NOTES FROM THE LABORATORY

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It is not unusual for a group of gemmologists to disagree over the colour of a gemstone. Some coloured diamonds in particular tend to possess colours which are most difficult to describe. However, when you see a diamond actually change from one distinct colour to another before your eyes it can shake any confidence you may have in your own eyesight.

Such was the case when late one afternoon I decided to make a start on identifying the nature (natural or treated) of the colour of a 2.02 ct brilliant-cut diamond.

All I really had time for was to make out my work-sheet, giving a full description of the stone, and a short microscopic examination before it had to go into the safe for the night. On the work-sheet I stated in a most positive fashion that the colour of the stone was green. The next morning when the safe was opened I immediately retrieved the envelope containing the diamond, took it to a work-bench and removed the stone from it. There before me lay a brilliant *yellow* stone! After checking the envelope to make sure that it was the one I put in the safe the night before (it was) I decided to check the stone's weight against my record, but as I picked it up to take it to the balance its colour started to change through various shades of yellow and yellow/green until it was back to the colour it was the night before.

These so called 'chameleon diamonds' have been reported upon before,[1] and the change has been variously described as being associated either with changes in temperature or in the amount of light reaching the stone. A manufacturer would notice the effect because it is said that these stones glow red on 'the wheel'[1] and change to yellow shortly afterwards, from which they return to their normal green at room temperature; whereas a trader might become aware of the type of stone he had in a similar manner to that in which I had become aware of the peculiarities of this stone.

The colour change from green to yellow, unless one includes the slight cooling which may occur if the stone is placed in a safe overnight, is usually described as being dependent upon a

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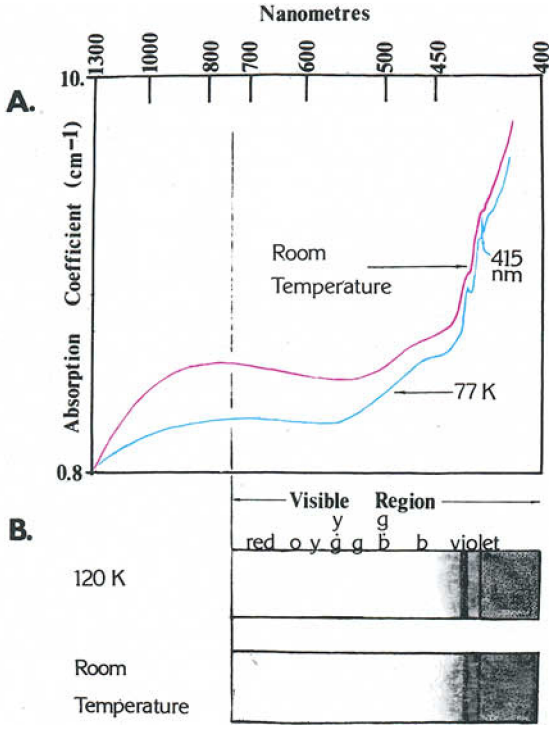

FIG. 1 The room and low-temperature spectra of a 'chameleon' diamond. (A) As a recorded absorption curve and (B) as seen with the hand spectroscope.

temperature increase, such as placing the stone on a hot plate, rather than a decrease; and so it was interesting to discover that when we reduced the temperature of this stone to at first 120K in the Laboratory and then to 77 K at King's College, London, whilst recording the spectra reproduced in Figure 1, the colour of this stone once again became a brilliant yellow.

The differences between the room and low temperature spectra depicted in Figure 1 are quite evident. The general appearance of the spectrum at room temperature is approaching that of a normal Type 1b with a weak 415 (Type 1a) peering out of the gloom and an unusual absorption hump covering the yellow, orange, red and N.I.R.—the area of greatest transmission being in the green. At the lower temperatures there is clearly a sharpening up of the 415, but more importantly there is a lessening of the absorption hump in the red, orange and yellow, allowing the stone to transmit to a greater extent in this region as well as in the green, thus resulting in a yellow stone.

One assumes that changes of a similar nature may take place when the stone is heated; however, we restricted ourselves to room and low temperature spectroscopy only.

The luminescence effects produced by this stone were—long-wave ultraviolet, a very strong bright yellow followed by a very strong greenish phosphorescence; short-wave ultraviolet, a strong and bright yellow/green followed by a very strong greenish phosphorescence; and x-rays, a blue/green followed by a strong green phosphorescence.

* * *

An item that we have seen quite a number of examples of over the past few years is the imitation crystal.

One interesting specimen made to imitate ruby was composed of fragments of natural and synthetic (Verneuil) ruby held together by an adhesive and coated in mica. More often than not though, these imitations are made to resemble emerald, in particular the type of mica-coated rough that emanates from East Africa.

Sometimes produced with obvious crystal form, as in the example shown in Figure 2, or more convincingly with the minimum of form, the basic material for this type of imitation is either very poor quality emerald, beryl or glass. The poor quality emerald or beryl varieties may be manufactured either by slicing the crystal down its length and gluing the two pieces back together with a green adhesive and then coating the whole in mica, or by hollowing out the crystal, infilling with a green substance and then coating the base with a matrix-like material.[2]

The example in Figure 2 is made of green glass and has just enough mica adhering to the surface (Figure 3) to be reasonably convincing.

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FIG. 2 An imitation emerald crystal made of glass and with mica adhering to its surface (see Fig. 3).

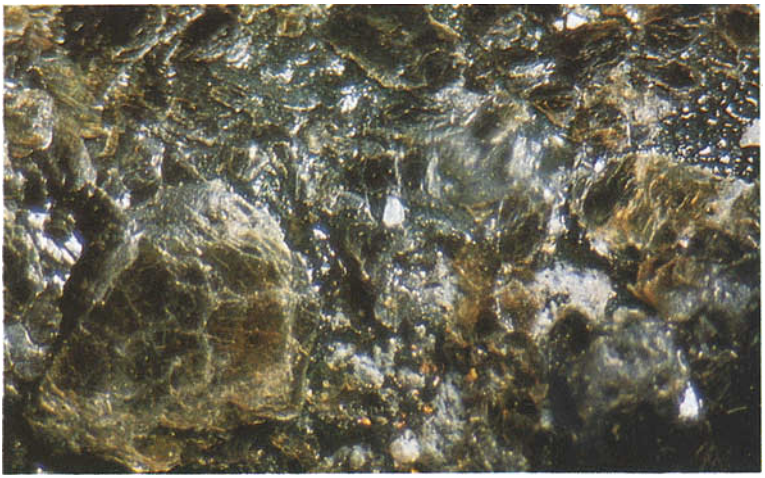

FIG. 3 The mica adhering to the surface of the imitation crystal in Fig. 2.

FIG. 4 A colourless natural sapphire before exposure to x-rays.

FIG. 5 The same natural sapphire as seen in Fig. 4 after a ten-minute exposure to x-rays.

A requirement of most gem identification processes is that whenever possible they must be of a non-destructive nature; indeed in the vast majority of cases gemstones can be and are identified in this manner. There are some cases, though, where the only means left open to one require either a 'minor' destructive test, such as taking an insignificant scraping from the girdle of a stone for an x-ray powder diffraction photo, or something possibly more destructive such as attempting to fade or drive off the colour of a suspected x-ray irradiated stone.

Understandably not many people are very happy when we ask for permission to test for colour stability upon being presented with a yellow sapphire for examination, but in some instances it is essential having established the natural origin of such a stone that a test of this nature is carried out. Some colourless sapphires may attain a yellow colour quite quickly when they are irradiated with x-rays,[3] but this colour is not permanent, and exposure to sunlight will gradually return the stone to its normal light yellow or colourless state.

It didn't take long to realize when recataloguing the laboratory collection recently, that a 'colourless' stone found in a stone paper marked 'irradiated "yellow" sapphire' (Figure 4) was not a misplaced stone but one that had been x-ray irradiated and had subsequently faded. It took just a ten minute exposure at a setting of 45 kV and 19 mA in close proximity to the x-ray tube window to reproduce the yellow colour (Figure 5).

When a little while ago we were confronted with two large apparently natural, intensely orange sapphires of a hue that we had not observed in nature before, quite naturally our suspicions were aroused. The two stones weighing 32.44 and 8.72 ct (Figure 6) were very similar in colour to the Verneuil type synthetic orange sapphire and did not approach the colour of the heat treated yellow sapphires we see so many of nowadays (Figure 6). The refractive indices were normal for corundum, but the absorption spectrum (Figure 7) was a very intense version of that expected from a synthetic yellow sapphire—the stones were strongly absorbing in the blue and violet and there were no signs of any sharp lines in the red or the 'normal' sapphire bands in the blue.

To the unaided eye the stones had a somewhat cloudy appearance and a microscopic inspection revealed the cause of this to be light reflecting from a multitude of dust-like particles within

FIG. 6 Four small heat-treated yellow sapphires and two large orange sapphires which owe their colour at least partly, if not wholly, to heat treatment.

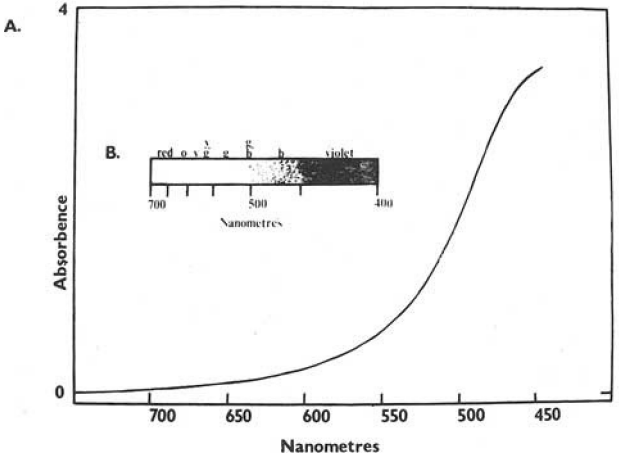

FIG. 7 The absorption spectrum of the smaller of the two large orange sapphires seen in Fig. 6, (A) as a recorded absorption curve and (B) as seen with the hand spectroscope.

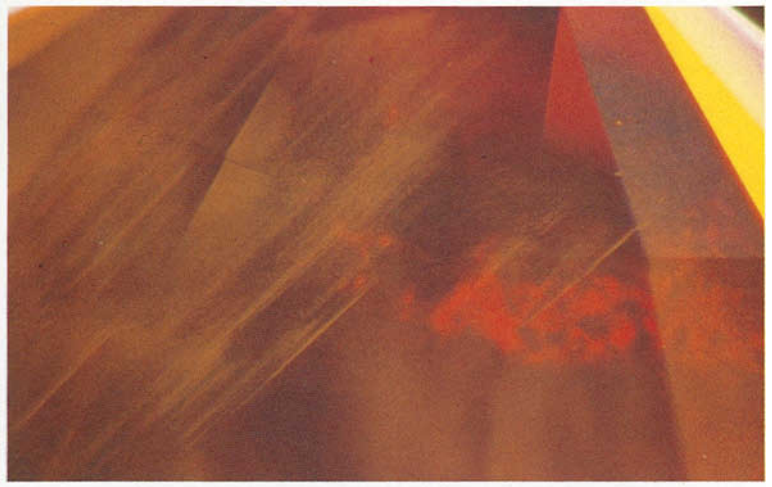

FIG. 8 Fine dot-like 'silk' producing a cloudy appearance to the orange sapphires seen in Fig. 6.

FIG. 9 Fine dot-like 'silk' orientated around small angular areas in the orange sapphires seen in Fig. 6.

FIG. 10 A 'Bombay Bunch' of natural pearls.

the stones. Over all these particles took on the appearance of very fine 'silk' (Figure 8), and they were generally orientated in specific directions travelling through the stones, although as may be seen in Figure 9, the particles also tended to orientate themselves around small angular areas. Also included were other indications that these were natural sapphires that had undergone heat treatment, such as the glassy centres to some feathers.[4,5] However, as both the colour and its intensity were so unusual, it was felt that a colour stability test was definitely needed, firstly because of some recent reports of colour instability in some heat-treated yellow sapphires, and secondly just in case they were treated stones that had been subject to further x-ray treatment. Unfortunately permission to attempt to fade these stones under controlled conditions was refused, and so the situation remained unresolved.

* * *

Even though many thousands of natural pearls pass through the laboratory every year, it is quite rare nowadays for them to be submitted as 'Bombay bunches'.[6] The first such bunch to be submitted for many months (Figure 10) reminded us just what a work of art these items are in themselves.

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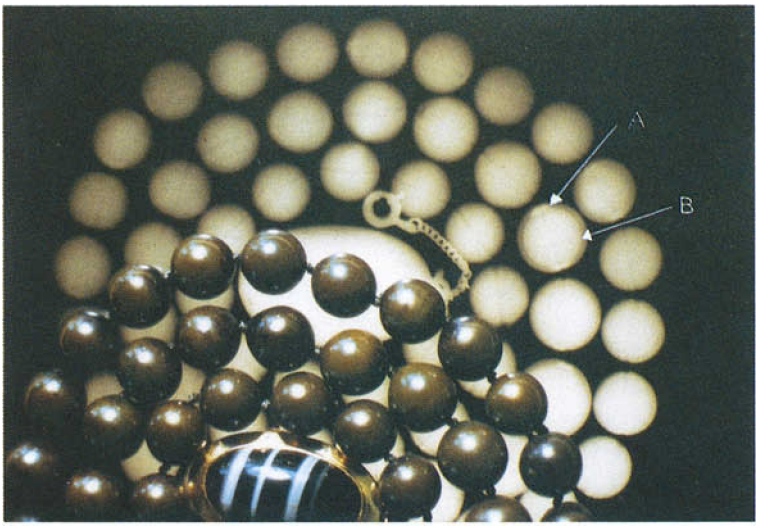

FIG. 11 A necklace of mottled brown stained cultured pearls together with the x-ray picture, showing the demarcation between the bead and the outer layers (B) and the way in which the stain alters the normal x-ray transparency of the demarcation (A).

The radiograph taken of an unusually coloured—mottled brown—'pearl' necklace (Figure 11) revealed not only that the pearls were cultured but also the way in which the stain had altered the normal x-ray transparency (11A) of the demarcation between the bead and the 'pearl' layer (11B).

* * *

Being a trade laboratory, naturally the majority of our work concerns the identification of the more important commercial stones, and so it is a most welcome break from routine when something outside this grouping is submitted. When the bead in Figure 12 was placed before us (looking for all the world like a lacquered peach stone), upon an eye inspection all sorts of suggestions about its possible identity were put forward, many of them not very complimentary.

The microscopic inspection revealed that the bead had a fairly thick coating of clear plastic (Figure 13), and, as this would inhibit an accurate SG determination and completely rule out RI measurements, it was quickly realized that, although the structure, visible now, appeared to be similar to that of coral, this was so distorted by the coating that we would have to resort to a destructive test so that we could 'get at' the structure without having to peer through the plastic.

We were fortunate in this case that the client had included in his instructions to us 'test to destruction if necessary'. Taking him at his word we sliced the bead into two, when a very coarse coral structure became all too obvious in its nakedness (Figure 14). A small drop of dilute hydrochloric acid was then placed on the newly exposed area as a confirmatory test, causing effervescence.

* * *

In the July 1979 issue of this *Journal*[7] I reported the occurrence in the visible spectrum of three brown diamonds of an absorption line that had previously only been associated with treated diamonds. Since then we have observed this line at 637 nm not only in brown stones but also in yellows and two 'odd' green stones.

I subsequently reported[8] that we were reasonably satisfied that when the 637 was observed in the sort of strengths described (that is, either very weak at room temperature or only visible at 120 K) and in the sort of stones described (that is, Type 1b in which the

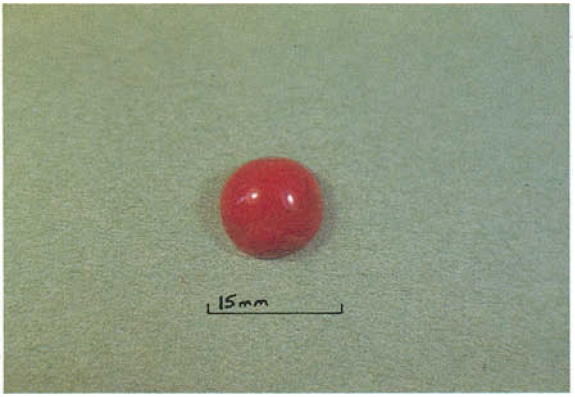

FIG. 12 A plastic coated coral bead.

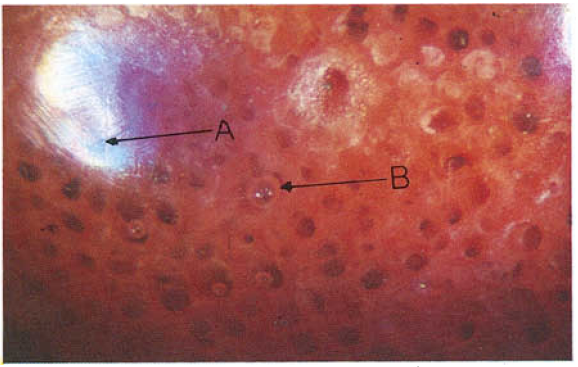

FIG. 13 (A) light reflecting from the surface of the clear plastic coating and (B) gas bubbles in that coating of the coral bead in Fig. 12.

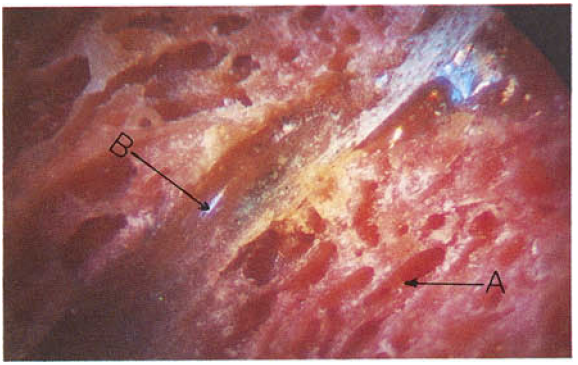

FIG. 14 (A) the 'coarse coral structure' and (B) the plastic coating adhering to the sides of the drill hole of the bead in Fig. 12.

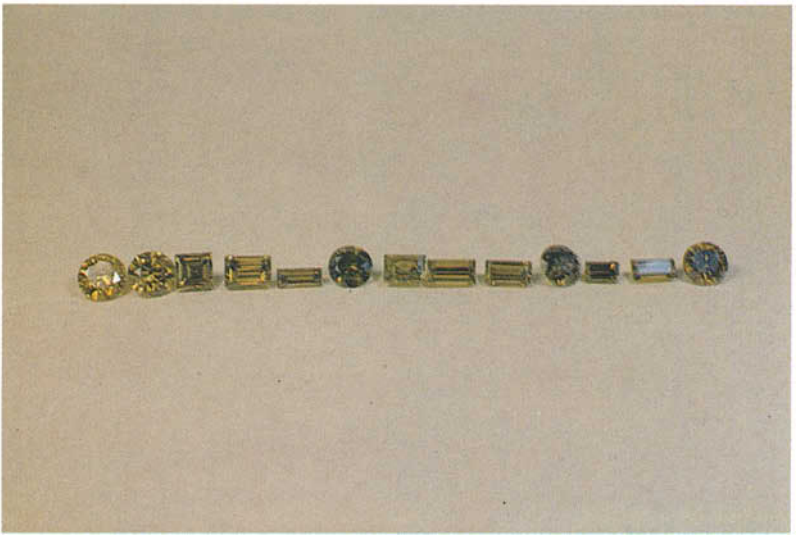

FIG. 15 Thirteen coloured diamonds which not only all come from the same item of jewellery, but also all contain the 637 nm absorption line in their spectra when examined at low temperatures.

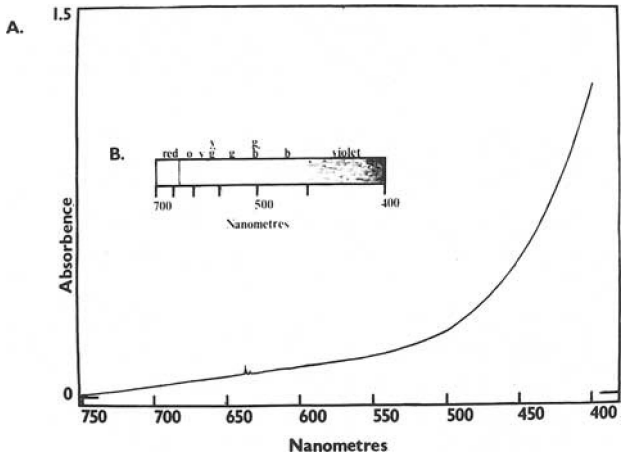

FIG. 16 The low temperature absorption spectrum typical of the stones seen in Fig. 15, (A) as a recorded absorption curve and (B) as seen with the hand spectroscope.

body colour was obviously due to the naturally occurring absorption in the violet and blue) it should be treated as yet another naturally occurring radiation-related band. We still believe this to be the case today.

During the four year period between the first report and a few weeks ago only 19 stones in which the naturally occurring 637 nm lines could be observed were examined in this Laboratory. More recently a further 15 have been added to that number. Somewhat incredibly though, 13 of this latest 15 (Figure 15) not only came from the same item of jewellery, a brooch, but also they were the only stones in that item.

Of these 13 stones, there was only one that revealed the 637 rather weakly at room temperature, all the rest only revealed the line at 120 K. Figure 16 shows the absorption curve of one of the two other stones at 77 K, this curve being fairly typical for the type of stone.

It is not too unusual with coloured diamonds to find the same type of stones, with similar spectra, in the same item of jewellery. This occurs quite often because the person making up the item has successfully matched the stones for body colour and in so doing has chosen stones of the same 'type'. What is unusual about this instance is that these stones are far from being matched for colour; as can be seen in Figure 15 the colours range from a distinct yellow through to brown.

* * *

A very high percentage of the coloured diamonds submitted to us for examination turn out to be naturally coloured, and so, when a treated stone turns up, although it may not be a pleasurable experience for the owner, for us this must undoubtedly produce a sense of achievement in that the lengthy procedures we strictly adhere to in the examination of coloured diamonds do pay dividends.

One such stone is worth a mention here, because it is so often said that only very low quality diamonds are irradiated, and in our experience this is very often not the case, certainly purity-wise if not colour-wise.

The stone weighed exactly 6 ct and was marquise shaped with a nice yellow body colour (Figure 17). Under both long-wave and short-wave ultraviolet radiations the stone fluoresced yellow/green and there was no phosphorescence. The absorption spectrum

FIG. 17 A 6.00 ct treated yellow diamond.

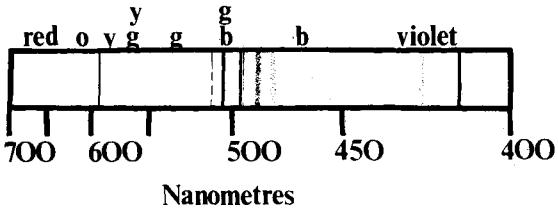

FIG. 18 The absorption spectrum, as seen with the hand spectroscope at 120 K, of the treated diamond seen in Fig. 17.

(Figure 18) at 120 K was unmistakably that of a treated stone with a strong 595 nm line and very strong lines at 503, 496 and 415 nm, the first three being induced by irradiation and subsequent annealing and the last naturally occurring. Whilst the 415 was fairly strong there was no 478 nm absorption, and therefore it is fair to assume, although it is by no means certain, that the pre-treatment colour of this stone would not have been significant, fancy-diamond-wise, but would have been evident under normal colour-grading standards.

Whilst we were not asked to grade the stone for purity, it is quite normal for us to give all coloured diamonds a thorough microscopic examination, and it was found that this stone at 10× magnification was clear of any internal imperfections apart from a few cleavages in the area of the girdle, which could have been removed to produce a flawless or loupe-clean stone.

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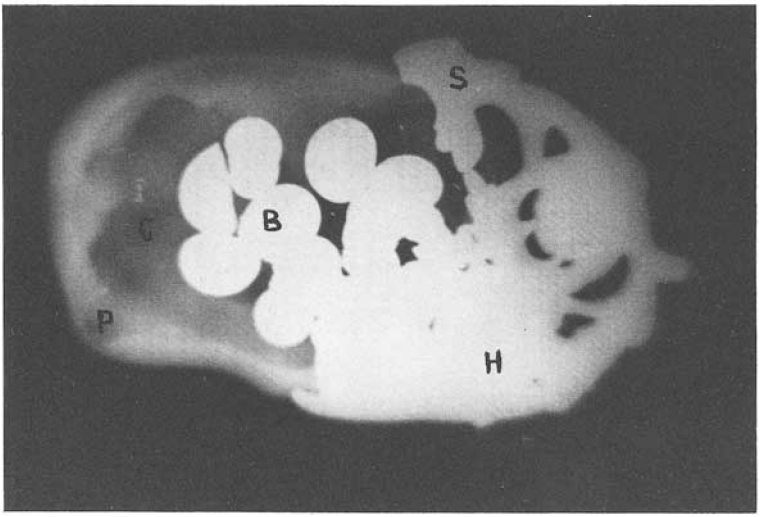

FIG. 19 The radiograph of a pendant-set large hollow baroque pearl, inside the cavity of which can be seen a number of beads with an opacity to x-rays similar to that of the setting. (P) pearl: (C) cavity: (B) beads: (S) setting: (H) the area of the setting which hides the 'cap'.

During one of our recent courses a participant produced a large baroque pearl, which seemed to be an ideal subject with which to demonstrate how direct radiography may help with pearl identification. The resulting picture, however, revealed a little more than was expected.

The pearl itself, which was mounted as a pendant, with the setting (S in Figure 19) covering the major portion of one 'corner', was found to be little more than a comparatively thin layer of nacre surrounding a large cavity (P and C respectivly). Within the cavity could be seen a number of beads (B) with an opacity to x-rays comparable with that of the setting. A further microscopic inspection exposed the 'cap' that had to be present. It was well hidden under the setting in the area marked H in Figure 19.

A fair assumption about the history of the pearl might be that at some stage it had been drilled and subsequently to this the area around the hole had collapsed, at which time it could have been decided to infill the cavity with a number of beads to give the pearl the weight expected of a pearl of that size.

* * *

The generally small 'Biwa' type[9] non-nucleated cultured pearls, when seen in long single or multi-row necklaces, are for the most part quite easy to identify when a radiograph is taken, and most people who handle pearls can virtually identify these necklaces just by seeing their shape and colour (usually very white). Increasingly, however, today we are seeing the much larger single non-nucleated cultured pearl in which the colour and shape vary considerably. It is certainly not possible without the aid of x-rays to even hazard a 'professional guess' about the origin of these pearls, and even when a direct radiograph is taken sometimes it is not the easiest of tasks to identify them.

Figure 20 shows some of the shapes, sizes and colours that are at present on the market. All eleven were identified as non-nucleated cultured pearls by direct radiography.

* * *

There are some gemmologists who appear to be able to find the most extraordinary items on their travels, and not least amongst these is one of the Laboratory's regular benefactors, Chris Cavey. During one of his visits with us latterly he placed in my hand what appeared to the naked eye to be a miniature portrait mounted as a fob (Figure 21). He then proceeded to tell us that he had bought it in a badly worn condition, and by way of offering a clue to its identity he added that he had had it *repolished.*

FIG. 20 11 non-nucleated cultured pearls.

A microscopic examination unveiled the true nature of the object. It was clearly made entirely of glass and was all one piece (not a mosaic) and the portrait, which was quite detailed (Figure 22) was continuous from one side of the object to the other with very little if any deviation—something similar to the name of a seaside town running through a long 'stick of rock' (candy)—therefore, no matter how many times it was repolished (provided there was still some of the item left!) the 'portrait' would remain practically unaltered.

Very often as gemmologists we are awe-struck by the artistic arrangement of included material in natural gemstones, but it is not so often that we are able to let out a gasp of admiration for the artistic achievements, within our sphere, created by man. This for me was certainly a case for the latter.

A week or so prior to Mr Cavey showing us this lovely object, he brought in five most attractive orange stones (Figure 23), the colour and lustre of which did not strike me as being peculiar to any of the more common gemstones. They had, he said, been offered to him as clinohumite from the U.S.S.R.

Clinohumite ($4Mg_2SiO_4.Mg(F,OH)_2$) [10] is a monoclinic member of the Humite group, and its constants are given in Dana's

FIG. 21 A coloured glass 'portrait' set as a fob.

FIG. 22 Detail of the portrait in Fig. 21.

FIG. 23 Five faceted examples of clinohumite averaging approximately 3 ct.

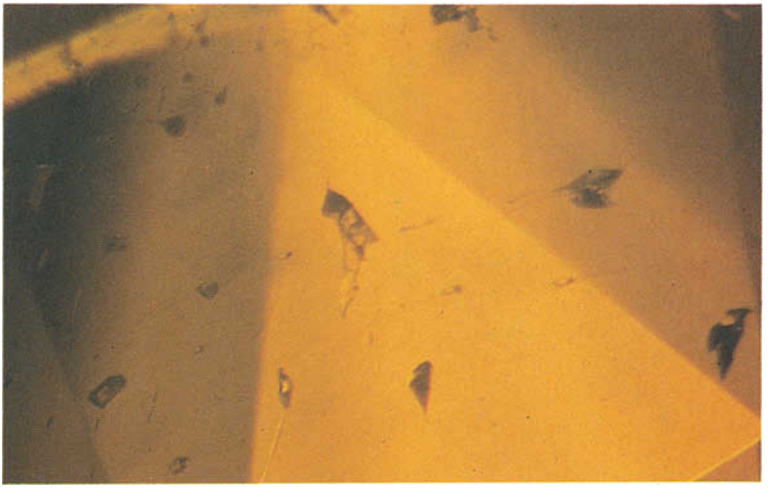

FIG. 24 2-phase inclusions in a clinohumite.

FIG. 25 A group of crystals in a clinohumite.

FIG. 26 Twin planes in a clinohumite.

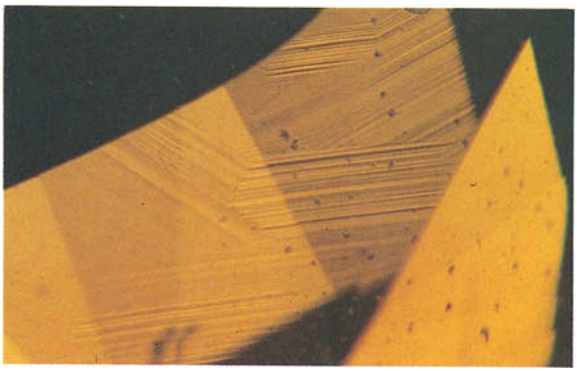

FIG. 27 Angular growth zoning in a clinohumite.

Textbook of Mineralogy[10] as RI α = 1.62 - 1.66, β = 1.64 - 1.67, γ = 1.65 - 1.69; SG 3.1 - 3.2 and H. 6 - 6.5; and one of the occurrences cited is near Lake Baikal in Siberia.

The average weight of these five stones was approximately 3 ct, the largest stone weighing 4.59 ct. Their SGs ranged from 3.223 - 3.230 and their RIs were α 1.630 - 1.633, β 1.644, γ 1.662 - 1.665 with DR varying from 0.031 to 0.033, all of which fall reasonably close to the range recorded by Dana.

By way of confirming their identity Steve Kennedy took a small powder scraping from the girdle of the smallest stone and produced an x-ray powder diffraction photograph which proved to be characteristic of clinohumite.

Under the microscope all the stones were a feast for the eyes. Figures 24-27 show the main types of included features (2-phase and other cavity types in Figure 24, crystal groups in Figure 25, twin planes in Figure 26 and growth zoning in Figure 27), and of course the double refraction was clearly evident upon viewing the facet edges through the stones.

The absorption spectrum for each stone resembled closely that produced in Figure 7 in that there were no sharp absorption lines or bands, just a strong absorption of wavelengths shorter than 450 nm in the visible region.

* * *

When something a little unusual is submitted to the Laboratory for examination it sometimes occurs that in the coming weeks several other examples of the same material are placed before us. However, in the weeks, months or indeed years, following this surge we may not be required to examine it again.

At the end of last year the pearl trade became very worried about the number of large mauve pearls entering the market, and we were asked to examine a number of examples. All were examined by direct radiography and none revealed any evidence on the radiographs which might have indicated a cultured origin, either non-nucleated or nucleated. Indeed the only structure revealed, indicated a natural origin. Apart from a re-examination of one of these pearls recently, we haven't seen any new examples, since the initial 'surge'.

FIG. 28 Two very pale pink natural pearls flanking a natural mauve pearl weighing 16.47 ct.

FIG. 29 The 'flat' side of the centre pearl in Fig. 28.

An example of the type of pearl involved is depicted in Figures 28 and 29. In Figure 28 it has been placed between two very pale pink natural pearls weighing 4.96 and 22.36 ct. The mauve pearl itself weighs 16.47 ct and measures 16.38 × 8.81 mm, and in Figure 29 the difference in colour between the base and the top of the pearl can be noted, and a peculiar central area can also be seen.

Whilst we could find no evidence to indicate that this pearl or any of the others we examined were cultured, it must be said that it should not be long before non-nucleated cultured pearls of a similar colour to these are available in this country.[11]

* * *

In the early part of 1983 we noticed an advertisement in one of the trade publications in which the various merits of the 'Angelo pearl' were being expounded. The descriptions given of these 'pearls' and some of the claims made intrigued us enough to request a sample of the material from the U.K. distributors, who immediately forwarded to us a number of loose specimens for our examination. All were fully drilled beads measuring approximately 5.5 mm in diameter.

A major portion of the advertisement hinges around the fact that 'the core' of the Angelo pearl 'is made of fragments of fresh water shells which are found in the waters of the Mississippi River', and it is stated further that 'only first class shell material for the core is used and is identical to that implanted in oysters for the nucleus of the cultured pearl'.

Shell based imitation pearls are not new.[12, 13] In 1978[14] Farn described an imitation pearl that produced a Laue diffraction pattern typical of a cultured pearl; it was in fact a lacquered mother of pearl bead, and before I received these samples, this was the sort of 'pearl' I was expecting to see—but this was not to be the case.

Firstly, by way of confirming the manufacturers' statements about the nucleus of the samples, one was sliced into two, whereupon it could be seen that they were in fact constructed of a solid central bead and some kind of coating to that bead (Figure 30). As the central bead effervesced when a small drop of hydrochloric acid was brought into contact with it and as it also had a banded structure (Figure 32) along with other properties

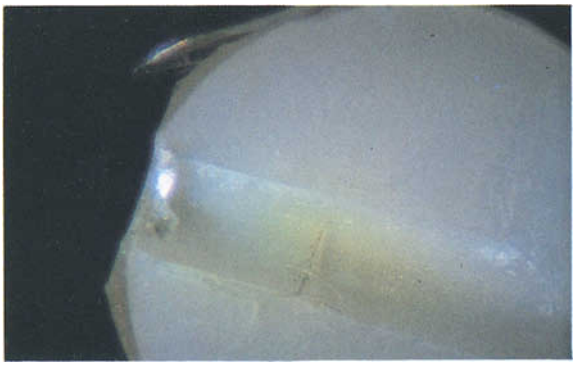

FIG. 30 A sliced 'Angelo' imitation pearl showing the shell bead centre and the thickness of the outer layer.

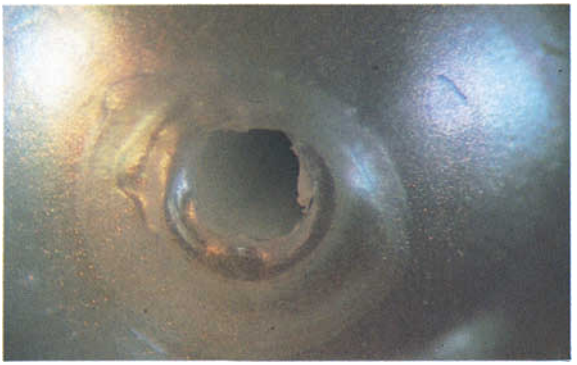

FIG. 31 The 'Angelo' imitation pearl showing a discretely speckled play of colour.

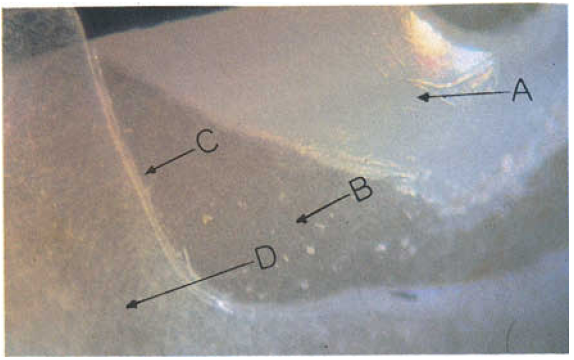

FIG. 32 The 'layers' of the 'Angelo' imitation pearl. (A) The shell bead nucleus showing a banded structure. (B) The layer closest to the bead, (C) the layer in which the 'play of colour' appeared to be confined and (D) the outermost layer.

which are characteristic of Mother of Pearl, such as x-ray induced fluorescence and phosphorescence and the production of four- and six-spot Laue diffraction patterns, the manufacturers description of the central core would seem to be accurate. The specific gravity of the whole pearl was found to be 2.65.

To the unaided eye there seemed to be a little more 'life' to these Japanese manufactured 'pearls' than there normally is with imitation pearls, although to the experienced eye they were 'not quite right' in comparison to the natural or cultured pearl. Under magnification it was found that this 'extra life' could be attributed to a discretely speckled play of colour (Figure 31) and that the coating consisted of three easily separable layers (Figure 32). The play of colour appeared to be confined to the centre layer, C in Figure 32, and did not seem to be present in the two other layers of this 'plastic like' material. Imitation pearls usually 'leave me cold' but I was really quite impressed with the visual impact of these. However, it remains to be seen just how resistant the coating is to damage from contact with the various cosmetics.

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