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4 **Geochemical study of the luting and coating of medieval watercraft from**
5 **the ship timbers discovered in the citadel of al-Balīd, Oman: composition**
6 **and origin**

7

8

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36

37 **Abstract**

38 Ship timbers were discovered during the excavation of the Islamic site of al-
39 Balīd recycled in the buildings of its citadel and the Great Mosque. These
40 fragments stripped from the hulls of Indian Ocean medieval sewn vessels form
41 the largest collection of archaeological evidence for the ships sailing during the
42 Middle Islamic Period (10th–15th century CE). More than one third of the
43 timbers bears traces of a bitumen mixture used as luting and coating to seal the

44 ships planking to waterproof and protect their hulls, a practice generally
45 associated with sewn-plank construction.

46 This paper presents the result of the GC-MS, carbon and hydrogen isotope on
47 chromatographic fractions, and X-Ray diffraction analyses on twenty-two
48 samples of the luting extracted from fifteen timbers from al-Balīd. The nature of
49 the constituents of the amalgam is determined showing that the luting consists of
50 bitumen mixed with mineral matter, fat or oil and two triterpenic resins, dammar
51 (Dipterocarpacea) and frankincense.

52 The chemical signature of the bitumen indicates that it was likely sourced from
53 two seeps in southwest Iranian: Mamatain and Ain Gir-Chersch Mehrgir-
54 Dehluran. The origin of the bitumen might suggest that the luting was applied
55 during the construction or repairing of Indian Ocean vessels in boatyards along
56 the coasts of the Arabian/Persian Gulf or Southern and Eastern Arabia.

57

58

59 **Keywords:** al-Balīd, Oman, medieval sewn plank boats, timbers, luting
60 amalgam, waterproofing agents, bitumen, resin, fat, oil, steranes, terpanes, GC-
61 MS analyses.

62

63 1. Introduction

64

65 Bitumen was an essential material in the history of the Arabian/Persian Gulf
66 ¹since early times. People have collected this substance from natural seeps in
67 Mesopotamia and Iran and used it for multiple purposes such as mortar, sealant,
68 glue and waterproof agent (Connan 1999, 2012; Connan and Deschesne 1995).
69 Because of its versatility, this material was widely traded in the Gulf since the
70 Neolithic, as far as Oman in the Arabian Sea (Connan and Carter 2007; Connan
71 and Van de Velde 2010).

72

73 Bitumen also played a crucial role in the development of boat building and
74 seafaring in Mesopotamia and the Gulf. Coastal communities used this
75 substance to waterproof the hulls of their reed and wooden vessels, prompting
76 both regional and international maritime trade, and enabling the flow of goods
77 between Mesopotamia, the Gulf and Indus Valley (Cleuziou and Tosi 1994,
78 2000; Carter 2002). The practice of using natural bitumen to coat vessels
79 persisted in southern Iraq (Ochsenschlager 2004; Thesiger 1954, 1964) and Iran
80 until recently (Cooper et al. 2020).

81

82 Yet, our knowledge of the use of this material in boatbuilding and seafaring
83 context during the medieval period is minimal. Due to the scarcity of medieval
84 shipwrecks in the Indian Ocean, most of the early and middle Islamic
85 archaeological bitumen is associated with the production of ceramic vessels,

86 ¹-Hereafter called the Gulf

87 such as torpedo jars, the inside of which is sealed with a thin layer of this
88 substance (Connan et al. 2020, 2021; Stern et al. 2008, Durand 2021, Lischi et
89 al. 2020, Tomber et al. 2020).

90

91 Our knowledge instead relies primarily on vague historical sources
92 mentioning a wide range of materials used to seal the hulls of watercraft in the
93 middle Islamic period. Moreover, terms such as bitumen, tar, pitch and resin are
94 often used indiscriminately for substances that look very similar in terms of
95 colours and physical properties but are produced from different materials
96 (Forbes 1955). Since it is virtually impossible to distinguish all these substances
97 by the naked eye, references to pitch and tar might actually indicate bitumen and
98 vice versa.

99

100 Recent excavation at the Islamic site of al-Balīd (2010–2018) (Fig.1) brought
101 to light several ship timbers of medieval vessels involved in the Indian Ocean
102 maritime trade (Belfioretti and Vosmer 2010; Ghidoni 2021; Pavan et al. 2018;
103 Vosmer 2017, 2019). These pieces of maritime material culture display the
104 distinctive traits of the Indian Ocean sewn-plank technique, providing invaluable
105 information about boatbuilding techniques and materials in the medieval period.
106 A considerable number of the al-Balīd timbers retained preserved “bitumen”¹ on
107 their edges and surfaces used to lute the planking seams and make the hull
108 watertight.

109

110

111

112 **2. The site of al-Balīd**

113

114 During the medieval period, the southern coast of Oman – corresponding today
115 to the Governorate of Dhofar - was characterized by the flourishing of several
116 settlements, involved in the long-distance trade across the Indian Ocean. Al-
117 Balīd, known at that time by the name *Zafār*, gained a role of leadership among
118 all these centres, becoming the most important urban settlements along the
119 southern coast of Oman (Newton and Zarins 2017). Having its flourishing
120 period between the 13th and the 15th century, when it is mentioned in various
121 sources (Ibn Baṭṭūṭah 1962; Smith 1985; Yule 1871) al-Balīd was a pivotal
122 centre for the international network developed by the Rasūlids which involved
123 Arabia, Africa, India and, furthermore, China and East Asia (Pavan and Visconti
124 2020).

125 *Zafār* and the whole coast of Dhofar are mentioned as centres of export
126 for local products, such as frankincense, ambergris, myrrh, dragon’s blood,
127 aloes, benzoin, liquid storax, camels and ostriches, but also as a place of

¹ The term “bitumen” is used here without presuming its chemical composition which could be either pure bitumen or a bituminous mixture of oil, resin and pure bitumen as illustrated in the chemical analyses below.

128 distribution for goods arriving from the interior of the country or other regions
129 of the Arabian Peninsula such as horses, or from Africa, such as rhino horns and
130 ivory (Pavan forthcoming).

131 The wealth of the ancient town finds is evinced by the numerous high-
132 quality and expensive pottery vessels manufactured in Egypt, Iran, and China,
133 but also in the medium-quality products, such as Yemeni, Indian, Chinese, and
134 Far Eastern ceramics which were found in the city.

135 Evidence of contacts with East Africa are barely represented by pottery
136 items, as most of the goods were perishable, but they include some samples of
137 copal and quite a good number of coins issued by the Sultans of Kilwa, who
138 ruled on the eastern coast of Tanzania, between the 10th century and the first half
139 of the 16th century (see Annucci in Pavan et al. 2020). The city of al-Balīd
140 covers a surface of 64 ha and it has roughly the shape of an irregular rectangle
141 (Fig. 2A). The site is apparently crossed by two major roads with smaller streets
142 encircling buildings which presumably stood rather isolated. The defensive
143 system was integrated by four towers towards the ocean, two others along the
144 western and eastern sides of the site. The side facing north apparently lacks
145 gates and only a couple of semi-circular towers have been identified at the edges
146 of the northern fortification system. Considering also the presence of a ditch, it
147 is possible that the northern side has more of a divisionary purpose (separating
148 the interior from the exterior) than a functional one. An internal gate, located in
149 the north-central part of the site (Area B) was excavated by P. Costa (1979) and
150 it was related to a possible inner wall delimiting an earlier smaller city.

151 The city itself could be divided into three main areas: a western one, with the
152 most important buildings such as the citadel (*Husn*) (Fig. 2B) and the Grand
153 Mosque located around the largest public square of the city; a central part, with
154 private large houses and mosques, including a funerary one; and a wide,
155 easternmost, area which was possibly used to stock, load and unload goods and
156 animals (Fig. 2A). This sector, which is now completely flat, except for a few
157 mounds surrounded by columns, reveals the presence of small mosques and
158 could have hosted temporary shelters and structures built of perishable
159 materials.

160 During the last four years (2016-2020) excavations have been mainly focused
161 on the citadel (*Husn* al-Balīd). Six constructional phases have been identified so
162 far on the basis of the data emerged from the archaeological investigations,
163 recent studies on the materials (pottery, small finds, coins) and the results
164 achieved during the activities carried out by the previous archaeological
165 missions working at the site. To date only the last three phases (VI, V, and IV)
166 have been stratigraphically investigated because the works still did not reach the
167 earliest occupational levels. The investigated phases cover a time ranging from
168 the end of the 13th to 18th century (Pavan et al. 2018, 2020).

169

170

171 3. The timbers

172

173 During several excavations at al-Balīd's citadel and Grand Mosque,
 174 archaeologists have discovered more than fifty timbers, identified as parts of
 175 hulls of sewn boats (Belfioretti and Vosmer 2010; Ghidoni 2021; Pavan et al.
 176 2018; Pavan et al. 2020; Vosmer 2017, 2019). These ship remains form the
 177 largest collection of archaeological evidence for ships sailing in the Indian
 178 Ocean during the Middle Islamic Period. Having fulfilled their maritime
 179 purpose, the timbers were recycled in a terrestrial context, reused as structural
 180 elements within the masonry of the site's buildings. The builders of al-Balīd
 181 used the straight timbers as levelling within the walls of the citadel to help
 182 distribute the weight of the masonry (Borgese et al. 2019), and as ceiling planks,
 183 beams, shelves and lintels (Fig. 3) (Ghidoni 2022). Evidence of boat part
 184 recycling has been found on the Red Sea coast of Egypt at Berenike
 185 (Sidebotham 2008), at Quseir al-Qadim (Blue 2006; Blue et al. 2011) and the
 186 guardroom ceiling of Fort Jesus in Mombasa (Prins 1982). The vast majority of
 187 the al-Balīd timbers consist of hull planks bearing similarities with recent sewn
 188 boats, and displaying the traits of the Indian Ocean sewn-plank construction
 189 Fig.4), such as:

190

- 191 - continuous sewing through holes regularly spaced along their edges;
- 192 - vegetal fibre sewing cordage recessed in rebates on the outside of the hull;
- 193 - a caulking roll (wadding) between the planking seams and the sewing
- 194 inside the hull;
- 195 - frame lashings;
- 196 - oblique dowels connecting adjacent planks;
- 197 - and luting and coating material.

198

199 The timbers are different sizes and display various fastening techniques,
 200 indicating that they once belonged to different vessels. Most of the planks (86
 201 %) have rebates on one side: small grooves or channels between the sewing
 202 holes and the edge of the plank. This evidence indicates a single-wadding
 203 technique typical of the western Indian Ocean (Belfioretti and Vosmer 2010;
 204 Pavan et al. 2018, 2020), where these rebates connect matching holes between
 205 two planks to accommodate and protect the stitches on the outside of the hull.
 206 Inboard, the sewing is over a cushion of vegetal material (wadding) and firmly
 207 compresses it on the plank's seam. A few planks had no rebates, suggesting
 208 instead the presence of wadding under the stitching on both sides of the hull in a
 209 pattern called double-wadding (Pavan et al. 2018, 2020; Vosmer 2017),
 210 observed in the 8th-9th centuries Phanom-Surin and Belitung shipwrecks (First
 211 Regional Office of Fine Arts 2016; Flecker 2000, 2010). An illustration by
 212 Johannes Baptista van Doetechum (c.1554–1606) shows vessels sailing off Goa
 213 with a caulking roll on each side of the hull (Van Linschoten 1605) pointing to

214 the use of this fastening system in the western Indian Ocean at the end of the
215 16th century. This technique was also documented in recent sewn vessels used
216 along the east coast of India (Kentley 1985, 2003).

217

218 Botanical analyses carried out on the timbers indicate that Indian Ocean
219 boatbuilders used a wide range of wood species to build medieval ships. The
220 species identified include teak (*Tectona grandis*), jujube (*Ziziphus mauritiana*),
221 *Terminalia* spp., *Anogeissus* spp., *Caesalpinia* spp., and palm (*Palmae*: species
222 unspecified) (Belfioretti and Vosmer, 2010; Capretti et al. 2010; Ghidoni 2021;
223 Vosmer 2019). The sewing cordage was visually identified as coir (coconut
224 husk), a fibre that was widely employed in western Indian Ocean sewn
225 watercraft throughout history (Chittick 1980; Edye 1834; Hornell 1942; Hourani
226 1963; Stanley 1869; Vosmer 1997).

227 The species diversity indicated by the botanical analyses also reflects a
228 variety of characteristics, ranging from high-quality boatbuilding wood for hull
229 planking, such as teak and some species of *Anogeissus*, to relatively poor
230 choices, such as palm and jujube, the latter better suited for frames than planks.
231 Moreover, the timbers distribution is wide, being native to various areas,
232 including East Africa, India and Southern Arabia.

233

234 Radiocarbon dating analysis on sixteen samples indicates that the felling of
235 the trees from which these timbers were fashioned occurred between the late 10th
236 and 15th centuries CE, suggesting a similar period for the construction of the
237 boats (Belfioretti and Vosmer 2010; Pavan et al. 2018; Vosmer 2017, 2019;
238 Ghidoni 2021).

239

240

241 **4. Bitumen evidence from al-Balīd**

242

243 One interesting aspect of the al-Balīd timbers is the presence, in more than a
244 third of the collection, of traces of a black substance resembling bitumen
245 (Belfioretti and Vosmer 2010; Pavan et al. 2018; Ghidoni 2021). This substance
246 occurs on planks either with or without rebates.

247

248 A thin layer of “bitumen” is preserved on the faying edge- the surface where two
249 planks are joined together-of seven timbers, pointing to the practice of luting,
250 the use of a malleable material to seal the seam between two adjoining planks
251 before their final fitting during the assemblage of the hull (Fig. 5A-B). The
252 construction method of sewn hulls makes it impossible to make them watertight
253 by driving caulking into their seams, in the way used in nailed ships, because the
254 sewing cordage would be in the way. This luting activity is suggested by various
255 ethnographic records from the western Indian Ocean (Severin 1985; Shaikh et
256 al. 2012; Vosmer et al. 2011; Weismann et al. 2019).

257

258 In eight timbers the “bitumen” is located on the former inner surface of
259 the plank, between the sewing holes and the edge. The best examples are
260 BA1104065.454 and BA0604145.175, which preserved a great deal of the
261 substance ranging in thickness between 2 and 5 mm (Fig. 5C-E). A thin dark
262 coat of the material is visible under the wadding in BA0604128.73 and
263 BA0604128.74, indicating that medieval boatbuilders smeared bitumen on the
264 hull seams prior to the sewing of the planks (Fig. 5D-F).

265

266 Four planks display traces of bitumen on the outer surface. All but one, Wo37
267 (Fig. 6A) have a flat surface with no rebates, suggesting the presence of the
268 wadding outboard. Traces of bitumen are located around the stitching holes of
269 timber Wo37, and extend towards the centre of the plank, suggesting that
270 bitumen covered the hull outboard, most likely below the waterline to increase
271 its water tightness and address construction issues, and protect it against marine
272 crustacea and molluscs such as barnacles and shipworms (*Teredo navalis*)
273 (Woods Hole Oceanographic Institute 1952).

274

275 The practice of using bitumen, tar or pitch, in Indian Ocean medieval
276 watercraft, is suggested by a number of illustrations in the *Maqāmāt* of al-Ḥarīrī
277 (Nicolle 1989, fig. 22; Bibliothèque Nationale, Ms Arabe 6094, fol. 68) and
278 Persian miniatures (Eastman 1950, pl. XII; Weismann 2002, fig. 21;
279 Bibliothèque Nationale de France, Suppl. Persan 641 f. 59) depicting black
280 hulls. Bitumen-coated vessels have persisted in Iraq (Thesiger 1954;
281 Ochsenschalger 2004), where they are still in use in the marshlands in the
282 southern region of the country (Jeffrey Rose, personal communication, February
283 2017). Five sewn *baggāras/lāmeles* from Iran, which were recently acquired by
284 Qatar Museums, were coated outboard with a thick layer of “bitumen” (Fig. 6B),
285 proving that the practice of using this material to waterproof watercraft persisted
286 in a maritime context until at least the second half of the 20th century (Cooper et
287 al. 2020).

288

289 In the case of timbers BA0604159.263 and BA0604172.69, fragments of
290 the same plank, a thick layer of this substance covers most of the former inner
291 surface (Fig. 6C), showing “bitumen” was also sometimes used inboard (Cooper
292 et al. 2020) (Fig. 6D). This would have protected the stitching against abrasion
293 and sun exposure, preventing the breakage of the cordage, thus extending the
294 life of the sewing.

295

296 In three timbers, “bitumen” appears to have been used to seal and smooth
297 irregular surfaces. In the case of timber BA0301-106, it covers an uneven
298 section near the sewing holes, to make it flush with the rest of the plank.
299 Similarly, medieval boatbuilders smeared “bitumen” near the sewing holes of

300 timber Wo73, to seal a crack, likely caused by the drilling of an additional hole
301 to either repair or reinforce the sewing. On plank Wo52, “bitumen” luted a
302 broken section of a dowel.

303

304 The site of al-Balīd also provides evidence of “bitumen” not associated
305 with the ship timbers. Recent excavations of the citadel have discovered a lump
306 of this substance (X35). The piece is small, measuring 60 x 100mm, and
307 provides no hints such as impressions of wood or cordage that could help to
308 reveal its purpose (Fig. 7). However, it is possible that the lump was imported
309 from the bitumen sources in Persia and Mesopotamia and stored at the site to be
310 used in boat construction or repair. Alternatively, shipwrights at al-Balīd could
311 have even stripped it from other vessels and stored it at the site to be reused.

312

313 Lastly, a layer of “bitumen” of irregular thickness was found lining the inside
314 of a cylindrical metal vessel (X43) (Fig. 8). The “bitumen” is likely to be pure
315 because it appears different from that seen on the ship planks and the lump X35,
316 having a very smooth and almost glassy texture with no inclusions. The fact that
317 the bitumen occurs primarily on the sides of the metal container may point to the
318 use of the substance to seal the inside of the container, in a practice previously
319 observed in ceramic vessels (Connan et al. 2020). However, it is very likely that
320 this evidence might indicate pure bitumen stored in the vessel for subsequent
321 use. Firstly, its thickness, exceeding 5mm, appears rather excessive if compared
322 with that generally used to seal ceramic vessels in the region (Connan et al.
323 2020; Lischi et al. 2020). The material of the container also appears to
324 strengthen this interpretation. It is indeed unlikely that a metal container would
325 require an internal bitumen lining since, differently from unglazed ceramics, it
326 would not have needed to be sealed. Moreover, a metal container could have
327 been used to prepare the bituminous mixture used in the luting and coating of
328 the vessels.

329

330

331 **5. Experimental**

332

333 *5.1 Archaeological samples*

334

335 We extracted 25 “bitumen” samples from 15 timbers, the lump (X35) and the
336 seemingly pure bitumen inside the metal container (X43) (Table 1 and Fig. 7), to
337 determine their chemical and mineralogical composition. The former helps to
338 trace the origin of the bitumen, while the latter provides insights into the
339 inclusions added to the bitumen to create the amalgam for maritime use.
340 Moreover, the samples were extracted to determine whether there are
341 differences in the “bitumen” origin and mineralogical composition which might
342 be related to chronology or location and function of the “bitumen” on the

343 timbers. Lastly, we intended to investigate possible similarities between the
 344 bitumen from the timbers and those from the lump and metal container, which
 345 might indicate different stages of the bitumen amalgam preparation. Overall, the
 346 comparison between the samples provides an opportunity for insights into the
 347 use and trade of bitumen during the medieval period, as well as about maritime
 348 activities, such as boat building, repair and maintenance in the western Indian
 349 Ocean and, perhaps, at the port of al-Balīd.

350

351 The sampling process was carried out with a scalpel, using gloves to avoid any
 352 contamination of the specimens. The samples, ranging between 2-10mm thick,
 353 were taken from most of the timbers, particularly those with a thick layer of
 354 “bitumen”:

355 - Eleven samples from nine planks where “bitumen” was located inboard

356 between the sewing holes and faying edge of the plank, under the wadding;

357 - Three samples from three planks where the substance was on the faying
 358 edge of three planks;

359 - Four samples from the outboard coating of four planks;

360 - Three samples from the inboard coating of two timbers (BA0604172.69 and
 361 BA0604159.263), which are, in fact, fragments of the same plank;

362 -Two samples from two timbers, where the substance was used to fill a
 363 broken dowel hole (Wo52) and to smoothen the damaged surface of the
 364 timber, around the sewing holes outboard (BA03031-106);

365 - One sample from the bitumen lump X35;

366 - One sample from the metal container X43.

367

368 Because some of the timbers had (bore) traces of “bitumen” on either their
 369 edges and between the sewing holes and the edge (under the wadding and
 370 sewing), more than one sample was extracted from those timbers.

371

372 ***5.2 Oil seeps***

373

374 In order to trace the origin of the al-Balīd bitumen from the ship timbers, metal
 375 container and lump, the characteristic biomarkers and carbon and deuterium
 376 isotopes present in the ancient samples were compared against modern
 377 compositional datasets obtained from oil seeps analysed in southern Iran and
 378 Iraq (Hit, Mosul area and Kurdistan) (Fig. 9). This reference database has been
 379 consulted in previous studies (Stern et al. 2008; Connan 2011; Connan et al.
 380 2014, Connan et al. 2020). Some complementary data were recently acquired on
 381 new oil seeps in southwest Iran given by Buyuk Ghorbani and on oils seeps
 382 from Kurdistan provided by Rzger Abdulkarim Abdula and Mohammed W.
 383 Alkhafaji which significantly increased the geographical coverage.

384

385 ***5.3 Analytical procedures***

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5.3.1 Bitumen identification

All archaeological and geological samples were subjected to the same analytical procedure at GeoMark Research Ltd. The dichloromethane extract was deasphalted using n-hexane. The deasphalted fraction was separated into saturated hydrocarbons, aromatic hydrocarbons, and resins using gravity flow column chromatography employing a 100-200 mesh silica gel support activated at 400°C prior to use. Hexane was used to elute saturates, dichloromethane to elute the aromatic hydrocarbons and dichloromethane/methanol (50:50) to elute the resin (NSO) fraction. Following solvent evaporation, the recovered fractions were quantified gravimetrically. The C₁₅₊saturated hydrocarbon fraction was subjected to molecular sieve filtration (Union Carbide S-115 powder) after the technique described by West et al. (1990). An aliquot of the total alkane fraction was not fractionated by silicalite in order to preserve access to the n-alkanes.

GC-MS of the C₁₅₊branched and cyclic hydrocarbon fractions was performed using an Agilent 7890A (split injection) interfaced to an Agilent 5975C mass spectrometer. The HP-2 column (50m x 0.2mm, 0.11µm film thickness) was temperature programmed from 150 °C to 325 °C at 2 °C/minute and held for 10 minutes. The mass spectrometer was run in the selected ion mode (SIM), monitoring ions *m/z* 177, 191, 205, 217, 218, 231 and 253 amu for branched and cyclic alkanes. The quantitative data were produced using peak surface.

To determine the absolute concentration of individual biomarkers, a deuterated internal standard (d₄-C₂₉sterane, Chiron lab, Norway) was added to the C₁₅₊branched/cyclic hydrocarbon fraction. Response factors (RF) at 221 for the deuterated standard to hopane (*m/z* 191) and sterane (*m/z* 217) authentic standards were found to be 1.4 for terpanes and 1.0 for steranes. Concentration of individual biomarkers were determined using the following equation: Conc. (in ppm) = (Ht biomarker) (ng standard)/ (Ht standard) (RF) (mg B/C fraction).

The C₁₅₊saturates, C₁₅₊aromatics, asphaltenes and resins were analysed for their respective carbon isotope (δ¹³C, VPDB) compositions. The asphaltenes and resins were also analyzed for their stable hydrogen isotope compositions. Details for all of the methods for stable isotope analyses are described in Connan et al. (2021).

5.3.2 Identification of fats, oils and resins

428 Analyses were carried out at the Avignon University IMBE-IRPNC laboratory.
429 Ten mg of samples were extracted three times with 1 mL of dichloromethane,
430 then centrifuged, finally the supernatant was derived with BSTFA/TMCS (99/1,
431 v/v) (Sigma Aldrich) and the solution was injected into a GC-MS device. The
432 analytical procedure was described in Mezzatesta et al. (2021a). The analyses
433 were carried out with a ThermoFisher apparatus equipped with an 8-slot AI3000
434 automatic injector and a Focus GC chromatograph coupled to an ITQ 700 ion
435 trap type mass spectrometer. The capillary column used was a GOLD TG-5MS
436 Thermo trace (5% diphenyl and 95% dimethyl polysiloxane) with an internal
437 diameter of 0.25 mm, a length of 30 m and a film thickness of 0.25 μm . The
438 carrier gas used was helium with a constant flow rate of 1 $\text{mL}\cdot\text{min}^{-1}$. The
439 injector, the transfer line and the ion trap were respectively at 250, 300 and 200
440 $^{\circ}\text{C}$. The electronic impact source had an ionization energy of 70 eV, an
441 ionization time of 25,000 μs and a scan in the mass range of 40-650 m/z . The
442 injection volume of each sample is 1 μL in splitless mode.

445 6. Results and discussion

447 6.1 Gross composition of the extractable organic matter

448
449 Gross composition data are reported in Table 2. The extractable organic matter
450 covers a wide range, from 2% to 77.4% /weight of the sample. The average
451 value, deduced from the treatment of the 25 samples gave 33.8 % / weight and is
452 higher compared to the average value of 355 archaeological bituminous
453 mixtures from the Near and Middle East, mainly Iraq, which is 23.4 % (Connan
454 2012). The coating of reed boats sailing in Mesopotamia and the Gulf during the
455 Neolithic and Bronze Age (5100-1900 BCE) was found to be 17.9 % / weight.
456 The average values for hydrocarbons (sat. + aro.) and asphaltenes of al-Balīd
457 samples are respectively 6.0 and 73.4 % which means that the mixtures are
458 matching the composition of archaeological bitumens (Connan 2012). At that
459 level of interpretation, based on gross composition only, this preliminary
460 conclusion about bitumen occurrence remains a hypothesis in regard to the real
461 constituents of the caulking mixtures.

462 Plots of the gross composition data (Table 2), in two ternary diagrams
463 (Figs. 10 and 11) differentiate the samples. Most samples cluster in the area rich
464 in polar components, however the samples Nos. 2360 and No. 3212 are located
465 apart (Fig. 10). The specific character of samples Nos. 2360 and 3212 is
466 confirmed when considering the plot of hydrocarbons *vs.* resins *vs.* asphaltenes
467 (Fig. 11). A third sample joins this group, namely No. 2362. All other samples
468 cluster in an area with low hydrocarbons and various amounts of resins. Two
469 samples, Nos. 3227 and 3210, are extremely devoid of saturates and aromatics
470 and may not contain bitumen but some other components: resins, fats, oils.

471 Complementary detailed investigations will provide some answers. No. 3227
472 (Wo52) appears to be a filler over the end of a dowel driven obliquely from the
473 outside of the plank into the faying edge of the plank above (now missing). It
474 may have been used as a filler to smooth the surface of the plank. Gross
475 composition of sample No. 3212 (Fig. 10) matches those of a pure bitumen with
476 11.1 % saturates and 14.4 % aromatics, i.e. a rather rich composition in
477 hydrocarbons. This sample is extremely soluble in dichloromethane (61% by
478 weight/ sample). Sample No. 3226 (Fig.11) is a slab consisting of a
479 characteristic archaeological bituminous mixture of mostly asphaltenes and few
480 hydrocarbons.

481

482 **6.2 Carbon and deuterium isotopes**

483

484 Carbon and deuterium isotopes data are listed in Table 3. A plot of $\delta^{13}\text{C}_{\text{sat}}$ (‰ /
485 VPDB) vs. $\delta^{13}\text{C}_{\text{aro}}$ (‰ / VPDB) of al-Balīd data (Fig. 11) in comparison to
486 linear trend curves obtained from data on 32 oil seeps of Iran and 28 oil seeps of
487 Iraq, show that some samples (Nos. 3212, 3226, 2934bis, 3211) are close to the
488 straight lines defined by natural oil seeps while the others are located outside of
489 these references. One may notice that the sample No. 3212, discovered in an
490 iron container and which is obviously a pure bitumen with its shiny conchoidal
491 fracture, and the sample No. 3226, which is a typical bitumen slab, are falling on
492 straight lines defined by the reference oil seeps. Most of other samples are
493 located in the upper part of the diagram. This feature suggests that these samples
494 are composite mixtures with “recent” additives (resins, fish oils, fats, etc.) which
495 shift the bitumen carbon values of aromatics towards enriched values.

496 A plot of $\delta\text{D}_{\text{asp}}$ (‰ / SMOW) vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ / VPDB) in Figure 13,
497 exhibits a dispersed pattern on both parameters with $\delta\text{D}_{\text{asp}}$ (‰ / SMOW) values
498 and $\delta^{13}\text{C}_{\text{asp}}$ (‰ / VPDB) covering a wide range respectively from -180 to -60
499 (‰ / SMOW) for δD and -25 to -29 (‰ / VPDB) for $\delta^{13}\text{C}$ (Fig. 13). The same
500 plot restricted to oil seeps from Iran and presented in Figure 14 limits the δD
501 range from -30 to -110 (‰ / SMOW). The report of this result in Figure 13,
502 defined two zones: one area clustering the samples with bitumen only and
503 another zone where bitumen is likely mixed with other ingredients to be defined
504 by specific additional analyses. The split of samples into two populations is
505 confirmed when considering both $\delta^{13}\text{C}_{\text{asp}}$ (‰ / SMOW) and $\delta^{13}\text{C}_{\text{resin}}$ (‰ /
506 SMOW) in the plot of Figure 15 where samples containing bitumen only are
507 identified using deuterium data.

508 As a consequence, the data mean that both polar fractions are affected by
509 the suspected additives and that their $\delta^{13}\text{C}$ data may be also partly altered. One
510 should remember such a possibility when considering the diagram T_s/T_m vs.
511 $\delta^{13}\text{C}_{\text{asp}}$ (Fig. 20) as reference to trace the origin of bitumen.

512

513 **6.3 Mineralogical composition**

514

515 The mineralogical compositions, obtained by X-Ray diffraction on residue left
516 after the dichloromethane extraction, are reported in Table 4. As a general
517 statement, the major minerals (calcite, dolomite, quartz, feldspar, clays, gypsum,
518 anhydrite) detected in the samples are those which were previously identified in
519 archaeological bitumens from various sites in the Near East (Forbes 1955;
520 Connan 1988; Connan et al. 2005; Connan 2007; Connan 2012; Badel and
521 Kramm 2014). In addition to these conventional minerals, other specific
522 minerals (siderite, hematite, talc, halite, etc.) have been found. These minerals
523 reflect the available tempers incorporated to the bitumen when the caulking
524 mixtures were prepared and therefore may be variable with the dockyard where
525 the caulking paste was elaborated. Bitumen could have also been mixed near its
526 source and exported ready to be used, or even recycled multiple times, its
527 original composition changed in various harbours and boatyards. To explore
528 such hypotheses, the samples containing clay minerals (kaolinite and illite) were
529 identified in Figure 16. Seven samples correspond to samples which have
530 $\delta^{13}\text{C}_{\text{Casp}}$ (‰ / VPDB) between -27.3 and -25.5 and $\delta^{13}\text{C}_{\text{Cresin}}$ (‰ / VPDB)
531 between -26.3 and -25.0, i.e., samples which are obviously coming from Iran
532 and more likely from Khuzistan and Fars. All the samples with illite also have
533 kaolinite except for sample No. 3226. The plot of gypsum occurrence in Figure
534 17 leads to samples mostly with the same isotope characteristics and therefore
535 again samples from Iran.

536

537

538 **6.4 Biomarkers: steranes and terpanes (Table 5)**

539

540 Ratios, calculated on the basis of severely altered patterns where some
541 molecular structures are questionable, were discarded from the record.

542 Characteristic fingerprints of well-preserved samples are shown in Figure
543 18. Sample No. 3215 is typical of an oil seep from Iran. Terpanes do show the
544 occurrence of 18 α (H)-oleanane and a moderate Ts/Tm. Steranes are rather
545 enriched in diasteranes 27Sdia and 27Rdia. Sample No. 3226 has a different
546 terpane fingerprint with no oleanane and a low Ts/Tm ratio. Steranes are also
547 different with very low diasteranes and no visible C27S and 27R diasteranes.

548 Sterane composition, expressed as % of C27 $\beta\beta$ R+S, %C28 $\beta\beta$ R+S,
549 %C29 $\beta\beta$ R+S (not shown) differentiates two groups of samples. Samples Nos.
550 2935, 3210, 3220, 3227, 3228 (Table 5) are depleted in C27 and C28 steranes
551 and therefore are more biodegraded than other samples which display well
552 preserved patterns. To trace the possible origin of bitumens, two molecular
553 parameters already used in a previous study (Connan et al 2020) were
554 considered. A plot of 18 α (H)-oleanane vs. $\delta^{13}\text{C}_{\text{Casp}}$ (‰ / VPDB) in Figure 19A
555 shows that sample No. 3215 falls within the population defined by oil seeps

556 from Mamatain (Khuzestan) whereas some other samples, located as al-Balīd,
557 are included in the area defined by oil seeps from Ilam-Lorestan (Ain Gir,
558 Sarkan, Siah Kuh, Dehluran, Chersh Mehrghir, Naft Safid) but not exclusively
559 for oil seeps of Litedan in eastern Iran (Hormozgan Province) and Dalaki in
560 Bushehr are also matching. Four samples (Nos. 2382, 3209-3208, 2935 and
561 2935bis) gave unexpected $18\alpha(\text{H})$ -oleanane values in regard to their
562 corresponding $\delta^{13}\text{C}_{\text{Casp}}$ (‰ / VPDB) data. These samples have already been
563 defined as mixed samples where bitumen is not alone. The addition of other
564 organic ingredients may be responsible for the significant shift in the $\delta^{13}\text{C}_{\text{Casp}}$
565 (‰ / VPDB).

566 The comparison of results obtained on al-Balīd samples to samples of oil
567 seeps from Iraq (Mosul area, Hit and Kurdistan, Fig. 19B), indicates that few
568 samples in Iraq (Mishraq and Qaiara in the Mosul area and Sangaw in
569 Kurdistan) match the data of the al-Balīd samples. Three samples fall in the area
570 defined by Mishraq-Qayara in the Mosul area and Sangaw in Kurdistan and
571 three samples (Nos. 3209, 3218 and 2362) match the Zakho oil seeps located in
572 the far north of Iraq, close to Turkey.

573 As a consequence, the molecular and isotopic data tend to favour an origin
574 of the bitumen in two major areas: Mamatain (Khuzestan) and Dehluran-Ain
575 Gir-Chersh Mehrghir (Ilam). Some seeps of Iraq cannot be ruled out for both
576 their molecular and isotopic characteristics are similar to those recorded in oil
577 seeps from Iran however they are further north and rather unlikely as more
578 proximate Iranian sources are available.

579 The plot of T_s/T_m vs. $\delta^{13}\text{C}_{\text{Casp}}$ (‰ / VPDB), also currently used for
580 correlation studies, confirm the diversity of the samples from al-Balīd (Fig. 20).
581 The comparison of these data to those of oil seeps from Iraq (Fig. 21) and Iran
582 (Fig. 22) brings some additional information about sources. Some samples from
583 Iraq (Mishraq and Qaiara in the Mosul area, Sangaw and Pungala in south
584 Kurdistan, and Zhako in northern Iraq) fall within the zone defined by al-Balīd
585 samples but most samples do not match the al-Balīd archaeological samples.
586 The same diagram, built with oil seeps from Iran (Fig. 22), does show that 10
587 samples from al-Balīd match the oil seeps from Mamatain on one side and
588 Dehluran-Ain Gir-Siah Kuh- Chersch Mehrghir on the other side.

589
590 The source of some samples remains unknown, but one may remember
591 that the carbon isotopes on asphaltene are not fully reliable and ascribed to
592 bitumen for they are disturbed by the other organic substances present as
593 suggested by the $18\alpha(\text{H})$ -oleanane data which are unexpected in regard to their
594 corresponding carbon isotope values. In addition, one may not exclude the use
595 of mixtures of oil seeps from different sources to prepare the medieval caulking
596 material as determined elsewhere (Connan et al. 2020; Connan et al. 2018). Oils
597 from different origins (J.Connann, unpublished) have been identified on the hull
598 of a *shahoof*, a small vessel from Dubai, formerly part of the EISCA collection

599 at the Eyemouth Maritime Centre, Scotland, and recently acquired by the Oman
600 Across the Ages Museum (OAAM) in Manah, Oman.

601

602 *Composition of the organic mixtures: bitumen, fats and resins*

603 In this study, 11 samples were analysed (samples Nos. 3210, 3215, 3218, 3221,
604 3222, 3223, 3225, 3226, 3227, 3228 and 3229) in order to determine the
605 presence of organic matter.

606 The samples Nos. 3210 and 3227 demonstrated the presence of fatty
607 acids which are characteristic of fatty matter. It is here difficult to determine the
608 nature of this fatty matter because of the absence of specific fatty acids and/or
609 sterols. In sample No. 3210, the proportion of fatty acids such as palmitic,
610 stearic and oleic acids indicated a well-preserved oil while in sample No. 3227,
611 the occurrence of a large proportion of dicarboxylic fatty acids such as adipic,
612 pimelic, suberic and azelaic acids indicated the presence of an oil that is
613 naturally degraded with time (Mezzatesta et al. 2021b). The chromatographic
614 analyses of the samples No. 3221, No. 3222 and No. 3226 showed only
615 molecules of hopanes and steranes interpretable as the presence of bitumen. For
616 the other six samples referred as Nos. 3215, 3218, 3223, 3225, 3228 and 3229,
617 ursane and oleanane molecules were detected. These molecules are markers
618 indicating the presence of a triterpenic resin but the nature of this resin is
619 unknown considering the lack of specific biomarkers detected and/or identified.

620

621 Taking into account these new results, Figure 19 has been updated in
622 Figure 23 which shows that the preliminary evaluation of sample compositions,
623 carried out on the basis of carbon and deuterium isotopes of asphaltenes was
624 relatively accurate. Complementary investigations, carried out by Philippe
625 Schaeffer and Pierre Adam (Strasbourg Institut of Chemistry, UMR 7177,
626 Strasbourg University) on eleven archaeological samples and three copal
627 samples, excavated near tower 10, in the external part of the city wall of al-
628 Balīd, have shown that copal is not part of the ingredients used in the
629 bituminous mixtures analyzed on planks. Two triterpenic resins have been
630 identified as mixtures: dammar (Dipterocarpacea) and frankincense. The
631 presence of fats and bitumen has been confirmed by this additional study.

632

633

634 **7. Discussion**

635

636 *Bitumen*

637 One important outcome of the analysis on the luting and coating of the al-Balīd
638 timbers is that the substance used is a bitumen-based amalgam. This aspect is
639 particularly interesting because historical and archaeological evidence indicate
640 that Indian Ocean boatbuilders used a variety of different materials to seal the

641 planking of sewn boats. In the 10th century Abū Zayd al-Sīrāfī remarks that the
 642 seams and sewing holes of the boats of Siraf were caulked with oil mixed with
 643 other substances (Al-Sīrāfī and Ibn Faḍlān 2014). Portuguese sources describe
 644 the use of pitch, bitumen and resin, in the vessels they saw along the coasts of
 645 East Africa and western India in the 16th century (Agius 2007; Moreland 1939;
 646 Prins 1986; Stanley 1869). A dark substance on the surfaces and edges of the
 647 Quseir al-Qadim sewn-ship timbers has been tentatively interpreted as bitumen,
 648 but no scientific analysis has been carried out to confirm the identification (Blue
 649 et al. 2011). The builders of the 9th-century Belitung shipwreck mostly used a
 650 lime-based putty to seal the hull planking, posts and through-beams (Flecker
 651 2000), though bitumen was found encrusted on one of its through-beams
 652 (Burger et al. 2010).

653
 654 The apparent homogeneity in luting material in the al-Balīd timbers clearly
 655 contrasts with the heterogeneity indicated by the textual sources and other ship
 656 remains of the Indian Ocean. It may also suggest that the timbers could have
 657 belonged to vessels from a particular region of the Indian Ocean, which
 658 privileged the use of this substance (see below). Considering that the date and
 659 thickness of the timbers (Ghidoni and Pavan 2022) point to vessels of different
 660 sizes and functions sailing between the late 10th and the mid-15th centuries, it
 661 also suggests that bitumen amalgams were commonly used for luting and
 662 coating in the Indian Ocean for at least four centuries.

663
 664 This apparently common use of bitumen in the medieval Indian Ocean is also
 665 important because this material is predominantly associated with Neolithic and
 666 Early Bronze Age boatbuilding in the region, and more generally to reed-built
 667 vessels. It is assumed that from the Iron Age, bitumen was replaced by other
 668 substances, at least for coating watercraft, and thought to have gradually
 669 disappeared in the Islamic era (Vosmer 2007). However, the evidence from al-
 670 Balīd indicates that the use of this material in the Indian Ocean boatbuilding
 671 context persisted well into the medieval period.

672
 673 *Bitumen in 19-20th-century Indian Ocean sewn boats*

674 The common use of a bitumen-based compound in medieval boatbuilding as
 675 suggested by the al-Balīd timbers also contrasts with the evidence from more
 676 recent sewn vessels of the Indian Ocean, which indicates various materials.
 677 Resin appears to have been the most common material for luting in ethnographic
 678 records of the Indian Ocean (Hornell 1942; Hill 1958; Severin 1985; Agius
 679 2002; Vosmer et al. 2011). Omani boatbuilders used it to impregnate the
 680 wadding of the *beden seyad*, a sewn fishing boat documented in Muscat in 1839
 681 (Pâris 1843). In East Africa, a paste made with mangrove bark was the preferred
 682 luting for the *mtepe* planking (Lydekker 1919). In fact, bitumen-based luting and
 683 coating are seldom attested in sewn boats in the region with only a few cases

684 from the Gulf and Oman. The sewn *baggāraslāmeles* of Hormuzgan, southern
 685 Iran, in the collection of Qatar Museums, have their hulls covered inboard and
 686 outboard by a black substance only provisionally identified as bitumen (Cooper
 687 et al. 2020). However, although no chemical analysis was carried out to
 688 scientifically identify the substance, interviews with Iranian boatbuilders
 689 confirmed that this type of boat was generally coated with bitumen (Kalantar et
 690 al. forthcoming). Chemical analysis carried out by the authors of this paper
 691 identified bitumen as the luting of an Omani *kambārī*, a sewn vessel on display
 692 in the Museum of the Frankincense Land, Salalah. However, this boat is a 1980s
 693 reconstruction (Weismann et al. 2019), built for display only, and the bitumen
 694 used in the luting could be a product recently used by the builders and might not
 695 reflect past practices and materials. Other evidence from Indian Ocean sewn
 696 boats might point to the use of bitumen luting. The “pitch” sealing the sewn
 697 *beden* planking recorded by Chittick (1980) in Hafun, Somalia, could also have
 698 been bitumen, since no analysis was carried out to confirm the nature of the
 699 material. Similarly, Shaikh (2012) mentions coal-tar being used, but it is not
 700 clear whether the substance was identified scientifically.

701

702

703 *Bitumen mixture*

704 The other important outcome of the geochemical analysis is that the al-Balīd
 705 luting samples are not made exclusively of pure bitumen. Instead, medieval
 706 boatbuilders added different matter to the bitumen to create a compound, where
 707 bitumen is only one of the many ingredients. This is certainly not a surprise, but
 708 rather a confirmation of a practice used in boatbuilding—as well as in other
 709 terrestrial purposes—in Mesopotamia, the Gulf, and perhaps Arabian Sea, in the
 710 Neolithic and Bronze Age (Cleuziou and Tosi 1994; Connan *et al.* 2005;
 711 Connan and Van de Velde 2010; Potts 1997: 100; Vosmer 2003a; 2003b).

712

713 The reason for adding modifiers of different material to the bitumen is to change
 714 its physical properties and obtain a compound suitable for the use in a
 715 boatbuilding context. To be used as luting, and more generally as a sealing
 716 agent, this amalgam has to be waterproof; maintain a certain flexibility or
 717 plasticity which prevent it from cracking; avoid melting or becoming too fluid
 718 when subjected to heat and sunlight; and maintain adherence to the timber under
 719 a variety of conditions, particularly when wet (Vosmer 2003a).

720

721 Tests carried out during the construction of a hypothetical 3rd millennium BC
 722 bitumen-coated reed-built vessel (Vosmer 2000; 2001; 2003a; 2003b; 2007),
 723 indicate that the use of mineral additives improves the working properties of
 724 bitumen. The use of calcium hydroxide – Ca(OH)₂ or in particular, calcite
 725 (calcium carbonate - CaCO₃) which is the predominant mineral additive in the
 726 samples from al-Balīd, increases the strength and durability of the bitumen

727 without compromising its flexibility (Vosmer 2003b; Vosmer 2007).
728 Mineralogical analysis shows that boatbuilders added a large quantity of calcite
729 in the bitumen used to coat reed vessels in the Gulf and Mesopotamia during the
730 3rd millennium BC (Connan et al. 2005). This indicates not only that this
731 practice lasted several millennia, but that during this period the bitumen mixture
732 has not changed much and that similar amalgams were used for different
733 purposes in both reed and wooden vessels.

734
735 The mineral matter in the bitumen amalgam also makes the bitumen resistant to
736 heat exposure. This would have been a major issue in Mesopotamia and the
737 Gulf, with extremely high temperatures in summer, causing pure bitumen to
738 ‘creep’ easily, again reducing its sealing properties. Even today, calcium
739 carbonate is added to various hydrocarbon polymers to improve their impact
740 resistance and working properties, as well as being used in bituminous cements
741 and bituminous paving (ASTM 2019).

742
743 Lastly, being a considerable part of the total compound’s weight, this mineral
744 matter increases the volume of the bitumen. This would have been an important
745 factor for ancient boatbuilders, enabling them to reduce the amount of bitumen
746 used thus saving on such a valuable and expensive material.

747
748 The presence of triglycerides (natural fats and oils from animal and plants) in
749 the amalgam increases the adhesive properties of bitumen (Cleuziou and Tosi
750 1994; Vosmer 2003b). Bitumen is in fact hydrophobic and does not adhere
751 easily with wood, which is hydrophilic. Natural fats and oils in small percentage
752 (2.5-3%) act as “antistripping” agents and help the bond between different
753 materials (Vosmer 2007). Their use is further necessary after the addition of the
754 mineral (inorganic) matter which reduces the adhesion of the bitumen. Another
755 important function of triglycerides is to increase the plasticity of the amalgam,
756 when the addition of minerals might make it hard and brittle, and prone to crack.

757
758 The results of the bitumen analyses do not show any correlations between their
759 geochemical signature and their location within the timbers. It does not look as
760 though medieval boatbuilders used different amalgams whether they used the
761 bitumen on the faying edge of plank or on the plank under the wadding. This
762 evidence might point to the presence of a “maritime” bitumen amalgam, used
763 for the luting, caulking and coating of Indian Ocean vessels, its differences in
764 chemical composition depending to where it was prepared. This product
765 intended for boatbuilding could have been mixed near the sources and exported
766 to maritime centres in the Indian Ocean as a ready-made product. Alternatively,
767 Indian Ocean boatyards could have imported pure bitumen from the Iranian
768 sources and that boatbuilders mixed it *in situ* during the construction of the
769 boats, using the same compound regardless of its location and function.

770

771 The bitumen geochemical analysis does not suggest any particular trend or
772 change of the mixture in time. Although variations occur within the chemical
773 and mineralogical composition of the samples examined, these appear too
774 random to suggest intentional modifications or changes of the amalgam by
775 Indian Ocean boatbuilders during the period indicated by the date of the timbers.
776 It is also important to note that bitumen is a material that can be recycled
777 multiple times, and some, if not all, of the bitumen in the al-Balīd timbers could
778 have been stripped from old vessels and reused in the building and repairing of
779 other boats.

780

781 Because of the scarce evidence of bitumen products associated with boats from
782 this period it is not possible to compare the amalgams from al-Balīd with other
783 archaeological 'bitumens' from the region. However, the al-Balīd mixtures
784 appears rather unique for these are diversified amalgams ranging from pure
785 bitumen with minerals to complex mixtures with bitumen, oil, resin and
786 minerals which have been rarely observed for this period.

787

788

789 *Mixture contamination (materials unintentionally added to the amalgam)*

790 One should be aware that the presence of calcite, resin, and, possibly, oil and fat,
791 in the bitumen, could also be explained by the fact that these materials are
792 generally associated to boatbuilding in the Indian Ocean, and could perhaps not
793 have been part of the original mixture. For example, fish oil was widely used to
794 preserve and maintain the planking of both sewn and nailed wooden vessels in
795 the Indian Ocean until recently (Agius 2002; Al-Hijji 2001; Bowen 1952;
796 Donaldson 1979; Hornell 1942, 1970; Johnstone and Muir 1962; Lorimer 1915;
797 Miles 1919; Vosmer 1997). Various medieval sources mention the use of fish
798 and whale oil to coat the hulls of ships sailing in the Indian Ocean (Agius 2007),
799 a practice that appears to have been in use in the Gulf since at least the 3rd
800 millennium (Cleuziou and Tosi 1994). Moreover, oil was also used on the
801 stitching to protect it and keep it moist, extending the life of the ship (Severin
802 1982), while experiments with dry, wet and oiled sewing fibres of *Arenga*
803 *pinnata* and coir (coconut husk fibre) demonstrated that soaking in oil increased
804 the strength of the fibres (Tyrrell 2019). By smearing oil on the hull planking,
805 medieval boatbuilders would have applied it on the luting, unintentionally
806 mixing it with the bitumen amalgam.

807

808 Quicklime (CaO), obtained from burnt shells and corals, and goat fat are the
809 main ingredients of the traditional antifouling (Arabic: *shaham*;
810 Malay/Tamil/Chinese: *chunam*) of Indian Ocean watercraft (Agius 2002; Yule
811 and Burnell 1886). The hulls of vessels were periodically coated with this
812 material outboard below the waterline to protect them against ship worms

813 (*Teredo navalis*) and barnacles (Agius 2002; Lorimer 1915; Vosmer 2007, 2017;
814 Weismann et al. 2014). References to this practice date back to the medieval
815 period in Arab, Indian and Chinese ships, as mentioned by al-Mas'ūdī (1861)
816 and Marco Polo (Yule 1871). The use of a lime-based substance is also indicated
817 by archaeological evidence from medieval shipwrecks from China and
818 Southeast Asia (Flecker 2001; 2007; Li 1989). Timber Wo37 from al-Balīd
819 bears traces of a white substance on its outer surface which resembles *chunam*.
820 The amount of substance was too little to allow scientific analyses to determine
821 its chemical composition and the identification of the material. However, its
822 colour and texture match those of the Indian Ocean antifouling, corroborating
823 information provided by historical sources. As in the case of oil, *chunam* would
824 have coated the planking seams and luting, perhaps leaving some traces in the
825 bitumen below.

826
827 Seven samples reveal the presence of resin in the bitumen mixture. Apart from a
828 16th century Portuguese source reporting vessels in East Africa caulked with
829 black “pitch” mixed with resin and incense (Prins 1986), resin is not usually
830 associated with bitumen in boatbuilding. A lump of bitumen discovered in
831 Akkaz, Kuwait, and dated to the Partho-Sasanian Period is the only other
832 archaeological evidence for the use of resin in a bituminous compound (Connan
833 2011). The function of this mixture is still not clear, but it appears very similar
834 to substances used for mummification in Pharaonic Egypt (Connan 2011), and it
835 might not have used in a maritime context. As we have previously seen, resin
836 was widely used with the same purpose of bitumen in other sewn boats of the
837 Indian Ocean and often mentioned in the historical sources. Perhaps the bitumen
838 luting in the al-Balīd timber was applied when the vessel was resewn and
839 smeared over a previous luting made of resin, or *vice versa*, causing the two
840 substances to mix together.

841
842 Overall, the evidence mentioned above raised the possibility that some of the
843 materials in the bitumen amalgam on the al-Balīd timbers were not originally
844 added to the mixture by the boatbuilders. The fact that it is likely that some of
845 these materials occurred on the hull of medieval vessels, might suggest that their
846 presence in the amalgam could have been instead the result of an accidental
847 mixing at a later stage, when these elements and the bituminous mixture came
848 into contact.

849
850 Chemical analysis provides insights into the sources of the natural bitumen used
851 in the al-Balīd timbers, suggesting southwest Iran seeps for most of the samples.
852 A possible Iranian origin corroborates the only documentary source on the topic
853 by the Dominican missionary Jordanus (1863), who, in the early 14th century,
854 reports that “in Persia are springs, from which flows a kind of pitch”. A similar
855 origin for the bitumen samples is significant, because it points to this

856 geographical area as the main source of bitumen in the Indian Ocean during the
857 medieval period. It also appears to confirm a trend noted for earlier periods
858 based on the study of bitumen lining torpedo jars, which indicated a shift from
859 Iraqi to Iranian sources between the 1st and the 10th century CE.

860
861 Very little is known about the use and trade of bitumen in the medieval period,
862 but this material would have probably been transported from the natural seeps of
863 the interior to the coastal centres in the southern Iranian coast, such as Siraf,
864 Qays (Kish) and Hormuz. Unfortunately, the geochemical analysis cannot tell us
865 whether the bitumen would have reached coastal areas already mixed and ready
866 to use, or the amalgam was prepared at the boatyards before being used.

867
868 One important question raises from the identification of the bitumen sources:
869 can the origin of the bitumen help us to determine the places where the boats
870 luted with it were built? It is difficult to answer this question because material
871 origin in the Indian Ocean does not necessarily match with the places where
872 these materials were used.

873 It is plausible that at least some of the al-Balīd timbers belonged to ships built in
874 city ports in southern Iran. Siraf is probably the closest to the bitumen sources
875 and had strong links with southern Arabia and particularly East Africa, the latter
876 being the region where many of the timber species from the al-Balīd timbers are
877 native. Historical sources also inform that the city imported exotic wood from
878 India and East Africa (Agius 2007; Chittick 1977), while the pottery type
879 discovered during the excavations also reveals a link with East Africa
880 (Priestman 2021).

881
882 Ships from Siraf were so common in southern Arabia and Red Sea, that the
883 Arabian Sea until Yemen was also known as *Bahr Fāris*, “the sea of Fars”,
884 where most of the “shipbuilders and seafaring men are Persians” (Hourani
885 1963). Hourani (1963) also states that it is probable that in the 10th century ships
886 from Siraf reached East Africa by sailing along the coast to Aden and round the
887 Somali coast, instead of choosing a direct route to Cape Guardafui, to avoid
888 Indian pirates from Socotra. Diaspora from Siraf helped the growth of ports like
889 Jedda and Aden and contributed to the development of East African centres like
890 Mogadishu, Kilwa and Malindi (Agius 2007).

891
892 However, the city of Siraf declines in the 10th century, a date earlier than the
893 date range of the al-Balīd timbers (late 10th-15th centuries CE). The island of
894 Qays (Kish), which flourished in the 11-12th centuries, and where some of the
895 merchants of Siraf moved after the city’s demise, might be a better candidate as
896 a boatbuilding place for the “al-Balīd ships”. Historian Yāqūt claims that the
897 ruling house of Quays had Southern Arabian origin and controlled the Arabian
898 Sea and was in fact the ruler of Oman (Goitein and Friedman 2007).

899

900 New Hormuz would have also been an important boatbuilding place. It was an
901 important city port, actively involved in the Indian Ocean trade, with a seaborne
902 empire controlling coastal centres in Oman and in the Gulf (Agius 2007).

903

904 Unfortunately, none of these Iranian coastal sites have provided direct evidence
905 for boatbuilding facilities, let alone the use of bitumen in a maritime context,
906 which might link the al-Balīd timbers to ships from southern Iran. In fact, the
907 probable Iranian origin of the al-Balīd bitumen does not necessarily imply that
908 boats were built near the material source. In a world like the Indian Ocean, with
909 intertwined material trade networks connecting various centres along its coasts,
910 it is likely that bitumen was one of the materials traded. As in the case of timber
911 for shipbuilding, lacking in places like the Arabian Peninsula, Red Sea and the
912 Gulf, luting material such as bitumen had to be imported for the construction of
913 vessels in these regions.

914

915 However, the use of bitumen as opposed to other substances, might hint towards
916 some regions of the western Indian Ocean, or exclude others, as possible places
917 where the “al-Balīd” boats were built or repaired. For example, ethnographic
918 records indicate that the preferred material to lute Indian sewn boats from the
919 Lakshadweep, Kerala and Goa was a mixture made of resin (Shaikh et al. 2012;
920 Varadarajan 1993; Severin 1985, Vosmer et al. 2011), which is largely available
921 in those areas, and of good quality (Edye 1835; Regert et al. 2008). In East
922 Africa, boatbuilders of the sewn *mtepe* smeared a thick paste of local mangrove
923 bark to lute its hull (Hornell 1941). Although these are relatively recent
924 evidence, one might consider it unlikely that these areas would have needed to
925 import bitumen from Iran, when they could use locally available and cheaper
926 material. The bitumen luting of the al-Balīd timbers, therefore, might point to
927 boatbuilding places in the Gulf, Arabian Peninsula, and Red Sea, which have no
928 luting material such as resin and are relatively close to the Iranian bitumen
929 sources.

930

931 In the Gulf, Bahrain is one possible origin for some of the “al-Balīd boats”,
932 because of its long maritime history and proximity to Iran. Archaeological
933 excavations at Qal‘at al-Bahrain indicate that the site imported bitumen for
934 millennia until the 16th century, the sources of which were exclusively Iranian
935 during the medieval period (Connan et al. 1998).

936

937 Some of the al-Balīd timbers could have belonged to vessels built in al-Balīd. A
938 city so deeply involved in maritime trade would have certainly had shipbuilding
939 facilities. Newton and Zarins (2017), who directed the excavation at al-Balīd
940 between 2005 and 2012, have suggested that a large complex located in the
941 southeast limit of the site could be interpreted as a drydock. The large number of

942 ship timbers recycled in the citadel of the site also strongly point to the building
943 and repairing of watercraft (Ghidoni and Pavan 2022). Occurrence of
944 frankincense in the bituminous mixtures suggests at least repairs of boats at al-
945 Balīd. Moreover, the pure bitumen in the metal container discovered in al-
946 Balīd, might also be a clue for these activities at the site premises. Bitumen
947 could have been imported to al-Balīd and there mixed with other materials to
948 create the amalgam to lute the ships built or repaired at the site.

949 Lastly, one should also consider that the bitumen amalgam on the al-Balīd
950 timbers might not have necessarily been applied during the construction of the
951 vessel, but at a later stage. The sewing cordage of sewn boats had to be regularly
952 replaced, and it is also possible that the luting on the al-Balīd timbers might
953 have been smeared on when the vessels had been re-sewn.

954

955

956

957

958 **Conclusion**

959

960 Chemical and mineralogical analyses of the luting of the al-Balīd timbers offer
961 information about the sources, processing and use of bitumen in the Indian
962 Ocean during the medieval period. They also yield insights into boatbuilding
963 materials and practices in the region, deepening our understanding of sewn-
964 plank construction during the middle Islamic period. The identification of the
965 timber luting as a bitumen-based compound suggests that bitumen was the
966 predominant material used by the coastal communities of the western Indian
967 Ocean to seal the planking of their vessels. The data from al-Balīd also indicates
968 that the use of bitumen amalgams to waterproof hulls of vessels in the region,
969 generally associated to much earlier periods, continued in the 10-15th centuries,
970 revealing the key role played by bitumen in the Indian Ocean boatbuilding
971 context for millennia.

972 Only a limited area of al-Balīd has been excavated, and a large number of ship
973 timbers are visible in the masonry and rubbles of the buildings. Future
974 excavations will offer the opportunity for further analysis to determine whether
975 there are changes in bitumen sources and variation in its mixture through time.

976

977

978

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980

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996

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1435 **Figure Captions**

1436

1437 Figure 1- Map showing the location of al-Balīd, Oman. (Photo: A. Ghidoni).

1438

1439 Figure 2- (A): Aerial view of al-Balīd with the main buildings excavated (Photo:
 1440 A. Pavan). (B): general view of the citadel (*husn*) where the ship timbers were
 1441 discovered (Photo: M. Massa).

1442

1443 Figure 3- (A): A ship beam recycled as a lintel over a small window. (B and C):
1444 Ship planks used as levelling courses in the walls of the al-Balīd citadel.
1445 (Photos: A. Ghidoni).

1446
1447 Figure 4-Outer surface of timber BA0604145.175 (above) and the main features
1448 of western Indian Ocean sewn-plank construction technique (below).

1449
1450 Figure 5- (A): “Bitumen” on the edge of timber BA1104065.454 suggesting the
1451 practice of luting the seams of the planks before their fitting. (B): The luting
1452 process, in this case carried out using *dammar* resin, during the construction of
1453 the al-Hariri Boat, a reconstruction of a 13th century Indian Ocean sewn
1454 merchant vessel based on the illustrations of the *Maqamat* al-Hariri. (C-E): A
1455 thick layer of “bitumen” covers the section between the sewing holes and the
1456 plank’s edge on timbers BA0604145.175 and BA1104065.454. (D-E): The
1457 boatbuilders of the *Jewel of Muscat*, a reconstruction of a 9th century sewn cargo
1458 vessel based on the Belitung shipwreck discovered in Indonesia, used dammar
1459 resin to seal the seams of the planks after their fitting and before commencing
1460 the sewing. (Photos: A. Ghidoni).

1461
1462
1463 Figure 6- (A): Timber Wo37 displaying a thin layer of “bitumen”, along with a
1464 white substance, on the outer side. (B): A *baggāra/āmele* from Iran, recently
1465 acquired by Qatar Museums (QM), coated outboard with a thick layer of
1466 “bitumen”. (C): “Bitumen” The boatbuilders of the *Jewel of Muscat*, a
1467 reconstruction of a 9th century sewn cargo vessel based on the Belitung
1468 shipwreck discovered in Indonesia, used dammar resin to seal the seams of the
1469 planks after their fitting and before commencing the sewing. Covers most of the
1470 inside face of timber BA0604172.69. (D): The thick inboard bitumen coating of
1471 a *baggāra/āmele* from Qatar Museums.

1472
1473 Figure 7- Bitumen lump (X-35) discovered in the recent excavations of the
1474 citadel.

1475
1476 Figure 8- Metal vessel (X43) with a coat of bitumen inside.

1477
1478 Figure 9- Map showing the reference oil seeps analyzed in Iran (after Connan et
1479 al. 2020).

1480
1481 Figure 10- Gross composition data in a ternary diagram: Saturates (%) vs.
1482 Aromatics (%) vs. Polars (%).

1483
1484 Figure 11- Gross composition data in a ternary diagram: Saturates + Aromatics
1485 (%) vs. Resins (%) vs. Asphaltenes (%).

- 1486
 1487 Figure 12- $\delta^{13}\text{Caro}$ (‰ /VPDB) vs. $\delta^{13}\text{C}_{\text{sat}}$ (‰ /VPDB): comparison of al-Balīd
 1488 samples to reference curves from 32 oil seeps of Iraq and 28 oil seeps of Iran.
 1489
- 1490 Figure 13- $\delta\text{D}_{\text{asp}}$ (‰ / SMOW) vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB): expected composition
 1491 of al-Balīd samples.
 1492
- 1493 Figure 14- $\delta\text{D}_{\text{asp}}$ (‰ / SMOW) vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) for oil seeps of Iran.
 1494
- 1495 Figure 15- $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) vs. $\delta^{13}\text{C}_{\text{resin}}$ (‰ /VPDB): comparison of al-
 1496 Balīd samples to the reference curves of 8 oil seeps from Iran and 32 oil seeps
 1497 from Iraq. Identification of al-Balīd samples referred as containing bitumen
 1498 only.
 1499
- 1500 Figure 16- $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) vs. $\delta^{13}\text{C}_{\text{resin}}$ (‰ /VPDB) of al-Balīd samples
 1501 with the identification of their clay minerals: kaolinite, kaolinite + illite, illite.
 1502
- 1503 Figure 17- $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) vs. $\delta^{13}\text{C}_{\text{resin}}$ (‰ /VPDB) of al-Balīd samples
 1504 with the identification of samples containing gypsum.
 1505
- 1506 Figure 18- Mass fragmentograms of steranes (m/z 217) and terpanes (m/z 191) of
 1507 two samples of al-Balīd: No. 3215 and No. 3226. Significance of abbreviations:
 1508 21St = pregnane , 22St=20-methylpregnane, 27 $\alpha\alpha\alpha$ S= 5 α ,14 α ,17 α -cholestane
 1509 20S, 29 $\alpha\alpha\alpha$ R= 5 α ,14 α ,17 α -stigmastane 20R, 29 $\alpha\alpha\alpha$ R= 5 α ,14 α ,17 α -
 1510 stigmastane 20R, 27Sdia = 13 β ,17 α -diacholestane (20S), 27Rdia = 13 β ,17 α -
 1511 diacholestane (20R), Ts= 18 α ,22,29,30-trisnorneohopane, Tm = 17 α ,22,29,30-
 1512 trisnorhopane, C29 $\alpha\beta$ = 17 α ,21 β -30-norhopane, C31 $\alpha\beta$ H = 17 α ,21 α -hopane,
 1513 C31 $\alpha\beta$ HS = 17 α ,21 β -30-homohopane(22S)
 1514
- 1515 Figure 19- Plot of 18 α (H)-oleanane vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB). Comparison of al-
 1516 Balīd samples with (A) -60 oil seeps from Iran, (B) 44 oil seeps from Iraq.
 1517
- 1518 Figure 20- Plot of Ts/Tm vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) for the al-Balīd samples.
 1519
- 1520 Figure 21- Plot of Ts/Tm vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) for the oil seeps of Iraq:
 1521 comparison to the al-Balīd samples.
 1522
- 1523 Figure 22- Plot of Ts/Tm vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB) for the oil seeps of Iran:
 1524 comparison to the al-Balīd samples.
 1525
- 1526 Figure 23- $\delta\text{D}_{\text{asp}}$ (‰ / SMOW) vs. $\delta^{13}\text{C}_{\text{asp}}$ (‰ /VPDB): modified figure 13
 1527 according to the results of complementary chemical investigations.

1528

1529 Table 1- Basic information about the al-Balīd samples analyzed.

1530

1531 Table 2- Gross composition data on the al-Balīd samples

1532

1533 Table 3- Isotopic data on the al-Balīd samples

1534

1535 Table 4- Results of the mineralogical analysis of some samples of al-Balīd

1536

1537 Table 5- Molecular data on the al-Balīd samples

1538



Musandam

IRAN

GULF OF OMAN

U.A.E

Batinah Coast

Muscat

Sur

Ra's al-Hadd

SAUDI ARABIA

OMAN

Masirah

ARABIAN SEA

Dhofar

Hallaniyat Islands

**Al-Balīd
(Salalah)**

YEMEN

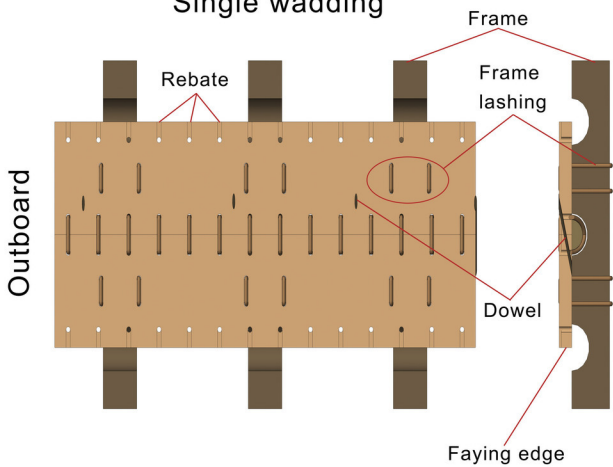






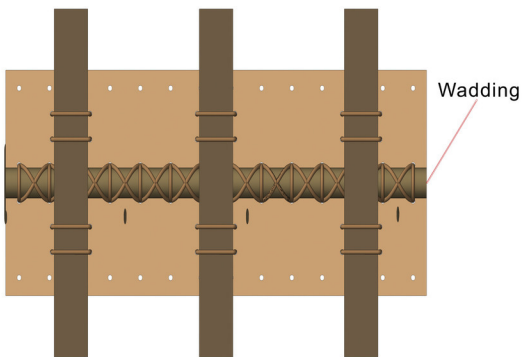
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Single wadding



Outboard

Inboard



Wadding





A



B

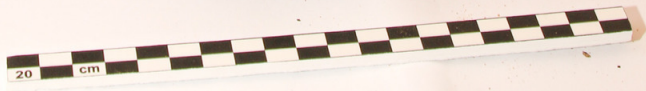


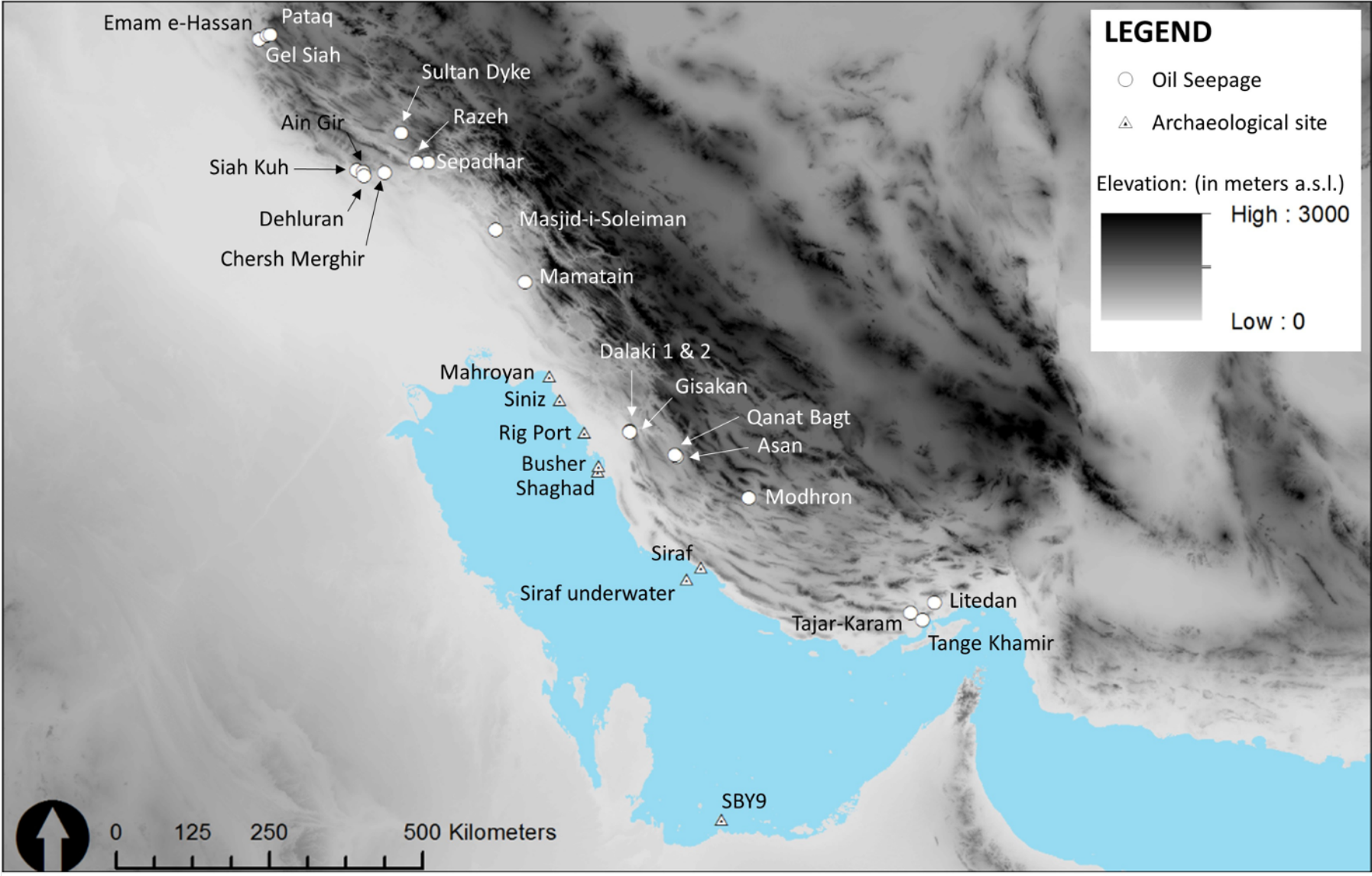
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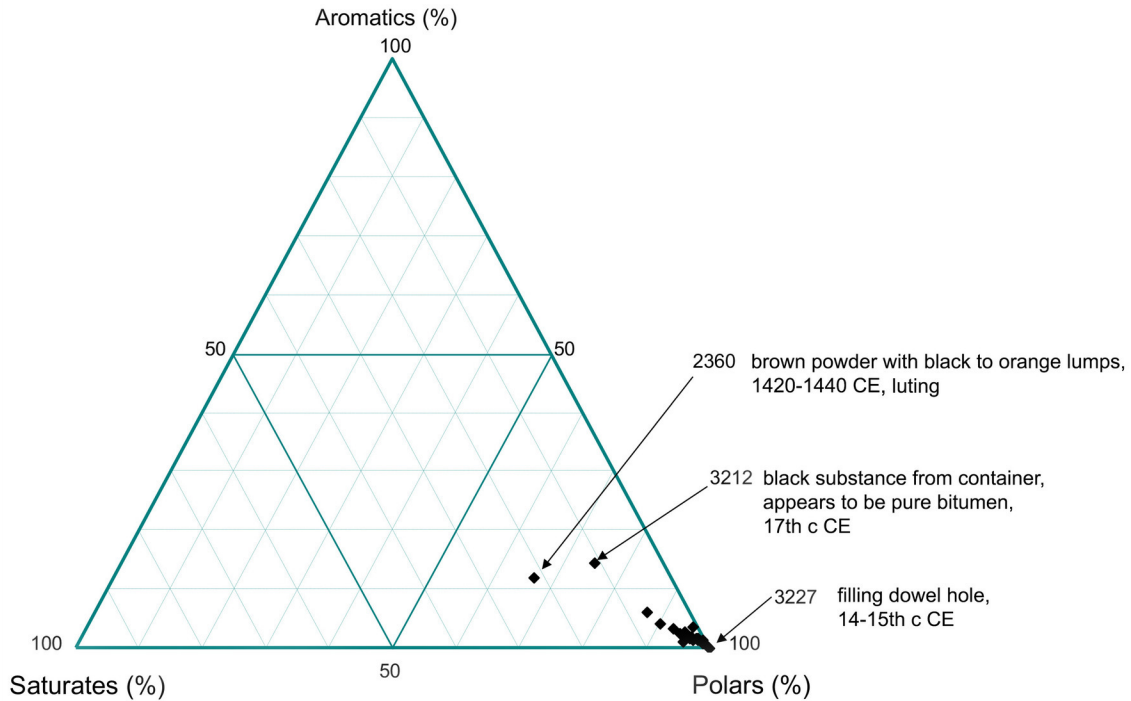


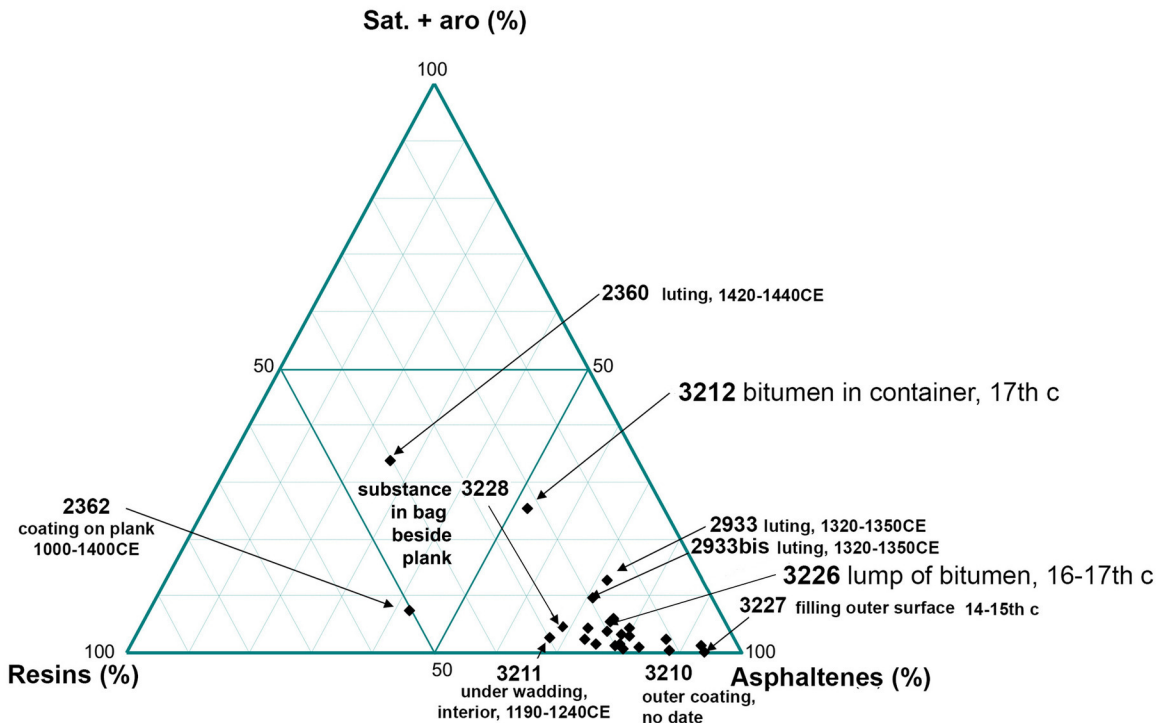
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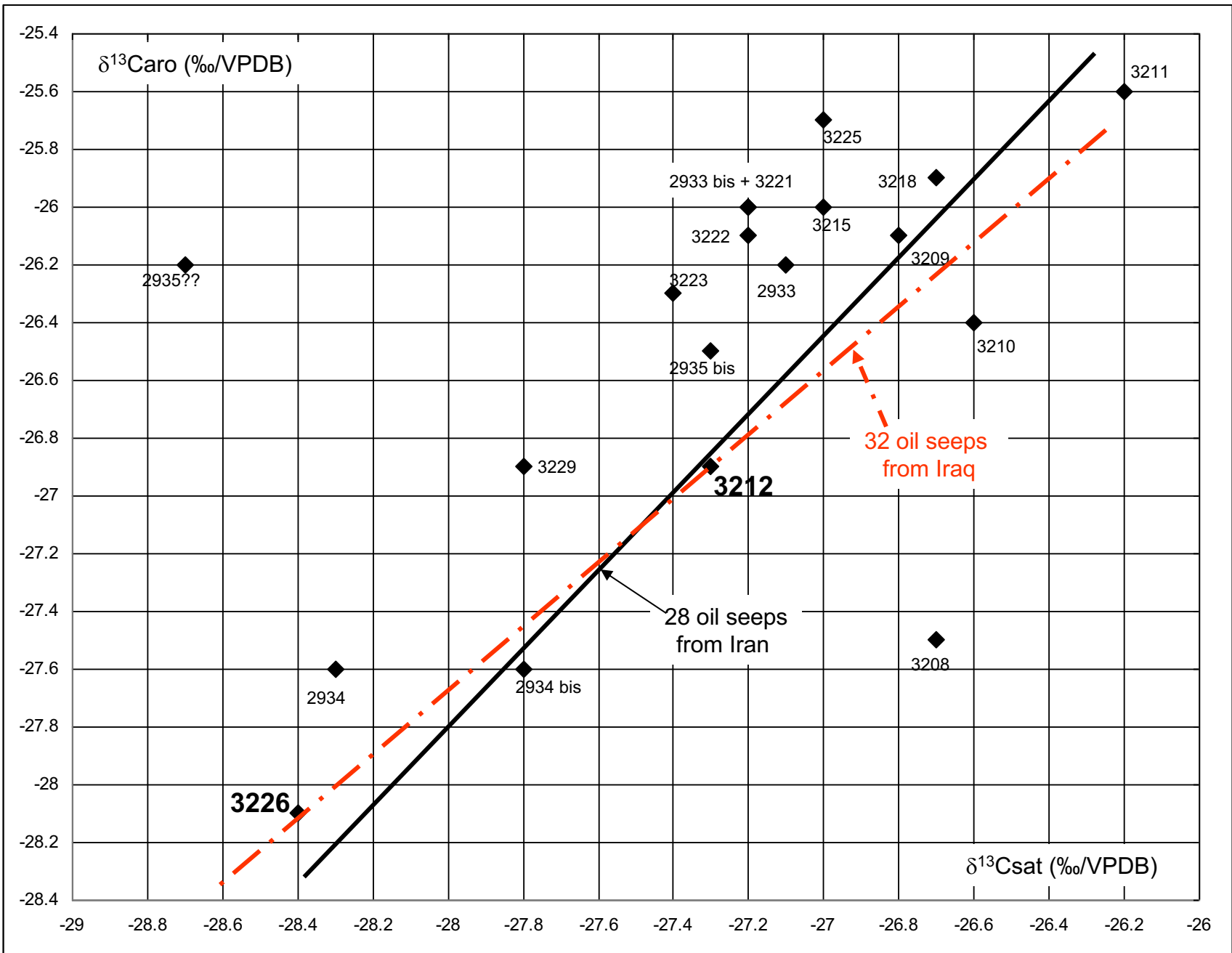


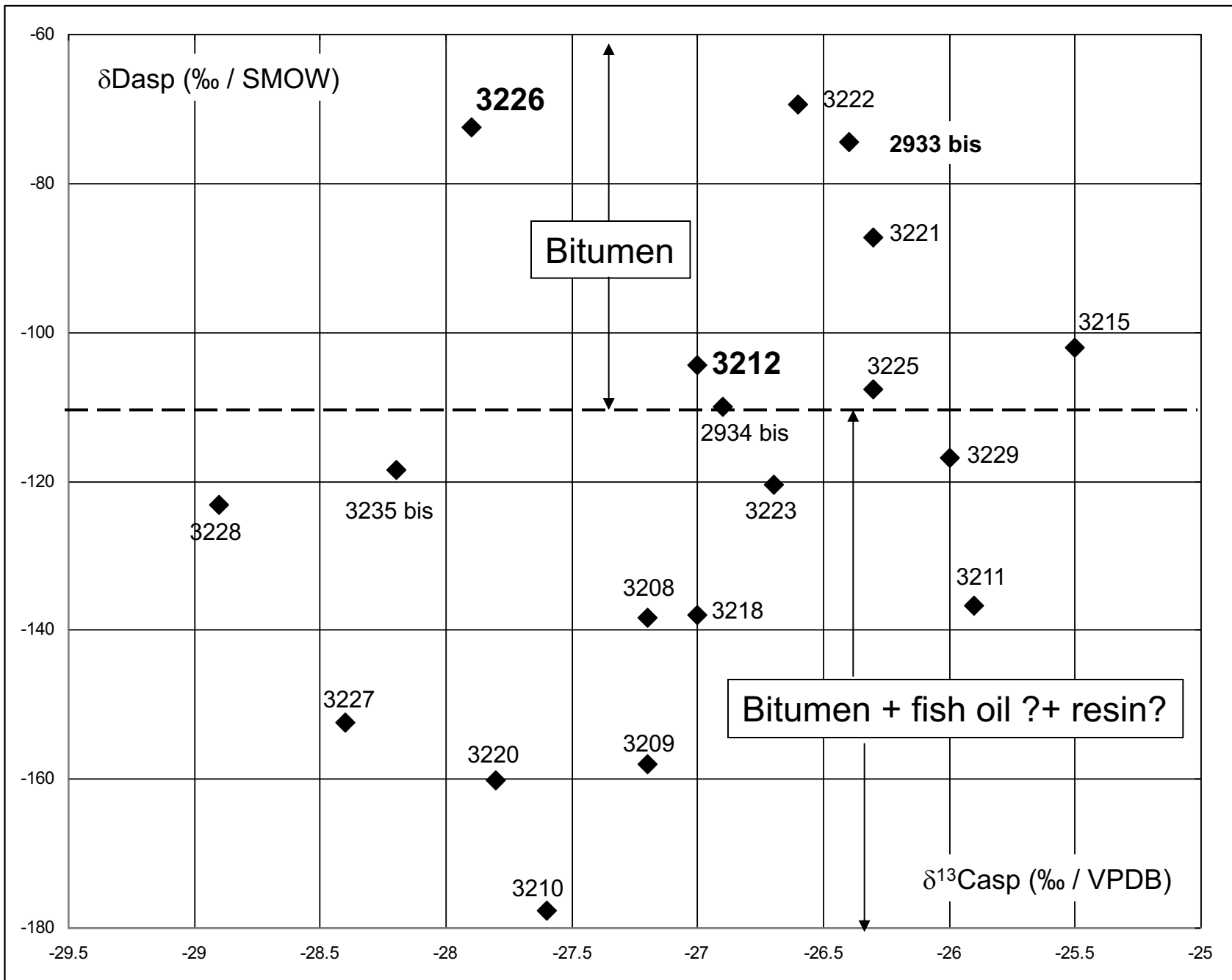


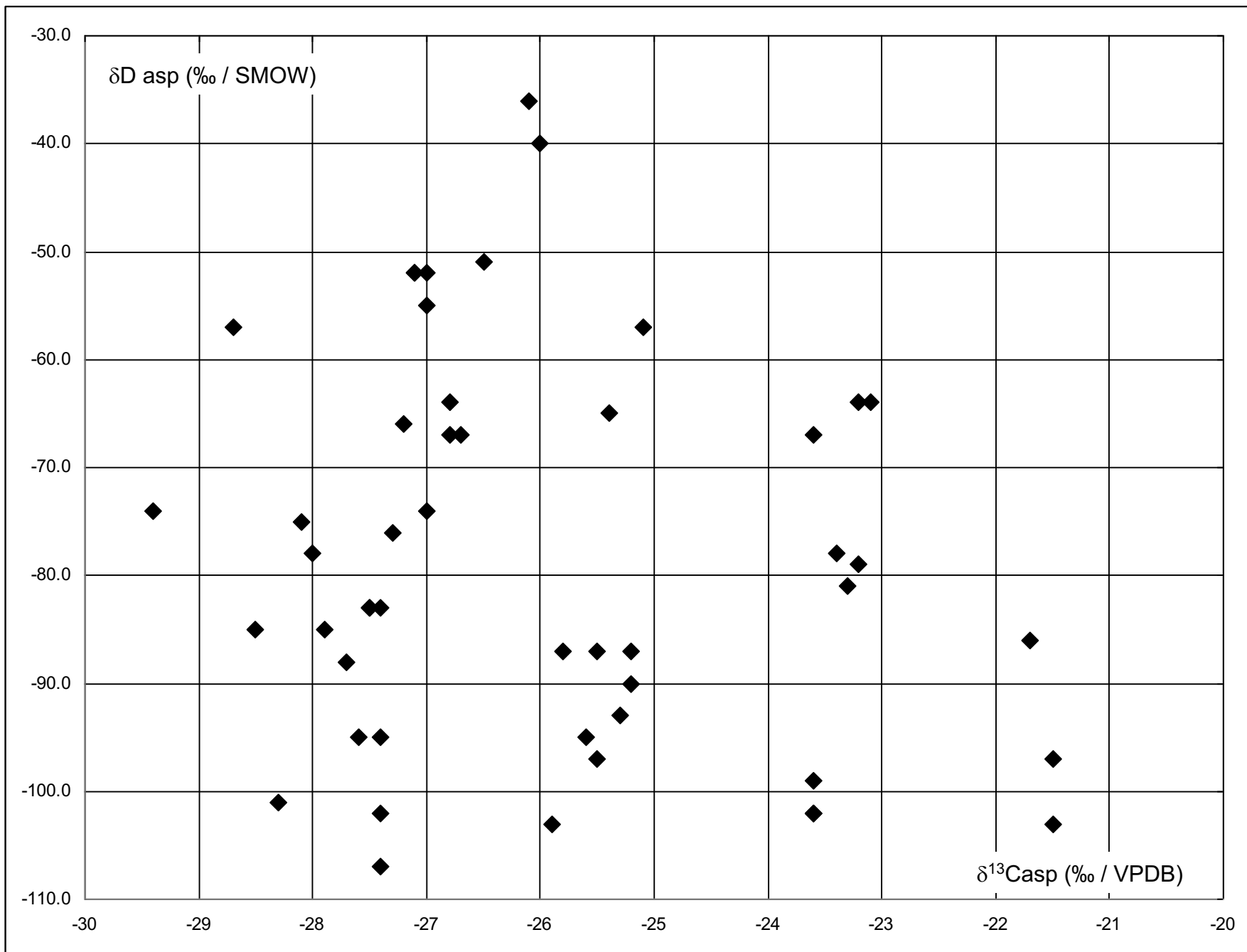


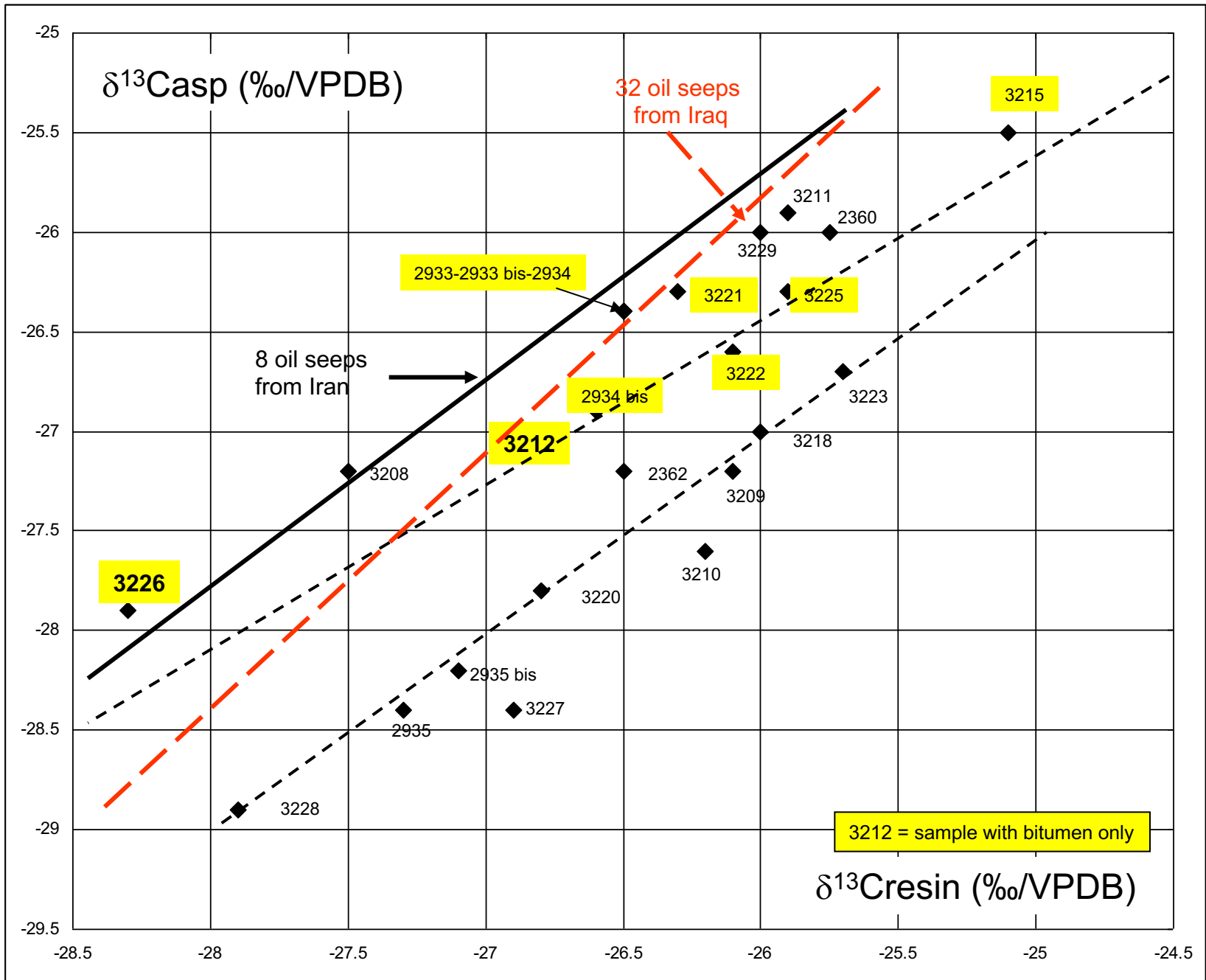


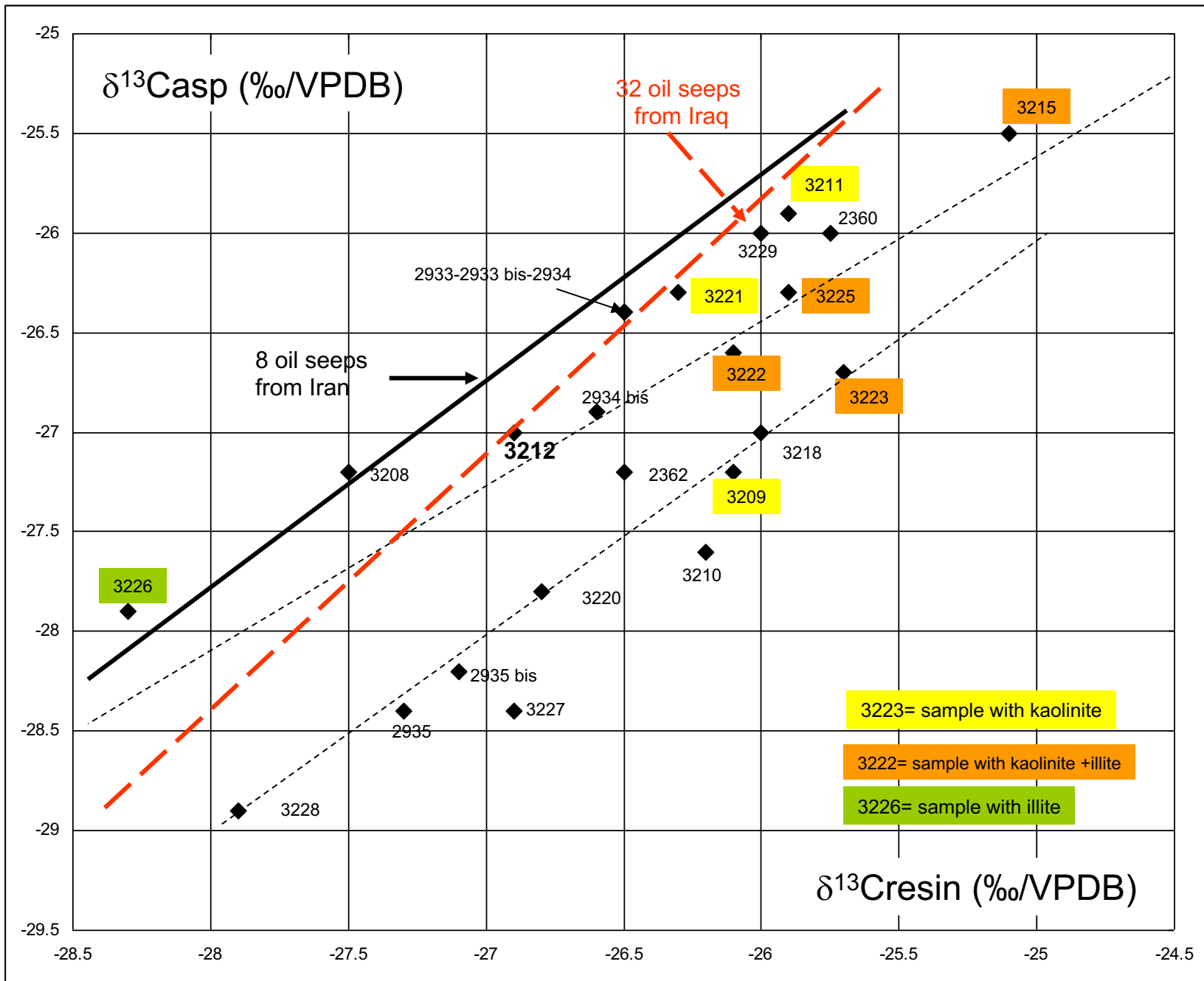


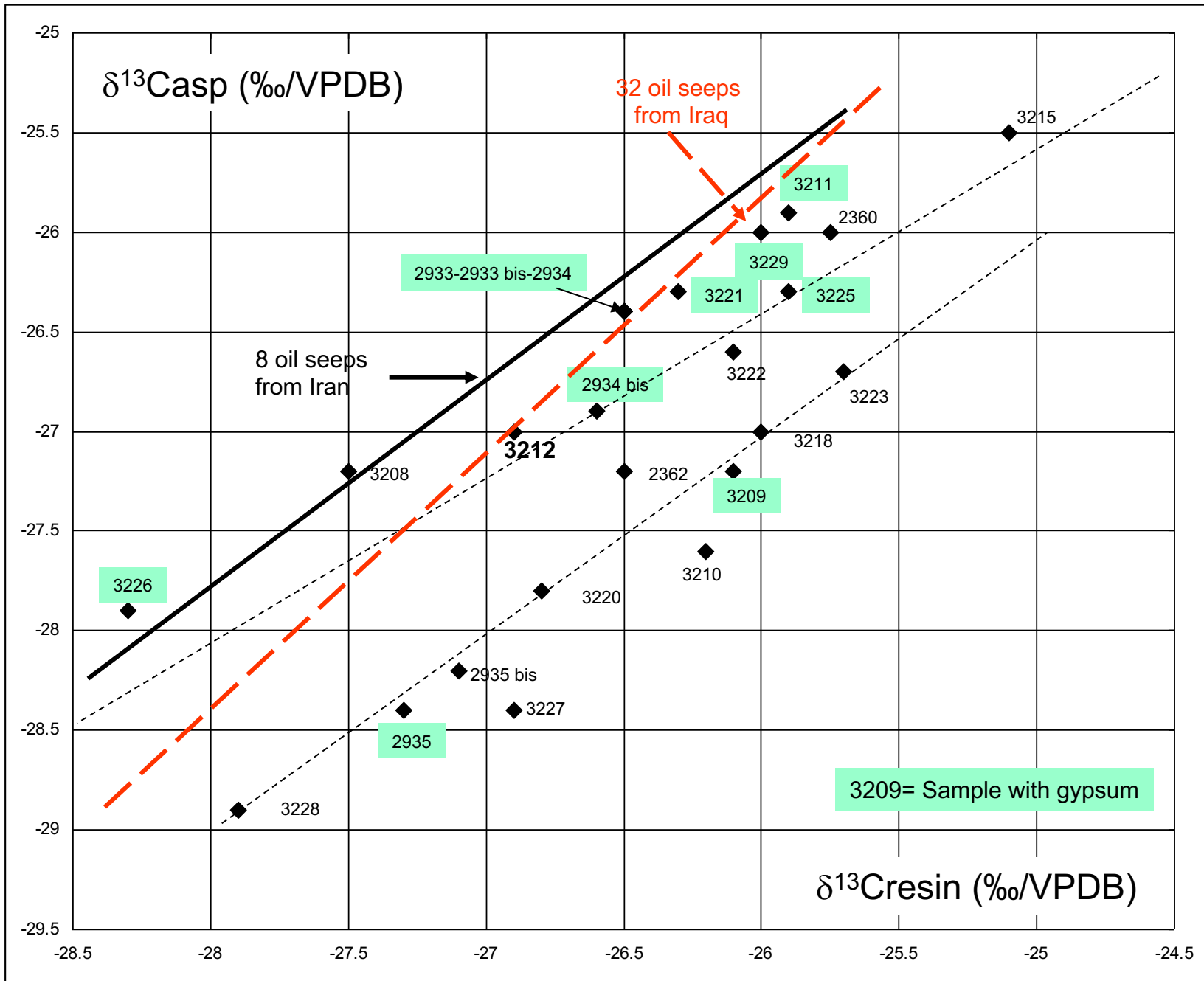






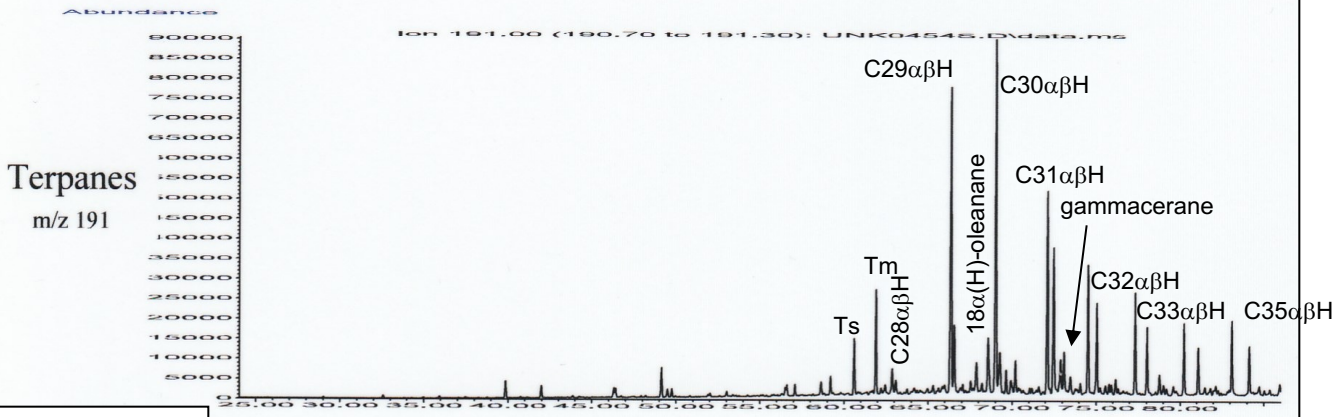




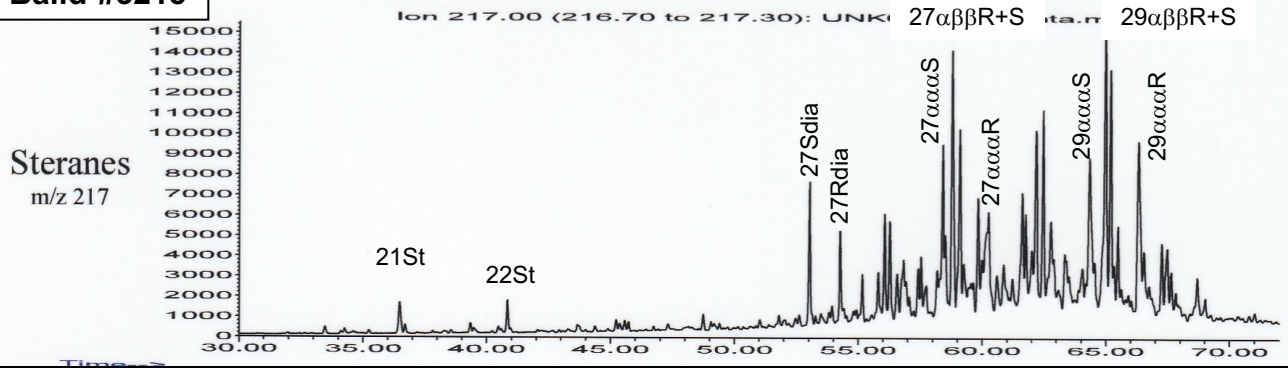


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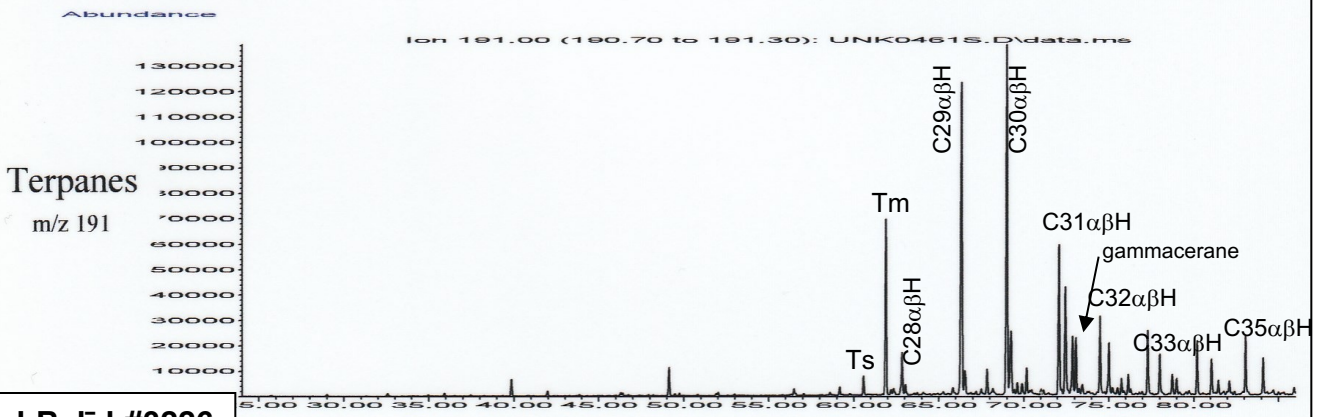


al-Balid-#3215

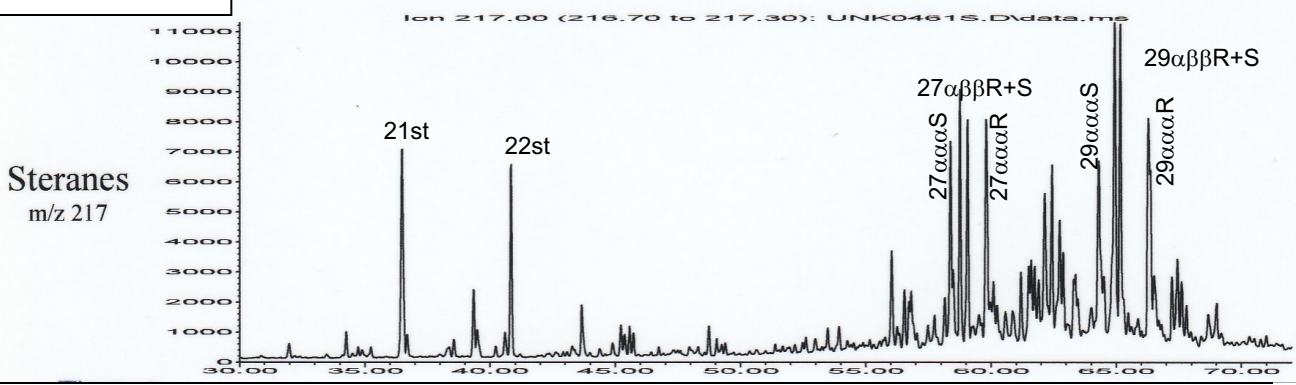


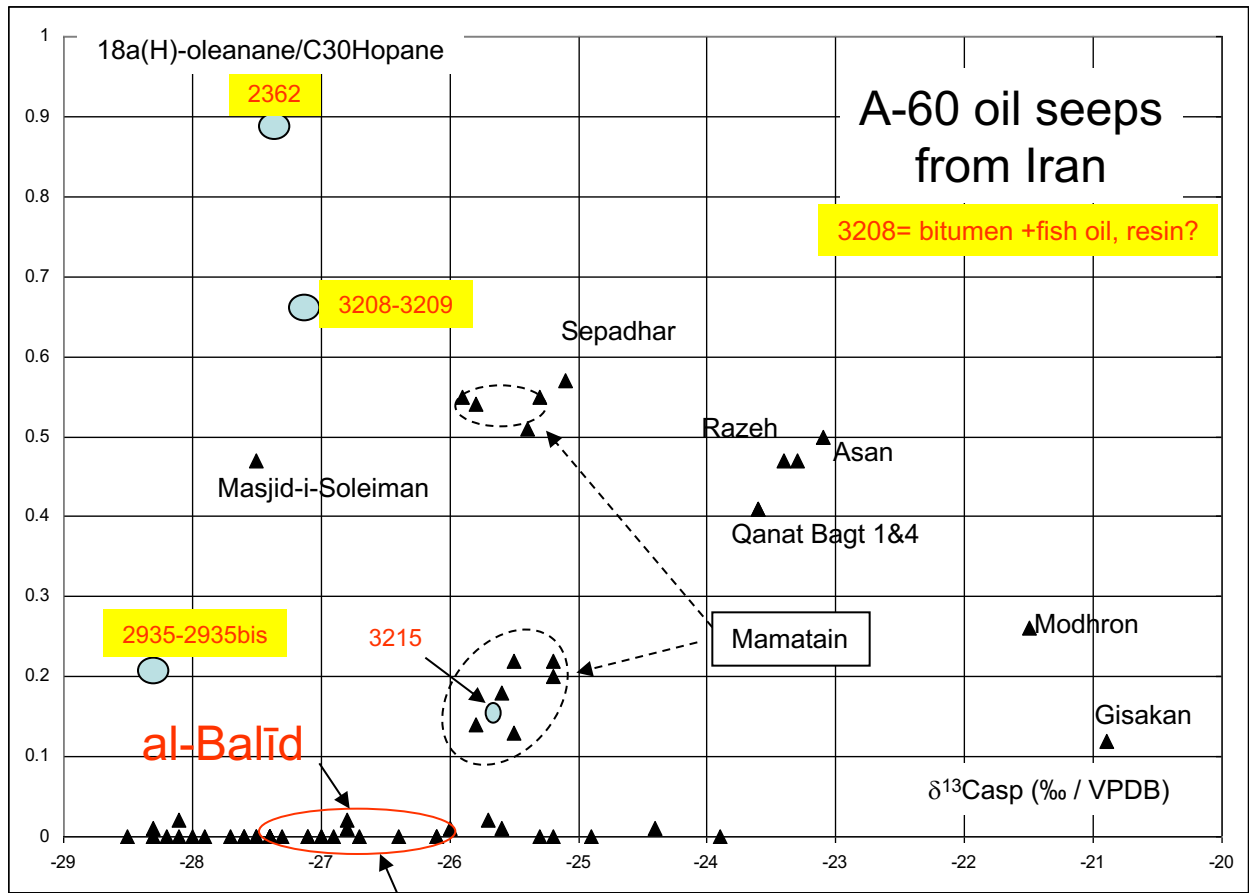
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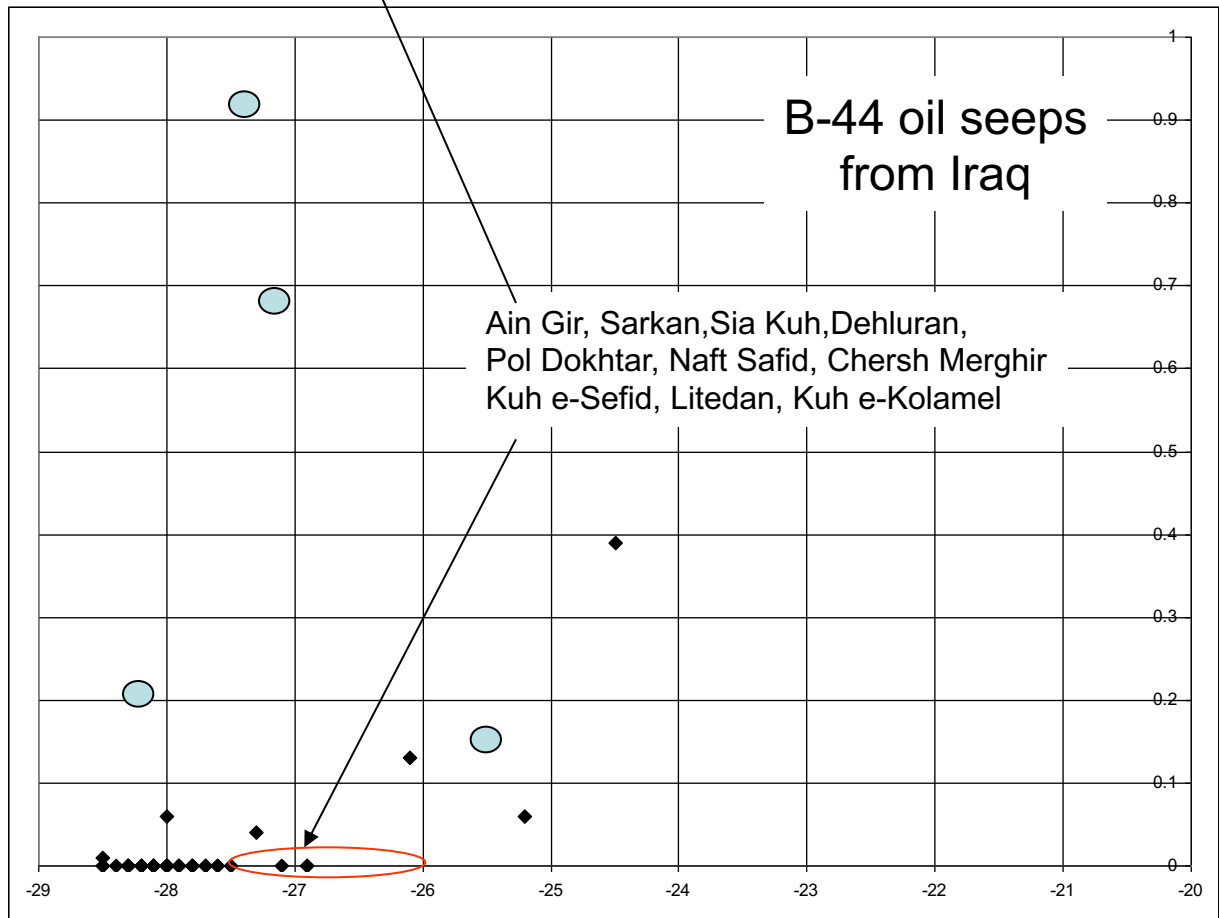


al-Balid-#3226

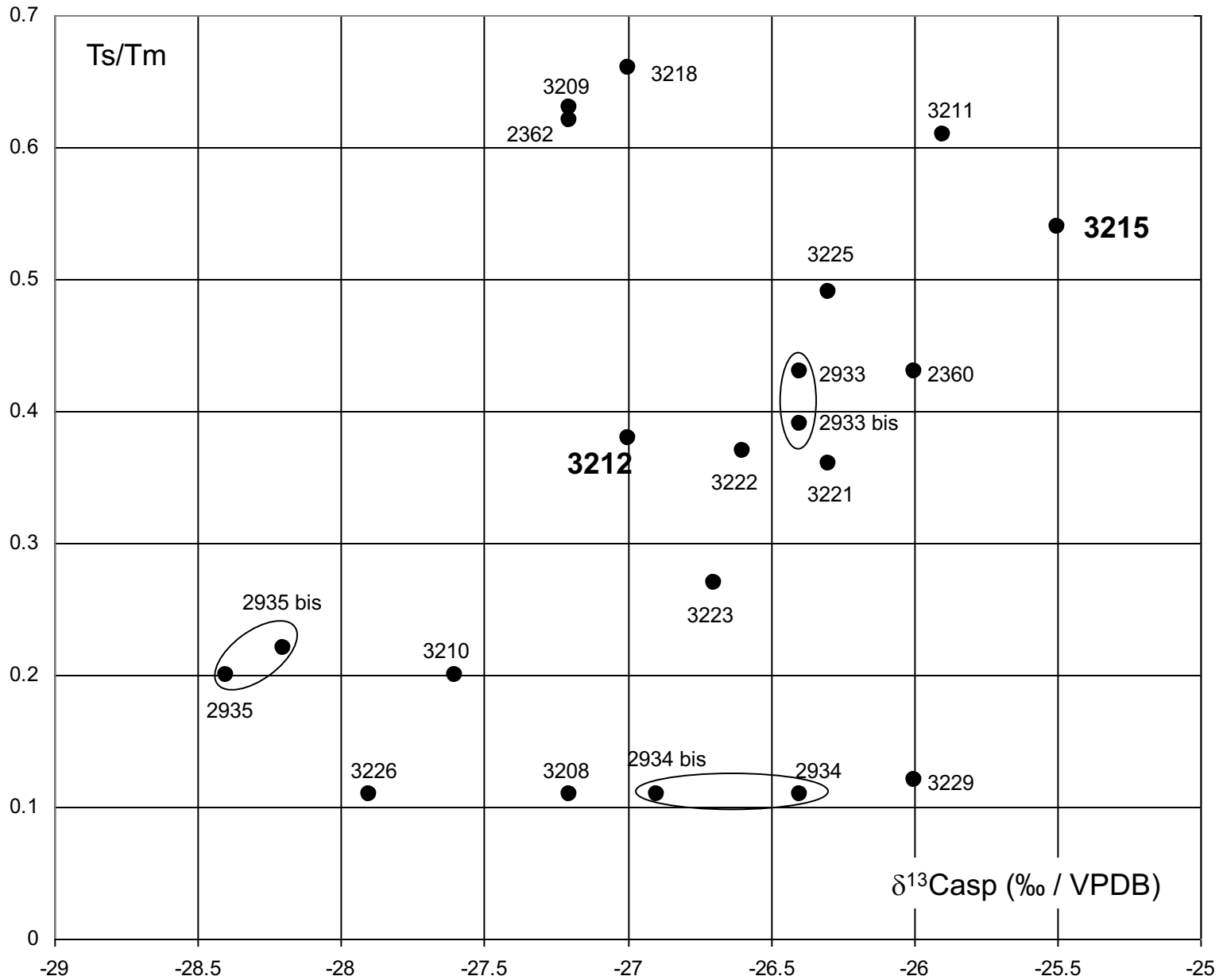


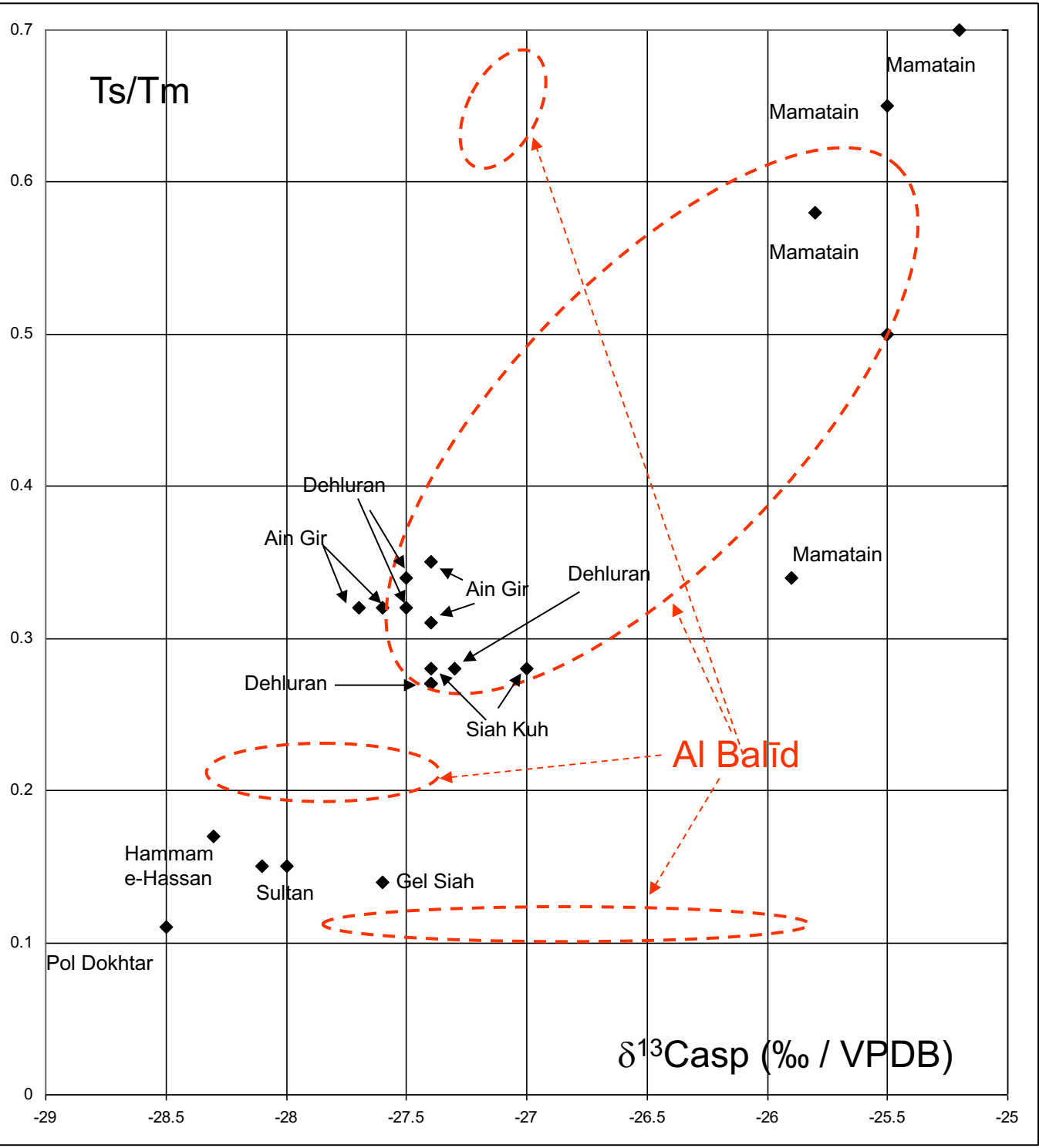


A



B





δD_{asp} (‰ / VSMOW)

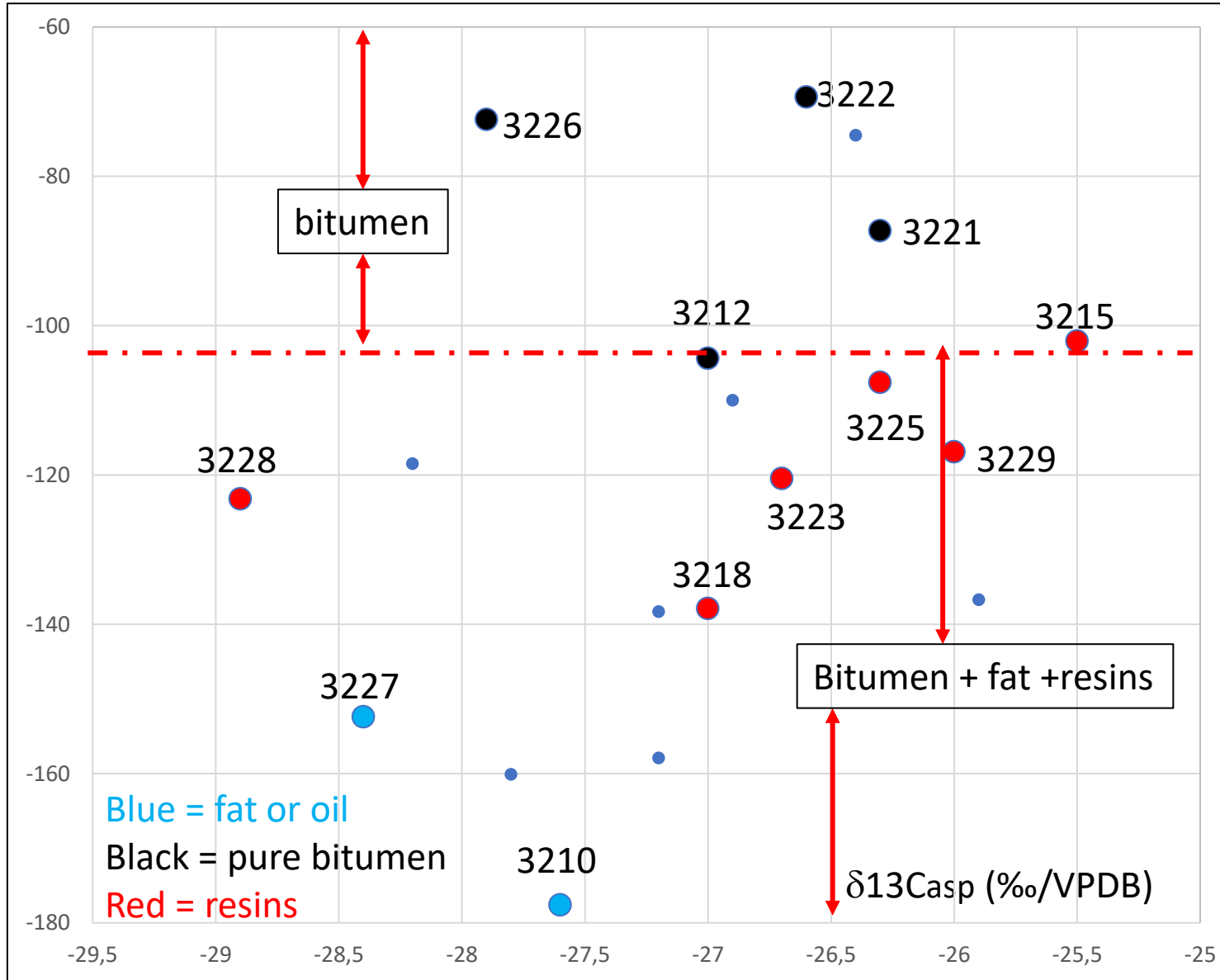


Fig.22

archaeological reference	lab number	date range	type of sample	comment
BA 0604128.74A	2360	1420-1440 CE	on plank seam	brown powder which shows black to orange lumps. Pitch according to touch and odor?
BA 0604128.74B	2361	1420-1440 CE	substance on stitching to hold the hull planks together (probably coconut fibre rope)	brown powder with numerous minerals. Microscopic aspect: yellow-orange powder. Resins?
BA 0604172.69A	2362	1000-1400 CE	coating on plank	pitch aspect
BA120621-1	2933	1320-1350 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber- 21/06/2012	bituminous mixture with very fine minerals, occurrence of wood fiber. Very hard texture
	2933 bis	1320-1350 CE		big black lump, rather homogenous, extremely hard, vegetal debris visible in the freshly broken mass, bituminous aspect
BA 1104065.454.1	2934	1020-1160 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber- 21/06/2012	very hard and black sample with fine minerals
	2934 bis			numerous small black grains with vegetal debris visible in the mass
BA 1104065.450-1	2935	1020-1170 CE	luting sample from boat plank	
	2935 bis			composite sample with vegetal fibers + bituminous mixture. Bituminous mixture selected for analysis
BA1104065.454	3208	1020-1160 CE	dark substance on the inside of the plank between the stitching and the edge	black plate with traces of wood on one side
BA0604159.263	3209	1280-1440 CE*	dark substance on the inside of the plank	black plate with big lumps of wood on one side. On the other side, smooth whitish surface in which minerals are not clearly visible
WO37	3210			numerous grains with some traces of wood. The finest grains are yellow (resin aspect?)
BA0604145.175	3211	1190-1240 CE*	well preserved plank with bitumen on the inner surface	typical bituminous mixture with numerous mineral grains clearly visible in the mass. Vegetal debris probably present
fragmentary container in iron with cylindrical shape	3212	17th century CE	solidified material inside container which appears as a pure bitumen	no residue after extraction, rare cristallized minerals
BA0301-106	3215	10-15th CE	traces of thick and dark substance on the outside of the plank around the sewing holes	black sample with traces of vegetals on the surface and in the mass
BA0604172.69	3218	1280-1440 CE*	dark substances on most of the inside plank	numerous grains which some minerals (quartz?). Some wood imprints
Wo 73	3220	10-15 th CE	substance found near the edge of the plank between and inside the stitching holes	sample with rope and imprints of wood
Wo 98B	3221	1380-1418 CE*	substance found in the inner face of the plank, near the edge and above the seam	black plate whose texture seems homogenous. If there are minerals present, they are extremely fine
Wo 98B	3222	1380-1418 CE*	thick layer on the edge of the plank	black lumps, rather homogenous. If there are minerals present, they are extremely fine . Wood imprints at surface
Wo 98A	3223	1384-1422 CE*	substance on the outer surface of the plank, between the stitching holes and the edge.	very black sample with wood debris at surface. Very fine minerals in the mass
Wo 98D	3225	14-15th CE	Substance found on the inside of the plank, near the edge	very black plate with extremely fine minerals in the mass.
Husn 17.107 X35	3226	16-17th CE	lump of bitumen discovered in the citadel (Husn) of al-Balid	very black bituminous mixture with very fine minerals
Wo 52	3227	14-15th CE	bitumen inside a dowel on the surface of the plank	Faily pure bitumen or resin because brown-yellow appearance on small pieces
BA01.11.01	3228	1044-1098 CE*	small fragments of bitumen (or resin?) in a bag next to the plank	bitumen aspect with very fine minerals. Wood imprint with wood debris in the mass.
BA0604128.74	3229	1420-1440 CE*	dark substance under the stitching on the inner surface of the plank	brown powder with some minerals (quartz) . A piece of seashell and some pieces of wood

date range	type of sample	lab number	EO (% weight/sample)	sat, (% / EO)	aro, (%/EO)	sat,+aro, (%/EO)	res, (%/EO)	asp, (%/EO)	polars = res,+asp (%/EO)
1420-1440 CE	On plank seam	2360	38.5	21.8	12.1	33.9	36.6	25.7	62.3
1000-1400 CE	coating on plank	2362	37.4	4.2	3.4	7.6	48.1	42	90.1
1320-1350 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	2933	27.1	7	6	13	15.4	71.6	87
1320-1350 CE		2933 bis	31.7	5.7	4.2	9.9	19.3	70.8	90.1
1020-1160 CE	luting sample from boat plank discovered in the Husn (Fortress) at al-Baleed-gate timber-21/06/2012	2934	24.4	1	1.6	2.6	11	86.4	97.4
		2934 bis	25.4	1.3	1.8	3.1	16.7	80.2	96.9
1020-1170 CE	luting sample from boat plank	2935	24.3	0.8	0.7	1.5	6.3	92.1	98.4
		2935 bis	57.5	1	1.6	2.6	22.8	74.6	97.4
1020-1160 CE	dark substance on the inside of the plank between the stitching and the edge	3208	14.4	0.7	0.7	1.4	20.1	78.5	98.6
1280-1440 CE*	dark substance on the inside of the plank	3209	31.8	0.3	0.6	0.9	19	80.1	99.1
		3210	35.5	0.3	0.3	0.6	11.5	88	99.5
1190-1240 CE*	well preserved plank with bitumen on the inner surface	3211	29.7	1.2	1.6	2.8	29.9	67.2	97.1
17th century CE	solidified material inside container which appears as pure bitumen	3212	68.3	11.1	14.4	25.5	22.1	52.3	74.4
10-15th c CE	traces of thick and dark substance on the outside of the plank around the sewing holes	3215	25.7	2.1	1.4	3.5	18	78.5	96.5
1280-1440 CE*	dark substances on most of the inside plank	3218	46.5	0.4	0.7	1.1	16.3	85.6	101.9
10-15th c CE	Substance found near the edge of the plank between and inside the stitching holes	3220	38.2	0.5	1.3	1.8	19	79.2	98.2
1380-1418 CE*	substance found in the inner face of the plank, near the edge and above the seam	3221	30	1	3.4	4.4	15.6	77	92.6
1380-1418 CE*	Thick layer on the edge of the plank	3222	31.2	3.5	2.6	6.1	17.8	76.1	93.9
1384-1422 CE*	Substance on the outer surface of the plank, between the stitching holes and the edge,	3223	31.3	2.2	1.8	4	20.1	75.9	96
14-15th c CE	Substance found on the inside of the plank, near the edge	3225	29	2.5	2	4.5	22.9	72.6	95.5
16-17th c CE	Lump of bitumen discovered in the citadel (Husn) of al-Balīd	3226	27.4	2.6	2.9	5.5	18.6	75.9	94.5
14-15th c CE	Bitumen inside a dowel on the surface of the plank	3227	77.4	0.1	0.1	0.2	6.1	93.7	99.8
1044-1098 CE*	Small fragments of bitumen (or resin?) in a bag next to the plank	3228	2.1	3.5	1.2	4.7	28.2	67.1	95.3
1420-1440 CE*	dark substance under the stitching on the inner surface of the plank	3229	27.6	1.1	1.4	2.5	24.4	73.1	97.5

location	date range	type of sample	lab number	$\delta^{13}\text{C}_{\text{sat}}$ (‰ / VPDB)	$\delta^{13}\text{C}_{\text{Caro}}$ (‰ / VPDB)	$\delta^{13}\text{C}_{\text{res}}$ (‰ / VPDB)	$\delta^{13}\text{C}_{\text{asp}}$ (‰ / VPDB)	$\delta\text{D}_{\text{res}}$ (‰ / SMOW)	$\delta\text{D}_{\text{asp}}$ (‰ / SMOW)
Al-Balid	1000-1400 AD	On plank seam	2360			-25.75	-26		
	1000-1400 AD	coating on plank	2362			-26.5	-27.2		
	1320-1350 AD	luting sample from boat plank	2933	-27.1	-26.2	-26.5	-26.4		
	1320-1350 AD	discovered in the Husn (Fortress)	2933 bis	-27.2	-26	-26.5	-26.4	-70.6	-74.5
	1020-1160 AD	luting sample from boat plank	2934	-28.3	-27.6	-26.5	-26.4		
		discovered in the Husn (Fortress)	2934 bis	-27.8	-27.6	-26.6	-26.9	-123.9	-110
	1020-1170 AD	luting sample from boat plank	2935	-28.7	-26.2	-27.3	-28.4		
			2935 bis	-27.3	-26.5	-27.1	-28.2	-139.4	-118.5
	1020-1160 AD	dark substance on the inside of the plank between the stitching and the edge	3208	-26.7	-27.5	-27.5	-27.2	-139.8	-138.3
	1280-1440 AD*	dark substance on the inside of the plank	3209	-26.8	-26.1	-26.1	-27.2	-165.8	-157.9
			3210	-26.6	-26.4	-26.2	-27.6	-194.9	-177.6
	1190-1240 AD*	well preserved plank with bitumen on the inner surface	3211	-26.2	-25.6	-25.9	-25.9	-137.8	-136.7
	17 th century AD	solidified material inside container whic appears as pure bitumen	3212	-27.3	-26.9	-26.9	-27	-109	-104.4
	10-15th AD	traces of thick and dark substance on the outside of the plank around the sewing holes	3215	-27	-26	-25.1	-25.5	-134.8	-102.1
	1280-1440 AD*	dark substanceson most of the inside plank	3218	-26.7	-25.9	-26	-27	-159.3	-137.9
	10-15 th AD	Substance found near the edge of the plank between and inside the stitching holes	3220		-26.7	-26.8	-27.8	-167.7	-160.1
	1380-1418 AD*	substance found in the inner face of the plank, near the edge and above the seam	3221	-27.2	-26	-26.3	-26.3	-78.4	-87.3
	1380-1418 AD*	Thick layer on the edge of the plank	3222	-27.2	-26.1	-26.1	-26.6	-77.9	-69.4
	1384-1422 AD*	Substance on the outer surface of the plank, between the stitching holes and the edge.	3223	-27.4	-26.3	-25.7	-26.7	-120.4	-120.5
	14-15th AD	Substane found on the inside of the plank, near the edge	3225	-27	-25.7	-25.9	-26.3	-103.8	-107.6
	16-17th AD	Lump of bitumen discovered in the citadel (Husn) of al-Balid	3226	-28.4	-28.1	-28.3	-27.9	-72.2	-72.4
14-15th AD	Bitumen inside a dowel on the surface of the plank	3227		-26.9	-26.9	-28.4	-141.7	-152.4	
1044-1098 AD*	Small fragments of bitumen (or resin?) in a bag next to the plank	3228		-27.7	-27.9	-28.9	-117.7	-123.2	
1420-1440 AD*	dark substance under the stitching on the inner surface of the plank	3229	-27.8	-26.9	-26	-26	-124	-116.9	

n° échantillon	site	date range	Quartz	Calcite CaCO3	Dolomite CaMg(CO3)2	Albite NaAlSi3O8	Gypsum (CaSO4, 2H2O)	Anhydrite (CaSO4)	Bassanite (CaSO4 0.5H2O)	Muscovite- 2M#1	Kaolinite	Illite	Clinoenstatite MgSiO3	Microcline KAlSi3O8	Halloysite Al2O3.2SiO2 .2H2O	Talc Mg3Si4O10(O H)2	Hematite (Fe2O3)	Siderite (FeCO3)	Halite NaCl	
3208	Al-Baleed	1020-1160 AD	xxxx	xx	xx	x				x										
3209		1280-1440 AD	xxxx	xx		x	x			x	x						xx			
3211		1160-1260 AD	xxx	xxxx		x	x		x	x	x						x			
2934bis		1020-1160 AD	xxxx	xx	x	x	x	x		x										
2935		1020-1170 AD	xxxx	xxx	x	x	x	x		x										
3228		1028-1184 AD	xx	xxxx																
2933bis		1320-1350 AD	xx	xxxx	x	x	x	x		x										
3218		1260-1400 AD	xxxx	xx	x	x				x				x	x		x			
3221		1300-1369 AD	xx	xxxx		x	x	x				x				x				
3222		1300-1369 AD	xxx	xxxx	x	x						x	x	x	x				x	
3223		1304-1364 AD	xxxx	xxxx	xx	x					x	x	x	x	x		x			
3215		10-15th AD	xxx	xxxx	xx	x					x	x	x	x	x		x			
3225		14-15th AD	xx	xxxx	x	x	x					x	x		x					
3226		16-17th AD	xxxx	xxxx	x	x	x				x		x		x					
3229		1400-1460 AD	xx	xxx		x	x		x		x				x					
3232		Al Baleed-Chunam	14-15th AD	x	xxxx		x	x												x

 calcite
  magnesian calcite (Mg.064Ca.936)(CO3)

Lab number	C30hopane ppm	Ts/Tm	Olean/Hop	G/C31HR	ster/terp	Rear/Reg	C27βR+S	C28βR+S	C29βR+S	C29βS/C29αR	steranes	terpanes
2360		0.43	0	0.32							perserved	preserved
2362		0.62	0.91	0.25							altered	preserved
2933	116	0.43	0.03	0.44	0.16	0.17	26.7	33.4	39.9		slightly altered?	preserved
2933 bis	93	0.39	0.02	0.52	0.22	0.29	27.8	31.4	40.8	0.98	slightly altered	well preserved
2934	760	0.11	0.01	0.56	0.11	0.12	24.7	25.5	49.8		preserved	preserved
2934 bis	439	0.11	0.01	0.55	0.14	0.12	25.1	24.5	50.4	1.77	preserved	preserved
2936	116	0.33	0.02	0.51	0.09	0.08	16.7	35.1	48.3		slightly altered	preserved
2935 bis	124	0.22	0.2	0.43	0.32	0.15	14	9.8	76.3		altered	slightly altered?
3208	681	0.11	0.01	0.53	0.13	0.13	24	23.4	52.6	1.62	preserved-siighly altered	preserved
3209	242	0.63	0.65	0.22	0.55	0.31	22.5	22.6	54.9	0.66	altered	slightly altered?
3210	5	0.2		0.06	8.16	0.04	4.7	17.9	77.4	0.26	severely altered	severely altered
3211	116	0.61		0.2	0.42	0.63	29.9	27.5	42.6	30.2	altered	altered
3212	48	0.38	0.01	0.18	0.24	0.26	30.3	24.6	45.1	1.19	preserved	preserved
3215	256	0.54	0.15	0.22	0.35	1.27	28.6	31.2	40.2	1.36	well preserved	well preserved
3218	175	0.66	0.66	0.22	0.54	0.32	23.3	23.7	52.9	0.75	altered	partially altered
3220	15	2.48		0.14	3	0.04	4	14.8	81.2	0.31	severely altered	severely altered
3221	255	0.36	0.01	0.56	0.19	0.27	27.6	32.6	39.8	0.95	partly altered	preserved
3222	183	0.37	0.01	0.54	0.19	0.25	27.5	29.4	43.2	1.11	slightly altered	preserved
3223	284	0.27	0.02	0.49	0.2	0.21	29.5	26.5	44	1.32	slightly altered	preserved
3225	141	0.49	0.07	0.46	0.21	0.4	23.1	31.3	45.6	1.29	slighly altered	preserved
3226	815	0.11	0	0.53	0.18	0.06	31.5	23.6	44.9	1.43	preserved	preserved
3227	2		1.13	0.77	4.8	0.1	6.8	12.6	80.6	0.13	severely altered	severely altered
3228	22		0.18	0.4	0.3	0.77	19	13.8	67.2	0.71	severely altered	severely altered
3229	694	0.12	0.03	0.52	0.17	0.09	28.3	21.9	49.8	1.3	preserved	preserved