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RADIOCARBON, ESR AND THERMOLUMINESCENCE (TL) DATING OF ARCHEOLOGICAL MONUMENTS IN AZERBAIJAN

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This study sought to enhance the precision of C-14, ESR and TL dating of archaeological monuments in Azerbaijan. The charcoal samples was collected from various sites (Polutepe, Shomutepe, Goytepe, Pahsatepe, Boyukdash-Anazaga, Burugtepe, Alkhantepe, Gebele-Selbir, Uzun Reme) for C-14 measurements. The counting vial was transferred into the liquid scintillation analyzer Tri-Carb 3100TR and allowed to count for a period of 300 to 1000 minutes depending on the size, age, and precision requirements. As a scintillation solution, 1 ml of commercially available SIGMA-ALDRICH liquid scintillation mixture PPO/POPOP in toluene was added. In this study the Radiocarbon dating method was used to date the age of charcoal samples from the archeological monuments in Azerbaijan. Calibration results was calculated with 68% and 95% probability (0=1950). Teeth and pottery samples were collected from the following archaeological sites: Jalilabad-Polutepe, Fuzuli-Leletepe, Ismayilbeytepe, Agstafa-Hesensu, Berde-Emirli-3, Jalilabad-Pashatepe, Gobustan-Anazaga, Gobustan-Boyukdash, Gobustan-Kichikdash, Jalilabad-Khudutepe, Chukur-Gebele, Mingechevir, Jalilabad-Elikomektepe, Gebele-Galayeri, and Yardimli-Arvana. The ESR and TL methods were employed to ascertain the age of the samples obtained from the aforementioned sites.
Keywords: *dating, ESR, TL, Azerbaijan, archeology, radiocarbon*

INTRODUCTION

Radiocarbon (^{14}C) dating is a fundamental technique used to identify the age of carbon-bearing materials up to about approximately 40,000-50,000 years [1]. However, its application to oil, a substance formed from ancient organic material millions of years old, presents unique challenges and opportunities [2–10]. This paper discusses the applications of ^{14}C dating related to the analysis of samples up-taking carbon from various periods in Azerbaijan. We have collected samples from the monuments of Polutepe, Shomutepe, Goytepe, Pahsatepe, Boyukdash-Anazaga, Burugtepe, Alkhantepe, Gebele-Selbir, Uzun Reme in Azerbaijan. Thermoluminescence (TL) dating and Electron Spin Resonance (ESR) dating are both methods used to date minerals and artifacts based on their exposure to radiation. While they share some similarities, they operate on different principles and are suited to different types of materials and dating ranges. Here's a comparison of both methods. TL dating is useful for artifacts and sites where the material has been exposed to heat or sunlight, while ESR dating excels in dating fossil teeth and marine deposits over much longer time spans. Both methods (Table 1) contribute significantly to archaeology, geology, and understanding human evolution [1]. The objective of this study was to enhance the precision of ESR and TL dating of archaeological monuments in Azerbaijan [2–10]. The artefacts were gathered from a range of archaeological locations and their chronological age was established. This process provides valuable information on the sequence of events at different locations, helping to construct a broader understanding of historical or prehistoric activities across various sites.

Table 1.

Comparison of TL and ESR Dating

Aspect	Thermoluminescence (TL) Dating	Electron Spin Resonance (ESR) Dating
Principle	Measures light emitted during heating to release trapped electrons	Measures trapped electrons based on their magnetic resonance
Materials	Ceramics, heated rocks, sediments	Tooth enamel, shells, quartz, corals
Age Range	Up to ~500,000 years	100,000 to 5 million years
Clock Reset	Last heating or exposure to sunlight	Initial formation or last exposure to sunlight
Accuracy	Can be less precise than ESR for older samples	Suitable for dating much older samples, including fossils
Applications	Archaeology, ancient pottery, and sediments	Paleoanthropology, dating human fossils and ancient teeth

Table 1 shows the comparison of TL and ESR dating methods.

MATERIAL AND METHODS

The benzene synthesis line for radiocarbon dating from the sample's original state to benzene is schematically described [11–16]. As many laboratories we also use silica-alumina catalysts for the cyclization of acetylene to benzene. The catalyst can be readily reactivated by heating in air at 570^0C, which causes the oxidation of the Cr(lll) and Cr(lV) to Cr(Vl).

The assumption in carbon-14 dating is that the analyzed sample has undergone only radioactive decay within the years since it ceased interaction with the biosphere. However, the archaeological artifacts and geological specimens are usually found embedded in or contaminated by other carbon-containing materials, which affect the carbon-14 content of samples. The reliability of the dating of old charcoal samples depends on the chemical treatment of the sample to remove any external 14C while leaving reliable fractions for dating. Charcoal samples of 25 grams were treated by an acid-alkali-acid (AAA) pretreatment [17–21] method after removal of visible contaminants. The concentration of the acid (HCI) and alkali (NaOH) -0.5% .

Benzene production was carried out by the following reactions:

a) $2C + 2 Li = Li_2C_2$
b) $Li_2C_2 + H_2O = C_2H_2 + Li_2O$
c) $3C_2H_2 = C_6H_6$

In order to remove possible ammonia compounds, acetylene is passed through phosphoric acid. A chromium activated alumina-silica catalyst was used for conversion of acetylene to benzene at room temperature. Benzene was then evaporated out of the catalyst at 120°C and collected in a vacuum at liquid nitrogen temperature. The synthesized benzene [22–26] was transferred into 20 ml low-potassium glass counting vials and increased to 3 ml volume by adding petroleum-derived benzene. As a scintillation solution, 1 ml of commercially available SIGMA-ALDRICH liquid scintillation mixture PPO/POPOP in toluene was added. The counting vial was then be transferred into the liquid scintillation analyzer Tri-Carb 3100TR and allowed to count for a period of 300 to 1000 minutes depending on the size, age, and precision requirements. Radiocarbon dating by TriCarb 3100TR is a well-established absolute dating technique widely used in different areas of research for the analysis of a wide range of organic materials. Precision levels of the order of 0.2–0.3% in the measured age are nowadays achieved while several international intercomparison exercises have shown the high degree of reproducibility of the results.

The following diagram illustrates the configuration of the vacuum line employed in the synthesis of benzene. The benzene synthesis proceeds from left to right (Figure 1).

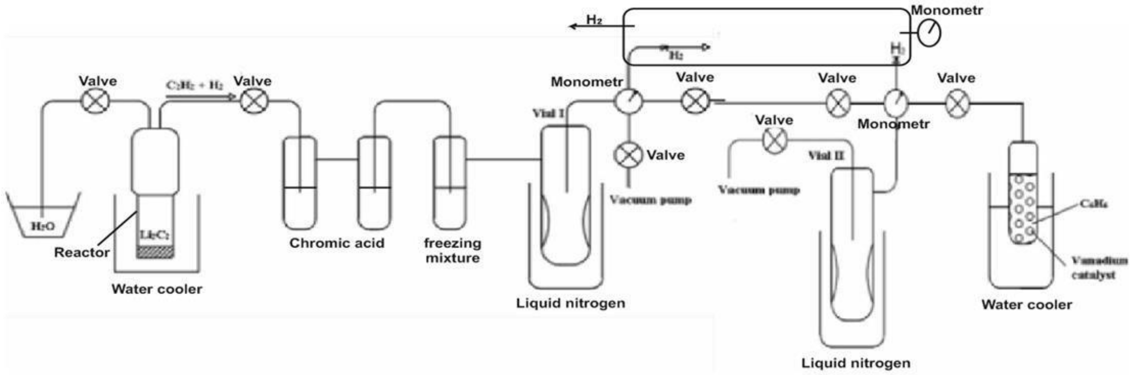

Fig. 1. Vacuum line employed in the synthesis of benzene

The charcoal was converted with 95% efficiency to carbide by the reaction (a) in the reactor. Reaction proceeded readily at 700C for 15 min. and then the temperature was raised to 900C. At a final temperature of 900C the production of carbide [27–32] was completed. After that, the hydrolysis of the lithium carbide was carried out in the reactor, which was running water-cooled, by feeding distilled water at a rate of about 100ml/min. It proceeded with a quantitative yield according to reaction (b). The excess lithium always reacts with the evolution of hydrogen gas, which is not needing in our procedure.

The gases which develop from the hydrolysis reaction are passed through the following group of traps. The first of the two traps on the right of the reactor which are consist of chromic acid are for keeping the excess gases. In the third trap, which is cooled with a freezing mixture consisting of acetone and dry ice, gases are almost completely dried. Passing through these following three traps, the acetylene is perfectly dried and can be ready for condens in the following ―cold finger‖, which is liquid nitrogen-cooled. At that point, the hydrogen gas,which is cannot freeze at the temperature of ―cold finger‖, is pumped with vacuum pump [33].

An alternative approach to the dating of ancient artefacts is electron spin resonance (ESR) analysis, which permits the imposition of a longer time limit than is possible with traditional radiocarbon dating. The capacity to identify paramagnetic centres in materials as ancient as $1x10^6$ years, due to their extended mean life (approximately $1x10^8$ years), offers significant potential for addressing a number of issues in geology, archaeology, and paleoanthropology [1]. Moreover, the ESR method can be employed as a supplementary technique for cross-referencing and/or verifying the results of the radiocarbon dating method. The foundation of ESR dating is the realization that certain materials, particularly dental enamel with its extended lifespan, generate paramagnetic centers when subjected to natural ionizing radiation. The sample preparation and ESR measurement procedures were conducted in accordance with established standard techniques (Figure 2). As illustrated in the accompanying illustration, the enamel was initially removed from the teeth using a dental drill and water cooling.

The 2-3 mm mean thickness enamel was then placed in a 30% NaOH solution for one day to disinfect it and separate any remaining dentine [2–10]. A dental drill was used to strip around 55±5 µm from inside and outside of the enamel surface to ensure that alpha radiation had no effect. In total 1.2 gr. enamel was collected and it was air-dried at room temperature for three days.

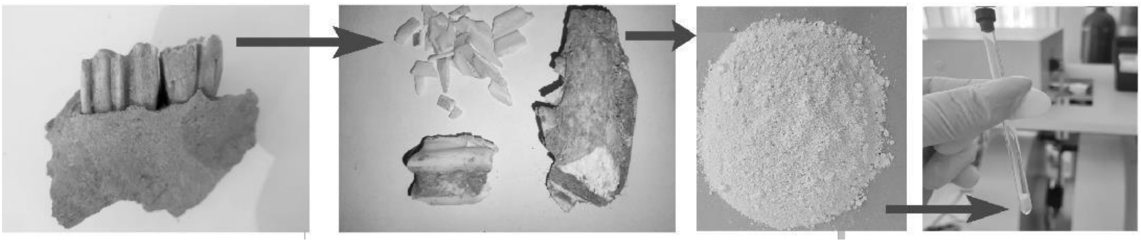

Fig. 2. Tooth sample preparation for ESR dating

Enamel powder was divided into six equal parts and each aliquot was placed inside glass tubes (Suprasil) for irradiation [11–16]. This area is designated for the preparation of samples, which are processed under controlled lighting conditions. The physical preparation area is equipped with the requisite materials for the optimal treatment of the samples prior to chemical preparation, including saws, dentist's drills, agate mortars, sieves, mills, hot plates, scales, and a magnetic separator, among other items [17–21]. The chemical preparation area has fume hoods along with all other laboratory material necessary for acid attacks or the separation of minerals through dense liquids.

Conversely, the Quartz inclusion method is utilized for the purpose of TL dating. The Harshaw TLD 3500 Manual Reader is employed for the measurement of TL characteristics of samples. The dose rate of the 60Co source was determined by the Magnettech Miniscope MS400 EPR Spectrometer using individually wrapped, barcode-labeled BioMax Alanine Dosimeter Films (developed by Eastman Kodak Company) [22–26]. The irradiated samples were weighed to 5±0.5 mg and read out after one day in an N_2 atmosphere in a Harshaw 3500 manual reader using the linear heating rate of 5ºC/s.

In order to determine the natural dose rate soil samples were collected in close proximity to the pottery sample. Uranium, Thorium and Potassium concentrations in soil were measured by gamma spectrometry Canberra GR4520 which has a low-level gamma spectrometry [27–32] system with 15 cm lead shielding and high-resolution GeHP hyper-pure germanium detector [33], having 43.5% resolution efficiency for 661.6 keV. Dose rates and age calculations were conducted using the online DRAC version 1.2.

RESULTS AND DISCUSSION

Table 2 shows the results of the C-14 measurements of the monuments in Azerbaijan. Calibration results was calculated with 68% and 95% probability (0=1950).

Radiocarbon dating was performed on charcoal samples collected from nine archaeological sites. Calibrated dates span a wide chronological range (Table 1), confirming long-term habitation across different cultural phases (Table 2):

Neolithic and Early Chalcolithic (7th–5th millennia BC): Goytepe (7746±88 BP; 6646–6476 cal BC) and Shomutepe (5801±96 BP; 4783–4542 cal BC) confirm early farming and sedentism. Polutepe samples (6220±85 BP and 6380±99 BP) and Pahsatepe (6704±88 BP) align with intensive Chalcolithic occupation.

Late Chalcolithic to Early Bronze Age: Burugtepe (6888±31 BP) and Alkhantepe (5079±29 BP) fall within this transitional phase. Upper Paleolithic: Boyukdash-Anazaga yielded the oldest radiocarbon age (12644±210 BP; 13413–12514 cal BC), indicating early human presence during the terminal Paleolithic. Iron Age and Historical Periods: Uzun Reme (3390±26 BP; 1732–1631 cal BC) corresponds to the Late Bronze–Early Iron Age. Gebele-Selbir (1105±11 BP; 899–988 cal AD) represents early medieval activity.

Dose rates and age calculation results of TL and ESR measurements are illustrated (Table 3) as follow.

Table 2.

Ages of various monuments in Azerbaijan

Sample name	14C age (BP)	± error	Cal BC(68%) 1-sigma	CalBC(95%) 2-sigma	Material
Polutepe (sample 1)	6220	85	5303 – 5052	5364-4951	charcoal
Polutepe (sample 2)	6380	99	5473 – 5224	5531-5069	charcoal
Shomutepe	5801	96	4783-4542	4901-4446	charcoal
Goytepe	7746	88	6646 – 6476	6906-6422	charcoal
Pahsatepe	6704	88	5713-5556	5749-5477	charcoal
Boyukdash-Ana-zaga	12644	210	13413-12514	13681-12210	charcoal
Burugtepe	6888	31	5796-5726	5877-5714	charcoal
Alkhantepe	5079	29	3952-3805	3959-3796	charcoal
Gebele-Selbir	1105	11	899-988	893-993	charcoal
Uzun Reme	3390	26	1732-1631	1750-1613	charcoal

Table 3 shows the results of the TL and ESR dating measurements of the monuments in Azerbaijan.

TL dating was conducted on ceramic artifacts and fired soil from 11 sites, producing ages ranging from ~15,000 BP to ~2,200 BP (Table 3):

Upper Paleolithic: Gobustan-Anazaga (15013±1324 BP) confirmed very early human activity, matching nearby radiocarbon results. Chalcolithic to Bronze Age (8th–3rd millennia BC): Sites such as Fuzuli-Leletepe (7919±455 BP), Agstafa-Hesensu (7913±324 BP), Jalilabad-Pashatepe (7826±513 BP), Ismayilbeytepe (6908±117 BP), and Jalilabad-Polutepe (6400±530 BP) showed strong consistency, suggesting widespread Chalcolithic settlement. Gobustan-Boyukdash 6835±383 BP) and Kichikdash (5655±233 BP) further support long-term cultural continuity. Late Periods: Chukur-Gebele (2230±510 BP) reflects Iron Age to historical occupation based on dating of a ceramic water pipe.

The TL dates strongly correlate with the C-14 chronology, especially at sites like Polutepe and Boyukdash, enhancing confidence in the results.

ESR dating was applied to 18 samples, including tooth enamel, deer antler, and mollusc shells. The results demonstrate a wide chronological range, offering additional insights into occupational phases: Chalcolithic and Early Bronze Age (8th–3rd millennia BC): Polutepe samples (7421±130, 7774±129, 7960±146 BP), Agstafa-Hesensu (8432±416 BP), Berde-Emirli-3 (7937±258 BP), and Khudutepe (8225±123 BP) confirm consistent early occupation across the region.

Table 3.

TL and ESR Ages of various monuments in Azerbaijan

Sample name	TL age (BP) ± error	Material
Jalilabad-Polutepe	6400±530	Ceramic
Fuzuli-Leletepe	7919 ± 455	Ceramic
Ismayilbeytepe	6908 ± 117	Ceramic
Agstafa-Hesensu	7913 ± 324	Ceramic
Berde-Emirli-3	8189 ± 850	Ceramic
Jalilabad-Pashatepe	7826 ± 513	Ceramic
Gobustan-Anazaga	15013 ± 1324	Fired soil
Gobustan-Boyukdash	6835 ± 383	Ceramic
Gobustan-Kichikdash	5655 ± 233	Ceramic
Jalilabad-Khudutepe	7116 ± 343	Ceramic
Chukur-Gebele	2230±510	Ceramic water pipe
Sample name	**ESR age (BP) ± error**	**Material**
Jalilabad-Polutepe (sample 1)	7421±130	Tooth
Jalilabad-Polutepe (sample 2)	7774±129	Tooth
Jalilabad-Polutepe (sample 3)	7960±146	Tooth
Agstafa-Hesensu	8432±416	Tooth
Berde-Emirli-3	7937±258	Tooth
Gobustan-Boyukdash	1894±97	Tooth
Gobustan-Boyukdash	14402±267	Mollusc shell
Gobustan-Kichikdash	2620±304	Tooth
Gobustan-Kichikdash	4233±364	Deer antler
Jalilabad-Khudutepe	8225±123	Tooth
Chukur-Gebele	2550 ± 200	Tooth
Mingechevir	275800 ± 10390	Tooth
Jalilabad-Elikomektepe (sample 1)	5959+122	Tooth
Jalilabad-Elikomektepe (sample 2)	5782+118	Tooth
Gebele-Galayeri	5514± 124	Tooth
Yardimli-Komani	4775±664	Tooth
Yardimli-Saribulag	5356±199	Tooth

Bronze to Iron Age: Elikomektepe (5959±122 and 5782±118 BP), Galayeri (5514±124 BP), and Komani (4775±664 BP) support continued settlement into the later Bronze and early Iron Age. Multiple Phases at Gobustan Sites: Gobustan-Boyukdash shows two very different ESR ages: a relatively recent 1894±97 BP (tooth) and an ancient 14402±267 BP (mollusc shell), indicating long-term or stratigraphically complex occupation. Gobustan-Kichikdash likewise reflects multiple use phases with a 2620±304 BP (tooth) and 4233±364 BP (deer antler). Historical and Early Medieval Periods: Chukur-Gebele (2550±200 BP) aligns with Iron Age occupation. Pleistocene Occupation: The most surprising result comes from Mingechevir, where a tooth sample yielded an ESR age of

275,800±10,390 BP. This suggests a much earlier, possibly Middle Pleistocene occupation. While requiring further validation, it opens new possibilities for early hominin presence in the region.

CONCLUSION

This study successfully applied a multi-method dating approach—Radiocarbon (C-14), Thermoluminescence (TL), and Electron Spin Resonance (ESR)—to establish a comprehensive and reliable chronological framework for key archaeological sites across Azerbaijan. The integration of these scientific methods allowed for the cross-validation of results and revealed a long sequence of human occupation, ranging from the Upper Paleolithic to the early medieval period.

Radiocarbon dating of charcoal samples confirmed continuous habitation during the Neolithic and Chalcolithic periods, particularly at sites such as Goytepe, Shomutepe, and Polutepe. TL dating of ceramics further supported these findings, while ESR dating of teeth and other materials expanded the chronological range and confirmed activity during the Bronze and Iron Ages.

Significantly, some ESR and TL results—especially from Mingechevir and Gobustan—suggest much earlier, potentially Pleistocene, human presence in the region. These findings underscore the archaeological richness of Azerbaijan and highlight the importance of continued excavation, sample contextualization, and advanced dating analysis.

Overall, the study demonstrates the effectiveness of combining radiometric and luminescence dating techniques in archaeological research. It provides a solid foundation for understanding the cultural development and settlement patterns of ancient populations in the South Caucasus and opens new possibilities for exploring deeper prehistoric layers in future investigations.

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AZƏRBAYCAN ƏRAZİSİNDƏ YERLƏŞƏN ARXEOLOJİ ABİDƏLƏRİN RADİOKARBON, EPR VƏ TL METODLARLA TARİXLƏNDİRİLMƏSİ

A.S. Əhədova

Təqdim olunan iş Azərbaycan Respublikası ərazisində yerləşən arxeoloji abidələrin C-14 metodu ilə mütləq tarixləndirilməsinə əsaslanır. C-14 ölçmələri üçün müxtəlif ərazilərdən (Polutəpə, Şomutəpə, Göytəpə, Pəhsətəpə, Böyükdaş-Anazağa, Buruqtəpə, Alxantəpə, Qəbələ Səlbir, Uzun Rəmə kömür nümunələri toplanmışdır. Hesablayıcı flakon Tri-Carb 3100TR Maye Sintilyasiya Analizatoruna yerləşdirildi və ölçüdən və dəqiqlik tələblərindən asılı olaraq 300 ilə 1000 dəqiqə arasında dəyişən müddət ərzində hesablandı. Toluolda 1 ml SIGMA-ALDRICH PPO/POPOP maye sintillyasiya qarışığı (kokteyl) sintilasiya məhlulu kimi əlavə edildi. Bu tədqiqatda Azərbaycanda arxeoloji ərazilərdən alınan kömür nümunələrinin yaşını təyin etmək üçün radiokarbon tarixləndirmə üsulundan istifadə edilmişdir. Kalibrləmə nəticələri 68% və 95% (0=1950) ehtimalı ilə hesablanmışdır. Digər tərəfdən, Azərbaycanda arxeoloji abidələrin ESR və TL tarixləndirmə üsulları ilə də mütləq yaşları tədqiq olunmuşdur. Diş və saxsı nümunələri aşağıdakı arxeoloji ərazilərdən toplanmışdır: Cəlilabad-Polutəpə, Füzuli-Lələtəpə, İsmayılbəytəpə, Ağstafa-Hesensu, Bərdə-Əmirli-3, Cəlilabad-Paşatəpə, Qobustan-Anazağa, Qobustan-Böyükdaş, Qobustan-Kiçikdaş- Xudutəpə, Çukur-Gəbələ, Mingəçevir, Cəlilabad-Elikoməktəpə, Qəbələ-Qalayeri və Yardımlı-Arvana. Yuxarıda qeyd olunan yerlərdən alınan nümunələrin yaşını təyin etmək üçün ESR və TL metodlarından istifadə edilmişdir.

Açar sözlər: *tarixləndirmə, EPR, TL, Azərbaycan, arxeologiyaб,* radiokarbon

ДАТИРОВАНИЕ АРХЕОЛОГИЧЕСКИХ ПАМЯТНИКОВ АЗЕРБАЙДЖАНА С ПОМОЩЬЮ РАДИОУГЛЕРОДНОЕ ЭСР И ТЕРМОЛЮМИНИСЕНТА

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Представленная работа была попыткой повысить точность датирования археологических памятников Азербайджанской Республики по методу С-14. Образцы древесного угля были собраны из различных мест (Полутепе, Шомутепе, Гѐйтепе, Пахсатепе, Беюкдаш-Аназага, Буругтепе, Алхантепе, Гебеле-Сельбир, Узун Реме) для измерений С-14. Счетный флакон был помещен в жидкостный сцинтилляционный анализатор Tri-Carb 3100TR и оставлен для подсчета в течение периода от 300 до 1000 минут в зависимости от размера, возраста и требований к точности. В качестве сцинтилляционного раствора был добавлен 1 мл коммерчески доступной жидкостной сцинтилляционной смеси SIGMA-ALDRICH PPO/POPOP в толуоле. В этом исследовании метод радиоуглеродного датирования был использован для датирования возраста образцов древесного угля из археологических памятников Азербайджана. Результаты калибровки были рассчитаны с вероятностью 68% и 95% (0=1950). Другой цель данного исследования было повышение точности датирования археологических памятников Азербайджана методами ESR и TL. Образцы зубов и керамики были собраны со следующих археологических памятников: Джалилабад-Полутепе, Физули-Лелетепе, Исмаилбейтепе, Агстафа-Хесенсу, Берде-Эмирли-3, Джалилабад-Пашатепе, Гобустан-Аназага, Гобустан-Бѐюкдаш, Гобустан-Кичикдаш, Джалилабад-Худутепе, Чукур-Гебеле, Мингечевир, Джалилабад-Эликомектепе, Гебеле-Галаери и Ярдымлы-Арвана. Методы ESR и TL были использованы для определения возраста образцов, полученных с вышеупомянутых памятников.

Ключевые слова: *датирование, ЭСР, ТЛ, Азербайджан, археология, радиоуглерод.*