was an officer in the Air Force Reserve and anticipating his call-up, which did occur in Spring of 1941, Mr. Shipley approached Dean Edward H. Kraus and Professor Chester B. Slauson of the University of Michigan, for a Director of Education. In June of 1940, Richard T. Liddicoat, Jr. was hired. One year later in the spring of 1941 Alfred L. Woodill, Robert Shipley's nephew, who would later become Executive Director of the American Gem Society, also joined the organization. Shipley was very concerned by what might happen to GIA during a war, and he turned the Institute over to the Board of Governors acting for the industry so that an endowment fund could be raised to insure the existence of the Institute through a war-time period. The expected decimation of enrollments did not occur, and ably assisted by Virginia Hinton and Elizabeth Brown on the educational side, Shipley weathered the war years and the Institute emerged in excellent condition.

With the help of the G.I. Bill, which went into effect, as far as GIA was concerned, in late 1946, the Institute prospered and the student body was enlarged enormously during this period. Many people who were to become key factors in the growth of the Institute joined the staff. Liddicoat who had returned from the service in February of 1946, was working with George Switzer, Ph.D., who later became Curator of Gems & Minerals at the Smithsonian Institution, and during this period Lester Benson, Kenneth Moore and Robert Crowningshield were all added to the staff, as well as Lawrence Copeland and others. Mark C. Bandy, Ph.D., was on the staff for a fairly short period of time and opened GIA's Laboratory in New York City in the Fall of 1948. GIA's first Eastern Laboratory had been opened in Boston by Edward Wigglesworth who was serving GIA without compensation. Upon his death in 1945, the laboratory was closed and did not reopen until Bandy and Crowningshield went to New York in 1948.

It was in 1948 that Shipley announced his intention of retiring in 3½ years, planning to step down at the end of 1951. Circumstances led to a 3-month postponement of that retirement date, which was then made March 31, 1952. Thus, after 21 years at the helm of GIA, Robert M. Shipley retired.

The years following his retirement were spent largely in South Laguna Beach in an area of the California coast the Shipleys loved. His beloved wife, Bea, passed away in July of 1973.

On February 18, 1977, three days before his 90th birthday, a vital Robert M. Shipley visited GIA's new headquarters in Santa Monica. A reception was held in his honor by GIA's Board of Governors and the staff. He charmed the student body with a fascinating recounting of salient events from the early days of GIA.

Robert M. Shipley lived a long and productive life. He will be missed by many, but he left his mark.

Differentiation of Black Pearls

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Introduction

The term black pearl has been generally used without clear-cut definition. In addition to naturally colored pearls, various kinds of artifically colored pearls are also called "black pearls." The naturally colored black pearls (hereinafter to be referred to as cultured black pearls), which are barely known to the world, are obtained from black-lipped pearl oysters.

Our main purpose is to describe the history and nature of the cultured black pearls. Methods are presented for distinguishing them from other artificially stained black pearls, especially from pearls blacked by the so-called "silver salt treatment."

Cultured Black Pearls

History and Present Situation

Reportedly, the first trial of black pearl cultivation in Japan, mainly blister pearl, using black-lipped pearl oysters (Pinctada margaritifera) was carried out in 1912 in the Miyakojima Islands in Okinawa Prefecture (the Ryukyu Islands at that time). For several decades since then, a great deal of effort has been made by many pearl culturers to cultivate black pearls. However, all their attempts failed because of the great difficulty involved in the cultivation technique. In the early 1970's, the present Ryukyu Pearl Co., Ltd., succeeded in producing black pearls on a profitable basis. This was nearly 20 years after the initiation of the project in 1951 on Ishigakijima Island, Okinawa.

At present, three main regions produce cultured black pearls: Yaeyama Islands (Ishigakijima, Iriomotejima and other), Tahiti and the Fiji Islands.

Although their existence has been known by some professionals for about 60 years, black pearls of gem quality, namely, perfectly round, real black and flawless, have rarely been seen in the market. The reason for the scarcity is the inability of most

Figure 1. Left: black-lipped pearl oyster. Right: Akoya pearl oyster.

Figure 2. Pearls from black-lipped pearl oysters. From left to right: silver, gold, brown-black, black and green-black.

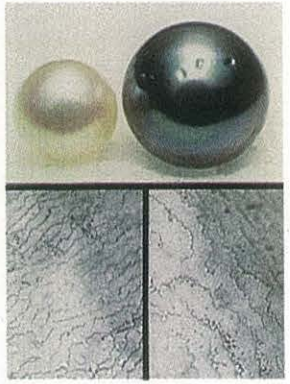

Figure 3. Microscopic observation of stripe patterns of Akoya pearl (left) and cultured black pearl (right) (60x).

Figure 4. Scanning electron microscope observation (x15,000) of the cut surface of a black pearl (above) and its schematic diagram (below).

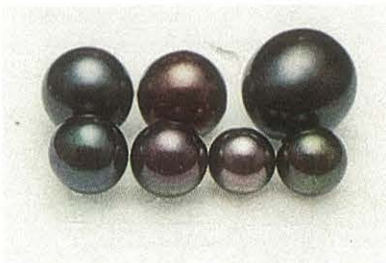

Figure 5. Artificially blackened pearls.

Figure 6. Blue pearl (left) and black pearl (right). Outer appearance (above) and sections (below).

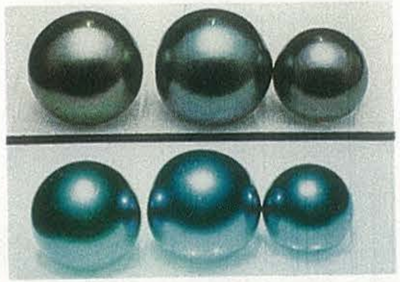

Figure 7. Cultured black pearls. Image of conventional color film (above) and infrared color film (below).

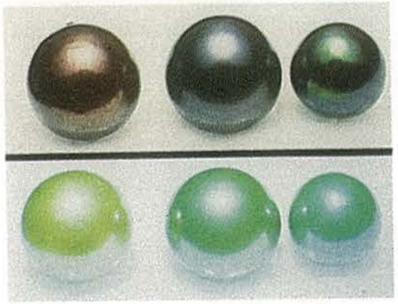

Figure 8. Silver treated black pearls. Conventional color film (above) and infrared color film (below). Compare the infrared image with that of Figure 7. The color tone is quite different.

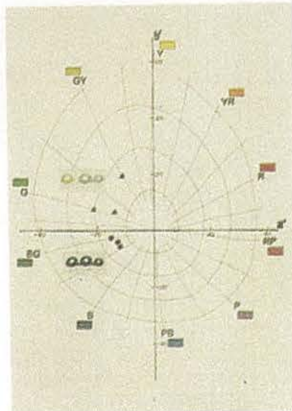

Figure 9. Chromaticity coordinates.

growers to cultivate them.

Presently, the quantity of gem quality black pearls is so limited that the annual production is estimated to be about 2,000.

Culturing Method

Black-lipped pearl oysters are large bivalves which live in tropical or subtropical seas. The inner surface of the shell is an intensively iridescent white silver. The edge of the shell is lined with a characteristic brown-black belt *(Figure 1.)*

The process of cultivation is very similar to that of ordinary pearls: A tiny bead which is made of mussel shell (called the "nucleus") is inserted into a black-lipped pearl oyster together with a piece of the mantle from another living oyster. The oyster is then placed in its seawater habitat and cultured so that the nacre layer can be formed around the nucleus. As the black-lipped pearl oysters are very sensitive, many of them, after having the nucleus inserted in their bodies, reject the nucleus or die. Only 20 or 30 per cent of them eventually serve as pearl-producing oysters during the two-year cultivation period. Of the pearls harvested from these surviving oysters, only 5 to 10 per cent are of gem quality.

Color Tone

Cultured black pearls may be classified into five groups depending on their color. For convenience the following designations are given: silver, gold, brown-black, green-black and black. Among them, the brown-black, green-black and black are tentatively called "black pearls." Silver and gold pearls can also be obtained from the Akoya and silver-lipped pearl oysters as well as black-lipped pearl oysters. Therefore, because they are more plentiful, they are of less value than the brown-black, green-black and black pearls and, of course, cannot be called black pearls *(Figure 2)*.

Structure and Composition

As is well known, the characteristic iridescence and color tone of pearls are derived from the pearl layer which is formed over the nucleus. Microscopic observation of the pearl layer discloses a regularly layered structure of aragonite, a polymorph of calcium carbonate similar to calcite. The characteristic striped pattern of the layers as observed over the surface through the microscope is shown in *Figure 3.* The layered crystal plates are recognized on the cut surface *(Figure 4)* through the use of the electron microscope.

Lying between the crystal layers are organic sheets, specific to pearls, which play a role of sticking the crystals together. The major component of the organic sheets is a hard protein called "conchiolin." Other substances such as glycoproteins and pigments also have been identified.

The only reason black pearls are black is that the brown-black pigment, characteristic of black-lipped pearl oysters, is part of their organic sheets. As far as this pigment is concerned, it has not been chemically studied yet. Only one report on the composition of their shells, not of pearls, makes it possible to guess the composition of the black pigment.*

*Yoshishige Horiguchi Bull. Japan. *Soc. Sci. Fish.* **25**, 391-401, 675-679 (1959).

Analogous (Imitation) Black Pearls

Pearls Blackened by Silver Salt Treatment

These are blackened pearls very similar to the cultured black pearls previously mentioned. They have been on the market since the 1930's and are more popular than the cultured ones. Several methods are being used to make pearls black, among the most popular is the "silver salt treatment." The coloration is based on the chemical reaction using various kinds of silver salts to make silver precipitate in the nacre so that the color of the nacre turns black. Thus blackened pearls are very similar in color to the cultured ones. They also have the same color of brown-black, green-black and black, which makes it difficult even for the experts to distinguish them from cultured black pearls by observation alone. Examples of this treatment can be seen in *Figure 5.*

Blue Pearls

It is the blue pearl which is often considered a black one by mistake, although it is not an imitation of the

TABLE 1.

Comparison of the Mechanisms of Color Formation Between Conventional Color Films and Infrared Color Films

Conventional Color Films

spectrum zone	blue	green	red	infrared
film layer sensitivity	blue	green	red	
dyes formed in film layers	yellow	magenta	cyan	
resulting colors in transparency	blue	green	red	

Infrared Color Films

spectrum zone	blue	green	red	infrared
film layer sensitivity	blue	green	red	infrared
film layer sensitivity after yellow filter		green	red	infrared
dyes formed in film layer		yellow	magenta	infrared
resulting color in transparency		blue	green	red

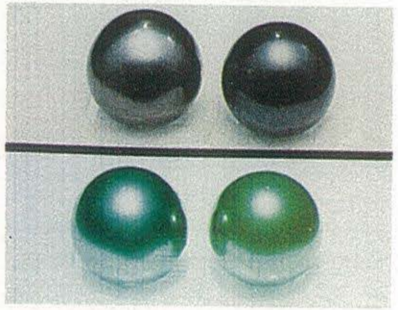

Figure 10. Brown-black pearls. At left is the cultured black pearl; at right the silver treated. Conventional color image (above) and infrared image (below).

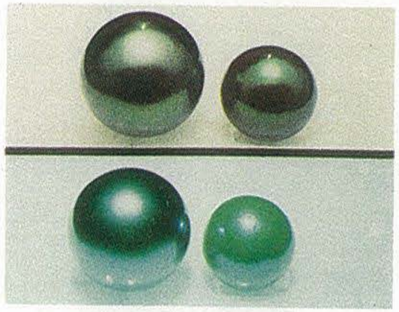

Figure 11. Green-black pearls. Left: cultured black pearl. Right: silver treated. Conventional color image (above) and infrared image (below).

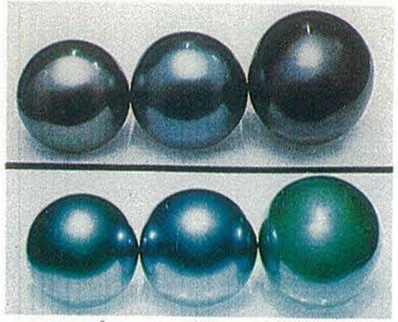

Figure 12. On the right is the imitation. In the center and on the left are cultured black pearls.

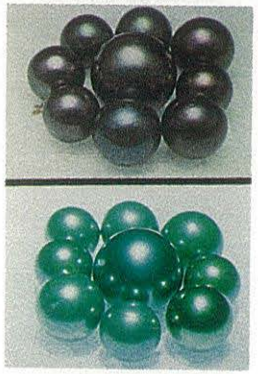

Figure 13. The cultured black pearl (center) is surrounded by silver treated black pearls.

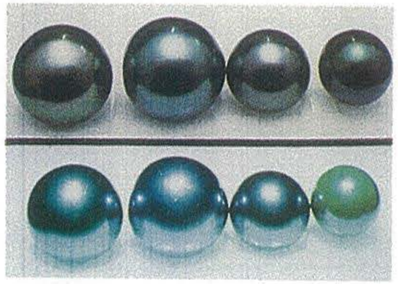

Figure 14. At the extreme right is the treated pearl. The others are genuine.

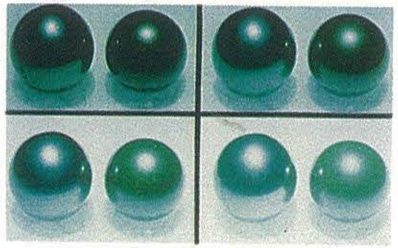

Figure 15. Images of infrared color film with varying exposures. Above left: optimum exposure. Above right: one scale in excess of the optimum exposure. Below left: three scales in excess. Below right: five scales in excess. The cultured black pearl (to the left of all four pictures) is clearly distinguished from the silver salt treated pearl (to the right of all four pictures).

black pearl. Most of the blue pearls are obtained from Akoya pearl oysters and not from the black-lipped oyster. They have bluish colors of transparent pale blue, pale green or deep blue, from which they derive their name.

Further explanation is needed in regard to the cause of blue tones. The tones chiefly come from colored contaminants in the nacre or between the nacre and nucleus. During the period of pearl cultivation, some organic matter becomes incorporated in the shell due to the physiological changes of the pearl oyster. The organic matter causes a brown blemish which gives the pearl a blue appearance when seen through the translucent nacre.

Although the name "blue pearl" often makes one think the blue pearl is a black one, the structure and composition of a blue pearl differ from that of a black pearl, as shown in *Figure 6.*

Differentiation Methods

It should be remembered that in this present article, the genuine black pearl means cultured black pearls having color tones of brown-black, green-black or black which are obtained from black-lipped pearl oysters. These are of gem quality. On the contrary, the blackened pearl means pearls colored by the silver salt treatment as previously described.

There has been an increasing demand by jewelers to differentiate these two types of pearls since appraisers often have been faced with the difficulty of doing it. We tried some methods, among which the following differentiations were found to be useful. Infrared color film yielded especially good results.

Differentiation by Color Image On Infrared Film

When the cultured black and blackened pearls are photographed with color infrared film (Kodak, Ektachrome Infrared Film, IE 135-20), a clear difference between the two can be observed in the color tone of the images. (See *Figures 7 and 8.*)

For a better understanding of the technique, first let us discuss infrared film. Conventional color film consists

TABLE 2.

Analysis of the chromaticity on the infrared images

		Y	x	y	L*	a*	b*
Figure 8	1	49.93	0.3322	0.3702	76.03	-11.48	19.06
(Ag treated	2	49.26	0.3014	0.3423	75.61	-13.82	6.50
Akoya pearl)	3	33.63	0.2878	0.3547	64.69	-21.20	7.14
Figure 7	1	34.21	0.2758	0.3152	65.12	-12.92	-4.50
(Cultured	2	34.57	0.2734	0.3107	65.40	-12.33	-6.01
black pearl)	3	16.29	0.2642	0.3225	47.34	-15.81	-3.05

*measured by spectrophotometer (Hitachi M 307).
calculated according to 0-0 SI 10 W 10 (JIS Z 8722)

of three image layers sensitive to blue, green, and red, whereas the image layers of infrared color film are sensitized to green, red and infrared. Accordingly, the differences in intensity of infrared ray reflections on materials results in differences of color tones from the images (see *Table 1*).

As shown in *Figures 7 and 8*, cultured black pearls give blue images while, in contrast, images of blackened pearls are greenish blue to yellow-green. Whatever different colors – brown-black, green-black or black – cultured black pearls may have, they give the same blue images which are quite different from those of blackened pearls. Other examples as seen in *Figures 10, 11, 12, 13* and *14* show this fact. These color characteristics can be used as one of the effective methods of differentiation.

Next, the analysis of the chromaticity was carried out on the images of *Figures 7* and *8*. (See *Table 2* and *Figure 9*.)

As indicated in *Table 2* and *Figure 9*, the chromaticity of the cultured black pearls belongs to the blue region, while blackened pearls have their chromaticity in the region from green to yellow with probable contamination of red. Although the mechanism has not been proven yet, the color difference between the two kinds of black pearls seems to result from the infrared reflection rates being different between genuine and imitation black pearls. This difference is due to the infrared color film which gives a red image when it is exposed to infrared rays.

There are two requirements necessary in order to obtain a clear differential image. First, the light source should be a Xenon lamp. The intensive emission spectrum of this lamp in the infrared region (0.82-0.99 μ) may contribute to the successful differentiation of the two pearls. Secondly, a series of images photographed with varying exposure is advisable, from optimum to excessive, since it is rather difficult to differentiate between them with only a single photo. By photographing in a series as recommended, clear differentiation will be obtained. *Figure 15* shows the difference in color tone of images taken at various exposures. Notice that the difference between the two becomes clearer with excessive exposure.

Differentiation by X-Ray Fluorescence Analysis

If pearls are treated with silver salt, silver will be detected by X-ray fluorescence analysis*. Since cultured pearls do not contain silver, the extraordinary content of it suggests that it has been treated with silver salt. The most favorable reason for using this analysis is that it can analyze the elemental composition without destruction of the samples. Elemental composition of the surface layer of pearls (pink pearls from Akoya pearl oyster), cultured black pearls and blackened pearls are shown in *Table 3.*

As expected, silver was detected only in the blackened pearls. The X-ray fluorescence analysis may be used as one of the methods for distinguishing cultured black pearls from the blackened ones. The only *unfavorable drawback* to this analysis is that the *sample pearls turn to brown-black from X-ray irradiation.*

Table 3 shows that presence of potassium and strontium in the cultured black pearl. Further studies are required in order to confirm which conditions, physiological ones of the oyster or environmental ones such as seawater, nutrition, and so forth, cause the presence of these elements.

Differentiation by Hardness Measurement

The nacre of the blackened pearl is more fragile due to the alteration of its surface by various chemicals during the coloring process, especially silver salt treatment. By measuring the tendency of the decrease in its hardness with a micro hardness testing machine, we can judge whether sample pearls are chemically treated or not. A decrease of about 40 per cent in the hardness caused by the coloring process was demonstrated by a Vickers hardness testing machine. In addition, an indication of surface deterioration, such as exfoliation and cracks are occasionally found on blackened pearls by careful observation with a microscope.

Other Methods of Differentiation

None of the blackened pearls fluoresce under ultraviolet irradiation. The cultured black pearls, on the other hand, especially those of middle grade, emit yellow-red fluorescence from the hollow and the micro niches on the surface. The emission is limited to the spot of such an irregularity on the surface (the cultured black pearls of high quality as previously defined emit no fluorescence because of the complete smoothness of the surface). However, there are so many exceptional cases in these properties that careful testing is required to adequately identify the cultured black pearl.

TABLE 3

Element Composition of the Surface Layer of Pearls by X-ray Fluorescence Analysis.

Samples	Elements														
	Ag	Sr	Zn	Cu	Ni	Fe	Co	Mn	Cr	Ca	K	S	P	Si	Al
Akoya pearl	–	++	–	–	–	–	–	–	–	+++	+	–	–	–	–
Black-lipped pearl	–	+++	–	–	–	–	–	–	–	+++	++	–	–	–	–
"Ag treated" Akoya pearl	++	++	–	–	–	–	–	–	–	+++	±	–	–	–	–

+++ : most clearly detected
++ : clearly detected
+ : detected
± : not clearly detected
– : not detected

*Being irradiated with a powerful X-ray, each element contained in the sample is excited to emit characteristic X-rays, the wave lengths of which are measured spectroscopically to confirm the presence of a corresponding element in the irradiated area.