (REE). Zircon was

44 ablated with a laser raster 15 μm wide using fluence and pulse rates of ~5 J/cm^2 and 20 Hz,

45 during a 45 second analysis (15 sec gas blank, 30 sec ablation) that excavated a line ~5 μm deep.

46 Ablated material was carried by a 1.15 L/min He gas stream to the nebulizer flow of the plasma.

47 Quadrupole dwell times were 5 ms for Si and Zr, 200 ms for ^{49}Ti and ^{207}Pb, 80 ms for ^{206}Pb, 40

48 ms for ^{202}Hg, ^{204}Pb, ^{208}Pb, ^{232}Th, and ^{238}U and 10 ms for all other HFSE and REE; total sweep

49 duration is 950 ms. Background count rates for each analyte were obtained prior to each line

50 analysis and subtracted from the raw count rate for each analyte. For concentration calculations,

51 background-subtracted count rates for each analyte were internally normalized to ^{29}Si and

52 calibrated with respect to NIST SRM-610 and -612 glasses as the primary standards. Ablation

53 pits that appeared to have intersected glass or mineral inclusions were identified based on Ti and

54 P signal excursions, and data from those analyses were generally discarded. U-Pb dates from

55 these analyses were considered valid if the U-Pb ratios appeared to have been unaffected by the

56 inclusions. Signals at mass 204 were normally indistinguishable from zero following subtraction

57   of mercury backgrounds measured during the gas blank (<100 cps ^{202}Hg), and thus dates are

58   reported without common Pb correction. Rare analyses that appeared contaminated by common

59   Pb were rejected based on mass 204 greater than baseline. Additionally, elements sensitive to

60   micro-inclusions of apatite, monazite, rutile, oxides, and melt inclusions (e.g., P, Ti, LREE) were

61   monitored if there were excursions in these elements, these analyses were rejected and not

62   included for interpretations in this study. Temperature was calculated from the Ti-in-zircon

63   thermometer (Watson et al., 2006). Because there are no constraints on the activity of TiO_2 in the

64   source rocks, an average value in crustal rocks of 0.8 was used and an activity of SiO_2 of 1.0 was

65   used based on the presence of quartz in all samples (Hayden and Watson, 2007).

66   For U-Pb and ^{207}Pb/^{206}Pb dates, instrumental fractionation of the background-subtracted

67   ratios was corrected and dates were calibrated with respect to interspersed measurements of

68   zircon standards and reference materials. The primary standard Plešovice zircon (Sláma et al.,

69   2008) was used to monitor time-dependent instrumental fractionation based on two analyses for

70   every 10 analyses of unknown zircon. A polynomial fit to the primary standard analyses versus

71   time yields each sample-specific fractionation factor. A secondary bias correction was

72   subsequently applied to unknowns on the basis of the residual age bias as a function of

73   radiogenic Pb count rate in standard materials, including Seiland, Zirconia, and Plesovice zircon,

74   or similar materials of known age and variable Pb content. A polynomial fit to the secondary

75   standard analyses with Pb count rate yields each sample-specific bias correction. Radiogenic

76   isotope ratio and age error propagation for all analyses includes uncertainty contributions from

77   counting statistics and background subtraction. Because the detrital zircon analyses are

78   interpreted individually, uncertainties from the standard calibrations are propagated into the

79   errors on each date. These uncertainties are the local standard deviations of the polynomial fits to

80   the interspersed primary standard measurements versus time for the time-dependent, relatively

81   larger U/Pb fractionation factor, and the standard errors of the means of the consistently time-

82   invariant and smaller ^{207}Pb/^{206}Pb fractionation factor. These uncertainties are ~2% (2σ) for

83   ^{206}Pb/^{238}U and ~1% (2σ) for ^{207}Pb/^{206}Pb. Additional details of methodology and reproducibility

84   are reported in Rivera et al. (2013).

85

86   ***ID-TIMS analysis***

87   U-Pb geochronology methods for isotope dilution thermal ionization mass spectrometry

88   follow those previously published by Davydov et al. (2010) and Schmitz and Davydov (2012).

89   All analyses were undertaken on crystals previously mounted, polished and imaged by

90   cathodoluminescence (CL), and analyzed by LA-ICPMS. Zircon crystals were subjected to a

91   modified version of the chemical abrasion method of Mattinson (2005), whereby single crystal

92   fragments plucked from grain mounts were individually abraded in a single step with

93   concentrated HF at 190°C for 12 hours. Zircon fragments were dissolved in Parr bombs at 220

94   °C for 48 h. Dissolved zircon solutions were subsequently dried down, redissolved in 100 μl 6 N

95   HCl and converted to chlorides in Parr bombs at 180 °C for 12 h, after which solutions were

96   dried again and brought up in 50 μl 3 N HCl. U and Pb were isolated by anion exchange column

97   chromatography using 50 μl columns and AG-1 X8 resin [200–400 mesh, chloride form

98   (Eichrom); Krogh, 1973].

99   The U-Pb aliquot was loaded in a silica gel emitter (Gerstenberger & Haase, 1997) to an

100   outgassed, zone-refined Re filament. Isotopic determinations were performed using an IsotopX

101   PhoeniX-62 TIMS. A correction for mass-dependent Pb fractionation was applied based on

102   repeated measurements of NBS 982 (Catanzaro et al., 1968) Pb on both the Daly ion counter

103    $[0.16 \pm 0.03 \%)$ amu^{-1}; 1s] and the Faraday cups $[0.10 \times (1 \pm 0.02 \%)$ amu^{-1}; 1s]. Uranium was

104   run as an oxide (UO_2) and measured in static mode on Faraday detectors equipped with 10^{12} Ω

105   resistors. The U mass fractionation for the same analyses was calculated using the $^{233}U/^{235}U$ ratio

106   of the double spike solution (0.99506 % ± 0.01 %, 1s).

107   U-Pb dates and uncertainties for each analysis were calculated using the algorithms of

108   Schmitz and Schoene (2007), the U decay constants of Jaffey et al. (1971), and a value of

109    $^{238}U/^{235}U = 137.88$. Uranium oxide measurements were corrected for isobaric interferences using

110   an $^{18}O/^{16}O$ value of 0.00206. Uncertainties are based upon non-systematic analytical errors,

111   including counting statistics, instrumental fractionation, tracer subtraction, and blank subtraction.

112   All non-radiogenic Pb was attributed to laboratory blank with a mean isotopic composition

113   determined by total procedural blank measurements. These error estimates should be considered

114   when comparing our $^{206}Pb/^{238}U$ dates with those from other laboratories that used tracer solutions

115   calibrated against the EARTHTIME gravimetric standards. When comparing our dates with

116   those derived from other decay schemes (e.g., $^{40}Ar/^{39}Ar$, ^{187}Re-^{187}Os), the uncertainties in tracer

117   calibration (0.05%; Condon et al., 2015; McLean et al., 2015) and U decay constants (0.108%;

118   Jaffey et al., 1971) should be added to the internal error in quadrature. Quoted errors for

119   calculated weighted means are thus of the form $\pm X(Y)[Z]$, where X is solely analytical

120   uncertainty, Y is the combined analytical and tracer uncertainty, and Z is the combined

121   analytical, tracer and ^{238}U decay constant uncertainty.

122   **G8 (Anorthosite from Glen Mountains Layered Complex):** CL-imaging of the 50

123   largest zircon crystals from this sample revealed a homogenous population of moderately

124   luminescent, oscillatory to sector zoned crystals. Nine grains were selected for CA-TIMS

125   analysis on the basis of the uniform, predominant CL pattern. Chemical abrasion in concentrated

126   HF at 190°C for 12 hours resulted in moderate dissolution of the zircon crystals. All eight

127   analyses are concordant and equivalent, with a weighted mean ^{206}Pb/^{238}U date of 532.49 ±

128   0.12(0.28)[0.62] Ma (MSWD = 0.93), which is interpreted as dating the crystallization of this

129   anorthosite.

130   **AE-389 (Rhyolite from the Arbuckle Mountains):** CL-imaging of the 100 largest

131   zircon crystals from this sample revealed a homogenous population of moderately luminescent,

132   oscillatory to sector zoned crystals. A lesser number of crystals have irregularly shaped,

133   relatively non-luminescent cores overgrown by aforementioned luminescent, oscillatory rims.

134   There are also a few zircon crystals that are poorly luminescent but are oscillatory zoned and are

135   considered to be xenocrysts. Eight grains were selected for CA-TIMS analysis on the basis of the

136   uniform, predominant CL pattern. Chemical abrasion in concentrated HF at 190°C for 12 hours

137   resulted in moderate dissolution of the zircon crystals. All eight analyses are concordant and

138   equivalent, with a weighted mean ^{206}Pb/^{238}U date of 539.20 ± 0.15(0.30)[0.64] Ma (MSWD =

139   0.54), which is interpreted as dating the eruption age of this rhyolite.

140 **JP-22 (Rhyolite flow from base of volcanic succession at Bally Mountain):**

141   CL-imaging of the 100 largest zircon crystals from this sample revealed a homogenous

142   population of moderately luminescent, oscillatory to sector zoned crystals. A lesser number of

143   crystals have irregularly shaped, relatively non-luminescent cores overgrown by aforementioned

144   luminescent, oscillatory rims. There are also a few zircon crystals that are poorly luminescent but

145   are oscillatory zoned and are considered to be xenocrysts. Eight grains were selected for CA-

146   TIMS analysis on the basis of the uniform, predominant CL pattern. Chemical abrasion in

147   concentrated HF at 190°C for 12 hours resulted in moderate dissolution of the zircon crystals.

148   All eight analyses are concordant and equivalent, with a weighted mean ^{206}Pb/^{238}U date of

149 530.98 ± 0.14 Ma(0.29)[0.62] Ma (MSWD = 0.94), which is interpreted as dating the eruption

150 age of this rhyolite.

JP-120 (Rhyolite flow from top of volcanic succession at Bally Mountain):

152 CL-imaging of the 100 largest zircon crystals from this sample revealed a homogenous

153 population of moderately luminescent, oscillatory to sector zoned crystals. A lesser number of

154 crystals have irregularly shaped, relatively non-luminescent cores overgrown by aforementioned

155 luminescent, oscillatory rims. There are also a few zircon crystals that are poorly luminescent but

156 are oscillatory zoned and are considered to be xenocrysts. Thirteen grains were selected for CA-

157 TIMS analysis on the basis of the uniform, predominant CL pattern. Chemical abrasion in

158 concentrated HF at 190°C for 12 hours resulted in moderate dissolution of the zircon crystals.

159 All thirteen analyses are concordant and equivalent, with a weighted mean $^{206}Pb/^{238}U$ date of

160 530.70 ± 0.12 Ma(0.28)[0.62] Ma (MSWD = 0.81), which is interpreted as dating the eruption

161 age of this rhyolite.

WP16-2 (Mt. Scott Granite):

163 CL-imaging of the 100 largest zircon crystals from this sample revealed a homogenous

164 population of moderately luminescent, oscillatory to sector zoned crystals. A lesser number of

165 crystals have irregularly shaped, relatively non-luminescent cores overgrown by aforementioned

166 luminescent, oscillatory rims. There are also a few zircon crystals that are poorly luminescent but

167 are oscillatory zoned and are considered to be xenocrysts. Eight grains were selected for CA-

168 TIMS analysis on the basis of the uniform, predominant CL pattern. Chemical abrasion in

169 concentrated HF at 190°C for 12 hours resulted in moderate dissolution of the zircon crystals.

170 All eight analyses are concordant and equivalent, with a weighted mean $^{206}Pb/^{238}U$ date of

171 530.45 ± 0.14 Ma(0.29)[0.62] Ma (MSWD = 0.69), which is interpreted as dating the

172 crystallization of this granite.

WP18-1 (Cache Granite):

174 CL-imaging of the 55 largest zircon crystals from this sample revealed a homogenous

175 population of moderately luminescent, oscillatory to sector zoned crystals. A lesser number of

176 crystals have irregularly shaped, relatively non-luminescent cores overgrown by aforementioned

177 luminescent, oscillatory rims. There are also a few zircon crystals that are poorly luminescent but

178 are oscillatory zoned and are considered to be xenocrysts. Seven grains were selected for CA-

179 TIMS analysis on the basis of the uniform, predominant CL pattern. Chemical abrasion in

180 concentrated HF at 190°C for 12 hours resulted in moderate dissolution of the zircon crystals.

181 All seven analyses are concordant and equivalent, with a weighted mean ^{206}Pb/^{238}U date of

182 530.61 ± 0.13 Ma(0.29)[0.62] Ma (MSWD = 0.12), which is interpreted as dating the

183 crystallization of this granite.

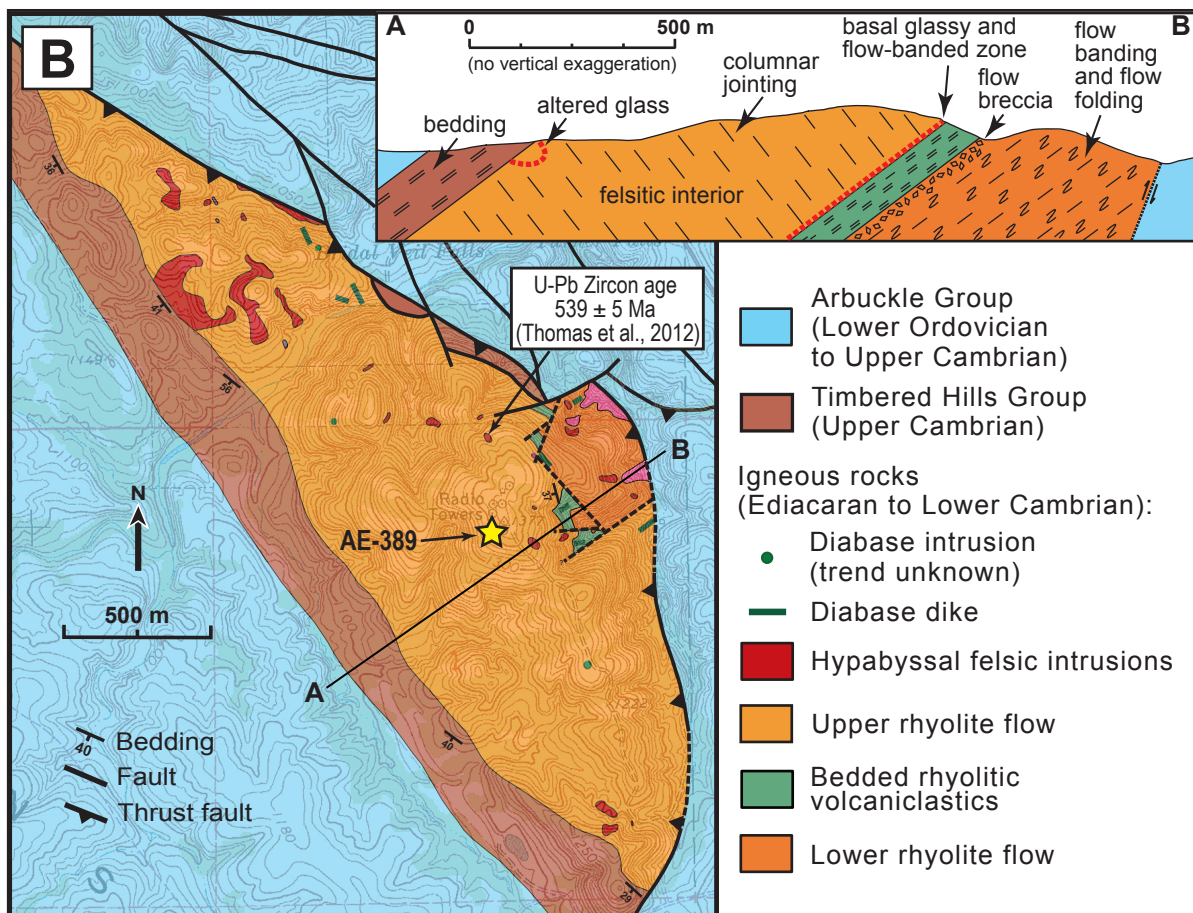184 **JPQ-71797 (Quanah Granite):**

185   CL-imaging of the 60 largest zircon crystals from this sample revealed a homogenous

186 population of moderately luminescent, oscillatory to sector zoned crystals. A lesser number of

187 crystals have irregularly shaped, relatively non-luminescent cores overgrown by aforementioned

188 luminescent, oscillatory rims. There are also a few zircon crystals that are poorly luminescent but

189 are oscillatory zoned and are considered to be xenocrysts. Eight grains were selected for CA-

190 TIMS analysis on the basis of the uniform, predominant CL pattern. Chemical abrasion in

191 concentrated HF at 190°C for 12 hours resulted in moderate dissolution of the zircon crystals.

192 Six of the eight analyses are concordant and equivalent, with a weighted mean ^{206}Pb/^{238}U date of

193 530.23 ± 0.14 Ma(0.29)[0.62] Ma (MSWD = 0.12), which is interpreted as dating the

194 crystallization of this granite.
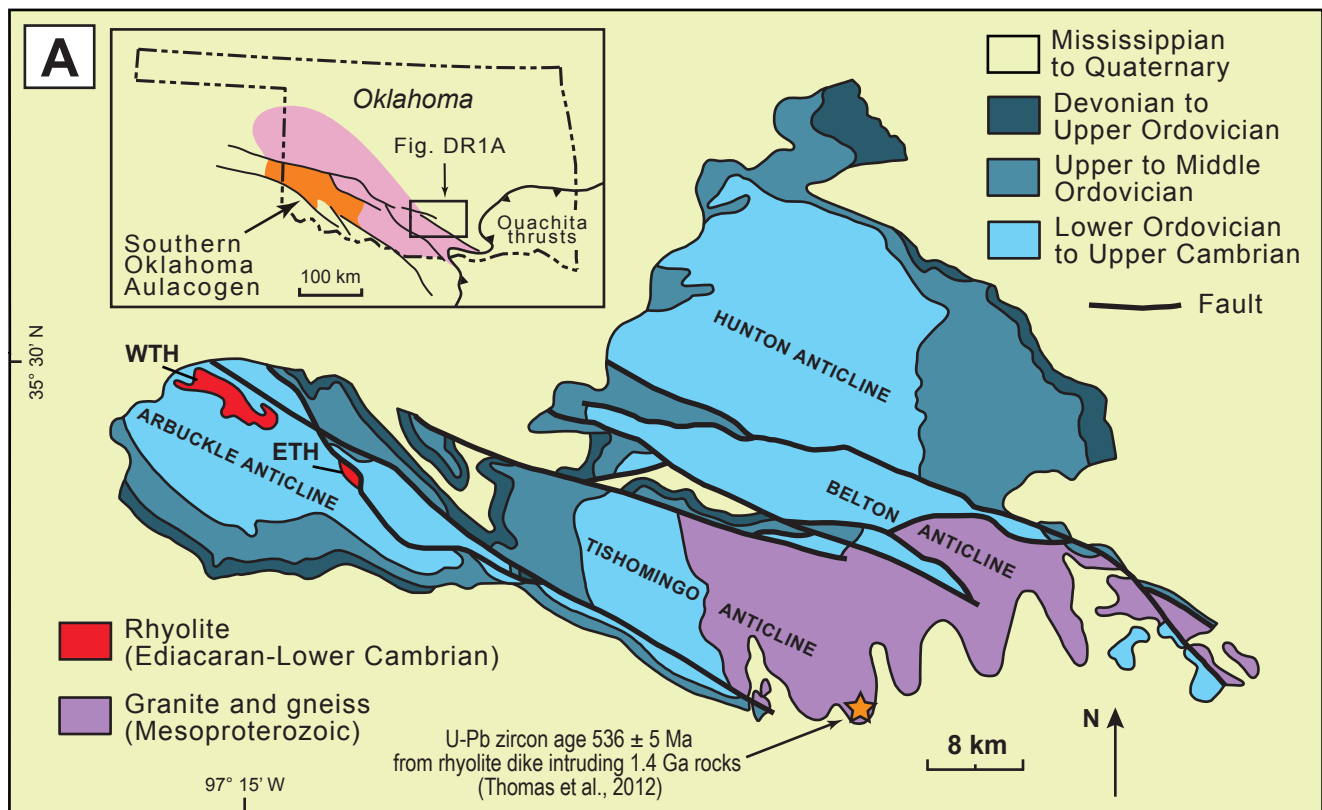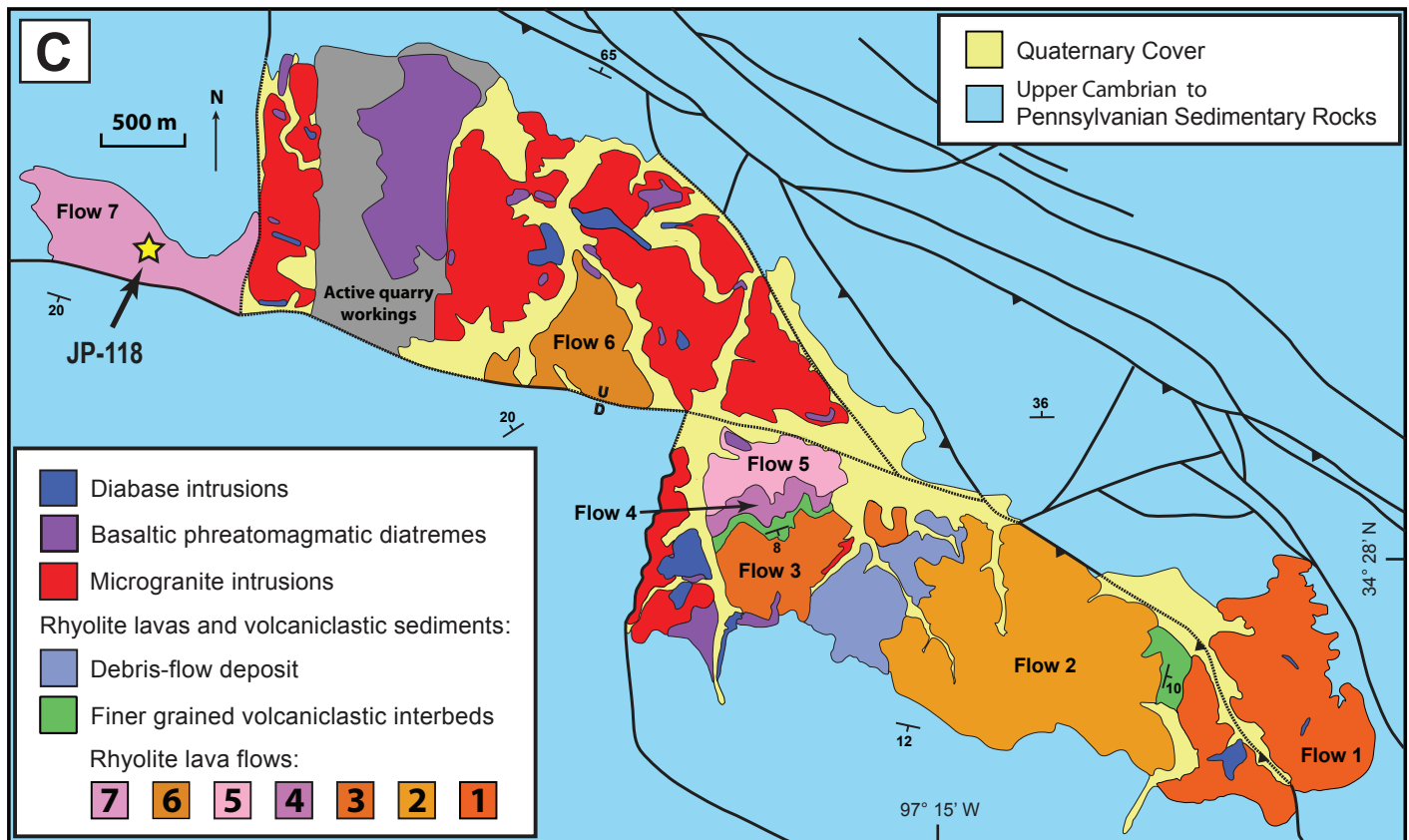
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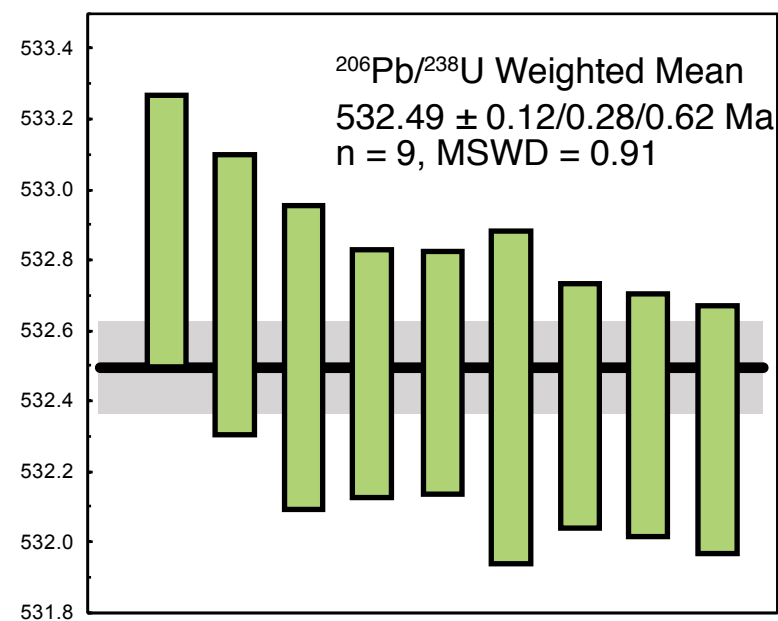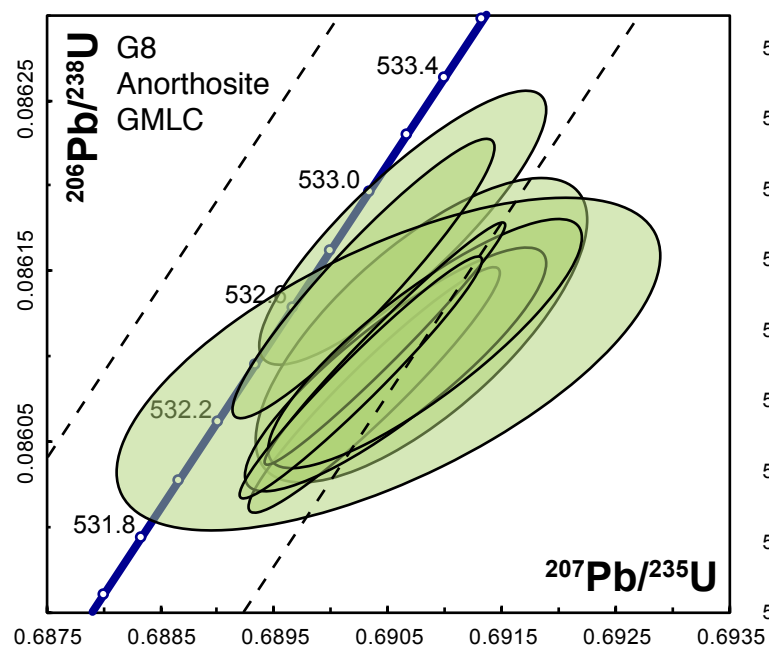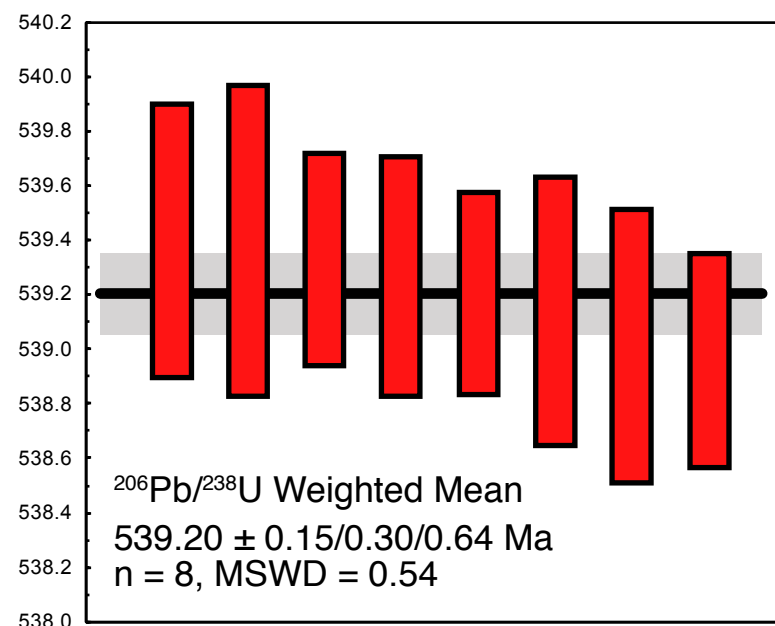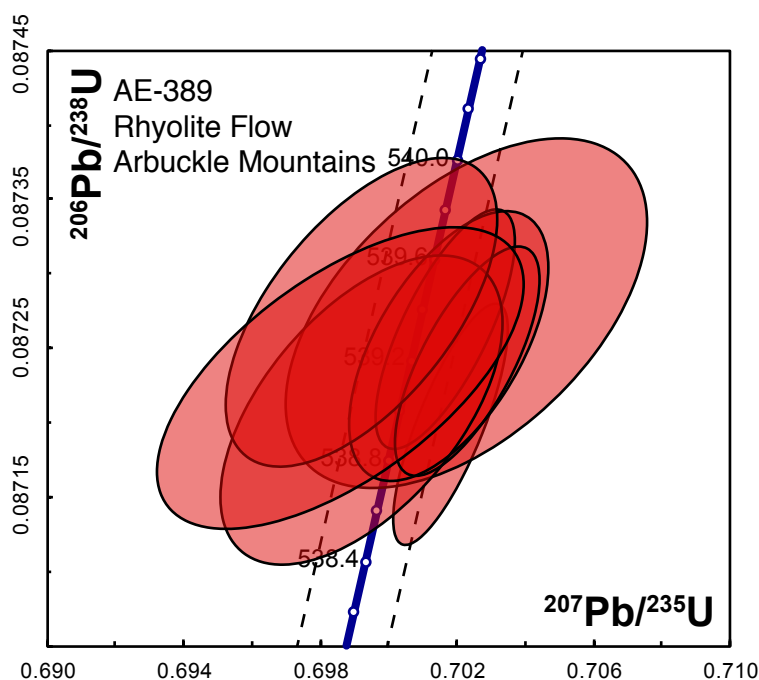246   *Contributions to Mineralogy and Petrology*, 151: 413-433.

A

Oklahoma

Fig. DR1A

Southern Oklahoma Aulacogen

Ouachita thrusts

100 km

35° 30′ N

Mississippian to Quaternary

Devonian to Upper Ordovician

Upper to Middle Ordovician

Lower Ordovician to Upper Cambrian

Fault

WTH

ARBUCKLE ANTICLINE

ETH

HUNTON ANTICLINE

BELTON ANTICLINE

TISHOMINGO ANTICLINE

Rhyolite (Ediacaran-Lower Cambrian)

Granite and gneiss (Mesoproterozoic)

U-Pb zircon age 536 ± 5 Ma from rhyolite dike intruding 1.4 Ga rocks (Thomas et al., 2012)

97° 15′ W

8 km

N

B

A

0   500 m

(no vertical exaggeration)

bedding

altered glass

columnar jointing

basal glassy and flow-banded zone

flow breccia

flow banding and flow folding

B

felsitic interior

U-Pb Zircon age 539 ± 5 Ma (Thomas et al., 2012)

AE-389

N

500 m

Bedding

Fault

Thrust fault

B

A

B

Arbuckle Group (Lower Ordovician to Upper Cambrian)

Timbered Hills Group (Upper Cambrian)

Igneous rocks (Ediacaran to Lower Cambrian):

Diabase intrusion (trend unknown)

Diabase dike

Hypabyssal felsic intrusions

Upper rhyolite flow

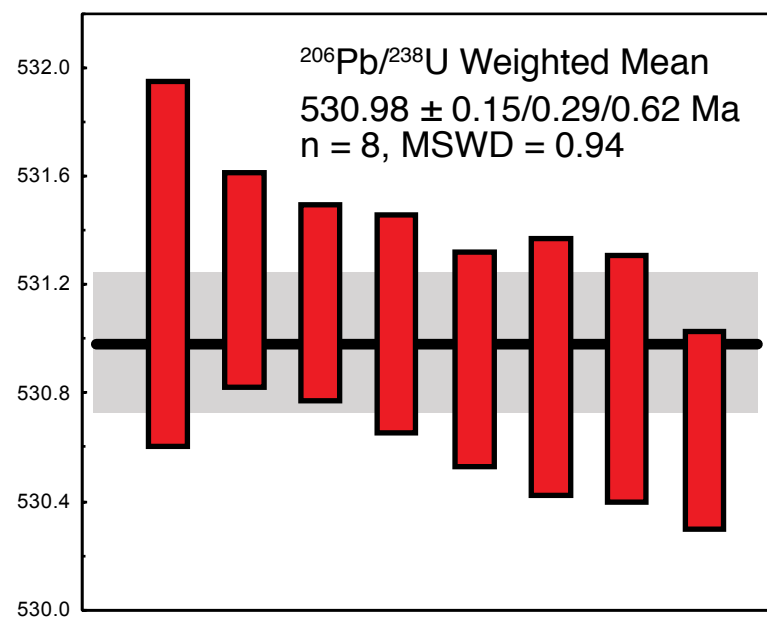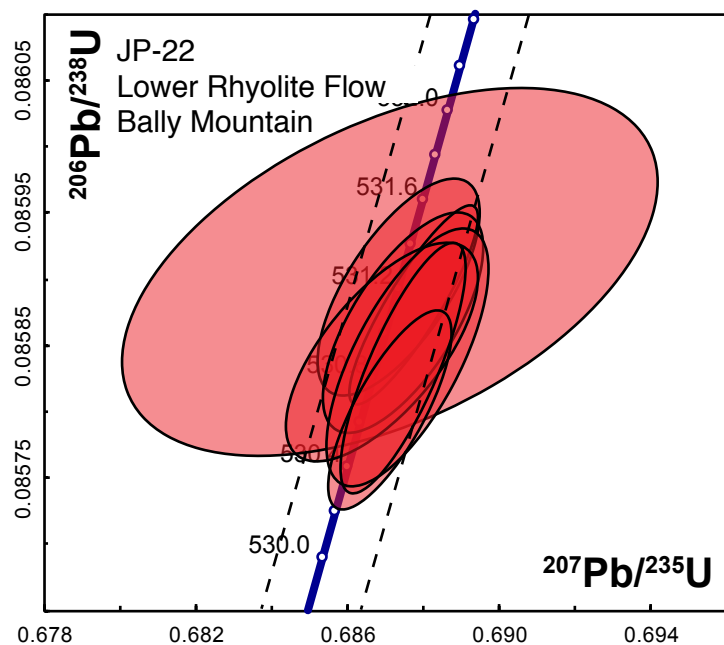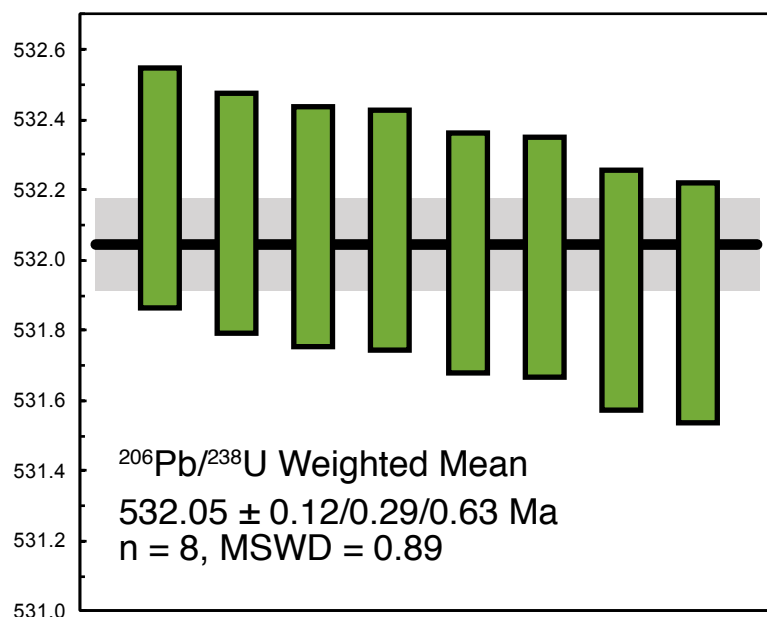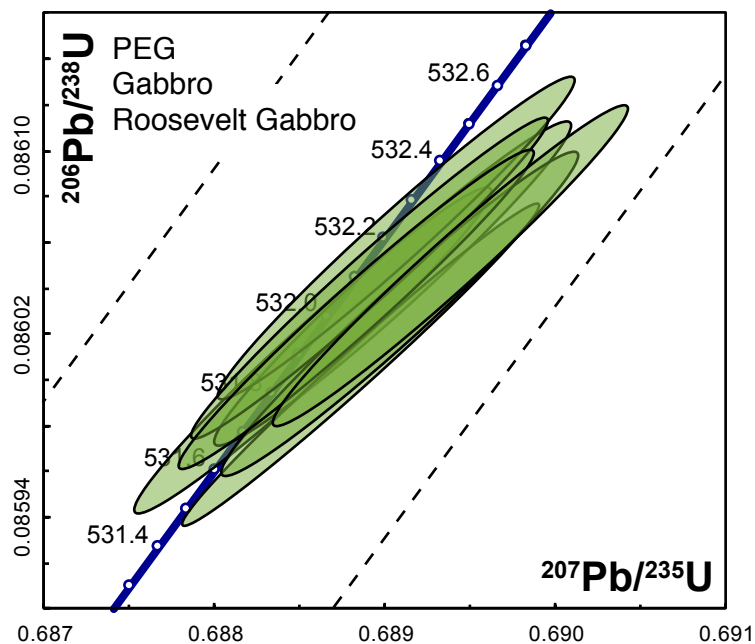Bedded rhyolitic volcaniclastics

Lower rhyolite flow

Fig. DR1: Geological setting of Arbuckle rhyolite samples. (A) Geological map of Arbuckle Mountains, modified from Ham (1973). WTH = West Timbered Hills, ETH = East Timbered Hills. Location of map is shown in inset. (B) Geological map and cross-section of igneous exposures in East Timbered Hills, modified from Eschberger et al. (2014). Location of dated rhyolite sample AE389 is indicated, along with location of hypabyssal felsic intrusion dated by Thomas et al. (2012). Faults are from Johnson (1990). (C) Geological map of igneous exposures in West Timbered Hills, modified from Boro (2015) and Toews (2015). Location of dated rhyolite sample JP-118 is indicated. Faults are generally taken from Johnson (1990).
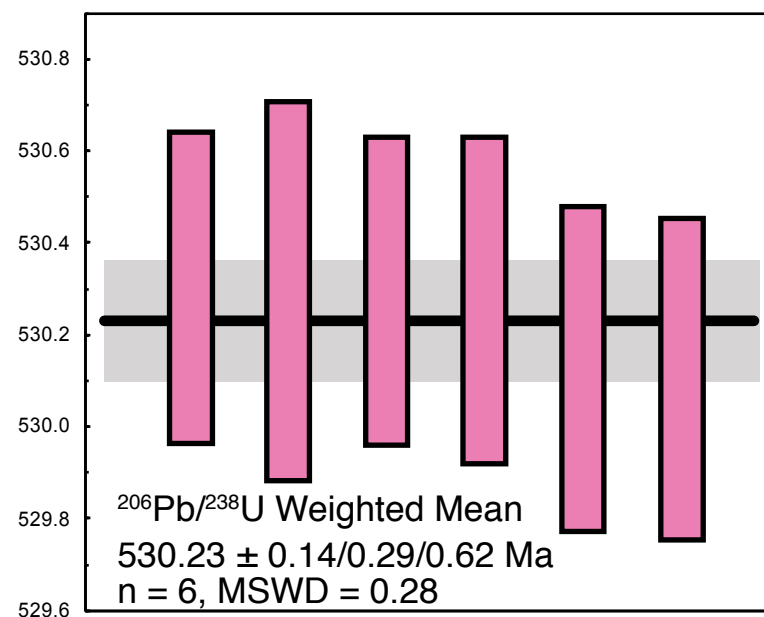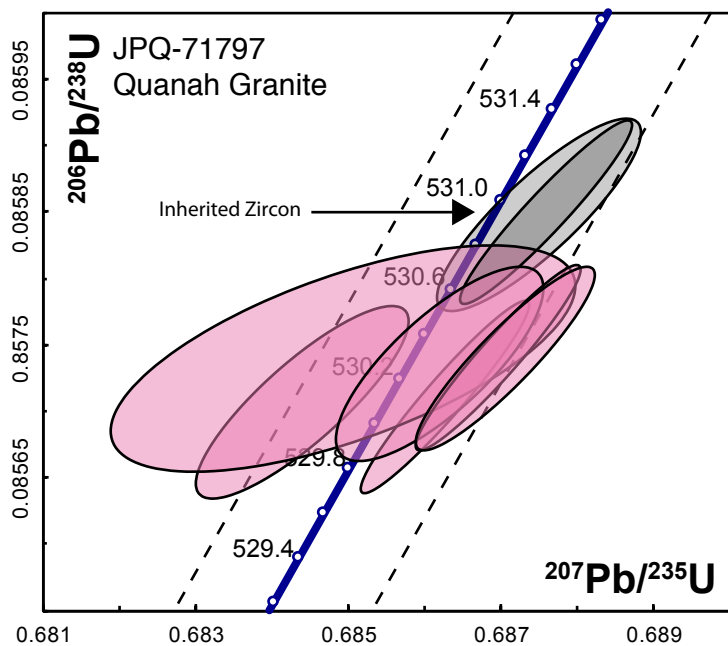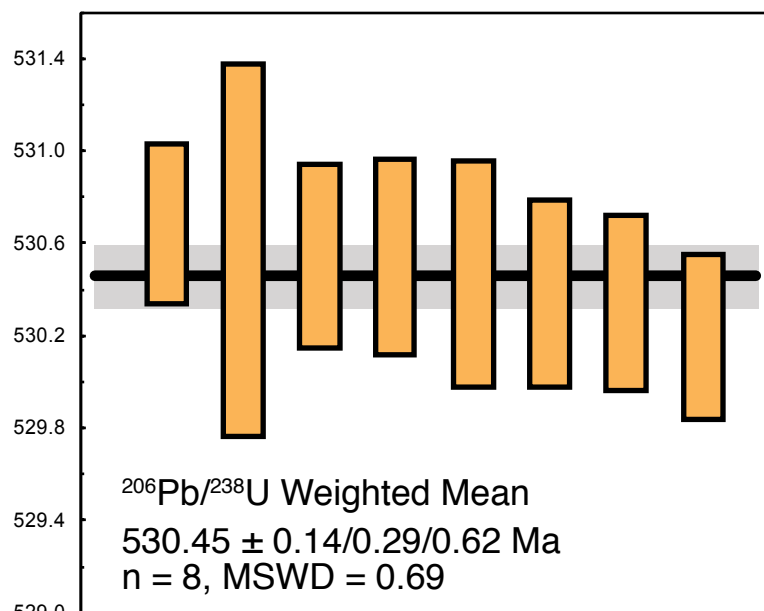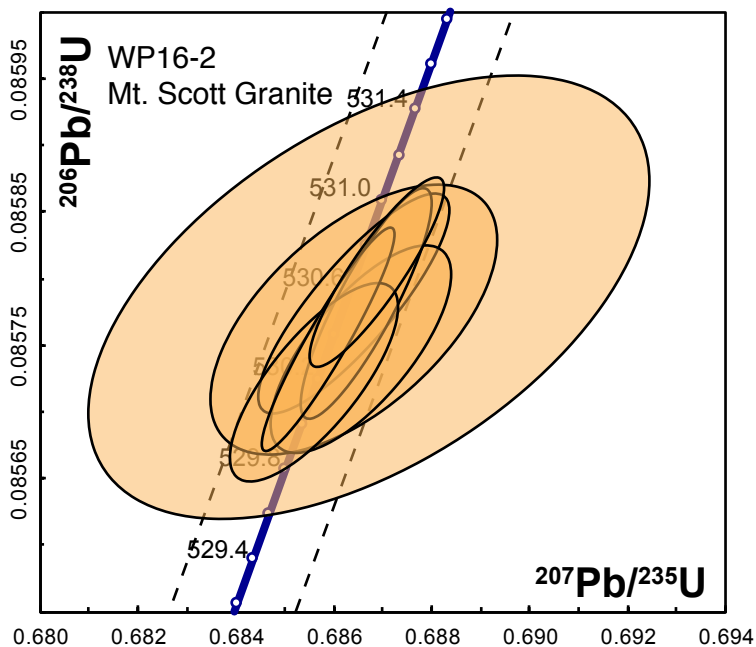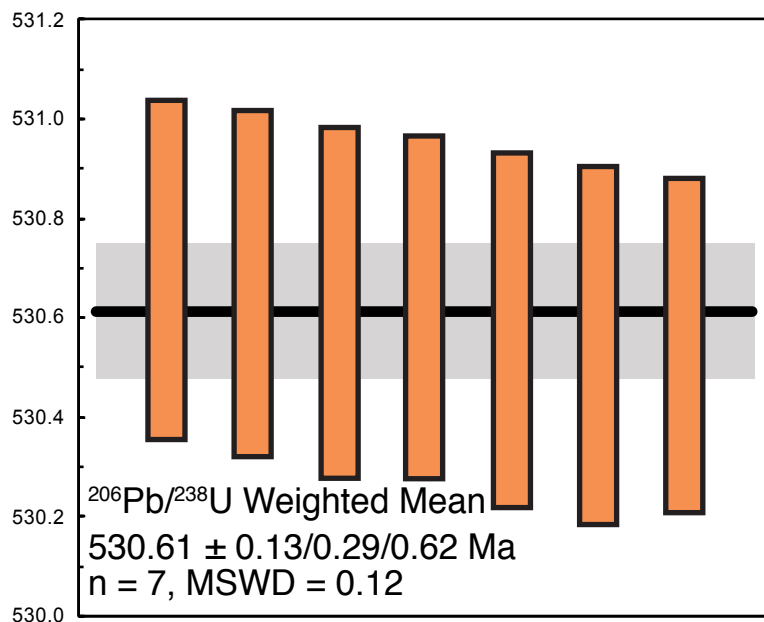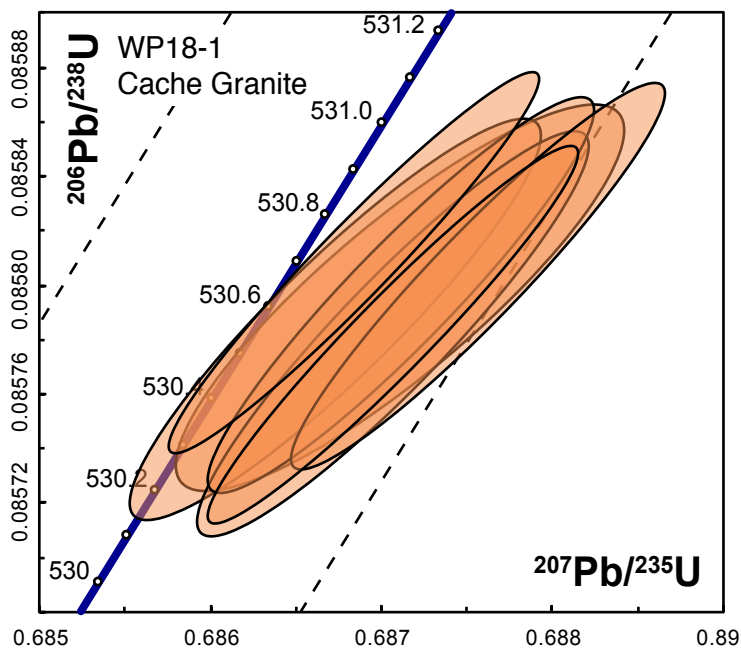
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WP18-1 Cache Granite

^{206}Pb/^{238}U Weighted Mean
530.61 ± 0.13/0.29/0.62 Ma
n = 7, MSWD = 0.12

WP16-2 Mt. Scott Granite

^{206}Pb/^{238}U Weighted Mean
530.45 ± 0.14/0.29/0.62 Ma
n = 8, MSWD = 0.69

JPQ-71797 Quanah Granite

Inherited Zircon

^{206}Pb/^{238}U Weighted Mean
530.23 ± 0.14/0.29/0.62 Ma
n = 6, MSWD = 0.28

Quoted errors for calculated ^{206}Pb/^{238}U weighted means are of the form ±X/Y/Z, where X is solely analytical uncertainty, Y is the combined analytical and tracer uncertainty, and Z is the combined analytical, tracer and ^{238}U decay constant uncertainty. Each ellipse indicates the analysis of a single zircon grain. Dashed lines show the error bounds of the concordia curve due to the uncertainty in the decay constants of U. MSWD refers to the mean square of the weighted deviates. Horizontal black in the the weighted mean panel indicates the weighted mean date and the grey band reflects the internal uncertainty. Colors of the ellipses and individual ^{206}Pb/^{238}U bars correspond to the color of the mapped unit from Figure 2 in the manuscript.

Sr and Nd Isotopic Data for the Wichita Igneous Province

Granites and rhyolites: ε_{Nd} = +1.9 to +4.5 (Wright et al., 1996; Gilbert and Weaver, 2010).

Basalts: ε_{Nd} = +1.9 to +4.1; $^{87}Sr/^{86}Sr_i$ = 0.70319 (Brueseke et al., 2016).

Diabase intrusions: ε_{Nd} = +1.6 to +5.1; $^{87}Sr/^{86}Sr_i$ = 0.70387 to 0.70484 (Hogan et al., 1995; Gilbert and Weaver, 2010; Lidiak et al., 2014).

Roosevelt Gabbro: ε_{Nd} = +3.3 to +5.1 (Gilbert and Weaver, 2010).

Glen Mountains Layered Complex: ε_{Nd} = +3.6 to +5.4; $^{87}Sr/^{86}Sr_i$ = 0.70359 (Lambert et al., 1988).

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