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DEPARTMENT OF EARTH SCIENCES AT THE UNIVERSITY OF ROME RADIOCARBON DATES I

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INTRODUCTION

We report here, in part, dates measured on samples from Italy since the construction of a new ^{14}C lab in 1990 for research in the fields of geology, paleomorphology and the biogeochemical cycle of carbon. We use liquid scintillation counting (LSC), following sample conversion to benzene through the four classical chemical steps: 1) combustion or hydrolysis to CO_2; 2) production of Li_2C_2; 3) hydrolysis to C_2H_2; 4) trimerization of acetylene to C_6H_6 with specific catalysts. The vacuum line for benzene synthesis has been designed following a comparative review of the literature (Tamers 1960; Noakes *et al.* 1963; Scharpenseel & Pietig 1970; Harkness & Wilson 1973; Polach, Gower & Fraser 1972; Gupta & Polach 1985), and the valuable suggestions of many colleagues from well-established labs abroad. The compact vacuum line is mostly modular with "O"-ring-joined components, and is equipped with greaseless stopcocks. We use commercial two-stage rotary pumps, pressure transducers and gauges; an electrovalve coupled with a pressure control unit supplies the purified oxygen for sample combustion while holding constant, just higher than 1 atm, the gas pressure inside the combustion tube.

The dates were measured by LSC of benzene in commercial low-K, 7-ml slim-line vials, previously checked for comparable background, in a Packard 2260 XL counter. The selection of fluor(s) suitable to operate the counter with the low-level option may be critical; a secondary wave shifter fluor is needed to achieve acceptable efficiency (Noakes & Valenta 1989). We overcame initial problems by using a scintillation cocktail made up by butyl - PBD and bis - MSB (13 and 1.3 mg $\cdot$ ml^{-1} benzene, respectively). Our results agreed with the data of others (Cook, Harkness & Anderson 1989; Cook *et al.* 1990) in that the cocktail is both fairly stable and resistant against chemical quenching with high counting efficiency. For the 3-g counting geometry, efficiency is better than 72% and background averages 1.400 ± 0.021 cpm.

Samples were counted for a minimum of 2000 min in 25-min intervals, in batches containing two background and two modern samples. Quenching is monitored with the parameter, t-SIE, supplied as a part of the software package by the counter manufacturer. Data from the counter, collected on-line onto a floppy disk, are loaded to a custom-made software program for statistical and age calculation (Calderoni & Venanzi 1989).

Charcoal and wood samples were boiled with 3 N HCl followed by rinsing with water and, when tested positive for humic matter, the latter was extracted with 0.1 N NaOH. Paleosol pretreatment included hydrolysis at 100°C with 6 N HCl and, when possible, extraction with 1 N NaOH of the humic fraction of the bulk humic matter.

We report dates according to the suggestions of Stuiver and Polach (1977), *i.e.*, in years BP, based on the conventional Libby half-life for ^{14}C of 5568 years, and where necessary, corrected for isotopic fractionation ($\delta^{13}C$ values are reported as per mil deviation from the PDB standard). Oxalic Acid I and ANU Sucrose are the modern reference standards; benzene prepared from industrial coke was used as background, and to dilute smaller samples to the selected geometry. The quoted errors, ± 1 σ confidence level, include the standard deviations of count rates for unknown, background and modern samples. Also included are the known laboratory uncertainties contributed by benzene dilution and weighing and $\delta^{13}C$ measurement.

The procedures we adopted for quality assessment and control focus on β-acitivity measurement, accuracy and reproducibility throughout the dating process. We check performance of activity measurement monthly by running background, Oxalic Acid I and an internal-test solution prepared by diluting labeled benzene (400 dpm ml^{-1}, obtained from SURRC, Glasgow). Our criterion for reliable short- and long-term system performance is linear (horizontal) count rates *vs.* time. Deviations from linearity alert of system malfunction. We monitor the analytical precision and accuracy through the global dating process by running secondary standards and an internal tertiary standard three times a year, and after repair and/or modification of the benzene synthesis line. Table 1 lists the results for the reference materials.

TABLE 1. Results for Replicate Measurements of Secondary and Tertiary Standards

Material	Percent of Modern (pMC)
Sucrose ANU	150.91 ± 1.04; 149.92 ± 1.00; 150.55 ± 1.02; 149.38 ± 1.06; 150.13 ± 1.05
Cellulose IAEA	130.96 ± 1.03; 129.85 ± 1.08; 130.68 ± 1.05;
Commercial charcoal*	99.02 ± 0.79; 99.74 ± 0.81; 98.47 ± 0.85

*Commercial, pre-bomb (1948–1950) charcoal is used in Rome as the "in house" tertiary standard since 1964 (Alessio, Bella & Cortesi 1984).

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GEOLOGICAL SAMPLES

Mt. Calvario series

Samples are from a two-paleosol (PAs) suite of Late Quaternary age, overlying Cretaceous micritic limestones, interbedded with polygenetic, tectonic breccia, and underlying a gravel unit. The site was exposed during quarrying on the south slope of Mt. Calvario (1743 m asl), Sangro River valley, central Apennines, 40 km east of Aquila, Abruzzi region (42°52′13″N, 14°03′16″E). The PAs record two phases of slope stability, both long enough for pedogenesis. After its formation, the lowermost PA experienced many neotectonic events, the last one being responsible for its burial. This is documented by an overall subvertical fault, measuring over 1 m of downthrow. The date for the uppermost, undisturbed PA provides a minimum age for a significant paleoseismological event that occurred during the Holocene (Calderoni *et al.* 1990). The PAs samples were collected and submitted August 1990 by G. Calderoni and L. Serva (ENEA-DISP, Rome).

38,500 ± 2000

Rome-125. Mt. Calvario 1 $\delta^{13}C = -27.3‰$

Humic acids and humin fractions from sandy silt (2 mm–2 μ) of the lowermost PA, *ca.* 50 cm thick, outcropping 10 m above the quarry ground level.

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Rome-126. Mt. Calvario 1 **39,800 ± 2200**
$\delta^{13}C = -27.0‰$

Humic acids and humin fractions from silty clay (<2 μ) of the lowermost PA. Concordance with Rome-125, indicating that the apparent age of humic matter is unaffected by the PA particle size, could also mean that primitive organic carbon redistributed during pedogenesis.

Rome-127. Mt. Calvario 2 **4650 ± 75**
$\delta^{13}C = -27.5‰$

Humic acids and humin fractions from the bulk of the uppermost PA, 30 cm thick, are probably likely a relict of B horizons of an andosol, exposed 12 m above the quarry ground level.

General Comment: The PA samples were previously hydrolyzed for 4 h at 100°C with 18% HCl for complete removal of both carbonate bedrock fragments and fulvic acids. Ages are consistent with the stratigraphy. The organic carbon content (1.72, 2.05 and 4.27%: Rome-125, -126, -127, respectively) decreases antithetically with age and particle size. By contrast, the C/N ratio for all the humic matter in the PAs is almost constant (mean = 8.3 ± 1.0), suggesting common pathways of pedogenesis and diagenesis.

Mounts of the Laga Chain series

The southern flank of the Laga Mountain chain in the central Apennines was selected for neotectonic and paleoseismological study because of historical accounts of seismic events and evidence of active tectonics. Dislocation features, *e.g.*, dissected alluvial terraces of the Tronto River and late Würmian fans of Campotosto, are particularly apparent at the piedmont belt of the chain (Bachetti *et al.* 1990). Recent road construction at the eastern edge of Piano di Montereale, Aterno River valley, Aquila, central Apennines (42°34′19″N, 13°22′35″E) exposed a sedimentary sequence. Here, a fluviatile, swampy layer yielded brown wood fragments, partly replaced by pyrite. Samples were collected and submitted 1989 by A. M. Blumetti, Department of Earth Sciences, University of Camerino.

Rome-128. Mounts of the Laga **39,500 ± 2700**

Heavily pyritized small wood fragments.

Comment: According to field data, the dated level rests on a small depression formed during the tilting of a section of the piedmont belt. Wood pyritization probably occurred during severe anoxic conditions that developed in the former basin of sedimentation. This resulted from significant, tectonically induced geomorphological changes that affected the drainage basin.

Middle Cesano River Basin series

Five wood samples from terraced alluvial sediments exposed in the middle valley of the Cesano River were dated as part of continuing research to establish a time scale for geomorphological variations of the central Apennines during the Late Quaternary. This reach of the Apennines has a unique feature, in that mountainous chains and basins are northwest-southeast-directed (Apennine direction), whereas most drainage systems flow northeastwards, probably owing to anti-Apennine trending faults that represent a preferential path of incision. Following tectonic displacements, the Cesano River eroded and accumulated sediments from the resulting catchment area. This was recorded by widespread fluvial terrace deposits along the main valley. Four groups of terraces have been defined according to elevation from the valley floor (Lipparini 1938; Selli 1954; Nesci &

Savelli 1990), from the older T1 (top) to the younger T4 (base). Preliminary dating of samples from nearby sites revealed that 4th- and, in part, 3rd-order terraces formed from the Late Pleistocene through the Holocene, although changes in individual streams effect significant variations in the terraces (Alessio *et al.* 1987). Samples originated from the walls of two clay quarries at St. Michele al Fiume (12°59′43″E, 43°39′37″N) and St. Lorenzo in Campo (12°56′43′E, 43°36′06″N), located in the Marchean Apennines close to the north bank of the middle Cesano River, at *ca.* 15 and 25 km, respectively, upstream from the sea coast. Collected and submitted 1990 by O. Nesci and D. Savelli, Institute of Geology, University of Urbino.

Rome-108. St. Michele al Fiume SN#1 **35,600 ± 1800**

Well-preserved dark wood from gray-blue, clayey-silty sand at the base of the section, underlying braided-stream sediments and overlying the Pliocene-age clayey-marl base.

Rome-109. St. Michele al Fiume SN#2 **37,300 ± 2000**

Rome-110. St. Michele al Fiume SN#4 **32,500 ± 1200**

Well-preserved brown wood samples from a fine-grained layer, underlying braided-stream sediments with festoon-like cross-stratification.

Rome-111. St. Lorenzo in Campo SN#5 **31,700 ± 1000**

Rome-112. St. Lorenzo in Campo SN#6 **37,300 ± 2200**

This section exposes the middle-to-upper part of 3rd-order terrace alluvium. Well-preserved, dark wood samples from small clayey-silty-sandy lenses contained in the lowermost pebbly layer.

General Comment: Samples from both sites are associated with a late phase of 3rd-order terrace fluvial deposits. Features include strip-shape, overall southwest-northeast trending and a thickness averaging 30 m. At present, the terraced suite outcrops from 5 to *ca.* 15 m above the talweg of the Cesano River Valley. Although the samples were partly *in-situ* trees and partly flooded tree branches, the ages indicate that the two dated sediment PAs are almost syngenetic and formed during the Würmian late glacial stage, *viz*, Pre-Bølling-Mstno.

Esino River valley series

This series contains the first dates for 3rd-order alluvial terraces of the middle Esino River valley, Matelica, central Apennine chain. Alluvial sediments, deposited here during Esino River floods, overlie the Miocene sandstone base of the Camerino basin, and their relationships with both 2nd- and 4th-order terraced sediments are recognizable. Samples originated from five exposures close to the river banks, and are representative of a significant reach of the valley. Samples were collected and submitted 1990 by M. Coltorti, Department of Earth Sciences, University of Camerino.

Rome-139. Esino River valley, Section 22 **31,800 ± 1100**

Scattered fragments of well-preserved charcoal from a sandy-silty layer at Casa Falceto IV (43°16′45″N, 12°59′57″E).

Rome-140. Esino River valley, Section 20 **41,000 ± 4000**

A dark macrofragment of wood from a clayey layer.

Rome-141. Esino River valley, Section 20 **>41,000**

Fragments of wood from a sandy-silty layer, 1 m below Rome-140, from Casa Felceto I (43°16′37″N, 12°59′46″E).

Rome-142. Esino River valley, Section 23 **30,200 ± 900**

Fragments of tree roots and peat from a sandy layer at Fosso Pagliano I (43°17′00″N, 12°59′39″E).

Rome-143. Esino River valley, Section 30 **32,700 ± 1200**

A macrofragment of dark, well-preserved wood from the lowermost clayey level at Casa Incrocca II (43°17′51″N, 12°59′08″E).

Rome-144. Esino River valley, Section 39 **23,500 ± 400**

Small charcoal fragments scattered through a lens.

Rome-145. Esino River valley, Section 39 **32,500 ± 1200**

Wood fragments 1.5 m below Rome-144 from the basal clayey layer at Case Pezza-Ansuini (43°19′31″N, 12°59′36″E).

General Comment: The dates indicate that 3rd-order terraces formed well before the Holocene in the Esino River valley and the northernmost valleys of the central Apennines, so far investigated. The origin of the terraces, probably triggered by dramatic neotectonic events and/or climatic changes, involved recurrent cycles. The beginning of such a catastrophic phase, however, is beyond the limits of ^{14}C dating.

Borgiano Deposit series

A sedimentary sequence consisting of intercalations of both slope-waste deposits and alluvial fan sediments was exposed during road construction near Borgiano Lake, the middle Chienti River valley, Marchean Apennines, central Italy (43°07′50″N, 13°12′00″E). The area is very interesting, in that recurrent neotectonic events badly fractured the carbonate bedrock, thus increasing both erosion and landslides. The deposit (Calderoni *et al.* 1991) is on 3rd-order terraced sediments (see Middle Cesano River valley series, above) and shows stratigraphic relationships with 4th-order Holocene alluvium. The sampled section shows numerous horizons of Rendzinic soil, intercalated with slope-waste debris and alluvial fan sediments at the contact with the basal slope and in the outer part of the deposit, respectively. Following stratigraphic analysis, the paleosol horizons were assigned to two suites, upper and lower, separated by significant lateral variations. The samples were collected and submitted 1989 by M. Coltorti, G. Pambianchi and F. Dramis, Department of Earth Sciences, University of Camerino.

Rome-129. Borgiano Deposit 1 **1620 ± 60**

Humic acids from the topmost humified layer of the upper paleosol suite. $\delta^{13}C = -28.1‰$

Rome-130. Borgiano Deposit 2 **1700 ± 65**
$\delta^{13}C = -27.5‰$

Humic acids from the topmost humified layer of the upper paleosol suite, 50 m apart from Rome-129.

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2270 ± 70
Rome-131. Borgiano Deposit 3 $\delta^{13}C = -25.5‰$

Fragments of well-preserved charcoal, associated with pottery, from a fireplace found in a shelter filled by gravels, underlying Rome-130.

4020 ± 70
Rome-132. Borgiano Deposit 4 $\delta^{13}C = -27.2‰$

Humic acids from the humified layer at the bottom of the upper paleosol suite.

Rome-133. Borgiano Deposit 5 **5870 ± 70**

Humic acids from the humified layer overlying the lower paleosol suite. $\delta^{13}C = -27.8‰$

8100 ± 80
Rome-134. Borgiano Deposit 6 $\delta^{13}C = -27.3‰$

Humic acids from the humified layer at the bottom of the lower paleosol suite, close to the base of the section.

General Comment: Dates on humic acids yield minimum ages for significant phases of slope stability (lasting long enough for pedogenesis) throughout most of the Holocene, including the arrival of humans to the area.

Orbetello Lagoon series

As part of research on the origin, fate and distribution of organic matter input in a lagoonal environment, four samples of basal sediments from Orbetello Lagoon (central Italy, Thyrrenian coast, Tuscany) were collected. The lagoon is shallow (1.5 m–30 cm), 27 km^2 wide and overlies part of a Miocene tectonic depression bordered seawards by sand banks; the two narrow channels communicating with the sea do not allow enough water turnover for preventing eutrophication. The study area was selected for little pollution, lack of significant continental input from streams and the existence of comprehensive geological, morphological and environmental records (Lazzarotto, Mazzanti & Mazzoncini 1964; Bartolini *et al.* 1977; Brambati *et al.* 1979). The samples were collected and submitted 1990 by M. Angeloni and R. Gragnani, ENEA-Casaccia, Rome.

$\Delta^{14}C = -95.91 \pm 7.63‰$
Rome-135. Orbetello 1 $\delta^{13}C = -18.3‰$

HCl-refractory organic matter from Station C_3 (42°26′08″N, 11°12′08″E), facing the village of Orbetello, *ca.* 3 km from land.

$\Delta^{14}C = -90.63 \pm 7.72‰$
Rome-136. Orbetello 2 $\delta^{13}C = -19.2‰$

HCl-refractory organic matter from Station C_4 (42°25′40″N, 11°16′26″E), at the southern lagoon apex, *ca.* 500 m from land.

$\Delta^{14}C = +64.83 \pm 8.52‰$
Rome-137. Orbetello 3 $\delta^{13}C = -18.5‰$

HCl-refractory organic matter from Station C_5 (42°26′49″N, 11°14′24″E), at the middle of the lagoon, *ca.* 500 m from land.

Rome-138. Orbetello 4 **$\Delta^{14}C = -51.27 \pm 7.81‰$**
$\delta^{13}C = -18.6‰$

HCl-refractory organic matter from Station C_6 (42°25′40″N, 11°11′55″E), at the western lagoon edge, *ca.* 5 km from land.

General Comment: We ran measurements on the fulvic acids- and proteinaceous-compound-free organic matter escaped to the intensive acidic hydrolysis performed for removing both carbonates and the most geochemically mobile organic compounds. The negative $\Delta^{14}C$ values for Rome-135, -136 and -138 reveal a significant mean residence time for the surficial, most stable organic matter, hardly predictable with the available data on enhanced lagoon eutrophication. Sample R-137 yields a post-bomb $\Delta^{14}C$ level approaching, to some extent, that of atmospheric $^{14}CO_2$ at the sampling year, thus suggesting a strikingly fast production through local biological pathways. This involves a marked drop in the $\delta^{13}C$ value from *ca.* −8‰ (mean for atmospheric CO_2) to the measured value, −18.5‰. Values for $^{13}C/^{12}C$ ratio support a prevailing marine origin for organic input; however, a secondary contribution from continental higher plants is suggested by the distribution pattern of *n* - alkanes in the dated organic matter (Monti 1991).

Lago Lungo series

Lago Lungo, *ca.* 7 km north of Rieti, Latium, central Italy (42°28′12″N, 12°40′17″E), presently a small pond, was an active sedimentation basin for alluvium from its catchment area during the Late Quaternary. In 1989, the sedimentary sequence was drilled to 81.50 m depth to study climatic change through pollen analysis and to establish a time scale for the Pleistocene-Holocene transition. The samples were collected and submitted 1990 by R. Pezzarossa, SIAGI Spa, Rome.

Rome-146. Lago Lungo 1 **8050 ± 100**

Small fragments of dark wood, 26.30–26.40 m deep in core. *$\delta^{13}C = -24.2‰$*

Rome-147. Lago Lungo 2 **>41,000**

Fragments of well-preserved brown wood, 36.10–36.20 m deep in core. *$\delta^{13}C = -23.7‰$*

General Comment: The dates were measured for preliminary evaluation of age and sedimentation rate. While sedimentological, geochemical and pollen analyses are underway, the age of Rome-146 is a significant chronostratigraphic constraint on the beginning of the Holocene.

Aeolian Archipelago series

The Aeolian Archipelago, consisting of seven islands, lies northwest of the northeast apex of Sicily (southern Tyrrhenian Sea), and is an important active volcanic arc in the Mediterranean Sea (Barberi *et al.* 1974; Keller 1979). The samples were dated as part of an ongoing study of tephrochronology for the recurrent explosive eruptions from the volcanic districts in central and southern Italy through the Late Quaternary.

Rome-148. Filicudi Island **>41,000**

Well-preserved bits of charcoal from a *ca.* 15-m-thick tephra sequence, containing the last pyroclastic deposits that blanketed the island, exposed close to the harbor of Filicudi Island (38°33′28″N, 14°34′49″E). Samples were contained in a layer of muddy, yellowish ash flow, in about the middle of the suite, underlying several ash and pumiceous layers. Their age suggests volcanic activity far older than that responsible for the "brown tuffs" spread throughout most of the Aeolian Islands (Calderoni, La Volpe and Lo Sito 1991). The samples were collected and submitted 1990 by A. Manetti, Department of Earth Sciences, University of Firenze.

Rome-149. Volcano Island **7680 ± 100**

Scattered charcoal fragments from a tephra layer exposed at Volcano Piano (38°23′10″N, 14°58′35″E). The date is important because it may reveal that "brown tuff" emplacement (Calderoni, La Volpe and Lo Sito 1991) lasted longer than previously believed. The samples were collected and submitted 1989 by R. Lo Sito, Department Geomineralogico, University of Bari.

Miscellaneous samples

Rome-150. Sorrento **>41,000**

Peaty, dark clayey sediment containing almost completely humified wood fragments, 14.70–14.90 m deep from a continuous core drilled 4 km west of Sorrento, Campania, southern Italy (40°37′32″N, 14°21′30″E). The sample underlay weathered tephra and lava fragments that can be firmly attributed to known phases of nearby volcanic areas. The sample was collected 1991 by S. Palomba, Centro Geologica Tecnica, Sorrento and submitted by D. Stanzione, Department of Earth Sciences, University of Napoli.

Rome-151. La Piantata **3000 ± 70**

Small, well-preserved fragments of tree branches partly charred at 12 m depth in a core from continuous drilling of a Middle Pleistocene lacustrine deposit at La Piantata, Urbino, central Apennines (43°44′22″N, 12°37′42″E). The young age reflects some recent reworking of the sediment suite. The sample was collected and submitted 1991 by F. Veneri, Institute of Mineralogy and Petrology, University of Urbino.

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