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# New ESR/U-series dates of the lowest Acheuleo-Yabrudian levels of Qesem cave

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## Abstract

Qesem cave is a Middle Pleistocene site located close to Tel Aviv, Israel, assigned to the Acheuleo-Yabrudian Cultural Complex (AYCC) of the Lower Palaeolithic. The site provides rich assemblages of knapped flint, animal remains and some human teeth making it of particular interest. Its location in the Levantine corridor confers a major interest to the understanding of human dynamics during the Middle Pleistocene.

A series of 6 herbivorous teeth from the base of the 11 m archaeological sequence of AYCC Qesem Cave was analysed by combined ESR/U-series method; this complements previous dating series carried out on teeth and on heated flints on higher parts of the sequence (Mercier et al., 2013; Falguères et al., 2016) The teeth were measured according a regular protocol for which each tissue was analysed by inductively coupled plasma-quadrupole mass spectrometry (ICP-Q-MS) for U-series using a combination of a Thermo iCAP-RQ mass spectrometer coupled to a Cetac Aridus III desolvator system, and ESR analyses were implemented on enamel tissue. All these data were combined in order to yield modelled ages.

The goal was to find out whether there was a time gap between the carbonates layers dated to 420 ka (Gopher et al., 2010) and archaeological layers of the AYCC found throughout the cave's sequence. The new results yield ages ranging between 220 and 430 ka confirming a great antiquity and a long duration of AYCC in the Levant.

**Key-words:** ESR/U-series, Qesem, Middle Pleistocene, AYCC

## Highlights

ESR/U-series on teeth in the lowest part of the Qesem sequence in order to have the maximum of AYCC extension.

## 1.Introduction

The site of Qesem Cave was excavated between the years 2001 and 2016 exposing a stratigraphy of some 11 meters of sediments (the bottom not reached yet). Based on lithic analysis, the finds throughout the sequence of the cave exposed to date were assigned to the Acheulo-Yabrudian Cultural Complex (AYCC). Two aspects of this entity have been exposed including a blade dominated industry known as the Amudian and a scraper (Quina and demi-Quina) dominated industry known as the Yabrudian. The two interfinger throughout the sequence the Amudian being most prominent while the Yabrudian is found in just a few parts of the cave and its sequence (Gopher et al. 2005; Barkai et al 2009, 2017; Barkai and Gopher 2013)

The cave was dated by U-series, TL and ESR and provided a range within MIS 11-7 (Barkai et al. 2003; Gopher et al. 2010; Mercier et al. 2013; Falguères et al. 2016). The latest dates (ca. 194 ka) were made on a karstic crust covering the latest archaeological remains in the sequence and we estimated, based on additional dates, that the latest use of the cave was ca. 220 ka (Gopher et al. 2010). The earliest dates made on karstic cave carbonates deposited on a rock shelf within the cave were ca. 420 ka and their relations with the archaeological layers was not fully clarified (Barkai et al. 2003; Gopher et al. 2010). The idea of sampling the lowest available part of the stratigraphic sequence of the cave was set forward in an attempt to find out when has the AYCC of Qesem Cave started and in order to find out whether there was a time gap between the carbonates dated to 420 ka and the earliest deposition of archaeological layers of the AYCC.

## **2.Context of the dated samples and methods**

The combined ESR/U-series (ESR-US) model takes into account both ESR and U-series data including radioelement contents, isotopic ratios, palaeodoses, cosmic dose and external gamma-dose rate allowing the reconstruction of the uranium uptake history in each dental tissue using a specific U-uptake parameter (Grün et al., 1988). The combined approach has been used for dating the entire Middle Pleistocene on both human and animal teeth (Grün, 2006; Falguères et al., 2010). The model cannot account for uranium loss though a new model, called the Accelerating uptake model, ESR-AU, allows an extension for samples exhibiting slight uranium leaching (Shao et al., 2012).

Six well preserved herbivorous teeth, from the deepest part of the Qesem cave stratigraphic sequence under the Shelf Area, at elevations ranging between 1035 and 1185 cm below datum (see [table S1](#), [Figure S1](#)), were analyzed by combined ESR and U-series method. The excavated area in these elevations is quite limited (7 m<sup>2</sup>); the samples for dating originate from Square D7 of the grid system excavated in the years 2014-2016 ([Figure 1](#)).

Sediments in this part of the cave remain damp throughout the year (less so in the summer) due to the near-by damp cave wall to the west and north of Square D7. The stratigraphy of this part of the cave consists of a series of alternating reddish-brown, dark reddish-brown clayish units all bearing lithic finds and faunal remains. A grey-brown unit with a concentration of stones of various sizes was exposed in the central and southern part of Square D7 and it can be clearly seen in the section. This unit was rich in lithic items and faunal remains. Field observations indicated that the sediments of this part of the cave frequently show an inclination to the west and south. This is the case for the top sediments of the area under the shelf possibly

indicating that the area was not occupied as the shelf ceiling was too low for daily human activities. However, deeper sediments under the shelf show both a) generally horizontal occupational deposits truncated by water activity near-by the western wall of the area under the shelf, and b) additional inclined sedimentary units in the lower excavated parts ([Figure 1](#)). Of note is a generally U-shaped feature running along the western wall of the area under the shelf filled up with yellowish, relatively fine grained and generally sterile sediments. This feature (never deeper than 60-70 cm) may represent a gully created by water coming in from the east-northeast in parallel to the occupation levels in this area and after occupations ceased.

The samples come from the grey-brownish (stony unit) and from the dark brown unit below it. The dosimetry samples are from the same two units.

The teeth were prepared at the Geochronology Lab of Department of “Homme et Environnement”, Muséum national d’histoire naturelle of Paris. Enamel, dentine and cement when present, were separated mechanically and their radioisotope contents measured by low background high precision gamma-ray spectrometry ([Yokoyama et NGuyen, 1980](#)).

U-series isotopic analyses of the Qesem teeth samples were performed on ICP-QMS at Paris Geochronology Lab, following the chemical protocol described in [Bischoff et al., 1988](#) and optimized in [Douville et al., 2010](#). The samples were dissolved in nitric acid together with a mixed  $^{236}\text{U}$ - $^{233}\text{U}$  and  $^{229}\text{Th}$  spike solution. U and Th were then separated by using anion exchange resins and purified with UTEVA resins. Thorium is then purified in a second anion resin column. Finally, U and Th are dried and dissolved in  $\text{HNO}_3$  2% for adjusted dilutions and isotopic analyses in ICP-QMS analyses (Thermo iCAP-RQ coupled to a CETAC III desolvator). The signal intensity of the different elements was measured under the pulse counting mode. For enamel, uranium and thorium were measured separately while for dentine and cement both elements were measured together. For ESR analyses, a part of the enamel, after cleaning its surface on both sides (inner and outer sides) to eliminate the effect of external alpha radiation, was ground, sieved and the 100-200  $\mu\text{m}$  fraction split into 14 aliquots. Thirteen of them were irradiated with a calibrated  $^{60}\text{Co}$  gamma-ray source from 63 to 16 000 Gy at the CENIEH Lab of Burgos, Spain. The irradiated aliquots were then stored in the laboratory several weeks to remove short-lived signals. The ESR measurements were performed on EMX Bruker ESR spectrometer at MNHN, Paris, using the following parameters: 1mW microwave power, 0.1mT modulation amplitude, room temperature (19°C), 10mT scan range, 4min scan time and 100kHz frequency modulation, at least 3 measurements were repeated for each dose on different days. The equivalent doses ( $D_e$ ) were determined from the asymmetric enamel T1-B2 signal at  $g = 2.0018$  ([Grün et al., 2008](#)). ESR intensities show a good reproducibility all their standard deviations being less than 4% of those means. The  $D_e$  were fitted from the experimental data using an Exponential plus Linear function (ExpLin) with Microcal OriginPro 8 software with  $1/I^2$  weighting. A single exponential saturation function (SSE) was also used for determining the  $D_e$  but it was observed that this function increases the  $D_e$  values with a lower root of mean square (RS) and a larger error range than that calculated using the ExpLin function and suggesting that the previous fitting is more appropriate than the SSE function which slightly overestimates the  $D_e$  values of 3 to 10% according the samples ([table S1](#), [Figure S2](#)).

ESR age calculations were carried out with the ESR-DATA program of Grün, (2009) using an alpha efficiency of  $0.13 \pm 0.02$  (Grün and Katzenberger-Apel, 1994) and Monte-Carlo beta attenuation factors (Brennan et al. 1997) based on the thickness of the tooth enamel and outer layers removed. The distance between the cave ceiling and the layers which have delivered the teeth is estimated to range between 12 and 15 meters and these values have been taken into account in the cosmic dose rate evaluation according to Prescott and Hutton (1994).

A 0% water content in the enamel has been used for the calculation. For dentine and cement,  $7 \pm 3$  % water content has been used. The sediment deeper than 1100 cm has 20% water content while those ranging between 1000 and 1100 cm have only 10% water content, determined by measuring the dry weight after putting the wet sediment in an oven at 35°C for one week.

The uranium content in the tissues varies significantly and the disequilibrium in the  $^{238}\text{U}$ -series was measured from the isotopic ratios and from the high resolution low background gamma-ray spectrometry for the beta dose rate calculation. The dose rate was calculated by using the dose rate conversion factors in Guérin et al. (2012). For the gamma dose contribution, in situ gamma-ray spectrometry equipped with a NaI probe was used to determine the external dose rate using the threshold method (Mercier and Falguères, 2007).

### 3.Results

U content varies from about 2 to 6 ppm in enamel, from 15 to 79 ppm in dentine, and from 31 to 73 ppm in cement (Table S1). These contents are similar to those measured in the Shelf Area at elevations around 500-600cm below datum (Falguères et al., 2016) that is, some five to six meters higher in the stratigraphic sequence of the cave.  $^{234}\text{U}/^{238}\text{U}$  ratio is less than 1 suggesting for some tissues a slight uranium leaching, especially in the QC1803 sample for which the ratio in the three tissues is lesser than 1 even with error range. The age ratio ( $^{230}\text{Th}/^{234}\text{U}$ ) is ranging between 0.338 and 0.770. The analytical uncertainty (at  $2\sigma$  level) is 0.2-2.4% for U-concentration, 0.6-2.3% for  $^{234}\text{U}/^{238}\text{U}$ , and 0.9-2.4% for  $^{230}\text{Th}/^{234}\text{U}$ . As the uranium content is relatively high in the analyzed tissues, it is important to have an idea of the eventual radon loss by crossing ICP-Q-MS and gamma ray spectrometric data (about 2/3 in the enamel and 4/5 in the dentine and in the cement).

Table 1 shows  $D_e$ , total dose rate, p-values for each tissue and ages.  $D_e$  values range between 171 and 244 Gy excepting for QC1801 which presents a higher  $D_e$  of 379 Gy. On the other hand this sample exhibits a high internal dose and p-value in the three tissues is comprised between 0 and -1 suggesting a different uranium incorporation than that observed in all the other teeth dominated by a recent uranium uptake hiding an older eventual leaching as suggested by the  $^{234}\text{U}/^{238}\text{U}$  ratios less than 1 for QC1803, QC1804 and QC1806 samples. This behavior is similar to that of the previously dated teeth from the shelf at elevation 550-600cm below datum (Falguères et al., 2016). Figure 2 shows the proportions between external and internal contributions versus the total dose. QC1801 exhibits an equivalent percentage between internal and external doses (53 and 47% respectively) while all the other samples have an internal dose rate ranging between 20 and 40% of the total dose. All these data for QC1801 advocate for a different geochemical environment than that of the other teeth.

The individual ages of the five remaining teeth are compatible within an error of 2 sigma and their weighted mean is  $338 \pm 10$  ka. The five teeth were subjected to a similar external dose rate except for QC1802 which presents a higher beta sediment dose due to the high content of uranium in the sediment, and an isochron analysis yields an age of  $351 \pm 20$  ka. Both ages are in agreement in spite of the bad  $r^2$  and suggest that the sediment including the AYCC artefacts, coeval with the herbivorous teeth, was deposited during the MIS10-9 period (375-300 ka) (Lisieki and Remo, 2005) and possibly somewhat earlier, around 400ka.

It is of note that apart from sample QC1803 [that shows an old date (422 ka) in the order of the earliest dates of the site], the other dates seem to quite well match the stratigraphy of the area.

#### **4. Discussion and conclusions**

These new chronological data from six animal teeth samples taken at the very base of the excavated stratigraphy (between 1035 and 1180 cm below datum) correspond well with the already published U-series, TL and ESR dates of the Qesem Cave sequence (Barkai et al. 2003; Gopher et al. 2010; Mercier et al., 2013; Falguères et al., 2016). The new ESR/U-series dates from the lowest part of Qesem Cave sequence completes our chronological picture of the time span of hominin bearing occupation levels at Qesem cave and confirms that hominins used the Cave during the end of MIS11 until the MIS8-7, as suggested previously. These new dates also show that no significant time gap existed between the beginning of the AYCC archaeological sequence and the deposition of the carbonates layer on top of the stone shelf dated to 420 ka (Gopher et al., 2010). The range of 420-220 ka consistently repeats itself in all the dating series we carried out in the cave since 2003.

The long stratigraphic sequence of the area under the shelf at Qesem Cave (starting at elevation ca. 500 and down to 1200 cm below datum) bears interfingering layers with Quina (and demi-Quina)-dominated Yabrudian assemblages and blade-dominated Amudian assemblages (Barkai et al., 2005). The assignment of the Qesem Cave sequence to the AYCC is thus well-based and has already been discussed in detail in the past (Barkai et al., 2009). Following the results presented in this paper, previous dating series of Qesem Cave, and available dates from other AYCC sites, we suggest to consider the range of 420-220 kya as the range for the AYCC of the Levant as a whole. This is in accordance with the available dates from AYCC sites (e.g., Tabun E, Misliya, Gamal Cave and the central Syrian sites) as summarized over a decade ago (see discussion in Gopher et al. 2010; Le Tensorer et al., 2007a, b).

However, the AYCC chronology in general (Qesem Cave included) may raise some issues worthy of discussion. These mainly concern the question of how the AYCC chronology relates to the late Lower Palaeolithic Acheulian (LPA) and how it relates to the beginning of the Early Middle Palaeolithic (EMP) Mousterian in the southern Levant (see some discussion in Gopher et al. 2010).

While we are not going to present a fully detailed comparative discussion about AYCC chronology, we may however note that:

a. Contemporaneity between the AYCC and the late LPA needs to be considered and remains an open question considering the limited geographical distribution of the AYCC (Figure 3). The case of LPA Holon

notwithstanding (see discussion in Gopher 2010; Porat et al., 1999), and regarding recently obtained (late) Acheulian dates (Revadim and Jaljuliya, as noted on in Zupancich et al. 2021, Shemer et al., in press), there seems to be some evidence for late LPA layers postdating 400 ka and some extent of overlap between the end of the LPA and the beginning of the AYCC should be accounted for. New results will corroborate or not this hypothesis. While we do not principally oppose the possibility of contemporaneity between the AYCC and the LPA, we think that such a statement is in need of stronger support.

b. Contemporaneity between the AYCC and the EMP Mousterian is less likely since in our view, a fair estimate for the beginning of the EMP would be no earlier than 220 ka and this would favour an AYCC/Mousterian chronological border around this time. A good example of a dated sequence with both AYCC and EMP Mousterian is that of Hayonim Cave. Although the AYCC (layer G) was not dated, the EMP Mousterian layers started as shown by a series of TL and ESR dates, around 220 ka (Mercier et al., 2007; Bar-Yosef, 2000). The range we suggested for the AYCC looks quite much more in place following the Hayonim Cave dates and other Mousterian dates in the region (see references in Gopher et al., 2010). The few dates of EMP Mousterian layers in the region predating 220 ka include the TL dates of Mousterian Tabun layer D of around 270 ka (Mercier et al., 1995) or ca. 250 ka (Mercier and Valladas, 2003), even though these results disagree with those obtained by ESR around 200 ka (Grün and Stringer, 2000). Another point of interest is the AYCC layer of Misliya; while the TL dating for this layer suggested a possible range between 250-170 ka, the dating of the recently published Misliya MP hominin led the excavators to suggest an MP range that rather starts around 200 ka (Hershkovitz et al., 2018).

c. We may conclude that the Qesem Cave dating results obtained by three different methods are reliable and range from 420-220 ka. That would mean that the AYCC was an entity of a long duration, comparable to that of the MP Mousterian. It shows a whole range of cultural innovations [lithic technology, the use of fire, hunting strategies, economy and more] separating it from the Acheulian though the border line should be precised by the dating (in progress) of Revadim and Jaljulia. The AYCC also differs from the MP Mousterian, clearly in lithic technology and typology. We would thus view the AYCC as a significant chapter in human cultural evolution in the Near East. No more Acheulian – not yet Mousterian would be a good descriptive summary for the AYCC although it is, in a way, a somewhat technical description, and may hint at a linearity (or continuity?) that does not necessarily represent the deep-past histories we discuss. In many ways, the AYCC seems to rather represent a „deviation“ of sorts from the „Main (Acheulian-Mousterian) Road“ that came in, and was then left out, not before introducing a significant array of innovation and change in many realms of human behavior and adaptation.

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Samples	depth (cm)	D <sub>e</sub> (Gy)	and cosmic (μGy/a)	(α+β) enamel (μGy/a)	(μGy/a)	Enamel	Dentine	Cement	(ka)
QC1801	1035	378,9 ± 6,3	811 ± 85	924 ± 179	1735 ± 207	-0,16 ± 0,17	-0,62 ± 0,13	-0,75 ± 0,10	218 +26 / -25
QC1802	1065	221,9 ± 3,7	617 ± 25	186 ± 21	803 ± 44	1,64 ± 0,20	1,31 ± 0,20	---	277 +17 / -15
QC1803	1125	243,6 ± 3,1	404 ± 21	157 ± 31	561 ± 35	6,11 ± 0,63	3,61 ± 0,42	2,90 ± 0,36	434 ± 35
QC1804	1150	170,6 ± 2,5	390 ± 21	101 ± 25	491 ± 21	1,52 ± 0,12	0,15 ± 0,02	0,11 ± 0,01	347 ± 24
QC1805	1175	244,3 ± 6,5	418 ± 19	196 ± 23	614 ± 35	3,02 ± 0,33	2,80 ± 0,36	---	399 +29 / -27
QC1806	1180	197,9 ± 3,2	450 ± 19	101 ± 10	551 ± 27	4,08 ± 0,36	2,37 ± 0,28	---	360 +21 / -19

Table 1: D<sub>e</sub> in Gy, annual dose rate in microGy/year, p-values and ESR/U-series ages in ka of the Qesem teeth. Errors are given with one sigma

## Figures caption

Figure 1: Stratigraphic sketch of the lowest levels of Qesem Cave in which the dated teeth were taken between 1200 and 1000 cm below datum (red circles). The grey brown and stony sediment containing the teeth is heterogeneous rendering the external dose measurement difficult. The black squares indicate the gamma measurements locations.

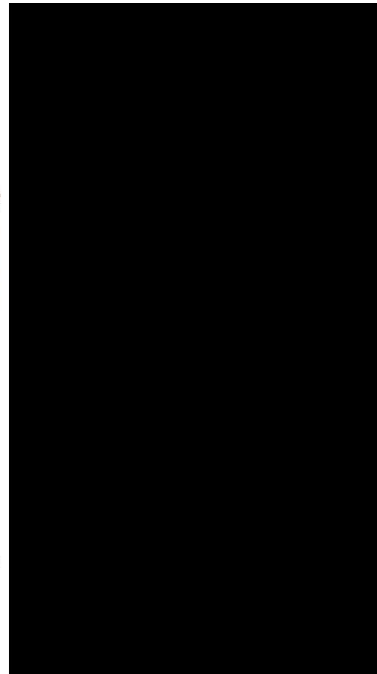
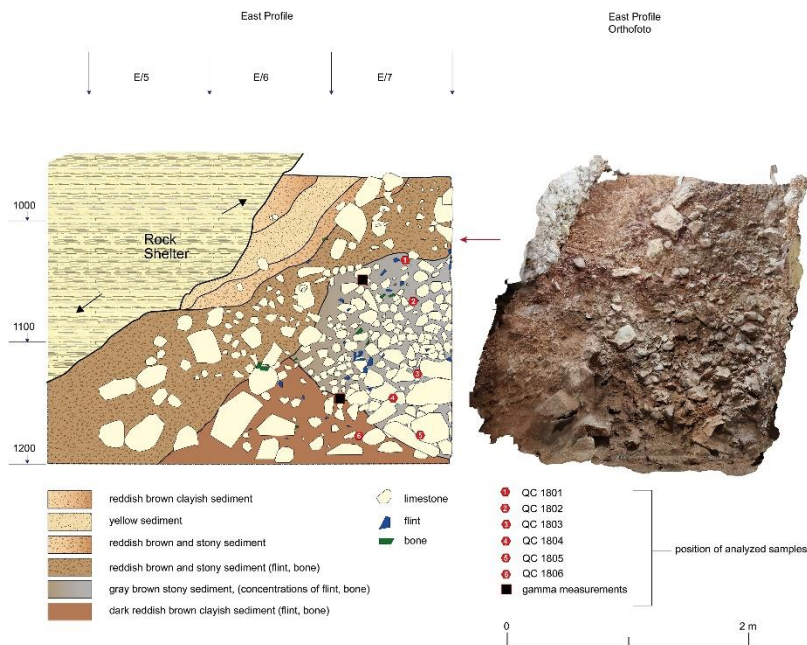


Figure 2: External and internal dose rate contribution for the analysed teeth

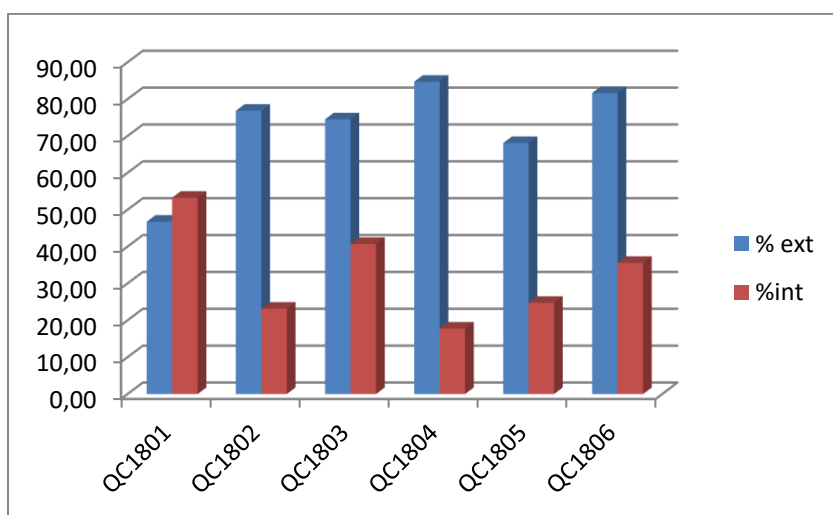


Figure 3: Succession of different cultures in Middle East showing the difficulties to separate clearly the AYCC from the LPA on one hand and from the EMP in other hand.

GBY = Gesher Benot Ya‘aqov (Rink and Schwarcz, 2005) ; Evr.Q = Evron Quarry (Porat and Ronen, 2002; Shemer et al., 2019) ; N.H. = Nahal Hesi (Zaidner et al., 2018); KMW. = Kefar Menachem West (Malinsky-Buller et al., 2016); Rev.= Revadim (Marder et al., 2007); Jal. = Jaljuliya (Shemer et al., submitted; Zupancich et al., 2021) ; Ho. = Holon (Porat et al., 1999) ; Qes. = Qesem (Gopher et al., 2010; Mercier et al., 2013; Falguères et al., 2016, this work) ; Hum = Hummal (Le Tensorer, 2007b); Hay. = Hyaonim (Mercier et al., 2007; Bar-Yosef, 2000); Tab. = Tabun (Mercier et al., 1995; Mercier and Valladas, 2003; Grün and Stringer, 2000; Schwarcz and Rink, 1998; Grün et al., 1991; McDermott et al.,1993) ; Mis. = Misliya (Herskovitz et al., 2018).

For Qesem, w.m.a. is the weighted mean age and i.a. is the isochron age calculated on the basis of 5 teeth. The brown rectangle corresponds to the calcite located at the base of the archaeological infilling.

B/M is the Brunhes/ Matuyama boundary. The vertical lines represent the age domain for each site dated by several methods.

