

This **Supplemental Material** accompanies

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**Table S1 provides total-fusion Ar/Ar analytical data.**

**Table S2 provides step-heating Ar/Ar analytical data.**

### **Supplemental Material: $^{40}\text{Ar}/^{39}\text{Ar}$ Dating Methods**

The  $^{40}\text{Ar}/^{39}\text{Ar}$  laser-heating dating method was applied to one sample of vitric (nearly aphyric) Kirker Tuff (37°59.279'N, 121°54.696'W) obtained from the base of the unit exposed on the south slope of a tributary of Kirker Creek, southeast of Pittsburg, California (Figs. 20 and 21<sup>1</sup>).

Two variants of the  $^{40}\text{Ar}/^{39}\text{Ar}$  technique were used, and two mineral phases were examined. The single-crystal total-fusion (SCTF) technique was applied to individual plagioclase phenocrysts, in which a single grain is rapidly heated to melting in a single step, or for a subset of the grains, after a low-power “degassing” step. The second technique uses a rampable-power laser to date single crystals by incremental heating (SCIH); both plagioclase and biotite phenocrysts were analyzed by this method. All processing and analyses were carried out at the Berkeley Geochronology Center (BGC).

Mineral phases were concentrated from bulk rock using standard separation techniques, including gentle hand crushing, sieving from 200 to 1000  $\mu\text{m}$ , and magnetic separations. Biotite was further separated using polytungstate heavy liquids at a density of 2.85  $\text{g}/\text{cm}^3$  to concentrate the heavier, fresher material. Plagioclase was treated in 5% HF for 15 min, followed by distilled water. Both phases were then checked under binocular microscope to obtain the final separations (mostly in the 200–300  $\mu\text{m}$  range).

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<sup>1</sup> Figures 29–34 appear at the end of this file. All other cited figures appear in the chapter itself.

The completed separates for  $^{40}\text{Ar}/^{39}\text{Ar}$  dating were irradiated in the Cd-lined CLICIT position of the Oregon State University TRIGA reactor in two batches (BGC irradiation numbers 462 for 10 h, and 472 for 5 h). Irradiation 462 employed sanidine from the Fish Canyon Tuff of Colorado as a monitor mineral (orbitally referenced age of  $28.201 \pm 0.023$   $1\sigma$  Ma; Kuiper et al., 2008), while 472 used sanidine phenocrysts from the Alder Creek Rhyolite of California (orbitally referenced age =  $1.1848 \pm 0.0006$  Ma; Niespolo et al., 2017). Reactor-induced isotopic production ratios for these irradiations were:  $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 2.65 \pm 0.02 \times 10^{-4}$ ,  $(^{38}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 1.96 \pm 0.08 \times 10^{-5}$ ,  $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 6.95 \pm 0.09 \times 10^{-4}$ ,  $(^{37}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.24 \pm 0.16 \times 10^{-4}$ ,  $(^{38}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 1.220 \pm 0.003 \times 10^{-2}$ , and  $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.5 \pm 0.9 \times 10^{-4}$ . Atmospheric  $^{40}\text{Ar}/^{36}\text{Ar} = 298.56 \pm 0.31$  (Lee et al., 2006), and decay constants followed Min et al. (2000).

Following several weeks for radiological “cooling” of the samples after irradiation, the grains were analyzed individually either by the SCTF (Table S1) or the SCIH (Table S2) technique: biotite by SCIH, plagioclase by both SCIH and SCTF. In the SCIH technique, individual phenocrysts are incrementally heated in 5–10 steps (depending on grain size and gas yield) at progressively increasing power to fusion. In the SCTF technique, an initial subset of grains (analyses 27674-01–27674-10) was heated in a single step to fusion, whereas another subset (27674-11–27674-23) was given a very low-power “degas” step prior to a high-power fusion step in an attempt to enhance the radiogenic argon concentration in the final fusion step. These analyses were conducted on a Nu Instruments Noblesse noble-gas mass spectrometer, featuring a high-efficiency ionization source and simultaneous multi-isotope measurement using all ion-counting electron multiplier detection systems. In total, 23 grains of plagioclase were analyzed by SCTF, while 10 grains of biotite and five grains of plagioclase were analyzed by SCIH (note that an additional 11 grains of plagioclase were tested with a low-power degas step

and rejected due to the likelihood that they were not actually primary plagioclase phenocrysts, but rather quartz, old detrital feldspar, or unknown altered material; Table S2). Additional details of the dating process utilized at BGC are provided in Deino et al. (2010).

## **<sup>40</sup>Ar/<sup>39</sup>Ar Dating Results**

### ***Single Crystal Total-Fusion Analyses***

Figure 28 shows the age probability-density spectrum for the SCTF analyses. A prominent mode composed of 13 grains is present at ca. 29 Ma, which is interpreted as the primary eruptive age. Two much younger grains are present (at ca. 10 Ma and ca. 4.8 Ma), but these contrast with all other analyses by SCTF, as well as biotite and plagioclase analyzed by SCIH (see below), and they are interpreted as altered material. Another four grains are clearly older detrital contaminants, ranging ca. 80–150 Ma. By filtering the population based on removing outliers that exhibit ages with significant departure from the median age (using the value of two normalized median absolute deviations from the median), these and one other grain were eliminated, yielding a weighted-mean age for the remaining group of  $29.04 \pm 0.38$  Ma. An “inverse isochron” regression ( $^{36}\text{Ar}/^{40}\text{Ar}$  vs.  $^{39}\text{Ar}/^{40}\text{Ar}$ ) was calculated from the 12 valid total-fusion runs (Fig. 29), and the age derived from this analysis was accepted as the reference age for SCTF experiments on the Kirker Tuff plagioclase of  $29.12 \pm 0.39$  Ma ( $1\sigma$ ).

### ***Single Crystals by Incremental Heating***

Incremental release experiment results are plotted as apparent age versus percent of total  $^{39}\text{Ar}$  released in Figure 30 for biotite and Figure 31 for plagioclase. Release patterns were examined for age consistency, where grains that had a near-uniform release pattern (“apparent age plateaus”) were deemed more geologically reliable than those with irregular age patterns. The plateau identification algorithm used here (following Fleck et al., 1977) delineates the set of

contiguous steps encompassing the greatest percent of  $^{39}\text{Ar}$  released that exhibits an acceptable mean square of weighted deviates (MSWD), with a threshold probability >95% that the observed scatter is caused by analytical error alone and that geological scatter is not demonstrated. A plateau must comprise at least 50% of the total  $^{39}\text{Ar}$  release and consist of at least three consecutive steps. The age and uncertainty assigned to the plateau are weighted means (weighted by the inverse variance of each step) and modified standard error (MSE; standard error expanded by root MSWD if MSWD >1).

Each of 10 biotite grains tested yielded a plateau across almost the entire release pattern, except characteristically the first one or two steps comprising ~5%–15% of the total  $^{39}\text{Ar}$  released; the anomalously young age of this initial release is attributed to slight alteration of the biotite. After the initial outgassing, plateaus extended to the end of the release and ranged from 29.53 to 27.82 Ma (weighted mean =  $28.54 \pm 0.13$  Ma). “Inverse isochron” regression ages of the plateau steps from each experiment, calculated on a grain-by-grain basis (Figs. 32, 33), and plotted on an age-probability density plot (Fig. 34), yielded a weighted-mean age of  $29.25 \pm 0.32$  Ma, within error of the plateau age mean. The isochron age is taken as the reference age of the biotite.

Similar to the biotite, the individual plagioclase grains subjected to incremental heating ( $n = 5$ ) yielded plateaus across virtually the entire release pattern (Fig. 31), except for the initial step in three of the experiments. Plateau ages ranged from 29.17 to 25 Ma, but it should be noted the uncertainties of the plateau ages varied widely (1.7%–44%) due to grain size and Ca/K ratio (Fig. 34). Inverse isochrons of the plateau steps (Fig. 33) yielded the age-probability distribution shown in Figure 34. The isochron ages give a weighted-mean age for plagioclase of  $29.19 \pm 0.60$  Ma, taken as the reference age for the plagioclase step-heating experiments.

A weighted-mean age of the three isochron-derived results (plagioclase SCTF =  $29.12 \pm 0.39$  Ma; plagioclase SCIH =  $29.19 \pm 0.60$  Ma; and biotite SCIH =  $29.25 \pm 0.32$  Ma) yields  $29.197 \pm 0.065$  Ma for the overall reference age of the Kirker Tuff.

Figure 29. Inverse isochron regression ( $^{36}\text{Ar}/^{40}\text{Ar}$  vs.  $^{39}\text{Ar}/^{40}\text{Ar}$ ) calculated from the included total-fusion runs. “ $^{40}\text{Ar}/^{36}\text{Ar}$  Int.” is the “trapped”  $^{40}\text{Ar}/^{36}\text{Ar}$  composition of the suite calculated from the  $y$ -axis intercept.

Figure 30. Biotite single-crystal incremental heating (SCIH) release spectra, showing apparent age and percent radiogenic  $^{40}\text{Ar}$  against the percentage of the total  $^{39}\text{Ar}$  released in each experiment. The total-gas integrated age, given at the bottom of each graph, was obtained by isotopically summing the individual steps weighted by the inverse variance, with the error calculated as the modified standard error (MSE; standard error expanded by root MSWD if  $\text{MSWD} > 1$ ) at  $1\sigma$ . The apparent age plateau was calculated as the mean age weighted by the inverse variance, with the error as the  $1\sigma$  MSE.

Figure 31. Plagioclase single-crystal incremental heating (SCIH) experiments.

Figure 32. Inverse isochron regressions calculated from the plateau steps of each biotite single-crystal incremental heating (SCIH) experiment.

Figure 33. Plagioclase inverse isochron regressions from the plateau steps of each single-crystal incremental heating (SCIH) experiment.

Figure 34. Results of isochron regressions from single-crystal incremental heating (SCIH) experiments on biotite (red squares) and plagioclase (black circles). Open and closed symbols represent grains omitted and included in the final result, respectively. Analyses were excluded if their ages fell more than two median absolute deviations from the median age. (A) Mean Ca/K ratio of individual grains. (B) Rank order of the isochron age results. (C) Age-probability density spectra of the biotite and plagioclase analyses, with weighted means.

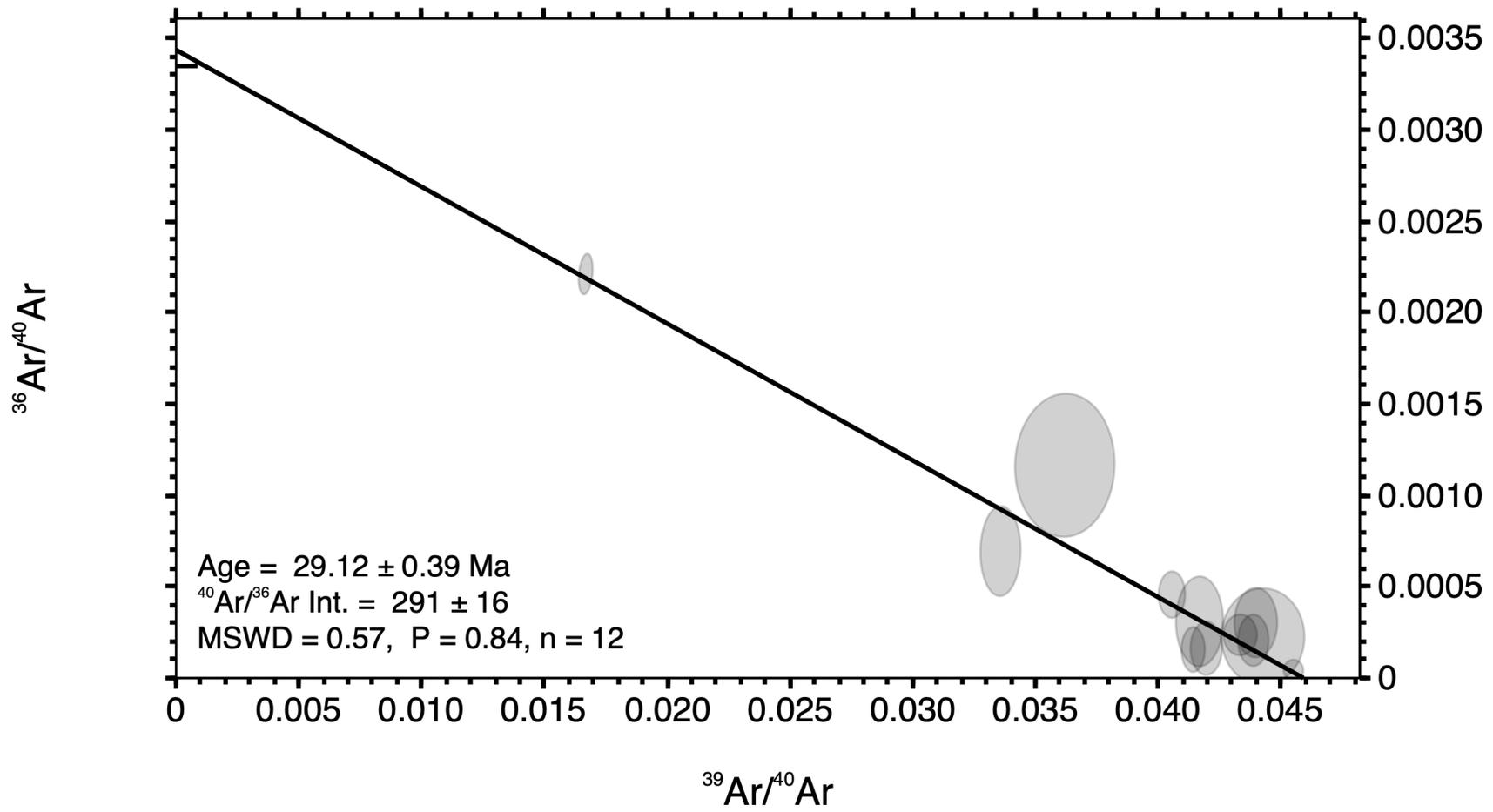


Figure 29

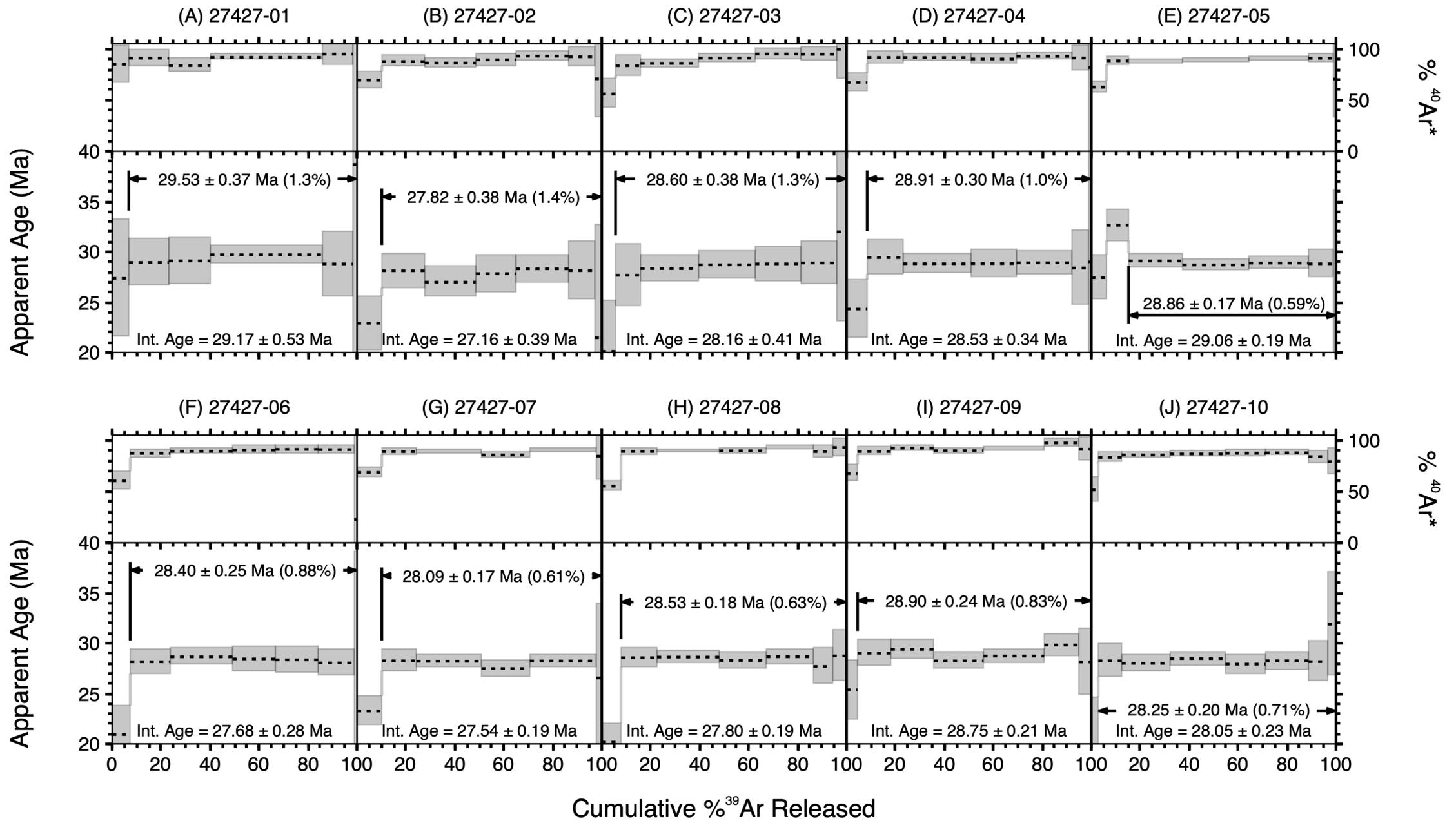


Figure 30

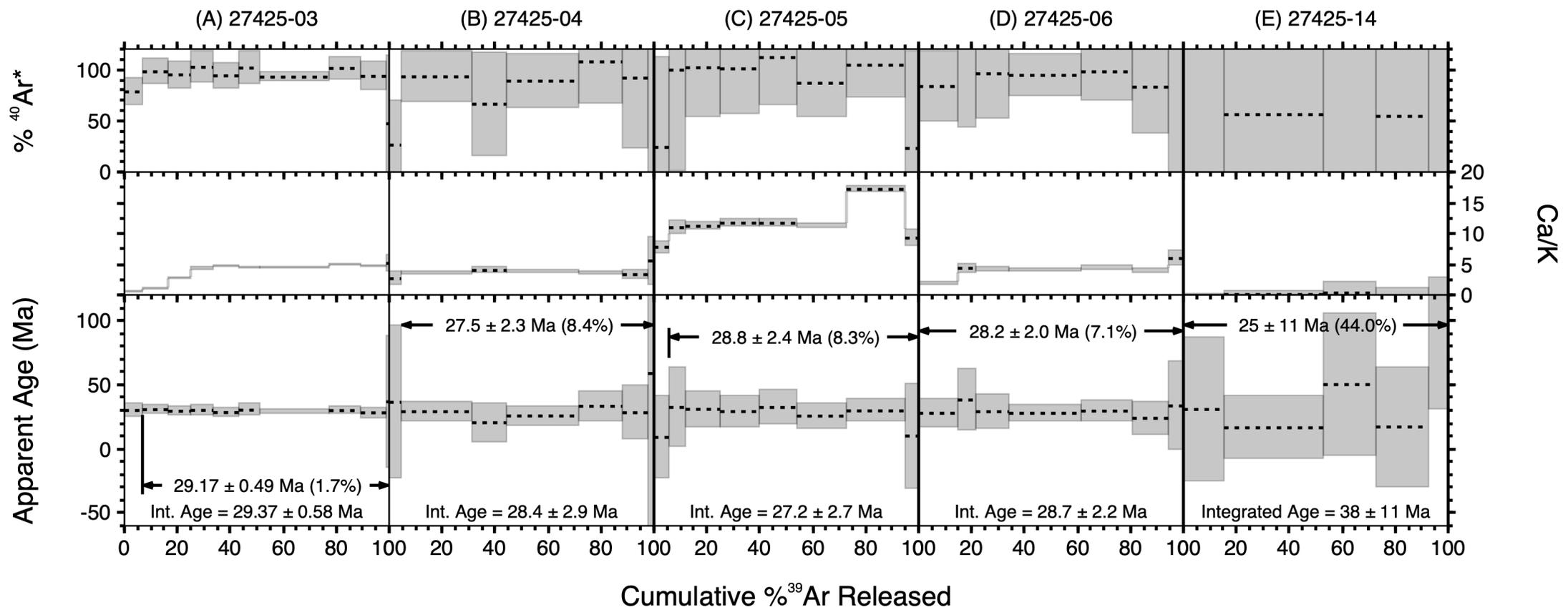


Figure 31

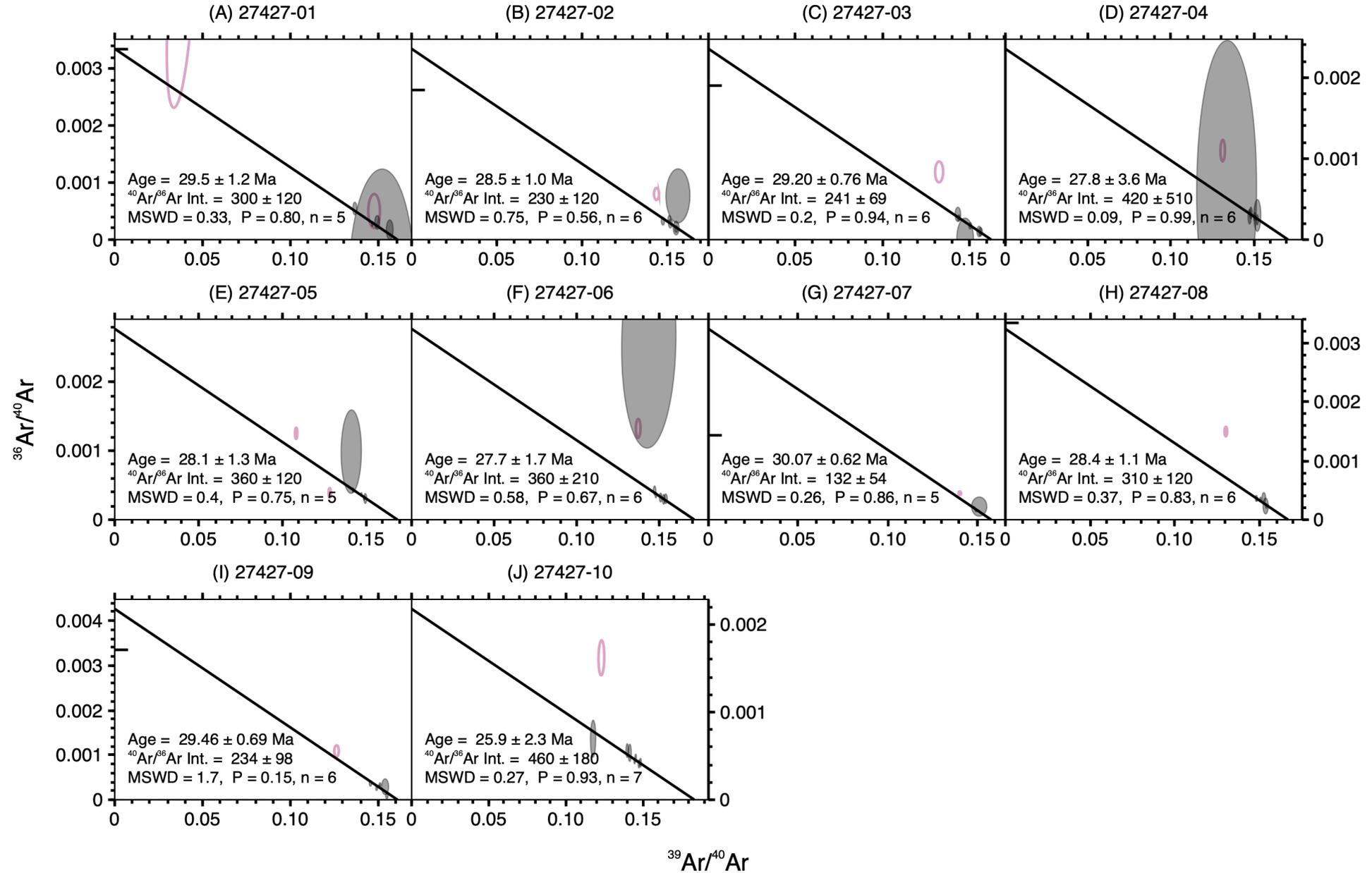


Figure 32

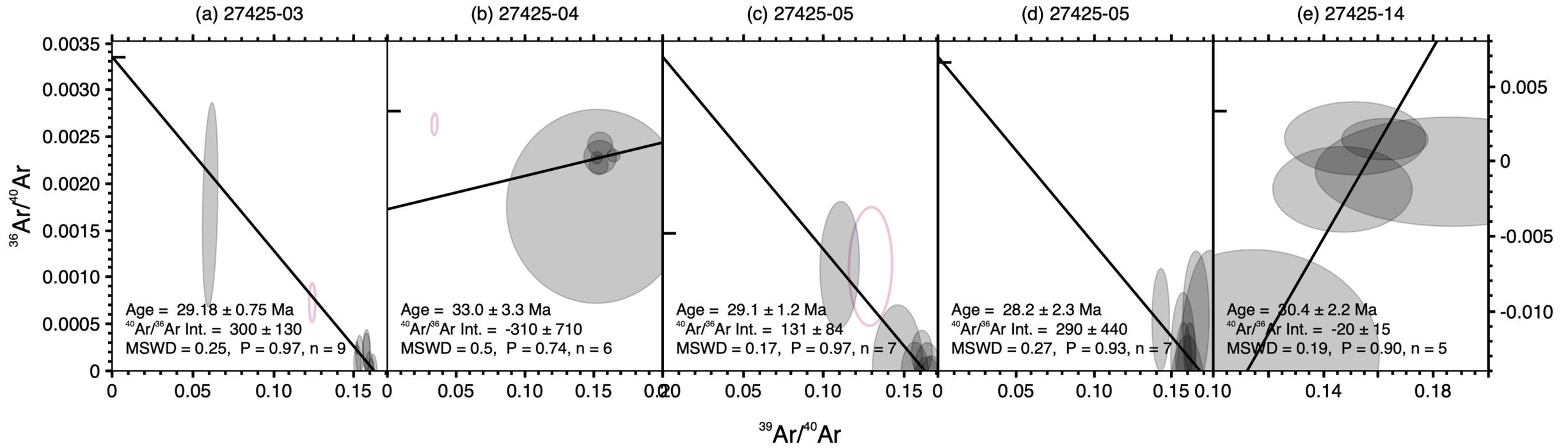


Figure 33

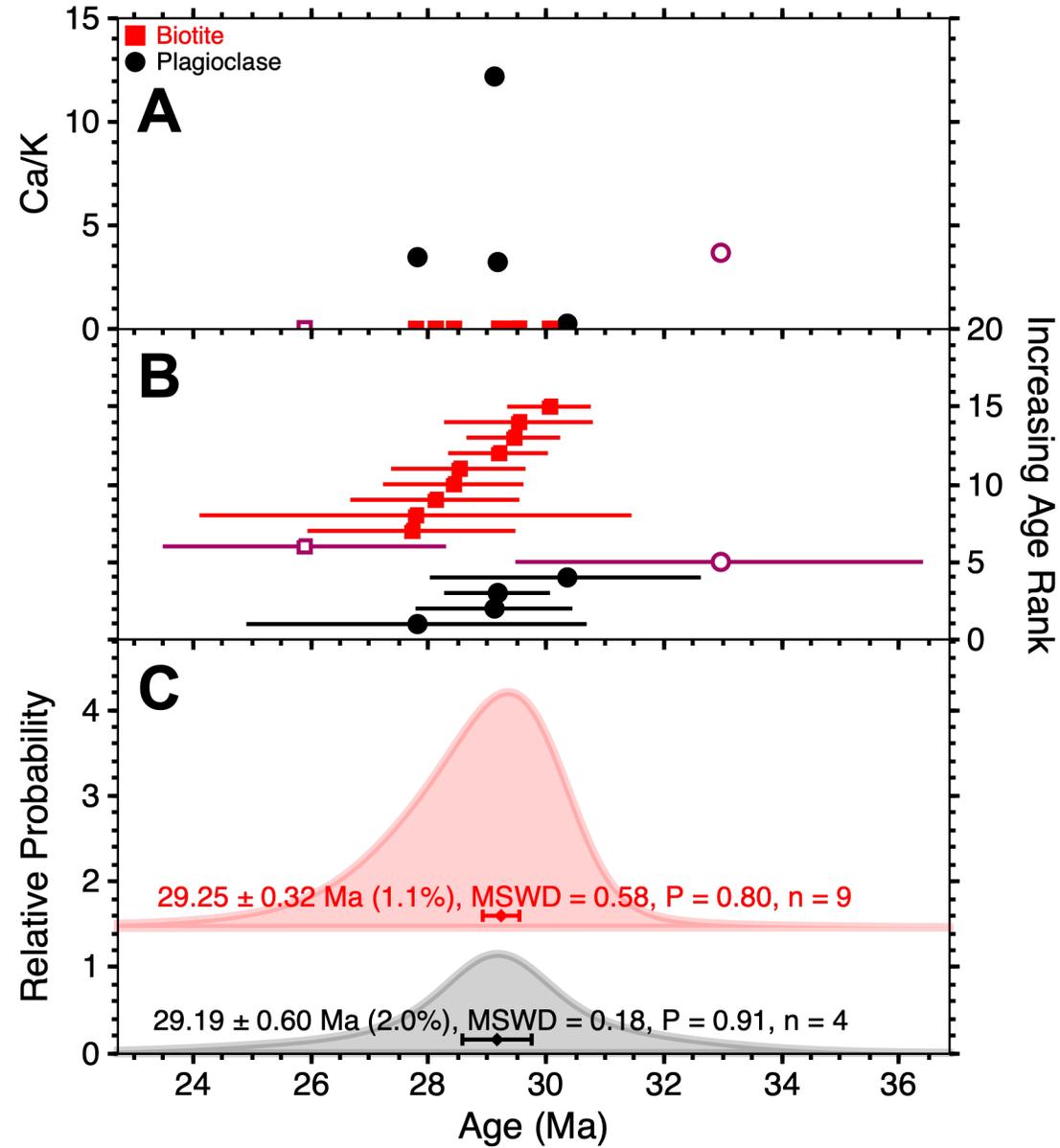


Figure 34