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Reconstruction of the original organic richness in weathered Silurian shale outcrops (Murzuq and Kufra basins, southern Libya)

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ABSTRACT

The early Silurian in North Africa and Arabia was characterised by widespread deposition of organic-rich shales in palaeo-depressions. The unit represents an important hydrocarbon source rock in the region and can be detected easily in well logs because of strong uranium-related natural radiation. In exposures, however, organic matter is commonly heavily oxidised through weathering so that identification of the unit in the field is difficult. Uranium and pyrite framboids appear to be less vulnerable to weathering and may be used to identify intervals of originally organic-rich shales in exposures. Framboids are discrete spheroidal aggregates of pyrite microcrystallites and their size distribution is thought to be controlled by palaeo-depositional bottom-water redox-conditions. Analyses of fresh Silurian organic-rich shales from a core reveal a close correspondence, for the most part, between total organic carbon, total gamma-ray response, uranium content (as determined by spectral gamma-ray) and framboid parameters. Feasibility tests of the concept have been carried out at two exposures in southern Libya and may form the basis for improved Silurian organic-rich shale distribution maps and more precise age models for Silurian organic-rich depositional phases in northern Gondwana.

INTRODUCTION

Lower Silurian organic-rich ('hot') shales are widely distributed over the northern Gondwanan shelf, and, though laterally discontinuous, constitute important hydrocarbon source rocks (Lüning et al., 2000) (Figure 1). In wireline logs they are characterised by strong gamma-ray peaks (Figure 2) related to a high content of authigenic uranium that 'precipitated' under reducing conditions during deposition (Wignall and Myers, 1988). Distribution maps of this organic-rich unit are based mostly on hydrocarbon exploration well data and are generally only available for central parts of the Palaeozoic Saharan basins (Figure 1), where most of the wells are located. The presence or absence of the lower Silurian organic-rich shales at the basin margins (Figure 3) are largely unknown, because the Silurian shales that crop-out here are usually intensely weathered, with the organic matter largely oxidised and palynomorphs destroyed down to depths of several tens of metres. Present-day arid weathering in the Sahara was preceded by more humid weathering conditions before ca. 3,000 B.C. (e.g. Szabo et al. 1995). Field studies of black shale weathering profiles in the USA carried out by Petsch et al. (2000, 2001) indicated a 60-100% total organic carbon (TOC) loss in highly weathered samples relative to initial, unweathered TOC content.

Two techniques are proposed that may help to identify the Silurian 'hot' shale in Saharan exposures, and which have the potential to facilitate correlation of subsurface well data with outcrop data for improved basin-wide interpretations. The first technique, pyrite framboid grain size and abundance analysis (Wilkin et al., 1996, 1997), has been calibrated on a set of fresh samples from a well core (E1-NC174) from the central part of the Murzuq Basin (southwest Libya) (Figure 3). Framboids are discrete spheroidal aggregates of pyrite microcrystallites (e.g. Rickard, 1970), have typical grain sizes of 2-15 μm in black shales with the size distribution thought to be controlled by bottom-water redox-conditions (Wilkin et al., 1996). The second technique, outcrop-based spectral gamma-ray measurements (e.g. Lüning and Kolonic; in press), has been successfully applied in a past exploration campaign at the western margin of the Kufra Basin (southeast Libya, Figure 3) where elevated uranium radiation was detected in the basal part of the Silurian succession, indicating presence of shales that may have been organic-rich before weathering (Eales and Lüning; in prep.). Both techniques are based on the evidence that uranium and (at least the shapes of) pyrite framboids in the Saharan Silurian "black" shale exposures are less vulnerable to destruction by weathering than the organic matter.

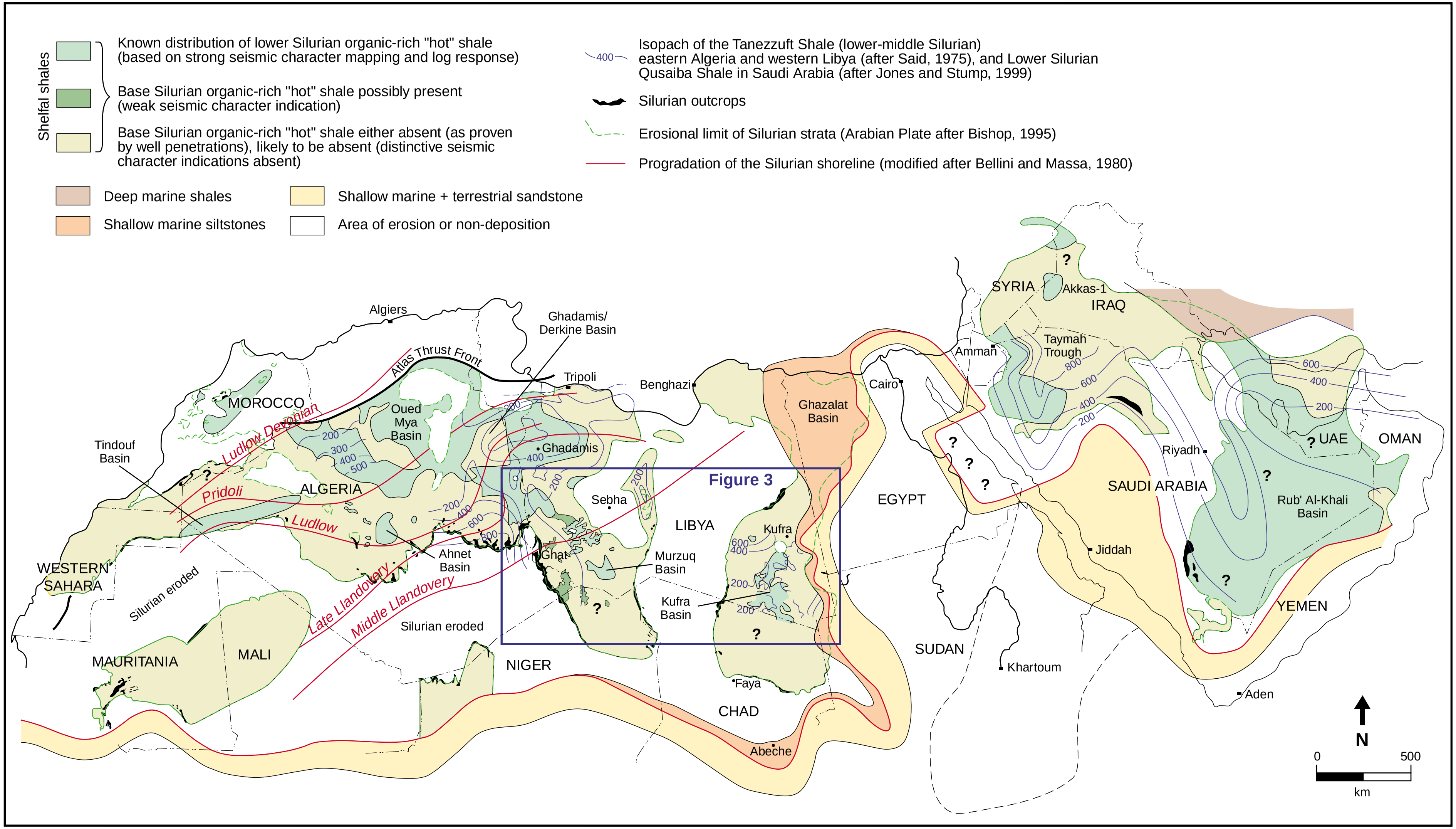

Figure 1: Known distribution of Silurian shales and early Silurian organic-rich shales in north Gondwana (from Craig et al., in prep.). Blue frame marks Libyan area enlarged in Figure 3.

MATERIAL AND METHODS

The Silurian organic-rich shale core studied comes from the exploration well E1 drilled in 1997 in the Murzuq Basin concession NC174, operated by LASMO Grand Maghreb Ltd and its partners (Davidson et al., 2000) (Figures 2, 3). The core is 17.4 m long and contains the whole of the organic-rich unit, plus the transitions to the under- and overlying organically leaner (but still organically rich) shales. A portable spectrometer (3" x 3" detector, GRS-2000, GF Instruments, formerly Geofyzika, Brno) was used for the core spectral gamma-ray measurements. Measurements were taken at 0.1 m intervals with a duration of one minute. Initial biostratigraphic ages for the hot shale reported in Lüning et al. (2000, their Figure 11) have been modified slightly because the new core spectral gamma-ray measurements revealed a 3 m shift between core and the original wireline log depths. For the spectral gamma-ray measurements in the field in southeast Libya, a Geofyzika GS-512 spectrometer (also 3" x 3" detector) was used with a 3 minute measuring interval.

The framboid analyses of the core thin sections were carried out with a Zeiss Axio-phot oil immersion microscope on a set of 22 samples distributed over the whole length of the core. Statistical framboid diameter data are based on counts of 150-250 framboids per sample. Framboid abundance counts were carried out in 10 fields of view under x1,000 magnification and averaged to one field of view.

For the geochemical investigations the samples were pulverised in an agate mortar. Organic and inorganic carbon were measured using a LECO CS-300 Carbon-Sulphur analyser (precision of measurements ± 3%). For TOC determination, inorganic carbon was carefully removed by repetitive addition of 0.25 N HCl. Calcium-carbonate ($CaCO_3$) contents were calculated following $CaCO_3 = (C_{inorg} - C_{org}) \times 8.33$. For total digestion analyses samples of about 50 mg were digested in a mixture of 3 ml HNO_3 (65%), 2 ml HF (40%) and 2 ml HCL (36%) of supra-pure quality at 200°C and 30 kbar in closed Teflon vessels (Heinrichs et al., 1986). After drying by evaporation the residue was re-dissolved with 0.5 ml HNO_3 (65%) and 4.5 ml deionised water.

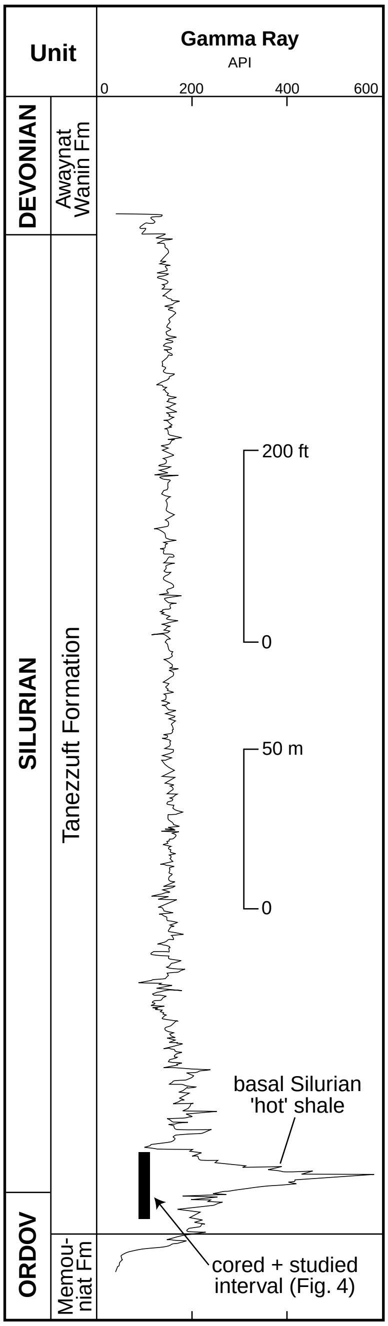

Figure 2: Total gamma-ray log of well E1-NC174. Latest Ordovician-early Silurian Tanezzuft Shale Formation with the basal radioactive, organic-rich 'hot' shale, overlying Ordovician sandstones of the Memouni at Formation (modified after Lüning et al., 2000).

E1-NC174 CORE (MURZUQ BASIN)

The Lower Silurian 'hot' shale in the E1-NC174 core consists of homogenous dark grey, hard shales with various degrees of mm- to sub-mm-scale pyritic lamination and banding. The pyritic laminations rarely appear to be slumped. Only a few medium to light grey shale intercalations commonly occur in the core. In some horizons larger, mm- to 10-mm-scale pyrite lenses and crystals are present. High TOC values of up to 13%, hydrogen indices between 300 and 400 (mgHC / gTOC) and the presence of type II kerogen indicate that these Silurian 'hot' shales are excellent oil-prone source rocks. Tmax values obtained from Rock-Eval pyrolysis (432-443°C) confirm an early mature level of thermal maturation. Biomarker analysis performed after desulphurisation of the total extract of the hot shale shows abundance of short-chain *n*-alkanes (C16-C22) and long-chain

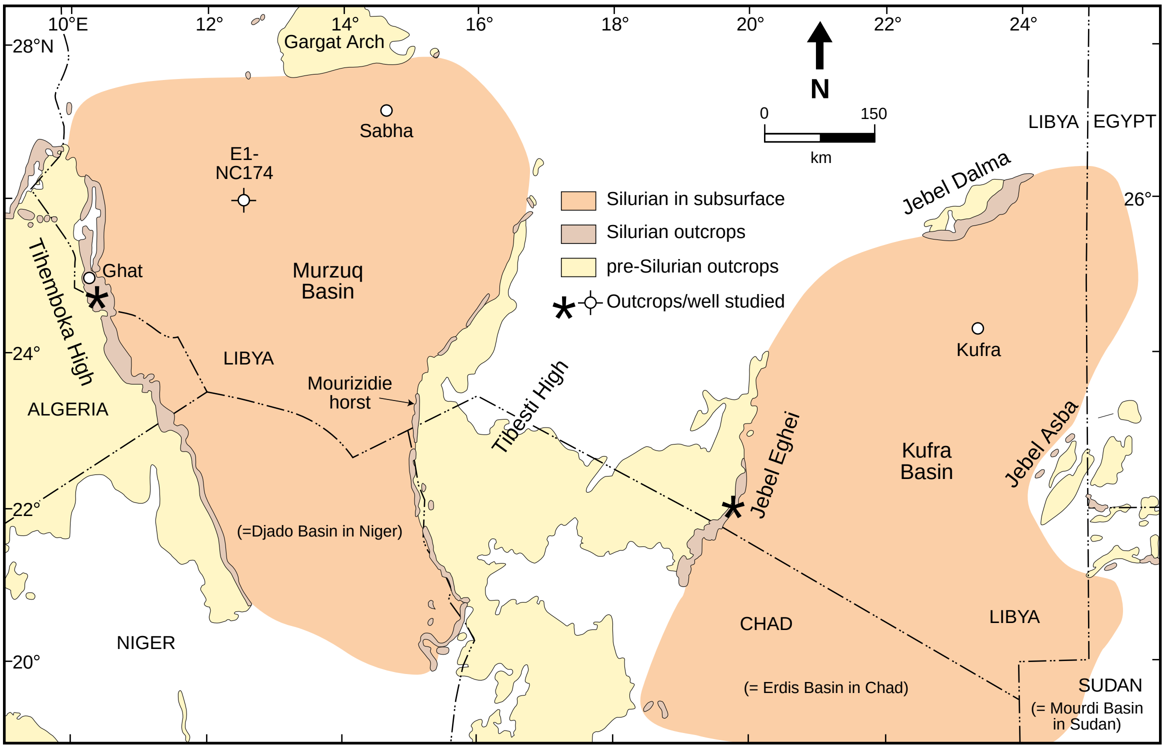

Figure 3: Locations of well E1-NC174 and the basal Silurian outcrops near Ghat and in Jebel Eghei.

(C25-C35) *n*-alkanes with no obvious odd-over-even predominance, steranes, hopanoids and acyclic isoprenoids. This composition indicates the presence of dominantly marine algal/bacterial organic matter. The carbonate content is low (1-7%).

The 'hot' shale in the core is clearly marked by high natural radiation values, as shown on both the total gamma ray wireline log (Figure 2) and in the total and uranium core spectral gamma-ray data (Figure 4). Notably, the gamma-ray and TOC trends in the Silurian organic-rich shales in the Sahara are strikingly similar (see also Lüning et al., 2000) so that the total, and in particular the uranium gamma-ray signals, can generally be used here as proxies for the vertical TOC distribution. Individual framboid diameters in the samples studied mostly range from 3-10 µm. Mean diameters for the different samples range only from 4.0-4.8 µm so that some of the variability observed may fall into the error range. Nevertheless, comparison of the gamma-ray/TOC trends with the framboid results reveals a close correspondence between the two datasets. The peak TOC interval generally coincides with the maximum framboid abundance as well as with minimum mean diameter and minimum size variability (standard deviation) (Figure 4). These results are supported by the framboid size model of Wilkin et al. (1996, 1997; see also Wignall and Newton, 1998) who postulated that framboid sizes in sediments are strongly dependent on the oxygen content in the water column. Generally, oxygen-poor conditions (i.e. often associated with organically richer sediments) are thought to be characterised by an increased formation of smaller framboids that are less variable in size than those in more oxygen-rich water columns (Wilkin et al., 1996). Similar relationships are also developed in the studied Silurian organic-rich shale, so that framboids may be used here as indicators for water-column oxygenation.

In a crossplot of the mean versus the standard deviation of the framboid size distribution, the samples with the highest TOC (interpreted as representing peak anoxia) plot in a separate field in the lower left corner of the diagram, marked by low mean framboid diameters and small standard deviations (Figure 5). Samples from the shale intervals underlying and overlying the peak anoxia zone (with increasing and decreasing TOC trends, respectively), generally plot to the right and upper right of the peak

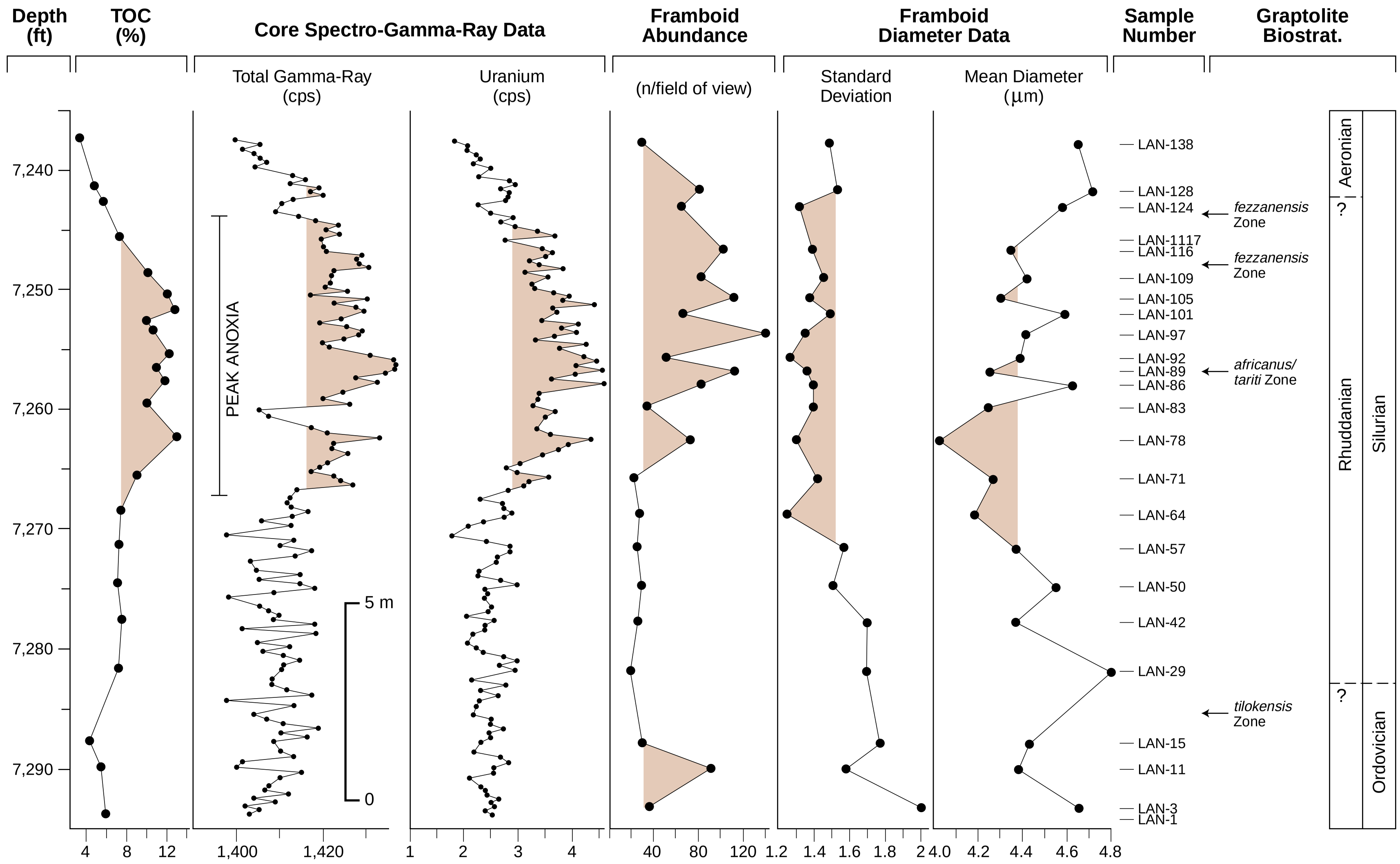

Figure 4: Correlation of TOC, core spectral gamma-ray, framboid and graptolite data of the basal Silurian 'hot' shale core of well E1-NC174. Interpreted zone of "peak anoxia" is based on maximum organic richness.

anoxia field. Nevertheless, all of the samples studied fall into the euxinic plot field, as defined by Wilkin et al. (1996) (Figure 5b), indicating that oxic-dysoxic conditions were not reached in the studied interval. The ecological changes recorded in this Silurian organic-rich shale core, therefore, only reflect variations within a fully euxinic regime. This also explains the rather subtle, nevertheless characteristic numerical changes observed in the standard deviation and mean diameter of the framboids, compared to greater variations in similar studies by e.g. Wignall and Newton (1998, 2001) whose studied successions also included oxic-dysoxic horizons.

The correlation of the apparently genetically interlinked TOC, spectro-gamma-ray and framboid trends in the studied Silurian organic-rich shale core, sets a first standard to be used and tested in more detailed Silurian outcrop studies. At outcrop, the original concentration of the more-or-less completely oxidised organic matter may now be approximated using the (less altered) framboid and spectral gamma-ray data.

GHAT OUTCROP SAMPLE

A feasibility test of the framboid-TOC-correlation technique has been carried out on strongly weathered grey and red shales that were collected in 1998 just above the base of the Silurian (Tanezzuft) shale succession in a field section near Ghat at the western margin of the Murzuq Basin (Figure 3). Unfortunately, spectral gamma-ray measurements were not carried out. The graptolite fauna of this horizon contains *Neodiplograptus africanus* (Legrand) (Figure 6), indicating the *africanus* / *tariti* Biozone of the Lower Llandovery (Rhuddanian). This age corresponds to the age of the peak of the interpreted anoxia as dated using graptolite biostratigraphic analysis in the E1-NC174 core (Figure 4). Therefore, the Ghat samples are also likely to have been organically rich prior to oxidization which results in present-day residual TOC values that typically lie around 0.05-0.2%.

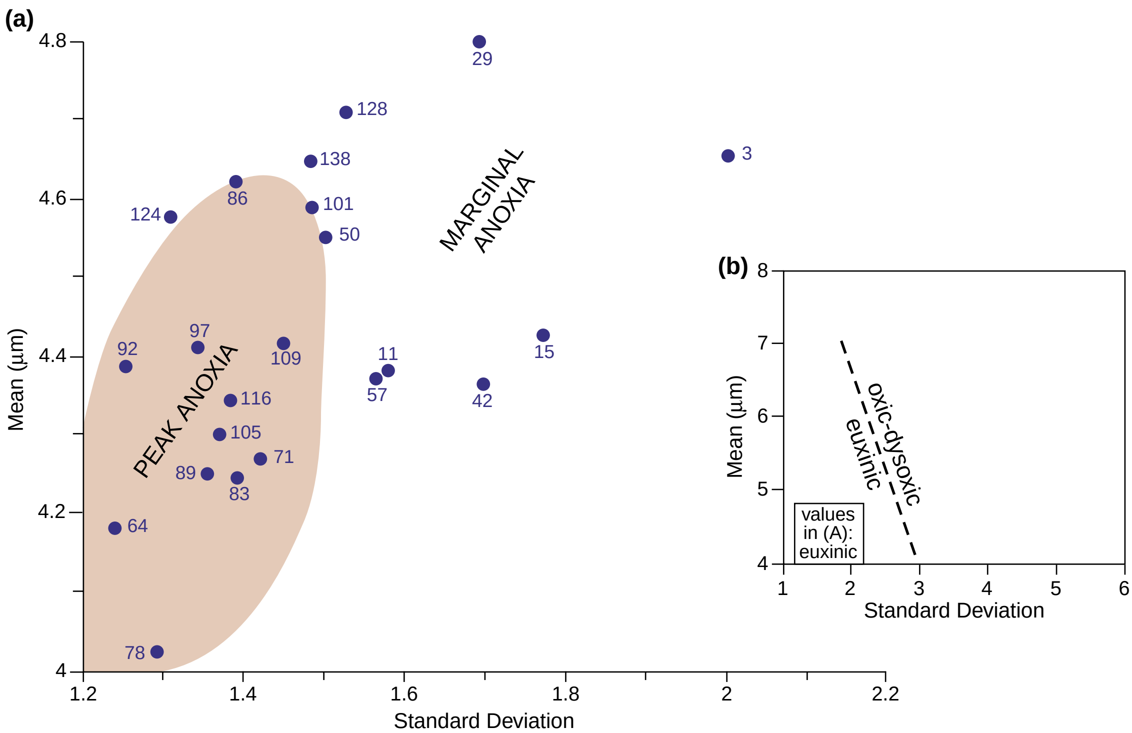

Figure 5: Plots of the mean versus the standard deviation of the framboid size distribution. (a) Studied Silurian 'hot' shale E1-NC174. Samples from the peak anoxia interval (see Figure 4) plot in a distinct field in the lower left corner of the diagram marked by low mean framboid diameters and small standard deviations. (b) Typical plot fields for sediments deposited under oxic-dysoxic and euxinic conditions according to Wilkin et al. (1996). Notably, all samples analysed from the E1-NC174 core plot in the euxinic facies field.

Although two of the three Ghat samples were severely weathered to such an extent that any pyrite framboids were completely destroyed in the thin sections, one (red-coloured) sample contained framboids and framboid-like structures that are thought to represent altered framboids (Figure 7a). Despite the framboids being partially destroyed and having increased sizes due to incipient recrystallisation, their abundance is comparable to that in the fresh core samples from the E1-NC174 well (Figure 7b), so that similar, oxygen-poor depositional conditions may be assumed. The samples originate from the very surface of the exposure. Future studies with moderate excavation (e.g. 0.5 m depth) may produce shale samples from outcrop that even better preserve the original framboid diameters. The presence of graptolites in shales alone already indicates low oxygen conditions at the sea floor because otherwise their rhabdosomes would have oxidised and/or been destroyed by bioturbation.

In a study of black shale weathering profiles in the USA, Petsch et al. (2000) found that pyrite loss coincides with, or precedes, TOC loss during weathering. It is unclear whether this vulnerability to weathering affects framboids and larger pyrite crystals to a similar degree. Despite this vulnerability of pyrite to destruction by weathering, the statistical framboidal size and abundance parameters in not completely weathered black shales, may still allow full reconstruction of the depositional redox conditions, as long as the framboidal shapes and/or abundances are generally unaltered.

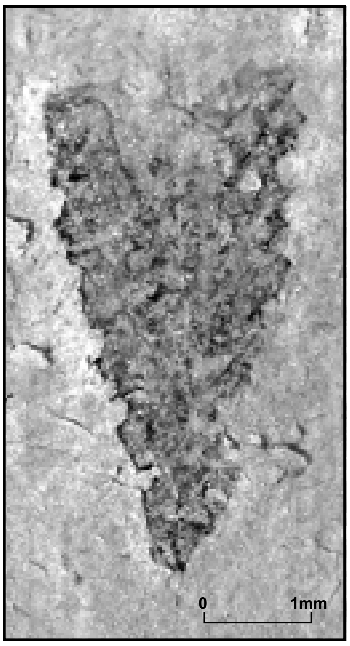

Figure 6: *Neodiplograptus africanus* (Legrand), index graptolite for the Lower Llandovery *africanus* / *tariti* Biozone (Figure 4) from a Silurian outcrop in the Ghat area (southwest Libya). Specimen deposited as BGS FOR 5357 in the British Geological Survey collections, Keyworth.

KUFRA OUTCROP SPECTRAL GAMMA-RAY

Silurian shales are also exposed around the Kufra Basin in southeast Libya and the basal shale interval was found exposed in one locality in Jebel Eghei at the western margin of the basin (Seilacher et al., 2002; Eales and Lüning; in prep.) (Figure 3). Natural uranium radiation values of particular horizons in this basal interval are up to double (~10 ppm) the normal lean shale baseline values (~5 ppm, as measured in the organically lean middle and upper parts of the Silurian Tanezzuft Formation in the same area). This elevated uranium content is interpreted as a relict of the radioactive hot shale, and therefore can be used to identify the unit at exposure even though the original organic matter is now oxidised. In the same strata circular shapes (20 mm diameter) with a fine radial structure where detected which Seilacher (2001: p. 52ff, his Figure 11e) interpreted as leached-out pyrite disc shadows, characteristic of originally organic-rich shales.

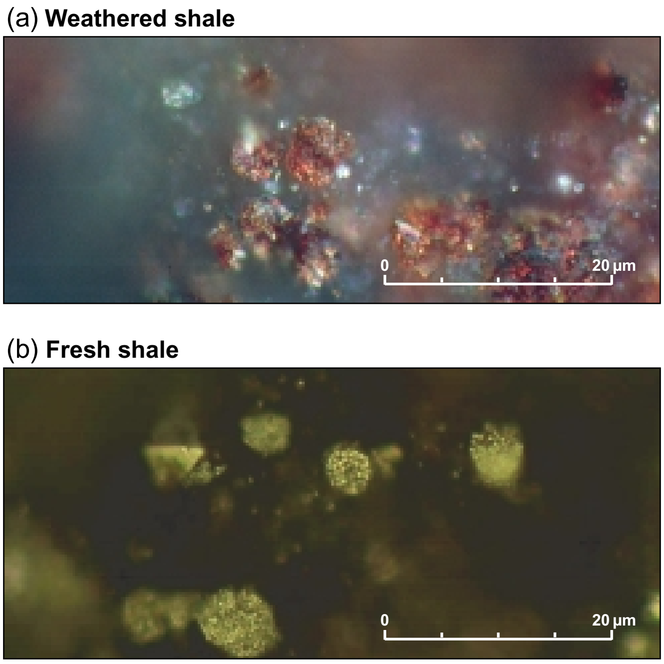

Figure 7: Framboids in the Silurian 'hot' shale in the Murzuq Basin. (a) Strongly weathered outcrop sample from the Ghat region. (b) Fresh core sample (LAN2-64) from well E1-NC174. Location map in Figure 3.

DISCUSSION

Both gamma-ray and framboid techniques, when combined, may help in mapping the presence or absence of the organic-rich basal Silurian shale interval at exposure in the Saharan Palaeozoic basins. Once the hot shale has been identified at outcrop, it can be dated accurately by high-resolution graptolite biostratigraphy, i.e. using macrofossils that are usually destroyed in well cutting samples but are more resistant than palynomorphs to surface and near-surface weathering processes. It is hoped that this approach will help increase our understanding of synchronous versus diachronous events during Silurian anoxic phases in North Africa and the Arabian Peninsula. While the Early Llandovery (Rhuddanian) hot shale described in this study may be the most important in the southern Libyan basins, a second significant anoxic phase occurred during the Late Llandovery / Wenlock in parts of northern Gondwana, including the Ghadames / Berkine Basin (Lüning et al.; in press a) (Figure 1).

Naturally, the correlation of natural uranium radiation, TOC, and framboid properties depends on a complex set of ecological parameters of which certain properties may change laterally and temporally. It is therefore clear that any such proxies are only valid for stratigraphically and regionally well-defined and calibrated sedimentary systems (see also Schmoker, 1980, 1981). For example, the Frasnian hot shale in North Africa (Lüning et al.; in press b) is characterised by significantly lower uranium radiation levels than the Silurian 'hot' shales at comparable organic richnesses. Radiation levels are even lower in the late Cenomanian-early Turonian organic-rich units in Morocco and framboids are dramatically less abundant (Lüning and Kolonic; in press). More detailed studies are obviously necessary to test the two methods and their validity in greater detail.

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