

CHARLES UNIVERSITY, PRAGUE RADIOCARBON MEASUREMENTS I

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ABSTRACT. We converted to CO₂ samples of organic materials, Quaternary carbonate rocks and carbonates extracted from groundwater. We measured ¹⁴C radioactivity in a proportional counter with an effective volume of 723ml filled to 0.28MPa. A mechanical box with an inside cylinder of a plastic scintillator, 4cm thick, arranged in anticoincidence provides the shielding. We present a review of radiocarbon ages of Quaternary and archaeological samples.

INTRODUCTION

The Charles University radiocarbon dating laboratory has been in operation at the Department of Hydrogeology and Engineering Geology, Faculty of Science, since 1972. The laboratory was established to conduct research in radiocarbon dating groundwater, the Quaternary period and archaeology.

Our equipment consists of a vacuum line processing samples to carbon dioxide which is used as filling gas, a proportional gas counter with massive and anticoincidence shielding and measuring and recording devices.

SAMPLE PROCESSING

After macroscopic impurities are removed from the samples, they are dried and crushed if necessary. Carbonates are removed by boiling in 2% HCl and washing until a neutral reaction occurs. Humic acids are dissolved with a 2% NaOH solution at 80°C for 24 hours and precipitated from the solution with 2% hydrochloric acid. The sample is finally washed in distilled water until neutral and dried. The dried sample is then burned in a double quartz tube in an oxygen stream using cobaltcobaltic oxide as the catalyst, purified on silver wool and absorbed in CO₂-free 2% sodium hydroxide solution with CuSO₄ added to remove H₂S. From the alkaline solution, the CO₂ is evolved by acidification with citric acid and subsequently dried and purified by sequentially trapping in dry ice liquid nitrogen, hot CuO and hot Ag traps to remove traces of sulphur and nitrogen. Afterwards, the CO₂ is filled into the detector. All the chemicals were p.a.¹ grade purity.

Carbon is extracted from groundwater in the field either by precipitating bicarbonates by barium chloride as barium carbonate (water sampling for ¹⁴C analysis, IAEA nd) or by trapping the bicarbonates on a basic ion exchanger (Crosby & Chatters 1965). In either case, CO₂ is evolved by phosphoric acid and is absorbed in CO₂-free sodium hydroxide solution with added CuSO₄. Afterwards, CO₂ is evolved by citric acid and treated as above.

Calcium carbonate samples of, eg, sediments and shells, are treated in the same way as BaCO₃ extracted from groundwater.

¹*pro analysi* = for analysis, in analytical chemistry, means the purity grade

MEASURING TECHNIQUES

After chemical treatment and purification, the sample, in the form of CO₂, is expanded into a proportional counting system consisting of an internal proportional gas counter and anticoincidence and massive shielding.

All construction materials of the counting and shielding equipment were selected and checked for low radionuclide contamination (Tykva 1974: 177–179). The detector section of the counting arrangement in a mobile light-tight case consists of an internal proportional counter surrounded by an anticoincidence cylinder of a plastic scintillator, 4cm thick, with photomultipliers at both ends. The counter in the case is connected by tubing to the filling line and, through a cable, to a preamplifier. The counter, of 723ml effective volume, is made from a copper tube purchased from an old brewery pipeline in which a silver-coated tube of pure quartz is inserted as a cathode. The central anode is a 30 μ m diameter molybdenum wire.

The light-tight case is inserted into a shielding box consisting of several layers. The outer lead was purchased from 60- to 80-year-old water-supply pipelines, the inner low-level lead from Bolidens A S, Sweden. Both materials were processed in special melts with protection against fallout and radionuclide contamination. Soft steel covering the lead was manufactured analogously on special order. Polymers containing dispersed boric acid are used as neutron shielding. The details of the arrangement were described earlier (Šilar & Tykva 1977).

At 0.28MPa filling pressure of pure CO₂ (83 mmol C at 20°C), we obtain a 400V working plateau with a slope of < 1%, working voltage of 5.35kv and a background of 1.92 \pm 0.03cpm. The count rate of 0.95 oxalic acid standard “modern” is between 10.5 and 10.8 \pm 0.1 cpm.

The results of ¹⁴C measurements are calculated to ¹⁴C ages using a Hewlett-Packard 9845 computer. Corrections are incorporated for barometric pressure, room temperature, dilution of sample if necessary, initial ¹⁴C activity of groundwater samples and δ^{13} C.

¹⁴C ages are calculated using the Libby half-life of 5568 \pm 30 years. Counting errors are expressed at 1 σ confidence level. Calibrated ages, if presented, were calculated according to Damon, Long and Wallick (1972) before the new calibration curves (Stuiver & Kra 1986) were published.

ARCHAEOLOGIC SAMPLES

Egypt

Samples of Egyptian origin were submitted mostly by Eugen Strouhal, National Museum, Prague, from the Náprstek Museum collections of Asian, African and American cultures, a section of the National Museum in Prague. Samples were also submitted from other museums that contributed to the exposition of mummies at the National Museum in 1971 and from the findings of the Czechoslovak Institute of Egyptology, Charles University, in Egypt. Other sources are also indicated.

CU-38. Coffin wedge**2470 \pm 260**

Sample A 3263 of the Natural History Inst Slovak National Museum, Bratislava. Tamarisk wood (*Tamarix articulata* V) (Březinová & Hurda 1976).

General Comment: sample was diluted with inactive CO₂ due to small amount of available wood. Coffin allegedly originated during Saitic period, lasting until beginning of Late periods (664–500 BC), according to Egyptologic dating by M Verner (Strouhal & Vyhnánek 1979).

CU-77. Mummy wrapping **1970 ± 130**

Comment: mummy wrapping supplied by Museum of Fine Arts, Philadelphia, USA, for autopsy carried out by international team, Feb 1, 1973, Wayne State School of Medicine, Detroit. Sample was labelled PUM II. Dated to 2120 ± 70 BP (170 ± 70 BC, Cockburn *et al* 1975) by R Stuckenrath, Smithsonian Institution.

CU-88. Mummy wrapping **2770 ± 120**

Part of mummy wrappings, sample no. P 624 c from Náprstek Museum, Prague.

Comment: well-preserved tissue was identified as flax (Strouhal, pers commun, 1979). According to embalming techniques, mummy P 624 b should date to Third Intermediate period, 1087–664 BC. According to Egyptologic dating of coffin by M Verner, mummy should date to Greek period, 332–31 BC (Strouhal & Vyhnánek 1979). Calibrated age, 1028 ± 144 BC, agrees with age derived from embalming techniques rather than with date of coffin. Hence, possibility that older mummy was displaced into newer coffin cannot be excluded (Šilar 1979).

CU-89. Mummy wrapping **3290 ± 130**

Part of mummy wrappings P 623 c, dark disintegrating flax cloth, from Náprstek Museum.

Comment: mummy could not be dated by examining embalming techniques due to bad preservation of mummy. Egyptologic dating by M Verner assigns coffin to Greek period, *ie*, 332–31 BC (Strouhal & Vyhnánek 1979). Neither calibrated, 1691 ± 160 BC, nor uncalibrated age of sample agrees with style of coffin. Displacement of older mummy into newer coffin seems more probable here than for CU-88.

CU-79. Block of wood **2770 ± 140**

Sample P 4662 is from Egyptian coffin from collection of Náprstek Museum. Dated to provide authenticity of sample, which proved to be genuine, made during Third Intermediate period (1087–664 BC).

Abusir series

Samples from archaeological site on edge of Western Desert above Abusir (29°54'N, 31°13'E), royal cemetery of 5th Dynasty, 30km south of Cairo on west bank of Nile River. Site was investigated by Czechoslovak Institute of Egyptology, Charles University. Coll and subm by E Strouhal. Wood samples identified by Březinová and Hurda (1989).

CU-413. Abusir J 1628 **2360 ± 170**

Wood (*Ficus* sp) from coffin of Tomb J 1628, from area around mastaba of Ptahshepses, secondary cemetery.

CU-414. Abusir J 1555 **2070 ± 160**

Wood (*Acacia arabica*) from coffin of Tomb J 1555, from area around mastaba of Ptahshepses.

CU-437. Abusir A 15 P 5448 **2490 ± 150**

Linen of mummy wrappings, P 5448, from Burial A 15 inside mastaba of Ptahshepses. Sample from Náprstek Museum, Prague.

Comment: dated to compare sample from inside mastaba with two samples from tombs situated around mastaba. We proved contemporaneity of samples using 2σ (Šilar 1989).

CU-415. Abusir 1/XX/76 3290 ± 170

Wood from coffin. Isolated tomb south of mastaba of Princess Khekeretnebti.

Comment: this tomb was ca 1km south of mastaba of Ptahshepses; result points to beginning of New Kingdom.

Saqqara series

Textiles and charcoal buried in shafts and chapels of tomb of Horemheb, Saqqara (29°51'N, 31°14'E) were investigated by Egypt Exploration Society, London and Antiquity Museum Leyden, in joint expedition. Coll and subm by E Strouhal.

CU-484. Saqqara I D 2450 ± 150

Charcoal from Shaft I, Room D, mixed with bones.

CU-488. Saqqara I F 2790 ± 150

Charcoal from Shaft I, Room F, mixed with bones.

CU-489. Saqqara IV N 4360 ± 160

Charcoal from Shaft IV, Room N, mixed with burials N 1 and N 2.

CU-490. Saqqara II A 59 2540 ± 220

Linen covering convexity of Skull S 9 found in Shaft II, Room A.

Comment: sample CU-489 is distinctly different from CU-484, -488 and -490, which confirms older age of original burial in Shaft IV, Room N compared with other samples. Date is even too old if time of construction of tomb in second half of 14th century BC is considered. Wood that yielded charcoal was apparently very old re-used piece. Other three samples were mutually similar. Their mean age falls in Third Intermediate period, 1087–664 BC.

Kalabsha and Wadi Qitna series

Samples of two close archaeological sites, Wadi Qitna (23°33'N, 32°55'E) and Kalabsha (23°34'N, 32°55'E) were investigated by joint Charles University and Náprstek Museum expeditions. Coll and subm by E Strouhal (1984).

CU-132. Kalabsha KS t K 20/57 1500 ± 100

Fragments of wood probably from funeral bed.

CU-133. Wadi Qitna WQ t 407 1730 ± 120

Fragments of wood probably from funeral bed.

CU-134. Wadi Qitna WQ t 74 1620 ± 100

Fragments of wood probably from funeral bed.

Comment: all samples belong to pre-Christian Ballana (X group) culture existing in Nubia from 2nd half of 3rd century until beginning of 6th century.

Iraq

CU-75. Aqar Quf **3330 ± 140**

Stalks of reed coll by J Šilar identified as *Phragmites australis* (Cav) (Kosinová, pers commun, 1976) from straw bands reinforcing adobe structure of ziggurat at western border of Baghdad (33°20'N, 44°20'E).

CU-141. Aqar Quf **3220 ± 110**

Stalks of reed, origin same as CU-75.

Comment: according to al-Tikriti (1970), Aqar Quf was founded by King Kurigalzu I at beginning of 15th century BC during Kassite period.

CU-180. Tell es-Sawwan **7240 ± 150**

Charcoal from Tell es-Sawwan at Samara (34°12'N, 43°53'E), Iraq. Lumps of charcoal up to 5mm in diameter with some admixture of silt. Subm by Fuad Safar, Directorate of Antiquities, Baghdad, Iraq.

Comment: dated by Theresa Carter, University Museum, Philadelphia, Pennsylvania as P 856 – charcoal lumps from gypsum pit, Level 3, Floor 3, 1.5m below surface – at 7299 ± 86 (Fuad Safar, pers commun, 1973).

GEOLOGIC SAMPLES

Kuwait

Oolitic sediments and shells from Persian Gulf coast of Kuwait. Samples coll by F Picha at Mena Saud (28°44'N, 48°28'E), Al-Khiran (28°39'N, 48°28'E) and coastal cliff south of Al-Khiran (28°33'N, 48°28'E).

CU-74. Al-Khiran **$\delta^{14}\text{C} = 105.07 \pm 1.85$ pMC**

Shells of living *lamellibranchs* from beach.

$\delta^{13}\text{C} = +1.0\text{‰}$

CU-70. Al-Khiran **380 ± 160**

Recent oolitic sand from top of present barrier beach, ca 7m asl, ooids including cement.

CU-99. Al-Khiran **1570 ± 120**
 $\delta^{13}\text{C} = +4.8\text{‰}$

Younger oolitic limestone from ridge ca 6m asl forming ancient barrier beach, ooids; cement was separated.

CU-109. Al-Khiran **1990 ± 130**

Younger oolitic limestone, from same sample as Cu-99, inner part of ooids; cement was separated.

CU-97.	Coastal cliff	4030 ± 140
	Layer of older oolitic limestone, ca 10m asl, ooids; cement was separated.	
CU-110.	Coastal cliff	4750 ± 140
	Older oolitic limestone, inner part of ooids; from same sample as CU-97.	
CU-98.	Coastal cliff	3070 ± 130 $\delta^{13}\text{C} = +4.0\text{‰}$
	Older oolitic limestone, separated cement from between ooids; from same sample as CU-97.	
CU-73.	Al-Khiran	22,850 ± 1220
	Ridge of quartz oolitic sandstone, ca 9m asl, ooids.	
CU-137.	Al-Khiran	2870 ± 110 $\delta^{13}\text{C} = +2.9\text{‰}$
	Inner part of shells of fossiliferous horizon, ca 1m asl.	
CU-182.	Al-Khiran	1990 ± 110 $\delta^{13}\text{C} = +2.5\text{‰}$
	Inner part of shells of fossiliferous horizon, ca 1m asl.	
CU-138.	Mena Saud, quarry	2930 ± 110 $\delta^{13}\text{C} = +4.1\text{‰}$
	Inner part of shells of fossiliferous horizon.	
CU-181.	Mena Saud, quarry	3520 ± 120 $\delta^{13}\text{C} = +2.5\text{‰}$
	Inner part of shells of fossiliferous horizon.	

Comment: oolitic sediments form ridges parallel to coast. Oldest sediments are found inland; younger ones are located progressively closer to beach (Pícha 1978). CU-99, -109, -97, -110 and -98 relate to respective samples. Inner part of ooids showed lowest ^{14}C activity and cement between grains, highest. ^{14}C activity corresponds to general stratigraphy and position of sediments. ^{14}C ages of the Pleistocene sediments seem to be very low due to recrystallization of aragonite. Lower ^{14}C age of cement indicates that atmospheric CO_2 was involved in subaerial diagenetic process. ^{14}C age of well-preserved mollusk shells seems to be lower than their allegedly Pleistocene age (Šilar 1980). Emergence rate of shore is between one and several millimeters per year which corresponds to rate of emergence recorded for mouth of Persian Gulf and Qatar (Vita-Finzi 1979).

Cuba

Corals of the Jaimanitas Formation

Corals from Rincón de Guanabo region (23°08'N, 82°11'E) west of Havana, Cuba. Coll and subm by V Náprstek, Dept Geol, Charles Univ. Dated as part of stratigraphic study of Jaimanitas Formation (Náprstek 1978).

CU-90.	Guanabo	28,500 ± 1500
	<i>Diploria strigosa.</i>	

CU-92. Guanabo 17,800 ± 450

Acropora sp.

CU-94. Guanabo 24,000 ± 1100

Heliastrea sp.

Comment: all three samples were prepared of single block of unaltered rock from which prisms of pure carbonate of coral skeleton were cut for ^{14}C and generic determination. Coral limestones of Jaimanitas Formation should be dated according to youngest date of *Acropora* sp, 17,800 ± 450 BP, which corresponds stratigraphically to Upper Wisconsin and not to Sangamonian interglacial, as assumed previously. Geological explanation of considerable scatter of ^{14}C dates of samples is that rock-forming corals are bioclasts of various ages that were repeatedly redeposited.

Czechoslovakia

Tufa samples

Svatý Jan pod Skalou series

Tufa from Svatý Jan pod Skalou (49°59'N, 14°10'E) from Holocene stratotype profile studied by V Ložek (1967), were dated paleontologically by means of mollusks. Coll by V Ložek (1967) and J Šilar. $\delta^{13}\text{C}$ was measured at Institut für Radiohydrometrie (Rauert, pers commun, 1975). ^{14}C and paleontologic ages were correlated. ^{14}C ages were calculated assuming initial activity was 70% modern ^{14}C . This value was adopted from initial activities of karst groundwaters in Central Europe as determined by MA Geyh (1972), and has been considered equal to activity of spring water from which tufa precipitated. Spring contains modern water at present.

CU-4. Svatý Jan pod Skalou 3700 ± 190
 $\delta^{13}\text{C} = -9.8\text{‰}$

Yellowish white porous tufa from Layer 37, 0.30m above base of outcrop.

CU-5. Svatý Jan pod Skalou 3390 ± 170
 $\delta^{13}\text{C} = -9.3\text{‰}$

Yellowish white foam sinter from Layer 32, 2.80–2.95m above base of outcrop.

Comment: age of Layer 37 is Epiatlantic (Kovanda, pers commun, 1975). ^{14}C and paleontologic ages agreed assuming the ^{14}C activity was 70% modern ^{14}C .

Vyšné Ružbachy series

Tufa from Vyšné Ružbachy (49°18'N, 20°34'E).

CU-285. Fossil compact tufa 38,700 ± 6850
 $\delta^{13}\text{C} = +8.1\text{‰}$

Sample J 29 subm by V Hanzel, Geol Survey GÚDŠ, Bratislava. $\delta^{13}\text{C}$ was measured by Demovič, Hoefs and Wedepohl (1972). ^{14}C age is uncorrected.

CU-145. Modern porous tufa on green plants 40.01 ± 1.82 pMC

Coll by J Šilar. Apparent ^{14}C age is 7360 ± 150 BP, used for correcting ^{14}C age of CU-285.

Comment: Vyšné Ružbachy tufa precipitates from warm springs containing CO₂ of endogenic origin. Water circulates in confined system in Triassic limestones covered with Paleogene flysch, recharge area of which is located on slopes of High Tatra Mts 19km west of springs. We have dated tufa to Pleistocene.

CU-179. Písek **5390 ± 130**

Wood excavated from alluvial deposits of Otava River, Písek (49°19'N, 14°10'E). Subm 1976 by J Černý, Charles Univ. Sample was used as reference.

Comment: comparative dating at Institute of Nuclear Research, Debrecen, resulted in Deb-159: 5340 ± 160 (Szalay-Csongor, pers commun, 1981).

Valča series

Clayey calcium carbonate sediments (mud), foam sinter (tufa) and embedded fragments of wood from limnic Holocene sequence in valley in Carpathians at Valča (49°03'N, 18°53'E) were dated. Deposit is Holocene stratotype and was also dated using malacologic, paleobotanic and paleomagnetic methods. Samples for ¹⁴C dating were collected by V Ložek and J Šilar from deposit by erosion in concave bank of creek. Aim was to correlate ¹⁴C age determinations of wood, tufa and mud. Samples were treated in standard way (see SAMPLE PROCESSING, above). These results are arranged from top to bottom layers as described by Vaškovský and Ložek (1976). Depths are reported in meters below surface.

CU-610. Valča **6260 ± 190**

Wood from Layer 14, 6.0m.

CU-641. Valča **7300 ± 210**

Wood from Layer 14, 6.0m.

CU-605. Valča **8150 ± 220**

Foam sinter from Layer 14, 6.0m

CU-618. Valča **8180 ± 200**

Foam sinter from Layer 14, 6.0m; same sample as CU-605.

CU-630. Valča **5600 ± 180**

Wood from Layer 15, 6.20m.

$\delta^{13}C = -29.4\text{‰}$

CU-187. Valča **5790 ± 140**

Wood from Layer 15, 6.20m.

CU-606. Valča **10,390 ± 450**

Mud from Layer 15, 6.20m.

CU-639.	Valča	10,400 ± 330
Mud from Layer 15, 6.20m; same sample as CU-606.		$\delta^{13}C = -7.4\text{‰}$
CU-649.	Valča	7330 ± 190
Wood from Layer 21, 6.8m.		
CU-643.	Valča	5650 ± 180
Wood from Layer 22, 8.1m		$\delta^{13}C = -27.2\text{‰}$
CU-645.	Valča	6180 ± 190
Wood from Layer 22, 8.1m.		$\delta^{13}C = -26.8\text{‰}$
CU-647.	Valča	9710 ± 240
Mud from Layer 22, 8.1m.		
CU-629.	Valča	8830 ± 210
Mud from Layer 24, 8.55m.		
CU-644.	Valča	7798 ± 210
Wood from Layer 25, 8.55m.		$\delta^{13}C = -29.4\text{‰}$
CU-185.	Valča	7430 ± 150
Wood from Layer 25, 8.85m.		
CU-616.	Valča	10,700 ± 260
Foam sinter with sandy tufa from Layer 31, 10.20m.		
CU-637.	Valča	11,650 ± 300
Foam sinter with sandy tufa from Layer 31, 10.30m.		$\delta^{13}C = -8.3\text{‰}$
CU-617.	Valča	10,950 ± 250
Sandy tufa from Layer 32, 11.6m.		
CU-638.	Valča	11,050 ± 280
Sandy tufa from Layer 32, 11.6m.		$\delta^{13}C = -6.2\text{‰}$

Comment: carbonate sediments consist partly of clayey earth material with plastic consistency rich in calcium carbonate (mud or marl according to conventional sedimentologic terminology) and of intercalations of tufa (foam sinter and sandy tufa). In Layers 14–25, wood is abundant as scattered fragments of branches or as standing tree trunks. ^{14}C dating of wood has shown that Atlantic/Boreal border, which had been considered to be in Layer 22, according to paleontologic dating, should be shifted somewhat lower below Layer 25. Deposit originated as limnic sediments in small lake behind barrier of tufa which was later destroyed by erosion. ^{14}C dating of clayey

sediment (mud) has not proven reliable due to contamination by allochthonous clastic calcium carbonate. Similar ages of thick sequence of sediments without systematic increase with depth show very rapid sedimentation. Occurrence of carbonate sediments together with Atlantic-age wood agrees with paleontologic dating and provides evidence that these sediments originated during humid climatic period as indicated by foam sinter precipitation in mid-European karstlands (Ložek 1985).

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