CHAPTER 3

STRATEGIES AND METHODS FOR SOURCING STONE AND METAL ARTIFACTS

CHAPTER INTRODUCTION: STRATEGIES AND METHODS

In order to address the lines of inquiry outlined in Chapter 1, it is necessary to know precisely what kinds of rocks and minerals are present in Harappa's artifact assemblage and, for select varieties of those materials, to ascertain which geologic sources they were most likely acquired from. The purpose of this chapter is to review the various research strategies and methods that were employed to accomplish those two tasks.

Reeves and Brooks (1978) outlined a series of steps (Figure 3.1) for successfully determining the geologic provenience of rock and mineral artifacts, which will serve as a guide for the presentation of this chapter. I begin with a discussion of the importance of utilizing the extensive body of geologic literature relating to South Asia as the primary reference source for locating the natural occurrences of the different rock or mineral types being investigated (Step A). Emphasis is also placed on the benefits of directly working with geologists. The discussion then shifts to the geologic field studies that were necessary for both collecting a representative range of geologic samples from each potential source area (Step B) and for confirming or refuting the existence and/or nature of certain rock and mineral occurrences. I then move on to the issue that ultimately underlies the success or failure of any stone or metal artifact sourcing study – demonstrating that the chemical, isotopic or mineralogical variability *between* the different geologic sources under examination is greater than the variability *within* any individual source (Step C). Many factors contribute to successful source discrimination, such as the choice of sampling strategies (discussed in relation to Step B) and analytical methods (discussed in relation to steps D and E). One issue, which is often not given due attention, relates to the selection of the appropriate geographic scale on which to define stone or metal "sources." After examining the issue of scale and the expectations of provenience resolution

Figure 3.1 Steps for successful determining the geologic provenience of rock and mineral artifacts (adapted from Reeves and Brooks 1978: 364-365).

A. Locating the natural occurrences of the rock or mineral type being investigated.

B. Collection of a representative range of samples from each potential source area.

C. Demonstrating that the chemical / isotopic / mineralogical variability between different geologic sources is greater than the variability within individual sources.

D. Establishing a set of analytical parameters that will allow geologic sources to be distinguished from one another with a high degree of confidence.

E. Analysis of rock and mineral artifacts and assignment to a probable geologic in accordance with the criteria established in D.

stemming from it, I proceed on a series of overviews outlining the various methods used to identify and/or characterize archaeological and geologic samples for this study. Also discussed are the analytical methods that were chosen to evaluate the quantitative data obtained from the characterization of selected varieties of stone or metal. Through the application of these methods, parameters were established that allow different geologic sources to be distinguished from one another with a high degree of confidence and artifacts to be assigned to them based on their chemical/isotopic/mineralogical properties (steps D and E).

In the conclusion of this chapter, I stress that statements regarding the geologic provenience of stone or metal artifacts are always *provisional*, regardless of how comprehensive or statistically significant the datasets they are based on may appear to be.

RESEARCH STRATEGIES

In this section the research strategies that were used to identify potential rock and mineral sources, assemble a collection of geologic samples for analysis and to define suitable scales of provenience resolution are discussed.

Using primary geologic reference materials to locate potential sources

There have been several major broad-scale studies (Fentress 1976; Lahiri 1992; Ratnagar 2004) of Harappan trade networks that examined multiple varieties of stone and metal to construct models of proto-historic resource access and exchange. However, to identify the rock and mineral sources that were potentially used in the past, those researchers relied heavily upon colonial-era British Government district gazetteers and secondary references such as the source identifications suggested or cited by the writers of early excavation reports. Consequently, their interpretations have serious limitations due to the imprecise, incomplete and occasionally spurious nature of their principal reference materials. I am not suggesting that those types of sources are always wrong or have no value and should be ignored. On the contrary, the reports of late 19th and early 20th century civil servants and archaeologists are sometimes the sole source of information on certain mineral deposits. I myself frequently cite these references throughout this book. However, they alone do not provide a comprehensive picture of South Asian rock and mineral resources and, because their writers usually did not visit geologic occurrences themselves, misidentification of source locations and of the materials themselves could easily have been made. For these reasons, literature of this kind should not be considered the "best sources" (Possehl 1999: 173) to turn to for primary reference material when delineating potential resource areas for rock and mineral artifact provenience studies.

A substantial body of scholarly literature relating to the geology of South Asia exists and was accessed for this study as the *primary reference material* for locating potential sources of the rock and mineral artifacts found at Harappa and other sites. Among the most useful publications were those produced by national government agencies such as the Records, Memoirs and Bulletins put out by the Geological Survey of India and the Geological Survey of Pakistan. Publications by state agencies, such as the Department of Mines and Geology, Government of Rajasthan were also valuable sources data. Dozens of university geology departments in both India and Pakistan regularly publish journals, conference proceedings and books detailing the geologic resources of the state or region that they represent. For decades institutions such as Pakistan's *Centers of Excellence* in Geology (Peshawar) and in Mineralogy (Quetta), the Geological Society of India (Bangalore) and the Wadia Institute of Himalayan

Figure 3.2 The author conducting fieldwork in Balochistan, Pakistan. Clockwise from top left - Consulting topo sheets in Muslimbagh, with levies in the Kanrach Valley, sampling steatite at Ugasai Nasir, and collecting bitumen in the Bolan Pass.

Geology (Dehra Dun) have conducted and published groundbreaking geologic research. Public reports produced by organizations like the Gujarat Mineral Development Corporation (GMDC) and the Federally Administered Tribal Areas Development Corporation (FATADC) are also excellent sources of data on mineral resources in those areas. Newer geologic overviews (e.g. Bender and Raza 1995; Kazmi and Jan 1997; Ramakrishnan and Vaidhyanadhan 2008) have been published within the last decade or two that are far more accurate and comprehensive than earlier ones. Lastly, the unique nature of the Subcontinent's geology has attracted researchers from around the world who have collaborated with their South Asian colleagues and published their results in a wide range of international journals.

Field-checking and sampling potential Harappan rock and mineral sources

After thoroughly searching the geologic literature in order to identify the potential sources of rock and mineral artifacts found at Harappa, the next task was

Figure 3.3 **[A]** Dr. S.R.H. Baqri (Pakistan Museum of Natural History) in the Rohri Hills, Sindh. **[B]** The sample-laden truck during my fieldwork with Dr. Baqri. **[C]** Dr. Khalid Mahmood (Centre of Excellence in Mineralogy, University of Balochistan) at the Tor Tangi steatite mine, Zhob District, Balochistan. **[D]** Khawar Akbhar (Geological Survey of Pakistan-Karachi) near Duddar, Las Bela District, Balochistan.

to visit those sources and to obtain samples from them for use in comparative analyses. This could only be accomplished by implementing a strategy of extensive geologic field work (Law 2008b). In a region as vast and diverse (geographically, geologically, culturally and politically) as northwestern South Asia this was an enormous undertaking, but one that was absolutely essential to the success of my research (Figure 3.2). I realized early on that I would need to work in close collaboration with Pakistani and Indian geologists (Figure 3.3). The first and most obvious reason is because geologic materials (their properties,

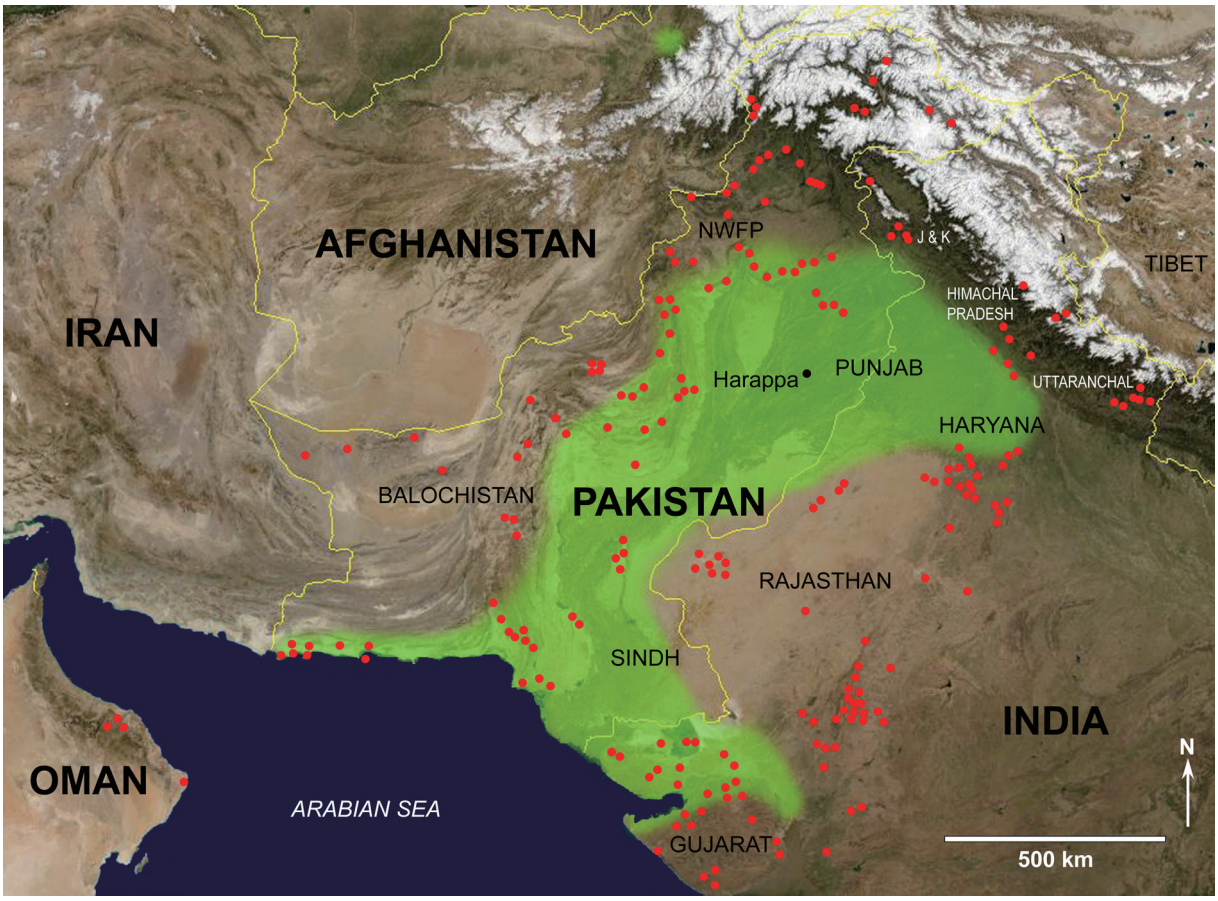

Figure 3.4 Locations (indicated by red dots) sampled ca. 2000-2010.
The green shaded area represents the approximate extent of the Indus Civilization (ca. 2600-1900 BC).

the processes that create them and their economic uses) are the focus of their discipline. Field geologists know their research areas intimately and, in the course of their surveys, often see and note old mines, working areas and sometimes even archaeological sites. Geochemists and economic geologists can provide valuable insights into the nature, variability and locations of mineral deposits and it is often the case that source samples for initial analyses can be acquired from their existing collections. Finally, the disciplines of archaeology and geology share many common features (stratigraphy, time depth, change) and most geologists that I had the privilege to work with have had a great personal interest in the human past.

Since the year 2000, I have visited and/or acquired samples from approximately 200 potential Harappan stone and metal sources in Pakistan, India and Oman (Figure 3.4). From most of them, I was able to collect a representative range of materials so that intra-source and extra-source macroscopic, compositional and/or isotopic variability could be satisfactorily assessed. Sampling strategies varied depending on the type of rock or mineral being studied (details on individual varieties are provided in upcoming chapters) and on the geographic extent over which a "source" occurred (an issue discussed in the next section). In most instances, a minimum of 20 to 25 samples per source were obtained, which is generally considered to be an amount sufficient for making statistically meaningful assessments and comparisons (Malyk-Selivanova *et al.* 1998: 667; Truncer *et al.* 1998: 25). Although no formal collection procedures were employed (such as the laying out of a transect or grid across a deposit and taking samples at predetermined or random intervals), with the help of my colleagues in the geosciences, a concerted effort was made to collect samples that were representative of a deposit, both spatially and

in terms of the full range of macroscopic varieties present in each locality. Whenever possible (and again depending on the type of material), samples were removed from fresh exposures using a geologic hammer rather than taken from loose contexts such as surface scatter or mine tailings.

Field surveys were also essential for reasons that went beyond just compiling a collection of geologic comparative materials. Misidentification/misrepresentation of a rock or mineral deposit sometimes happens, even in the geologic literature. By personally visiting a reported occurrence I was able to confirm or refute its existence and/or to clarify the nature of the material found there. My survey of steatite sources (Chapter 7) illustrates the benefits of employing this strategy. Deposits of steatite were reported in the northern part of the Zhob District, Balochistan at two locations (Ahmad 1975: 135). When I visited the region I found that it was actually chlorite and serpentine, rather than steatite, which occurred at those locations. Field-checking also helped to clarify the nature of steatite deposits worked along the margins of the Peshawar Valley at Jamrud (Abbas *et al*. 1967) and Kund (Qaiser *et al*. 1980). Visits to those deposits revealed that the materials occurring there were of an extremely low-grade and not at all of the quality Harappans used for manufacturing purposes. Lastly, in the course of a field survey previously unpublished sources may be identified, as was the case when I visited several unreported steatite mines in the Las Bela District of southern Balochistan. Although these deposits were well known locally and had apparently been worked for quite some time, to my knowledge no direct reference to them had ever appeared in print.

Tapping other sources of information: Johris, pansaris and pattarwalas

During my travels across South Asia I picked up a great deal of useful information regarding the sources and uses of rocks and minerals from various *johris*, *pansaris* and people I broadly refer to as *pattarwalas*.

A johri is stone jewelry seller. Although in some instances a group of them have congregated in a permanent location, such as in Jaipur's famous "johri bazaar," in most cases they are individuals who, in advantageous temporary locations, have set up portable display cases (kabats) filled with rings, amulets, necklaces, prayer beads, as well as various loose beads, cabochons and miscellaneous bits of worked and unworked stone. It was from a johri named Sufkara Abbaas (Figure 3.5 A), who had his kabat set up in front of Abdullah Shah Ghazi's tomb in Karachi, that I learned an important steatite, serpentine and chlorite source area discussed by Vidale and Shah (1990) was located relatively close by that city and not, as reported (ibid), near the distant town of Turbat. Mr. Abbaas supplied me with a range raw materials and finished ornaments from the source area, which, using the information he provided, I shortly afterwards visited myself (discussed in Chapter 7). It was during my first research trip to Pakistan in 2000 that Mark Kenoyer showed me the usefulness of talking with johris (Figure 3.5 B) and since then I have rummaged through kabats in places as far flung as Khairpur, Agra and Islamabad (Figure 3.5 C, D & E).

Pansaris are purveyors of traditional "medicinal herbs, crude and refined inorganic medicinal preparations, as well as drugs of animal origin commonly used by the practitioners of indigenous medicine" (Singh 2001: 190). Because rocks and minerals are ingredients in many of their remedies, I made it a point to visit pansari shops in different regions of the study area (such as the one in Bannu, NWFP pictured in Figure 3.6 A) and question the proprietors about the uses and origins of those materials. Often times I would take a set of samples. Figures 3.6 B & C shows the owner of shop in New Attock City, Punjab Province, Pakistan and the group of rocks and minerals that I purchased from him. Many (but not all) of these same materials have been recovered in raw form at Harappa and,

Figure 3.5 Visiting johris.

[A] Mr. Sufkara Abbaas discusses a sample of Wayaro steatite in front of Abdullah Shah Ghazi's tomb in Karachi. **[B]** Dr. Mark Kenoyer looks through a johri's kabat in Karachi. **[C]** Mr. Ashiq Hussain, Khairpur, Sindh. **[D]** A johri in Agra, Uttar Pradesh. **[E]** The author looks through a johri's kabat in Islamabad.

thus, it possible some might have been for medicinal purposes. Pieces of galena (lead sulfide) purchased at this pansari shop and another in Karachi proved to be very informative comparative samples in my studies of Harappan lead artifacts (see Appendix 12.7).

I gathered a tremendous amount useful information from various individuals that I will collectively refer to here as *pattarwalas* or "stone people." Included in this category are the agate bead-makers or *akik-walas* of Khambhat, Gujarat (Figures 3.7 A & B), whose production techniques and material record were documented in detail by Kenoyer, Vidale and Bhan (1991, 1994). I was able to identify many of the same stones that Harappans used among their diverse stocks of raw materials and to learn, if not the exact locations, at least the approximate source areas for important types such as the hard-to-find black and white jasper (Figure 3.7 C). Wherever I went I sought out the carvers of millstones, querns and mortars (Figures 3.7 D & E). They not only provided information on the sources of the raw materials they used but also on the properties that, for grinding purposes, made stone from particular locations preferable to just any old

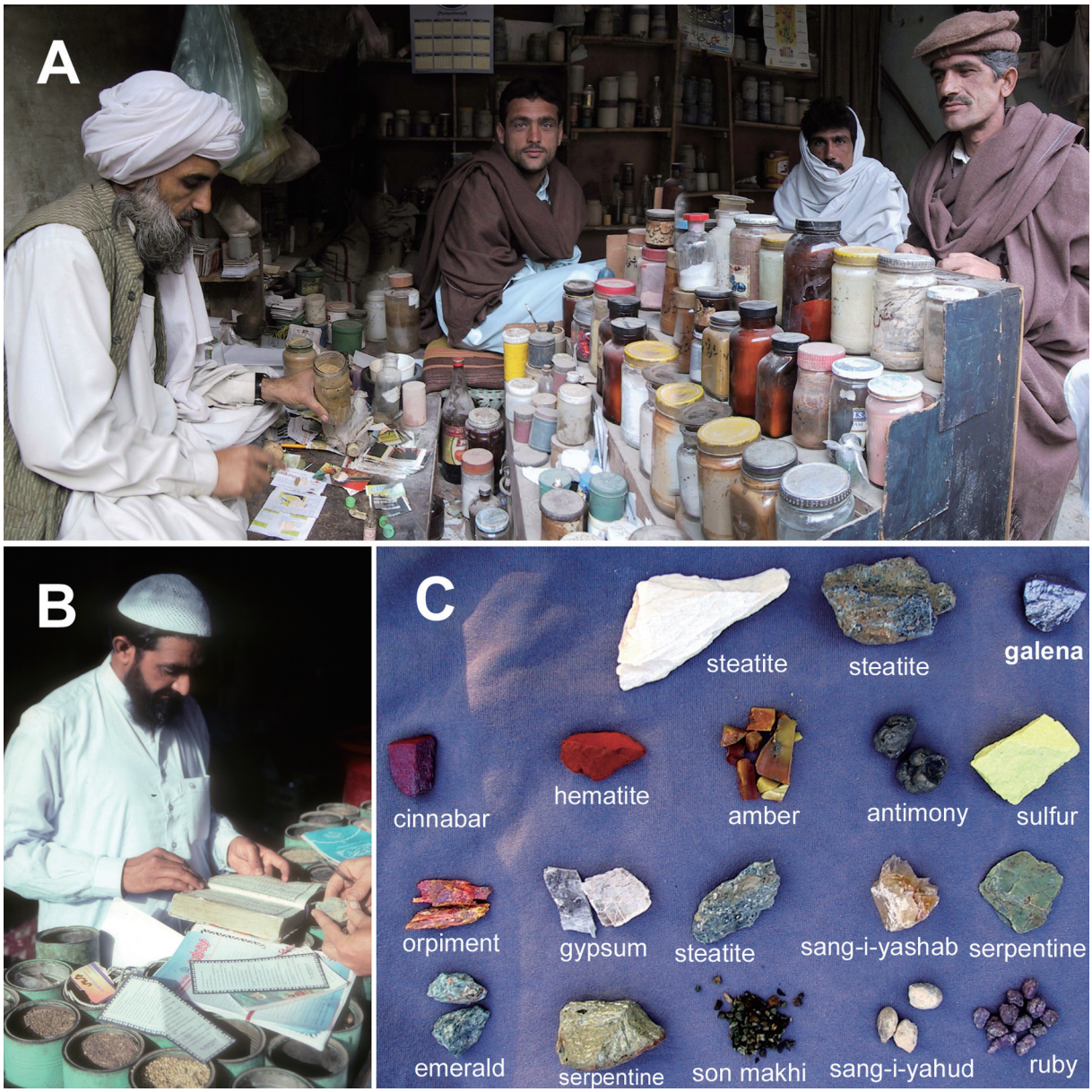

Figure 3.6 Visiting pansaris.

[A] A pansari prepares remedies at his shop in Bannu, NWFP. **[B]** A pansari in New Attock City, Punjab Province, Pakistan consults a manual of traditional medicines. **[C]** The set of medicinal rocks and minerals collected from the New Attock City pansari.

run-of-the-mill rock. Finally, I was fortunate enough to meet by chance individuals in many different places across South Asia (Figures 3.7 F, G & H) who had a deep interest in and knowledge of the geology of their local areas. These pattarwalas were most happy to show me their collections, share some samples, and even personally guide me occurrences of stone that I sought.

The "Provenience Postulate" and Defining a Geographic Scale of Provenience Resolution

Underlying the present study is the assumption known as the "provenience postulate" (Weigand *et al.* 1977: 24). This assumption holds that determining the source of a stone or metal artifact "is possible as long as there exists some qualitative or quantitative chemical or mineralogical difference between natural sources that exceeds the qualitative or quantitative variation within each source" (Neff 2000: 107-

Figure 3.7 Various pattarwalas.

[A] An agate bead-maker in Khambhat, Gujarat. **[B]** "Akik-wala" sign in Khambhat. **[C]** Bead roughouts of black jasper with white bands. **[D]** Millstone carvers in Agra, Uttar Pradesh. **[E]** A women carving querns and mullers, Katmandu, Nepal. **[F]** Mr. Aslam displays a specimen of fossiliferous limestone he collected in Las Bela, Balochistan **[G]** Mr. Bhagat Chang rummages through his collection of crystals from the Parvati Valley, Himachal Pradesh. **[H]** Mr. Malik collects hematite near Shin Kai, North Waziristan.

108). A successful outcome to provenience research is, therefore, largely dependent upon the natural properties of the rock and mineral sources being examined, adequate sampling of those sources (discussed above) and the application of suitable methods for both characterizing materials collected from them and analyzing the resulting data (discussed below). However, an additional factor that has great bearing on the success of a study is the consideration given to the geographic scale on which "sources" are defined and the expectations of provenience resolution that stem from the definition of this scale.

For example, certain types of stone occur as well-circumscribed bodies (outcrops/pockets/zones) of material that, individually, have highly distinctive chemical compositions. Obsidian – a volcanic glass for which the geologic proveniences of artifacts made from it can frequently be resolved down to the level an individual outcrop (Williams-Thorpe 1995), is probably the best example of this type. At the other extreme are materials that occur (either contiguously or intermittently) across broad geographic areas and are fairly homogeneous throughout. Cackler and others (1999) had difficulty differentiating individual chert outcrops in northern Belize because they were all, in essence, part of a single extensive geologic formation. Such a situation, when it occurs, need not always mean that the geologic provenience of an artifact is irresolvable. A "source" can be defined as either a single location or a collection of many locations in "geographic space" (Neff 1998: 116). Depending on the extent of the study area and the diversity of the geology within it, the scale at which a "source" is defined may be expanded to include materials sampled from multiple geologically related locations.

The current examination of Harappan acquisition networks involves numerous rock and mineral types and multiple scales of provenience resolution. The locations where gem-quality vesuvianite-grossular garnet (Chapter 9) and high-quality agate (Chapter 8) can be found are limited in number and in geographic size. Deposits of steatite (Chapter 7) and limestone (Chapter 11), on the other hand, are much more numerous and occur over extremely broad areas. Extensive sampling, characterization and analysis of the latter two materials have indicated that, in some instances, "sources" are best defined at a regional scale. For a material like steatite it may, in the end, only be possible to make a statement such as "the stone that these artifacts are composed of appears to have been derived from deposits located in the NWFP of northern Pakistan." For steatite artifacts from a site situated within that region, like Sarai Khola, this would provide little information other than the material was probably acquired locally. However, in terms of Harappa – a site for which potential steatite sources lay roughly 300 to 900 km away in all directions, this level of resolution is more than sufficient to provide valuable insights into the extent and directions of long-distance resource acquisition networks.

METHODS OF MATERIAL IDENTIFICATION AND CHARACTERIZATION

In this section, I review the various methods that were used to identify and/or characterize the archaeological and geologic materials examined for this study.

Visual inspection/comparison and basic mineralogical testing

The 56,000+ stone and metal artifacts recovered during HARP excavations were initially classified by rock or mineral type based on visual inspection by HARP co-director Dr. Mark Kenoyer, who has had nearly 40 years of experience examining lithic materials from archaeological sites in South Asia. For this study, I re-examined the majority of

these artifacts primarily to familiarize myself with the range of materials found at the site but also to locate artifacts for which initial identifications needed to be revised or clarified. Illustrated rock and mineral handbooks and field guides (Pellant 2002; Pough 1988) were especially valuable tools in this effort. Direct comparisons were made between artifacts and geologic samples that I had collected from sources around the Greater Indus region. In May of 2000, with the permission of the Director-General of Archaeology and Museums, Government of Pakistan, I assembled a "traveling" set of samples that contained 200 rock and mineral artifacts from Harappa (all of them small non-diagnostic fragments) representing the full range of material varieties and sub-varieties present at the site. These archaeological samples were compared to geologic samples in the extensive collections housed at the Geological Survey of Pakistan's museum in Quetta, the Department of Geology, University of Peshawar and the Pakistan Museum of Natural History in Islamabad. Numerous professional geologists from these institutions generously provided their expert assessments of the identities and the probable origins the various rock and mineral artifacts in the set. Their identifications enabled me to plan a comprehensive field survey for the purpose of collecting my own geologic samples for comparative analyses.

Simple, non-destructive mineralogical tests were conducted on a number of archaeological samples. The most common test used was that to determine a stone artifact's *density*, which "is a fundamental and characteristic property of each mineral and, as such, is an important determinative property" (Rapp 2002: 21). The density of a mineral is expressed as its *specific gravity* (SG) – the ratio of its weight to the weight of an equal volume of water. A Hanneman direct reading specific gravity balance was used at Harappa to make SG measurements on several hundred artifacts. Another basic test used was that of a mineral's *hardness*, that is, its resistance or susceptibility to abrasion (scratching) relative to ten minerals on an ordinal scale first developed by Friedrich Mohs in 1812 (Appendix 3.1). Mineral types that resembled one another could often be differentiated using a simple scratch test. For example, a translucent green flake composed of vesuvianite-grossular garnet (hardness ≈ 7) will scratch feldspar (hardness 6) and so can be easily be distinguished from an identical looking serpentine flake (hardness ≈ 4), which will not.

X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) analysis enables one to unambiguously determine the identity of crystalline substances (Henderson 2000: 10). Over 100 rock and mineral artifacts from Harappa were characterized using this technique, which involves bombarding a small amount of powdered rock sample with X-rays so as to cause the electrons within it to vibrate. The vibrating electrons reflect a portion of the X-ray radiation as waves that reinforce themselves in an effect called *diffraction* (Klein and Hurlbut 1977: 277). The patterns that the diffraction effects create are recorded and provide precise information about the atomic structure(s) of the mineral(s) within the sample. It is the only technique used here that can accurately distinguish between mineral *polymorphs* (minerals sharing the same chemical composition but having different crystal structures). For example, quantitative data on the abundance of silicon dioxide in a sample can be obtained using electron microprobe analysis (discussed below), but only with XRD is it possible to determine which polymorph (*quartz*, *tridymite* or *cristobolite*) it is (Henderson 2000: 11).

The majority of the XRD analyses made for this study were conducted at the at the S. W. Bailey X-ray Diffraction Laboratory, Department of Geoscience, University of Wisconsin-Madison on either a Scintag PADV X-ray diffractometer or a Rigaku Rapid II X-ray diffraction system. Diffraction data were output in digital form and interpreted using the program

JADE 6.0. Some analyses were made at the Center of Excellence in Geology, University of Peshawar on an older instrument that did not possess a computer interface. The XRD patterns were recorded on paper "strip-charts" and the peak positions and relative intensities had to be manually measured and recorded. These data were then interpreted using mineral phase search manuals published by the International Centre for Diffraction Data. Both the Madison and the Peshawar diffractometers were run at 40 kv and 30 ma and, for most samples, scans were run at a 2-theta angle from 5° to 65° with a .02° step size and a .25 second count time.

Using the old XRD in Peshawar and the Scintag XRD in Madison required that a small amount of material be ground to a fine powder for analysis. Thus, because it was a destructive method, only archaeological raw material debris fragments were analyzed using these instruments. In 2009, the Rigaku XRD was installed in Madison. With this instrument, artifacts can be directly X-rayed without powdering. This has permitted the mineralogical composition of a number of small artifacts to be non-destructively determined. The Rigaku XRD employs a molybdenum target and so the 2-theta values of spectra output by this instrument are different from those made using the Scintag XRD, which employs a copper target (the peak patterns are identical, however). In one instance, I modified the Rigaku-made spectra of steatite microbeads (Appendix 7.14 Figure 2) to be comparable to the Scintag-made spectra of experimentally heated steatite chips (Appendix 7.12 Figure 3). The remaining Rigaku-made XRD spectra included in this study (these are individually noted) are unmodified.

Electron microprobe analysis (EMPA)

Electron microprobe analysis (EMPA) is both a method with which to acquire compositional data on solid materials as well as a powerful micro-imaging tool (Reed 2005). Samples are affixed in epoxy within a tubular analysis cartridge and then a flat surface is ground, polished and given a thin carbon coating. Upon this surface the "probe" can focus a beam of electrons on an analytical area as small as 1 μm or micron (0.001 millimeter). This makes it a useful tool for examining rocks having multiple mineral phases and minute inclusions. Chemical characterizations can be done using either the energy dispersive spectrometry (EDS) or wavelength dispersive spectrometry (WDS) capabilities of the probe. EDS measures the X-ray energy emitted from the area under the beam of electrons and permits quick reconnaissance and qualitative chemical characterizations of materials (Lund 1994). WDS measures electrons diffracted by the crystal structure of the material under the beam (Lund 1995). When calibrated with known standards, highly accurate quantitative chemical data can be obtained using WDS. Micro-imaging of materials using backscattered electrons (BSE) works on the same principle as scanning electron microscopy. All EMPA of archaeological and geologic samples in this study was conducted under the direction of Dr. John Fournelle at Department of Geoscience, University of Wisconsin-Madison on either a Cameca SX50/51 electron microprobe or a Hitachi variable-pressure scanning electron microscope (VP-SEM) with EDS capability.

Spectrometric analysis

Spectrometric (spectroscopic) analysis includes many different methods and types of instruments (Pollard and Heron 1996: Chapter 2). Highly accurate data on the elemental composition of a substance can be collected by observing the spectrum of light emitted when the atoms composing it are excited (atomic emissions spectrometry). Elemental as well as isotopic data may be obtained by directly detecting ionized atoms that have been separated according to their mass-to-charge ratios (mass spectrometry). Excitation or ionization of a sample

for analysis can be achieved by various means but the use of an inductively-coupled plasma (ICP) torch is becoming increasingly common (Taylor 2000).

Spectrometric analyses were conducted on artifacts and geologic samples composed of alabaster, limestone and various metals (lead, silver and copper). Limestone and metals were analyzed using the two ICP spectrometers at the Laboratory for Archaeological Chemistry (hereafter LARCH), University of Wisconsin-Madison, under the direction of Dr. T. Douglas Price and Dr. James Burton. The first instrument was an Applied Research Labs Model 3520 inductively-coupled plasma - atomic emission spectrometer (ICP-AES), which can detect and quantify dozens of elements at sub-parts-per-million concentrations (see Burton and Simon 1993 for elemental detection limits and precision typical of this instrument). The second was a Finnegan MAT ELEMENT I high resolution, magnetic-sector inductively coupled plasma-mass spectrometer (ICP-MS), which can obtain elemental and isotopic data at concentrations in the parts-per-quadrillion range. Full details regarding sample preparation and analysis, which varied according to the material being analyzed, are provided in Chapter 11 for limestone and in Chapter 12 for metal artifacts.

The analysis of certain artifacts and geologic samples required the use of spectrometers not available at the LARCH. The sulfur isotope compositions of alabaster (Chapter 10) and lapis lazuli (Appendix 4.4) were determined by Dr. Chris Eastoe at the Isotope Geochemistry Laboratory, Department of Geosciences, University of Arizona using a continuous flow isotope ratio mass spectrometer (CFIRMS). High precision strontium isotope assays of alabaster samples were made by Drs. Joel Blum and Andrea Klaue at the Department of Geosciences, University of Michigan–Ann Arbor, using a thermal ionization mass spectrometer (TIMS). Full details on these instruments, sample preparation and analysis are provided in the Chapter 10. A small number of lead samples examined in this study were analyzed on a Neptune multiple-collector inductively-coupled-plasma magnetic-sector mass-spectrometer (MC-ICP-MS) by Dr. Emily Peterman at the W.M. Keck Isotope Laboratory in the Earth and Marine Science Department, University of California-Santa Cruz.

Instrumental neutron activation analysis (INAA)

Instrumental neutron activation analysis (INAA) – a highly accurate and precise method for quantifying the major, minor and trace element compositions of materials, has been used by archaeologists around the world in efforts to determine the proveniences of a wide range of artifact types (Glascock and Neff 2003). In South Asian archaeology, this technique has, up until now, been exclusively applied to provenience studies of ceramic artifacts such as figurines (Possehl 1994), stoneware bangles (Blackman and Vidale 1992) and pottery (Méry and Blackman 1999; R.P. Wright 1984). For this study, INAA was used to characterize geologic samples and artifacts composed of chert, steatite, agate-carnelian, vesuvianite-grossular garnet and limestone.

All rock samples subjected to INAA were prepared for analysis at the LARCH. Fresh, unweathered material was preferred for analysis. For geologic samples, either a small chip (approximately one gram) was struck from a freshly broken surface toward the interior of the sample and then crushed into a coarse powder with a steel mortar and pestle or, for softer stone (steatite and some limestones), a tungsten carbide drill was used to burr-off the exterior surface of a small area on a sample and then drill directly into the fresh material, creating powder in the process that could used for analysis. Drill bits were carefully cleaned after each was sample was taken. Should contamination from the bit itself have occurred, it would be recognizable as a tungsten "spike" in the INAA spectrum during analysis (Truncer *et al*. 1998: 29). Archaeological samples

were first cleaned in an ultrasonic bath of purified water. Removal of material was dependent on the size nature of the artifact. Generally around 0.5 to 1 gram of material was chipped off of hard stone artifacts and lightly pounded in the steel mortar into smaller (< 1 mm) pieces. The pieces were examined under low magnification and those that were free of cortex, patina or other surface features were selected for analysis. Artifacts made of softer stone were sampled using the tungsten drill described above. For both geologic samples and artifacts, exactly 200 mg (± 1 mg) of sample was loaded into polyethylene vials. Using a diamond scorer, sample numbers were etched onto the vials, which were then sealed by friction welding.

Sample analysis was conducted at the University of Wisconsin's Nuclear Reactor (UWNR) research facility by the team supervised by lab director Robert Agasie. INAA provides precise data on the elemental composition of a material. Elements within samples are first made radioactive, or *activated*, by exposing them to a neutron flux, after which the gamma ray emissions produced as radioactive elements decay were detected and counted (see Glascock and Neff 2003 for full details on this technique). A series of vials containing a standard (Canadian Centre for Mineral and Energy Technology Reference Soil Sample SO-4) were irradiated with the samples to calibrate for variations in exposure to the neutron flux (Robert Agasie and Kevin Austin, UWNR *personal communication*). Depending on an element's half-life, different irradiation and count times were necessary. For the "short" count at the UWNR, each vial was irradiated for 3 seconds and, after a 15 to 16 minute decay time, a 300 second count was performed. For the "long" count, sample vials were irradiated for 7,200 seconds and, after a decay time of approximately seven to ten days, a 3,600 second count was performed. The UWNR facility employs a high purity Ortec Geranium Detector coupled with a PCAII PC-based multi-channel analyzer to detect specific gamma ray emissions emitted by the irradiated samples to determine the amounts of individual elements present within them. The fractional proportions of the elements detected in each sample were reported in parts per million (ppm). After the results were screened of elements that failed to be detected in all samples or had high count-rate standard deviations, the data could be subjected to multivariate statistical analyses.

METHODS OF DATA ANALYSIS

The application of the characterization methods outlined above to the different sets of archaeological and geologic samples examined for this study resulted in large amounts of raw, highly varied types of quantifiable data. Because these results were used to determine the probable geologic provenience of stone and metal artifacts (and, ultimately, to support statements about the behaviors of ancient Harappans), the methods by which they were evaluated had to be carefully chosen. Much has been written regarding the numerous approaches to the analysis of quantifiable archaeological (Baxter 1994, 2003; Shennan 1997) and geologic data (Davis 1986). In this section, I discuss the methods utilized in this study and the reasons for choosing to employ them.

Preliminary data analysis / bivariate plotting

Prior to any statistical manipulation, multiple combinations of isotopic or elemental (after being log normalized) values in a dataset were compared on simple bivariate plots using the program DataDesk 6.0. This exploratory procedure was conducted to determine if, at this initial level, groups of samples from different geologic sources could be clearly differentiated from one another. For sets composed of isotopic data, like those produced for alabaster (Chapter 10) and lead (Chapter 12), this was indeed

possible through bivariate (and in some cases even univariate) plotting of measured values alone. For other datasets, such as that for limestone (Chapter 11), bivariate plots of selected elemental values produced reasonably good separation between sources but multivariate analysis was found to work somewhat better. In such instances, both methods will be presented for comparison. For most of the rock or mineral datasets, however, these preliminary analyses indicated that groups of samples belonging to different geologic sources could not be easily distinguished using simple bivariate plots alone. More robust, multivariate approaches were required.

Multivariate approaches

Choice of methods

For the purposes of study, canonical discriminant analysis (CDA) was deemed the most suitable method for using multivariate data to differentiate sets of samples from various geologic sources and to assign provenience to archaeological samples. CDA is different from other statistical approaches such as principle component analysis or cluster analysis – two methods widely employed in provenience studies of archaeological ceramics (Glascock *et al*. 2004), in that it begins with the presumption that a dataset already has a well-defined structure (Baxter 1994: 185). There is no need to assess patterns of variance or clustering in an effort identify structures that may (or may not) represent meaningful groups, simply because groups with known members have already been defined in the dataset. Here, the known groups are the sets of geologic samples that I have personally collected from sources across Pakistan and India. Although the degree to which it is possible to differentiate the various geologic sources within a dataset is initially unknown, there is never any ambiguity whatsoever as to whether or not the samples representing those sources actually belong to them. When groups are known *a priori*, CDA is the most suitable multivariate technique to use (ibid.: 81). It has been used in provenience studies of several of the same materials examined in this study including steatite (Truncer *et al*. 1998), chert (Craddock *et al*. 1983) and limestone (Holmes and Harbottle 2003).

Certain situations arose throughout this study for which the use of a supplementary multivariate statistical method proved to be worthwhile. For instance, a large set (n = 141) of archaeological steatite from Harappa was analyzed (Chapter 7) and it is useful to know if multiple geochemically distinct groups of materials may be represented among the samples making up that set. Cluster analysis was chosen as the appropriate method with which to evaluate this possibility. This method, which actually encompasses a many different techniques (Baxter 1994: 140), can be used in tandem with CDA as a way to validate and graphically complement observed clusters of unknown samples that, in the case of stone artifacts, may be from the same geologic formations (ibid.: 165, 204-206).

All multivariate analyses were made using the statistical program SPSS 11.0.

Canonical Discriminant Analysis

Canonical discriminant analysis (CDA) was used to make geologic provenience determinations for rock and mineral artifacts in five of the upcoming chapters. For details on the complex mathematics underlying this method one should seek out Michael Baxter's treatises on statistical applications in archaeology (Baxter 1994, 2003). Here, I provide general overviews of the two main features of CDA: discrimination and classification.

- Discrimination

CDA makes two important presumptions of a dataset: 1) that it is composed of distinct groups whose individual members are known and 2) that it contains all possible groups (Baxter 1994: 185-186). During the analysis of a dataset, one or more linear combinations of variables called *discriminant functions*

are generated, each of which are intended to produce the maximum degree of separation (discrimination) possible between the groups of individual cases being assayed. If a set is composed of two groups then just one discriminant function is possible as there is only a single dimension between them that can be evaluated. Analysis of datasets made up of larger numbers of groups results in the generation of multiple functions because additional dimensions can be considered. Displaying the results of analyses involving three or more groups in a two dimensional format is accomplished by creating a bivariate plot of the dataset using the *first* and *second* functions (which are the first and second most significant discriminators) as the axes. Individual members of a dataset are plotted by their *discriminant scores*, which are the values that result when discriminant functions (unstandardized canonical discriminant function coefficients) are applied to each case. In essence, CDA collapses the multiple measurements made for a case down into a single variable (Davis 1986: 479). The *optimal* end result is a scatterplot on which each group is represented by a separate distinct cluster of datapoints (cases), all of which actually belong to the groups they are in.

Optimal separation between groups of samples in a dataset is not always achieved, however. The clusters of datapoints representing a group may overlap with one another – sometimes considerably. Visual examination of scatterplots is the really not the best way to accurately assess how well groups were differentiated using CDA. Discrimination success is better evaluated through "cross-validation" (Baxter 1994: 204). SPSS 10.1 has a cross-validation feature called "leave-one-out" classification. In this procedure, each member of the dataset is omitted from the group it belongs to and classified (a process discussed in the following section) in relation to the dataset as an *ungrouped case*. A percentage is generated based on the number of cases in the dataset that were correctly assigned to the groups that they actually came from. This percentage provides a general indication of how good group separation is and a way to compare discrimination success from different stages of analysis.

- Classification (and misclassification)

The same discriminant functions that were generated to differentiate known groups can also applied to individual cases of unknown origin to classify them according to which group or groups they most closely resemble. These "unknowns" (stone artifacts as well as the geologic samples left out of their groups for cross-validation purposes) are treated *ungrouped* cases and each is placed (according to its discriminant scores) on a bivariate plot in relation to the defined groups of a dataset. The point in space where the mean of a group's members' discriminant scores is situated is called a *group centroid*. An ungrouped case's similarity/dissimilarity with the two or more group centroids in a dataset is established in terms of *Mahalanobis distance* – a statistical measurement that takes into account correlations between variables (Baxter 2003: 70). An individual case shares one Mahalanobis distance value with each group and it is classified as belonging to (or predicted to most likely belong to) the group for which that value is the smallest.

Two quick caveats need to be made regarding CDA classifications/predictions. First, because the method presumes that a dataset contains all possible groups, every ungrouped case considered receives a *predicted group membership* (PGM). This does not mean that the cases definitively belong in the groups that they have been assigned to or, for that matter, to any of the other groups in a set. There is always the chance that the classification for an archaeological sample (ungrouped case) will change when materials from additional geologic sources (known groups) are eventually added to the dataset. Secondly, there is the possibility that an artifact may also be misclassified even when the

source from which it derived is represented as a group in the dataset. Misclassification might occur because of poor separation (discrimination) due to geochemical similarities between the sources being considered and/or because an artifact is an outlier that is situated nearer to the centroid of a source different from its own. In cases when the *first* PGM is questionable, it is sometimes useful to examine the *second* PGM (determined by the second nearest group centroid). In certain cases it *could* be the actual source. Classifications made throughout this book are evaluated on a case-by-case basis in light of overall cross-validation success percentages as well as, occasionally, second PGM determinations.

Cluster Analysis

"Cluster analysis is the generic term for a wide range of methods for discovering homogeneous groups or clusters in a set of data" (Baxter 2003: 90). The various approaches employed in this study fall into a category of methods known as *hierarchical clustering*, which essentially works by either building up (agglomerative) or breaking down (divisive) a dataset into groups based on different measurements of their members' similarities/dissimilarities. Discussions of the different algorithms and distance measurements that may be applied to a dataset to produce clusters can be found in any one of several excellent books written on statistical analysis in archaeology (Baxter 1994, 2003; Shennan 1997). Although some consider the *average linkage* technique to one of the better hierarchical methods (Shennan 1997: 254), there is no one method that is clearly to be preferred over others. Multiple methods can be applied in an exploratory manner to observe how they compare. If the "structure in the data is reasonably clear and captured by all of the competing methodologies" then one may be "reasonably confident that a revealed structure is 'real'" (Baxter 1994: 160).

The result of a cluster analysis (CA) is most commonly displayed as a *dendrogram* – a series of connected straight lines branching out like limbs on a tree. The terminal ends of the branches signify individual cases. Similarity between any two given cases is represented by their distance to each other along the branches of the tree rather than by their proximity to each other on its terminal end. Determining the number of separate clusters that are represented on a dendrogram (in a dataset) is very much a subjective endeavor and, once again, can be facilitated by comparing multiple clustering strategies.

CHAPTER CONCLUSION: STATEMENTS OF PROVENIENCE

For this study, an effort was made to be as comprehensive as possible with regard to locating the geologic sources that Harappans potentially acquired rock and mineral resources from, to collect representative samples from as many of those sources as possible; to define the best scale of provenience resolution, to employ the most appropriate methods of material identification and characterization and to choose those analytical methods that were best-suited for examining the different types of data that were produced. Nonetheless, <u>any</u> statement made in this book regarding the geologic provenience of a stone or metal artifact should always be considered as *provisional*. The study area is vast and the possibility exists that the true source of a particular material may not have been located and/or sampled. Artifact provenience determinations may need to be revised when additional sources are eventually considered. The strongest statement of provenience that can be made at present or, for that matter, at any time in the future (regardless of how many sources and samples are eventually incorporated into a dataset) is: "given all of the sources examined, artifact X appears most chemically (or isotopically or mineralogically) analogous to geologic samples analyzed from source

Z or from source region Z." Still, although always provisional, such a statement and the data that it is based upon can be used to construct a compelling argument for a link between Harappa and a specific source area.

Prior to constructing such arguments, however, it is first necessary to present Harappa's rock and mineral artifact assemblage in detail and examine its spatial and temporal distribution at the site.