MORTAR DATING

International Meeting

Maison de la Recherche

25 au 27/10 2018

LabEx Sciences archéologiques de Bordeaux (LaScArBx)

programme financé par l'ANR - n° ANR-10-LABX-52

MoDIM 2018 – PROGRAM

THURSDAY, 25 OCTOBER 2018

- 08:30 – Registration (ongoing through the day)
- 09:00 – Opening

Session 1: Characterization of materials for dating. Chairperson: Gilberto Artioli.

- 09:30 – Keynote lecture by Gilberto Artioli, "Radiocarbon dating of complex mortars: can we assess the chances of successful dating?"
- 10:30 – Asscher et al., "Characterization of ancient mortars by combined XRD, NMR and FTIR techniques"
- 10:50 – Coffee break
- 11:10 – Ricci G. et al., "The castle of Cannero, Lake Maggiore, Italy: an insight into XIV-XVI century mortar techniques and their bearing on the radiocarbon dating of complex mortars"
- 11:30 – Poduska et al., "Assessing the feasibility of electrophoretic separation of $CaCO_3$ polymorphs for archaeological applications"
- 11:50 – Hayen and Boudin, "Historic mortar characterisation, useful tools for sample selection and data interpretation of mortar dating"
- 12:10 – Válek et al., "Radiocarbon dating potential of lime binders used in Prague in Medieval Period"
- 12:30 – Lunch break at the Archéopôle d'Aquitaine
- 14:00 – Michalska et al., "How various carbonate components can affect the pretreatment protocol in radiocarbon dating of mortars"

Session 2: Radiocarbon dating. Chairperson: Elisabetta Boaretto.

- 14:20 – Keynote lecture by Elisabetta Boaretto, "Radiocarbon dating of archaeological carbonate materials: difficulties, new directions and applications"
- 15:20 – Coffee break
- 15:40 – Caroselli et al., "Radiocarbon dating of dolomitic mortars from the Convent Saint John, Müstair (Switzerland): first results"
- 16:00 – Ringbom et al., "Delayed hardening in dating mortar"

Poster session (16:20-18:00)

- Barrett and Reimer, "Mortar Dating: The use of FTIR and TGMS for screening samples and understanding variation in dating results"
- Barta et al., "Roman building I. at Bratislava castle: characterization of mortar samples for ^{14}C dating"
- Javel et al., "Preliminary study of the lime mortars of the architectural complexes of Nanteuil-en-Vallée : Contributions and perspectives for the understanding of the construction sites, from Gallo-Roman Antiquity until the French Revolution"
- Kabacińska et al., "Radiation defects in lime mortars and plasters studied by EPR spectroscopy"

- Ponce-Antón et al., “Comparison of sample preparation methods for radiocarbon dating of mortars. Case study of Irulegi Castle (Navarre, Spain)”
- Sanjurjo-Sanchez et al., “OSL dating of earthen mortars from a medieval building in NW Spain: Santa Mariña de Augas Santas (Ourense)”
- Sanjurjo-Sanchez et al., “Radiocarbon and OSL dating of lime mortars of the Archaeological Ensemble of Tarraco (Les Ferreres Aqueduct), World Heritage Site”
- Tirelli et al., “Integrated dating of the Modena cathedral vaults construction and restoration (Northern Italy)”

Visit to the IRAMAT-CRP2A laboratory (18:00-19:00)

FRIDAY, 26 OCTOBER 2018

Session 2: Radiocarbon dating (continued). Chairperson: Elisabetta Boaretto.

- 08:30 – Coffee (no registration)
- 09:00 – Scott and Hajdas, “Why consider a mortar measurement inter-comparison?”
- 09:20 – Hajdas et al., “Towards routine ^{14}C dating of mortar at the AMS laboratory ETH Zurich”
- 09:40 – Toffolo et al., “Radiocarbon dating of anthropogenic carbonates using the thermal decomposition method”
- 10:00 – Coffee break

Session 3: Luminescence dating. Chairperson: Petra Urbanová.

- 10:20 – Keynote lecture by Petra Urbanová, “Single grain OSL dating of mortars in archaeology of architecture: state of the art”
- 11:20 – Panzeri et al., “Luminescence and radiocarbon dating of mortars at Milano-Bicocca laboratory”
- 11:40 – Sanjurjo et al., “OSL dating of lime mortars in a singular building of NW Spain: Santa Eulalia de Boveda (Lugo)”
- 12:00 – Guibert et al., “Modeling light exposure of grains during mortar making: consequences for Optically Stimulated Luminescence Dating”
- 12:20 – Lunch break at the Archéopôle d'Aquitaine

Session 4: Open discussion (14:00-18:00). Chairperson: Pierre Guibert.

Dinner at Café Maritime (20:00)

SATURDAY, 27 OCTOBER 2018

Excursion to Saint-Seurin and Saint-Emilion:

- 09:00 meeting at Saint-Seurin church
- 10:30 break
- 11:00 bus to Saint-Emilion
- 12:00 arrival to Saint-Emilion

- 12:30 lunch
- 14:00 guided tour
- 17:00 return to Bordeaux
- 18:00 expected arrival to Bordeaux

ABSTRACTS – ORAL PRESENTATIONS

Radiocarbon dating of complex mortars: can we assess the chances of successful dating?

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Abstract

Despite the recent substantial advances and efforts in mortar preparation, characterization, and in radiocarbon dating protocols, the routine dating of complex mortar systems seems to be elusive, at least based on the contrasting results of the MODIS round robin exercise. On one hand simple lime-based mortars with a one-step carbonation history are known to yield reliable dates of the binder emplacement. On the other hand such an ideal situation is hardly encountered in practical cases, where radiocarbon dating of the carbonated binder is variously affected by geologic carbonate, delayed hydraulic reactions, formation of long-term phases containing carbonate, and secondary alteration processes.

Through a number of case studies, it is shown that appropriate mineralogical characterization of the mortar greatly enhances the understanding of the reaction history of the material, thus allowing for the preliminary assessment of the "dateability" of the binder. However, the understanding of the reaction sequence might not be sufficient for a successful dating, because of the impossibility to physically or chemically separate the anthropogenic and geogenic carbonates or the carbon-containing phases in the binder formed at different times. The mineralogical composition and microstructural features of most common prehistoric and historic lime mortars are described, and a series of parameters are proposed that can be efficiently used to assess the chances of extraction of datable binder fractions. The measurable parameters are related to the hydraulic character of the binder, the chemical complexity of the system, and the extent of post-carbonation reactions. Several experimental techniques may be necessary to support and control the dating procedure, including luminescence spectroscopy and stable isotope mass spectrometry.

Characterization of ancient mortars by combined XRD, NMR and FTIR techniques

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Abstract

Ancient hydraulic mortars are complex system having chemical and mineralogical features inherited from (1) the original formulation, (2) the extent of the hydraulic reaction, and (3) the open or close environment in which the material evolved. Since many of the hydraulic products are amorphous, disordered, or poorly crystalline, their identification is often problematic, though it is important in order to understand the nature and development of the binder material. Further, several of the amorphous and crystalline phases produced during hydraulic reactions or subsequent long-term evolution may have a detrimental effect on the binder fraction commonly used for radiocarbon dating, the major problem commonly being the crystalline phases hosting CO_2 or CO_3-2 species in the structure. Layered double hydroxide or Afm phases are especially troublesome in this respect.

Here we show results from different pre-Roman and post-Roman mortars and plasters, reviewing the results from different characterization techniques. We discuss the different information obtained on wet- and dry-separated fractions by MAS-NMR and FTIR measurements. It is argued that the information extracted on the binder fraction with the two spectroscopic methods can be correlated and used to describe the short range atomic structure of the products and the extent of the hydraulic reactions, provided that long-range information (mineral phases) by XRD measurements and local microstructure and chemical information on the reaction interfaces by SEM/EDS measurements are used to constrain the system.

Several case studies are discussed, mainly from the first millennia BC, when different sources of hydraulic reactive materials were used, encompassing charred plant remains, crushed pottery and amorphous volcanic glass.

Radiocarbon dating of archaeological carbonate materials: difficulties, new directions and applications

Elisabetta Boaretto[1]

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Abstract

The presence of wood ash, plaster, mortar or cement in an archaeological site indicates the use of pyrotechnology. These materials are so-called “anthropogenic carbonates” ($CaCO_3$) and are the results of activities related to high temperature. When identified in archaeological contexts they could be perfect materials for radiocarbon dating and set the chronology of such activities or technologies.

Attempts to date by radiocarbon content these carbonate materials have been proven very complex due to the nature of the sample, diagenesis over time and difficulties in identifying the original fraction formed during or immediately after the temperature event. Several programs of dating pyrogenic carbonates have been pursued with different degrees of success. Yet no rules or analytical tools have been proven sufficient to qualify the pyrogenic carbonate for radiocarbon dating.

Can we reliably identify the original material or trust unexpected results for chronology?

We have developed a few methods (e.g. grinding curve with FTIR) and applied Raman spectroscopy and XRD to identify phased/minerals (e.g. aragonite) that are of great help in prescreening and dating. Some directions are not fully explored, like the use of SEM-CL for archaeological materials. As a general application of radiocarbon dating to pyrogenic carbonates is not yet available, the interest in these archaeological materials should still hold. Changes in the pyrogenic carbonate over time and recorded by the variable radiocarbon concentration might be the key to understand environmental interactions. Maybe these materials should be regarded as “living minerals”!

Radiocarbon dating of dolomitic mortars from the Convent Saint John, Müstair (Switzerland): first results

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Abstract

Absolute dating of mortars is crucial to date the construction phases of archaeological sites and to confirm or challenging existing chronologies. The method of selective dissolution, showed promising results in dating Roman and Medieval mortars (Hajdas et al. 2012) and in the frame of the mortar dating intercomparison study (Hajdas et al. 2018). The presence of hydromagnesite or other Mg-compounds in a mortar is a potential source of error for the dating process because the presence of hydromagnesite (or even brucite) indicates a slow hardening of the mortar and therefore the possibility of delayed absorption of calcium carbonate (Hayden et al. 2016, 2017). But, since the speed of acid hydrolysis of dolomite is considerably slower than calcite, the chance of contamination from dead carbon content by the aggregate may be limited.

In Müstair the situation seems perfectly suitable in order to verify if the 14C dating can be applied to mortar made by dolomitic raw material because the age of some masonries was clearly established by dendrochronology. About 5000 mortar samples belonging from the 8th to the 20th century where collected in more 30 years of archaeological excavations on site. Such quality and number of samples is very rare and provides a unique opportunity for further analytical studies. In particular, the presence in some samples of embedded charcoal fragments can provide complementary independent data necessary to support or deny the results of mortar dating. Furthermore, lime lumps and bulk mortars show different 14C contamination and therefore they resulted to be highly complementary for a reliable mortar dating (Lindroos et al. 2018). In this study a comparison of results obtained by radiocarbon dating of bulk mortars, lime lumps and charcoal fragments from the same dolomitic mortar is presented in relation with the petrographic characterization of the mineralogical phases content.

Modeling light exposure of grains during mortar making: consequences for Optically Stimulated Luminescence Dating

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Abstract

Dating building materials is one of the essential means for a better understanding of the construction history. Previous researches showed that the practice of re-using bricks originating from older structures was frequent. That is the reason why we have been focusing on mortar dating for the last 7 years. The basic premise of mortar dating by OSL is that quartz in the sand used for making mortar is optically zeroed during the preparation process (optical bleaching). The moment to be dated is the last exposure of sand grains to light, before being embedded within the masonry and hidden from light. But one of the problems encountered is the frequent partial and heterogeneous bleaching of grains that led us to use systematically the single grain technique (SG-OSL) [1].

A new way of statistical treatment (the so-called EED model, as exponential exposure distribution [2]) will be presented and discussed, as the result of the latest development of the relevant theoretical approach. Finally, a fair agreement between OSL and reference ages for a series of samples from a variety of ages and situations was obtained even in case of poorly bleached material. We will also present some cases for which some difficulties when dating them by SG-OSL still remain (low sensitivity mortar samples, samples resulting from a mixture of sediment and calcareous stones...). Anyway, actual situation of OSL dating methodology justifies the systematic use of SG-OSL in dating masonries.

[1] Urbanová, Guibert. A methodological study on single grain OSL dating of mortars: comparison of five reference archaeological sites. Geochronometria, 44, 77-97, 2017.

[2] Guibert, Christophe, Urbanová, Blain, Guérin. Modeling incomplete and heterogeneous bleaching of mobile grains partially exposed to the light: towards a new tool for single grain OSL dating of poorly bleached mortars, Radiation Measurements, 107, 48-57, 2017.

Towards routine ^{14}C dating of mortar at the AMS laboratory ETH Zurich

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Abstract

An interest in direct dating of mortar is growing, as are the chances for cross check of the used preparation methods. Complexity of the mortar requires a holistic approach such as petrographic studies. However, at the moment the method developed and applied in our laboratory is solely based on accuracy of ^{14}C analysis i.e., their agreement with expected/known age of the monuments. The fact that mortars, stucco and fresco i.e., anthropogenic carbonates have different physical and chemical properties is explored in our method. In a process of sieving we separate fine fraction of carbonates (45-63 µm). This fraction is then trapped for AMS analysis after being dissolved in concentrated phosphoric acid. The first 6 sec (2 x 3 sec dissolution interval) are considered to originate from the anthropogenic carbonates. Two more fractions (each 3 sec) are collected to observe the change in ^{14}C ages. In most of the cases the 3rd and 4th fractions are significantly older indicating the presence of old carbonates. We will present and discuss the results obtained on samples collected from sampling sites with known age as well as our results for MODIS inter-comparison project [1].

[1] Hajdas I, Lindroos A, Heinemeier J, Ringbom Å, Marzaioli F, Terrasi F, Passariello I, Capano M, Artioli G, Addis A, Secco M, Michalska D, Czernik J, Goslar T, Hayen R, Van Strydonck M, Fontaine L, Boudin M, Maspero F, Panzeri L, Galli A, Urbanova P, and Guibert P. 2017. Preparation and Dating of Mortar Samples-Mortar Dating Inter-Comparison Study (Modis). *Radiocarbon* 59: 1845-1858.

Historic mortar characterisation, useful tools for sample selection and data interpretation of mortar dating

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Abstract

The binder of historic mortars is traditionally produced by burning lime stone in a kiln. Depending on the source of the raw materials, the type of fuel used and the conditions reached during calcination and slaking, a lime binder with varying degrees of hydraulic properties and lime hydrate content (calcium or magnesium hydrate) is obtained. Whereas the hydraulic phases will, in the presence of moisture, react in a short notice throughout the entire thickness of the constructional element, the absorption of atmospheric carbon dioxide by the lime hydrates slowly proceeds from the external surfaces inward, hence, delaying the moment of hardening by carbonation. Furthermore, the carbonation process is influenced by moisture, possibly leading to binder leaching or secondary deposition of calcium carbonate. From the aggregate, limestone or shell fragments of varying sources (of geogenic origin) and in varying amounts could have end up in the mortar sample, while certain aggregate types, e.g. volcanic sand or finely crushed ceramics, may interact chemically with the lime hydrate to form new minerals which enhance the hydraulic properties of the mortar. Such an aggregate-lime interaction, known as the pozzolanic reaction, proceeds slowly and may involve secondary carbonate formation. In addition, the lime stone from the binder production may have been incompletely burnt, leaving trace remnants of lime stone behind in the binding mass of the historic mortar. Historic mortars are a complex puzzle depending on their source materials and the conditions present over their entire lifespan. An overview is presented of the various processes involved to evaluate the potential risks involved for the mortar dating process and to support the radiocarbon data interpretation. Sample screening based on mortar characterisation is emphasized prior to the mortar dating process.

How various carbonate components can affect the pretreatment protocol in radiocarbon dating of mortars

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Abstract

Different types of mortars obtained in the limestone and dolomite burning process were successfully dated with the use of different preparation [1, 2, 3]. However, depending on detailed composition, structure, porosity, specific gravity, those variable carbonates could affect the dating process with varying intensity [4].

In order to investigate the behaviour of different carbonates after applying the same mechanical and chemical preparation as used for archaeological mortars during ^{14}C pretreatment procedure, tests of leaching reaction have been performed on the group of natural carbonaceous rocks.

Additionally, also different shells were used in experimental research, to observe their decomposition rate in orthophosphoric acid, and their ability to penetrate the suspension.

This research shows how different the behavior of various carbonates could be and why in some cases the dating of suspension, in comparison to grain fractions, gives the results closer to the real one, while in other cases the suspension is much more exaggerated than the samples prepared otherwise.

[1] Heinemeier J, Ringbom Å, Lindroos A, Sveinbjörnsdóttir ÁE 2010. Successful AMS 14C dating of non-hydraulic lime mortars from the medieval churches of the Åland Islands, Finland. Radiocarbon 52(1):171–204.

[2] Lindroos A, Orsel E, Heinemeier J, Lill J-O, Gunnelius K. 2014. 14C dating of Dutch mortars made from burned shell. Radiocarbon 56(3):959–68.

[3] Ringbom Å, Lindroos A, Heinemeier J, Sonck-Koota P. 2014. 19 years of mortar dating: learning from experience. Radiocarbon 56(2):619–35.

[4] Hajdas I, Lindroos A, Heinemeier J, Ringbom Å, Marzaioli F, Terrasi F, Passariello I, Capano M, Artioli G, Addis A, Secco M, Michalska D, Czernik J, Goslar T, Hayen R, Van Strydonck M, Fontaine L, Boudin M, Maspero F, Panzeri L, Galli A, Urbanova P, Guibert P. 2017. Preparation and dating of mortar samples-Mortar Dating Inter-comparison Study (MODIS). Radiocarbon 59(6):1845-1858.

Luminescence and radiocarbon dating of mortars at Milano-Bicocca Laboratory

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Abstract

Radiocarbon and luminescence (both Thermoluminescence, TL, and Optically Stimulated Luminescence, OSL) are the main dating techniques in construction dating.

TL is a well-established method for absolute dating of masonry. Nevertheless, the frequent practice of reuse or the use of unfired natural materials, such as mud bricks, adobe or stones, makes TL dating almost useless in some applications. In recent years OSL dosimetry of quartz extracted from mortar sand aggregates has been developed after the confirmation that quartz grains are generally bleached during mortar manufacturing. Although some attempts were made to find reliable OSL dating protocols, OSL mortar dating is not a routine method so far. The main issue is that the quartz grains contained in the mortar may be only partially or non-homogeneously bleached leading to an overestimation of the sample age. The development of the Single Grain technique allowed a better identification of the well bleached quartz grains, which represents a fundamental step for reliable dating. However, the identification of the bleached grains is not always successful, indicating that further investigations are needed to develop suitable dating protocol. For what concerns the radiocarbon technique a crucial aspect is the correct identification and selection of the anthropogenic calcite; the lack of a proper method of discrimination leads to a severe age overestimation.

In light of these considerations, a discussion of the dating results we obtained applying both luminescence and radiocarbon dating will be shown, evidencing the critical aspects of material selection and possible parameters that influence the reliability of the dating analysis. A number of case studies on samples from northern Italy applying both Multigrain and Single-Grain OSL techniques will be presented as well as the results of a few radiocarbon results obtained using the acid digestion technique.

Assessing the feasibility of electrophoretic separation of $CaCO_3$ polymorphs for archaeological applications

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Abstract

We demonstrate a proof-of-principle method to separate particles of two $CaCO_3$ polymorphs, calcite and aragonite, based on surface charge density differences that affect electrophoretic mobility values. When particles are dispersed in aqueous media, their surfaces become charged, and the net surface charge density determines the extent to which the particles can be moved in the presence of electric fields. When calcium carbonate particles are dispersed in water, the particles acquire a negative surface charge density due to the release of Ca ions and/or adsorption of anions. However, this surface charge is typically so small that the particles do not have enough electrostatic repulsion to prevent agglomeration. We show that phosphate additives, which are commonly used to reduce particle aggregation, have a serendipitous added benefit of stabilizing $CaCO_3$ against dissolution. We also show that calcite and aragonite powders show significant differences in their electrophoretic mobility distributions in polyphosphate-containing suspensions. However, the mobility differences among archaeologically relevant samples, such as lime plasters and chalk, are not consistently different enough to make this a reliable separation strategy. In other words, electrophoretic separation of calcite and aragonite appears to be possible in theory, but it is not trivial to put into practice. Nevertheless, this study is important because it takes a new and fundamentally different approach to non-destructive separation of archaeological materials, using surface chemistry differences.

The castle of Cannero, Lake Maggiore, Italy: an insight into XIV-XVI century mortar techniques and their bearing on the radiocarbon dating of complex mortars

Giulia Ricci[1], Michele Secco[1], Gilberto Artioli[1], Fabio Marzaioli[2], Filippo Terrasi[2], Isabella Passariello[2], Federica Badino[3], Simone Simonetti[3], Paolo Lampugnani[4], and Francesca Garanzini[5]

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Abstract

The chemical and mineralogical complexity of ancient mortar systems often poses challenges both in the characterization and the separation of the binder fraction. This may have important effects on the results of subsequent radiocarbon dating, because of the carbonate component incorporated in the hydraulic products, notably in the layered double hydroxide or Afm phases. The situation is further complicated if Mg phases are present, either as derived from dolomitic carbonate during the preparation of the putty, or because of reactive magnesium silicates participating to the hydraulic reactions. The different kinetics of carbonation between the Ca phases (lime, portlandite) and the Mg phases (periclase, brucite) induces different maturation times, and furthermore enhances the delayed formation of carbonate-containing double layered hydroxides of the hydrotalcite-type, with severe effects on the radiocarbon dating of the architectural structures. To investigate in detail the effect of the Mg phases on the resulting radiocarbon dates, a series of Mg-containing mortars and plasters from the Castle of Cannero, on the Italian Lake Maggiore, were investigated. The castle was built on a narrow time span (1519 and 1521 by Ludovico Borromeo) and some of the dates obtained on the mortars/plasters can be critically compared to stratigraphically consistent charcoal samples and even dated graffiti made by the mason workers. The collected samples are composed by hydrated Mg-carbonates (as hydrotalcite) causing the strength development of the binder, likely related expansive process and density increase during hydration. The detailed sequence of mineral reactions obtained by Scanning Electron Microscopy (SEM), combined with careful checking by X-Ray Powder Diffraction (XRPD) of the mineral phases present in the binder fraction separated for radiocarbon dating, yields a preliminary understanding of the timing of the carbonation reactions and the complex relationship to the hydraulic products in Mg-containing mortars.

Mortar Radiocarbon Dating: contamination effects and sample characterization. The case studies of medieval sites in the Mediterranean area

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Pharmaceutical Science and Technology – via Vivaldi 43, Italy

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Abstract

The determination of the age of the ancient structures is one of the most important issues in the study of historical buildings and their evolution. In this paper a multi-analytical approach has been carried out in order to highlight the critical phases in the process of radiocarbon dating of mortar. In detail, a multi-analyses procedure useful to obtain the complete characterization of the samples and the choice of the proper parameters in the implementation of sample preparation protocol (CRYO2SONIC), will be presented.

Different analyses have been performed on mortar samples, that allow to collect clues about the presence of possible dating contamination sources, through identification of mineralogical phases and nature of the aggregates.

Polarized Light Microscopy (PLM) on thin sections, X-Ray Diffraction (XRD), Isotope Ratio Mass Spectrometry (IRMS), Thermogravimetry (TG) and differential scanning calorimetry (DSC) with simultaneous FTIR spectroscopy for the Evolved Gas Analysis (EGA) and Fourier Transform Infrared (FTIR) spectroscopy, were adopted for determining the main mineralogical and textural features of samples (e.g. binder and aggregate composition, B/A ratio) in order to correctly discriminate the different carbon dioxide sources.

Finally, some applications of the whole procedure at samples coming from different archaeological contexts of various periods (Andalusian castles, Medieval site UNESCO of Modena and the Florence Baptistery) will be presented.

Delayed Hardening in Dating Mortar

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Abstract

There is extensive proof of delayed hardening of mortar samples deep in the walls or in mortars made water resistant through mixing in terracotta or bricks.

Ever since 1994, when drilling for original mortars in an exterior wall in Finström church, Åland, have we been aware of the risks of delayed hardening in mortar sampling. We came across running mortar from deep within the wall, mortar that had not yet hardened. These results of mortar analysis indicated that this church was to be built in 200 years. The same northern wall of the church has later convincingly been dated to the 13th Century.

In 1997 we got further confirmation of the risk, when testing a big core of mortar drilled through the wall in the church of Saltvik, Åland. Samples were taken from the core at regular intervals at successive depths. The result was that the deeper into the wall we reached, the younger the mortar.

At a Roman villa, Torre de Palma, in the Iberian peninsula, extensive mortar dating was implemented. There were plenty of opportunities to test samples of cocciopesto, mortars mixed with terracotta or bricks to improve the impermeability, and to compare these with results of lime mortars from the same construction. The inevitable result was that the cocciopesto represented delayed hardening.

In Trajan’s Market, Rome, we traced signs of delayed hardening on a wall where missing bricks provided an opportunity to take successive samples of pozzolana from different depths into the wall. Again, the result was that the deeper the sampling of the mortar, the younger the result.

OSL dating of lime mortars in a singular building of NW Spain: Santa Eulalia de Boveda (Lugo)

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Abstract

The Temple of Santa Eulalia de Boveda (Lugo, Spain) is a very singular building located in Lugo, Galicia (NW Spain). It has been proposed that the building was constructed in different historical periods, based on observed architectural features and the use of several building materials, being basically granite rock, bricks and lime mortars. The mortars have been characterised and compared with the identified construction phases, but the absence of absolute ages directly obtained by dating building materials hindered the knowledge of the absolute chronology of the building. From architectural features, the building has been dated back to the Roman, Paleochristian or Early medieval periods by different authors. In a first attempt to get an absolute chronology, five brick samples corresponding to different architectural phases were dated by thermoluminescence, but heterogeneous results indicated that they were probably reused, some of them dating back to the Roman period, as observed in other buildings of Western Europe for the medieval period. Thus, optically stimulated luminescence (OSL) dating has been used to date lime mortar samples. Previously, the dated mortars were studied and characterized. Eleven lime mortar samples were taken from different parts of the building that correspond to different historical phases. Coarse quartz grains of the aggregate sand were separated and used for luminescence measurements and dose rates were assessed to get OSL ages. The obtained ages have allowed obtaining a complex chronology that indicates an historically continuous use of the building, providing ages younger than expected. These results corroborate that the paintings and the decorative reform do not correspond with the initial construction but were made in the early medieval period. This fact has strong archaeological and historical implications, showing the continuity or recovery of roman painting tradition in the beginnings of the Asturian kingdom.

Why consider a mortar measurement inter-comparison?

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Abstract

From the early days of radiocarbon dating, laboratories have been accustomed to participating in inter-comparisons, specifically to demonstrate the comparability of their measurements but also to test the effects of new developments, including different pre-treatment methods. This is especially important when the samples require intricate and complex pretreatments. The results of an inter-comparison provide valuable information to laboratories and confidence for users in the robustness and reliability of the results. The goals of a mortar inter-comparison would be to ensure that the laboratory methods used (both chemistry and instrumental) are appropriate and properly validated and that the results are traceable and thus linked to internationally recognised standards. These goals are entirely in keeping with widely accepted principles in analytical science, namely systems of analytical quality control (QC) as the fundamental basis for overall quality assurance (QA) and quality management. Part of quality assurance is concerned with establishing and maintaining primary standards and reference materials (with known activities/concentrations) and then the subsequent routine measurement of those standards and reference materials within the laboratory. An inter-comparison provides insight into the comparability of measurements (and thus bias, accuracy and precision of those measurements) and may also be able to characterise materials that can form the basis of recognised reference materials. We propose such an inter-comparison, involving in the first instance identification of a small number of mortars (suitably tested for homogeneity), recruitment of the laboratories who will take part, followed by distribution of the samples, and analysis of the results to inform laboratories on the variability in the results.

Radiocarbon dating of anthropogenic carbonates using the thermal decomposition method

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Abstract

Anthropogenic carbonates ($CaCO_3$), such as ash and lime plaster, are pyrotechnological products commonly found at archaeological sites. These materials contain radiocarbon, and in principle could be used to obtain accurate age determinations and thus significantly contribute to the establishment of absolute chronologies for the past 50,000 years. However, this is not feasible because the original isotopic signature of pyrogenic $CaCO_3$ is often altered by diagenesis and/or mixing with geogenic contaminants, resulting in major age offsets. In addition, it is difficult to isolate and separate the pristine pyrogenic fraction from the geogenic and diagenetic ones. Here we present a characterization approach of anthropogenic carbonates that includes infrared spectrometry, thin section petrography, X-ray diffraction and scanning electron microscopy coupled with high-resolution cathodoluminescence, with which we identified the pyrogenic $CaCO_3$ fraction. The latter was isolated through density separation and its carbon dioxide content recovered by thermal decomposition. The carbon aliquots thus obtained allowed the isotopic characterization of the carbonate fractions and the reconstruction of their diagenetic history, resulting in radiocarbon measurements of archaeological samples from different periods and geographic areas.

Single grain OSL dating of mortars in archaeology of architecture: state-of-art

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Abstract

SG-OSL dating of mortars is nowadays becoming a new support in analyzing the construction history of ancient buildings. The aim of the present paper is to make an overview of the progress achieved in this dating application since the very first attempts 18 years ago [1]. The special focus will be given to methodological developments performed in Bordeaux research center IRAMAT-CRPAA in the last few years and to their progressive integration into archaeological research [2,3,4].

The basic premise in OSL dating of mortars is that quartz in the sand used for making mortar is optically zeroed during the preparation process. The moment to be dated is the last exposure of mortar to light, before being embedded within the masonry and hidden from light. The first OSL dating tests on known-age archeological mortars were performed by a standard single aliquot OSL technique, indicating potential but raising also some important shortcomings linked in particular with the problems of poor bleaching [5].

A deeper research, focused on dating of known-age monuments, was carried out between 2012 and 2015 in IRAMAT-CRPAA. It consisted in an important methodological innovation: a systematic use of so-called “single grain analyses” (SG-OSL). This technique, relatively recent in the field of luminescence dating [6], allows overcoming the problems of heterogeneous light exposure. However, the change of scale of analyses from hundreds to single grains required to reassess our perception of the material in terms of its heterogeneity and to develop new concepts of data treatment [2]. Therefore, the tools for convenient assessment of “single grain” measurements, taking into account to bleaching mechanism of mortars, have been developed [3]. Contrary to usual practice, we also insist on systematic evaluation of microdosimetric characteristics in studied samples [2]. Thanks to the all aforementioned methodological advances, we are currently able to assess reliability of dating results obtained by internal control of the method itself which is a fundamental requirement for the integration of the SG-OSL dating in archaeological research in order to date the mortars of unknown age.

By its nature, SG-OSL dating requires participation of an expert on the archaeological excavation since the factors linked to architectural development of the monument and past interventions need to be evaluated *in situ* in order to determine environmental radioactivity and therefore to provide reliable dating results. Such way of working stimulates deeper interconnection of different scientific disciplines which is being concretized via multi-institutional interdisciplinary MoDAq project we conduct since 2016. As a result of this research inter-connecting archaeometry, stratigraphic archaeology, typo-chronology and history, we were able to precise chronology and demonstrate long continuity in occupation of several emblematic early medieval churches, some of which will be discussed within this paper [4, 7].

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Radiocarbon dating potential of lime binders used in Prague in Medieval Period

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Abstract

The beginning of the use of lime as a mortar binder for masonry structures at Prague castle area is in general dated to the end of the 9th century when the church of Our Lady was erected. Mortars from various buildings dating up to the 15th century have been characterised by means of optical microscopy, SEM EDS, thermal analysis, XRF, XRD and ratio determination by acid dissolution during the last 15 years. This characterisation has been also accompanied by experiments replicating historic lime technologies, mapping of local historic geological resources and by studying written sources in archives. This approach brought a relatively comprehensive understanding of the mortar materials used in that period in Prague. Overall, the mortars were all rich in binder and showed a certain chemical and mineralogical similarities. They were composed mostly from feebly to moderately hydraulic lime and a siliceous sand of fluvial nature. The origin of the raw material is in the southwest vicinity of Prague of Devonian age. Aim of this recent study was to utilise the relatively detailed understanding of the materials used at Prague castle area including the technological processes of binder production and processing and to evaluate them in terms of their potential for the use carbon dating. It specifically focused on characterisation of course binder related particles present in the historic mortars and the predominant hydraulic nature of these binders. For this purpose, three archaeological mortar samples representing different construction periods and buildings were dated by AMS using three separated CO_2 fractions. The obtained data are discussed in depth with regard to estimate the potential of a future use of the radiocarbon dating method for the site. It also offers an overview regarding the understanding of the use of natural hydraulic lime in Mediaeval Period and its hardening processes.

ABSTRACTS – POSTER PRESENTATIONS

Mortar Dating: The use of FTIR and TGMS for screening samples and understanding variation in dating results

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Abstract

Mortar can offer the most secure context from which to date material on an archaeological site. However, there is no consensus regarding the most suitable approach to use. Several approaches exist, including: dating of pure lime lumps (e.g. Pesce and Ball 2012); the isolation of a fine lime binder suspension following cryogenic breaking and ultrasonification, Cryo2Sonic (e.g. Carmine et al. 2015); the sequential dissolution of a sieved fine fraction (e.g. Lindroos et al. 2007); and nano fraction isolation (Ortega et al. 2012). These methods, while often produce positive results, can be too reliant on prior chronological information to provide confidence in the interpreted dates. Also, there can be considerable variation in the ages produced by different methods (e.g. Hajdas et al. 2017).

To both understand the differences observed between several prominent mortar dating methods (lime lumps, Cryo2Sonic, sequential dissolution) and find a suitable method of screening and evaluating the reliability of samples, Fourier transform infrared spectroscopy (FTIR) and thermogravimetric mass spectrometry (TGMS) were applied to mortar samples (from historical buildings) at various stages of preparation. In particular, ν_2/ν_4 IR absorption ratios (e.g. Chu et al. 2008) and carbonate decomposition temperatures (Fabbri et al. 2014) were used as proxies for the degree of anthropogenic calcite still present in samples.

A comparison between the dating and characterization results is presented, along with an assessment of the reliability of each method, based on intra-comparison of dates, historical information and characterization results. The feasibility of using FTIR and TGMS as a means of screening and evaluating the reliability of samples is promising but there are several areas of concern which will be discussed.

Roman building I. at Bratislava castle: characterization of mortar samples for ^{14}C dating

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Abstract

We present current research of mortars, first results of which were introduced at Zurich 2015 and Edinburgh 2016 meetings. Petrography and scanning electron microscopy of the mortar layer showed that majority of aggregate is quartz sand, with small pieces of limestone and ceramic. Steep age profiles for two samples (46-75 μm, H_3PO_4, 3 fractions/sample) indicated severe contamination and postdepositional alterations. A large difference between first fractions (2146±35BP, 2334±25 BP) predating the archaeological record (1st cent. calBC – 1st cent. calAD) testified to heterogeneous character of the mortar layer.

New analyses show that mortars from different locations within the building contain, beside quartz sand, different kinds of carbonaceous component as limestone aggregate and not totally burnt limestone fragments, recrystallisation is present within pores and around aggregates. Taking into account the composition of analysed mortars, different types of pretreatment [1, 2] are applied to check the possibility to exclude all the factors that interfere with the opportunity of getting a real age of mortars setting time. Each mortar is dated at least three times. The obtained results reflect the mortars composition and the influence of environmental conditions.

This work was supported by the Slovak Research and Development Agency under contract no. APVV-14-0550.

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Preliminary study of the lime mortars of the architectural complexes of Nanteuil-en-Vallée: Contributions and perspectives for the understanding of the construction sites, from Gallo-Roman Antiquity until the French Revolution

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Abstract

The abbey of Notre-Dame of Nanteuil-en-Vallée is a monastic site located between Poitiers and Angoulême (France). The site was dismantled during the French Revolution: the abbey church and part of the complex served as a quarry and as a source of building materials for the surrounding buildings, as evidenced by the many visible uses in the village. Seven excavation campaigns carried out between 2011 and 2017 allowed archaeologists an important study of the monastic complex. Those archaeological operations have identified many phases of construction and occupation since the Gallo-Roman antiquity to the present day.

An almost exhaustive sampling of lime mortars has been carried out within the structures accessible and observed since 2011. More than 200 mortar samples were therefore taken from the various buildings and masonry of all periods. The study of architectural binders and their comparison allows us to identify masonries and construction phases. But one of the objectives is also to observe changes or constancy in the practices, recipes and supply logic in the choice of materials for the manufacture of lime mortars. The work carried out in recent years at IRAMAT-CRP2A shows that lime mortar also provides chronological information by using SG-OSL technique. In a first step, 14 lime mortar samples were taken for SG-OSL dating: 3 samples from the "Trésor" whose chronology remains problematic, and 11 from the masonries observed during the excavations of 2017 immediately covered. These first samples will allow us to test the sensitivity of the grains and to consider, or not, SG-OSL mortar sampling on the whole stratigraphy in the nave of the church.

In order to question the supply and the use of the building materials, the abbey gives us the opportunity to study the raw materials used in a large chronological sequence from Gallo-Roman Antiquity to the present day.

Radiation defects in lime mortars and plasters studied by EPR spectroscopy

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Abstract

Electron Paramagnetic Resonance (EPR) spectroscopy is a well-established method of dating based on trapped charges, applied to various crystalline materials, including carbonates, bones and teeth. It provides a detailed insight into the structure of radiation defects – paramagnetic centers generated by irradiation, without the need of a painstaking sample preparation, often challenging in other methods. Using EPR we studied the effect of gamma radiation on lime mortars and plasters from Sveta Petka church in Budinjak, Croatia [1] and ancient settlement Hippos in Israel [2], to analyze the process of defect generation occurring during sample history. Analysis of the complex spectra revealed the presence of radiationinduced species of carbonate origin, as well as connected with additives and impurities, such as sulphates and nitrates. Since, as it has been recently shown, radiation defects can also be generated, instead of bleached, in pure calcite by UV radiation [3], we investigated also the effect of UV exposure on lime mortars. Our results can lead to deeper understanding of generation and bleaching mechanisms of paramagnetic species, which is crucial for identifying the issues, especially related to light exposition, affecting the accuracy of age determinations in trapped-charge dating methods.

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Comparison of sample preparation methods for radiocarbon dating of mortars. Case study of Irulegi Castle (Navarre, Spain)

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Abstract

In this study a combination of two different mechanical separation procedures in sample preparation for radiocarbon dating were performed. The refined materials obtained by each procedure were converted to carbon dioxide by sequential dissolution in order to compare ^{14}C ages yielded from the preparation products. The lack of the written sources of Irulegi Castle (Navarra, Spain) until AD 1230 makes mortars a potential material to be dated in order to determine building age. Previous to the mechanical separation the mineral composition of mortars was analyzed by petrographic microscopy, X-ray diffraction and cathodoluminescence. The sample preparation for AMS dating consisted on two different mechanical separations: 1.The mortar was crushed using pliers and then sieved using increasingly fine mesh widths ranging between 20–500 µm. The grain-size fraction 46–75 µm of bulk mortar was used for dating. 2. The mortar was manually crumbled and disaggregated by means of an ultrasonic bath. Particle-fractionation technique routinely used in soil and clay mineralogy studies were performed in order to isolate a pure neoformation calcite fraction of binder. Based on the differential settling of particles in a liquid a grain-size fraction < 2 µm was obtained for radiocarbon dating. A total of twenty-one ^{14}C measurements from mortar binder of three samples from the Keep Town of the Irulegi Castle were performed. Nine measures were carried out for mortars prepared by the first mechanical separation and twelve for mortars prepared by the second respectively. Additionally, charcoal fragments and a tooth from within one of the mortar samples were also dated.

OSL dating of earthen mortars from a medieval building in NW Spain: Santa Mariña de Augas Santas (Ourense)

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Abstract

The historical-archaeological building named *Basílica da Ascensión* y *Forno da Santa* corresponds to an unfinished church built on a previous structure that was transformed into a crypt. It is located in the bottom of a small depression in the location of Armeà, Allariz (Ourense, NW Spain). Previous archaeological studies have established that the construction of the crypt is the result of a sequence of constructive phases dating back to the Iron Age. Important changes occurred in the Early and Late Medieval periods and finally the with the beginning of the construction of the unfinished church in the 14th Century. However, absolute ages would be needed to confirm this chronology. In a recent project, dating the building phase was attempted for first time. Due to the absence of any datable material in the crypt (granite blocks and earthen mortars), absolute dating was attempted by optically stimulated luminescence (OSL) on the earthen mortars. Eight mortar samples were taken from the crypt, seven of them were successfully dated. Coarse quartz grains have been measured to assess the equivalent doses, based on the use of several grain sizes for each sample. Dose rates have been estimated by three different methods. Results show that the ages of samples are correlated, and that the chronological framework of the building is large, with the oldest mortars dating back to the 6th Century. Historically this date is that one of the earlier sure Christian building in the region built to commemorate the martyrdom place of a local Saint. This study has allowed to cross archaeological, anthropological, documentary and analytical data, so that today we can interpret this sequence, and build a coherent historical narrative explaining the transformation of an Iron Age Sauna into a church linked to the cult of Santa Mariña.

Radiocarbon and OSL dating of lime mortars of the Archaeological Ensemble of Tarraco (Les Ferreres Aqueduct), World Heritage Site

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Abstract

The 'Les Ferreres' Roman aqueduct, also called 'Pont del Diable' (Catalan for 'Devil's bridge') is a significant part of the Archaeological Ensemble of Tarraco, a Word Heritage Site since 2000. The aqueduct reaches a maximum height of 27 meters and is 217 meters long. It is composed by two levels of stone arches: 25 arches in the upper section, and 11 in the lower one. Historical and archaeological interpretations point to the construction of the aqueduct under the rule of Emperor August (63 BC – 14 AD) to supply water to the city of Tarraco. From 2010, an integral restauration was carried out funded by the Spanish 'Ministerio de Fomento' and the Tarragona city council. The project comprised a diagnosis of conservation problems to suggest solutions to them, to clean the monument, to investigate the stone provenance and to date the monument. A complete research work was carried out on the stone blocks and mortars, this revealed small quantities of joint lime mortar used to build the aqueduct. However, lime mortars were used to cement ashlars that lie on directly on the rock or to correct imperfections of the stone blocks, and to build the water channel (specus) of the aqueduct. The lime mortars were firstly studied and characterised, before applying absolute dating methods: radiocarbon and optical stimulated luminescence dating (OSL). AMS radiocarbon dating was applied to the lime of 7 mortar samples, while OSL was used on quartz grains within the mortar aggregate of 8 samples. The complexity and heterogeneity of the mortar components added complexity to the dating process in both techniques. However, the obtained results are quite consistent with the presumed age of the aqueduct.

Integrated dating of the Modena cathedral vaults construction and restoration (Northern Italy)

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Abstract

The Modena cathedral was built between the 12th and the 13th century, but the vaults were progressively added to the main framework during the 15th century. Some of the vaults were built using either a gypsum or a lime binder. The vaults carry the scars of several earthquakes and some were even repaired using different binders, gypsum or lime. The result is a complex patchwork of lime repairs over original gypsum vaults and gypsum repairs over original lime vaults.

After the last damaging earthquake in 2012, the anti-seismic reinforcement project was the occasion to try an integrated approach to date the ancient mortars by radiocarbon sequential dissolution [1] and optically stimulated luminescence of the sand aggregate [2] and to date the bricks using thermo-luminescence. The different techniques were applied in order to date:

1) the original vaults construction timing;

2) the restoration portions built after the main earthquakes.

One of the main goals was to investigate the timing when the vaults suffered the main damage and to correlate the main restoration works the earthquake chronology deduced from the catalogue of the historic earthquakes.

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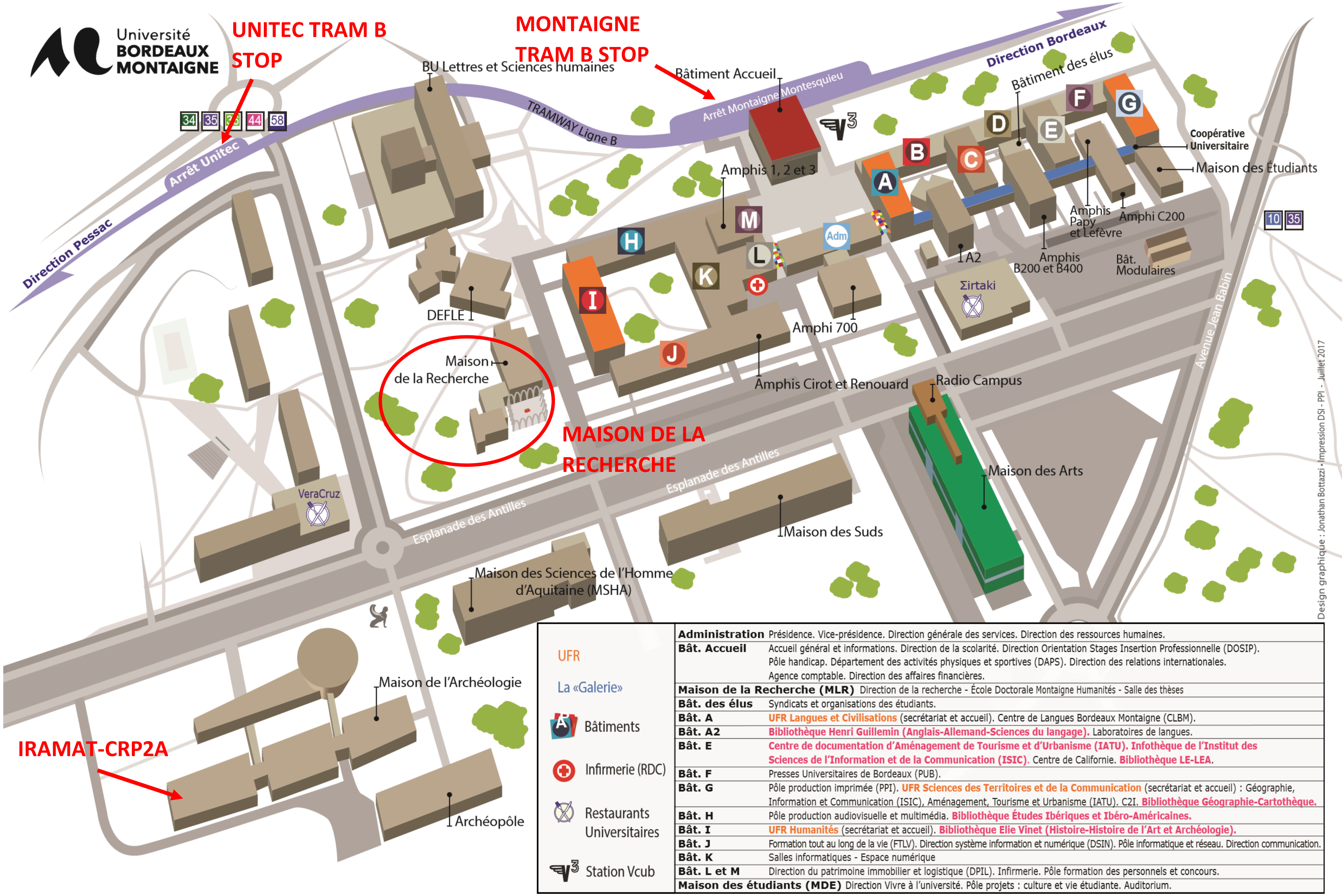

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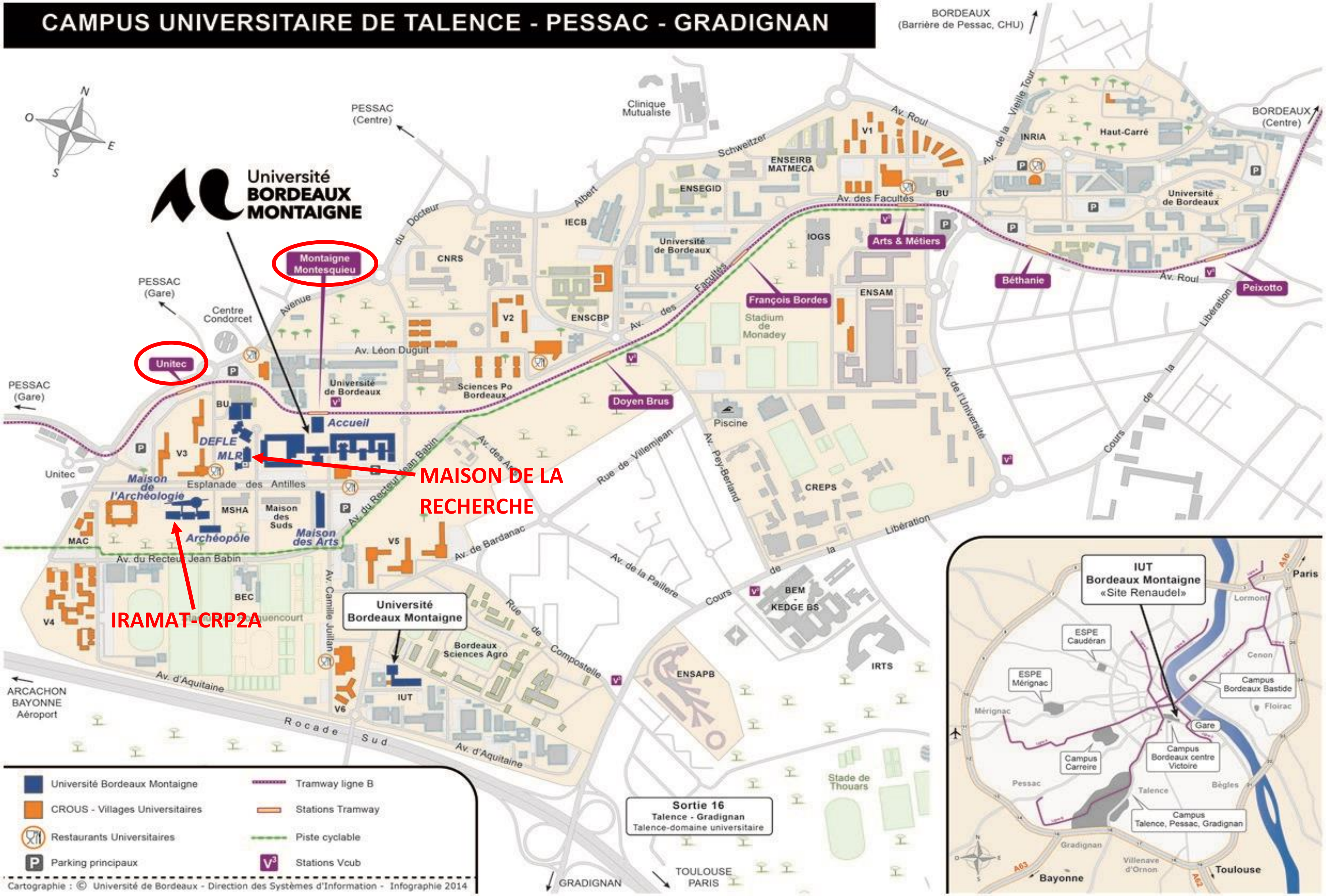
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