# 1. DRY AND DUSTY PERIODS IN THE MESOPOTAMIA REGION FROM 2560-2450 BC AND 2310-2020 BC, INFERRED FROM STALAGMITE CHEMISTRY

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## ABSTRACT

Human-timescale palaeo-climate records are a critical tool for exploring the relationship between past societies and their environment. I present here a stalagmite from the southern side of the Alborz mountains in northern Iran, a region directly impacted by dust events originating in the Mesopotamia region. The stalagmite formed continuously through the 3rd mill. BC, producing a 1500-y climate and environmental record able to be sampled up to annual resolution. The stalagmite record suggests a significant increase in Mesopotamia-sourced dust events from 2560-2450 BC (110 y) and 2310-2020 BC (290 y), relative to the rest of the 3rd mill. BC. The events demonstrate threshold behaviour in dustiness of the Mesopotamia region, due to either enhanced aridity, stronger winds, or change in soil properties or vegetation cover. Additional information on regional rainfall amount proceeding and during the dusty periods is found in the same Iranian stalagmite and in other East Mediterranean speleothem records, which indicate regional and local dry conditions coincident with the dust events.

**Keywords:** Speleothem; Mg/Ca ratio; stable isotopes; Aeolian dust; Palaeo-climate records; Mesopotamian archaeology; environmental changes; arid periods; third millennium BC; 4.2 kyr event; population dynamics; landscape change; urbanization; climate impacts

## INTRODUCTION

Stalagmites, calcium carbonate structures formed in caves, contain multiple geochemical proxies which record past changes to the environment on local and regional scales. In this chapter I review the fundamental features of the stalagmite archive which can be used in climate and environment reconstructions. I then discuss our current understanding of the climate and environmental conditions of the 3rd millennium BC in interior Mesopotamia based on a precisely-dated multi-proxy north Iran stalagmite record, as well as other palaeo-climate records in the region. The north Iran stalagmite was collected from an area which is directly impacted by both the region's westerly winter storm fronts as well as the summer Shamal dust storms, an ideal location for recording past rainfall and dust events which originated in the interior Mesopotamia region.

## SPELEOTHEM ARCHIVES

Speleothems are mineral deposits formed within caves. Stalagmites, a type of speleothem, are dripstones formed from dripping water, which grow vertically from the cave floor. Stalagmites are the most common speleothem used to investigate past climatic and environmental change and are found in most regions of the world.

The drip waters which grow the stalagmite layers originate as meteoric water. As meteoric water flows through the soil above the cave, and then into the limestone, it acquires various elements, such as calcium, carbon, and magnesium. Some elements contain chemical signatures related to surface environment changes, such as rainfall or dust events. These elements are captured within the growing stalagmite layers, thus forming a proxy record of past environment and climate change which is preserved for thousands of years.

A variety of microanalytical techniques developed over the past two decades now allow for high-resolution sampling of stalagmite layers for reconstructing past climate. Annual or even sub-annual stalagmite climate records are now possible when stalagmite vertical growth rate is ~100 µm/yr or greater, which is common in warm environments. Sampling techniques include micromilling (up to ~50 µm spatial resolution), laser ablation (up to ~10 µm spatial resolution), and secondary ionization mass spectrometry (up to ~10 µm spatial resolution) (see Baldini et al. 2021 and citations within). In a global synthesis of annually laminated stalagmites from twenty-three caves across six continents, the average stalagmite vertical growth is 93 µm/yr (Baker et al. 2021).

## STALAGMITE CLIMATE AND ENVIRONMENTAL PROXIES

Several geochemical features in the stalagmite layers are used to construct proxy records, which are used to infer past rainfall, temperature, and/or environmental events. In this section I review common interpretations of four of the more frequently used proxies measured in sequential stalagmite layers: oxygen isotope ratios, carbon isotope ratios, alkaline element-to-calcium ratios, and trace element concentrations.

### Oxygen isotope ratios

Stable oxygen isotopes $^{18}O$ and $^{16}O$ fractionate during phase changes (e.g., water vapor to liquid water), and hydrologic processes associated with these phase changes correlate with climate variables, such as temperature or rainfall. The ratio of $^{18}O$ to $^{16}O$ in samples is used to infer past fractionation processes. Oxygen isotope ratios are expressed in delta-notation, relative to a standard, following the equation,

$$\delta^{18}O = \frac{\left({}^{18O}/_{16O}\right)_{sample} - \left({}^{18O}/_{16O}\right)_{standard}}{\left({}^{18O}/_{16O}\right)_{standard}} \times 1000$$

Processes associated with oxygen isotope fractionation in the hydrologic cycle are discussed in detail in Lachniet (2009) and are briefly summarized here.

The first control on $\delta^{18}O$ in the hydrologic cycle is the isotopic composition of the source water from which atmospheric vapor originates. Seawater $\delta^{18}O$ of the seas surrounding the Middle East region varies both spatially (e.g., LeGrande et al. 2006) and temporally. Processes that affect seawater $\delta^{18}O$ include precipitation and evaporation, river runoff, ocean and sea circulation patterns, and land ice expansion or melt over longer timescales. Planktonic foraminifera calcite $\delta^{18}O$ in some cases can be used to estimate surface water $\delta^{18}O$ variations in the past (e.g., Almogi-Labin et al. 2009).

Oxygen isotopes are fractionated during evaporation from the source water, and again during raindrop formation from the atmospheric vapor. $^{18}O$ is preferentially incorporated into the liquid phase during rainout, lowering the $\delta^{18}O$ value of the remaining vapor in the atmosphere. Hence through ongoing rainout from a vapor source, the rainwater and remaining vapor $\delta^{18}O$ progressively lower as the fraction of vapor remaining decreases (Dansgaard 1964). This process is known as Rayleigh distillation, and is temperature dependent, as vapor condensation requires air mass cooling (Dansgaard 1964). Processes that can cool the air source include orographic uplift, advection, convection, convergence, or frontal lifting. High $\delta^{18}O$ moisture may also be recycled back to the atmosphere from evaporation of soil, lakes, and rivers. Air parcels on multiple trajectory paths all with unique rainout histories may

converge and mix prior to arrival over a cave site. Variable air parcel trajectories through the year can cause seasonal $\delta^{18}O$ signatures in rainfall.

The local, at-site $\delta^{18}O_{rain}$ 'amount effect' is the observed lowering of $\delta^{18}O_{rain}$ values with increased rainfall amount over an averaged timespan (Dansgaard 1964; Rozanski et al. 1993; Risi et al. 2008). The 'amount effect' is documented most strongly at tropical island sites but has also been observed in the extratropics (Bar-Matthews et al. 2003). The 'amount effect' is explained by two processes: re-evaporation of falling rain and the diffusive exchanges with the surrounding vapor, and recycling of subcloud layer vapor feeding the convective system by convective fluxes (Risi et al. 2008).

Meteoric water slowly travels through the surface soil and carbonate bedrock in the vadose zone (above the water table), where some of the water may evaporate (raising the $\delta^{18}O_{water}$ value), and/or mix with existing waters in the karst system (diluting the $\delta^{18}O_{rain}$ signal amplitude). Notably, evaporative fractionation of $\delta^{18}O_{water}$ in the soil zone, as well as 'selective recharge' – allowance of only high intensity, lower $\delta^{18}O_{rain}$ storm event waters to progress through the karst to the cave chamber – are highly likely in water-limited environments (Pape et al. 2010; Cuthbert et al. 2014; Markowska et al. 2016).

Calcium carbonate oxygen forms from aqueous dissolved inorganic carbonate (DIC) oxygen. The amount of exchange time needed for dripwater $\delta^{18}O_{H2O}$ to equilibrate with $\delta^{18}O_{DIC}$ is on the order of several 10,000 s (Dreybrodt & Scholz, 2011). As such, a sufficiently long time (hours) is needed for the $\delta^{18}O$ of the rainwater to be reflected in the drip water carbonate species. The residence time of a drip water varies from site to site, and drip to drip, but waters forming stalagmites are from seepage flow or fissure flow, not fast conduit flow, and thus almost all stalagmite-forming drips have karst residence times longer than several hours.

Given the multitude of fractionation processes that affect rainwater/dripwater/stalagmite $\delta^{18}O$, a modern empirical relationship between recorded climate parameters (such as rainfall amount and temperature) and measured collected rainwater $\delta^{18}O$, or modern grown calcite in the cave, is often relied upon to determine a climate-proxy relationship. Multiple records from the same cave or from regional caves which demonstrate reproducible stalagmite $\delta^{18}O$ signals add confidence to the regional nature of the records and the interpretation as a climate signal.

## Carbon isotope ratios

The ratio of stable carbon isotopes $^{13}C$ to $^{12}C$ within the surface environment and within the karst system is dependent on several local processes. Carbon isotopes are also expressed in delta-notation, relative to a standard, following the equation,

$$\delta^{13}C = \frac{\left({}^{13C}/_{12C}\right)_{sample} - \left({}^{13C}/_{12C}\right)_{standard}}{\left({}^{13C}/_{12C}\right)_{standard}} \times 1000$$

A discussion of the main controls on stalagmite $\delta^{13}C$ is provided in Fohlmeister et al. (2020) and discussed briefly here.

Carbon isotopes in stalagmites are originally derived from carbon dioxide gas in the soil and upper karst air, and from carbonate bedrock which is dissolved by seeping waters. The $\delta^{13}C$ value in $CO_2$ is dependent on the vegetation type – C3 plants produce lower $\delta^{13}C$ values ($\delta^{13}C_{CO2}$ ~ -27 to -24‰) than C4 plants ($\delta^{13}C_{CO2}$ ~ -14 to -12‰) (Cerling 1984) – and reflects the amount of vegetation and amount of biologic and microbial activity in the surface environment. More biologic and microbial activity leads to a greater proportion of carbon derived from plants and microbes, which has a lower $\delta^{13}C$ value than atmospheric $CO_2$ (Holocene $\delta^{13}C_{CO2}$ ~ -6.4‰, Elsig et al. 2009). Thus, relatively low stalagmite $\delta^{13}C$ may be reflective of surface environmental conditions which are more favourable to biologic and microbial activity, such as warm and wet. Relatively low stalagmite $\delta^{13}C$ may also be interpreted as a hypothesized shift to more C3 surface vegetation which is not water stressed, and/or a smaller proportion of C4 vegetation.

As $CO_2$ degasses from the drip water, temperature-dependent fractionation processes occur, in which the lighter $^{12}C$ isotope is preferentially incorporated into gaseous $CO_2$. The degassing process progressively raises $\delta^{13}C_{DIC}$ and $\delta^{13}C_{CaCO3}$ through ongoing carbonate precipitation, which can occur along any drip path open to the low-$pCO_2$ cave atmosphere. Carbonate precipitation prior to the drip water reaching a stalagmite is known as prior calcite precipitation or prior aragonite precipitation (PCP/PAP). The degree of PCP/PAP and the magnitude of progressive $\delta^{13}C$ increase along the drip path is dependent on i) the $pCO_2$ gradient between the water phase and gaseous phase, and ii) the length of the period the water is in contact with low $pCO_2$ air before reaching the stalagmite tip. Generally, greater PCP/PAP, and consequently higher $\delta^{13}C$ values, are interpreted as drier environmental conditions (slower and less water flow allows for more PCP). Other proxies, such as alkaline elements (see below), are also affected by PCP/PAP and are used together with stalagmite $\delta^{13}C$ records to form a stalagmite multi-proxy climate or environmental interpretation.

### Ratio of alkaline elements to Ca (Mg/Ca, Sr/Ca, Ba/Ca)

PCP has a progressive impact on drip water X/Ca ratios and subsequent calcite X/Ca ratios, where X is magnesium (Mg), strontium (Sr), or barium (Ba) (e.g., Tremaine et al. 2013). Mg, Sr, and Ba are soluble minor elements found in bedrock, and are added to cave drip water solution as cations alongside calcium (Ca) during bedrock dissolution. When the drip water enters an open environment and carbonate begins to precipitate, the Ca cation is preferentially incorporated into the crystal structure relative to Sr and Ba, and to an even larger extent relative to Mg (e.g., Day et al. 2013; Wassenburg et al. 2020). Similar to the Rayleigh distillation process in which remaining vapor and subsequent rainwater $\delta^{18}O$ values progressively decrease due to the preferential rainout of $^{18}O$ relative to $^{16}O$, the PCP process progressively increases the X/Ca ratio of drip water cations and subsequent calcite formation due to the preferential incorporation of Ca relative to Mg, Sr, and Ba in the carbonate structure. As greater PCP leads to higher X/Ca ratios, and greater PCP is interpreted as reflective of drier environmental conditions (slower and less water flow allows for more PCP), higher X/Ca ratios in the stalagmite are interpreted to reflect drier environmental conditions.

### Aeolian deposition on karst surface

The leaching of isotopes or elements from aerosol deposits by meteoric water or complete dissolution of aerosol deposits can transport chemical evidence of aerosol activity above the cave to the stalagmite. Elemental or isotopic ratios of aerosol particulates must be distinctive from the background carbonate bedrock chemistry in order for these signals to be recognizable in a stalagmite record. A small number of records of dust events (e.g., Goede et al. 1998; Frumkin et al. 2004; Ünal-Imer et al. 2016; Carolin et al. 2019) and volcanic ash deposits (e.g., Siklósy et al. 2009; Braun-Badertscher et al. 2014; Jamieson et al. 2015) constructed from stalagmite proxies are found in the literature.

## STALAGMITE GEOCHRONOLOGY

Stalagmite layers are dated using the U-Th disequilibrium method, which is briefly described here. Stalagmite layers precipitate with a trace amount of the radioactive uranium nuclide $^{234}U$ (~$10^{-11}$ to $10^{-10}$ g/g) acquired from the dissolved carbonate bedrock. $^{234}U$ is soluble and is transported in the drip water to the top of the stalagmite. The karst rock dissolution and subsequent stalagmite precipitation process fractionates $^{234}U$ and $^{230}Th$, members of the $^{238}U$-$^{206}Pb$ decay chain, resulting in non-steady-state radiogenic conditions. Over thousands of years post this drip water dissolution fractionation process and stalagmite layer formation, all members within the $^{238}U$-$^{206}Pb$ decay chain return to equilibrium state, as $^{234}U$ decays to $^{230}Th$ ($^{234}U$ half-life 245,600 years, Cheng et al. 2013), which decays to $^{226}Ra$ ($^{230}Th$ half-life 75,580 years, Cheng et al. 2013), followed by the remaining decay chain members' decay to stable $^{206}Pb$. The U-Th age equation is the solution to the set of differential equations which model the multi-member return to steady-state equilibrium through growth and decay following the drip water dissolution fractionation process (Bateman 1910; see also Ivanovich et al. 1992). By measuring $^{234}U/^{238}U$ and $^{230}Th/^{238}U$ activity ratios in a stalagmite carbonate sample using a multi-collector inductively-coupled plasma mass spectrometer (e.g., Hellstrom 2003; Hoffmann et al. 2007; Cheng et al. 2013), the age of the initial bedrock dissolution and subsequent precipitation of a particular stalagmite layer can be derived iteratively using the U-Th age equation.

Approximately 20-200 mg calcite is usually required for each age analysis, depending on the uranium concentration in a particular stalagmite and the sensitivity of the mass spectrometer. Aragonite stalagmites typically have much higher U concentration, and therefore require less sample, ~5-20 mg. Age precision on stalagmite subsamples using the U-Th method can be achieved in the per mil range, provided there is a significant concentration of radionuclides for measurement (generally at least 100 ppb U in the stalagmite).

Age accuracy in stalagmite U-Th dating is dependent on the level of external contaminants which are captured within the stalagmite crystal structure during precipitation. Detrital and organic matter particulates contain trace amounts of U and Th nuclides, with thorium contaminants largely hindering U-Th age measurements. It is difficult to separate detrital or organic matter contaminants physically or chemically from the stalagmite during U-Th age sampling, which involves milling or slicing ~20-200 mg of sample along a growth layer. Estimated contaminant radiogenic $^{230}Th$ values are therefore subtracted from the total measured radiogenic $^{230}Th$ values post-analysis using an estimated ratio of $^{230}Th$ to $^{232}Th$ at age zero (Cheng et al. 2000). $^{232}Th$ is the stable, abundant isotope of thorium, whose presence in the stalagmite is from original contamination only, not radiogenic growth, and is therefore used as a contaminant tracer.

The average $^{230}Th/^{232}Th$ atomic ratio of bulk earth surface silicates, commonly used as a best estimate to correct for detrital Th contamination in stalagmite age samples, is ~4 ppm. Studies have suggested local detrital $^{230}Th/^{232}Th$ ratios within various cave sites to more likely range from 4 to 160 ppm, with some measurements and calculations suggesting that the ratio in the detrital matter deposited within the stalagmite may have changed over time (e.g., Hellstrom 2006; Ridley et al. 2015; Carolin et al. 2016). Overestimating the detrital $^{230}Th/^{232}Th$ ratio results in calculated U-Th ages too young, while underestimating the detrital $^{230}Th/^{232}Th$ ratio results in calculated ages too old. Age inaccuracies due to an incorrect detrital $^{230}Th/^{232}Th$ ratio estimate may range from tens to thousands of years, depending on the amount of detrital contaminant in the stalagmite. High confidence in U-Th age accuracy should therefore only be associated with stalagmites with low contamination reflected by low $^{232}Th$, and relatively high radiogenic $^{230}Th$ for measuring.

## Westerlies storm fronts and Shamal dust events

Precipitation in the Mesopotamia region, as well as the Taurus to Zagros Mountain ranges surrounding it to the north and east, occurs predominantly in the winter and spring seasons (Evans et al. 2004; 2006; Raziei et al. 2012; 2013). Moisture is transported largely from the Eastern Mediterranean, but may also be sourced from the Red Sea, Gulf, and the Arabian Sea, with mid-tropospheric troughs forming over the Eastern Mediterranean, Syria, Jordan, and Iraq (Evans et al. 2004; 2006; Raziei et al. 2012; 2013). Orographic uplift over the Zagros and Taurus mountains leads to heavy precipitation, some of which crosses over the Zagros mountains into northwestern Iran (Fig. 9.1a). The winter rains provide important water resources to northern and southern Mesopotamia, both in rain fed regions and through transport by the Tigris and Euphrates rivers.

Dust storms in the region originate in the Tigris-Euphrates alluvial plains (Cao et al. 2015), and occur in both the summer and winter seasons (Hamidi et al. 2013). The summer Shamal winds, strong northwesterlies near the surface, transport dust across Mesopotamia and the Persian Gulf, as well as parts of the Arabian Peninsula (e.g., Middleton 1986; Notaro et al. 2015; Yu et al. 2016), and west and central Iran (Givehchi et al. 2013; Hamidi et al. 2013) (Fig. 9.1b).

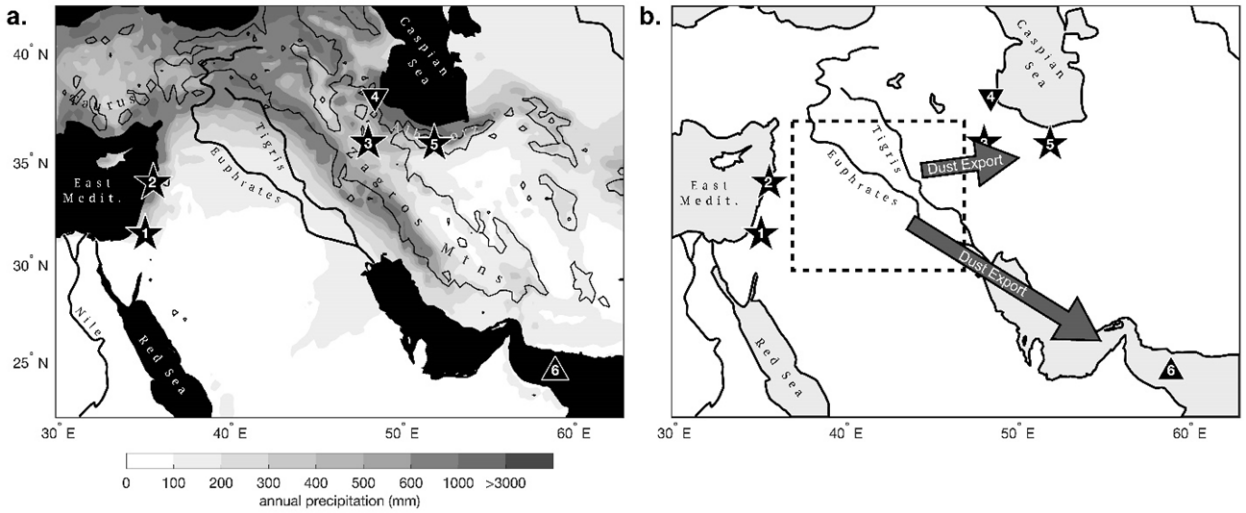


**Figure 1.1: Annual precipitation in Mesopotamia and the surrounding region and dust export from the Tigris-Euphrates alluvial plains. (a) Shading indicates long-term average annual precipitation 1979-2021 (Hersbach et al. 2019). Note coloring increments are nonlinear above 600 mm annual rainfall. Contours show 1500 masl elevation. Major mountain ranges, seas, and rivers (modern location) are labeled. Location of paleoclimate records discussed in the text are indicated by numbers in markers, 1: Soreq (Bar-Matthews et al. 2011), 2: Jeita (Cheng et al. 2015), 3: KT-3 (Andrews et al. 2020), 4: Lake Neor peat bog (Sharifi et al., 2015), 5: GZ14-1 (Carolin et al. 2019), 6: Gulf of Oman marine sediment core (Cullen et al. 2000) and coral (Watanabe et al., 2019). (b) Arrows indicating direction of summer Shamal winds and dust storm export to neighboring regions. Numbered markers are the same as (a). Image by author**

## Stalagmite GZ14-1: An archive of precisely-dated Mesopotamia dust events

Gol-e zard cave (35.84°N, 52.00°E) is located on the southern side of the Alborz mountains, ~50 km East-North-East of Tehran (Fig. 9.1 marker 5). The cave entrance is at 2,535 masl, ~1,400-1,700 m above the surrounding Iranian plateau to the south. A thick blanket of breccia covers the region, which is Tertiary in age. There is no exposure of bedrock (geological formation) at the site. Vegetation above the cave consists of sparse alpine brushes. Inside the cave there are active drips, and many damaged stalagmites. The cave site is today visited by tourists and locals.

Stalagmite sample GZ14-1 was collected at the back of the cave, ~5 m above the river level, in May 2014. There were active drips in the area when the stalagmite sample was collected. Humidity appeared near 100% in the back of cave where GZ14-1 was collected during the field site visit.

Limestone rock samples chipped from areas above the cave, as well as one fast-drip water sample (<2 secs between drips), were analysed for trace elements and stable isotopes at the University of Oxford. The rock samples are predominantly calcite. Some bedrock samples contained a percentage of quartz and some contained trace amounts of gypsum.

Stalagmite GZ14-1 was sliced in half vertically using a tile saw and slabbed. A high-resolution scan of Sample GZ14-1 with U-Th age sampling and proxy record sampling marks is shown in Fig. 9.2. XRD analysis confirms the sample is calcite.

Calcite samples of 80 to 230 mg were drilled using a hand-held dental drill at various distances from the top of the stalagmite for U-Th age analysis. Sample drill depth into the stalagmite slab was ∼2 mm to 3 mm. U and Th isotopes were measured using a Nu Plasma multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) at the University of Oxford (detailed procedures in Carolin et al. 2019). Individual ages and 95% confidence intervals were calculated using an in-house Monte Carlo script that incorporates chemical blank errors, analytical uncertainties, and an estimated initial $^{230}Th/^{232}Th$ ratio range of 2-10 ppm (uniform distribution). The age model with 68% and 95% confidence ranges was produced using the OxCal Version 4.3 Poisson process deposition model [$k_0$= 1 $cm^{-1}$, $\log_{10}(k/k_0) = U(-2,2)$], with interpolation (Bronk Ramsey 2008; Bronk Ramsey et al. 2013).

The working half of GZ14-1 was mounted to a NewWave micromill for proxy powder sample milling. Samples were milled in a trench along the stalagmite growth axis at 500 and 250 µm step intervals for initial low-resolution sampling, followed by 100- and 50-µm step intervals for high-resolution sampling. Powders ∼500 to 1,000 µg were collected individually using aluminium spatulas and stored in compressed air-cleaned plastic 2 mL centrifuge tubes.

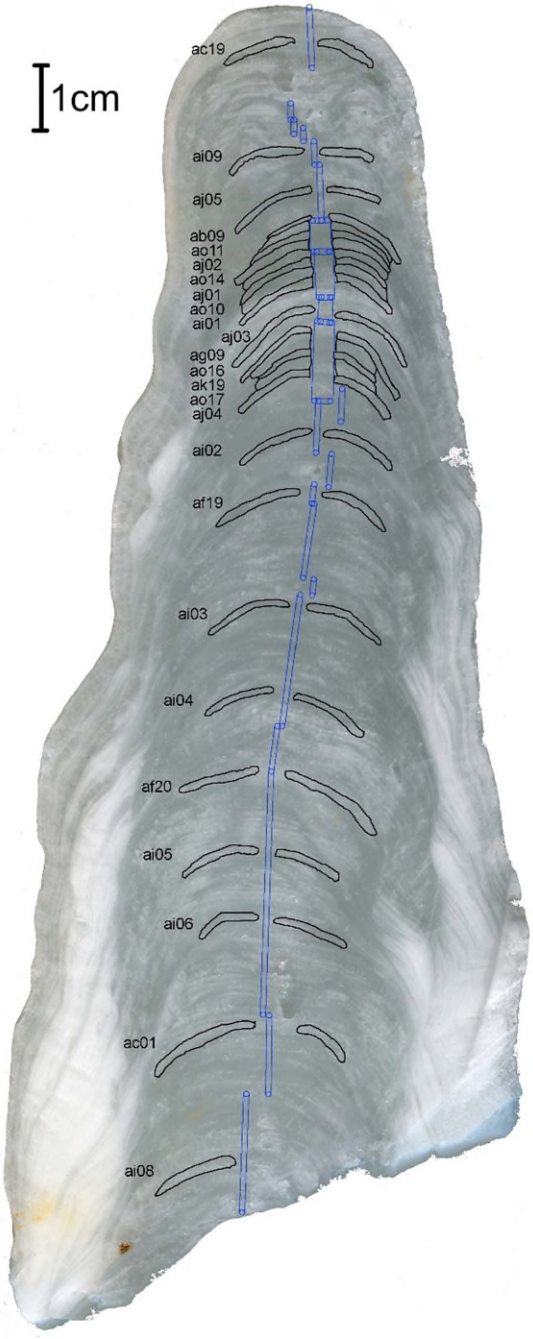


**Figure 1.2: High-resolution scanned image of Stalagmite GZ14-1. Milled trench-style combined stable isotope and trace element powder sampling along growth axis shown. There is a gap near the top of the stalagmite in milled samples as the stalagmite became too porous for sampling. Individual U-series age trenches alongside growth axis shown with labeled sample number. From Carolin et al. 2019, Figure S7. Reproduced with permission**

Between 80- to 100-µg of calcite was removed from the storage tubes and analysed for a suite of elements using a Thermo Scientific Element 2 ICP-MS at the University of Oxford. Calibration standards bracketed every 20 samples to correct for instrument drift. Trace element-to-Ca ratios were determined using the 'ratio' method (Rosenthal et al. 1999). A secondary standard was measured every 10 samples to calculate precision and accuracy (average relative accuracy error 5%, average relative precision error 2%, 1σ). Between 30- to 60-µg of calcite was removed from the storage tubes and analysed for $\delta^{18}O$ and $\delta^{13}C$ using a Thermo Scientific Delta V isotope ratio mass spectrometer (IRMS) coupled to a Kiel V carbonate device at the University of Oxford. Each batch of samples was measured with calibration standards and evenly scattered secondary standards. Precision and accuracy were calculated using the secondary standards' long-term average and standard deviation ($\delta^{18}O$ = ±0.07‰, $\delta^{13}C$ = ±0.05‰, 1σ). All GZ14-1 measured data are available at https://www.ncdc.noaa.gov/paleo/study/28710.

The U-Th age model and thin section analysis indicate that stalagmite GZ14-1 grew with no recognizable vertical growth hiatuses between 3210-1730 BC (Carolin et al. 2019) (Fig. 9.3). The age model has an average age error of 31 y (1σ) (Table 9.1), with larger errors during slower vertical growth periods. GZ14-1 grew relatively quickly throughout the majority of the record (>130 µm/y). However, in two periods, the vertical growth rate drops below 100 µm/y, to ~15-20 µm/y: 2620-2430 BC and 2370-1960 BC (start to end dates) (Carolin et al. 2019) (Fig. 9.4). The decreased growth rate is suggestive of drier local conditions; however, other factors not directly related to rainfall, such as temperature, interval between drips, and drip water chemistry, can also affect stalagmite vertical growth rate (see text above).

| Sample ID | Distance from top (mm) | U (ppb) | $^{230}Th/^{232}Th$ (ppm) | Corrected age (BCE) | Age error, 1s (y) | Detrital Th correction applied (y) |
|---|---|---|---|---|---|---|
| ac19 | 6.5 | 98 | 680 | 1730 | 40 | -32 |
| ai09 | 23.8 | 144 | 4500 | 1890 | 20 | -5 |
| aj05 | 29.9 | 140 | 6100 | 1890 | 40 | -4 |
| ab09 | 34.5 | 158 | 1440 | 1940 | 50 | -15 |
| ao11 | 36.0 | 88 | 5000 | 2080 | 90 | -5 |
| ao08 | 37.4 | 99 | 3800 | 2240 | 110 | -6 |
| aj02 | 38.9 | 98 | 30000 | 2210 | 50 | -1 |
| ao14 | 40.2 | 95 | 15000 | 2200 | 90 | -2 |
| ~~aj01~~ | ~~41.5~~ | ~~106~~ | ~~73~~ | ~~2100~~ | ~~150~~ | ~~-350~~ |
| ao10 | 42.6 | 130 | 6600 | 2430 | 40 | -4 |
| ai01 | 43.8 | 140 | 8200 | 2370 | 20 | -3 |
| aj03 | 48.7 | 165 | 2700 | 2410 | 30 | -9 |
| ag09 | 52.0 | 155 | 2410 | 2420 | 40 | -10 |
| ao16 | 53.8 | 120 | 1190 | 2530 | 40 | -21 |
| ak19 | 55.5 | 115 | 1690 | 2640 | 80 | -15 |
| ao17 | 57.2 | 114 | 16000 | 2560 | 60 | -2 |
| aj04 | 58.9 | 139 | 28000 | 2630 | 30 | -1 |
| ai02 | 67.0 | 111 | 6500 | 2680 | 30 | -4 |
| af19 | 77.0 | 103 | 5900 | 2780 | 50 | -4 |
| ai03 | 94.8 | 102 | 1850 | 2830 | 30 | -15 |
| ai04 | 109.0 | 114 | 4200 | 2930 | 30 | -7 |
| af20 | 121.0 | 122 | 1180 | 3020 | 40 | -23 |
| ai05 | 134.0 | 96 | 8800 | 3020 | 30 | -3 |
| ai06 | 144.7 | 98 | 9200 | 3010 | 30 | -3 |
| ac01 | 161.1 | 122 | 810 | 3130 | 50 | -35 |
| ai08 | 183.3 | 92 | 4500 | 3210 | 40 | -6 |

**Table 1.1: Stalagmite GZ14-1 measured U/Th isotope activity ratios and calculated ages. Distance is distance from the top of the stalagmite, with smaller distance corresponding with younger age. U (ppb) is uranium concentration in calcite. 230Th/232Th in the sample provides a metric for judging the magnitude of detrital contamination – small values indicate relatively large detrital contamination. The detrital 230Th/232Th estimate (at age 0, the time of deposition) used for the age correction is a range of 1-10 ppm, uniform distribution. Corrected ages were calculated using the radiogenic isotope half-lives found in Cheng et al. 2013. Age errors are calculated using an internally developed Monte Carlo simulation. One sample (sample aj01) had exceptionally large detrital contamination and the corrected age was out of chronological order with the neighboring; hence this sample has been discarded from the final age model. Data provided in Carolin et al. 2019**

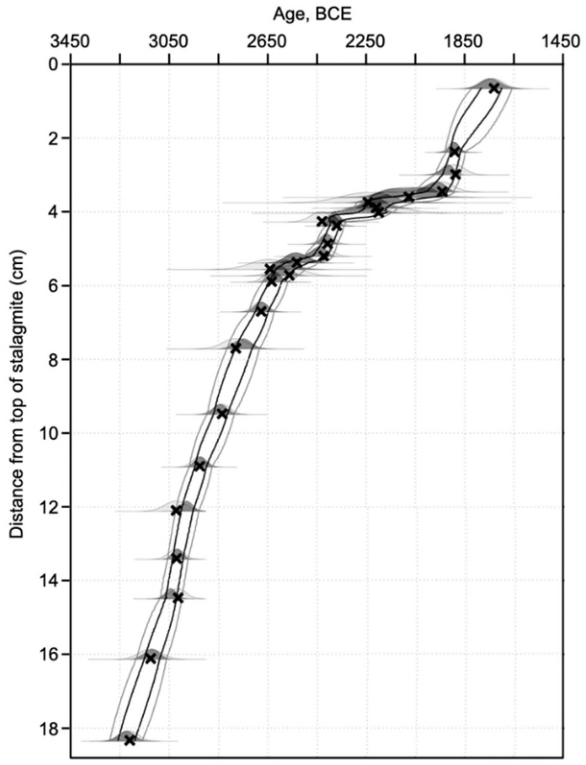


**Figure 1.3: GZ14-1 age v. depth plot with OxCal Poisson process deposition age model 68% (black) and 95% (dark gray) confidence ranges. Individual un-modeled U-Th ages are plotted as black 'x' shapes. Individual modeled age distributions are shown in dark gray (68%) and light gray (95%). Adapted from Carolin et al. 2019. Reproduced with permission**

The ratio of Mg/Ca in GZ14-1 exhibits sudden changes coincident with the periods of slow vertical growth. Mg/Ca abruptly increases at the start of two periods, lasting from 2560-2450 BC and 2310-2020 BC (Carolin et al. 2019) (Fig. 9.4). The age error on the start and end of these periods ranges between 40 y and 70 y (1σ), which is relatively large in comparison to most individual U-Th ages in GZ14-1. This is due to the very slow growth rate of this interval, and difficulty in U-Th sample extraction with limited sample size. The abrupt increases in GZ14-1 Mg/Ca are interpreted as increases in Mesopotamia-sourced dust events (see section 1.1.4). Modern dust events which reach Tehran are sourced from the deserts of Syria and Iraq (Givehchi et al. 2013). Mineralogical studies show that dust sourced from the Mesopotamia region contains dolomite (Hojati et al. 2012; Ahmady-Birgani 2015), which has a large Mg/Ca ratio. Mg leached off the dust by meteoric water above the cave would add extra Mg cations to the drip waters, of which a portion would precipitate in the stalagmite calcite. A greater dust flux and deposition over the Gol-e zard cave site is thus suggested to have resulted in shifts to larger Mg/Ca values in the stalagmite record (Carolin et al. 2019).

During the two periods of heightened Mg/Ca, GZ14-1 $\delta^{18}O$ exhibits a gradual increase up to 1‰ higher followed by a decrease back to baseline values (Carolin et al. 2019) (Fig. 9.4). Collected Tehran rainwater (50 km SW of the cave site) between 1962 and 1972 (International Atomic Energy Agency's Global Network of Isotopes in Precipitation (GNIP) database) demonstrates a negative correlation between $\delta^{18}O_{rain}$ and annual average precipitation amount (51% of the variance in $\delta^{18}O_{precip}$ is predictable from precipitation amount) (Carolin et al. 2019). The increased stalagmite $\delta^{18}O$ values are therefore interpreted as periods of drier conditions in the area. GZ14-1 $\delta^{13}C$ does not show significant changes coincident with either the abrupt shifts in Mg/Ca or the more gradual $\delta^{18}O$ increases. This suggests that the karst environment, such as surface vegetation type or amount of microbial activity in the soil, of this high elevation (>2500 masl) cave did not change significantly as a result of the drier conditions at lower elevations.

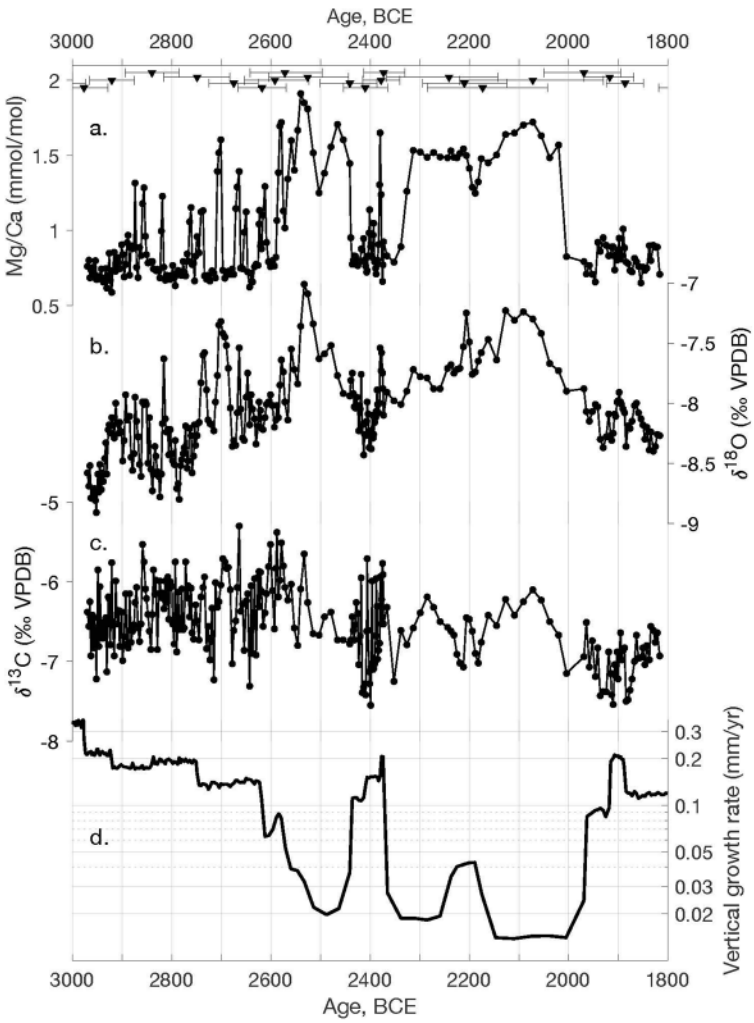


**Figure 1.4: GZ14-1 (a) Mg/Ca (millimoles per mole), (b) δ18O, and (c) δ13C (‰) plotted above the (d) mean vertical growth rate. The vertical growth rate is plotted as a 20 y moving average of the annually interpolated OxCal-modeled mean vertical growth rate. Mg/Ca is interpreted as a record of Mesopotamia region dust activity, with higher values indicating greater dust deposits due to strength of dust storm and/or frequency of dust events. δ18O is interpreted as local and/or upstream trajectory rainfall amount, with higher δ18O values related to drier conditions. Sampling frequency is 2-5 yrs in the faster growth section, and 12-17 yrs in the slower growth section. Data used in this figure was originally published in Carolin et al. 2019. Image by author**

## Paleoclimate evidence of third millennium BC dry events on a regional scale

The two periods of increased Mg/Ca in the GZ14-1 record demonstrate threshold behaviour in dustiness of the Mesopotamia region, due to either enhanced aridity, stronger winds, or change in soil properties or vegetation cover. Several factors suggest a drier regional climate coincident with the heightened dusty periods. First, within the same stalagmite, $\delta^{18}O$ gradually increases within the two increased Mg/Ca periods. Under modern conditions, rainwater monitoring in Tehran (50 km from the cave site) has demonstrated an anti-correlation between interannual precipitation amount and $\delta^{18}O_{precip}$ (Carolin et al. 2019). Thus, the $\delta^{18}O$ increase is interpreted as a local and likely also upstream trajectory decrease in rainfall amount, supportive of a dry environment suggested by the Mg/Ca dust interpretation. Second, on a regional scale, southern Levant Soreq cave speleothem $\delta^{13}C$ (Bar-Matthews et al. 2011) shows small increases in $\delta^{13}C$ coincident with the GZ14-1 Mg/Ca and $\delta^{18}O$ increases (Fig. 9.5). The authors interpret $\delta^{13}C$ changes to reflect changes in soil-organic carbon, with higher values indicating drier conditions (Bar-Matthews et al. 2011). The Soreq centennial $\delta^{13}C$ variations overlay a more gradual trend in $\delta^{13}C$ and $\delta^{18}O$ toward higher values beginning around 2800 BC and ending around 2000 BC (Bar-Matthews et al. 2011), which is interpreted as indicating a long-term trend toward drier conditions in the southern Levant through the third millennium BC. In the northern Levant, the Jeita cave $\delta^{18}O$ record also shifts to higher values by ~ 0.5‰ between 2500-2000 BC, with the pattern similar to the GZ14-1 Mg/Ca abrupt increases, though not aligned in age (Cheng et al. 2015) (Fig. 9.5). The offset in age could be due to combined age error in both age models. Finally, in the northern Red Sea, a sediment record shows a clear, anomalous +2‰ increase in planktonic $\delta^{18}O$, interpreted as drier conditions and enhanced evaporation in the region, from 2250 to 2050 BC (±100 y; 1σ), with perhaps an earlier period of less extreme aridity (indicated by a 0.3‰ $\delta^{18}O$ increase) from 2550 to 2450 BC (±100 y; 1σ) (Arz et al. 2006).

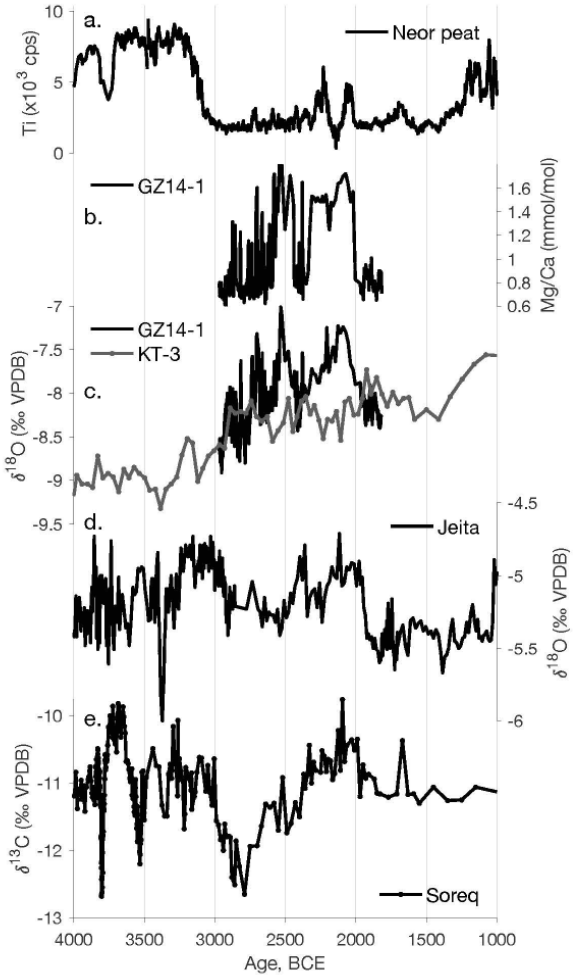


**Figure 1.5: Environmental and climate changes in the Middle East region over the third millennium BC. Site locations are shown on Figure 1. (a) Neor peat core Ti intensity interpreted as measure of aeolian input (Sharifi et al. 2015). (b) Iran stalagmite GZ14-1 Mg/Ca interpreted as a dust proxy, with greater Mg/Ca values indicating increased dust deposition on karst surface (Carolin et al. 2019). (c) Iran stalagmite GZ14-1 δ18O plotted on top of Iran stalagmite KT-3 δ18O, same y-axis (Carolin et al. 2019; Andrews et al. 2020). Higher δ18O values interpreted as local and/or upstream relatively drier conditions. (d) Jeita cave stalagmite δ18O, with higher values interpreted as relatively drier climate (Cheng et al. 2015). (e) Soreq speleothem δ13C, with higher values interpreted as drier surface conditions (Bar-Matthews et al. 2011). Image by author**

The two GZ14-1 centennial events are not reproduced in climate and environmental proxies of Holocene stalagmite KT-3 from Katalekhor Cave (Fig. 9.5; Andrews et al. 2020), ~300 km west of Tehran on the eastern side of the Zagros topographic highs (Fig. 9.1, marker 3). A broad increase in $\delta^{13}C$ begins around 2350 BC in KT-3, an extension of KT-3's long, gradual increase in $\delta^{18}O$ through the mid-to-late Holocene, both suggesting a general trend toward drier conditions from the third to the 1st millennium BC (Andrews et al. 2020). The lack of abrupt $\delta^{18}O$ events in KT-3 may be because Katalekhor Cave is situated in the core of the winter precipitation zone. Under modern conditions, there is less interannual variability in rainfall than is modelled at the Gol-e zard cave site area, thus making it less prone to severe climate events. The longer-term trend in KT-3 $\delta^{18}O$ is mirrored in GZ14-1 $\delta^{18}O$. Abrupt dust events are also not evident in KT-3 elemental records. Element ratio signatures in a stalagmite, however, are dependent on the overlying bedrock chemistry and the drip water seeping path of a particular drip to a stalagmite tip. Thus, it is not unusual that stalagmites from different cave sites do not replicate each other's X/Ca records.

Several paleo-dust records exist in the region which support a shift to dry and dusty conditions at the end of the third millennium BC, in agreement with the GZ14-1 stalagmite record. A dust record interpreted from an XRF scan of lithogenic elements such as titanium (Ti) abundance in a Neor peat mire core indicates relatively enhanced dust activity within the 2500-2000 BC period (Sharifi et al. 2015). A sediment core record from the Gulf of Oman shows an abrupt increase in Mesopotamia-sourced dust deposition around 2150 BC (± 100 y, 1σ) (Cullen et al. 2000), and fossil corals also collected in the Gulf of Oman indicate a significant and anomalous increase in seasonality around the same time, suggesting an uncharacteristic increase in the number of winter shamal days (dry dust events in the Mesopotamia region) (Watanabe et al. 2019). Winter shamal transports cold and dry air to western Asia, and cold and wet conditions to the Gulf of Oman (Watanabe et al. 2019). Notably, the two Gulf of Oman archives (sediment core and coral) record one period of dustiness centred around at 2150 BC (± 100 y, 1σ), with no earlier event at ~2500 BC (Watanabe et al. 2019; Cullen et al. 2000). Taken with the GZ14-1 stalagmite results, the collection of paleoclimate records demonstrates the presence of two arid periods, but indicate that the later of these – centred around 2150 BC – is of

larger amplitude and has a greater spatial extent, apparently influencing the broad Middle East region. The precise chronology of the GZ14-1 record allows the duration of these two centennial-scale events to be quantified and demonstrates the 2150 BC-centred event was of longer duration, as well as larger extent, than the earlier 2500 BC event (~290 y versus ~110 y).

## Implications of heightened Mesopotamia dust events during the latter 3rd Mill. BCE

Lawrence et al. 2021 analyse trends in population and settlement organization across Northern Mesopotamia and the Northern Levant from 4000 to 1000 BC, using Summed Probability Distributions (SPD) of radiocarbon dates. The authors find a surge in urbanization beginning around 3000 BC, increasing significantly from 2850 BC before a decline beginning around 2250 BC. The greatest amount of growth in both urban site area and rural site counts occurred in the drier landscapes of the region (Lawrence et al. 2021). The expanded use of marginal, drier landscapes may have been driven by the population's new focus on textile production, as sheep and goat are able to be pastured in more marginal environments (Wilkinson et al. 2014; Lawrence et al. 2015). Subsequent ecological degradation and desertification may have resulted from overgrazing and agricultural intensification (de Gruchy et al. 2016; Lawrence et al. 2021). Heightened dusty periods are inferred from the Iranian stalagmite record, 2560-2450 BC and 2310-2020 BC (± 30-70 y, 1σ). The latter period is thought to have the largest regional extent and climatological impact based on agreement between the collection of paleoclimate records around the Middle East. One hypothesis is that the first period of enhanced dustiness was predominantly an artifact of human-induced landscape change in the Mesopotamia region, while the second longer period of dustiness was a combination of landscape change and anomalously drier climate. On a longer broader scale, stalagmite data from the Eastern Mediterranean and western Iran both indicate a gradual trend toward drier conditions from the beginning to the end of the third millennium BC.

Increasing aridity from the beginning to the end of the 3rd mill. BC increased the need for quality irrigation and landscape management in Southern Mesopotamia, and increased the population size within suitable environments as marshland environments shrunk (Kennett & Kennett, 2006; Altaweel, 2019; Engel & Brückner, 2021; Lawrence et al., 2021). The deltas and irrigation network in the region thus became increasingly important to survival of the Sumerian city-states due to the drying environment through the 3rd mill. BCE.

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