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EXPERIMENTAL DETERMINATION OF THE ^{14}C INITIAL ACTIVITY OF CALCAREOUS DEPOSITS

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ABSTRACT. We have reconstructed the initial activity of calcareous sediments by 1) measuring the ^{14}C activity of recent pre-bomb-test calcareous deposits, 2) calculating the ratio of the ^{14}C activity of the calcareous sediment to that of the adjacent coeval wood, and 3) extrapolating ^{14}C activity of the deep lacustrine sediment layers to the surface, assuming a constant sedimentation rate. We show here that the uppermost sediment is affected by the global increase of atmospheric ^{14}C activity due to thermonuclear bomb tests in the early 1960s. ^{14}C activity of submerged aquatic plants showed values similar to ^{14}C activity of DIC. Thus, organic residue of the sediment cannot be used for reliable ^{14}C dating because of the unknown fraction of aquatic plant detritus. The initial activity, A_0, obtained in a case study of the geochronology of carbonates in the Plitvice Lakes area, ranged from 70.5 to 72.2 pMC for Lake Prošće sediment, and from 75.9 to 76.7 pMC for Lake Kozjak sediment. These values also agree with calculated values based on the downstream increase of ^{14}C activity of DIC in freshwater open to the atmosphere.

INTRODUCTION

Chemical and isotopic composition of dissolved inorganic carbon (DIC) reflects geochemical processes of groundwater formation. The $\delta^{13}C$ value close to zero points to marine limestone as the main source of carbon, whereas a negative $\delta^{13}C$ value indicates carbon of biogenic origin. A high ^{14}C activity of DIC is typical of recent groundwater having a very short mean residence time. The initial activity, A_0, is defined as the ^{14}C activity of carbonate sediment at the moment of precipitation, and represents the percentage of the carbon of biogenic and/or atmospheric origin in the total DIC. A_0 is of primary interest in calculating the ^{14}C age of calcareous deposits. In an early study of groundwater dating, Geyh (1973) ranged A_0 values according to the geology of the catchment area. Other studies of the initial activity of calcareous deposits (Thorpe, Otlet & Sweeting 1980; Srdoč *et al.* 1983, 1986a; Pazdur 1988; Pazdur, Pazdur & Szulc 1988; Horvatinčić *et al.* 1989; Pentecost *et al.* 1990) showed that the A_0 depends on geological setting, geochemical processes, vegetation and climate, which are, in principle, site-specific. A temporal change of A_0 is not very likely over a period of stable environmental conditions, as demonstrated by our data for Holocene samples. The A_0 value remained constant throughout a 12-m-long sediment core spanning ≈8 ka (Srdoč *et al.* 1986b). Pazdur *et al.* (1988) documented the dependence of A_0 on the environmental conditions of tufa deposition. They obtained lower A_0 values in fast-flowing turbulent streams. Moreover, measurements of ^{14}C activity of DIC in springs and in stream water showed a systematic increase of ^{14}C activity and $\delta^{13}C$ along the water course (Thorpe, Otlet & Sweeting 1980; Srdoč *et al.* 1986a; Pentecost *et al.* 1990) due to exchange between atmospheric and dissolved CO_2.

Such complex and site-specific physico-chemical mechanisms involved in the DIC formation and carbonate precipitation lead to serious difficulties and erroneous results in the application of theoretical models (for a review, see Fontes 1983), as it was shown by comparing the measured and the calculated groundwater activities (Krajcar Bronić *et al.* 1986).

Under isotopic equilibrium conditions, the isotope enrichment factor, ε_{bc}, between bicarbonates dissolved in water and precipitated carbonate, $\varepsilon_{bc} \approx \delta_b - \delta_c$, is probably <0.5‰ (Fontes 1983; Dulinski & Rozanski 1990), where δ_b and δ_c are the $\delta^{13}C$ values for bicarbonates and solid carbonate, respectively. Thus, the difference in ^{14}C activity of DIC, which consists mostly of bicarbonates, and that of carbonate precipitated from freshwater, is <0.1% modern carbon (pMC). Hence,

the simplest method for determining A_0 would be to measure recent ^{14}C activity, either of DIC or of naturally precipitated calcium carbonate, if ground and surface waters had not become contaminated with bomb ^{14}C. Consequently, the present ^{14}C activity of dissolved bicarbonates in water cannot be used to determine the A_0 of carbonate that precipitated hundreds or thousands of years ago.

We describe below three independent experimental methods for determining A_0 based on measured ^{14}C data, and discuss their limitations. We applied these methods in case studies dealing with the geochronology of calcareous deposits in several karst regions of the Dinaric Alps, Croatia. Most of the research was done at the Plitvice Lakes area, where a comprehensive set of ^{14}C dates was obtained and published in our date lists (Srdoč *et al.* 1987, 1989). Figure 1A shows the Plitvice Lakes profile.

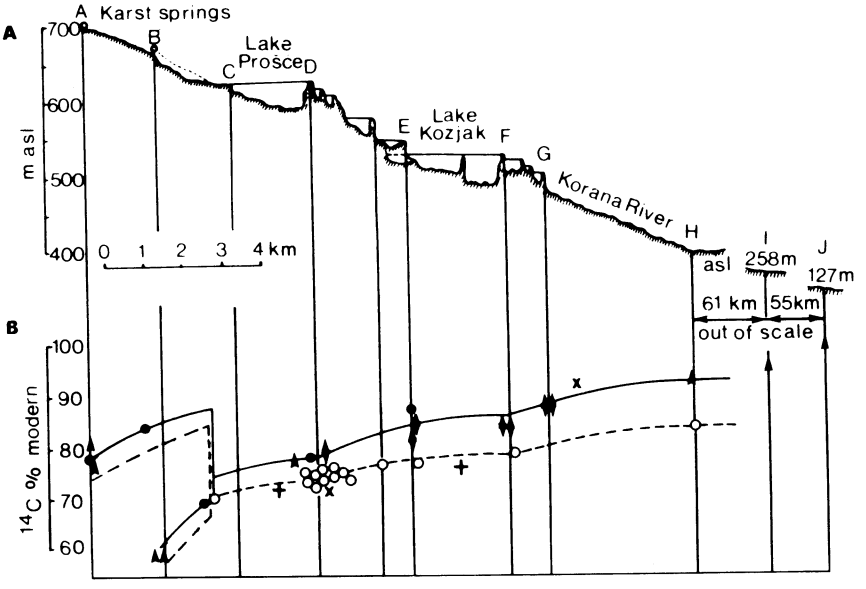

Fig. 1. A. Plitvice Lakes profile, Plitvice National Park, Croatia. B. ^{14}C activity of bomb-contaminated recent tufa (•), DIC in water (▲) and carbonate deposited on polyamide mats (♦). ^{14}C activity of tufa (○) and aquatic plants (x) collected before 1945, and the uncontaminated top layer of lake sediment (+). Calculated DIC ^{14}C activity according to the Srdoč *et al.* (1986a) model: — for recent (1980–1983) atmospheric ^{14}C activity; - - - before bomb tests.

MEASUREMENT OF ^{14}C ACTIVITY OF RECENT CARBONATES

Measurements of ^{14}C activity of DIC and recent tufa deposited on polyamide mats showed a systematic increase of ^{14}C activity from karst springs to the Korana River mouth (Fig. 1B). The increase of ^{14}C activity and the corresponding changes in $\delta^{13}C$ (Table 1) were attributed to the exchange of carbon isotopes between atmospheric CO_2 and DIC, and absorption of CO_2 developed by root respiration and decay of terrestrial plants. Srdoč *et al.* (1986a) developed a model based on measured ^{14}C and ^{13}C concentrations of DIC, atmospheric CO_2 and terrestrial plants, describing changes in carbon isotope composition of DIC along the water course. By replacing bomb-contaminated values of DIC with pre-bomb atmospheric activity of 100 pMC, it was possible to calculate the A_0 for calcareous sediments at any location along the Korana River. Table 1 shows the A_0 values according to this model, which will be compared with A_0 obtained by the methods described below.

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TABLE 1. ^{14}C activity of bomb-contaminated DIC and recent calcareous deposits, A(t), in the Korana River catchment area. Sampling sites are sorted in a downstream direction. $A_{atm}(t)$ is the atmospheric ^{14}C activity for the year of sampling. The A_0 values for the pre-bomb-test era were calculated by Srdoč *et al.* (1986a). The $\delta^{13}C$ value of DIC is the mean value of several measurements at the particular location.

Sample no.	Sampling site	Year	A(t) (pMC)	$A_{atm}(t)$ (pMC)	A/A_{atm} (pMC)	A_0	$\delta^{13}C$ (‰PDB)
Z-702	Crna Rijeka stream	1979	69.1	130	53.1	66	−12.5
Z-754	Bijela Rijeka stream	1980	85.3	128	66.6	75	−12.6
Z-1059	Confluence site	1982	71.5	124	57.7	70	−11.5
Z-1416	Lake Prošće	1984	77.9	122	63.8	72	−11.1
Z-1029	Lake Ciginovac	1982	73.2	124	59.0	74	−10.1
Z-1031	Špiljski garden	1982	77.5	124	62.5	74	
Z-1068	Rječica	1982	89.0	124	71.8	76	
Z-980	Burget	1981	85.4	126	67.7	76	−10.0
Z-1011	Burget	1982	83.8	124	67.6	76	−10.0
Z-1067	Plitvica stream	1982	84.7	124	68.3	77	−12.7
Z-1012	Plitvica waterfall	1982	87.9	124	70.9	77	−11.7
Z-1276	Novakovića brod	1984	88.2	122	72.2	80	−9.4
Z-1065	Korana R. headwater	1982	89.5	124	68.4	80	
Z-659	Korana R. headwater	1979	90.0	130	69.1	81	
Z-658	Korana waterfall	1979	91.3	130	70.2	82	
Z-704	Korana bridge	1979	90.9	130	69.8	83	−9.5
Z-1019	Korana Drežnik	1982	92.4	124	75.5	83	
Z-1063	Korana Tušilović	1982	90.8	124	73.2	87	−11.4

We attempted to correlate recent, bomb-contaminated DIC ^{14}C activity with mean atmospheric CO_2 activity of the same year, when the DIC sample was collected. In a rapid turnover in the carbon cycle, which includes carbon uptake by terrestrial plants from atmospheric CO_2 *via* photosynthesis, followed by biodegradation of plant detritus and DIC formation, the ratio $A(t)/A_{atm}(t) \times 100$ (Table 1) should approach A_0, as measured before the bomb-test era (Srdoč *et al.* 1986a). Table 1 also presents the mean atmospheric ^{14}C activity for each year, determined as the mean value of ^{14}C activity of atmospheric CO_2, annual plants and leaves, and tree rings (Obelić *et al.* 1986, and our recent unpublished data). These data agree with the "clean air" ^{14}C activity of the lower troposphere of the northern hemisphere (Levin *et al.* 1989). It is evident that the $A(t)/A_{atm}(t) \times 100$ values increase downstream, but they are approximately 10 pMC lower than the A_0 values determined by applying model of downstream increase of ^{14}C activity (Fig. 1B). Increased atmospheric ^{14}C activity has a significant influence on the increase of ^{14}C activity of DIC in surface water along the water course. On the contrary, the response of ^{14}C activity of DIC at springs to changes in atmospheric ^{14}C activity is delayed and damped (Srdoč 1986), and obliterated by large seasonal variations. This can be explained by a gradual decay of refractory fraction of organic matter in topsoil (Scharpenseel and Becker-Heidmann 1989) resulting in CO_2 having lower ^{14}C activity than the contaminated atmospheric CO_2.

^{14}C ACTIVITY RATIO OF CARBONATE AND ORGANIC MATTER

Organic Matter of Terrestrial Origin

Measurement of ^{14}C activity of terrestrial plants (driftwood, leaves) embedded in sediment helps to determine the A_0 of the matrix material. Assuming that the organic matter found in the carbonate

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sediment is of terrestrial origin and coeval with the surrounding sediment, the initial ^{14}C activity is calculated as the ratio of the ^{14}C activity of sediment and organic matter, both expressed in pMC. We emphasize that the terrestrial origin of the organic matter has to be checked by $\delta^{13}C$ measurements.

Wood fragments were found embedded in sediments in both Kozjak and Prošće Lakes. Adjacent sediment was carefully collected and ^{14}C activity of both samples was measured (Srdoč *et al.* 1986b). The calculated A_0 thus obtained was 72.2 pMC and 75.9 pMC for Prošće and Kozjak Lakes, respectively (see Table 4).

Layers of organic detritus embedded in tufa were found near Hajduković in a pit formed by karstification of a calcareous river terrace in Plitvice National Park (Fig. 2). In the pit, 3–4 m deep, 5–6 m in diameter, thick layers of tufa of various textures are intercalated with dark layers of charred organic detritus with the remains of partially carbonized leaves (Fig. 2). At the bottom of the pit, we found partially rotted hollow tree trunks, 25–30 cm in diameter. We made ^{14}C analyses of tufa, organic detritus and wood, and present the calculated initial ^{14}C activities in Figure 2. The A_0 values ranged from 77.0 to 77.8 pMC, in accordance with A_0 calculated from contemporary ^{14}C activity of Plitvica stream (77 pMC, see Table 1), except that of 71.6 obtained as the ^{14}C activity ratio of tufa inside the hollow trunk and wood. A plausible explanation is that the most reliable cases are those where the calcareous coating adheres to the coeval organic substratum. Filling a hole in a tree trunk does not necessarily imply the contemporaneity of the materials in question.

In waters supersaturated with carbonates, all submerged surfaces are covered by a calcareous crust in about 1 or 2 years. The ^{14}C activity of calcareous coating is practically the same as that of DIC (Fig. 1B). During our research in the Plitvice National Park, we discovered several examples of pre-bomb-test wood coated with tufa. Table 2 presents the results of these ^{14}C activity measurements. These A_0 values are similar to those obtained by Srdoč *et al.* (1986a) at the same locations. Several samples (such as Z-847 and Z-848) showed high ^{14}C activity of calcareous coatings com-

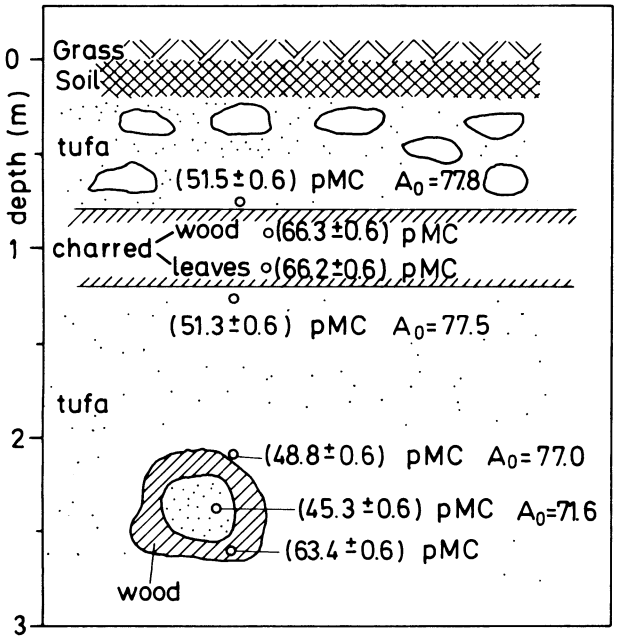

Fig. 2. Schematic diagram of the Hajduković pit, Plitvice National Park. The ^{14}C activity of calcareous deposits and organic detritus, as well as the calculated A_0 values, are shown at sampling points.

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pared to known ^{14}C activity of pre-bomb tufa, and the calculated A_0 is too high. We concluded that bomb-^{14}C-contaminated calcareous crust coated the uncontaminated wood. These examples show that the described method should be applied cautiously when either inorganic or organic matter is contaminated with bomb-produced ^{14}C.

Encrusted wood branches were retrieved from the "Perinka ponor" swallow hole in Lika, central Croatia (Srdoč *et al.* 1989). Pre-bomb-test calcareous deposits on wood enabled us to determine that A_0 = 68.5 pMC for calcareous deposits in the Gacka River catchment area (Z-1907 to -1910, Table 2).

A watermill on the Krka River in the low-land flow, North Dalmatia presented an interesting finding. The mill has been inactive since 1908, when the Krka River was diverted into a hydroelectric plant. Calcareous deposits on the wooden parts of the mill yielded an A_0 value of 75.8 pMC (Table 2).

TABLE 2. A_0 values based on the ratio of ^{14}C activity of calcareous coating and adjacent organic matter of terrestrial origin

Sample no.	Sample description	^{14}C activity (pMC)	A_0 (pMC)
Plitvice Lakes area			
Z-853	Plant stalks	93.3 ± 0.8	
Z-855	Tufa	68.9 ± 0.6	74.1
Z-856	Tufa	69.6 ± 0.8	74.9
Z-1307	Wood covered with tufa	97.2 ± 0.7	74.3
Z-1306	Tufa	72.2 ± 0.6	
Z-857	Moss adjacent to tufa	72.8 ± 0.6	72.8
Z-847	Wood coated with tufa	98.1 ± 0.6	90.2
Z-848	Tufa coating on wood	88.5 ± 0.7	
"Perinka ponor" swallow-hole, Gacka River			
Z-1907	Calcareous deposits	67.7 ± 1.1	69.0
Z-1908	Wooden beam	97.8 ± 1.4	
Z-1909	Tufa coating on twig	68.7 ± 1.1	
Z-1910	Twig	101.4 ± 1.4	68.0
Krka River catchment area, North Dalmatia			
Z-2323	Calcareous coating on wood from watermill (*ca.* 1908)	75.8 ± 0.8	75.8

Pazdur, Pazdur and Szulc (1988) also used this method comparing the ^{14}C age of the tufa layer with ^{14}C ages of over- and underlying peat layers, and obtained an A_0 of (77 ± 1) pMC for the Trzebienice site in Poland.

Dating the Organic Component of Sediment

The ^{14}C activity ratio of carbonates and organic residue gives the A_0, if the organic matter in carbonate is of terrestrial origin, and if it is coeval with the sediment. We made such an analysis on lake sediments from the Plitvice National Park. Table 3 presents the results. The calculated A_0

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values are higher than that obtained by other methods described in this paper for the same sediment, and are generally too high for the investigated area of Plitvice Lakes (see Fig. 1B).

Marčenko *et al.* (1989) showed that submerged aquatic plants take CO_2 from water for their photosynthesis. As a result, the $\delta^{13}C$ values of aquatic plants are significantly lower (from −32 to −48‰) than that of terrestrial plants (−28‰ on the average in the Park area), and their ^{14}C activity is close to that of DIC. Some plants use both the atmospheric and dissolved CO_2 simultaneously. Thus, these plants are not suitable for ^{14}C dating, unless the initial activity of incorporated carbon is known. Further, fossil remains of such plants buried in lake sediments show an erroneous ^{14}C age of the sediments. ^{13}C measurements of the organic fraction of sediments, presented in Table 3, show that organic matter from lake sediments consists of a mixture of terrestrial and aquatic plants. Consequently, the use of ^{14}C activity of organics in lake sediments may introduce an error in calculating the A_0 value if the organic residue contains carbon derived from aquatic plants.

TABLE 3. The ratio of ^{14}C activity of sediment and organic residue

Lake and sediment depth (m)	^{14}C activity Sediment (pMC)	Org. res. (pMC)	A_{sed}/A_{org} (pMC)	A_0 mean* (pMC)	$\delta^{13}C_{org}$ (‰PDB)
Lake Prošće					
0.3	67.0	86.2	77.7		−30.5
4.3	52.0	65.5	79.4		
8.3	38.4	44.8	85.6		
10.3	34.5	37.8	91.2		
				72.0	
Lake Kozjak					
0.03	89.5	111.3	80.0		−33.6
0.07	75.4	91.6	82.5		−29.9
0.2	73.5	82.1	89.5		−30.2
6.7	57.9	65.0	89.1		
8.8	48.6	56.1	86.7		
				76.0	

*Mean A_0 value from Table 4

However, recent true aquatic plants collected before the bomb-test period can be used for A_0 determination. The ^{14}C activity of moss (*Cratoneurum commutatum*) (Z-857, Table 2) collected at Plitvice Lakes around 1920 and stored in the Zagreb Botanical Garden Museum, agrees very well with A_0 calculated by using other methods described in this paper.

Pazdur, Pazdur & Szulc (1988) estimated the apparent age, equivalent to A_0, of tufa from several sites in Poland by direct comparison of ^{14}C dates of carbonates and organic fractions. This was checked by paleobotanical analyses and by measurement of $\delta^{13}C$ content for terrestrial origin. The authors obtained A_0 values ranging from 61 pMC to 89 pMC, depending on lithologically different forms of tufa or calcareous sediment.

A_0 OBTAINED FROM LAKE MARL ^{14}C ACTIVITY BY THE EXTRAPOLATION METHOD

The ^{14}C activity of a depth profile, expressed in pMC, plotted *vs.* sediment depth, gives a straight line, provided the sedimentation rate is uniform. The slope of the regression line gives the sedimentation rate. The intercept, *i.e.*, extrapolation to the sediment surface, gives the initial

activity. The method is applicable to steady conditions of carbonate precipitation and a constant sedimentation rate throughout the sediment depth or over a fairly long section (*e.g.*, 2–3 m) of the profile. Such extrapolation is not applicable for turbulent sedimentation and slumping, *e.g.*, in the case of the Lake Ontario sediment profile (Buchardt & Fritz 1980).

Care must be taken of the increase of natural ^{14}C activity due to thermonuclear bomb tests after World War II in the uppermost layer of the sediment. The thickness of the layer contaminated with bomb ^{14}C depends on the sedimentation rate. Isotope and various other analyses of the very young sediments provide, however, valuable environmental information and reflect the magnitude of modern pollution.

Several shallow sediment cores were retrieved from Lake Kozjak (see Fig. 1A) to study anthropogenic influences on lakes and lake sediments, as described by Srdoč *et al.* (1992). The uppermost 9 cm of the core showed a non-linear increase of ^{14}C activity compared to layers immediately below 9 cm. The layer free of bomb ^{14}C, 9–13 cm deep, gave an A_0 of 76.7 pMC, which agrees well with A_0 values obtained by the other methods used (Table 4).

TABLE 4. Initial ^{14}C activity of lake sediments obtained by different methods

Method	A_0 (pMC) Lake Prošće	Lake Kozjak
Ratio of ^{14}C activity of sediment and of wood embedded in sediment	72.2	75.9
Extrapolation to pre-bomb surface ^{14}C activity of lake sediment	71.1*	76.1**
Uppermost layer of sediment free of bomb ^{14}C	70.5	76.7
Calculated A_0 (Srdoč *et al.* 1986a)	72.4	76.0

*12-m-long sediment core
**2-m-long sediment core

Sediment cores, 12-m long, were retrieved from Kozjak and Prošće Lakes (Srdoč *et al.* 1986b). The uppermost section, 20-cm thick, was excluded from the study because of contamination with bomb ^{14}C. ^{14}C dating of the Lake Prošće core revealed a uniform sedimentation rate (1.6 mm yr^{-1}) throughout the profile. In Kozjak Lake, the extrapolation method can be applied only to the upper 2 m of the sediment core due to sedimentation disturbances *ca.* 2 ka ago (Srdoč *et al.* 1986b). In the upper sediment layer, the sedimentation rate of 0.76 mm yr^{-1} was determined. The calculated A_0 is 71.1 pMC for Lake Prošće (Fig. 3) and 76.1 pMC for Lake Kozjak. Constant $\delta^{13}C$ and $\delta^{18}O$ values throughout the profiles (Srdoč *et al.* 1986b) indicate stable environmental conditions during the sedimentation period and hence, the constant A_0 value (Pazdur 1988).

The initial ^{14}C activities of these lake sediments obtained by different methods (Table 4) agree well. The mean A_0 value for Lake Prošće is (72 ± 1) pMC, and that for Lake Kozjak is (76 ± 1) pMC.

CONCLUSION

Three experimental methods for determining the ^{14}C initial activity of calcareous deposits were applied and tested in a karst region of the Dinaric Alps. We concluded:

1. Although ^{14}C activity measurement of DIC, recent tufa, sediment or other forms of calcareous deposits is the simplest method, A_0 values are too high because of contamination

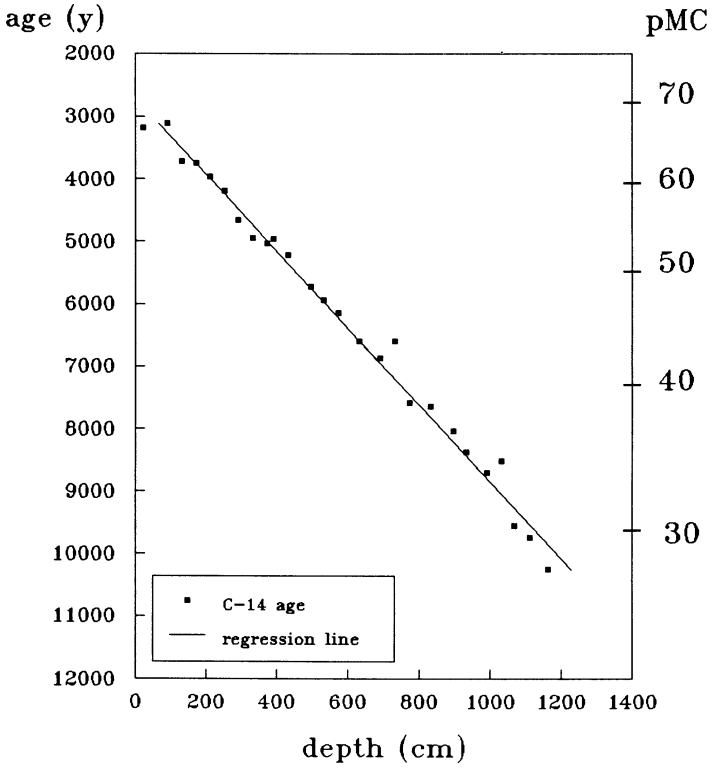

Fig. 3. ^{14}C age and ^{14}C activity in pMC of the 12-m-long sediment core retrieved from Lake Prošće, Plitvice National Park. The regression line shows the sedimentation rate is 1.6 mm yr^{-1} and the A_0 = (71.1 ± 0.7) pMC throughout the sediment depth.

of carbonates with bomb ^{14}C. Pre-bomb-era samples, if available, give a reliable A_0 value for the investigated area.

2. ^{14}C activity of a relatively long lacustrine sediment profile extrapolated to the surface yields an A_0 value based on a constant sedimentation rate. The precipitation conditions should be checked by $\delta^{13}C$ measurements, and the uppermost layer contaminated with bomb ^{14}C should be discarded.
3. The ratio of ^{14}C activities of coeval carbonate and organic matter of terrestrial origin (wood, leaves) gives a reliable A_0 value. The origin of organic material should be checked by measuring $\delta^{13}C$. Aquatic plants buried in sediment that use DIC from freshwater for photosynthesis have ^{14}C activities similar to those of DIC, and they should not be used for calculating A_0. The same holds for organic residue of lake marl, which usually contains a substantial fraction of aquatic plant carbon.

In order to obtain reliable A_0 values, at least two of these methods should agree. We obtained fairly consistent A_0 values for the Plitvice Lakes.

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