New datings of Amudian layers at Qesem Cave (Israel): results of TL applied to burnt flints and ESR/U-series to teeth

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Acheuleo-Yabrudian Cultural Complex (AYCC) emerged in this region during Paleolithic times. At the end of the Lower Paleolithic period, the Acheuleo-Yabrudian Cultural Complex (AYCC) emerged in this region. The AYCC includes three major lithic industries: Acheulo-Yabrudian dominated by handaxes and Quina scrapers; Yabrudian dominated by Quina scrapers; and Amudian dominated by blades. The blade-dominated Amudian industry of the AYCC is consistently present at Qesem Cave while the Quina, scraper-dominated, Yabrudian industry appears in stratigraphically distinct units in two areas of the cave. The Amudian artifact collection contains a sequence of MIS 10 and 8. The sequence of MIS 10 and 8 is the first attempt to apply the TL and ESR/U-series dating methods at this site and these methods yielded results which are generally in agreement. They support a time interval of hominid-bearing occupation of the areas of the cave where Amudian artifacts were recovered during MIS 8 and likely 9 for the Deep Pit Area, and during MIS 8 and possibly 7 for the Upper part of the sequence (Square K/10 and the Eastern Microfauna-Bearing Area). An older occupation of the cave is also conceivable on the base of two dating results (MIS 11).

1. Introduction

Connecting Africa and Eurasia, the Levant is naturally considered as a corridor which enabled human migrations during Paleolithic times. At the end of the Lower Paleolithic period, the Acheulo-Yabrudian Cultural Complex (AYCC) emerged in this region where it remained confined. This cultural complex is thus known in a limited series of sites, from which the most famous is probably the Tabun Cave — Mount Carmel — (Garrod and Bate, 1937). At Tabun, the chronology of the AYCC industries has been questioned for a long time (Grün and Stringer, 2000) but the last series of dates seems to indicate that this cultural complex could be older than 260 ka in this site (Mercier and Valladas, 2003). Additional chronological results gave also indications about the antiquity of this cultural complex as, for example, the U-series dating of a flowstone covering the Acheuleo-Yabrudian layers discovered at Jamal Cave (Weinstein-Evron et al., 1999), or the thermoluminescence dates obtained for the Early Levantine Middle Paleolithic (‘Tabun D-type’ industry) at Hayonim Cave (Mercier et al., 2007), which post-date the Acheulo-Yabrudian industries.

The AYCC includes three major lithic industries: Acheulo-Yabrudian dominated by handaxes and Quina scrapers; Yabrudian dominated by Quina scrapers; and Amudian dominated by blades. The blade-dominated Amudian industry of the AYCC is consistently present at Qesem Cave while the Quina, scraper-dominated, Yabrudian industry appears in stratigraphically distinct units in two areas of the cave (Barkai et al., 2009). The Amudian lithic assemblage shows a very early and well established blade production technology, systematically used for the serial production of predetermined laminar items (Shimelmitz et al., 2011). Blades in small numbers do appear in Yabrudian assemblages, and were made by Amudian blade-production standards. Scrapers, including Quina scrapers, are dominant in Yabrudian tool assemblages. Qesem Cave is one of the rare sites which provided exclusively Acheuleo-Yabrudian finds clearly dominated by the Amudian
industry (Barkai et al., 2003, 2005; Gopher et al., 2005; Shimelmitz et al., 2011), and thus offers the opportunity to enhance our knowledge of this very early blade industry and precise its chronological position. Moreover, the Qesem Amudian bearing layers yielded also human dental remains showing affinities with those of modern populations recovered in the Middle Paleolithic sites of Skhul and Qafzeh (Hershkovitz et al., 2011).

1.1. General presentation of the cave

Qesem Cave is situated 12 km east of the Mediterranean coast of Tel Aviv (Fig. 1) in a hilly limestone terrain. The cave is a sediment-filled chamber estimated at ~20 × 15 m in size and ~10 m high (Gopher et al., 2005) although a larger and deeper chamber yet to be excavated was recently uncovered. Since the discovery in 2000, ongoing excavation has exposed ~9.5 m of deposits containing sediments of natural and anthropogenic origins. The stratigraphical sequence is divided into two parts: the lower (~5.5 m thick) was deposited in a closed karstic chamber, while the upper part (~4.5 m thick) was deposited when the cave was more open as indicated by the presence of calcified rootlets (Karkanas et al., 2007). Today, the roof of the cave is missing in its entirety due to road construction (Fig. 2) and just the infilling and the wall of the cave are exposed. It is probable that only parts of the roof, on top of the rock shelf and the upper area (N-Q/8-12) survived and were destroyed by road construction.

The nature of the rich lithic and faunal assemblages, supported by a plethora of U-series dates (Barkai et al., 2003; Gopher et al., 2010) indicates that the infilling sequence corresponds to Middle Pleistocene deposits. These deposits were continuously affected since their deposition by recurring subsidence, erosion, and possibly fracturing or cracking changing the cave's shape, and finally acting as post-depositional agents (Frumkin et al., 2009). As indicated by the presence of ashes, the use of fire is attested all along the sequence and micromorphological studies indicate that fire was more intensively used in the upper part of the sequence than in the lower part (Karkanas et al., 2007). As a consequence, numerous burnt flints (Lemorini et al., 2006) and bones (Stiner et al., 2009, 2011) were discovered in the various deposits.

The lithic assemblages of the cave are dominated by the Amudian blade industry of the AYCC (Gopher et al., 2005, 2010; Barkai et al., 2005, 2009; Shimelmitz et al., 2011). This complex refers to the chrono-cultural entity postdating the Lower Paleolithic Acheulian, predating the Middle Paleolithic Mousterian and corresponds to Jelinek’s “Mugharan Tradition” (Jelinek, 1981, 1992). Another lithic industry of the AYCC is the scraper-dominated Yabrudian that appears at Qesem Cave in three stratigraphically and spatially distinct areas (Barkai et al., 2009; Lev, 2010).

The Amudian industry is characterized by systematic blade production (Shimelmitz et al., 2011), and many of the tools were made on blades, including backed and retouched blades, burins, end scrapers and especially abundant naturally backed knives. A flake component also exists in the Amudian, and side scrapers appear in variable, generally low frequencies (Barkai et al., 2005; Gopher et al., 2005; Barkai et al., 2009). Handaxes are rare at Qesem Cave and only a few were found in Amudian layers. Use-wear analysis of the Amudian assemblages documented the outstanding state of preservation of the flint items and indicated that butchering was the main activity at the site (Lemorini et al., 2006). Carcasses were processed mainly with blade cutting tools (knives) used specifically for skinning, disarticulation, and cutting meat while a specific type of recycled, double-ventral flakes was used for cutting meat too (Barkai et al., 2010). The Yabrudian industry shows significantly lower frequencies of blades compared to the Amudian and is dominated by Quina scrapers (up to 50% of the shaped tools). Yabrudian has hitherto been found in three distinct and limited areas in the cave; one under a rock shelf in the central part of the cave (elevations 560–600 cm below datum), one in the southwestern part of the cave (elevations ca. 600–630 cm below datum), and one by the eastern wall of the cave (elevations 130–150 cm below datum) and they all appear to be contemporaneous, in the sense of field relations, to adjacent Amudian contexts.

The faunal assemblages of the site are dominated by fallow deer but other species such as auroch (Bos), horse (Equus), wild pig (Sus), red deer (Cervus) and tortoise (Testudo) are present. Mostly prime-aged fallow deer were hunted and selected body parts were transported into the cave. Cut marks and burning signs were found

Fig. 1. Location of the Qesem Cave and other important Paleolithic sites.

Fig. 2. Picture of the cave before the road construction.
on quite an impressive number of the bones (Stiner et al., 2009, 2011) bearing indications of butchering and marrow extraction. The presence of bones in clear association with indications of human activities, the patterns of body part selection and transportation, the large scale of human manipulation (cutting, fracturing, burning, marrow extraction) and the lack of evidence for non-anthropogenic accumulation argue in favor of a faunal assemblage that was brought to the cave by humans.

A study of human dental remains (Hershkovitz et al., 2011) indicated that the hominins inhabiting Qesem Cave were not Homo erectus but rather most similar to later modern populations (e.g. Skhul/Qafzeh) of this region, notwithstanding some Neanderthal affinities as well. Based on a newly developed bio-energetic model it was recently suggested that dietary stress in the Middle Pleistocene Levant caused by the disappearance of elephants triggered the demise of H. erectus who was highly dependent on very large animals. Those in the population better adapted to the new diet constraints — namely hunting larger numbers of smaller and faster animals in order to provide sufficient caloric intake to compensate for the loss of the elephants — indicate the appearance of a new hominin lineage (Ben-Dor et al., 2011). This suggestion is consistent with the recently published innovative model for the evolution of Pleistocene human populations of Europe (Bernídez de Castro and Martínón-Torres, 2012) suggesting the Levant as a Central Area of Dispersals of Eurasia (CADE), an “origin region” for human species biodiversity.

1.2. Previous chronological studies

The absolute/radiometric dating project of Qesem Cave was designed to include two major aspects based on the availability of potential samples in the cave. One line of work was directed toward the dating of speleothems present on-site by way of its being a karstic system that was active for a long time. Speleothems were sampled from various parts of the cave and dated by the U-series method. The first series of $^{230}$Th/$^{234}$U dates suggested that the occupation of the cave began around 380 ka and ended possibly around 200 ka (Barkai et al., 2003). A later project of U-series dating concluded that the human occupation started sometime around 420 ka and ended shortly before 200 ka (Gopher et al., 2010). In some cases, dated speleothems enabled offering dates for specific contexts: for example, sample QE-06-1 is a speleothem crust found under the eastern part of a rock shelf and it has been shown, following micromorphological analysis, that it is a pure speleothem formed in situ on top of unworked sediments. The speleothem itself showed no signs of dissolution or re-precipitation and yielded an age of 299 ± 30/–30 ka which is therefore a minimum age for the 5.5 m of the sediment sequence below it. As indicated by this example, speleothems are formed naturally and consequently, they need to be related to the human presence and activities in the cave by inference.

In order to overcome this limitation, a second line of work was to get dates from burnt flints and animal teeth both of which were clearly brought into the cave by the inhabitants and are thus directly related to human activities. The dating methods applied to these samples, i.e. Tholuminescence — TL — to burnt flints, and combined Electron Spin Resonance and U-series methods — ESR/U-series — to teeth, are then major to establish the chronology of Qesem Cave and their results constitute the topic of this paper.

2. TL and ESR/U-series dating

The first stage of this TL and ESR/U-series dating project was largely devoted to an attempt in defining the chronological range of the cave deposits and thus, samples were taken in all parts of the sequence readily exposed and available in summers 2004 and 2005 (Fig. 3). The results of this stage are presented here but future TL/ESR dating stages are underway and will be devoted to specific areas of the cave and specific contexts (like contexts where human teeth were found, constructed hearths, etc.) and to the resolution of specific questions (sedimentation rate, chronological correspondence of Yabrudian and Amudian activity areas, etc.).

2.1. Context of the dated samples

Sampling and dosimetry for the TL and ESR/U-series dating project started in 2004. The rescue excavation in 2001 reached bedrock in the southern part of the cave which was refilled and covered by the new road soon after the excavation. Thus no access to the southern lower part of the caves’ sequence was possible when resuming excavations in 2004. The samples presented in this paper are thus mainly from the top 1.5 m of the lower stratigraphic sequence of the cave and the bottom/middle parts of the upper stratigraphic sequence. The top 2.5 m of the upper sequence as well as the deepest 2.5 m of the lower sequence (known to date) were not dated yet.

Three major areas were studied and sampled for TL and ESR/U-series dating:

1). Square K/10 at elevations 375–415 cm below datum is situated in the central part of the cave (Fig. 3). Only half of square K/10 was excavated in 2001 showing part of a thick (40–50 cm) archaeological palimpsest of soft, dark-brown sediments with a very dense concentration of flint artifacts and animal bones. This unit is sandwiched, top and bottom, by cemented sediments. It is a stratigraphically clear context and quite distinct.

2). The Eastern Microfauna-Bearing Area is located in squares L-M-N/13–14–15 where rich assemblages of micromammal and reptile bones were recovered from archaeological horizons at elevations 320–545 cm. The area is close to the original eastern wall of the cave and includes the lower part of the upper sequence. The sediments appear in different shades of brown; they are generally soft with small angular or sub-rounded stones and include at least three inclined thin horizons of dark brown to black sediment starting at elevation 430 in the east and reaching elevation 495 in the west (Fig. 4). The inclined beds continue to the west and reach square H–13 of the Deep Pit thus revealing a direct connection between the upper and lower sequences (Fig. 3). A deeper look at the sediments and formation processes in the lower part of this area was provided by a detailed micromorphological study of a 25 cm thick sediments block, collected in square L/14 (nearby the teeth sampled for ESR/U-series) and slightly deeper (between elevations 495 and 520 cm). This block belongs to the uppermost part of the Lower sequence based on sedimentological and depositional considerations (Karkanas et al., 2007; Maul et al., 2011).

3). The Deep Pit Area in the present central part of the cave was originally excavated as the ‘Deep Sounding’ in 2001 (two square meters). The area suffered a collapse of sediments due to the heavy rains of winter 2001 and has thus been enlarged in 2004–2006 (Fig. 3). Since 2008 this area is re-excavated in the hope of reaching bedrock again. In this paper we thus relate to the Deep Pit Area, sampled for TL and ESR/U-series in 2004–2006, as a stratigraphic context not fully understood that seems to have witnessed rather significant post depositional processes as it is located at the center of the main chamber of the cave.

All sediments of the Deep Pit Area belong to the Lower Sequence except for a brownish layer at the top of the Deep Pit stratigraphy that belongs to the Upper Sequence of the cave. This includes a
stony unit consisting of limestone boulders in the western and eastern sections of the Deep Pit which is almost two meters thick in the west. The deposition of the limestone boulder unit is not clear yet, but to our best understanding at the moment it might represent a major pile of stones that accumulated at the center of the cave, a process well known at aging karstic caves (e.g. Frumkin et al., 2009). The micromorphological study based on relevant sediment samples taken in 2004 and 2005 (Karkanas et al., 2007) shows, amongst other things, fine charcoal fragments and burnt bones as well as in situ undisturbed burnt layers with recrystalized wood ash (i.e. hearths) in the western section of the Deep Pit which is clearly an in situ deposition. The observed features suggest that the area of deposition was rather inside a humid closed karstic environment (see also Frumkin et al., 2009).

2.2. Dating methodology

In this study the TL and ESR/U-series methods were applied to burnt flints and herbivorous teeth, respectively. These methods rely on dosimetric measurements performed on these materials but share dosimetric information related to the environment of the samples. In the following are presented the environmental dose rates determined in situ for the three above mentioned areas.

The environmental dose rates were measured by inserting artificial dosimeters in the sediments. A total of ca. 50 dosimeters was planted in the different layers and stayed in place for at least one year, recording the gamma and cosmic rays. In Table S1 (Supplementary Data) are given the gamma dose-rates deduced after subtracting the current cosmic dose-rate for the dosimeters.
relevant to the dated samples (flints and teeth). The gamma dose-rate values vary from ca. 180 to 460 $\mu$Gy/a, and these variations seem dependent on the areas. In square K/10, the dose-rate values vary from 310 to 460 $\mu$Gy/a, with 4 out of 5 values below 380 $\mu$Gy/a. In the lower part of the Deep Pit Area the distribution of the dose rates is more heterogeneous, varying from 178 to 412 $\mu$Gy/a. These variations can be explained by the presence of numerous limestone blocks of different sizes in the sediment. Indeed, limestone has a very low U content compared to that of clay. Moreover, limestone blocks of different sizes are randomly distributed within the sediments of the Deep Pit and the western rim of this area consists of an almost solid unit of limestone boulders (Fig. 4): all these heterogeneities make the evaluation of the gamma dose-rate received from each sample particularly difficult to estimate.

The doses measured with the dosimeters were used for estimating the gamma dose-rate to which each sample, be it a flint or a tooth, was exposed. On Fig. 3 are given the location of the samples (flints and teeth) and dosimeters.

2.2.1. The thermoluminescence (TL) dating method

Numerous flint artefacts showing marks of past heating have been recovered all along the archaeological sequence enabling the application of the Thermoluminescence (TL) dating method. Flint is actually well known as a good natural dosimeter recording all doses to which it is exposed. The TL method, which was successfully applied to several Near-Eastern archaeological sites (Valladas et al., 1987, 1988, 1998; Mercier et al., 1995a,b; Mercier and Valladas, 2003), allows the determination of the time elapsed since the sample has reached in the past a temperature in excess of $\sim 400 ^\circ$C. Heating of a flint was likely accidental, taking place in the habitually used fireplaces spread throughout the cave’s sequence, but it lead to the zeroing of the dose previously recorded in its crystal lattice.

Estimating the dose newly accumulated since this past heating (zeroing) event, called the Paleodose or Equivalent Dose (ED), one can evaluate this time period given that, in parallel, the total dose-rate, i.e. the sum of all doses absorbed by the sample in one year, can be accurately determined.

2.2.1.1. Samples selection and preparation. Fifty six flint samples showing marks of past heating were selected and preliminary TL measurements were performed to check if they had been sufficiently heated in the past for totally zeroing their geological TL signal. The forty specimens that passed this test successfully were prepared following the procedure described by Valladas (1992) which first consists in cutting each flint with a diamond saw in order to isolate its internal part. This procedure aims at selecting a volume of sample not irradiated by alpha and beta particles coming from the surrounding sediments, which are radiative components difficult to estimate with reliability. The internal fraction of the flint is generally used for TL measurements and radioisotope analyses. However, the examination of the flint cut sections indicated that some of them, like QS9 and QS48b, are made of blotches of different

Fig. 4. Eastern Microfauna-Bearing Area.
colors (gray and white) which are suspected to have radioelement contents significantly different from each other (Selo et al., 2009). We then tried to isolate a homogenous part by eliminating all visible colored blottches. After crushing this part, a fraction of the powder was sampled for Neutron Activation Analysis (NAA) in order to determine the radioelement contents — in uranium (U), thorium (Th) and potassium (K) — of the specimen. These radio-isotopes induce what is called the internal dose-rate, mainly due to alpha and beta particles originating from the flint itself. To check the impact of a possible heterogeneous distribution of U within a sample, different grain sizes were measured for seven flints. Table S2 (supplementary data) shows that the U contents obtained on the different fractions are within ±10 for all the samples tested (total of 7 specimens) except for the heterogeneous samples QS9 and QS48b which exhibits variations by a factor 3 and are particularly critical because such large variations can induce differences in the TL properties.

For TL measurements, the 100–160 μm fraction was selected by sieving and then chemically treated with HCl and rinsed with distilled water. After drying, the powder was divided in sub-samples which were irradiated with a calibrated Cs-137 source (dose-rate: ~1.17 Gy/min; Valladas, 1978). The measurement of the induced TL signals was done with a home-made TL equipment (Valladas et al., 1994), with a heating rate of 5 °C/sec, and the UV-blue component of the emission spectrum was selected with appropriate optical filters (MTO 380 nm). These measurements allowed to determine the variations of the TL signal as a function of the absorbed dose, and for each sample, the Equivalent Dose was then calculated following a standard procedure (Mercier et al., 1992), after checking that the plateau test, which allows characterizing the efficiency of the past heating in zeroing any previous dose, was passed.

Additional experiments were performed for determining the relative efficiency of alpha to beta particles in inducing a TL signal. This was done in irradiating different aliquots of each sample with either beta rays, from a Sr-90 source, or alpha rays, from a Am-241 source (Valladas and Valladas, 1982).

2.2.2. The combined ESR/U-series dating method

The combined ESR/U-series method (ESR-US model), taking into account both ESR and U-series data including radioelement contents, isotopic ratios, equivalent doses and external gamma-dose rate, allows the reconstruction of the history of uranium uptake in each dental tissue using a one parameter (p-value) diffusion equation (Grün et al., 1988). Its application to herbivorous teeth (Bahain et al., 1992; Grün, 2009) implies consideration on the incorporation, and thus a better estimation of the age (Falguères et al., 1997; Grün et al., 1998).

2.2.2.1. Samples selection and preparation. Among the thirteen herbivorous teeth initially selected, seven showed a good state of preservation and were prepared for analyses. The samples (four bovid teeth, two horse ones and one of Rhino) come from two different sections in the site. Two teeth QS5054 and QS5055 were sampled in square M/14, between 475 and 485 cm below datum. The five other samples (QS5052, 0503, 0507, 0508, 0510) were taken in an area where the majority of the analyzed burnt flints were found (Fig. 3).

The dental tissues (enamel, dentine, and cement when present) of each tooth were separated and cleaned mechanically using a dentist drill. Enamel was then ground, and sieved. Nine enamel aliquots (about 100 mg each) of the 100–200 μm grain-size fraction were irradiated respectively with doses of 200, 320, 500, 800, 1250, 2000, 3200, 5000, 8000 Gy, using a calibrated Co-60 source (IBL gamma source, LNHB, CEN Saclay). ESR-signal intensities of irradiated and natural aliquots were measured with an EMX Bruker X-band spectrometer, with the following measurement conditions: 10 mW microwave power, 0.1 mT amplitude modulation, 10 mT scan range, 80 ms time constant, 100 kHz modulation frequency. Each measurement was repeated 3 times. The equivalent doses \( D_{eq} \) were determined from the asymmetric enamel signal at \( g = 2.0018 \) by the additive method, using an exponential fitting (Grün, 1989; Yokoyama et al., 1985).

U-series analyses were made for each dental tissue. Samples (between 0.1 and 3 g) were dissolved in HNO3, and spiked with \(^{234}\)U\(^{228}\)Th. U and Th were then first separated using an anion exchange resin column (Dowex 1 × 8; 100–200 mesh) in HCl-form. The separated fractions of U and Th were then purified with an UTEVA resin column and a second anion exchange resin column, respectively. Prior to the analysis of U and Th using alpha-ray spectrometry, the two elements were extracted by TTA/benzene solution and deposited on steel discs.

ESR-US ages were calculated with the ESR-DATA program (Grün, 2009), using an alpha efficiency of 0.13 ± 0.02 according to Grün and Katzenberger-Apel (1994), and Monte-Carlo beta attenuation factors based on the thickness of the tooth enamel and outer layers removed (Marsh et al., 2002). In addition, water content was estimated to be 3 wt% in the enamel and 7 wt% in the dentine and cement. The environmental (external gamma and cosmic) dose rates were deduced from the analysis of the TL dosimeters. Gamma-ray spectrometry was used to determine the radioelement contents (U, Th and K) of sediments in which the teeth were collected and the external beta dose rate to which the dental tissues had been subjected was calculated according to Adamiec and Aitken (1998). In measuring these sediment samples, no significant disequilibrium in the U-series was detected. The effect of Ra and Rn loss in each tissue was determined by combining the alpha-ray and gamma-ray data (Bahain et al., 1992).

2.3. Dating results

The dating results (Table 1 with TL ages, 2 and 3 with ESR-US data) are displayed in four batches on Fig. 5, starting with samples of the mid upper sequence (elevations 375–415 cm below datum; TL ages) from Square K/10, followed by the dates from the Eastern Microfauna-Bearing Area in the lower part of the upper sequence (elevations 475–485 cm below datum; ESR-US ages), and finally the Deep Pit Area (TL and ESR-US ages) in two batches — one in which samples come from the upper part of the Deep Pit (upper part of the lower sequence, elevations 560–660 cm below datum) and a second, for samples from the mid lower sequence (elevations 660–750 cm below datum).

2.3.1. Square K/10

The 8 selected burnt flints from Square K/10 have been found at elevations between 375 and 415 cm below datum, and yielded consistent ages. For this set of samples, the environmental dose-
rate is ~400 μGy/a, typical of an environment enriched in limestone fragments and poor in clay (Mercier et al., 1995b). In contrast to this homogenous dosimetry, these samples show very different U contents, ranging from 0.16 to 3.11 ppm, which generate internal dose-rates between ~80 and 1512 μGy/a. The corresponding equivalent doses D are correlated to these internal dose-rates, showing also large differences (from ~143 to 412 Gy). This good correlation leads to consistent TL ages (Table 1) indicating the reliability of these individual results: from them, a mean age of 257 ± 20 ka was obtained after sample QS9 was discarded because of its U distribution heterogeneity (Table S2). Square K/10 yielded a rich blade-dominated Amudian industry (Barkai et al., 2009; Shimelmitz et al., 2011) and a use wear study of the industry has shown that blades were used mainly for meat cutting (Lemorini et al., 2006). These results indicate that the material unearthed from Square K/10 should be attributed to MIS 8 and possibly to the beginning of MIS7.

### 2.3.2. The Eastern Microfauna-Bearing Area

Two teeth QS0504 and QS0505 from Square M/14 at elevations 475–485 cm below datum, some 60–70 cm below the lower elevation of the dated flints from Square K/10 were dated. As for U-series dates from the sample Q6 flowstone (see Gopher et al., 2010), they exhibit an uranium content ranging between 4.2 and 7.6 ppm in enamel and ~75 ppm in dentine which is higher than the U concentration observed in the other teeth (except QS0508) (Table 2).

Even though these two samples show very different U-series and ESR data, they yielded similar ages if we take into account the associated uncertainties (Table 3). QS0505 has an Δt three times higher than that of QS0504. A significant difference in the internal dose is also observed mainly due to the great difference of uranium content in the two teeth. Similarly, the isotopic ratios are different and range between 0.7 (dentine) and 0.9 (enamel) for ^230^Th/^234^U. The calculated ages (280 ± 40–73 and 252 ± 18 ka) are similar in spite of these different data expressed by p-values between 0 (linear uptake, LU) and ~1 (early uptake, EU) for QS0505 and by a recent uranium uptake (p-values > 0) for QS0504. These results are statistically rather similar to those obtained on the 8 burnt flint samples of Square K/10, despite the difference of elevation (~60 cm), and should also be attributed to MIS 8. The Eastern Microfauna Bearing Area yielded a blade-dominated Amudian industry (Barkai et al., 2009; Shimelmitz et al., 2011) as well as a rich assemblage of micromammals and reptiles (Maul et al., 2011). Two human teeth were found in this area in Square M/13 at elevation 360–370 cm below datum (Hershkovitz et al., 2011).

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**Table 1**

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Location square</th>
<th>Z (cm)</th>
<th>U (ppm)</th>
<th>Th (ppm)</th>
<th>K (%)</th>
<th><strong>α</strong>-sensitivity (μGy/a/10^{1.1} a)</th>
<th>Dose-rate (μGy/a)</th>
<th>De (Gy)</th>
<th>Age (ka)</th>
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<td>375–380</td>
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<td>19.71</td>
<td>1021 ± 462</td>
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<td>208</td>
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<td>0.01</td>
<td>0.011</td>
<td>24.34</td>
<td>240 ± 95</td>
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<td>208</td>
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<td>K10</td>
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<td>1.14</td>
<td>0.09</td>
<td>0.026</td>
<td>14.27</td>
<td>278 ± 189</td>
<td>39</td>
<td>208</td>
</tr>
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<td>QS16</td>
<td>K10</td>
<td>385–390</td>
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<td>0.13</td>
<td>0.036</td>
<td>17.36</td>
<td>176 ± 115</td>
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<td>14.45</td>
<td>202 ± 134</td>
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<td>0.12</td>
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<td>14.21</td>
<td>100 ± 81</td>
<td>41</td>
<td>208</td>
</tr>
<tr>
<td>QS22</td>
<td>K10</td>
<td>400–405</td>
<td>0.16</td>
<td>0.03</td>
<td>0.016</td>
<td>14.60</td>
<td>42 ± 37</td>
<td>41</td>
<td>208</td>
</tr>
<tr>
<td>QS26</td>
<td>K10</td>
<td>410–415</td>
<td>0.79</td>
<td>0.02</td>
<td>0.007</td>
<td>18.04</td>
<td>240 ± 122</td>
<td>43</td>
<td>208</td>
</tr>
<tr>
<td>QS30</td>
<td>F13d</td>
<td>560–565</td>
<td>0.61</td>
<td>0.06</td>
<td>0.027</td>
<td>15.52</td>
<td>162 ± 111</td>
<td>43</td>
<td>208</td>
</tr>
</tbody>
</table>

**Fig. 5.** TL (dark dots) and ESR/U-series (open dots) ages as a function of depth (below datum).
2.3.3. The upper part of the Deep Pit Area

For burnt flints coming from the upper part of the Deep Pit Area, at a depth between 50 and 635 cm below datum, TL ages (Table 1) are highly scattered (from ~200 to ~420 ka). One can however notice that 5 of the 8 samples yielded ages between 235 ± 26 and 285 ± 32 ka, quite close to the previously presented ages. Two of the three other samples (i.e. QS31a & QS48b) have apparent relatively young ages (~200 ka) but they showed white and gray zonations which were difficult to remove during preparation. Yet, Neutron Activation Analyses (NAA) performed on samples showing this kind of zonation (Table S2, supplementary data) had indicated that these zones may have different U contents, with variations up to a factor of 3, from ~1 to ~3 ppm (Selo et al., 2009). In consequence, it has been suspected that the determined U content is not fully representative of the fraction used for TL analyses. Thus, in calculating mean ages, these two results (indicated with star in Table 1) have been discarded. Most interesting is the case of sample QS0508 which was homogenous, zonation is not suspected and its U content (0.61 ppm) remains on the average of most of other samples. Consequently, the only possibility for this sample to have an age close to the other ones (~270 ka) is to suppose that its external dose-rate was ~800 μGy/a. However, such a high external dose-rate is not realistic at Qesem Cave because of the nature of the sediment (dominated by limestone blocks and fragments in these layers). Moreover, no dosimeter recorded such a high value (see Table S1). Therefore, the possibility remains that this heated artifact is not in its original location and comes from an older occupation of the cave. We can only speculate how this flint item reached the position in which it was found: it may have been retrieved in the past from its original context or discarded before being reused or recycled, a phenomenon well represented throughout the layers of the cave (e.g. Barkai et al., 2010), or it was displaced by post depositional processes. Whatever the reason is, this sample yielded the oldest TL date; it is interesting to notice that this result is identical to the oldest date obtained for the U-series (Gopher et al., 2010) and close to the one obtained by ESR-US on QS5003 (388 ± 23 – 22 ka). In discarding unreliable samples (i.e. QS31a and QS48b) as well as QS30 because of its surprising great age, a mean TL age of 261 ± 16 ka (corresponding to MIS 8) is obtained for the Upper part of the Deep Pit Area.

ESR/U-series analyses have also been performed on 4 teeth (QS5002, QS5003, QS5007, QS5008) found in this area. These teeth have been discovered at elevations ranging between 570 and 630 cm below datum. Except for QS5008, the U content is quite similar in the teeth ranging between 0.85 and 1.18 ppm in enamel and between 22 and 42 ppm in dentine and cement tissues. QS5008 exhibits a U-content which is 10 times higher in the enamel and 2–4 times higher in the dentine than that observed in the other samples. These high concentrations lead to an annual dose value 5 times higher and an De 2 or 3 times higher compared to that of the other samples. QS5008 is the only sample presenting a 234U/238U ratio >1. The other samples exhibit a 234U loss (or 238U excess?) which can reach 10% versus the QS5008 isotopic ratio. These data suggest that the teeth underwent a complex geochemical history with probably a U-leaching followed by a very recent uptake which occurred in all the teeth including QS5008 as proven by the p-values (all greater than –1) and resulting in scattered ages ranging from ~225 to ~388 ka. Because of this complex history, these last results are considered to be only indicative of the time range.

Taken as a whole, most of the selected TL and ESR/U-series results obtained for samples from the Upper part of the Deep Pit Area,

Table 2

<table>
<thead>
<tr>
<th>Sample</th>
<th>Faunal species</th>
<th>Square</th>
<th>Elevation (cm)</th>
<th>U (ppm)</th>
<th>234U/238U</th>
<th>232Th/238U</th>
<th>228Ra/238U</th>
<th>T enamel (mm)</th>
<th>Removed enamel (mm)</th>
<th>D0(Cy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>QS0505</td>
<td>Bovid</td>
<td>M14</td>
<td>475–489</td>
<td>72.81</td>
<td>1.280 ± 0.033</td>
<td>0.900 ± 0.033</td>
<td>0.24</td>
<td>1.41</td>
<td>0.19–0.19</td>
<td>661 ± 7</td>
</tr>
<tr>
<td>QS0504</td>
<td>Bovid</td>
<td>M14</td>
<td>430–435</td>
<td>75.15</td>
<td>0.991 ± 0.023</td>
<td>0.480 ± 0.017</td>
<td>0.28</td>
<td>1.05</td>
<td>0.10–0.06</td>
<td>264 ± 6</td>
</tr>
<tr>
<td>QS0507</td>
<td>Horse</td>
<td>F14</td>
<td>570–575</td>
<td>1.19</td>
<td>0.999 ± 0.026</td>
<td>0.350 ± 0.017</td>
<td>0.60</td>
<td>1.25</td>
<td>0.07–0.13</td>
<td>197 ± 4</td>
</tr>
<tr>
<td>QS0503</td>
<td>Bovid</td>
<td>G15</td>
<td>585–590</td>
<td>24.75</td>
<td>0.930 ± 0.022</td>
<td>0.363 ± 0.014</td>
<td>0.30</td>
<td>1.11</td>
<td>0.04–0.10</td>
<td>184 ± 5</td>
</tr>
<tr>
<td>QS0508</td>
<td>Bovid</td>
<td>116</td>
<td>600–605</td>
<td>94.48</td>
<td>1.015 ± 0.016</td>
<td>0.712 ± 0.019</td>
<td>0.24</td>
<td>1.70</td>
<td>0.08–0.12</td>
<td>550 ± 9</td>
</tr>
<tr>
<td>QS0502</td>
<td>Horse</td>
<td>G15</td>
<td>625–630</td>
<td>1.11</td>
<td>0.983 ± 0.039</td>
<td>0.361 ± 0.018</td>
<td>0.78</td>
<td>1.13</td>
<td>0.09–0.11</td>
<td>147 ± 3</td>
</tr>
<tr>
<td>QS0510</td>
<td>Rhinoceros</td>
<td>D 116</td>
<td>700–705</td>
<td>11.25</td>
<td>1.148 ± 0.031</td>
<td>0.970 ± 0.037</td>
<td>0.31</td>
<td>1.20</td>
<td>0.08–0.12</td>
<td>550 ± 9</td>
</tr>
</tbody>
</table>

Table 3

<table>
<thead>
<tr>
<th>Sample</th>
<th>Square</th>
<th>Z (cm)</th>
<th>Age (ka)</th>
<th>p-Values</th>
<th>p Values</th>
<th>Age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>QS5002</td>
<td>M14b-d</td>
<td>475–480</td>
<td>235 ± 40</td>
<td>0.23</td>
<td>0.28</td>
<td>23 ± 18</td>
</tr>
<tr>
<td>QS5004</td>
<td>M14b-d</td>
<td>480–485</td>
<td>235 ± 40</td>
<td>0.23</td>
<td>0.28</td>
<td>23 ± 18</td>
</tr>
<tr>
<td>QS5007</td>
<td>F14d</td>
<td>570–575</td>
<td>235 ± 40</td>
<td>0.23</td>
<td>0.28</td>
<td>23 ± 18</td>
</tr>
<tr>
<td>QS5003</td>
<td>G15c</td>
<td>585–590</td>
<td>235 ± 40</td>
<td>0.23</td>
<td>0.28</td>
<td>23 ± 18</td>
</tr>
<tr>
<td>QS5008</td>
<td>I16</td>
<td>600–605</td>
<td>235 ± 40</td>
<td>0.23</td>
<td>0.28</td>
<td>23 ± 18</td>
</tr>
</tbody>
</table>
except two results (QS0503: 388 ± 23/–22 ka and QS30: 421 ± 38 ka), can be attributed to MIS 8, as it was the case for samples from Square K10 and the Eastern Microfauna Bearing Area. But as indicated by Fig. 5, these dates do not correlate with the depth.

2.3.4. The lower part of the Deep Pit Area

The 8 burnt flints from the lower part of the Deep Pit (depth > 660 cm below datum) come from Squares H/14 and I/16, on the east side of this pit, and from Squares F/13d and F/15d, on the west side. The environmental dose-rates recorded by the dosimeters are relatively low but they are scattered, ranging from ~312 to 444 μGy/a. This spread is observed at the scale of one square meter: for instance in Square I/16, dose-rates of 312 and 409 μGy/a were measured for depths of 660 and 690 cm, respectively. This variability could be explained as already noted by the presence of blocks of limestone which are almost free of radioactivity, as compared to fine sediments present between these blocks where the dosimeters could be inserted.

The TL ages are also relatively scattered in this sector, varying from 204 ± 19 to 308 ± 43 ka, even though 5 samples out of 8 provided ages older than 242 ka. Two of the other three samples, QS41a and QS41b, have very low radioelement contents and have therefore relatively low internal dose-rates (<170 μGy/a). Their apparent ages are consequently significantly dependent on the environmental dose-rates used for calculation. If the dosimetric environment of these pieces (defined as a sphere of ~30 cm of radius in which gamma rays are generated by the decay of radioelements) was relatively rich in limestone, the external dose-rate to which these samples have been exposed was certainly lower than the value recorded by the closest dosimeter. For instance, one can easily show that if samples QS41a and QS41b had received an external dose-rate equal to the lowest dose-rate measured in this sector, i.e. 312 μGy/a, their apparent ages would be ~275 and ~279 ka, respectively, indiscernible to the ages of the other 5 samples. Even though one has no reason to prefer one external dose-rate over another, this simple calculation indicates how sensitive the apparent ages of these two samples are. On the contrary, sample QS54 has a relatively high U content, in comparison to most of the other samples (1.46 ppm) leading to an internal dose-rate almost twice the value of the external dose-rate. The age of this sample (204 ± 19 ka) is therefore less dependent on a misfit between the estimated external dose-rate and the value it actually received during burial. Without considering the two samples QS41a and QS41b, a mean TL age of 264 ± 36 ka is obtained for the set of samples found at a depth greater than 660 cm.

Only one tooth (QS0510) coming from the lower part of the Deep Pit, found at elevations 700–705 cm below datum was available for combined ESR/U-series analyses. Among all the considered samples, this rhino tooth exhibits the lower uranium content in its tissues (0.18 ppm for enamel and 11.24 ppm for dentine). The isotopic ratios are comprised between 1.15 and 1.17 for $^{234}$U/$^{238}$U and between 0.97 and 0.77 for $^{230}$Th/$^{234}$U, for enamel and dentine tissues respectively. The annual dose is low compared to some teeth found in the pit at a little higher elevation. $P$-values range between 0 and ~1 (~0.40 and ~0.98 for enamel and dentine respectively) and yielded an age of 326 ± 20/–18 ka. It then falls in the time range covered by the TL ages (see Fig. 5) which are, as in the Upper part of the Deep Pit Area, not ordered by depth, probably indicating stratigraphic disturbances. However, considering the dating results obtained for the Upper part of the Deep Pit Area, it seems likely that the Lower part of this area can be correlated to MIS 8 and possibly 9.

3. Discussion

When looking at the results after the selection process has been made (Fig. 5), it seems that the TL and ESR/U-series ages increase with depth in the upper part of the sequence (i.e. Square K/10 and the Eastern Microfauna-Bearing Area), ranging from 217 ± 35 to 289 ± 25 ka and from 283 ± 40/–37 to 252 ± 18 ka, respectively. Two dates inversions exist however if we do not consider at first sight the associated uncertainties: one explanation could be that local disturbances affected the sediments, moving for instance a piece vertically, but it is more likely that these inversions which are of the same order than the individual age uncertainties (~25–30 ka), are related to the difficulty in estimating the dosimetric parameters. Consequently, for the sake of the discussion here, one has to consider the results obtained in an area as a whole and then define the time range covered by the dates. Following this remark, the results shown in Fig. 5 indicate that the sediments of Square K/10 and the Eastern Microfauna-Bearing Area were likely deposited during MIS 8. However, considering the uncertainties associated with the TL and ESR/U-series individual results and even though the two dated samples recovered in the Eastern Microfauna-Bearing Area came from a greater depth, it is too early to conclude that the sediments were deposited in this area a long time before those of Square K/10.

For the Upper and Lower part of the Deep Pit Area, all results except two are in the ~200–350 ka time interval, and do not seem to increase with depth. Nevertheless, looking at the results distribution and considering the conclusions drawn for Square K10 and for the Eastern Microfauna Bearing Area, this suggests that the more likely time interval for deposition of the sediments from the Upper and Lower parts of the Deep Pit is MIS 8, and possibly 9. One has also to consider the two results which gave ages greater than ~350 ka, possibly indicating an older occupation of the site during MIS 11 or 10. The location of these samples in the Upper Part of the Deep Pit Area remains questionable and the above mentioned stratigraphic problems identified in this area need to be tackled.

Considering the chronological available database for Lower Paleolithic Acheulian in the Levant that predates the AYCC and the earliest dates available for the Middle Paleolithic Mousterian, postdating the AYCC (see discussion in Gopher et al., 2010 and references therein), the results obtained in this study provide further support on the attribution of the AYCC to MIS 8 and 9, thus positioning the end of the Lower Paleolithic in this region. The fact that the distinct Amudian industry appears all along the Qesem Cave sequence and the fact that the Yabrudian industry is known in different parts of the stratigraphy from top (at elevation 130–150 cm below datum), through elevations 560–600 cm below datum, and somewhat deeper in a newly excavated area in Squares B-E/15–16 (reaching elevation 630 cm below datum) supports the suggestion that these two industries of the AYCC would be contemporaneous at Qesem Cave.

4. Conclusion

The application of the TL and ESR/U-series dating methods at Qesem Cave allowed, despite the various problems discussed above, to obtain results which are generally in agreement. These new chronological data support a time interval of hominid-bearing occupation at the cave, likely during MIS 8 (and possibly at the beginning of MIS 7) for the Upper part of the sequence (Square K/10 and Eastern Microfauna-Bearing Area) and MIS 8 and likely 9 for the Deep Pit Area. An older occupation of the cave before MIS 8–9 is also conceivable on the base of two dating results (MIS 11–10). This time interval is therefore similar with those defined on the basis of the U-series dates obtained at Qesem Cave (Gopher et al., 2010 and
references therein) suggesting no time gap between the growth of speleothems and the occupation period. Moreover, since ~2.5 m of sediments above the highest dated samples and over 2.5 m of sediments below the deepest dated samples were not dated yet, the range of dates presented in this paper correlates to the middle part of the human occupation at the cave.

In the future, the application of the ESR/U-series and TL dating methods to flints and animal teeth that have been brought into the cave at least as early as 420 ka, will probably contribute to refine the chronology of the Acheuleo-Yabrudian Cultural Complex and of the human remains found at the cave, and to precise the time duration during which the Amudian and Yabrudian industries were produced.

Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jas.2013.03.002.

References


