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DATING LIME MORTAR BY OSL METHOD, WEST CHERCHELL THERMAL BATHS: A CASE STUDY

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ABSTRACT

The city of Cherchell is known as an ancient city, civilizations have succeeded in the latter from the Phoenicians to the Turkish period. The western thermal baths of this city are little known, Stéphane Gsell attributes their construction to the end of the 2nd beginning of the 3rd century [1] unlike Pensabene [2] which indicates that these dates are not based on any reliable argument, it adds to this problem dating the initial construction, the problem of remodeling during its use or the current plan seems to be symmetrical like the thermal baths of Rome or certain thermal baths in North Africa such as Carthage, Timgad [3]. So in this case an absolute dating is required, the dating of the Arian lime mortar mixed with the quartz sand used for the construction by the OSL dating method used by myself (Bouzetine and all 2021)[4] offers a great possibility of resolving these problems on all that it gives very reliable results.
To do this we took samples of lime mortars MC01 from the southern part assumed to be symmetrical to the northern part, MC02 the part assumed to be added later on the west side and mortar MC03 for the assumed initial part.
A characterization by SEM-EDX and chemical analysis by fluorescence dates from the late 2nd early 3rd century and the western part was built a little later, the shape of the baths was almost certainly symmetrical.

KEYWORDS

Cherchell, Baths, OSL Dating, Lime Mortar

CITATION

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Introduction.

The small port of Cherchell in Algeria occupies the site of Caesarea of Mauretania which was one of the most important cities in North Africa in Roman antiquity. Heiress of Iol, which was perhaps one of the capitals of the Numidian king Massyle, Micipsa, then of the Moor Bocchus, it was renamed Caesarea by king Juba II, the son of Juba I, the ally of Pompey, whom Augustus placed in 25 BC. BC at the head of the restored Mauretanian kingdom. Ptolemy succeeded his father Juba in AD 23. AD; but in 40 it was eliminated by Caligula and Caesarea naturally became the capital of the new province of Mauretania Caesariana created by the emperor Claudius. It was to keep this function of provincial capital until the end of Antiquity. If in the 16th century, the Turks came close to restoring its role as capital for a moment, throughout the Middle Ages it was no longer a town of secondary importance and Arab historians even consider it to have disappeared. Its modern renaissance dates from the settlement of the Andalusians expelled from Spain. [5]

Within these ruins is a thermal bath complex called the Western Baths or Sultana's Pleasure.

The construction of these thermal baths is usually placed at the end of the 2nd-beginning of the 3rd century: this dating, which is not improbable, is however not based on any precise argument. We will only note that Ionic capitals, which could come from a portico preceding the building and therefore belong to the original decoration of the building, can be dated to the last decades of the 2nd century [6].

In this work we will carry out a direct dating by luminiscence on the quartz contained in lime mortar used with the solid brick to build this monument.

Geographic location

Cherchell is a coastal town located approximately 100 km from the capital Algiers Figure.1. Figure 2 shows the location of the west thermal baths in relation to the plan of Ceasarea of Mauretania and a satellite photo of the site taken on May 29, 2024 Google site figure .3, figure 4 gives the ground plan of the thermal baths and the locations where the lime mortar samples MC01, MC02 and MC03 were collected. This choice of the locations of the baths is not random, as we see that the plan of these baths is a symmetrical imperial plan like that found in Rome and certain baths in North Africa, except in two parts where the samples MC01, MC02 were carried out in Figure 4. So we seek to date the monument through the MC03 samples and to check whether there were any remains or not through the dates provided by the three samples.

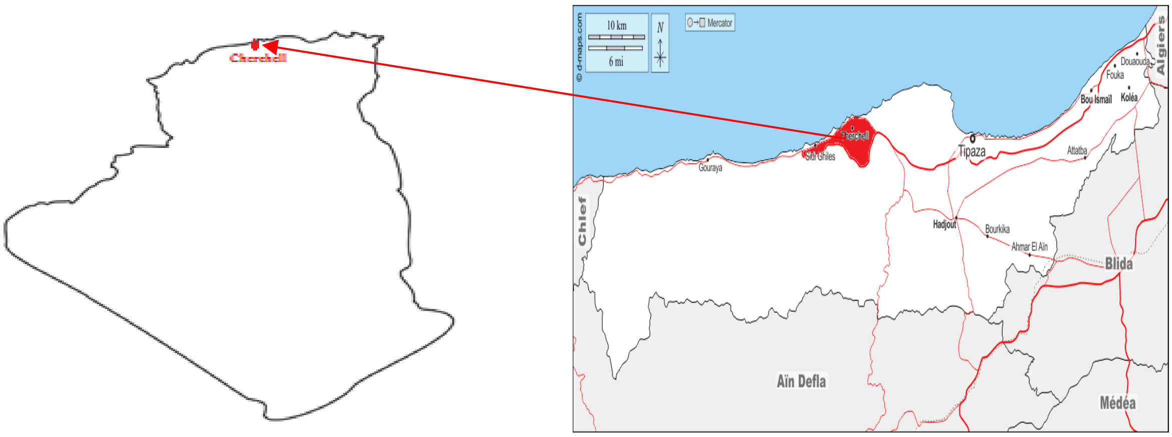

Fig. 1. Geographic location of Cherchell

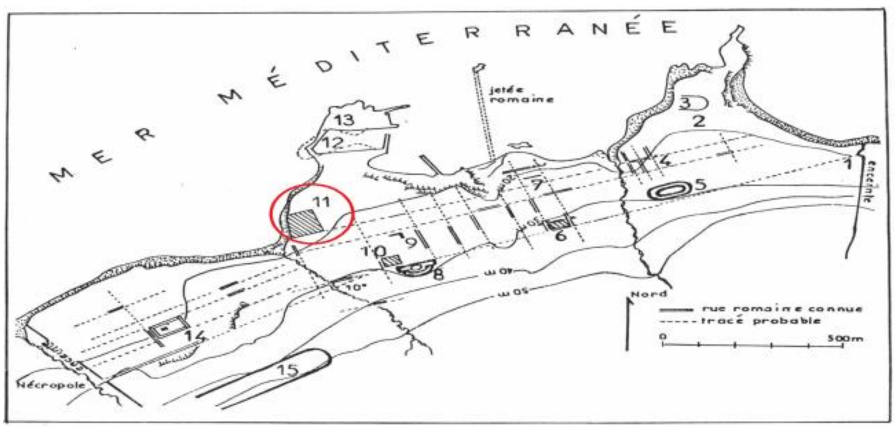

***Fig. 2.** Restored plan of Ceasarea of Mauretania **(**N° 11: West thermal baths) [7]*

Fig. 3. Satellite image of the west thermal baths[8]

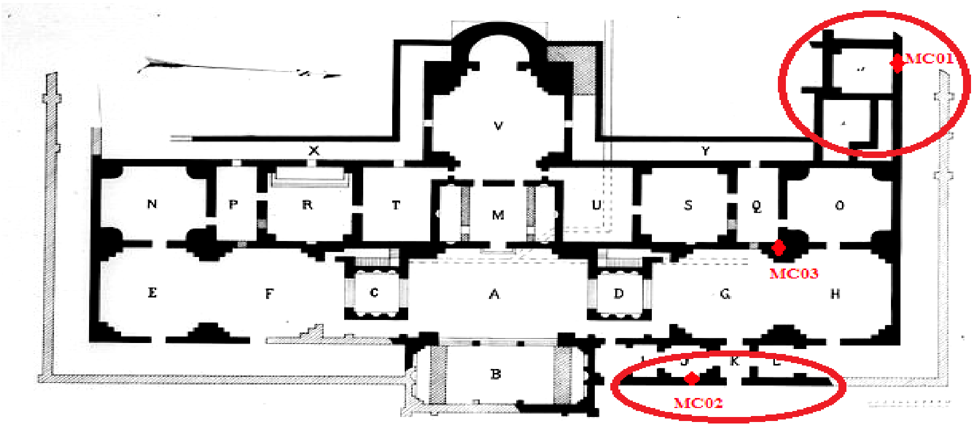

Fig. 4. Ground plan of the West thermal baths and the sampling locations [9]

MATERIEL AND METHOD

Materials
Characterization of mortars:
• **X mapping**

The three mortar samples are analyzed under SEM-EDS as shown in Figure 5, Figure 6 and Figure 7.

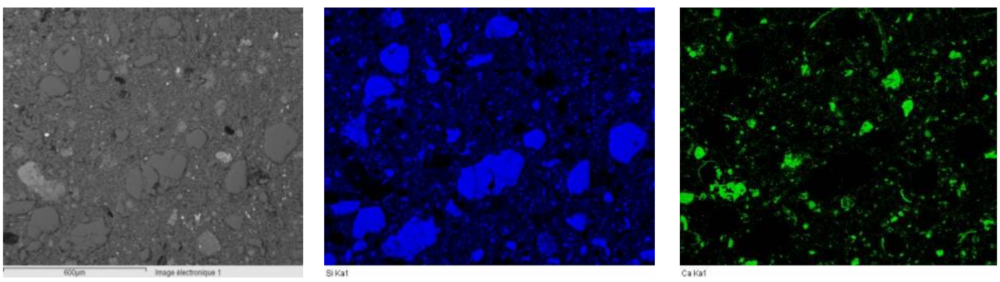

Fig. 5. Mortar MC01

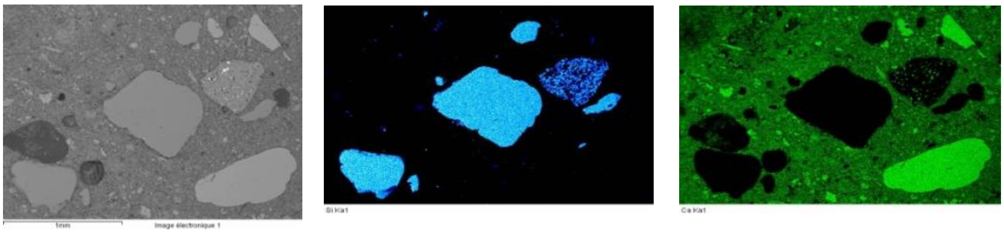

Fig. 6. *Mortar MC02*

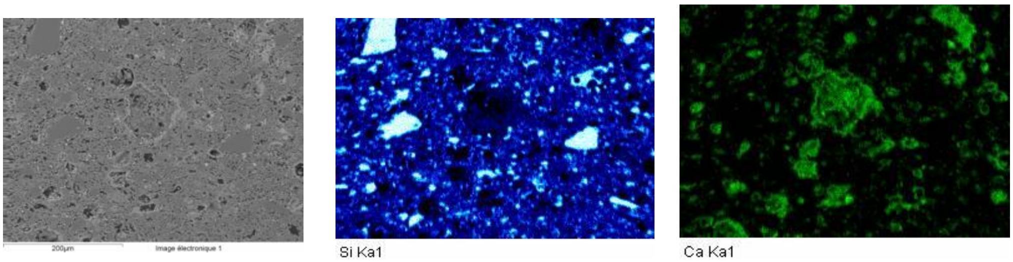

Fig. 7. *Mortar MC03*

The X mapping images show that the three mortars are composed mainly of CaCO3 (limestone matrix) and SiO2 (quartz filler) but with different contents and particle sizes.

- **Chemical analysis by X-ray fluorescence (XRF)**

The chemical analysis is carried out using the pressed powder method, the results are presented in Table.1.

Table.1. Results of chemical analysis by fluorescence of the three mortars

Oxyd (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	P_2O_5	SO_3	LOI
MC01	33,57	7,32	2,12	32,11	1,03	0,2	0,2	0,04	0,06	0,01	23,01
MC02	20,13	1,2	0,8	43,43	0,06	0,05	0,03	-	-	0,3	34,49
MC03	35,14	6,88	1,88	30,22	0,8	0,16	0,32	0,03	0,01	0,13	24,35

The MC01 and MC03 mortars are very similar in terms of chemical composition, quartz and calcite content, considerable presence of alumina, unlike the MC02 mortar which is much richer in calcite and low in silica and alumina.

Method

Principle of the method:

The optically stimulated luminescence method is based on the existence of natural radioactivity. The radioelements present in the materials studied, mainly potassium, uranium and thorium, contain naturally radioactive isotopes which will give rise to spontaneous disintegration by emitting ionizing radiation and energetic particles presenting different powers of penetration into the material.

The physical interpretation of the luminescence phenomenon is based on the "band theory" Figure 8. The interaction of ionizing radiation with matter consists of providing electrons with a sufficient quantity of energy to lead to the ionization of atoms. The energy given up to the electrons allows their transition from the valence band to the conduction band. In an ideal crystal, that is to say without defects, these electrons would lose their excitation energy quite quickly and return to the initial state, in the valence band. However, in a real crystal the electrons will be temporarily trapped by defects in the crystal. In this study, interest is focused on defects in the quartz crystal.[10]

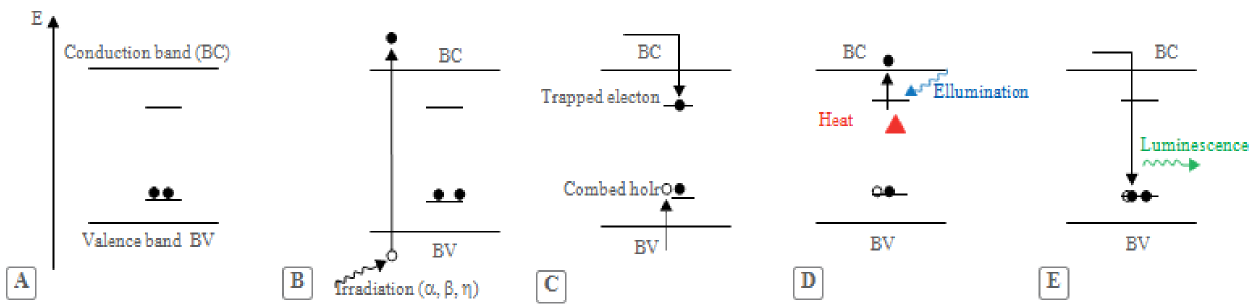

Fig. 8. *Physical interpretation of the phenomenon.*

The crystal structure of quartz, made up of silicon and oxygen, consists of a three-dimensional network of SiO_4 tetrahedral. It can present several types of defects Figure 9. The imperfections responsible for the existence of luminescence is point type defects which are divided into two groups: intrinsic defects corresponding to changes in arrangement within the framework of the crystal lattice and extrinsic defects linked to the presence of external impurities.

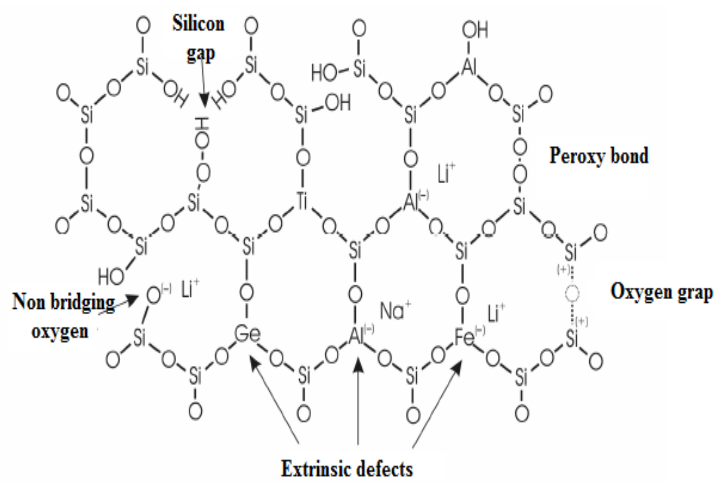

Fig. 9. *Schematic representation of the main point defects in quartz (after Götze et al., [11]*

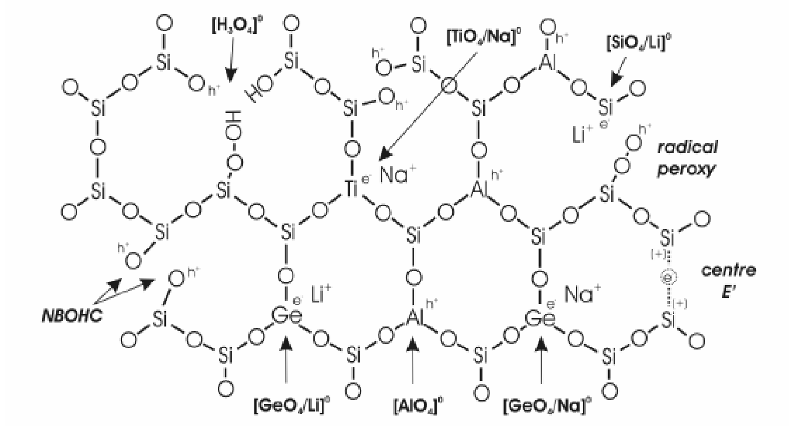

Fig. 10. *Schematic representation of the electron (e-) and hole (h+) centers created after irradiation of quartz at room temperature[12]*

Intrinsic defects in figure 10 correspond to atomic vacancies in the crystal lattice (oxygen or silicon vacancies), to the presence of an atom in an interstitial position (for example the presence of atoms which fit into the empty spaces of the crystal or the presence of an additional oxygen leading to the formation of a peroxide bond) or to a movement of an atom of the crystal backbone towards an interstitial position. This last type of defect is due to the breakage of a Si-O bond under the action of ionizing radiation or during a significant rise in temperature.

Extrinsic defects are associated with the presence of impurities in substitution or in an interstitial position.

The substitutions of atoms without modification of the crystal lattice must satisfy a condition of compensation of electric charges. During the formation of quartz, silicon (Si^{4+}) can be replaced by ions with the same formal charge such as germanium (Ge^{4+}) and titanium (Ti^{4+}) to form the defects noted respectively $[GeO_4]0$ and $[TiO_4]0$, but also by aluminum (Al^{3+}) or iron (Fe^{3+}).[13]

Luminescence dating is a paleodosimetric method. In this sense it is based on the fact that natural minerals play the role of dosimeters, recording the dose of irradiation coming from the natural radioactivity of the object itself and its environment. We thus measure by luminescence (restitution in the form of light) Figure 11 the quantity of energy accumulated since the zero moment to be dated: the archaeological dose. This makes it possible to determine the duration of exposure to natural irradiation, also knowing the dose rate absorbed annually by the mineral "dosimeter", which is assumed to be constant over time. The method of measuring the archaeological dose by luminescence is generally done according to

two processes which give their name to two twin methods:

• heating of the mineral up to temperatures around 500°C, this is thermally stimulated luminescence (or thermoluminescence, TL);

• illumination, this is then optically stimulated luminescence (OSL).

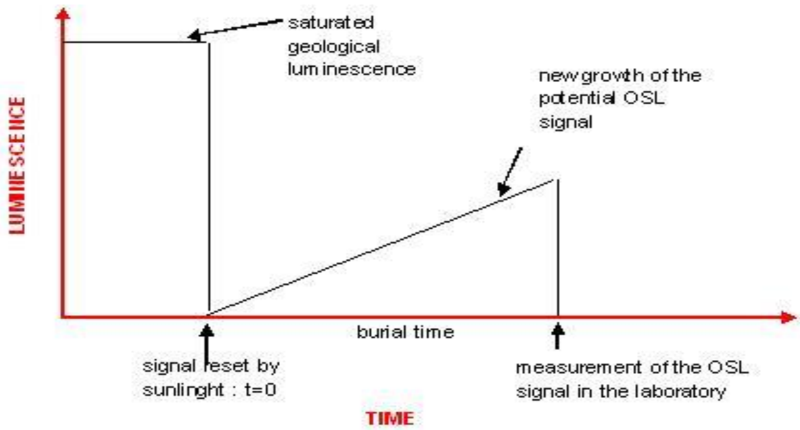

Fig. 11. *Evolution of luminescence over time (by Chantel Tribolo)*

The age measurement, which is a measurement of the duration of exposure to natural radioactivity since time zero to be dated, amounts to determining two quantities:

• the archaeological dose, or quantity of energy (reduced to the unit of mass of matter) accumulated since the zero moment to date, via a trapping of the charges by thermal stimulation (TL) or optical in the visible or near infrared (OSL). The unit of dose in the SI system is the gray, 1 Gy = 1 $J.kg^{-1}$;

• the annual irradiation dose or annual quantity of energy absorbed by the material which was used for the luminescence measurement (Gy/year).

Age is obtained by the ratio of the archaeological dose to the annual dose.

$$Age = \frac{(\text{Equivalent dose in (Gy)})}{(\text{Annual dose (Gy / an)})}$$

Extraction of quartz

We have chosen to use the technique single aliquot quartz inclusions for dating:

- Finely grind the Areirenne lime mortar.

- Attack with a dilute solution of HCl (HCl 1M) to remove the carbonates, followed by several rinses with water;
- Attack with a 30% hydrogen peroxide solution for 48 to 72 hours in order to remove organic matter; followed by several rinses with water.
- Attack with a mixture of Hexafluorosilicic acid (H_2SiF_6 31%) and nitric acid (HNO_3 68%) (mixture of concentrated solutions in a respective volume ratio 9: 1) for 48 to 72 hours in order to dissolve the feldspars and the aluminosilicates of the material

The equivalent dose (archeological dose):

For the measurement of the equivalent dose, we used the technique of small quartz inclusions (fine grain technical quartz), (Blain et al., 2007)[14].

The protocol that was used to determine the beta equivalent dose of mortar lime is based on the work of Ian Bailiff (Bailiff and Holland, 2000[15]; Bailiff, 2007[16]). It is a protocol derived from SAR (Single Aliquot Regenerative), a procedure very universally used for dating sedimentary deposits, as described by Murray and Wintle (Murray & Wintle, 2000[17]; Wintle and Murray, 2006 [18]).

Unlike the traditional SAR protocol, the protocol implemented for lime mortar takes into account the fact that the materials have been strongly heated in the past and that inevitably there is a progressive variation in the sensitivity to irradiation during measurements, linked to the progressive filling of very deep traps with electrons from "optical" or photo-sensitive traps (McKeever and Chen, 1997[19]).

Table 2 describes the protocol used.

Alpha efficiency factor

The efficiency factor alpha was not measured here but set equal to 0.03 ± 0.01, which corresponds to the usual values of this parameter for the OSL of quartz.

Results and Discussion.

Determination of equivalent doses

The same protocol as that presented in Table 2 was used, but with defined temperature preheats (200 ° C for MC01 and 220 ° C for MC02 and MC03). However, we have modified the regeneration doses so as to adapt them to the value of the equivalent dose in Table 4 which we were able to estimate more precisely with the measurements made during the preliminary study of the preheating conditions.

Table 2. Protocol for measuring the equivalent dose of fine grains extracted from lime mortar for a test sample (disc containing quartz

Step	Operation	Measure height
Natural signal measurement		
Step 0	Preheatig 10s@T_p (10 seconds at preheating temperature Tp)	
	Reading signal OSL Nat at 125°C during 40s	L_n
	Preheating 10s@T_p	
	Reading signal OSL residual at 125°C during 40s	RL_n
	Irradiation by dose test D_0 (2.98 Gy)	
	Preheating 10s@T_p (10 seconds at T_p preheating temperature)	
	Lecture signal OSL test à 125°C durant 40s	T_n
	Preheating 10s@T_p	
	Reading signal OSL residual at 125°C during 40s	RT_n
Measurements of regenerated OSL signals (steps i = 1 to 5)		
Step 1	Irradiation with a regeneration dose Di = {2.98; 5.96; 8.94; 0; 2.98, 11.92} Gy for the preheating tests Di = {1.49; 2.98; 4.47; 0; 2.98, 5.96} Gy for the measurement of equivalent doses	
	Preheating 10s@T_p (10 seconds at T_p preheating temperature)	
	Lecture signal OSL Nat à 125°C durant 40s	L_i
	Preheating 10s@T_p	
	Reading signal OSL residual à 125°C during 40s	RL_i
	Irradiation by dose test D_0	
	Preheating 10s@T_p (10 seconds at T_p preheating temperature)	T_i
	Reading signal OSL residual at 125°C during 40s	
	Preheating 10s@T_p	
	Reading signal OSL residual at 125°C during 40s	RT_i
	If i <6 Go to the next step, otherwise go to the next aliquot	

Table 3. Determination of the conditions for measuring the equivalent dose of lime mortar on the basis of OSL recycling reports

Sample	Number of discs for measuringthe equivalent dose Average	quantity of material deposited in the discs	Equivalent dose (Gy)
MC01	24	0.65mg	3.98±0.10
MC 02	31	0.32mg	3,83±0.14
MC03	28	0.63mg	4,08±0.09

Table 4. Determination of equivalent doses of lime mortar,

Sample	Preheating temperature (C °)	180°C	200°C	220°C	240°C
MC01	*Recycling ratio*	0.96 ± 0.06	0.95 ± 0.09	1.04 ± 0.11	1.24 ± 0.15
MC 02	*Recycling ratio*	1.21 ± 0.14	0.88 ± 014	1.13 ± 0.13	1.32 ± 0.10
MC03	*Recycling ratio*	0,99 ± 0.13	0.92 ± 0.1	1.02 ± 0.13	1.26 ± 0.14

Annual radiation dose

The annual dose is calculated with:

$$\dot{d} = k.\, \dot{d}_{\alpha} + \dot{d}_{\beta} + \dot{d}_{\gamma} + \dot{d}_{cosm}$$

The k factor is the efficiency factor of alpha particles compared to beta and gamma.(Aitken, 1985[20]).

Radiochemical measurements

The alpha and beta component is determined from radiochemical measurements of the samples to be dated by gamma spectrometry with low background noise (Guibert et al., 2009b [21]). Possible additional measurements by ICP MS are carried out on the luminescent grains which allow the measurement of archaeological dose, when it is estimated that these grains contain radioelements in significant quantity (De Corte et al, 2006 [22]; Vandenberghe et al, 2008[23]; Guibert et al, 2009a[24]; Urbanová et al, 2018 [25]). By working on the small inclusions of quartz as we did here three lime mortar , MC01,MC02 and MC03, it is however not necessary to measure their contents in radioelements because we suppose in this case verified the hypothesis of radiochemical continuity and alpha irradiation between the fine grains and their local environment.

In order to determine the environmental component of irradiation, taking into account the position of the samples in the architectural structure and the surrounding materials which are known to have rather low radioactivity (terracotta and limestone). The value chosen is: 0.60 ± 0.20 mGy / year reflect this level of radioactivity. It also contains a cosmic term which we estimate to be around 0.2 mGy / year.

Results of the annual dose assessments

Table 5. Humidity and radioactive elements lime mortar. Uncertainty is a standard deviation

Sample	H_2O (%)	U(^{238}U) (ppm)	U(^{226}Ra) (ppm)	U(^{210}Pb) (ppm)	Th (ppm)	K (%)
MC01	0.0	2.66 ± 0.15	2.78 ± 0.02	3.99 ± 0.33	9.32 ± 0.14	1.31 ± 0.02
MC02	0.0	1,88 ± 0.18	1.83 ± 0.01	1.96 ± 0.29	8.35 ± 0.13	1.30 ± 0.03
MC03	0.0	2.37 ± 0.21	2.38 ± 0.04	3.76 ± 0.35	9,22± 0.15	1.30 ± 0.02

The annual dose values and their components are presented in Table 6.

Table.6. Components of the annual dose and total annual dose

Sample	d_α (mGy/an)	$\dot{d}_\beta$ (mGy/an)	$\dot{d}_{env}$ (mGy/an)	Annual total dose ($\dot{d}$) (mGy/an)
MC01	0.40 ± 0.14	1.20 ± 0.05	0.60 ± 0.20	2.20 ± 0.23
MC02	0.34 ± 0.12	1.23 ± 0.04	0.60 ± 0.20	2.17 ± 0.24
MC03	0.41 ± 0.13	1.18 ± 0.04	0.60 ± 0.20	2.19 ± 0.24

Table 7 presents for each sample of lime mortars, the archaeological dose, the annual dose and the age in years compared to 2024, as well as the details of the systematic and statistical uncertainties.

Table.7. OSL dating results lime mortar of thermals baths of Caesaria.

Sample	Equivalent dose (Gy)	Annual dose (mGy/an)	Age (years)	Incertainty. (years)	Date (years)
MC01	3.98±0.10	2.20 ± 0.23	1809	81	215±81 AD
MC02	3,83±0.14	2.17 ± 0.24	1765	75	259±75AD
MC03	4,08±0.09	2.19 ± 0.24	1863	82	161±75AD

Conclusions

The results obtained on the dating of the Plais des Thermales or West Thermal Baths of Caesarea, are in agreement with the archaeological data, Sthephane Gssel estimated the construction of these thermal baths dates back to the end of the 2nd-beginning of the 3rd century date obtained on the sample of mortar MC03 which displays a date of 161±75AD in its asymmetrical form. Towards the south we and after the dating of the mortar MC01 which displays a date of 215±81 AD very close date with certain uncertainties confirms that the construction of the southern part belongs to the plan which leads us to believe that the plan was perfectly symmetrical with the presence of a construction symmetrical to the northern construction as shown in Figure 12, verification is difficult because this location is built by the current occupants of the site

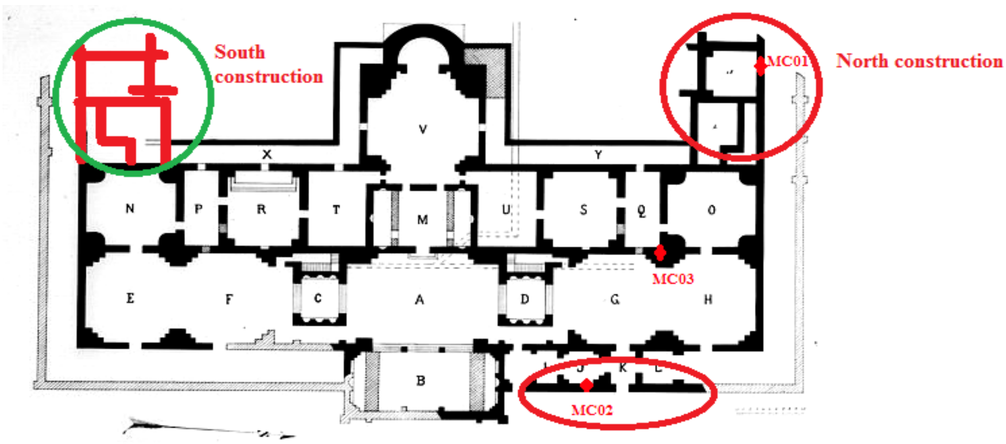

Fig. 12. Estimated plan of the palace of the thermal baths

For the cabins of the western part their dating (sample MC02) displays a date a little far 259±75AD compared to the first two we can say that they were built about a century later, which supports this hypothesis is the nature of the mortar of lime used which is different from the first two.

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