Flint mining in prehistory recorded by *in situ*-produced cosmogenic ^{10}Be

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The development of mining to acquire the best raw materials for producing stone tools represents a breakthrough in human technological and intellectual development. We present a new approach to studying the history of flint mining, using *in situ*-produced cosmogenic ^{10}Be concentrations. We show that the raw material used to manufacture flint artifacts ≈300,000 years old from Qesem Cave (Israel) was most likely surface-collected or obtained from shallow quarries, whereas artifacts of the same period from Tabun Cave (Israel) were made of flint originating from layers 2 or more meters deep, possibly mined or quarried by humans.

The first archaeological evidence of the use of stone tools dates to ≈2.5 million years ago (1). In prehistory, one of the most widely used raw materials was flint, a microcrystalline form of quartz. Because flint quality varies, the choice of raw materials for producing tools is important; flint mined from underground is generally more easily workable than surface-collected material, which is not always present in large quantities and usually weathered by atmospheric agents (2, 3). There are only a few reports of flint mining sites in the early Paleolithic, such as the Acheulian complex at Isampur (India) (≈1.0 million years B.P.),[¶¶] the Lower-Middle Paleolithic in Mount Pua (Israel) (≈200,000 B.P.) (5), and the Middle Paleolithic in Qena (Egypt) (≈50,000 B.P.) (6). The approach presented in this article can be used to directly analyze flint artifacts from different stratigraphic layers in prehistoric caves, leading to information on the provenance of the raw material. We show that the analysis can determine whether the raw material originated from deep layers (1 m or more), possibly mined by humans. The application of this method will contribute to our understanding of the history of flint mining in different regions of the world and can be expanded to other raw materials.

^{10}Be *in Situ* Production in Flint Minerals

The interaction of showers of high-energy primary and secondary cosmic ray particles with the atmosphere and shallow matter in the earth's crust produces a number of long-lived cosmogenic isotopes by nuclear reactions (7, 8). The cosmogenic isotope *in situ* buildup in rocks has been extensively studied both theoretically and experimentally by accelerator mass spectrometry methods of analysis (ref. 9 and references therein). *In situ* cosmogenic production was shown to involve a complex balance between various geophysical processes and parameters: (*i*) altitude- and latitude-dependent cosmic-ray particle fluxes; (*ii*) proton and neutron absorption coefficients in the earth's crust (the mean attenuation length for spallation reactions in rocks is about $\Lambda \approx 160$ g/cm^2, and the average rock density is $\rho = 3$ g/cm^3); (*iii*) the erosion rate of surface rocks; (*iv*) the burial history of rocks; and (*v*) the production rate by slow (stopping) and fast muons, penetrating particles produced as secondary particles in the shower caused by cosmic particles. The case of cosmogenic ^{10}Be ($T_{1/2} = 1.5$ million years) *in situ* production in quartz (SiO$_2$) has received particular attention largely because of the stability of the target matrix. The main reaction, occurring between the surface and 1.5–2 m of depth, is spallation of oxygen (and, to a lesser extent, silicon) by high-energy nucleons. Measured ^{10}Be production rates scaled to sea-level (and high-latitude) range between 4.5 and 5.5 atoms per gram per year (see, for example, refs. 10 and 11). The contribution of muons is minor (≈2%) at the earth's surface but becomes dominant at depths greater than ≈2 m because of a much larger attenuation length (5300 ± 950 g/cm^2 for fast muons) (12–14). Measurements of *in situ* produced ^{10}Be in surface and subsurface quartz are found to depend critically on the local surface erosion rates, determining the residence times of a mineral on the surface or at a given depth. These rates were shown to vary greatly according to climatic and geographic situations, generally between ≈2 m per million years in dry and arid locations (15, 16) and 20–40 m per million years in rainy areas (17).

We emphasize the key feature that if a flint nodule was extracted by deep mining (1 m or more) to provide raw material for the manufacture of tools, the artifacts will necessarily bear a low ^{10}Be content signature. Typical concentrations measured in deep-lying quartz minerals are constrained to values of the order of 10^4 to 10^5 ^{10}Be atoms per gram, whereas surface quartz displays much wider concentration distributions, up to several times 10^6 ^{10}Be atoms per gram (15–17). Subsequent ^{10}Be radioactive decay or buildup in a flint artifact, which was deposited in a cave, is negligible for periods less than ≈10^5 years. Radioactive decay could be important for older archeological samples. On the other hand, artifacts manufactured from flint collected at or close to the surface will probably have higher ^{10}Be contents, depending on their exposure histories.

We show here that flint nodules are closed systems with respect to *in situ* ^{10}Be and that it is possible to distinguish between deeply quarried material and surface collection or shallow mining of raw material used in the manufacture of flint tools.

Sample Descriptions

Several groups of samples were measured (Table 1). They are as follows.

Group 1: Deeply Buried Flint Nodules. We analyzed two deep-lying flint samples from nodules extracted from 1.4 and 0.9 m below surface at the site of Ramat Tamar, south of the Dead Sea, at ≈50 m below sea level. According to ref. 18, the Ramat Tamar nodules were formed ≈90 million years ago, together with the Turonian limestone in which they are still embedded. Another sample was collected from a road-cut through Mt. Carmel in northern Israel (8 m below the surface). These three nodules

Table 1. ^{10}Be concentrations in the measured samples

Specimen	Provenance	Depth, cm	^{10}Be, 10^5 atoms per g
Buried			
LRT2	Negev	90	0.15 ± 0.07
LRT1	Negev	140	0.22 ± 0.04
LRT1	Negev	140	0.08 ± 0.03
LRT1	Negev	140	0.03 ± 0.03
BeAl8	Galilee	800	0.07 ± 0.06
BeAl8	Galilee	800	0.12 ± 0.05
Surface			
32	Negev	—	0.10 ± 0.07
32	Negev	—	0.26 ± 0.11
32 Z	Negev	—	0.33 ± 0.06
32 Z	Negev	—	0.27 ± 0.05
33	Negev	—	0.76 ± 0.11
COLw/o p	Negev	—	1.86 ± 0.12
COLw/o p Z	Negev	—	1.76 ± 0.10
LRT7	Negev	—	0.81 ± 0.18
LRT7	Negev	—	0.92 ± 0.21
LRT7	Negev	—	1.14 ± 0.15
LRT7 Z	Negev	—	1.27 ± 0.14
LRT8	Negev	—	0.23 ± 0.06
LRT8 Z	Negev	—	0.16 ± 0.04
BeAl9	Galilee	—	0.27 ± 0.11
BeAl9 Z	Galilee	—	0.22 ± 0.03
RM1	Galilee	—	0.59 ± 0.06
RM3	Galilee	—	0.25 ± 0.06
Qesem			
QC5		365–370	0.18 ± 0.06
QC1		375–380	0.41 ± 0.05
QC8		590–610	0.15 ± 0.04
QC10		615–620	0.32 ± 0.05
QC12		595–640	0.46 ± 0.05
QC13		665–670	0.67 ± 0.06
QC16		670–675	1.39 ± 0.08
QC7		750–840	0.17 ± 0.04
QC14		780–810	0.53 ± 0.06
Tabun			
TB1		822	0.12 ± 0.07
TB3		831	0.09 ± 0.08
TB4		839	0.08 ± 0.09
TB22		965	0.10 ± 0.08
TB21		1090	0.14 ± 0.10
Ramat Tamar			
LRT12a		—	0.08 ± 0.03
LRT6		—	0.13 ± 0.04
LRT6 Z		—	0.27 ± 0.03
LRT12b		—	0.28 ± 0.05
LRT12b		—	0.14 ± 0.04
LRT12b Z		—	0.24 ± 0.03

The ^{10}Be atoms per g of flint and the depths at which the samples were collected are listed. —, Collected on the surface. The depths of the Qesem and Tabun samples refer to datum, which represents the surface of the highest preserved sediment in the site. See the text for sample descriptions and Table 2 for the method of data analysis. Z, measured in Zurich. Quoted uncertainties are random errors resulting from statistical counting and background subtraction. The overall systematic uncertainty, composed of the error (2%) in the calibration of the Weizmann standard relative to the ETH/PSI standard and of the uncertainty (2.6%) of the ETH/PSI standard (see text), is 3.5%.

were probably never exposed at the surface after rock formation. These samples were chosen to determine the amount of ^{10}Be found in deeply buried flint nodules.

Group 2: Surface Collected Flints. This set of eight flints was collected from surface exposures at different locations in Israel (Negev Desert and Galilee). Samples LRT7 and LRT8 (see Table 1) are chert rocks present on the extant surface in Ramat

Tamar. These flints were collected to have at least a small reference distribution of random flint collection from the present surface. However, the present distribution of surface flints is not necessarily the same as that of surface raw material exploited in ancient times.

Group 3: Neolithic Flint Artifacts from a Quarrying Site. This group consists of flint artifacts from an archaeological site in Ramat

Tamar. This Neolithic quarrying complex includes a village, flint quarries (1.5–2 m deep), and workshops (19), providing archeological evidence that these artifacts were produced with the same material as Group 1. These artifacts were produced ≈10,000 years ago and then left on the surface, where they can still be found. This group was chosen as a test of our hypothesis and to determine whether quarried material left on the surface is contaminated by the relatively abundant atmosphere-produced [10]Be. Although an exposure time of 10^4 years is negligible for [10]Be buildup for this application, it should be sufficient to test whether the flint behaves as a closed system with respect to *in situ*-produced [10]Be.

Group 4: Acheulo-Yabrudian Flint Artifacts from Tabun Cave. This group consists of five flint artifacts from the lower levels of Layer E of Tabun Cave (Mt. Carmel). Tabun Cave has a long stratigraphic section. It is perhaps the most important prehistoric cave in the region, because it serves as the type locality to which all other chronologies, based on flint tool typologies and radiometric dates, refer. Layer E is from the Acheulo-Yabrudian period (≈350,000–200,000 B.P.) (20). The origin, and hence the exposure history, of the raw material used for the manufacture of these flints is unknown. The flints were deposited in a cave, and because almost all karstic caves in the Levant have thick limestone or dolomitic roofs, we assume that the flints were shielded from cosmic rays. The flints were subsequently covered by layers of sediment. The sediments are, for the most part, dry. The stratigraphic depths at which the artifacts were found are listed in Table 1.

Group 5: Acheulo-Yabrudian Flint Artifacts from Qesem Cave. This group consists of nine artifacts from Qesem Cave (central Israel). Qesem Cave is a newly discovered prehistoric cave located on the coastal plain of Israel east of Tel-Aviv. The preliminary dating of the Acheulo-Yabrudian layers indicates an age range of 350,000–200,000 years B.P. (21). This is consistent with the typology of flint artifacts found at Qesem Cave, which is comparable to that of Tabun Layer E. The sediments are, for the most part, dry. There is no evidence of prehistoric flint quarries in the vicinity of either Tabun or Qesem caves.

Analytical Procedures
The cleaning procedure for flint and the extraction of Be is based on Kohl (22) and Ivy (23). The flint is first crushed into small particles (<50 μm), and organic and carbonate material is then removed by acid dissolution (3 M HCl + 3 M HNO_3). Because of mass loss (30–40%) during crushing and cleaning (between three and five steps), it is necessary to start with a flint artifact of at least 20–25 g. Several etching steps with 2% HF in an ultrasonic bath are performed to remove [10]Be of atmospheric origin; the criterion used in this procedure is to reach a steady-state content of stable Al of 100–400 ppm, monitored by inductively coupled plasma MS. After the addition of 1 or 0.5 mg of Be (Aldrich Atomic Absorption 1% HCl solution of Be), used as chemical carrier, the cleaned silica is slowly dissolved in Teflon beakers in 40% HF (≈40–60 ml) and 70% $HClO_4$ (≈20 ml). The residue is fumed at least three times with 5 ml of 70% $HClO_4$ to eliminate the remaining HF. The residue is then dissolved in 1 M HCl, and hydroxide precipitation is performed at pH 8.5. This step removes Ca. The hydroxides are subsequently dissolved in 8 M HCl, and Fe is separated with diisopropylether. Al and Be are separated from the 1 M HCl solution by using a cation exchange column (Sigma AG 50W-X8). Beryllium is eluted with 1 M HCl and aluminum with 4.5 M HCl. The two separated fractions are precipitated as hydroxides at pH 8.5 and then ignited in the oven at 850°C to obtain BeO and Al_2O_3; the latter is stored for [26]Al analysis. The entire chemical procedure is performed in Teflon containers to reduce to a

minimum the presence of [10]B, which severely interferes with the [10]Be measurement. The BeO material, mixed with Nb powder for bulk (BeO:Nb ≈1:20 in mass) is then pressed in a Cu sample holder to be inserted in the ion source of the accelerator mass spectrometry facility (24) at the 14UD Pelletron Koffler accelerator of the Weizmann Institute. BeO$^-$ ions produced by Cs$^+$ sputtering were selected and accelerated with a terminal voltage of 8 MV. [10]Be^{3+} ions, after magnetic and velocity analysis, are transported to the detector. The latter is essentially composed of two parts: a Xe-filled cell, which stops interfering [10]B ions (flux at detector ≤4.5 × 10^5 [10]B^{3+} ions per second), and an isobutane-filled ionization chamber where [10]Be ions are completely stopped. The measurement of the partial and total energy loss in the gas identifies the ions unambiguously. The measurement sequence consists of alternate measurements of [9]Be (charge current) and [10]Be (counting). Both measurements are averaged over the transmission curve of the accelerator by scanning the accelerating terminal voltage; this procedure has been shown to reduce uncertainties due to the different ion-optical behavior of [9]Be^{3+} and [10]Be^{3+} (mainly caused by the Coulomb explosion of the BeO$^-$ molecular ion).

The [10]Be/[9]Be ratio, r, is measured relative to an internal [10]Be standard. The number of [10]Be atoms in the processed BeO material is obtained by multiplying r by the amount of [9]Be carrier used in the chemical procedure. This number includes the [10]Be contribution introduced during the chemical procedure. This contribution [(0.8 ± 0.1) × 10^6 [10]Be atoms] is estimated from an average of procedure blank measurements (using the [9]Be carrier without flint sample). Final values were obtained by subtracting the procedure blank background from the measured values of [10]Be atoms. The internal [10]Be standard used at the Weizmann Institute was calibrated at the EN Tandem Accelerator of Eidgenössische Technische Hochschule Paul Scherrer Institute (ETH/PSI) by comparison with an ETH/PSI standard. These measurements gave an average value of [10]Be/[9]Be = (1.10 ± 0.02) × 10^{-11} for the Weizmann Institute internal standard (25). Eight BeO samples were also measured at ETH/PSI.

Results and Discussion
Table 1 shows the results of [10]Be concentration measurements in flints.

Fig. 1 shows the [10]Be concentration frequency distributions derived from table 1 for the different groups. The distributions are normalized to the number of samples measured in each group. The samples prepared from buried nodules show very small [10]Be contents, of the order of 0.1 × 10^6 to 0.2 × 10^6 [10]Be atoms per gram of flint. These values are consistent with the saturation concentration due to muonic interaction only (see, for example, ref. 12). The similarity between the distributions of Ramat Tamar artifacts and of buried nodules is in agreement with the archaeological evidence that Ramat Tamar flint artifacts were manufactured in the Neolithic from deeply mined raw materials. It also confirms our earlier observation (26) that flint, even when exposed on the surface, is not contaminated by atmosphere-produced [10]Be and behaves as a closed system with respect to *in situ* [10]Be. Interestingly, Tabun artifacts are observed to have concentrations similar to the buried nodules and to the Ramat Tamar set. Both the surface flints and the Qesem Cave artifacts, on the other hand, have a much wider distribution of [10]Be contents. The behavior of the surface-collected set indicates different exposure times and erosion histories at each location, because not all of the samples are from the same area. For samples from Qesem cave, the possibility of shallow as well as deep mining together with surface collection cannot be excluded. Shallow mining seems to have been used at the Lower-Middle Paleolithic site at Mt. Pua (Israel) (5), where signs of multiple shallow quarrying locations, piles of rock debris, and many examples of flint nodules can be found.
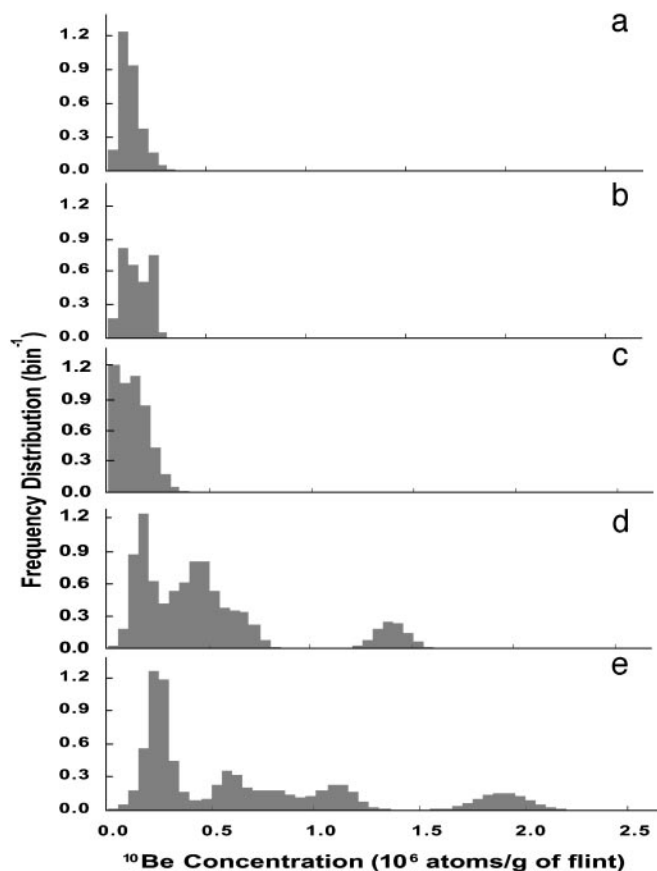
Fig. 1. Distributions of *in situ* produced ^{10}Be concentrations measured in flint samples for buried nodules (*a*) (*n* = 3), Ramat Tamar artifacts (*b*) (*n* = 3), Tabun cave artifacts (*c*) (*n* = 5), Qesem cave artifacts (*d*) (*n* = 9), and surface-collected flint (*e*) (*n* = 8). See the text for details on the sample groups. The *y* axis represents the number of samples per bin of concentration (*x* axis). Each distribution is normalized to the number of measured samples in the group.

Because of the limited number of samples in each set, a statistical analysis is useful to estimate the level of confidence to which one can establish similarity or dissimilarity between measured pairs of sets. Table 2 lists the results for different pairs. The statistical test in the second and third columns estimates the probability that both members of a pair of sets are randomly sampled representations of a single unknown distribution (or, more technically, of two parent distributions whose mean values are the same). This probability for the buried set and Qesem artifacts is very low. In contrast, the result of the test for the buried set and Tabun samples states that no significant difference exists between these two sets of samples. The same conclusion holds for the buried nodules and the Ramat Tamar artifacts. The data show that the Tabun artifacts were most likely manufactured from flint originating in layers 2 m deep or deeper. This finding suggests that humans in this region in the Lower-

Table 2. Statistical test results

Pair of sets	Prob. I, %	Prob. II, %
Buried–Qesem	1	4
Buried–Tabun	>99	92
Buried–Ramat Tamar	>99	83
Buried–Surface	1	3
Surface–Qesem	42	53

The table compares the distributions of ^{10}Be concentrations for pairs of sample sets (see text). The second and third columns list the results of a Wilcoxon nonparametric sum of rank test (27). The test estimates the probability (Prob. I) that both members of a pair of sets have a single parent distribution with no *a priori* assumption of the shape of the parent distribution. In order to estimate the effect of the experimental errors (s_i) of individual data points (c_i) in each set (see Table 1) on the probabilities of the second column, the following procedure was followed. For each sample set 1,000 randomly sampled sets were generated by the expression $c_i = c_i + r_i s_i$ (j = 1,000), where r_i is a random number obtained from a standardized Gaussian distribution. The Wilcoxon test (identical to the second column) was applied to each pair of randomly sampled sets. The third column (Prob. II) lists the mean of the obtained probabilities for each pair of sets. No significant difference between Prob. I and Prob. II is observed.

Middle Paleolithic were already mining, and hence investing efforts to obtain quality flint nodules. It is conceivable that the flint was derived from shallower depths in outcrops exposed on cliffs or from rapidly eroding exposures. For these scenarios we would, however, have expected to find a larger range in ^{10}Be concentrations. Conversely, the data from Qesem cave establish that its artifacts were not made exclusively from deeply buried flint.

Interestingly, the one artifact (QC16) from Qesem cave, having the highest ^{10}Be concentration and introduced as a control sample, was of poor quality flint; the raw material from which the artifact was made was presumably collected on the surface.

Although the number of samples analyzed in the present study is limited, the results are statistically significant and demonstrate the potential of this new methodology for exploring the history of flint mining. The immediate future prospect is to systematically investigate the differences in ^{10}Be concentrations in flint artifacts from different stratigraphic layers in Qesem and Tabun caves, in order to document the development of flint mining in the region. It will also be of much interest to determine whether mined flint was used for the manufacture of certain tool types and not others (4).

The methodology described here can, in principle, be applied to other rock types used for the production of artifacts. Together with petrographic and geochemical analyses providing information on flint provenience, the ^{10}Be methodology described here will result in a more complete picture of the manner in which humans developed the cognitive abilities to optimize the use of raw materials for tool production.

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