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CARBONATE-WATER ISOTOPIC TEMPERATURE SCALE

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By Samuel Epstein, Ralph Buchsbaum, Heinz Lowenstam, and Harold C. Urey

ABSTRACT

A relationship between temperature and relative O^{18} abundance in calcium carbonate in marine shells has been determined. If the relative O^{18} abundance of the water in which the shell grew is known, the temperature can be determined to an accuracy of $\pm 1.0°C$. The relative O^{18} contents of marine waters increase with salinity and, in the case of surface marine waters, with salinity and temperature.

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ILLUSTRATIONS

INTRODUCTION

Urey *et al.* (1951) discuss a method for measuring paleotemperatures that depends on a variation in the oxygen −18 abundances in calcium carbonate deposited in the sea. McCrea (1949; 1950, p. 849) investigated the isotopic composition of calcium carbonate precipitated inorganically from water and determined the temperature coefficient for the abundance of O^{18} in calcium carbonate relative to water as a function of temperature; he found a value of −5.4°C permil increase in the ratio of O^{18} to O^{16} in the calcium carbonate. McCrea gave a calculation for this quantity including estimates of the effect of the translational and hindered rotational degrees of freedom in the solid state on the distribution functions, and arrived at a value of 5.1. Assuming free rotation in the solid state Urey (1947, p. 562) calculated a value of −5.7 for the same quantity. This research aims to determine this quantity and to set up an equation relating temperature to the difference in isotopic composition of samples of carbon dioxide prepared from organically precipitated calcium carbonate and an arbitrarily selected reference sample of carbon dioxide.

The relationships between temperature and O^{18} abundance in calcium carbonate from marine shells and that from inorganic calcium carbonate precipitates are similar. But a number of special considerations must be taken into account in the case of marine shells to find the temperature of the sea water in which the marine animals lived: (1) the isotopic composition of the oxygen of the oceans; (2) the collection of marine shell-bearing animals which grow in water of constant and known temperatures; and (3) the problem of impurities in the carbon dioxide prepared from marine shells.

AKNOWLEDGMENTS

stitute for grants which have made this work possible, and we wish to extend our most sincere thanks for this help. During recent months the work has also been supported under Task Order XXVIII, Contract N6ori-20 of the Office of Naval Research.

RELATIVE O^{18} CONTENTS OF MARINE WATERS

Since the ratios of vapor pressures of H_2O^{16} to H_2O^{18} are 1.008 and 1.011 at 25°C and 0°C respectively (Riesenfeld and Chang, 1936, p. 127), evaporation and condensation continuously change the O^{18} contents of bodies of water. The magnitude of the variation of O^{18} in the oceans is important to the determination of a valid temperature scale and to the general problem of the determination of temperature of unknown samples, because this thermometer depends essentially on the magnitude of the difference between the O^{18} contents of the calcium carbonate and the water in which it was precipitated. Consequently a series of O^{18} determinations were made on samples of sea water, most of which were made available to us by H. Pettersson, who collected them on the ALBATROSS expedition (1949, p. 182). The method of determining the relative O^{18} content of water samples involves the equilibration of carbon dioxide with the water at 25°C and analysis of an aliquot of the gas. The carbon dioxide so equilibrated is compared with our standard working sample of carbon dioxide prepared from the calcium carbonate of belemnites of the Pee-dee formation using a high-precision mass spectrometer (McKinney, McCrea, Epstein, Allen, and Urey, 1950). The analyses are relative only, but are correct within a probable error of about $0.2\%_0$ of the isotope ratio. The results of these analyses are given in Table 1. The unit used in this table is defined by the following equation:

$$\delta(O^{18}) = \left(\frac{R_{sample}}{R_{standard}} - 1\right) 1000,$$

where R_{sample} and $R_{standard}$ are the ratios of $CO^{16}O^{18}$ to CO_2^{16} for the sample and standard reference gas, respectively.

Apparently, the isotopic composition of oxygen is not homogeneous throughout the oceans. Deviations from the mean isotopic composition

TABLE 1.—RELATIVE O^{18} CONTENT OF MARINE WATERS

Location	Depth of point of collection (Meters)	Temperature (°C)	Salinity (‰)	$\delta(O^{18})$ (‰)
30°47'N. 25°25'W.	10	—	37.44	-0.0_1
	1000	—	35.43	-0.5_9
26°25'N. 34°00'W.	5000	—	34.9	-0.2_1
14°27'N. 66°13'W.	5000	4.2	34.9	-0.7_4
14°15'N. 71°02'W.	0	27.7	36.0	0.4_2
02°52'S. 88°20'W.	20	18.8	34.4	-0.3_6
	500	7.5	34.6	-0.5_0
	1000	4.5	34.6	-0.5_5
13°25'S. 149°30'W.	0	27.7	35.8	-0.2_3
	1000	4.3	34.6	-0.9_3
	4000	1.4	34.7	-0.6_5
07°45'S. 120°30'E.	10	29.5	34.5	0.5_7
10°06'N. 52°45'E.	20	29.3	35.5	0.0_6
	500	10.7	35.0	-0.2_5
	2000	2.9	34.8	-0.9_0
35°41'N. 21°50'E.	50	16.5	38.8	0.6_9
	2000	13.6	38.7	0.8_4
00°13'S. 18°26'W.	25	22.6	35.6	0.0_4
	500	7.1	34.7	-0.5_0
	2500	3.0	34.9	-0.3_8
	7500	1.4	34.8	-0.4_4
28°05'N. 60°49'W.	25	28.5	36.8	0.3_8
	500	16.3	36.3	-0.1_8
	2000	3.7	35.0	-0.5_9
43°04'N. 19°40'W.	2000	3.6	35.0	-0.6_7
President Channel, Puget Sound	171	9.4	31.4	-1.9_4
Middle Channel, Puget Sound	157	7.4	33.1	-0.9_1
Encenada, Mexico	~0	19	33.5	-0.8_1
St. Tomas, Mexico	~0	15	33	-0.7_7
Monterey Bay, California	~0	11	33.8	-0.8_5
La Jolla, California	~0	15	34.0	-0.7_6
Bermuda	~0	18–29	—	0.6_4

of the oceans occur in the waters close to the mouth of a river, as is the case for the President Channel location. If water of fully marine composition is considered, then the maximum variation of O^{18} in ocean water is $1.7\%_0$, which would represent about 10°C variation in temperature determination. Clearly, it is necessary to determine the O^{18} content of the water in which a marine shell grew if this particular shell is to be used in correlation of temperature with the O^{18} content of organically precipitated shell.

On further examination of the data an attempt was made to establish a correlation between the O^{18} content and the salinity of the water. Such a correlation is possible because processes which tend to increase or decrease the O^{18} of the water also tend to increase or decrease
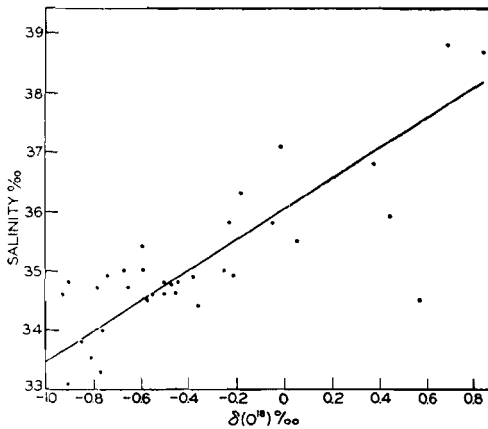

FIGURE 1.—PLOT OF SALINITY VS. O¹⁸ CONTENT FOR OCEAN WATERS

the salinity. The plot of salinity against relative O^{18} content is shown in Figure 1. The points scatter badly, but a regular trend is evident, and a straight-line relationship is assumed between salinity and O^{18} content of the water. The relationship is only approximate as was to be expected. Extrapolation of the line to zero salinity gives $-13.5\%_{00}$ as the relative O^{18} content of fresh water.

The approximate linear relationship between salinity and O^{18} abundances suggests that it might be more rigorously followed if limited to surface waters. Since surface salinity and surface temperature show some indication of being related linearly, we have plotted in Figures 2 and 3 salinity vs. O^{18} content and temperature vs. O^{18} content respectively for the waters of Table 1 coming from a depth of 25 meters or less. The plots indicate rather definite linear relations, but the data are too meager to justify any definite conclusion. We shall continue this investigation as we secure more water samples, particularly from higher latitudes.

The mean isotopic composition of the oceans will be close to that of the water of mean salinity, and we have selected this as having $-0.47\%_{00}$ relative to our standard gas and

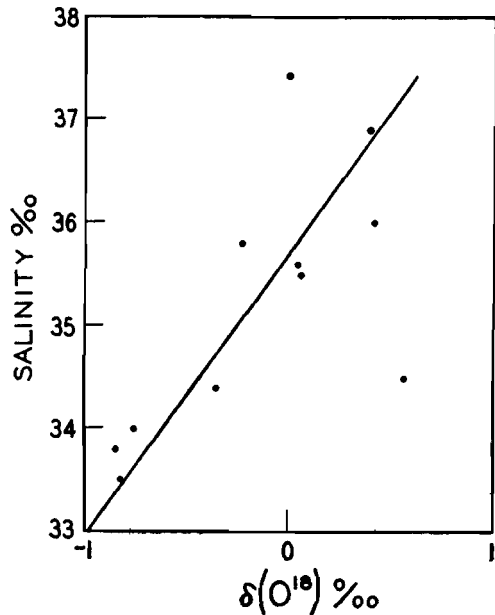

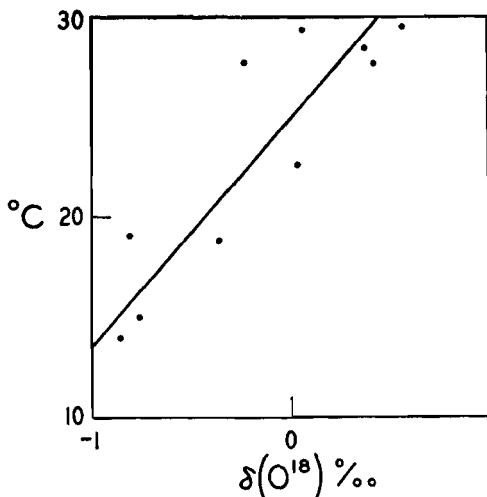FIGURE 2.—PLOT OF SALINITY VS. O¹⁸ CONTENT FOR SURFACE OCEAN WATERS

FIGURE 3.—PLOT OF TEMPERATURE VS. O¹⁸ CONTENT FOR SURFACE OCEAN WATERS

corresponding to $34.8\%_{00}$ salinity. This gives $-13.0\%_{00}$ as the isotopic composition of fresh water relative to that of sea water of $34.8\%_{00}$ mean salinity. This difference is larger than that expected for a fractionation equal to the ratio of vapor pressures, and substantially greater than that for any fresh-water samples investigated by us. Lake Michigan is representative and

analyzes as $-6.2\%_{oo}$ relative to our mean sea water.

This large variation of isotopic composition of sea water decreases the reliability of temperature measurements by this method unless a sample of water is analyzed at the same time and a correction for the isotopic composition of the water is made on the carbon dioxide analyses. This error, some 2.5 times as large as expected, is disappointing, especially when paleotemperatures are considered. The melting ice of high latitudes must have a lower O^{18} content than has fresh water of the temperate zone. The whole problem of water evaporation from the oceans and its precipitation over the land and oceans is very complex, and our data are not sufficiently extensive to justify a more detailed discussion.

A correlation between salinity and O^{18} content is useful in that salinity data which are quite readily available for present-day oceans can give an approximate correction for the isotopic abundance if water samples are not available for mass spectrometric analyses. We hope the distribution of O^{18} between phosphate and water will give another thermometer with a different temperature coefficient, thus making possible the elimination of the water phase.[1]

ISOTOPIC TEMPERATURE SCALE

Marine Shells Selected and their Growth Temperatures

One problem in establishing a temperature scale using marine calcareous-shelled animals was to get shells which grew at known temperatures. To grow certain marine animals under controlled temperatures in the laboratory is a lengthy and difficult project. Nevertheless, constant-temperature baths were set up at three Pacific Coast marine laboratories,[2] and these

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[1] M. Steinberg is developing this possibility in our laboratory.

[2] Oceanographic Laboratory, University of Washington, Friday Harbor, Washington. We are indebted to Dr. Thomas G. Thompson and Dr. Emery Swan for assisting in this work, and also to Dr. C. Barnes for help in locating suitable areas for collecting. At the Hopkins Marine Station we are indebted to Dr. Rolf Bolin for help in looking after one of the experimental tanks. At Scripps Institution of Oceanograph, La Jolla, California, we are indebted to Dr. Carl Hubbs who assisted us in setting up one of the experimental tanks and in making collections in lower California.

were stocked with calcareous organisms with notched or drilled shells. It was thought that if repair occurred, the new shell, when analyzed, would yield reliable data which could be used to fix points on the temperature scale. Not all of the material is in, and some of the attempts were unsuccessful. However one of the experimental batches of animals has been analyzed and the results are presented later. Consequently, shell-bearing marine animals were collected from locations which have a minimum annual variation of temperature. The species of animals chosen were attached or sedentary forms which live in relatively confined areas so that the point at which collection was made would be representative of the condition under which the animals grew their shells. Animals were collected from marine locations which had records of maximum constancy and which had a minimum of contamination with freshwater drainage. Collections were made in the cooler waters of Puget Sound, intermediate conditions of Monterey Bay, and the warmer waters of certain areas along the coast of lower California in Mexico. In addition, shells from the still warmer waters of Bermuda were analyzed.

Particular care was taken to estimate carefully the temperature at which the particular fragment of shell used for analyses grew. For example, the samples analyzed from Santa Tomas, Mexico, included one large red abalone, *Haliotis rufescens*, and one large black abalone, *Haliotis cracherodii*, and two keyhole limpets, *Fissurella volcano*. The *Haliotis* were collected August 25, 1949, and thus the outer few millimeters of the rim of the shell grew within the months May to August. Dr. C. L. Hubbs measured temperatures at several stations in this region during September 1948, May, July, and August 1949, and also observed the daily variation on August 18, 1949. From these measurements we conclude that 15°C is a reasonable average temperature. The variations in temperature during these months is about the same as the daily variation from 14.4°C to 17.0°C. Thus a probable error of ±1°C seems a reasonable estimate. A complete cross section of the shell of the keyhole limpets was taken so that it was necessary to estimate the average annual temperature. The temperature in this location goes as low as 11°C and as high

TABLE 2.—DESCRIPTION OF SHELL SAMPLES USED FOR DETERMINATION OF TEMPERATURE SCALE

Location	Specimen		Part of specimen used	Est. Temp. °C	Water analysis
	No.	Name			
Middle Channel, Puget Sound 48°22′30″N., 122°58′45″W., 130–157 meters, July 22, 1949.	1	Mussel *Volsella modiolus*	Cross section of complete half shell	7.4	-0.9_1
	2	Brachiopod *Terebratulina transversa*	Complete half shell		
President Channel, Puget Sound 48°41′50″N., 122°59′30″W., 171 meters July 16, 1949	3	Mussel *Volsella modiolus*	Cross section of complete half shell	9.4	-1.9_4
	4	Brachiopod *Terebratulina transversa*	Cross section of complete half shell		
Hopkins Marine Station, Pacific Grove, Calif.	5	Red abalone *Haliotis rufescens*. At low tide level May 14, 1949	At least a year's growth of shell	12.6	-0.8_6
	6	Giant keyhole limpet *Megathura crenulata* 50 m. off-shore, 3 m. Aug. 15, 1949	Cross section of complete shell	11.5	
Santa Tomas, Lower California, Mexico Aug. 27, 1949 3–6 meters	7	Red abalone *Haliotis rufescens*	Last ½ cm of large shell	15	-0.7_7
	8	Black abalone *Haliotis cracherodii*	Last ½ cm of large shell	15	
	9 10	Limpets *Fissurella volcano*	Cross section of 2 complete shells	14	
Between Encenada and Morro Points, Lower California, Mexico. 3 meters, Aug. 25, 1949	11	Red abalone *Haliotis rufescens*	Edge of shell	20	-0.8_1
Bermuda, Tank in laboratory	12	Snail (conch) *Strombus gigas*	Regenerated shell	31	0.6_4
	13	Bivalve, *Pinna* sp.	Regenerated shell (part)	29	

as 17°C. The average temperature of this sample of the shell was thus estimated to be 14°C within an error of less than 3°C and perhaps as close as ±1°C. Similar considerations of temperature data made available to us led to the assignment of other temperatures, and similar uncertainties exist in the estimate.

Table 2 describes the location at which the marine animals were found, the estimated temperature of the fragment of shell used for analy-

ses, and the relative O^{18} of the water collected with the sample.

The carbon dioxide was prepared from the marine shells by reacting the calcium carbonate with approximately 100 per cent ortho phosphoric acid in an evacuated vessel. The carbon dioxide was trapped in a liquid nitrogen cooled trap and pumped free of the more volatile impurities, then passed through traps covered

by a solid CO_2-acetone bath to freeze out such less volatile impurities as water.

The samples of the shells described in Table 2 were then cleaned of obvious impurities and

isotopes of the calcium carbonate during their deposition in their complex shell structure. But not enough information has been obtained to justify this conclusion regarding these groups

TABLE 3.—ISOTOPIC ANALYSES OF ORGANIC CALCIUM CARBONATE SAMPLES

Each isotopic analysis is a mean of at least four sample preparations. The measurements were all within the accuracy of $\pm 0.2\%_o$.

Number of specimen (Table 2)	Observed Temp. °C	Analysis (No heat treatment)	Analysis (With heat reatment)	Water correction	Corrected analysis (No heat treatment)	Corrected analysis (With heat treatment)
1	7.4	1.4_2	0.3_6	0.4_4	1.8_7	0.8_0
2	7.4	1.3_4	0.1_3	0.4_4	1.7_8	0.5_7
3	9.4	0.5_4	-1.2_7	1.4_7	2.0_1	0.2_0
4	9.4	-0.2_1	-1.1_4	1.4_7	1.2_6	0.3_3
5	12.6	0.5_0	-0.5_1	0.3_8	0.8_8	-0.1_3
6	11.5	1.0_6	-0.3_1	0.3_8	1.4_5	0.0_7
7	15	0.9_2	-0.9_7	0.3_0	1.2_2	-0.6_7
8	15	0.9_5	-0.8_1	0.3_0	1.2_5	-0.5_1
9	14	0.6_0	-0.4_3	0.3_0	0.9_0	-0.1_3
10	14	0.4_6	-0.5_0	0.3_0	0.7_6	-0.2_0
11	20	0.2_7	-1.8_0	0.3_4	0.6_1	-1.4_6
12	31	-0.5_8	-2.2_7	-1.1_1	-1.6_9	-3.3_8
13	29	---	-1.7_5	-1.1_1	---	-2.8_6

the carbon dioxide prepared. Table 3 gives the results of analyses when this method of preparation of carbon dioxide is used. The specimen numbers correspond to those in Table 2. The results of the sixth column are plotted in Figure 4.

The data show a correlation between temperature at which the marine animals grew and the relative abundance of O^{18} in the carbon dioxide prepared from the shells. But the deviations from a linear or nearly linear relationship are much larger than the expected experimental error. The difference in analyses for shells collected simultaneously from the same location eliminated the possibility that the poor correlation between temperature and relative O^{18} content was due to incorrect estimation of the temperature at which the animals grew.

The possibility that the animals did not lay down their shells in equilibrium with the surrounding water seemed to be unlikely because the relative O^{18} contents of calcium carbonate showed a definite correlation with temperature. Some preliminary results obtained during the investigation indicate that perhaps the echinoderms and corals fractionate the oxygen

of invertebrates. In any case, the shells here used in the determination of the temperature scale were those which are deposited external to the animals' soft parts.

Comparison of results of McCrea with those of Table 3 indicated that the possible errors in the present work were of the order of magnitude of $1\%_o$ of the mass 46 to mass 44 ratio. Since impurities of mass 46 present to the extent of three parts per million would introduce an error equivalent to $1\%_o$ in the ratio, the usual chemical methods for analysis of impurities would be impractical in this case. Therefore, the criterion for valid analyses became the consistent correlation between temperature at which the shells were laid down and the measured change in relative abundance of mass 46 in the carbon dioxide.

Extraction of Carbon Dioxide, Problems of Impurities, and Results

The method of collecting the ion currents in the mass spectrometer gave measurements of the abundance ratio of mass 46 to the sum of masses 43, 44, 45, and 47. Consequently,

although the ratio of mass 46 to mass 44 would be relatively insensitive to small concentrations of impurities of masses 43, 45, and 47, these had to be considered. Purification of samples
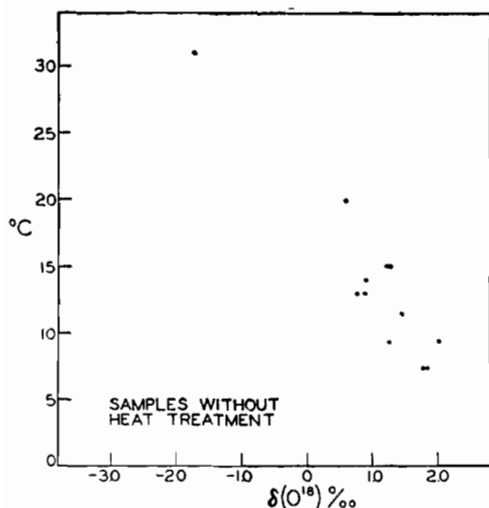

FIGURE 4.—PLOT OF O^{18} CONCENTRATION VS. TEMPERATURE: CALCIUM CARBONATE SAMPLES WITHOUT HEAT TREATMENT

could then either decrease or increase the value of the 46 to 44 ratio depending on whether purification eliminated impurities of mass 46 or of masses 43, 44, 45, and 47.

Many molecules or radicals formed in the ionizing chamber of the mass spectrometer, both organic and inorganic can contribute to the ions of mass 43 to 47. Some of these can be C_2H_5OH, CS, CH_3COOH, BCl, NO_2, N_2O, and propane. The first attempts were focussed on the possible impurities in the carbon dioxide gas. Several carbon dioxide samples from different marine shells were mixed with small volumes of bromine gas and irradiated by strong light, with the hope that bromination would shift the mass range of the impurities beyond the 43 to 47 range. Some samples of carbon dioxide were mixed with Van Slyke's (1940, p. 509) oxidizing solution or passed over hot CuO. Organic impurities would then be converted to carbon dioxide, and if present in trace amounts should not affect the 46 to 44 ratio of the carbon dioxide sample as a whole. Samples were also mixed with traces of ammonia and water to hydrolize compounds like boron

trichloride, and passed over hot copper to reduce possible traces of nitrogen oxides.

In all these cases no appreciable effect on the isotopic analyses was observed.

Samples of carbon dioxide from the shells were also mixed with fluorine. Only where there was evidence of incomplete separation of the carbon dioxide and fluorine and subsequent contact with glass was there a change in O^{18} analysis. These changes were erratic for any one source of gas samples and varied from $-0.3\%_{00}$ to $-200\%_{00}$ of the 46 to 44 ratio. This was obviously due to SiF^+ of mass 47 adding to ion beam 44 and thus showing a decrease in the ratio of 46 to 44.

A more successful approach to the purification problem involved treatment of the powdered calcium carbonate before preparation of the carbon dioxide. The powder was heated at 400°C for 60 minutes with a slow stream of helium passing over the powder. The helium was first purified by passing it over copper oxide at 750°C and then through a liquid nitrogen cooled trap. With this treatment the relative O^{18} contents of the calcium carbonate from shells was decreased in most cases, and in a reproducible manner.

Experiments to determine the length of time necessary for heating the calcium carbonate powder to attain constancy of O^{18} analysis with further heating showed that heating at 400°C for 15–30 minutes was necessary to "purify" the samples tried. A safety factor of two was adopted, and the samples were heated for an hour. Since the equilibrium pressure of carbon dioxide over calcium carbonate is 10^{-6} atmospheres at 400°C, it was necessary to test this treatment for possible loss of carbon dioxide and fractionation of the oxygen isotopes. Two samples of calcium carbonate, one normal in O^{18} and the other enriched by $100\%_{00}$, were precipitated inorganically (using sodium carbonate and calcium chloride). Samples of each were analyzed without the heat treatment and with heat treatment. The analyses of each variety of calcium carbonate checked, showing that heat treatment did not change the analyses of such samples. Clearly, it was the organically precipitated calcium carbonate alone which was affected by the heat treatment. To shorten the time for the experiments the powdered

derms and corals are not particularly useful in relation to the paleotemperature scale because of extensive infiltration of the fossils. The Foraminifera should be investigated for, even if not useful over long periods of time, they may be of interest for the investigation of more recent temperatures.

The variation of O^{18} content in marine and fresh waters, together with precise methods of measurement of the small quantities involved, makes possible an investigation of meteorological effects of considerable interest. The fractionation of the oxygen isotopes in evaporation and precipitation of water should give some information in regard to these processes as they occur in nature. We intend to continue our studies on the isotopic composition of water since the problem bears so directly on the measurements of paleotemperatures.

CONCLUSIONS

The variation in isotopic composition of marine waters is appreciable in terms of the isotopic temperature scale. When corrections are made for the isotopic composition of sea water in which mollusks and brachiopods have lived, a linear relationship between temperature and the O^{18} concentration exists. The slope of the curve agrees approximately with calculated values and with that secured when inorganically precipitated calcium carbonate samples crystallized at known temperatures are

used. Organically precipitated calcium carbonate gives reproducible analyses if it is first heated to 475°C for 15 minutes and then reacted with phosphoric acid to prepare carbon dioxide, the gas used in the mass spectrometer.

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ADDENDUM

We have delayed publication of this paper while it was in galley proof because we discovered disturbing variations in our temperature determination. We traced this to the use of heated copper oxide to remove impurities from helium gas passed over our samples during the heating process. Small amounts of oxygen from the copper oxide were present in the helium, and the carbon dioxide formed apparently exchanged in some cases with the calcium carbonate of the samples. Removal of all oxygen by means of a charcoal trap at liquid nitrogen temperature corrected these difficulties. Our tentative corrected temperature equation becomes $t = 14.8 - 5.41\delta$. The slope now agrees with McCrea's value. We will publish details of this temperature scale shortly.