Significance of the Age and Identity of a Volcanic Ash Near DeSoto, Kansas, with Respect to the Enclosing Terrace Deposits

by

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Submitted to the Department of Geology and the Faculty of the Graduate School of the University of Kansas in partial fulfillment of the requirements for the degree of Master of Science

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April 9, 1987
Date thesis accepted

1987

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ABSTRACT

A layer of volcanic ash within terrace fill is located north of DeSoto, Kansas. Terraces along the Kansas River have not been worked on extensively since the 1950's and there were no absolute dates associated with them. The ash was determined to be within fill of a Buck Creek terrace remnant, which had been assigned an Illinoian age. A determination of the identity of the ash was based on shard shape and color, phenocryst assemblage, expected geographic distribution of different ashes, glass chemistry, magnetic polarity, and fission-track age of the glass shards. All this information indicates that the ash is the 0.62 my Lava Creek B Ash from the Yellowstone area. This ash has been used as a marker for the Late Kansan. Though there is presently disagreement as to dates for stage boundaries within the Pleistocene, a Late Kansan ash can not be within Illinoian fill. A precise statement as to the relationship between glacial stages and the formation of the Buck Creek terrace must await agreement on the dates of Pleistocene stage boundaries.

The methodologies of both Dr. John Boellstorff and Dr. Charles Naeser, respectively, were followed in dating the glass shards, and Naeser's techniques were followed in an unsuccessful attempt to date zircon phenocrysts. Several problems exist in determining glass and zircon fission-track ages, including uncertainties in neutron fluence determination,

fission decay rate of ^{238}U, and proper procedure for etching and counting glass shards. Though alluded to in the main text, a discussion of these problems is reserved for an appendix. It is the author's opinion the a ζ factor method of age determination should be used and an interlaboratory age standard set up for glass fission-track dating. A decision as to whether to use the bulk etch technique or polished slide technique should be based on research goals, and the age and morphology of the shards. Though the polished slide technique probably results in more precise age determinations; the bulk etch technique is much faster for fine grained, young ashes and should yield reasonable dates. Its use as a preliminary method to determine suitability of a particular ash sample for fission-track dating is recommended.

ACKNOWLEDGMENTS

Without the help of a great many people, this thesis would not have been possible. First, I would like to express my appreciation to the Department of Geology at the University of Kansas for their generous financial support. This research was also funded by a Sigma Xi Grant-in-Aid of research and a portion of a National Geographic Society grant awarded to Dr. Larry Martin of the Systematics and Ecology Department at KU. Dr. Jan Schreurs of the Corning Museum of Glass generously donated one of the dosimeters used. Several ash samples were irradiated within the Georgia Institute of Technology Research Nuclear Reactor with the help of Drs. Bob Kirkland and Ratif Karam; while the KU radiation safety officer, Dr. Benjamin Friesen, helped in the reacquisition of my samples following irradiation. Dr. Kenneth Kodama of Lehigh University (Bethlehem, PA) determined the magnetic polarity of the ash. I had many helpful discussions with Dr. Glen Izett of the U. S. Geological Survey, Denver about phenocrysts and expected geographic distributions of different Pleistocene ash falls and he also performed glass chemistry analyses.

Drs. Albert Rowell and Anthony Walton assisted with photographic work, and many other KU faculty provided help and support along the way. Dr. Gerald James of the Kansas State Geological Survey helped me in my attempt to identify clays by

X-ray diffraction. I had extensive helpful talks with both Dr. Curtis Sorenson of the KU Department of Geography and Ms Ula Moody of Kilgore College (Kilgore, TX) about the soils and general stratigraphy of my terrace. I appreciate the time Dr. Gunther Schlager of the Systematics and Ecology Department at KU spent with me in deciphering statistics.

Dr. John Boellstorff (presently at Amoco Production Research, Tulsa) spent time teaching me his fission-track methodology at his University of Nebraska lab and also generously donated samples of his dosimeter and sample irradiation containers. His subsequent help through several phone calls and letters is also much appreciated. Dr. Charles Naeser of the U. S. Geological Survey, Denver helped in innumerable ways, including spending much time in his lab and on the phone helping me with fission-track dating, irradiating glass and zircon samples, and opening his lab to my use on several occasions. Without the help of Drs. Charles and Nancy Naeser, this thesis would not have been possible.

In a different way, this is also true of the support received from my fellow graduate students at the University of Kansas, especially Karen Spray, Steve Smith, Jean Hall, and Brian McNeice - thank you all! Thanks to Jeff Coble who performed several heavy mineral and magnetic separations for me, and to Jennifer Sims at the Kansas State Geological Survey for allowing me to make use of her drafting talents.

I would also like to express my thanks to Mr. Dalaba (owner of the property on which my terrace is located) and his family for their general friendliness throughout the course of my fieldwork. Thanks to my committee members Drs. Ernest Angino and Randy Van Schmus who provided help of many sorts over the past few years. Last, but far from least, I would like to express my thanks for the continuous help and encouragement I received from my mentor Dr. Wakefield Dort. Thank you for all those times you were available at strange hours for long discussions on everything.

Preface

The research undertaken for preparation of this report fits naturally into two categories. Primary motivation for the study was an effort to obtain and interpret an absolute date for terrace alluvium, a heretofore unknown aspect of Kansas River history. However, this attempt led to encounters with the intricacies of fission-track dating of volcanic ash and the necessity for evaluating differing laboratory procedures.

The dichotomy within the research required that the report also be in two parts, each of which could stand alone. Part I describes the conventional geological investigation of a sedimentary sequence and associated land forms, an absolute date obtained for one specific horizon, and possible correlations with existing relativistic chronologies. Part II describes and evaluates laboratory procedures used to obtain the absolute age of a volcanic ash present in the sedimentary sequence.

PART I

STRATIGRAPHY, AGE, AND INTERPRETATION OF A VOLCANIC ASH-BEARING SEQUENCE NEAR DESOTO, KANSAS

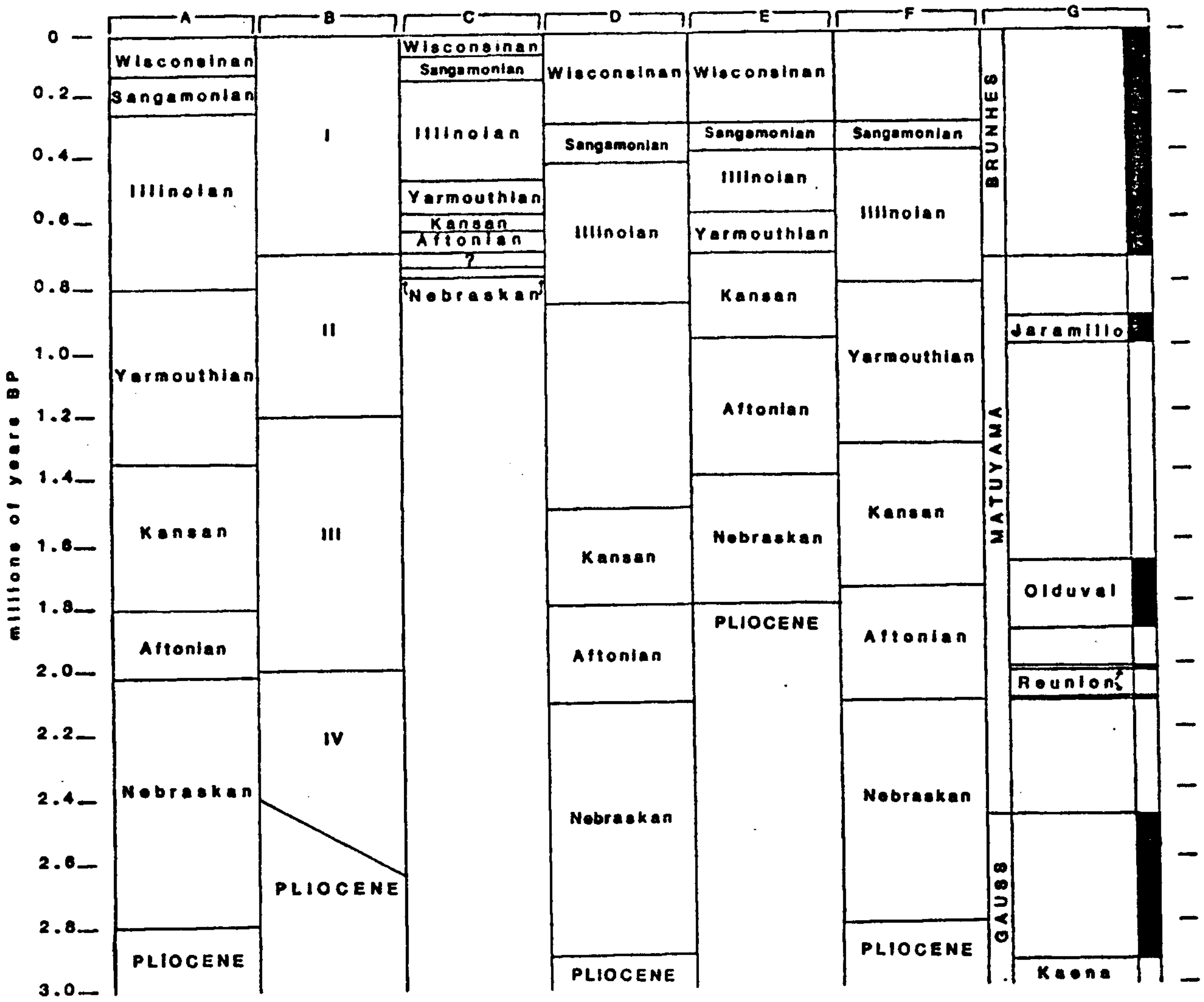
INTRODUCTION

Alluvial terraces are common along many rivers and streams. Obtaining an understanding of the formation and age of these terraces can contribute to an understanding of the geologic history of the entire watershed. However, it is difficult to assign an age to an alluvial terrace. As the terrace itself is a two-dimensional surface, there is no material to which one can assign an absolute date. The fill below a terrace usually can be assigned only a relative date with respect to the age of materials with which it is in contact; for example, the Buck Creek fill truncates Kansan glacial deposits and is capped by a well-developed (Sangamon?) soil and/or discontinuous Peorian Loess (Davis and Carlson, 1952).

While the timing of terrace formation with respect to the stages of Pleistocene chronology has frequently been discussed, the absolute dates of these stages are not agreed upon (see Boellstorff, 1978; Dube, 1985; Reed and Dreezen, 1965). A comparison of some of these chronologies is in table 1. The existence of datable material within terrace fill would thus be of great value. A volcanic ash is an excellent candidate (Swineford, 1963).

Approximately 1.8 km north of DeSoto, Kansas a terrace and associated fill have been exposed in a driveway cut. This

Table 1. Comparison of recently published chronologies for the Pleistocene. A) Beard, 1969. Marine, based on planktonic data, paleotemperatures and magnetic polarity information from Gulf of Mexico cores. B) Boellstorff, 1978a. Continental record correlated with oxygen isotope stages in the Gulf of Mexico and dating at Vrica, Italy; suggests revising or dropping Pleistocene stage names and uses "chronozones". C) Dube, 1985. Tentative Midcontinent record, based on best dates of type sections. D) Dube, 1985. Marine conceptual Midcontinent, based on correlation of paleotemperatures, eustatic cycles, and oxygen isotope stages in the Gulf of Mexico. E) Van Eysinga, 1978. Accepted as standard by the Geological Society of America. F) Smith, 1985. Marine record, based on all available data from the Gulf of Mexico and Vrica, Italy. G) Mankinen and Dalrymple, 1979. Magnetic polarity, black = normal.


A
B
C
D
E
F
G
0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
millions of years BP
Wisconsinan
Sangamonian
Illinoian
Yarmouthian
Kansan
Aftonian
Nebraskan
PLIOCENE
I
II
III
IV
PLIOCENE
Wisconsinan
Sangamonian
Illinoian
Yarmouthian
Kansan
Aftonian
?
?Nebraskan?
Wisconsinan
Sangamonian
Illinoian
Kansan
Aftonian
Nebraskan
PLIOCENE
Wisconsinan
Sangamonian
Illinoian
Yarmouthian
Kansan
Aftonian
Nebraskan
PLIOCENE
Sangamonian
Illinoian
Yarmouthian
Kansan
Aftonian
Nebraskan
PLIOCENE
BRUNHES
MATUYAMA
GAUSS
Jaramillo
Olduvai
Reunion?
Kaena

location is herein named the Dalaba Site after the present property owner. The terrace fill contains four main sedimentary units including a volcanic ash (see figure 1). The lowermost sediment is alluvium consisting of alternating sand and clay. A paleosol was developed in this alluvium and then partially removed by erosion. A volcanic ash was deposited on the eroded surface, followed by deposition of lacustrine sediments. Finally, a loess cover was deposited on the terrace surface.

There are four terraces generally recognized in the lower Kansas River valley, their names and heights are listed in table 2. The terraces along the lower Kansas River were mentioned in various reports during the mid 1930's and 40's. Serious study began in the early 1950's with work by Davis (1951) and Carlson (1952). At that time it was thought that one terrace had been formed during each of the last three of the four periods of continental glaciation of classical Pleistocene terminology. Obtaining an absolute date on material within one of these terraces can be used toward testing this hypothesis.

Davis and Carlson (1952) made detailed studies of those terraces between Lawrence and Topeka, Kansas. They stated that the Kansas River, as it exists today, formed during the Kansan glaciation as an ice-margin stream, though a precursor stream existed during the Nebraskan glaciation. The valley of the Kansas River was filled with up to 80 feet of glacial outwash, forming the Menoken Terrace surface. During the Illinoian

Figure 1. Terrace face with 40 cm grid. The four main sedimentary units are labeled as interpreted. A paleosol is developed in the upper part of the alluvial sequence - note dark horizon below light-colored ash. The base of the paleosol is approximately coincident with the top fence strand. (Clippers = 1 m) (June, 1984)


West
East
loess
lacustrine
ash
(paleosol)
alluvium

TERRACES OF THE LOWER KANSAS RIVER VALLEY

Name	Height	Age
Holliday Complex	(part of floodplain?)	Recent
Newman	2.5 m above floodplain	Wisconsinan
Buck Creek	11m above floodplain	Illinoian
Menoken	24-30m above floodplain	Kansan

adapted from Davis and Carlson, 1952

Table 2. The ages listed are those generally assumed for the respective terraces. The heights are averaged from Davis and Carlson (1952). The Newman Terrace was largely covered by water during the 1951 Kansas River flood (McCrea, 1954). The Holliday Complex contains several low scarps, and though generally recognized as a terrace, it was included as part of an irregular floodplain surface by Davis and Carlson.

glaciation, the valley was incised 50 to 60 feet and then aggraded to 35 feet above the modern floodplain level. The resulting surface, preserved only at the mouths of tributary streams, is known as the Buck Creek Terrace. Another cut-fill cycle occurred during the Wisconsinan glaciation forming the Newman Terrace. This terrace is still being aggraded by exceptionally severe floods. A fourth possible terrace, the Holliday Terrace Complex, was defined by McCrea (1954) and named by Dufford (1958). It is a complex of several small, scarped surfaces which Davis and Carlson (1952) included as irregularities on the modern floodplain surface.

The terrace at the Dalaba Site is probably a Buck Creek Terrace remnant on the basis of comparisons of terrace height and fill with published data. Both the Buck Creek Terrace (surface) and the underlying alluvium have been called Illinoian (Davis and Carlson, 1952), but on the basis of very little evidence. If the included volcanic ash can be assigned an absolute date, it can then be used to obtain a better defined age for the terrace. The ash was initially inferred by the author to be a Pearlette ash (e.g. a Yellowstone source ash) on the basis of color, shard shape, and the absence of biotite phenocrysts.

Part I is organized in the order in which information was obtained. Descriptions of the general geomorphology and stratigraphy of the terrace fill are presented first. There then follows a discussion of identification of the volcanic ash, first on the basis of petrographic evidence, then according to its fission-track age. These data lead to conclusions regarding the significance of its presence at this specific locality. Details of laboratory procedures and analyses are given in Part II.

GEOMORPHOLOGY

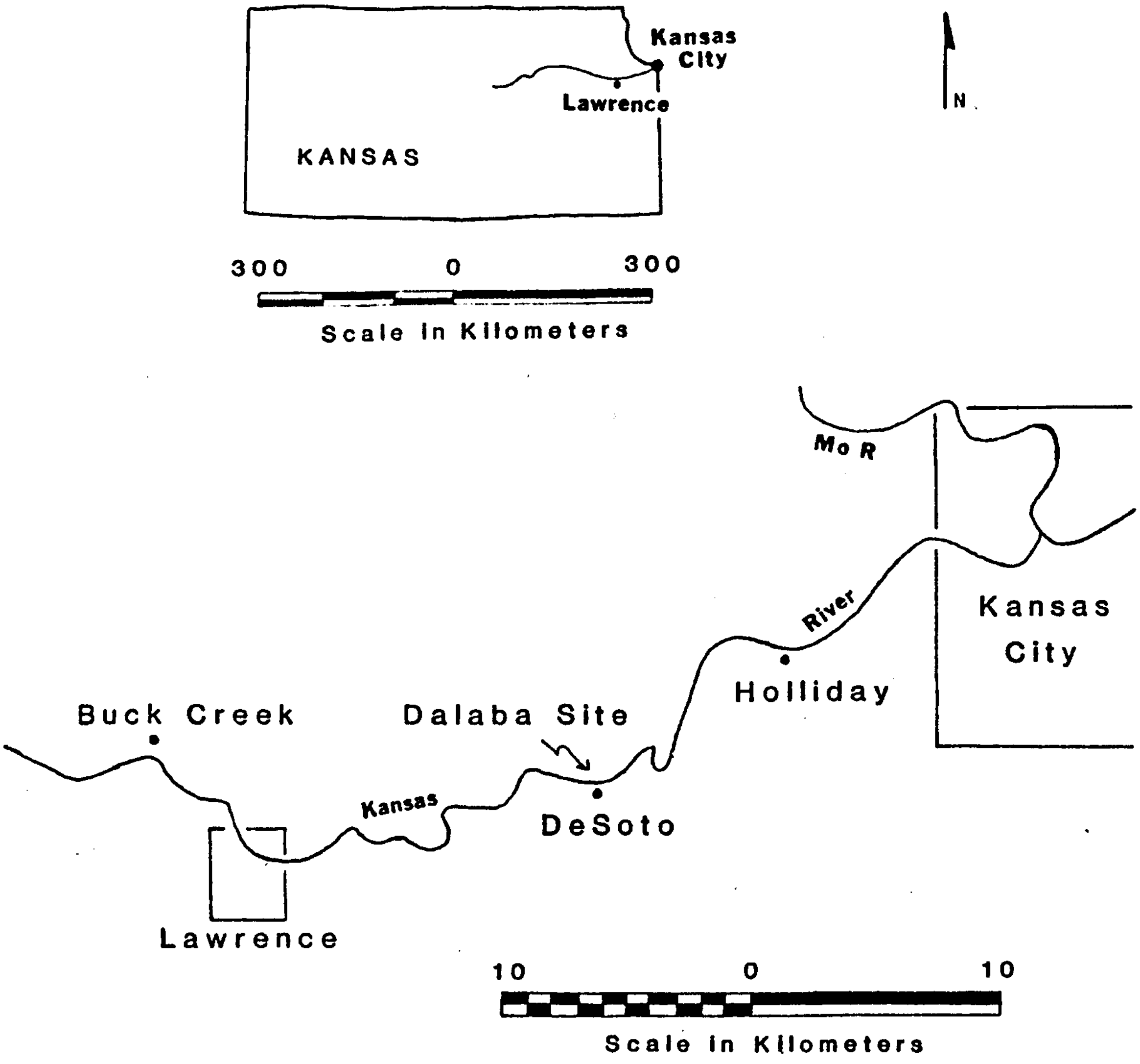
Site Description

The ash is exposed in a driveway cut along the face of a small, triangular terrace remnant on the north side of the Kansas River approximately 1.8 km north of DeSoto, Kansas, off Leavenworth County road 26 (see figures 1 (in Introduction) and 2). The Land Office Grid location is SW 1/4, NE 1/4, NE 1/4, sec 21, T. 12S, R. 22E. This locality has been designated as the Dalaba Site by the author. The Stanton Limestone of the Lansing Group, Missourian Stage of the Pennsylvanian crops out on the valley walls to either side of this terrace remnant (see figure 3.)

The main face of the exposed terrace fill is about four meters (12 ft) high and about five meters (16 ft) long. The terrace surface is at an elevation of nearly 815 ft (250 m).[1] This is about 35 feet (10 m) above the floodplain. Elevations of the rolling uplands can reach 1000 ft (305 m) within a mile of the Kansas River. Drainage in the uplands is generally good. Mean annual rainfall is about 35 in/yr (8.9 cm/yr) and the mean annual temperature is about 54°F (12.2°C).

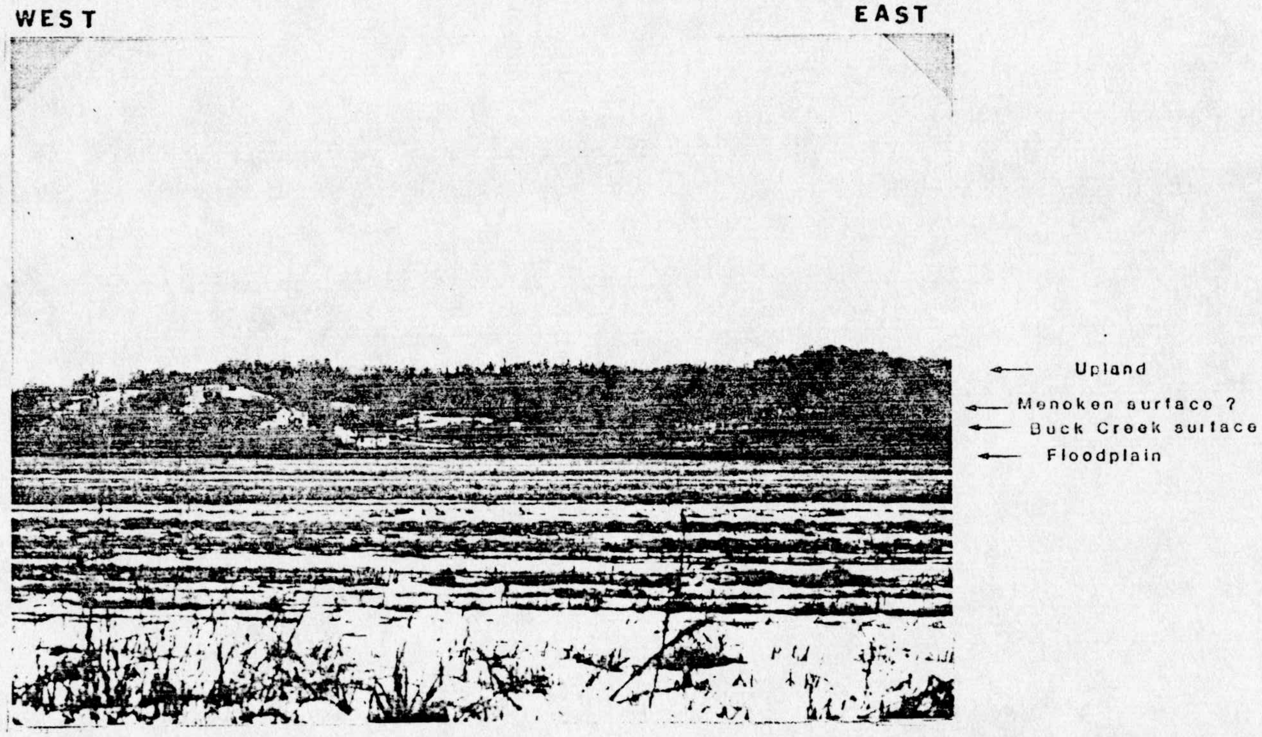
[1] Measurements in both English and metric systems are used throughout this report. When the original measurement was made in the English system, a metric equivalent will be given. English equivalents to metric measurements will be given only to add clarity to a specific discussion. The first measurement is given in the original system of measurement.

Figure 2. Location map. The terrace containing the volcanic ash is located across the Kansas River from the city of DeSoto, Kansas, at the position marked "Dalaba Site".



Adapted from Hollen, 1982

Figure 3. Geomorphic context of the terrace. In the foreground is the modern Kansas River floodplain. The snow delineates the labeled land surfaces in the background. Limestone crops out from the valley wall at a level below the Buck Creek surface both east and west of the terrace remnant. The diagonal slope is the Dalaba residence driveway. (Dec., 1985)


WEST
EAST
Upland
Menoken surface ?
Buck Creek surface
Floodplain

Four terraces have been recognized in the lower Kansas River Valley (Davis and Carlson, 1952; McCrea, 1954; Holien, 1982), their names, heights and presumed ages are listed in table 2 in the Introduction. A determination of the identity of the terrace remnant at the Dalaba Site is complicated by its small areal extent. The following methods were used to establish which terrace is present at the Dalaba Site.

Though the terrace surface has been scraped by a bulldozer by the property owner, it is still about 815 ft (250 m) in elevation (the ash is at an elevation of 810 ft (O'Conner, 1971)). The Newman Terrace is flooded during major floods (McCrea, 1954), while the present terrace surface at the Dalaba Site is about 15 ft (4.5 m) above the level reached by the last great flood in 1951. The Buck Creek Terrace is at an elevation of 880 ft (270 m) at Buck Creek, KS, and at 785 ft (240 m) at Holliday, KS, (Davis and Carlson, 1952) which indicates the elevation at DeSoto should be approximately 815 ft (250 m). Elevations for the Menoken Terrace at Lawrence, KS, and Holliday, KS, are 925 ft (280 m) and 890 ft (270 m), respectively (Davis and Carlson, 1952). These towns are approximately equidistant up and down river from DeSoto. Thus, if the remnant were part of the Menoken Terrace, its surface should be at an elevation of about 900 ft (275 m). The elevation of the terrace remnant at the Dalaba Site is therefore consistent with the elevation of the Buck Creek Terrace.

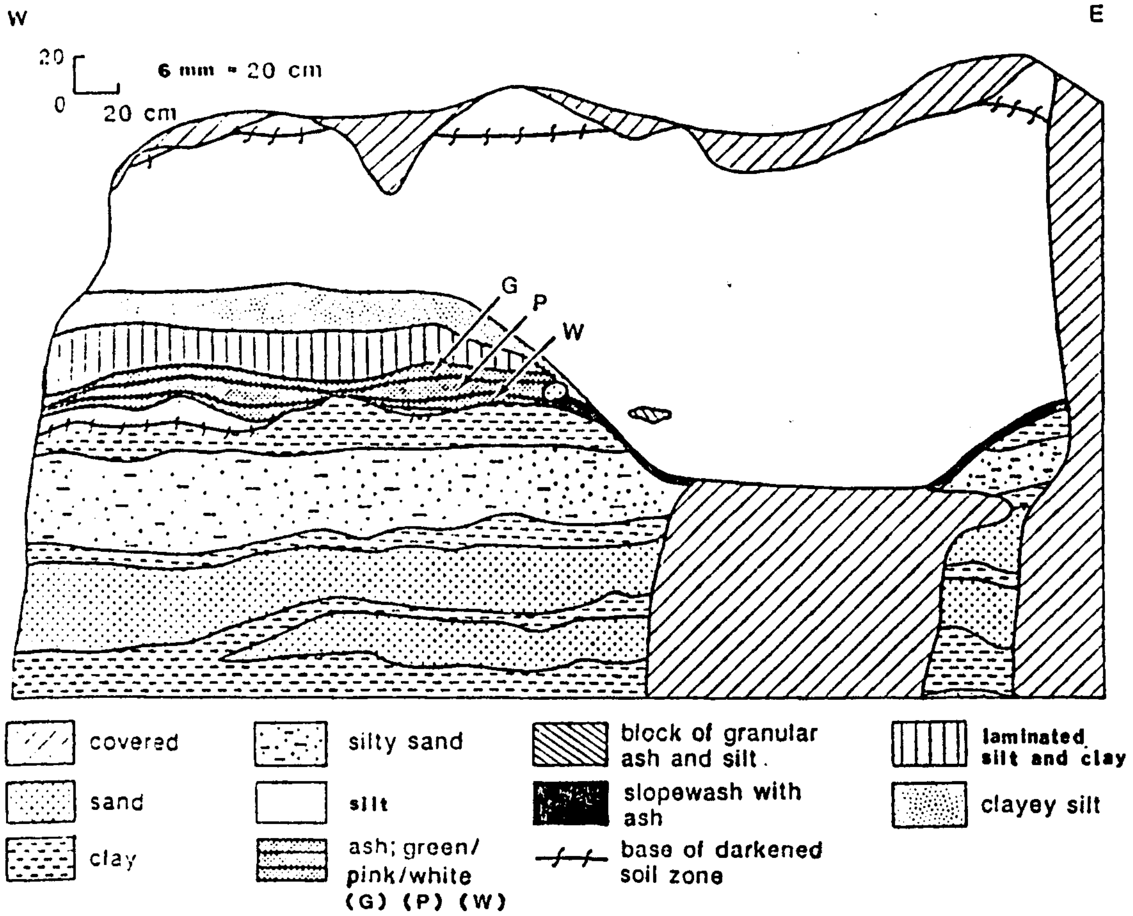
The Menoken Terrace fill has a bedrock floor which is about 20 ft (6 m) above the present floodplain (Davis and Carlson, 1952). Though bedrock crops out of the valley wall to either side of the terrace at the Dalaba Site, there is no evidence that the terrace fill is lain on a bedrock floor above present floodplain level. The description of Buck Creek fill given by Davis and Carlson (1952) includes the presence of a well-developed soil (which they called Sangamon) at the surface and a discontinuous loess (Peorian) cover. The terrace at the Dalaba Site contains a paleosol developed in the alluvial fill and a relatively thick loess mantle. For these additional reasons, the terrace is most likely a Buck Creek Terrace remnant.

Stratigraphy and Sedimentology

The terrace fill at the Dalaba Site consists of four main units. These are the lower alluvial sequence, paleosol, volcanic ash and lacustrine material, and a loess cover (figure 4). A different sedimentary environment is required for the deposition and/or preservation of each of these four main units. A general description of each of these units follows. Detailed descriptions and particle size analyses of each sub-unit can be found in Appendix A.

The lower portion of the exposed terrace fill consists of alternating sand and clay. Nearly all contacts are sharp, commonly outlined by sesquioxides. One sand layer pinches out

Figure 4. Schematic profile of the exposure of the terrace face. Compare this drawing with figure 1. (Drawn June, 1984.)


W
E
20
0
6 mm = 20 cm
20 cm
G
P
W
covered
sand
clay
silty sand
silt
ash; green/ pink/white (G) (P) (W)
block of granular ash and silt.
slopewash with ash
base of darkened soil zone
laminated silt and clay
clayey silt

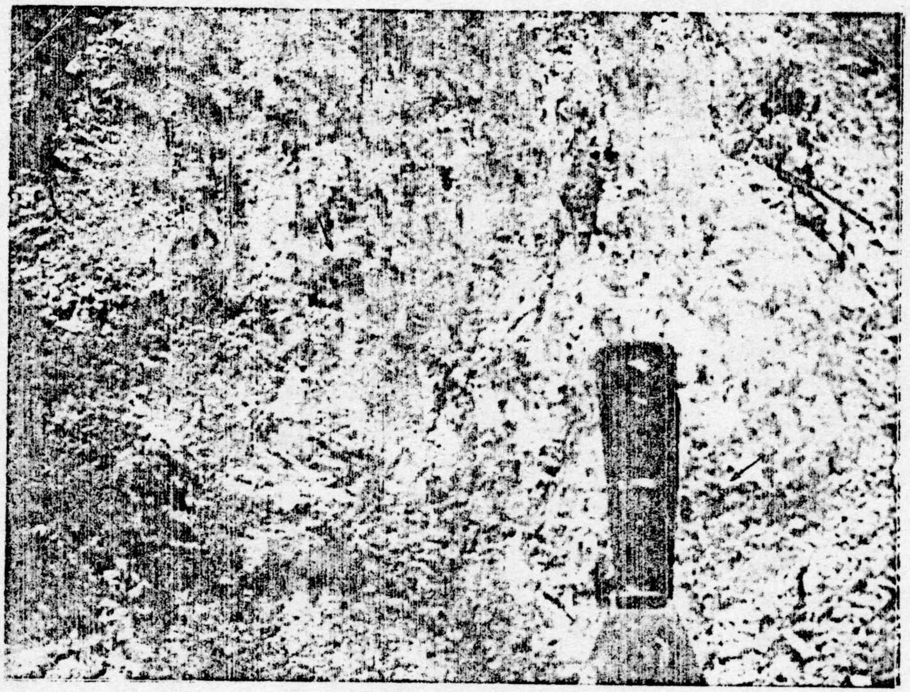
within the limits of the exposure.

Within the upper part of the alluvial sediments the presence of manganese nodules and root casts outlined with sesquioxides is evident (figure 5). Upon closer examination, a vertical color change from dark brown (7.5YR4/4) to pale brown (10YR6/3) is visible within portions of the uppermost clay sub-unit. Particle size analysis of these upper alluvial sub-units (see Appendix A) does not show nearly the alternation expected for sand to clay alluvial sub-units. Weak blocky structure and clay skins (clay accumulations around soil particles) are also present. These observations suggest the presence of a paleosol at the top of the alluvial sequence. Pedogenic processes have reduced the particle size variations at the top and it is likely that added organic matter has caused the color change. The lower limit of observed pedogenic influence is within the lowest clay unit depicted in figure 4, approximately 150 cm below the color change.

This darker portion of the uppermost alluvial clay is not present across the entire exposed terrace front. Indeed, the contact between this clay and the overlying ash is very sharp and irregular. Thus, it is probable that a substantial portion of the paleosol has been removed by erosion. Moody (pers. comm., 1985) interpreted the upper part of what remains as a buried B2 horizon.

The overlying unit is a volcanic ash composed almost

Figure 5. Lower horizons of paleosol. Many mottles are visible on the scraped surface. Several root casts are present, some marked by arrows. Black manganese nodules are also common. Location of the view on figure 1 is the scraped area to the lower left of the measuring tape. (Trowel = 12 cm.)



entirely of glass shards. Wilcox (1965) stated that in order to preserve ash, sedimentation should be continuous, such as in a lake or valley bottom. The lower contact includes irregularities such as burrows and upward prominences of the paleosol. There is no recognizable sediment between the paleosol and the ash, thus erosion and deposition were probably not widely separated in time. These suggest that though erosion had taken place fairly recently, the area was covered with standing water when the ash fell. The preservation of a sharp upward protrusion of paleosol (see figure 6) indicates that the water was not flowing, or it would have been removed.

The ash bed consists of three different portions which are white, pink, and green colored, respectively (see figure 7). The entire ash bed is 10 to 20 cm thick. The lowest portion consists almost entirely of glass shards and is white (10YR8/2) in color. This portion is only 0 to 7 cm thick and is interpreted as an air-fall ash. As Swineford (1963, p. 360) stated, "It is difficult to imagine how segregations of shards 95 percent pure formed under any conditions other than those that immediately followed the eruption." Further evidence includes the combination of an extremely sharp and irregular lower contact, and gradational upper contact; preservation of delicate skeletal zircon phenocrysts; and the composition of the upper two colored portions. The pink and green portions contain increasing amounts of clay, respectively, and are interpreted as secondary ash

Figure 6. Basal ash contact. As can be seen in figure 1. the basal contact contains both upward protuberances (as above) and down-ward projecting burrow fills. This view is a close-up of a portion of the contact. It is probable that the ash was deposited in a standing body of water not long after an erosional event. (Trowel = 12 cm)

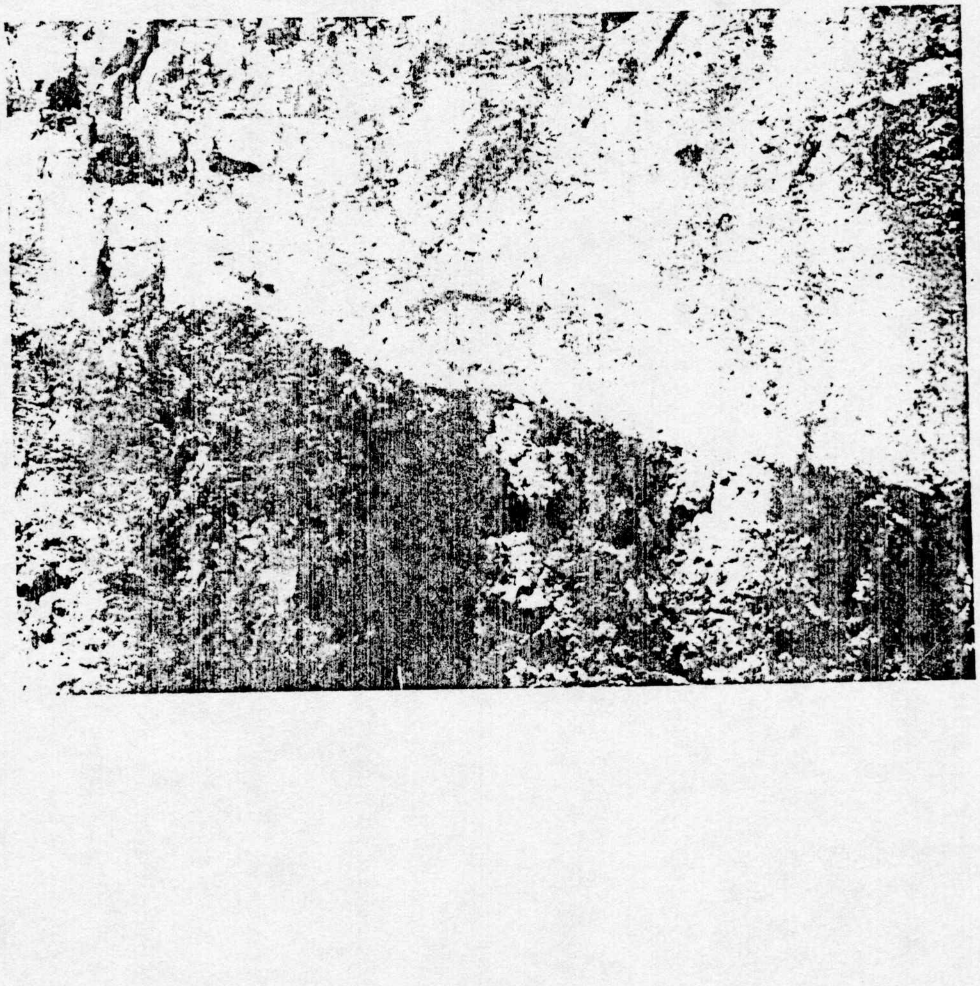
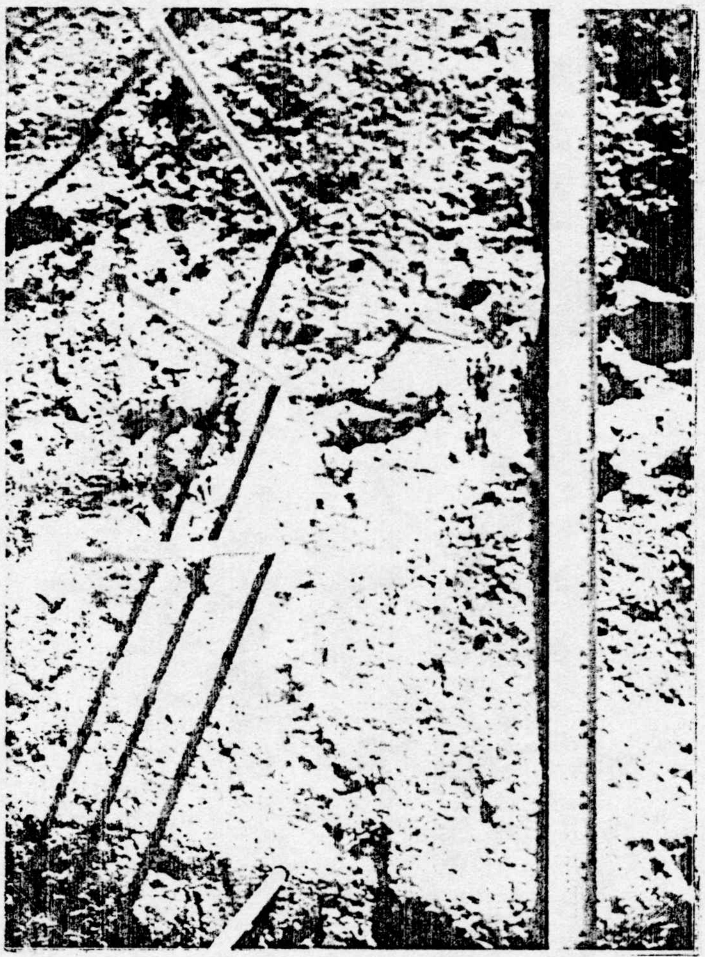


Figure 7. Colored ash layers. The approximate contacts of the three differently colored layers of the ash are marked by the dowels. These are the green pink, and white layers, respectively. Note the friable appearance of the white layer in contrast to the clay-rich appearance of the two upper layers. The transition zone is blocky and is best developed to the right of the tape measure. The brown area to right center is a filled burrow.

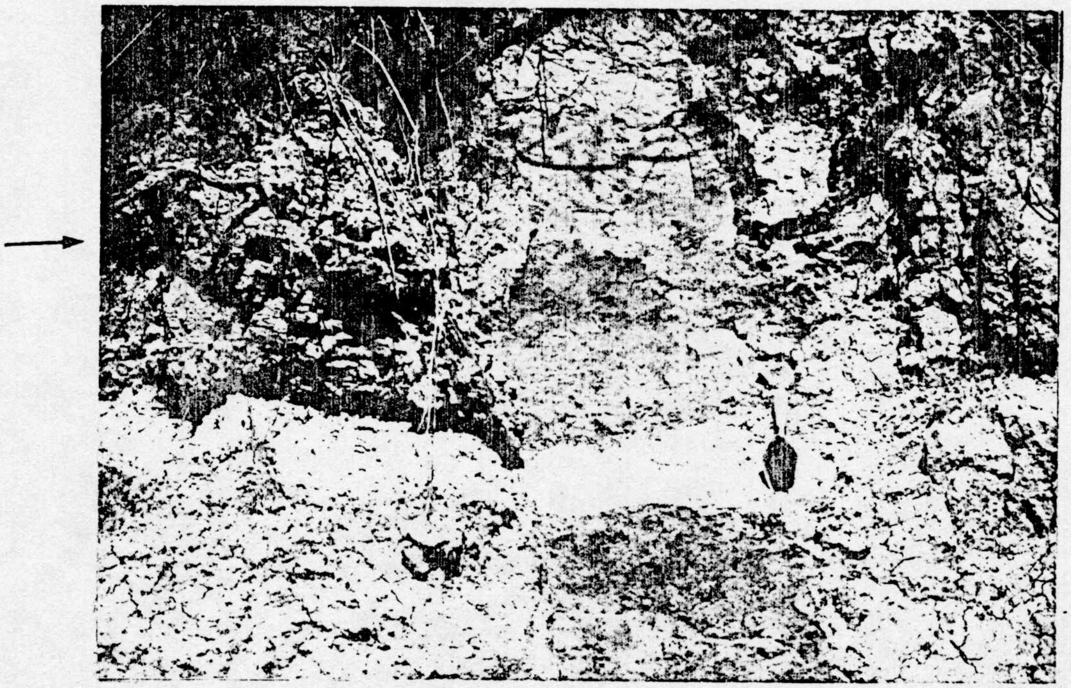


deposits washed in from surrounding areas. The central (pink) portion makes up the bulk of the ash thickness and was probably washed into the depositional basin shortly after the original air-fall. Using the Munsell color system, the color ranges from pinkish white (7.5YR8/2) to very pale brown (10YR7/4) in color, averaging pink (7.5YR8/4). The upper (green) portion varies from light olive gray (5Y6/2) to grayish yellow green (5GY7/2) in color and is a very plastic, though glass-rich clay.

An alteration zone up to two centimeters thick is present between the green portion and the overlying lacustrine sediments. This zone is very blocky in texture and probably consists of highly altered ash. This zone, though somewhat mottled, is approximately grayish green (10GY6/2) in color.

The material directly above the ash can be parted along horizontal laminations. This sub-unit is more clay-rich than the overlying loess; it can be ribboned between the fingers and shows clay-cracks when dry (see figure 8), unlike the loess. This can be attributed to the alternating silt and clay laminations, much like varves, which are present. This material was interpreted as lacustrine by Moody (pers. comm., 1985). Moody also located clay skins near the center of the lacustrine unit and interpreted the presence of a weak B paleosol horizon. The contact between the lacustrine sediment and the overlying material is erosional. A slight color change also occurs. The lacustrine material is pale brown (10YR6/3) while the overlying sediment is brown to

Figure 8. Lacustrine unit. Laminations within the lacustrine unit (above the ash) are visible in the unscraped portion to the left in the figure. In the scraped portion, note the color change at the same level at which the laminations disappear (arrow). The darkened zone of the paleosol is also visible in the scraped portion. (Trowel = 35 cm)



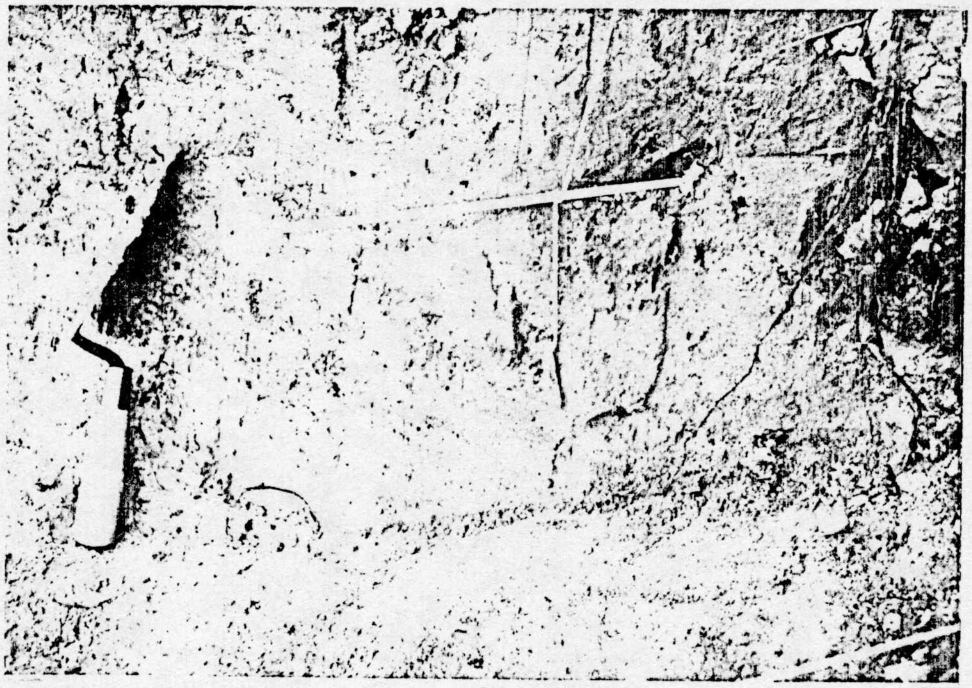
yellow brown (10YR4-5/4). Manganese nodules are common throughout this sub-unit as well as throughout the underlying ash.

All the sedimentary units discussed thus far are cut by a large channel-like feature which can be seen in the re-entrant near the east end of the exposed terrace face (see figure 1). On both sides of this feature a wispy trail of ash particles can be traced downward from the level of the erosionally truncated primary ash bed and could even be followed across the excavated floor of the re-entrant about 10 cm out from the present vertical terrace face (see figure 9). Four small (about 1 X 2 cm) pebbles of siltstone and chert were located in the east corner of the re-entrant behind the ash trail within the fill. A few similar pebbles were found at various locations in the fill, but never more than about 30 cm from the edge of the feature. A block of material (about 28 X 8 cm in size) composed of granular silt and ash particles was found about 35 cm out from the ash trail within the fill. Its location is visible in figure 1 and it is shown schematically in figure 4. As is also shown in figure 4, there is an anomalous 33 X 15 cm block of sand located at the eroded end of the ash.

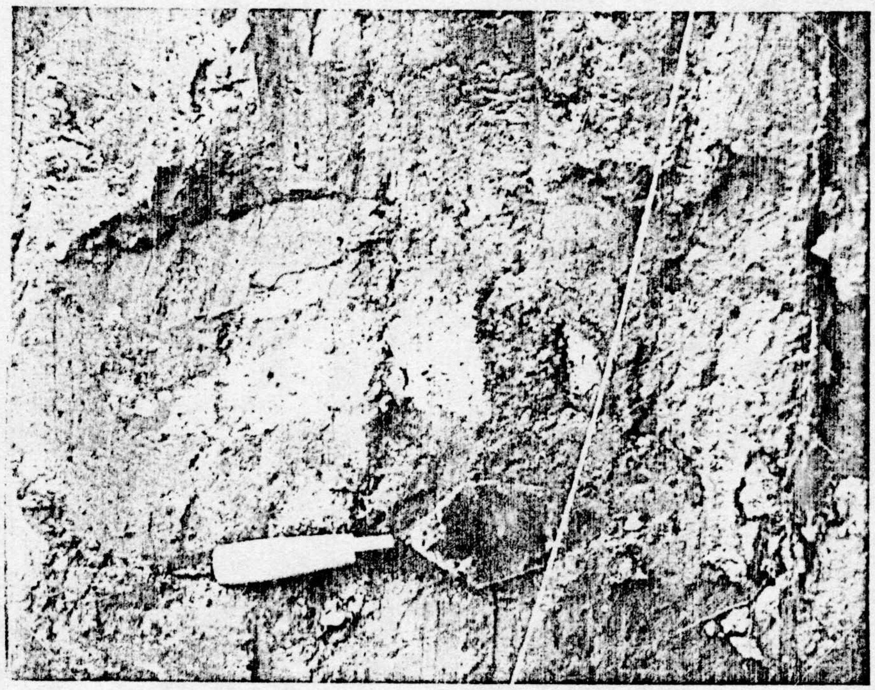
All of these occurrences can be explained by postulating the existence of a channel cut into the sediments approximately parallel to the present exposure. The re-entrant in which this channel is visible has been eroded back farther into the terrace

Figure 9. Ash trail. The ash trail is visible on the west wall of the re-entrant just left of the arrow in figure 1. The arrow marks the location of 10 B. A) Close-up of the ash trail at the base of the excavated re-entrant as it starts across the floor. The arrow indicates the largest of several ash patches along the trail. The ash trail marks the contact between the gray alluvial clay and the tan loess channel fill. B) Block of granule sized ash and silt. The location of this block is indicated on both figures 1 and 4. Though cut by a borrow to the right, the contacts are very sharp. It is possible this block was emplaced in the loess fill when it was frozen. C) Ash trail on the east wall of the re-entrant. A large burrow cuts the trail behind the tape measure.

D) Close-up of the base of the eastern ash trail. Note the pebbles (arrows) present in the loess fill. It is not known why the eastern trail is more wispy than the western trail. (Trowel = 35 cm)



A



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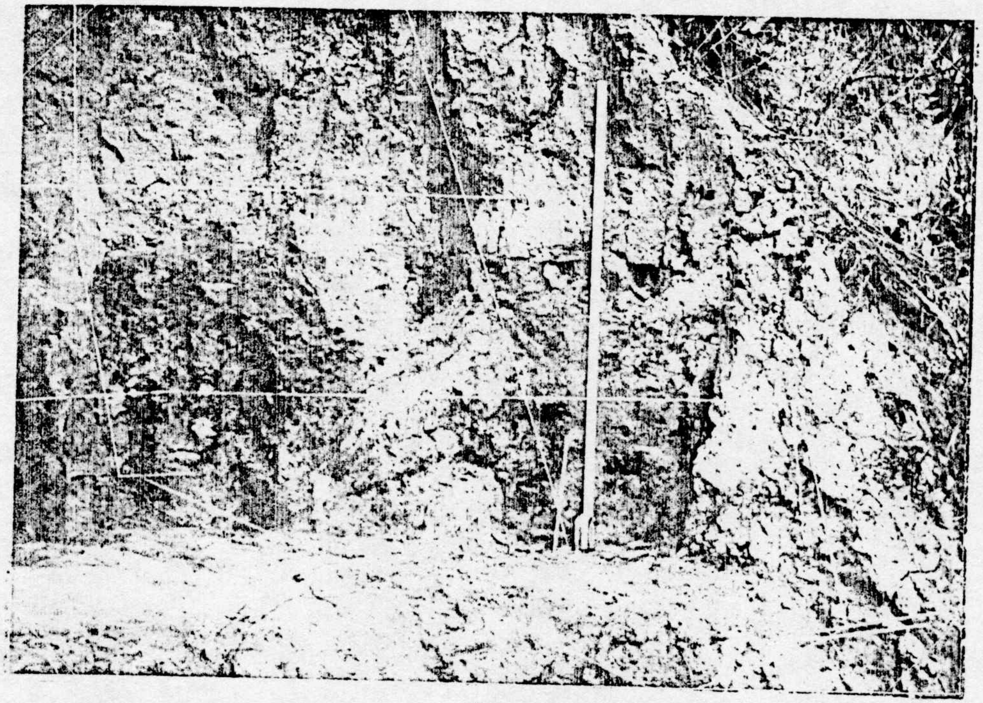

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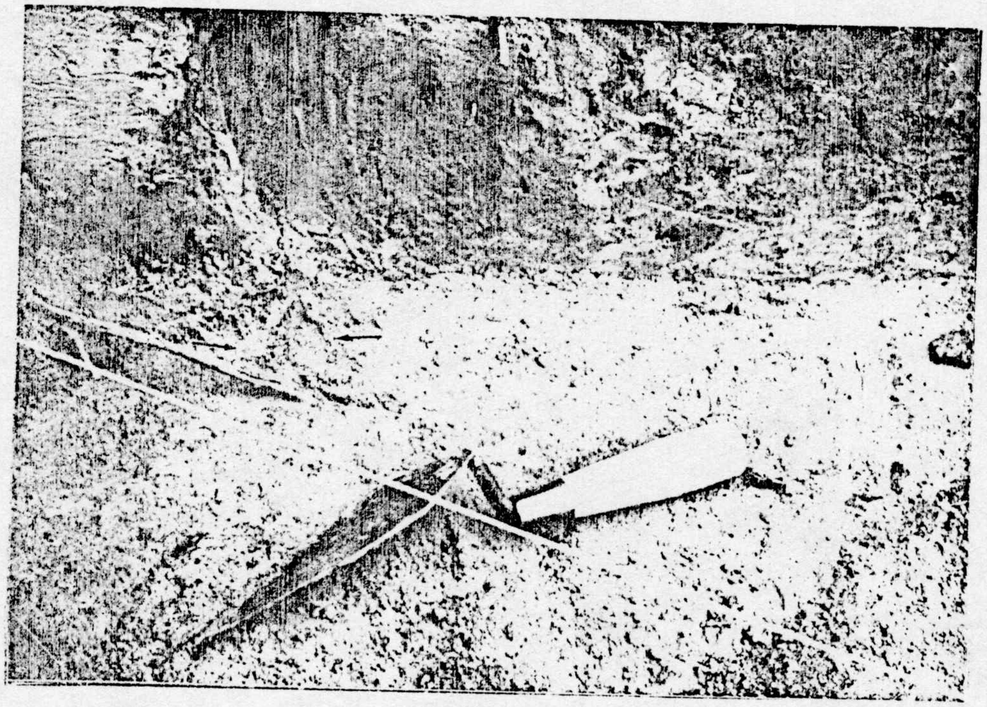


D

face than the portion in which the ash is present and thus has cut into the side of the channel. The ash trail is interpreted as slope wash from erosion of the main ash body down the south side of the channel. This explains its presence on both apparent (E/W) sides of the channel-like feature and indicates that the ash was once much more extensive. Further information, derived from taking core samples, on the location and extent of this channel is in Appendix A.

In fact, the land owner reported that the ash once cropped out along his present driveway (10 meters away) before he bulldozed the face of the terrace further back. He stated that the ash had the same general appearance and thickness as in the present exposure. Small blocks of granular ash and silt, similar to that in figure 9, as well as small concentrations of ash are also present intermittently both east and west of the scraped terrace face at the same general level as the ash bed. These are interpreted as being analogous to the ash trail, and indicate that the ash was once much more extensive than at present.

The material above the lacustrine sub-unit on the main terrace face is a clayey silt. This material overlays the eroded edges of both the lacustrine sub-unit and the ash. It then grades into a very mixed unit which is present to varying extents all the way around the lower perimeter of the channel-like feature. The ash trail is included within this mixed sub-unit.

The mixed sub-unit is interpreted as slope-wash with varying amounts of each of the eroded units as components. The contact between the clayey silt sub-unit and the overlying loess is gradational. Moody (pers. comm.; 1985) located a B22 soil horizon within this material (which extended into the lacustrine sub-unit below) on the the basis of the presence of clay skins. The area in which the lacustrine material accumulated was probably at least occasionally moist after continuous deposition of lacustrine sediment ceased. It is possible that it was the accumulation of loess which filled in the pond.

As has been previously stated, the uppermost unit is interpreted as loess. This material is massive and composed primarily of silt sized quartz. The accumulation of generally homogeneous silt of so great a thickness is hard to explain by any mechanism other than as air-borne silt. Its presence near a major river valley within an area of known loess deposition supports this conclusion. Moody (pers. comm., 1985) located several incipient soil horizons within this unit. A modern soil is developing within the loess beneath the present terrace surface.

IDENTIFICATION OF THE VOLCANIC ASH

The presence of various Cenozoic volcanic ash lentils within the Central Plains has been known for a long time (Merrill, 1885). The name "Pearlette Ash" was given to ash in a lentil along Crooked Creek Valley in Meade Co., Kansas by Cragin (1896). Frye and Leonard (1957, p.6) state that this ash fall "occurred in the last phase of Kansan deposition". The Pearlette Ash can be distinguished from other Pleistocene ashes on the basis of petrographic and chemical analyses including: shard shape, color, and number and type of vesicles; phenocryst assemblage; and glass chemistry. Boellstorff (1973a and b) found the Pearlette Ash to be actually four separate ashes of different ages, but all from a Yellowstone source area. It is now generally accepted that three ashes from the Yellowstone source area reached the Central Plains (Izett and Wilcox, 1982). These ashes can be distinguished from one another by the magnetic polarity of the ash (Reynolds and Larson, 1972), and by fission-track dating of the glass shards (Boellstorff, 1973a) or zircon phenocrysts (Naeser et al, 1973).

Three different sets of names are in use for the Pearlette family of ashes (see table 3). Izett et al. (1970) recognized that the Pearlette Ash on the Great Plains consisted of at least two different air-fall ashes. By 1971, the air-fall equivalents of the three major pyroclastic events in Yellowstone had been

Comparison of Names for Pearlette Family Ashes of the Central Plains

Boellstorff, 1976 glass f-t	Naeser *et al.*, 1973 zircon f-t	Izett and Wilcox, 1982 magnetic polarity sanidine K/Ar
Pearlette (restricted) 0.61 my	Type O Pearlette 0.6 my	Lava Creek B normal 0.62 my
Hartford 0.74 my	--------	---------
Coleridge 1.21 my	Type S Pearlette 1.2 my	Mesa Falls reversed 1.27 my
Borchers 1.97 my	Type B Pearlette 2.0 my	Huckleberry Ridge horizonal 2.02 my

Table 3. At least two sets of informal names have been suggested for the members of the Pearlette family of ashes since it was recognized as being more than a single ash-fall. Since then, much work has been done on the Yellowstone source area and the ash-falls have been correlated with their ash-flow tuff equivalents on the basis of magnetic polarity, chemical and petrographic means (Reynolds, 1975). The dates listed were obtained by fission-track (f-t) dating of the glass shards and zircon phenocrysts, respectively, and by K/Ar dating of sanidine phenocrysts within the ash-flow tuffs. It has been suggested that the flow names be used for their ash-fall equivalents (Izett and Wilcox, 1982). The Hartford ash has been found in relatively few localities and never (to the author's knowledge) in stratigraphic association with the Pearlette ash (restricted). There is no Yellowstone flow of this age and it is generally assumed to be a Lava Creek B Ash.

identified within the Great Plains and were provisionally termed; Pearlette type O ash, Pearlette type S ash, and Pearlette type B ash by Naeser et al. (1971). Boellstorff (1973a) objected to the basis on which these names had been applied and proposed that the Pearlette type O ash was actually two different ashes. In order to avoid confusion, he retained the "Pearlette" name for only the youngest Yellowstone ash. The other names were derived from geographic localities close to designated type areas. These names are as follows; Pearlette ash (restricted), Hartford ash, Coleridge ash and Borchers ash.

The Pearlette controversy on the Plains heightened interest in study of the pyroclastic material within Yellowstone Park itself. In 1975, Reynolds conclusively correlated the air-fall ashes to their correlative tuffs based on magnetic polarity and published chemical and petrographic data. A map of Pearlette family ash occurrences (Izett and Wilcox, 1982) uses the Yellowstone ash-flow tuff names. This set of names will be used throughout this thesis, except for specific references to use of the Borchers ash as an interlaboratory standard in Boellstorff's methodology. The Yellowstone names are as follows; Lava Creek B Ash, Mesa Falls Ash and Huckleberry Ridge Ash.

In an effort to identify the ash at DeSoto (herein called the DeSoto ash) several techniques were used in addition to absolute dating. These techniques were determination of glass chemistry, petrography, general morphology of the shards, and magnetic polarity.

There are three source areas from which volcanic ash was carried to the Great Plains. Each source yielded a distinctive form of glass shards with distinctive physical and chemical properties. The ashes which reached the Great Plains from each of these three source areas are the Guaje Ash from the Valles Caldera in north central New Mexico, the Bishop Ash from Long Valley Caldera in central California, and the Pearlette family of ashes from the Yellowstone calderas in northwestern Wyoming and adjacent parts of Idaho and Montana. Table 4 lists these and some related ashes in chronological order and summarizes their respective properties.

The Guaje Ash is fine-grained and light gray in color, with the glass shards being mainly bubble-junction and bubble-wall types with a few elongate-pumiceous shards (Izett et al., 1972). This ash has a glass fission-track age of 1.4 ± 0.2 my. and is correlated with the Guaje Pumice Bed which has a K/Ar date of 1.4 million years. Phenocrysts in this ash include sanidine, clinopyroxene, and magnetite. Izett et al. (1972) made special notice of the lack of plagioclase, even in the correlative tuff.

The Bishop Ash is chalky white and contains abundant biotite phenocrysts (Izett et al., 1970). The glass shards of this ash are minutely pumiceous. Other phenocrysts which occur

Table 4. Comparison of Pleistocene ashes of the Central Plains. The Tsankawi and Lava Creek A Ashes did not reach the Central Plains and are included for comparison. See table 6 for a comparison of glass chemistry for these ashes. Note different magnetic polarities of the Yellowstone ashes and the different appearance of the Bishop Ash, the nearest ash chronologically to the Lava Creek B Ash.

Sources: Boellstorff, 1973b
Borchardt, *et al*., 1972
Izett *et al*., 1970
Izett *et al*., 1972
Izett and Naeser, 1976
Izett and Wilcox, 1982
Reynolds and Larson, 1972

Comparison of Pleistocene Ashes of the Central Plains

Name	Source	Age (my)	Shard Shape	Color	Phenocrysts Present	Diagnostic Phenocrysts	Magnetic Polarity
Green Mountain Resevoir	Long Valley Caldera, CA	0.4	pumiceous	chalky white		biotite	normal
Lava Creek A	Yellowstone, WY	0.6					normal
Lava Creek B	Yellowstone, WY	0.6	bubble wall, bubble junction	silver gray	qtz, olig, san, f-aug, g-b hnbd, mag, il, zir, chev, al, ap	colorless zircon moderate 2V sanidine	normal
Bishop	Long Valley Caldera, CA	0.7	pumiceous	chalky white	biot, qtz, Na - plag, san, zir, ap, al, sph, il, mag, hnbd (?)	biotite low 2V sanidine	normal
Tsankawi	Jemez Mountains, NM	1.1					reversed
Mesa Falls	Yellowstone, WY	1.2	bubble wall, bubble junction	silver gray			reversed
Guaje	Jemez Mountains, NM	1.4	bubble wall, bubble junction, and some elongate pumiceous	light gray	qtz, san, f-hdbg, zir, chev, al, mag, il (?)	no plagioclase or hornblende	reversed
Huckleberry Ridge	Yellowstone, WY	2.0	bubble wall, bubble junction	silver gray		pink zircon	horizontal

in lesser quantities include quartz, sodic plagioclase, low 2V sanidine, and lesser zircon, apatite, allanite, sphene, ilmenite, and magnetite, and possibly hornblende (Izett _et al_., 1970). This ash has been dated by K/Ar at 0.7 my..

Members of the Pearlette family of ashes are all silver gray in color (Izett _et al_., 1970). The glass shards are primarily of bubble-wall and bubble-junction type. Phenocrysts for these ashes include quartz, oligoclase, moderate 2V sanidine, ferroaugite, green-brown hornblende, and lesser chevkinite, allanite, zircon, apatite, ilmenite, and magnetite (Izett _et al_., 1970). The color of the zircon phenocrysts varies among the individual ashes. Those from the Huckleberry Ridge Ash are pink, while those from the Lava Creek B Ash are colorless (Naeser _et al_., 1971). This color difference probably reflects the age difference between these ashes, since zircon color changes as radioactive damage accumulates (Naeser, 1986, pers. comm.). Radiometric (K/Ar) ages of these ashes are: Lava Creek B Ash - 0.62 my., Mesa Falls Ash - 1.27 my., and Huckleberry Ridge Ash - 2.02 my. (Izett and Wilcox, 1982). The Lava Creek A Ash (0.62 my.) has a small areal distribution and will not be considered further herein.

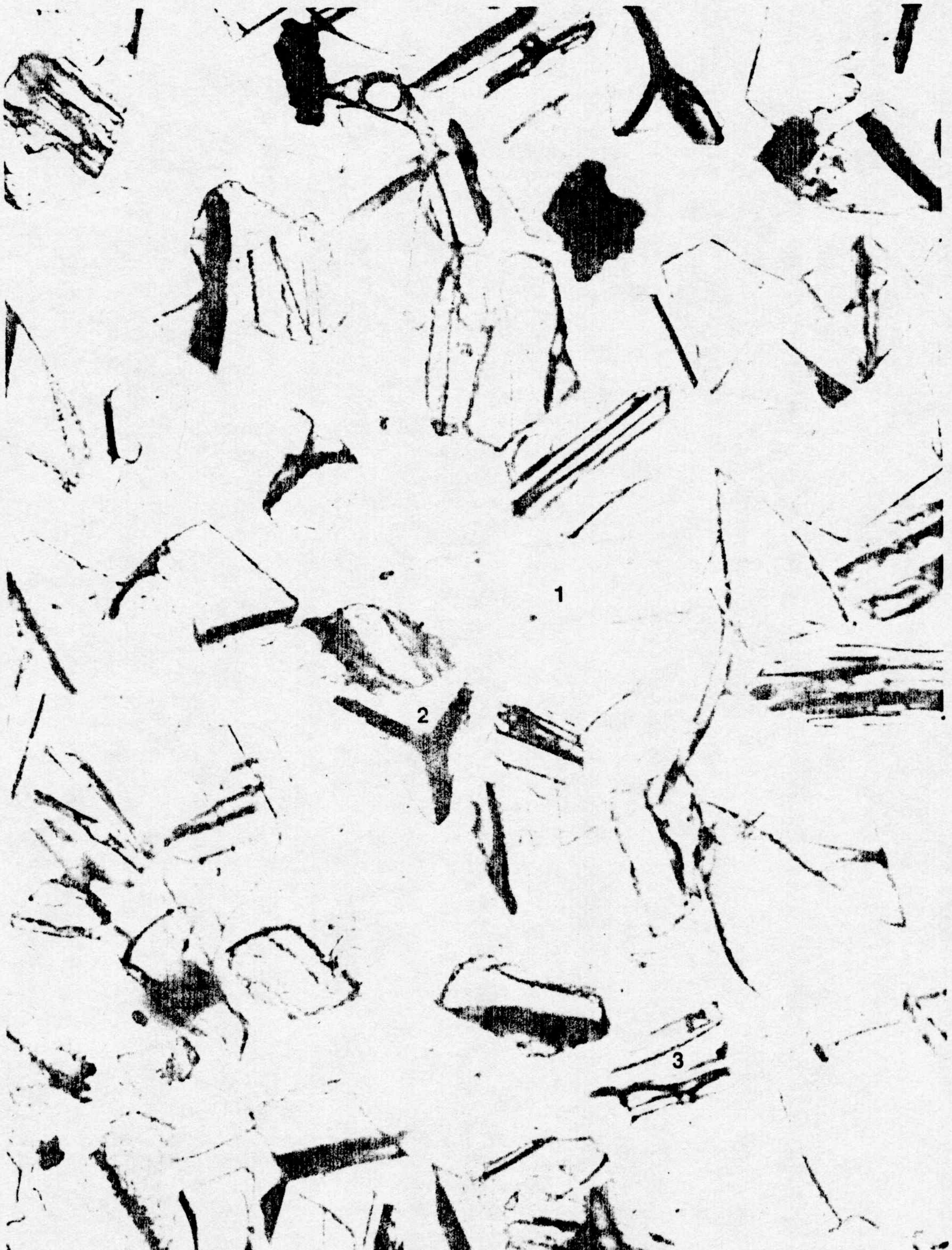
Though the DeSoto ash is creamy white (10YR8/2, white) in outcrop, once the shards have been cleaned, it is distinctly silver gray in color (about mid-way between white, 10YR8/1 and light gray, 10YR7/1). The shards are of bubble-wall and bubble-junction types (see figure 10). Few shards have more than a single rib and less than 5% have numerous vesicles. Truly pumiceous shards are very rare. The phenocryst assemblage of this ash will be described below.

Petrography

Several procedures were used to determine the petrography of the DeSoto ash. The ash bed consists of three differently colored portions (see figure 8 in the section on geomorphology). In an effort to determine a reason for these differences, each portion was examined by X-ray diffraction. The white, air-fall ash was wet sieved through two different sieve stacks and examined under the microscope. Both heavy liquid and magnetic separations were performed.

In order to determine the size composition of the ash, a bulk sample of the white ash was set to soak overnight in 0.1 N sodium oxalate to deflocculate any clays. It was then wet sieved through a U. S. Standard sieve stack consisting of 60 mesh (0.25 mm) and 170 mesh (0.088 mm) screens. Only a gentle stream of water was used and all material was saved. Four fractions were calculated; +60, -60+170, -170, and the clay fraction i.e. that fraction which did not settle from the -170 mesh collection bucket within about five minutes. These sieve sizes were selected because they represented the simplest breakdown of meaningful sizes for this ash. The 60 mesh screen

Figure 10. Morphology of the DeSoto ash. Flat bubble wall (1) shards are highly desirable for fission-track dating. The ribbed shards are bubble junction shards (2). Vesicular shards (3) in the DeSoto ash have elongate, stretched vesicles. This is the 0.125 - 0.088 mm (-120+170 mesh) sieve fraction as seen in plain light.


1
2
3

separates out medium sand and larger particles. These accounted for only 10% of the total sample. About 40% is in the -60+170 size fraction which includes fine and very fine sand. Fifty percent of the sample passed through the 170 mesh screen. Of this portion, 40% did not settle out rapidly and can be considered clay sized.

A different procedure was used in processing of the white ash in order to recover glass shards and zircons for dating. The ash was first spread to dry in an air conditioned room, since heating the ash might anneal it, causing fission-tracks to disappear. The dry ash was then sieved through a stack of 30 (0.589 mm), 120 (0.125 mm), and 230 (0.065 mm) mesh screens to remove the unusable large debris and fine material from the ash. The resulting -30+230 mesh fraction was left to soak overnight in a 0.1 N sodium oxalate solution to deflocculate and clays. This fraction was then sieved with a strong stream of water through a 60, 120, 170, and 230 mesh sieve stack. The most desirable size of shard to date is the -60+120 mesh fraction (Boellstorff, pers. comm.; 1984), however, little glass of this size was present in the sample. Therefore, the -120+170 mesh fraction was saved for dating. The -170+230 fraction was retained in the hope of obtaining datable zircon phenocrysts.

The white ash separates into three main fractions, (+60, -60+230, and -230) each of which has a different set of characteristics. The majority of the +60 size fraction consists of insoluble white, irregular and/or tubular masses, though about 5% consists of flat glass shards. There are also some modern root fragments. These white masses are unaffected by either concentrated HCl or 30% H_2O_2. An analysis of this material by both x-ray diffraction and fluorescence is described below.

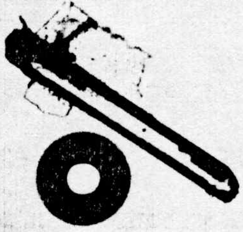
The -60+230 mesh fraction is about 60% glass, though proportions of other constituents vary within subfractions of this size category. A colorless mineral steadily increases from about 5 to 15% from the -60+120 mesh to the -170+230 mesh fraction. Some specimens have conchoidal fracture, while others are blocky and appear to have at least one discernible cleavage. Birefringence is low. At least some of this material is probably quartz. White, fluffy, opaque balls account for approximately 25 to 30% of the sample and become less abundant in the smaller size subfractions, while the amount of other minerals increases. These other minerals account for less than 5% of the sample. There is also a small quantity of black to orange opaque nodules in this size fraction. They account for about 5% of the -60+120 mesh fraction, but less than 1% of the -170+230 mesh fraction.

The -230 mesh fraction is about 50% glass, 20% colorless, birefringent material, 5% other minerals, and 25% clay-like material. An analysis of this clay-like material by X-ray diffraction is described below.

Heavy liquid and magnetic separations were performed to help in concentrating and identifying these other minerals. The glass was removed by using concentrated zinc bromide with a specific gravity of about 2.45. The heavy fraction was then further separated in bromoform of specific gravity 2.85. Minerals heavier than this were run through a Franz magnetic separator at magnetic susceptibilities of 0.1 A, 0.2 A, 0.3 A, 0.5 A, and 1.0 A with a 10°side tilt. The nonmagnetic fraction was further separated with methylene iodide of specific gravity 3.32 and then these two subfractions rerun through the magnetic separator (set at 1.5 A) at a 5° slant.

Only two of the mineral species found can be considered as phenocrysts of this ash. Most of the minerals occur as rounded fragments and are obviously detrital. These detrital grains may have been added by burrowing and/or pedogenic processes, as well as by sampling techniques. In order to be assured of the phenocrystic origin of a particular mineral fragment, it must have glass attached to it. A few mineral fragments, mostly zircons, appeared to be in good condition, but did not have glass. One variety of zircon, a clear, long prismatic (400 X 40μ) variety, occasionally occurred as delicate skeletal crystals and frequently had attached glass. These are considered to be phenocrysts. Other zircons included stubby, barrel-shaped colorless and light pink varieties which were never seen to have attached glass (see figure 11). The only other

Figure 11. Phenocrysts from the DeSoto ash. A) Colorless zircon with attached glass, 0.33 mm long. B) Colorless skeletal zircon with attached glass, 0.24 mm long. Note good crystal faces on these zircons C) Detrital colorless zircon, note rounded crystal faces and lack of glass, 0.13 mm long. D) Chevkinite with attached glass, 0.39 and 0.12 mm long. All viewed in plain light.



A

B

C

D

mineral occurring with attached glass is chevkinite, a cerium titanosilicate. These long and slender (450 X 50μ), striated, golden brown crystals make up the majority of the > 2.85, 0.5A fraction. Other heavy minerals which are relatively common are magnetite and epidote (?), these are considered to be detrital.

The phenocryst assemblage of the Lava Creek B Ash includes both colorless zircon and chevkinite, as indicated in table 4. As the Huckleberry Ridge Ash contains pink zircon, the absence of glass on the pink zircons found (especially in light of the presence of glass on the colorless zircons) is significant.

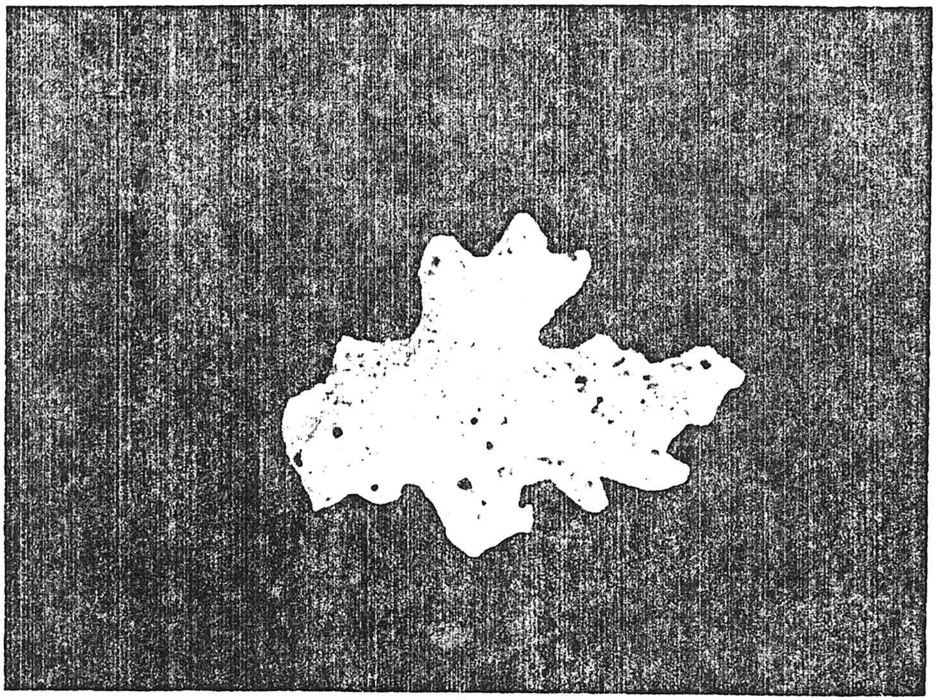
Analytical Results

X-ray diffraction analyses were run to try to determine if there were a mineralogic reason for the differently colored layers within the ash bed and to identify unknown components of the ash. The samples run included: bulk samples from the white, pink, and green colored layers; and the following portions of the white ash - the processed glass; the -230 mesh size fraction; the -60+230 mesh, < 2.85 specific gravity fraction; and the white masses from the +60 mesh size fraction. Novaculite (SiO_2) was used as a standard. No clay peaks were noted on any of the diffractograms and, other than quartz, no minerals were identified. Though the three differently colored layers of the ash did not yield identical diffractograms, they are broadly similar and no reason for the different colors was readily apparent. The -230 mesh size fraction and processed glass have similar diffractograms in that they each have a broad shoulder beginning at about 20° 2θ, but no well defined peaks.

In an effort to determine the identity of some of the more enigmatic materials, x-ray fluorescence (XRF) was utilized. An hypothesis for the identity of the white masses was that they were amorphous material derived from leaching of the ash. In order to test this hypothesis, approximately 365 grams of processed glass was leached in swirling distilled water for nine days. The resulting salts were fused and analyzed by XRF. No peaks not attributable to the flux were present. Fused disks were also made of the white masses, the black opaque nodules, and of the processed glass. Nearly 90% of the mass of the white masses was lost on ignition. Since there was no reaction of this material when tested with HCl (and thus it is not a carbonate), it is likely that it is largely organic. The form of several individual masses suggests that they formed around plant roots (see figure 12). Small dark nodules were found in both the paleosol underlying the ash and in the ash bed itself during field work. The XRF at the University of Kansas is not currently calibrated to analyze for manganese and the total percentage of oxides accounted for was less than 100%. It is likely that these dark nodules are manganese nodules.

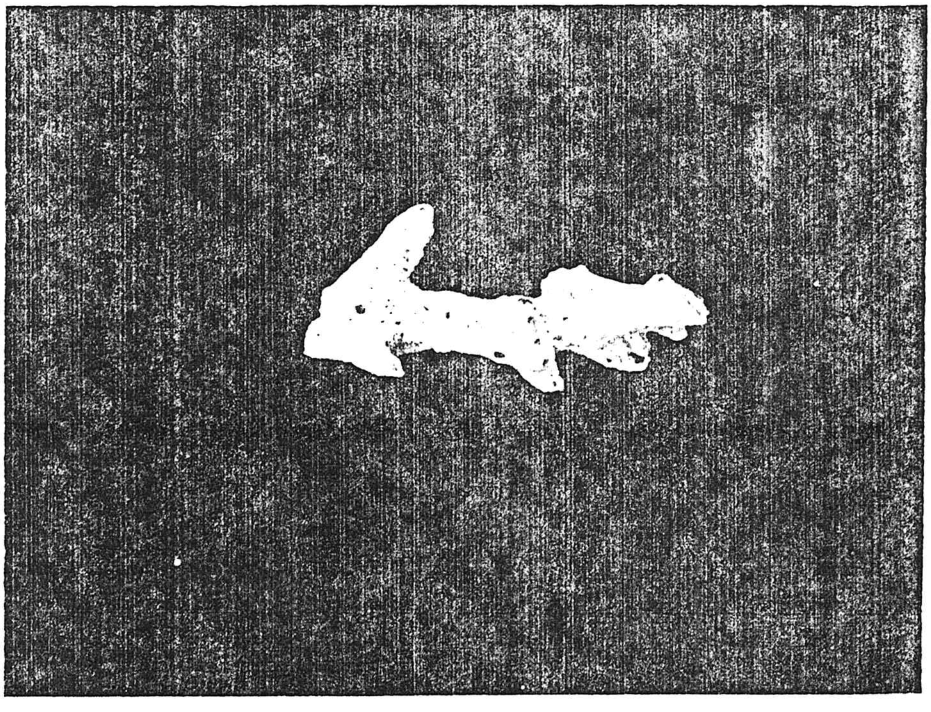
The bulk chemistry of the glass phase of the ash was analyzed in order to establish further the identity of the DeSoto

Figure 12. White masses of unknown origin. These forms are largely hollow (note circular apertures) and are frequently root-like, as are those above. A) 6.5 mm long. B) 6.0 mm long.



A

B



ash. The electron microprobe is said by Smith and Westgate (1969) to be the best analytical method by which to analyze ash chemistry. The authors listed several reasons for this choice, the primary one of which is the problem of contaminants. The bulk chemistry of an ash can be altered by admixing of detrital materials and by small inclusions and microcrystallites within the shards. Individual shards can be analyzed by electron microprobe, hence eliminating these problems. In preparation of samples, it is best to use diamond dust for polishing as aluminum oxide may leave excess aluminum (Moody, pers. comm.; 1985).

Unfortunately, an electron microprobe capable of the precision necessary for this work was not available for the duration of the study. For this reason, samples of cleaned ash were sent to Dr. Izett of the U. S. Geological Survey in Denver for analysis (sodium oxalate was not used during cleaning of this sample). The glass was dissolved in hydrofluoric acid and analyzed by induction coupled plasma atomic absorption spectrometry. The results of this analysis are listed and compared with other volcanic ashes occurring in Kansas and Nebraska in table 5. The results are compatible with an identification of Lava Creek B Ash (Izett, pers. comm.; 1985). The high zinc content is probably an artifact of the use of zinc chloride in separating out the glass fraction from the bulk ash. The relatively high manganese content may be related to the presence of manganese nodules within the ash bed.

Table 5. The chemistry of the glass phase of the DeSoto ash is compatible with an identification of Lava Creek B Ash. Na and K are exchangable cations and thus are not good elements for comparative work. See text for comments on high Mn and Zn contents. Values given are averaged without standard deviations from each indicated source.

Notes:

total Fe as FeO

* generally included as Lava Creek B Ash

x ppm Mn

References:

1. Izett, *et al*., 1970
2. Izett *et al*., 1972
3. Boellstorff, 1973a
4. Izett, for this work
5. Borchardt *et al*., 1972

Methods:

A. electron microprobe analysis
B. Atomic absorption spectroanalysis
C. Colorimetric analysis
D. Standard gravimetric methods
E. not given by source
F. Induction coupled plasma atomic absorption spectroanalysis
G. Instrumental neutron activation analysis

Chemistry of the Glass Phase of Selected Volcanic Ashes

Ash Name	Source Method	weight percent oxide (unless indicated otherwise)								ppm		
		SiO_2	Al_2O_3	$FeO^{\#}$	CaO	Na_2O	K_2O	TiO_2	MnO	Zn	Sm	Rb
DeSoto	4F		11.3	1.4	0.53	3.24	4.70	0.12	320^{x}	160		200
Lava Creek B	1A	72.4	11.8	1.41	0.59	3.00	5.32	0.11				
	1B			1.33	0.50	3.10	5.28			90		
	2B				0.53	3.01	5.41	0.11	0.04	82		215
	2C	70.9	12.3	1.36								
	2D	72.84	11.83	1.48	0.54	3.10	5.32	0.12	0.04			
	3E			1.1					280^{x}		12	
Hartford*	3E			1.14					280^{x}		13	
Bishop	1A	73.0	12.0	0.69	0.52	3.46	4.5	0.08				
	1B								265^{x}	30		175
	3B			0.64					200^{x}		5	
	5G			0.64		3.34	4.88		240^{x}		4	
Mesa Falls	1A	72.4	11.7	1.27	0.77	2.6	6.0	0.12				
	1B			1.15	0.49	2.44	6.11			70		
	2B				0.54	2.93	5.48	0.10	0.04	70		200
	2D	72.99	11.86	1.29	0.58	2.48	6.13	0.11	0.03			
	2C	72.6	11.5	1.13								
	3E			1.0					240^{x}		11	
Huckleberry Ridge	2D	72.86	11.86	1.57	0.55	3.14	5.34	0.12	0.04			
	3E			1.2					280^{x}		14	
Guaje	2B			1.29	0.26	3.50	5.26		0.08	120		330
	2C	71.7	11.6					0.05				

Since all the Pearlette ashes are from the same source area, their chemistry is rather similar. Though it is relatively straightforward to determine if an ash is chemically similar to the Yellowstone source ashes, identifying which particular ash a sample is from can be difficult, as can be seen in table 5.

Paleomagnetic studies offer yet another method by which to differentiate these chemically similar ashes (Reynolds and Larson, 1972). Fortunately, the magnetic polarity of each of the three major Yellowstone source ashes is different. The Lava Creek B Ash is normally polarized, while the Mesa Falls Ash is reversely polarized. Amazingly, the Huckleberry Ridge Ash seems to have been erupted during a period when the earth's magnetic poles were changing, it is horizontally polarized.

Samples of the DeSoto ash were sent for paleomagnetic determination to Dr. Kenneth Kodama of Lehigh University (Bethlehem, PA). These samples were obtained by carving 1 cm^2 pedestals into the ash *in situ*, covering the pedestal with Devcon 5 minute epoxy, and placing a 1 cm^3 plastic box over the pedestal. The orientation of magnetic north and the dip of the surface of each box were measured with a Brunton compass and written on each box. The pedestal was then cut off and the boxes capped. At Lehigh, one sample was demagnetized at 100, 200, 400 and 800 oersted. Two other samples were demagnetized at just 800 oersted. It was determined that the DeSoto ash has normal polarity (Kodama, pers. comm.; 1984). This is consistent with an identification as Lava Creek B Ash.

Fission-Track Dating

Fission-track dating involves the counting of tracks created by the spontaneous fission of ^{238}U. When first discovered, these tracks could be seen only with the magnification available with an electron microscope. By etching the damaged zone created by passage of a nuclear fragment, it is possible to see fission-tracks at magnifications of $\geq$ 500X through an optical microscope.

Since the rate of formation of fission-tracks is constant, one need know only the amount of ^{238}U present in a material and the fission-track density to obtain a date. This date refers to the time elapsed since the material became capable of retaining tracks, i.e. since it cooled from an originally higher temperature. The first fission-track dates were published during 1964 (Fleischer *et al.*, 1975).

The easiest method by which to determine the ^{238}U concentration in a material to be dated is by comparison with a standard having a known uranium concentration. This is done by utilizing the constant isotopic ratio of ^{235}U to ^{238}U. Both the sample and the standard are irradiated together in a nuclear reactor. Thermal neutrons induce ^{235}U (but not ^{238}U) in the materials to fission. The neutron fluence (Φ) (number of neutrons striking a square centimeter area of material) is determined by counting the induced track density (ρ_i) in the standard (or dosimeter), thereby permitting the ^{238}U concentration of the sample to be calculated.

The population method of fission-track dating was used to determine an age for the DeSoto ash. In this method it is assumed that there is a homogeneous distribution of uranium in the ash. The ash is divided into two splits, one of which is used to determine the spontaneous track density. The other split is sent to a nuclear reactor to be irradiated. A piece of standard dosimeter glass is sent along with the ash sample to be irradiated at the same time, since the number of neutrons which strike the ^{238}U nuclei within the sample (the nuclear fluence) determines how many induced tracks will be formed. The ratio of these three track densities (spontaneous, induced, and dosimeter) allows the age to be determined.

A more specific discussion on the theory of fission-track dating, and some of the problems involved comprises Part II of this report. However, for the purposes of Part I, it can be stated that an age of 0.58 ±.09 my was obtained by fission-track dating a sample of the DeSoto ash.

CONCLUSIONS

All of the results derived from this work agree with a correlation of the DeSoto ash with the Lava Creek B Ash. These include shard shape and color, phenocryst content and color, glass chemistry, magnetic polarity, and presumed geographic extent. The fission-track ages obtained (see table 9 in Appendix A) do not preclude this correlation. Therefore, the ash at DeSoto is interpreted to be 0.62 million years old based on K/Ar dates for the Lava Creek B Tuff in Yellowstone (Izett and Wilcox, 1982). Depending on which timescale is accepted, the 0.62 million year time line may fall anywhere from the Aftonian interglaciation to the Illinoian glaciation (see table 1 in the Introduction).

The ash is situated in terrace fill above a paleosol developed in alluvium and below a loess deposit. The terrace remnant at the Dalaba Site fits the description of the Buck Creek Terrace best (see Geomorphology), and thus has an assumed stratigraphic age of Illinoian (Davis and Carlson, 1952). In order for a soil to develop in which the pedogenic influence can be readily seen to a depth of nearly two meters, a fairly long length of time must pass. This indicates that the alluvium must have been deposited well before 0.62 million years ago. Referring again to table 1 in the Introduction, it is apparent that a definitive determination of the stage during which the ash was deposited can not be made at this time. It seems highly unlikely, however, that the major paleosol at DeSoto is the Sangamon Soil, which Davis and Carlson (1952) thought capped Buck Creek Terrace fill. It is possible that the paleosol is a Yarmouthian soil, though the classic Aftonian sequence in Iowa contains a volcanic ash which has been correlated with the Lava Creek B Ash (Boellstorff, 1978b). The following scenario is postulated.

During original development of the Kansas River valley during the Kansan (Frye and Leonard, 1952) and possibly even the Nebraskan (Heimg and Hawe, 1963), the Menoken Terrace was formed. This terrace was likely classic in form, the terrace surface correlating with the height of a former floodplain surface. As the Kansas River entrenched itself, abandoning the Menoken Terrace (possibly after capturing drainage which had previously flowed south through the McPherson valley), and then began to aggrade again, it is possible that the terraces created at different stream mouths were not strictly coeval or concordant.

The author suggests that the Buck Creek Terrace may actually consist of a series of only generally coeval terraces. The alluvium at DeSoto may have been deposited during the late Kansan or even the early Yarmouthian. The paleosol developed on the resulting stable surface during the Yarmouthian. The ice front during the Illinoian glaciation was far north of the Kansas River valley, and it is not known exactly what effects that glaciation might have had on the area. It is possible that an effect of the Illinoian glaciation was increased downcutting as meltwater discharged into the Mississippi River. The channel which is eroded through the ash may have formed during this time. Loess deposition probably occurred during the Illinoian and may have been augmented during the Wisconsinan.

Before any of this can be rigorously tested, more absolute dates must be determined and the dates for the Pleistocene stages must be agreed upon. It is certain, however, that the entire terrace fill sequence at the Dalaba Site was not deposited during one cut-fill cycle, or time period, and the implied conclusion that the alluvial terrace fill is of Illinoian age appears to be impossible.

APPENDIX A: Detailed Terrace Stratigraphy

Sediment description

In order to more fully investigate the terrace fill sediments, a trench was excavated into the slump at the exposed terrace base. Figure 4 from the Geomorphology section is reproduced here as figure 13 with an addendum which includes these deeper units. Also indicated on figure 13 is the location of each particle size analysis sample taken. These data are enumerated in table 6. A detailed description of each unit follows. There was no effervescence present in any unit. Thicknesses were recorded at the location indicated on figure 13. The unit names are the same as those in table 6.

modern soil - 0 to 23 cm; dark brown (7.5YR4/4) silt loam; granular; friable; gradual boundary.

silt - 23 to 130 cm; brown (10YR5/3 to 7.5YR5/4 or 4/4) silt loam; granular; friable; gradual boundary.

clayey silt - 130 to 142 cm; brown (10YR5/3 to 7.5YR5/4 or 4/4) silt loam to silty clay loam; granular; friable; gradual boundary.

laminated silt and clay - 142 to 160 cm; pale brown (10YR 6/3) silty clay loam to silty clay; platy with silty and more clay-rich laminae; friable; sharp boundary.

transition - 160 to 162 cm; olive gray to grayish green (5Y5/2 to 10GY6/2) clay; blocky; firm; sharp boundary.

Figure 13. Schematic profile of the terrace face with extra units added. Arrow at top marks location of thickness measurements cited in the detailed description in the text. Samples 14 and 16 from green and white ash, respectively, located just above and below sample 15. Sample 17 is from the white ash in line with samples 13 and 21.

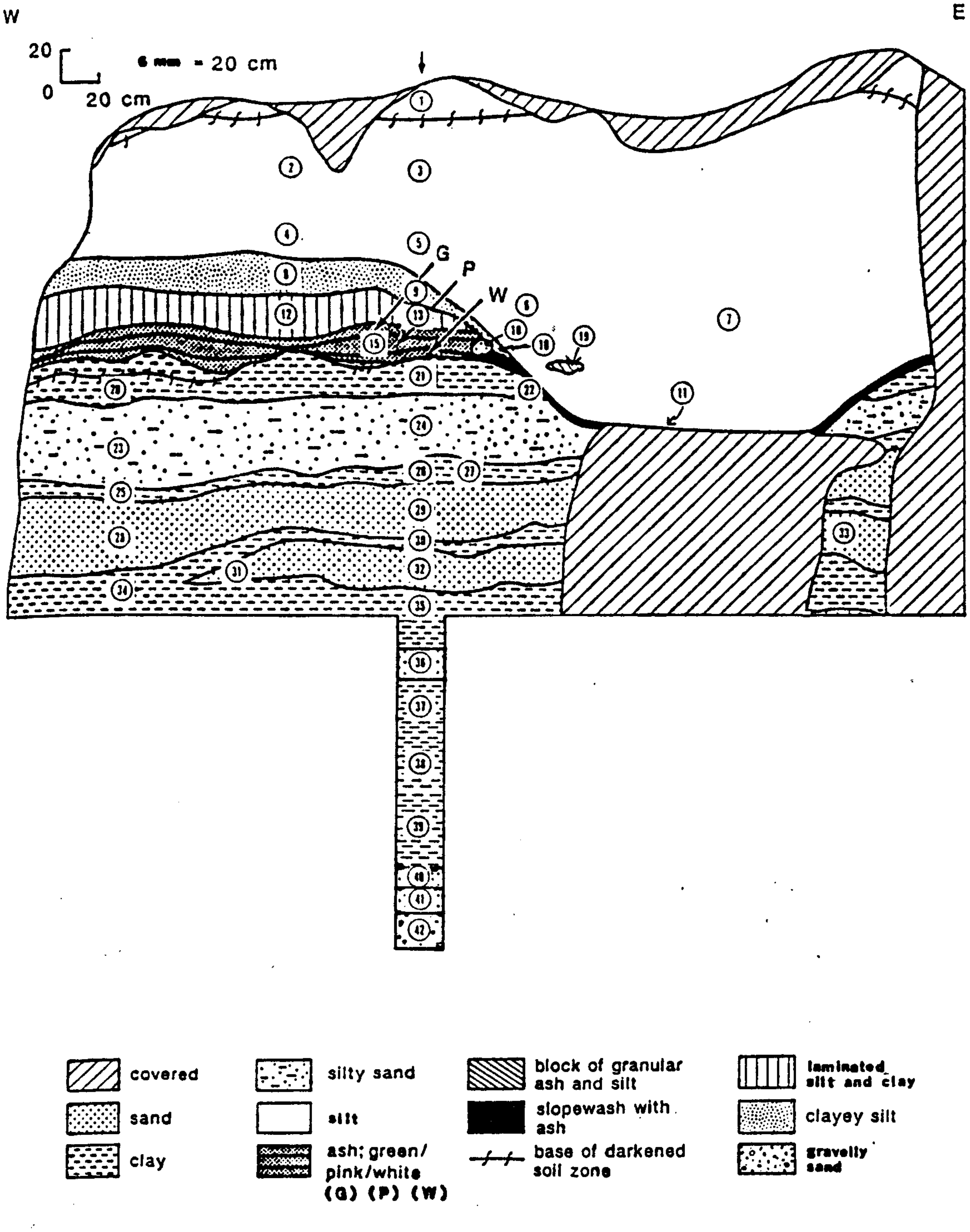

W
E
20
0
20 cm
6 mm = 20 cm
1
2
3
4
5
G
P
W
6
7
8
9
12
13
15
16
18
19
11
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
covered
sand
clay
silty sand
silt
ash; green/pink/white (G) (P) (W)
block of granular ash and silt
slopewash with ash
base of darkened soil zone
laminated silt and clay
clayey silt
gravelly sand

Table 6. Data were gathered at several different times and combined. For one set of analyses, both the 2 hour clay settling time and the 6 hour 52 minute clay settling time were recorded. Though these are generally very similar, note the large quantity of fine silt in the ash samples, especially sample 15, the pink colored ash. Percent sand calculated from the 2 hour clay times.

Particle Size Data

sample #	% Sand	% Silt	% Clay	~7 hr Clay	unit	
1	31	48	21	-	modern soil	
2	25	52	23	23		
3	20	50	30	-		
4	23	48	29	28	silt	
5	27	54	19	-		
6	29	49	22	22		
7	18	54	28	28		
8	11	52	37	37	clayey	
10	25	50	25	25	silt	
9	29	54	17	-	slope	
11	31	52	17	17	wash	
12	5	44	51	45	laminated	
13	17	48	35	-	silt and clay	
14	9	42	49	41	green	
15	9	36	55	37	pink	
16	23	40	37	29	white ash	
17	29	42	29	-	white	
18	18	52	30	-	sand ball	
19	12	46	42	-	ash + silt	
20	25	40	35	29		
21	27	44	49	-	clay	
22	15	52	33	29		
23	33	48	19	17		
24	41	46	13	-	sand	
25	21	44	35	31		
26	19	50	31	-	clay	
27	13	32	55	47		
28	59	23	18	17		↑
29	50	41	9	-	sand	
30	15	42	43	-	clay	alluvium
31	21	50	29	-		
32	37	52	11	-	sand	
33	21	60	19	19		
34	15	42	43	39		
35	29	40	31	-	clay	
36	87	3	10	-	sand	↓
37	21	56	23	-		
38	19	60	21	-	clay	
39	17	56	27	-		
40*	99	0	1	-	sand to	
41	97	2	1	-	gravelly	
42*	99	0	1	-	sand	

* Gravel data (wt%)

sample #	<2mm	2-4mm	>4mm
40 ~~15~~	86.0	11.5	2.5
42 ~~17~~	59.8	24.6	15.6

ash - 162 to 185 cm; three subunits: Green - 162 to 167 cm; grayish yellow green to light olive gray (5GY7/2 to 5Y6/2) clay; blocky to massive toward base; firm; gradual boundary. Pink - 167 to 178 cm; very pale brown to pink (10YR8/3 or 7/4 to 7.5YR8/2 or 4) clay; massive; friable; gradual boundary. White - 178 to 185 cm; white (10YR8/1 or 2) clay loam; massive; very friable; Mn nodules present; sharp, undulatory boundary.

alluvium to base of described column

185 to 221 cm; brown (10YR5/3) clay loam to silty clay loam; massive friable; many distinct mottles; round, up to 1 cm root casts outlined with sesquioxides; common Mn nodules increasing with depth; gradual boundary.

221 to 246 cm; pale brown (10YR6/3) loam; massive; friable; many mottles and small sesquioxide concretions; common root casts and Mn nodules; sharp, undulating boundary.

246 to 265 cm; pale brown (10YR6/3) silty clay loam; massive; friable; many prominent root casts outlined with sesquioxides; many Mn nodules; sharp boundary outlined by discontinuous sesquioxide accumulation.

265 to 286 cm; light gray (10YR7/2) loam; massive; friable; many prominent brownish yellow (10YR6/8) mottles; Mn nodules more common with depth; sharp boundary.

286 to 295 cm; light brownish gray (2.5YR6/2) silty clay with lighter colored hazy subvertical streaks; blocky; firm; clay skins; sharp boundary.

295 to 318 cm; light brownish gray (2.5YR6/2) silty loam; weak blocky structure; friable; weak clay skins; few small, but distinct gray mottles decreasing in frequency with depth; sharp boundary.

318 to 348 cm; light brownish gray (2.5YR6/2) clay loam; blocky; firm; clay skins; few distinct mottles; sharp boundary outlined with a sesquioxide layer.

348 to 363 cm; very pale brown (10YR7/3) loamy sand; massive; very friable; sharp boundary at 5 mm thick continuous sesquioxide layer.

363 to 452 cm; grayish brown (2.5YR5/2) silty clay to silt loam; blocky; firm; root casts outlined with sesquioxides common to 400 cm; common distinct round brownish yellow (10YR6/8) sesquioxide concretions with gray centers, 5 mm to 3 cm in diameter to 430 cm, becoming more diffuse with depth till no more appear; sesquioxide layer above 2.5 cm sandy zone near base; sharp boundary at ~3mm well indurated sesquioxide pan.

452 to 461 cm; pale brown (10YR6/2) sand; massive; loose; mineralogy: quartz with some feldspar and magnetite, grains rounded; sharp boundary at sesquioxide layer.

461 to 471 cm; light brownish gray (10YR6/2) sand; massive; loose, but some "cling"; mineralogy same as above; sharp boundary.

471 to at least 490 cm; strong brown (7.5YR4/4) to red (2.5YR4/6 gravelly sand; alternating layers are sesquioxide coated, partly indurated, and uncoated loose sand; 10 cm X 3 mm partially coated shale and 2.5 X 2.5 X 7 cm coated chert pebbles present in ~25 cm wide exposure; mineralogy same as above, except grains frosted; no lower boundary reached.

These lowermost three sand units may well represent a point bar deposit, with occasional lag deposits and clay drapes. The paleosol was not encountered within the described column and no particle size data were gathered for it. The texture is the same as that of the alluvial clay unit it caps, and the boundary is gradual. In contrast to the clay, which is a paler brown (10YR5/3), the paleosol is redder and darker brown (7.5YR4/4).

The particle size data as shown in figure 14 indicate the alluvial character of the lower sediments well. Pedogenic processes have damped the swing from clay to sand toward the top of the paleosol. With the exception of the sand which pinches out, there is a smooth increase in sand percentage (and corresponding decrease in silt percentage) with depth until nearly pure sand is reached.

Within the units at the top of the column, there is an increase in clay content with depth. This may reflect a trend toward wetter conditions. Once clay was blown in, it was trapped. This trend reaches a maximum within the laminated clay and silt unit, which has been interpreted as lacustrine.

Coring data

Seven (two-inch diameter) cores were taken from the top of the terrace using a Giddings mobile soil-sampling drill. The locations of these cores are shown in figure 15 with respect to the exposed face of the terrace fill. The sediments within these cores, the column described earlier, and a column within the "channel" are represented in figure 16.

Ash was present to some extent in four of the cores. The sediment overlying the ash was nearly always different, however. Loess overlay green and pink (secondary) ash in core 4 and discrete chips of pink ash in core 5. These chips

Figure 14. Variation of particle size with depth. The depths shown are representative and are not actual sample depths. Datum was chosen as the base of the profile in figure 13 and the lithology uses the same symbols. Above datum, the numbers are averaged from several analyses; those below datum are from single analyses. The uppermost analysis is from the modern soil. The loess is quite variable, with clay and sand percentages each ranging from 20 to 30; this variability does not correlate with depth. Clay increase from the base of the loess to the ash. Note the sharp increase in particle size from the colored, secondary ash just above the 140 cm depth to the white, primary ash. Below the ash, the alluvial character of the sediment is obvious, though pedogenic processes have somewhat dampened the size swings and increased the silt content of the sands above datum.

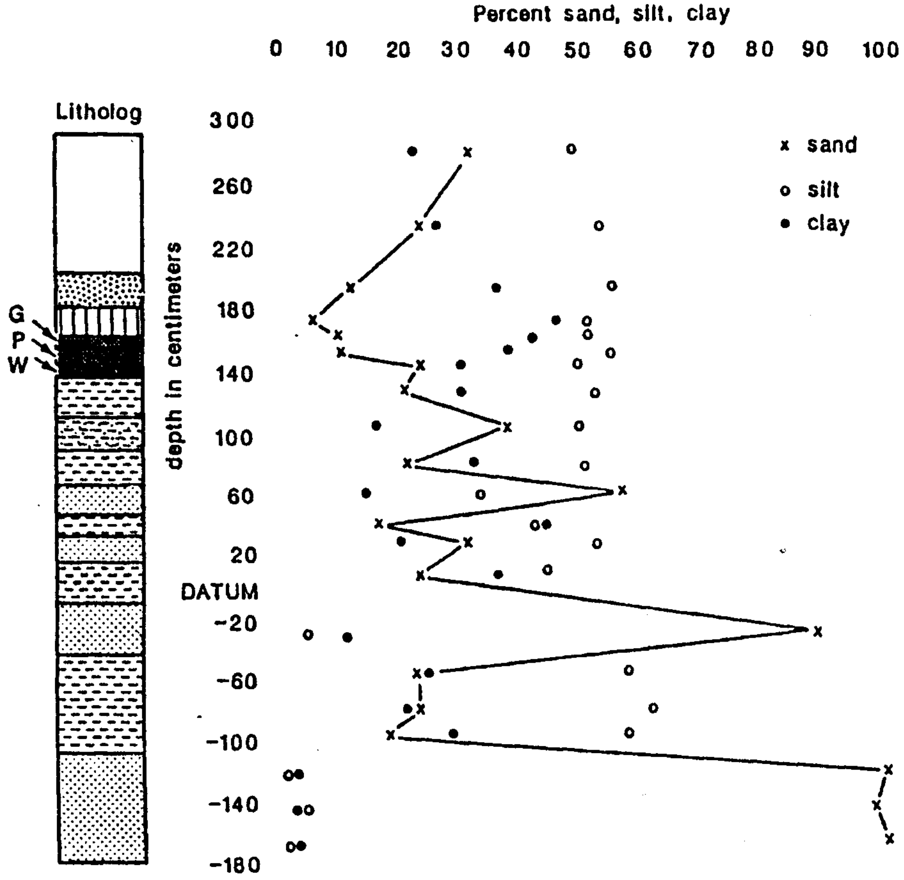

Percent sand, silt, clay
0
10
20
30
40
50
60
70
80
90
100
Litholog
G
P
W
depth in centimeters
300
260
220
180
140
100
60
20
DATUM
-20
-60
-100
-140
-180
x sand
o silt
● clay

Figure 15. Location of cores. A total of seven cores were taken from the terrace surface. The terrace face of figures 4 and 13 is located between the arrows on this diagram.

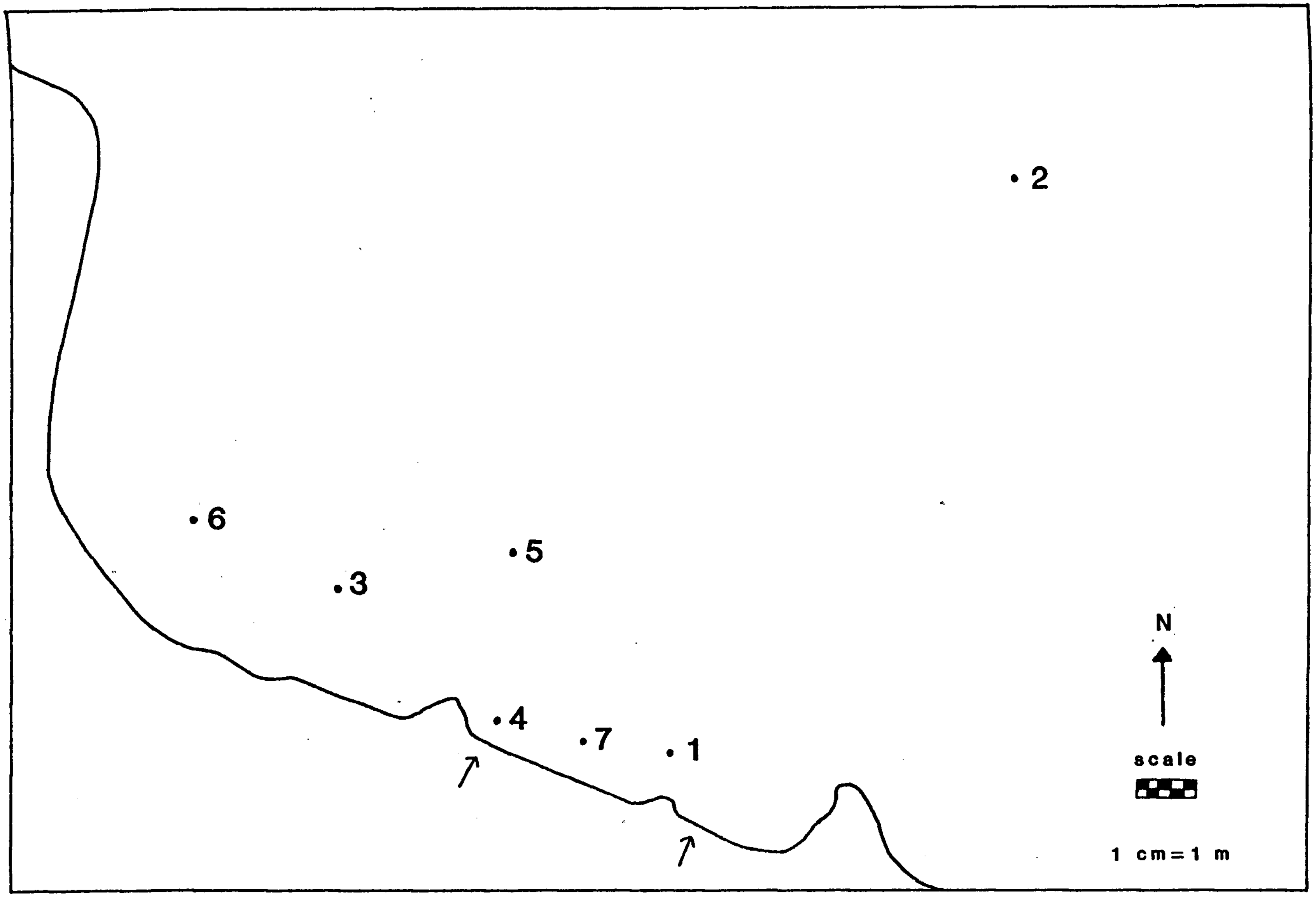
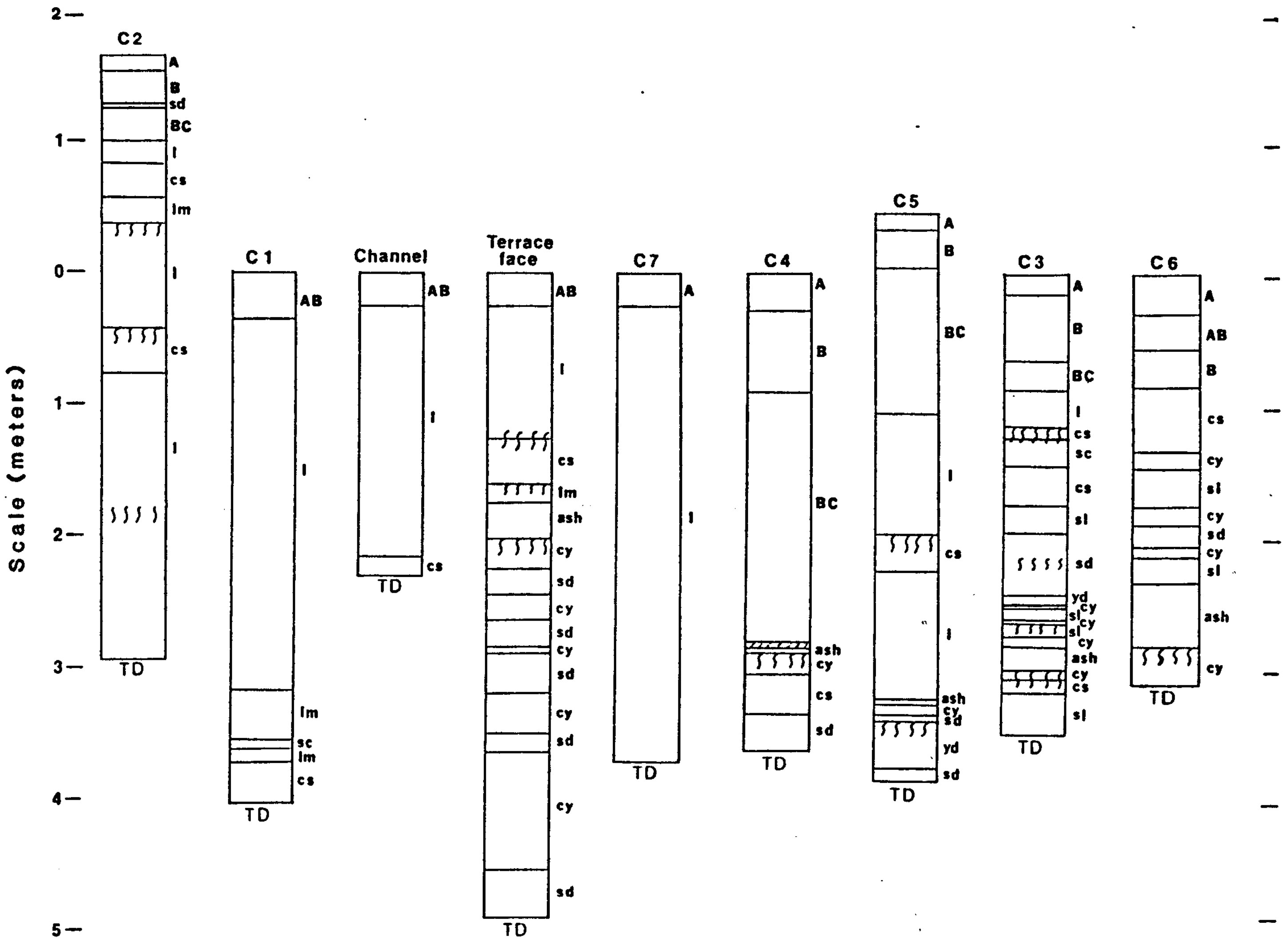

2
6
5
3
4
7
1
N
scale
1 cm = 1 m

Figure 16. Core descriptions. Datum is the surface of the terrace at the front face. Parent material of the modern soil is loess in all cores.

Key:

A - organic-rich A horizon of modern soil
AB - transitional, organic-poor, clay-enriched
B - clay enriched, frequently reddened B horizon
BC - transitional, leached, Mn nodules and/or mottling present
l - loess (C horizon of modern soil), calcareous silt, friable
cy - clay
sl - silt (alluvial)
sd - sand
cs - clayey silt
sc - silty clay
lm - laminated silt and clay
ash - evidence of ash, not necessarily airfall ash
ʃʃ - evidence of a paleosol horizon; darkening, clay skins, Mn nodules, etc.



Scale (meters)
2
1
0
1
2
3
4
5
C2
A
B
sd
BC
l
cs
lm
l
cs
l
TD
C1
AB
l
lm
sc
lm
cs
TD
Channel
AB
l
cs
TD
Terrace face
AB
l
cs
lm
ash
cy
sd
cy
sd
cy
sd
cy
sd
cy
sd
TD
C7
A
l
TD
C4
A
B
BC
ash
cy
cs
sd
TD
C5
A
B
BC
l
cs
l
ash
cy
sd
yd
sd
TD
C3
A
B
BC
l
cs
sc
cs
sl
sd
yd
cy
sl
cy
sl
cy
ash
cy
cs
sl
TD
C6
A
AB
B
cs
cy
sl
cy
sd
cy
sl
ash
cy
TD

are up to 3 cm long and are randomly oriented. They appear to be rip-up clasts. The sediment beneath these chips is alternating clay and white sand. A full ash section including green, pink and white air-fall ash is present in both core 3 and core 6. The sediment overlying the green ash in these cores is alternating clay and silt of alluvial character, however, silt overlies the ash in core 6 and clay overlies it in core 3.

The only cores in which laminated lacustrine deposits were retrieved were cores 1 and 2. Core 1 ended in nonlaminated clayey silt. A faintly laminated horizon occurred within this clayey silt beneath the lacustrine unit. Core 2 contains a faintly laminated unit above the loess. It is possible that ash is present below the base of the core. However, this is not likely (especially in core 1), based on the relative depth of the the ash in other cores and in the exposed terrace face.

A paleosol underlies the ash in cores 3 and 6. In core 4, the underlying sediment is a sesquioxide stained clay with abundant Mn nodules, thus this is probably a deeper level of the paleosol which is not darkened. The sediments below the pink ash chips in core 5 are also stained with sesquioxides and contain Mn nodules. Slightly deeper in core 5 there are abundant Mn nodules and root casts.

In the cores which do not contain ash, loess is present to considerable depths. It is possible ash was never reached, but is present in these locations. However, based on the erosional truncation of the ash bed exposed on the terrace face, it is more likely that the ash, if ever present, has been removed in these locations.

It is evident from the highly variable cores obtained from this relatively small area that the sedimentological history of the terrace fill is complex. This history can generally by explained by the scenario briefly presented below, and in more detail in the conclusions of the main text.

Alluvium was deposited in which a paleosol developed. Some of this alluvium was eroded away by a stream oriented approximately parallel to both the present terrace face and the Kansas River. In some areas, standing water must have existed in which the ash and lacustrine sediments could accumulate. In other areas, more alluvium was deposited. The channel must have down-cut further or meandered toward the present terrace face in order to erode away the ash and some lacustrine material in order to truncate these units. The block of sand present at the end of the ash on the terrace face represents either a block of older alluvium from a level above the ash which was eroded out after the ash fell, or is an eroded block of younger alluvium.

The entire area was eventually buried by loess. Several soils are present, thus the loess was not all deposited at one time. Though no unaltered loess is present in core 6, the soil present is the same as that elsewhere and thus it probably has loess as parent material. As the property owner has bulldozed away an unknown quantity of material from the terrace surface, true depths below the surface can not be obtained. The true character of the original surface of the sediment is likewise unknown.

Part II

Procedure for Fission-Track Dating and Evaluation of Differing Analytical Techniques

INTRODUCTION

Part I of this report presented a description and interpretation of the stratigraphic sequence of sedimentary units present in a small exposure near DeSoto, Kansas. A terrace surface overlying these sediments may correlate with the Buck Creek Terrace of standard nomenclature for the Kansas River valley. One of the sedimentary units is a volcanic ash which, on the basis of physical and mineralogical characteristics and magnetic polarity, is clearly shown to be the Lava Creek B Ash. At its source in the Yellowstone area, a K/Ar age of 0.62 my has been determined for this material. In contrast, when originally described by Davis and Carlson (1952), the Buck Creek Terrace was assigned an Illinoian age and a soil developed in the upper part of the alluvial fill was believed to be of Sangamon age. Despite present debate about Pleistocene nomenclature and chronology, the Sangamon Soil cannot be 0.62 my in age (see table 1 in the Introduction to Part I). Determination of an absolute age for the DeSoto ash therefore becomes of paramount importance for verifying of modifying the sequence and chronology of previously proposed episodes in the development of the Kansas River valley. This can best be accomplished by fission-track dating.

As noted in Part I, fission-track dating involves the counting of tracks created in a host substance by the passage of ionized fragments formed by spontaneous fission of ^{238}U. The rate of formation of fission tracks is a constant that can be determined. Therefore, to obtain the age of a specimen, one need only know the amount of ^{238}U present and the number (or density) of fission tracks that have developed since the specimen cooled sufficiently to become able to retain the tracks.

The easiest method by which to determine the ^{238}U concentration in a material to be dated is by comparison with a standard having a known uranium concentration. This is done by utilizing the constant isotopic ratio of ^{235}U to ^{238}U. Both the sample and the standard are irradiated together in a nuclear reactor. Thermal neutrons induce ^{235}U (but not ^{238}U) in the materials to fission. The neutron fluence (Φ) (number of neutrons striking a square centimeter area of material) is determined by counting the induced track density (ρ_i) in the standard (or dosimeter), thereby permitting the ^{238}U concentration of the sample to be calculated. A more specific discussion on the theory of fission track dating, and some of the problems involved, is in Appendix B.

The population method of fission-track dating was used to determine an age for the DeSoto ash. In this method it is assumed that there is a homogeneous distribution of uranium in the ash. The ash is divided into two splits, one of which is used to determine the spontaneous track density. The other split is sent to a nuclear reactor to be irradiated. A piece of standard dosimeter glass is sent along with the ash sample to be irradiated at the same time, since the number of neutrons which strike the ^{235}U nuclei within the sample (the nuclear fluence) determines how many induced tracks will be formed. The ratio of these three track densities (spontaneous, induced, and dosimeter) allows the age to be determined.

Two separate methodologies were used by the author to obtain glass fission-track dates. These are the methodologies of Dr. John Boellstorff (developed at the University of Nebraska, Lincoln, now at Amoco Research, Tulsa) and Dr. Charles Naeser (U.S. Geological Survey, Denver). There are some fundamental differences in the approaches used by these two scientists. The two main differences are in preparation of the glass shards for counting and in determination of the nuclear fluence used in irradiating the ash.

Boellstorff performs a bulk etch of the ash after cleaning. The etch time is calculated so that enough glass will be dissolved to remove any tracks formed by surface uranium contamination. The shards are then transferred to a microscope slide and the fission-tracks within the shards are counted in water. The standard glass used for fluence determination is a piece of microscope slide for which Boellstorff has calculated an appropriate equation which will be described below. The tracks are counted directly on the polished and etched glass slide standard. Boellstorff also redates three or more subsamples of the Borchers ash (now generally called the Huckleberry Ridge Ash, see Introduction of Part I) along with each irradiation in order to check the fluence determined from glass dosimeters and also to serve as an intra-laboratory standard. Samples of both the DeSoto ash and the Borchers ash, along with several glass dosimeters, were sent to the Georgia Institute of Technology Research Nuclear Reactor (a graphite reactor) for irradiation and dated by this method.

For Naeser's method, the shards must be embedded in epoxy and polished before etching. This polishing removes enough glass to eliminate any tracks which may have formed from surface uranium contamination. Fission-tracks are then counted on the exposed portions of shards on these slides. The ash dated using this procedure was irradiated by Naeser in the U.S.G.S. TRIGA reactor in Denver. The nuclear fluence was determined by using the National Bureau of Standards (NBS) fission-track glass standards. In order to eliminate the need to repolish and etch the NBS glass after each irradiation, and thus preserve it, the external detector method is frequently used. In this method the tracks are counted on a piece of track detector instead of on the glass itself. A piece of muscovite detector was attached to the NBS glass standard before irradiation. One of the two fission fragments from the original ^{235}U nucleus which split during fission will form a track in the detector, while the other fragment will form a track in the glass.

Since glass shards anneal at relatively low temperatures (Storzer, 1970), especially when hydrated (Lakatos and Miller, 1972), an attempt to date the more stable zircon phenocrysts was made. Zircon is dated by the external detector method. The zircon crystals are embedded in FEP teflon and etched before irradiation so that the spontaneous tracks are counted in the zircon itself. A piece of muscovite detector is then attached to the etched teflon chip over the zircons and the entire package irradiated. The induced tracks are then counted in the muscovite detector after it is etched.

Irradiations were performed in the same reactors used by Boellstorff and Naeser, respectively, in their own work. Particulars of their respective methodologies are located in Appendix C. Following is a brief discussion of the different dosimeters used in the research and methods of fluence determination.

Neutron Fluence Determination

Fission-track glass standards are used to determine the fluence within the reactor to which the irradiated shards have been exposed. The thermal neutron flux is the rate at which neutrons bombard a substance ($n/cm^2/sec$), while the fluence is the integrated flux over time (n/cm^2). Differences in the chemistry of these various glasses as well as differences in the method by which each was initially calibrated, including the different energy spectra generated by various reactors, can result in a different fluence being determined from the use of different standard glasses. Three different glass standards were selected for use on this project. More detailed information is located in Appendix C.

Most workers engaged in fission-track research within the United States use National Bureau of Standards (NBS) Fission Track Glass wafers. These wafers (12 X 3 mm) can be purchased in a set containing four non-irradiated glass wafers and two wafers irradiated by the NBS. Four different uranium levels are available. These glasses are a subset of NBS's series of Trace Elements in Glass standards. NBS standard SRM962a, which contains 37.38 ppm U, was used as one standard. Naeser uses the analogous NBS standard, SRM612 of the Trace Elements in Glass series, as his dosimeter.

Boellstorff uses a 1/2 by 1/4 inch (~13 X 6 mm) chip from a set of microscope slides which were calibrated to Fleischer's original set of dosimeters as his standard. These chips were also used in this research. This is the same procedure used by Fleischer, Price, and Walker (1965) in their pioneering studies on fission-tracks. The uranium distribution was found to be uniform and present at a concentration of 0.7 ppm (Boellstorff, pers. comm.; 1985).

A third glass standard was obtained from the Corning

Museum of Glass. The Corning glass CN-1 (1 in^2 (2.54 cm^2)), contains approximately 40 ppm U. It was chosen as the third dosimeter because it is the only specially made natural uranium glass standard presently available. It was cut into pieces the same size as Boellstorff's standard chips for ease in handling. The effects of using glass standards prepared with depleted uranium and/or containing other trace elements are outlined in Appendix C.

The neutron fluence is determined from the NBS standards by directly comparing the fission-track density in the dosimeter (ρ_o) irradiated along with the samples to that in an identical piece of glass irradiated previously by the National Bureau of Standards under known conditions. Both irradiated and nonirradiated specimens are included in the package of dosimeters which can be purchased from the NBS.

The fluence (Φ) can be determined for the Boellstorff standards directly from ρ_o. The original equation described by Fleischer and Price (1964) was modified by Boellstorff (Equation 1) to fit his standards, since they contain more

$$\Phi = 1.13 \quad 0.03 \times 10^{11} \rho_o \qquad \text{eqn 1}$$

uranium than Fleischer's original standards. In order to use Equation 1, it is essential that the etching conditions be replicated as exactly as possible.

For the Corning standard CN-1, neither an equation nor a pre-irradiated specimen exist. The fluence was thus calculated by a method analogous to that described for the NBS dosimeters. Since the neutron fluence can be expressed as a function of a constant and the dosimeter fission-track density (Equation 1), once the value of this constant is determined for a specific dosimeter, only ρ_D is necessary to calculate Φ . This constant is termed a B factor by Fleischer et al. (1975) and is discussed further in Appendices B and C. Thus, if the fluence can be determined by any other method for a reactor run, including a different dosimeter, a B factor can be calculated for the dosimeter lacking an irradiated pair. The B factor for the Corning glass was calculated from the specimen irradiated in Denver using the fluence Naeser had determined for this run from his NBS dosimeter. This B factor was then used to calculate the unknown fluence for the Georgia Tech reactor.

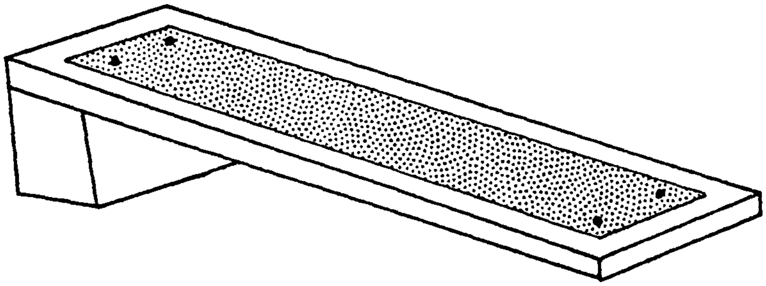
In order to determine ρ_D , the dosimeters must be etched in order to make the fission-tracks large enough to count under an optical microscope. Following is the procedure for the determination of ρ_D in each of the dosimeters.

Preparation of standards

Before irradiation, each piece of dosimeter was cleaned with alcohol to remove as much uranium contamination from dust, etc. as possible. The dosimeters should be placed at several locations within the irradiation package so that possible variations in the nuclear flux during irradiation can be mapped.

After irradiation, the glass pieces, including the pre-irradiated NBS standard, were each glued to a glass microscope slide using Buehler epoxy. The original surface was removed by grinding in one direction on a slant board (see figure 17) to which a piece of automotive wet-dry 400 grit paper had been attached. The slide was rotated by 180° after each pass to prevent the slide from becoming thinner on one side than the other. Grit of this size creates scratches which are about 25 microns deep (Buehler, 1976). This guarantees that when the scratches are removed, any tracks seen will have been formed in four-pi geometry, (e.g. from uranium atoms which may have been either above OR below the final glass surface) and that none of the fission-tracks counted are from uranium contamination on the original surface, since the etched fission-tracks are only about 12 microns long (Boellstorff, 1973a). These scratches were removed by grinding them in a similar fashion with 600 grit paper in a perpendicular direction. Polishing was done on a Buehler Ecomet III polisher/grinder using Buehler Microcloth polishing cloth. This fuzzy cloth yielded better results than flat linen clothes, because the thick slides rapidly tore the linen. The 600 grit scratches were removed by moving the slide along a radius of the polishing disk with 1000 SiC grit. Final polishing

Figure 17. Slantboard. Apparatus used to grind slides so only unidirectional scratches would be formed.



was done similarly, but with a six micron diamond slurry. It is best to keep scratches unidirectional to limit confusion of polishing pits with fission-tracks (Naeser, pers. comm.; 1985). The slides were cleaned in a weak Alconox solution in an ultrasonic cleaner for several minutes, rinsed with tap water, and squirted with alcohol to dry.

Etching techniques

The etching procedures for use with Equation 1 are much different than those for the other glasses. The object is to replicate the conditions for which the equation was derived instead of to create well-developed fission-tracks. Boellstorff's microscope slide standard was etched as per his directions (five seconds in 48% HF at 20°C). Tracks can be seen because glass damaged by the passage of a fission fragment dissolves faster than undamaged glass. Unfortunately, five seconds is not long enough to develop fission-tracks fully. The differential etch rate has not had a chance to allow tracks that are nearly parallel to the dosimeter surface to be seen (see Appendix C). After awhile, the critical angle at which a fission-track axis must penetrate the glass surface in order to be seen stabilizes and the fission-track density will no longer change with etch time (assuming the uranium concentration is homogeneous). The time it takes for this to occur depends on several factors, including the chemistry of the glass.

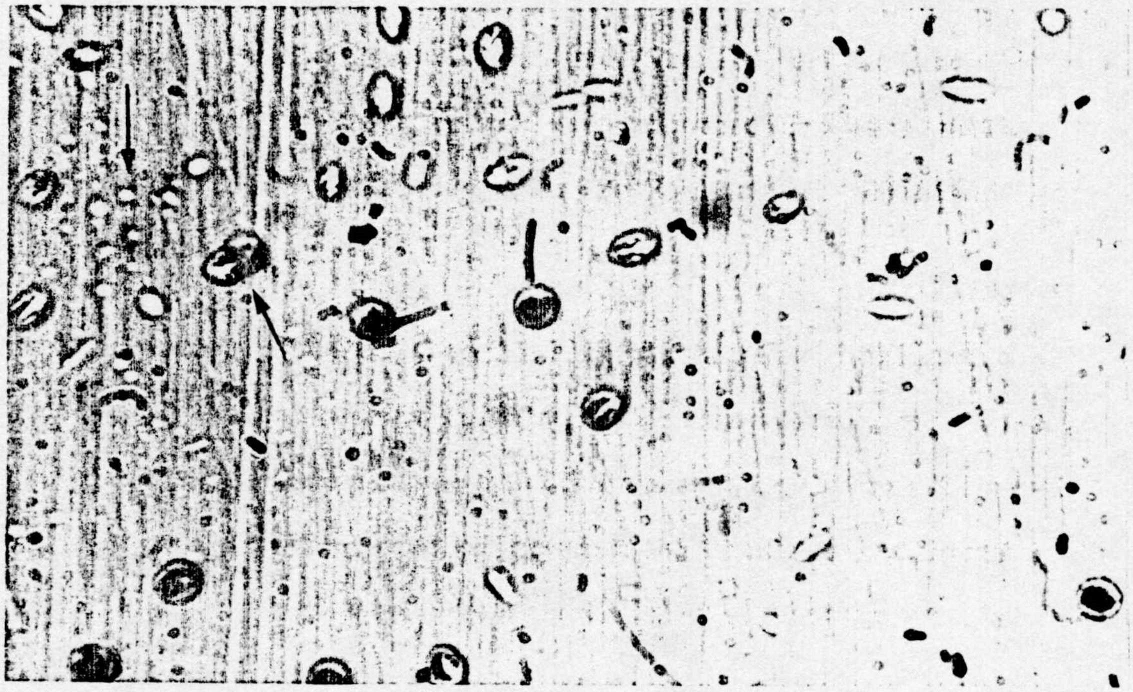
The etching conditions are thus critical for the Boellstorff glass. The procedures used to obtain these etching conditions were as follows. The acid was cooled in an ice bath on a magnetic stirrer until the temperature stabilized at 20°C. Two pieces of the dosimeter (which had been irradiated at Georgia Tech) were glued to a microscope slide which was then held in a bulldog clip and agitated in fresh 48% HF. So as to best approximate a true etching time of only five seconds, the slide was initially pulled from the acid just after four seconds passed and then held in a 1000 ml beaker of distilled water for several minutes. The slide entered the water just before five seconds, resulting in the etch stopping quite near the proper time. After the initial rinse, the slide was held under running tap water to be sure all traces of HF were removed.

The remaining standards (including the other Boellstorff standards) were all etched at room temperature (26°C) in 24% HF as per Naeser's procedure (Briggs, 1978). Fifty milliliters of 48% HF were slowly poured into 50 ml of distilled water in a 150 ml plastic beaker. The mixture was poured back and forth several times to ensure complete mixing. The 24% HF was allowed to cool in a covered beaker for 30 minutes, since HF has a relatively high heat of dissolution. Thirty seconds was determined to be a satisfactory etching time by experimentation with duplicates of each standard. Several factors, including glass chemistry, effect the etching efficiency, thus the time must be determined separately for each type of glass.

The NBS dosimeter irradiated by the NBS was etched simultaneously with a wafer irradiated along with the samples sent to Georgia Tech. The remaining Boellstorff standards (one of which had been irradiated in Georgia and the other in Denver) were then etched together, followed by the Georgia/ Denver pair of Corning standards. The slides were held in a bulldog clip and agitated in acid. At thirty seconds they were transferred to a 1000 ml beaker of distilled water to stop the etch. They were then thoroughly rinsed in running tap water.

When the etch was checked by viewing the slides at 600X under a microscope, it was discovered that on many of the slides the tracks were decorated with an unknown substance. Rinsing the slides for fifteen seconds in a 3M solution of HNO_3 succeeded in removing the substance. This procedure is similar to that suggested by Carpenter and Reimer (1974) for cleaning dosimeters prior to irradiation. Boellstorff later commented that this occurrence is normal, he sometimes uses a toothbrush and liquid soap to clean the glass slides (pers. comm.; 1986). Immediately prior to counting, the slides were cleaned by rubbing gently with liquid dish soap and a finger tip, as had been suggested by Boellstorff (pers. comm.; 1984).

Figure 18. Fission-tracks in a dosimeter. Fission-tracks are conical and are readily visible in dosimeter NBS-SRM962. A cluster of etchant "dishes" is present in the upper left (vertical arrow). Light will focus to a point in the base of a fission-track as the focus knob of the microscope is turned, but sweeps across etchant pits. The vertical lines are polishing scratches. A double track (diagonal arrow) is also present. (630X, plain light)



Counting techniques

Counting was done at 600X under a Bausch and Lomb microscope with a transmitting light objective and halogen light source. Approximately 1000 tracks should be counted for each fluence determination. A 1 mm^2 grid with 100 square subdivisions was used for counting. The fission-track count varied substantially between the dosimeters because the ^{235}U concentration was also highly variable. For the NBS and Corning dosimeters, fission-track counts were recorded for each small square. The density was so low in the Boellstorff dosimeter that counts for the entire grid of 100 squares were recorded.

The large expanse of good quality glass makes fission-track counting of the dosimeters relatively fast and easy. Fission-tracks should be cone-shaped and come to a sharp point. Light should move along the slope of the fission-track as the focus knob is moved up and down. The etchant caused pitting of the glass surface, creating shallow "dishes", but these should not be counted (see figure 18). All the scratches are unidirectional, so the occasional track-like pitted scratch can be identified as such.

In regions with a high density of fission-tracks, counting can become difficult. The points of light in the base of each track can usually be resolved. If the tracks can not be differentiated, the reactor run must be repeated with either a lower nuclear fluence or a different dosimeter, depending on the fission-track density within the irradiated samples.

Glass Fission-Track Dating

Sample collection

The ash is located in a terrace fill along the front of which a driveway has been cut (see figure 1 in the Introduction to Part I). The face of the fill was scraped clean and then approximately another inch of material removed from the surface of the ash in order to minimize the possibility of contamination. The ash bed itself varies in thickness, as was discussed earlier. A trowel was used to remove samples from the thick portion of the white airfall ash, trying to minimize the amount of reworked ash and extraneous material while maximizing the percentage of basal ash retrieved. The effects of gravity settling caused the lowermost portions of the ash to contain the highest percentage of large shards and phenocrysts. Ash was removed to a depth of about one foot back into the terrace face. Approximately 10 gallons of ash were obtained.

Sample preparation

The white ash was processed as described previously in the section on petrography. The resulting glass fraction (-120+170 mesh, 0.125 - 0.088 mm) was then rinsed and decanted several times. It was then agitated for several minutes in an ultrasonic cleaner to remove fines from inside vesicles. The ash was treated with concentrated hydrochloric acid and 30% hydrogen peroxide solutions, respectively, in order to remove any carbonates or organics. In order to promote quick drying, the ash was then centrifuged at 3000 rpm for eight minutes. Finally, the clean ash was spread out on paper towels underlaid by aluminum foil to dry in an air conditioned room.

Before dates can be obtained, a portion of the ash must be irradiated in a nuclear reactor. As stated above, this is the best method for determining the uranium concentration of the glass shards.

Boellstorff's method

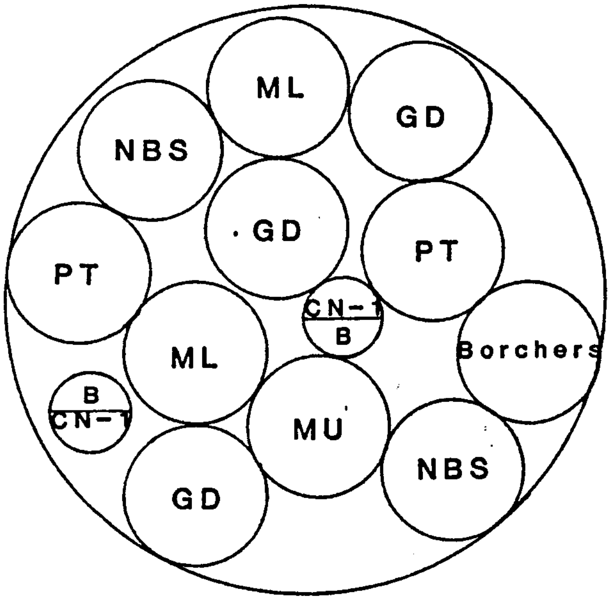
Irradiation procedure - Approximately 10 grams of clean ash were irradiated in position V-43 of the Georgia Institute of Technology Research Nuclear Reactor by Dr. Ratif Karam. The reactor power was 250 Kw with a sub-cadmium neutron flux of approximately 1.4×10^{11} n/cm^2/sec. Thus irradiation for 80 minutes yields a neutron fluence of 6.7×10^{14} n/cm^2, a figure within 5% of the fluence used by Naeser when he irradiated the author's other sample in Denver.

Since the fluence is a determining factor in the number of tracks which will be induced in the glass shards (see above and

Appendix C), it is important to choose an exposure which will produce a countable track density. It is desirable to have a relatively high density of tracks so that counting will be fast. If the density is too high, however, tracks may obscure each other and make counting difficult. The sample vessel was rotated at 1 rpm to try to minimize the effect of flux variations within the reactor, as suggested by Boellstorff (pers. comm., 1984).

Ash samples were placed in approximately 5.5 X 1.5 cm polyethylene plastic vials. The Boellstorff standard (B) and Corning standard (CN-1) were placed into similar, though smaller vials of 2.5 X 1 cm. The National Bureau of Standards (NBS) glass disks were placed in the larger vials, as they would not fit in the smaller vials. Standards were placed at two or more radii and at two or more elevations within the one quart bottle in which samples are irradiated, as suggested by Dr. Kirkland (pers. comm., 1984), because the flux within a reactor varies. Figure 19 is a diagram of the location of the different standards and samples within the one quart irradiation vessel. Unfortunately, the location of each vial was merely mapped and the vials were not separately labeled as to location. The radiation safety officer at the University of Kansas later unpacked the contents of the irradiation vessel when it was returned from Georgia Tech, thereby destroying all record of positioning. Thus, it was not possible to map any flux variations that may have occurred.

Figure 19. Packing arrangement within irradiation vessel. Each small circle represents a vial. The split circles are stacked smaller vials, the upper vial is listed on top. GD = DeSoto ash, CN-1 = Corning Museum of Glass 40 ppm uranium standard glass, B = Boellstorff's standard glass (MU, ML, and PT are other ashes). The Borchers ash was irradiated and counted as an interlaboratory standard.


ML
GD
NBS
GD
PT
PT
CN-1
B
ML
Borchers
B
CN-1
MU
NBS
GD

Etching techniques - Since the hydrofluoric acid (HF) etchant dissolves glass, plastic containers must be used for all procedures. The procedure given the author by Boellstorff (pers. comm., 1984, 1985) was slightly modified in an attempt to reduce any differential etching of the induced and spontaneous tracks.

A 24% HF solution was made by pouring 50 ml of concentrated (i.e. 48%) HF and 50 ml of distilled water back and forth between two plastic beakers several times. HF acid has a very high heat of dissolution, therefore the acid was then allowed to cool back to room temperature. Two 1000 ml beakers with 800 ml of distilled water in each were set on magnetic stirrers to quickly and thoroughly disperse the ash when stopping the etch. Etching was done in 1 pint plastic bottles from which the tops had been removed. These were set in a sonic cleaner in order to keep the ash continuously agitated. One tablespoon of irradiated ash was placed carefully at the base of one container and one tablespoon of nonirradiated ash was placed in another container. Care was taken to ensure that no ash clung to the side of either container. This quantity of ash was found to be the largest amount which could be efficiently etched at one time. Fifty milliliters of 24% HF was poured into each container. These were occasionally swirled by hand.

Two minutes was determined by Boellstorff (1973a) to be adequate to remove any tracks which may have formed from surface uranium contamination for his mid-continent Pleistocene ashes and was used for this study. After two minutes, the etch was stopped by pouring the contents of each container into a separate beaker containing 800 ml of swirling distilled water. The stirrer was immediately turned off and the water poured off as soon as the ash settled. It is possible that mild etching is still occurring at this point since the ash is in a weak acid. Therefore, the 1000 ml beakers were refilled and decanted several times.

Finally, the ash was retrieved by filtering through a vacuum filter. Most of the DeSoto shards had dissolved completely by this time; however, what remained were the thick, flat, nonpumiceous shards desirable for counting. It is easier to count tracks on flat, broad expanses of glass than on the tiny ridges between the vesicles in pumiceous glass. Most of the Borchers ash (interlaboratory standard), being coarser, survived the etch procedure. The etch time can not be reduced substantially because enough material must be removed to be sure no tracks from surface uranium contamination still exist (Boellstorff, 1976).

Counting techniques - Counting of tracks was done under an optical Bausch and Lomb microscope with a halogen lamp and a transmitting light objective at 600X. A random sample of the ash was transferred with an eye dropper from the vial to a glass microscope slide. The slide was covered with a large cover slip and water added carefully along an edge as necessary to keep the slide from drying.

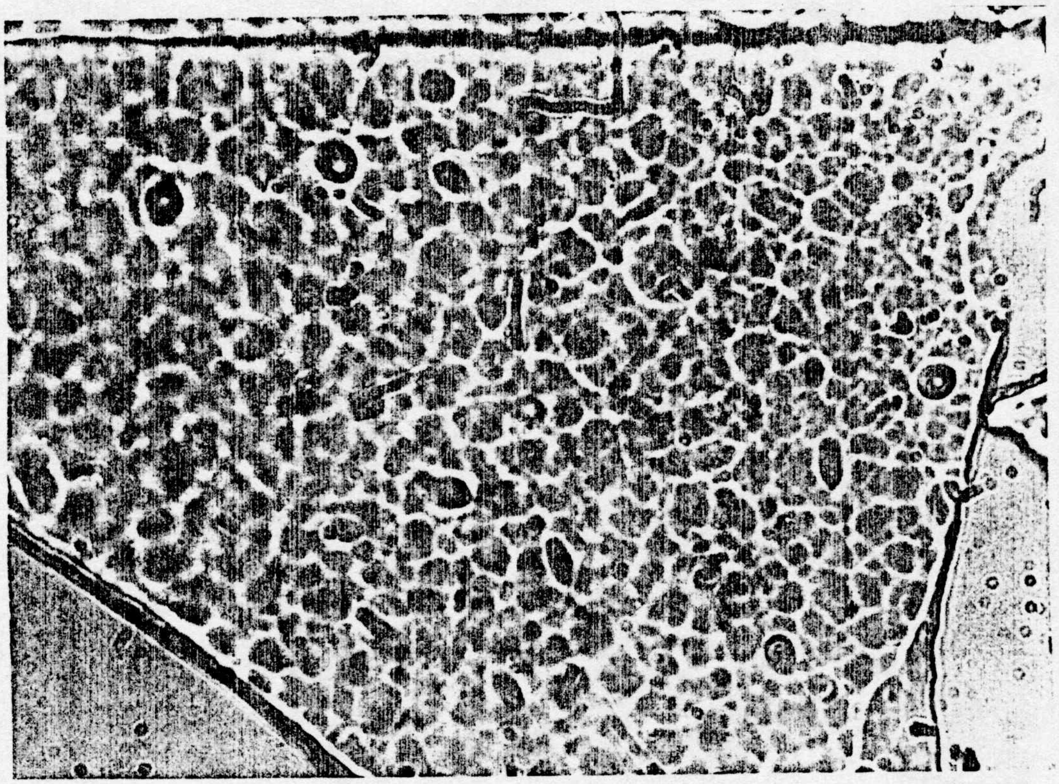
The area of glass counted was determined by use of a counting grid. The grid used here was 1 mm^2 and divided into 100 square subdivisions. The area of good quality glass (no vesicles, crystalites, etc.) which can be found with a reasonable frequency is determined by scanning the shards under an optical microscope. The DeSoto ash was counted using an area of 4 X 4 squares and the Borchers ash using an area of 5 X 5 squares (compare figures 10 and 20). Three hundred areas of spontaneous tracks and one hundred areas of induced tracks should be counted.

The most efficient method to survey a slide is to scan across it with one axis fixed. This was accomplished on the available microscope by immobilizing one of the axis adjustment knobs with masking tape. The slide was scanned until an adequately sized shard was located. The grid was then positioned appropriately on the shard, and all tracks within the counting area were counted. The grid should be positioned with the shard slightly out of focus to inhibit selecting biased placements based on track location within the shard. The focus needs to be moved up and down constantly while counting. Tracks are conical and the light will move along the track as the focus knob is turned, ending in a dot of light (see figures 18 and 21).

Figure 20. Morphology of Borchers ash shards. Note the overall larger size of these shards compared to those of the DeSoto ash in figure 10 at the same magnification. Bubble wall (1), bubble junction (2), vesicular (3), and pumiceous (4) shards are present. The cloudiness of some shards is caused by small amounts of attached clay. This is the 0.25 - 0.147 mm (-60+100 mesh) sieve fraction as seen in plain light.

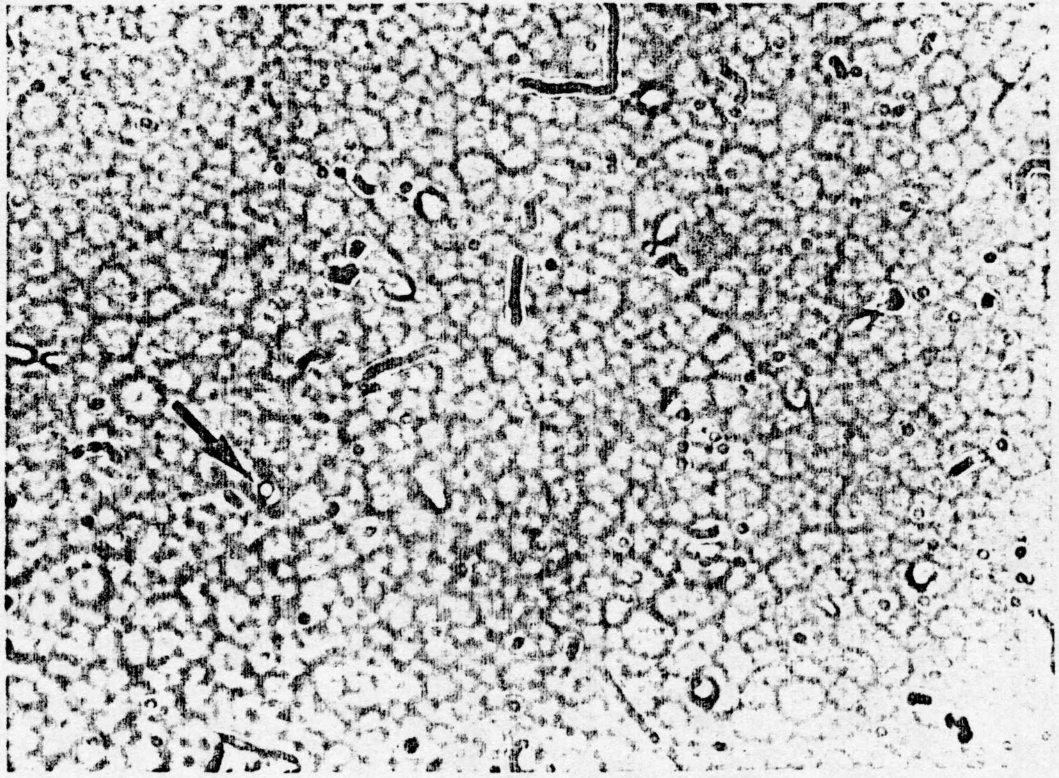

3
1
4
2

Figure 21. Fission-tracks in the DeSoto ash - Boellstorff's method. A) Fission-tracks on a curved shard surface. By moving the focus knob of the microscope, tracks on the left side of the figure and the underside of the shard can be brought into focus and counted. Note the etched surface texture of the shards. B) Etch-through. Since it can be determined that one track etched all the way through the shard (arrow), it should be counted as two tracks, one starting from the base and the other from the top of the shard. This is because the total area counted is actually twice that scanned since tracks on both sides of the shard are counted. If it were not clear how many tracks, if any, were actually present, the shard should not be counted. Etched-through tracks are easily confused with small vesicles. (630X, plain light)



A

B



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As the entire shard was etched, tracks on BOTH sides will be revealed. If the focus knob is not turned far enough to focus on both these sets of tracks, the resultant track density will be in error. On thin shards, tracks can etch all the way through (see figure 21). Counting in areas where this has occurred was avoided. It is very difficult to tell one track from two tracks which have joined, or either of these from a vesicle. It can be difficult to distinguish surface pitting caused by the etchant from tracks. These pits are usually "dish-shaped" while tracks are conical (see figure 18). This is one reason why it is essential to continually turn the focus knob up and down. Light acts differently inside a cone from inside a partial sphere. Shadows should converge near the center, not sweep across a potential track. Subsequent scans were several grid widths from the previous scan to avoid recounting the same shards.

The number of tracks counted in each area, including the zeros, was recorded. Then the spontaneous track density is calculated from the nonirradiated shards and the induced plus spontaneous track density from the irradiated shards. Since both sides of each shard were counted, the area used in the calculations is actually twice the grid area scanned.

Age calculation - To determine the reactor flux for the irradiation from the standard glasses, the dosimeter track density is required (see Neutron Fluence Determination). The age of the Borchers ash is calculated first to check the fluence calculated by counting tracks in the dosimeter. This ash has been repeatedly dated at about 2 my. If a different age is obtained, there is an error in counting or elsewhere. If this occurs, the dosimeter and Borchers ash should be recounted to establish that the calculated track densities are accurate. If the fluence determination is still in error, a decision must then be made as to whether the problem is large enough to warrant discarding the sample data. This depends on the accuracy needed to answer the question being asked by the worker. If not, one then determines an appropriate fluence by back calculating from the Borchers ash age. This fluence is then used to determine the age of the unknown ash.

The ages reported for each sample generally should represent the mean and one standard deviation (binomial) of at least three age determinations made on subsets of the ash sample (Boellstorff, 1973a). The age obtained from the original Borchers ash calculation should also be reported to give an additional indication of the accuracy of the age determination. Since this ash has been repeatedly dated, it can be used as an interlaboratory standard (Boellstorff, 1976) to determine at least general directional biases of different workers and reliability of the reported ages.

The version of the age equation used by Boellstorff

(Equation 2) has taken into account the slight amount of alpha decay which occurs and combined the various constants.

$$A = 14.95 \times 10^{9} \log \left[1 + 9.50 \times 10^{-18} \frac{\rho_s \Phi}{\rho_i} \right] \qquad \text{eqn 2}$$

In equation 2; A is the time over which fission-tracks have been stored, Φ is the nuclear reactor fluence, ρ_s is the spontaneous fission-track density, and ρ_i is the induced track density. This equation is the same as that used by Naeser (Equation 5), except for the use of log base 10 instead of the natural logarithm.

Naeser's method

Irradiation procedure - Approximately three grams of the DeSoto ash were sent to Dr. Charles Naeser to be irradiated in the U. S. Geological Survey TRIGA Reactor in Denver. One piece each of Corning glass standard (CN-1) and Boellstorff's standard were included in order to directly compare the nuclear flux within the Denver and Georgia reactors, respectively. The irradiated ash was returned to the author folded within a flat 1/2 inch square foil package, while the standards were returned as loose chips. This sample had been irradiated along with NBS-SRM612 standard glass, the fission-track dosimeter generally used by Naeser.

A suitable neutron fluence can be determined using equation 3:

$$\Phi = 1.63 \times 10^{7}\ T \qquad \text{eqn 3}$$

where Φ is the total neutron fluence in n/cm^2 and T is the expected age in years (modified from Naeser, 1976). If the normal neutron flux (n/cm^2/sec) is known for a specified power level, the exposure time can be readily obtained. Naeser calculated that a fluence of 6.96×10^{14} n/cm^2 had been used for the samples sent to him by counting the tracks in a muscovite track detector placed on the NBS standard during the irradiation.

Slide preparation - There is really only one way to make these slides. Repeated experiments with different epoxies and various manufacturing techniques failed. An epoxy smear will curl up and fall off the slide during etching; while ash sprinkled on top of an epoxy blob will aggregate and sometimes sink, leaving pits scattered over the slide surface and too little glass at any one level when the slide is polished. The pits are covered with nonembedded shards which have not been polished.

Naeser uses Buehler epoxy and a grooved teflon block to make slides. The Buehler epoxy is clear, hardens in about three days at room temperature, and has a viscosity low enough to permit it to flow easily and entrap shards. The teflon block contains a central trough approximately 1 mm deep. A small pinch of ash is scattered over the center of the trough and a few drops of epoxy dropped onto it. The labeled slide is inverted over the epoxy and a weight placed over it until the epoxy cures. The slides can be popped off by flexing the teflon block. Detailed directions can be found in USGS Open-File Report 76-190 (Naeser, 1976).

Naeser suggested using coffee can lids and microscope slide chip spacers to make slides at the University of Kansas. This never worked particularly well with the Hillquist epoxy on hand. Though it cures in about 24 hours at room temperature, it seemed too viscous to embed the shards. Heat cured epoxy can not be used because the tracks might become annealed, and thus disappear, during curing. In the end, several slides were made by the author in Denver using Dr. Naeser's equipment.

After the epoxy has thoroughly cured, the slides must be polished. Naeser uses 400 grit to expose the shards and remove any fission-tracks which may be present due to surface uranium contamination, 600 grit to remove the 400 grit scratches, and six micron diamond paste to polish the slides.

It is important to maintain unidirectional scratches in order to maximize the chance of removing them with the next lower grit size and to facilitate differentiating fission-tracks from polishing scratches. At the University of Kansas, this was accomplished by using the technique described earlier for preparation of the standards. Four to six passes down the slant board at moderate pressure were generally sufficient to expose the shards. After final polishing, the glass shards should be highly reflective when tilted into the light under a microscope. The slides were cleaned in a mild Alconox solution in an ultrasonic cleaner and thoroughly rinsed.

Etching techniques - It is important that irradiated and nonirradiated slides be etched under identical conditions. Different etching conditions can result in a different proportion of fission-tracks becoming countable on the slides (see Appendix B). The ratio of spontaneous to induced fission-tracks should depend only on age and reactor fluence. Therefore, a bulldog clip (having a capacity of four slides) was used to hold one irradiated and three nonirradiated slides back to back for simultaneous etching. The ratio of tracks (ρ_s/ρ_i) should be calculated only from slides etched together.

As etching time is dependent on glass chemistry, a few trials must be run in order to determine the optimum etch duration. Tracks should be sharp bottomed and in random orientations. Experiments indicated that fifty seconds was a suitable etch duration for the DeSoto ash.

A 150 ml plastic beaker containing 100 ml of 24% HF prepared as described earlier for the standards was used to etch the slides. The slides were dipped into the beaker, and moved back and forth gently. After etching, the slides were put into a 1000 ml beaker of distilled water and agitated for several minutes. Afterwards, the slides were held under running water for several more minutes to be sure all the acid was removed. In order to be consistent in the treatment of samples and standards, the slides were then dipped into a weak nitric acid (HNO_3) solution for fifteen seconds. See the section on Preparation of Standards for an explanation.

Upon checking the slides after etching, the glass surfaces did not look "right". Many pitted areas existed and overall shard/track quality seemed low. Since the author had not seen shards like these when working in Dr. Naeser's lab, it was assumed the problem was related to incomplete polishing and that the irregular surface visible was caused by etched scratches. Therefore, the slides were reground and etched, being extremely careful to be sure all the etched tracks were ground away. As it turned out, the problem was caused more by the small grain size of the DeSoto ash and its morphology, as well as the microscope equipment available. The pitted surface was caused by the etchant removing a thin layer of epoxy from over a portion of a shard and then etching unpolished glass. The general haziness of the shards is probably attributable to using a transmitted light objective instead of the reflected light objective used by Naeser. Portions of many shards get lost in a "glow" caused by light refracting as it passes through the many shard/epoxy interfaces. See figure 22 for examples of the problem as seen after re-etching.

Figure 22. Fission-tracks in the DeSoto ash - Naeser's method.

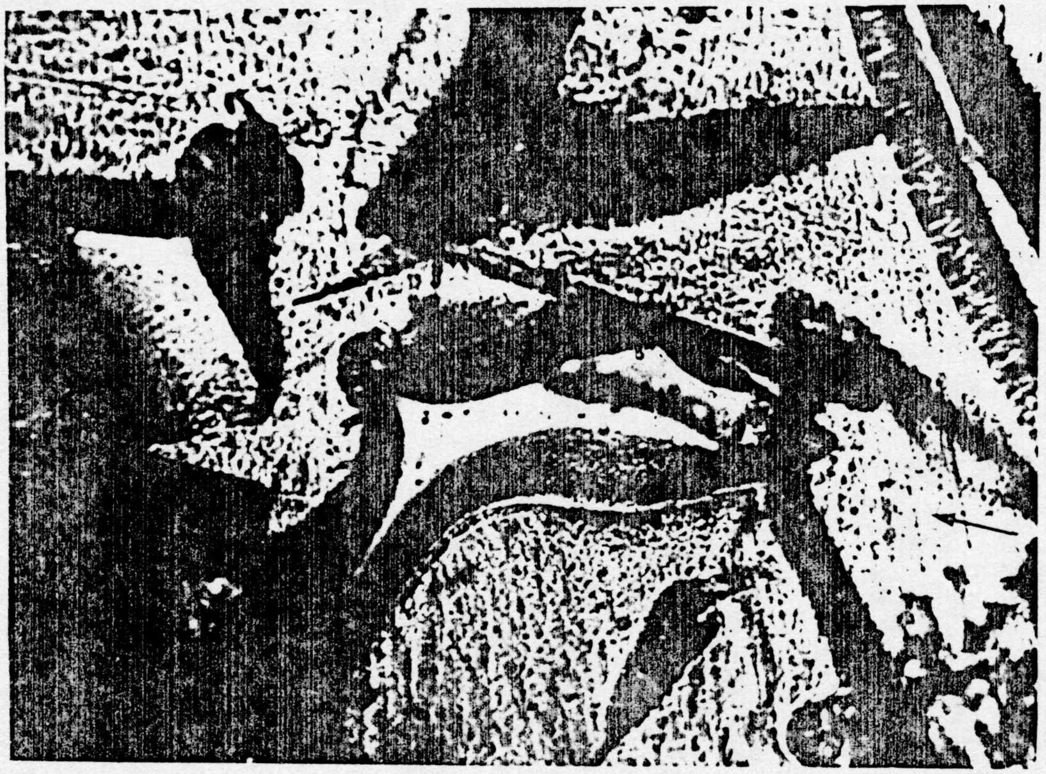
A) Portion of a polished slide at 250X. Note the relatively low proportion of polished glass at the surface. The area of the central shard above the rib (light/dark stripe) can not be adequately seen to count because of refraction problems. The shard marked by the arrow is covered by a thin layer of epoxy.

B) Portion of a polished slide at 250X. Fission-tracks are visible (arrows) in the central shard. Both shards in the upper right are uncountable because of refraction problems.

C) Nearly etched out tilted shard with a fission-track at 630X. From the top are: epoxy "wall" (former side of shard), basal cast of the shard, polished glass, shadowed epoxy.

D) Shard at 630X containing fission-tracks and a scratch. The linear arrangement and spherical shape of the scratch pits (arrow) distinguishes them from the conical fission-tracks.

All viewed in plain light.



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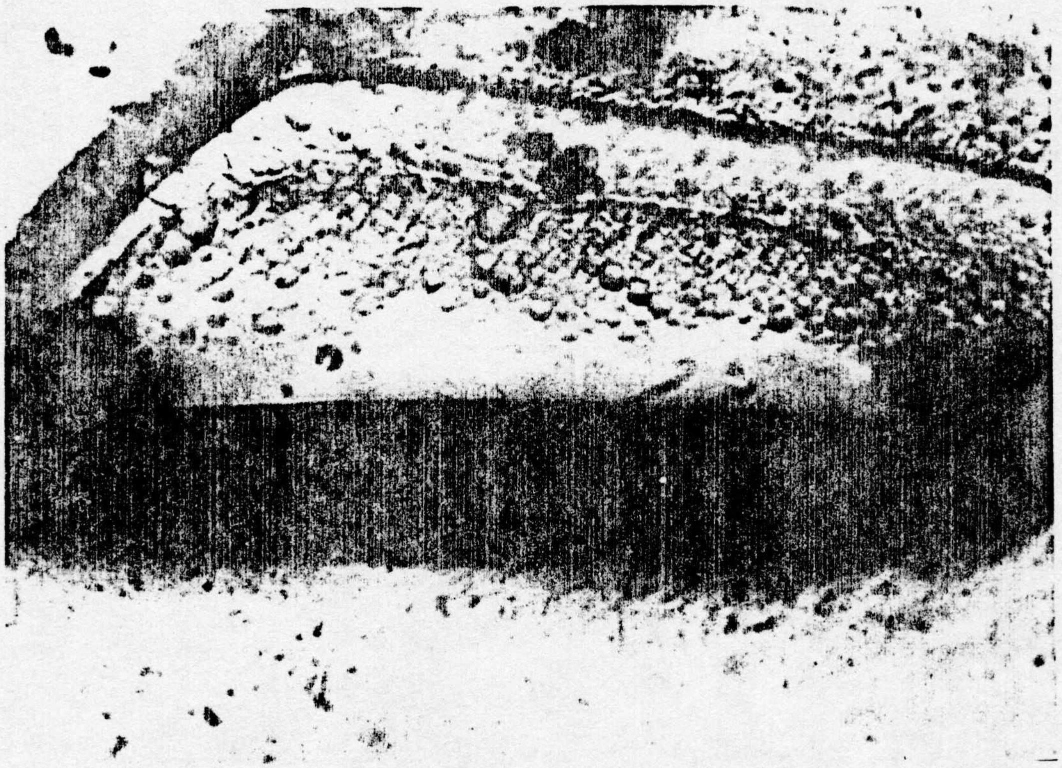
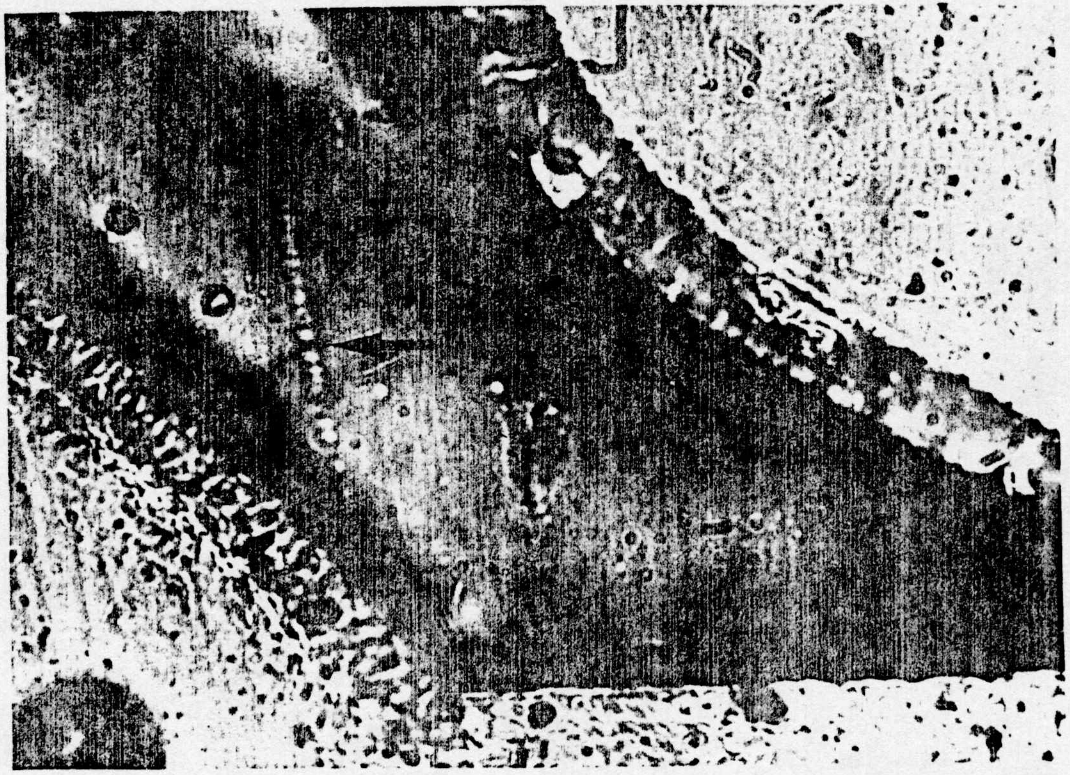
B



C

D



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Counting techniques - It is very important to count tracks only where polishing scratches are visible. Tracks in other areas (e.g. the bases of vesicles) may have been created by uranium lying on the shard as surface contamination. Glass area was determined by point counting. A 1 mm^2 grid with 100 intersections was used during counting. The number of times an intersection falls on countable glass was recorded, as well as the number of tracks seen anywhere within the grid for each field of view. An average of only 1% of the slide surface contained countable glass, i.e. glass which was not shadowed or otherwise not readily visible, which did not contain polishing scratches, etc..

Counting was done as described earlier. The area of highest shard density was outlined on each slide to minimize time spent counting essentially pure epoxy. This entire outlined area was then scanned. Adjacent E-W grids were counted across the entire area. The N-S axis was then moved 0.5 axis location indicators (about 1 1/2 grids) after each E-W pass to prevent recounting of shards.

As these fission-tracks are on polished glass surfaces, their appearance is very similar to those on the standards (compare figures 18 and 22). At least a thousand tracks should be counted on both the irradiated and nonirradiated ash slides, respectively. Given the low glass density, as well as low track density in the young, fine grained DeSoto ash, it proved impossible to obtain this number of spontaneous tracks.

Age calculation - The area of good glass scanned was calculated by first determining the percentage of glass (as opposed to epoxy or uncountable glass) in relation to the total area scanned. The spontaneous plus induced track density can then be determined for the irradiated ash and the spontaneous track density determined for the nonirradiated ash. These values were substituted into Equation 4 to obtain the age of the sample:

$$A = \frac{1}{\lambda_D} \ln \; 1 + \frac{\lambda_D \sigma I \rho_s \Phi}{\lambda_F \rho_i} \qquad \text{eqn 4}$$

where A is the time over which fission-tracks have been stored, λ_D is the decay rate of uranium due to α decay, σ is the cross section of uranium, I is the isotopic ratio of $^{235}U/^{238}U$, Φ is the neutron fluence, λ_F is the decay rate of ^{238}U due to spontaneous fission, and ρ_s and ρ_i are the spontaneous and induced fission-track densities, respectively. Upon substitution of values for the constants ($I = 7.26 \times 10^{-3}$, $\lambda_D = 1.54 \times 10^{-10}$/yr, $\lambda_F = 6.85 \times 10^{-17}$/yr and $\sigma = 582 \times 10^{-24}$ cm^2) this equation becomes:

$$A = 6.49 \times 10^{9} \ln \left[1 + 9.50 \times 10^{-18} \frac{\rho_s \Phi}{\rho_i} \right]. \qquad \text{eqn 5}$$

The neutron fluence is determined by counting tracks in the NBS standard glass. The standard deviation of any particular count is merely the inverse of the square root of the number of tracks counted, normalized to percent, since fission-tracks follow Poissonian statistics (Naeser, 1976). Thus if 250 tracks were counted, the standard deviation would be about 6.3%, or 16 tracks. To calculate the uncertainty of an age, take the inverse of the number of tracks counted for both the spontaneous and induced counts, add these together and take the square root. Normalize this number to percent to obtain the standard error of the age.

Zircon Fission-Track Dating

Zircon phenocrysts were obtained from the DeSoto ash as described in the section on petrography. The tracks on individual zircon crystals are counted and dated instead of two separate populations, as for glass (irradiated and nonirradiated). The zircon crystals are embedded in an FEP teflon disk and etched in a eutectic mixture of KOH and NaOH at 210°C. They are then covered with a uranium-poor muscovite detector before irradiation. After irradiation, the spontaneous track density is counted in the zircon crystal and the induced track density is counted in the muscovite detector.

At least six zircon crystals need to be counted for an acceptable age determination (Naeser, 1976). The age of each crystal is determined using equation 5 in the same fashion as for glass dates. The neutron fluence is determined by counting the induced track density in a muscovite detector which overlies a dosimeter during irradiation. Standard Poissonian statistics can be used to analyze the data because the two track densities can be directly related i.e. the covariance can be calculated. Several papers have been published recently on zircon fission-track statistics. The following present several different approaches: Green, 1981; Galbraith, 1981; and Johnson *et al.*, 1979.

RESULTS OF FISSION-TRACK DATING

Flux Determination

The fluences of the nuclear reactors at the U.S.G.S. in Denver and at Georgia Tech were calculated using a variety of different techniques and with four different dosimeters. The Denver irradiation package contained three dosimeters, including Naeser's NBS-SRM612 dosimeter, a 1/2 X 1/4 inch (13 X 6 mm) chip of Corning CN-1 dosimeter and a similar chip of Boellstorff's dosimeter. The Georgia irradiation package contained a total of four chips each of the Corning and Boellstorff dosimeters and two wafers of NBS-SRM962a dosimeter. The results are listed in table 7 and will be refered to below by line number.

The Denver samples were irradiated for 35 minutes in the outer ring of the Lazy Susan at a reactor power of 200 Kw. The total neutron fluence was calculated by Naeser to be $6.96 \times 10^{10} \pm .02$ n/cm^2. The external detector method was used along with NBS-SRM612 dosimeter (which had been calibrated with copper foil). A total of 2292 tracks were counted in the muscovite detector for the determination by Naeser.

The fluence calculated by Naeser was used along with the fission-track density (ρ_o) calculated for the Corning dosimeter irradiated in Denver (sample CN-1-N) in order to calculate a B factor (see Neutron Fluence Determination or Appendix C) of

line #	Standard	sample number	location irradiated	tracks counted	area counted ($X10^{-2}$ cm^2)	track density ($X10^5$ t/cm^2)	fluence ($X10^{14}$ n/cm^2)	way fluence calculated
1	RT-3	RT-3	NBS	1208	1.037	1.165	4.37	Cu foil
2	RT-4	RT-4	NBS	1394	1.382	1.008	3.87	Cu foil
3	NBS-SRM 962a	NBS-G1	Georgia	1429	1.037	1.378	5.17 5.29	ratio with RT-3 ratio with RT-4
4	CN-1	CN-1-G1	Georgia	4028	1.037	3.885	5.75	B factor calculated from CN-1-N
5	CN-1	CN-1-N	Denver	3251	0.6912	4.703	6.96	C. W. Naeser using NBS 612 and Cu foil
6	Boellstorff	BN	Denver	380	5.60	.0660	7.45	Boellstorff's equation
7	Boellstorff	BG1	Georgia	983	11.52	.0853	9.64	"
8	Boellstorff	BG1	Georgia	983	11.52	.0853	9.00	B factor from BN
9	Boellstorff	BG2	Georgia	957	11.52	.0831	9.39	B's eqn, 5 sec etch

Table 7. Results of the neutron fluence determinations for the Georgia and Denver nuclear reactors using a variety of different dosimeters and techniques. See text for an explanation of each of these calculations.

6.76×10^{-10} tracks/n. This B factor was used to determine the neutron fluence of the Georgia Tech reactor using dosimeter CN-1-G1 and its fission-track density. For the dosimeter used by Boellstorff and irradiated in Denver (BN), a B factor of 9.48×10^{-12} tracks/n was determined. This B factor was used together with dosimeter BG1 to determine the fluence of the reactor at Georgia Tech.

For the Georgia reactor, a reactor power of 250 Kw results in a flux of about 1.4×10^{11} n/cm^2/sec (Kirkland, pers. comm.; 1984). To obtain a total neutron fluence of 6.72×10^{14} n/cm^2 an irradiation time of eighty minutes should be used. The actual fluence was calculated using each of the three standards and a variety of methods. These fell into two groups, one above and the other below the requested fluence value.

The NBS sends two pre-irradiated wafers with each order for dosimeters. These two wafers have been irradiated in different reactor positions (RT-3 and RT-4) and fluence levels by NBS. The fluence for the Georgia Tech reactor was obtained by comparing counted fission-track densities for each of these respective dosimeters and NBS-SRM962a (NBS-G1). The resultant fluence determinations are the same within the uncertainty limits and is about 5.2×10^{14} n/cm^2 (line 3). When using the B factor calculated from dosimeter CN-1-N and fission-track counts from dosimeter CN-1-G1 (line 4), a value of 5.75×10^{14} n/cm^2 was obtained. Though similar, these values are statistically different and both are considerably less than the calculated fluence value. As the NBS pre-irradiated dosimeters were calibrated with copper, these other values are also considered to be copper calibrated. The copper calibration values were chosen over the gold values also reported by NBS because copper values are in standard usage. The gold values would yield higher fluence determinations.

The other group of fluence values for the Georgia Tech reactor was obtained using Boellstorff's standards BG1 and BG2. A value of 9.39 X 10^{14} n/cm^2 was calculated for dosimeter BG2 using Equation 1 and the five-second etch for which this equation was intended (line 9). Using dosimeter BG1 and the same equation, but with the thirty second etch used for the other dosimeters, resulted in a fluence value of 9.64 X 10^{14} n/cm^2 (line 7). A higher value is expected since the five second etch is not long enough to develop all the tracks, as explained above. A third determination was made by calculating a B factor from dosimeter BN and Naeser's fluence detemination (which is copper calibrated) and dosimeter BG1 (line 8). Dosimeters BG1 and BN were etched simultaneously, and a value of 9.00 X 10^{14} n/cm^2 was obtained. This value is similar to the values obtained using Equation 1 (lines 7 and 9). Dosimeter BN yielded a fluence of 7.45 X 10^{14} n/cm^2 for the Denver reactor using Equation 1 (line 6), higher than the value of 6.96 X 10^{14} n/cm^2 obtained by Naeser using NBS-SRM612 (copper calibrated) for the Denver reactor.

However, as stated above, this is expected to some degree because BN was etched for longer than proper for use with Equation 1.

Boellstorff (1980) found that the uranium calibrated standards consistently yielded fluence values about 1.5 times greater than fluence values calculated from NBS dosimeters and copper calibration. When his dosimeter is used with the longer etch (not proper for Equation 1), the value obtained is about 1.1 times greater than that determined by Naeser. This would seem to indicate that the higher fluence values obtained using Boellstorff's dosimeters were at least partially a result of uranium calibration. However, when a B factor calculated from a copper-calibrated fluence determination was used to obtain a fluence for the Georgia reactor, a high uranium-like value resulted (line 8). Thus, it is not known why these values are so much higher than those determined using the other dosimeters or the calculated fluence level.

Glass Fission-Track Age

It was determined from petrographic, chemical, and paleomagnetic data that the DeSoto ash is the Lava Creek B Ash from Yellowstone. Therefore, the age determination was more a verification of identification than anything else. The age as determined by Boellstorff's method will be discussed, followed by that obtained using Naeser's method for both glass and zircon ages. Counting data are listed in table 8 and the calculated

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Sample Track Counting Data

	spontaneous tracks			induced tracks*		
sample	#ctd	area ($X10^{-2}cm^2$)	ρ_s ($X10^3$)	#ctd	area ($X10^{-2}cm^2$)	ρ_i ($X10^3$)
Borchers	214	7.20$^+$	2.97	2101	2.880	72.95
GD	50	3.69$^+$	1.36	1467	1.843	79.59
GA	8	0.041	2.0	201	0.021	94.8
035	2	0.006	3.1			
036	2	0.015	1.3			
037	4	0.019	2.1			

* induced plus spontaneous data, spontaneous not yet subtracted

+ areas shown twice that scanned since tracks on both sides of the shards are revealed and counted, thus true glass areas recorded.

Table 8. Both the Borchers ash and GD samples of the DeSoto ash were irradiated in Georgia and counted following Boellstorff's methodology. GA is the sample of DeSoto ash irradiated in Denver and counted following Naeser's methodology. The numbered subsets shown for the spontaneous tracks are the data derived from each of the three individual polished slides counted. The totals are recorded in the full GA line. (# ctd = number of fission-tracks counted.)

ages are summarized in table 9.

As stated earlier, the Borchers ash was dated along with the DeSoto ash in order to determine the "goodness" of the fluence determination. One hundred areas of 1.44 X 10^{-4} cm^2 each were counted on the irradiated shards and 250 areas were counted on the non-irradiated shards. The fluence used was the average of the two uranium-calibrated determinations, or 9.52 X 10^{14} n/cm^2. This value gave an age of 2.49 my for the $\sim$2 my old Borchers ash. Obviously, this value is too high. Using the fluence obtained by averaging the lower copper calibrated NBS and Corning determinations (5.40 X 10^{14} n/cm^2 (lines 3-4, table 7)) gave an age of 1.42 my. This is too young, though Naeser states that all labs except Boellstorff's get glass fission-track dates of between 1.3 and 1.5 m.y. for the Borchers ash. (pers. comm., 1986).

As the author did not need a particularly precise age in order to verify the identity of the DeSoto ash as the Lava Creek B Ash, the fluence values given above were averaged to obtain a value of 7.5 X 10^{14} n/cm^2. Though this is not an acceptable technique, this fluence value gave a date of 1.96 my! When the high uranium-calibrated fluence (average of lines 7 and 9, table 7) and the value for λ_F (8.46 X 10^{-17} /yr) suggested by Thiel and Herr (1976) for uranium-calibrated dosimetry are used, an acceptable age of 2.11 my is obtained.

In order to calculate an age for the DeSoto ash, 100 areas

Fission-track ages of the DeSoto ash

sample name	ρ_s ($X10^3 t/cm^2$)	ρ_i ($X10^3 t/cm^2$)	fluence ($X10^{14} n/cm^2$)	age (my)		method
Borchers	2.972	69.98	9.52	2.49	.7	B's fluence
	"	"	5.40	1.42	.4	N's fluence
	"	"	7.5	1.96	.6	average
	"	"	9.52	2.11	.6	=
GD	1.36	78.2	9.52	1.02	.3	B's fluence
	"	"	5.40	0.58	.2	N's fluence
	"	"	7.5	0.80	.2	average
	"	"	9.52	0.82	.2	=
GA	2.0	93	6.96	0.9	.6	fluence
035	3.1	92	"	1.4	.8	obtained
036	1.3	93	"	0.6	.8	by
037	2.1	93	"	1.0	.9	Naeser

Table 9. The Borchers ash is the intralaboratory standard used by Boellstorff and proposed by him as an interlaboratory standard (see text). His accepted age for this ash is 1.96 my. GD is the sample of the DeSoto ash dated following Boellstorff's methodology. Each of these was dated using several choices for the fluence determination. These are: Boellstorff's microscope slide and Equation 1; the value derived by averaging the NBS and Corning standards; using Naeser's fluence determination techniques; the value derived by averaging the above two numbers; and a calculation using Boellstorff's fluence, but the decay rate calculated using uranium calibrated standards. GA is the sample of DeSoto ash dated following Naeser's methodology. The numbered subsets are the data determined for each individual slide counted, the numbers listed beside "GA" are the totals of these three slides. See the text for a further explanation of these different methodologies.

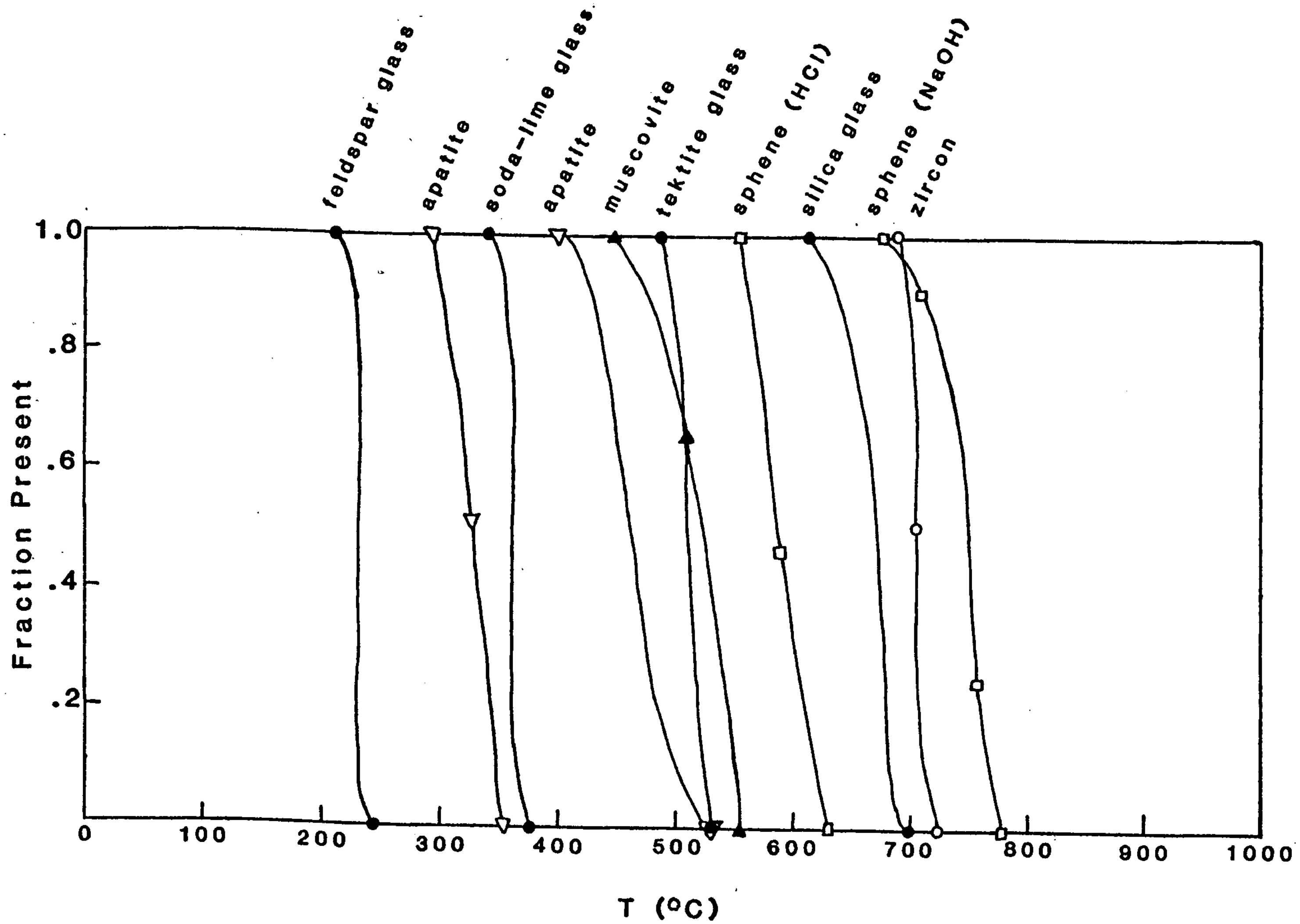
of 9.22 X 10^{-5} cm^2 were counted on the irradiated shards and 200 similar areas were counted on the non-irradiated shards. Using the average 7.5 x 10^{14} n/cm^2 fluence value gave a date of 1.02 ± .15 my. (As only one age determination was made, uncertainties are given using the standard 15% uncertainty ascribed to fluence determinations (Fleischer and Price, 1964; and Boellstorff, 1973).) Even using just physical characteristics, chemistry, and magnetic polarity for correlation, the DeSoto ash can only be the Lava Creek B Ash. This date does not allow for this identification. When recalculated using the fluence determined from copper calibrated dosimeters (5.40 n/cm^2), an age of 0.58 ± .09 my was obtained. This is quite close to the accepted age of 0.62 my for the Lava Creek B Ash. A third recalculation, using the higher value for the fluence and the Thiel and Herr λ_f value yields an age of 0.82 ± .12 my. Though still high, this date is ~23% lower than the age obtained using the American standard value of 6.85 X 10^{-17} /yr for λ_f and the same fluence value (1.02 ± .15 m.y.).

This 23% difference seems particularly significant. Thiel and Herr (1976) stated a 20% difference in experimental parameters (age, dosimetry, etc.) would result if the 6.85 X 10^{-17}/yr value for decay rate were used instead of 8.46 X 10^{-17}/yr. Naeser _et al_. (1973) stated that Boellstorff's ages tend to be 20 to 25% too high in comparison to K-Ar ages and zircon fission-track ages. Similar results have been obtained by other workers

(Seward, 1979). This value of about 20% was again cited by Naeser (pers. comm.; 1985) as the extent to which most Cenozoic ash is annealed.

Feldspar glass (rhyolitic) anneals at a lower temperature in annealing experiments (see figure 23) than other materials which are regularly dated, especially zircon. Hydration of glass increases the ease with which annealing occurs (Lakatos and Miller, 1972). Naeser _et al_. (1980) found that over 90% of the volcanic ash samples they dated had glass fission-track ages younger than those of coexisting zircon phenocrysts. The results of this experiment are shown in figure 24 and most of these glass dates are about 20% lower than the zircon dates. When Boellstorff performed a similar experiment, his glass dates were nearly the same as the zircon dates (see figure 25). Seward (1980) and Boellstorff and Alexander (1980) both attribute this to Boellstorff's use of a different dosimeter (uranium calibrated). Seward, however, challenges Boellstorff's ability to directly compare ages obtained using different dosimeters, stating that Boellstorff's higher fluence was "spuriously correcting" his glass dates (1980). This, and the following discussion on age correction is presented only to show that disagreements exist on proper procedure for fission-track dating among experts in the field. Some of these issues are discussed further in Appendix C. A thorough consideration of annealing and fluence determination is beyond the scope of this work.

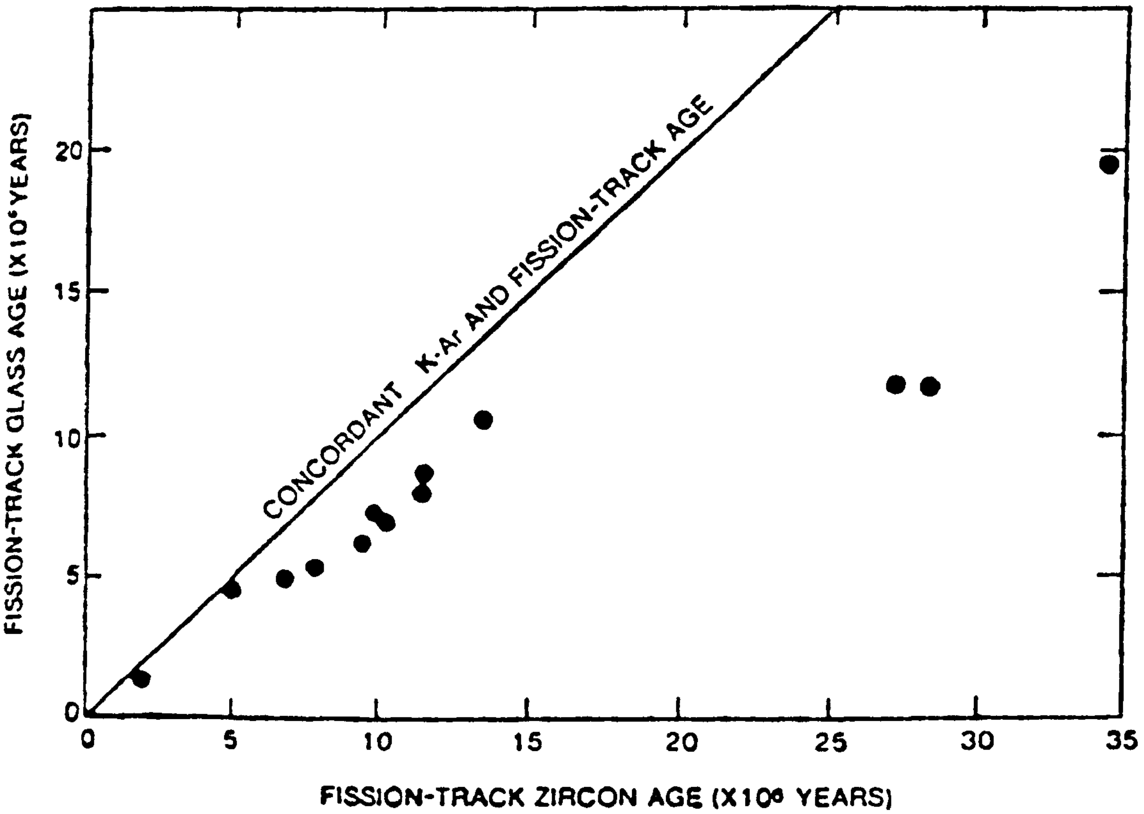
Figure 23. Annealing temperatures for materials commonly dated by the fission-track method. Most volcanic ash shards, including those of the DeSoto ash, are feldspar glasses. Microscope slide glass is soda-lime glass, while tektites are meteoritic glass. Note that the etchant used can affect annealing seen. Feldspar glass anneals at a lower temperature than any other material which is commonly dated. Hydration lowers this temperature further. Apatite is the only mineral for which data have been gathered by two independent labs, the reason for its double listing.



after Naeser, 1976

Track Fading After 1 Hour Anneal

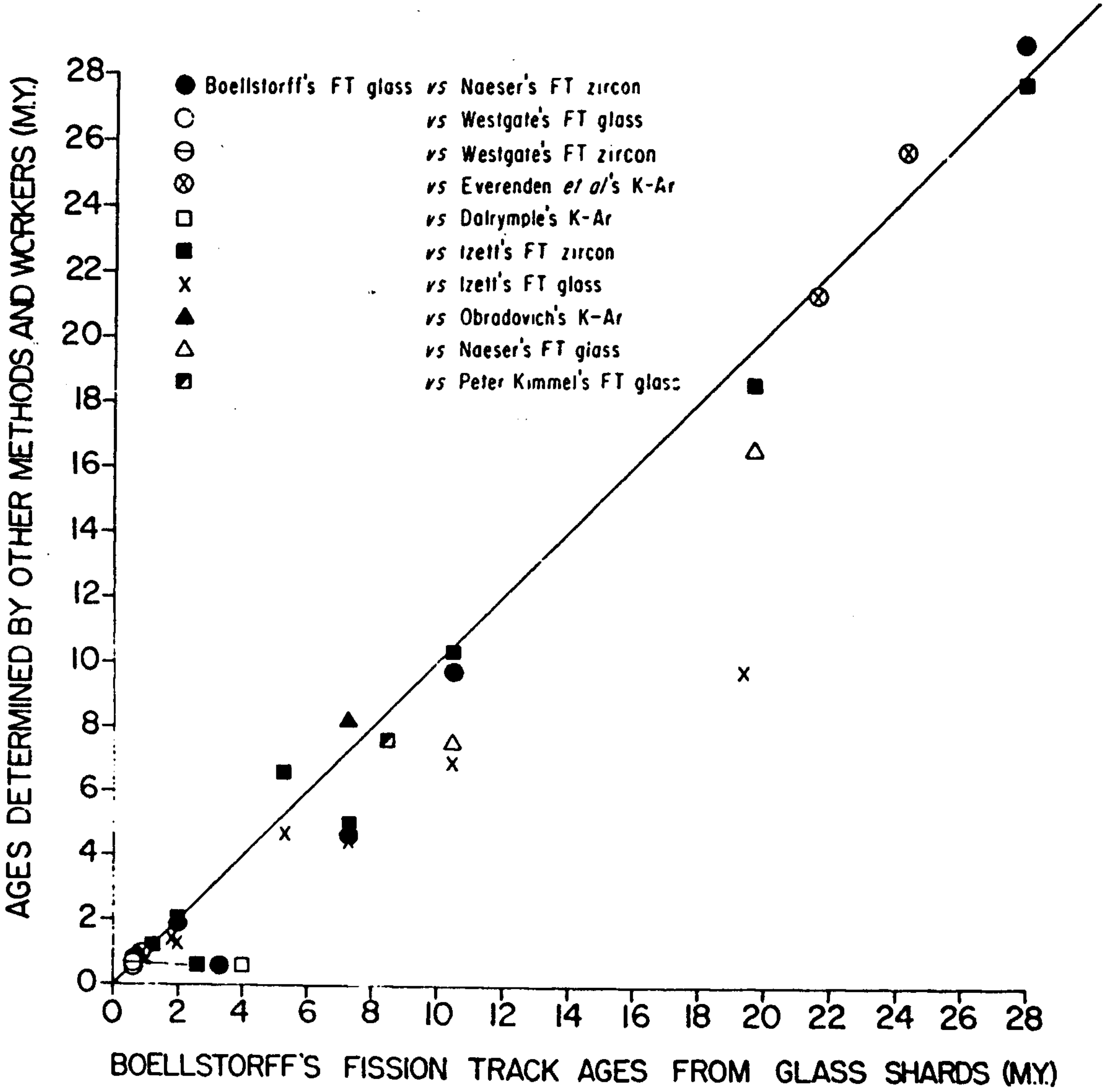
Figure 24. Comparison of glass and zircon fission-track ages as per Naeser. The fission-track dates obtained by Drs. Charles Naeser and Glen Izett on hydrated glass shards are generally about 20% lower than dates obtained on co-occurring zircon phenocrysts.



From Naeser et al., 1980

Figure 25. Comparison of glass fission-track ages and ages determined by other methods as per Boellstorff. Boellstorff obtains ages for glass shards which are comparable to those obtained by other workers using a variety of methods. Note that the older glass dates obtained by Izett and Naeser, respectively, fall below the equal age line, as in figure 24.

(Figure obtained from J. D. Boellstorff, unpublished.)


● Boellstorff's FT glass vs Naeser's FT zircon
○ vs Westgate's FT glass
⊖ vs Westgate's FT zircon
⊗ vs Everenden et al's K-Ar
□ vs Dalrymple's K-Ar
■ vs Izett's FT zircon
x vs Izett's FT glass
▲ vs Obradovich's K-Ar
△ vs Naeser's FT glass
▨ vs Peter Kimmel's FT glass
AGES DETERMINED BY OTHER METHODS AND WORKERS (M.Y.)
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
BOELLSTORFF'S FISSION TRACK AGES FROM GLASS SHARDS (M.Y.)

In 1969, Storzer and Wagner developed a method by which to correct annealed fission-track ages. They derived a curve which correlated the ratio of the diameter of laboratory annealed etched tracks to the diameter of the original etched tracks and track density. Thus the proper, unannealed track density can be determined. In practice, the diameters of the spontaneous and induced tracks are compared (Seward, 1979). However, Boellstorff (1975) has found that spontaneous and induced track diameters for Cenozoic ashes in general seem to vary by about 15%. His feeling (1984, pers. comm.) is that the difference in activation energy for the formation of spontaneous and induced tracks, respectively, may be the cause for up to 15% of this size difference. Boellstorff further states that the correction Storzer and Wagner would ascribe to this 15% difference would result in his ages being 20% higher than accepted dates. Naeser, (1986, pers. comm.) responds that if activation energy were the cause, one should not be able to find examples where the sizes of spontaneous and induced tracks are the same, and he does. It is obvious that the entire field of dosimetry and glass fission-track dating needs to be carefully analyzed and reappraised as to accuracy and correct procedure.

The DeSoto ash is so fine grained that very little glass was countable on the polished slides made for Naeser's procedure. A total of 229 grids (1 mm^2) was counted on the irradiated glass slide. An average of only 1.61% of this area contained polished glass exposed at the surface. A total of 201 tracks was counted on this irradiated slide. On the three non-irradiated slides, 688 grids yielded only eight spontaneous tracks. Though the data base is quite small, the date obtained (0.90 ± .32 my) is adequate to include the accepted age for the Lava Creek B Ash. The uncertainty was calculated using Poissonian statistics as discussed in the section "Naeser's method - Age calculation" above. The fluence used in the calculations was that obtained by Naeser from his NBS-SRM612 dosimeter, which is copper calibrated.

Zircon Fission-Track Age

It was decided to try and date zircons from the ash because glass anneals readily. Unfortunately, few zircons were actually obtained from the ash for reasons detailed in Appendix D. Of these few zircons, about half fell out of the FEP teflon disk during etching. When cleaving the muscovite detector, a perfectly smooth cleavage flake was never obtained. Naeser and the author decided to give it a try anyway.

No date was ultimately derived for several reasons. A lack of good zircons was primary, however. Dating of the one or two useable zircons (those with no blemishes such as crystallites, and for which a good image existed in the muscovite detector) was not attempted because of problems with counts in the Fish Canyon zircon standard. The date obtained for the standard of 49.4 my is about twice the proper age of 27.2 my. Though the detector image was not of high quality, it is not known why the age was so far off. For these reasons, the zircons were not dated.

CONCLUSIONS

The fission-track dating methods of Boellstorff and Naeser vary considerably in detail. These differences can be relegated to two main areas, flux determination and sample preparation. Many of the factors involved in choosing which particular procedures to follow have been stated earlier in the text or can be found in Appendices B and C. This section is concerned primarily with summarizing some of these factors and pointing out areas where more work is needed.

There are many problems relating to reactor dosimetry. The choice of which dosimeter to use is intertwined with considerations such as which decay rate to use and what facilities are available to the worker. The use of a standard equation (as opposed to comparing track densities) to calculate reactor fluence is probably not accurate. There are too many variables involving differences in etching and counting criteria for one equation to work reliably for different workers. Thus, a dosimeter, for which a duplicate irradiated under known conditions is available, must usually be chosen. In the United States, this generally means the National Bureau of Standards dosimeters at present. The details of dosimetry and the different values obtained using various calibration techniques needs to be worked on. Boellstorff (1980) suggested the creation of a uranium-calibrated standard having an irradiated/nonirradiated pair in addition to the creation of some agreed on interlaboratory dating standard. Naeser, (1986, pers. comm.) stated that the ζ factor (see Appendix B), which eliminates separate determinations of neutron fluence and fission decay rate, will probably become standard in fission-track dating in the future.

The choice of sample preparation before counting is strongly related to the size, shard shape, and age of the ash to be dated. It is easier to see the tracks on polished glass surfaces, and to control the etching conditions of slides than of bulk ash. However, unless the ash has plenty of large, flat shards, very little glass will actually be countable using the polished slide method. For young ashes, the worker must be prepared to either spend a very long time counting, or base the dates on only a few tracks. It takes less time to count more tracks using the bulk etch technique. Though track identification can be more difficult, this technique can yield general dates with far less time and effort than it takes to polish and count the large number of slides required for precise dates. This is particularly true if an interlaboratory standard is used. It is reasonable to assume that if the track identification criteria and etching techniques of the worker were adequate to obtain a correct date for the standard, the date on the unknown is probably also correct.

The author feels that bypassing problems of fluence determination and fission decay rate by use of a ζ factor is a good procedure. This should be augmented by the development of an interlaboratory glass fission-track age standard so that the ages obtained by different workers can be compared. Which counting technique to use depends to some extent on the goals of the research and the size and morphology of the ash to be dated. If highly precise dates are necessary, use of polished slides will probably yield more accurate results. However, as stated above, to obtain general ages of young and/or fine grained ashes, the bulk etch technique should result in adequate dates with far less effort expended. Perhaps the bulk etch technique should be routinely used to check the feasibility of dating a particular ash sample by either technique.

APPENDIX B

Theory of Fission-Track Dating

Review of nuclear physics

The basis of any isotopic dating technique lies in the inherent instability of a naturally occurring element and the predictable rate at which an unstable element will break down into a stable element. Nuclear binding energy within the nucleus of an element is maximized when the number of protons (atomic number = Z) is approximately equal to the number of neutrons (neutron number = N); thus, these elements are most stable. As the mass number (A = Z+N) increases, the ratio of N to Z actually increases substantially from 1:1. This results from the repulsive forces between protons becoming stronger than the nuclear binding forces as more protons are added to the nucleus (see figure 26).

Atoms which have the same number of protons (and thus are the same element), but different numbers of neutrons are called isotopes. There are relatively few naturally occurring radioactive isotopes. Though all elements with more than 83 protons are radioactive, all those which are naturally occurring are the result of the decay of ^{238}U, ^{235}U, or ^{232}Th. These three long-lived radioactive isotopes occur because "they have not yet completely decayed since the time of synthesis of the

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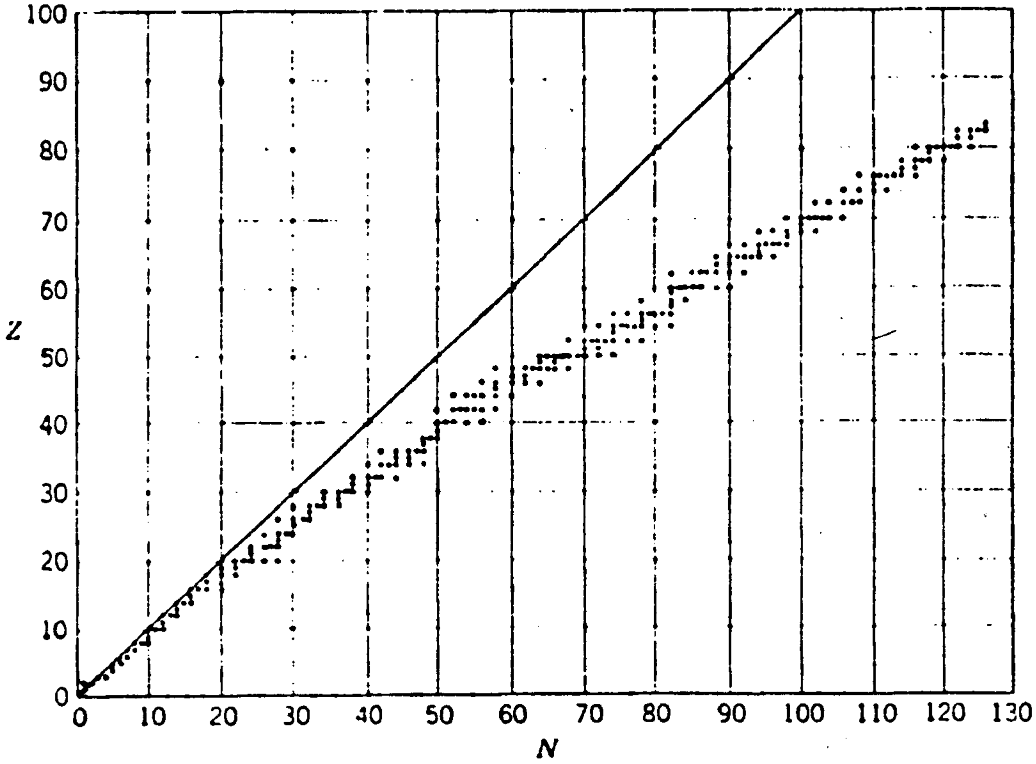
Figure 26. Plot of atomic number versus neutron number. When the stable nuclei are plotted with atomic number (Z) versus neutron number (N), there is an increase in the number of neutrons relative to the number of protons.



From Friedlander et al., 1981

elements..." (Faure, 1977). Others (e.g. ^{14}C, ^{10}Be, ^{32}Si, etc.) are continuously being created by various natural nuclear reactions. Recently the introduction of man-made nuclear fallout has increased the number of radioactive isotopes present in the environment.

Radioactive decay

There are five modes by which isotopes decay. These are named by the primary type of nuclear particle which is transferred to or from the nucleus during the decay process.

The first of these, alpha decay, occurs primarily in atoms having a Z of at least 58. An alpha particle consisting of two protons and two neutrons (a He nucleus) is emitted from the nucleus of the parent. Thus Z for the daughter isotope will decrease by two and N will also decrease by two. If the daughter is left in an excited state, the excess energy will be emitted as gamma rays.

There are two types of beta decay, negatron and positron decay. In negatron decay, an electron (negatron) is expelled as a neutron is transformed into a proton. This type of decay causes Z to increase by one and N to decrease by one. In positron decay, a proton is transformed into a neutron, a positron (positive analog of an electron), and a neutrino. Therefore, Z will decrease by one and N increase by one. The mass number remains unchanged in either case. Gamma rays are emitted in both types of beta decay if the daughter isotope is left in an excited state.

In electron capture, a fourth decay mode, the nucleus captures an extranuclear (shell) electron which results in a decrease of Z by one as a proton is transformed into a neutron. Excess energy will be emitted in the form of a neutrino. If the daughter is left in an excited state, gamma rays will be emitted. X-rays are emitted as the shell electrons refill vacancies left by the capture process.

The fifth type of radioactive decay is spontaneous fission. In atoms with a mass number of greater than approximately 100, electrostatic repulsion is greater than the forces holding the nucleus together (Faure, 1977). The nucleus may then spontaneously divide into two highly charged daughter nuclei which are unequal in size (Friedlander et al., 1981). In addition to the two nuclear particles, alpha particles, neutrons, various light fragments, and a large quantity of energy are released. Fission can be naturally induced if enough particles strike susceptible ^{235}U or ^{232}Th nuclei.

History of fission-track dating

Nuclear fission was discovered by Hahn and Strassman in 1938 (Faure, 1977). As two charged nuclei resulting from fission travel through a substance, they create a damage zone or fission-track. In 1959, Silk and Barnes took the first direct photographs of fission-tracks (Fleischer et al., 1975). However, the process of viewing these tracks with an electron microscope caused the tracks to anneal and thus disappear. Therefore, it was not until Fleischer, Price and Walker discovered that the tracks could be etched, and hence made larger and more permanent, that fission-track dating became feasible (Fleischer et al., 1975). During 1964 the first fission-track ages were published. Fission-tracks have since been used for dating a variety of minerals and glasses covering a wide range of geologic time. Thus, fission-track dating has a wide applicability in geochronology and archaeology.

In the discussion which follows, the processes of track formation and etching will be discussed first. Following will be a discussion of the age equation and several of the problems associated with its use.

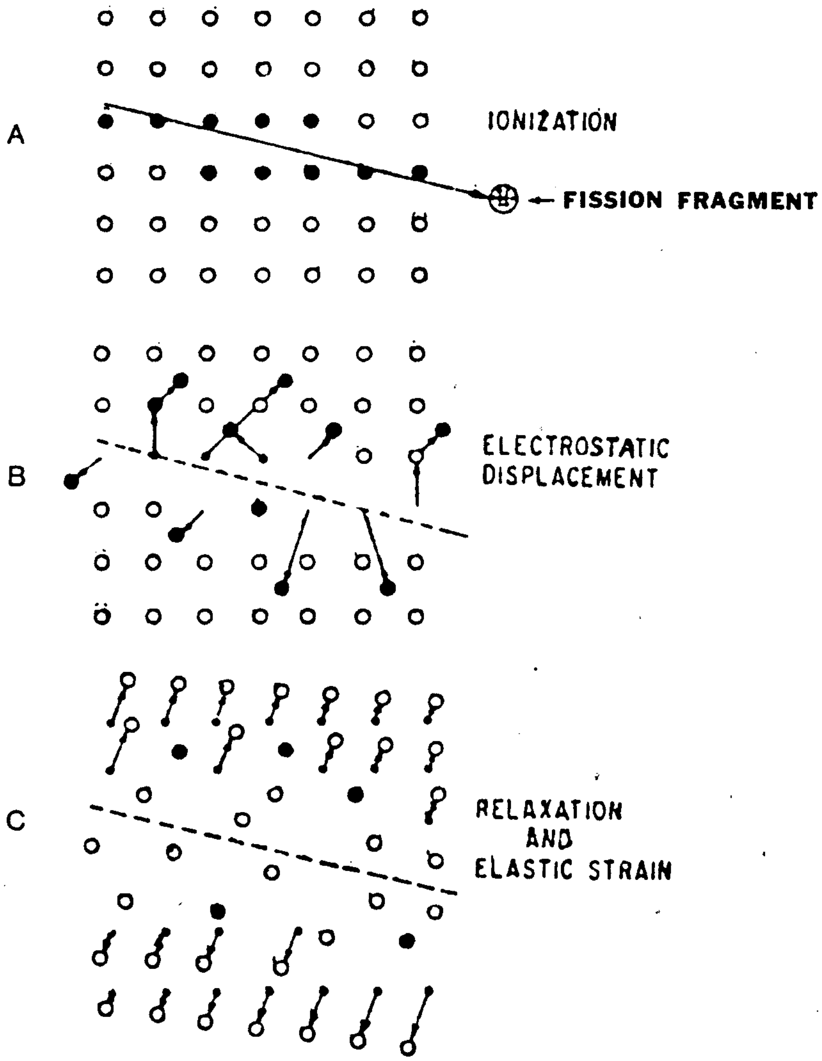
Basis of fission-track dating

Fission-track dating involves the counting of tracks created by the spontaneous fission of ^{238}U within a substance. As this is the only naturally occurring isotope which is known to form spontaneous fission-tracks in significant quantities, it is necessary only to measure the amount of ^{238}U present and the fission-track density to obtain an age once the rate at which these tracks accumulate is known (about 10^{-16} per year (Fleischer *et al*., 1975)). This age represents the time elapsed since the material became capable of retaining tracks, i.e. since it cooled from an originally higher temperature. Examples of materials which can be dated include volcanic glass and phenocrysts (zircon and apatite), metamorphic events (apatite, zircon, and sphene), heat treatment of lithic artifacts (obsidian), and firing dates of some types of pottery (glaze and zircons within the temper).

Fission-tracks are formed by the interaction of the nuclei resulting from fission with the constituent atoms of the host material. When fission occurs, the two fission fragments travel away from each other in opposite directions (see figure 27). These nuclei are stripped of most of their electrons and as they move through the host they pick up electrons from other atoms (Fleischer *et al*., 1975) and are thus ionized. The passage of these highly ionized particles causes electrostatic imbalances within the host lattice. Electrons from atoms along the path of the fission fragments are loosened and ejected, creating a zone of positive charge. These ionized atoms then move apart by electrostatic repulsion, forming a track. The track area is very porous and may even be a fine channel (Price and Walker, 1962).

Because of their small size, fission-tracks can be seen in their natural state only with the magnifications available on an electron microscope. In the process of viewing them, however, the tracks anneal, and thus disappear (electrons from the

Figure 27. Formation of fission-tracks. A) Creation of ionized zone within a lattice by the passage of an ionized fission fragment. B) Disruption of the lattice caused by mutual repulsion of the ionized atoms (solid circles) within the lattice. C) Final form of a fission-track. The area containing a low density of atoms will etch faster and thus form countable tracks in a material upon magnification of at least 500X.



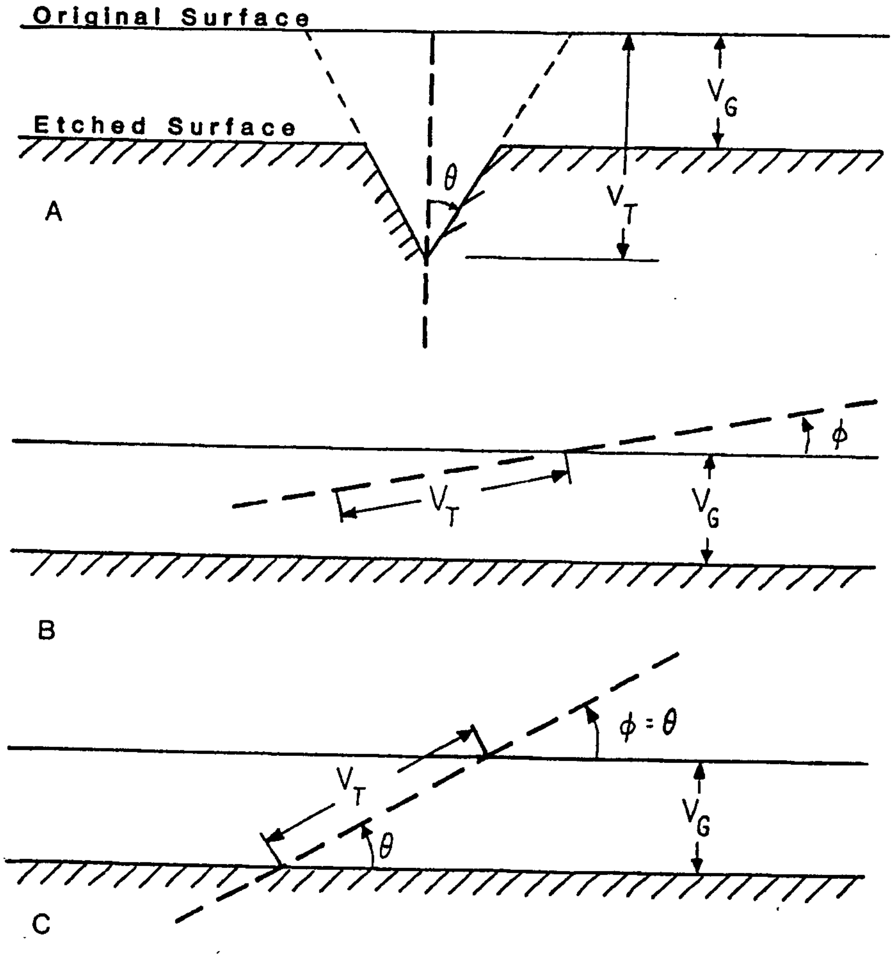
Adapted from Fleischer et al., 1975

microscope beam are furnished to the affected atoms along the track, allowing them to move back to their previous locations). In order to prepare a material for fission-track dating, the tracks must be made larger and more permanent. This is done by etching. The strained area of the material will dissolve faster in an etchant than will the undamaged portion. The original channel width is typically on the order of 25 angstroms, though the elastic-strain field (see figure 27) increases this width to about 100 angstroms; channel length is usually 20 to 30 microns (Price and Walker, 1962).

Whether a track is revealed by etching is dependent upon its geometry (see figure 28). If the angle at which the track intersects the surface of the material is too shallow, the track will be erased instead of enhanced as etching proceeds. Therefore, a fission-track must intersect the surface of the material at an angle greater than a specific critical angle in order for preferential etching of the track to take place. The measure of this angle depends upon the characteristics of the material and the etchant used, the energy of the fission fragment, and the thermal history of the material. After etching, tracks can be viewed through an optical microscope at a magnification of $\geq$ 500X.

As mentioned above, the thermal history of a material has an effect upon whether or not a track will be revealed by etching.

Figure 28. Fission-track etching geometry. In order for a track to be revealed, the inclination of the track (θ) must be greater than half the angle formed by a vertical etch pit, ie the critical angle (ϕ). Otherwise, the etch rate in the undamaged glass (V_G) will be sufficient to remove the track. The increased rate of etching along a track (V_T) is what permits any tracks to be revealed. A) Vertical etch pit. B) Fission-track to shallow to be revealed. C) Fission-track at the critical angle.



Adapted from Fleischer and Hart, 1972

Generally, as temperature rises, atoms move more quickly within a lattice. When atoms move back into lattice positions which were vacated as a fission product passed, the track closes at that point. This prevents the etchant from proceeding farther down the track at the increased rate of dissolution. If this happens often enough, the track will become annealed and no preferential etching can take place.

Determination of an age

Determining a fission-track age requires the counting of two sets of tracks and the determination of the neutron dose. The first of these, the spontaneous track density (ρ_s), is the number of tracks created by spontaneous fission of ^{238}U. It is expressed as follows:

$$\rho_s = (A\lambda)\ N_v\, c^{238} R^{238} \eta^{238} \qquad \text{eqn 6}$$

where A is the time over which spontaneous fission tracks have been stored, λ is the decay rate by fission of ^{238}U, N_v is the number of atoms per unit volume in the material, c^{238} is the fraction of those atoms which are ^{238}U, R^{238} is the length of the etchable track of a ^{238}U fission fragment, and η^{238} is the etching efficiency. The equation can be broken into two parts, where $A\lambda$ represents the fraction of ^{238}U atoms which fission in time A, and $N_v\ c^{238}\ R^{238}\eta^{238}$ represents the number of ^{238}U atoms per unit area that are within range R of the etched surface and can lead to etched tracks at that surface. (Fleischer et al., 1975) Possible reasons why a track might not be revealed by etching have been discussed above.

Variations in the ratio of ^{235}U to ^{238}U have been found in nature only in association with uranium ore (Fleischer et al., 1975). Therefore, the most straightforward method for obtaining the ^{238}U concentration in a material is to irradiate the sample with thermal neutrons in a nuclear reactor in order to induce fissioning of ^{235}U within the sample and thereby determine its concentration. The induced track density (ρ_i) is the second track count which is required for an age determination. It can be expressed by an equation analogous to that for ρ_s:

$$\rho_i = (\sigma \Phi) N_v c^{235} R^{235} \eta^{235} \qquad \text{eqn 7}$$

where is the cross section for fission of ^{235}U and Φ is the neutron fluence in the reactor. Neutron flux is the number of neutrons which pass through a 1 cm^2 area per second, while neutron fluence is the integrated flux over time in neutrons per square centimeter. The first term ($\sigma \Phi$) represents the fraction of ^{235}U which has fissioned; and as in Equation 1, N_v c^{235} R^{235} η^{235} represents the number of ^{235}U tracks which can be counted.

The cross section (in barns) of a nuclide represents the probability of a nuclear process, such as fission, occurring. Since the probability of a reaction taking place is directly proportional to the cross-sectional area of the target nucleus, cross section has the dimensions of area (1 barn = 10^{-24} cm^2). Even if a particle passes outside the area of the nucleus proper, it may still cause a reaction to occur. For this reason, cross section may be thought of as "... the apparent cross-sectional area of the bombarded particle as seen by the incident particle ...", the size of which depends on both "... the properties of the matter through which the radiation passes ... and on ... the properties of the radiation itself" (Chase and Rabinowitz, 1962, p. 115).

An induced fission-track is created when a thermal (slow) neutron impinges on the nucleus of a ^{235}U atom. The number of tracks created is dependent upon the neutron fluence and the ^{235}U concentration. A measure of the fluence can be obtained by determining the track density in a piece of dosimeter glass (ϱ_D) of known uranium concentration. The dosimeter track density can be expressed as:

$$\varrho_D = B\,\Phi \qquad \text{eqn 8}$$

where $$B = \sigma N_v\, c^{235} R^{235}\, \eta^{235}. \qquad \text{eqn 9}$$

The terms in Equation 9 are analogous to those in Equation 7, only for the dosimeter glass as opposed to the sample.

Equations 6 through 8 can be combined to yield:

$$A = \zeta \left(\frac{\rho_s}{\rho_i}\right) \rho_D$$ eqn 10

where $$\zeta = \frac{\sigma C^{235} R^{235} \eta^{235}}{B\lambda C^{238} R^{235} \eta^{238}}$$ eqn 11

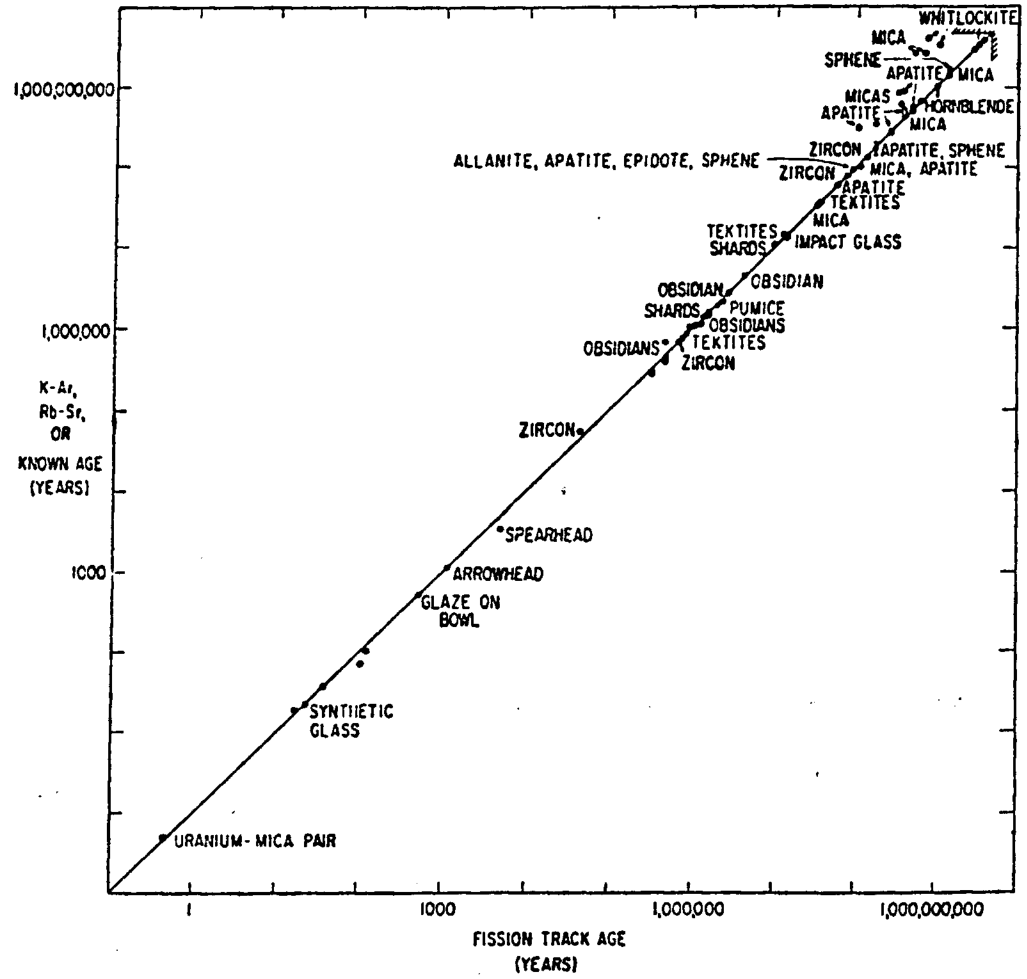
Of the terms in Equation 11, and are nuclear constants, and clearly do not vary. The isotopic ratio $\frac{C^{235}}{C^{238}}$ has not been found to vary in nature except as stated above. R and η are very nearly equal for the two isotopes of uranium and their ratio is constant. For each type of dosimeter glass, the terms in B are constant. Thus, once determined, ζ can be considered a constant.

Assumptions in the use of the age equation

The age equation as presented in Equation 10 is linear with time and uranium concentration, but should be used only for ages of less than 10^9 years. For ages greater than this, the variation in quantity of ^{238}U caused by alpha decay becomes significant and the equation must be slightly altered. The applicability of the age equation over this broad time range is shown in figure 29 (Fleischer et al., 1975).

There are generally three assumptions made in using the age equation. First is that all the spontaneous tracks are caused by fission of ^{238}U and all the induced tracks are caused by fission of ^{235}U. Second is that the same etching and counting techniques were used in obtaining ρ_s and ρ_i so that their ratio (ρ_s/ρ_i) will be

Figure 29. Age range for fission-track dating. Fission-track dating has been shown to yield correct dates for materials ranging from 1/2 year to over a billion years old. The range for a specific material depends on its uranium content, less uranium is required for the older ages. Points plotting significantly above the line have been annealed by thermal events.



From Fleischer et al., 1975

accurate. Lastly is the assumption that γ is a constant proportionality factor.

Though the values in γ were discussed above, two require additional comment. The decay rate of ^{238}U by fission has been measured repeatedly and a variety of values from 5.3×10^{-17}/yr to 12×10^{-17}/yr has been determined in various laboratories and at various times (Fleischer et al., 1975). There are two values which are now frequently used in fission-track age determinations, 6.85×10^{-17}/yr and 8.46×10^{-17}/yr. In the United States the value 6.85×10^{-17} is more frequently applied because it "has yielded geologically meaningful ages for geochronologists using the NBS standards" (Seward, 1979). It was derived by obtaining the weighted average of a determination made using synthetic mica detectors and uranium foil and a determination which required the K/Ar and Rb/Sr ages of a suite of minerals to agree with fission-track ages determined for them using mica detectors (Roberts et al., 1968). The second decay rate value was determined by fission-track dating of man-made uranium glasses (Thiel and Herr, 1976) and is used mostly in Europe.

Thiel and Herr (1976) claim that use of the lower decay rate will cause a 20% systematic deviation in experimental parameters. As the age of the glass dated by them was known with far more precision than this, use of the lower decay rate would have yielded erroneous results. They state that uranium foils can interfere with reactor neutrons and possibly result in a low decay rate being obtained. At this time, this problem is unresolved.

There is also a slight variation in the cited values for the cross section for fission of ^{235}U (σ). A value of 580 X 10^{-24} cm^2 was given in a 1981 nuclear chemistry text (Friedlander _et al_., 1981). Naeser (1976) uses a value of 582 X 10^{-24} cm^2 in his fission-track determinations. Most recently, General Electric (Walker _et al_., 1984) published a value of 583 X 10^{-24} cm^2. Even so, this variation in the value of σ causes a less than 0.5% difference in the age obtained as used in the age equation.

Spontaneous fission involves tunneling through an energy barrier called a Coulomb barrier (Friedlander _et al_., 1981). The attractive nuclear forces of the nucleus must be overcome before a particle can escape. These forces create an impediment to fission. Induced fission occurs whenever enough energy is supplied to a susceptible nucleus to overcome this barrier. Some nuclides, especially those with an odd number of neutrons (such as ^{235}U) have a large cross section for fission by thermal neutrons. The remaining susceptible nuclides (such as ^{238}U) can be induced to undergo fission with fast neutrons. Figure 32 shows a comparison of the energy needed to fission ^{235}U and ^{238}U. Of those nuclides which can be fissioned with the relatively slow thermal neutrons, only ^{235}U is naturally occurring in concentrations great enough to yield a significant number of tracks (Walker, 1984). Thus it is reasonable to assume that all the induced tracks counted are the result of fission of ^{235}U.

However, there is a discrepancy in how thermal neutrons are defined for different purposes. A thermal neutron, in general, is defined as having an energy of between approximately 2 X 10^{-8} MeV and 5 X 10^{-7} MeV (Fleischer et al., 1975). As used in the fission-track age equation, a thermal neutron is assumed to have an energy of 2.53 X 10^{-8} MeV (Boellstorff, pers. comm., 1986). This is also the value used in figure 30 for determining the fission cross section of ^{235}U.

Thermal neutrons are produced in a nuclear reactor by slowing the neutrons formed during fission by passing them through a moderator, commonly water or graphite (Friedlander et al., 1981). The proportion of thermal to fast neutrons resulting is commonly reported as a cadmium ratio e.g. the ratio of the neutron fluence seen by a detector shielded by cadmium to the total neutron fluence. Unfortunately, this definition for a thermal neutron includes all those with an energy level of $<$ 5 X 10^{-7} MeV.

This definition introduces two complications. First, the thermal cross section of ^{235}U varies with neutron energy (as shown in figure 30). Thus it is no longer constant, as assumed in the age equation. Also, ^{232}Th can be induced to fission with energy as low as 4 X 10^{-7} MeV (Boellstorff, pers. comm., 1986).

Figure 30. Comparison of uranium isotope cross-sections. A) The ^{235}U isotope can be induced to fission at low neutron energy levels. Cross-section values normalized to 582 barns at 0.0253 eV. B) ^{238}U fissions spontaneously in nature. To induce fissioning, large neutron energy levels are necessary (note scale change).

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Thus when thermal neutrons are defined as sub-cadmium neutrons, instead of as neutrons with an energy of 2.53 X 10^{-8} MeV, some ^{232}Th fission-tracks may be formed. This fact also further complicates the use of dosimeters which contain thorium to measure neutron fluence (see table 10 in Appendix C).

There are four types of natural tracks which could potentially be found in terrestrial samples: those from the spontaneous fission of ^{238}U; those from the spontaneous fission of ^{244}Pu; tracks produced by recoil of heavy nuclei after alpha decay of ^{232}Th, ^{235}U, or ^{238}U; and "tracks produced by the scattering of alpha particles from the normal constituents of silicate minerals" (Fleischer _et al_., 1975). This last type is not usually discussed in the literature and Fleischer _et al_. (1975, p. 161) dismiss them with the statement that they "have not proved useful". Presumably there is little chance of confusion between one of these tracks and a fission-track. The half life for ^{244}Pu is short in comparison to the time since its synthesis and it is thus considered an extinct isotope. Tracks caused by fission of ^{244}Pu have not been found in terrestrial samples (Fleischer _et al_., 1975). The tracks produced by recoil of heavy nuclei after alpha decay have not been observed with an SEM in any material other than mica (Fleischer _et al_., (1975). Naeser (1986, pers. comm.) stated that "Alpha recoil tracks are very short ... and the ... pits are less than 0.5 microns deep. Therefore, they ... cannot easily be mistaken for fission-tracks". Thus, none of these track sources should contribute to errors in the ρ_s/ρ_i ratio.

Fleischer *et al*. (1975) listed a variety of other possible mechanisms by which tracks can be formed which were considered to be unimportant. These include: the spontaneous fission of naturally occurring elements other than those discussed above; fission induced by α , β or γ activity; induced fission of heavy nuclides by fast or thermalized neutrons produced in other reactions; fission induced by cosmic ray primaries or secondaries (e.g. neutrons formed by collision of the primary cosmic ray with nuclei of the material); and spallation (e.g. loss of parts of the nucleus) caused by cosmic ray secondaries. As the earth is shielded from cosmic rays by its atmosphere, all cosmic ray track sources are of negligible importance for most terrestrial samples. Detectable induced track densities have been found in samples low in uranium, but rich in other very heavy elements, however. In many studies of tracks, Fleischer *et al*. (1975) have not found a significant density of natural fission tracks which might have been caused by anything other than spontaneous fission of ^{238}U, excepting cases where the dated mineral was embedded in uranium ore. Thus, if samples are selected with some care, it can be assumed that all the spontaneous tracks are caused by spontaneous fission of ^{238}U.

Several causes for variation in the ρ_s/ρ_i ratio have been discussed above. There are, however, also procedural considerations which must be adhered to if the track ratio is to accurately reflect reality. As the etching procedure can cause variations in the countable track density, it is essential that identical conditions be used when etching spontaneous and induced tracks. Boellstorff (pers. comm., 1985) has stated that spontaneous tracks are often smaller than induced tracks. Thus it is important to be sure not to overlook the smaller tracks when counting. Consistent errors of this type can be calculated into a personal ζ factor (as counting efficiency) for use when calculating the age of a sample.

Use of the ζ factor is highly recommended by Hurford and Green (1983) for a variety of reasons. They found that there were unexplained errors in the response of both Co foil activation monitors and occasionally in the NBS-SRM612 dosimeter itself which could cause estimates of neutron fluence to be radically different from the true values. The comparative method of determining neutron fluence (comparing induced track densities in preirradiated and worker irradiated specimens of a dosimeter), has the problem that a subjective decision on the part of the worker as to what will be considered a track is introduced into the fluence calculation. The problems in determining an absolute value for the fission decay rate have been discussed above.

For these reasons, Hurford and Green have rejected this method of age determination as "grossly imprecise". Their method involves repeated calibration of a uranium glass dosimeter against a suite of minerals whose ages have been determined by other means, such as K/Ar. This removes the necessity of separate determinations of neutron fluence and fission decay rate as well as any systematic counting "errors" by combining them all in one factor. Since determination of the ζ factor depends on track etching and counting conditions, as well as track identification criteria, etc.; a ζ factor must be determined for each worker. Therefore, it is suggested by Hurford and Green that a suite of minerals and dosimeters be kept at each laboratory so any new worker may derive a "personal ζ factor" by recounting them.

APPENDIX C

A Consideration of the Different Standards Used and some of the Problems with the Fission-Track Dating Technique

The fluence within the nuclear reactor during irradiation is determined by counting fission-tracks in a standard glass with a known uranium concentration. The fluence cited by the reactor operator can be off by as much as 20% (Naeser, 1976), so an independent measurement is necessary. This is even more evident when it is noted that while the thermal neutron fluence in the age equation refers to the senso stricto 2.53×10^{-8} MeV thermal neutrons, many reactors report thermal neutron fluence in the sub-cadmium ($< 5 \times 10^{-7}$ MeV) sense (see Appendix B). In order to maintain a countable density of fission-tracks (neither rare nor overlapping), a dosimeter containing a uranium concentration similar to that of the samples is desirable. Three different glass standards were selected for use on the this project.

The National Bureau of Standards (NBS) Fission Track Glass wafers are used by most researchers within the United States. These wafers are purchased in a set containing both nonirradiated and irradiated samples, and thus make convenient dosimeters. They are a subset of NBS's series of Trace Elements in Glass standards. This series contains more than sixty different trace elements, which have been analyzed by many different chemical techniques (Carpenter and Reimer, 1974).

In their pioneering studies on fission-tracks, Fleischer, Price, and Walker (1965) used chips from microscope slides. Boellstorff uses as his dosimeter chips from a similar set of slides which have been calibrated against Fleischer's original set of dosimeters. The uranium distribution was found to be uniform and present at a concentration of 0.7 ppm (Boellstorff, pers. comm.; 1985). A detailed chemical analysis of this glass has not been performed.

Walker requested the Corning Museum of Glass to prepare some uranium glass standards for use in fission-track dating (Schreurs, pers. comm.; 1984). Unfortunately, this original dosimeter glass (U-1) was lost in a flood in 1972. Since then, Dr. Jan Schreurs, of Corning, has prepared several other uranium glasses. Corning glass CN-1 contains approximately 40 ppm U and was chosen as the third dosimeter.

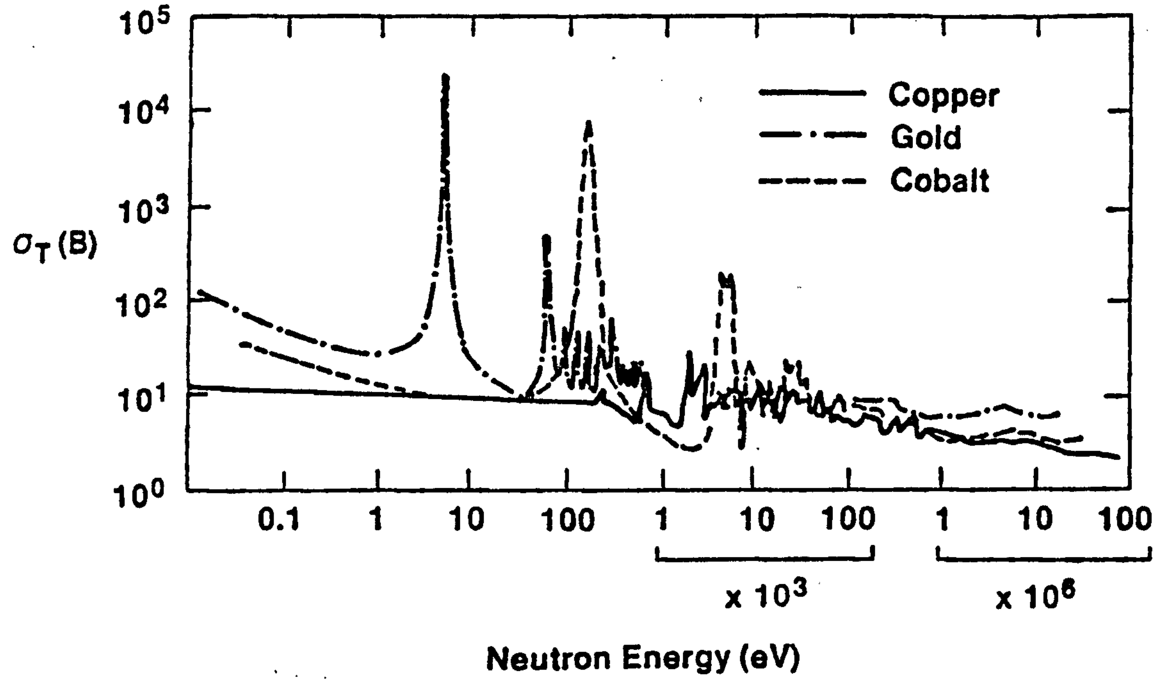
Calibration of dosimeters

Glass standards are usually calibrated by means of counting the activity produced in metal foils irradiated along with the glass. The National Bureau of Standards calibrated their fission-track standards using copper and gold foils "since many laboratories routinely use these foils" (Carpenter and Reimer, 1974, p. 3). During irradiation of the standards, every tenth irradiation vessel (called a rabbit) contained one piece each of gold and copper foil. The certificate enclosed with the standards upon receipt from the NBS also listed noncertified fluence values obtained from iron and cobalt foils. For the same irradiation in the NBS Research Reactor Irradiation Position RT-4 the following fluences were determined (all are reported as X 10^{14} n/cm^2): copper - 3.87 ± 0.07, gold - 4.17 ± 0.08, iron - 3.87 ± 0.08, and cobalt - 3.79 ± 0.12 (NBS Certificate, SRM962a, 1984).

The choice of which foil to use as a standard affects the resultant fluence determination because of the variation in the reaction of different elements to irradiation. One of these factors is the cross section, or probability of a nuclear process occurring, as discussed in Appendix B. Various elements react dissimilarly to the spectra of energies present within a nuclear reactor, thus a precise knowledge of the energy spectra is necessary in order to accurately determine the fluence. This is usually not the case. Figure 31 shows the relationship between cross section and neutron energy for several metals which are commonly used for dosimeter calibration. This factor is too variable to account for within a standard calibration equation.

To try and minimize this variable, Boellstorff advocates using uranium foil, since uranium is the element with which fission-track dating is concerned. Fleischer and Price (1964) used uranium foil in the original calibration of their microscope

Figure 31. Comparison of cross-sections of metals commonly used to calibrate dosimeters. Compare with figure 30 (^{235}U). Note the wide range in cross-sections (eg probability of a nuclear process occurring) depending on neutron energy. Thermal neutrons (those to which the age equation refers) have an energy of 0.0253 eV. Frequently, "thermal" neutrons are equated with "sub-cadmium" neutrons, which have an energy of < 0.5 eV, much greater than that for thermal neutron _senso stricto_. Many reactors, including those of the TRIGA type (USGS reactor) create many neutrons with an energy of > 10 eV. Thus a wide energy spectrum may be present during an irradiation. This is the reason Boellstorff advocates calibration with uranium foil. At least this wide energy spectrum is striking the same element in both foil and dosimeter, thus reducing the number of unknowns.



From Carpenter, 1984

slide standards. A piece of uranium foil was included along with the standard in each of a series of irradiations at different fluence levels. The uranium foil was then analyzed for the resultant ^{140}Ba and ^{99}Mo activity and a fluence determined. The results were graphed along with the induced track density in the dosimeter glasses. This produced a straight-line equation, useful only for this particular dosimeter, in which the only variable is the induced fission-track density of the dosimeter.

Thiel and Herr (1976) noted a problem with interference between uranium foils and reactor neutrons which may cause uranium foils to yield erroneous fluence values. However, Boellstorff feels that this is only a significant problem if thick foils are used, and that in any rate, the error is less than that caused by using a glass standard doped with depleted uranium and then calibrated by comparison to activity induced by an unknown energy spectra in a foil made of any material other than uranium (1986, pers. comm.). The author is unsure which procedure is truly "best". Unfortunately, the choice of decay rate in the age equation is related to this choice of dosimeter calibration technique and thus contributes to the uncertainty of ones results. (Compare Thiel and Herr, 1976; Roberts _et al_., 1968; and Seward, 1979.)

Determination of neutron fluence for a sample run

Though an equation has been developed for use with Fleischer's standard, and adjusted for use with Boellstorff's standard, other dosimeters must make use of a comparative technique for neutron fluence determination. The NBS has provided for this by sending both preirradiated and nonirradiated glass in each package of dosimeters ordered. But since the track density in glass is hard to determine with good reproducibility (Naeser, 1976), it is essential that the NBS-irradiated/researcher-irradiated pair be etched at the same time. This requires the researcher to repolish and etch the NBS irradiated wafer each time it is used. An external detector eliminates this necessity. This method involves taping either a piece of uranium-poor muscovite or Lexan polycarbonate to the polished surface of a sample before irradiation. One of the fission fragments resulting from each fission event of ^{235}U within range of the surface enters the detector and leaves a fission-track. The etch conditions for these detectors are less sensitive to varying etch conditions than is glass. Thus the external detector method is frequently preferred to counting fission-tracks directly on the glass.

Another potential problem with the NBS dosimeters is the presence of other elements within the glass (see table 10). These other elements can have an effect on the fluence determination when calculated by the external detector method.

Table 10. NBS-SRM962 is the National Bureau of Standards fission-track standard and is a subset of NBS-SRM612 (Trace Elements in Glass Standard), thus they have the same chemistry. CN-1 is a Corning Museum of Glass uranium standard glass, and U-1 was the original uranium standard made by Corning. B is Boellstorff's microscope slide fission-track standard. Note the wide variety of values for the isotopic ratio of ^{235}U to ^{238}U.

Chemistry of the Standard Glasses

	Base Glass Chemistry (wt %)						Trace Elements (ppm)			
Standard	SiO_2	Na_2O	CaO	MgO	Al_2O_3	K_2O	Th	B	U	Isotopic ratio
NBS-SRM962[a]	72	14	12	-	2	-	37.79[1]	32[2]	37.38	418[4]
NBS-SRM612[a]	72	14	12	-	2	-	37.79[1]	32[2]	37.38	-
CN-1[b]	72.7	16.7	5.1	3.5	1.4	0.4	~.4	-	~40[3]	137.77
U-1[c5]	-	-	-	-	-	-	-	-	40.9	277.8
B[d]	-	-	-	-	-	-	-	-	0.7	"natural"

Sources

a) Carpenter and Reimer, 1974
b) Schreurs, pers. comm., 1984
c) Schreurs <u>et</u> <u>al</u>., 1970
d) Boellstorff, pers. comm., 1985

Notes

1) ^{232}Th only 2) ^{32}B only

3) average of 9 determinations using 5 different methods

4) obtained by inverting ^{235}U atom percent, thus number is a bit low

5) "very pure matrix glass" Contains depleted U of ~½natural values.

Both thorium and boron "emit charged particles which may be recorded in many external detectors" (Carpenter and Reimer, p. 2, 1974). Also, at neutron energy levels of $> 4 \times 10^{-7}$ MeV, ^{232}Th will fission (Boellstorff, pers. comm., 1986). The NBS glasses contain substantial quantities of these elements. However, natural materials also contain many of these same elements. No other trace elements were purposefully added to the Corning glass in an effort to make it a "pure" uranium standard. Boellstorff's dosimeter is likely to contain small amounts of a variety of trace elements, including thorium, since this glass was not originally produced for analytical purposes.

Another difference between these three standards is the type of uranium each contains. The NBS dosimeters have been doped with depleted uranium. Each of the different uranium concentration levels has a different ^{235}U isotopic abundance, though none of these is equivalent to natural abundances. The Corning glass was doped with pre-war undepleted uranium, thus its isotopic abundance is the same as that of natural uranium-containing materials. The microscope slide glass used by Boellstorff contains natural uranium. This is only a potential problem when determining uranium concentration levels as opposed to fluence levels, however, since the uranium is homogeneous within each series of standards.

One final consideration is the availability of a pre-irradiated specimen. If a sample irradiated under presumably known conditions does not exist, then the straightforward comparative technique can not be used.

As stated above, it is possible to develop an equation to determine neutron fluence for a particular dosimeter. There are several problems with this technique, however. Different researchers can have slightly different fission-track identification criteria which can compound the problem of reproducibility of fission-track counts due to etching differences. There is no way to account for these differences in a standard equation. When a pre-irradiated/ nonpre-irradiated pair are etched and counted together, these effects are minimized.

Lacking an irradiated/non-irradiated pair of dosimeters to work from, there are only two other methods by which to calibrate a dosimeter. The first is to date a series of materials of known age and calculate the neutron fluence (Φ) from the age equation. This method is not practical for routine work, though it should always be used when initially calibrating a standard.

The other method is analogous to that described for the NBS dosimeters. Since the fission-track density (ρ_o) can be expressed as:

$$\rho_o = B\,\Phi, \qquad \text{eqn 12}$$

if the fluence for a reactor run can be determined by another method, such as a <u>different</u> dosimeter, that fluence can be ascribed to the fission-track density counted in the original dosimeter. An unknown neutron fluence can then be calculated by using this B factor whenever the same glass and etching conditions are used. This method can take into account differences in counting criteria (a B factor can be determined for each worker). Etching conditions also become less problematic since the two dosimeters can be etched simultaneously. In the end, the choice of which dosimeter to use depends upon the facilities available to the researcher and the research goals for the particular project.

APPENDIX D

Methodology used for obtaining and fission-track dating zircon phenocrysts

A description of the procedures used in an attempt to obtain zircon fission-track dates for the DeSoto ash is outlined below. This attempt was made because zircon is much more resistant to thermal annealing of fission-tracks than is glass.

Sample Preparation

One gallon of the dried ash was left overnight in 0.1 N sodium oxalate to deflocculate any clays. The ash was then wet sieved through a 30, 60, 170 mesh sieve stack, saving the -170 mesh fraction. After each portion of ash was sieved, time was allowed for the larger and heavier portion of the -170 mesh fraction to settle. Most of the clay-sized fraction was then decanted and the residue reserved. This was done because the zircons are small and elongate, thus there is a high probability of many being in the -170 mesh fraction. This method was found to be easier than using the 230 mesh sieve because it clogs too rapidly.

In order to concentrate the heavy minerals, the reserved portion of the -170 mesh fraction were centrifuged in concentrated zinc bromide ($ZnBr_2$) with a specific gravity of about 2.45. Three to four grams of ash were added to 100 ml of $ZnBr_2$ in a large (175 ml) plastic centrifuge tube. After 10 minutes at 2000 rpm, a firm disk of mostly glass could be lifted off the top of the $ZnBr_2$. In order to conserve heavy liquid, the heavies were filtered off only after several centrifuge runs. More ash was added to the tube, the $ZnBr_2$ topped off, and the contents thoroughly mixed for intermediate runs.

When the -60+170 mesh fraction was centrifuged in $ZnBr_2$, the upper glass disk did not become firm enough to remove easily. In order to separate the two weight fractions far enough to allow pouring off the light fraction, extravagant quantities of $ZnBr_2$ had to be used. An attempt to use a separatory funnel was abandoned because it took too long for the separation to occur and only small quantities of ash could be processed at a time. For these reasons, bromoform, which has a higher specific gravity (2.85) was used to process the -60+170 mesh sieve fraction. It was not used for the -170 mesh samples because it is expensive and is a carcinogen.

Separatory funnels were used along with bromoform to obtain zircons from the -60+170 mesh fraction. Approximately 3.5 g of ash were put in each 125 ml funnel. The funnels were filled with bromoform and the mixture stirred thoroughly every five minutes for the first half hour of settling. Stirring helps prevent zircons from sticking to the light weight glass shards and thus floating to the top.

After at least three hours of settling time, the heavy fraction was drained into a beaker. Acetone was added to thin the bromoform and allow easier cleaning of the resulting fractions. The bromoform can be reclaimed for future use by adding water. The water will attract the acetone and the resulting mixture floats on the more dense bromoform.

The heavy minerals were then retrieved by filtering. Paper filters are quite fibrous and the author was concerned that the tiny zircon crystals might become trapped in these fibers. For this reason, smooth millipore filters were used. Unfortunately, these filters dissolved in the small amount of acetone still mixed with the heavy minerals and created a glue-like substance. In an attempt to retrieve the zircons, they were treated with a variety of solvents, but were never completely cleaned. The next step in separating zircons should be to perform a magnetic separation. However, this was not attempted for several reasons, primary of which was incomplete cleaning of the zircons.

Slide preparation, irradiation and etching

After discussing with Naeser the difficulties encountered when filtering the zircons, it was decided to proceed. The zircons should not have been damaged; and the silicates, apatite and sphene, might have been removed in the process. (One hour in warm 48% HF will dissolve these minerals.) The following is a brief synopsis of the procedure Naeser and the author used in Denver for zircon fission-track dating. Detailed instructions can be found in Naeser (1976).

The zircon crystals were spread over an area of about 1 cm^2 on a glass slide which was then placed on a hot plate. The zircons were covered with an FEP teflon disk and a second glass slide. This package was pressed together until the teflon softened and the zircons became embedded. The teflon disk was then glued to a glass slide with epoxy and the grain mount polished following the same procedures as discussed earlier for the glass slides.

The polished zircons were then etched in a eutectic mixture of KOH and NaOH at 210°C. After 24 to 48 hours (depending on age, uranium concentration, etc.), the zircon surfaces will be covered with randomly oriented, funnel-shaped fission-tracks. Since the direction of movement of the fission fragment with respect to the crystallographic axes has an effect on etch rate, under-etching results in fission-tracks having an apparent preferred orientation. Over-etching causes loss of the zircon grain because it becomes too porous to be held by the teflon. This can cause problems with zoned crystals as some parts may etch much faster than others.

After etching, a muscovite detector is taped to the teflon disk. A clean, fresh surface is obtained by using double stick tape to cleave the muscovite. The slide is now ready to be irradiated.

The interlaboratory standard for zircon fission-track dating is zircon crystals from the Fish Canyon Tuff of Colorado (dated at 27.2 ± 0.7 my) (Seward, 1979). A sample of Fish Canyon zircons and two pieces of NBS-SRM962a dosimeter, all covered with muscovite detectors, were irradiated by Naeser in Denver for 35 minutes at 200 Kw.

Counting Procedure

The induced fission-track density is counted on the muscovite detector after it is etched. The spontaneous density is counted directly on the zircons. No induced tracks will show up on the zircons because etching was done prior to irradiation.

After the zircons have been irradiated, the muscovite is removed. A pin is used to punch holes through the muscovite into the teflon disk to enable the images of specific zircons to be found in the muscovite when counting. So many induced tracks are made in the muscovite detector that the image of each zircon crystal will be clearly visible after etching. Muscovite is etched for 16 minutes at 20°C in 48% hydrofluoric acid. The muscovite is attached adjacent to the teflon disk on a glass slide so the two are positioned as open pages of a book.

A 1 mm^2 grid with 100 square subdivisions was used for counting. Magnifications of about 1000X are needed for zircon fission-tracks and of about 500X for the muscovite. A zircon/muscovite image pair is located using the punched holes and grid. A counting area is delimited within the zircon crystal and its corresponding image in the muscovite detector to be sure the same area is counted on both. Tracks counted on the zircon crystal and its image in the muscovite are recorded separately. Zircon fission-tracks are funnel-shaped so a spot of light moves up and down the length of the track as the focus is moved up and down. Fission-tracks in muscovite are diamond-shaped. Virtually everything seen in the specially selected detector will be a track.

Age determination

Zircon fission-track ages are determined using the same equation as for glass fission-track ages. The spontaneous fission-track density is obtained by counting tracks in the zircon crystal, and the induced fission-track density is obtained by counting tracks in the muscovite detector. The detector was in two-pi geometry with the zircon crystal, and thus contains tracks from ^{235}U atoms which lay below it within the zircon crystal. For comparison, the fission-tracks visible on the polished surface of the zircon crystals were formed in four-pi geometry (e.g. tracks formed from ^{238}U atoms which lay both above

AND below the polished surface are present). For this reason, the induced track density must be multiplied by two before the age calculation is performed. As stated above, zircons from the Fish Canyon Tuff are used as an interlaboratory age standard.

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