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A record of Pleistocene climate from a stalactite, Nerja Cave, southern Spain

C. Jiménez de Cisneros^{a,*}, E. Caballero^a, J.A. Vera^b, J.J. Durán^c, R. Juliá^d

^a Estación Experimental del Zaidín (CSIC), Profesor Albareda, 1, 18008 Granada, Spain

^b Departamento de Estratigrafía y Paleontología, Facultad de Ciencias, Universidad, 18071 Granada, Spain

^c Instituto Geológico y Minero de España, Ríos Rosas, 23, 28003 Madrid, Spain

^d Instituto Jaime Almera (CSIC), Lluís Sole i Sabaris, 08028 Barcelona, Spain

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Abstract

A study of stable isotopes ($\delta^{18}\text{O}$, $\delta^{13}\text{C}$) of a uranium-series dated aragonitic stalactite from Nerja Cave (Málaga, southern Spain) was carried out in order to determine the conditions of deposition in isotopic equilibrium and non-equilibrium. We obtained a record of climate change from 190 000 to 160 000 years ago. A series of carbon ($\delta^{13}\text{C}$) and oxygen ($\delta^{18}\text{O}$) isotopes analyses have been carried out in parallel to the stalactite growth axis. Sampling was done in each growth layer. A curve of the secular $\delta^{18}\text{O}$ aragonite variations for the stalactite suggests cooler climate conditions in southern Spain for the period studied compared to the present.

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1. Introduction

Carbonate rocks are subject to dissolution by groundwater-forming karstic features ranging in size from microscopic vugs to passages tens or hundreds of kilometres in length. The advantage of caves for palaeoclimatic studies is the yearly stability of climatic conditions within most of them. Studies have suggested that the oxygen isotopic composition of cave dripwater is constant through the year, and approximately equal to

the mean annual precipitation outside the cave (Schwarcz et al., 1976; Yonge et al., 1985). In addition, the common observation that temperature in any deep cave is close to the mean annual temperature aboveground (Wigley and Brown, 1976) provides the fundamental principle on which palaeoclimatic studies on speleothems are based. Dorale et al. (1992), Bar-Matthews et al. (1999, 2000), Williams et al. (1999), Linge et al. (2001a,b) among others, have demonstrated the possibility of using speleothems (including stalactites, stalagmites) for palaeoclimatic reconstruction. Studies of stalactites can provide highly resolved information on climatic and environmental cycles in continental regions (Harmon et al., 1978a,b; Reyes et al., 1993).

* Corresponding author.

E-mail address: cisneros@eez.csic.es
(C. Jiménez de Cisneros).

Isotopic palaeotemperatures are recorded by a speleothem only if calcite or aragonite is deposited in isotopic equilibrium with the solution from which it is deposited. That is, the relation, being α the fractionation between calcite or aragonite and water:

$$\alpha_{c-w} \equiv ({}^{18}\text{O}/{}^{16}\text{O})_c / ({}^{18}\text{O}/{}^{16}\text{O})_w = K(T)$$

must be satisfied, where K is the equilibrium constant for the fractionation of oxygen isotopes between calcite (c) and water (w) for a given temperature (T). [Hendy \(1971\)](#) demonstrated that some speleothems are deposited in isotopic equilibrium with the associated dripwater and that these can be recognised by their constant oxygen isotopic composition within a single growth layer. Seepage water in caves isotopically resembles average meteoric water ([Harmon and Schwarcz, 1981](#)), thus making the temperature in the cave directly related to the average surface temperature ([Caballero et al., 1996](#)). Consequently, changes in oxygen isotopic composition of seepage waters should parallel changes in local meteoric water. However, in reality, the interpretation is not straightforward. [Fornaca-Rinaldi et al. \(1968\)](#), [Hendy and Wilson \(1968\)](#), [Fantidis and Ehhalt \(1970\)](#), [Hendy \(1971\)](#), and others, have established that not all speleothems are deposited in conditions of isotopic equilibrium with the water of origin, and, therefore, are not usable for palaeotemperature interpretations. Kinetic fractionation may occur when rapid evaporation takes place during deposition, or where there is a rapid loss of CO_2 from the solution to the cave atmosphere. If the relative humidity in the cave is high, e.g. close to 100%, kinetic fractionation should be limited ([Gascoyne, 1992](#)).

[Bar-Matthews et al. \(1999\)](#) have established the climate of the eastern Mediterranean region of the last 60 ka, using a high-resolution study of the oxygen and carbon isotopic composition of speleothems from the Soreq Cave, Israel. They concluded that the very low $\delta^{18}\text{O}$ values of the speleothems represent a deposition during relatively warm conditions, that the $\delta^{18}\text{O}$ values of rainfall were about 2‰ lower than at present and that annual rainfall was almost twice as high as today.

[Bar-Matthews et al. \(2000\)](#) have analysed the connection between the timing of sapropel events in the eastern Mediterranean Sea and the time of low $\delta^{18}\text{O}$ events in speleothems of the Soreq Cave. They suggested that the low $\delta^{18}\text{O}$ events in this cave speleothems constrain the maximum duration of the sapropel formation. [Repinski et al. \(1999\)](#) provide a record of climate changes for the period between 4400 years ago to the present from stable isotope analyses and uranium-series dating of a stalagmite from Cold Air Cave, South Africa. [Frumkin et al. \(1999\)](#) have produced an almost continuously dated record of a climate change encompassing isotopic stages 6–1 in Jerusalem. [Vesica et al. \(2000\)](#) have recognised isotopic stages 9, 7, 5e, 5c and 5a from oxygen isotope and U-series studies of speleothems on Mallorca, Spain.

In this paper, the results from uranium-series dating and stable isotope analyses of a stalactite from Nerja Cave, southern Spain, are presented. The main objective is to discuss the isotopic characterisation of speleothems deposited at the Nerja Cave site and, consequently, the establishment of the conditions of deposition. We demonstrate the feasibility of extracting a high-resolution record of Pleistocene climate change from speleothem in southern Spain.

2. Area description

Nerja Cave is situated in the easternmost part of Málaga province, in southern Spain ([Fig. 1](#)). The local climate is dry Mediterranean, with a mean annual temperature of 20°C. This area is characterised by a vegetation dominated by grasses using the C_3 photosynthetic pathway. This cave is an excellent fossil record of its own history and the palaeoclimatic and neo-seismotectonic evolution of the area where the cave is located. The beginning of the karstification period which gave rise to its origin started at least by Pleistocene times, as early as 800 000 years ago, although it is possible that the genesis of the cave started earlier. On the other hand, throughout the history of the cave different phases of active growth responsible for the formation of large vol-

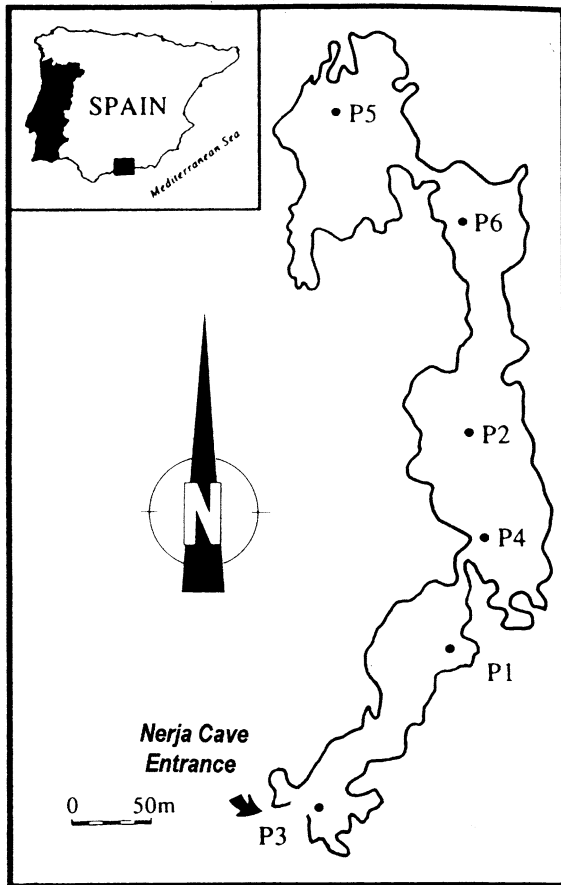


Fig. 1. Plan of Nerja Cave, showing the cave interior with the location of the halls. Inset: situation of the cave in Málaga, southern Spain.

umes of speleothems can be identified (Durán et al., 1993).

3. Materials and methods

The materials from which the Nerja Cave complex is mainly formed belong to the Middle–Late Triassic dolomitic marbles of the Betic Internal Zones (Alpujarride Complex). Discordant Pliocene and Quaternary sedimentary deposits appear near the coast (Sanz de Galdeano, 1990). Nerja Cave has a vertical difference of 62 m, and its profile is practically horizontal (Fig. 1). The cave consists of numerous halls with a north–south orientation and the estimated volume

known for the cave is around 300 000 m³ (Durán, 1994). The cave presents areas with differing degrees of ventilation, and in some areas, for example, the relative humidity is high, being close to 100%, with a CO₂ concentration between 800 and 900 ppm; these features allow the precipitation of speleothems in isotopic equilibrium (Reyes et al., 1993) with seepage waters.

Several speleothem generations are present inside the cave, and some of them have been dated (Durán et al., 1993). The antiquity of the older group of speleothems exceeds 800 ka (isotopic stages 29–17), the second one belongs to isotopic stage 9 (328 ka), the third group is 260 ka old (isotopic stage 7), the fourth between 180 and 110 ka (isotopic stage 6 and 5), the fifth between 100 and 60 ka (isotopic stage 5), and the sixth group dates from the Holocene (isotopic stage 1) (Durán et al., 1993).

The focus of this study is one stalactite which was collected between the Lanza and Montaña halls (P5–P6; Fig. 1). Other samples of present-day speleothems were taken from the Cataclismo–Organos halls (P1; Fig. 1). The stalactite was sectioned perpendicular to the growth layers so that the internal lamination could be recognised easily (Fig. 2). The specimen consisted of an alternation of white and light-brown layers that can be visually differentiated. The stalactite is 170 mm long and has a diameter of 50–70 mm. It is possible to differentiate two growth stages in this sample (Fig. 2):

(1) A well laminated first stage, showing a sparry texture, white colour (7.5YR 8/1), and six growth bands (1–5 mm thick).

(2) A second stage with micritic texture and darker colour (7.5YR 7/2), showing five growth bands (2–8 mm thick).

The mineralogical composition of the samples was determined by powder X-ray diffraction (XRD) analysis using a Philips 1730 diffractometer equipped with a Cu X-ray tube and a graphite monochromator. Results from the XRD analysis show that all of the studied material consists of aragonite.

The sampling for this isotopic study was carried out using a dental-drill. Samples for stable isotope analysis of less than 20 mg were drilled. The drill

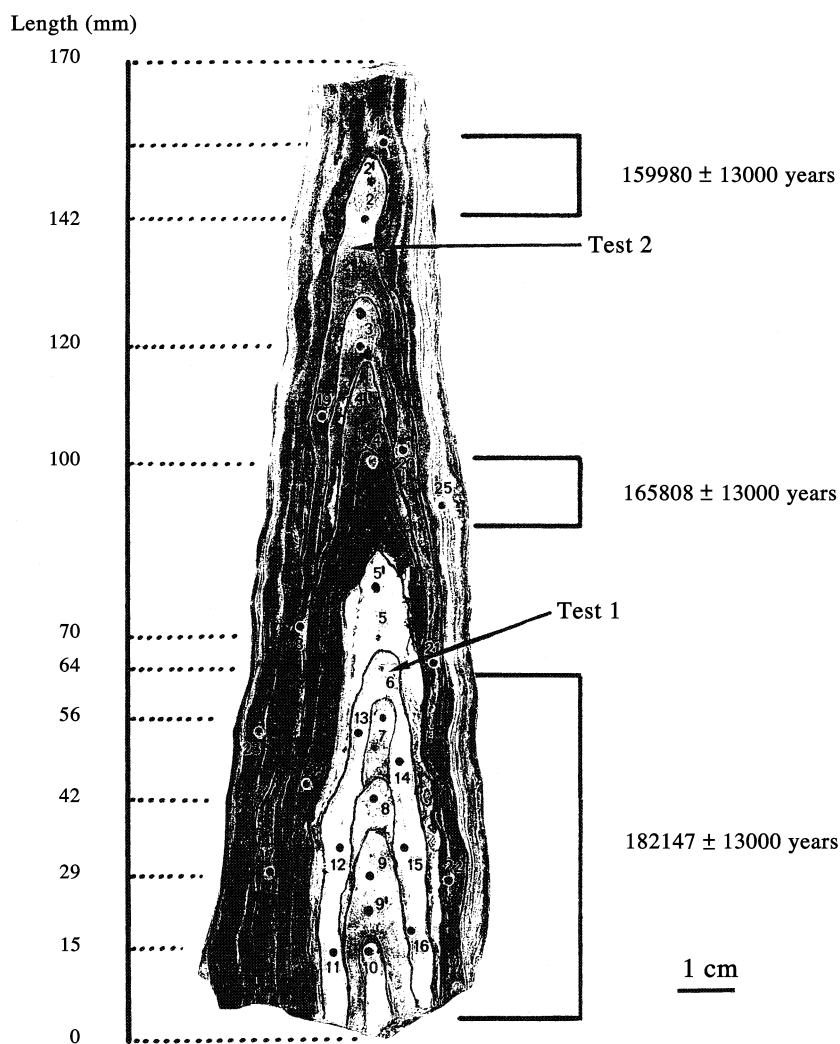


Fig. 2. Photograph of the cross-section of the 170-mm long stalactite from Nerja Cave. Sampling sites for the U-series and $\delta^{18}\text{O}$, $\delta^{13}\text{C}$ analyses are indicated. The black dots indicate the positions of the $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ samples. Tests 1 and 2 indicate the locations of the two single-layer test series

was cleaned with HCl and water in between samples, in order to avoid sample contamination. Results are reported as ‰ vs. the Pee Dee Belemnite (PDB) standard established by Craig (1961) for carbonates. Delta values were calibrated against the PDB using in-house working standards of known isotope composition (Reyes et al., 1989). Palaeotemperatures were calculated using the Grossman and Ku (1981) equation. Analytical precision was 0.1 ‰ for $\delta^{18}\text{O}$ and 0.05 ‰ for $\delta^{13}\text{C}$. The methodology used to determine $\delta^{18}\text{O}$

and $\delta^{13}\text{C}$ in the carbonate samples consisted of transforming the carbonate into CO_2 , using ultra-pure phosphoric acid at 25°C (McCrea, 1950). The CO_2 extracted was analysed using a Finnigan-Mat 251 mass spectrometer. Isotopic determinations of both $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ in carbonates were carried out in the Stable Isotope Laboratory at the Estación Experimental del Zaidín (CSIC), Granada.

For the dating of the samples the uranium-series disequilibrium method of Ivanovich and Har-

Table 1
Carbon and oxygen isotopic values along a two-growth layer of stalactite ET

Tests	$\delta^{13}\text{C}$ (‰ PDB)	$\delta^{18}\text{O}$ (‰ PDB)	Temperature (°C)
<i>Test 1</i>			
ET-11	−2.8	−3	11.5
ET-12	−2.9	−3.1	11.8
ET-13	−2.9	−3.3	12.3
ET-6	−2.7	−2.7	10.5
ET-14	−2.2	−3	11.4
ET-15	−1.5	−2.9	11.1
ET-16	−1.9	−3.3	12.3
<i>Test 2</i>			
ET-17	−2.5	−3.7	13.9
ET-18	−4.9	−3.2	12.1
ET-19	−4.7	−4	14.9
ET-2	−4.1	−3.8	14.2
ET-2'	−4.3	−3.6	13.3
ET-2''	−4.3	−3.9	14.4
ET-20	−2.9	−3.8	14.1
ET-21	−5.5	−3.5	12.9
ET-22	−5.8	−3.4	12.8

mon (1992) was used. It was performed at the Instituto Jaume Almera, CSIC, Barcelona. This method is based on the uranium solubility in natural waters while the thorium content in these natural waters is negligible. During the speleothem formation, CaCO_3 traps the uranium present in the water and the uranium-series clock is reset. The dating method is based on the fact that uranium is co-precipitated with aragonite from natural waters that are essentially free of thorium. The ^{230}Th (daughter isotope) derived from the decay of ^{234}U (parent isotope) is post-depositional. The $^{230}\text{Th}/^{234}\text{U}$ ratio is commonly used by dating of speleothems. The radiochemical procedure for U and Th isolation follows the method described by Bischoff and Fitzpatrick (1991). The radioisotope activities were determined by alpha spectrometry and the age calculation according to Rosenbauer (1991).

4. Results

4.1. Hendy test

Using Hendy' criteria (Hendy, 1971; Bar-Mat-

thews et al., 1993), the stalactite was tested along a single growth layer and was cut perpendicular to its length in order to expose its growth laminae and to permit checks for secondary alteration (Bar-Matthews et al., 1997). Two single-layer sample series were tested according to Hendy (1971) in order to determine whether the deposition had taken place under conditions of isotope equilibrium. Analysis of individual growth bands is commonly referred to as the 'Hendy test' (Linge et al., 2001a,b). Each of the series included seven or nine samples drilled along the same growth layer. The 'Hendy test 1' (samples 11, 12, 13, 6, 14, 15, and 16) corresponds to the internal layer; its results are given in Table 1 and graphically in Fig. 3. The 'Hendy test 2' (samples 17, 18, 19, 2, 2', 2'', 20, 21, and 22) corresponds to the external layer; its results are given in Table 1 and graphically in the Fig. 3. Table 1 shows the palaeotemperatures calculated using the equation for temperature-dependence for aragonite (Grossman and Ku, 1981). Palaeotemperature mean values of 11.6°C and 14.6°C were obtained for 'Hendy test 1' and 'Hendy test 2', respectively. Table 2 shows the isotopic values of present-day speleothems. In this case the $\delta^{18}\text{O}_{\text{SMOW}} = -4.6\text{‰}$ mean value of the Cataclismo–Organos halls has been used. The temperature values obtained are similar to the one prevailing at present (19–21°C).

4.2. U-Th series and stable isotopes

The calculated ages from uranium-series $^{230}\text{Th}/^{234}\text{U}$ are included in Table 3. The results for the stable isotope analysis of samples drilled along the growth axis are shown in Fig. 2 and Table 3 (dating of the base and the top of the stalactite). A uniform stalactite growth rate indicates an accretion rate of about 0.47 cm/1000 yr. The first period of growth of the stalactite occurred between 190 000 and 180 000 years ago and the second period between 170 000 and 160 000 years ago. Fig. 4 shows the variation of $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ from the base to the top of the stalactite vs. time. Overall, there is a good correlation between $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ throughout. The $\delta^{18}\text{O}$ values vary from -1.08‰ to -4.76‰ and the $\delta^{13}\text{C}$ vary from -0.23‰ to -6.89‰ . The most enriched

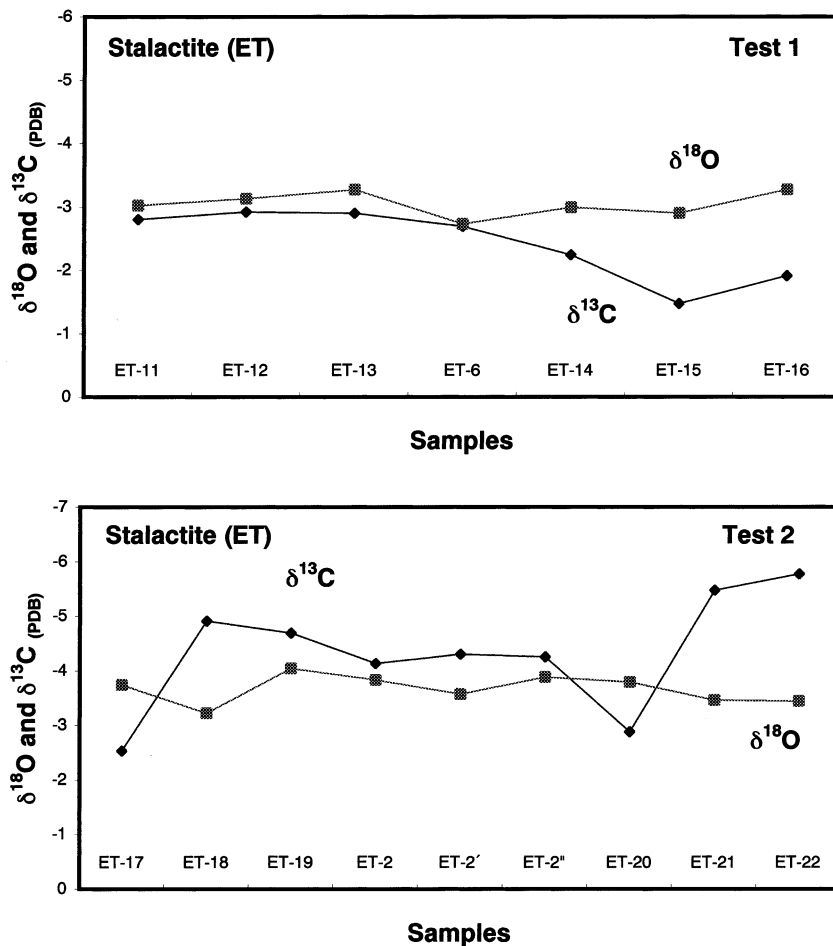


Fig. 3. Stable isotope variations along the single-growth layers, tests 1 and 2.

values of ^{18}O are found during the eldest growth period (190 000–180 000 years ago). The most depleted values are recorded during the period between 170 000 and 160 000 years ago.

5. Discussion

Several authors have indicated that CO_2 depletion together with some degree of water evaporation are responsible for ^{18}O and ^{13}C enrichment during speleothem deposition (Fornaca-Rinaldi et al., 1968; Hendy and Wilson, 1968; Duplessy et al., 1969; Fantidis and Ehhalt, 1970; Hendy, 1971; Gewalt, 1981; Schwarcz, 1986). Hendy (1971), in a study of the conditions of speleothem

formation, showed that some speleothems are deposited in oxygen isotopic equilibrium. Besides, he noted that they can be recognised by the fact that the oxygen isotopic composition measured along a single growth layer is constant; slight inconsistencies that are observed must not be correlated with changes in the isotopic composition of carbon. Talma and Vogel (1992) have suggested that isotopic equilibrium conditions can occur in the centre at the same time as kinetic/evaporative fractionation occurs at the flanks.

The 'Hendy test', carried on our material, supports the conclusion that isotopic equilibrium conditions occurred during the time of deposition in Nerja Cave. Thus its stable oxygen signal is related either to cave temperature, isotopic com-

Table 2

Carbon and oxygen isotopic values of present-day speleothems

Present-day speleothems	$\delta^{13}\text{C}$ (‰ PDB)	$\delta^{18}\text{O}$ (‰ PDB)	Temperature (°C)
CN-1	-5.9	-4.9	20.1
CN-2	-5.6	-4.7	19.4
CN-3	-5.3	-4.7	19.4
CN-4	-5.9	-5.2	21.1
CN-5	-5.6	-5.4	21.8
CN-6	-5.8	-4.6	19
CN-7	-6.7	-4.8	19.7
CN-8	-2.5	-4.9	20.1

position of dripwater, or both. In both cases, the isotopic variations of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ show no significant relation. In order to confirm this, the corresponding linear regressions between both parameters was established, giving very low correlation coefficients, $r=0.13$ and $r=-0.47$ to stages 1 and 2, respectively. According to Hendy's criteria (1971), this may suggest strong evidence of equilibrium deposition of this speleothem with the water ground, thus allowing this stalactite to be used as a palaeoclimatic indicator. The isotopic values obtained in this paper are similar to the ones reported by Jiménez de Cisneros (1994), Durán (1996), and Vesica et al. (2000) in speleothems from the southern Iberian Peninsula and the island of Mallorca.

Another way of testing the significance of the

Table 3

Carbon and oxygen isotopic values as function of time of stalactite ET

Sample	$\delta^{13}\text{C}$ (‰ PDB)	$\delta^{18}\text{O}$ (‰ PDB)	$^{230}\text{Th}/^{234}\text{U}$ (years)
ET-1	-5.8	-4.5	
ET-2	-4.1	-3.8	159 980 ± 13 000
ET-2'	-4.3	-3.6	
ET-2''	-4.3	-3.9	
ET-3	-6.4	-4.8	
ET-3'	-6.3	-4.6	
ET-4	-6.9	-4.6	
ET-4'	-6.8	-4.7	
ET-4''	-6.6	-4.8	
ET-4'''	-6.5	-4.8	
ET-5'	-1.9	-3.1	
ET-5	-1.6	-2.6	165 808 ± 13 000
ET-6	-2.7	-2.7	
ET-7	-2	-2.8	
ET-8	-2.1	-2.7	
ET-9	-1.8	-3.3	
ET-9'	-2.1	-3.5	
ET-10	-0.2	-1.1	182 147 ± 13 000

isotopes is by substituting the modern isotope values of precipitation, dripwater ($\delta^{18}\text{O}_w$) and of the stalactite ($\delta^{18}\text{O}_c$), into the equation for temperature-dependence (Grossman and Ku, 1981) for aragonite and compare these against the measured air temperature. Caballero et al. (1996) studied the isotopic composition of the Nerja Cave seepage waters and found that the mean isotopic value of the latter can be considered to

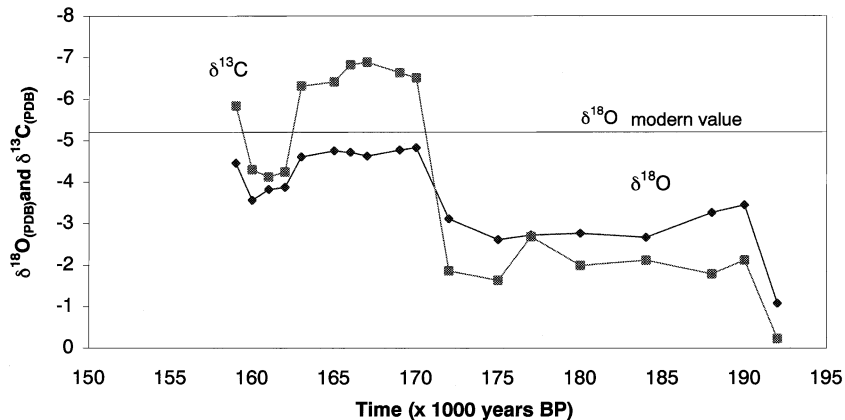


Fig. 4. Variations in $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ of speleothem plotted vs. the age of deposition. This curve can be considered to represent the curve of relative climatic change for southern Spain during Pleistocene times.

represent the mean isotopic value of the precipitation waters at the site of the cave, with a $\delta^{18}\text{O}_{\text{SMOW}} = -5.2\text{‰}$. This value is similar to the mean value obtained at the Lanza Montaña halls (Caballero et al., 1996). Therefore, we believe it can be used to determine the temperature formation of the speleothems into the equation for temperature-dependence for aragonite, which is slightly different relative to calcite:

$$T(^{\circ}\text{C}) = 19 - 3.52(\delta^{18}\text{O}_{\text{c}} - \delta^{18}\text{O}_{\text{w}}) + 0.03(\delta^{18}\text{O}_{\text{c}} - \delta^{18}\text{O}_{\text{w}})^2 \text{ (Grossman and Ku, 1981).}$$

When inserting the value $\delta^{18}\text{O}_{\text{SMOW}} = -5.2\text{‰}$ in the equation of Grossman and Ku (1981), the calculated temperatures are 11.6°C for test 1 and 14.6°C for test 2. We suggest, based on the Hendy test and the above discussion, that the past regional mean temperature can be determined from the composition of the present values of $\delta^{18}\text{O}_{\text{w}}$ and $\delta^{18}\text{O}_{\text{c}}$ and it is assumed that in the past temperature was cooler than at present.

The $\delta^{13}\text{C}$ values of speleothems can also provide important palaeoclimate information because the carbon isotopic composition of seepage water is strongly influenced by the uptake of soil- CO_2 . The $\delta^{13}\text{C}$ values in the studied stalactite show large variations (-6.83‰ to -0.23‰). These fluctuations are often explained by changes in vegetation (Dorale et al., 1992, 1998), the most obvious changes being related to photosynthetic pathways. When the correlation between $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ is good, as it is in this case where $r = 0.942$, we have a clear correlation between hydrology and vegetation above the cave.

If combined with the $^{230}\text{Th}/^{234}\text{U}$ ages, the $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values for stalactite help to reconstruct a continuous palaeoclimate record over the period between 190 000 and 160 000 years ago, being the interval during which this stalactite was formed (Fig. 4). The speleothem record contains several very sharp changes in $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$, being the signs of rapid cooling and warming trends. Two stages can be deduced from the vertical variations in the ^{18}O and ^{13}C content alongside the growth axis of the studied stalactite, a colder phase and a warmer one.

In order to interpret the $\delta^{18}\text{O}$ fluctuations in palaeoclimatic sense, the relationship between $\delta^{18}\text{O}$ and temperature must be assessed. The palaeoclimatic significance of the observed secular variation in $\delta^{18}\text{O}$ (calcite/aragonite) was demonstrated. Variations in climate will affect $\delta^{18}\text{O}$: (1) with a change in cave temperature, and (2) with decrease in air temperature at the surface. A tendency of the $\delta^{18}\text{O}$ of rain or snow and thus of dripwater to decrease (Dansgaard, 1964) causes a depletion in $\delta^{18}\text{O}$ of the calcite. Secular variations in the oxygen isotopic composition of aragonite in these deposits are largely the results of temperature-dependent changes in the isotopic fractionation between aragonite and water and of changes in the $\delta^{18}\text{O}$ of the dripwaters from which the speleothem was formed. The relatively enriched oxygen isotope ratios in the oldest part of the stalactite indicate a colder period (Fig. 4). Our palaeoclimate records are generally concordant with the data obtained in isotopic studies of deep marine sediments and glacial ice cores, confirming that we are recording global climatic events. Comparison of the palaeotemperature curve with the isotopic curves published by several authors (Emiliani, 1966; Emiliani and Schackleton, 1974; Williams et al., 1988) permits differentiation of the isotopic stage boundary 7/6 (end of 7 and beginning of 6). During this interval, the phreatic level of the waters in the cave decreased and a dry and cold interval was started which changed to a new interval (Durán, 1996). These results are concordant with the ones obtained in this work and confirm the alteration of a colder stage followed by a warmer one. Gascoyne (1981) showed a frequency distribution for speleothems dated by the $^{230}\text{Th}/^{234}\text{U}$ method from northwest England which clearly demonstrated significant growth during isotopic stage 7, 5, 3 and 1. Durán (1989) also showed a frequency distribution of speleothems for the last 400 000 years in the Iberian Peninsula with maximum growth in isotopic stages 9, 7, 6, 5 and 3.

6. Conclusions

The speleothem record presented here shows

significant shifts in $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ isotopic values that suggest changes in climate during the interval studied. The palaeoclimatic record from this stalactite contributes to enhanced understanding of climate variability in this region of southern Spain.

We present a curve of relative climatic changes in Nerja Cave, which forms the expression of climatic change for the southern Iberian Peninsula some time during the Middle Pleistocene. Two stages can be deduced from the variations in the ^{18}O and ^{13}C content alongside the growth axis of the studied stalactite, i.e. a colder and a warmer phase (12°C and 15°C). The most enriched values of ^{18}O in the studied stalactite are found during the eldest period of growth (190 000–180 000 years ago). The most depleted values are recorded between 170 000 and 160 000 years ago.

The $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ measurements of aragonitic speleothems indicate that deposition in the Nerja Cave occurred under conditions of isotopic equilibrium. This isotopic equilibrium was confirmed by the low correlation between the values of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ in the single layer.

The oxygen and carbon isotope variations measured on the $^{230}\text{Th}/^{234}\text{U}$ dated stalactite from Nerja Cave indicate that during the Middle Pleistocene temperature was cooler than at present in the south of Spain.

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