7. Some problems in connection with C^{14} dating of tests of Foraminifera

By

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ABSTRACT.—A C^{14} dating of foraminiferal tests has been made on deep-sea core material collected from the Algiers-Provençal Basin in the Western Mediterranean Sea. During the course of the experiments some difficulties arose concerning the preparation of samples. A systematic investigation of the reagents employed demonstrated the importance of using water completely free of carbon dioxide for the preparation of the samples.

Results obtained from examinations of the petrography, chemical composition and grain-size constitution of the core material demonstrated the necessity of using test fragments of a size not less than 44 μ in order to obtain a reliable dating; increased accuracy may follow the use of even coarser material. In this connection the C^{14} dating demonstrated that the 4–44 μ fraction might contain as much as approximately 75 % of *older* foreign material since the age obtained for this fraction was too great by roughly two half-lives in one case, i.e. by more than 10,000 years. The material <4 μ also gave too great an age.

In connection with this preliminary investigation of the cores it appears that the temperature rise of the surface water in the Western Mediterranean Sea began contemporaneously with that in the Atlantic Ocean or possibly a little earlier.

RÉSUMÉ. — On a procédé à une datation, par la méthode du C^{14}, de tests de Foraminifères de carottes provenant des grandes profondeurs du bassin Algiers-Provençal dans la Méditerranée occidentale. Plusieurs problèmes, exposés ici, se sont présentés lors de la préparation des échantillons. Une étude systématique des réactifs employés a montré la nécessité de n'utiliser que de l'eau dépourvue de dioxyde de carbone.

Les résultats obtenus par des études de la pétrographie, de la composition chémique et de la granulométrie des échantillons, a révélé que seuls les fragments de tests plus petits que 44 μ fournissaient des résultats sérieux et que la précision était encore meilleure avec du matériel plus grossier. À ce propos, la détermination du C^{14} a prouvé que la fraction de 4 à 44 μ contiendra environ 75 % de matériaux étrangers, *plus anciens*, l'âge obtenu pour cette fraction étant presque deux demi-vies plus grand, c'est-à-dire plus de dix mille ans. Le matériel <4 μ a lui aussi indiqué un âge trop élevé.

En connection de cette étude préliminaire, il ressort que le réchauffement des eaux superficielles de la Méditerranée occidentale a commencé en même temps, ou peut-être un peu plus tôt, que dans l'Océan Atlantique.

Contents

Introduction

In deep-sea sediments the temperature changes which occurred during the Pleistocene period are usually indicated as variations in the frequency of the most temperature-sensitive species of the microfossil assemblage. Among these fossils the planktonic foraminiferal tests provide the most trustworthy indications of the climatic variations of this period, since their concentration varies in accordance with the temperature changes in the surface water of the sea and because successive accumulations of tests normally occur in chronological order (ERICSON *et al.* 1956). During the last decade several papers dealing with such investigations have been published (ARRHENIUS 1952*a*, EMILIANI 1955, 1957, EWING and DONN 1956, RUBEN and SUESS 1955, ERICSON *et al.* 1956, 1961, BROECKER *et al.* 1960*a* and others) and particular attention has been given to conditions in the Atlantic and Pacific Oceans. These investigations have given rise to the fairly general opinion that the last more significant rise in the surface water temperature of the Atlantic occurred about 11,000 years ago.

In connection with a petrographical study of some sediment cores from the Western Mediterranean Sea the problem of dating a given deposit was encountered by one of the authors (K.G.E.). Similar problems with material from the same area had previously been touched upon by R. TODD, 1958, and OLAUSSON, 1960 and 1961.

The present paper deals with an investigation into the dating of three sediment cores collected in June 1948 from the deepest part of the Southern Algiers-Provençal Basin during the cruise of the Swedish Deep-Sea Expedition with the "Albatross" under the direction of HANS PETTERSSON (1957). The location of the coring operations and the numbers of the sediment cores are shown in Fig. 1. By means of the Kullenberg piston core sampler the cores were taken from a depth varying from 1325 m to 2782 m; the cores have lengths of 9–10 m (KULLENBERG 1955). A detailed description of core No. 210 will be published by ERIKSSON in the Reports of the Swedish Deep-Sea Expedition, Vol. VIII, No. 7, and the two other sediment cores will be treated later in the same publication. The present paper deals mainly with the C^{14} dating of foraminiferal tests in the deep-sea sediments of the Mediterranean and pays special attention to some possible sources of experimental errors.

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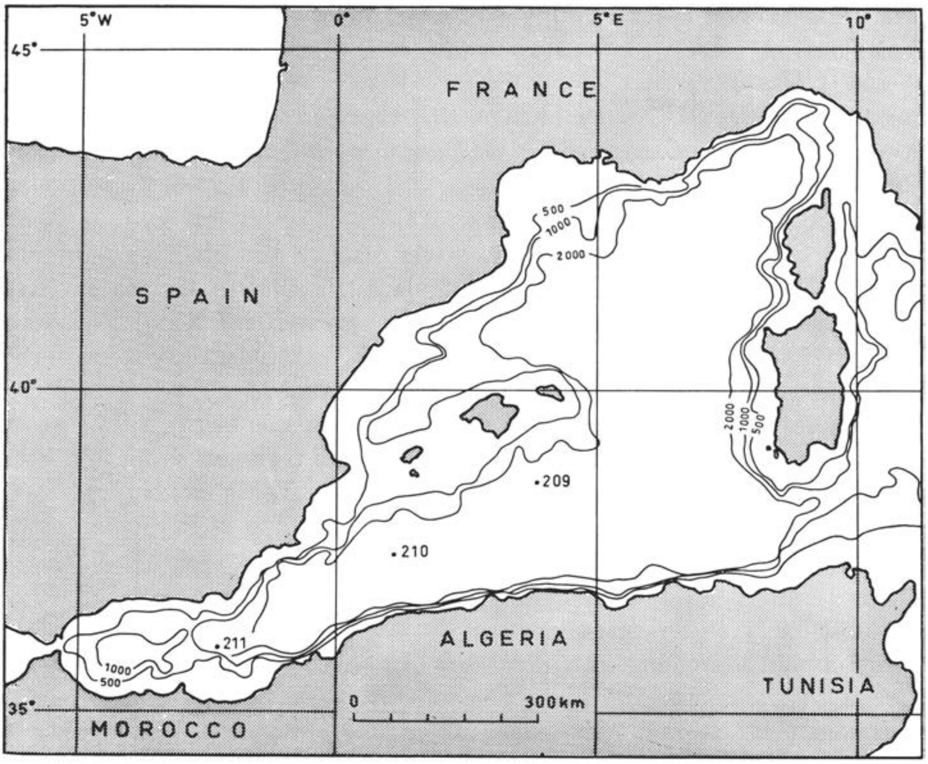

Fig. 1. Sites of coring in the Western Mediterranean Sea.

The Late Glacial—Postglacial Boundary

One of the more important climatic changes during the Pleistocene Period, which has been distinctly registered in the different fossil assemblages, especially in the continental deposits (plant remains such as pollen grains and seeds, skeletal remains of animals, etc.) and which has been dated by the C^{14} method with a comparatively high degree of accuracy, is the amelioration which led to the various climates of the Postglacial times. In the marine sediments this Late Glacial—Postglacial boundary is to be found in a zone in which a Foraminifera assemblage consisting of mainly cool-tolerant species is replaced, at higher levels, either entirely or partially by a more warm-tolerant assemblage closely similar to the fauna at present inhabiting the sea. The mid-point of the transition zone is used to define the boundary between the two climatic phases since this change in fossil assemblage is a reflection of the temperature rise in the surface water of the sea.

In the Western Mediterranean Sea the Foraminifera species *Globigerina pachyderma* (D'ORBIGNY) was the most important of the true cool-tolerant species during the Würm Ice Age (R. TODD 1958). The most important of the true warm-tolerant species during Postglacial times are *Globigerinoides sacculifera* (BRADY), *Globigerinella aequilateralis* (BRADY) and *Globorotalia truncatulinoides* (D'ORBIGNY). Accordingly the boundary between the Late Glacial and Postglacial phases has been located in the transition zone between the cool-tolerant assemblage dominated by *Globigerina pachyderma* and the relatively warm-tolerant assemblage dominated by the other three species mentioned above (PHLEGER 1947, 1953; F. PARKER 1958).

Owing to the exchange of surface waters between the Mediterranean Sea and the Atlantic Ocean, the fossil assemblages in adjacent regions of these water masses can be expected to correspond to a great extent. These conditions imply that in the transition from Late Glacial to Postglacial the change in the assemblages in these two regions should roughly coincide.

An estimate of the date at which the temperature rise began will be given in the present paper, but this topic will be treated in greater detail in a later publication (ERIKSSON, Repts. Swed. Deep-Sea Exp., Vol. VIII, No. 7).

The Sediment

The sediment in the deeper parts of the Algiers-Provençal Basin is mainly a greyish mud alternating with a few beds of pure clay or sand. The carbonate content varies between 20 and 50 % and the mean value is c. 40 %. The calcareous material is to great extent authigenic, consisting of test fragments of planktonic and benthonic Foraminifera, Pteropoda, Coccolithophoridae, and of other planktic and benthonic organisms. It also contains a significant amount of wind-borne material derived from the surrounding continents. According to analyses of three samples of aeolian dust from the Sahara currently being blown out over the Atlantic (Cap Verde Basin), the dust contains between 25 and 40 % of calcareous material (RADCZEWSKI 1939).

The main parts of the sediments occur *in situ* and generally exhibit a fairly dense and homogeneous structure. At some levels, however, beds of re-deposited material occur; such deposits have been transported either by slip or by some type of density flow. They usually have a heterogeneous or graded bedded structure.

As mentioned above, wind-borne materials play a big rôle in the Mediterranean area in determining the sediment composition and accumulation rate both in the sea and on the land. In the central part of the Algiers-Provençal Basin two principal types of aeolian deposit may be distinguished; one is a fine-grained type in which the chief mode is the finer half of the silt-size fraction (aeolian dust, supplied almost annually by the sirocco winds) while the other is more coarse-grained with the chief mode in the coarse silt or the fine sand fraction (aeolian sand, supplied sporadically and only seldom by very strong storms). These types may have the same or different parent materials; they consist mainly of quartz and calcareous fragments in varying proportions

Table 1. Contamination of "infinitely" old material with recent material due to different dispersing media.

Laboratory No.	Dispersing medium: Medium (1 liter)	Dispersing medium: Wash liquid (0.5 liter)	Subsequent treatment	Apparent age years[b]	δC^{13} ‰	Net counting rate c.p.m. (contamination)
U-287	Dist. H_2O	Dist. H_2O	—	$33{,}800^{+2400}_{-1900}$	−7.5	0.14 ± 0.03
U-288	NH_4OH 0.1 %	NH_4OH 0.1 %	—	$32{,}000^{+1600}_{-1400}$	−8.2	0.18 ± 0.03
U-289	Dist. H_2O	Dist. H_2O	Dil. HCl (pH = 4.0)[a]	$37{,}400^{+3600}_{-2500}$	−7.5	0.09 ± 0.03
U-290	Boiled dist. H_2O	Boiled dist. H_2O	—	> 40,000	−7.7	0.02 ± 0.03
U-291	NH_4OH 0.1 %	NH_4OH 0.1 %	—	$36{,}500^{+3100}_{-2300}$	−7.3	0.10 ± 0.03
U-292	Dil. HCl (pH = 4.0)	Dil. HCl (pH = 4.0)	—	> 40,000	−7.5	0.05 ± 0.03

[a] Following filtration the sample was treated with 5 cm^3 of dil. HCl (pH = 4.0) and dried in the oven at 105° C.

[b] The apparent ages are the results, within the limits of error, corresponding to the net counting rates, due to contamination. The errors given are the statistical errors. The accepted value of 5570 years is used for the half-life of radiocarbon. The δC^{13} values give the C^{13} enrichment relative to the Chicago PDB standard (CRAIG 1961). 95 % of the net counting rate of the NBS oxalic acid gives 9.31 c.p.m. in the proportional counter.

depending upon the composition of the parent material or upon selection during transport. Thus, in a deposit rich in aeolian sand, foraminiferal tests are either very rare or absent; in a sediment poor in such sand, tests may be abundant.

With one exception none of the samples examined in the present investigation was taken from beds rich in aeolian sand material largely because of the scarcity of tests in such material and also in order to avoid contamination as far as possible from coarser wind-borne calcareous components.

Preservation and Preparation of the Material

The sediment cores, numbered and divided into suitable portions, were wrapped in double sheets of acetate foil, placed in aluminium tubes and then immediately transferred to the cold storage room of the ship. After the return of the Expedition the cores were kept in cold storage at the Oceanographic Institute in Göteborg, in order to preserve their natural moisture and to prevent subsequent structural and chemical changes (cf. ARRHENIUS 1952*b*). In 1957 one of the present authors (K.G.E.) brought the cores to the Geological Institution in Uppsala where they were transferred into flat, air-tight boxes of galvanized iron plate each containing a layer of water at the bottom (cf. NORIN 1958, p. 23) and again preserved in cold storage.

Before removing a sample for dating in the C^{14} laboratory, the calcareous material was separated from the other components of the sediment. The very first samples were dispersed with aqueous ammonia and then either sieved, in cases where material >44 μ was to be used, or separated by the Atterberg decantation method, where material with a lower limit of 4 μ was required, in accordance with the standard methods for some grain-size analyses.

The Core Material under Investigation

The foraminiferal tests are usually most abundant in the 50–100 μ size grade; sometimes a coarser size grade may predominate. In the latter case the deposition of the larger tests is usually associated with a marked change in the sedimentation conditions resulting from changes in the principal wind direction followed by changes in the speed of the surface current. The concentration of tests in the sediment is fairly irregular and generally low. Thus a rough estimate of the test concentration in core No. 210, as it appears in thin-sections under the microscope, gives normally less than 10 %. At some levels in which a strong accumulation of tests occurs, the test frequency may be much greater.

It has previously been demonstrated that for sediment material from the bottom of the Atlantic Ocean, the fine carbonate fraction (<74 μ) gives a greater age than the coarser carbonate fraction (RUBEN and SUESS 1955; ERICSON *et al.* 1956). In the present investigation of material from the Algiers-Provençal Basin it was necessary, however, to use shell material >44 μ, owing to the low test concentration in the cores and in order to obtain enough material for the radiocarbon dating without using too great a portion of the sediment core.

Despite the use of this finer grade of material it was still not possible to generate sufficient carbon dioxide for a normal radiocarbon dating, and instead the gas was diluted so that the proportional counter could be filled to the normal working pressure of three atmospheres. The ratio between the total pressure and the pressure of the gas generated from the sample is termed the dilution factor and is denoted below by Sp.

It was previously suggested by ARRHENIUS (1952*a*) and further discussed by *inter alia* EMILIANI (1957) and ERICSON *et al.* (1961) that at some levels in the sediment cores the bottom sediments had been reworked by the action of mud feeders. According to studies of the three cores which were investigated such mixing seems to have little effect on the dating result.

The risk of error due to the time required for the components in the sediment to settle is negligable according to the figures given in Table 15 on p. 253 by KUENEN (1953). Thus, for example, foraminiferal tests with a diameter of 20 μ need three days to settle 100 metres. In the present instance the depth varies between c. 2000 and 3000 metres and the settling period corresponds to 60 to 90 days; this is negligable in comparison with the geological ages involved.

Table 2. C^{14} dating of calcareous material of different grain sizes.

Sample	Laboratory No.	Grain size of material	Dispersing medium	Primary radiocarbon age B.P.	Dilution factor Sp.	δC^{13} ‰
20903:155–145	U-293	$>44\,\mu$	Dist. H_2O	$14{,}200^{+480}_{-460}$	2.5	-3.2 ± 0.5
	U-294	$4–44\,\mu$		$26{,}600^{+830}_{-760}$	1.0	-1.2 ± 0.5
	U-295	$<4\,\mu$		$17{,}300^{+300}_{-290}$	1.0	0.0 ± 0.5
21107:440–428	U-296	$>44\,\mu$	Boiled dist. H_2O	$16{,}700^{+1200}_{-1100}$	7.2[a]	$+0.1\pm0.5$
	U-297	$4–44\,\mu$		$22{,}300^{+750}_{-690}$	1.6	-2.3 ± 0.5
	U-298	$<4\,\mu$		$21{,}200^{+430}_{-410}$	1.0	0.0 ± 0.5

[a] Due to losses of material during the purification of the carbon dioxide the value of Sp. is higher than is implied by the amount of test material.

Radiocarbon Dating

The radiocarbon datings were performed by one of the present authors (I.U.O.) at the Uppsala Radiocarbon Laboratory (OLSSON 1958, 1961). The initial results were calculated using a sample of NBS oxalic acid as a reference material (GODWIN 1959), assuming a half-life of 5570 years for C^{14} (GODWIN 1962) and including corrections for deviations in the C^{13}/C^{12} ratio; no corrections were made, however, for the apparent age of the sea water, which is probably about 600 years in this area (BROECKER and OLSON 1959, 1961). The mass-spectrometer analyses were performed at Karolinska Institutet, Stockholm, under the supervision of Dr. R. RYHAGE.

The figure of 600 years for the surface water seems to be about 150 years greater than the corresponding mean value for the Atlantic. This may be attributed to upwelling phenomena which can occur with extraordinary strength so that water from the deeper regions mixes with the surface water to a greater extent than usual. According to W. S. BROECKER, R. GERARD, M. EWING and B. C. HEEZEN (1960*b*) and G. S. BIEN, N. W. RAKESTRAW and H. E. SUESS (1963) the apparent age of deep water may be several hundreds of years more than that of the surface water. The apparent age of 600 ± 200 years can therefore be assumed for the Mediterranean Sea. The existence of some variations in the C^{14}/C^{12} ratio occurring on a world-wide scale during geological times has been disregarded in this discussion.

In the first attempts to correlate the results of the C^{14} datings performed on core material from the Western Mediterranean Sea with results relating to the

Table 3. C^{14} dating of calcareous material from cores

Sample	Laboratory number	Grain size of material	Dispersing medium	Primary radiocarbon age years B.P.	Dilution factor Sp.
20903:155–145	U-293	$>44\,\mu$	Dist. H_2O	$14{,}200^{+480}_{-460}$	2.5
21001:25–21	U-140	$>62\,\mu$	Dist. H_2O NH_4OH Tap water	6,450 ± 190	2.4
21001:29–23	U-255	$>44\,\mu$	Dist. H_2O	5,880 ± 100	1.0
21002:48–45	U-39	Total	—	10,380 ± 120	1.0
21002:65–62	U-141	$>62\,\mu$	NH_4OH Tap water	5,460 ± 220	3.5
21002:100–96.5	U-25	Total	—	11,780 ± 160	1.0
21003:117–107	U-256	$>44\,\mu$	Dist. H_2O	$8{,}980^{+360}_{-330}$	4.2
21004:192–181	U-299	$>44\,\mu$	Boiled dist. H_2O	11,660 ± 260	1.8
21007:398–390	U-251	$>44\,\mu$	Tap water	13,180 ± 300	1.7
21107:440–428	U-296	$>44\,\mu$	Boiled dist. H_2O	$16{,}700^{+1200}_{-1100}$	7.2[a]

[a] Due to losses of material during the purification of carbon dioxide the value of Sp. is higher than is implied by the amount of test material.
[b] The analyses are made by R. Todd (1958).

Atlantic large discrepancies were obtained. Moreover inconsistent results were given by a series of determinations on material from the same core. This might be assumed to arise either from storage or preparation of the samples or to too low a grain size limit for the material selected for dating.

To avoid possible contamination due to absorption of carbon dioxide from the air by alkaline solutions, trials were made with different reagents to produce dispersion of the sample. To facilitate a systematic investigation of the error introduced during the preparation of the samples, fragments of an oyster shell of Tertiary Age from Cyprus (kindly placed at our disposal by Dr. Fritz Brotzen, Geological Survey of Sweden, Stockholm) were treated with one of the following reagents; aqueous ammonia, distilled water from the laboratory tank and the same water either acidified or boiled.

The shell was first washed with distilled water and then treated with dilute hydrochloric acid to remove the surface layer. As a result of this treatment the shell (initially 169 g) lost 34 g corresponding to about 20% in weight. Such a procedure has previously been tested on several shell samples (Olsson and

Nos. 209, 210 and 211, Western Mediterranean Sea.

δC^{13} ‰	Estimated apparent age of water	Radiocarbon age corrected for estimated apparent age	Half-life correction (from 5570 to 5730 years)	Corrected radiocarbon age	Predominating Foram. spec. acc. to Eriksson (1964)[b] Warm-tolerant Foram.	Cool-tolerant Foram.
-3.2 ± 0.5	600 ± 200	$13{,}600\pm520$	+2.87 %	14,010	Roughly Equal	
-2 ± 1		$5{,}850\pm280$		6,040	+	
-2.3 ± 0.5		$5{,}280\pm230$		5,450	+	
-3 ± 1		$9{,}780\pm240$		10,080	+	
-6 ± 1		$4{,}860\pm290$		5,020		+
-2 ± 1		$11{,}180\pm260$		11,520	+	
-5.5 ± 1		$8{,}380\pm410$		8,640	+	
-2.6 ± 0.5		$11{,}060\pm330$		11,400	Roughly Equal	
-10 ± 1		$12{,}580\pm360$		12,960		+
$+0.1\pm0.5$		$16{,}100\,{}^{+1200}_{-1150}$		16,600		+

BLAKE 1961–1962). The shell was then crushed in a mortar to a grain size corresponding roughly to the size range of the foraminiferal tests and thoroughly mixed. From this fairly homogeneous sample 6 aliquots were taken and treated with the different liquids mentioned above (Table 1).

The effect produced by contamination, which is easily introduced during treatment of a sample with aqueous ammonia, is given in Fig. 2. From the curve it is seen for example that the date for the transition from the Late Glacial to the Postglacial phase can easily be 500 years too low if the sample is not prepared with caution. The corresponding error introduced during a radiocarbon determination of a sample roughly 30,000 years old can easily be −5000 years.

From the description of the sediment core it is evident that the finer fractions of the calcareous material often consist of minute calcareous fragments supplied either by air (aeolic dust) or, to a lesser extent, by river. In the coarse fraction the calcareous material consists almost entirely of foraminiferal tests and shell fragments of Pteropoda. The choice of 44 μ as the limit between the fine and coarse fractions was justified from a consideration of the grain composition of the core material (Fig. 3 and e.g. Plate No. 3 in ERIKSSON 1964).

In two cases analyses of different fractions were performed to show how

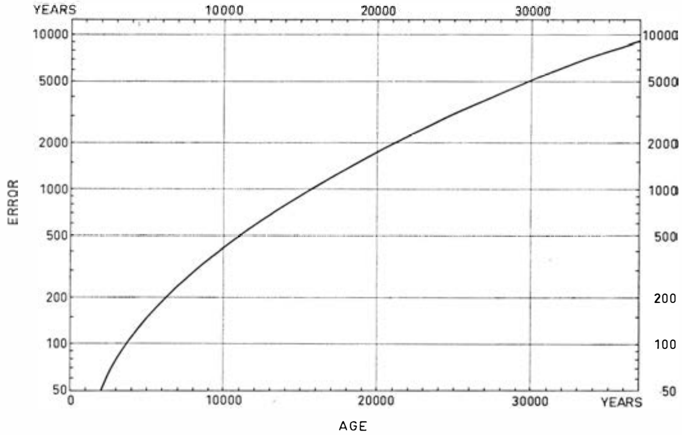

Fig. 2. Curve showing the error in age due to an arbitrarily chosen contamination by recent material corresponding to 0.2 c.p.m. in a counter with a net counting rate for the reference sample of 9.3 c.p.m.

severe the contamination by wind-borne material can be in the Mediterranean area (cf. Table 2). Judging from the microscopical examination of the samples, the foreign materials in the component described as "Calcareous fragments" do not exceed 1 %. The relative amounts of the various components (foraminiferal tests, calcareous fragments and other constituents) in the different fractions are shown in Fig. 3. From Fig. 4 it can be seen that a contamination corresponding to 1 % of old material in the total calcareous material produces an error of +80 years.

From Table 3 it is evident that where the total material has been used the dates for the samples are too high by comparison with the other samples. Accordingly, the dating results for samples Nos. U-25 and U-39 must be rejected. For this preliminary determination of the beginning of the last temperature rise in this area the discussion will therefore be restricted to samples Nos.

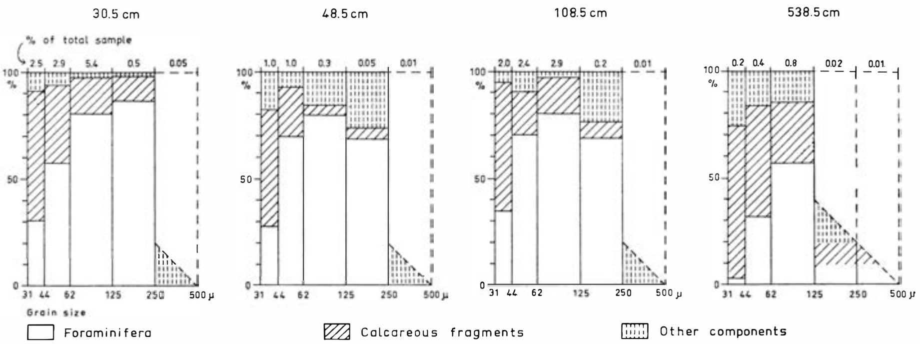

Fig. 3. Grain-size histograms showing the percentage of relatively entire foraminiferal tests, calcareous fragments (mainly Pteropoda shell fragments and broken foraminiferal tests) and other components (terrigenous minerals etc.) in some samples from core No. 210. The percentages were determined microscopically by counting small samples of the various fractions.

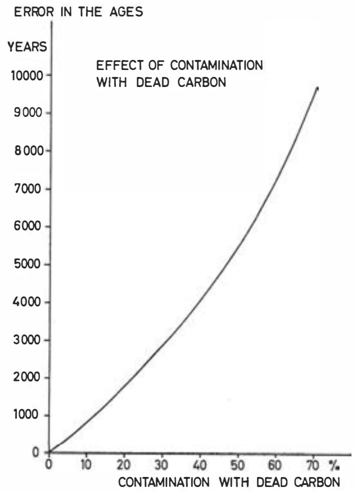

Fig. 4. The error in age as a function of the percentage of contamination by an "infinitely" old material. The curve is valid for samples of all ages.

U-251 and U-256 in core No. 210, No. U-296 in core No. 211 and No. U-293 in core No. 209. The statistical error is about ± 300 years for most of these dating results. The error in the estimated apparent water age can be set at ± 200 years (cf. above). It can be assumed that the overall contamination would produce a result rather too low than too high; consequently it is suggested that these errors should be estimated at about $\pm^{100}_{300}$ years (cf. above). Accordingly most of the results should be quoted with errors amounting to about ± 500 years at least. It should be stressed, that according to current practice the uncertainty in the value of the half-life is not included in this figure.

Conclusions

This investigation has shown that the sediment fraction $<44\ \mu$ from the Mediterranean Sea sediments contain a considerable amount of old material. This has led, in one instance, to an increase of 10^4 years in the age of a sample examined by the radiocarbon dating technique. The investigation has also shown that unless certain precautions are taken contamination of the sample by atmospheric and hence "young" carbon dioxide may occur during the dispersion process. Thus, boiled or acidified distilled water is to be preferred as the dispersing agent.

To judge from the present datings as they relate to foraminiferal test frequency of the most temperature-sensitive species, the surface water temperature in the Western Mediterranean Sea began to rise about 13,000 years ago. A more detailed geological discussion of this result will be published in a forthcoming paper.

Institute of Quaternary Geology and Institute of Physics, University of Uppsala, September 30th, 1963, Sweden.

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