## **GSA DATA REPOSITORY 2011143**

### Otero et al.

**Table DR1.** Location, systematic attribution, sample number, sample type and oxygen isotope compositions of the fish fossil samples. (d) is for diagenesis suspected

| Location                   | Taxon  | Environment  | Sample  | Sample  | d18O (PO4)  |  |  |  |  |
|----------------------------|--|--|---|---|---|--|--|--|--|
| Toros-Menalla : 7 Ma       |  |  |   |   |   |  |  |  |  |
| TM 82                      | Hydrocynus   | marginal waters  | 19-M82-h  | 4 teeth   | 20,19   |  |  |  |  |
| TM 90                      | Polypterus   | Swamp  | 30-M90-p  | 4 scales  | 20,90   |  |  |  |  |
| TM 254                     | Hydrocynus   | open waters  | 31-M90-p<br>36-M254-h<br>37-M254-b  | 5 teeth   | 19,23<br>16,40<br>18.06   |  |  |  |  |
| TM 337                     | Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Tetraodon  | marginal waters<br>marginal waters   | 40-M337-h<br>51-M337-h<br>49-M337-t   | 5 teeth<br>33 teeth   | 18,29<br>18,58<br>19,36   |  |  |  |  |
| TM 266                     | Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Tetraodon<br>Tetraodon | open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters | 11-M266-h<br>12-M266-h<br>39-M266-h<br>41-M266-h<br>42-M266-h<br>43-M266-h<br>43-M266-h<br>48-266-t<br>50-266-t | n teeth<br>n teeth<br>6 teeth<br>33 teeth<br>33 teeth<br>33 teeth<br>33 teeth<br>33 teeth<br>38 plates<br>26 plates | 17,10<br>18,05<br>18,12<br>16,08<br>16,62<br>16,90<br>17,06<br>17,10<br>18,44 |  |  |  |  |
| TM 267                     | Hydrocynus<br>Hydrocynus   | open waters  | 24-M267-h<br>25-M267-h  | 5 teeth<br>8 teeth  | 15,86<br>16,43  |  |  |  |  |
| Kossom Bougoudi : 5.5–5 Ma |  |  |   |   |   |  |  |  |  |
| KB 03<br>KB 07             | Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus   | open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters   | 7-B3-h<br>8-B3-h<br>44-B3-h<br>45-B3-h<br>53-B3-h<br>26-B7-h<br>32-B7-h   | n teeth<br>n teeth<br>33 teeth<br>33 teeth<br>33 teeth<br>5 teeth<br>3 teeth  | 18,94<br>19,90<br>18,67<br>19,03<br>19,14<br>19,74<br>17,63                   |  |  |  |  |
| Kolle : 5–4 Ma             |  |  |   |   |   |  |  |  |  |
| KL 02                      | Hydrocynus<br>Hydrocynus<br>Hydrocynus   | open waters<br>open waters<br>open waters  | 13-L2-h<br>14-L2-h<br>54-L2-h   | n teeth<br>n teeth<br>33 teeth  | 18,97<br>20,24<br>19,16   |  |  |  |  |
| KL 02 (chann               | <b>el)</b><br>Hydrocynus<br>Polypterus   | marginal waters<br>marginal waters   | 15-L2c-h<br>29-L2c-p  | n teeth<br>4 scales   | 18,21 (d)<br>18,20 (d)  |  |  |  |  |
| Koro-Toro : 3.5–3 Ma       |  |  |   |   |   |  |  |  |  |
| KT 13<br>KT 12             | Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Hydrocynus<br>Tetraodon  | open waters<br>open waters<br>open waters<br>open waters<br>open waters<br>open waters   | 9-T13-h<br>10-T13-h<br>16-T12-h<br>46-T12-h<br>56-T12-h<br>47-T12-t<br>55-T12-t                                 | n teeth<br>n teeth<br>33 teeth<br>33 teeth<br>30 plates<br>23 plates  | 19,57<br>20,05<br>19,76<br>19,58<br>19,86<br>18,99<br>20,14                   |  |  |  |  |
|                            | 1000000  | opon waters  | 00-112-1  | 20 plates   | 20,14   |  |  |  |  |

### **Appendix DR1. Sample preparation**

Teeth (jaw teeth of *Hydrocynus*), tooth plates (beak elements of *Tetraodon*) and ganoid scales (*Polypterus*) were cleaned by ultrasonic cleaning in deionized water. Depending on the concerned fish, bulk analyses were performed on 10 to 33 teeth, on about 20 tooth plates from one or two beak elements, or on 5 to 10 scales. Well-preserved enamel was manually selected on each tooth by hand-picking following rough crushing in an agate mortar.

## Appendix DR2. Oxygen isotope analysis of tooth enamel phosphate

Measurements of oxygen isotope ratios of apatite consist in isolating  $PO_4^{3-}$  using acid dissolution and anionexchange resin, according to a protocol derived from the original method (Crowson et al., 1991) and slightly modified (Lécuyer et al., 1993). Silver phosphate was quantitatively precipitated in a thermostatic bath set at a temperature of 70°C. After filtration, washing with double deionised water, and drying at 50°C, 15 mg of Ag<sub>3</sub>PO<sub>4</sub> were mixed with 0.8 mg of pure powder graphite. <sup>18</sup>O/<sup>16</sup>O ratios were measured by reducing silver phosphate to CO<sub>2</sub> using graphite reagent (O'Neil et al., 1994; Lécuyer et al., 1998). Samples were then weighed into tin reaction capsules and loaded into quartz tubes and degassed for 30 minutes at 80°C under vacuum. Each sample was heated at 1100°C for 1 minute to promote the redox reaction. The CO<sub>2</sub> produced was directly trapped in liquid nitrogen to avoid any kind of isotopic reaction with quartz at high temperature. CO<sub>2</sub> was then analyzed with a GV Prism<sup>TM</sup> mass spectrometer at the Laboratory UMR CNRS 5125 'PEPS', University Lyon 1. Isotopic compositions are quoted in the standard  $\delta$  notation relative to V-SMOW. Silver phosphate precipitated from standard NBS120c (natural Miocene phosphorite from Florida) was repeatedly analyzed ( $\delta^{18}O = 21.67 \pm 0.18$ ; n = 15) along with the silver phosphate samples derived from the Neogene Chadian fish remains.

## Appendix DR3. 818O and 8D composition of six water samples collected in February 2006 in Chad

Aliquots of 200  $\mu$ l of water were automatically equilibrated at 40°C with CO<sub>2</sub> and H<sub>2</sub> and analyzed using a MultiPrep<sup>TM</sup> system on line with a GVI IsoPrime<sup>TM</sup> dual inlet IRMS. Internal reproducibilities were typically ±0.03‰ for  $\delta^{18}$ O and ±0.5‰ for  $\delta$ D. External reproducibilities of  $^{18}$ O/ $^{16}$ O and D/H measurements were established at about ±0.1‰ and ±1‰, respectively, by scaling raw data to the "true" isotopic ratios of SMOW, SLAP and GISP international standards.

The measurements are presented below in a table giving the name sample and their location, and in the diagram  $\delta D=f(\delta^{18}O)$  showing the samples (blue spot), the regression line (blue line) and also the Meteoric Water Line (MWL) in red. Note the Deuterium and <sup>18</sup>O enrichment of the residual water collected in a drying pond (Guileba, TCH\_W5)

| Sample | Location                      | δ <sup>18</sup> Ο | SD   | δD    | SD   |  |  |
|--------|-------------------------------|-------------------|------|-------|------|--|--|
|        |                               | ‰                 |      | ‰     |      |  |  |
|        |                               | SMOW              |      | SMOW  |      |  |  |
| TCH_W1 | well ≈15°LatN (desert, Salal) | 4.77              | 0.05 | 16.1  | 0.03 |  |  |
| TCH_W2 | well ≈16°LatN (desert, TM)    | -2.46             | 0.05 | -33.6 | 2.0  |  |  |
| TCH_W3 | well ≈16°LatN (desert, TM)    | -0.26             | 0.04 | -29.7 | 1.0  |  |  |
| TCH_W4 | well ≈16°LatN (desert, TM)    | -2.83             | 0.06 | -37.7 | 0.5  |  |  |
| TCH_W5 | pound ≈13.3°LatN (Guileba)    | 26.14             | 0.08 | 100.6 | 1.3  |  |  |
| TCH_W6 | well ≈13.3°LatN (Guileba)     | -7.69             | 0.01 | -54.4 | 2.7  |  |  |
|        |                               |                   |      |       |      |  |  |

*Note:* The wells in Salal and Guileba are those of the village; the wells in the desert were dug by OO and MHT (about 2 m deep) close to three different sites in the Toros-Menalla area (TM).



# **Appendix DR4. Diagenesis**

Isotopic exchange under inorganic conditions has little effects upon the oxygen isotope composition of phosphate, even at geological scale (Kolodny et al., 1983; Lécuyer et al., 1999). Well-preserved and unbroken teeth were preferentially selected to isolate tooth enamel that is considered as the most robust biomineral for preserving pristine oxygen isotope ratios. Although no method is available to demonstrate definitely whether the oxygen isotope composition of tooth phosphate was affected by diagenetic processes, several arguments are in favor of the preservation of the primary isotopic record. Based on the clustered phosphate chemical yields close to 40% measured during the wet chemistry procedure, the original stoechiometry of tooth enamel was preserved. A strong argument supporting the preservation of the oxygen isotopic record is provided by the relation observed between the size of water bodies and the mean  $\delta^{18}$ O values of fish teeth that lived in the contemporaneous late Miocene sites from Toros-Menalla. If early diagenetic processes had occurred, they would have most likely homogenised  $\delta^{18}O_p$  values of all vertebrate remains whatever the physiology and ecology of the corresponding taxa. It appears on the basis of our data set that diagenetic effects - if they occurred - were weak enough to preclude the resetting of the original oxygen isotope compositions of fossil fishes. However, two samples from a same peculiar horizon in KL2 (Otero et al., 2009) were excluded from the dataset because they do not match any of these criteria (Table DR1).

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