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Is Strontium-isotope stratigraphy a reliable tool for dating shallow-marine platform carbonates at the Barremian-Aptian transition? Review of western Tethyan case studies

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Abstract: Strontium-isotope measurements on Lower Cretaceous marine rocks derive from belemnite material sampled in ammonite-constrained basinal successions. A group of values with a narrow range across the Barremian/Aptian boundary does not allow the separation of the uppermost Barremian (*Martelites sarasini* ammonite zone) from the lower Aptian *pro parte* (*Deshayesites oglanlensis*-*D. forbesi* ammonite zones). Growing numbers of studies applied Sr-Isotope Stratigraphy (SIS) on Barremian-Aptian shallow-marine sequences (Urgonian facies) in order to solve controversial results obtained by using different shallow-water biological time markers. Based on re-examination of case studies, we conclude that Sr-isotope values can neither be used to prove nor to disprove the location of the putative Barremian/Aptian boundary based on biostratigraphy. Pending more data available, SIS should be used with caution for dating ammonite-free carbonate sediments in the corresponding time interval.

Key-words:

- Strontium Isotope Stratigraphy;
- Cretaceous;
- Ammonites;
- Urgonian platform;
- Tethys

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Résumé : ***La stratigraphie isotopique du Strontium est-elle une méthode fiable pour dater les plates-formes carbonatées à la transition du Barrémien à l'Aptien ? Révision de cas d'études en Téthys occidentale.-*** Les mesures de l'isotope du Strontium dans des roches carbonatées marines du Crétacé inférieur proviennent de restes de bélemnites récoltés dans des séries de bassins datées directement par ammonites. Autour de la limite Barrémien/Aptien, une gamme étroite de valeurs du Strontium ne permet pas de distinguer le Barrémien supérieur (Zone d'ammonite à *Martelites sarasini*) de l'Aptien inférieur pro parte (zones d'ammonite à *Deshayesites oglanlensis* et *D. forbesi*). Pourtant, l’application de la Stratigraphie Isotopique du Strontium (SIS) sur des séquences marines carbonatées barrémo–aptiennes (à faciès urgonien) apparaît dans un nombre croissant d’études, essentiellement afin de résoudre les datations souvent controversées des marqueurs biologiques d’environnements peu profonds. La révision de ces cas d'étude montre que l’utilisation des valeurs de l'isotope du Strontium n’est pas un outil fiable pour prouver ou réfuter la localisation de la limite Barrémien/Aptien telle que déduite par la biostratigraphie. Dans l'attente de données complémentaires, la SIS doit être utilisée avec précaution pour dater des séries carbonatées non datées directement par ammonites dans l'intervalle de temps concerné.

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Mots-clefs :

- stratigraphie isotopique du Strontium ;
- Crétacé ;
- ammonites ;
- plate-forme urgonienne ;
- Téthys

1. Introduction

Extensive data have been collected on the changes of the strontium isotopic record ($^{87}Sr/^{86}Sr$) of Lower Cretaceous marine rocks and fossils over the three last decades (*e.g.*, Jones *et al.*, 1994; Bralower *et al.*, 1997; Howarth & McArthur, 1997; McArthur *et al.*, 2001, 2004, 2012; Masse & Steuber, 2007; Burla *et al.*, 2009; Bodin *et al.*, 2009, 2015; Huck *et al.*, 2010, 2011, 2012, 2013; Mutterlose *et al.*, 2014; Huck & Heimhofer, 2015). Strontium isotope measurements from well-preserved, originally calcitic, shell material (oysters, rudists, belemnites, planktonic foraminifera) are assumed to reflect the isotopic composition of the sea-water (Veizer, 1989; Mearon *et al.*, 2003; Strohmenger *et al.*, 2010), but its record has also been developed as a chronostratigraphic tool using external calibration by biological (ammonites, planktonic foraminiferas, calcareous nannofossils) and/or geochemical ($\delta^{13}C$, $\delta^{18}O$) markers on reference basinal series (*e.g.*, Jones *et al.*, 1994; McArthur *et al.*, 2001, 2004; Bodin *et al.*, 2009, 2015).

In recent years, Strontium-Isotope Stratigraphy (SIS) has been used to date ammonite-free, shallow-marine carbonate sequences of Barremian-Aptian age (Urgonian facies) in order to solve controversial results using orbitolinids and/or rudists as time markers (*e.g.*, Masse & Steuber, 2007; Burla *et al.*, 2009; Huck *et al.*, 2011; Huck & Heimhofer, 2015). Our paper addresses this problem by a re-appraisal of the Barremian-Aptian strontium isotope record with respect to the standard Mediterranean ammonite zonation, and re-examine SIS-derived datings of well-documented Urgonian series of the western Tethys. We will try to decipher the underlying causes of the mismatch between this technique and biostratigraphy.

2. Generalities on the Barremian-Aptian transition

In the Mediterranean Tethys, the upper Barremian includes four ammonite zones according to the current standard zonation discussed by the IUGS Lower Cretaceous Ammonite Working Group (Reboulet *et al.*, 2014) (Fig. 1). The two uppermost zones are the acme of uncoiled (*I. giraudi* Zone) and recoiled (*M. sarasini* Zone) Heteroceratidae that provide long-distance correlative bioevents at the scale of the Tethys (Klinger *et al.*, 1984; Delanoy, 1997; Lehmann *et al.*, 2015).

By means of ammonites, the base of the Aptian is defined at the First Appearance Datum of the Deshayesitidae (*e.g.*, Rawson, 1983; Birkelund *et al.*, 1984; Reboulet *et al.*, 2011, 2014), and more specifically at the FAD of *Deshayesites oglanlensis*. Unfortunately, this ammonite bioevent has so rarely been documented as a result of ammonite-free stratigraphic interval (SE France - Delanoy, 1995; SE Italy - Landra *et al.*, 2000; Tunisia - Lehmann *et al.*, 2009; NE Spain - Bover-Arnal *et al.*, 2016; Turkmenistan - Cecca *et al.*, 1999a; Japan - Ando *et al.*, 2002), syn-depositional hiatus (Spain - Aguado *et al.*, 1997) or erosion (North Italy - Lukeneder *et al.*, 2012), while continental conditions prevailed over most of the Boreal Realm at that time (Lehmann *et al.*, 2015, and references therein).

The lower Aptian, formerly referred as Bedoulian in the Cassis-Roquefort-la-Bédoule unit-stratotypical succession of southern France (Fabre-Taxy *et al.*, 1965; Moullade *et al.*, 2000), is divided into four ammonite zones (Fig. 1). However, the definitions of their boundaries have been much debated in the past decades (*e.g.*, Casey *et al.*, 1998; Ropolo *et al.*, 1999, 2000a, 2000b, 2006; Moreno-Bedmar *et al.*, 2009, 2010, 2012, 2014; Bersac & Bert, 2012, 2015; Frau *et al.*, 2015), and no consensus has been yet reached regarding their vertical extension and long-distance correlation. This is the case for the *D. forbesi-D. deshayesi* zones including the spreading and culmination of the Ocean Anoxic Event (OAE) 1a (Moreno-Bedmar *et al.*, 2009). The latter refers to the worldwide, massive deposition of organic-rich black shales in deep basinal settings associated to a major perturbation in the global carbon cycle (Menegatti *et al.*, 1998). The carbon isotope curve of the OAE 1a has been divided in eight diagnostic segments (C1 to C8) by Menegatti *et al.* (1998), among which the black shales deposition culminates through the C3 *pro parte* to C6 isotopic segments in the Alpine Tethys and its surrounding areas (Li *et al.*, 2016, and references therein).

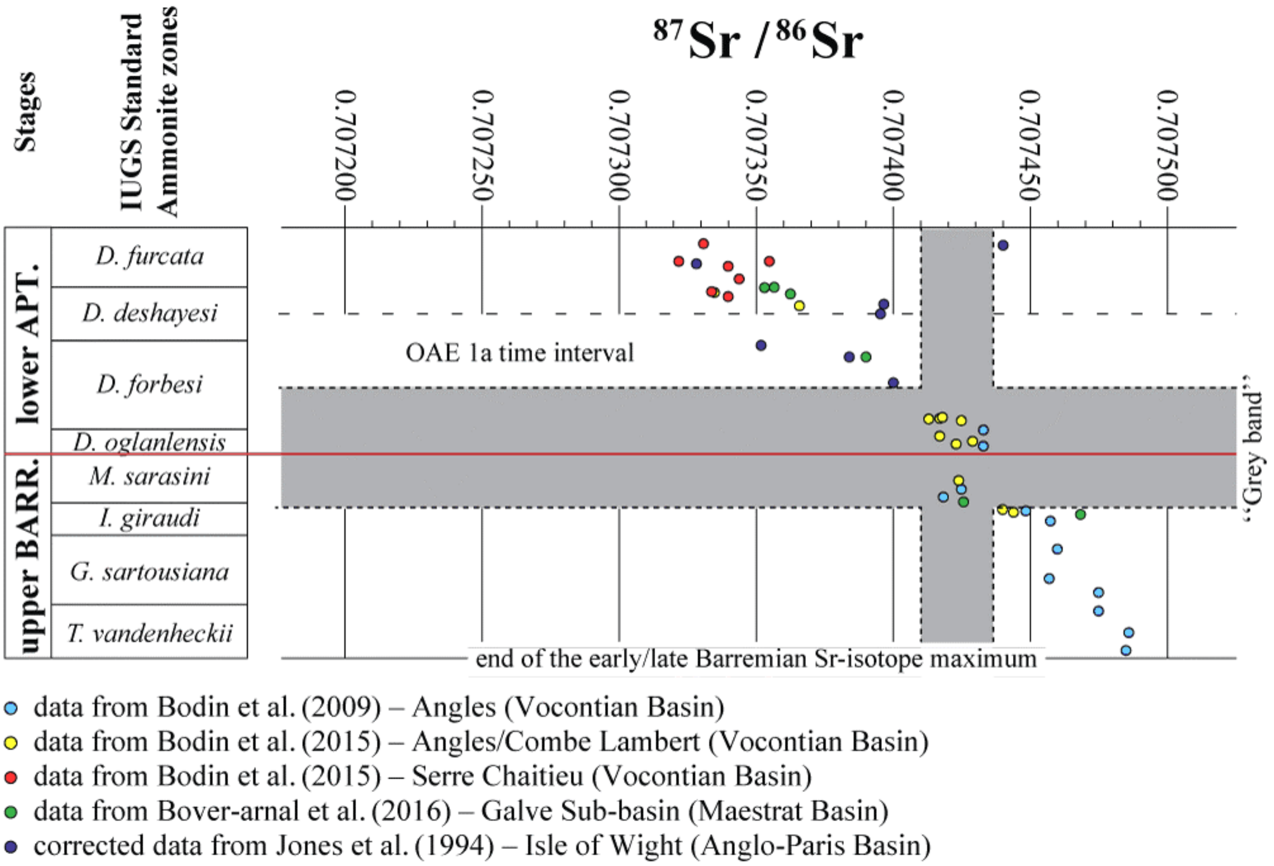

Figure 1: Succession of the Barremian-Aptian marine $^{87}Sr/^{86}Sr$ ratio from reference ammonite-bearing successions of the western Tethys (based on JONES *et al.*, 1994; BODIN *et al.*, 2009, 2015; BOVER-ARNAL *et al.*, 2016) plotted with the standard Mediterranean ammonite zonation (REBOULET *et al.*, 2014). Note that the "grey band" spans the SIS data below the OAE 1a time interval, and the ratio does not separate the uppermost Barremian (*Martelites sarasini* Zone) from the lowermost Aptian (*Deshayesites oglanlensis-D. forbesi pro parte* zones).

Reinterpretation of the ammonite-bearing successions of southern France (FRAU *et al.*, 2015, work in progress) and northern Spain (MORENO-BEDMAR *et al.*, 2009, 2012; NAJARRO *et al.*, 2011) indicates that the OAE 1a-related time interval encompasses the upper *D. forbesi* and lower *D. deshayesi* standard zones. These results contradict the current ammonite age-calibration of the OAE 1a used in the two last versions of the Geologic Time Scale which located this event in the upper part of the *D. deshayesi* Zone (GRADSTEIN *et al.*, 2012; OGG *et al.*, 2016). This misinterpretation relies on the use of the *D. deshayesi* Zone as defined by BRÉHERET (1997) and ROPOLO *et al.* (2000b, 2006) in the reference successions of the Vocontian (Angles-Combe Lambert-Glaise composite section) and South Provence basins (Cassis-Roquefort-la-Bédoule) which proved to be erroneous (MORENO-BEDMAR *et al.*, 2009; FRAU *et al.*, 2015, work in progress).

3. Sr-isotope trends in the Barremian-Aptian transition

Sr-isotope data from ammonite-bearing beds at the Barremian-Aptian transition are limited and derive from the reference successions of southern England (JONES *et al.*, 1994; MCARTHUR *et al.*, 2004), southeast France (BODIN *et al.*, 2009, 2015) and northeast Spain (BOVER-ARNAL *et al.*, 2016). Each case study is discussed below and synthesized in Figure 1.

3.1. Southern England

JONES *et al.* (1994) provided the first high-resolution Sr-isotope curve based on ammonite-constrained, belemnites and oysters from the Middle Jurassic to Lower Cretaceous of Great Britain. These data stand as the reference SIS curve for the Boreal Cretaceous, together with the revised data of MCARTHUR *et al.* (2004). Regarding our studied interval, JONES *et al.* (1994) provided two values from the upper Barremian of the Speeton Clay (Yorkshire Coast) and nine values from the lower Aptian of the Lower Greensand (Isle of Wight).

Upper Barremian values derive from belemnites collected in association with the ammonites *Parancyloceras elegans* and *P. denckmanni* defining the first two zones of the upper Barremian in Boreal settings (Mutterlose & Bornemann, 2000). According to Jones *et al.* (1994), and later confirmed by McArthur *et al.* (2004), the *P. elegans* Zone records the most positive Sr-isotope values of the Lower Cretaceous, around 0.707489 if we acknowledge the correction proposed by Bralower *et al.* (1997) adding 0.000025 to the original values.

Lower Aptian values derive from beds belonging to the *Prodeshayesites fissicostatus*, *Deshayesites forbesi*, *Deshayesites deshayesi* and *Tropaeum bowerbanki* ammonite zones as defined by Casey *et al.* (1998). Isotope values fluctuate from 0.707396 to 0.707430, if we acknowledge the same correction proposed by Bralower *et al.* (1997). Long-distance correlation of Lower Greensand ammonite zones with the Mediterranean standard zonation is still a matter of debate (Moreno-Bedmar *et al.*, 2009, 2014; Bersac & Bert, 2012, 2015), but external calibration by carbon isotope (Gröcke *et al.*, 1999) suggests that the Lower Greensand ammonite zones encompass the C2 *pro parte* to C7 isotopic carbon segments of Menegatti *et al.* (1998); an interval spanning the *D. forbesi* Zone *pro parte* to the *D. furcata* Zone in reference ammonite-constrained successions of the Mediterranean Tethys (Frau *et al.*, work in progress).

3.2. Southeast France

Bodin *et al.* (2009) documented Sr-isotope values from belemnites collected in the upper Hauterivian, Barremian and lowermost Aptian beds from the Vocontian-type Angles section (southeast France), that was formerly designed as unit-stratotype for the Barremian Stage (Busnardo, 1965).

Sr-isotope measurements describe a long-lasting increase through the upper Hauterivian and lower Barremian, ranging from 0.707466 to 0.707524, with plateau values occurring in the *K. nicklesi-M. moutonianum pro parte* zones. Maximum value (*i.e.*, 0.707524) falls in the uppermost *K. compressissima* Zone. A decreasing trend follows with minimum values achieved in the lowermost *M. sarasini* Zone (*i.e.*, values equal to 0.707418 and 0.707425). Sr-isotope values in the lower Aptian beds assigned to the *D. oglanlensis* (pro *Tuarkyricus*) Zone and lower *D. forbesi* (pro *D. weissi*) Zone by Delanoy (1995) are only based on two measurements equal to 0.707433.

Bodin *et al.* (2015) complemented the Sr-isotope curve by new measurements (10 samples) from the *I. giraudi-D. forbesi pro parte* zones in the lateral equivalent Combe Lambert section (see details in Delanoy, 1995). Values vary little in this time interval and belong to a continuum ranging from 0.707448 to 0.707413

Additional Sr-isotope measurements were documented in two other Vocontian sections (Serre Chaitieu and Glaise), which exhibit an undisturbed OAE 1a succession (locally referred to as Niveau Goguel - see details in Bréheret, 1995). This interval spans the C3 to C6 isotopic carbon segments (Bodin *et al.*, 2009, Fig. 6). Unfortunately, only data from post-OAE 1a levels assigned to the upper *D. deshayesi* and lowermost *D. furcata* zones are given by the authors, because the Niveau Goguel and its enclosing strata are devoid of belemnite remains. Sr-isotope measurements are significantly low and range from 0.707366 to 0.707305.

3.3. Northeast Spain

Bover-Arnal *et al.* (2016) documented two Sr-isotope values obtained from oysters calibrated by ammonite occurrences in the shallow-water platform carbonates of the Maestrat Basin (NE Spain): 0.707466 in beds of the upper *I. giraudi* Zone, and 0.707425 in beds of the lower *M. sarasini* Zone. From the same area, a value of 0.707390 was measured on a rudist shell from a *Lithocodium*-bearing horizon in the lower part of the marl-dominated, ammonite-bearing Forcall Formation. According to Bover-Arnal *et al.* (2016), this horizon is coeval with the major positive carbon shift that mirrors the C4 isotopic segment of Menegatti *et al.* (1998), calibrated with the uppermost *D. forbesi* Zone (Moreno-Bedmar *et al.*, 2010). Three additional Sr-isotope measurements have been documented from rudist shells of the Villaroya de los Pinares Fm. Bounded by ammonites dating the *D. deshayesi-D. furcata* zones. The average value is 0.707356.

3.4. Standard Sr-isotope curve

The Sr-isotope compiled curve established by Bodin *et al.* (2009, 2015), supplemented by the data of Jones *et al.* (1994) and Bover-Arnal *et al.* (2016), shows the following features and trends (Fig. 1):

(i) Sr-isotope measurements achieve maximum values through the lower Barremian and record an overall decreasing trend in the upper Barremian *pro parte* (*T. vandenheckii-I. giraudi* zones). The ratio is characterized by high values comprised between ~0.707475 and 0.707500. This trend conforms well to that established in Boreal settings by Jones *et al.* (1994) and McArthur *et al.* (2001, 2004), although it does not match with the biostratigraphic definition of the lower/upper Barremian boundary by means of ammonites between the Boreal and Tethyan standard scale (Mutterlose *et al.*, 2014);

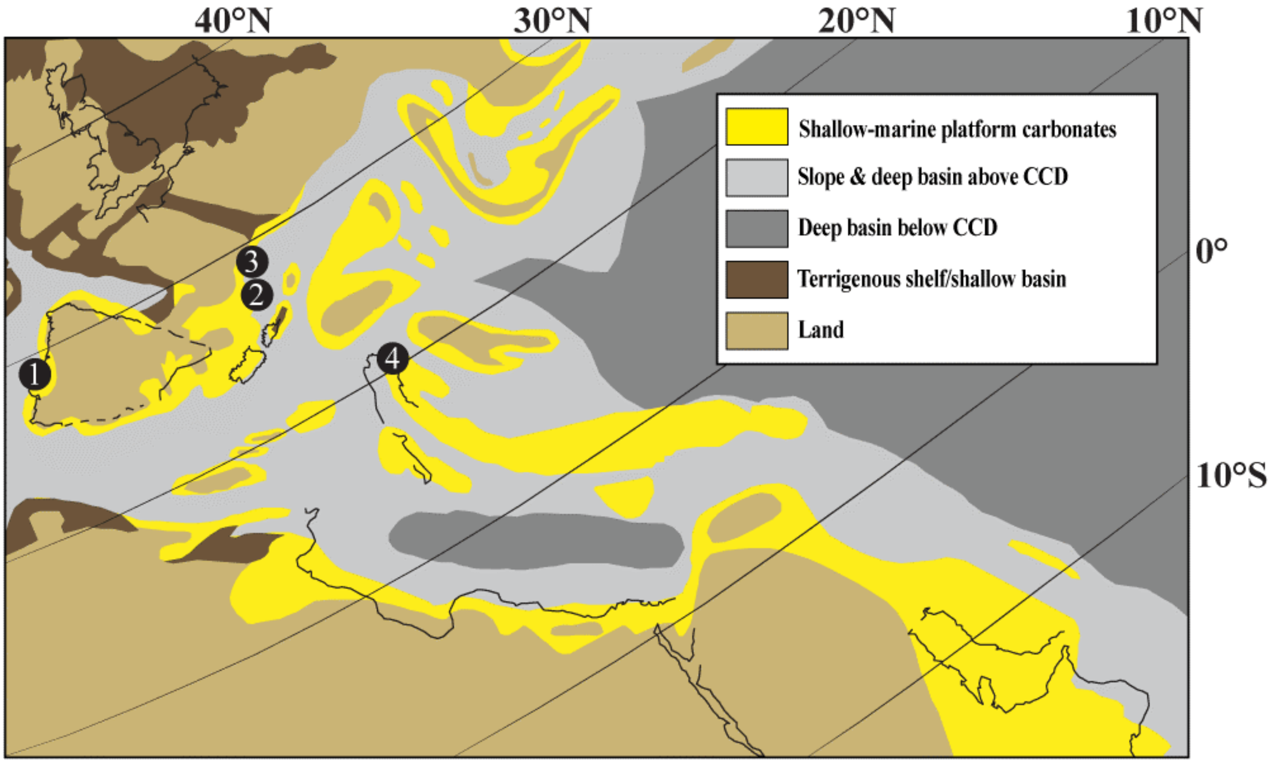

Figure 2: Barremian-Aptian palaeogeographic map of the western Tethys and location of the case studies (modified from Steuber *et al.*, 2005). Numbers refer to case studies: 1. Estremadura (Portugal) 2. southern Provence (France), 3. northern Subalpine Chains (France), 4. Istria (Croatia).

(ii) A group of Sr-isotope measurements is bounded by values of 0.707435 to 0.707410 and forms a break in the overall decreasing trend of the Sr-isotope curve. This group of values plots in a "grey band" in which SIS is unreliable for distinguishing the uppermost Barremian (*M. sarasini* Zone) from the lowermost Aptian (*D. oglanlensis*-*D. forbesi pro parte* zones);

(iii) Measurements from the OAE 1a-related time interval are limited in both realms but values are lower than a threshold fixed at 0.707400 through the *D. forbesi*-*D. deshayesi pro parte* zones;

(iv) A newly recognized decreasing trend of Sr-isotope values ranges from 0.707366 to 0.707305, which characterises post-OAE 1a levels and spans the upper *D. deshayesi* to *D. furcata* zones.

4. SIS in Urgonian platform carbonates: Review of case studies

SIS based on shell material has been widely used for dating ammonite-free, carbonate platforms of Barremian-Aptian age (Urgonian facies) in the western Tethys (Fig. 2). This is the case in eastern Portugal (Burla *et al.*, 2009; Huck *et al.*, 2012), southern (Huck & Heimhofer, 2015) and south-eastern (Huck *et al.*, 2011, 2013) France, and south-western Croatia (Huck *et al.*, 2010). Results and interpretations of these studies are exposed and discussed below. Note that carbon-isotope stratigraphy was frequently used in support of SIS-derived datings in these case studies. The reliability of this method is highly questionable in Lower Cretaceous shallow-marine carbonates, and its use for stratigraphic purpose most often led to controversial uncertainties (see recent discussion in Godet *et al.*, 2016). This problem is beyond the scope of this work and will be addressed elsewhere.

4.1. Estremadura

In Estremadura (eastern Portugal), Sr-isotope measurements were performed by Burla *et al.* (2009), and re-examined by Huck *et al.* (2012), from oysters sampled in the Urgonian Cresmina Fm., including the Barremian-Aptian transition according to bio- and chemostratigraphic markers (Fig. 3). Values are in the range of 0.707405 to 0.707433 (actually 0.707385 to 0.707457 if acknowledging error bars) for the rudistid limestones (Ponta Alta Mb.), which are assumed to be of early Aptian age with the occurrence of caprinid rudists (Rey, 1992; Burla *et al.*, 2008), and 0.707407 to 0.707424 (actually 0.707379 to 0.707450 if acknowledging error bars) for the overlying orbitolinid-bearing beds (Praia de Lagoa Mb.) of late early and/or early late Aptian age (Burla *et al.*, 2008), that also include angiosperm pollen (Heimhofer *et al.*, 2007) and calcareous nannofossils (Da Gama *et al.*, 2012).

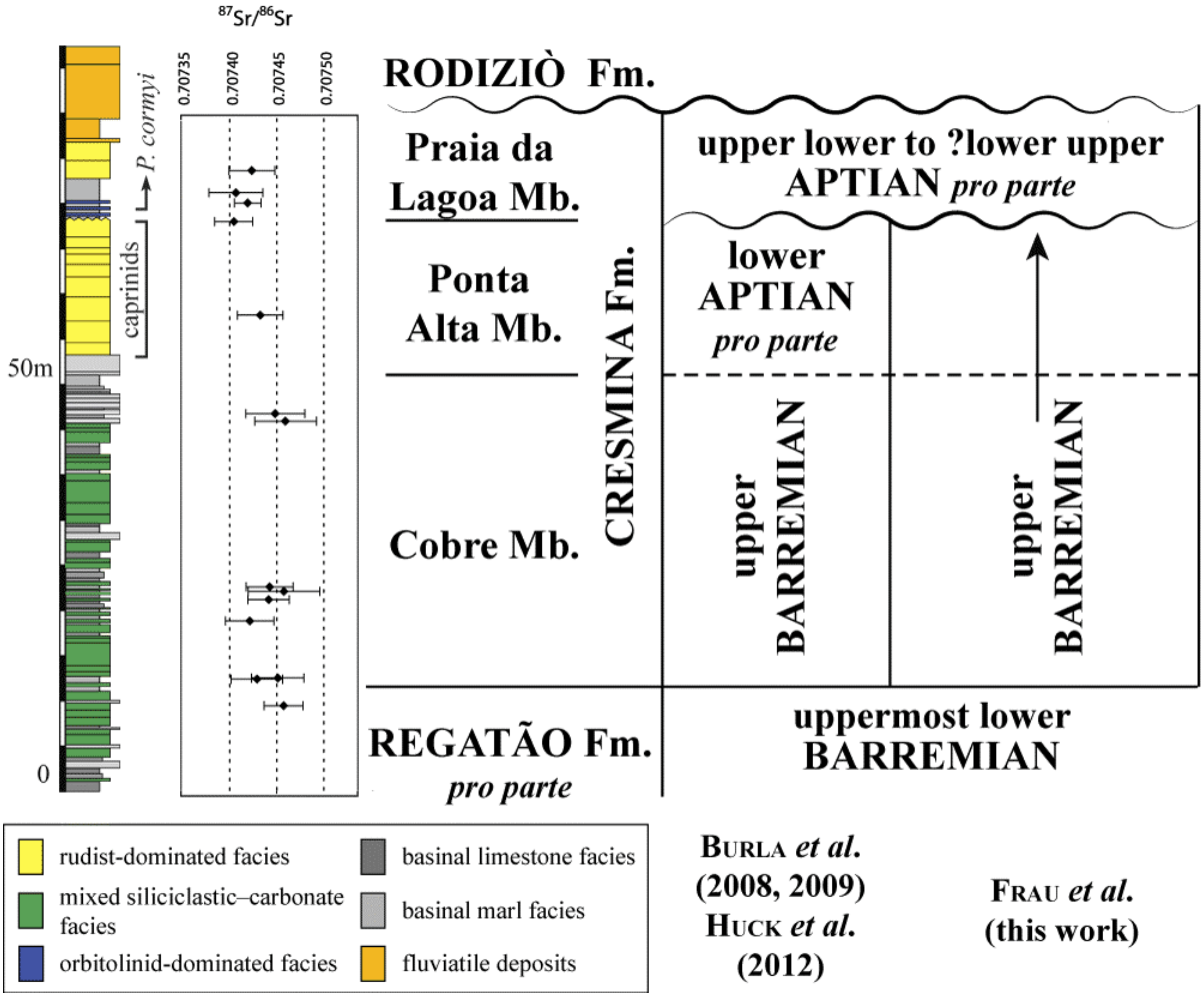

Figure 3: Litho- and Sr-isotope stratigraphy of the Cresmina section (Estremadura, Portugal) with stratigraphic interpretations proposed by BURLA *et al.* (2008, 2009) and FRAU *et al.* (this work). The late Barremian age of the Ponta Alta Mb. follows the recent age-assignment of the Urgonian-type, North Provence caprinid-bearing beds (see FRAU *et al.*, accepted). Sinuous lines mark major discontinuities.

The range of values is almost similar for both members and, if plotted onto our compiled curve, it spans most of the upper Barremian and the entire lower Aptian including the "grey band". Although the occurrence of the orbitolinid *Praeorbitolina cormyi* in the Praia de Lagoa Mb. pinpoints the upper lower Aptian boundary (its range being constrained by ammonites to the *D. deshayesi* to *D. furcata* Zones in southeast France - SCHROEDER *et al.*, 2010, and references therein), the calibration of the caprinid-bearing Ponta Alta Mb. is open to discussion and could be either latest Barremian or earliest Aptian in age (Fig. 3).

It is noteworthy that the dating of the caprinid-bearing beds of the Mediterranean Tethys mostly relies on the comparison with the Urgonian-type, North Provence platform series (southern France), in which the caprinid-bearing unit (U^2 Unit *sensu* LEENHARDT, 1883) up to now assigned to the *D. oglanlensis*-*D. forbesi pro parte* zones by indirect correlation with the surrounding basinal settings (MASSE, 1995, 2003; MASSE & FENERCI-MASSE, 2011). Isolated Sr-isotope values from rudist shells measured in the U^2 Unit also fall in the "grey band" (*i.e.*, 0.707418 to 0.707449 - MASSE & STEUBER, 2007), and cannot be confidently used to pinpoint the age of the Urgonian-type caprinid-bearing beds by SIS. Their direct calibration is thus a challenging issue that could have major consequences on dating the demise of the Urgonian rudistid-platform ecosystems at the scale of the Mediterranean Tethys. Note that the current dating of the U^2 Unit has been recently challenged by FRAU *et al.* (2017) who alleged a latest Barremian age; this age is now confirmed by the discovery of overlying ammonite occurrences (FRAU *et al.*, 2018).

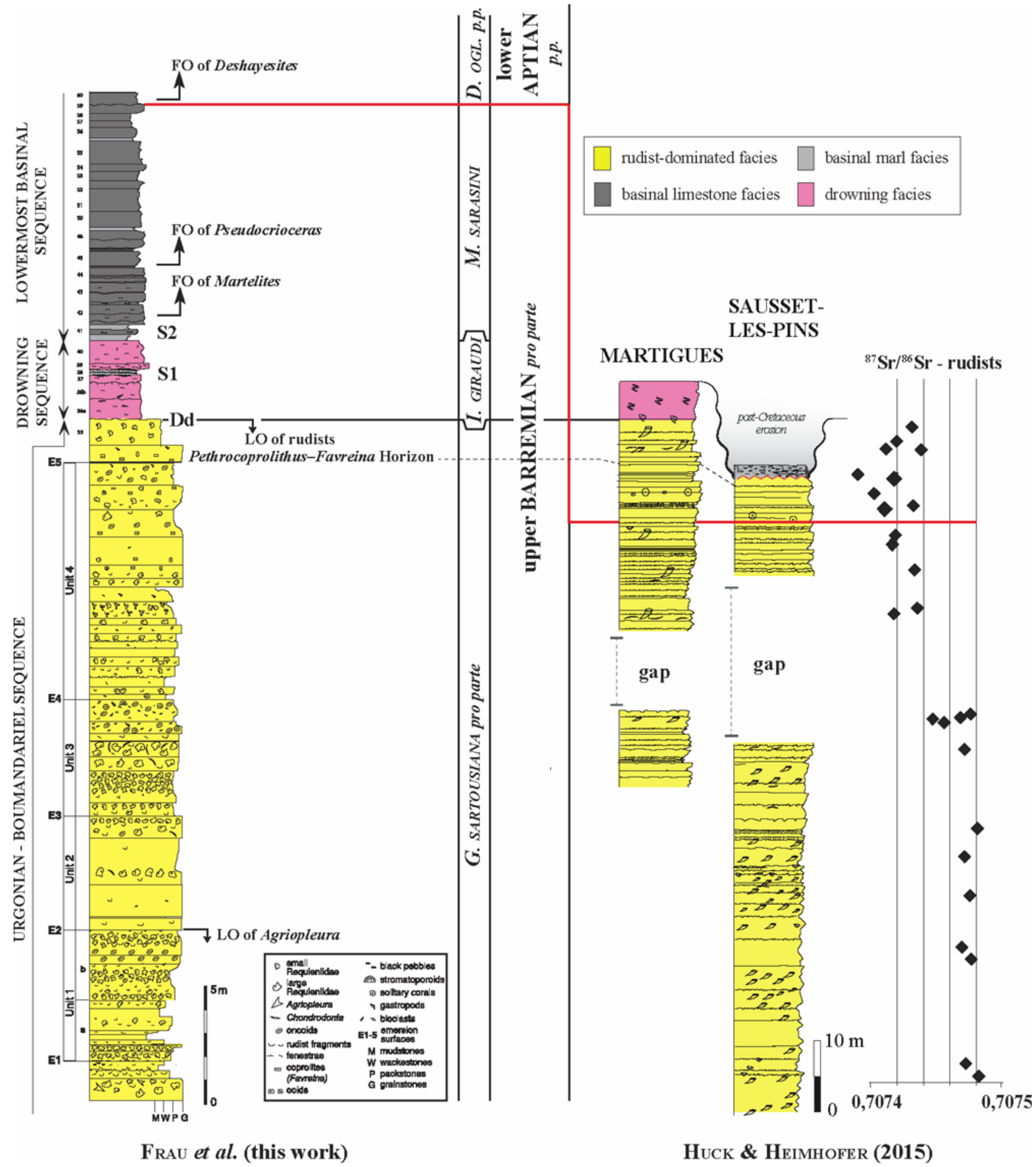

Figure 4: Synthetic stratigraphic section of the Barremian-Aptian transition in southern Provence with main litho- and bio-events of the Boumandariel (yellow), drowning (rose) and lowermost basinal (blue) sequences (modified from FENERCI-MASSE *et al.*, 2005; MASSE & FENERCI-MASSE, 2011), and comparison with the litho- and Sr-isotope stratigraphy published by HUCK & HEIMHOFER (2015). Note that major ammonite bio-events support the location of the Barremian/Aptian in the lowermost basinal sequence (red line *sensu* FRAU *et al.*, this work).

4.2. Southern Provence

High-resolution, Sr-isotope measurements were performed by HUCK & HEIMHOFER (2015) on the Boumandariel sequence *sensu* FENERCI-MASSE *et al.* (2005) from the western part of the Nerthe Massif (Martigues and Sausset-les-Pins sections) in southern Provence. This sequence, divided into five units (*i.e.*, E1 to E5), represents the topmost part of the rudist-rich Urgonian series of southern Provence (MASSE *et al.*, 2001; FENERCI-MASSE *et al.*, 2005; MASSE & FENERCI-MASSE, 2006) (Fig. 4). By contrast with North Provence, the caprinid-bearing beds are absent due to the progressive deepening of this region, which lead to the deposition of the thick, ammonite-bearing succession of the South Provence Basin (MASSE & FENERCI-MASSE, 2011; FRAU *et al.*, 2016).

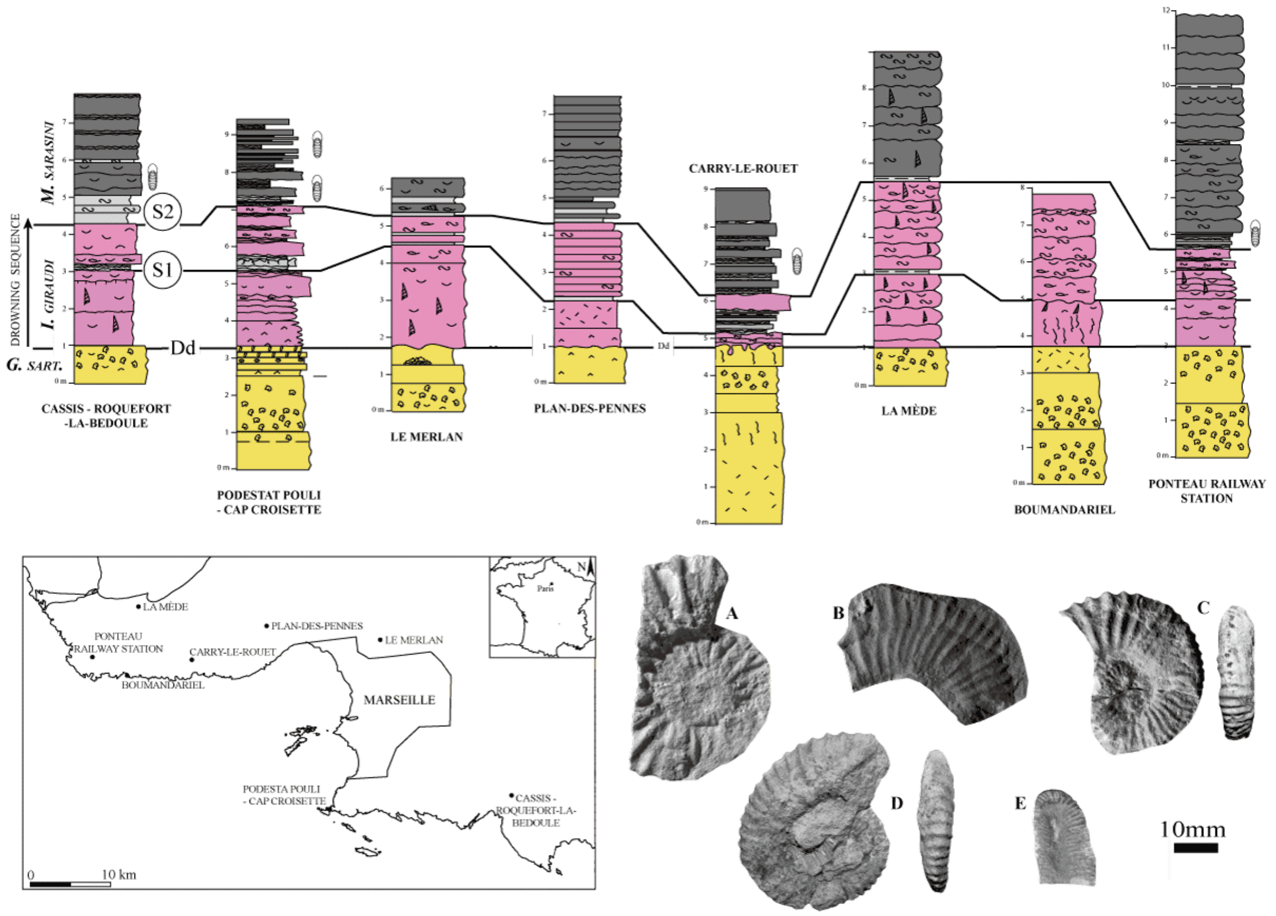

Figure 5: Stratigraphic correlations of the uppermost Barremian through the Martigues-Marseille region with the first illustration of age-diagnostic heteroceratid ammonites from the Nerthe Massif: (a) and (b) specimens MPP-CR.1 and MPP-CR.2 of *Calanquites* gr. *katsharavai* from Carry-le-Rouet; (c) and (d) specimens FSL.108074 and FSL.108083 of *Martelites* sp. from Gare de Ponteau; (e) specimen FSL.108077 *Heteroceras* sp. from Gare de Ponteau. All ammonites are deposited in the Musée de Paléontologie de Provence (MPP) and the Faculté des Sciences de Lyon (FSL).

Sr-isotope measurements given by Huck & Heimhofer (2015) were obtained from rudist shells from the E1 to E5 units, essentially requieniids and monopleurids (Fig. 4). At Sausset-les-Pins, the values fluctuate from 0.707324 to 0.707479, while at Martigues, the range narrows from 0.707418 to 0.707448. In both cases, Sr-isotope values encompass a spectrum which apparently covers the upper Barremian and lower Aptian if plotted onto our compiled curve. Values range from 0.707411 to 0.707324 at Sausset-les-Pins and were interpreted to be indicative of the *D. oglanlensis*-*D. forbesi* zones by Huck & Heimhofer (2015); assigning *de facto* a late early Aptian age to the post-Urgonian deposits. According to these authors, the putative Barremian/Aptian boundary falls in the upper part of the Boumandariel sequence but the existence of a major hiatus spanning the *G. sartousiana* and *I. giraudi* zones was presupposed by the authors.

It is noteworthy that Huck & Heimhofer (2015) overlooked the extensive litho- and biostratigraphic data collected from the Urgonian and post-Urgonian series of South Provence (Moullade *et al.*, 2000; Masse & Fenerci-Masse, 2011; Frau *et al.*, 2016). Indeed, this series is laterally continuous from the Nerthe Massif to the so-called Calanques Massif, which houses the Bedoulian-type series. Four guide levels, *i.e.*, the last occurrence of the rudist genus *Agriopleura* located at the E2 emersive surface (Fenerci-Masse *et al.*, 2005), the *Pethrocoprolithus*-*Favreina* calichified horizon located at the boundary between the E4 and E5 units (Masse, 1966, 1976), and the two black shale horizons S1 and S2 (= Niveau Taxy *sensu* Moullade *et al.*, 2000) of the drowning sequence (Masse & Fenerci-Masse, 2011), are essential for documenting this continuity and support the synchronous demise of the Urgonian regime through the study area (Fig. 5). Although no ammonites have yet been reported in the drowning sequence, three ammonite assemblages occur throughout the lowermost basinal sequence (Fig. 5). The lower assemblage is dominated by the Heteroceratidae (*Martelites*, *Heteroceras*, *Calanquites* and endemic genera), which progressively disappear and are replaced by the Ancyloceratidae *Pseudocrioceras*, and then by the Deshayesitidae *Deshayesites* (see Delanoy *et al.*, 1997; Ropolo *et al.*, 1999, 2000a, 2000b, 2006; Frau *et al.*, 2016). The first two assemblages characterize the lower and upper subzones of the *M. sarasini* Zone, while with unanimous agreement the third one begins the Aptian stage in the study area (Delanoy *et al.*, 1997; Cecca *et al.*, 1999b; Ropolo *et al.*, 1999, 2000a, 2000b; Reboulet *et al.*, 2011, 2014). These ammonite assemblages also occur in the Nerthe Massif. As an example, we report the occurrence of the Heteroceratidae *Calanquites* gr. *katsharavai* at Carry-le-Rouet, as well as the association of *Martelites* sp. and *Heteroceras* sp. at the Gare de Ponteau section (Fig. 5). These taxa date the lowermost *M. sarasini* Zone and confirm the age-assignment of the drowning sequence to the *I. giraudi* Zone through the study area (Frau *et al.*, 2016).

In summary, there is hardly any doubt that the Barremian/Aptian boundary falls in post-Urgonian basinal series and that the Boumandariel sequence is of early late Barremian age. It is currently assigned to the upper *G. sartousiana* Zone in agreement with the Last Appearance Datum (LAD) of the rudist genus *Agriopleura* (see Masse & Fenerci-Masse, 2015), although Clavel *et al.* (2014) argued an older Barremian age by orbitolinid occurrences. In any case, the foregoing highlights that the calibration of the Barremian/Aptian boundary proposed by Huck & Heimhofer (2015) using SIS is erroneous.

4.3. Northern Subalpine Chains

Sr-isotope measurements were performed by Huck *et al.* (2011, 2013) on rudist shells from the Urgonian series *pro parte* in the Cluses-Forclaz area (Bornes-Aravis massifs, Northern Subalpine Chains). The studied part of the section overlies outer platform deposits dominated by bioclastic-peloidal facies. This succession includes two rudist accumulation units (*i.e.*, Lower and Upper Urgonian limestone members *sensu* Wermeille, 1996, Unit 15 and 18 *sensu* Trabold, 1996; segments CL7-CL9 and CL11 *pro parte*-CL13 *sensu* Huck *et al.*, 2013), that are separated by an orbitolinid-rich episode, which is considered by the authors to be the local equivalent of the so-called Lower *Orbitolina* Beds cropping out in the Southern Subalpine Chains (Trabold, 1996) (Fig. 6). Note that the upper rudist accumulation unit likely consists of foraminifer-peloidal facies grading upward into quartz-rich sands topped by a prominent discontinuity. Post-Urgonian deposits correspond to the Helvetic Greensandstones Fm., the basal part of which corresponds to the upper Aptian Aujon Beds as defined by Pictet (2016 and references therein).

According to Huck *et al.* (2011), Sr-isotope values of the lower rudist accumulation unit encompass the maximum early-late Barremian change-over from increasing to decreasing values, while values of the upper unit falls in the "grey band" as herein defined. Accordingly, Huck *et al.* (2011) assigned a late Barremian-early Aptian age to the rudistid units and the orbitolinid episode of the Cluses-Forclaz area. The age would conform to the Urgonian Limestone Fm. *sensu* Arnaud *et al.* (1998) cropping out in the Southern Subalpine Chains.

In our opinion, the high Sr-isotope values of the lower rudist accumulation (ranging between 0.707476 and 0.707495), followed by a decreasing trend through the CL8-9 segments (average values of 0.707461) can be interpreted in two different ways with respect to the Sr-isotope reference composite curve (i) early-late Barremian change-over from increasing to decreasing values as Huck *et al.* (2011) did or (ii) internal variability within the early Barremian Sr-isotope plateau values. Unfortunately, biological markers documented in the lower rudist accumulation by Trabold (1996) do not provide independent evidence for interpreting the Sr-isotope curve. Indeed, the orbitolinid taxa documented by Trabold (1996, Fig. 4.2) in the lower rudist accumulation unit fall in a time interval that spans the lower to lower upper Barremian without specifying the ammonite zone. Going further, the Highest Occurrence Datum of *Agriopleura* observed at the CL9-CL10 segment transition by one of us (J.-P.M.) cannot be confidently considered to be the LAD of this rudist genus although it similarly falls at a short-term emersive surface indicated by both litho- and geochemical markers (Huck *et al.*, 2011), as in the southern France record (see § 4.2).

Above, a set of six Sr-isotope values was documented in the upper rudist accumulation unit while no confident measurement has been possible for the orbitolinid-rich episode due to values below the standard threshold (Huck *et al.*, 2011, Fig. 5). However, the authors pointed out that "the uppermost Cluses section, beneath the drowning unconformity, is characterized by altered $\delta^{13}C$ values and $^{87}Sr/^{86}Sr$ ratios due to pervasive recent weathering". SIS cannot be, therefore, specifically used to prove or disprove the late Barremian-early Aptian age assigned to the two rudist accumulation units. Only the potential occurrence of caprinid rudists in the upper rudist accumulation unit could bring biostratigraphic refinement with respect to the Southern Subalpine Chains. However, to date no age-diagnostic rudists have been reported in this area. As a result, the equivalence between the orbitolinid-rich beds of the Cluses-Forclaz area with the Southern Subalpine Lower *Orbitolina* Beds is still a moot point which needs further investigation.

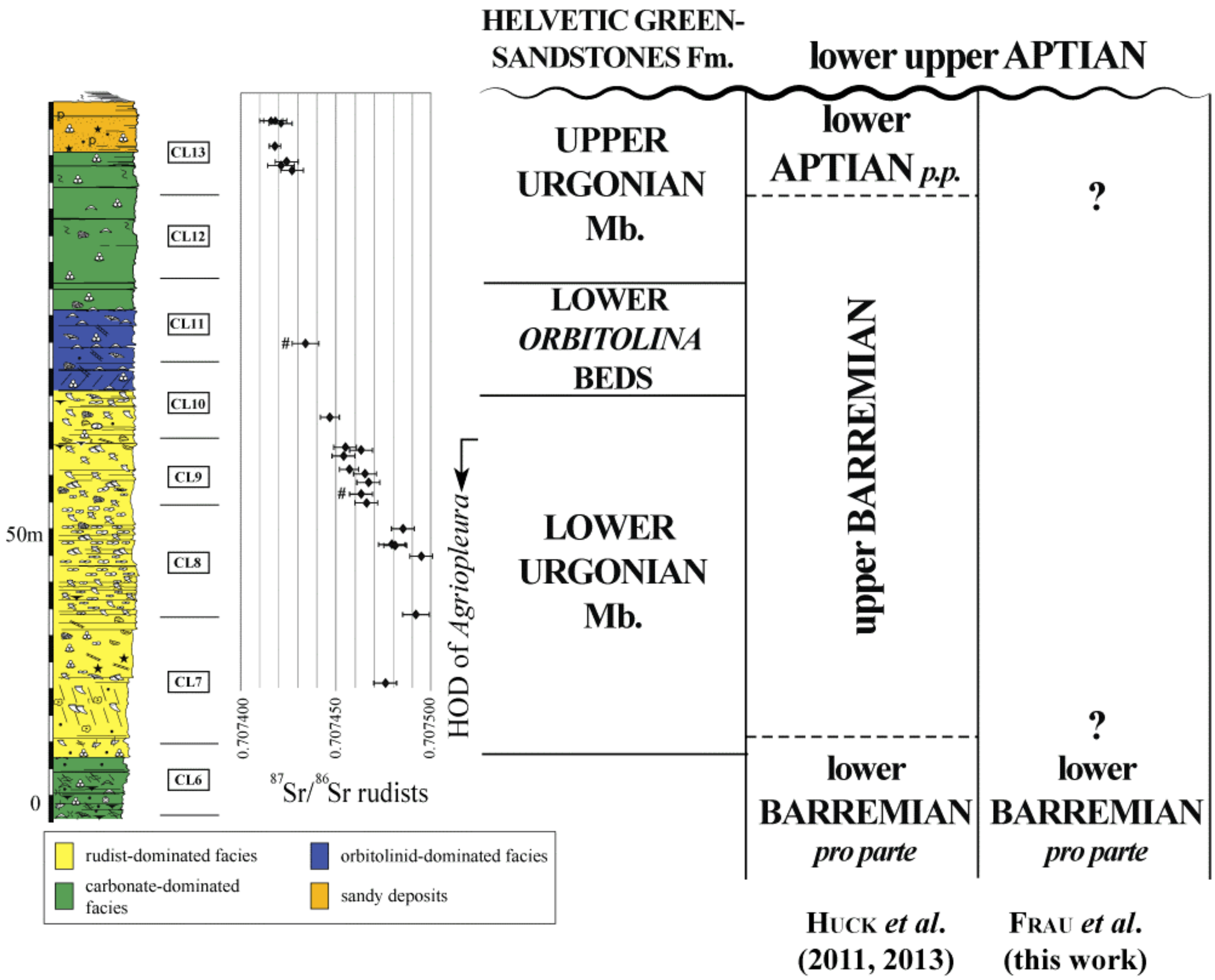

Figure 6: Lithostratigraphy and Sr-isotope stratigraphy of the Cluses section (Northern Subalpine Chains, SE France) with stratigraphic interpretations proposed by HUCK *et al.* (2011, 2013) and FRAU *et al.* (this work). Sinuous line marks the final emersive discontinuity at the top of the Urgonian series. Symbol (#) indicate the rudist samples with strontium concentrations below the threshold of 1000 ppm (see HUCK *et al.*, 2011).

4.4. Istria

SIS based on requieniid rudist shells from the *Lithocodium-Bacinella* episode of the Kanfanar section (Istria, Croatia) was documented by HUCK *et al.* (2010) (Fig. 7). Values are in the range of 0.707395 to 0.707421, and fall in the "grey band", thus preventing a precise calibration. HUCK *et al.* (2010) converted the Sr-isotope values into numerical ages (*i.e.*, 124.5 ± 0.4 Ma and 124.0 ± 0.4 Ma) using the LOWESS method of HOWARTH & MCARTHUR (1997). Referring to GRADSTEIN *et al.* (2004), these numerical ages were associated with a late Barremian-early Aptian age, including the spreading and culmination (*pro parte*) of OAE 1a. Unfortunately, HUCK *et al.* (2010) overlooked the occurrence of the age-diagnostic orbitolinid, *P. cormyi*, in the lower part of the *Bacinella-Lithocodium* episode (MASSE *et al.*, 2004), which ranges in the *D. deshayesi-D. furcata* zones (*op. cit.*), and postdates the OAE 1a-related time interval. Consequently, Sr-isotope values cannot be used to pinpoint the position of the *Bacinella-Lithocodium* episode of Kanfanar with respect to the lower/upper Aptian boundary. Nevertheless, one may notice that the Croatian *Bacinella-Lithocodium* episode is younger than the Oman ones which predates the FO of *P. cormyi* and are apparently associated with the OAE 1a time interval (see MASSE *et al.*, 1998a, 1998b; IMMENHAUSER *et al.*, 2005; SCHROEDER *et al.*, 2010).

5. Concluding remarks

Based on the Sr-isotope compiled curve, the group of values comprised between 0.707435 and 0.707410 represents a "grey band" in which SIS is unreliable for separating the uppermost Barremian (*M. sarasini* Zone) from the lower Aptian *pro parte* (*D. oglanlensis-D. forbesi* zones). Potentially, values ranging from 0.707485 to 0.707440 tend to indicate an early late Barremian age, while values lower than 0.707410 are strictly younger than the upper lower Aptian (upper *D. forbesi-D. furcata* zones time interval). Following our foregoing review, little doubt exists

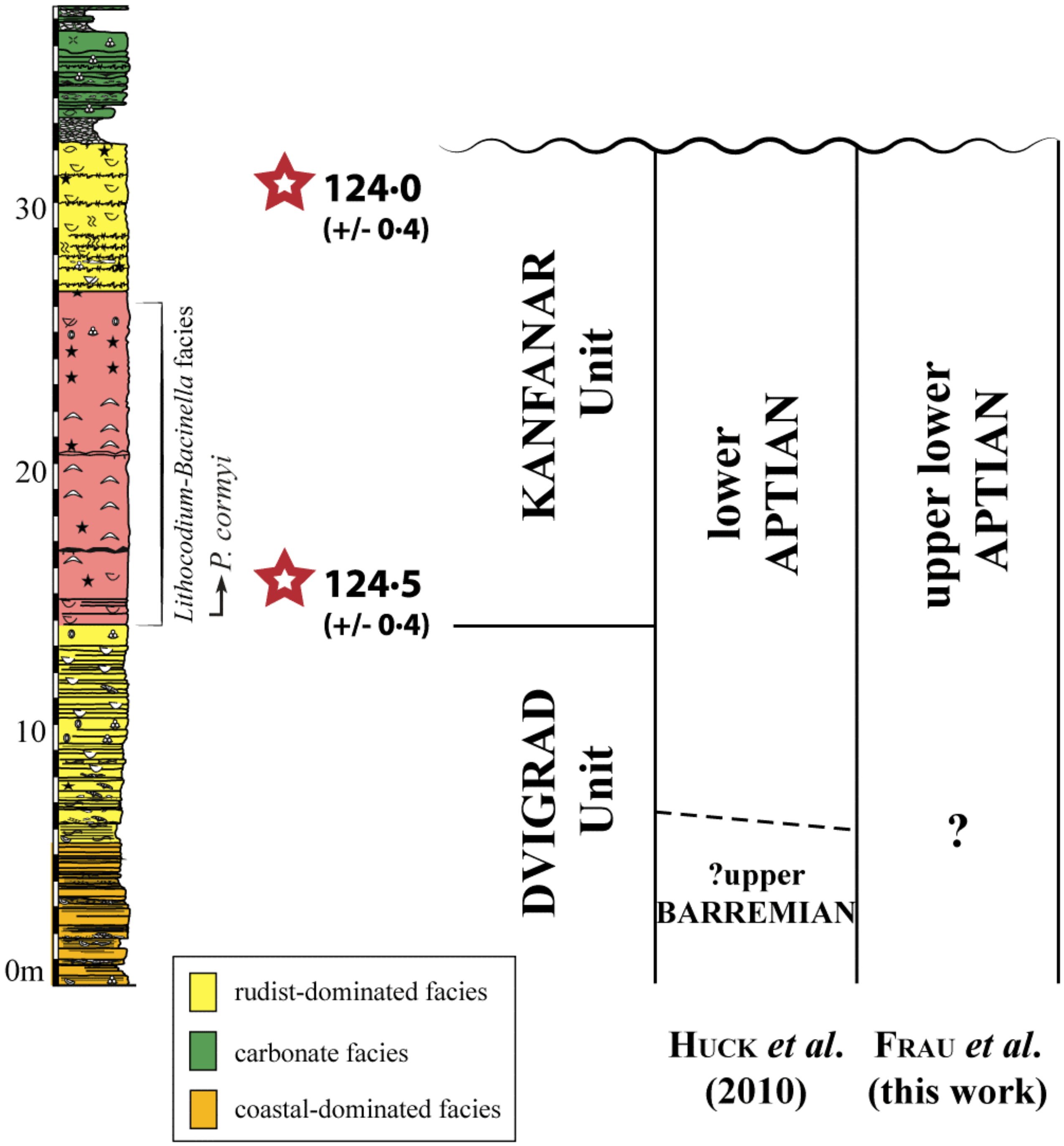

Figure 7: Lithostratigraphy and Sr-derived numerical ages of the Kanfanar section (Istria, Croatia) with stratigraphic interpretations proposed by HUCK *et al.* (2010) and FRAU *et al.* (this work). Sinuous line marks a major discontinuity.

that Sr-isotope values cannot be specifically used to prove or disprove results based on shallow-marine biological markers applied to the Urgonian sequences at the Barremian-Aptian transition. The value of 0.707427 assumed to be indicative of the Barremian/Aptian boundary by HOWARTH & MACARTHUR (1997) and HOSSEINI *et al.* (2016) therefore cannot be used with confidence.

The conversion of Sr-isotope values to numerical ages subsequently assigned to a stratigraphic interval is also a source of great imprecision, but this is mostly due to the imperfect calibration of numerical ages with stage boundaries. Although SIS-derived ages commonly tend to match biostratigraphic data at the stage level in the lower Cretaceous (see exception in GODET *et al.*, 2011, versus CHAROLLAIS *et al.*, 2013), their resolution is, notwithstanding exceptions, generally too low for defining ammonite zones and subzones in shallow-marine carbonates. In some cases, biological markers should be more precisely integrated with specific parts of the indeterminate SIS segments, and thus to improve correlations.

For the study interval, the compiled Sr-isotope curve is supported by a limited number of measurements, especially for the OAE 1a time interval. The scarcity of control points precludes the recognition of possible temporal variations within a given ammonite zone and/or the existence of some internal variability. For instance, the apparent similarity of Sr-isotope values between the *M. sarasini* and *D. forbesi* zones is possibly a sampling artefact, but until more data become available, the Sr-isotope curve should continue to be used with extreme caution for dating ammonite-free stratigraphic intervals.

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