New $^{40}Ar/^{39}Ar$ ages for the Spearhead and Civet Cat Canyon Members of the Stonewall Flat Tuff, Nye County, Nevada-Evidence for systematic errors in standard K-Ar age determinations on sanidine

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NEW $^{40}Ar/^{39}Ar$ AGES FOR THE SPEARHEAD AND CIVET CAT CANYON MEMBERS OF THE STONEWALL FLAT TUFF, NYE COUNTY, NEVADA—EVIDENCE FOR SYSTEMATIC ERRORS IN STANDARD K-Ar AGE DETERMINATIONS ON SANIDINE

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This paper presents the results of new age determinations of the Stonewall Flat Tuff using both single-crystal laser-fusion $^{40}Ar/^{39}Ar$ and conventional K-Ar dating methodologies. These determinations are the outgrowth of geologic mapping of the Tolicha Peak 15-minute quadrangle and the recognition that standard K-Ar age determinations made during the last several years on alkali feldspars in tuffs from the southwestern Nevada volcanic field were in some cases significantly younger than ages obtained from overlying rock units. The new radiometric dates of 7.5 to 7.7 Ma show that Stonewall Flat Tuff is as much as 20% older than heretofore believed and demonstrate the existence of appreciable systematic errors that may occur in the analysis of alkali feldspar using the conventional K-Ar technique.

The Stonewall Flat Tuff consists of two sheets of slightly peralkaline silicic ash-flow tuff erupted from the Stonewall Mountain volcanic center (Noble and others, 1984; Hausback and Frizzell, 1986; 1987; Weiss and Noble, 1989). The lower sheet, the Spearhead Member, is the most widespread (fig. 1), whereas the overlying Civet Cat Canyon Member is less voluminous and more restricted in areal extent.

K-Ar ages of about 6.3 Ma had been reported for both members by Kistler (1968) and Noble and others (1984, 1988). Armstrong and others (1972) and Foley (1978), however, had presented ages on the Stonewall Flat Tuff and the Stonewall Mountain volcanic center that were older than 7 Ma. The ages reported by Noble and others (1984) were, nevertheless, accepted as providing the best estimate of the Stonewall Flat Tuff because they were more recent and because they agreed very well with the earlier age of Kistler (1968). Additional complexities were indicated by the spread in ages obtained on the Civet Cat Canyon Member (Noble and others, 1988) and the two conventional K-Ar ages of 6.2 ± 0.2 and 7.3 ± 0.3 Ma (specimens DN-CST and 3DN8-18A, respectively, this paper) obtained on sanidine from exposures in the vicinity of the Bullfrog Hills, one of which is a million years older than the heretofore accepted "best" age of 6.3 Ma.

SAMPLE DESCRIPTION AND PREPARATION

Dates reported in this study were determined from radiometric analysis of sanidine separates, four analyses are by $^{40}Ar/^{39}Ar$ methods and two are by conventional K-Ar methods. Two of the samples are from exposures of the Spearhead Member (specimen BH86N9) and Civet Cat Canyon Member (BH86N33) within the Tolicha Peak 15-minute quadrangle to the east and southeast of Stonewall Mountain. The other two samples are from exposures of the Spearhead Member near Goldfield and the Black Mountain volcanic center. These latter two samples (specimens LABCANO81 and 83G SPEAR) are sanidine separates that had previously been dated by conventional K-Ar techniques, yielding 6.3 ± 0.2 Ma (Noble and others, 1984, table 3). These four samples were analyzed by $^{40}Ar/^{39}Ar$ methods. The final two samples (DN-CST and 3DN8-18A) are from exposures of the Spearhead Member in the vicinity of the Bullfrog Hills and were analyzed by conventional K-Ar methods.

All specimens analyzed consist of partly to densely welded, glassy ash-flow tuff. Pumice fragments were used for LABCANO81 whereas the others were separated from bulk tuff. Samples were prepared for $^{40}Ar/^{39}Ar$ dating by simple crushing, sieving, and hand-selection of feldspar grains under the binocular microscope. Grains that appeared altered or cloudy, or contained large mineral, glass, or fluid inclusions, or that had adhering glass were discarded. Standard heavy liquid and magnetic separation methods were employed on samples LABCANO81 and 83G SPEAR. Crystal concentrates were washed sequentially with 10% HCl, 5% HF, and distilled water solutions for approximately 5 minutes each in an ultrasonic bath.

Sanidine concentrates for the conventional K-Ar analyses were made by crushing, heavy-liquid, magnetic, electrostatic, and hand picking procedures.

All the grains dated are believed to be sanidine, based on direct chemical analysis of a number of alkali feldspar separates from the Stonewall Flat Tuff and on the lack of gridiron twinning common in anorthoclase.

$^{40}Ar/^{39}Ar$ ANALYTICAL PROCEDURES

Details of the analytical process for the single-crystal, laser-fusion $^{40}Ar/^{39}Ar$ dating as employed herein are given in Deino and others (1990). Briefly, the samples were first irradiated in a core facility of the Omega West reactor of the Los Alamos National Laboratory. Samples BH86N33 and BH86N9 were irradiated for 10 hours, while 83G Spear and LABCANO81 received 8 hours of irradiation. After a cooling period, the samples were transferred to the vacuum extraction system for overnight bakeout. Individual feldspar grains were then fused with a focused Ar-ion laser beam applied for 10–40 seconds. The released gasses were scrubbed of reactive species, followed by Ar isotope measurement in a low-blank mass spectrometer (MAP-215). The dating process for each analysis takes about 30 minutes and is fully automated.

Sanidine from the Fish Canyon Tuff, MMhb-1, and an intralaboratory sanidine standard (referenced to MMhb-1) were used to monitor the neutron flux. A reference age of 27.84 Ma was used for the Fish Canyon Tuff sanidine (Cebula, and others, 1986, corrected for the updated age

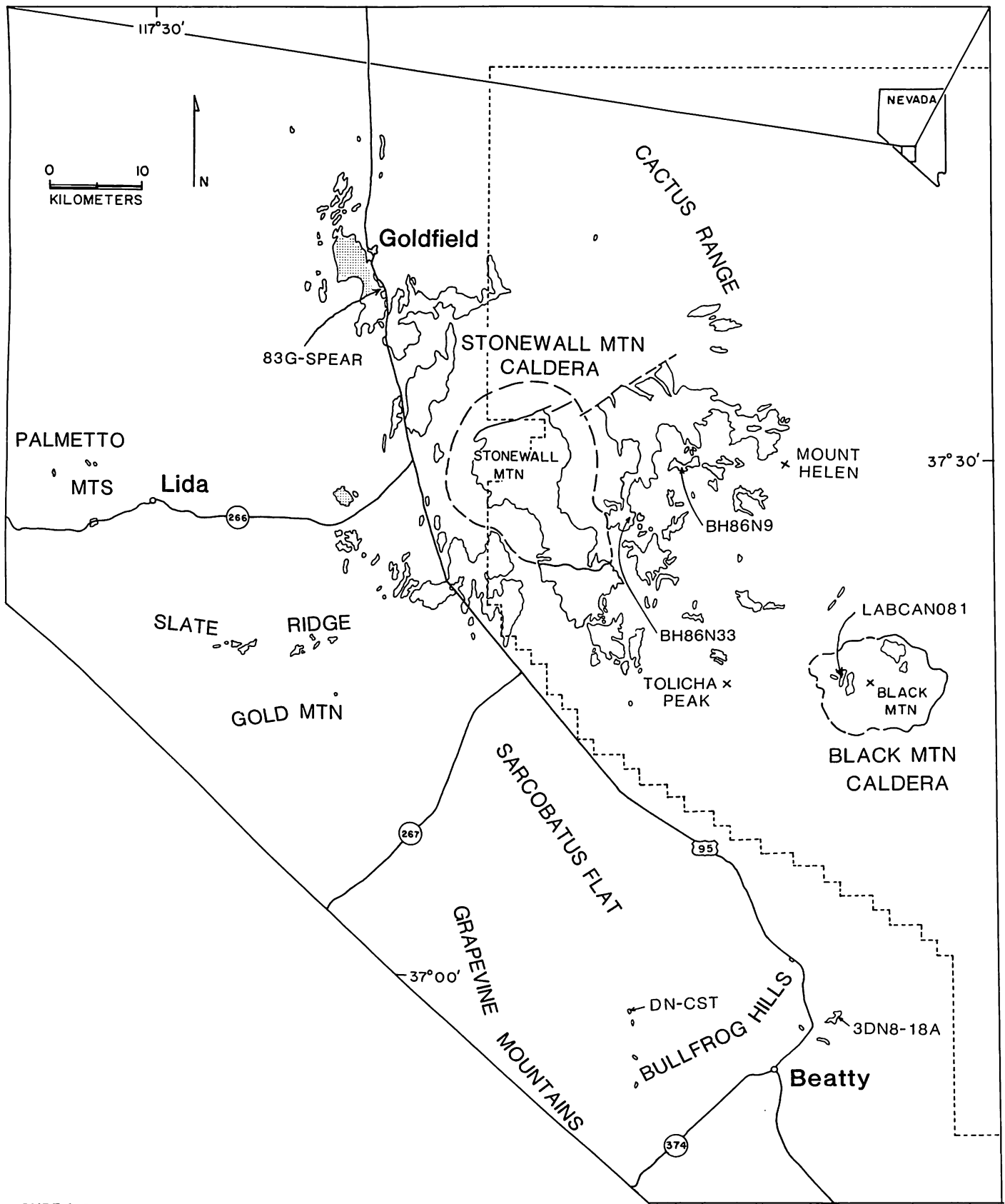

FIGURE 1. Map showing the distribution of the Stonewall Flat Tuff, Nye and Esmeralda Counties, Nevada. Arrows point to sample localities of this paper; stipple shows outcrops of the Stonewall Flat Tuff covered by basaltic lava; short dashed line shows the boundary of the Nellis Air Force Bombing and Gunnery Range. Compiled from published maps as revised by Noble and others (1984), mapping of Hausback and Frizzell (1988), Weiss and Noble (1989), and unpublished mapping of Noble and Weiss (1983–1990) and Frizzell and Hausback (1986–1987).

of monitor MMhb-1 of 520.4 Ma, Samson and Alexander, 1987).

Weighted mean ages were calculated using the inverse variance of the 1σ analytical uncertainties, as the weighting factor as in Taylor (1982), while 1σ standard errors of the mean (s.e.m.) were calculated according to Samson and Alexander (1987).

K-Ar ANALYTICAL PROCEDURES

Samples DN-CST and 3DN8-18A were analyzed by standard isotope dilution procedures as described by Dalrymple and Lanphere (1969). Potassium analyses were performed by lithium metaborate flux fusion flame photometry techniques, the lithium serving as an internal standard (Ingamells, 1970). Argon analyses were performed using a 15.2-cm-radius Neir-type mass spectrometer. The precision of the data shown is the estimated analytical uncertainty at one standard (Cox and Dalrymple, 1967). It represents uncertainties in the measurement of radiogenic ^{40}Ar and K_2O based on experience with hundreds of replicated analyses in the U.S. Geological Survey laboratories in Menlo Park. Mass discrimination of the spectrometer is routinely determined on the basis of multiple

TABLE 1. $^{40}Ar/^{39}Ar$ analytical data, Stonewall Flat Tuff, Nye Co., Nevada.

Lab ID#	Ca/K	$^{36}Ar/^{39}Ar$	$^{40}Ar^*/^{39}Ar$	Moles ^{40}Ar $\times 10^{14}$	$\%^{40}Ar^*$	Age (Ma) ± 1σ[§]
Civet Cat Canyon Member (BH86N33):						
J = 0.007269 ± 0.000005						
1200-01	0.1582	0.00015	0.577	6.4	93.5	7.54 0.03
1200-02	0.0957	0.00007	0.576	5.8	97.1	7.54 0.03
1200-03	0.0569	0.00007	0.572	5.7	96.7	7.49 0.03
1200-04	0.1050	0.00005	0.578	7.7	97.9	7.56 0.02
1200-05	0.1333	0.00015	0.577	6.5	93.3	7.55 0.03
1200-06	0.0964	0.00013	0.577	3.3	93.9	7.55 0.04
1200-07	0.1479	0.00034	0.574	8.7	85.6	7.51 0.03
1200-08	0.1828	0.00021	0.577	8.4	91.0	7.55 0.02
					Wtd. Ave. =	7.54 0.03
Spearhead Member (BH86N9):						
J = 0.007255 ± 0.000005						
1201-01	0.0323	0.00019	0.590	11.5	91.3	7.71 0.12
1201-02	0.0363	0.00006	0.580	9.0	97.1	7.57 0.04
1201-03	0.0358	0.00011	0.583	6.4	94.8	7.61 0.06
1201-04	0.0284	0.00001	0.585	6.0	99.2	7.64 0.03
1201-05	0.0377	0.00002	0.580	6.6	98.7	7.58 0.03
1201-06	0.0303	0.00053	0.585	11.0	78.7	7.64 0.36
1201-07	0.0353	0.00008	0.584	10.4	96.2	7.62 0.04
1201-08	0.0359	0.00003	0.584	7.9	98.4	7.63 0.02
					Wtd. Ave. =	7.61 0.03
Spearhead Member (83G Spear):						
J = 0.005512 ± 0.00001						
1761-1	0.0359	0.00022	0.765	0.6	92.0	7.59 0.13
1761-2	0.0318	0.00040	0.794	0.2	86.9	7.88 0.58
1761-3	0.0429	0.00036	0.776	0.5	88.0	7.70 0.16
1761-4	0.0339	0.00040	0.725	0.5	85.5	7.19 0.14
1761-5	0.0357	0.00017	0.758	0.1	93.8	7.52 0.07
1761-6	0.0839	0.00008	0.817	0.3	97.4	8.11 0.26
					Wtd. Ave. =	7.53 0.19
Rejected as too old (detrital contaminant suspected):						
1761-7	0.4386	0.00064	1.193	0.2	87.3	11.83 0.55
Spearhead Member (LABCAN081):						
J = 0.005512 ± 0.00001						
1759-1	0.1120	0.00279	0.665	0.2	44.6	6.60 0.83
1759-2	0.0353	0.00024	0.763	1.6	91.5	7.57 0.05
1759-3	0.0363	0.00005	0.804	0.4	97.9	7.98 0.20
1759-4	0.0349	0.00007	0.798	1.1	97.2	7.91 0.07
1759-5	0.0337	0.00007	0.774	1.6	97.2	7.68 0.04
					Wtd. Ave. =	7.69 0.13

[§]Errors in age quoted for individual runs are 1σ analytical uncertainty; errors in age quoted for weighted means are 1σ weighted standard deviation. Ca/K is calculated from $^{37}Ar/^{39}Ar$ using a divisor of 0.51. $^{40}Ar^*$ refers to radiogenic argon. 'Moles ^{40}Ar' refers to the estimated total moles of ^{40}Ar released during fusion based on spectrometer sensitivity considerations. $\lambda = 5.543 \times 10^{-10}$ y^{-1}. Isotopic interference corrections: $(^{36}Ar/^{37}Ar)_{Ca} = 2.58 \times 10^{-4}$, $(^{39}Ar/^{37}Ar)_{Ca} = 6.7 \times 10^{-4}$, $(^{40}Ar/^{39}Ar)_{K} = 2 \times 10^{-3}$.

analyses of purified air. The constants used in age determination are those from the Subcommission of Geochronology (Steiger and Jager, 1977).

RESULTS

Analytical $^{40}Ar/^{39}Ar$ data for the laser-fusion analyses are provided in table 1. In total, eight $^{40}Ar/^{39}Ar$ analyses were obtained from the Civet Cat Canyon Member, while 20 were obtained from the Spearhead Member. The radiogenic ^{40}Ar content of most analyses was greater than 85%, indicative of unaltered material. Analyses of 83G SPEAR and LABCAN081 yielded about an order of magnitude less gas ('Moles ^{40}Ar' in table 1) than BH86N33 and BH86N9 due to smaller grain size; this difference is reflected in the greater analytical uncertainties of the former (~0.08 vs. 0.03 Ma). The overall weighted mean of the three sample means of the Spearhead samples is 7.61 ± 0.03 Ma (1σ standard deviation; 0.01 s.e.m.). The single Civet Cat sample yielded a weighted mean age of 7.54 ± 0.03 Ma (0.01 s.e.m.).

The analytical results and K-Ar ages determined for samples DN-CST and 3DN8-18A are given in the "K-Ar Sample Descriptions" section below.

DISCUSSION

Previous determinations of the age of the Civet Cat Canyon and Spearhead Members by the conventional K-Ar method have all yielded results younger (by as much as 20%) than the laser-fusion $^{40}Ar/^{39}Ar$ results reported here. Ages of about 6.3 Ma have been reported for both members by Kistler (1968) and Noble and others (1984, 1988). Armstrong and others (1972) presented three K-Ar ages from the Spearhead Member that average 7.0 Ma (ages recalculated using presently accepted constants). Foley (1978) presented K-Ar age determinations of 7.48 ± 0.20 Ma (biotite) and 7.19 ± 0.54 Ma (alkali feldspar) on the Civet Cat Canyon Member and dates of 7.52 ± 0.17 Ma (biotite) and 7.36 ± 0.12 Ma (alkali feldspar) on a stock that cuts intracaldera tuffs of the Civet Cat Canyon Member on Stonewall Mountain. We also report here two new conventional K-Ar determinations of 6.2 ± 0.2 and 7.3 ± 0.3 Ma (specimens DN-CST and 3DN8-18A, respectively) obtained recently on sanidine from exposures of the Spearhead Member in the vicinity of the Bullfrog Hills (Weiss and others, 1988). The spread in K-Ar ages obtained from the Civet Cat Canyon Member indicate that dating alkali feldspar by conventional K-Ar methods gives widely discrepant ages.

While such discrepancies are not usually this large (e.g., Samson and Alexander, 1987), we have found that K-feldspar ages of middle to late Tertiary ash-flow tuffs in the Great Basin produced by the conventional K-Ar technique are often younger than corresponding $^{40}Ar/^{39}Ar$ ages. Commonly, young conventional ages on K-feldspar are attributed to insufficient fusion times and inadequately high fusion temperatures, such that not all $^{40}Ar^*$ is expelled from the melt. However, we have encountered young ages even when the feldspar melt was vaporized during fusion. Apparently, a significant amount of the $^{40}Ar^*$ released by the melt is trapped through ion implantation on the alkali mirror formed on the walls of the fusion bottle during the higher-temperature steps of RF excitation. We have demonstrated this effect by remobilizing the alkali mirror with a flame and measuring the argon recovered.

SAMPLE DESCRIPTIONS

1. *DN-CST* K-Ar
Spearhead Member, Stonewall Flat Tuff (at Currie Well in the western Bullfrog Hills; SW/4 NE/4 S22,T11S,R45E; 36°58.09'N, 116°55.27'W; Bullfrog 15' quad., Nye Co., NV). *Analytical data:* K_2O = 6.61%, $^{40}Ar^*$ = 5.87555 x 10^{-11} mole/g, $^{40}Ar^*/^{40}Ar$ = 75.6%. *Collected by:* D. C. Noble, University of Nevada, Reno. *Analyzed by:* E. H. McKee, U.S. Geological Survey. *Comment:* Sample consisted of flat-lying, partially welded, glassy ash-flow tuff overlying basalt dated at 8. 1 Ma (Marvin, and others, 1989).
(sanidine) 6.2 ± 0.2 Ma

2. *3DN8-18A* K-Ar
Spearhead Member, Stonewall Flat Tuff (in first drainage N of Beatty Wash; NE/4 SE/4 S23,T11S,R47E; 36°57'47"N, 116°40'55"W; Bare Mountain 15' quad., Nye Co., NV). *Analytical data:* K_2O = 6.76%, $^{40}Ar^*$ = 7.06712 x 10^{-11} mole/g, $^{40}Ar^*/^{40}Ar$ = 40.0%. *Collected by:* D. C. Noble and K. A. Conners, University of Nevada, Reno. *Analyzed by:* E. H. McKee, U.S. Geological Survey. *Comment:* Sample consisted of glassy, partially welded, flat-lying ash-flow tuff.
(sanidine) 7.3 ± 0.3 Ma

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