Dissolved iron in the Cretan Sea (eastern Mediterranean)

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Abstract

Dissolved iron (Fe) distributions in the water column of the Cretan Sea show consistent patterns of elevated surface mixed layer concentrations (mean values of 1.44 and 1.95 nmol L^{-1} in March and September, respectively) that rapidly decrease with depth to low and relatively uniform concentrations in deep waters (circa 0.5 nmol L^{-1}). These features are consistent with inputs of dissolved Fe to surface waters and with rapid scavenging to low values in deep waters. The main source of surface dissolved Fe is probably Saharan atmospheric dust, the supply of which is variable depending on sporadic dust storm events and transport to the Cretan Sea. The short residence times estimated for dissolved Fe in the mixed layer (0.7–2 yr) emphasize the rapid removal of this element from surface waters and are consistent with recent mass balance calculations for Fe in the western Mediterranean Sea, in which rapid removal of Fe to sediments is needed to provide a balance in the budget. The deep-water concentrations are strikingly constant and similar to or slightly lower than those reported for the open ocean. The high surface concentrations we observed would be ample to support substantial primary production, and it is likely that the macronutrients, and phosphorus in particular, limit production in this region.

Iron (Fe) is an essential nutrient for phytoplankton, bacteria, and other marine microorganisms, and the role of this metal as a potentially limiting factor for phytoplankton has come under increasing scrutiny since the initial ''iron hypothesis'' was put forward by Martin et al. (1988) to explain limited productivity in high-nutrient low-chlorophyll (HNLC) oceanic systems. Experiments such as IRONEX and SOIREE have now clearly demonstrated that Fe additions can stimulate phytoplankton growth in HNLC zones (Watson 2001), although many questions still remain about the forms of Fe present, uptake mechanisms, and the impact of Fe on export carbon production. The distributions, sources, and sinks of Fe in seawater have recently been reviewed (De Baar and De Jong 2001). There is a coherent trend in reported data for all regions, with increasing concentrations approaching the continental margins (up to circa 100 nmol L^{-1}), while in the open ocean, dissolved Fe has typically low concentrations in surface waters (0.02–1 nmol L^{-1}) and higher (0.4–2.0 nmol L^{-1}) concentrations in deep waters. The rapid removal of dissolved Fe through association with biotic and abiotic particles and its loss from the water column results in Fe having a short residence time (De Baar and De Jong 2001), and upper-ocean concentrations should largely reflect local inputs (e.g., aeolian and shallow benthic sources). Johnson et al. (1997) summarized the available accurate data for many open-ocean environments and noted that Fe typically showed a nutrient-like vertical distribution. They argued that in deep waters, Fe is maintained in solution at a relatively constant concentration as an organic complex, rather than through a solubility mechanism or as a result of a balance between in situ regeneration and removal processes. The relative importance of pseudoequilibria, organic complexation, and size fraction (i.e., association with colloids) in maintaining deep-water concentrations of dissolved Fe is yet to be resolved (De Baar and De Jong 2001).

Despite the biogeochemical importance of Fe in the ocean, there is still limited accurate information on this element because of difficulties with contamination during collection and analysis. The limited Fe data for the Mediterranean as a whole are summarized by De Baar and De Jong (2001), and only a handful of data exist for the eastern Mediterranean (Saager et al. 1993; Van Den Berg 1995). Deep-water concentrations of about 1 nmol L^{-1} have been reported, although higher vales were measured over the isolated and small Bannock and Tyro anoxic brine basins (Saager et al. 1993).

An important feature influencing the geochemistry of the Mediterranean Sea is the proximity of the Sahara Desert to the south and the input of dust from this source to the Mediterranean (Guerzoni et al. 1999). A small fraction of the Fe in dust comes into solution on contact with seawater (Jickells and Spokes 2001), and given the relatively high abundance of Fe in the dust, such inputs would be expected to be an important source of dissolved Fe to surface waters. It has been suggested that iron inputs associated with Saharan dust may indeed stimulate primary production (Saydam 1996), despite the eastern Mediterranean Sea and contiguous Cretan Sea being one of the most oligotrophic marine ecosystems known, and this region is generally regarded to be phosphorus (P) limited (Krom et al. 1991). Recent data on Fe from the western Mediterranean Sea (Sarthou and Jeandel 2001) imply an important impact of biota on removal of Fe from

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Table 1. Location of the sampling stations in the Cretan Sea.

Station	Position	Water column depth (m)
MSB1	36°04.50N 25°17.00E	1,775
MSB2	35°44.70N 25°06.00E	1,363
MSB3	36°00.00N 23°53.60E	1,191
MSB4	36°15.00N 24°06.30E	911
MSB5	36°11.60N 24°41.00E	1,235
MSB6	36°00.00N 25°42.30E	1,286
MSB7	35°40.00N 26°13.00E	2,273

Fig. 1. Sampling stations in the Cretan Sea.

the water column, and they imply as well that the low residual concentrations of Fe after biological activity may lead to changes in species succession or even growth limitation.

The present article reports new data on dissolved Fe for a series of stations in the Cretan Sea (a component of the eastern Mediterranean) in two contrasting seasons and discusses the implications of these first detailed data for the biogeochemistry of Fe in the eastern Mediterranean Sea.

Materials and methods

Samples were collected during March (early spring) and then September–October (late summer) of 1997 from the research vessel *Aegaeo* at a series of stations in the Cretan Sea (Table 1). In March, Sta. MSB1, MSB2, MSB3, MSB6, and MSB7 were occupied, and in September–October, all stations shown in Fig. 1 were occupied. A limited number of samples was collected in both March and September, approximately 1 km away from the island of Milos, to ascertain any influence of its hydrothermal activity on Fe concentrations in the adjacent water column.

Water samples were collected using 10-liter Teflon-coated Go-Flo bottles, fitted with Teflon taps and mounted on a CTD (Seabird) rosette system. Components of the CTD system that were regarded as having contamination potential were covered in plastic film. The March samples were taken directly into acid-cleaned 1-liter low-density polyethylene bottles (Nalgene). The Go-Flo tap and 1-liter bottle were both maintained within a large resealable plastic bag during this operation to minimize potential contamination. The sample was then filtered in a Class 100 laminar flow hood through 0.4-μm–pore size Nuclepore polycarbonate membranes held in a rigorously acid-cleaned polysulfone filtration system (Nalgene). Sample bottles were stored in the dark in resealable polyethylene bags for return to the Southampton laboratory. During the September/October cruise, the Go-Flo bottles were pressurized with filtered nitrogen, and the seawater was passed directly through 0.4-μm–pore size filters held in in-line PTFE holders. Samples collected using CTD systems similar to those used here have been shown to provide uncontaminated samples equivalent to
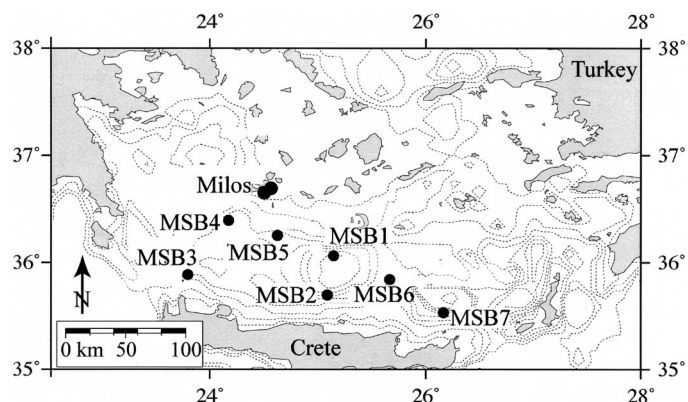
those obtained using the established system of Go-Flo bottles on Kevlar line (Wu and Luther 1994; Sohrin et al. 2000). Some surface samples were collected using a pole technique, as described by Sherrell and Boyle (1988). These were vacuum filtered using the method described above. Within 1 week of collection, all samples were stabilized by acidification with 1 ml of quartz sub-boiling (Q) distilled nitric acid per liter of seawater.

Fe was separated and concentrated from the seawater in the Southampton laboratory using a modification of the chelation and solvent extraction procedure of Statham (1985), which allows simultaneous extraction of Fe, manganese, and other metals. In overview, the Fe present in a 200-g aliquot of seawater is complexed with a mixed dithiocarbamate reagent, and the complexes are repeatedly extracted into chloroform. The chloroform is evaporated on a hot plate, and the residue is repeatedly oxidized by heating with small volumes of concentrated quartz sub-boiling distilled (Q) nitric acid. The final acid aliquot is made up to a volume of 1.6 ml with Q water, giving an overall concentration factor of approximately 125. Iron in the concentrate is determined by graphite furnace atomic absorption spectrophotometry. All analytical work was done in a Class 100 dedicated trace-metal clean laboratory fitted with clean wet stations for handling the chloroform and acid. Throughout the sampling and analytical procedures, all materials coming into contact with samples were rigorously cleaned (Hart 2000). With the storage and analytical procedures used, the Fe measured will include simple ionic species, colloidal and organically and inorganically complexed forms of the element, and will thus provide a measure of total dissolved (<0.4 μm) Fe.

The performance of the analytical procedure is summarized in Table 2. The accuracy of the analyses is shown by good agreement of certified values of NASS-4 for Fe and those obtained here. Some samples from the March cruise did appear to be suspect relative to the bulk data and this was evident from the divergence of data from the overall smooth profiles for Fe. With improved filtration and sample handling during the following September cruise, only about 4% of the total data set was regarded as suspect, and these data were excluded. Nutrient data and salinities obtained from samples collected from each Go-Flo bottle were used

Table 2. Blanks, detection limits, precision, and accuracy. Blanks and detection limits are based on nine separate sets of data.

Average and range (in brackets) of blanks (nmol L^{-1})	Average and range (in brackets) of detection limits (2.5 times standard deviation) (nmol L^{-1})	Precision (% coefficient of variation, at 1.51 nmol L^{-1})	NASS-4 (Certified value is 1.88 ± 0.28 nmol L^{-1})
0.17 (0.07–0.23)	0.19 (0.09–0.34)	4.7	1.95

to help identify any bottles that had not closed at the correct depth. These samples are excluded from the discussion.

Nitrate plus nitrite concentrations were determined by conventional autoanalyzer procedures (Grasshoff et al. 1999). Precision of measurements was ±5%, and the detection limit was 0.1 μmol L^{-1}.

Results

The data are displayed in Web Appendix 11 at http://www.aslo.org/lo/toc/vol_50/issue_4/1142a1.pdf, and Fig. 2 shows the vertical distributions of dissolved Fe in March

A) March

B) September

Fig. 2. Dissolved Fe at stations occupied in the Cretan Sea in (A) March 1997 and (B) September/October 1997.

1997 and September/October 1997, respectively. The most obvious features of the distributions for both seasons are the elevated concentrations in the surface mixed layer (means for March and September, 1.44 and 1.95 nmol L^{-1}, respectively) and the low and relatively consistent deeper water values that are reached rapidly below the mixed layer in both seasons (mean concentration $\geq$ 600 m = 0.46 nmol L^{-1}). These deep-water concentrations are lower than most previously reported data in the Mediterranean Sea (*see* summary of data in De Baar and De Jong 2001) but are similar to those recently reported by Sarthou and Jeandel (2001) in the western basin.

The mixed layer depths in March and September (as identified by temperature and salinity variations) are, respectively, ~125 and ~50 m. The formation of the shallower warmer thermocline in September reflects surface heating over the summer. In the water column adjacent to the island of Milos in September, which is known to have active hydrothermal sites (Dando et al. 1999), dissolved Fe concentrations were higher throughout the water column than at other stations (maximum 16.0 nmol L^{-1}), thus indicating a benthic, presumably hydrothermal, source of Fe. The surface samples collected in March have lower concentrations (1.93 and 4.88 nmol L^{-1}).

Discussion

Deep waters—The different water masses in the Cretan Sea and their exchange with adjacent zones are discussed by Theocharis et al. (1999). Deep waters in the Cretan Sea (>~680 m) consist primarily of Cretan Deep Water in the eastern and central areas, overlain with a mixture of waters of east Mediterranean and Aegean origin. Within the precision of the Fe data, there are no features clearly related to water masses. The overall low concentrations reflect the scavenging and removal processes discussed by Sarthou and Jeandel (2001). The average concentration reported here is similar to those described by De Baar and De Jong (2001) and Johnson et al. (1997) for oceanic waters generally and by Wu et al. (2001) for the North Atlantic Ocean, which indicates that a similar mechanism to that in the open ocean may exist here for maintaining deep-water concentrations above solubility controlled levels. If this mechanism involves complexation by one or more organic ligands, as proposed by Johnson et al. (1997), the origin of the ligands must be from the adjacent Atlantic Ocean, in situ formation within the Mediterranean, or, possibly, atmospheric inputs. A significant fraction of the "dissolved" Fe (<0.4 μm) is expected to be in the colloidal size range (Wu et al. 2001).
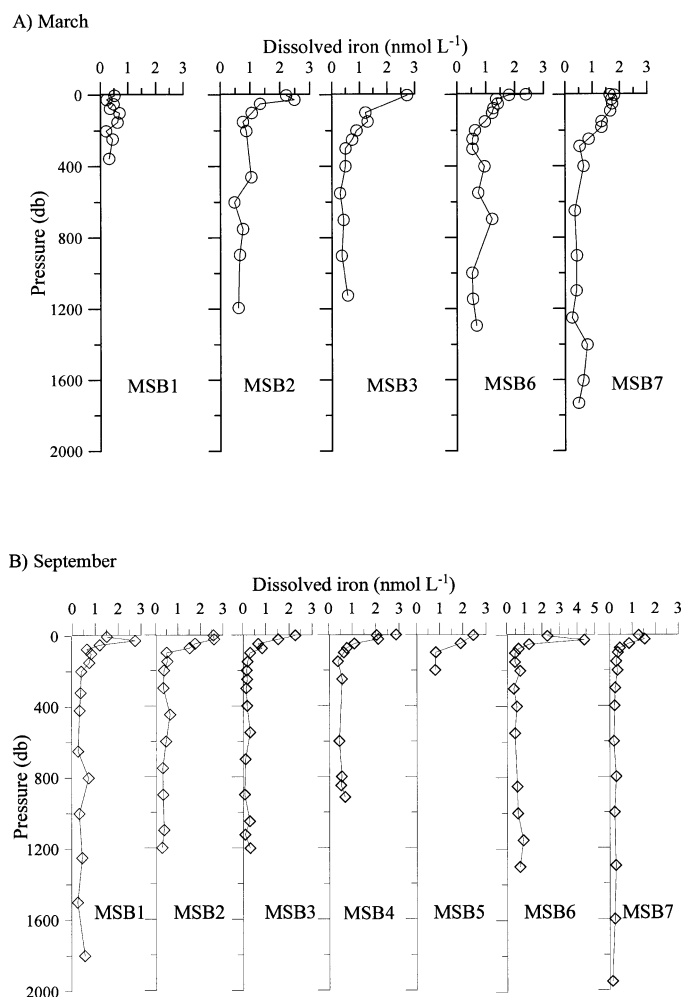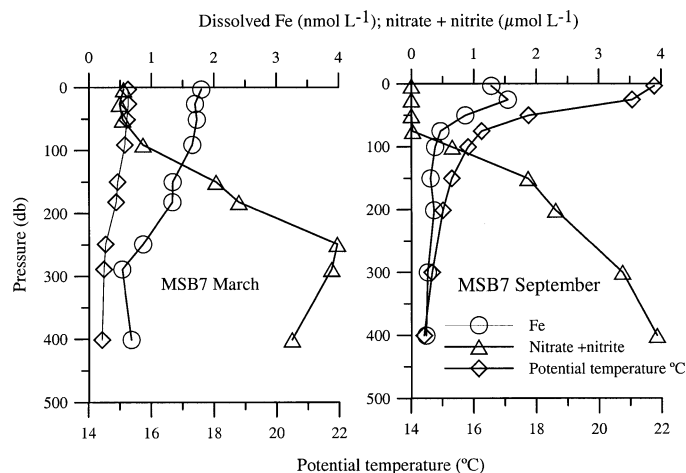
Fig. 3. Dissolved Fe, potential temperature, and nitrate plus nitrite for station MSB7 in March and September.

Upper water column—The uppermost part of the Cretan Sea water column is derived from Levantine Surface Water, Modified Atlantic Water, and diluted Black Sea Water (BSW) (Poulos et al. 1997). Evaporation during the summer months leads to increases in salinity in these surface waters. Theocharis et al. (1999) observed central cyclonic and western anticyclonic eddies in the Cretan Sea, which, though they may shift in space and extent between seasons, appear stable over time and affect the vertical and lateral distribution of both intermediate and surface waters. Within the central cyclonic eddy, homogenization of salinity down to 150 m is observed at MSB1 and MSB6 in March, where waters of intermediate depths have been transported to the surface.

The eastern Mediterranean and Cretan Seas are some of the most oligotrophic waters on the planet (e.g., Krom et al. 1991), and primary production and chlorophyll concentrations are extremely low (Gotsis-Skretas et al. 1999; Ignatiades et al. 2002). Winter mixing leads to increased surface-water nutrient concentrations, and the highest productivity is observed in the spring. Solar heating over the summer produces a warm shallow thermocline, and the macronutrients (nitrogen, P) are reduced to extremely low concentrations in this layer as a result of biological activity. This behavior is typified at station MSB7 (Fig. 3), where in the spring, the deep mixing is evident as low but detectable nitrate plus nitrite present in the surface waters. By the autumn the nutrients have been reduced to nondetectable concentrations, and a distinct nutricline has been formed. Dissolved Fe reflects these changes in the upper water column as a result of mixing, with high surface-water Fe being mixed with low deep-water Fe over the winter. At the end of the summer, the shallow thermocline contains high Fe concentrations that rapidly fall to background levels in deeper waters. This general pattern of high surface Fe being mixed down into deeper waters by the spring and being contained in the shallow thermocline formed over the summer is evident in the data shown in Fig. 2.

Trace metals with short residence times relative to the average residence time for water in the Mediterranean (~100 yr, Béthoux et al. 1998) will be expected to have distribu-

tions reflecting input and removal processes within the basin (e.g., aluminum; Guerzoni et al. 1999). The relatively high dissolved Fe concentrations observed here in surface waters of the Cretan Sea may reflect a range of sources of Fe, including rivers, Atlantic Ocean water entering the Mediterranean, nearshore sediments, hydrothermal activity, and atmospheric inputs.

Rivers—Riverine inputs to the eastern Mediterranean and to the Mediterranean as a whole are small (Guieu et al. 1997) and localized and will not affect offshore sites such as the Cretan Sea. A surface-water mass that may contain elevated Fe is the BSW that enters the northern Aegean through the Dardanelles. However, only modest increases in Fe concentrations over Aegean background values have been observed in BSW, and those have been observed only close to source (Hart 2000).

Atlantic waters—The surface waters flowing from the western to eastern basins of the Mediterranean through the Sicilian Strait are derived principally from North Atlantic Surface Water entering through the Strait of Gibraltar. These Atlantic inflow waters are reported to have low dissolved Fe concentrations in the range 0.65–0.93 nmol L^{-1} (Morley et al. 1997) and thus cannot be a significant source of Fe to the eastern Mediterranean.

Sedimentary sources of Fe—Elevated concentrations of dissolved Fe relative to the open ocean are a common feature of coastal and shelf waters (e.g., Muller et al. 1994; Boye et al. 2003; and as discussed by De Baar and De Jong 2001). The main source of the Fe in these systems is reported to be the sediments and the result of recycling of organic matter (Elrod et al. 2004). If such sources were present along the coast of Crete and the adjacent mainland, it is possible that some Fe may be advected in surface waters to the stations sampled here. More detailed sections away from the coast would be needed to identify such a source.

Hydrothermal sources of Fe—A series of active hydrothermal sites have been identified in the Aegean Sea (Dando et al. 1999), including several islands along the Hellenic volcanic arc on the northern edge of the Cretan Sea. Milos is one of these islands, and it has been previously studied for seawater and sediment enrichment of metals (Dando et al. 1999) and is a potential source of dissolved metals to Aegean surface waters. The concentrations of dissolved Fe very close to the island, however, show only a modest increase over those in the open Aegean, and thus this signal is not expected to propagate far given the short residence time of Fe in the water column. If the hydrothermal inputs of Fe at Milos are typical of volcanic islands in the Hellenic arc, hydrothermal sources of Fe are unlikely to have a significant impact on waters tens to hundreds of kilometers away from these islands in the Cretan Sea.

Atmospheric sources—Atmospheric deposition has been shown to be an important source of trace metals to the Mediterranean (e.g., Guieu et al. 1997; Guerzoni et al. 1999; Herut et al. 2001). Mean annual atmospheric mass fluxes

observed at Crete (21 g m^{-2}) and Israel (50 g m^{-2}) were found to be the highest in the Mediterranean. In comparison to riverine discharge, 70% of insoluble particles in the eastern Mediterranean were from the atmosphere, compared to only 22% in the central and 19% in the western Mediterranean (Guerzoni et al. 1999). The trace-metal composition of aerosols transported to the Mediterranean can be regarded as having an anthropogenic-rich 'background' component from Europe, upon which large sporadic pulses of Saharan crust-rich dust are superimposed (Guerzoni et al. 1999). Despite much variability, there is a clear seasonal cycle of dust incursions over the Mediterranean, with minima in the winter months and increasing dust transport beginning over the east basin in spring and spreading west in the summer (Moulin et al. 1997). The main source of this dust over the eastern Mediterranean is due south in the Sahara Desert (D'Almeida 1986).

The contribution to dissolved Fe in surface waters from atmospheric particles is dependent on a variety of factors, including solubility, photoreduction to Fe II, and organic complexation of Fe in rainwater and seawater (Jickells and Spokes 2001). Guieu et al. (1997) provided an estimate of total dissolved atmospheric Fe inputs to the western Mediterranean (90–180 mol Fe km^{-2} yr^{-1}) that is based on the soluble fractions from both wet and dry deposition and is the best available estimate for the Mediterranean Sea as a whole. Although the solubility of Fe in these particles is low, ranging from 3–11% (Guerzoni et al. 1999), the reported overall input flux of dissolved Fe to surface waters is high because of the high total mass input of Saharan dust and because Fe is a relatively abundant constituent of this material.

An estimate of the annual increase in surface-water dissolved Fe as a result of atmospheric inputs can be made by taking an average mixed layer depth of 80 m and the Fe atmospheric input values of Guieu et al. (1997). The Fe increase corresponds to 1.13–2.25 nmol L^{-1} (i.e., values of the same order as those observed). The dissolved Fe flux values used here may be underestimates for the eastern Mediterranean, as dust inputs for this area (mean annual input 21 g m^{-2}) are about twice that of the western Mediterranean (Guerzoni et al. 1997). Additionally, surface Fe values will reflect the net effect of removal and input terms and will be dependent on the episodic nature of dust inputs. However, the concentrations calculated are consistent with a primarily atmospheric input of Fe.

An estimate of the residence time of dissolved Fe in the surface mixed layer of the Cretan Sea can be made assuming that the major Fe source is aeolian and that the system is at steady state, with removal largely occurring through biological uptake and vertical particle transport to deeper waters. Residence times for two seasons with differing mixed layer depths (corresponding to each cruise) are given in Table 3 and were obtained by dividing the mixed layer inventory of dissolved Fe per m^2 (calculated from data shown in Fig. 2) by the atmospheric flux of dissolvable Fe from aeolian particles.

These residence times of 0.7–2.0 yr are short, indicating the reactivity of Fe in these surface waters, but they are in-line with estimates for oceanic regions (De Baar and De Jong

Table 3. Estimates of residence times in the surface mixed layer with respect to atmospheric inputs. Figures in brackets are average mixed layer depths. The dissolved Fe flux values used in these calculations (90–180 mol Fe km^{-2} yr^{-1}) are taken from Guieu et al. (1997).

Date and mixed layer depth (m)	Mar 1997 (~125)	Sep 1997 (~50)
Residence time (yr)	1.0–2.0	0.7–1.3

2001); e.g., 1 to 20 yr for the upper 500 m of the Pacific (Landing and Bruland 1987), 0.8 yr in the upper 100 m of the Sargasso Sea, and 2 yr from modeling by Johnson et al. (1997). The short residence times reported here imply efficient removal of Fe from the water column. Ozturk et al. (2003) have demonstrated significant removal of water-column Fe during bloom conditions in Aegean coastal waters. Ignatiades et al. (2002) found higher average values for chlorophyll *a* and primary productivity for surface waters in March 1997 (0.301 mg m^{-3} and 0.508 mg C m^{-3} h^{-1}, respectively) relative to September 1997 (0.119 mg m^{-3} and 0.167 mg C m^{-3} h^{-1}, respectively), and the period of higher biological activity corresponds to the shortest residence times of Fe (March), presumably reflecting more active biological removal of the element at that time. The short residence times are also consistent with recent mass-balance calculations for Fe in the western Mediterranean Sea (Elbaz-Poulichet et al. 2001), in which rapid removal of Fe to sediments is needed to provide a balance in the budget for this element.

Implications of Fe concentrations for primary production—Primary production in the Cretan and eastern Mediterranean Seas is reported to be intimately linked with the availability of macronutrients and of P in particular (Krom et al. 1991). Thus, the highest productivity in the Cretan Sea occurs in spring, after winter mixing brings macronutrients to the mixed layer through erosion of the nutricline (*see* Gotsis-Skretas et al. [1999]; Ignatiades et al. [2002]; Fig. 3) and has decreased by the end of the summer, when macronutrients are at extremely low concentrations in surface waters. The data reported here show high surface Fe concentrations for both of these seasons that are well above any normal metabolic requirements (Hutchins and Bruland 1998), and, therefore, Fe is not obviously limiting microbial growth at these times. For there to be any limitation of larger phytoplankton growth by Fe, either biological removal of Fe from the upper water column must be significant and rapid, as suggested in the more productive western Mediterranean to explain low dissolved Fe concentrations (Sarthou and Jeandel 2001), and/or the atmospheric inputs must be reduced. Given the extremely low productivity of the Cretan and Mediterranean Seas and the greater inputs of Saharan dust (and thus Fe) to the eastern relative to the western Mediterranean Sea, it seems highly unlikely that Fe will be limiting for larger phytoplankton species. This view is consistent with recent work (Ozsoy and Saydam 2001) for the eastern Mediterranean that has reported that the flux of the bioavailable Fe fraction in most atmospheric wet deposition

events was sufficient to support the maximum primary production rates typical of the eastern Mediterranean Sea.

In order to more fully understand the biogeochemistry of Fe in these oligotrophic waters, more temporally detailed information is required on atmospheric inputs, the resulting dissolved Fe concentrations, and the response to these inputs of the phytoplankton and microbial populations present.

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