# SPECTRAL REFLECTANCE AND FLUORESCENCE CHARACTERISTICS OF NATURAL-COLOR AND HEAT-TREATED "GOLDEN" SOUTH SEA CULTURED PEARLS

#### By Shane Elen

A comparison study was made between the yellow and white nacre of the gold-lipped *Pinctada maxima* oyster shell and 65 yellow cultured pearls, both natural and treated color, produced from this mollusk. The yellow nacre of this shell has a characteristic absorption feature in the UV region between 330 and 385 nm; the strength of this feature increases as the color becomes more saturated. White shell nacre fluoresces very light blue or very light yellow to long-wave UV radiation, whereas yellow shell nacre fluoresces greenish to brownish yellow or brown. Natural-color yellow cultured pearls from *P. maxima* exhibited absorption and fluorescence characteristics similar to those of the yellow shell nacre. In contrast, the absorption feature in the UV was either weak or absent in yellow cultured pearls reportedly produced by a method involving heat treatment, and their fluorescence was generally very light blue or light yellow.

he popularity of "golden" cultured pearls from the South Seas (figure 1) has increased steadily over the last 10 years (Vock, 1997; "Prices of golden ...," 2000). As demand for these cultured pearls has grown, treated-color "golden" cultured pearls have also entered the marketplace. A portion of the South Sea yellow cultured pearls harvested are well shaped with few blemishes but with less desirable color-either very light (Federman, 1995) or uneven. The most common methods used to produce or enhance yellow coloration in South Sea "golden" cultured pearls involve treatment with a chemical or an organic dye. The fact that so many different chemicals and dyes could be used for this purpose complicates a comprehensive study of these treatment methods. However, inorganic chemicals are routinely identified using EDXRF; and dyes, since they usually are applied after the pearls are drilled, generally are easy to identify with magnification.

The Ballerina Pearl Co. (New York City) developed a method in the early 1990s to treat these poorly colored South Sea cultured pearls to produce a more uniform and desirable yellow. These were first marketed in 1993 with full disclosure that they were treated (Vock, 1997). Details of the proprietary process are not known, but it has been reported that the treatment, which is believed to be stable, is applied to undrilled cultured pearls and involves the use of heat without the application of dyes or bleaching (Vock, 1997). Inasmuch as the report did not exclude chemicals other than bleach, it is possible that chemicals also may be involved in the treatment. It is probable that other companies are treating "golden" pearls using a similar process.

The challenge for the gem and jewelry industry is to separate natural-color cultured pearls, of any color, from treated ones (Sheung, 1998). Although, as noted above, drilled treated cultured pearls generally can be distinguished from natural-color cultured pearls by microscopy (Hargett, 1989), undrilled cultured pearls—especially in the absence of obvious visual features—are much

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Figure 1. These natural-color and heattreated cultured pearls range from 12.5 to 13.6 mm: the natural-color cultured pearls are on the top right and bottom left. Note the area of "golden" nacre along the periphery of the white nacreous region in the goldlipped Pinctada maxima shell (18.0 cm in diameter). Photo by Maha Tannous.

more difficult to identify. The present report compares heat-treated yellow cultured pearls to natural shell nacre and natural-color yellow cultured pearls from the *Pinctada maxima* to identify criteria that can be used to separate treated from natural-color material, especially when the samples are undrilled.

#### BACKGROUND

The gold-lipped *P. maxima* oyster has a characteristic yellow to "golden" nacre inside the shell along the periphery of the white nacreous region (Gervis and Sims, 1992, p. 4; again, see figure 1). Pearls produced from this oyster, either natural or cultured, are typically white, "silver," or yellow (South Sea Pearl Consortium, 1996) and include so-called "golden" pearls.

The post-harvest color treatment of cultured pearls, of any color, falls into two categories: prior to drilling and after drilling. Heat treatment (Vock, 1997) and gamma irradiation (Ken-Tang Chow, 1963) may be performed pre- or post-drilling. As noted above, however, dyes and chemicals typically are used only after drilling (Komatsu, 1999), to facilitate their entry parallel to the nacre layers. Generally, such treatments affect the organic material (i.e., conchiolin) between the nacre layers; they have less influence on the crystalline nacre (Gauthier and Lasnier, 1990). The most obvious indication of these treatments (with the exception of irradiation) is an unusual color concentration in the form of a colored layer (visible in the drill hole) or a colored spot or streak visible on the surface (Komatsu, 1999). Color from dyes or chemicals typically becomes concentrated in surface defects such as cracks, pits, or blemishes (Newman, 1999, p. 94). Dimples or protrusions in the nacre are particularly likely to exhibit concentrations of color. These areas are often more porous (as indicated by a cloudy or milky appearance prior to treatment), which allows the dye or chemical to become concentrated (T. Moses, pers. comm., 2000). These features sometimes are eye-visible, but usually they can be detected only with microscopy.

Typically, detection of a treated yellow color in undrilled cultured pearls relies on the presence of surface color concentrations. When such features are absent, however, identification is more of a challenge. Inasmuch as the Ballerina process is applied prior to drilling, this is the situation for yellow cultured pearls treated by this technique.

Due to the nature of the chemicals and dyes typically used in most post-drilling treatments, many color-treated natural and cultured pearls can be identified using X-ray fluorescence (XRF) or Raman spectrometry (see box A). However, the author did not find these techniques useful for the heat-treated "golden" pearls tested for this study. Thus, the pur-

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# BOX A: ADVANCED IDENTIFICATION OF COLOR TREATMENTS IN NATURAL AND CULTURED PEARLS

Advanced testing often can be used to identify colortreated cultured pearls. These techniques include Xray fluorescence (XRF), Raman and luminescence spectrometry, and UV-Vis spectrophotometry. XRF is useful for detecting the presence of inorganic treatments such as silver salts (black) or iodine (yellow; figure A-1). In most cases, organic treatments and biological pigments cannot be detected by this method, but their presence can sometimes be determined using Raman spectrometry (figure A-2). UV-

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Figure A-1. The iodine (I) peaks in this energydispersive X-ray fluorescence (EDXRF) spectrum of a yellow Akoya cultured pearl indicate chemical treatment.



Figure A-3. In a UV-Vis (ultraviolet-visible) reflectance spectrum, an absorption maximum at 700 nm is characteristic of natural-color black cultured pearls from the Pinctada margaritifera oyster.

Vis spectrophotometry frequently is used to identify the presence of chromophores responsible for natural coloring in Tahitian black cultured pearls, as indicated by an absorption at 700 nm (Komatsu and Akamatsu, 1978; figure A-3). Luminescence can verify the presence of porphyrins (Miyoshi et al., 1987) responsible for the reddish brown fluorescence observed in natural-color black cultured pearls originating from the *Pinctada margaritifera*, *Pteria sterna*, and *Pteria penguin* oysters (figure A-4).



Figure A-2. In this Raman spectrum of an orangy yellow Akoya cultured pearl, the series of peaks above 1085 cm<sup>-1</sup> indicate treatment with an organic dye.



Figure A-4. This luminescence spectrum of a cultured black pearl from Pinctada margaritifera shows fluorescence peaks at 617 and 676 nm due to porphyrins, which are responsible for the reddish brown fluorescence to 365 nm UV or 405 nm blue light.



Figure 2. These five samples, each about 9 mm in diameter, show the range of color seen in the Pinctada maxima shell nacre samples. Photo by Maha Tannous.

pose of the present research was to determine other nondestructive spectral or fluorescence characteristics that could be used to aid in the identification of heat-treated yellow cultured pearls from the *P*. *maxima* that either are undrilled or have drill holes that are inaccessible for examination.

For convenience and brevity, natural-color yellow cultured pearls will henceforth be abbreviated as NYCPs, and reportedly heat-treated yellow cultured pearls as HTYCPs. Note, too, that the hue, tone, and saturation terms used to describe the colors of the shell and cultured pearl samples are based on the new (2000) GIA pearl grading system.

#### MATERIALS AND METHODS

To establish the characteristics of known *untreated* material, we obtained a total of 42 samples of nacre from seven gold-lipped *P. maxima* shells that originated from Australia (2 shells), the Amami Islands in Japan (2), and the Philippines (3). The samples, each approximately 9 mm in diameter, were removed with a diamond saw while the shells were completely immersed in water, since overheating could affect their color (Webster, 1994) and thus their fluorescence or spectral characteristics. Two samples of white nacre and four samples of yellow nacre were obtained from each shell, for a total of 14 white nacre and 28 yellow nacre samples.

In addition, 53 NYCPs, ranging from 9.1 to 15.7 mm, were obtained from several reputable sources. They were reportedly from Indonesia (5), the Philippines (23), Japan (3), Australia (6), and the "South Seas" in general (16). Forty-five were undrilled and eight were drilled. Twelve undrilled treated-color yellow cultured pearls, which were reportedly heat treated, were also obtained for the study; they ranged from 11.8 to 13.1 mm. We believe that these samples originated from the gold-lipped variety of the *P. maxima*; we could not confirm whether the process has been applied to cultured pearls from the silver-lipped *P. maxima*. No strongly



Figure 3. The natural-color yellow cultured pearls ranged from very light yellow to orangy (as shown here) or greenish yellow. These pearls are 11.6–13.6 mm in diameter. Photo by Maha Tannous.

saturated yellow cultured pearls of known pedigree, natural or treated, were available for characterization.

All of the cultured pearls were examined with a GIA Gem Instruments Mark VII microscope using fluorescent and fiber-optic lighting. Initially six NYCPs and six HTYCPs were analyzed with a Renishaw 2000 Ramascope laser Raman microspectrometer and a Thermo Noran Spectrace 5000 EDXRF spectrometer. Inasmuch as neither technique revealed any distinct differences between the natural- and the treated-color samples, further testing by Raman and EDXRF was discontinued.

UV-Vis reflectance spectra and fluorescence observations were obtained for each sample using a Hitachi 4001 spectrophotometer and a UVP model B100 AP long-wave ultraviolet lamp. Reflectance spectra were collected from 250 to 2500 nm, although only the pertinent data range (i.e., 250-700 nm) is illustrated in this article. At least two spectra were obtained in different regions for each cultured pearl that showed uneven color or fluorescence distribution. A total of 162 reflectance spectra were collected: 42 on the shell nacre samples, 94 on the natural-color cultured pearls, and 26 on the treated cultured pearls. Acquisition of luminescence spectra with a Spectronic AB2 luminescence spectrometer was unsuccessful, due to instrument configuration, sample shape, and the generally desaturated fluorescence colors. Therefore, fluorescence color and distribution were observed visually in a darkened room with the aid of UV contrast goggles.

## RESULTS

**Visual Appearance.** Fourteen of the shell nacre samples were white, and 28 ranged from light to dark yellow (see, e.g., figure 2). The natural-color cultured pearls ranged from very light yellow to orangy or greenish yellow (see, e.g., figure 3); a few exhibited slightly uneven color distribution. The HTYCPs ranged from light yellow to orangy yellow (see, e.g., figure 4), with the majority being light yellow.

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Figure 4. The heat-treated yellow cultured pearls ranged from light yellow to moderate orangy yellow. The samples shown here are 11.3–12.6 mm in diameter. Photo by Maha Tannous.

Small, localized spots of concentrated color were visible in 10 of the 12 HTYCPs, with either the unaided eye or magnification (figure 5). The remaining two did not exhibit any visible or microscopic evidence of treatment.

UV-Vis Reflectance Spectra and Fluorescence. Shell Nacre Samples. The UV-Vis spectra revealed a decrease in reflectance due to absorption between 330 and 460 nm for all 28 yellow shell nacre samples as compared to the samples of white shell nacre. This broad region of absorption is actually composed of two features: one in the UV region from 330 to 385 nm, with a maximum between 350 and 365 nm; and the other in the visible region from 385 to 460 nm, with a maximum between 420 and 435 nm. The spectra of the white shell nacre samples showed no absorption features in this broad region. Examination of the reflectance spectra for a series of five samples that ranged from white to dark yellow revealed an increase in the general absorption between 330 and 460 nm as the strength of the yellow coloration increased (figure 6).

Typically, dark yellow shell nacre exhibited moderate brown, greenish brown, or greenish yellow fluorescence, and light yellow shell nacre fluoresced light brown or light yellow (see, e.g., figure 7). Most of the white shell nacre samples showed moderate-to-strong very light blue fluorescence, although some appeared to exhibit a slight trace of yellow.

*Natural-Color Cultured Pearls.* The spectra for these samples (figure 8) exhibited absorption characteristics similar to those of the yellow shell nacre, except that the maximum in the UV varied from 350 to 385 nm. All but four of the 94 spectra obtained on these samples exhibited the two absorption features between 330 and 460 nm. In the four spectra that did not show these features, the regions



Figure 5. Small spots of concentrated color are evident within blemishes on this yellow cultured pearl that reportedly has been heat treated. Photomicrograph by Shane Elen; magnified 5×.

analyzed were white areas on unevenly colored samples; and these results were similar to the reflectance spectra obtained for white shell nacre. As the yellow color of the cultured pearls increased in saturation, the long-wave UV fluorescence progressed from light yellow or light brown, to greenish yellow, greenish brown, or brown.

Heat-Treated Cultured Pearls. Five of the 26 reflectance spectra obtained from the HTYCPs

Figure 6. These reflectance spectra of five shell nacre samples—ranging from white through dark yellow—show increasing absorption from 330 to 460 nm as the color intensifies. Note in particular the increasing absorption in the UV region from 330 to 385 nm.





Figure 7. The typical fluorescence reactions of natural- and treated-color cultured pearls when exposed to 365 nm (long-wave) UV radiation are illustrated here. Shown in normal lighting (left in each pair) and with long-wave UV (right) are: (A) white shell nacre, (B) yellow shell nacre, (C) 13.6 mm natural-color light yellow cultured pearl, (D) 12.5 mm natural-color orangy yellow cultured pearl, (E) 12.6 mm heat-treated light yellow cultured pearl, and (F) 12.6 mm heat-treated orangy yellow cultured pearl. Normal lighting photos by Maha Tannous; fluorescence photos by Shane Elen.

exhibited a weak absorption feature in the UV region around 345 nm (see, e.g., figure 9). These spectra represented five of the 12 treated cultured pearls studied. Spectra taken from other areas on these same five HTYCPs, and from the remaining seven HTYCPs, exhibited very weak or no absorption in the UV region. All 26 spectra showed an absorption feature in the blue region between 415 and 430 nm.

Generally, the fluorescence of the HTYCPs was slightly uneven, and appeared unrelated to the dis-

Figure 8. As was the case with the shell nacre samples, the reflectance spectra of the natural-color yellow cultured pearls showed increasing absorption in the 330–460 nm region with greater saturation of color.



tribution of bodycolor. In one HTYCP, however, fluorescence did appear to be related to the uneven distribution of the yellow color: Light yellow and light brown fluorescence corresponded directly to areas of light yellow and light orangy yellow. Eight of the HTYCPs, which were light in tone and saturation, fluoresced moderate to strong light yellow; two—also light in tone and saturation—exhibited a very light blue fluorescence. The sample with the

Figure 9. The reflectance spectra of the heat-treated yellow cultured pearls revealed very weak or no absorption in the 330–385 nm region. The presence of a weak UV absorption feature around 345 nm suggests that the orangy yellow cultured pearl exhibited some natural yellow coloration prior to treatment.



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Figure 10. These reflectance spectra of natural and treated light yellow (top) and orangy yellow (bottom) cultured pearls show a distinct difference in absorption in the 330–385 nm region. Note the lack of absorption in this region shown by the treated-color samples.

most saturated orangy yellow bodycolor fluoresced a light brownish orange. The spots of concentrated color seen in some HTYCPs (see figure 5) fluoresced a strong orange to long-wave UV.

#### DISCUSSION

**Confirmation of Reported Origin of Color.** One of the main challenges of performing a study of this kind is obtaining samples of known species and color origin. Ideally for pearls of natural color, witnessing the removal of the pearl from the oyster would be the method of choice. However, this is not always practical, so instead we must look to very reliable sources. Similarly, obtaining pearls treated by specific methods also can be very challenging.

None of the cultured pearls represented as being of natural yellow color showed any visible indications of treatment, whereas most of the cultured pearls reported to be heat treated did exhibit some visible evidence in the form of spots with concentrated color. In most cases, too, there was a distinct difference in reflectance spectra between yellow cultured pearls stated to be natural color and those that were reportedly heat treated. In addition, those stated to be natural color exhibited reflectance and fluorescence characteristics similar to the samples of shell nacre of similar color. These findings would appear to support the reported color origins for the cultured pearl samples used in this study.

Although no chemical study of the zoochrome (a naturally occurring pigment molecule found in the animal kingdom; Needham, 1974) was performed, the reflectance and fluorescence results indicate that the yellow coloration in *P. maxima* shells and NYCPs appears to be due to a common zoochrome regardless of their geographic origin.

Spectral Reflectance Characteristics. A broad absorption between 330 and 460 nm in the reflectance spectra was seen in the yellow nacre from both the gold-lipped P. maxima shell and the NYCP samples (figures 6 and 8), and thus appears to be characteristic of natural-color vellow nacre produced by this species. The strength of this absorption appeared to increase as the color became more saturated. The 420-435 nm maximum (in the blue region of the visible spectrum) is typical of reflectance spectra for yellow objects (Lamb and Bourriau, 1995), and is responsible for the yellow color observed. Although UV absorption is not necessary to produce yellow coloration, it does appear to be related to the same zoochrome that is responsible for the absorption in the blue region of the visible spectrum. The absence of this absorption maximum in the UV reflectance spectra obtained from white areas on unevenly colored cultured pearls is consistent with the reflectance results obtained for white shell nacre.

The HTYCPs exhibited an absorption in the blue region, as would be expected for a yellow cultured pearl, but there was a significant difference in the strength of UV absorption between similarly colored natural and treated cultured "golden" pearls (figure 10). The absence of the UV absorption feature in the HTYCPs indicates that the cause of their yellow coloration is different from that of the NYCPs tested. The fact that some areas of the treated cultured pearls showed a weak UV absorption feature suggests that these areas were a light yellow prior to treatment.

**Ultraviolet Fluorescence.** Fluorescence can be a valuable yet challenging identification technique, since many of the effects and color differences are

heat-treated yellow cultured pearls produced by <i>P. maxima</i> .				
Nacre color	UV absorption feature (330–385 nm)	Visible absorption feature (385–460 nm)	Fluorescence to long-wave UV	Comments
Natural white	None	None	Moderate to strong; very light blue or very light yellow	Generally very light blue fluorescence
Natural yellow	Distinct to strong	Present	Moderate; light yellow or light brown, to greenish yellow, greenish brown, or brown	Even distribution of body color will be accompanied by even fluorescence
Heated yellow	None to weak	Present	Moderate to strong; light yellow, very light blue, or light brownish orange	Even distribution of body color may be accompanied by uneven fluorescence

**TABLE 1.** Summary of identification characteristics for natural-color and heat-treated yellow cultured pearls produced by *P. maxima*.

quite subtle and can vary depending on the type of long-wave UV source used. It is important to observe the relationship between (1) the tonal distribution of the yellow color, and (2) the distribution and hue of the fluorescence (again, see figure 7). For the natural-color samples, the distribution of fluorescence matched the evenness or unevenness of the color distribution: An even yellow distribution corresponded to an even fluorescence distribution, and uneven color showed uneven fluorescence. An even color distribution with a patchy fluorescence is indicative of treatment. In lighter samples, however, slight tonal variations in nacre color can be quite difficult to see compared to the more obvious patchy fluorescence.

In addition, the fluorescence colors emitted by both natural- and treated-color light yellow cultured pearls can be similar, and thus they are less reliable as an indicator of treatment. The exception to this is the very light blue fluorescence observed in natural white nacre and in some light yellow HTYCPs. The fact that no NYCPs of light yellow color exhibited this fluorescence color would imply that the treated-yellow sample was originally white. (Caution must be observed, however, because some long-wave UV lamps emit enough blue visible light to render this observation unreliable.) The HTYCPs also lacked the greenish fluorescence component noted in many of the yellow shell nacre samples and NYCPs. Neither the NYCPs nor the yellow shell nacre exhibited the strong orange fluorescence seen in the spots of concentrated color of some of the HTYCPs; therefore, distinct spots of strong orange fluorescence would indicate treatment.

Inasmuch as the details of the heat-treatment process are unavailable, the effect that heat treatment might have on the individual components that contribute to fluorescence (i.e., conchiolin, aragonite plates, and pigments) was not investigated. It has been reported that the fluorescence of conchiolin can be affected by certain treatments (S, Akamatsu, pers. comm., 2001). However, the differences in fluorescence between natural- and treatedcolor cultured pearls may result from a combination of these components and not necessarily from any one component in particular.

**Identification of Heat-Treated Yellow Cultured Pearls.** Table 1 summarizes key identification characteristics for natural-color and heat-treated yellow cultured pearls produced by the *P. maxima* oyster. As indicated below, the ease with which HTYCPs can be identified depends greatly on whether the original cultured pearl had even or uneven color.

- 1. An HTYCP that exhibited white and yellow areas prior to treatment is likely to be the easiest to separate from natural color. After treatment, the white areas would appear yellow but would exhibit the UV spectral characteristics of white nacre; that is, the UV absorption characteristic of yellow nacre between 330 and 385 nm would be absent. In this type of treated cultured pearl, areas that formerly were white fluoresce a lighter color than do the yellow zones. The uneven fluorescence also may contrast with an even yellow coloration of the treated cultured pearl.
- 2. An HTYCP that exhibited evenly distributed, very light color prior to treatment may exhibit a UV absorption feature that is too weak for its apparent color (figure 10). Also, the fluorescence color might appear too light for the apparent tone and saturation of the treated cultured pearl.
- 3. Probably the most difficult to identify would be HTYCPs that were originally unevenly colored light and dark yellow. Nevertheless, this type of HTYCP is likely to exhibit uneven fluorescence, possibly in contrast to an even yellow coloration.

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Figure 11. Large, fine "golden" cultured pearls are a popular item in the jewelry mix. The natural-color cultured pearls in this strand range from 13.0 to 17.3 mm. Courtesy of Tara & Sons, New York; photo © Harold & Erica Van Pelt.

Again, the UV absorption feature would likely appear too weak for the apparent color of the treated cultured pearl.

If heat-treated cultured pearls are subsequently drilled, the color may appear to be concentrated in the surface layers of nacre when observed through the drill hole (T. Moses, pers. comm., 2000).

#### CONCLUSION

As "golden" cultured pearls from *P. maxima* become increasingly popular in the marketplace (figure 11), there is growing incentive to treat offcolor cultured pearls to produce these rich yellow to orangy yellow colors. As a result, the separation of treated-color from natural-color pearls has become a constant challenge for the gemologist. Although color treatment is relatively straightforward to identify in many drilled cultured pearls because of distinctive color differences in the layers visible through the drill hole, certain treatments—especially heat treatment—may be very difficult to detect in some undrilled cultured pearls.

The present study of white and yellow shell nacre from gold-lipped *P. maxima* shells showed a characteristic increase in absorption between 330 and 385 nm in the UV spectrum as tone and saturation increased from white to dark yellow. This absorption appears to be related to the zoochrome responsible for absorption in the blue region, which results in the yellow color of the nacre. The change in absorption with increasing tone and saturation was accompanied by a similar change in long-wave UV fluorescence, which progressed from very light blue or very light yellow, to light yellow or light brown, to greenish yellow, greenish brown, or brown as the colors of the shell nacre samples changed from white to dark yellow.

NYCPs from the *P. maxima* oyster exhibited characteristics similar to those of the yellow shell

nacre. However, the UV reflectance and fluorescence properties of HTYCPs of comparable color appeared more like those of very light yellow or white nacre. Although only 12 treated samples were obtained for the study, all 12 lacked the UV absorption feature characteristic of natural yellow color in *P. maxima*. Therefore, the fluorescence and, more importantly, the absence of UV absorption can help identify heat-treated undrilled "golden" cultured pearls.

Given that the UV absorption feature is a characteristic of natural-color yellow nacre in *P. maxima*, the absence of this feature would indicate a treated yellow color regardless of the treatment method used. Unfortunately, without further study of chemically processed or dyed "golden" pearls, we cannot state that the presence of this feature proves that the color is natural. Fortunately, these latter methods typically are applied to drilled cultured pearls and thus generally are easier to identify.

#### ABOUT THE AUTHOR

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