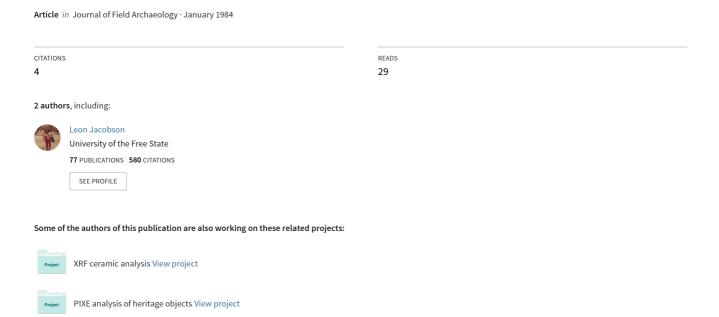
# Cultural sediment formation in open air sites and rock shelters on the Brandberg, Namibia





# **News and Short Contributions**

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# News and Short Contributions

# Field Report

# Cultural Sediment Formation in Open-Air Sites and Rock Shelters on the Brandberg, Namibia

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Sediment analysis of three open-air sites and two rock shelters in the Brandberg, Namibia, provided the opportunity to examine site formation and disturbance in an arid environment. Geomorphic processes peculiar to site settings at different scales are considered in explaining intersite and intrasite variation among sediment samples. The evidence for environmental trends and cultural patterning beneath this geomorphic overlay is discussed, and the sedimentary and geochemical configurations are attributed to cultural processes.

#### Introduction

Systematic study of the interaction between cultural and non-cultural processes, and between organic and inorganic inputs in site formation will increase resolution in the interpretation of archaeological residues. The evidence for cultural ordering preserved at archaeological sites is difficult to assess unless the effect of archaeosedimentary processes on site arrangement is also considered. Similarly, evidence for environmental trends in stratified archaeological contexts is difficult to interpret unless the cultural half of the sedimentary equation is known or can be held constant. Sediment analysis of

1. Karl W. Butzer, *Archaeology as Human Ecology* (Cambridge University Press: Cambridge 1982) 35–42.

three surface sites and two rock shelters in the Brandberg provided the opportunity to study site formation and disturbance in an arid, granitic, and high relief setting, with the objective of recovering evidence for environmental trends and cultural patterning they would otherwise mask.

The Brandberg is a large granite inselberg located on the eastern edge of the Namib desert in Western Damaraland, Namibia (FIG. 1). Inselbergs provide topographic and biotic variation in semiarid and arid environments and it is no surprise that Middle Stone Age (M.S.A.), Late Stone Age (L.S.A.), and macrolithic Brandberg assemblages (of the 16th–18th centuries A.C.) have been found in this area. The M.S.A. and L.S.A. material comes primarily from rock shelters, while Brandberg occurrences are usually associated with concentrations of free-standing stone structures.

Recent investigations in the Brandberg by L. Jacobson have focused on both abri and surface sites, including the Girls School Shelter and two settlements of open-air stone structures, Tsisab A and Tsisab 30 in the Tsisab ravine, as well as Lower Numas Cave and a presumed village site (NV 2), in the Numas valley.2 Twenty-four sediment samples were collected for analysis at the University of Chicago Paleoecology Laboratory in order to obtain information on past environmental changes from the shelter sites, and on the past functions of the structures that comprise the open-air sites. To weigh the evidence on these issues, it was necessary to reconstruct the geomorphic context of the sites, particularly the extent and nature of mechanical and chemical weathering, as well as the transport agencies and post-depositional alteration to which the samples have been exposed.

Laboratory analysis of the Brandberg sediment samples included particle-size analysis (by hydrometer and

2. Leon Jacobson, "Mid-Holocene to Recent Cultural Changes in the Brandberg, S. W. A.," in *Proceedings, IXth Union International des Sciences Préhistoriques et Protohistoriques Congrès* (Nice 1976); idem, "A Study of Functional Variability in the Later Stone Age of Western Damaraland, Namibia," unpublished B. A. Honors paper, University of Cape Town (Cape Town 1978).

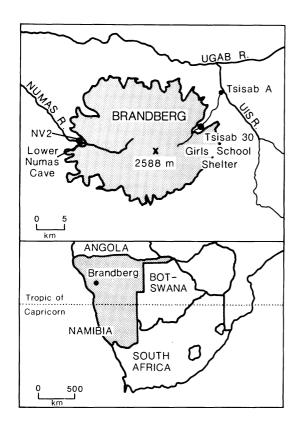


Figure 1. The Brandberg and its location in Namibia (South West Africa).

dry sieving), examination by binocular microscope, and determination of pH, calcium carbonate (CaCO<sub>3</sub>), organic matter (O.M.), and phosphorus (P) content. The results of the analytical program, including standard Folk textural parameters,<sup>3</sup> are presented in graphic form below. The source material for all clastic particles in the Brandberg samples is the Brandberg granite,<sup>4</sup> together with small quantities of stained and rounded, probably eolian, quartz grains; the samples, however, vary in terms of mineral proportions, chemical composition, and particle-size distribution.

# The Open-Air Sites

Samples were collected from two Brandberg industry sites, Tsisab 30 and NV 2, and from one abandoned Dama settlement, Tsisab A. At the latter, functions of the extant structures can be reconstructed quite confi-

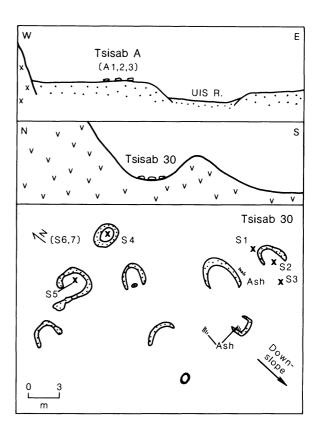


Figure 2. Schematic setting of Tsisab A and Tsisab 30 sites (above), and partial plan of Tsisab A structures, indicating sample locations (S1–S7). Bedrock is Mesozoic lava for Tsisab 30, and Brandberg granite for Tsisab A.

dently from 19th century A.C. ethnographic reports on the Dama, pastoralists probably responsible for the prehistoric Brandberg industry (FIG. 2). At each site samples were taken from within structures or features and from an adjacent spot less likely to have been affected by human activity. It was hoped that human effects on the sediments could be pinpointed, partially through intrasite comparison, and that these would form a pattern. It was expected that known kraals (stock pens) and huts of Tsisab A would have relatively high P and O.M. values, from dung as well as more general human use, while O.M. and P values from the prehistoric sites would provide a basis on which to confirm or refute customary inferences as to the functions of structures in Brandberg industry settlements by analogy with similar structures in Dama settlements. Similar ethnographic interpretations have been proposed by Maggs.<sup>5</sup>

Samples were collected from a hut, a kraal, and be-

5. T. M. O'C. Maggs, "Pastoral Settlement on the Riet River," SAfArchBull 26 (1971) 37-63.

<sup>3.</sup> Robert L. Folk, "A Review of Grain-Size Parameters," *Sedimentology* 6 (1966) 73–93.

<sup>4.</sup> F. D. I. Hodgson, "Petrography and Evolution of the Brandberg Intrusion, S. W. A.," *Special Publication of the Geological Society of South Africa* 3 (1973) 339–343.

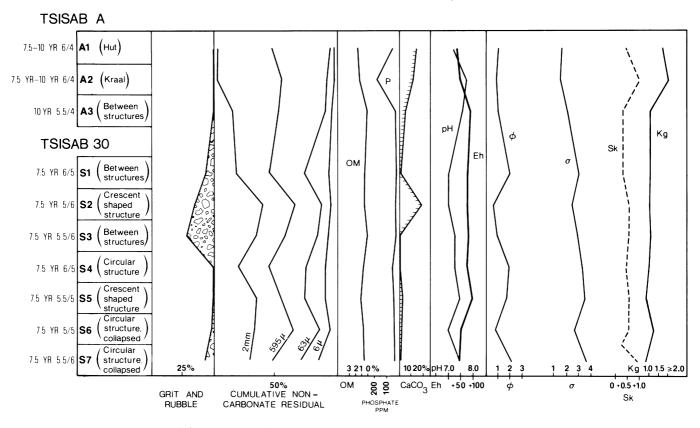


Figure 3. Analytical results from Tsisab A and 30. Cumulative Textures are shown for the fraction under 6.3 mm. Calcium carbonate determined by bulk loss in acid only.

tween structures at Tsisab A, which is situated at the confluence of the Tsisab and Uis rivers. At Tsisab 30, located on a platform near Girls School Shelter and with radiocarbon dates of 210 (Pta-1821), 275 (Pta-1820), and 420 (Pta-1783) B.P., 6 seven samples were collected: two from an area outside any structures; two from within crescent-shaped, probable hut structures; one from a circular structure thought to have been a kraal; and two from two small, collapsed, circular structures also thought to have been kraals. At NV 2, samples were removed from a centrally located cairn, one from the ground surface and one from the bovid burial beneath.

### **Site Formation**

All the samples from Tsisab 30 consist of superficially weathered granitic rubble and a small quantity of eolian quartz grains (FIG. 3). Mineralogically, the composition of all the samples is similar to that of the bedrock, but they fall into two different size categories. Samples S1, S4, S5, and S7 have smaller mean particle sizes and

6. J. C. Vogel and E. Visser, "Pretoria Radiocarbon Dates II," Radiocarbon 23 (1981) 43-80.

fewer pieces of rock coarser than 6.3 mm (usually intact granite) than samples S2, S3, and S6. The best explanation for this size difference probably is that S2, S3, and S6 have received coarse, slope-derived granite debris in addition to rubble from in situ weathering of granite, while samples S1, S4, S5, and S7 have been protected from such input by their greater distance from the slopes on either side of the site and/or the existence of stone walls between them and the slope.

The Tsisab A samples are ca. 80–90% quartz, only a small percentage of which is eolian. This indicates that the less resistant feldspars of the source granite have been selectively weathered, leaving the quartz intact. The latter is concentrated in the potential traction and saltation size grade, an indication of effective water sorting; and sand-grain rounding is usually good compared with all other Brandberg samples analyzed. The location of Tsisab A, near the confluence of two rivers, makes a fluvial origin probable. Within Tsisab A, sample A3 has a larger 210 µm to 2 mm component than samples A1 and A2; A3 represents soil between abandoned structures, instead of within one, and therefore has been less protected from continuing surface runoff.

At both Tsisab 30 and Tsisab A the intrasite variation

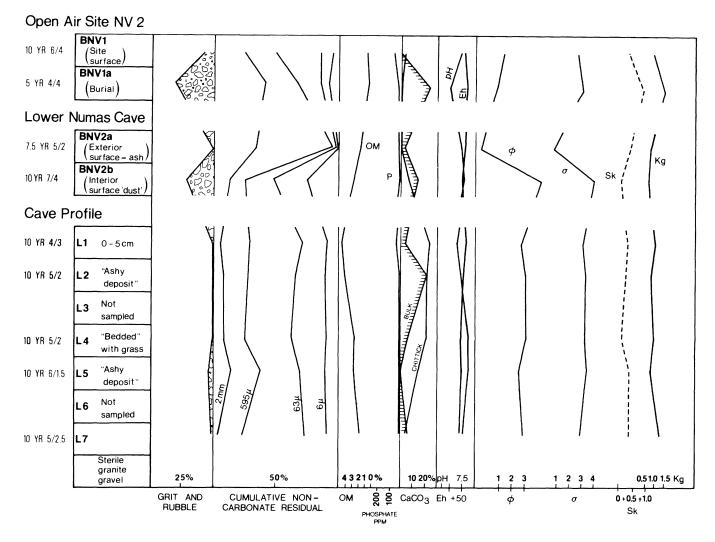


Figure 4. Analytical results from NV 2 and Lower Numas Cave. Calcium carbonate determined by bulk loss as well as by the Chittick method.

of the O.M. and P values corresponds closely to cultural patterns, rather than to variation in the character or intensity of geomorphic processes. The interpretation of this variability will, therefore, be sought in patterns of site use.

The NV 2 samples are primarily composed of local granitic debris, which is slightly less corroded than that of Tsisab 30 and has a slightly stronger, although still minor, eolian component (FIG. 4). Sample BNV 1 resembles the finer group of samples from Tsisab 30, especially S1 and S4, and is a surface sample from a roughly comparable topographic location. BNV la, from the burial itself, is of coarser grade. This difference in particlesize distributions, plus a decrease in grain-surface weathering, pH, P, botanical residues, and O.M., as well as an increase in CaCO<sub>3</sub> between BNV 1 and

BNV la, can in part be explained by continuing soil formation on the exposed surface. The samples from NV 2 do not, therefore, appear to encode information on past human activities.

# Site Use

Although the values for O.M. and P are very low for all the samples from Tsisab 30 and Tsisab A, they conform to a pattern that seems to confirm analogies drawn between Dama and Brandberg industry settlements, if time-dependent decay of these materials can be assumed. The highest value for organic matter was obtained for the Tsisab A hut structure, and the second highest for the Tsisab A kraal. The highest P value was obtained for the Tsisab A kraal. Among the Tsisab 30 samples,

O.M. peaks in the two crescent-shaped, hut outlines, while P peaks slightly in the ruins of the probable kraal.

# The Shelter Sites

Girls School Shelter, located near the foot of a steep slope on the edge of the Tsisab ravine, is formed by a single boulder (FIG. 5). The site contains ca. 1 m of deposit, the upper levels of which have been radiocarbondated to 720 (Pta-1773), 910 (Pta-1777), 2780 (Pta-1776), and 6510 (Pta-1547) B.P.<sup>7</sup> These levels have produced microlithic materials; the bottom two units of the deposit have produced materials suggestive of M.S.A. Sediment samples were collected from the bottom five levels (L3-L7), the topmost levels (L1-L2) consisting mainly of ash.

The Lower Numas Cave is a shelter formed by several interlocking granite boulders and was excavated by Rudner<sup>8</sup> and Jacobson<sup>9</sup> (FIG. 6). Radiocarbon dates of 2890 (Pta-178) and 2950 (Pta-179) B.P.10 from the Rudner excavations (equivalent to Level 2), and of 3950 (Pta-1623), 4180 (Pta-1295), and 4840 (Pta-1620) B.P.<sup>11</sup> from the Jacobson excavations (Level 5), suggest that the 80 cm of deposit at this site accumulated between ca. 3000 and 5000 B.P. The archaeological record includes a microlithic stone-tool assemblage, ostrich eggshell, an upper grindstone, broken slate pendants, a bone awl, and a gemsbok horn, favoring an L.S.A. designation. A total of seven samples was collected from Lower Numas Cave, representing six of eight stratigraphic units, as well as the ground surface just beyond the sheltered area.

# **Site Formation**

There seem to be more differences between the sediments from Girls School Shelter and Lower Numas Cave than there are among the individual levels of either site. As a group, the samples from the Girls School Shelter tend to have a coarser mean size fraction, and a higher percentage of grit/rubble (FIG. 7). They are also more positively skewed than the Lower Numas Cave samples. Given that the two deposits were formed from the same

# 7. Ibid

- 8. J. Rudner, "The Brandberg and its Archaeological Remains," Journal of the South West African Scientific Society 12 (1957) 7-44; idem, "Radiocarbon Dates from the Brandberg in South West Africa," SAfArchBull 27 (1973) 107-108.
- 9. Jacobson, 1978 op. cit. (in note 2).
- 10. J. D. Vogel and M. Marais, "Pretoria Radiocarbon Dates I," Radiocarbon 13 (1971) 378-394.
- 11. Leon Jacobson and J. C. Vogel, "Recent Radiocarbon Dates From the Brandberg," South African Journal of Science 71 (1975) 349; Vogel and Visser, op. cit. (in note 6).

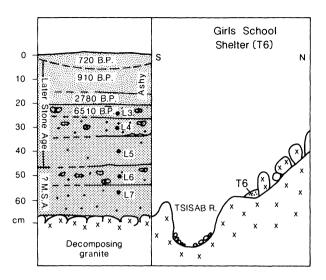


Figure 5. Schematic setting of Girls School Shelter with profile.

source material, this pattern of variation suggests that some spatial rather than temporal factor or process controlled their formation. Similarities between the Girls School Shelter and the Tsisab 30 samples (from a parallel topographic location) suggest that this spatial variable is site setting or, more precisely, the set of geomorphic processes characteristic of the specific topographic situation. The finer than 6.3 mm fraction of the Girls School Shelter samples is matched quite closely by the finer

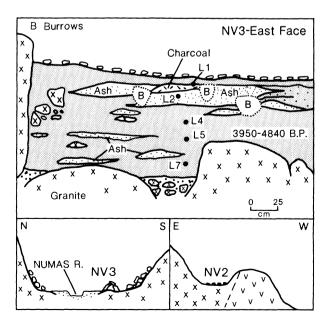


Figure 6. Schematic setting of NV 2 and NV 3 and profile of Lower Numas Cave (NV 3).

# GIRLS SCHOOL SHELTER

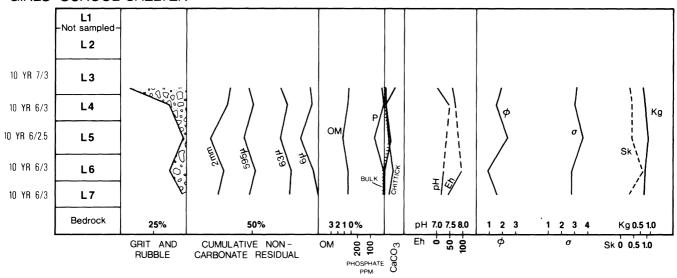


Figure 7. Analytical results from Girls School Shelter.

samples from Tsisab 30, but the relative amount of material coarser than 6.3 mm is paralleled best by the coarsest of the Tsisab 30 samples (S2, S3). Since the shelter site is situated at the base of a slope, that slope probably is the source of the coarse material. The different, symmetrical, and poorly sorted particle distribution characteristic of the Lower Numas Cave probably is compatible with a more completely enclosed shelter, acting as a better trap for fine sediments partly of cultural origin. In the Girls School Shelter and among the surface sites such fine components would have been both diluted and selectively washed or blown away. Higher O.M. values and quantities of organic material, especially charcoal, in the Lower Numas Cave samples reflect this factor as well as the intensity of human occupation and limited oxidation or rainwater eluviation within the enclosed shelter. The high CaCO<sub>3</sub> approximations for the first four levels can also be explained by lack of percolating water.

The temporal variability of the shelter sites can be adequately explained by postulating sediment contributions from human intervention and time-dependent decay of the organic compounds. In Girls School Shelter, the samples with the greatest proportion of material coarser than 6.3 mm are found below a major disconformity, and are therefore likely to represent a residual deposit, the associated "fines" of which were washed out or otherwise eroded. Sample L6, which also contains a relatively large amount of very coarse material, may reflect the same phenomenon. Level 5 is distinct from the other

Girls School Shelter levels because it has a relatively high clay content and a relatively low proportion of very coarse material. This sample, which comes from a level beneath the 6510 B.P. date but above the probable M.S.A. levels, can tentatively be called an artifact of relatively intense human occupation; the high O.M. and P values helped simulate the relatively high clay content.

Among the samples from Lower Numas Cave, the greatest contrast is between samples from Levels 1 to 7, and from the interior and exterior surfaces. While this contrast could be used as evidence that geomorphic processes that are at present active could not have formed the deposit, it can be, and therefore probably should be, interpreted in the context of the present environment. The particle-size distribution of the exterior surface is very similar to that of the fluvially sorted Tsisab A deposit; but instead of being 80-90% quartz, it is composed of a relatively fresh granitic debris. This sample is a fluvially sorted, but locally produced, igneous product that reflects geomorphic processes more similar to those of a floodplain than to those of a cave. The internal surface sample is somewhat enigmatic, containing both more grit/rubble and clay than do the cave fills. This internal deposit may reflect partial rock disintegration and eolian dust accumulated during the three thousand or so years since the shelter's last occupation. Finally, it can be noted that the O.M. and P values for Lower Numas Cave decline steadily with depth, suggesting a process of time-dependent decay.

# **Site Formation Processes and Past Environmental Change**

Since differential formation of the shelter deposits can be attributed to differences in topographic setting, to human interference, and to the passage of time, there appears to be no need to postulate changes in the climatically controlled rate and form of granite weathering, the most climatically sensitive factor in the system under consideration. The deposits sampled from the two shelters, however, do not overlap temporally and there is only minimal information for current and past background weathering processes in the shelters, or for the Brandberg as a whole. It is, therefore, impossible to rule out the possibility that some variability between the sites—the sampled portions of which are of different ages—or within the sites, resulted from changes in climate, particularly the minimum amount of moisture necessary for chemical, rather than mechanical weathering. An increase in chemical decomposition would produce more fine, granular debris, in contrast to the coarse mechanical products of exfoliation. The limited amount of very coarse debris in the cultural levels of Lower Numas Cave, compared with the surface rubble (BNV 2b), could therefore be explained by an increase in chemical weathering. Equally well, however, this might reflect rapid sedimentation as a result of human occupation, with a consequent dilution of exfoliated rubble.

#### Conclusion

Analysis of sediment samples collected from Tsisab A and Tsisab 30, stone-built structures in the Brandberg, suggests that variations in textural and geochemical values between structures of differing configuration cannot be attributed to geomorphic agencies, but instead give support for using ethnographic analogy to interpret structure function of the basis of shape. If low values of, and a time-dependent decay for, O.M. and P are accepted, the evidence suggests that, like the Dama, groups associated with the Brandberg industry used crescent-shaped structures for huts and circular ones for kraals. On the other hand, at NV 2 the difference between O.M. and P values is less a result of cultural activity than of soil formation. Samples from Girls School Shelter and Lower Numas Cave did not provide unequivocal evidence for environmental change in the Brandberg. The shelter analyses do illustrate the importance of separating cultural and noncultural sediment inputs and provide evidence for a phase of relatively intense human occupation. This is Level 5 at Girls School Shelter, which is later than the probable M.S.A. levels at this site, but older than 6510 B.P. (Level 3).

These conclusions are limited in their details by the

number of samples available, and the absence of an intensive geoarchaeological on-site investigation. They do, however, show that the sedimentary and geochemical configurations identified were essentially the product of cultural processes. This in turn suggests that geoarchaeological investigation can always be productive, even in the case of shallow open-air deposits of relatively recent age.

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Leon Jacobson, B.A. (Hons), is senior professional officer in charge of archaeology at the State Museum, Windhoek, Namibia. His research interests include the archaeology of arid environments and the application of nuclear chemistry to archaeological problems.

# Special Study

# **Organic Matter and Carbonates in Archaeological Sites**

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Organic matter and carbonates are two groups of compounds often cited as evidence of occupation, but often their content is not analytically calculated. A loss-on-ignition technique for determining organicmatter and carbonate percentages can quantify these constituents. As demonstrated for the Vosburg site, located on the Blue Earth River of south-central Minnesota, the technique can provide essential information concerning the mode of deposition and sources for matrices found within archaeological sites.

# Introduction

Human occupation contributes a variety of chemical compounds to the habitation surface. Archaeological studies have demonstrated that analysis of such compounds yields abundant information concerning prehistoric activity, enabling archaeologists to predict site boundaries, <sup>1</sup> to locate evidence of occupation in a profile, <sup>2</sup> and to quantify amounts of leaching. <sup>3</sup>

As unused food, refuse from house construction, and remains of the dead decompose, significant amounts of carbon, nitrogen, calcium, and phosphorus, as well as minute quantities of trace elements, are released. Individual compounds can be used to detect and to quantify particular archaeological materials. For example, phosphorus has been used to indicate the presence of bone, organic matter to identify areas of refuse and intensive habitation, and carbonates to determine density of shells or presence of limestone. Techniques for measuring phosphorus have been reported frequently,<sup>4</sup> but fewer

- 1. O. Arrhenius, "Investigation of Soil from Old Indian Sites," Ethnos 2-4 (1963) 122-136; Z. Goffer, M. Molcho, and I. Beit-Arieh, "The Disposal of Wastes in Ancient Beer-Sheba," JFA 10 (1983) 231-235; A. M. A. Griffith, "A Pedological Investigation of an Archaeological Site in Ontario, Canada: An Examination of the Soils In and Adjacent to a Former Village," Geoderma 24 (1980) 327-336; C. E. Heidenreich and S. Narratil, "Soil Analysis at the Robitaille Site, Part 1, Determining the Perimeter of the Village," Ontario Archaeology 20 (1973) 25-29; H. J. Lutz, "The Concentration of Certain Chemical Elements in the Soils of Alaskan Archaeological Sites," American Journal of Science 249 (1951) 925-928.
- 2. S. F. Cook and R. F. Heizer, "Studies on the Chemical Analysis of Archeological Sites," CPA 2 (1965) 11–102; T. F. Buehrer, "Chemical Study of the Material from Several Horizons from the Ventana Cave Profile," in E. Haury, ed., The Stratigraphy and Archaeology of Ventana Cave, Arizona (University of Arizona Press: Tucson 1950); J. E. Foss, "The Pedological Record at Several Paleoindian Sites in the Northeast," in W. S. Newman and B. Salwen, Amerinds and their Paleoenvironments in Northeastern North America (New York Academy of Sciences: New York 1977) 234–244; J. K. Stein and G. Rapp, Jr., "Archaeological Geology of Site," in G. Rapp, Jr. and S. E. Aschenbrenner, Excavations at Nichoria: Site, Environs, and Techniques (University of Minnesota Press: Minneapolis 1978) 234–257; R. M. Thorson and T. D. Hamilton, "Geology of the Dry Creek Site: a Stratified Early Man Site in Interior Alaska," Quaternary Research 7 (1977) 149–176.
- 3. Cook and Heizer, op. cit. (in note 2); J. F. Dormaar, "Effect of Boulder Flow on Soil Transformation under Tipi Rings," PAnt 21 (1976) 115–129; H. Laville, J. P. Rigaud, and J. Sackett, Rock Shelters of the Perigord, Geological Stratigraphy and Archaeological Succession (Academic Press: New York 1980); G. E. Mattingly and P. J. Williams, "A Note on the Chemical Analysis of a Soil Buried Since Roman Times," Journal of Soil Science 13 (1962) 254–258; J. K. Stein, "Geological Analysis of the Green River Shell Middens," Southeastern Archaeology (1982) 22–39; Stein and Rapp, op. cit. (in note 2).
- 4. R. C. Eidt, "A Rapid Chemical Field Test for Archaeological Site Surveying," AmAnt 38 (1973) 206–210; idem, "Detection and Examination of Anthrosols by Phosphate Analysis," Science 197 (1977) 1327–1333; R. C. Eidt and W. I. Woods, Abandoned Settlement Analysis: Theory and Practice (Field Test Assoc.: Shorewood, Wisconsin 1974); F. A. Hassan, "Sediments in Archaeology: Methods and Implications for Paleoenvironmental and Cultural Analysis," JFA 5 (1978) 197–213; idem, "Rapid Quantitative Determination of Phos-

archaeological publications have discussed the procedure for measuring organic matter and carbonate.

Because organic matter and carbonate are two compounds that are measured frequently by archaeologists, and that supply valuable information when quantified, I propose in this paper a simple loss-on-ignition procedure to quantify the organic matter and carbonate for archaeological sediments. A detailed description of the theory and mechanics of the technique will be followed by the results of a study from a site in Minnesota that will illustrate its potential in archaeological research.

# **Technique**

Methods for measuring organic matter and carbonates<sup>5</sup> in sediment are numerous. Organic matter is analyzed by oxidizing the sample in hydrogen peroxide,<sup>6</sup> or igniting it at high temperatures followed by measuring its weight loss.<sup>7</sup> Total carbon and organic carbon are usually analyzed by incinerating the sample and measuring the

phate in Archaeological Sediments," *JFA* 8 (1981) 384–387; D. F. Overstreet, "A Rapid Chemical Field Test for Archaeological Site Surveying: An Application and Evaluation," *Wisconsin Archaeologist* 55 (1974) 262–270; B. Proudfoot, "The Analysis and Interpretation of Soil Phosphorus in Archaeological Contexts," in D. A. Davidson and M. L. Shackley, *Geoarchaeology* (Westview: Boulder, Colorado 1976) 93–113; D. Proven, "Soil Phosphate Analysis as a Tool in Archaeology," *Norwegian Archaeological Review* 4 (1971) 37–50; A. Sjöberg, "Phosphate Analysis of Anthropic Soils," *JFA* 3 (1976) 447–454; W. I. Woods, "The Quantitative Analysis of Soil Phosphate," *AmAnt* 42 (1977) 248–252.

- 5. Organic matter is not the same substance as organic carbon. Organic matter includes fresh plant and animal residues, humus, and inert carbon forms such as charcoal, coal, and graphite. To convert the organic-carbon content to the organic-matter content, one traditionally multiplied the organic-carbon value by 1.724 as reported in J. W. Read and R. H. Ridgell, "On the Use of the Conventional Carbon Factor in Estimating Soil Organic Matter," *Soil Science* 13 (1922) 1–6. F. E. Broadbent, "The Soil Organic Fraction," *Advances in Agronomy* 5 (1953) 153–183, reports that this factor is not accurate, because the ratio of organic carbon to organic matter varies from surface horizons to subsurface horizons. A carbonate is any mineral containing a fundamental anionic unit of (CO<sub>3</sub>-2). The most common mineral form of carbonate is calcite, (CaCO<sub>3</sub>), or calcium carbonate, which is the major chemical substance of invertebrates' shells.
- 6. W. O. Robinson, "The Determination of Organic Matter in Soils by Means of Hydrogen Peroxide," *Journal of Agricultural Research* 34 (1927) 339–356.
- 7. F. E. Broadbent, "Organic Matter," in C. A. Black, ed., *Methods of Soil Analysis* (American Society of Agronomy: Madison, Wisconsin 1965) 1397–1400; W. E. Dean, Jr., "Determination of Carbonate and Organic Matter in Calcareous Sediments and Sedimentary Rocks by Loss-on-Ignition: Comparison with other Methods," *Journal of Sedimentary Petrology* 44 (1974) 242–248.

amount of carbon dioxide evolved.8 Carbonates are analyzed most often by determining the amount of carbon dioxide (CO<sub>2</sub>) evolved when an acid is introduced to the sample,9 or by measuring the weight loss of a sample after treatment with acid or burning at a specific temperature. 10 The loss-on-ignition method described in this paper, following a procedure described by Dean, 11 can measure both organic matter and carbonate with reliable accuracy.

# Theory of Loss-on-Ignition Method

When a dried powdered sample is heated, the organic matter (O.M.) contained in the sample will begin to ignite at 200°C and finish burning when the temperature reaches 550°C. When carbonate is ignited, the evolution of carbon dioxide (CO<sub>2</sub>) gas begins at 800°C and is completed when 850°C is reached. A simple mechanism for measuring the amount of these components in a sample is to measure the weight loss before and after a burn of 550°C and 1000°C. The percentage of weight lost after the two burns constitutes the determination of the percentage of organic matter and CO<sub>2</sub>, respectively, in the sample. The carbon-dioxide percentage can be used to calculate carbonate-ion concentration.

#### Procedure

- 1) A sample collected from an archaeological site is powdered with a mortar and pestle.
- 2) Ca. 3-5 grams of sample is placed in a size "O" (10 ml) ceramic crucible. The crucibles should be weighed to four decimal places after burning at 1000°C to clean them.
- 8. L. E. Allison, "Wet-combustion Apparatus and Procedure for Organic and Inorganic Carbon in Soil," Soil Science Society of America Proceedings 24 (1960) 36-40; L. E. Allison, W. B. Bollen, and C. D. Moodie, "Total Carbon," in Black, ed., op. cit. (in note 7) 1346-1366; J. C. Van Moort and D. De Vries, "Rapid Carbon Determination by Dry-combustion in Soil Science and Geochemistry,' Geoderma 4 (1970) 109-118; L. E. Allison, "Organic Carbon," in Black, ed., op. cit. (in note 7) 1367-1378; A. Walkley and I.A. Black, "An Examination of the Degtjareff Method for Determining Soil Organic Matter and a Proposed Modification of the Chromic Acid Titration Method," Soil Science 37 (1934) 29-38.
- 9. L. E. Allison and C. D. Moodie, "Carbonate," in Black, ed., op. cit. (in note 7) 1379-1396.
- 10. O. K. Galle and R. T. Runnels, "Determination of CO2 in Carbonate Rocks by Controlled Loss-on-Ignition," Journal of Sedimentary Petrology 30 (1960) 613-618; J. G. Konrad, G. Chesters, and D. R. Keeney, "Determination of Organic- and Carbonate-carbon in Freshwater Lake Sediments by a Micro-combustion Procedure,' Journal of Thermal Analysis 2 (1970) 199-208; W. N. Waugh and W. E. Hill, Jr., "Determination of Carbon Dioxide and Other Volatiles in Pyritic Limestones by Loss on Ignition," Journal of Sedimentary Petrology 30 (1960) 144-147.
- 11. Dean, op. cit. (in note 7).

- 3) The crucible (with sample), resting in a fire-brick holder, is placed in a drying oven and heated at 90°-100°C for one hour to dry the sample completely.
- 4) The crucible is transferred to a desiccator until it equilibrates with room temperature and is then weighed. This weight is the dry weight of the sample used in all calculations.
- 5) The crucible is placed in a preheated 550°C muffle furnace for exactly one hour. The sample is then removed from the furnace and cooled to room temperature (in the desiccator) and then reweighed. The difference between the weight when dry and the weight after the 550°C burn represents the weight of organic material in the sample.
- 6) The sample is then returned to the muffle furnace, now preheated to 1000°C, for an additional hour. Preheating the furnace to this temperature can sometimes take 3-4 hours depending on the type of furnace used. Once again the sample is cooled in a desiccator and weighed. The weight loss between the 550° and 1000°C represents the amount of CO2 evolved from carbonate minerals in the sample. If we assume that the sample's carbonate component is composed exclusively of calcium carbonate, we can convert the CO<sub>2</sub> to CaCO<sub>3</sub> by dividing the weight by 0.44, the fraction of CO<sub>2</sub> in CaCO<sub>3</sub>. If archaeologists are using the carbonate measurement to quantify shell density or limestone, then the assumption is valid.

# Accuracy of the Method

As with most simple laboratory techniques, the losson-ignition method does have a few shortcomings. The method does not differentiate between carbonate minerals (e.g., calcite or dolomite); the weight loss reflects the amount of carbon dioxide in a sample, which in turn must be converted to carbonate percentages assuming an initial carbonate mineralogy. When clay minerals are present, the water in their lattice structure is removed upon heating between 550° and 1000°C with a resulting weight loss. The amount of water driven off will be identified incorrectly as evolved carbon dioxide, especially in samples that are low in carbonate and high in clav.

Dean's experiments indicate that a sample with no carbonate but containing a large amount of clay will have a 1.0-1.8% loss in weight when burned between 550° and 1000°C. Samples containing 100% clay could yield a 5% weight loss. Dean suggests that for samples with a relatively high clay content, the assumption that the ignition loss between 550° and 1000°C represents CO<sub>2</sub> loss from carbonate will be in error by an amount that is directly proportional to the amount of clay present,



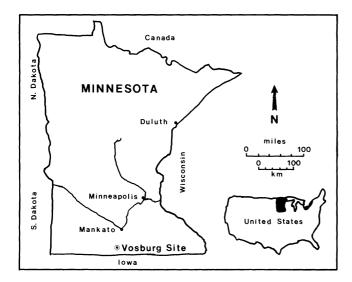


Figure 1. Location of the Vosburg Site in south-central Minnesota.

and inversely proportional to the amount of carbonate present. <sup>12</sup> If a sample has very low clay content, the loss-on-ignition technique reflects the carbonate content in the sample with an accuracy greater than 99%.

# **Example of Loss-on-Ignition Method Applied to Archaeological Research**

Samples from an Oneota site in southern Minnesota can be used to demonstrate the application of the loss-on-ignition method in archaeological research (FIG. 1).<sup>13</sup> The Vosburg site overlooks the Blue Earth River in Blue Earth County, 3 km sw of Winnebago in south-central Minnesota. Cultural remains cover an area of 12 acres. Most of the site has been homogenized within the plow zone. Radiocarbon dates ranging 605–1100 A.C. probably represent a number of occupations over that period.

Excavation was conducted by the University of Minnesota and the Science Museum of Minnesota under the direction of Drs. Guy Gibbon and Orrin Shane.<sup>14</sup> The

# 12. Ibid. 244.

- 13. The data reported here were collected while the author participated in the Blue Earth Valley Research Project during the summer of 1979. I provided an interpretation of the regional and local geological context of the Blue Earth Phase archaeological sites in Faribault and Blue Earth Counties. Results of the work are available in J. K. Stein, "Regional and Local Geological Context of Blue Earth Phase Archaeological Sites in Faribault and Blue Earth Counties," manuscript on file, Department of Anthropology, University of Minnesota (Minneapolis 1980).
- 14. Results of the Blue Earth Valley research are the subject of a dissertation being completed by C. A. Dobbs, Department of Anthropology, University of Minnesota, and can be found in C. A. Dobbs

subsurface material of the site, composed of gravelly outwash, was deposited sometime after 14,000 years ago from melting of the Des Moines ice lobe. This lobe originated in the Winnipeg Lowland, flowed south along the Red River Lowland and continued SE along the axis of the Minnesota River Lowland. During its advance, limestones, shales, and sandstones were incorporated into the ice. <sup>15</sup> The outwash at the Vosburg site contains granules of limestone, siliceous shale fragments, and pieces of sandstone. Two strata are observed in the site. A coarse sand is exposed at the surface continuing to a depth of 30–50 cm. A much more gravelly stratum underlies the sand consisting of alternating bands of gravel and sand.

These geological conditions have promoted the development of well drained, organically rich prairie soils. The soils are classified by the Soil Conservation Service<sup>16</sup> as part of the Dickenson Series, excessively well drained soils associated with the rolling landscape of southern Minnesota. Subsurface layers are often coarsely textured with occasional pockets of silty material. Subsurface horizons usually extend 0–20.3 cm (A horizon), 20.3–58.4 cm (B horizon), and 58.4–? cm (C horizon).

The soil at the Vosburg site closely resembles the representative profile of the Dickenson series except for its thicker A horizon (0–35 cm). Debris from human occupation has accelerated the buildup of organic material at the surface. The B horizon also extends further below the surface (65 cm), possibly because of the permeability of the parent material.

Pits were the predominant subsurface features encountered in the excavation. At least 67 pits were uncovered in an excavation covering 120 sq m. In any one excavation unit  $(2m \times 2m)$  determining the number of pits and their order of intrusion was often impossible. So many pits were discovered that Shane suggested that the site may have served for hundreds of years as a storage area for prehistoric people because the sediment pro-

and O. C. Shane III, "Oneoto Settlement Patterns in the Blue Earth River Valley, Minnesota," manuscript available at Department of Anthropology, University of Minnesota, Minneapolis, or Science Museum of Minnesota, St. Paul.

<sup>15.</sup> C. L. Matsch, "Quaternary Geology of Southwestern Minnesota," in P. K. Sims and G. B. Morey, *Geology of Minnesota: A Centennial Volume* (University of Minnesota Press: Minneapolis 1972) 548–560; H. E. Wright, Jr., C. L. Matsch, and E. J. Cushing, "Superior and Des Moines Lobes," in R. F. Black, R. P. Goldthwait, H. B. Willman, eds., *The Wisconsin Stage* (Geological Society of America: Boulder, Colorado 1973) 153–185.

<sup>16.</sup> P. O. Paulson et al. *Soil Survey of Blue Earth County, Minnesota*, U.S.D.A., Soil Conservation Service in cooperation with the University of Minnesota State Agricultural Experiment Station (St. Paul 1978).

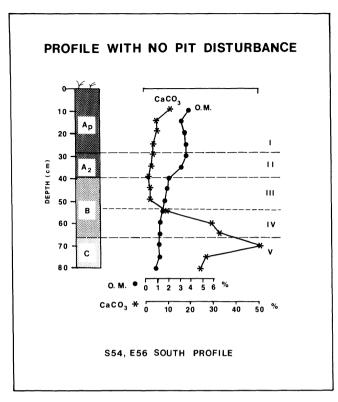


Figure 2. The organic-matter (O.M.) and carbonate (CaCO<sub>3</sub>) percentages determined for samples from a control profile; i.e., a portion of the archaeological site that contained no evidence of pit disturbance. Ap refers to plow zone. A2, B, and C refer to standard soil horizons. Roman numerals on the right side of the diagram differentiate the profile into zones for discussion purposes.

vided a quickly drained subsurface that could be easily excavated.17

At the Vosburg site occupation and intrusion of pits has introduced excessive amounts of organic matter to the substrate, masking the lower horizons of the solum and signifying the occurrence of A-horizon material at depths below its natural stratigraphic position. The losson-ignition method was utilized to measure the organicmatter content.

The carbonate content of the soils at the Vosburg site is inherited from the calcareous drift in which the site has formed. The carbonates, composed predominantly of limestone (CaCO<sub>3</sub>), are confined stratigraphically to the B and C horizons. To identify the position in the substrate of original B and C horizon material, often masked pervasively by organic matter, the loss-on-ignition procedure for carbonates was used.

A control profile, sampled at a location without pit disturbance, reveals the chemical composition of each horizon at the site (FIG. 2). Near the surface (zone I), carbonate content is less than 10% and organic matter is between 3-4%. In zones II, III, IV, and V, the organicmatter percentages decline. In zone V, the carbonate level rises sharply to values exceeding 20%.

An undisturbed profile such as seen in Figure 2 is extremely rare at the Vosburg site. Most profiles exhibit evidence of disturbances usually in the form of prehistoric pits. But not all pits are characterized by the same stratigraphy. Some display well defined layering similar in color and dimension to either an inverted or normal soil profile. Others, characterized by uniformly dark colors, are middens with abundant animal and plant remains. Several profiles display mottled coloration with a small number of artifacts mixed throughout.

The various types of pits can be distinguished by examining differences in sediment colors in the pit. But the identification of the pit contents and the processes responsible for pit filling are very difficult to determine from visual inspection alone. By quantifying organicmatter and carbonate content, the mode of sediment deposition and sediment source can be reconstructed more accurately.

### Chemistry of Three Pits of the Vosburg Site

Each pit was selected because its boundaries were distinct with no subsequent intrusion of other pits.

# Pit A

Samples taken from a pit with a bell-shaped outline were analyzed for their organic-matter content and carbonate percentages (FIG. 3). The stratigraphy in the profile indicated a reversal of the A, B, and C soil horizons as defined by the control profile. Zone I has a high organic-matter percentage (average 3.5%) and low carbonate value (6.5%) characteristically similar to an A horizon. Zone II is low in organic-matter (less than 2%) and high in carbonate percentages (greater than 20%), indicative of a C horizon. One sample, 40 cm below the surface, contains either A-horizon material or organic refuse that was thrown in during the filling process. Zone III has variable organic-matter content (1-4%) and low carbonate content (less than 10%), equivalent to the inverted B and A horizons. Another anomaly is seen at 100 cm depth with increased carbonate percentage. This sample consists of limestone gravel that was mixed with the A and B horizon. It was not observed in the profile because of its dark organic stains. Zone IV is the C horizon of the undisturbed solum.

The chemical data support the visual field interpretation that the stratigraphy constitutes an inversion of A,

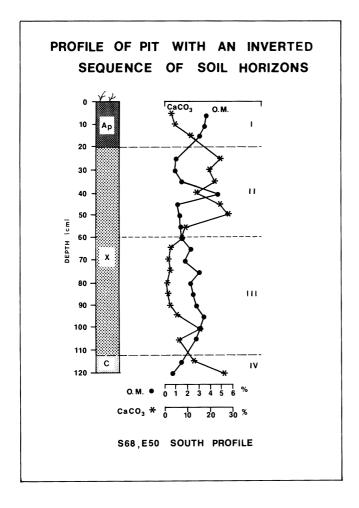


Figure 3. The organic-matter (O.M.) and carbonate (CaCO<sub>3</sub>) percentages determined for samples from a profile intersecting a subsurface pit. The stratigraphy of the pit suggests an inverted sequence of standard soil horizons, with the highest carbonate percentages in zone II. Ap refers to plow zone; X refers to archaeologically altered horizons; C refers to the standard soil horizon. Roman numerals on the right side of the diagram differentiate the profile into zones for discussion purposes.

B, and C soil horizons capped by another more recent A horizon (plow zone). The organic-rich deposit at the base of the bell-shaped pit was probably not derived from cultural debris because it contains few artifacts or ecofacts. The pit could have been filled by first dumping in material from the A horizon (possibly a mass of inorganic sediment held together by roots of prairie grass and the plant itself), then mixing with sediments from the B horizon followed by matrix from the gravel-rich C horizon. On the other hand, the organic-rich zone could have been produced by the decomposition of organic material such as the cultigens that were historically stored in such bell-shaped pits. The overlying sediment was then either thrown or washed in over the food debris.

#### Pit B

The second pit has no visually discernible layering. The organic-matter and carbonate percentages (FIG. 4) indicate only three horizons; an A horizon capping the pit, a C horizon below the pit, and a third intervening horizon composed of a mixture of A, B, and C horizons with the inclusion of cultural material. In Figure 4, zone I has an average organic-matter percentage of 4.0% and a carbonate average of 4.2%, values slightly above the range of an A horizon. In zone II, a black deposit, the chemical percentages are extremely variable with organic-matter percentages ranging from 1.5% to 4.4% and carbonate values from 2.9% to 27.4%. In zone III, the C horizon, average organic-matter content is 1.2% and the carbonate content averages 31.4%.

Close examination of zone II reveals subtle stratification. Lenses of black-stained gravel, concentrations of white ashy material, charred bones, and charcoal are discernible. Artifact density is greater in this pit than in any other.

The analytical data suggest that this pit is filled with midden and mixed with inorganic sediment from either B or C horizons. Subtle lenses within the fill represent dumping episodes. High percentages of carbonate signify areas where C-horizon material was mixed with occupational debris. Without chemical analysis, the source of the matrix would not be identifiable because of the masking effect of organic matter.

#### Pit C

The third pit illustrates a combination of processes (FIG. 5). Visually the pit is filled with midden overlain by a lightly colored gravel layer, which is overlain in turn by a dark layer. The chemical analysis indicates that zone I has characteristics of an A horizon, with an organic-matter average of 3.7% and carbonate content of 6.1%. The lowest zone (VI) records C-horizon values (organic matter-0.7%, carbonate-11.5%). The composition of the intervening zones suggests the operation of two different processes. Zones III-V are mixtures of midden and inorganic material from A and B horizons (carbonate levels are too low to be from a C horizon). Some artifacts were found within these zones. Above this midden is a layer (zone II) with chemical characteristics of a C horizon (average organic-matter values of 1.0% and carbonate of 17.8%, with almost no artifacts).

At some point the pit was excavated and partially filled with midden. Later, material derived from a C horizon was deposited in the pit. The source of the C-horizon sediment may have been the pit's own backdirt that was spread around the pit's opening, or material from another

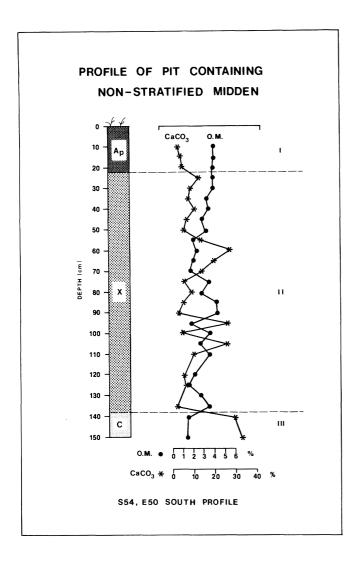


Figure 4. The organic-matter (O.M.) and carbonate (CaCO<sub>3</sub>) percentages determined for samples from a profile exposing a nonstratified midden. The stratigraphy is dominated by zone II where sediment from A, B, and C soil horizons has been mixed with inclusions of cultural materials. See caption for Figure 3 for description of abbreviations.

adjacent pit. The zone-II sediment was probably thrown into the pit purposefully because the horizontal banding of the gravel (not angled parallel to the paleo slope of the pit's outline) precludes natural slumping as the mechanism of infilling. The processes responsible for filling this pit appear to be a combination of midden deposition and inclusion of natural soil horizons.

# Potential for the Loss-on-Ignition Method

Some of the interpretations proposed for the three pits can be derived solely from visual observation. Additional chemical information, provided by the use of the

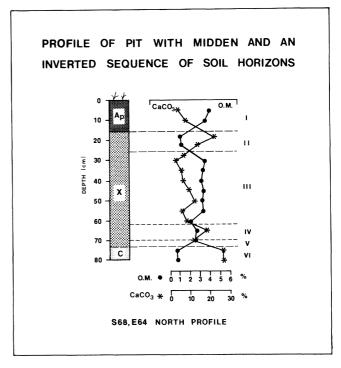


Figure 5. The organic-matter (O.M.) and carbonate (CaCO<sub>3</sub>) percentages determined for samples from a profile exposing midden and other disturbed zones. Zones III through V are midden, capped by zone II, a remnant of a C soil horizon transported from a nearby area. See caption for Figure 3 for description of abbreviations.

loss-on-ignition method, enhances the interpretation of pit stratigraphy where visual evidence is lacking. The source of the inorganic matrix of the midden can be assigned either to the A, B, or C soil horizons, or a combination of one or more horizons.

This example has illustrated only one way that the quantification of organic matter and carbonate can contribute to archaeological interpretation. The dark brown color of organic matter masked the objects and sediments in the Oneota refuse pits. To identify the sediment's source and the formational processes of the pits, the organic matter and carbonate needed to be quantified. The loss-on-ignition method provides an efficient, inexpensive means of analyzing organic-matter and carbonate content. These compounds are evidence that provide, along with lithics, ceramics, and bones, information to archaeologists regarding the behavior of prehistoric people.

# Acknowledgments

I wish to acknowledge the Science Museum of Minnesota and Dr. Orrin Shane for providing the material on which this research is based and Timothy Ready, Clark Dobbs, and Dr. Guy Gibbon for their aid during the excavations of 1979. I wish to thank S. E. Chernicoff, C. W. Beck, and F. A. Hassan for their helpful comments in the preparation and editing of this manuscript, and K. Kluge for drafting of figures.

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# **Announcements**

# **ICAZ Announces International Conference**

The Fifth International Conference on Archaeozoology will be held in Bordeaux, France between the 25th and 29th of August, 1986. This conference is sponsored by the International Council for Archaeozoology and is being organized by Pierre Ducos and his French colleagues with the support of various local and national French academic, research, cultural, and political bodies. There is no specific theme for the conference other than "taking stock of world archaeozoological research." Individual contributions and ideas for symposia are solicited so long as they fall within the definition of archaeozoology as "the study of animal remains connected with the settlements of ancient human groups, thus contributing to the knowledge of those groups." For registration forms please write to the organizer: Dr. Pierre Ducos, Ve Conférence ICAZ, C.R.E.P., St. André de Cruzières, France.

Previous International Conferences on Archaeozoology were held in Budapest (1971), Groningen (1974), Szczecin (1978), and London (1982). The last conference attracted more than 230 researchers from 32 countries including 41 from the U.S.A. The proceedings of that London conference are currently being published in

four volumes by British Archaeological Reports (Oxford) under the title *Animals and Archaeology*. The first volume, subtitled *Hunters and Their Prey*, has already appeared in *BAR International Series* 163 (Oxford 1983).

The International Council on Archaeozoology was formed in Groningen in 1974 in order to promote the study of and standards in the study of faunal remains from archaeological sites. Corresponding membership is available to anyone interested in archaeozoological or related research by writing the General Secretary: Dr. A. T. Clason, Biologisch-Archaeologisch Instituut, Poststraat 6, 9712 ER Groningen, The Netherlands.

# Association for Preservation Technology Conference

The Association for Preservation Technology is an international organization dedicated to the science and technology of the preservation of historic resources. Its members include architects, engineers, contractors, craftsmen, educators, and administrators. The APT Annual Conference will be held 19–23 September, 1984, in Toronto, Canada. The conference will address the issue of "Principles in Practice" by stimulating awareness of the technological consequences of particular philosophical stances in architectural conservation. Information regarding APT and the conference is available from Mr. Herb Stovel, Program Director, 77 Bloor Street West, Toronto, Ontario, Canada M7A 2R9 (Telephone [416] 965-5727).

# New Officers of the Asociación Venezolana de Arqueología

The annual meeting in October 1983 of the Venezuelan Association for the Advancement of Science (AsoVAC) was the scene for the election of the following new officers of the Asociación Venezolana de Arqueología (AVA) for 1983–1984. President Elect, Alberta Zucci; Past President, Fulvia Nieves; Secretary of Publications, Erika Wagner; Scientific Secretary, Kay Tarble; Treasurer, Miguel Angel Perera; Secretary of Conservation, Lilliam Arvelo. Also elected were the following to the Committee on Ethics: José M. Cruxent, J. Eduardo Vaz, and Victor Nuñez-Regueiro.

Information regarding AVA may be requested from Ms. Wagner at

Asociación Venezolana de Arqueología Apartado 1827 Departamento de Antropología, IVIC Caracas 1010A, Venezuela.