Cover Page



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Author: Talamo, Sahra Title: Refining 14C dating of bone >30,000 BP : establishing an accurate chronology for the Middle to Upper Palaeolithic transition in France Date: 2012-06-06

4 Establishing ¹⁴C dating at MPI-EVA

In this thesis I focus on the pretreatment of bone to obtain pure collagen and convert it into graphite for AMS measurements to obtain reliable radiocarbon dates. The individual steps of pretreatment involve extraction of collagen from bone, cleaning all the equipment used in the procedures and the conversion of collagen to graphite (graphitization). I chose to use the extraction method (method C in chapter 6; paper (Talamo and Richards, 2011)) that best avoids lab contamination. In the field I collected good quality bone samples, which were selected due to their potential for high carbon yields.

All the bone samples presented in this thesis were subject to the following pretreatment procedures, usually in batches of up to 12 samples:

- Entry in database
- Pulverisation of bone
- Decalcification
- Removal of humics
- Gelatinization
- Cleaning of the filters and checking for the removal of contamination
- Ultrafiltration
- Freeze drying

These procedures are outlined in detail below.

4. 1 Database entry

A S-EVA number is assigned to the sample and it is inserted in our database. Important fields of the sample record are S-EVA number, submitter name, sample code assigned by the submitter, name of the project or site, weight of the sample as received and a photo. All the subsequent pretreatment steps are entered into the database.

Submitter:	Sorresi 💌	S-EVA number*:	13662	(A)
Sample Code:	Y6-979 US 06.rc	Project:	Les Cottes 💌	
Material	Bone 💌	Data received:	8/18/2009	
		Weight received mg:	9635.5	Hidden value - Revision (die revision ist für das encounterte
Species:	Bison or Horse	Anatomical Part:	long bone	synchro-problem, dass access den write error nicht bringt):
Comments:	Chatellperron			
Specimen Picture:	1. if bmp - Select the whole versa 2.if jpg - Select the Frame. 1 "Object". Then "Create Fro To view, double click icon.	Bitmap and paste it to the ad hen from the Toolbar chooss m File'' and "Browse" to your	e "Insert" then	
	S-EVA13662.JPG			
* - must have valu ** - Create a new (Les. You have to enter a spec entry, but submitter, project, sa	ific value, or no data set can ample code, date entered and	be created. d material remains.	
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Figure 4.1 Entry page of the database at MPI

4.2 Pulverisation of bone

The bone is first cleaned by sand-blasting and then, using a dental drill, 500 mg of bone powder is taken. In the case of bone fragments a mortar is used to grind the bone. Bone powder is essential for a fast and efficient decalcification.

4.3 Decalcification

The sample is kept in 0.5M HCl at room temperature until no CO_2 effervescence is observed, which usually takes 4 hours. This interval is divided into two 2-hour segments after which the sample is rinsed in ultrapure water and centrifuged. Then it is kept overnight in 0.5 M HCl in a refrigerator.

4.4 Removal of humics

The following day 0.1M NaOH is added for 30 minutes to remove humics, which could have been introduced to the bone sample by ground water during the interval between burial and excavation. The NaOH step must be complemented by a final HCl step (15 minutes), to remove potential contamination from modern CO_2 taken up by the NaOH.

4.5 Gelatinization

The gelatinization step follows the method outlined in Longin (1971), at pH3 in a heater block at 75°C for 20h.

4.6 Cleaning of the filters and checking for the removal of contamination

The cleaning procedures for the ultrafilters are essential for a valid radiocarbon date (Higham, et al., 2006b). The ultrafilters are Sartorius "Vivaspin 15" of 30 KDalton size with 50ml plastic centrifuge tubes. The cleaning is designed to remove carbon-containing humectants. It is very important not to clean the filters more than 24 hours in advance as they may soften or dry out. The ultrafilters are rinsed 5 times in the centrifuge with ultrapure water for 15 to 20 minutes. Then they are bathed in ca 1 liter of ultrapure water in the ultrasonic bath for one hour, and after that rinsed 3 times.

Before the 4th centrifuge step, 1 ml of ultrapure water is added to one of the filters, and removed for analysis of remaining carbon. For this measurement the water sample is freeze-dried, another *ca*. 20 μ l ultrapure water is added and inserted with *ca*. 8 mg chromosorb into a large tin capsule. The amount of carbon is determined by combustion in the EA (see step below). The burn yield must be below 5 to 10 μ g C for this sample to indicate that the filter is not contaminated by carbon-containing humectants.

The Eeze-FilterTM' (Elkay Laboratory Products (UK) Ltd.) is bathed for 20 minutes in ca.1 liter of ultrapure water.

4.7 Ultrafiltration

The gelatine obtained in step 5 is filtered in the Eeze-filter to remove mineral particles. Then the liquid is transferred to the ultrafilter and centrifuged until the liquid in the filter is below 0.5 ml.

4.8 Freeze-drying

The filtered sample is frozen to a solid. The tube is sealed with parafilm and kept in a -28°C freezer. The tubes are kept in an inclined position so that the solution is thinly distributed along the tube, with no more than 10 mm at the thickest part. The samples stay in the freezer for at least 12 hours so that they are solidly frozen. Then the samples are transferred to the freeze-drier and lyophilized for 48 hours.

The specific lab protocol procedures for each sample (Figure 4.2) is entered into the database (Figure 4.3).

S-EVA	Notes	
	Total Bone	mg
Sample taken	mg Rest	mg
Date HCI for 2h	□ HCI for 2h □ Fridge all night_	
Date	HCI for 2h ロ Wash 3 NaOH 30min ロ Wash 3 HCI 15min ロ Wash 3	times H2O 🗆
Date Heater in with 10ml of Ph	13Time in	
Date Heater out	Time out	75°C for 20h
	Cleaning Eeze – Filter	
Date 20 minu	5	
Clea Date	ning Ultrafilter procedures	
15 min	centrifuge with H2O pure □	
	centrifuge with H2O pure □	
	in Ultrasonic water	
15 min	centrifuge with H2O pure □	
	centrifuge with H2O pure □	
	centrifuge with H2O	
Date Centrifuge and Samples i	n the fridge	
Date Freeze – dryer in	Time in	
Date Freeze – dryer out	Time out	
Collagen mg	•1 51	
Circa 0.5mg of Collagen for C/	N	
Sent Collagen to	mg Remaining Collagen at MPI Date Date	
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Radiocarbon Age	Collagen back from the AMS L	abmg

Collagen extraction protocol

Figure 4.2 Lab protocol with all the procedures made during the pretreatment

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3 sample					
* Submit Project Lab V	Worker Sample Pretreat Gr	aphite CN Send & R	Receive Sample Results T	Fest List	
Lab Worker:	Talamo 💌				
S-EVA number:	13662	Sample Code:	Y6-979 US 06.rc		
Date entered:	8/27/2009	Weight used mg:	1609.1		
HCL date:	8/31/2009	Rest mg:	7938.2		
Heater date:	9/1/2009	To be date:			
Centrifuge with ULF:	9/2/2009	% Collagen:	5851.272		
Freeze dry date:	9/7/2009				
Comments:					
Collagen mg:	27.5				
				\checkmark	
Record:	61 🕨 🕨 🕨 🖳 a	of 418			

Figure 4.3 Input of the lab protocol of the pretreatment and calculation of the % collagen

Important parameters are the date of the various steps, the weight used and the final weight of the collagen. At this point the collagen yield is available, which should be a minimum of 5 mg for 500 mg initial bone powder (1% yield limit). The minimum amount of collagen for graphitization is 3 mg.

4.1 Graphitization steps

All the collagen obtained after the pretreatment outlined above is graphitized according to the following procedures:

- Loading of collagen into tin caps
- Combustion in an Elemental Analyser (EA)
- Determination of carbon yield and C:N ratio
- Determination of $\delta^{13}C$ and $\delta^{15}N$ in a mass spectrometer
- Cleaning the CO₂ gas containers and conditioning of the iron catalyst
- Collection of CO₂ in the rigs
- Addition of hydrogen

- Conversion of CO₂ into graphite in the graphitizer
- Check of graphitization parameters
- Preparation of blank samples
- Preparation of shipment to an AMS facility and submission to the AMS laboratory for radiocarbon measurement.

4.1.1 Loading collagen into tin caps

The collagen is loaded into tin capsules, which are pre-cleaned in cyclohexane and acetone. An empty tin capsule is combusted to check that the blank contribution is < 2 µg C.

4.1.2 Combustion in Elemental Analyser (EA)

The collagen is combusted in the EA (CHN analyzer) system, in a sequence of up to 10 samples limited by the amount of available gas containers. Each sample combustion is preceded by the combustion of an empty tin capsule to purge the system. The sample is injected into the furnace together with a stream of helium and oxygen. The combustion furnace is at a temperature of 1000°C and with the addition of tin the combustion temperature reaches 1500°C; the subsequent reduction furnace is used to complete the combustion at 600 °C (Figure 4.4).



Figure 4.4 Elements of the graphitization: combustion in the EA (middle), $\delta^{13}C$ and $\delta^{15}N$ determination in the mass spectrometer (right) and the graphitizer (left).

The helium acts as carrier gas. The combustion products are sent through several gas chromatographic columns (GC) to purify and to separate the components of interest, nitrogen (N), CO_2 carbon (C), and hydrogen (H). This information is recorded by the EA software (Figure 4.5).

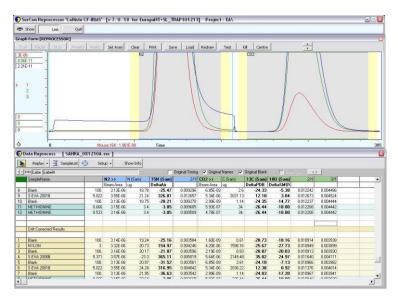


Figure 4.5 Protocol of the elemental analyser. The peaks represent the separation of $C(CO_2)$ and N

4.1.3 Determination of carbon yield and C:N ratio

After the successful combustion of a sample the key parameters for quality control of bone collagen are available. These are the amount of carbon and nitrogen in the sample, which is used to determine the C:N ratio, These data and isotopic data from the next step are then entered into the database (Figure 4.6)

ubmit Project Lab W	'orker Sample	Pretreat (Graphite C	N Sen	d & Receive	Sample Results	Test	List		
Lab Worker:	Talamo	~	N	ot to be dal	ed:					
S-EVA number:	2232	#	S	ample Code	B15 (20)				
Date preparation:	11/15/2007									
Mg of collagen:	0.52									
Results File Path:										
Results (Select the	13C	15N	%C	%N	C:N					
whole Excel work- sheet and paste it	-20.66	6.30	42.98	15.61	3.21					
to the adjoining frame or vice versa):										
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Figure 4.6 Input of isotope data into the database

4.1.4 Determination of $\delta^{13}C$ and $\delta^{15}N$ in a mass spectrometer

A small fraction, approximately 1%, of the purified gases are sent to the mass spectrometer (Figure 4.7), connected to the EA, to measure the stable isotope values (δ^{13} C and δ^{15} N) (Table 4.I).

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			Beam Area 1	5	DeltaPDB D	eltaSM0\						and the second	-
	test CO2 REFGAS	10.	5.06E-07 4.53E-07	0.	0.00	0.00	0.012031	0.004302		Magnified Sensor Di	play		
	cample1	10	4.5.3£-07 4.74E-07	40.	-25.00	-18.00	0.012031	0.004317		Tempera	tures	Sensors	ALC: NO.
4	sample2	10	4.78E-07	42.195	-24.91	-13.65	0.012034	0.004323					
5		10	4.63E-07	40.	-25.00	-18.00	0.012035	0.004325		Combustio	1000	Pressure 1	21
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	test	10.		45.074	-24.95	-16.86	8.010957	0.003943		GC2	525	Flow 2	359
	CO2 REFGAS	10.	4.53E-07	40	-25.00	-18.00	0.010956	0.003938		OUL.	525		222
	sample1	10		41.593	-24.99	-16.70	0.010956	0.003943			 1000000000000000000000000000000000000		
	sample2 CO2 REFGAS	10		41.564	-25.01	-16.94	0.010956	0.003942	The second s				
-	SWETIET WES	10	4.03E-07	40.	-25,00	-18.00	0.010306	0.005536					
		1											

Figure 4.70 utput page of the mass spectrometer for $\delta^{13}C$.

Name	Weight/Vol	¹³ C (Sam)	¹⁵ N (Sam)	С	Ν	C:N
	mg	DeltaPDB	DeltaAir	%	%	
EVA 0008 Nylon	4.092	-29.5	1.6	60.90	11.80	6.02
EVA 0008 Nylon	4.802	-29.5	1.6	60.90	11.80	6.02
Nylon 66 B2 Sample	4.708	-29.5	1.2	60.81	11.85	5.99
Nylon 66 B3 Sample	4.745	-29.5	1.2	60.83	11.80	6.01
	average	-29.54	1.52	60.86	11.83	
	stdev	0.04	0.24	0.05	0.03	

Table 4.1 Example of a determination of stable isotope ${}^{13}C$ and ${}^{15}N$ for the reference material of Nylon 66

4.1.5 Cleaning the CO_2 gas containers and conditioning of the iron catalyst

The CO₂ gas containers are glass tubes closed by metal valves, called rigs (Figure 4.8).



*Figure 4.8 CO*₂ *gas container (rig) filled with iron catalyst.*

The rigs are filled with 1.5 to 3 mg iron catalyst (Aldrich Chem. Co. <10micron 99.9%) and the optimal ratio of iron to carbon was determined to be 3 to 1 (Vogel, et al., 1984). To avoid contamination from absorbed CO₂ or particulates the iron and the glass surfaces are cleaned by adding H₂ (99.999%) at 500 mbar into the rigs and placing them in the oven at 450 °C for 1 hour.

4.1.6 Collection of CO_2 in the rigs

Most of the CO_2 is collected in a rig attached to the gas collection system (Figure 4.9) and is trapping using liquid N_2 .

Hydrogen is added to the frozen CO_2 in a quantity sufficient to guarantee a complete reduction of CO_2 . In our system an excess of H_2 is used with the ratio $H_2:CO_2=2.2:1$



Figure 4.9 Graphitization system manufacture by the Oxford laboratory

4.1.7 Conversion of CO_2 into graphite in the graphitizer

The rig is placed in the oven at 560 °C for 6 hours, where CO_2 is reduced to carbon and water vapour. The latter is removed by cooling one finger of the rig (Figure 4.10).



Figure 4.10 Reduction of CO_2 to graphite using iron as catalyst in an oven (top section); water vapour is removed by immersing the vertical finger of the rig into a cooling bath (left and right section).

4.1.8 Check of the graphitization parameters

During the reduction of CO_2 to carbon hydrogen is consumed at the ratio 2:1 with respect to carbon. Therefore the pressure in the rigs after reduction will be low reflecting the excess amount of hydrogen. Typically we use 400 mbar of hydrogen which results in a residual pressure of *ca*. 80 mbar. This pressure is checked by reconnecting the rigs to the gas collection system. All these parameters are entered in the database (Figure 4.11).

sample Submit Project Lab	Worker Sample Pretreat Gra	ohite CN Send &	Receive Sample Results Test List	
Lab Worker:	Talamo 💌	Not converted in	to graphite:	
S-EVA number:	2000 B	Sample Code:		
Date start:	12/10/2008	Log Reference:	SAHRA081012a	
Mg of collagen:	5.371	μg C:	G 1.651	
Final Pressure:	104 mbar			
			\checkmark	
cord: 🚺 🔳	75 🕨 🕨 💌 🖬 of	202		
		302		

Figure 4.11 Input of graphitization parameters into the database

4.1.9 Preparation of blank samples

All steps of pretreatment and graphitization may contribute exogenous carbon (contamination). Therefore ¹⁴C free material (old bone, Nylon 66, Pliocene wood) is pretreated and graphitized in the lab. These samples are called blank samples and they are prepared at the same time as the archaeological samples, and are also sent to the AMS facilities to establish the level of ¹⁴C activity in the blanks.

4.1.10 Preparation of shipment to an AMS facility and submission

At this point the samples are ready to be sent to an AMS facility, where the graphite will be pressed into a target and measured in batches in the accelerator. A batch of target usually consists of a number of samples, standards and blanks. For this thesis the samples were submitted to the laboratories of Oxford. Kiel and Mannheim/Zurich. All dates of shipments and the dating results are recorded in the database providing the final list of samples and their ages (Figure 4.12 - 4.13 - 4.14)

ample Submit Project Lab '	Worker Sample Pretreat Gr	phite CN Send & Receive Sample Results Test List	
Lab Worker:	Talamo 💌	Not to be sent	
S-EVA number:	2000 D	Sent Collagen:	
Date of shipment:	12/19/2008	Sent Graphite:	
Date expected:	2/16/2009	Sample Code:	
Sent to:	Oxford 💌		
Collagen mg sent:	7.8		
Collagen mg rest	43		
		\checkmark	
rd: 🚺 🖣	77 🕨 🕨 💌 🖳 a	382	

Figure 4.12 Table of shipment and dating results of a sample

Lab Worker:	i alamo 💌			
S-EVA number:	1601.1	Sample Code: SP-1461	Results PDF (Important Note: The F subfolder of the database file - That	means at present on
Sent to:	Kiel 💌	AMS Lab Nr.: KIA 37396	humfsshared in "humfsshared:Rese 14\Subfolder"]:	arch Projects\C
C14 Age: 38280	En+-1d: 560	Cal BP:	KIA Results\TalamoS090721.doc	
	Err+- 2dt	d °C: -12.81	Choose Document	Open Document
Result Graph Path:				
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Final EVA nu	mber:			
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Figure 4.13 Input of data as reported by the AMS facility

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S-Eva 9635 9636 13677 M 13678 13679 13671 13672 13672 13675 M 13676 M	Vertexteed (weight pretr. do 24-1258 Y4-279 US08 or 23-356 US08 rc 24-311 US04 rc Y4-51083 US04 9 Y6-1681 US08 or 23-362 US08 0 Y5-1654	Sent to Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10831 MAMS_10832 MAMS_10826 MAMS_10827 MAMS_10828 MAMS_10828	Sample Side Les Cottes Les Cottes Les Cottes Les Cottes Les Cottes Les Cottes Les Cottes Les Cottes Les Cottes Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9	49.7 22.9 30.1 20 16.1 13.4	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0	38130 43980 39460 37940 38310 33170 33570 39550 40430	470 650 540 460 250 270 560 610	
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S-Eva 9695 9696 9677 M 13677 M 13677 M 13677 M 13676 M 13675 M 13676 M 13663 13665	etreated (weight pretr. do Z4-1258 Y4-279 US08.0 Z3-356 US08.rc Z3-289 US08.rc Y4-311 US04.9 Y6-1681 US04.9 Y6-1681 US08.0 Z3-362 US08.0 Y5-1654 Y5-125 US 06 S5-557 US 06	Sent to Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10831 MAMS_10832 MAMS_10827 MAMS_10827 MAMS_10828 MAMS_10829 MAMS_10814 MAMS_10814	Sample Side Les Cottes Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9 1933.5	49.7 22.9 30.1 20 16.1 13.4 13.7	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0 0.71 0	38130 43980 39460 37940 38310 33170 33570 39550 40430 32550 34590	470 650 540 460 500 250 270 560 610 250 300	
S.Eva 9635 9636 13678 13679 13671 13672 13675 M 13663 13665 M 13667	Every Series of Code 24-1258 24-1258 24-1258 24-279 US08 to Z3-356 US08 to Z3-356 US08 to Z3-326 US08 to Z3-356 US08 to Z3-356 US08 to Z3-362 US08 0 25-1654 24-525 US 06 S6-557 US 06 Z4-3266 US 06.1	Sent to Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10831 MAMS_10832 MAMS_10826 MAMS_10826 MAMS_10828 MAMS_10828 MAMS_10814 MAMS_10814 MAMS_10814	Sample Side Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9	49.7 22.9 30.1 20 16.1 13.4	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0.71 0 1.91	38130 43980 39460 37940 38310 33170 33570 39550 40430 32550 34590 35754	470 650 540 460 500 250 270 560 610 250 300 318	
S-Eva 9635 9636 13677 M 13678 13679 13675 M 13676 M 13665 M	etreated (weight pretr. do Sample Code Z4-1258 Y4-279 US08 to Z3-356 US08 to Z3-356 US08 to Z3-289 US08 to Z3-289 US08 to Z3-289 US08 to Z3-289 US08 to Z3-361 US08 0 Z3-362 US08 0 Y5-1654 Y5-1225 US 06 S6-557 US 06 Z4-3286 US 06.1 Z4-3368 US 06.1	Sent to Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10832 MAMS_10832 MAMS_10826 MAMS_10828 MAMS_10828 MAMS_10828 MAMS_10814 MAMS_10814 UnK 41 MAMS_10814	Sample Side Les Cottes Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9 1933.5 826.6	49.7 22.9 30.1 20 16.1 13.4 13.7 15.8	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0.71 0 1.91 0	38130 43980 39460 37940 38310 33170 33570 39550 40430 32550 34590 35754 37330	470 650 540 460 250 270 560 610 250 300 318 430	
S-Eva 9635 9636 13678 13679 13671 13675 13676 13675 13676 13663 13665 13668 13668	Sample Code Z4-1258 Y4-279 US08 0 Z3-356 US08 0 Z3-356 US08 rc Z3-3289 US08 rc Y4-311 US04 Y5-1083 US08 0 Z3-352 US08 0 Z3-352 US08 0 Z3-352 US08 0 Y5-1654 Y5-1225 US 06 Z4-3268 US 06.1 Z4-3368 US 06.1 Z4-3368 US 06.1 US06.15 P5-785	Sent to Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10831 MAMS_10832 MAMS_10828 MAMS_10828 MAMS_10828 MAMS_10828 MAMS_10828 MAMS_10816 UnK 41 MAMS_10824 UnK 43	Sample Side Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9 1933.5 826.6 701.5	49.7 22.9 30.1 20 16.1 13.4 13.7 15.8 18.9	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0.71 0 1.91 0 2.69	38130 43980 39460 37940 38310 33170 33570 39550 40430 32550 34590 34590 35754 37330 33535	470 650 540 460 250 270 560 610 250 300 318 430 224	
S-Eva 9635 9636 13677 M 13679 13673 13671 13675 M 13675 M 13665 M 13665 M 13668 M 13669	Sample Code Z4-1258 Y4-279 US08 to Z3-356 US08 to Z3-356 US08 to Z3-356 US08 to Z3-359 US08 to Z3-361 US04 Y5-1081 US04 Y5-1081 US08 to Z3-362 US08 to Z3-362 US08 to Z3-362 US08 to Z3-362 US08 US-1061 Z4-3368 US-061 Z4-3368 US-061 US04 5bj R5-785 Z4-3368 US-061	Sent to Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim Mannheim	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10831 MAMS_10826 MAMS_10826 MAMS_10827 MAMS_10826 MAMS_10828 MAMS_10829 MAMS_10814 MAMS_10814 UnK 41 MAMS_10824 UnK 42	Sample Side Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9 1933.5 826.6 701.5 642.1	49.7 22.9 30.1 20 16.1 13.4 13.7 15.8 18.9 21.2	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0.71 0 1.91 0 2.69 3.3	38130 43980 39460 37940 38310 33170 33570 39550 40430 32550 34590 35754 37300 33535 37016	470 650 540 550 250 270 560 610 250 300 318 430 224 290	
S-Eva 9635 9636 13677 M 13679 13673 13675 M 13675 M 13663 13665 M 13668 M 13668 13668	etreated (weight pretr. do Sample Code Z4-1258 Y4-279 US08 to Z3-356 US08 to Z3-356 US08 to Z3-289 US08 to Z3-289 US08 to Z3-289 US08 to Z3-289 US08 to Z3-362 US08 0, Y5-1654 Y5-1225 US 06 Z4-3286 US 06.1 Z4-3368 US 06.1 Z4-3368 US 06.1 X6-205 US 06.07	Sent to Mannheim Mann	Lab Nr. MAMS_10803 MAMS_10804 MAMS_10830 MAMS_10832 MAMS_10832 MAMS_10826 MAMS_10828 MAMS_10828 MAMS_10828 MAMS_10814 MAMS_10816 UnK 41 UnK 42 UnK 42 UnK 40	Sample Side Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9 1933.5 826.6 701.5 642.1 542.9	49.7 22.9 30.1 20 16.1 13.4 13.7 15.8 18.9 21.2 11.6	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0.71 0 0.71 0 1.91 0 2.69 3.3 2.14	38130 43980 39460 37940 38310 33170 33570 39550 40430 32550 34590 35754 37330 335754 37330 33535 37016 35273	470 650 540 460 500 250 250 610 250 610 250 300 318 430 224 224 229 224	
S-Eva 9635 9636 13679 13671 13671 13675 13676 13663 13665 13668 13668 13668 13668 13668 13664	Sample Code Z4-1258 Y4-279 US08 to Z3-356 US08 to Z3-362 US08 0 Y5-1654 Y5-1225 US 06 Z4-3286 US 06.1 Z4-3368 US 06.1 Z4-3368 US 06.1 Z4-3368 US 06.1 Z4-3368 US 06.1 Z5-2785US 06 Y5-2785US 06.07 Y5-2785US 06.07	Sent to Mannheim Mann	Lab Nr. MAMS_10803 MAMS_10803 MAMS_10830 MAMS_10832 MAMS_10826 MAMS_10826 MAMS_10827 MAMS_10828 MAMS_10828 MAMS_10828 MAMS_10816 UnK 41 UnK 43 UnK 42 UnK 40 UnK 40 UnK 37	Sample Side Les Cottes	1466.8 7492 793.8 618.8 1011.5 1013.9 1933.5 826.6 701.5 642.1 542.9 1887.9	49.7 22.9 30.1 20 16.1 13.4 13.7 15.8 18.9 21.2 11.6 17.7	3.39 3.06 0 3.79 0.32 1.59 1.32 0 0 0,71 0 0.71 0 1.91 0 2.69 3.3 2.14 0.94	38130 43980 39460 37540 38310 38310 33570 40430 32550 34590 35754 37330 35754 37330 33535 37016 35273 40560	470 650 540 250 250 250 560 610 250 300 250 318 430 224 290 224 290 244 400	
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Figure 4.14 Example of a summary sheet of an archaeological site (Les Cottés)