# Chemical Characterization and Sourcing of Upper Great Lakes Cherts by INAA

**P.J. Julig, L.A. Pavlish, C. Clark and R.G.V. Hancock**

*Instrumental neutron activation analysis (INAA) is carried out on a series of ninety-three geological chert samples from three known source regions and six beach locations in the western Great Lakes and upper Midwest region. These chemical data are compared with those obtained from a lithic cache from the McCollum site (DiJa-1) on Lake Nipigon to determine the source(s) of the artifacts. A new method permitting whole artifacts to be analyzed by INAA and returned to their curators unaltered was employed to obtain the chemical data from these specimens. Some long distance imports (Knife River Flint) are present in the McCollum cache; however, several sources including Hudson Bay Lowland chert are represented. INAA is an appropriate non-destructive methodology for provenance studies of lithic artifacts whose sources cannot be readily determined by traditional means.*

## Introduction

In the study of lithic assemblages the identification of the artifacts' raw material is one of the primary concerns of the analyst. This forms the basis of many subsequent interpretations of prehistoric human behaviour, such as cultural group mobility, trade, social interactions, and the temporal and cultural aspects of raw material selection. The identification of lithic materials in archaeological assemblages is commonly based on macroscopic or low-power microscopic criteria. Several petrographic factors, however, may affect the accuracy of such identifications including: 1) visual' look-alikes from other known or unknown sources; 2) post-depositional modification to artifact surfaces; and, 3) the level of skill of the analyst in recognizing the range of variability within and between sources.

In the Upper Great Lakes region prehistoric cultures used a range of lithic materials from both primary and secondary sources. Pebble chert present in Lake Superior beach deposits, river gravels and glacial till is often identified by archaeologists as Hudson Bay Lowland chert or HBL (e.g. Clark, 1989). HBL has been reported as a minor component in Lake Superior region lithic assemblages from Paleo-Indian (Fox, 1975; Julig, 1988) to late prehistoric times (Holman & Martin, 1980). Extensive use of pebble cherts by prehistoric groups has also been reported in the northern Lake Michigan region (Binford & Quimby, 1972). The geological source region for HBL is the Hudson Bay lowland basin in northern Ontario. This chert is mainly Devonian age, from the Stooping River Formation (Sanford et al., 1968), and it commonly occurs as cobbles and pebbles distributed by Pleistocene glaciations in secondary deposits throughout the Canadian Shield. Within these regions HBL occurs as a high quality, waxy chert of variable colour (steel-grey through light brown, dark brown, and cream). Along the south shore of Lake Superior and around northern Lake Michigan it is, however, reported to be highly variable with respect to both quality and colour (Clark, 1989).

Julig (1988) has noted that HBL has tended to be a catch-all category for a broad range of archaeological and geological pebble cherts. The identification of some of these cherts is inconsistent with the criteria used to define HBL from the source region. In addition the brown colour varieties of HBL are often visually indistinguishable from Knife River Flint from North Dakota (Clayton et al., 1970).

The objectives of this study are fivefold:

1) to characterize chemically (using instrumental neutron activation analysis [INAA]) geological HBL chert from the source region;

2) to characterize the geological sources of several other cherts which can be visually similar to certain HBL colour varieties (e.g., Gunflint chert [GC], Knife River flint [KRF]);

3) to test samples of pebble "HBL-like" chert from Lake Superior and Lake Michigan beach deposits;

4) to compare the pebble "HBL-like" cherts to the various geological sources tested to determine their degree of chemical similarity; and

5) to compare the geological data with those obtained from the lithic cache of the McCollum

archaeological site, and to demonstrate a new approach to neutron activation analysis by which entire artifacts are placed in the reactor, analyzed, and subsequently returned undamaged to their curators.

## Analytical Methods

Sample sizes ranging between 200 and 600 mg were used for the neutron activation analysis of the geological samples. The archaeological samples from the McCollum site varied in size between 50 and 10,000 mg.

To determine the concentrations of uranium (U), dysprosium (Dy), barium (Ba), titanium (Ti), magnesium (Mg), silicon (Si), sodium (Na), vanadium (V), aluminum (Al), manganese (Mn), calcium (Ca), and potassium (K), which produce short-lived radioisotopes, all samples were irradiated serially for five minutes at a neutron flux of 2.5 x $10^{11}$ n.cm-2.s-1 in the SLOWPOKE Reactor Facility at the University of Toronto. After an eight to twelve minute delay, to allow for the decay of short-lived "Al to levels similar to those of the other radioisotopes, each sample was assayed using a five minute counting time with germanium-based gamma-ray detectors. The chemical concentrations were determined using the comparator method as discussed by Hancock (1978).

## Sub-Sampling Methodology

In order to obtain sub-samples in the range of 200-600 mg representative geological specimens of HBL, KRF and GC were reduced by bipolar techniques using a moose antler anvil and billet hammer. The nine geological sources tested are listed in Table 1. The unweathered interior matrix of the geological samples was used wherever possible to reduce the effects of surface chemical weathering. A range of colour varieties and quality of materials were tested for each locality. However, for some locations (Table 1) only a limited number of samples were available for analysis, and several HBL geological samples were weathered and of somewhat poor quality chert.

## Geological Samples

### Hudson Bay Lowland (HBL) Chert

As mentioned above, a primary objective of this study is to characterise HBL cherts from the primary source region in the Hudson Bay Lowland basin and adjacent areas of the Canadian Shield. This region experienced repeated glaciation during the Pleistocene and, as a result, the cherts have been widely distributed in regional secondary deposits. The original source region for HBL cherts (Figure 1) is underlain by nearly horizontally bedded Palaeozoic limestones and shales, covered by up to four separate tills, and overlain by Tyrrell sea marine sediments. In the vicinity of the Albany River in the western James Bay lowlands the Stooping River Formation containing this chert is of Lower Devonian age (Sanford et al., 1968). This formation is a sparsely fossiliferous grey limestone with nodular chert. Near the mouth of the Moose River there are lag deposits of abundant nodular chert produced by the sorting and winnowing of wave action (pers. comm. A.W. Norris 1991). During an archaeological survey along the lower Albany River (Julig 1982) no high-quality primary chert sources were located, but abundant geological cobble chert was present along river terraces and beaches and seventeen samples of these cherts were tested for this study (Table 1). Other geological HBL samples were obtained from the Severn River region in the northern Lowlands (five samples). The geological bedrock in this region is of Silurian (Severn River Formation) and Ordovician age (Bad Cache Rapids group) (Sanford et al., 1968), which also contain some chert. From the boundary of the Lowlands and Shield, four samples were obtained from Attawapiskat Lake, these being from beach gravels. As shown on Table 1, numerous colour varieties are represented, from light grey (10YR6/1) to dark brown (10YR3/3). High quality lustrous specimens are common in both the grey and brown colour varieties (Julig 1982). It was not possible within the context of this study to obtain in situ geological chert samples; however, a larger suite of samples is being sought for further studies.

### Gunflint Chert (GC)

Within the Gunflint Formation of the Precambrian bedrock in the Atikokan-Thunder Bay-Marathon area north of Lake Superior (Figure 1) are a variety of algal cherts. These Gunflint oolitic cherts are of Proterozoic age and contain some of the earliest fossils in the world in the form of stromatolites and other macrofossils (Glass 1972). The cherts of the lower Gunflint member are predominantly black, red and white; however dark *grey* to brown colour variations have been noted in the field and in geological collections (Table 1). Since some Gunflint chert varieties can overlap visually with HBL seven samples from two in situ localities were tested by INAA to determine whether they are chemically distinct from HBL. Since the Gunflint chert source area (Figure 1) was glaciated transport of this material certainly occurred along with that of the more distinctive iron-rich taconites of the

Figure 1.

Map of the study area showing geological source areas (No.'s 1-3); beach lake cobbles (No.'s 4-9); and the location of the McCollum archaeological site (No. 10).

**Table 1. Geological chert samples and source regions, and archaeological samples from the McCollum site.**

| | Geological or Field Designation | # of samples Analyzed | Collecting Locations | Munsell Colour (unweathered matrix) | Collector & Reference | Context & Comments |
|---|---|---|---|---|---|---|
| GEOLOGICAL CHERT SAMPLES AND SOURCE REGIONS | | | | | | |
| 1. | Hudson Bay Lowland Chert (HBL) | 17 | Albany River, Ontario | light yellow brown (10YR6/4)<br>light grey (10YR6/1)<br>dark grey (10YR4/1)<br>dark greyish brown (10YR3/2)<br>dark brown (10YR3/3) | P. Julig 1982 | river cobbles (some fossil inclusion) |
| | HBL | 5 | Severn River, Ontario | light grey (10YR7/1)<br>pale brown (10YR7/3) | J. Luc Pilon | river cobbles |
| | HBL | 4 | Attawapiskat Lake & Triangular Lake, Ontario | mottled white to light grey (10YR7/2) | Ont. Ministry of Culture & Communication W. Ross and P. Reid | beach cobbles medium to low grade chert |
| | | 26 Total | | | | |
| 2. | Gunflint Chert (GC) | 4 | Whitefish R., Lypster, Ont. | dark reddish brown (5YR3/2) to (7.5YR3/2) | ROM Dept. of Invertebrate Paleontology, C. Collins | in situ, (algal stromatolites present) |
| | GC | 3 | Kakabeka Falls, Ont. | dark grey (7.5YR4/0) | " | " |
| | | 7 Total | | | | |
| 3. | Knife River Flint (KRF) | 16 | Dunn County North Dakota | dark brown (10YR3/3) to dark yellowish brown (10YR3/4) | Clayton, 1970<br>S. Ahler<br>Julig et al., 1989b | cobbles from pit near primary aboriginal quarry area |
| | KRF | 9 | White Butte & Slope Counties, North Dakota | pale brown (10YR6/3) to dark yellowish brown (10YR4/6), and dark brown (10YR3/3) | S.Ahler | cobbles |
| | | 25 Total | | | | |

GEOLOGICAL CHERT FROM GREAT LAKES BEACH DEPOSITS

| | | | | | | |
|---|---|---|---|---|---|---|
| 4. | "HBL-like" | 11 | Lake Superior, McLain Park, Houghton County, Michigan | light grey (10YR6/1)<br>pale brown (10YR6/3) | C. Clark | beach pebbles |
| 5. | "HBL-like" | 2 | Lake Superior, Apostles Islands, Wisconsin | greyish brown (2.5Y5/2) | C. Clark | beach pebbles (banded, as Gunflint chert) |
| 6. | "HBL-like" | 2 | Lake Superior, Little Girls Point, Ontonagon County, Michigan | light grey (10YR7/1) | C. Clark | beach pebbles, low grade material (dolomite?) |
| 7. | "HBL-like" | 3 | Northwestern L. Superior, Grand Portage National Monument, Minn. | pale brown (10YR6/3) to light yellowish brown (10YR6/4) | C. Clark | beach pebbles |
| 8. | "HBL-like" | 7 | Lake Michigan Sleeping Bear Dunes National Lakeshore, Benzie County, Michigan | pale brown (10YR6/3) to light grey (10YR6/1) | C. Clark | beach pebbles |
| 9. | "HBL-like" | 10 | Lake Superior, Houghton and Keweenaw County, Michigan | light grey (10YR6/1)<br>light olive grey (5Y6/2)<br>light brownish grey (10YR6/2)<br>very dark brown (10YR2/2) | C. Clark | pebble chert, variable, some banded and mottled |
| | | 35 Total | | | | |

/Table 1 continued

Table 1 continued

ARCHAEOLOGICAL SAMPLES FROM McCOLLUM SITE (DiJa-1)

| | Artifact No. | Artifact Description | Weight of Sample (mg) | Colour |
|---|---|---|---|---|
| 10. | DiJa-1-1 | biface | 110.8* | brown (I0YR3/3) |
| | DiJa-4-1 | end scraper | 4150 | light grey (10YR5/1) |
| | DiJa-1-2 | end scraper | 2860 | pale brown (10YR6/3 to 10YR5/2) |
| | DiJa-1-3 | used flake | 1622 | light grey (10YR6/1) |
| | DiJa-1-4 | used flake | 1248 | light grey (10YR6/1) |
| | DiJa-1-5 | used flake | 1143 | grey brown (2.5YR5/2 |
| | DiJa-1-6 | used flake | 1006 | grey brown (2.5YR5/2) |
| | DiJa-1-7 | used flake | 1366 | light grey (10YR6/1) |
| | DiJa-1-8 | end scraper | 1781 | light grey (10YR6/1) |
| | DiJa-1-9 | end scraper | 1461 | tan to pale brown 110YR7/2) |
| | DiJa-1-10 | used flake | 1820 | mottled light/dark grey (10YR7/2 to 10YR4/1) |
| | DiJa-1-11 | used flake | 1257 | medium grey 15YR7/1) |
| | DiJa-1-12 | used flake | 781 | brown (KRF-like) (10YR3/3) |
| | DiJa-1-13 | used flake | 1167 | grey and white (5YR7/1 to 10YR8/1) |
| | DiJa-1-14 | used flake | 1806 | grey with cortex (I0YR6/1) |
| | DiJa-1-15 | scraper | 2097 | tan to **pale** brown (10YR7/2) |
| | DiJa-1-16 | used flake | 1893 | *grey* brown (2.5YR5/2) |
| | DiJa-I-17 | used flake | 931 | brown (10YR3/3) |
| | DiJa-1-18 | used flake | 1087 | grey brown (2.5YR5/2) |
| | DiJa-1-19 | used flake | 1188 | brown (10YR3/3) |
| | DiJa-1-20 | used flake | 632 | tan (10YR7/2) |
| | DiJa-1-21 | end scraper | 1346 | brown (10YR3/3) |
| | DiJa-1-22 | used flake | 462 | brown (10YR3/3) |
| | DiJa-1-23 | used flake | 1395 | brown (10YR3/3) |
| | DiJa-1-5-1 | flake | 1459 | tan (10YR7/2) |
| | DiJa-1-5-2 | flake | 2509 | grey (10YR6/1) |
| | DiJa-1-5-3 | flake | 605 | grey brown (2.5YR5/2) |
| | DiJa-1-5-5 | flake | 2456 | grey brown with cortex (2.5YR5/2) |
| | DiJa-1-5-6 | flake | 674 | tan (10YR7/2) |
| | DiJa-1-5-7 | flake | 1214 | grey (10YR6/1) |
| | DiJa-1-5-8 | flake | 749 | tan (10YR7/2) |
| | DiJa-1-5-9 | flake | 503 | brown (10YR3/3) |
| | DiJa-1-5-10 | flake | 1031 | light brown (10YR6/3) |
| | DiJa-1-5-11 | flake | 1136 | tan (10YR7/2) |
| | DiJa-1-5-12 | flake | 613 | brown (10YR3/3) |
| | DiJa-1-5-14 | flake | 915 | brown (10YR3/3) |
| | DiJa-1-5-15 | flake | 914 | brown (10YR3/3) |
| | DiJa-1-5-16 | flake | 377 | brown (10YR3/3) |
| | DiJa-1-5-17 | flake | 705 | tan (10YR7/2) |
| | DiJa-1-5-18 | flake | 844 | banded dark brown (7.5YR3/2) |
| | DiJa-1-5-19 | flake | 1797 | translucent brown (10YR6/3) |
| | DiJa-1-5-20 | flake | 368 | translucent brown (10YR6/3) |
| | DiJa-1-5-21 | flake | 287 | grey brown (2.5YR5/2) |
| | DiJa-1-5-22 | flake | 958 | tan (10YR7/2) |
| | DiJa-1-5-23 | flake | 404 | translucent brown (10YR6/3) |
| | DiJa-1-5-24 | flake | 1149 | translucent brown (10YR6/3) |
| | DiJa-1-5-25 | flake | 801 | banded brown (7.5YR3/2) |
| | DiJa-1-9-1 | flake | 872 | translucent brown (10YR6/3) |
| | DiJa-1-9-2 | flake | 1561 | tan (10YR7/2) |
| | Dija-1-9-3 | flake | 316 | brown (10YR3/3) |
| | DiJa-1-10-2 | flake | 708 | light brown (10YR7/3) |
| | DiJa-1-38 | point | 9923 | tan (10YR7/2) |
| | DiJa-1-40 | blade scraper | 85.5' | dark brown (10YR3/3) |
| | DiJa-1-41 | scraper | 50.7' | dark brown (10YR3/3) |
| | 47-PC-12 | flake | 889 | dark brown (10YR3/3) |
| | 47-PC-12(14-2-27) | scraper | 147.* | dark brown (10YR3/3) |
| | Total 56 | | | |

* specimens too large for largest containers are subsampled

Gunflint Formation. Lithic materials from the Gunflint formation were widely used by preceramic native cultures in the Lake Superior region (Julig 1988).

### Knife River Flint (KRF)

The source region of KRF is in western North Dakota (Figure 1) where it occurs in secondary deposits (Clayton et al., 1970). It ranges in colour from light brown to dark brown (Table 1) and is visually similar to some brown varieties of HBL (cf. Julig et al., 1989a,b). KRF was widely used by prehistoric cultures on the Northern Plains (Loendorf et al., 1984) and has been reported in archaeological assemblages from Alberta (Wormington and Forbis, 1965) to Ohio (Braun et al., 1982). KRF occurs in small quantities in archaeological contexts in the Western Great Lakes region (Wright 1987; Julig et al., 1991). A total of twenty-five geological samples of KRF from Dunn County and two other western North Dakota locations were analyzed; their visual characteristics are noted in Table 1.

### Pebble "HBL-like" Chert from Upper Great Lakes Beaches

Chert samples visually identified as "HBL" were collected from beaches at six locations from Lake Superior and Lake Michigan. These samples vary greatly in colour and quality. The sample locations from northwest Lake Superior to northeastern Lake Michigan represent a transect of about 500 km across the upper Great Lakes (Figure 1, Table 1). These thirty-five samples were also analyzed by INAA to determine their chemical correspondence with HBL from the geological source region.

## Results

The initial objectives were to characterize chemically geological HBL from its source regions and to compare these characterizations with those of visually similar KRF and Gunflint chert. Of the fifteen short half-life radioisotope-producing elements sought, the six most consistent and diagnostic in separating these sources are U, Al, Si, Cl and Mn. These six elements are paired [(x = Al & Si); (y = CI & Mn); (z = Dy & U)]; scaled [values are consistently adjusted to permit use of a ternary diagram: (x = AI*1000/Si); (y = C1/100 + Mn/10); (z = Dy + U)]; standardized [(x = (x/x+y+z)); (y = (y/x+y+z)); (z = (z/x+y+x))]; and, normalized [x+y+z = 1] (Figure 2a,b,c).

Using these six elements, three chemical groupings are evident for these chert types. Gunflint chert contains Mn levels at a concentration factor ranging to three times that in the other source samples (Figure 2a and Table 2). KRF is chemically distinct, clustering in the upper left portion of the ternary diagram on the basis of higher standardized and normalized U and Dy values (Figure 2a). Two HBL geological samples (#25 and #26, Table 2) have high Mn values, resulting in a rather broad chemical spread for this material (Figure 2a). However, these two samples had some weathered patina which may have contributed to these greater concentrations. Another possibility is that there may be several chemically distinct groups within the HBL field (Figure 2a) and this is being addressed in further research. However, at this point, the HBL, KRF and GC groups can be separated chemically on the basis of these six elements.

In the plotting of the Great Lakes pebble beach "HBL-like" samples against the three geological fields, the specimens fall within the HBL cluster (Figure 2b). The pebble chert samples from Lake Michigan beaches have greater Cl concentrations (Table 2), indicating some chemical as well as visual variability in these secondary chert sources. However, the low Mn concentrations of these Lake Michigan cherts indicate they are not derived from the Gunflint formation (GC).

## Archaeological Comparisons: McCollum (DiJa-1) Sample

This analysis of geological materials provides a basis for comparison with prehistoric archaeological lithic materials. The Late Archaic McCollum lithic and copper cache materials from the Lake Nipigon region of Ontario (Griffin and Quimby, 1961) was obtained from the Canadian Museum of Civilisation for the purpose of analysis by INAA. The lithic archaeological sample consisted of fifty-six items: twenty-nine flakes, one point, one biface, one lamellar blade and twenty-four scrapers and raclettes (Julig et al., 1991). These specimens included a range of colour varieties (Table 1), some of which were previously identified as KRF (Wright, 1987).

## Rationale for Method

Short-lived isotope-producing INAA with low flux reactor facilities like SLOWPOKE permits the non-destructive bulk analyses of very small specimens and whole artifacts without damage or need of excessive cool-off periods. This situation contrasts with that of high neutron flux reactors which tend

Table 2. Geochemical data from INAA of geological materials used in Figure 2a and 2b

| No. | sNo | Al(%) | Si(%) | Cl(ppm) | MN(ppm) | U(ppm) | Dy(ppm) |
|---|---|---|---|---|---|---|---|
| GEOLOGICAL MATERIALS: SOURCE REGIONS. | | | | | SOURCE: 1 | HUDSON BAY LOWLAND CHERT (HBL) | |
| 1 | 1 | 0.27 | 55.95 | 75.00 | 0.37 | 1.36 | 0.01 |
| 2 | 1 | 0.25 | 47.45 | 94.00 | 5.20 | 0.76 | 0.23 |
| 3 | 1 | 0.36 | 57.42 | 6.00 | 0.36 | 0.51 | 0.01 |
| 4 | 1 | 0.26 | 53.00 | 102.00 | 1.40 | 1.71 | 0.01 |
| 5 | 1 | 0.25 | 57.00 | 135.00 | 0.00 | 1.98 | 0.01 |
| 6 | 1 | 0.26 | 54.00 | 96.00 | 0.70 | 1.51 | 0.01 |
| 7 | 1 | 0.23 | 52.00 | 50.00 | 0.80 | 0.48 | 0.29 |
| 8 | 1 | 0.29 | 37.00 | 80.00 | 15.50 | 0.91 | 0.08 |
| 9 | 1 | 0.27 | 49.00 | 92.00 | 22.70 | 0.76 | 0.07 |
| 10 | 1 | 0.27 | 54.00 | 116.00 | 4.80 | 0.86 | 0.27 |
| 11 | 1 | 0.32 | 41.00 | 49.20 | 3.08 | 0.15 | 0.02 |
| 12 | 1 | 0.46 | 51.07 | 51.88 | 16.09 | 0.29 | 0.18 |
| 13 | 1 | 0.34 | 53.55 | 71.57 | 1.46 | 0.30 | 0.03 |
| 14 | 1 | 0.34 | 50.21 | 220.37 | 1.89 | 0.88 | 0.04 |
| 15 | 1 | 0.28 | 45.41 | 165.78 | 2.08 | 0.36 | 0.01 |
| 16 | 1 | 0.29 | 47.22 | 198.01 | 1.51 | 0.74 | 0.10 |
| 17 | 1 | 0.30 | 53.42 | 145.92 | 1.39 | 0.58 | 0.03 |
| 18 | 1 | 0.16 | 44.98 | 99.96 | 1.77 | 0.63 | 0.05 |
| 19 | 1 | 0.36 | 48.43 | 107.14 | 2.64 | 0.33 | 0.01 |
| 20 | 1 | 0.29 | 47.27 | 111.62 | 57.56 | 0.65 | 0.05 |
| 21 | 1 | 0.32 | 46.83 | 100.03 | 20.01 | 0.36 | 0.04 |
| 22 | 1 | 0.29 | 40.28 | 144.60 | 55.81 | 0.37 | 0.03 |
| 23 | 1 | 0.22 | 25.12 | 314.55 | 50.61 | 0.59 | 0.01 |
| 24 | 1 | 0.33 | 50.40 | 103.66 | 1.26 | 0.52 | 0.02 |
| 25 | 1 | 0.29 | 40.28 | 145.58 | 127.85 | 0.37 | 0.03 |
| 26 | 1 | 0.29 | 47.27 | 110.69 | 235.81 | 0.65 | 0.05 |
| GEOLOGICAL MATERIALS: SOURCE REGIONS. | | | | | SOURCE: 2 | GUNFLINT CHERT (GC) | |
| 27 | 2 | 0.25 | 46.84 | 122.00 | 456.12 | 0.07 | 0.13 |
| 28 | 2 | 0.28 | 48.70 | 122.00 | 380.18 | 0.49 | 0.07 |
| 29 | 2 | 0.23 | 45.01 | 75.00 | 479.23 | 0.44 | 0.29 |
| 30 | 2 | 0.23 | 44.19 | 32.00 | 343.71 | 0.23 | 0.25 |
| 31 | 2 | 0.24 | 46.37 | 72.00 | 589.05 | 0.55 | 0.13 |
| 32 | 2 | 0.21 | 42.44 | 60.00 | 163.69 | 0.37 | 0.13 |
| 33 | 2 | 0.26 | 49.02 | 66.00 | 147.49 | 0.30 | 0.08 |
| GEOLOGICAL MATERIALS: SOURCE REGIONS. | | | | | SOURCE: 3 | KNIFE RIVER FLINT (KRF) | |
| 34 | 3 | 0.24 | 52.63 | 6.89 | 2.21 | 7.31 | 0.09 |
| 35 | 3 | 0.22 | 49.10 | 45.00 | 2.94 | 7.36 | 0.09 |
| 36 | 3 | 0.24 | 54.47 | 20.80 | 3.78 | 3.64 | 0.14 |
| 37 | 3 | 0.25 | 52.66 | 31.60 | 4.24 | 3.83 | 0.11 |
| 38 | 3 | 0.21 | 51.30 | 11.79 | 6.36 | 6.56 | 0.60 |
| 39 | 3 | 0.22 | 50.27 | 52.71 | 8.71 | 8.89 | 0.62 |
| 40 | 3 | 0.20 | 46.79 | 21.44 | 2.03 | 4.78 | 0.20 |
| 41 | 3 | 0.22 | 54.30 | 35.43 | 2.16 | 5.43 | 0.21 |
| 42 | 3 | 0.19 | 47.56 | 21.90 | 2.98 | 5.16 | 0.42 |
| 43 | 3 | 0.25 | 49.03 | 30.53 | 2.87 | 5.30 | 0.43 |
| 44 | 3 | 0.25 | 49.13 | 38.31 | 6.01 | 4.78 | 0.01 |
| 45 | 3 | 0.25 | 50.50 | 65.00 | 9.13 | 1.78 | 2.42 |
| 46 | 3 | 0.26 | 48.74 | 77.00 | 3.98 | 1.95 | 2.50 |
| 47 | 3 | 0.24 | 52.27 | 48.00 | 0.89 | 3.96 | 0.07 |
| 48 | 3 | 0.24 | 50.90 | 65.00 | 0.88 | 4.64 | 0.05 |
| 49 | 3 | 0.23 | 51.20 | 74.00 | 0.92 | 4.69 | 0.07 |
| 50 | 3 | 0.24 | 47.30 | 32.00 | 1.15 | 3.70 | 2.04 |
| 51 | 3 | 0.28 | 52.00 | 56.00 | 1.52 | 6.71 | 0.80 |
| 52 | 3 | 0.25 | 51.40 | 31.00 | 1.30 | 6.43 | 0.71 |
| 53 | 3 | 0.29 | 50.30 | 84.00 | 1.34 | 1.39 | 1.88 |
| 54 | 3 | 0.22 | 45.40 | 77.00 | 1.89 | 1.69 | 2.05 |
| 55 | 3 | 0.25 | 50.40 | 66.30 | 1.96 | 2.14 | 3.97 |
| 56 | 3 | 0.23 | 47.30 | 61.00 | 1.61 | 4.48 | 0.16 |
| 57 | 3 | 0.24 | 53.30 | 29.90 | 9.50 | 5.12 | 0.11 |
| 58 | 3 | 0.23 | 51.60 | 29.90 | 4.14 | 4.96 | 0.23 |

/Table 2 continued

Table 2 continued

| No. | sN0 | AI(%) | Si(%) | CI(ppm) | MN(ppm) | U(ppm) | Dy(ppm) |
|---|---|---|---|---|---|---|---|
| GEOLOGICAL MATERIALS: CHERT FROM LAKE BEACHES<br>HOUGHTON COUNTY, MICHIGAN, McLAIN PARK, LAKE SUPERIOR | | | | | | SOURCE: 4 | |
| 59 | 4 | O.50 | 48.31 | 113.00 | 6.33 | O.44 | O.07 |
| 60 | 4 | O.50 | 48.36 | 121.00 | 5.37 | O.40 | 0.06 |
| 61 | 4 | O.39 | 49.13 | 39.00 | 3.50 | 0.34 | O.06 |
| 62 | 4 | O.42 | 47.68 | 38.00 | 6.26 | O.35 | O.04 |
| 63 | 4 | O.36 | 50.54 | 46.00 | 4.59 | O.26 | O.04 |
| 64 | 4 | O.36 | 50.19 | 52.00 | 2.64 | O.30 | O.04 |
| 65 | 4 | O.50 | 55.80 | 44.00 | 4.05 | O.53 | O.04 |
| 66 | 4 | O.38 | 48.73 | 41.00 | 5.72 | O.39 | O.04 |
| 67 | 4 | O.32 | 46.13 | 52.00 | 4.89 | O.45 | O.05 |
| 68 | 4 | O.50 | 48.48 | 115.00 | 6.29 | O.43 | O.08 |
| 69 | 4 | O.50 | 48.29 | 118.00 | 5.32 | O.39 | O.07 |
| GEOLOGICAL MATERIALS: CHERT FROM GREAT LAKE BEACH DEPOSITS<br>APOSTLES ISLANDS, WISCONSIN, LAKE SUPERIOR | | | | | | SOURCE: 5 | |
| 70 | 5 | O.33 | 52.15 | 51.00 | O.79 | O.47 | O.27 |
| 71 | 5 | O.32 | 48.49 | 50.00 | O.83 | 0.38 | O.21 |
| GEOLOGICAL MATERIALS: CHERT FROM GREAT LAKE BEACH<br>LITTLE GIRLS POINT, ONTONAGON, MICHIGAN, LAKE SUPERIOR | | | | | | SOURCE: 6 | |
| 72 | 6 | O.33 | 33.15 | 189.00 | 48.72 | O.80 | O.31 |
| 73 | 6 | O.39 | 41.98 | 145.00 | 26.79 | 0.52 | 0.25 |
| GEOLOGICAL MATERIALS: CHERT FROM GREAT LAKE BEACH DEPOSITS<br>GRAND PORTAGE NATIONAL MONUMENT, MINNESOTA, NW LAKE SUPERIOR | | | | | | SOURCE: 7 | |
| 74 | 7 | 0.33 | 50.70 | 37.00 | 3.01 | O.26 | O.16 |
| 75 | 7 | 0.30 | 47.38 | 35.00 | 2.06 | O.20 | O.09 |
| 76 | 7 | O.24 | 33.60 | 32.00 | 2.73 | O.17 | 0.08 |
| GEOLOGICAL MATERIALS: CHERT FROM GREAT LAKE BEACH DEPOSITS<br>SLEEPING BEAR DUNES NATIONAL LAKESHORE, BENZIE COUNTY, MICHIGAN | | | | | | SOURCE: 8 | |
| 77 | 8 | O.42 | 47.56 | 284.00 | 9.51 | 0.31 | O.06 |
| 78 | 8 | O.58 | 45.46 | 306.00 | 45.09 | O.57 | O.25 |
| 79 | 8 | 0.48 | 45.81 | 426.00 | 11.06 | 0.45 | O.11 |
| 80 | 8 | O.30 | 47.37 | 276.00 | 1.90 | 0.61 | O.04 |
| 81 | 8 | O.29 | 46.16 | 256.00 | 1.48 | O.59 | O.04 |
| 82 | 8 | O.39 | 46.66 | 810.00 | 3.81 | O.44 | 0.10 |
| 83 | 8 | O.46 | 44.37 | 14.00 | 5.34 | O.58 | O.15 |
| GEOLOGICAL MATERIALS: CHERT FROM GREAT LAKE BEACH DEPOSITS<br>HOUGHTON AND KEWEENAW COUNTIES, MICHIGAN. LAKE SUPERIOR | | | | | | SOURCE: 9 | |
| 84 | 9 | O.30 | 47.78 | 58.48 | 20.48 | O.37 | O.05 |
| 85 | 9 | O.29 | 51.92 | 51.06 | 4.06 | O.33 | 0.06 |
| 86 | 9 | O.31 | 45.84 | 59.04 | 1.17 | 0.46 | 0.31 |
| 87 | 9 | O.27 | 46.38 | 56.27 | 1.37 | O.98 | O.03 |
| 88 | 9 | 0.35 | 50.64 | 60.78 | 14.06 | O.30 | O.09 |
| 89 | 9 | 0.38 | 47.78 | 71.82 | 5.25 | 0.83 | O.03 |
| 90 | 9 | 0.28 | 43.38 | 55.59 | 11.38 | O.27 | O.09 |
| 91 | 9 | O.32 | 46.43 | 60.83 | 45.17 | O.27 | 0.04 |
| 92 | 9 | O.37 | 44.87 | 60.25 | 4.65 | 0.72 | 0.05 |
| 93 | 9 | O.37 | 44.36 | 96.36 | 117.80 | O.47 | O.23 |

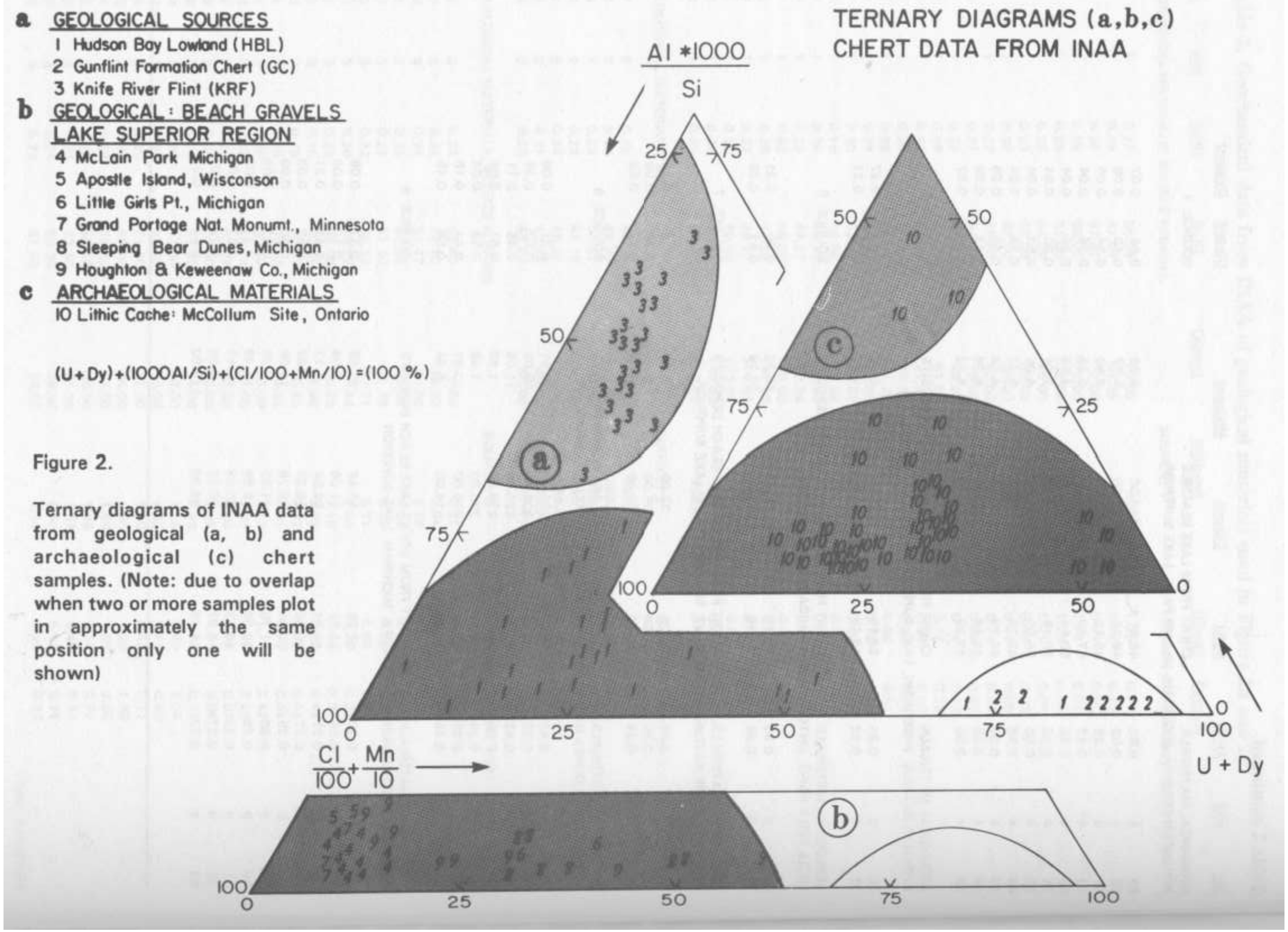

Figure 2.

Ternary diagrams of INAA data from geological (a, b) and archaeological (c) chert samples. (Note: due to overlap when two or more samples plot in approximately the same position only one will be shown) to create "hot" artifacts that are permanently lost to the analyst and curator. In some situations there are advantages to using INAA: 1) INAA will provide high sensitivity in favourable situations for a large number of elements; 2) methods for analyzing chemically complex rocks and minerals making up artifacts often require chemical separation or the addition of reagents to the sample dissolution, and these approaches may introduce contaminants into the sample in the form of reagents or particulate matter in the laboratory; prevention can be tedious and costly and INAA permits analysis prior to any chemical processing which may reduce the influence of contaminants; 3) with the development of new methods using the SLOWPOKE facility short-lived isotope-producing INAA can be carried out on a large range of sample sizes without chemical separation and more importantly, the sample can remain in its original form; 4) INAA greatly reduces laboratory handling time; 5) INAA is capable of assaying a large number of elements at one time. Multi-element analyses are often important in establishing correlations between trace elements. Sample handling time is further reduced if several elements are determined simultaneously because no greater time allocation to analyses is required; 6) reduced handling time and multi-element analytical potential permits a great deal more analytical research work per unit time to be carried out; and 7) the whole artifact analysis method permits items to be assayed in their entirety and returned to their curators undamaged and, because the results obtained are based upon the total artifact, the potential for sampling error is eliminated. Therefore, if bulk assay work is an objective, time is limited and non-destruction is essential, INAA is a viable alternative to other analytical methods (Hancock et al., 1990).

## Analytical Procedure for Archaeological Material

The fifty-six whole artifacts from the McCollum site were placed in three sizes of polyethlene containers (Olympic Plastics Co.). The large containers measuring approximately 2.5 cm x 5 cm (25ml) were used with thirty-six of the artifacts. Medium (7 ml) containers were required for seventeen artifacts, and three artifacts were placed in standard 1 ml containers. The size range between the largest (9923 mg) and smallest (50 mg) item was approximately a factor of 200 (Table 1). The majority of the artifacts (small scrapers and flakes) were in the 1000 to 2000 mg range. The reactor procedure used is analogous to the one discussed above for the geological materials with a larger irradiation site being required for the 25 ml containers. More importantly, the use of medium and large containers required that the standards derived by the comparator method be recalculated. With these recalculated standards the INAA-derived elemental concentrations for the whole artifacts from the McCollum site were determined (Table 3).

## Archaeological Results

The same suite of paired, scaled, standardized and normalized element concentrations are plotted for the McCollum artifact material in Figure 2c (No. 10) as are shown for geological materials in Figure 2a (Nos. 1-3) and 2b (Nos. 4-9). The majority of the artifacts fall within the HBL-like chemical field. But there are three artifacts that look like KRF and do fall within the KRF chemical field. In addition, the preliminary evaluation of the analyses of the materials suggests that there are several chemically distinct clusters within the McCollum cache. These results suggest that possible Michigan HBL-like cobble chert sources may have been used but without more geological and geochemical information precise location within this HBL cluster would be purely conjecture.

## Conclusions

The three primary geological chert sources tested, KRF, HBL and GC, are shown to be chemically distinctive. Pebble chert from Lake Superior and upper Lake Michigan beach deposits visually identified as "HBL" was shown to correspond chemically with the HBL field, although there is considerable visual and some chemical variability between the locations tested. Lithic analysts must be cautious in using only visual criteria to identify the geological sources of artifacts (e.g. Clark, 1984). Some dark brown HBL, KRF and Lake Superior cherts and agates are visually very similar. KRF chert from the Dakotas has now been chemically confirmed in upper Great Lakes archaeological assemblages (Julig et al., 1991) as previously reported by Wright (1987). These long distance imports occurred in small quantities in both utilitarian and mortuary contexts, and in assemblages that contained visually similar HBL and/or Lake Superior agates. Analysts must consider the possibility of multiple imports from different locations, rather than placing all unknown cherts in a default "HBL" category, often with an assumption that they are derived from local secondary sources.

The comparison of the data obtained from the

**Table 3. Geochemical data from INAA of McCollum materials used in Figure 2c.**

| No. | Sno | Al(%) | Si(%) | CI(ppm) | MN(ppm) | U(ppm) | Dy(ppm) |
|---|---|---|---|---|---|---|---|
| ARCHAEOLOGICAL MATERIAL: LITHIC MATERIALS FROM THE McCOLLUM CACHE, ONTARIO | | | | | | | |
| SOURCE: 10 | | (UNKNOWN GEOLOGICAL SOURCES) | | | | | |
| 94 | 10 | O.24 | 44.18 | 60.85 | 1.77 | 5.68 | O.05 |
| 95 | 10 | O.26 | 50.24 | 89.56 | 2.37 | 0.35 | 0.03 |
| 96 | 10 | O.17 | 46.62 | 68.95 | 2.18 | O.25 | O.02 |
| 97 | 10 | 0.21 | 46.44 | 78.16 | 43.54 | 0.30 | O.04 |
| 98 | 10 | O.14 | 47.24 | 73.46 | 0.82 | O.26 | O.06 |
| 99 | 10 | 0.17 | 46.81 | 133.80 | 3.67 | O.49 | O.06 |
| 100 | 10 | O.16 | 46.32 | 137.65 | 2.82 | O.64 | O.05 |
| 101 | 10 | O.18 | 46.25 | 128.32 | 2.96 | O.29 | O.04 |
| 102 | 10 | 0.19 | 45.32 | 92.64 | O.69 | O.25 | 0.01 |
| 103 | 10 | O.15 | 46.07 | 63.96 | 1.35 | O.12 | O.04 |
| 104 | 10 | O.17 | 43.20 | 97.44 | 1.86 | O.33 | 0.05 |
| 105 | 10 | 0.18 | 49.19 | 103.82 | 1.34 | O.32 | O.04 |
| 106 | 10 | O.16 | 45.39 | 139.71 | 1.26 | O.33 | O.04 |
| 107 | 10 | 0.28 | 48.95 | 120.46 | O.96 | O.69 | O.02 |
| 108 | 10 | O.14 | 46.31 | 113.59 | 1.67 | O.24 | O.07 |
| 109 | 10 | 0.24 | 44.62 | 95.37 | 4.50 | 0.32 | O.04 |
| 110 | 10 | O.17 | 43.96 | 82.04 | 1.21 | O.29 | O.07 |
| 111 | 10 | O.16 | 43.32 | 77.77 | 0.61 | O.21 | O.03 |
| 112 | 10 | 0.30 | 46.12 | 121.58 | 1.42 | O.74 | O.04 |
| 113 | 10 | O.34 | 45.51 | 108.24 | 1.94 | 0.51 | O.03 |
| 114 | 10 | O.15 | 46.37 | 111.21 | 0.93 | O.35 | O.42 |
| 115 | 10 | 0.29 | 47.77 | 144.58 | 1.30 | O.73 | O.23 |
| 116 | 10 | O.13 | 45.29 | 122.86 | 1.03 | 0.91 | 0.05 |
| 117 | 10 | O.34 | 48.00 | 233.86 | 9.88 | 1.13 | O.04 |
| 118 | 10 | 0.13 | 43.39 | 128.54 | O.89 | 0.68 | 0.04 |
| 119 | 10 | O.16 | 45.92 | 129.84 | 3.00 | O.40 | O.08 |
| 120 | 10 | 0.22 | 46.37 | 71.76 | 1.38 | O.26 | 0.02 |
| 121 | 10 | O.16 | 46.16 | 142.41 | 1.46 | 0.27 | O.02 |
| 122 | 10 | O.22 | 45.09 | 92.38 | 1.68 | O.29 | 1.55 |
| 123 | 10 | O.15 | 44.04 | 123.88 | 1.71 | 0.45 | O.04 |
| 124 | 10 | O.13 | 44.02 | 315.21 | 1.74 | O.43 | O.10 |
| 125 | 10 | O.18 | 45.95 | 141.81 | 3.68 | O.43 | 0.10 |
| 126 | 10 | O.19 | 46.59 | 167.22 | 3.09 | O.26 | O.07 |
| 127 | 10 | 0.28 | 44.39 | 100.86 | O.89 | 0.43 | O.13 |
| 128 | 10 | O.21 | 46.97 | 111.93 | 1.02 | O.31 | 0.22 |
| 129 | 10 | O.34 | 46.19 | 105.32 | 1.07 | O.46 | O.01 |
| 130 | 10 | O.15 | 44.23 | 132.92 | 2.88 | O.38 | 0.06 |
| 131 | 10 | O.20 | 45.55 | 125.46 | 2.45 | O.31 | O.01 |
| 132 | 10 | O.27 | 46.48 | 143.57 | 3.29 | O.30 | 0.03 |
| 133 | 10 | 0.35 | 47.23 | 141.6 | 2.04 | 21.98 | O.07 |
| 134 | 10 | O.28 | 46.38 | 83.93 | 3.71 | O.56 | O.05 |
| 135 | 10 | O.33 | 46.64 | 95.62 | 1.32 | 0.80 | O.04 |
| 136 | 10 | 0.13 | 47.01 | 106.11 | 1.11 | O.68 | O.03 |
| 137 | 10 | O.30 | 48.47 | 236.43 | 1.21 | 2.58 | O.02 |
| 138 | 10 | 0.36 | 48.68 | 141.46 | 1.68 | O.52 | O.04 |
| 139 | 10 | O.12 | 46.75 | 100.62 | O.59 | 0.35 | O.09 |
| 140 | 10 | 0.25 | 44.43 | 97.26 | O.92 | 4.04 | 0.03 |
| 141 | 10 | O.11 | 46.07 | 63.54 | 2.04 | 2.05 | O.09 |
| 142 | 10 | O.26 | 41.81 | 109.56 | 50.04 | O.38 | O.05 |
| 143 | 10 | O.30 | 47.26 | 74.16 | 0.53 | O.57 | 0.02 |
| 144 | 10 | O.14 | 45.33 | 120.45 | 1.25 | 0.33 | O.06 |
| 145 | 10 | O.33 | 47.41 | 147.49 | 2.19 | O.49 | O.02 |
| 146 | 10 | O.22 | 42.22 | 166.08 | 1.13 | 1.53 | O.02 |
| 147 | 10 | O.12 | 46.24 | 252.16 | 1.37 | 0.60 | O.01 |
| 148 | 10 | 0.12 | 46.20 | 99.81 | 1.88 | O.28 | O,11 |
| 149 | 10 | O.35 | 47.32 | 167.16 | 1.93 | O.39 | O.00 |
| 150 | 10 | 0.34 | 44.37 | 274.25 | 6.98 | 1.28 | O.28 |

analyses of the McCollum cache using non-intrusive short-lived isotope-producing INAA methods with those from geological materials shows that several McCollum artifacts are similar to KRF both in their visual and chemical makeup. Ongoing research may permit more precise sourcing of these HBL cluster McCollum materials, once a greater suite of in situ geological HBL has been chemically characterized. The ability to chemically analyze a group of artifacts without changing them in any appreciable way can, in many research situations, provide the analyst with a potential for obtaining greater numbers of samples and the curator with greater peace of mind.

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P.J. Julig
Department of Sociology and Anthropology, Laurentian University, Sudbury, Canada P3E 2C6
L.A. Pavlish
Archaeometry Laboratory, Department of Physics, University of Toronto, Toronto, Canada M5S 1A7 C. Clark
Midwest Archaeological Center, National Park Service, Federal Building, Lincoln, Nebraska, USA 68508
R.G.V. Hancock
SLOWPOKE Reactor Facility and Department of Chemical Engineering and Applied Chemistry, University of Toronto, Toronto, Canada M5S 1A4