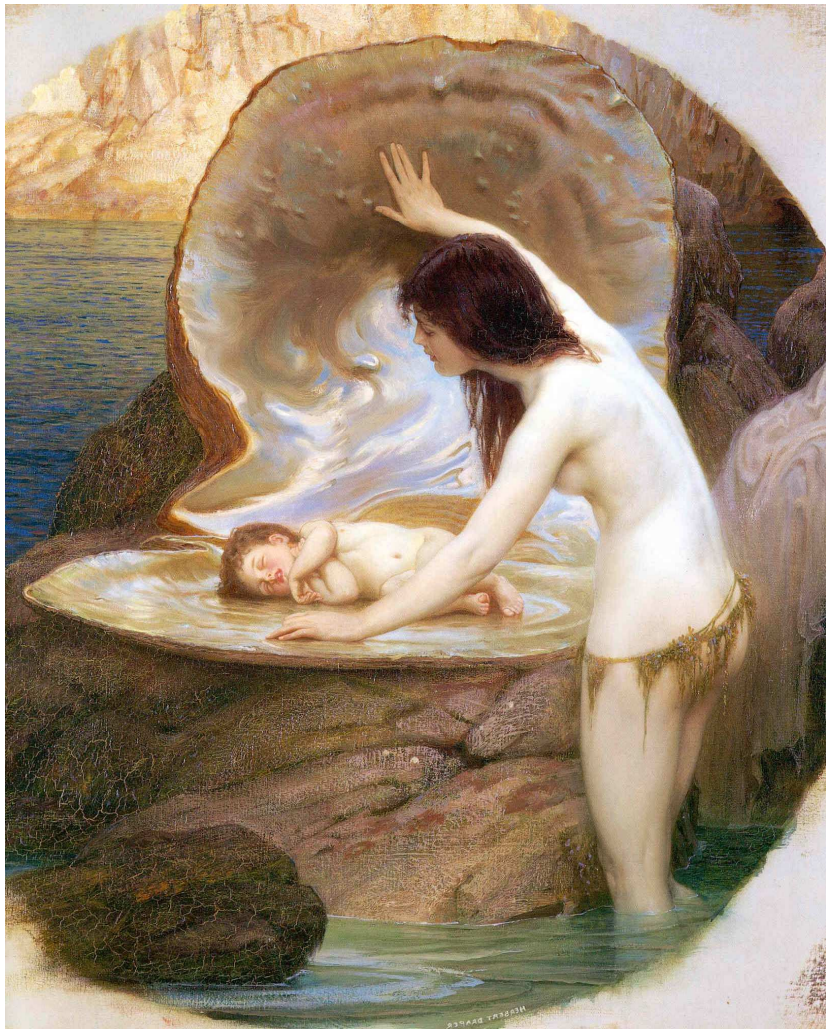


Pearls

Stefanos Karampelas

Introduction

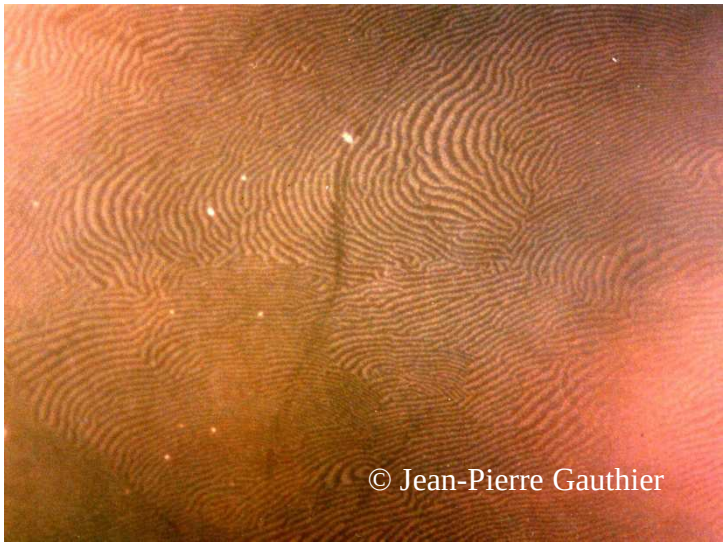
Pearls are some of the best examples of gems formed directly through biological processes, *i.e.* biomineralization.

Virtually all molluscs with shell made of calcium carbonate are able to produce pearls (bivalves, gastropods and cephalopods). However, not all pearls are attractive enough to be used in jewellery.

Nacreous vs non-nacreous

Pearls' internal structures architecture gives rise to desirable optical effects, such as iridescence, flames etc.

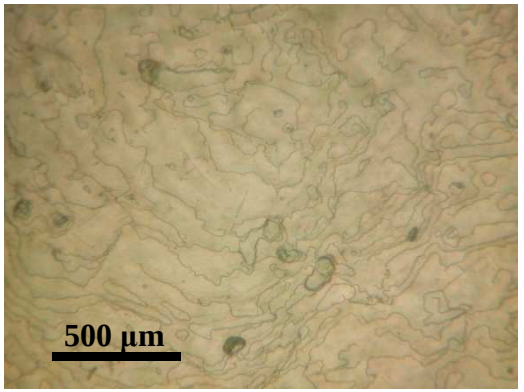
Two categories: nacreous and non-nacreous

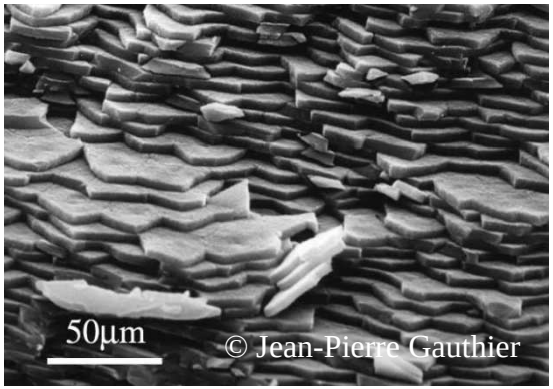


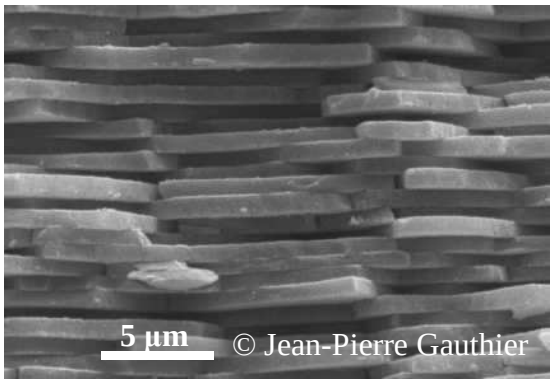
Nacreous pearls

Nacreous pearls are composed by alternating, concentric layers of **aragonite** (sometimes with small regions made of calcite or vaterite) and organic matter.

Each layer is a polygonal paving of aragonite tiles, about 3 to 5 micron across, and 0.4 to 1 µm thick.



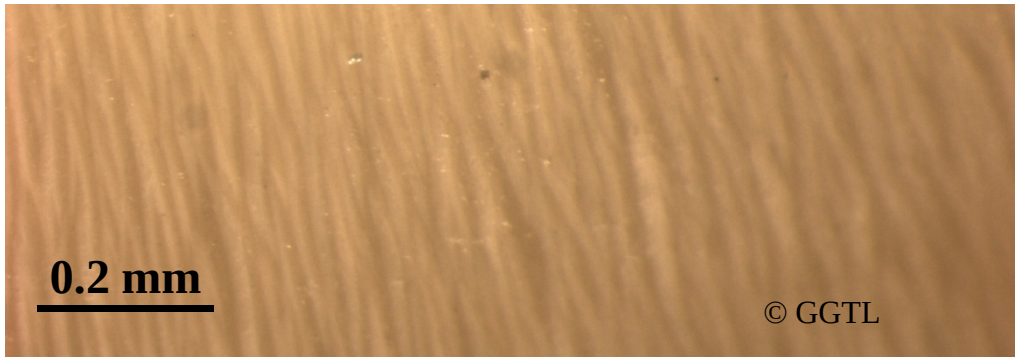




Non-nacreous pearls

The vast majority of non-nacreous pearls used in jewellery are also mainly made of aragonite.

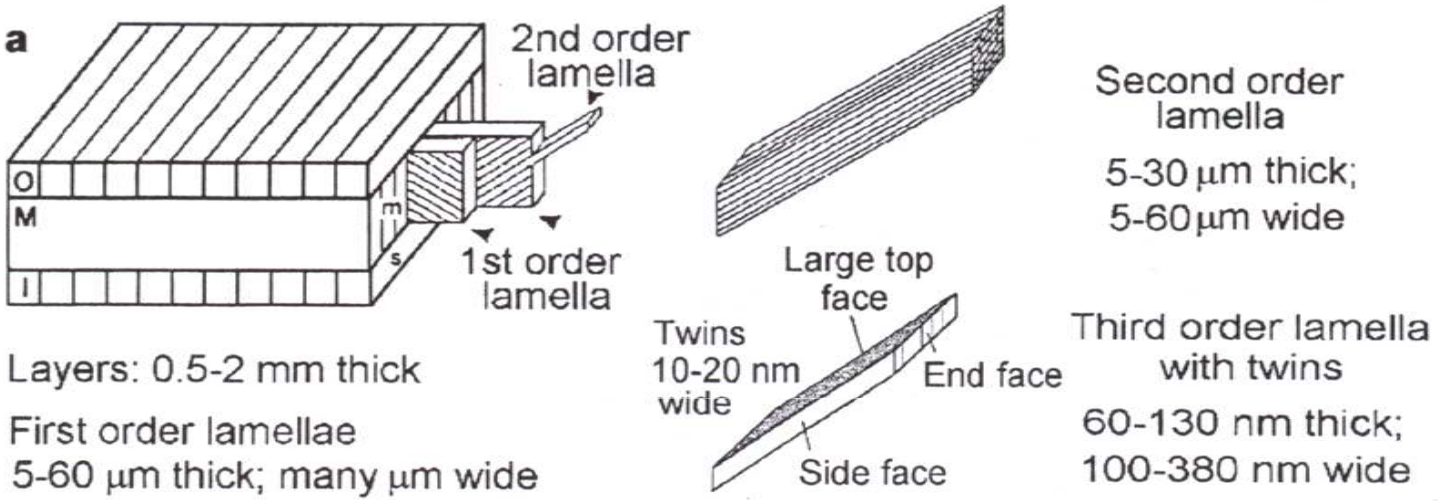
The most known is the flame structure.



Non-nacreous pearls

Flame structure

Kamat et al. 2000, *Nature*, Vol. 405, pp. 1036-1040.



It is due to the first, second and third order of fibrous aragonite.

Natural vs Cultured pearls

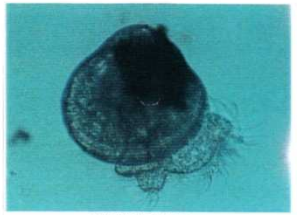
Natural pearls (NPs) are secreted accidentally by mollusks without human intervention.

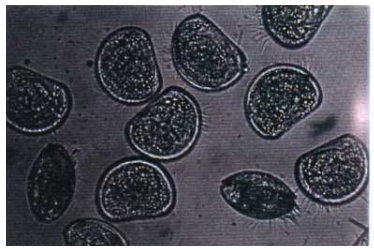
Cultured pearls (CPs) are produced after transplantation by man of a tissue (with or without the implantation of a bead).

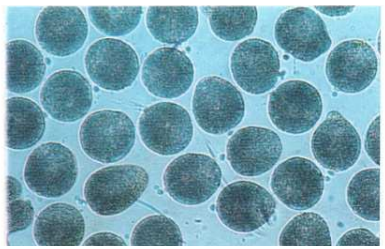
Today in the market: **99.8% cultured pearls** and **0.2% natural pearls**. **More than 99.9%** are found **into bivalve molluscs**. FW or SW.

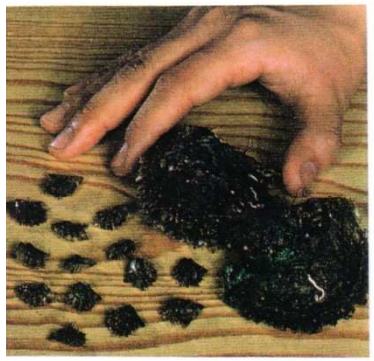
Cultured Pearls

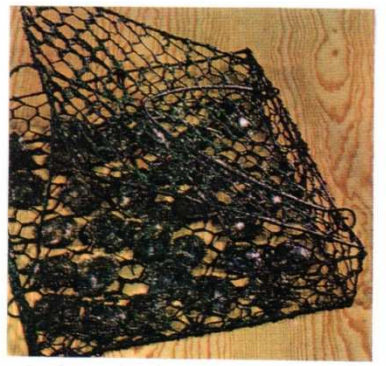
First step: Collecting spats
Between 12 and 24 months to do animals
from 5 to 10 cm.







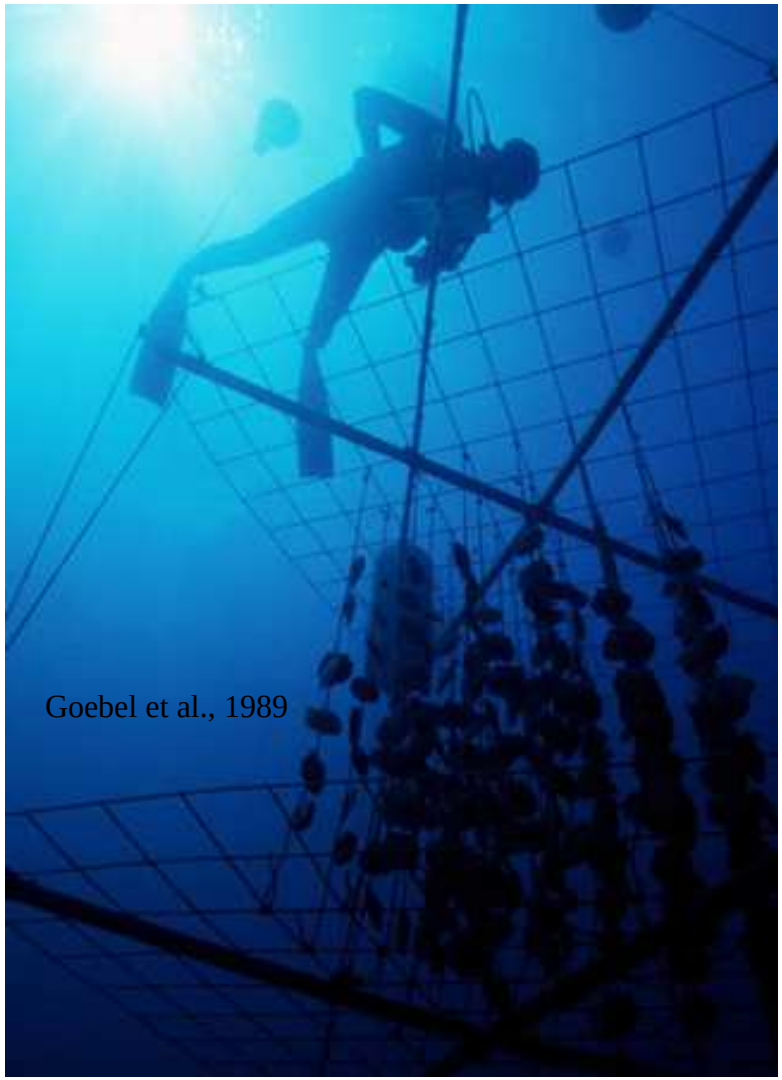




Sometimes → wild mollusc
or from hatcheries (GM to obtain more homogenous samples)

Cultured Pearls

The shell is sometimes drilled to put the at ropes during 3 to 12 months.



Goebel et al., 1989

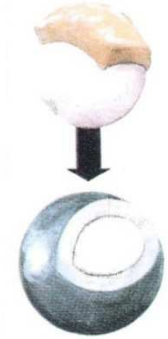
Cultured Pearls

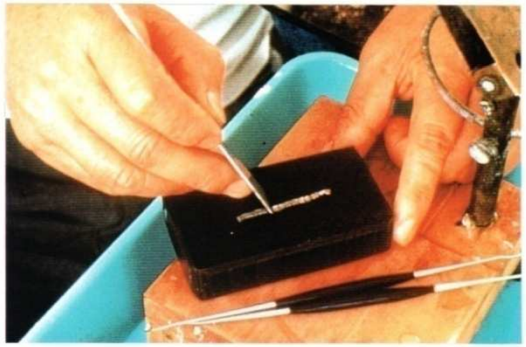
Second step:

grafting

The receivers are grafted when 2 years old.

Goebel et al., 1989





Cultured Pearls

Without bead

With bead

Weight: grams, carats, momme (3.75 grams)

SWCPs

Most **SWCPs** are **with bead**; a bead with a piece of tissue are transplanted into the gonad of the acceptor mollusc.

SWCPs

Mikimoto

The very first successful cultivation ("blister" pearls) were took place around 14th century in China.

The first successful experiments to cultivate round pearls took place at the end of 19th century in Mexico.

Patent for big scale "round" pearl cultivation around 1920 (Mikimoto).
Up to 1960s all the cultured pearls were "Akoya".

SWCPs

Akoya SWCPs: cultivated into *Pinctada fucata*, mainly in **Japan** and **China**, as well as Vietnam and Korea.

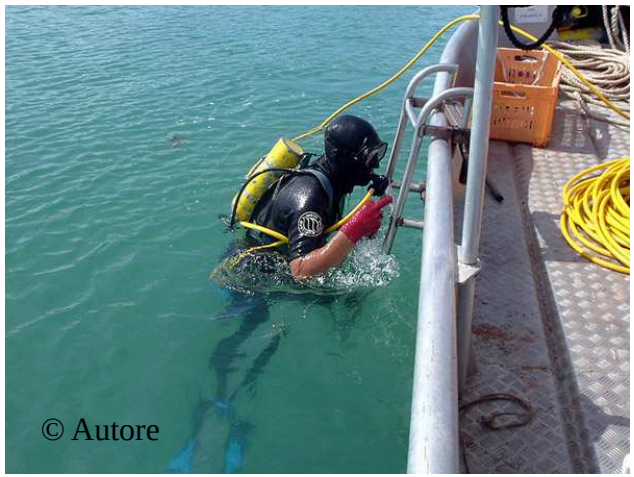
There are mainly beaded (some non-beaded SWCPs are also found accidentally) and yield from **7 to 9 mm** and **rarely up to 12 mm**.

There are mainly white, cream and rarely yellow and bluish. At **2010**, about **20 tons** of **top quality** reached the market.

© PearlParadise

SWCPs

“South sea” SWCPs: cultivated into *Pinctada maxima*, mainly in **Australia** and **Indonesia** as well as Philippines and Burma.



SWCPs

There are mainly beaded (some non-beaded SWCPs are also found accidentally) and yield from **10 to 14 mm** and **rarely up to 20 mm**.

There are mainly white, cream and yellow, rarely pink. At **2010**, about **15 tons** of **top quality** reached the market.

© Autore

SWCPs

“Tahitian” SWCPs: cultivated into *Pinctada margaritifera*, mainly in **French Polynesia** as well as in Cook Islands and Fiji.


Caseiro, 1993

There are mainly beaded (some non-beaded SWCPs are also found accidentally) and yield from **9 to 12 mm** and **rarely up to 16 mm**.

SWCPs

There are most commonly black to light grey as well as yellow to green, brown, gray-red, gray-blue, and grey-green. They can also contain strong overtones (i.e., secondary colors), including pink and purple.

At **2010**, about **15 tons** of **top quality** reached the market.


Elen, 2003


© Autore

SWCPs

“Mexican” SWCPs: cultivated into *Pteria sterna*, in **Mexico** (probably in Indonesia too).

Small scale cultivation

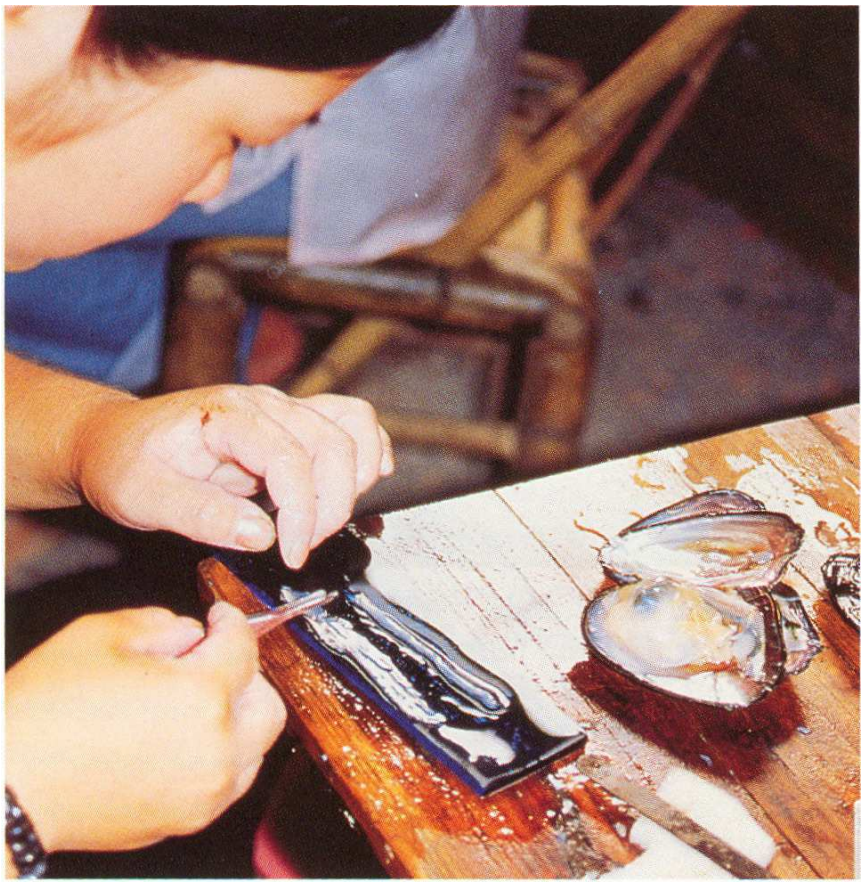
There are mainly beaded (some non-beaded SWCPs are also found accidentally) and yield from **7 to 10 mm** and **rarely up to 14 mm**.

SWCPs

Similar colors with the “Tahitian” SWCPs.

At **2010**, less than **3 kilos** of **top quality** reached the market.

FWCPs



Akamatsu et al., 2001

Most **FWCPs** are **without bead**.

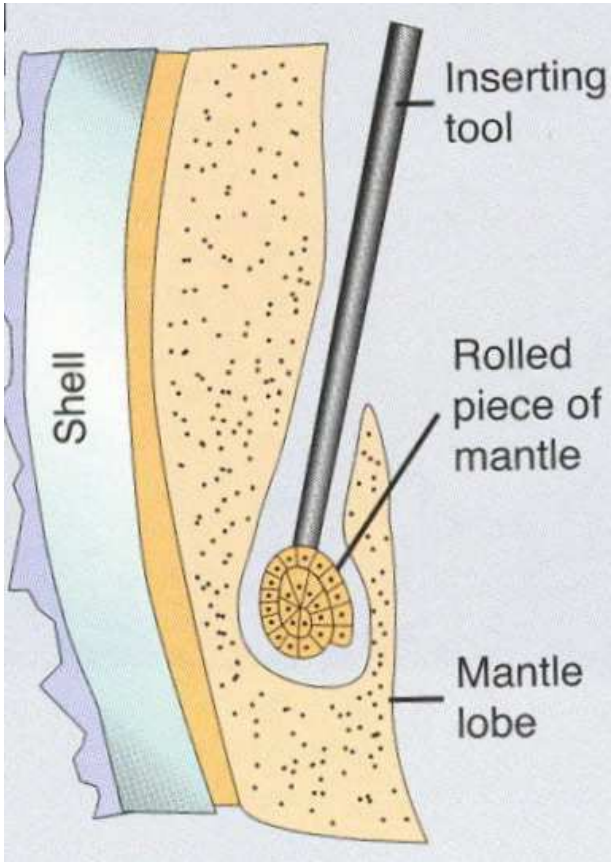
Slices of the (epithelial cells) tissue (sized: 4x4 mm) of the donor to transplanted to the acceptor (into the mantle).

FWCPs

Akamatsu et al., 2001

Spheres of 2 mm

FWCPs



Akamatsu et al., 2001

Tissue transplantation into (mantle) of the acceptor mollusk

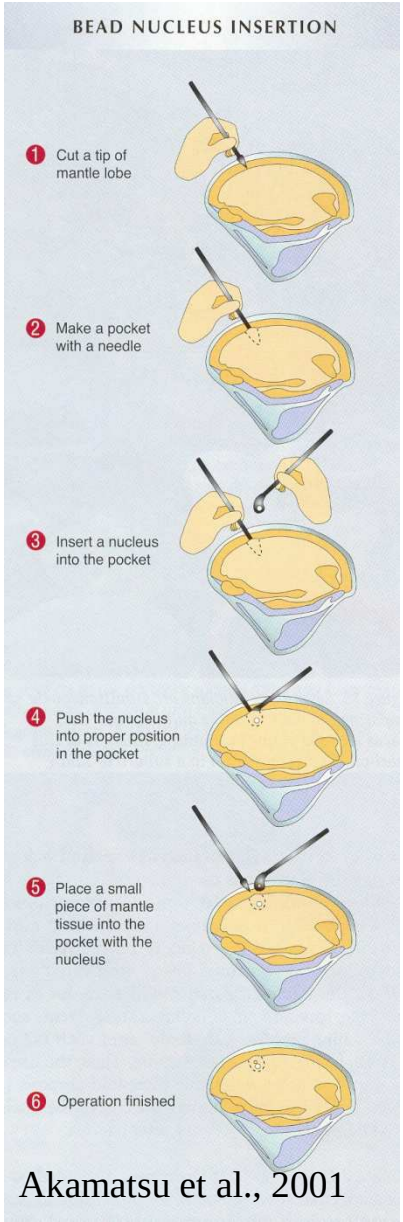
FWCPs

Akamatsu et al., 2001

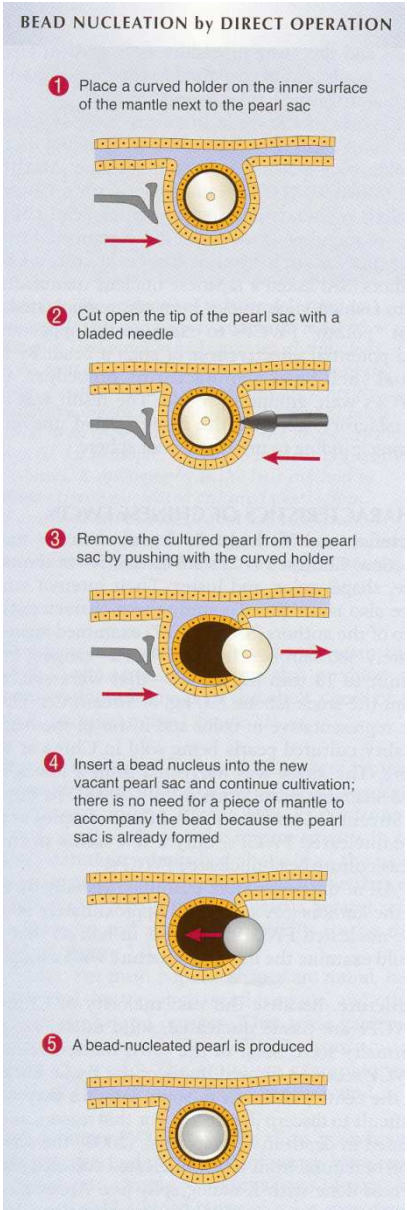
Up to 40 grafts
Lifetime up to 30y

Every 6 months they change location.
4 years for 7-8 mm

FWCPs



Akamatsu et al., 2001

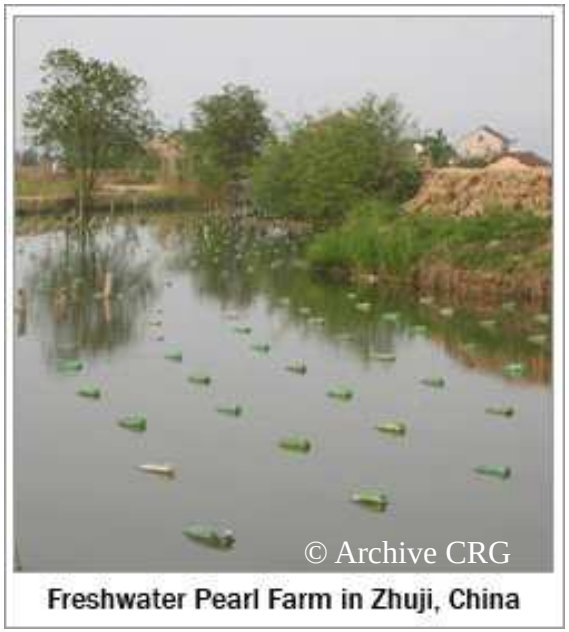


FWCPs

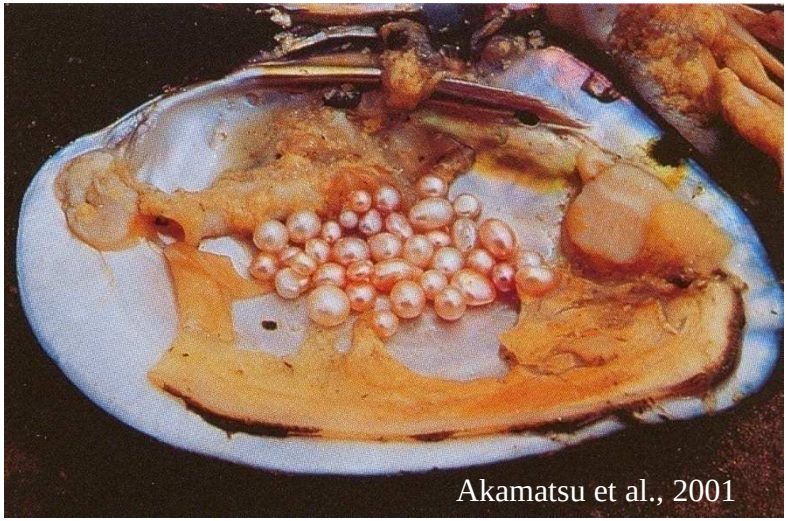
Japon, Biwa Lake
From 1960
H. schlegeli

FWCPs

Now *Hyriopsis sp.* is used



Freshwater Pearl Farm in Zhuji, China

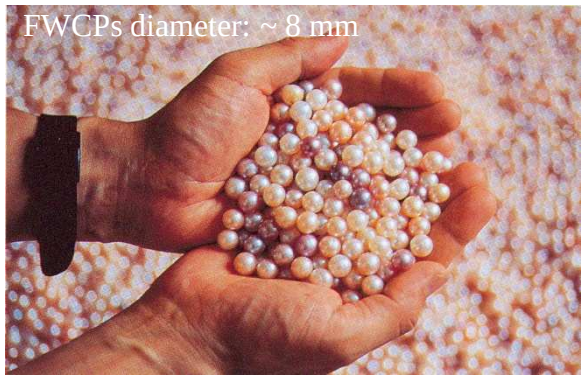


Change of the transplantation process

FWCPs

96% of the pearls found in the market are freshwater cultured pearls (**FWCPs**).

At 2006, 1500 tons are cultured in China, where 800 tons are suitable for jewellery (i.e., **~ 95%** of pearls found in the market) and about **75 tons of superior quality**.



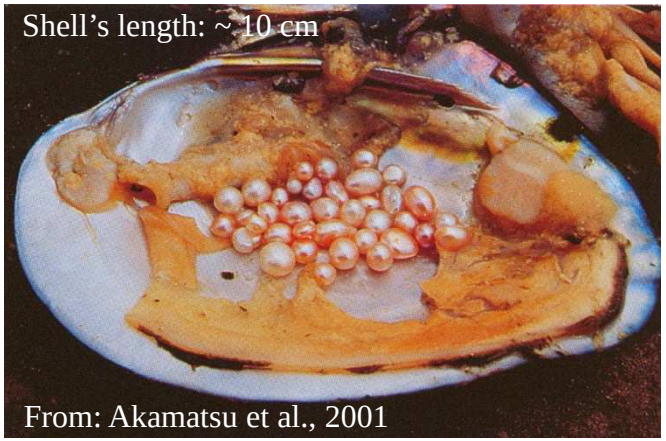
Akamatsu et al., 2001

The vast majority of FWCPs are cultivated after solely tissue graft transplantation.


© Frieden

FWCPs

The main mollusks used today for FWCPs cultivation are: *Hyriopsis cumingi*, *H. schlegeli* (Biwa cultured pearls), *H. schlegeli* x *cumingi* (Kasumiga cultured pearls). There cultured mainly in China but in Japan and Vietnam as well.



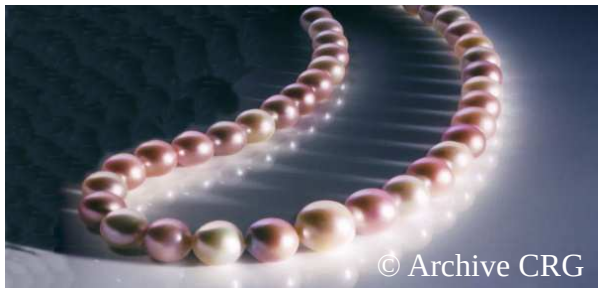
Hyriopsis cumingi

They yield from 6 to 8 mm and rarely up to 12 mm.

FWCPs

Recently, cross-breeds are used to improve the quality, colour and size of the FWCPs.

They can yield even more than 16 mm (round or near-round) of high quality. Various beads are also used. At **2010**, only **30 tons** of **top-quality** pearls reached the market.



FWCPs

Decoration,
medicine (20%)

FWCPs

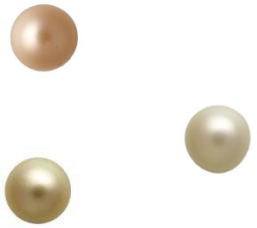
High quality FWCPs from fin 2010

With bead, up to 20 mm round

More than 1000 USD/pearl

A.k.a. "Ming" or "Edison" FWCPs

Small scale cultivation

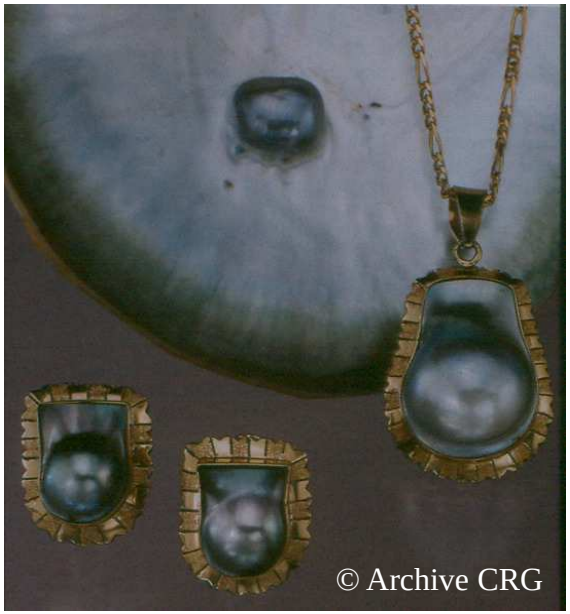


SWCPs are cultivated into *Pinctada radiata*, in **UAE**.

Exact numbers of cultured pearls are not yet known.

Small scale cultivation

SWCPs are cultivated into *Pinctada mazatlanica*, in **Mexico**.



Small scale cultivation

Haliotis sp.

Non-nacreous

Exact numbers of cultured pearls are not yet known.

Small scale cultivation

Strombus gigas

Natural pearls (nacreous)

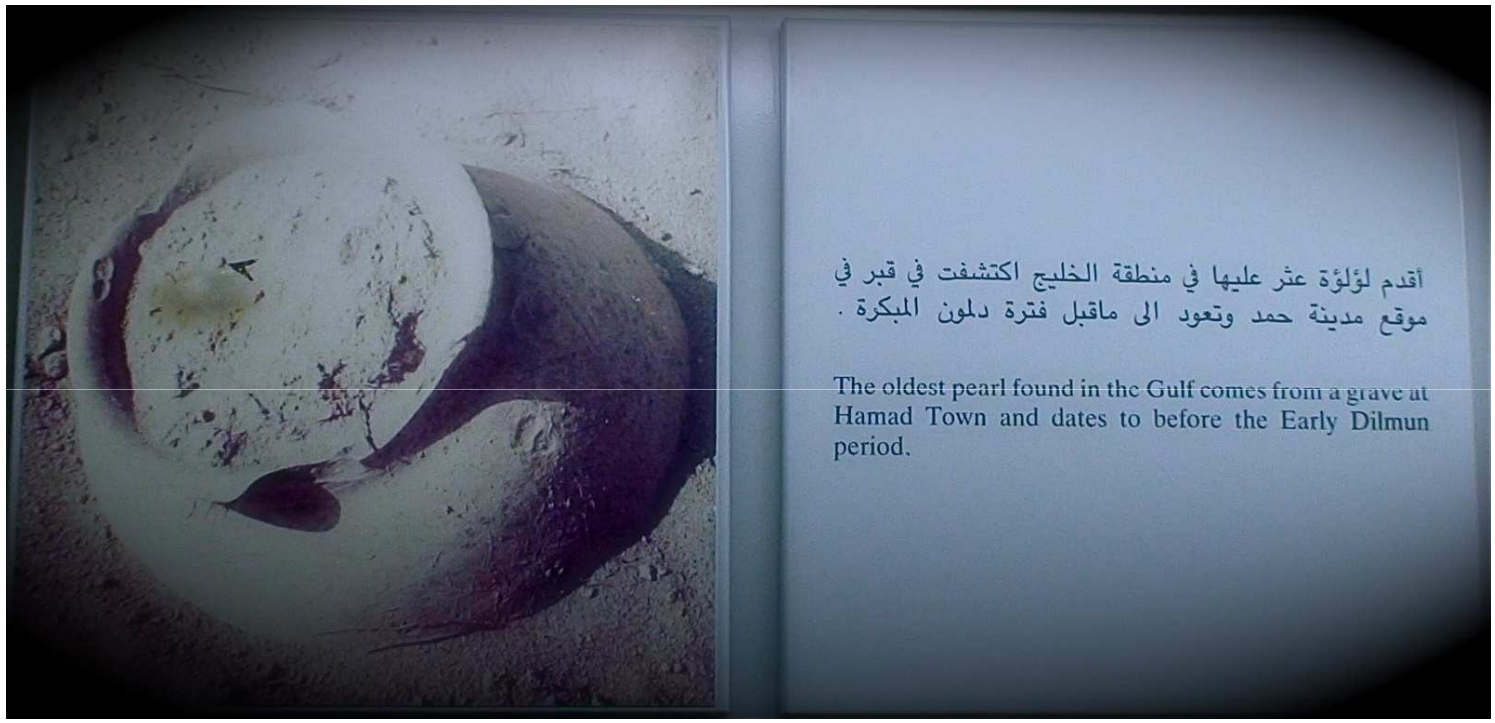
SWNP
P. maxima

SWNP
P. margaritifera
and *Pteria spp.*

FWNP

SWNP
P. radiata.

Natural pearls (nacreous)



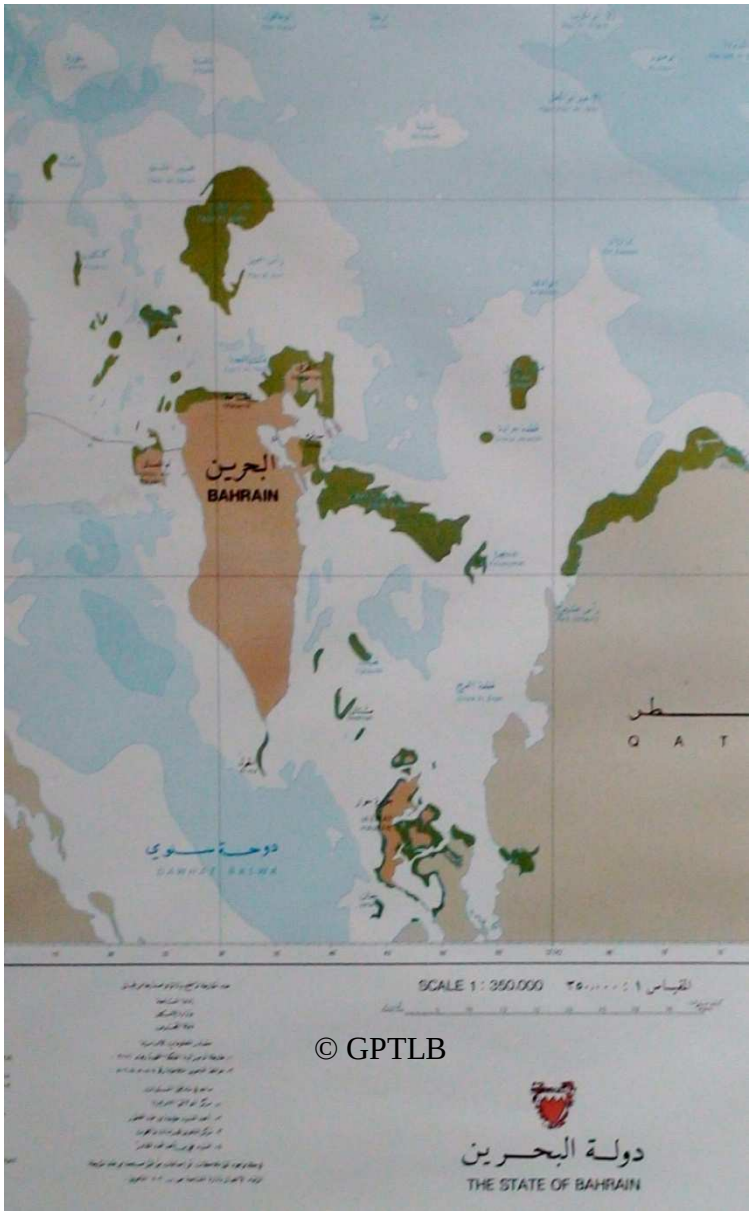
The oldest pearls found during an excavation; dated 2500 BC

Natural pearls (nacreous)

Natural pearls (nacreous)

19th century the 70-80% of the natural pearls were coming from the gulf

Natural pearls (nacreous)



Heirats…

Natural pearls (nacreous)

Natural pearls (nacreous)

Natural pearls (nacreous)

Bombay bunch

From 1 to 3mm, today it is cost more to drill and string the pearls than the pearls itself .

Natural pearls (nacreous)

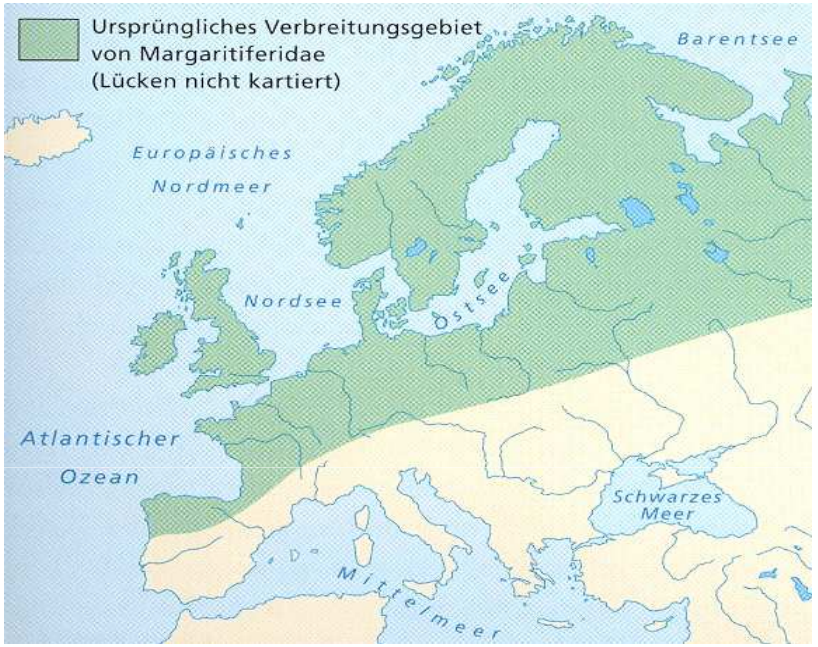
Natural pearls (nacreous)

Sri Lanka

Natural pearls (nacreous)

Freshwater pearls

Margaritifera margaritifera



Strack, 2000

Natural pearls (nacreous)

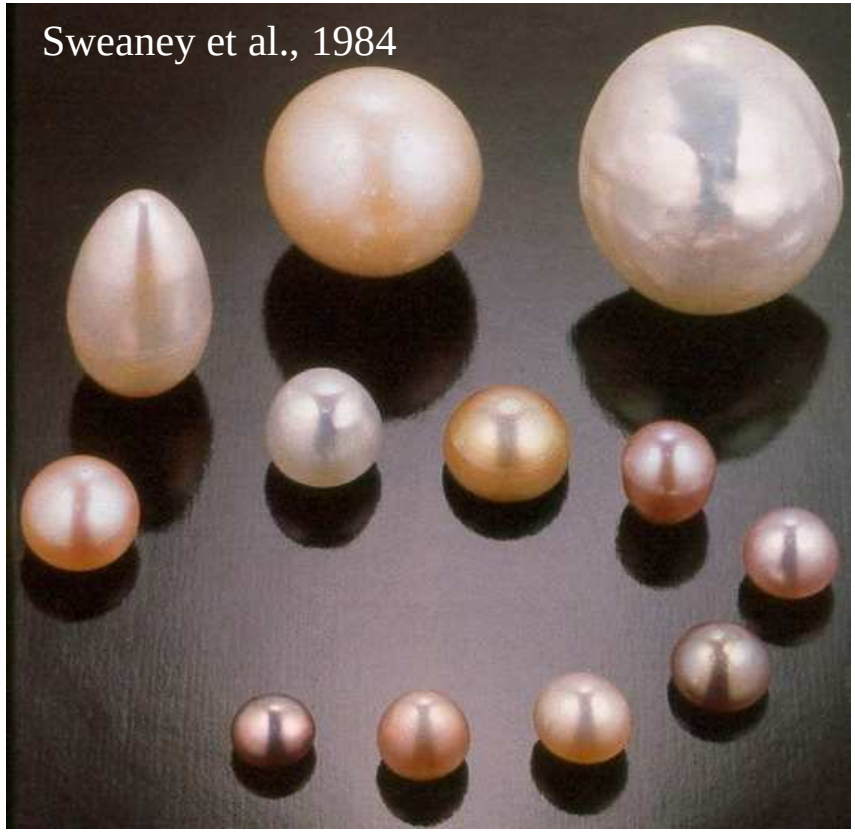
Freshwater pearls

Crown of Blanche of Valois

Crown of Charlemagne

Natural pearls (nacreous)

Freshwater pearls

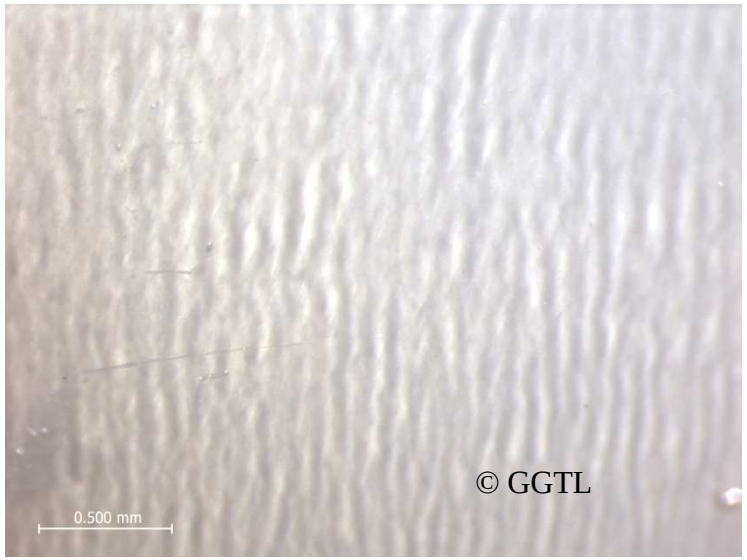


North America,
Megalonaias gigantea (Unio)
25.4mmx20.8mm to 7.5mm

More than 350 FW pearl producing molluscs species in US

Natural pearls (non-nacreous)

The vast majority of non-nacreous pearls used in jewellery are also mainly made of aragonite.



The most are fished today to the waters off Indonesia and Philippines.

Natural pearls (non-nacreous)

N°	ESPECE	carat	mm
1	Spondilus regius Cassis	2,75	9,15
2	madagascariensis Pleuroploca	11,50	28,73
3	gigantea	7,96	11,80
4	Atrina vexillium Pleuroploca	9,02	11,70
5	trapezium Pinctada	2,59	7,40
6	mazatlanica	3,43	9,00
7	Melo melo Pleuroploca	66,50	21,80
8	gigantea	8,18	11,90
9	stombus gigas	4,00	8,00
10	Pinctada radiata	2,89	10,00
11	Tridacna squamosa	31,88	16,20
12	Melo broderipii	45,70	18,50
13	Nautilus Pompilius Pleuroploca	7,53	10,90
14	gigantea	7,61	12,64
15	stombus gigas	3,00	8,00
16	Pinctada maxima	8,00	10,00
17	Haliotis iris Pleuroploca	7,00	12,00
18	gigantea Argopecten	112,00	24,40
19	purpuratus	3,67	8,46
20	Lopha cristagalli	7,44	10,00
21	Pinctada maxima, perle janus, un coté nacre, l'autre en calcite noire	3,97	9,50

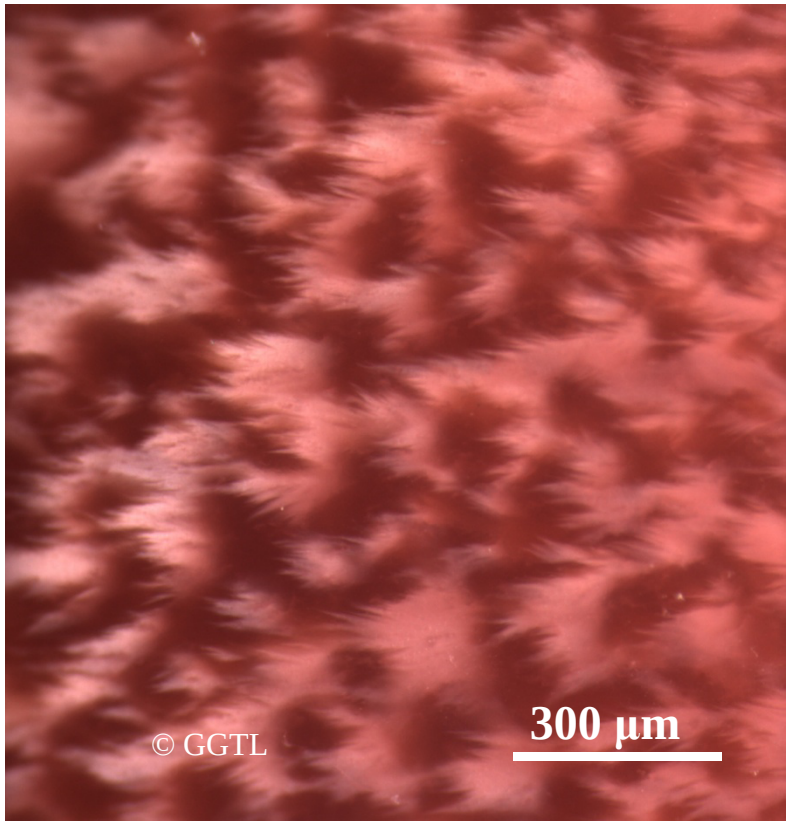
Natural pearls (non-nacreous)

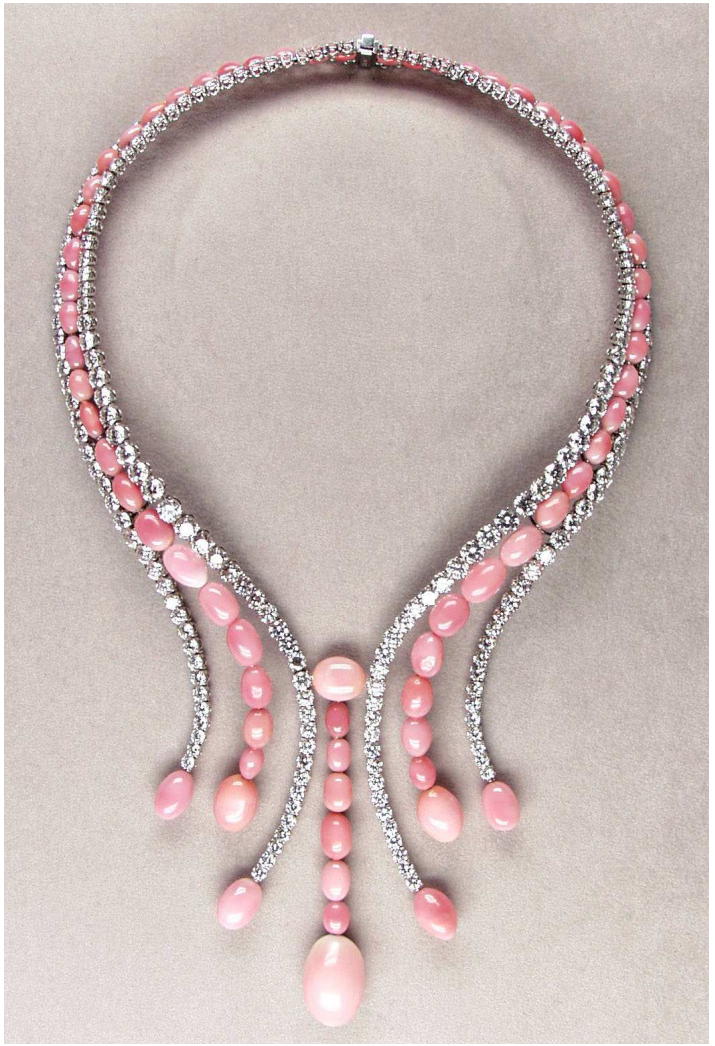
Strombus gigas

Queen conch pearls

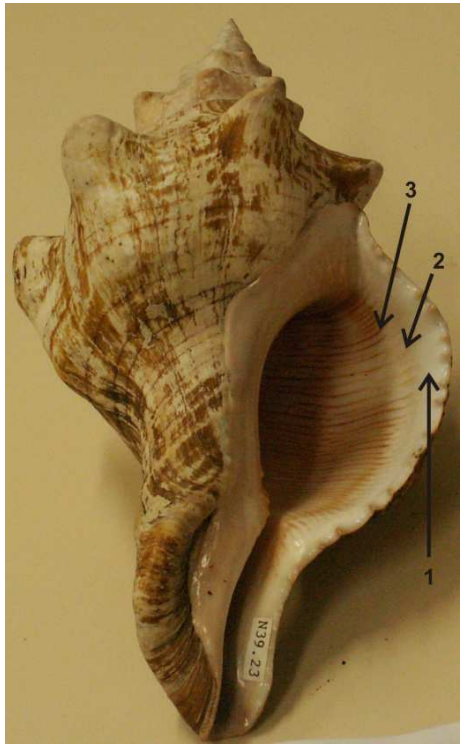
Natural pearls (non-nacreous)

Queen Conch pearls



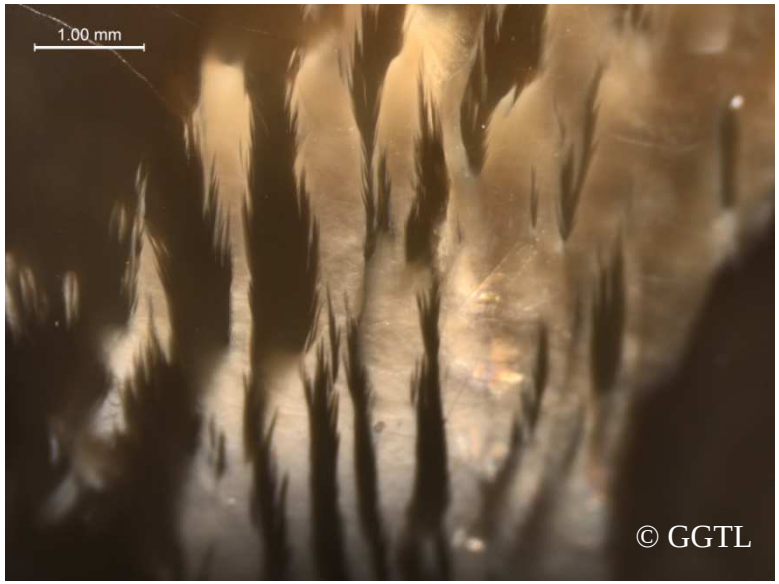


Natural pearls (non-nacreous)



Pleuroploca gigantea

(Horse) Conch pearls

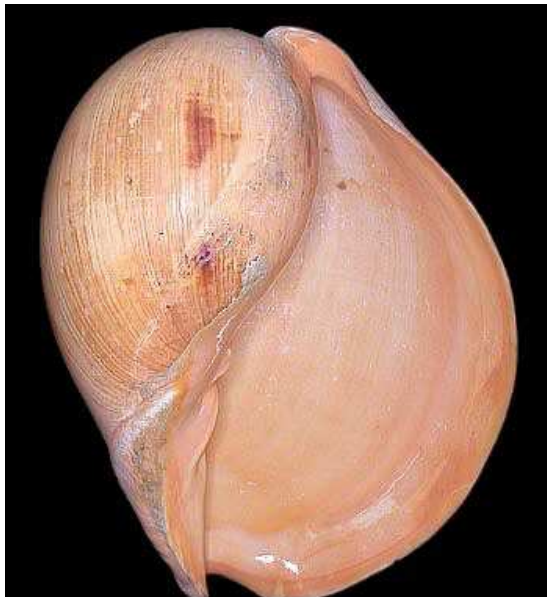


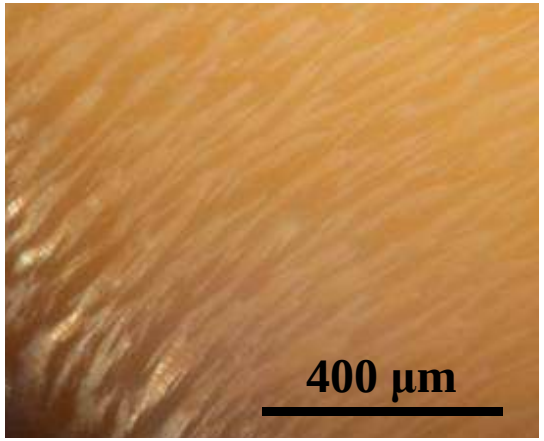
Natural pearls (non-nacreous)

Cassis ssp.

Emperor helmet pearls

Natural pearls (non-nacreous)





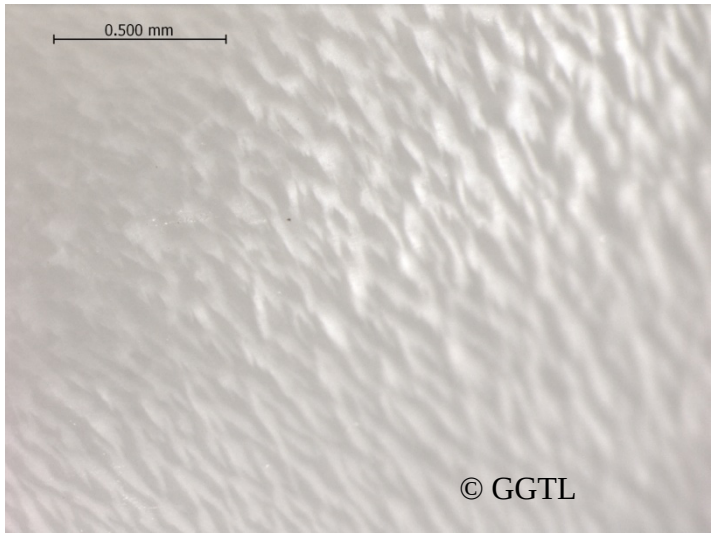
Melo ssp.

Melo pearls

Natural pearls (non-nacreous)

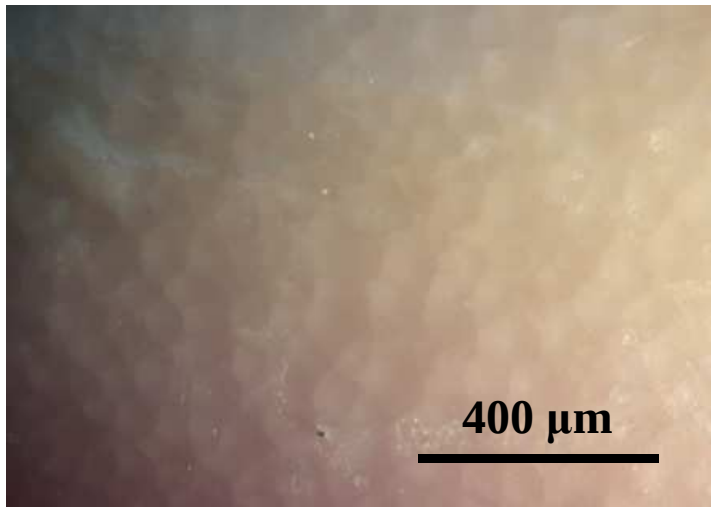
Tridacnidae family

Giant clam pearls



Natural pearls (non-nacreous)

Veneridae family
Quahog pearls

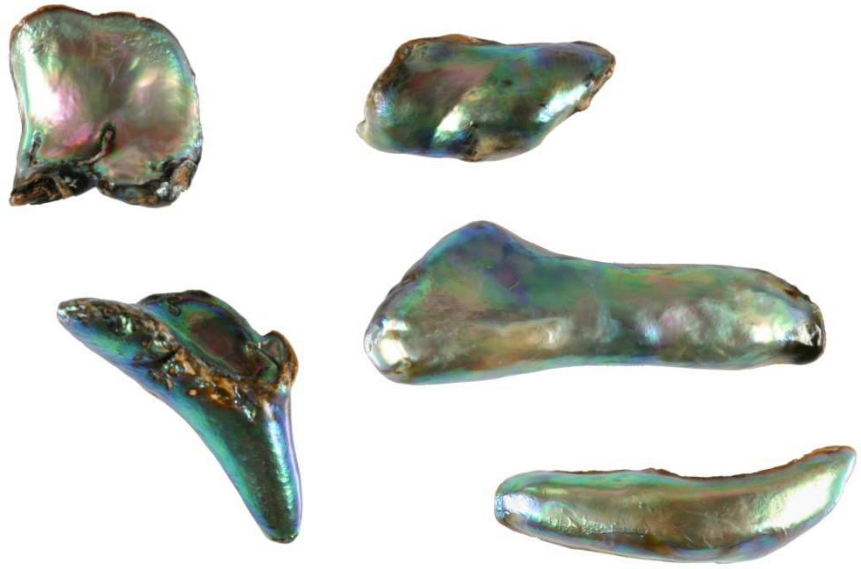


Honeycomb structure: prismatic arrangement of aragonite formed by parallel and adjacent prisms that do not strongly interlock long their mutual boundaries

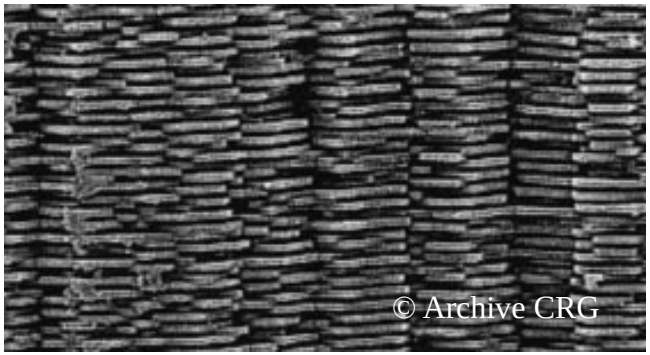
Natural pearls (non-nacreous)

Haliotis sp.

Abalone pearls



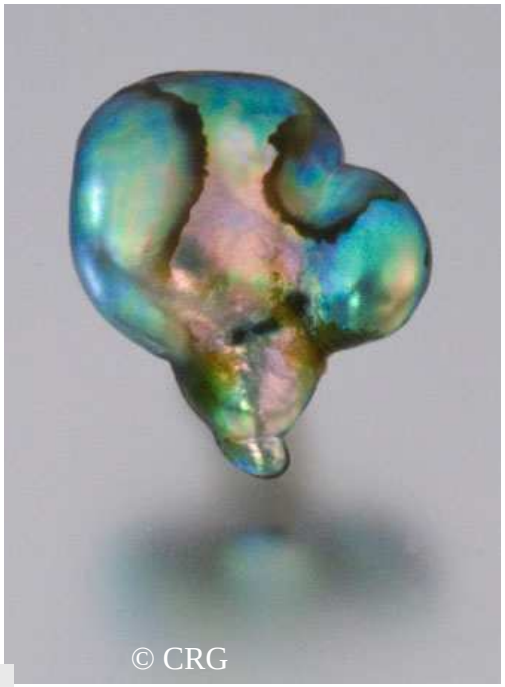
Aragonitic pearls



Natural pearls (non-nacreous)

Haliotis sp.

Abalone pearl



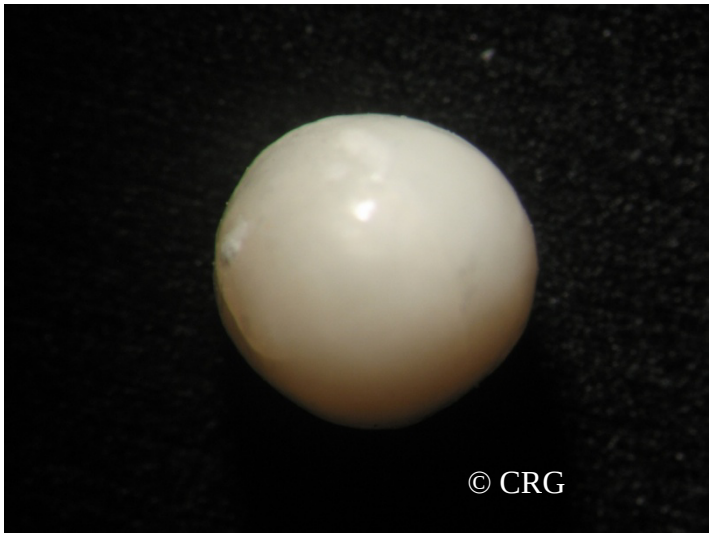
Natural pearls (non-nacreous)

Natural pearls (non-nacreous)

Crassostrea sp.

True oyster

Natural pearls (non-nacreous)



"Bigorneau" (winkle) pearl (cooked)

Littorina sp.

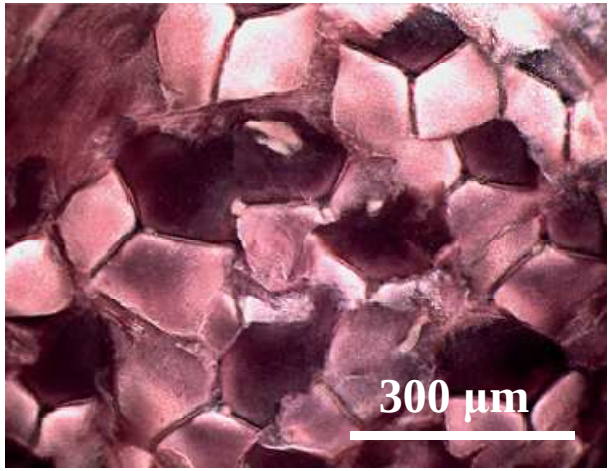
Natural pearls (non-nacreous)

Calcitic

Pectenidae family

Scallop pearls

Structures: ~ segmented patchwork of cells, with each cell comprising three differently oriented subsegments



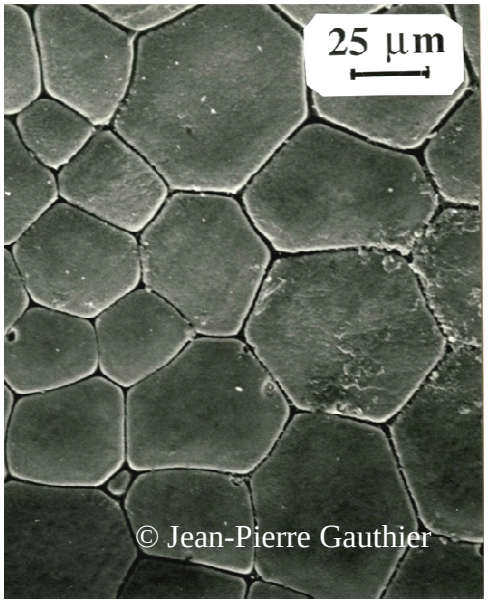
Natural pearls (non-nacreous)

Pinnidae family

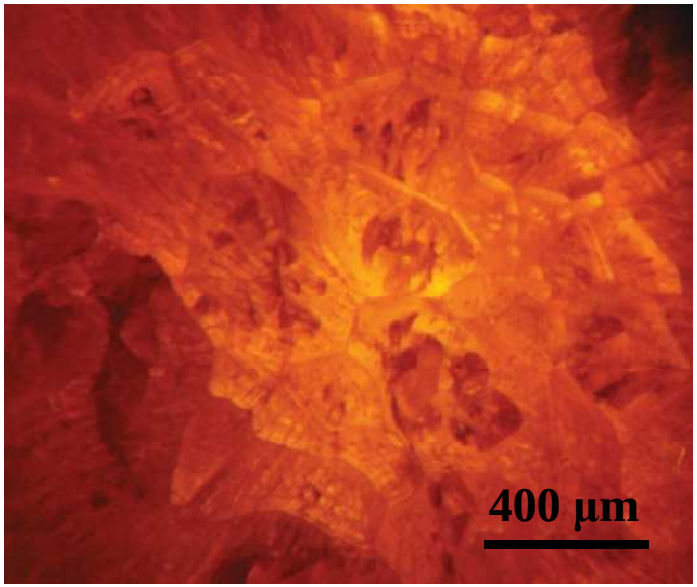
Calcitic

Pen shell pearls

Natural pearls (non-nacreous)



Columnar calcitic structures were observed with transmitted illumination



Natural pearls (non-nacreous)

Pinna nobilis

Calcite/aragonite

Pearl testing

Introduction/Pearl identification

Past (30 years ago):

Pearl or imitation?

FW or SW?

Natural or cultured?

Introduction/Pearl identification

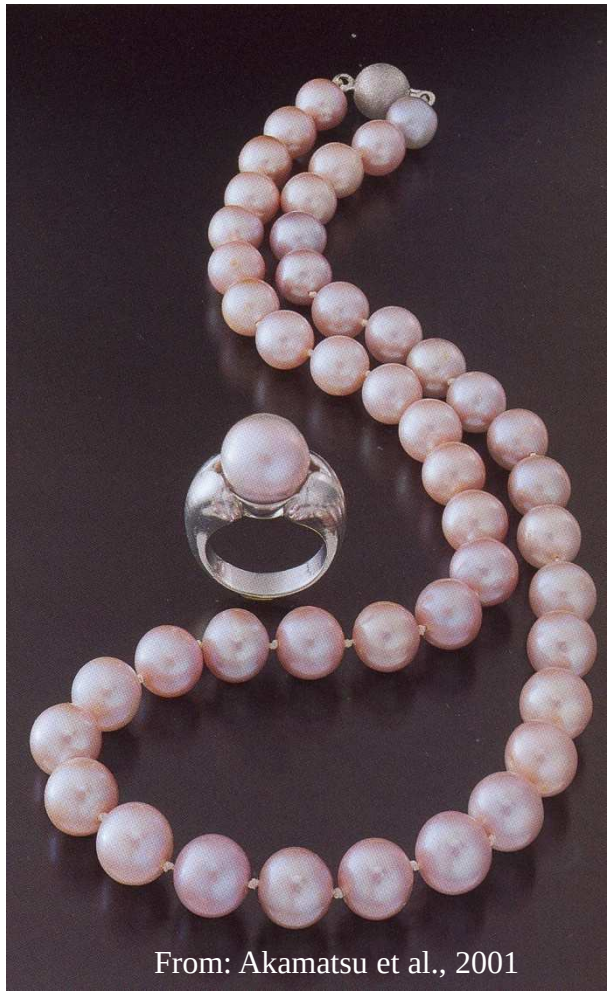
Today:

Pearls or imitation?

FW or SW?

Natural or cultured?

Bead nucleated or non-bead nucleated? If beaded, what kind of bead was used?



From: Akamatsu et al., 2001

Introduction/Pearl identification

Today:

Natural color?

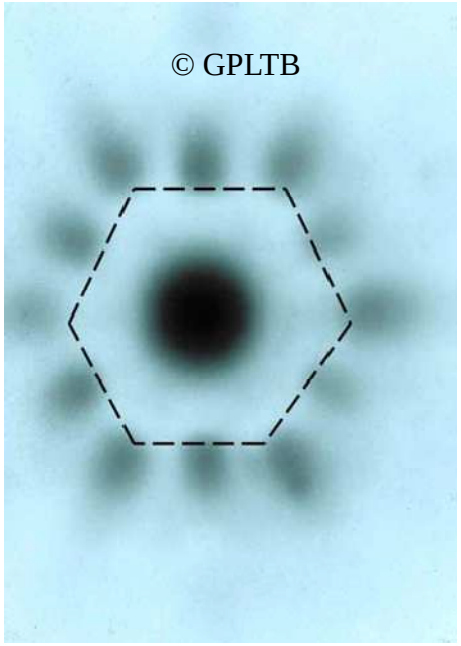
Mollusk?

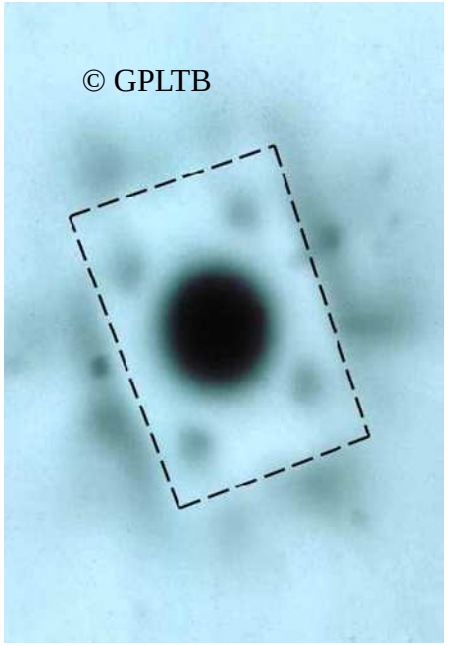
CITES protected?

Quality control of pearls

Analysis/ “Forgotten” tests

Lauegram





Candling

Analysis/Microscope

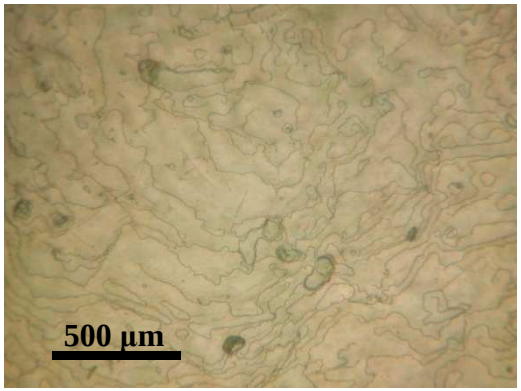
GÜBELIN GEM LAB

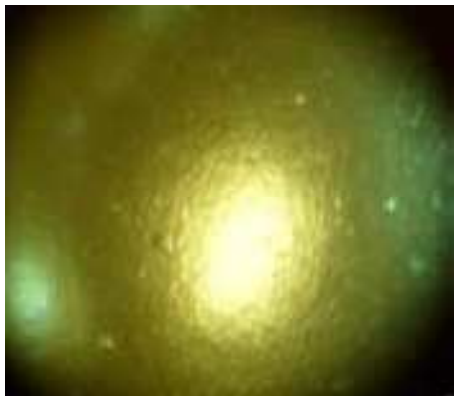
Microscope

Analysis/Microscope

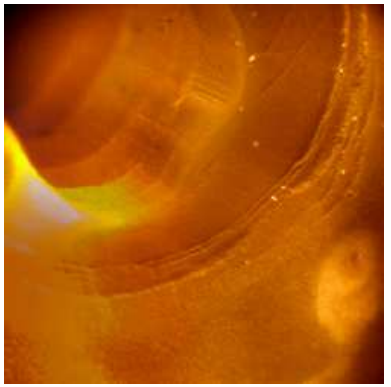
Before

Natural or imitation?





Beaded or natural (observation through the drill hole)?



Analysis/Microscope

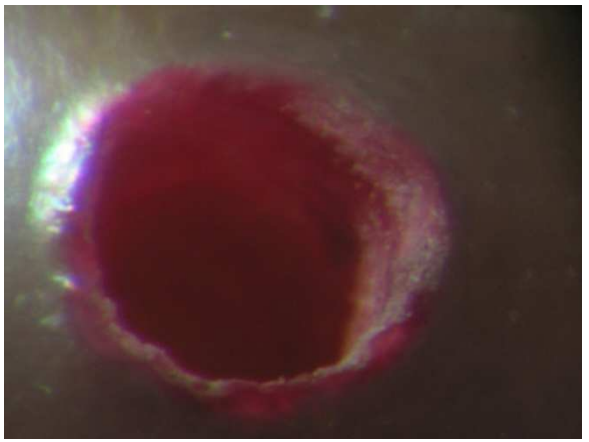
GÜBELIN GEM LAB

Today

Natural or imitation?

Beaded or not?

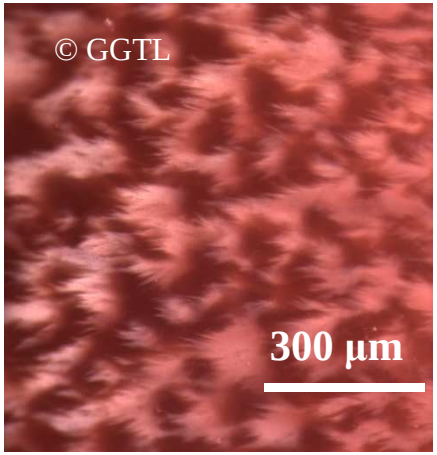
Color spots? Color treatment



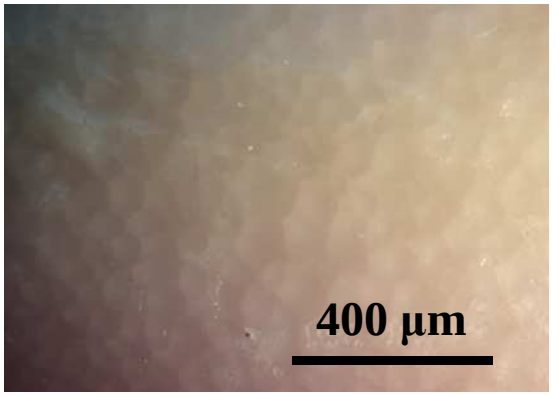
Analysis/Microscope

Nacreous or non-nacreous?

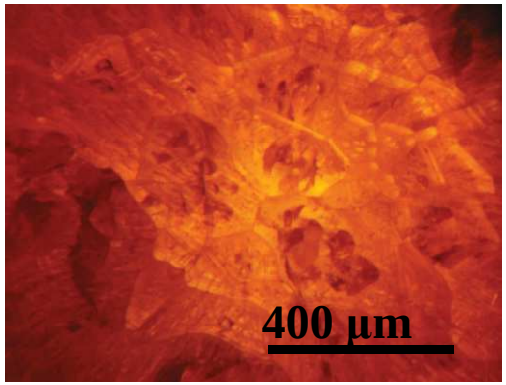
Mollusk?



Queen conch pearl
CITES protected

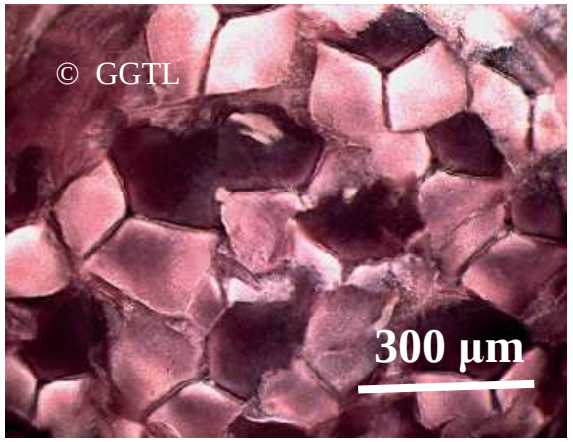


Quahog pearl



Pen pearl

Polished?



Scallop pearl

Analysis/UV lamps

Necklace with natural pearls from *Pteria ssp.* and *P. margaritifera*

LWUV

SWUV

<u>Color treated?</u>

Analysis/Luminescence to X-rays

X-ray cabin

FW vs. SW → FW luminesce (due to Mn?), attention to colored FW, and to SWCP with bead and thin nacre

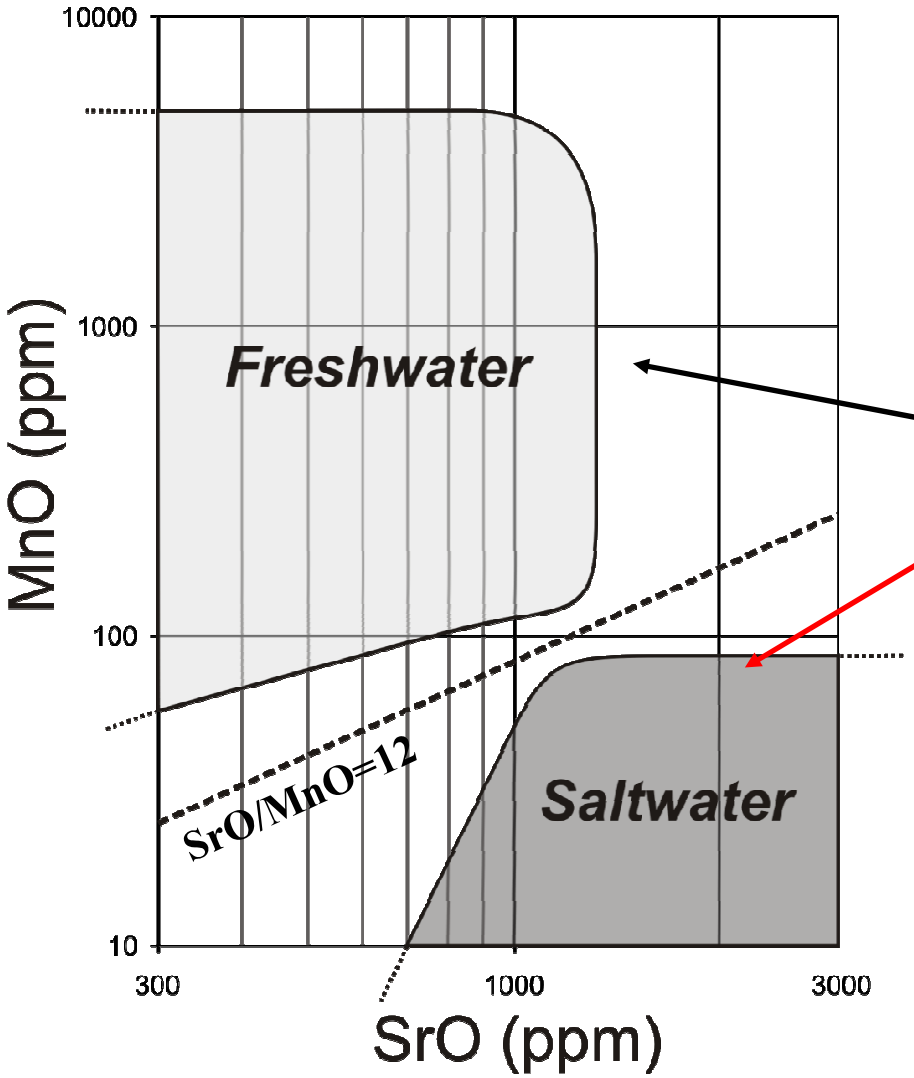
Analysis/EDXRF

EDXRF

SW vs. FW?

Inorganic dye (e.g. Ag? Br?)?

Analysis/EDXRF



Analysis/Radiography

X-radiography (similar to those for medicine)

Natural vs. Cultured pearl...

Silver salt treatment...

Analysis/Radiography

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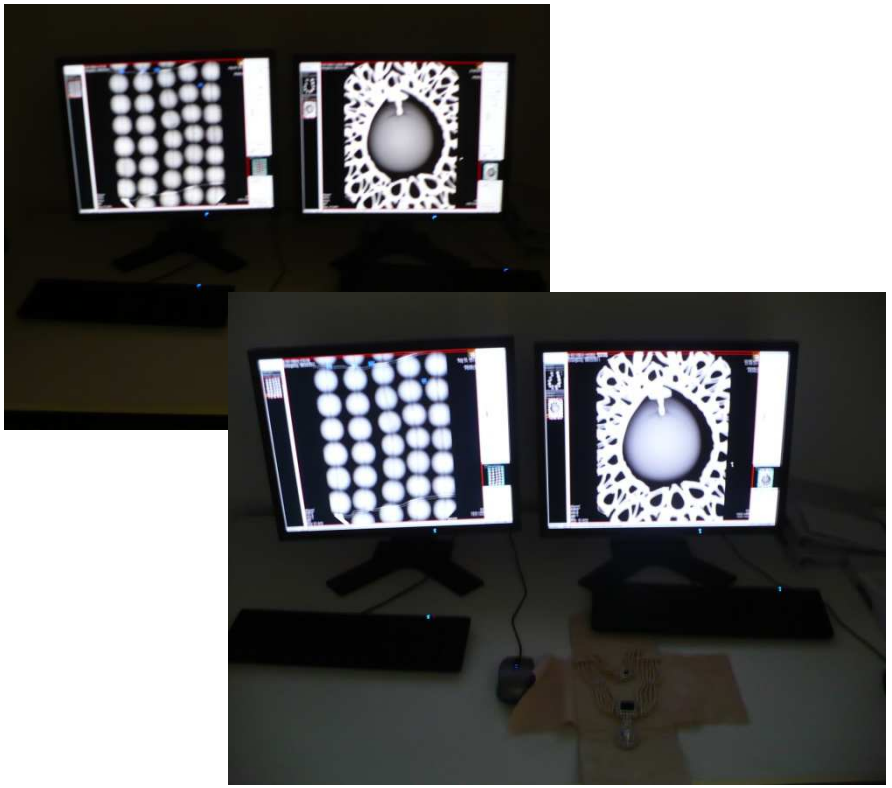
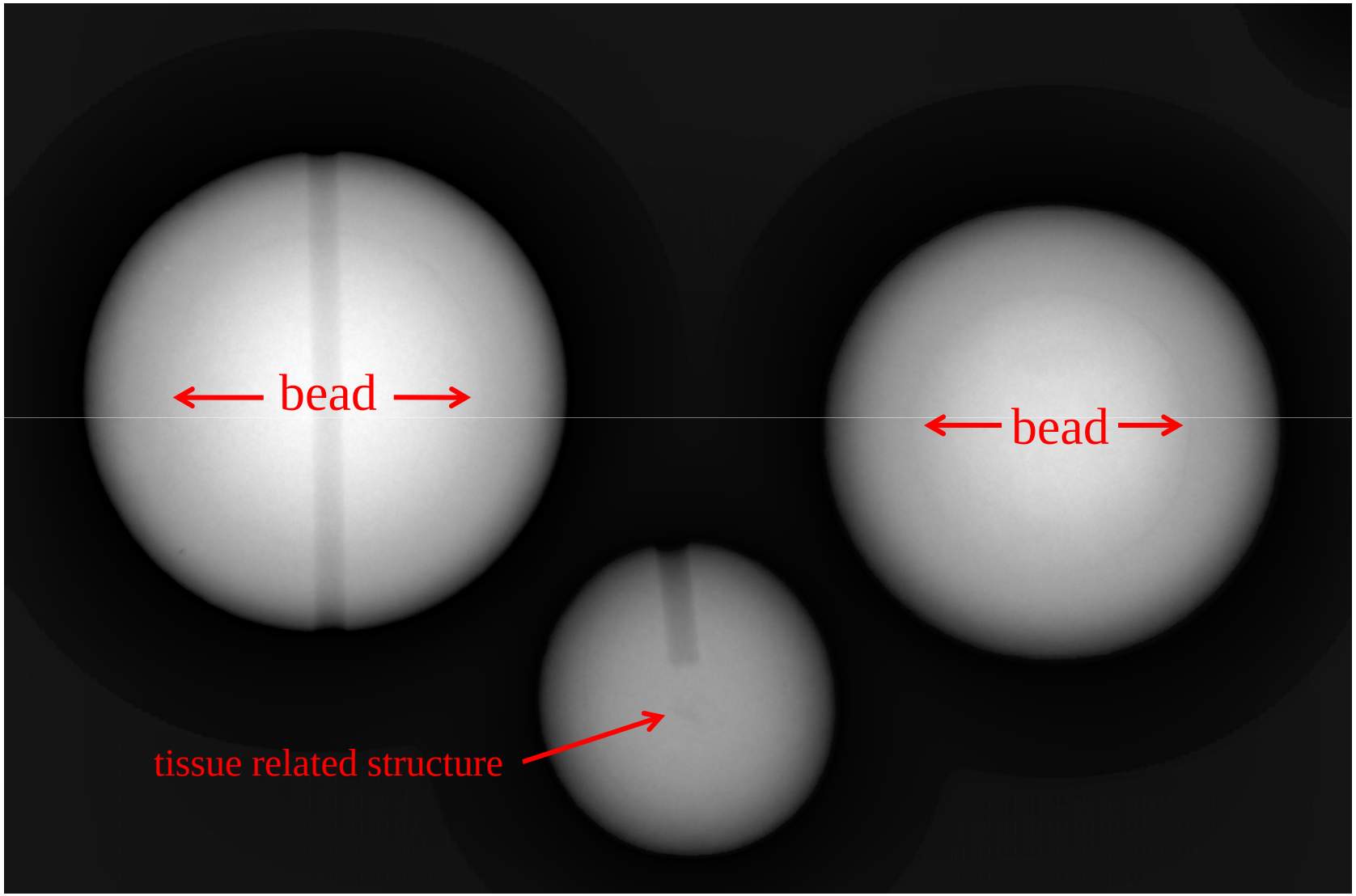
Films

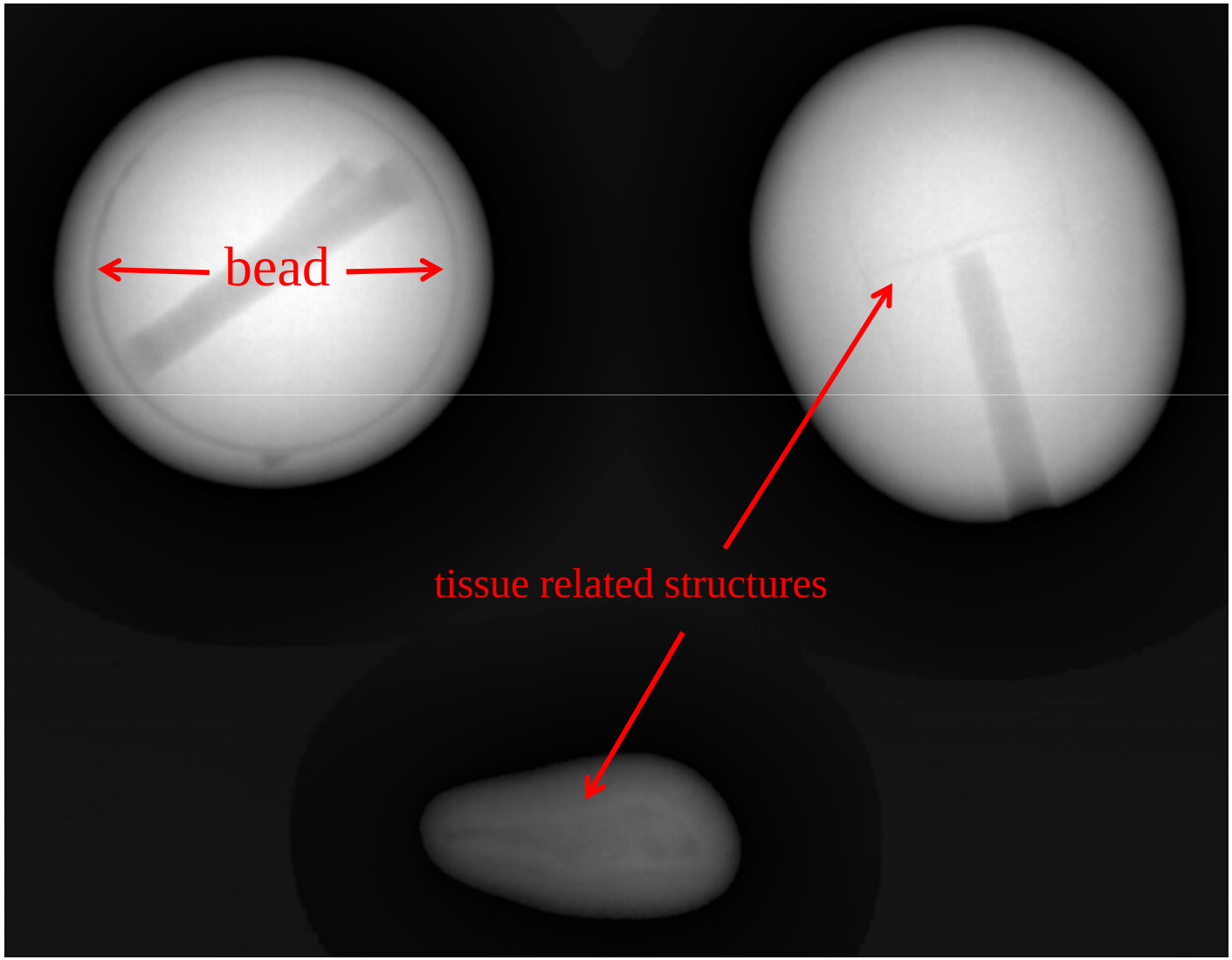
Digital



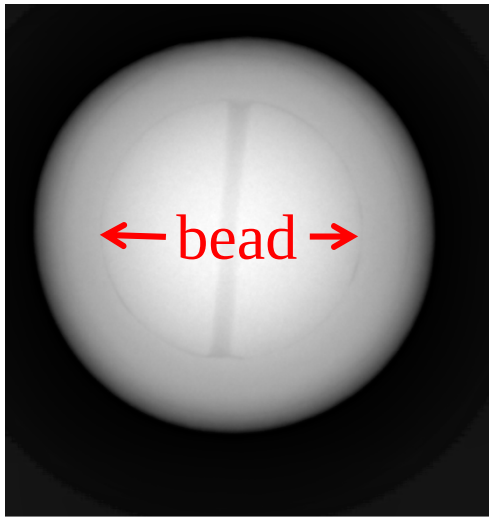
Analysis/Radiography



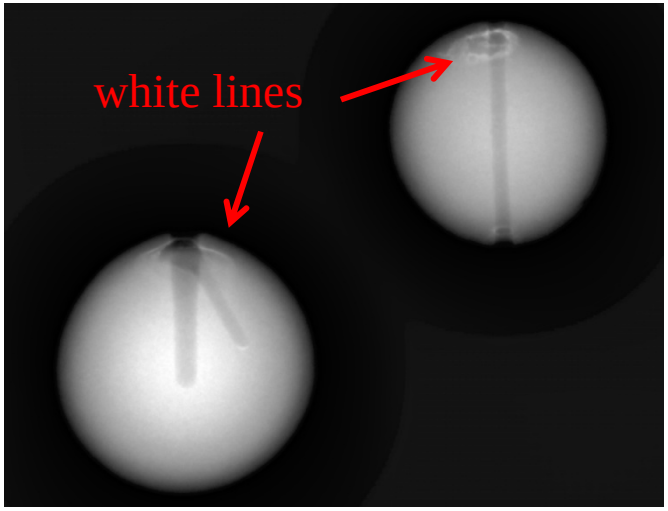
Analysis/Radiography

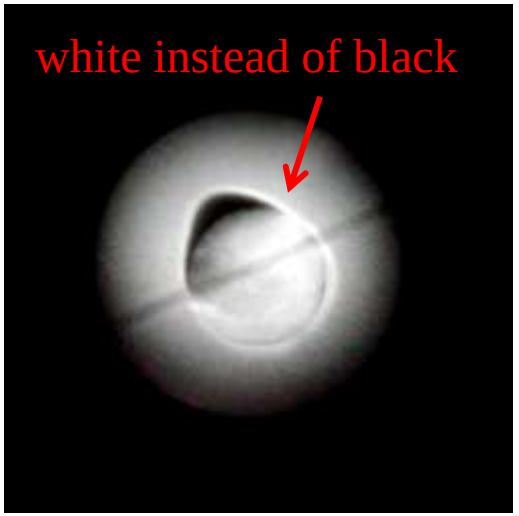


Analysis/Radiography



FWCP with drilled bead

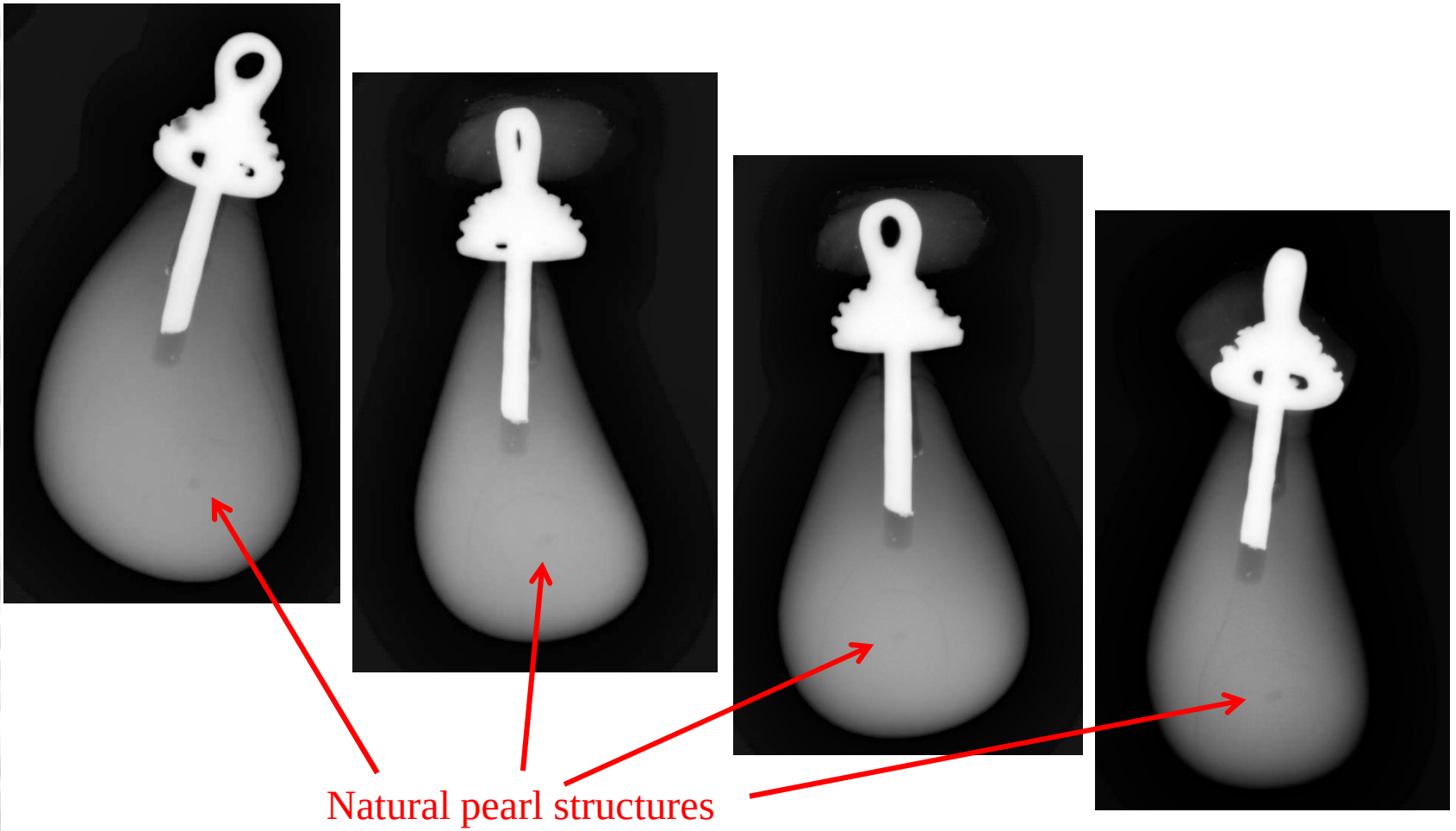




SWCPs treated with $AgNO_3$ after drilling

Methods/X-ray imaging

Digital



Sometimes in different directions; much easier with digital

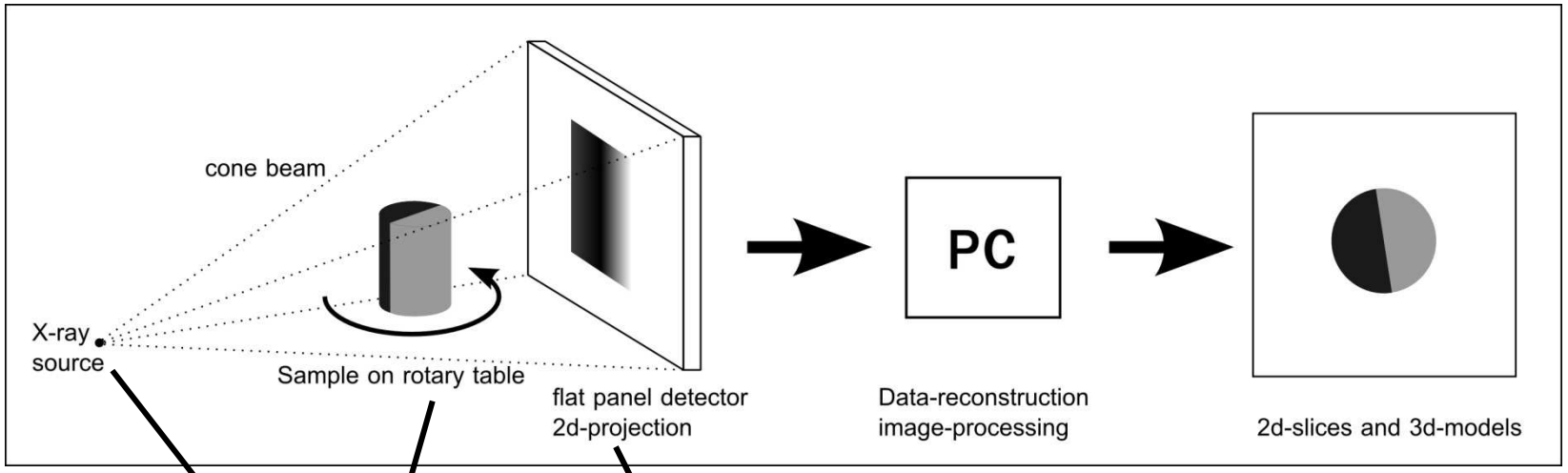
Analysis/Tomography

2.5 tons

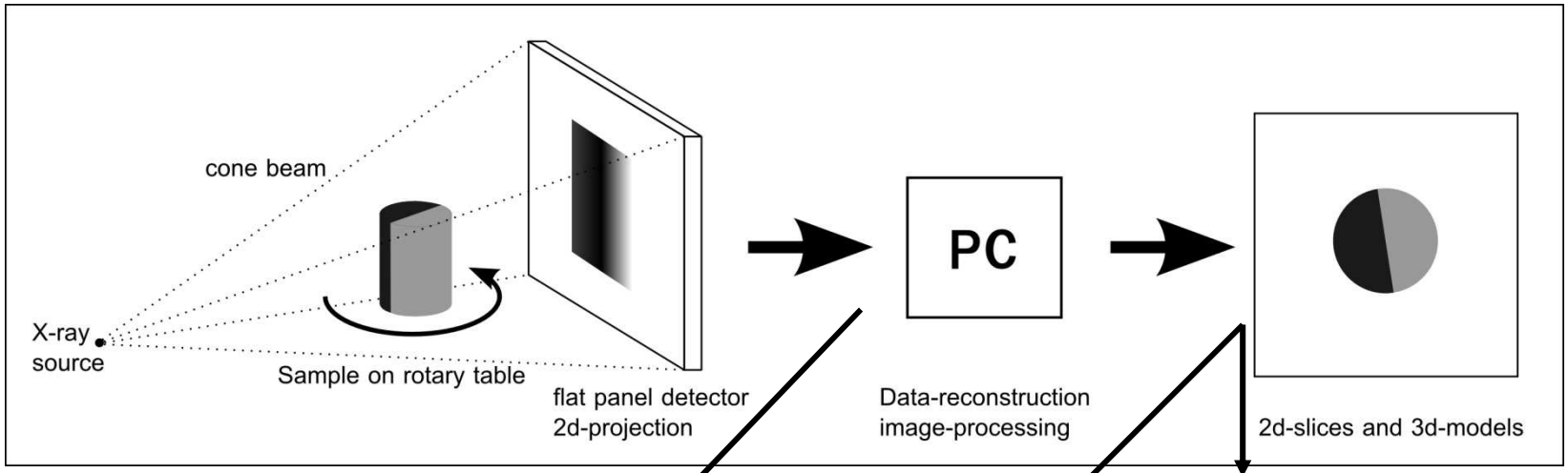
CT-alpha instrument

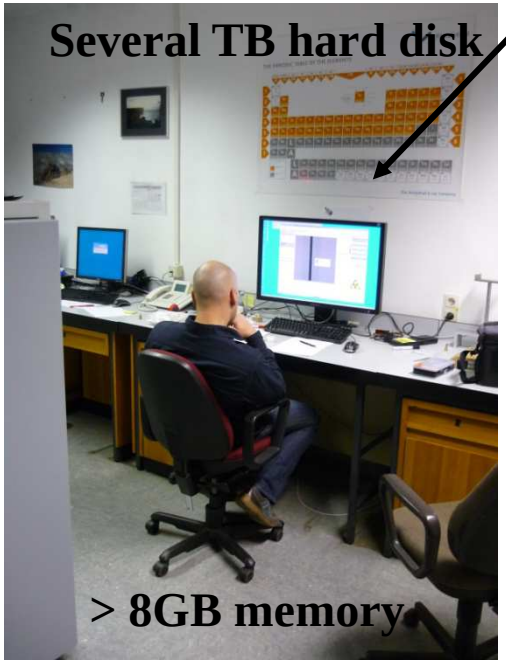
190x150x100 cm

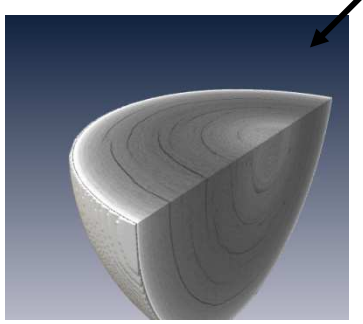
Analysis/Tomography

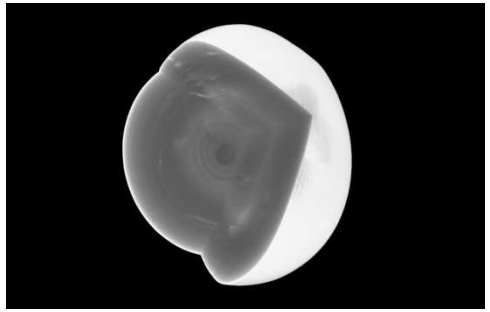


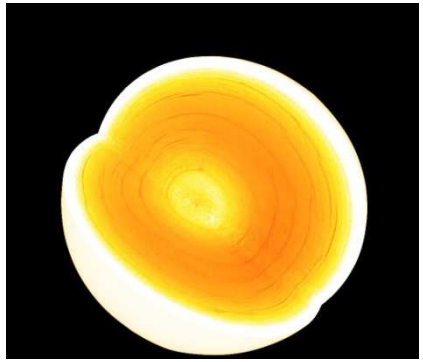
Analysis/Tomography

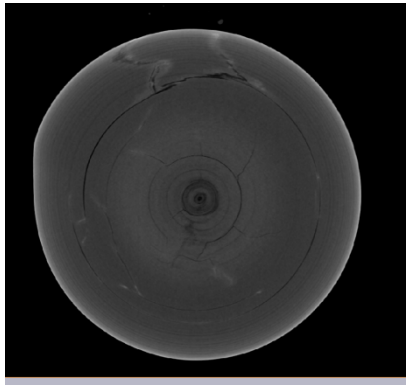






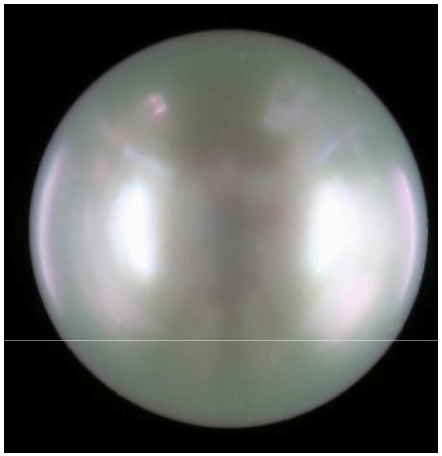


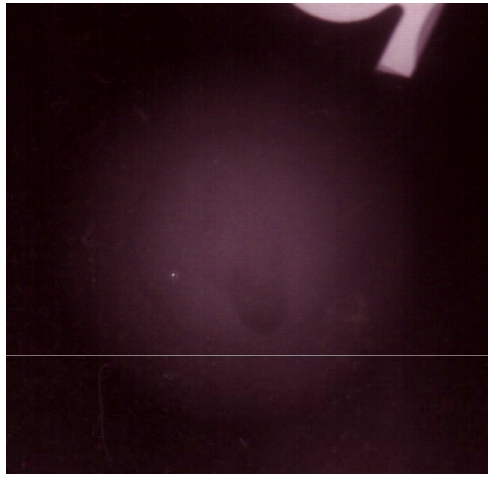


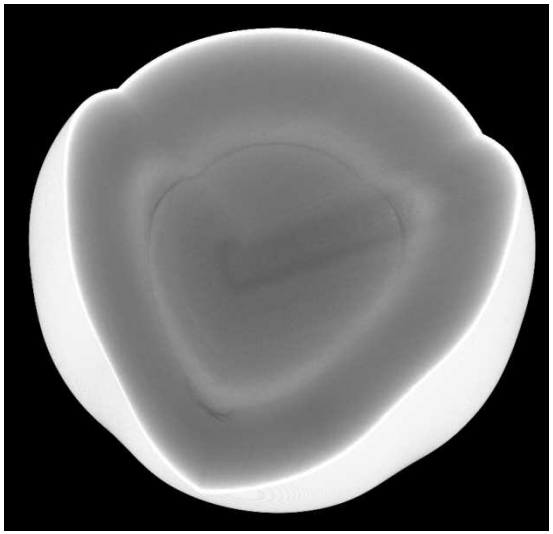


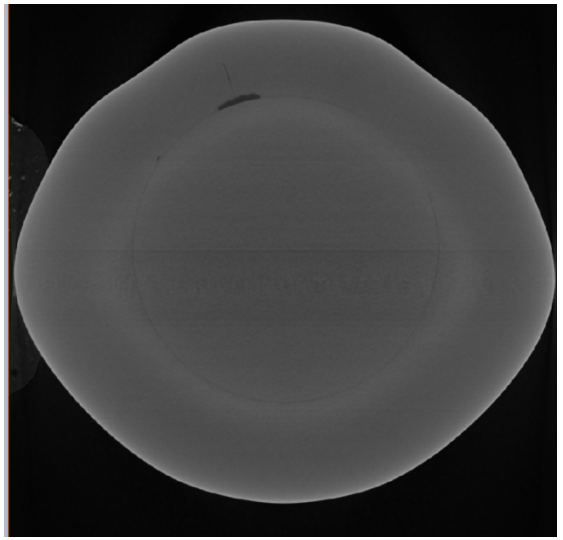
Results

White button shape BSWCP from *P. maxima*, 12.7x11.3 mm
Resolution of µ-CT: 13.8 µm



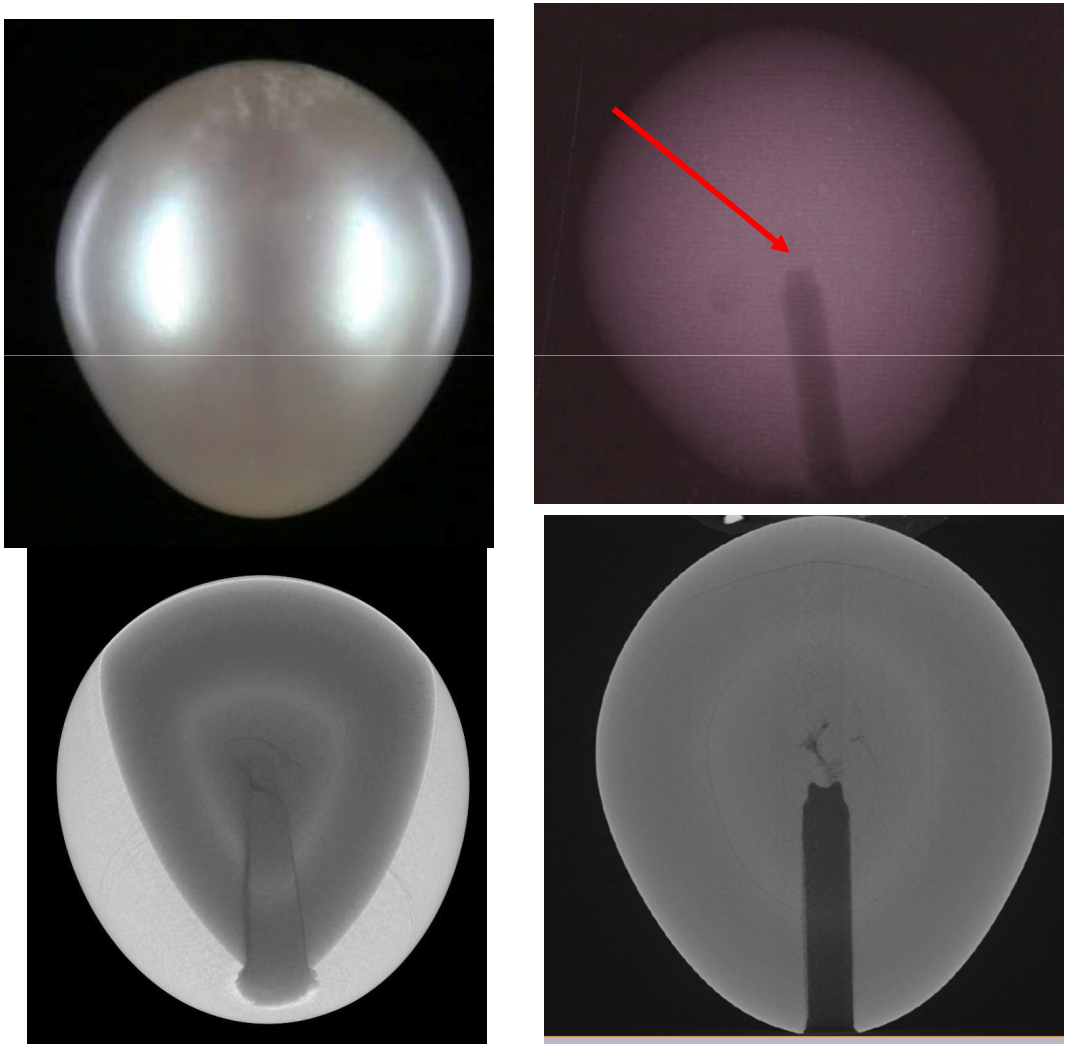






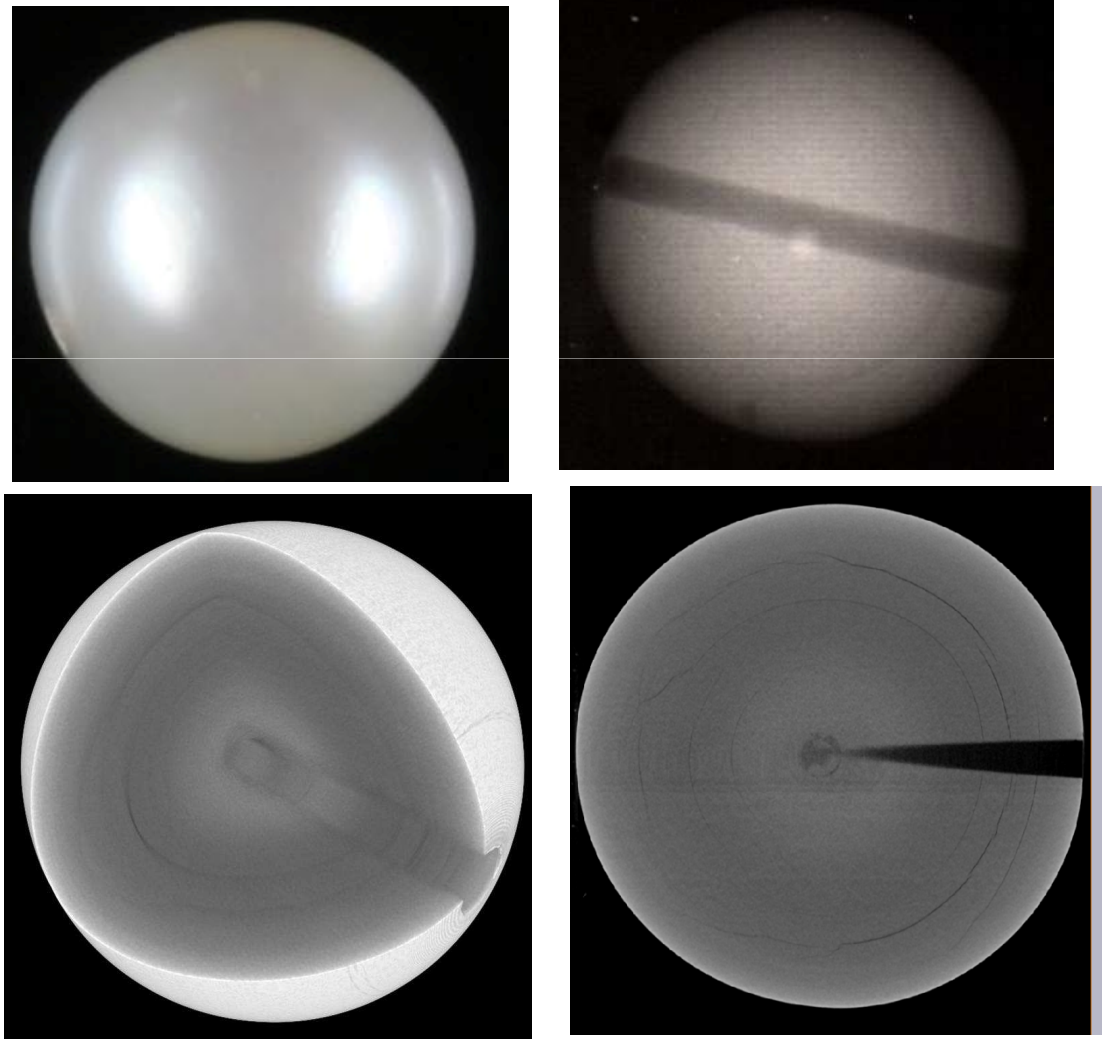
Analysis / Tomography NBFWCP

Light grey drop sh. NBFWCP *Hyriopsis spp.*, 10x8.8 mm
Resolution of µ-CT: 11 µm



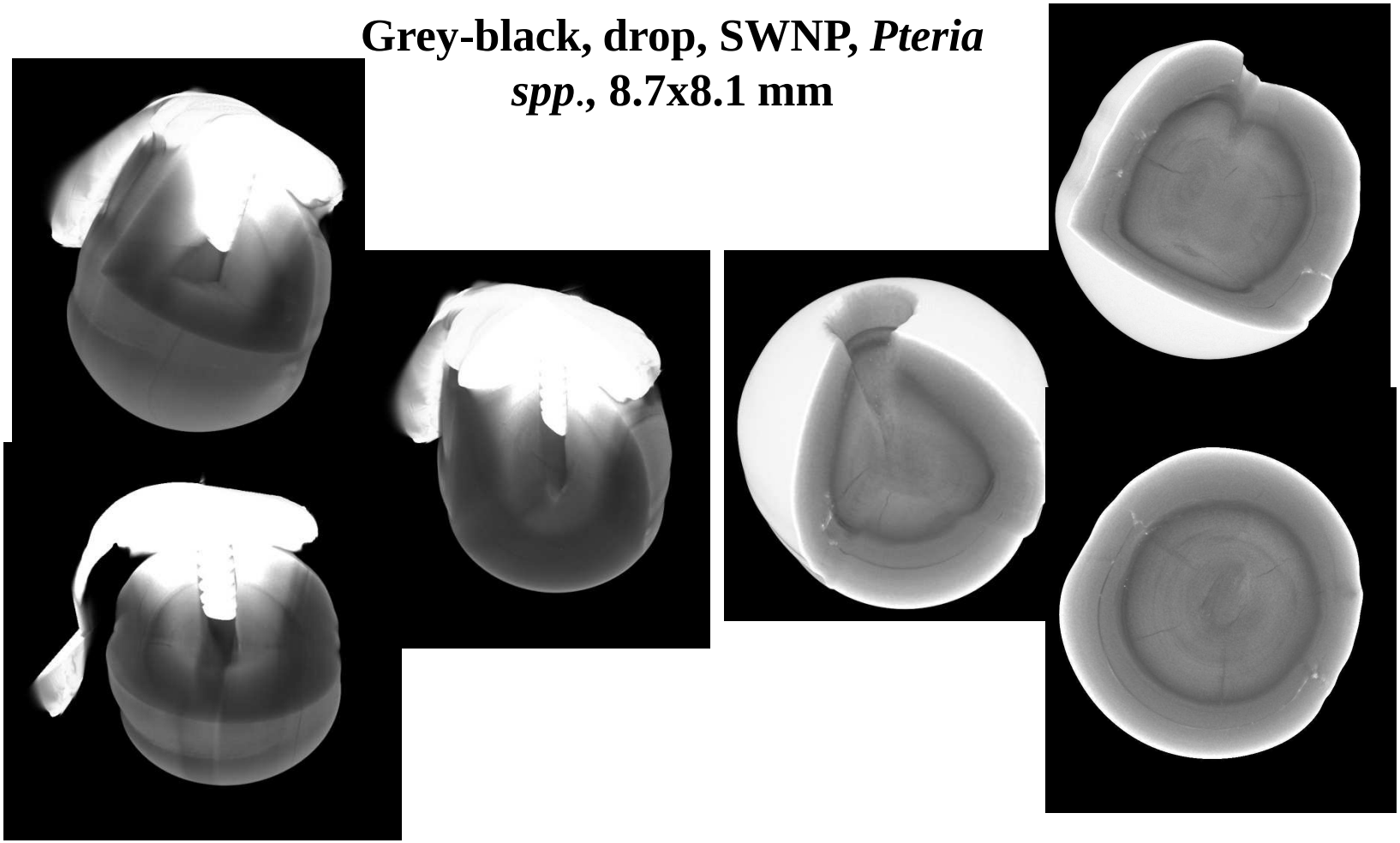
Analysis/ Tomography SWNP

Light grey, round, SWNP *Pinctada spp.*, 6.4 mm
Resolution of µ-CT: 7 µm



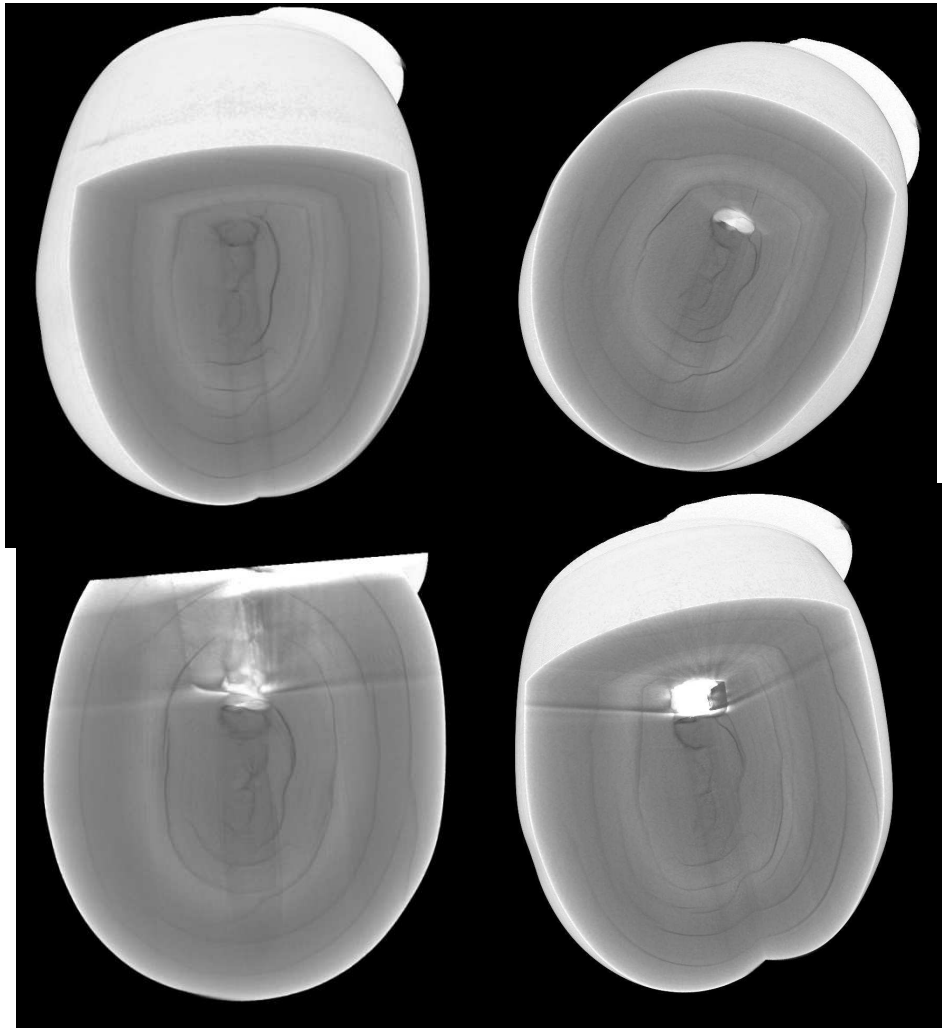
Results/Metal mounting

Grey-black, drop, SWNP, *Pteria spp.*, 8.7x8.1 mm



Results/Metal mounting

Grey-purple, drop, NBFWCP,
***Hyriopsis spp.*, 11x8.9 mm**



Results/ Strands

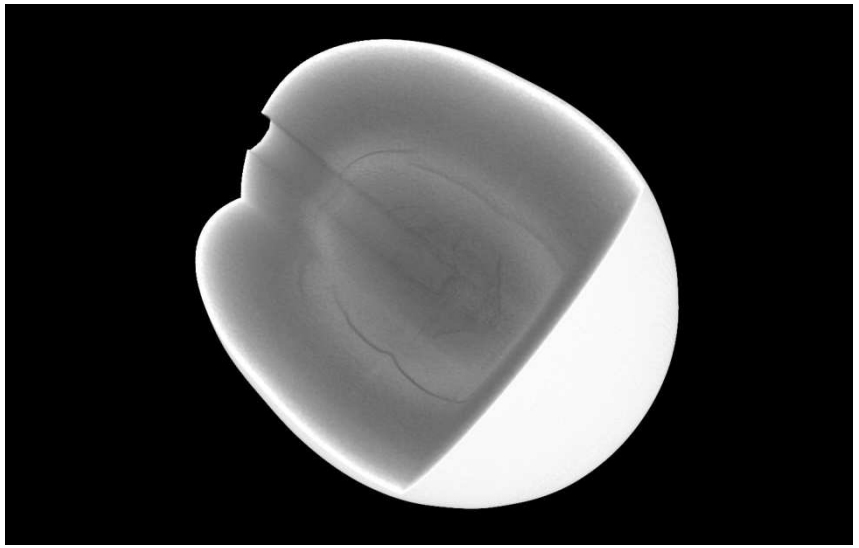
Possible only under exceptional conditions

Results/Time CT measurement

GÜBELIN GEM LAB

Time from sample mounting to model is > 4 hours

> 4h



Identification of the bead, if bead cultured pearl…

Analysis/Raman spectroscopy

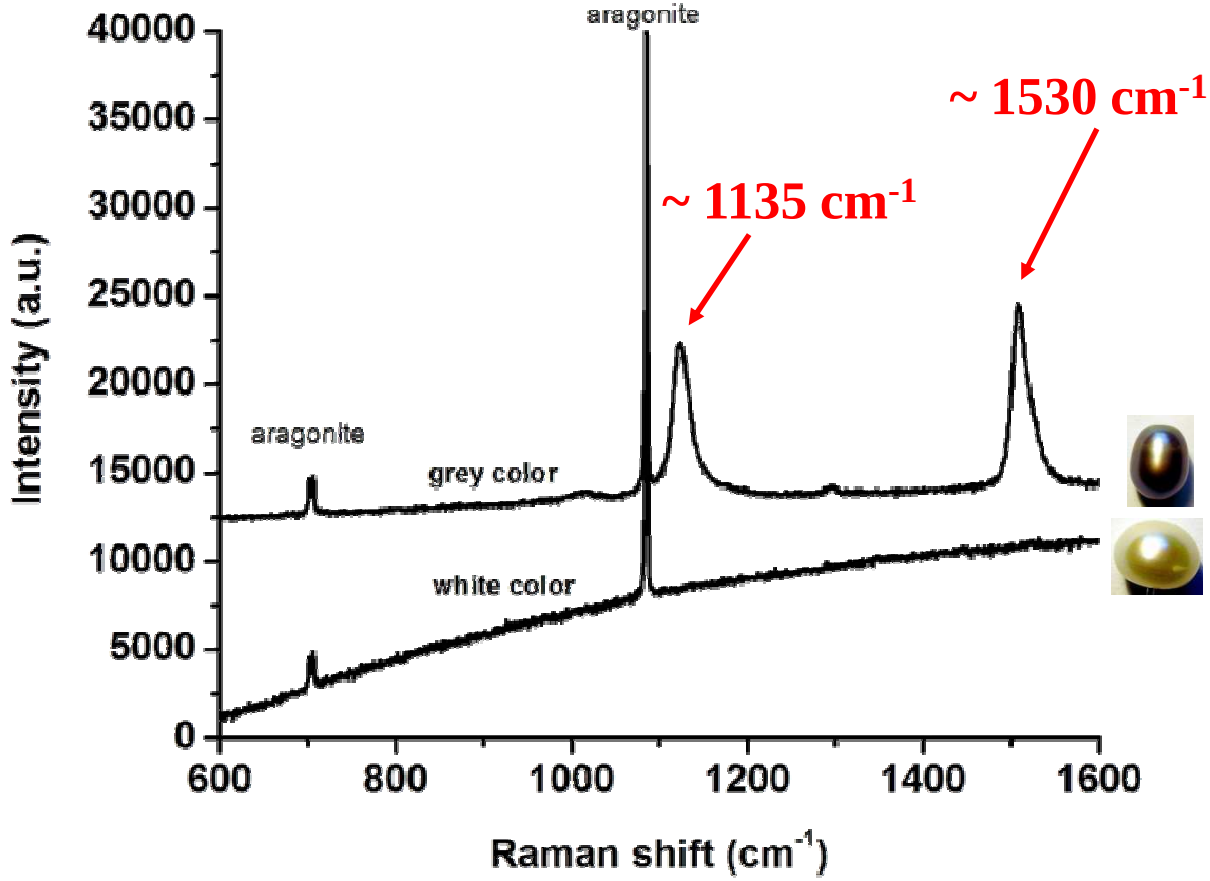
Raman spectrometer

Color authenticity of FWCPs...

Mollusk identification...

Methods/Raman spectroscopy

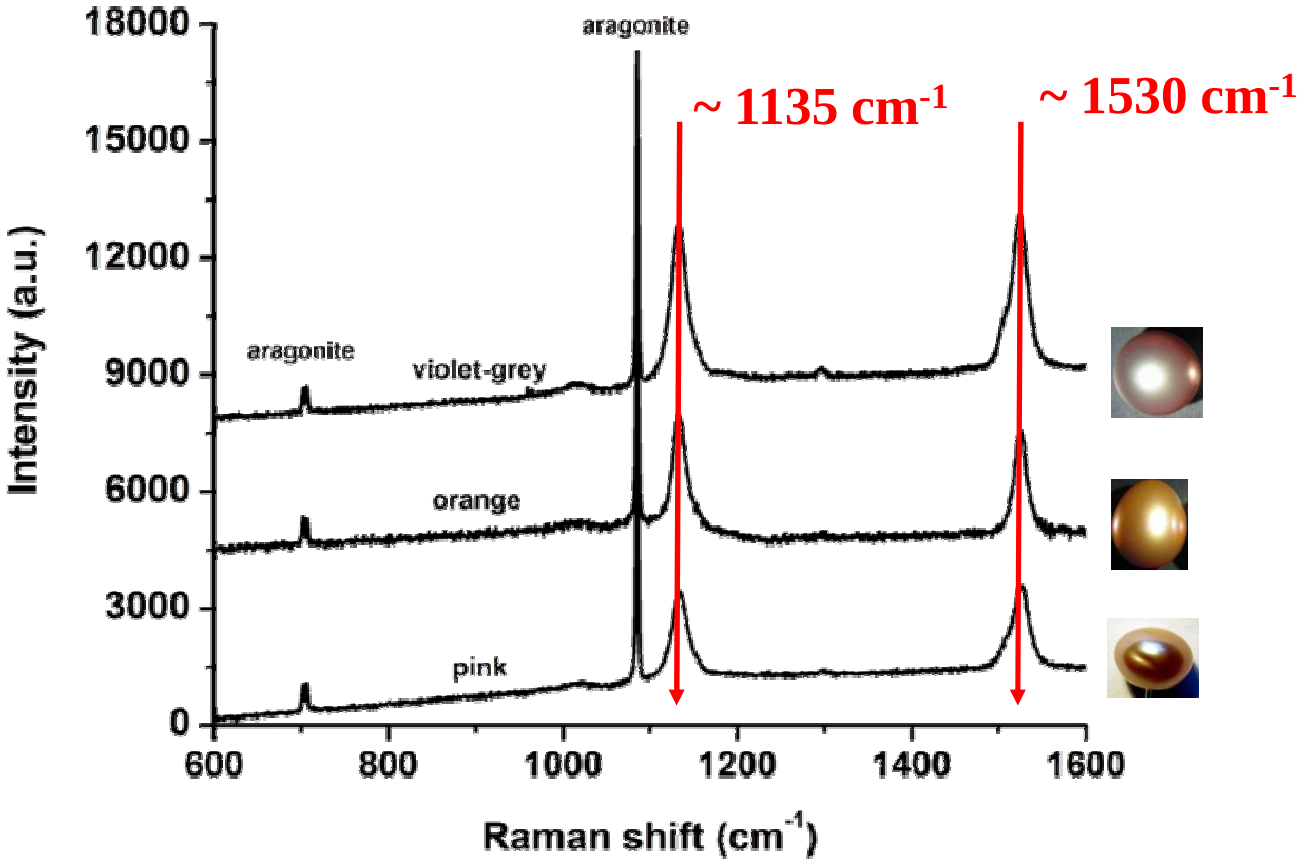
Natural-color FWCPs, Excitation: 514 nm



Two intense additional bands in natural-color FWCP.

Methods/Raman spectroscopy

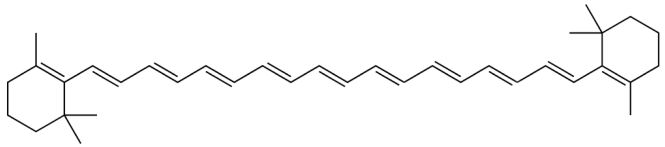
Natural-color FWCP, Excitation: 514 nm



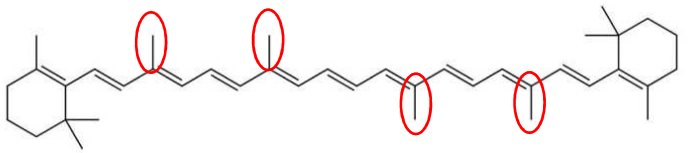
Peaks at 1130±15 cm^{-1} (C-C stretching) and at 1530±30 cm^{-1} (C=C stretching) characteristic of compounds named polyenes are observed in all natural-color FWC pearls.

Methods/Raman spectroscopy

Polyenes (polyacetylenes) are the compounds that contain one or more sequences of alternating double and single carbon-carbon bonds (polyenic chain), regardless of their terminal ends. Their general chemical structure is: R-(-CH=CH-)$_n$-R'.



Carotenoids are polyacetylenic molecules which additionally have four methyl (CH_3) groups attached in their polyenic chain. Their terminal ends may vary.



The majority of pigments found in nature containing polyenic chain belong to carotenoids family (vegetables, birds etc.).

Methods/Raman spectroscopy

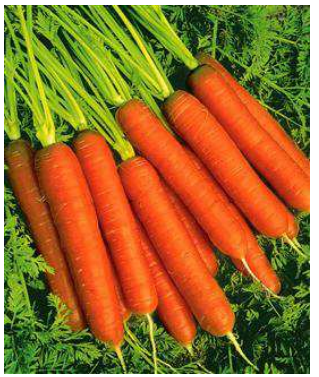
Polyenes

Ara sp.

Eolophus sp.

Carotenoids

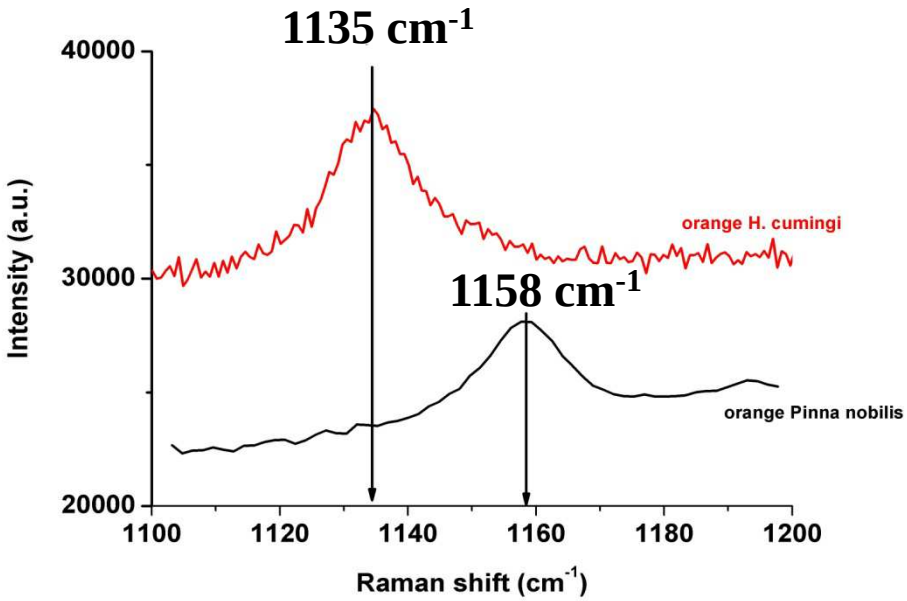
Serinus sp.



Methods/Raman spectroscopy

For simple polyacetylenic molecules the band at about 1135 cm^{-1}.

For polyacetylenic molecules this band is shifted about 25 cm^{-1} to approximately 1160 cm^{-1} (due to the methyl groups).



Pigments of natural colored cultured pearls from FWCPs contain no CH_3 groups their are not member of carotenoid family.

Methods/Raman spectroscopy

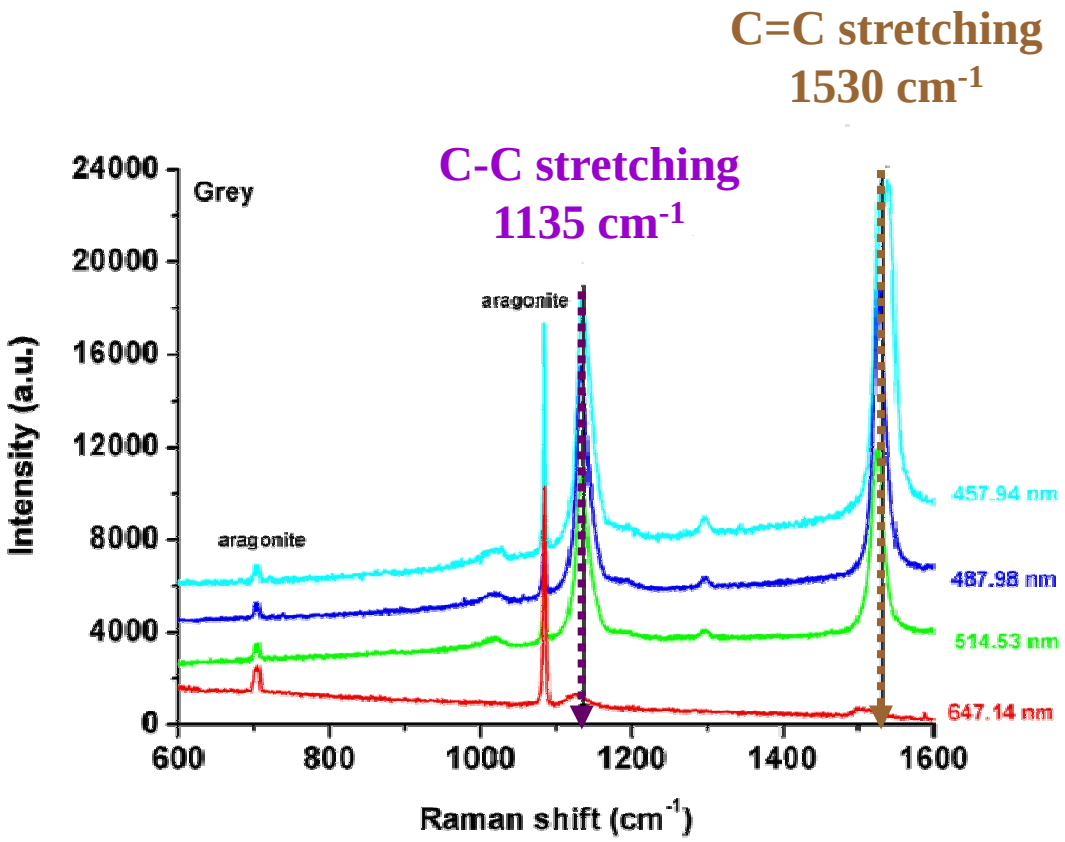
Polyacetylenic compounds present Raman resonant phenomena.

Their scattering enhanced by excitation in the absorption bands of the pigments.

Raman spectroscopy : Bands remain in the same position, changing the excitation wavelength. Just their intensities vary.

Changing excitation wavelength, variations in the position, shape and relative intensities of the two most intense bands are noted (mainly for C=C stretching band). Measurements were taken for the same point of the same sample.

Methods/Raman spectroscopy



Exact position of C=C stretching band depends on the polyenic chain length.

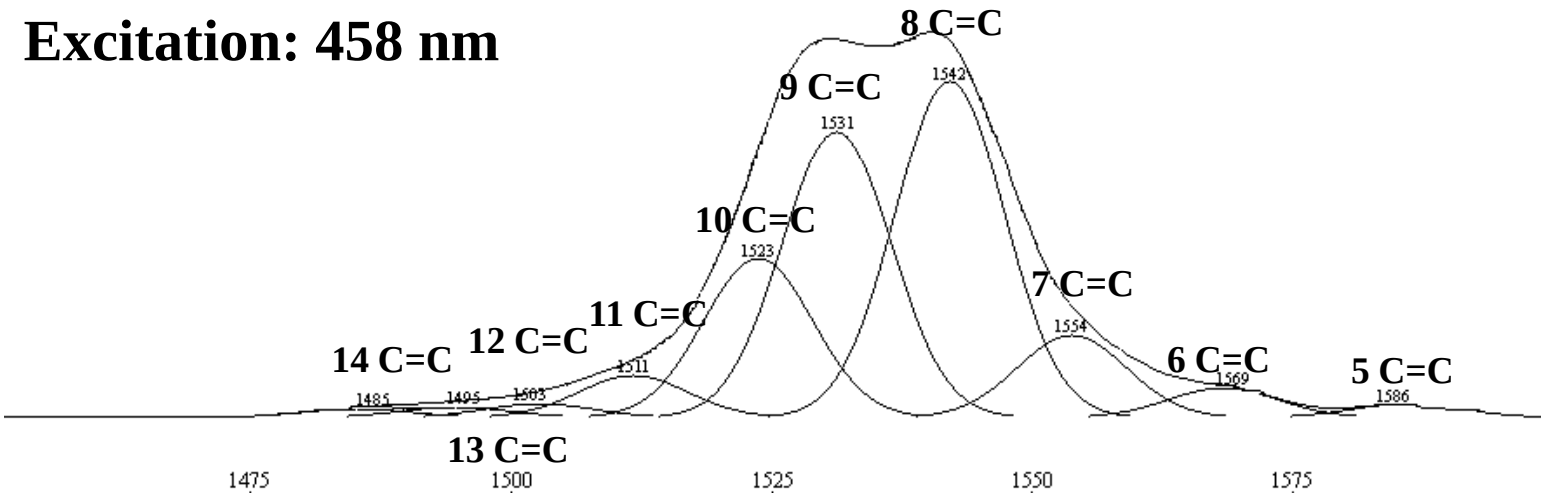
Barnard et al. 2006: ν_1=97.07ln(1/n)+1745 cm^{-1} for 3≤n≤12

n: number of double bonds in the polyenic chain.

Methods/Raman spectroscopy

Decomposition with constraints (using the same position and width for each component, and allowing only intensity to vary) of C=C stretching band.

Excitation: 458 nm



R-(-CH=CH-)$_n$-R' n=5-14

Methods/Raman spectroscopy

Natural-color FWCP

The same pearl may contain up to 10 pigments with *n*= 5-14.

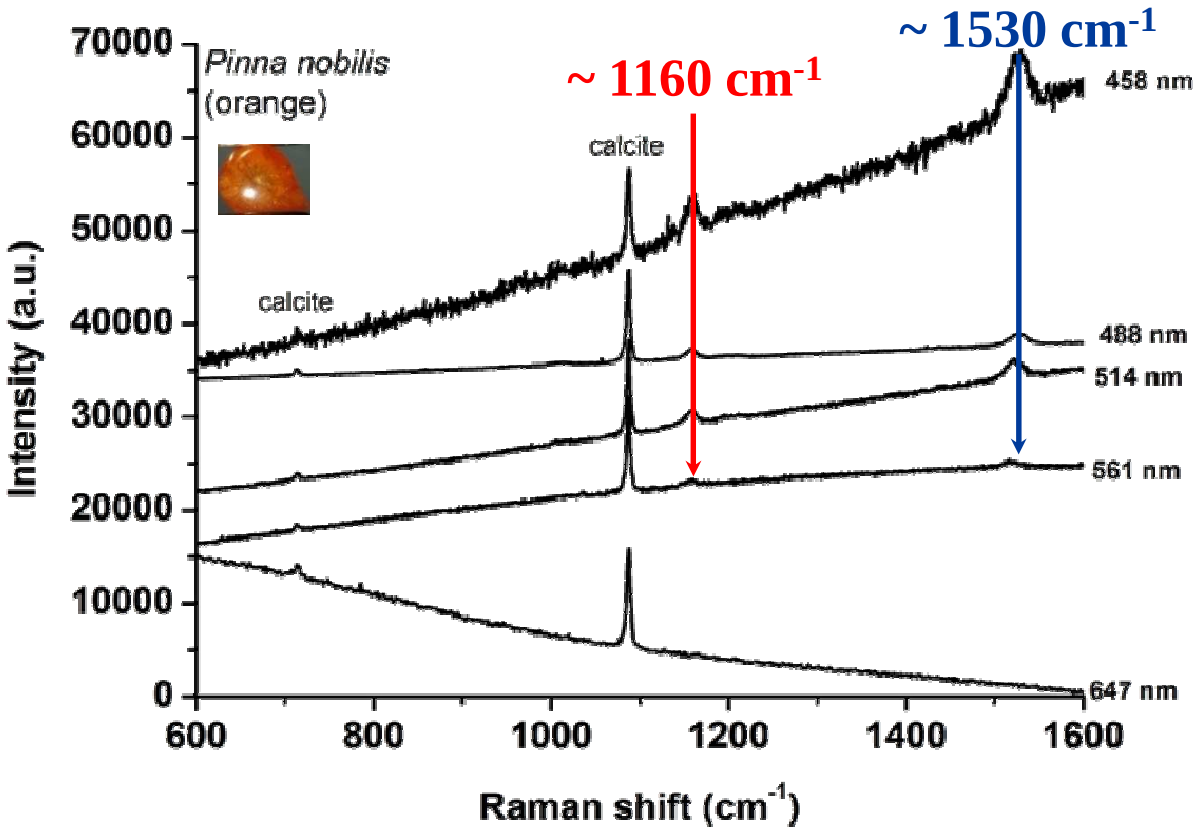
Different colors are explained by different mixtures not by the change of a single pigment.

Akamatsu et al., 2001

Similar measurements have shown that 15 species of pearls and/or the inner part of pearl producing mollusk contain “simple” polyenic molecules.

Methods/Raman spectroscopy

Mollusk identification



Peak at about 1160 cm^{-1} , thus carotenoid pigments.

Changing the excitation wavelength, variations in the position and shape of the two most intense bands are noted. Color is due to a mixture of carotenoids, not to a single pigment. The only pearl which contain carotenoid pigments.

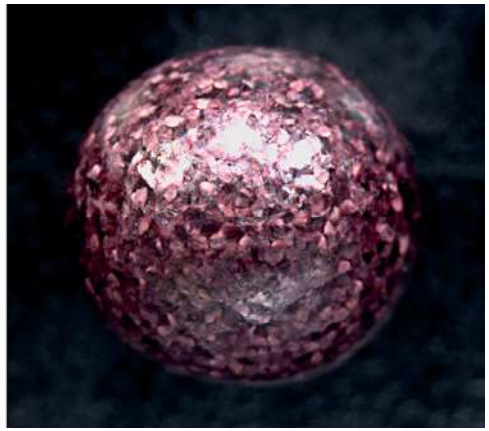
Methods/Raman spectroscopy

Mollusk identification

The only calcitic pearls with polyenic pigments

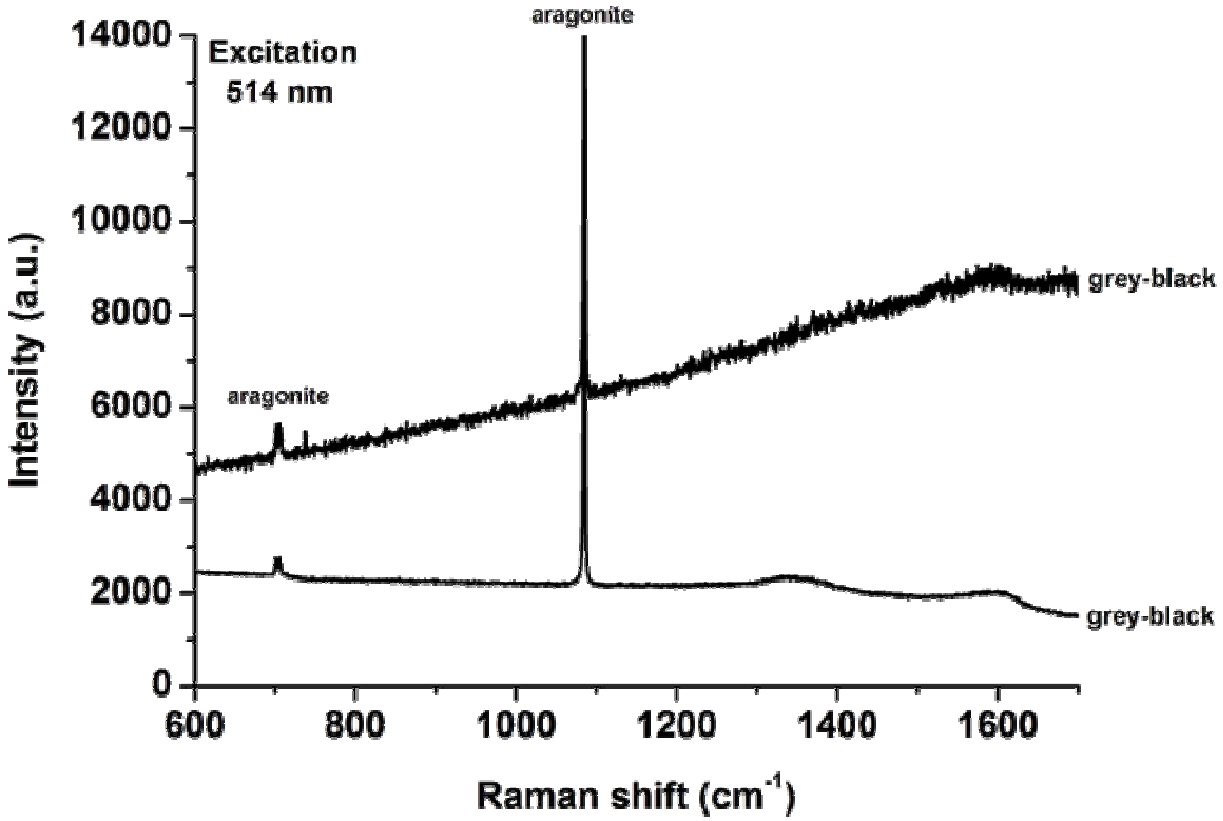
Pectenidae family

Scallop pearls



Methods/Raman spectroscopy

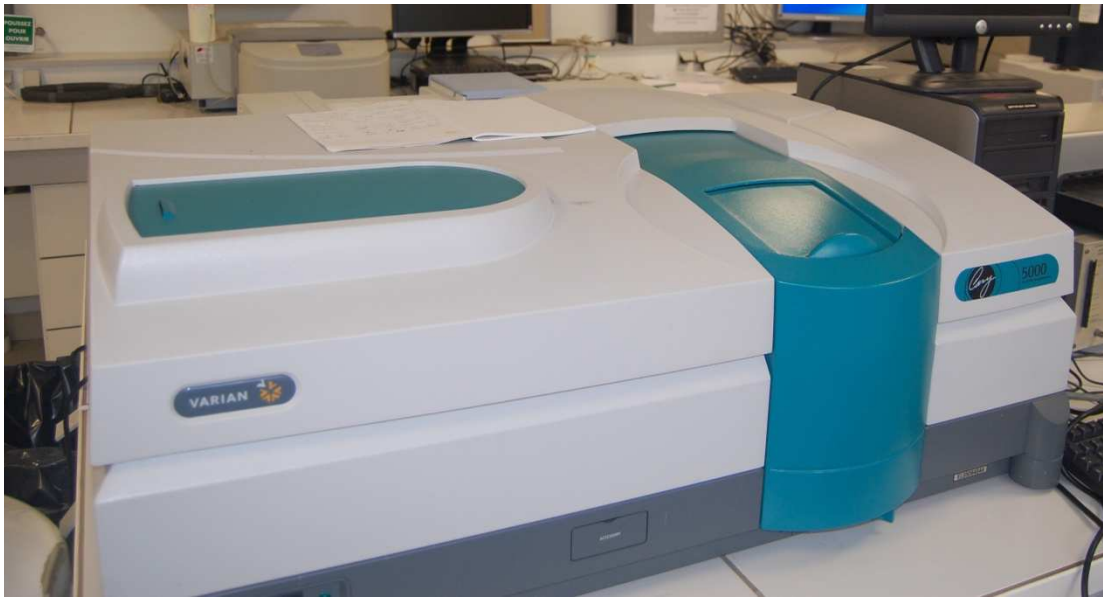
Treated-color FWCP



Some color-treated pearls show peaks at about 1350 and 1600 cm^{-1}. These peaks are characteristic of disordered carbon and it is probably because of pearls' organic matter decomposition.

Analysis/UV-Vis-NIR

UV-Vis-NIR Diffuse Reflectance Spectroscopy



Color authenticity...

Coating...

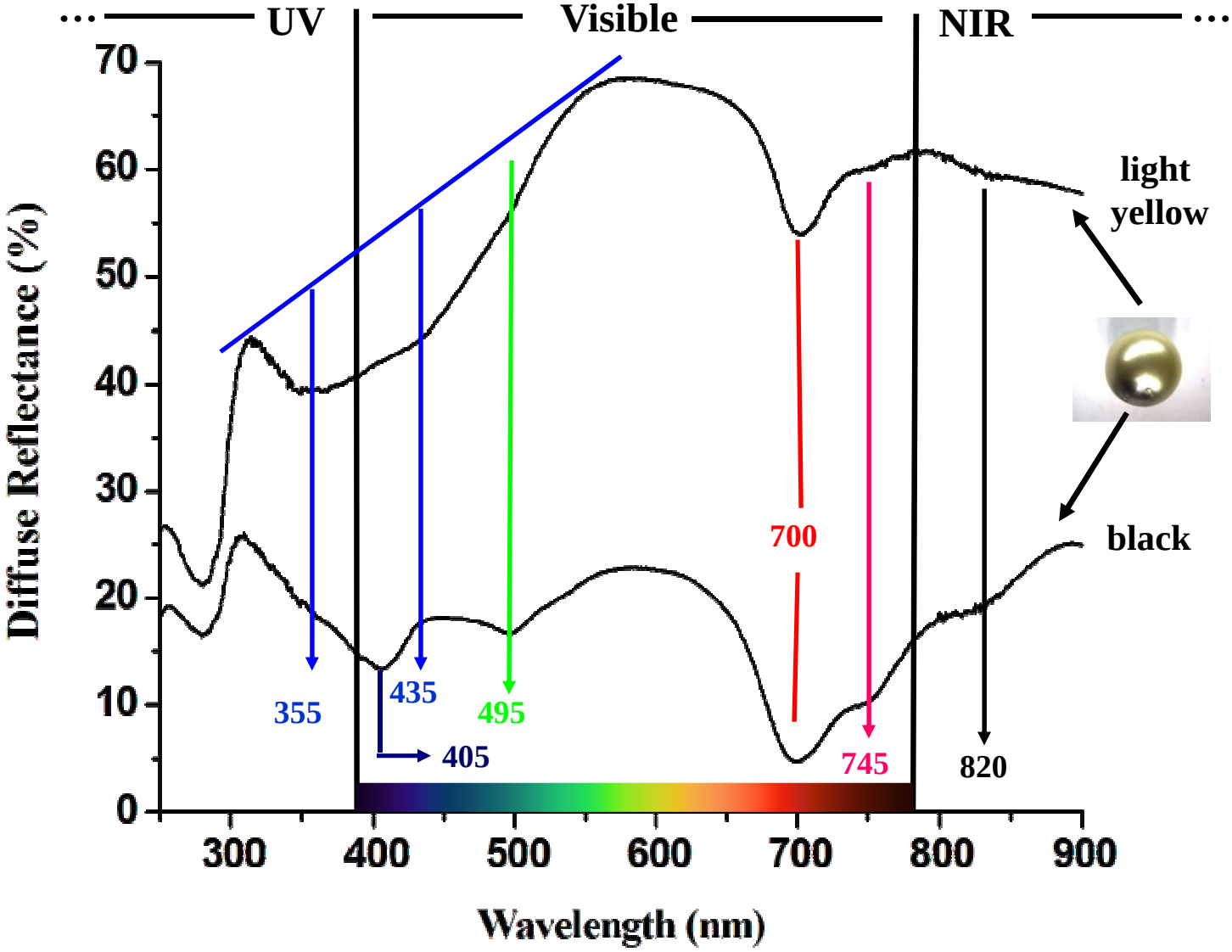
Bleaching...

Mollusk indetification...

Analysis/UV-Vis-NIR

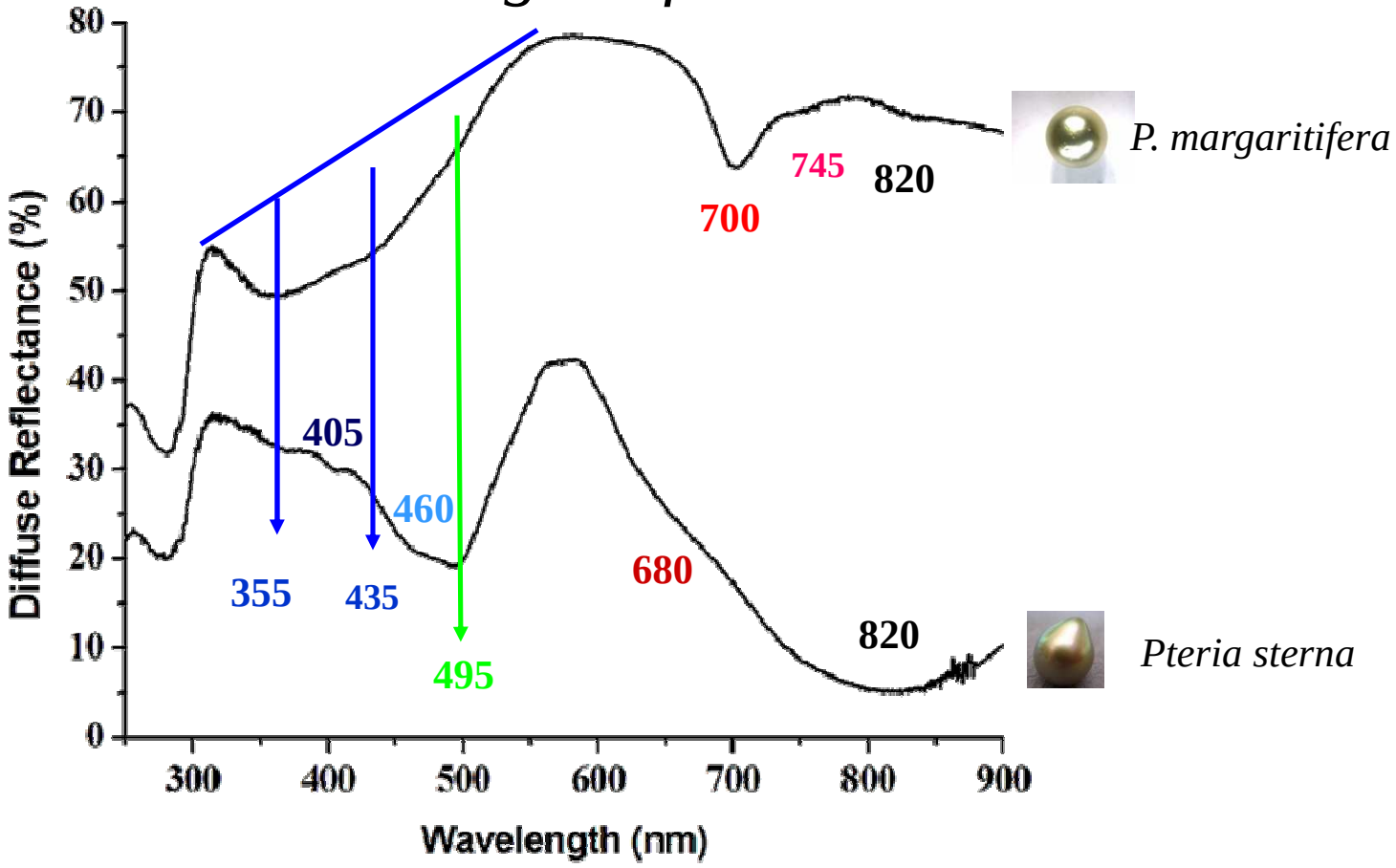
P. margaritifera

How to read diffuse reflectance spectra



Results / UV-Vis-NIR

Pteria sterna vs *P. margaritifera*

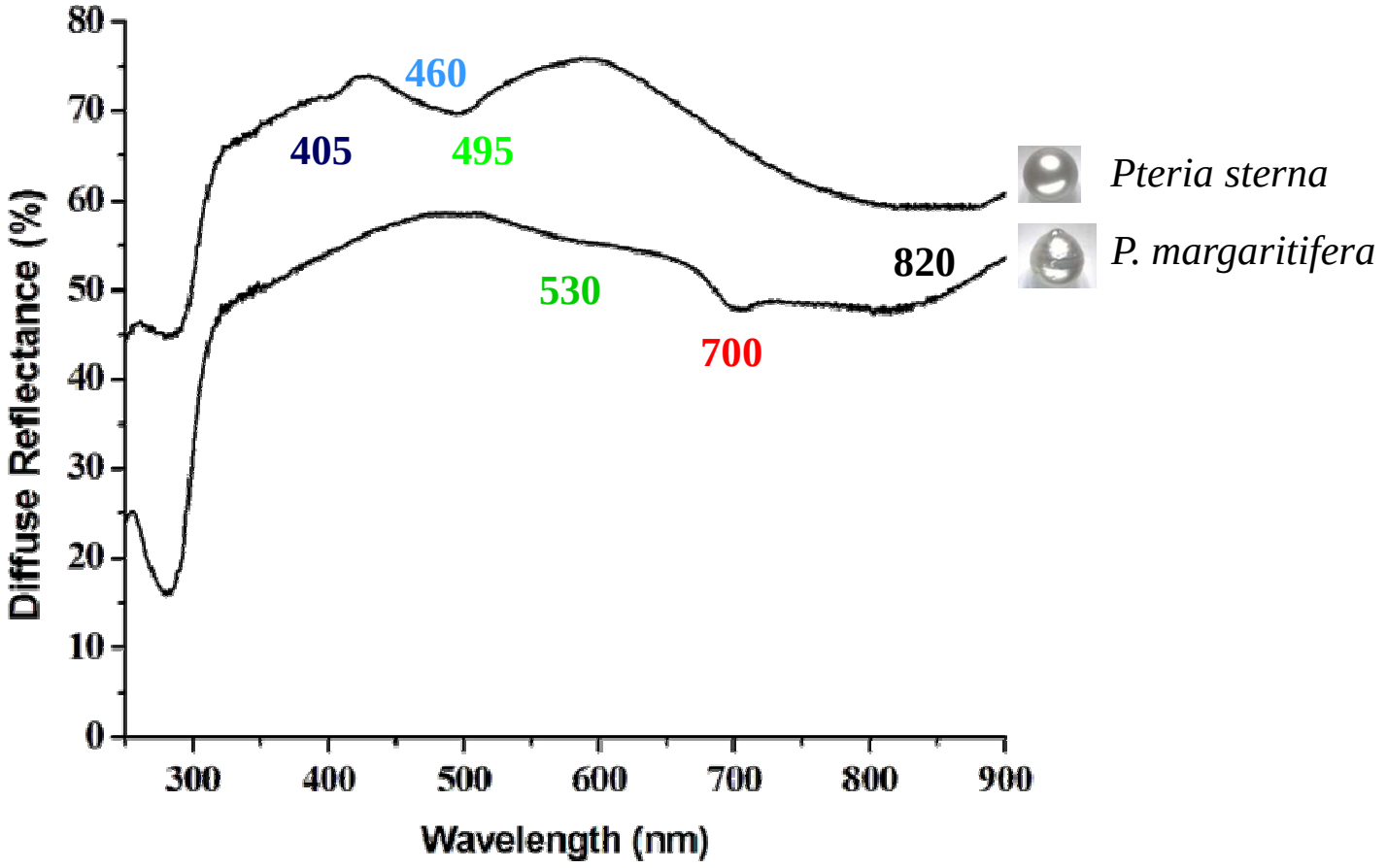


Both samples' colors are due to different absorptions.

No absorption at 700 nm on *Pteria sterna*'s.

Analysis/UV-Vis-NIR

Pteria sterna vs *P. margaritifera*



Both samples of white color could show some weak absorptions.
No absorption at 700 nm on *Pteria sterna*'s.

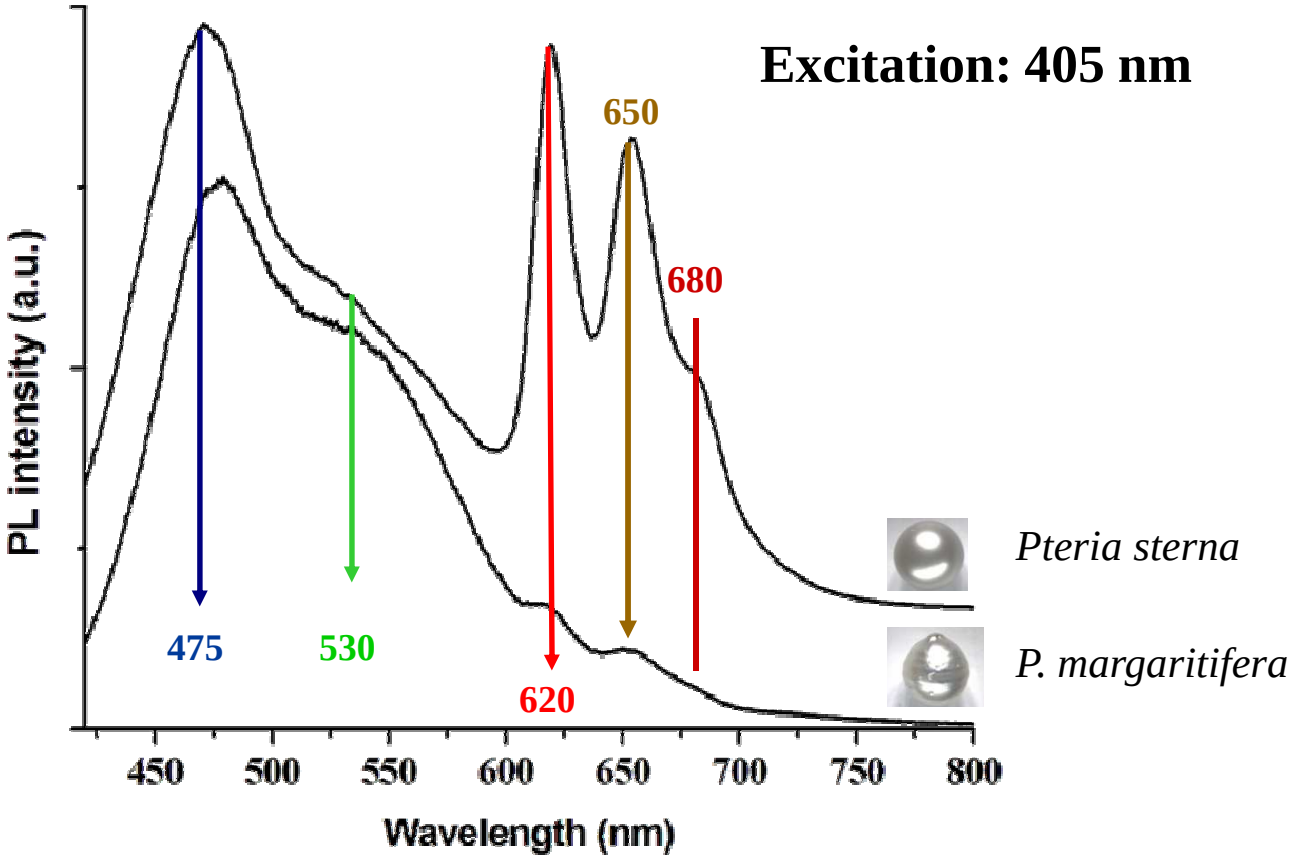
Analysis/Photoluminescence

Mollusk indetification...

Color authenticity...

Analysis/Photoluminescence

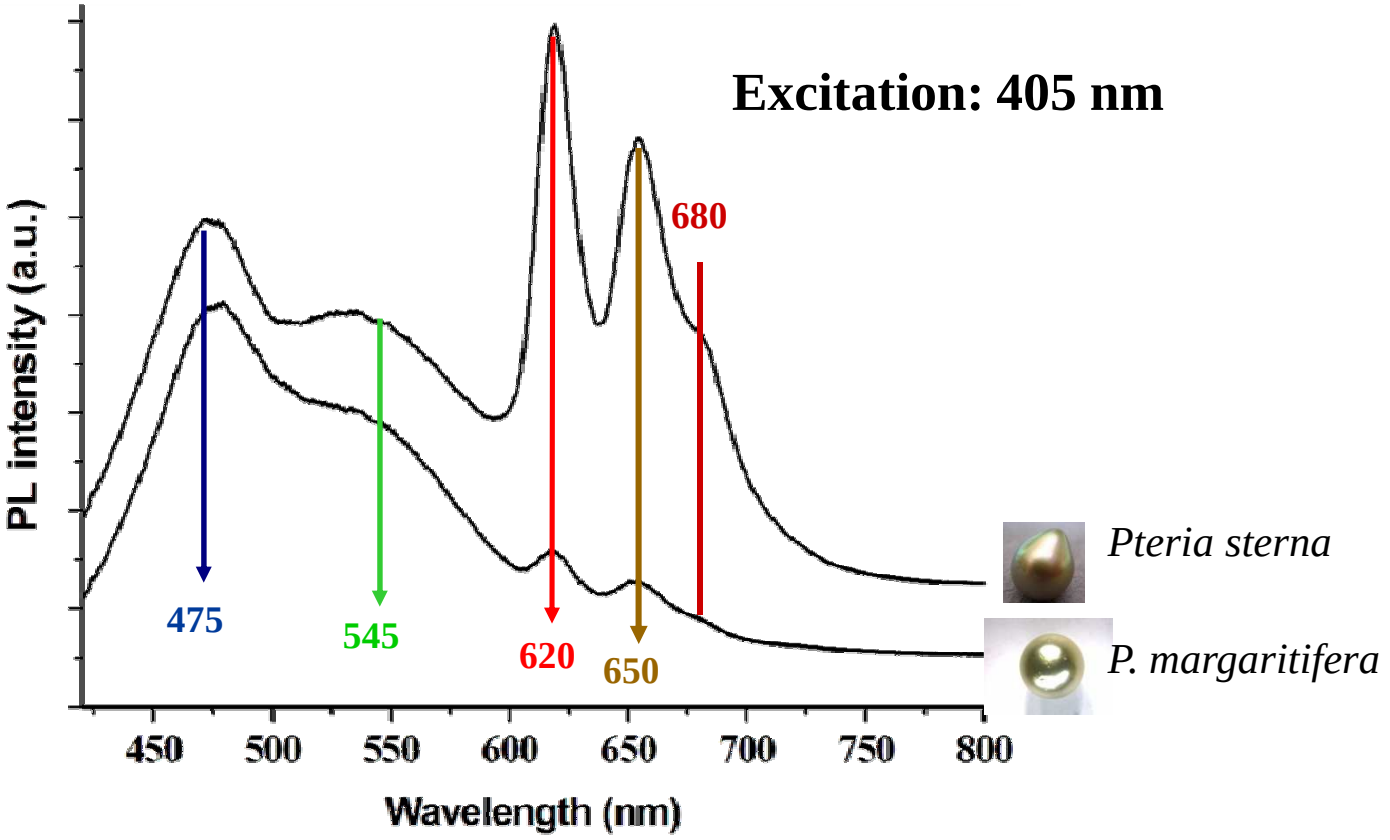
Pteria sterna vs *P. margaritifera*



The **PL in the blue part of *P. margaritifera* is most important** than those observed **in the red part**. Slight differences in the exact positions of PL bands (e.g. 475 nm, 530 and 620 nm).

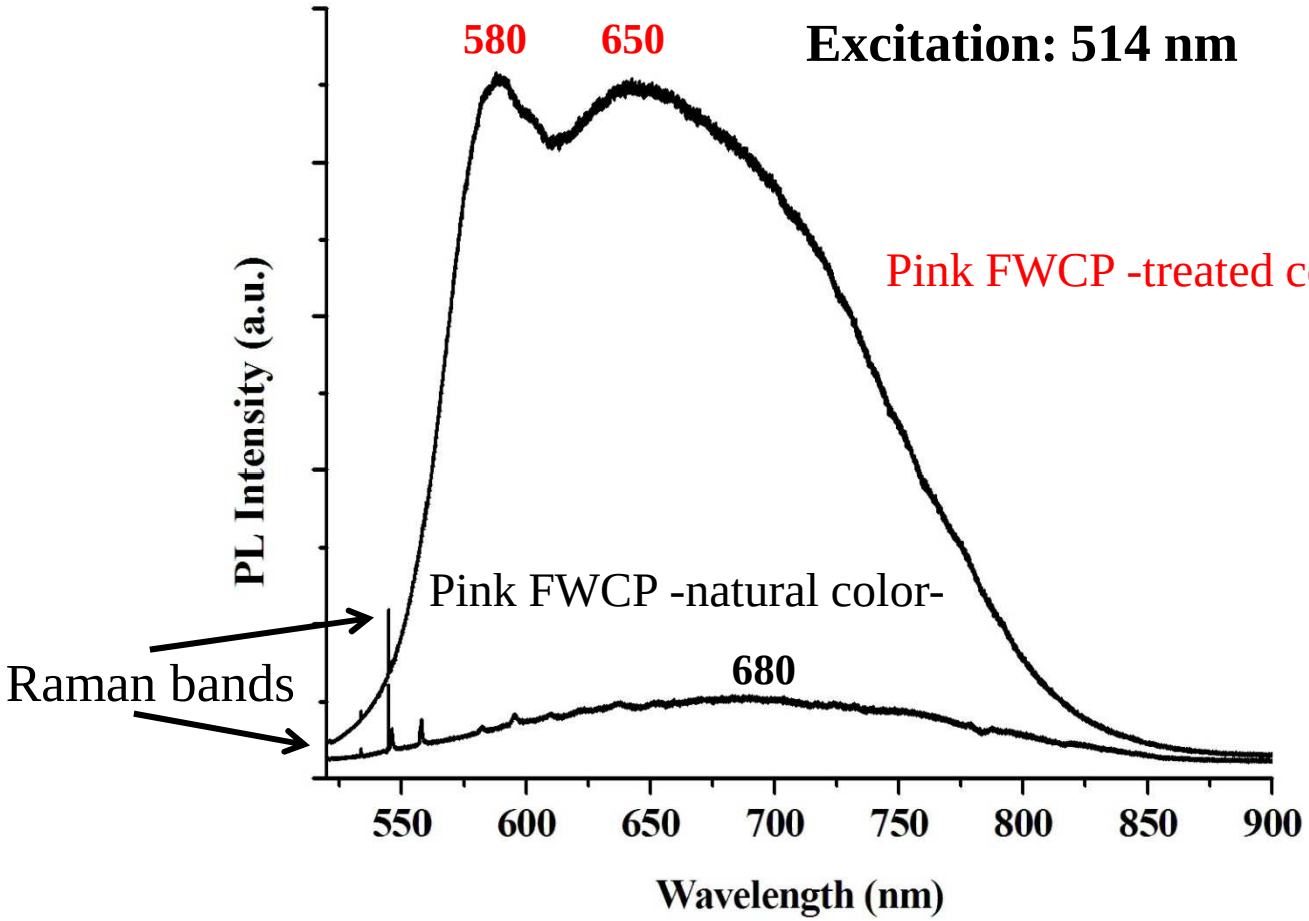
Analysis/Photoluminescence

Pteria sterna vs *P. margaritifera*



The **PL in the blue part of *P. margaritifera* is most important** than those observed **in the red part**. Slight differences in the exact positions of PL bands (e.g. 475 and 545 nm).

Analysis/Photoluminescence



The PL spectra of **natural color FWCPs** (large band centered at the red part **-680 nm-**; overtones because of the natural pigment) are different compared to treated-color FWCPs.

Analysis/Quality control of CP

Autore Five S's

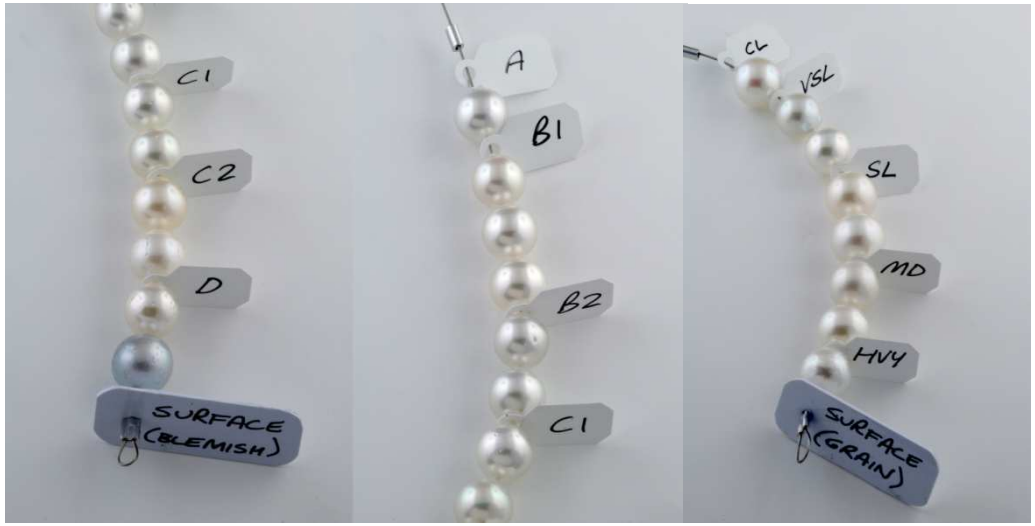
Shine *(lustre)*

Surface *(surface)*

Shade *(couleur)*

Shape *(forme)*

Size *(grandeur)*



Analysis/Quality control of CP

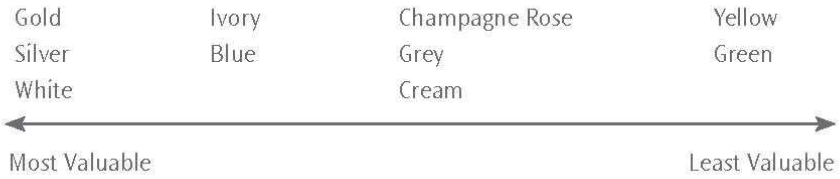
GÜBELIN GEM LAB

Autore Five S's

Shine *(lustre)*

Surface *(surface)*

Shade *(couleur)*

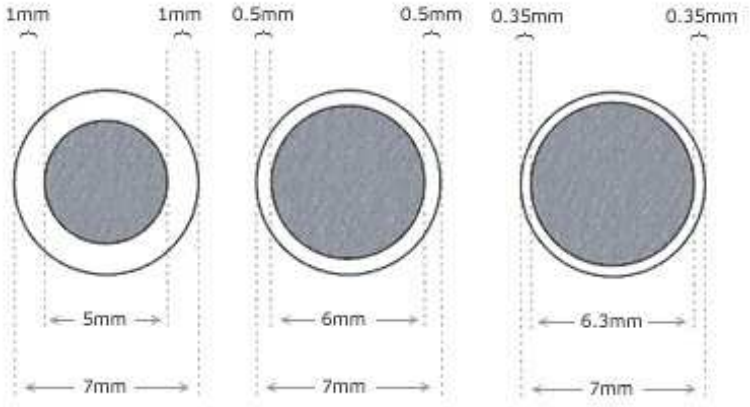


Shape *(forme)*

Size *(grandeur)*

Shapes: round, near round, drop, baroque

Analysis/Quality control of CP

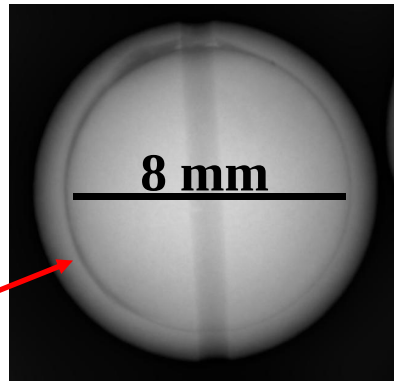


Estimation of nacre thickness depend of the host mollusk; e.g. excellent for:

P. maxima is >2.5 mm

P. margaritifera is >1.8 mm nacre thickness

P. fucata is >1.2 mm nacre thickness

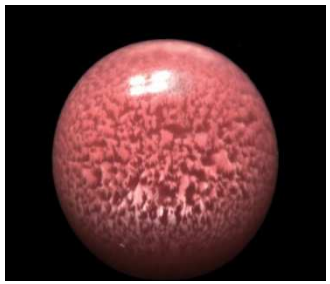


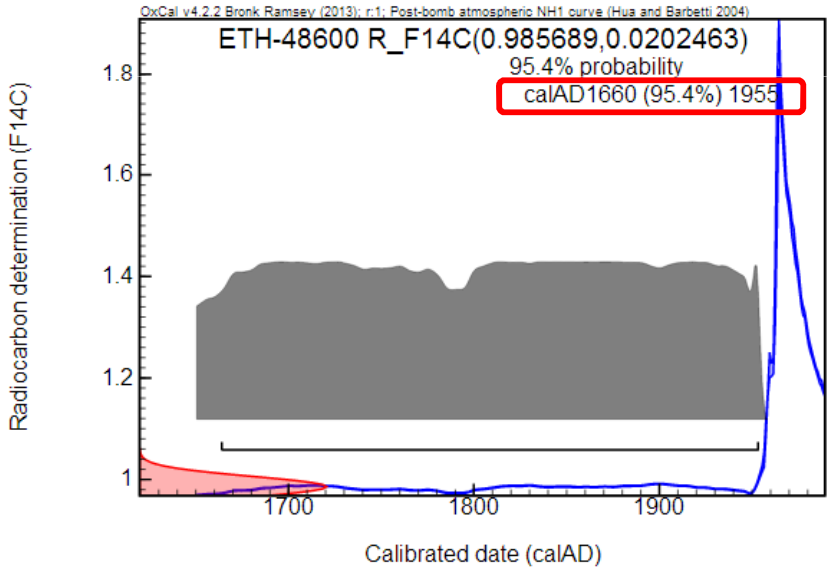
New approaches

Several relatively new methods were applied with limited applications (micro-destructive):

^{14}C age determination: Pearl from *Strombus gigas* (Queen conch pearls -CITES protected from 2006-) fished in Caribbean sea before 1970.

Strombus sp.

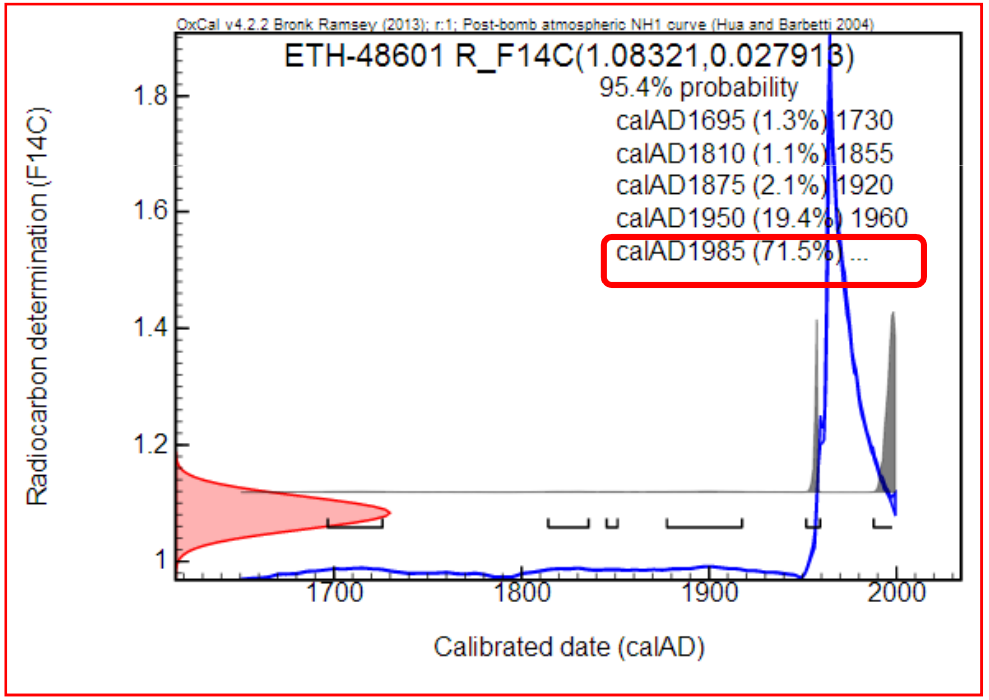




Queen conch natural pearl

New approaches

^{14}C age determination: Natural pearl from *Pinctada radiata* **fished** in **Arabian gulf** on **2011**.

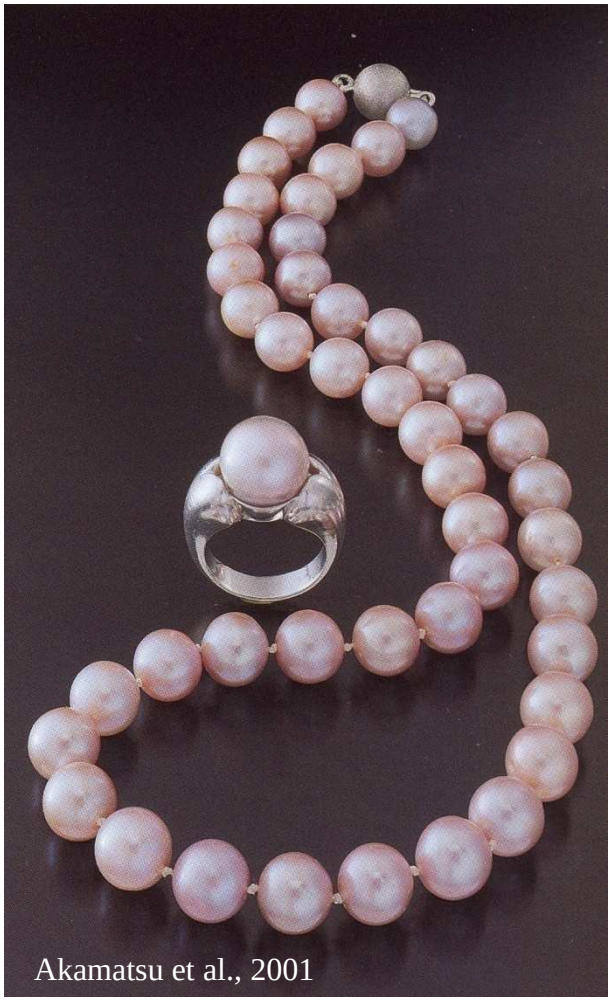


^{14}C age determination: Impossible for FW pearls

Present

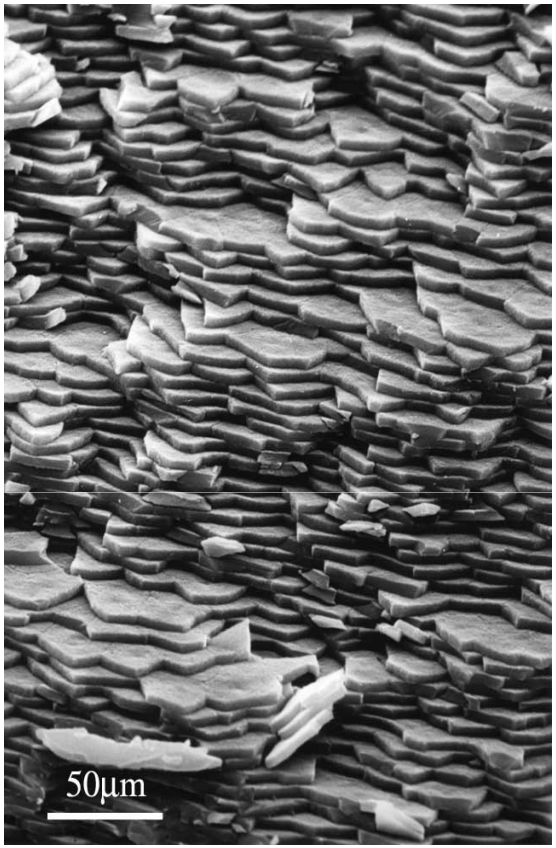
- Today **pearl testing is** more challenging and more **complicated** than ever.

- **Several methods have to be applied for pearl testing** (e.g. for a colored FWCP characterisation it is needed : X-radiography, EDXRF, X-ray luminescence, Raman spectroscopy, UV-Vis spectroscopy), high cost and time consuming…


Akamatsu et al., 2001

Wrap up

- Only **highly specialized labs have** the **instrumentation** can issue pearl reports.
- Samples with known origin (mollusk, year of cultivation etc.) needs to be re-studied
- **New methods are coming**…



Certificate

Is it worth a certificate?

From: Christie's

Two row necklace of 68 (35+33) natural SWpearls, sold about 6m euros

From: Christie's

59.92 ct natural SW pearl 200k euros


Thank you

Acknowledgments

- Centre de Recherche Gemmologique (CRG, Nantes, France)
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-Pr. Emmanuel Fritsch (University of Nantes, France)
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- Abeer Tawfeeq Al-Alawi (GPTLB, Manama, Bahrain)
- Georrge Bosshart (†)

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More information: s.karampelas@gubelingemlab.ch

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