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AMS RADIOCARBON DATING OF MORTAR: THE CASE STUDY OF THE MEDIEVAL UNESCO SITE OF MODENA

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Abstract

The carbon dioxide forming the binder during the set of a lime mortar reflects the atmospheric ¹⁴C content at the time of construction of a building. For this reason, the ¹⁴C dating of mortars is used with increasing frequencies in archaeological and architectural research. Mortars, however, may also contain carbonaceous contaminants potentially affecting radiocarbon dating. The Centre for Isotopic Research on Cultural and Environmental heritage (CIRCE) of the Second University of Naples (SUN) has recently obtained some promising results in mortar radiocarbon dating thanks to the development of a procedure (i.e. Cryo2SoniC) aiming to eliminate exogenous C contamination that may occur in a mortar.

The construction history of the UNESCO World Heritage Site of Modena (Italy) is still controversial and represents a challenging case study for the application of absolute dating methodologies for different reasons. From the point of view of ¹⁴C dating, for example, given the high percentage of carbonate aggregates composing these samples, Modena mortars represent an experimental test particularly indicative of exogenous carbon sources suppression ensuring methodology accuracy.

In this paper several AMS Radiocarbon dates were carried out on lime lumps with the aim to: i) verify procedure accuracy by a comparison of the results obtainable from lime lumps dated after different treatments (i.e. bulk lime lumps vs Cryosonic purified lime lumps); ii) compare the absolute chronology of the different building phases for the medieval UNESCO site of Modena, with that assumed by historical sources. Historical temporal constraints and mortar clustering, based on petrography, have been applied to define a temporal framework of the analyzed structure. Moreover, a detailed petrographic characterization of mortars was used both as a preliminary tool for the choice of samples and to infer about the lack of accuracy (when verified) of the applied mortar ¹⁴C dating procedure.

Introduction

Dating ancient buildings and establishing the chronology of construction phases represents an important issue for archaeologists. In many cases buildings have been developed in several construction phases along many centuries and/or have been subjected to restorations and re-phasing. During the setting, mortars naturally record the moment of construction and the opportunity to apply the ^{14}C dating to this class of materials constitutes a possible breakthrough in the field of absolute chronology analyses.

Since 1960s, mortars have been exploited as a potential material for radiocarbon dating [1] and despite the fact that this method appears very simple in its principles, some measured radiocarbon ages showed evident contradictions with respect to the expected historic ages [2-16].

The main exogenous carbon sources potentially affects the final measurements are:

- primary carbonates residues (calcinations relics) originating from the incomplete burning of the limestone during the mortar production process. They lead to an ageing effect producing sensitive ^{14}C age overestimation;
- limestone fragments used as aggregates, again, potentially ageing the ^{14}C dating;
- neogenetic carbonates precipitated after the interaction with the structure of ground water or rain, containing variable aliquots of dissolved inorganic carbon (DIC), potentially inducing rejuvenation (i.e. rain) or ageing (i.e. groundwater).

The Centre for Isotopic Research on Cultural and Environmental heritage (CIRCE) of the Second University of Naples (SUN) has recently obtained some promising results in mortar radiocarbon dating thanks to the development of a procedure aiming to eliminate contamination that may occur in a mortar by means of i) cryobreaking; ii) a single/double ultrasonication step (CryoSoniC [17] Cryo2SoniC [18, 19]); iii) centrifugation. This procedure is based upon the assumption that the carbonate belonging to the binder fraction is characterized by a more easily breakable structure compared to the one of geological origin. For this reason, the binder after ultrasonic breaking forms a suspension that may be easily separated by the other mortar components and easily recovered through high-speed centrifugation. Only the suspended fraction is used for the ^{14}C dating [20].

Among different elements characterizing ancient mortars, lime lumps are frequently observed due to the technological process of production [21]. Lime lumps in *sensu stricto* are binder-related nodules often rounded and porous, distinctly appearing in the mortar matrix [22]. The evident advantage of dating lime lumps is that they are made of pure binder, being potentially free of aggregates contamination. Recently, the possibility of lime lumps dating has been studied and implemented [23, 24, 25] but the contribution of calcination relics in the lime lumps cannot be *a priori* excluded because the petrographic characterization (i.e. optical microscopy on thin sections) cannot be performed directly on the sample to be dated. Commonly, bulk lime lumps have been dated according to the so-called pure lime lumps methodology [24]. This technique is based upon the selection of pure lime lumps after a detailed petrographic characterization of the mortar,. This technique showed a general good accuracy when applied on samples of known age [23].

Recently, the CIRCE group has compared radiocarbon dates obtained on bulk lime lumps, selected according to the main pure lumps properties, with the CryoSoniC produced fraction developed from the same material [20]. In details, for 3 samples coming from an Italian case study (Ponte della Lama) it was found a sensitive (statistically significant) difference between

the two methodologies because the bulk lime lumps have provided older dates than those produced by CryoSonic fractions. This study has suggested that a purification procedure may be necessary for dating lime lumps.

In this paper a multidisciplinary approach has been applied for a correct interpretation of the radiocarbon dating results of mortars/lumps. A comparison between a pure lime lumps like methodology and a CryoSonic purification of lime lumps is presented in order to investigate the possible discrepancies yielded from the application of these different procedures. Moreover, the preliminary petrographic analyses of mortars has been used for:

- identifying the unburned limestone relicts, calcareous aggregates and new formed calcite, considered as contaminants;
- identifying the presence of lime lumps that could be extracted from the mortar excluding the aggregate dead carbon contamination;
- defining mortar groups (i.e. clustering) by means of compositional and textural parameters, limiting the number of dating analyses to be performed;

To test the Cryosonic procedure, samples were chosen in order to represent an extreme case of double contamination by older (underburned fragments) and younger (neoformed calcite) carbon sources. The goal was to assess the accuracy of the methodology with respect to different contamination. Moreover, the chosen samples should allow to reconstruct the history of the medieval UNESCO World Heritage Site of Modena (Italy).

The Modena UNESCO case study

The Cathedral and the Civic Tower “Ghirlandina” were listed as UNESCO World Heritage site since 1997. The construction of the Cathedral started in 1099 as stated in an epigraph located on the facade. The inscription indicates the date of the excavation of the foundations (23 May 1099) and the setting of the first stone (6 June 1099).

The history of the Cathedral of Modena has been and it is still the subject of an open debate by historians. According to the most accepted hypothesis, the construction of the Cathedral proceeded from the apses to the façade and it was then completed with masonry connections on the lateral sides (fig. 1) but no agreement exists on the chronology of the construction phases [26-28].

The beginning of the construction of the Ghirlandina bell tower together with the Cathedral is still a hypothesis, as historical documents are not conserved. After the construction of the first floor there was a long interruption because of subsidence problems and the availability of ornamental stones [29]. The Campionesi Masters completed the 90 m-tall bell-tower in 1319 [30] (fig. 2).

This is the first attempt to provide an absolute dating of the Cathedral and Ghirlandina mortars with the aim to contribute to the debate of the UNESCO site development.

Materials and methods

Samples selection and preparation

Seven samples from C130 foundation core mortars of the Cathedral (position in fig. 1) and one from the basement of the Ghirlandina Tower (GM86B, fig. 2) were vacuum impregnated in epoxy resin (araldite) for the preparation of thin sections. The main mortars features under the optical microscope were qualitatively described using the UNI-Normal 11176 document [31] as a reference. Lime lumps found in the mortars were separated from the bulk under a stereomicroscope up to 75 magnifications.

The mortars texture is particularly complex given the presence of different lumps including lime lumps *sensu strictu*, underburned and overburned binder related particles, often difficult to be correctly distinguished with naked eyes or with the stereomicroscope.

Moreover, the samples were taken from foundations and walls partially buried and the presence of secondary calcite could be assumed. Indeed, a phenomenon of crystallization of secondary calcite within the pores, around the grains and in the bulk was highlighted by mortar optical analyses. These mortar samples could therefore be considered as doubly contaminated by dead carbon and secondary calcite.

Four lime lumps were selected (two from C130 and two from GM86): one lump from each mortar was measured with no pretreatment (bulk) and the other two were measured after CryoSoniC pretreatment (producing the so called susp-lump fraction). Therefore, the measurement of untreated lime lumps (C130_lump, 21 mg and GM86_lump, 10 mg) and their CryoSoniC product (i.e. C130_susp_lump, 32,88 mg and GM86_susp_lump, 27,49 mg) were radiocarbon dated. In details each lime lump, for the Susp purification, was crushed and put into a 50 mL beaker and submerged into an excess (about 40 mL) of de-ionized water. The beakers containing the samples underwent an ultrasonic attack for 30', in order to produce the suspension. The suspension was quickly siphoned after the ultrasonication to avoid the sedimentation into 50 mL centrifuge vials. The vials were centrifuged at 7874g for 5' and oven dried ($T = 80^{\circ}\text{C}$) overnight. The dried suspensions (C130_susp_lump, and GM86_susp_lump), the untreated lime lump (C130_lump and GM86_lump) and carbonate standards samples (i.e. IAEA C1 and C2) [32] were digested with phosphoric acid under vacuum for 2 hr at 85°C to produce CO_2 . The CO_2 delivered during the reaction was cryogenically purified by other gasses, reduced to graphite on iron powder catalyst according to the CIRCE sealed-tube reaction protocol (the zinc process) following Marzaioli et al. [33]. The graphite was dated by means of the CIRCE AMS system [34]. The ^{14}C isotopic ratios were converted to ^{14}C ages [35] and calibrated to absolute (i.e. calendar) ages with the OxCal 2.3 [36] using the IntCal13 atmospheric calibration data set [37].

Results and discussion

Petrographic analyses results

All the mortar samples are characterized by high amount of geologic carbonate contaminants (aggregates, fig. 3a) and a high amount of binder related particles (fig. 3a, b). The most frequent are the underburned fragments of the carbonate stones used to produce the lime. Their characteristic, structural and textural features show that they are marly limestones partially modified by the heating. The samples from C130 foundation core mortars showed the presence of newly formed calcite within the pores (fig.4a), around the aggregate grains (fig. 4b), in the mortar binder (fig.5a) but also within one lime lump (fig. 5b).

Radiocarbon dating

Measured radiocarbon age on C130_lump (1570 ± 38 B.P.) without the CryoSonic preparation compared to the expected age of the mortars, first half of the 12th century A.D., gave a calendar age of 412-571 AD, an older age than expected (fig. 6a and table 1). The selected lime lump was probably characterized by a not completely burned inner part. The presence of underburned sectors within the lumps made it difficult to distinguish them from the pure lime lumps because they often show similar shapes and features in the external part.

A significant effect of rejuvenation induced by the CryoSonic procedure is evident comparing C130_susp_lump with C130_lump (Fig 6b and table 1). The younger age could be explained by two possibilities: i) the crystallization of new calcite that occurs not only within the carbonate binder, as expected, but even within the lime lumps; ii) the inner part of the masonry delayed the carbonation reaction of about 300 years. Sonninen et al. [38] described examples where the complete carbonation was achieved after centuries. This calcite was not efficiently eliminated with the CryoSonic procedure: the measured radiocarbon age on the C130susp_lump was not in agreement with the estimated age of the masonry, first half of the 12th century A.D., in fact it has been dated to the 15th century (2 sigma= 1411-1511 and 1601-1615) (table 1).

A different situation was observed on the sample, GM86, coming from the basement of the Ghirlandina Tower. ^{14}C measurement of bulk lime lump (GM86_lump) and of its CryoSonic product (GM86_susp_lump) were used as precious tool to verify archaeological expectations (table 1). No further analysis on bulk mortar, which was known to be composed by a calcareous aggregate, has been performed. Their radiocarbon age lead to i) a statistical coherence between bulk lime lump and suspended lump; ii) a sensitive match with chronological reference, which fixed 1099 AD as a lower limit for this sample chronology (Fig 7 and table 1).

Observed RC dates on the lumps (pure lime lump like) and the CryoSonic produced Susp_Lump fraction showed a different behaviour being in agreement for the GM 86 and in disagreement for the C130 sample according to Marzaioli et al. [17,18]. This observation may be interpreted as a *a-priori* difference in the selected lumps: the latter (i.e. C130) may have contained inner calcination relicts, whereas the other (i.e. GM 86) was probably contamination-free. It must be emphasized that the selection of lumps does not guarantee that the data obtained are consistent with the archaeological ages. This can only be determined through a statistic of multiple dating obtained from different lumps from the same mortar, verifying the homogeneity among results. These considerations, even if based on a limited number of observations, indicate the necessity of the application of CrySonic procedure also to lime lumps.

Conclusion

We selected a complex case study to test the ^{14}C dating of mortars, which contained two main contaminants identified by petrography: dead carbon limestone fragments (from underburned particles and aggregates) and neomorphic calcite deposited by circulating fluids. In theory, the evident advantage of using lime lumps is that they are made of pure binder and are potentially free of contamination. For this reason, the dating of lumps from complex mortars with multicontaminant fractions appears to be particularly significant to test the different preparation techniques. It is well known that the dating of buried mortars from excavations or

foundations is potentially very problematic because affected by different possibilities of contamination, especially by secondary calcite growth. Actually, the samples collected from the foundation cores of the Cathedral were the ones that showed the largest difference with the expected age.

We dated untreated lime lumps and suspended lumps produced by means of CryoSoniC methodology in order to separate the contaminants.

In one case (C130) the untreated lump showed an older age than expected probably due to dead carbon coming from internal underburned areas. The CryoSoniC preparation actually produced a rejuvenating effect, but it yielded a younger age than expected. As demonstrated by petrography, in this mortar secondary calcite formed even inside lime lumps. A slow carbonation of the mortar producing the same rejuvenation effect can not be excluded.

Conversely, two other lumps, one prepared with CryoSoniC and the other untreated, yielded the expected age with an error of few tens of years. In this case the protocol succeeded in avoiding any contamination effect.

The historical-artistic hypothesis that the tower and the Cathedral were probably founded together in 1099 has been confirmed because the two lumps yielded compatible ages. Our results demonstrate the validity of the CryoSoniC procedure for the radiocarbon dating of pure lime lumps and indicate that detailed petrographic analyses are a fundamental support for the application of any dating methodology and for the correct interpretation of results.

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Fig. 1: The Modena Cathedral construction phases proposed by Peroni [27] with the location of the core C130;

Fig. 2: Ghirlandina construction phases proposed by Labate with the position of the sample GM86.

Fig. 3: a) Photomicrograph of the Ghirlandina foundation mortar characterized by old carbonate contaminants (aggregate) and a binder related particle (dark brown). b) Photomicrograph of the Ghirlandina foundation mortar with a large lime lump *sensu strictu* in the center.

Fig. 4: Photomicrograph of mortars from the C130 core a) newly formed calcite around the pores and b) around the aggregate grains.

Fig. 5: Photomicrograph of mortars from the C130 core: a) newly formed calcite within the pores and b) inside one lime lump.

Fig. 6: calibration curve for the samples (a) C130_LUMP and (b) C130_SUSP_LUMP

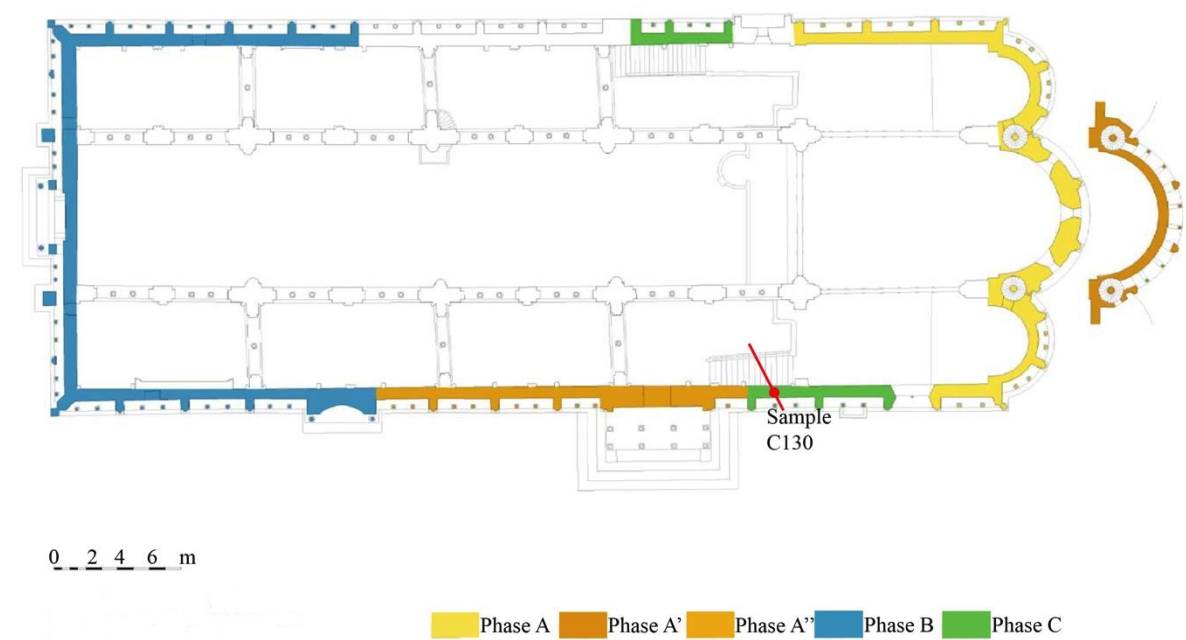
Fig. 7: calibration curve for the samples (a) GM86_LUMP and (b) GM86_SUSP_LUMP

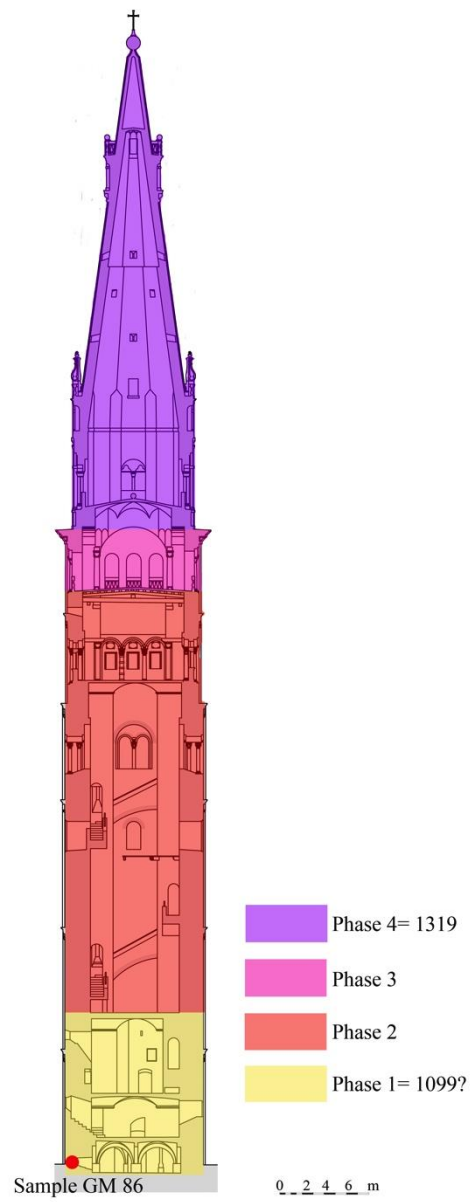
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Table 1: results of radiocarbon dating.

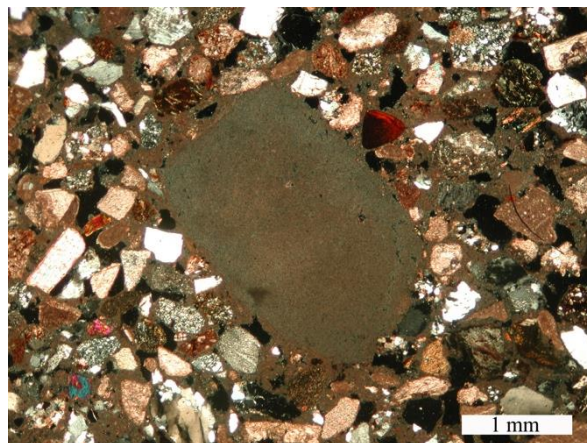
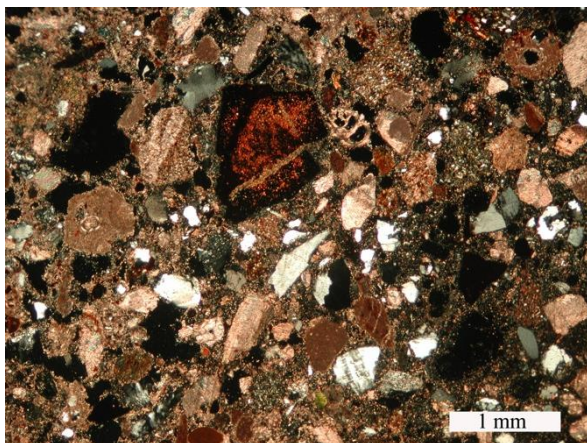
Figures

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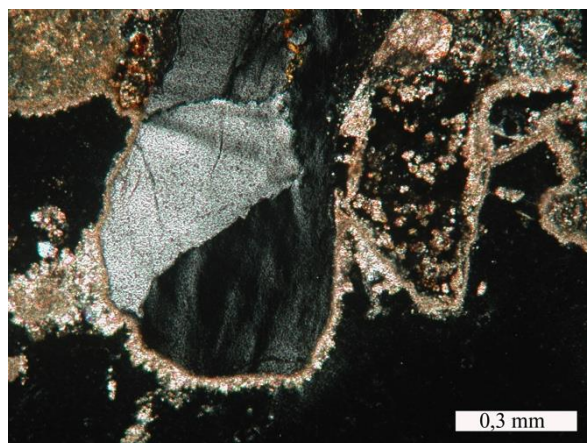
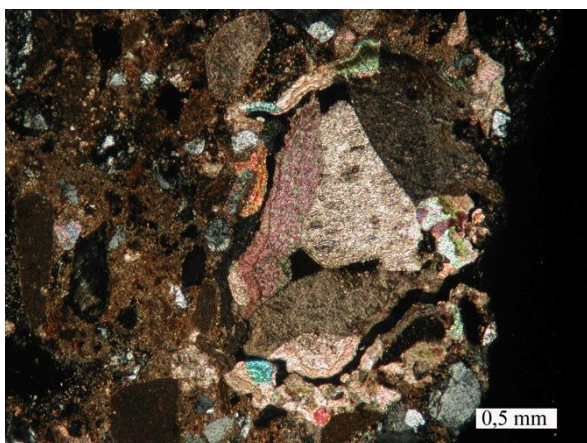




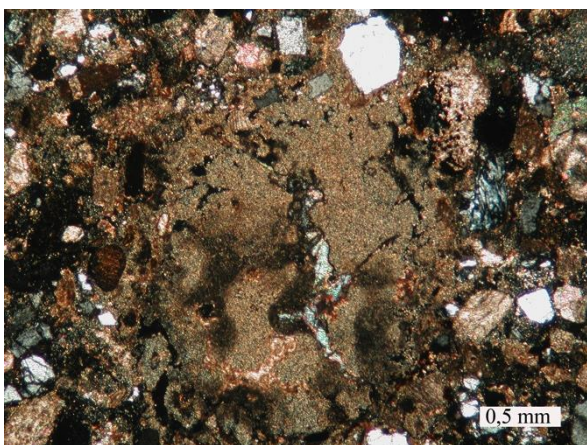
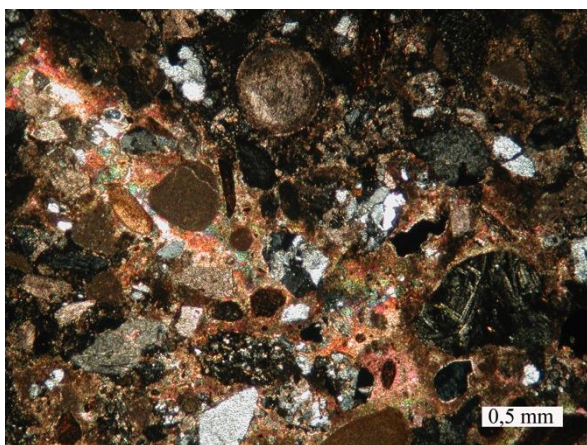
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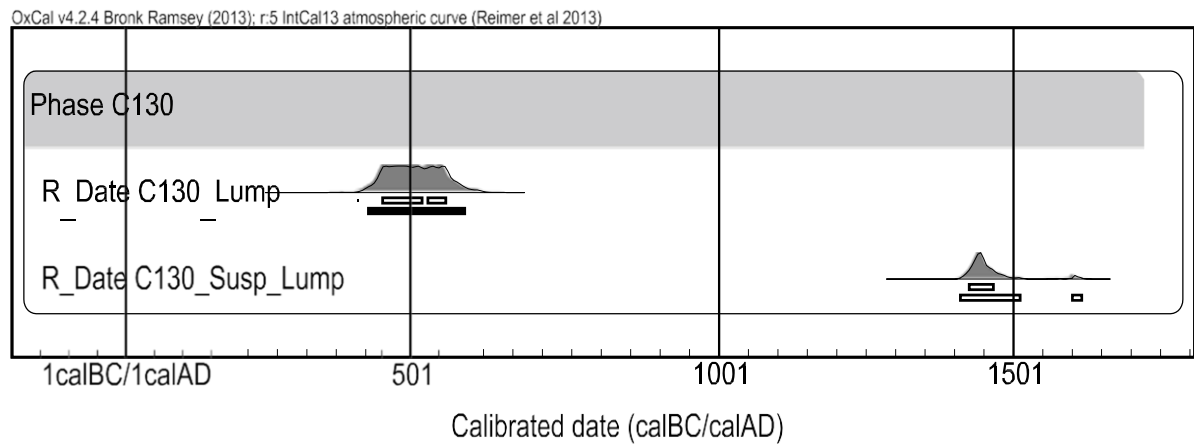
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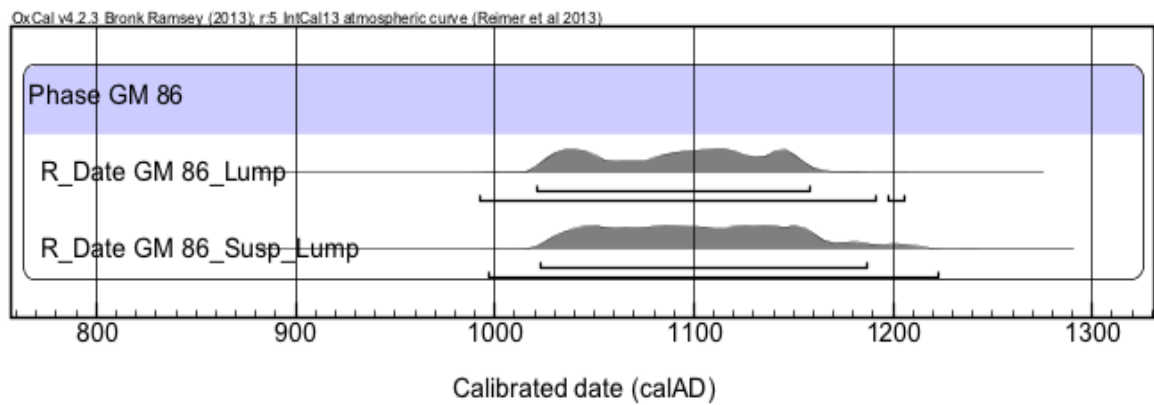


Table 1

Sample	Expected age	^{14}C age (BP)	Calibrated age 2σ	Notes
C130 lump Cathedral	1120-1130	1570 \pm 38	412-571	Contamination from secondary calcite
C130 susp_lump Cathedral	1120-1130	444 \pm 37	1411-1511 1601-1615	Contamination from secondary calcite
GM86B_lump Tower	1099-1106	953 \pm 33	1021-1158	
GM86B susp_lump Tower	1099-1106	929 \pm 38	1023-1186 1200-1205	