

DATA REPOSITORY: ANALYTICAL METHODS

U-Th-Pb Chemistry

Chips of coral interiors (aragonite; X-ray diffraction) were cut with a steel blade and a Dremel microdrill, washed four times in water in an ultrasonic bath, and then dried, weighed, dissolved, and spiked (U-Pb, ^{233}U - ^{236}U - ^{205}Pb ; U-series, ^{229}Th - ^{233}U - ^{236}U). U and Pb were separated on 250 μL columns by HBr methods (AG1x8 resin in Br⁻ form; see also Strelow and Toerien, 1966), and then Pb was purified of Ca traces on 100 μL columns. U, Th, and Pb were run on a Micro Mass sector 54 mass spectrometer, with Pb signals ($\sim 10^{-12}$ A for ^{208}Pb) measured in Faraday cups in static mode. Fractionation corrections for Pb were $0.12\% \pm 0.04\%/\text{amu}$, and the correction for full procedural Pb blanks was 50 ± 13 pg.

Corals for U-series analysis and U fractions from the U-Pb separation were prepared by iron coprecipitation and run by standard mass-spectrometric methods (Chen et al., 1986; Edwards et al., 1987). Spike calibration, chemical separation, and analytical protocols were confirmed by interlaboratory calibration with the University of Minnesota radioisotope lab and by U-series dating of known corals from Barbados (kindly provided by Larry Edwards).

Data Treatment

In corals, the initial coral ($^{230}\text{Th}/^{238}\text{U}$) is typically near zero, which generates a deficit in $^{206}\text{Pb}^*$ produced from ^{238}U . The same holds for an initial coral ($^{231}\text{Pa}/^{235}\text{U}$) near zero, giving a slight deficit in $^{207}\text{Pb}^*$ from ^{235}U . Modern and late Pleistocene oceans have a ^{234}U excess of about 15% (e.g., Chen et al., 1986; Edwards et al., 1987), which ultimately yields an excess of $^{206}\text{Pb}^*$. Combined, these effects yield a time lag of ~ 60 k.y. for growth in $^{206}\text{Pb}/^{208}\text{Pb}$ ratios after ~ 1 m.y..

U-Pb isochron ages and uncertainties were calculated at the 95% confidence level with Isoplot/Ex (Ludwig, 1999); the most precise ages were obtained with 3-D linear, concordia-constrained $^{238}\text{U}/^{206}\text{Pb}$ - $^{207}\text{Pb}/^{206}\text{Pb}$ - $^{208}\text{Pb}/^{206}\text{Pb}$ isochrons. Coral CCD-6058 has a $^{206}\text{Pb}/^{208}\text{Pb}$ intercept of 0.4906 ± 0.007 (MSWD=0.33), and a $^{206}\text{Pb}/^{207}\text{Pb}$ intercept of 1.217 ± 0.024 .

(MSWD=0.33). The low MSWD results from the estimate of uncertainty in blank level. Coral CCD-878 has a $^{206}\text{Pb}/^{208}\text{Pb}$ intercept of 0.513 ± 0.014 (MSWD=3.1), and a $^{206}\text{Pb}/^{207}\text{Pb}$ intercept of 1.28 ± 0.1 (MSWD=4.3). Because coral growth may occur over decades and in different seasons, the MSWD greater than 1.0 may stem from small variations of initial Pb acquired from the ambient marine setting.

StreLOW, F.W.E., and Toerien, F.S, 1966, Separation of lead (II) from bismuth (III), thallium (III), cadmium (II), mercury (II), gold (II), platinum (IV), palladium (II), and other elements by anion exchange chromatography: *Analytical Chemistry*, v. 38, p. 545-548.