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Blue sapphires from the Mambilla Plateau, Taraba State, Nigeria

A preliminary examination

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GIA Laboratory, Bangkok

August 16th, 2014



Figure 1: Parcel of 30 rough blue sapphires reportedly from the Mambilla area, Nigeria seen in Bangkok on June 05th 2014. The weight of the stones is between 1 and 5 carats. Photo: Vincent Pardieu © GIA.

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Introduction:

In April 2014, one of the authors (VP) was preparing a field expedition to visit sapphire deposits in Nigeria. During the preparations he contacted several West African gem merchants in Bangkok to arrange to see some samples and seek additional details about the sapphire deposits in Nigeria. This interest was fueled by the emergence of Nigeria as an important producer of gemstones over the past few years, and the lack of publications on the sapphires and sapphire deposits from and within the country. During one of these discussions he was shown a parcel of blue sapphires reportedly from a new deposit located on the Mambilla Plateau (Taraba State) in Nigeria (Figure 1). Over the following weeks, more parcels containing stones of similar appearance showed-up in Thailand, and also in Sri Lanka, as many Sri Lankan gem merchants reportedly rushed to Nigeria. All the people we met were very excited by the quality and the size of the stones being produced. Large clean stones were reported, some weighing between 100 and 300 carats with a nice blue color. Reports emerged that stones from the new deposit were being traded at very high prices. One stone reportedly sold for more than 1 million US dollars in Thailand. Photos of such stones rapidly started to appear on social media sites (Figure 2) and were also frequently encountered on the mobile phones of some West African gem merchants visiting Thailand.

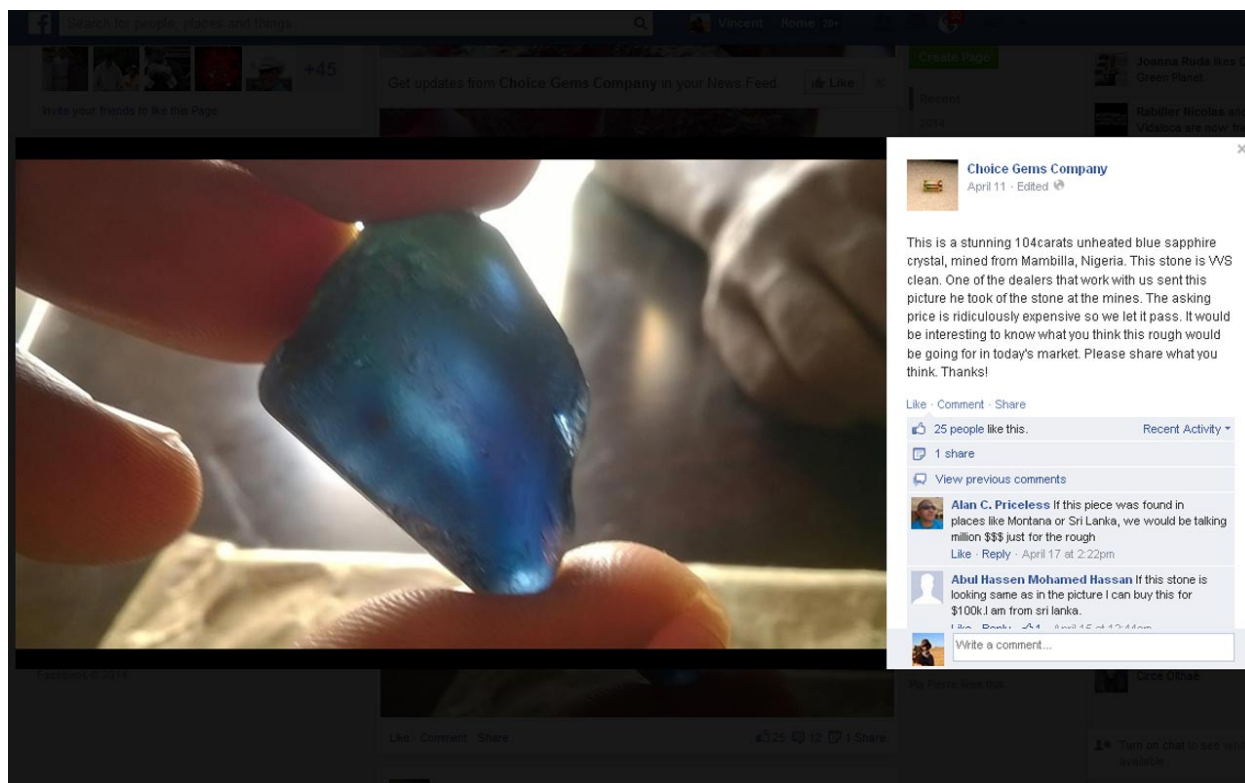


Figure 2: Photo of a large rough sapphire reportedly from Mambilla being shared openly on a popular social media site by a West African gem merchant as seen on April 11th 2014.

The new production looked slightly different to the material from the old diggings in the Mambilla area. The “classic” sapphire material from Mambilla was known to occur as small stones, usually with quite a dark blue color and a good crystallization (Michelou 2007) while the new material was much lighter and brighter (Figure 3).



Figure 3: Left: Some “classic” material reportedly from old diggings in the Mambilla area. Right: A parcel of material reportedly from the new deposit in the Mambilla area. These two groups look very similar except for the color which is much lighter for the stones from the new diggings. Photo: Vincent Pardieu © GIA.

After further discussions with West African people who claimed they had visited the area, it looked as though the new deposit was located about 10 km north of Guroji, a small village about 30 km north west of Gembu (Figure 4). After studying a geological map of the area and using Google Earth (Figure 7) this location makes sense as it is clear that Guroji is located within a basalt rich area.

Such a conclusion came as no surprise to VP as some areas like O Ta Prang close to Pailin in Cambodia also produce light blue sapphires that respond very well to heat treatment, while about 3 km further down the same stream an area called Suon Umpal is known to produce darker stones of lower commercial value (Pardieu 2009).



Figure 4: Local map (modified from Google Earth) of the Mambilla Plateau area with the approximate location of the new deposit shown, near Guroji according to the information provided by local miners. The old diggings producing small dark blue stones seem to be located near Gembu close to the border with Cameroon. The exact location of these deposits still needs to be confirmed. Note: The photos of the single stones illustrated in this map are of one stone weighing over 100 carats, reportedly from the new deposit, and another of about 8 carats courtesy Brume Jeroh. Map: Vincent Pardieu / GIA Laboratory Bangkok.

Sadly the GIA field expedition to Nigeria planned for the end of April 2014 had to be postponed and with the advent of the rainy season in Nigeria, it was difficult to plan another expedition before the completion of this report. Thus this study will only be a “preliminary report” as it is based on F type samples (per GIA Field Gemology department terminology, see Annex A) which means that these samples were collected from trustworthy West African sources in Bangkok. At the beginning of June 2014, as the field expedition was postponed, we decided to acquire some interesting parcels of fine quality rough sapphires, reportedly from the Mambilla area, in order to start a preliminary study on the new material. Our plans are to confirm these results in future after visiting the new sapphire mining area and completing a study of the samples collected on site at the time.

While working on this preliminary study we also met Mr. Saoud Kouyate, a Guinean merchant, in Bangkok, who claimed he had visited the deposit at the end of June 2014. According to him a Nigerian company called “Gendutse Investment” recently acquired the mining license for the area where the new discovery occurred. Mr. Kouyate was kind enough to put us in touch with Mrs. Hauwa Ibrahim, who works for the company in Nigeria. She sent us several photographs of the new mining site (Figure 5, Figure 6) and confirmed the location of the new mining site as being approximately 10 km north of Guroji village on the Mambilla Plateau.



Figure 5: General view of the new sapphire deposit on the Mambilla Plateau as seen at the beginning of July 2014. The diggings along the stream are clearly visible as is a village that has been built by the thousands of miners who moved in to work the new deposit. Photo: Modified from Mrs. Hauwa Ibrahim / Gendutse Investment, Nigeria.



Figure 6: Four different more detailed low resolution views taken from a video created by a mobile phone showing the diggings along the stream where sapphires are recovered. Most of the work is carried out by hand by the thousands of illegal miners who moved to the area after its discovery during spring 2014. Photos: Mrs. Hauwa Ibrahim / Gendutse Investment, Nigeria.

The photos in Figure 6 show that the deposit extends over approximately 2 km along a stream in a hilly area matching the Google Earth view of the area described by Mrs. Ibrahim. It seems that the miners built two large camps with several hundred houses covered with reflective aluminum roofs near the mining site. People mine using simple hand tools in rather shallow pits. After a few months of intense mining following the discovery of the new area, the sapphire mining activity reportedly decreased with the arrival of the rainy season in June 2014 and is expected to start again once the rainy season passes around September or October.

Background on Nigerian sapphires and their deposits:

Nigerian sapphires are known from different basalt related localities, all of them located within the central and eastern parts of the country (Kiefert and Schmetzer 1987; Kanis and Harding 1990; Michelou 2007; Raw_material_Research_and_Development_Council 2010):

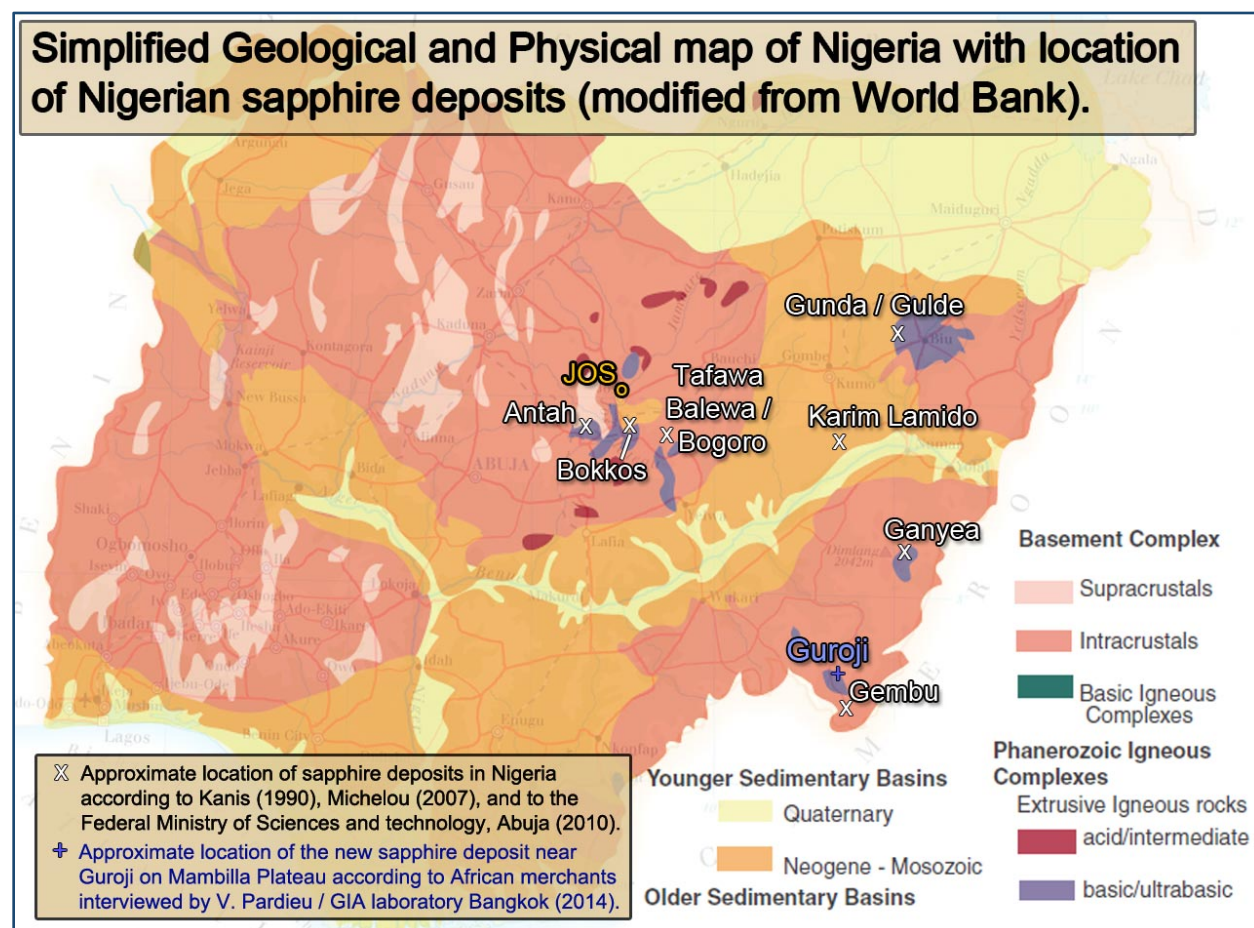


Figure 7: Sapphire deposits in Nigeria. Map modified from "Simplified Geological Map of Nigeria" by World Bank.

Near Jos, the main gem trading center in Nigeria, such sapphires are found in the Jemaa District (Kanis and Harding 1990) of the Kaduna State. According to Michelou, the main mining site in the Jemaa District, called “Antah”, produced significant quantities of sapphires over the past 25 years. It is probably the same location visited by Mr. Petch at the end of the 1980’s and studied by Kiefert (Kiefert and Schmetzer 1987), even though there is no mention of any specific mine in their article, only reference to the Kaduna State. Several other sapphire producing localities are reported by Michelou in 2007 and the “Federal Ministry of Science and technology” in 2010.

Mining for sapphires occurs near Jos at Gidan Waya (near Antah) in Kaduna State; near Bokkos in the Plateau State; and near Bogoro in the Bauchi State. Sapphires also reportedly occur near Gunda and Gulde in Yobe State; and/or Borno State; near Ganye in the Adamawa State; and near Karim Lamido in the Taraba State (J.C. Michelou, 2007 considered this deposit to have the best commercial potential at the time). Finally Michelou describes some mining near Gembu on the Mambilla Plateau near the border with Cameroon in the Taraba State. He describes the stones as usually being small but with a nice saturated blue color and good form. He added that the Gembu deposit extends to the other side of the border with Cameroon.

While Nigeria is a country rich in sapphire deposits, the stones are not considered very good quality by the market as most of them are too small, too dark or too included to be viewed favorably. Nevertheless, during a visit to Australia in 2012, a very experienced sapphire miner informed author VP that some sapphires found in Nigeria were amongst the best stones he saw from basalt related type material (Jack Wilson, Pers. Comm.).

Besides this positive comment it seems that the recent discovery of these large, transparent to milky, light blue sapphires from the Mambilla Plateau near Guroji, is something that was never encountered before from Nigeria in terms of quality.

Description of the rough sapphires from the new deposit near Guroji:

Sample description and selection:

After checking several parcels, reportedly from the new deposit on the Mambilla Plateau, the GIA Laboratory acquired, with the help of Martial Curti (G.G.), a parcel of 30 sapphires, with stones seen as being representative of the production from the new deposit (Figure 1). At first sight it was clear the sapphires from the new and old diggings were slightly different. The material obtained from the old diggings was much darker than the new material. Nevertheless the rest, aside from the size of the stones, appeared to have been mined from similar basalt related secondary deposits.



Figure 8: Parcel of 30 rough blue sapphires reportedly from the Mambilla area, Nigeria acquired in Bangkok on June 05th 2014 for the GIA reference collection. The weight of the stones is between 1 and 5 carats. Photo: Vincent Pardieu © GIA.

The sapphires ranged in color from light blue to deeply saturated blue. Interestingly no yellow or green material was seen in any of the parcels seen in Bangkok that originated from either the new or the old diggings on the Mambilla Plateau. But some yellow and greenish stones were reportedly found by traders in Nigeria (Jeroh Brume, Pers. Comm.). The dichroism seen in these sapphires is rather limited suggesting that the stones are not so rich in iron. This is very unusual for basalt related sapphires and a very positive point as greenish sapphires have a

lower market value than blue ones. The rough varied from hexagonal prismatic habits to some of a tabular habit. To find so many tabular pieces is again rather unusual for blue sapphires and another very positive element as it means lapidarists will be able to position the table more or less perpendicular to the c-axis and produce stones with attractive blue colors. While most stones showed some color zoning it did not appear to detract from the even blue color exhibited, another positive point. Finally the clarity ranged from clean to milky with a limited number of mineral inclusions and fissures as is commonly the case with sapphires from secondary deposits.

Note: The light colored semi-translucent milky sapphires are often referred to as “Geuda type” in the trade. These sapphires, even though they don’t look blue and transparent, are of specific gemological interest to the trade as their color and clarity may potentially be improved by heat treatment via techniques developed in Thailand at the end of the 1960’s (Coldham 2003; Pardieu 2006).

Features like the milky appearance, the absence of silky stones and the limited dichroism indicate that these sapphires are likely to possess a moderate iron content (no silky stones, no green or yellow stones), and some relatively high titanium (milky appearance), reminiscent of sapphires found around Pailin in Cambodia.

The rough often showed an etched rounded surface as it is commonly encountered on the surfaces of basalt related sapphires (Hughes 1997). Some surfaces were rounded from weathering and many had some surface reaching fissures filled with a dry mud-like (limonite) foreign substance as is the case in all secondary type deposits where rubies or sapphires are found in association with gravels within former river beds resulting from the weathering of basalt.

Overall, the parcels of sapphires seen from the new deposit on the Mambilla Plateau in Nigeria look particularly interesting with good combination of large size, nice form, high clarity and attractive colors.

For this preliminary study we selected:

- 5 stones (out of the 30 samples acquired) representative of the color and clarity of the sapphires from the new deposit. These were clean enough to be fabricated as wafers in order to collect spectroscopic data using UV-Vis-NIR and FTIR spectroscopy. Some also possessed interesting inclusions.
- 10 stones, reportedly from the old diggings on the Mambilla Plateau, in order to compare the new and old material.

After collecting data on these stones, we selected two stones for the first part of this study (see Figure 9): as we believe that they are representative of the clean and milky types of sapphires originating from the new deposit near Guroji:

- Sample 100309934812, light blue highly transparent stone, typical of the type of “facet” grade material that will be fashioned without heat treatment.
- Sample 100309934811, milky blue stone showing no visible dichroism and a Tyndall effect. Typically this “geuda” type material is likely to be heat treated before being fashioned.

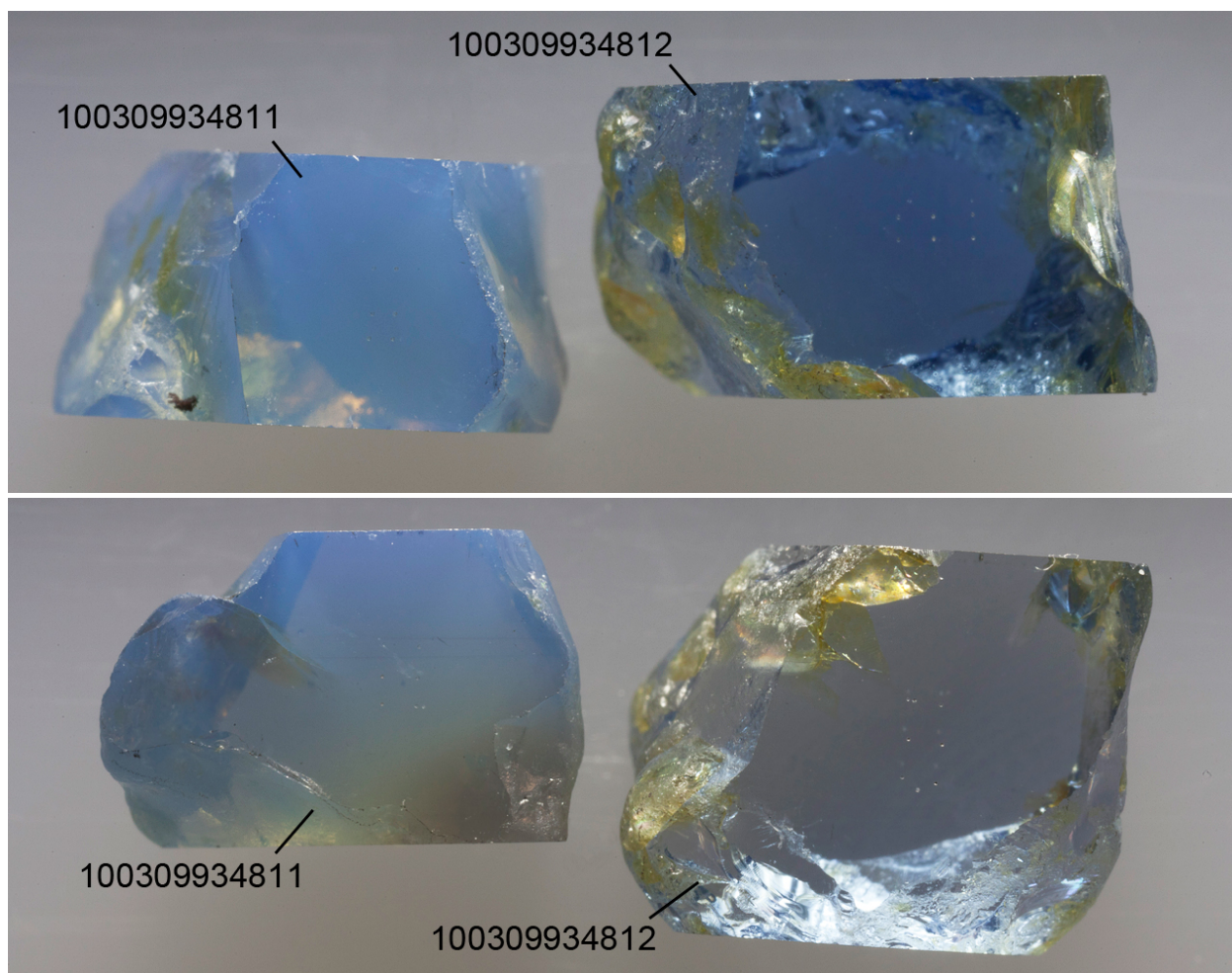


Figure 9: GIA reference samples 100309934811 (milky type) and 100309934812 (transparent type) seen after fabrication into wafers using natural light coming from above the photo. The top photo shows the stones in the direction of the c-axis, while the bottom photo shows them perpendicular to the c-axis. The transparent stone on the right shows dichroism while the milky blue stone on the left doesn't. Photos: Sasithorn Engniwat © GIA.

The gemological properties of the 2 samples are summarized in the following tables:

Table 1: Gemological properties of the 2 sapphire samples studied in this study.



Reference #	Max. RI	Min. RI	Birefringence	LWUV	SWUV	Sample type	Photo (transmitted light)
100309934811	1.770	1.761	0.009	Inert	Inert	Milky	
100309934812	1.769	1.760	0.009	Inert	Inert	Clean	

Table 2: Other gemological data/details of the 2 sapphires described in this study.

Reference #	Sample Category & Origin	Wafer Weight (carats)	Polished wafer orientation	Wafer path length (mm.)	Color (in daylight)
100309934811	F2 "New area, Guroji"	1.534	Parallel to the c-axis	3.530	Brownish blue
100309934812	F2 "New area, Guroji"	2.439	Parallel to the c-axis	4.081	Light blue

The other stones from the new and the old deposits on the Mambilla Plateau were used for the second and third parts of this study, focusing on:

- An inclusion study of the sapphires from the Mambilla Plateau.
- A trace element study of 5 sapphires from the new deposit on the Mambilla Plateau in comparison with sapphires from the old diggings and some stones from other basalt related deposits.

Sample Fabrication:

All the samples in this study were fabricated in the GIA Laboratory Bangkok by Jonathan Muyal and Victoria Raynaud. The samples were fabricated for 2 different purposes:

5 samples were found to be too included to collect high quality optical data from, so they were fabricated in order to document their inclusions. In some cases one or more windows were polished perpendicular to the c-axis, while in other cases, directions not specifically related to the c-axis were windowed in order to optimize the study of their internal features.

10 samples were found to be clean enough to be fabricated as “spectra quality samples”. To fit into this group the samples had to possess a clean area large enough to enable high quality reference spectra to be collected along the c-axis, and/or perpendicular to the c-axis.

- 4 samples were fabricated with one set of surfaces polished perpendicular to the c-axis.
- 2 samples were fabricated with one set of surfaces polished parallel to the c-axis.
- 4 samples possessed shapes good enough to be fabricated as optical wafers with two sets of surfaces carefully polished perpendicular and parallel to the c-axis of the crystal. One of them (sample 100309934812) was later sliced and fabricated again into 2 thin wafers in order to collect better quality spectroscopic data.

These 10 samples were subsequently polished with great care to enable good quality spectra to be collected. Optical path lengths of the wafers were measured with a Mitutoyo Series 395 spherical micrometer with an accuracy of 2 microns.

Instrumentation:

- Sample photography:

To document the color of our samples, we used a Canon EOS 5D camera with a Canon Macro MP-E 65mm lens adapted to a camera stand. In order to produce consistent results for each GIA reference sample the photographs were taken under exactly the same lighting conditions, with the reference samples being placed in a Logan Electric Tru-View 810 Color Corrected Light Box (5000K lamp). A neutral density filter was used to calibrate the camera light box combination to produce a neutral gray. High-resolution reference photographs

were then collected using transmitted light. As the reference photos were taken of wafers cut perpendicular or parallel to the c-axis, the color of the samples in the photographs taken using transmitted light can be considered representative of the color, respectively, of a nearly pure o-ray or e-ray.

Photomicrographs of internal features were captured at up to 180x magnification with a Nikon SMZ 1500 system using darkfield, brightfield, diffused and oblique illumination, together with a fiber-optic light source when necessary. *Note:* The field of view information in the captions was calculated based on the magnification power of the microscope. On each photo a scale has been added showing 25% of the total field of view for the sake of consistency. The measurement on the scale was rounded up to the nearest whole number after the decimal place for better clarity.

- **UV-Vis-NIR spectroscopy:**

Ultraviolet-visible-near infrared (UV-Vis-NIR) spectra were collected using a Hitachi U-2910 spectrophotometer for polarized ordinary and extraordinary rays, integrating with a polarizer accessory controlled by ThorLabs APT. We used 1.5 mm as the slit width. The spectra obtained were corrected by calculating the reflection loss from the index of refraction data and the data was converted to absorption coefficients using $\alpha = 2.303A/d$ where “d” is the depth in centimeters.

- **Infrared Absorption (FTIR) spectroscopy:**

Fourier-transform infrared (FTIR) spectroscopy was performed using a Thermo Nicolet 6700 FTIR spectrometer equipped with an XT-KBr beam splitter and a mercury-cadmium-telluride (MCT) detector operating with a 4x beam condenser accessory, and resolution was set at 4 cm^{-1} with 1.928 cm^{-1} data spacing. The spectra obtained were converted to absorption coefficients using $\alpha = 2.303A/d$ where “d” is the depth in centimeters.

- **Raman spectroscopy:**

Raman spectra were used to identify inclusions using a Renishaw inVia Raman microscope fitted with a 514 nm argon-ion laser. The Raman spectra were collected in the range 100 and 1500 cm^{-1} . The accumulation was set at 50 to improve the signal to noise ratio of the spectra. Spectra were calibrated using the 520.5 cm^{-1} line of a silicon wafer. In all cases, we used the RRuff database as a reference in our attempts to identify inclusions. Spectra comparisons were performed using Renishaw Wire (version 3.4) and/or Thermo Galactic “Spectral ID” (version 3.02) software.

- **Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS):**

For chemical analysis, we used LA-ICP-MS technology with a Thermo Fisher Scientific iCAP Q Induced Coupled Plasma - Mass Spectrometer (ICP-MS) coupled with a Q-switched Nd:YAG Laser Ablation (LA) device operating at a wavelength of 213 nm. Laser conditions used 55 μm diameter laser spots, a fluency of around 10 J/cm^2 , and a 15 Hz repetition rate. 12 spots were analyzed on each wafer. For the ICP-MS operations, the forward power was set at $\sim 1350 \text{ W}$ and the typical nebulizer gas flow was $\sim 1.00 \text{ L/min}$. The carrier gas used in the laser ablation unit was He, set at $\sim 0.60 \text{ L/min}$. The criteria for the alignment and tuning sequence were to maximize Be counts and keep the ThO/Th ratio below 2%. A special set of synthetic corundum reference standards were used for quantitative analysis. All elemental measurements were normalized on Al (internal element standard), this value approximates to the chemical composition of corundum.

Part 1: Preliminary study of two GIA reference samples reportedly from the new deposit near Guroji:

GIA Reference Specimen Number 100309934812:

GIA reference sample 100309934812 is an F2 type sample (See Annex A). The specimen weighed 3.427 carats before fabrication (Figure 10). It had a light blue color with weak dichroism and was particularly transparent. It is a typical facet grade sample. Such material will usually be faceted without requiring heat treatment. A few unhealed surface reaching fissures were filled with some yellow to brown foreign substance of natural looking origin (probably limonite).



Figure 10: GIA reference sample 100309934812 before fabrication. The photos were color calibrated and represent the same sample in different directions using transmitted light. The field of view is 1 centimeter. Photos: Sasithorn Engniwat © GIA.

The stone was carefully fabricated into two thin optical wafer samples 100309934812-A and 100309934812-B (Figure 11), weighing 0.911 cts and 0.908 cts respectively, with two sets of surfaces carefully polished perpendicular and parallel to the c-axis of the crystal.

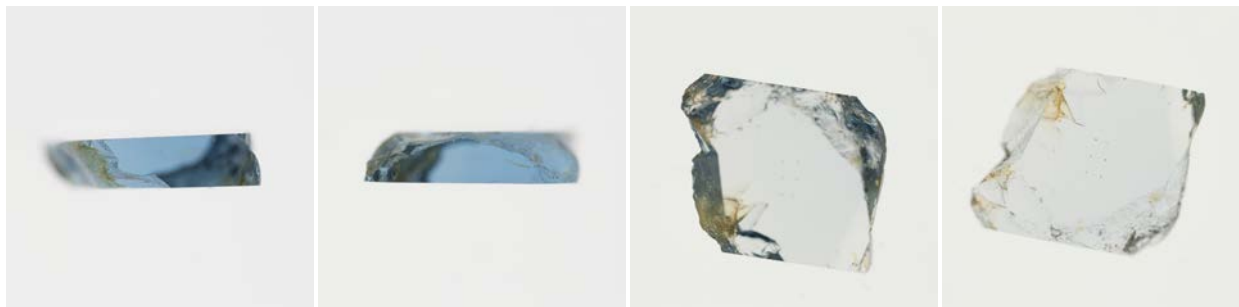


Figure 11: GIA reference sample 100309934812-B after fabrication. The photos were color calibrated and captured using transmitted light. In the two photos on the left, the sample is seen parallel to the c-axis of the crystal, while on the right it is seen perpendicular to the c-axis of the crystal. In this photo series, the dichroism is visible more clearly with the best color (medium blue) being in the direction of the c-axis, while perpendicular to the c-axis the color seen is a very light blue. The field of view is 1 centimeter. Photos: Sasithorn Engniwat © GIA.

When viewed under long-wave and short-wave UV light no reaction was observed.

Microscopic examination and Raman spectroscopy:

GIA reference sample 100309934812-B only contained a few inclusions consisting of very low density clouds of minute particles barely visible using a microscope at 40x magnification and fiber-optic illumination. When studying the sample in a very clean area with Raman spectroscopy, we collected, as expected, some very clear spectra that matched the RRuff database R040096 corundum reference spectrum almost perfectly.

UV-Vis-NIR and FTIR Spectroscopy:

Polarized UV-Vis-NIR spectra (o-ray and e-ray) were collected from the specimen in a clear area with no visible particles or mineral inclusions in the beam path.

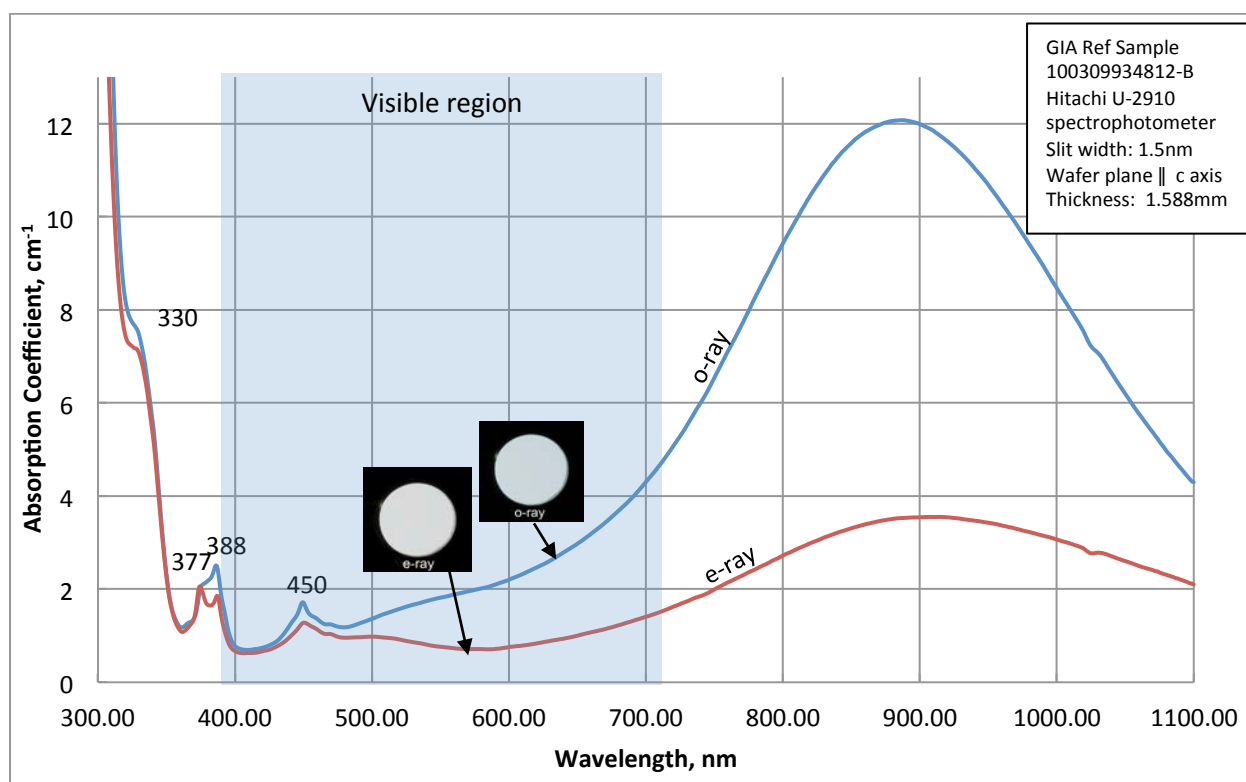


Figure 12: Polarized UV-Vis-NIR absorption spectra of GIA reference specimen 100309934812-B with inset color calibrated polarized photos of the beam path area for the o- and e-rays. Optical path length: 1.588 mm. Weight: 0.908 carats. Color: Even light blue.

The spectra collected (Figure 12) show a strong broad absorption band centered around 900 nm in the near infrared region. This absorption feature is characteristic of basalt related blue sapphires. It could be due to an Fe^{2+} - Fe^{3+} related broad band (Fritsch and Mercer 1993). Fe^{3+} related series are also visible at 377 and 388 nm in the UV region and at 450 nm in the violet region of the spectra. The 377 and 450 nm are associated with Fe^{3+} pairs while the 388 nm is associated with single Fe^{3+} ions. A broad band is also visible around 500 to 600 nm in the o-ray. This band is the main reason for the blue coloration in the sapphire as it absorbs light in the green, yellow and red parts of the visible spectrum (indicated in light blue on Figure 12). It can be attributed to some limited charge transfer absorption by Fe^{2+} - Ti^{4+} . The absorption edge can be observed at 307 and 308 nm for e-ray and o-ray, respectively. It is interesting to note that the minimum absorption levels for the o- and e-rays are about the same, with the e-ray being a little lower than that of the o-ray.

An infrared spectrum (Figure 13) was collected for the stone through the same area and orientation used to collect the UV-Vis-NIR spectrum. Some weak absorption features were observed at 3309 and at 3232cm^{-1} , the latter one being indistinct.

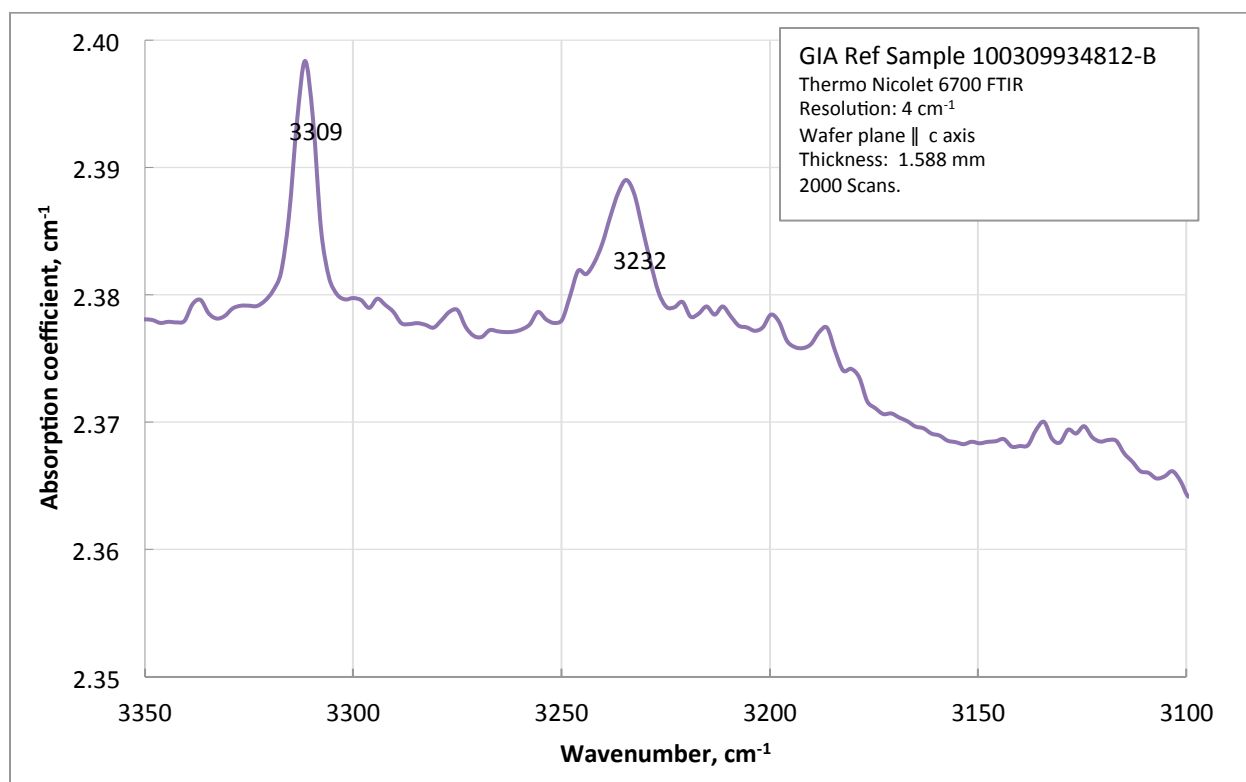


Figure 13: FTIR spectrum of GIA reference specimen 100309934812-B in the 3200 to 3340 cm^{-1} range. Optical path length: 1.588 mm. Weight: 0.908 carats. Color: Even light blue.

Chemistry:

LA-ICP-MS chemical data (Table 3) was collected on each side of GIA reference sample 100309934812. Six spots were analyzed on each side of the wafer, twelve spots in total (Figure 14) in the same area studied with FTIR and UV-Vis-NIR spectroscopy.



Figure 14: GIA reference sample 100309934812 showing the location of the 12 spots where LA-ICP-MS analysis was conducted for this preliminary study on each side of the wafer. Photo: Sasithorn Engniwat © GIA.

Table 3: LA-ICP-MS results for GIA reference sample 100309934812. “BDL” stands for “Below Detection Limit”, “BQL” stands for “Below Quantification Limit”

Specimen 100309934812 Spot number (color of the area)	9Be	24Mg	47Ti	51V	52Cr	56Fe	69Ga	90Zr	93Nb	178Hf	181Ta	182W	232Th
SP1 (light blue)	BQL	0.9	14.1	BDL	BDL	832.5	49.4	BDL	0.04	BDL	0.01	BDL	BDL
SP2 (light blue)	BQL	BQL	17.3	BDL	BDL	865.3	48.0	BDL	0.02	BQL	0.01	BDL	BDL
SP3 (light blue)	BDL	BQL	12.4	BQL	BDL	847.1	48.8	BDL	0.02	BDL	0.01	BDL	BDL
SP4 (light blue)	BDL	BQL	14.5	BDL	BDL	865.3	48.3	BDL	0.03	BQL	0.02	BDL	BDL
SP5 (light blue)	BDL	BQL	11.5	BQL	BDL	821.5	46.5	BDL	0.02	BDL	0.01	BDL	BDL
SP6 (light blue)	BDL	BQL	21.3	BDL	BDL	664.5	40.4	BDL	BDL	BQL	0.01	BDL	BDL
SP7 (light blue)	BDL	BDL	23.0	BQL	BQL	668.2	43.0	BDL	QDL	BQL	0.01	BDL	BDL
SP8 (light blue)	BDL	BDL	17.6	BDL	BDL	635.3	40.4	BDL	QDL	BQL	0.01	BQL	BDL
SP9 (light blue)	BDL	BQL	18.9	BDL	BQL	628.0	39.2	BQL	BDL	BDL	0.00	BDL	BDL
SP10 (light blue)	BDL	BDL	23.3	BDL	BQL	639.0	39.8	BDL	BDL	BQL	0.00	BQL	BDL
SP11 (light blue)	BDL	BDL	22.5	BDL	BDL	606.1	38.6	BDL	BDL	0.00	0.01	BQL	BDL
SP12 (light blue)	BDL	BDL	21.0	BDL	BDL	613.4	39.2	BDL	BDL	BQL	0.00	BDL	BDL
Detection Limits	0.1	0.3	0.8	0.1	0.3	0.9	0.0	0.00	0.00	0.00	0.00	0.00	0.00

This method enables the best comparison possible between the chemistry and the FTIR and UV-Vis-NIR spectra collected from the sample. Nevertheless the comparison has its limitations as the techniques have some significant differences. While FTIR and UV-Vis-NIR spectroscopy are techniques analyzing the whole volume of the wafer in the beam path area, the LA-ICP-MS method only analyzes the components within the micron sized spots tested. This is fine if the

sample is homogeneous, but the correlation might be difficult if the sample has an uneven chemical composition.

This sample was interesting as it was very transparent, possessed a homogeneous color and contained no visible inclusions at magnifications up to 40x. The chemical data collected showed a very limited amount of trace elements present. Only significant amounts of iron (Fe), gallium (Ga) and a small amount of titanium (Ti) were detected. It is interesting to note that although the iron content is significant for sapphire, it is actually very low for a basalt related sapphire. Traces of niobium (Nb) and tantalum (Ta) were also detected but in such insignificant quantities as to be of no importance. This chemistry is compatible with the absence of fluorescence under both long-wave and short-wave ultra-violet lighting.

Discussion about the data collected from GIA reference sample 100309934812:

On the UV-Vis-NIR spectra (Figure 12) we can see that the blue color for the sample can be explained by the presence of a broad absorption band for the o-ray starting around 450 nm and increasing towards the red part of the visible spectrum. It relates to inter-valence charge transfer absorption by $\text{Fe}^{2+}\text{-Ti}^{4+}$. It is interesting to see that the fine blue color seen here is only produced by an average of 17 ppma of titanium content associated with iron. The main absorption feature in the UV-Vis-NIR spectrum of each ray is the broad band centered between 800 and 1100 nm. This very strong band, seen in all basalt related sapphires, is absorbing outside the visible part of the spectrum and thus it is not responsible for the blue color seen by human eyes.

The chemical data results provided by LA-ICP-MS clearly show that titanium is present. With no magnesium or beryllium to interfere with a possible charge transfer with Fe^{2+} , the chemistry looks compatible with the information provided by the UV-Vis-NIR spectra. It is interesting to note that the iron content is rather low for a basalt related sapphire because iron is often found at levels of over 1000 ppma and sometimes even over 3000 ppma in such sapphires (see Part 3 of the study).

Raman analysis of the sample resulted in a clear corundum spectrum that matched spectra in the RRuff database reference closely, except for two minute absorption features. Thus no significant second phases (other minerals) are, as expected, present.

FTIR analysis also failed to detect the presence of any mineral inclusions. All these results are compatible with the chemical data collected from the sample proving that this sapphire, besides some significant iron content and traces of gallium and titanium, is particularly pure.

Overall it is interesting to see that results obtained from the same area of the reference sample using different techniques (Raman, FTIR, UV-Vis-NIR and LA-ICP-MS) did not produce any conflicting results, rather to the contrary, which is very comforting.

GIA Reference Specimen Number 100309934811:

GIA reference sample 100309934811 is an F2 type sample (see annex A). It is an interesting example of what traders refer to as “Geuda” type sapphire. This translucent sample has a milky blue appearance via reflected light and a yellowish brown color via transmitted light (Figure 17). This is a clear example of the Tyndall effect due to the presence of minute particles scattering the light (Figure 17). Interestingly it shows no visible dichroism. This can be explained by the presence of the minute particles. The o-ray and e-ray polarization are randomized by the particles as a result of many multiple reflections.



Figure 15: Tyndall effect in GIA reference sample 100309934811. The sample was placed on a black slightly reflective background and a white “daylight” type fluorescent illumination illuminated it from above. As can be seen blue color is scattered from the sides of the sample but when light reaches the lower part of the stone it becomes greenish and then yellow as most of the blue is scattered away. Photo: Vincent Pardieu © GIA.



Figure 16: Tyndall effect in blue sapphire from the Mambilla Plateau area, Nigeria. Using fiber-optic illumination from behind, it can clearly be seen that perpendicular to the direction of the incoming light some blue color is visible. On the other hand, when looking through the sapphire in the direction of the incoming transmitted light the sapphire appears yellow as the blue component of the light is scattered away. Note also that the reflection of the light passing through the stone onto the table is more yellow than the reflected light that didn't pass through the sapphire. Photo: Vincent Pardieu © GIA.

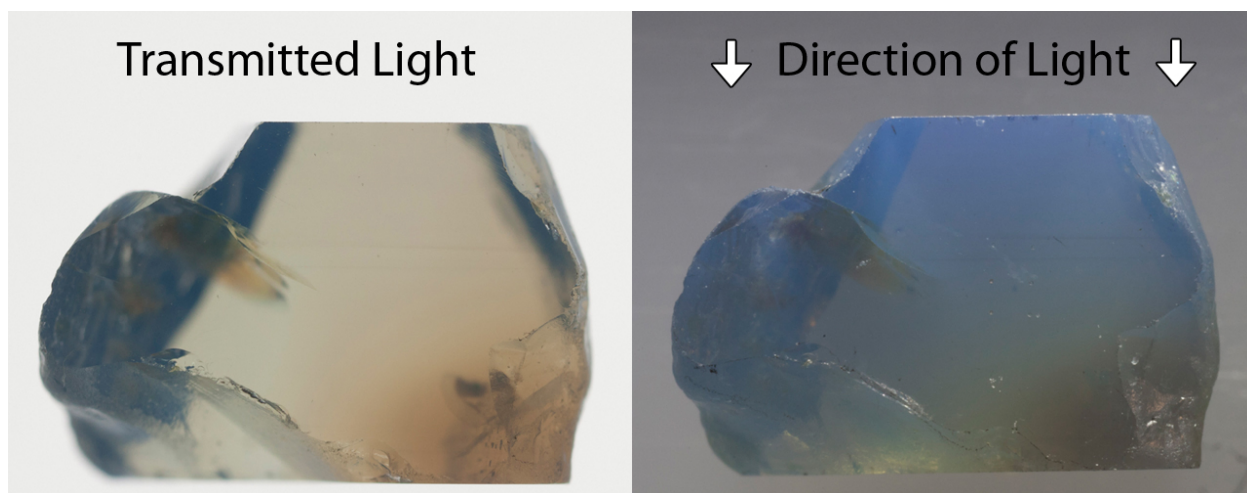


Figure 17: GIA reference sample 100309934811 seen using diffused transmitted light (left) and perpendicular to the direction of the light (right). The Tyndall effect is clearly apparent once again. This effect allows longer wavelengths (yellow, red) to be transmitted while shorter ones (blue, violet) are scattered. The result is that the sample appears blue from light scattering

when it is seen perpendicular to the direction of the light, while it appears yellow via transmitted light as the blue is scattered. Photos: Sasithorn Engniwat © GIA.

Such stones will usually be heat treated before fashioning in order to improve their color and transparency. It weighed 2.054 carats before fabrication (see Figure 18).

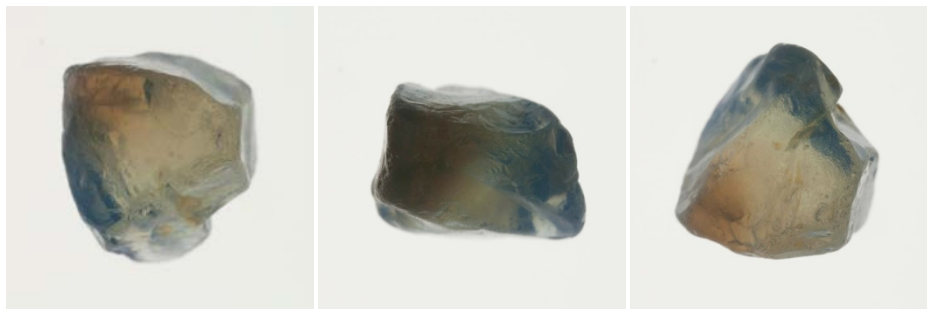


Figure 18: GIA reference sample 100309934811 before fabrication. The photos were color calibrated and represent the same sample in different directions using transmitted light. The field of view is 1 centimeter. Photos: Sasithorn Engniwat © GIA.

The stone was carefully fabricated as an optical wafer with two sets of surfaces polished perpendicular and parallel to the c-axis of the crystal (Figure 19). The fabricated wafer weighs 1.534 carats.

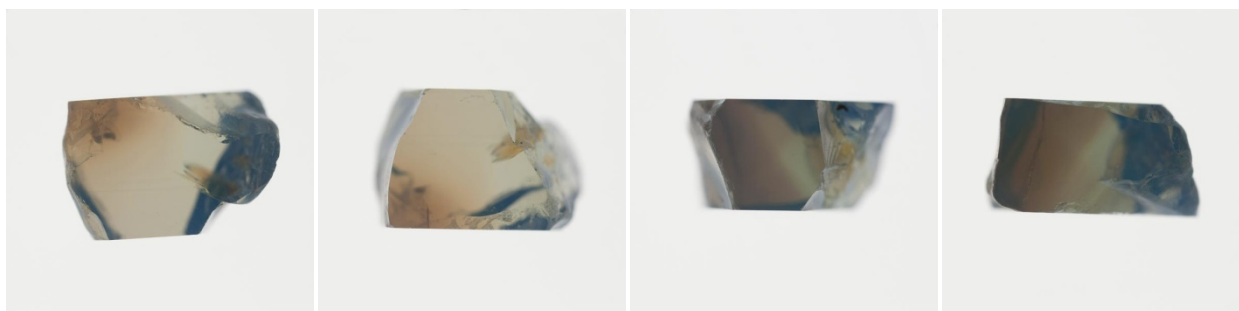


Figure 19: GIA reference sample 100309934811 after fabrication. The photos were color calibrated and represent the same sample using transmitted light. In the two photos on the left, sample 100309934811 is seen parallel to the c-axis of the crystal, while in the 2 photos on the right it is seen perpendicular to the c-axis of the crystal. It is apparent that the milky areas are not really even in the sample since one side is more brownish than the other. The field of view is 1 centimeter. Photos: Sasithorn Engniwat © GIA.

When viewed under long-wave and short-wave UV-light no reaction was observed.

Microscopic examination and Raman spectroscopy:

In GIA reference sample 100309934811 the inclusion scene, is composed of numerous bands of minute particles orientated in the direction of the c-axis.

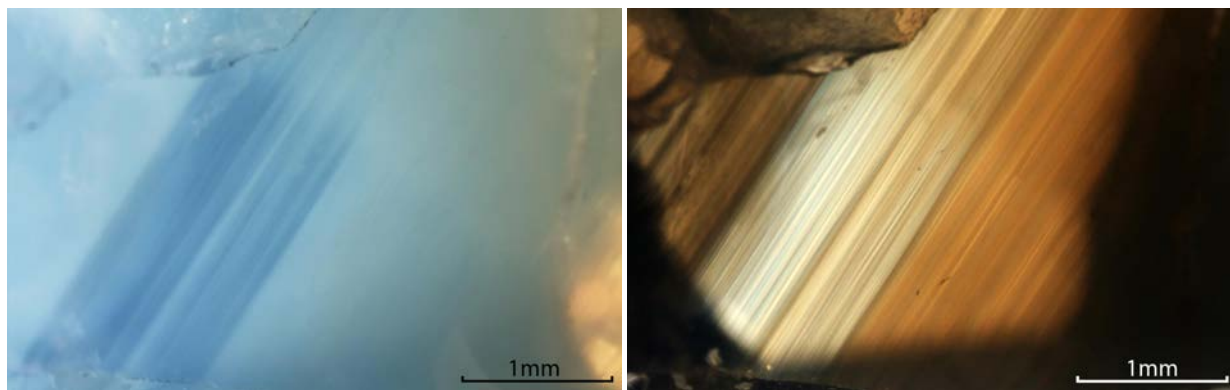


Figure 20: Left: GIA reference sample 100309934811, seen in the direction of the c-axis after fabrication, magnification 30x (field of view 4.1 mm). Left: Bands of whitish particles are visible scattering the light and giving the stone a milky bluish appearance (Tyndall effect) when viewed with fiber-optic lighting. Right: Under brightfield light the blue component of the white light is scattered away by particles, resulting in either yellow or brown milky bands depending of the particle concentration. Photos: Jonathan Moyal © GIA.

The Tyndall effect is also visible using the microscope as particles scatter the light and give the stone a milky blue appearance when studied via fiber-optic or darkfield illumination (Figure 20). When observed using brightfield illumination the particle rich sapphire appears yellow or brown (Figure 20, right) depending on the density of the bands of particles in specific areas as the wavelengths associated with the blue component are removed from the visible light by scattering.

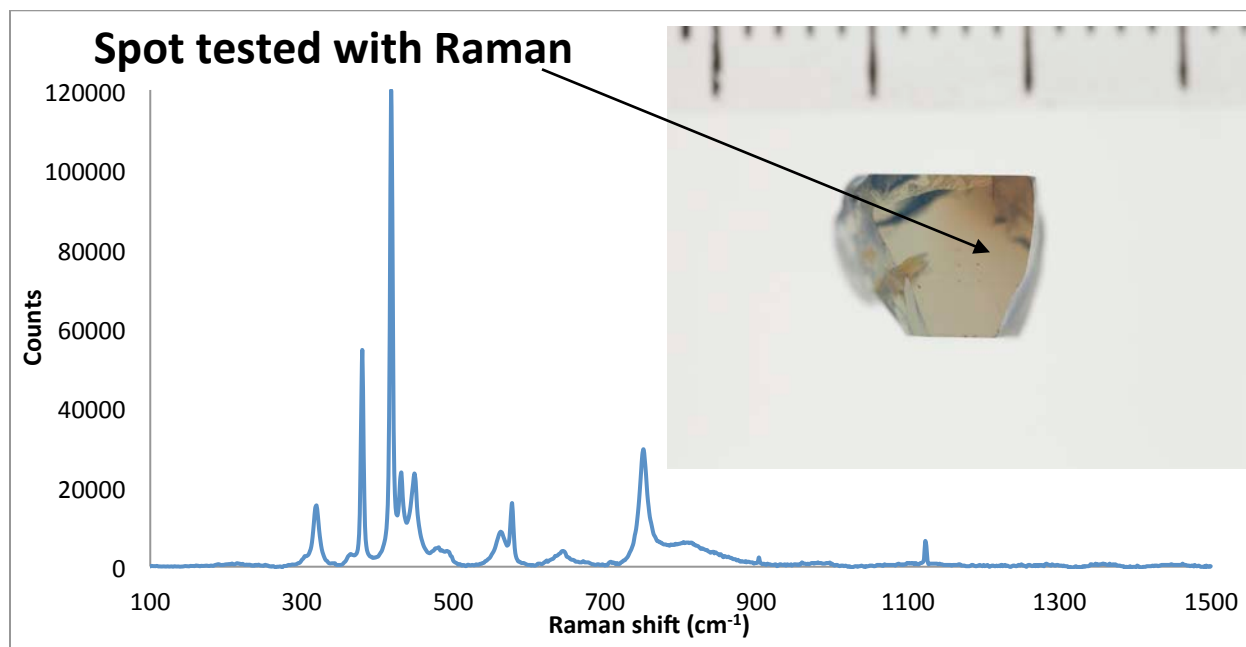


Figure 21: Raman spectrum of GIA reference sample 100309934811. It is clear that the spectrum collected in an area rich in minute particles is dominated by peaks associated with corundum © GIA.

Studying the sample with Raman spectroscopy in an area with some dense concentrations of minute particles, revealed, as expected, a spectrum (Figure 21) showing a combination of peaks indicating that corundum is present with at least one secondary mineral phase that could not be clearly identified.

UV-Vis-NIR and Infrared Absorption (FTIR) Spectroscopy:

Attempts to collect some polarized UV-Vis-NIR spectra from GIA reference sample 100309934811 in a similar way to those collected from sample 100309934812 proved unsatisfactory owing to the limited transparency of the milky sample containing particles that scattered the light. As particles scatter light they also scatter polarization in an unknown way. As a result we could not collect any valid optical data to present here. Light scattering from particles in gemstones is a very complex subject that goes beyond the scope of this study on sapphires from the Mambilla Plateau area in Nigeria, hence this issue will be discussed in a future publication. That being said, besides the absorption features related to particles scattered by light the UV-Vis-NIR data collected for the sample, was dominated by the presence of a broad band centered between 800 and 1100 nm that is characteristic of all basalt related sapphires.

An infrared spectrum (Figure 22) was collected for the stone through the same area where the attempts to collect UV-Vis-NIR data were carried out using the same orientation parallel to the c-axis.

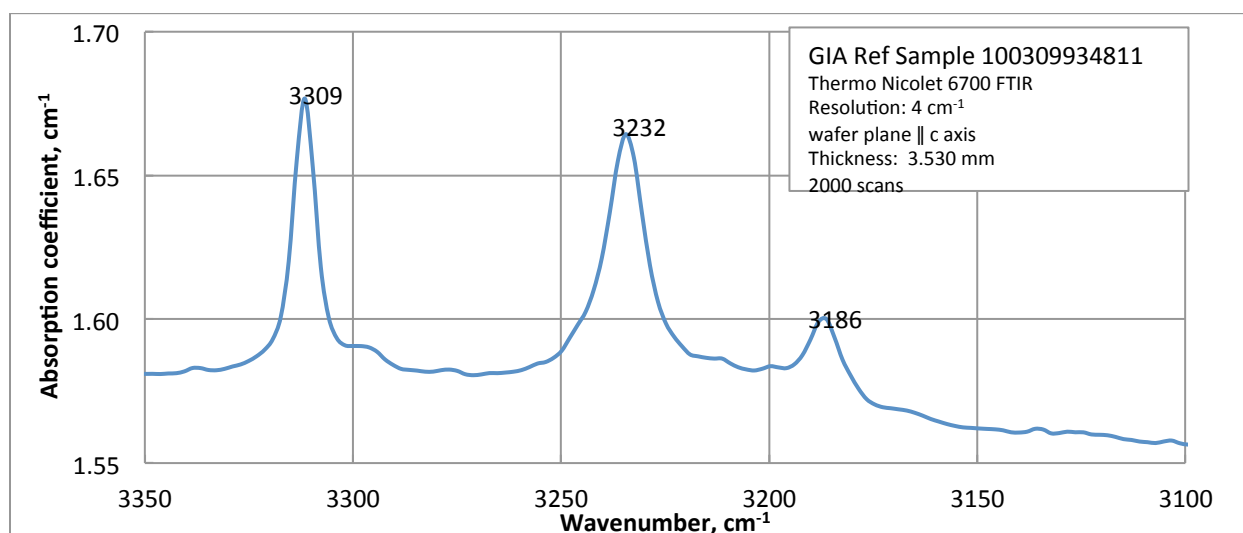


Figure 22: FTIR spectrum of GIA reference specimen 100309934811 in the 3100 to 3400 cm^{-1} range. Optical path length: 3.530 mm. Weight: 1.534 carats. Color: brownish yellow via transmitted light.

Some interesting absorption features were observed in the FTIR spectrum at 3309, 3232, and 3186 cm^{-1} . These are commonly found in natural unheated basalt related sapphires (Smith 1995). The 3309 cm^{-1} feature also commonly occurs in heated sapphires (metamorphic or basalt related) and in hydrothermal synthetic sapphires (Smith 1995).

Chemistry:

LA-ICP-MS chemical data (Table 4) was collected on each side of GIA reference sample 100309934811. Six spots were analyzed on each side of the wafer, twelve spots in total (Figure 23) in the same area studied with FTIR and UV-Vis-NIR spectroscopy.

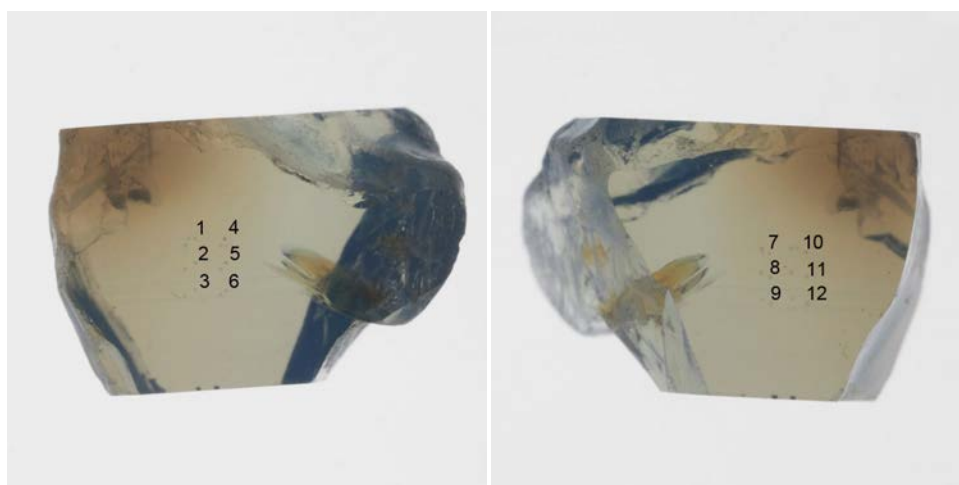


Figure 23: GIA reference sample 100309934811 showing the location of the 12 spots where LA-ICP-MS analysis was conducted for this preliminary study on each side of the wafer. Photo: Sasithorn Engniwat © GIA.

Table 4: LA-ICP-MS results for GIA reference sample 100309934811. “BDL” stands for “Below Detection Limit”, “BQL” stands for “Below Quantification Limit”.

Specimen 100309934811 Spot number (color of the area)	9Be	24Mg	47Ti	51V	52Cr	56Fe	69Ga	90Zr	93Nb	178Hf	181Ta	182W	232Th
SP1 (Milky brownish blue)	1.1	1.3	163.5	1.3	BQL	737.5	38.3	0.02	0.19	0.01	0.62	BQL	BDL
SP2 (Milky brownish blue)	1.4	1.2	175.9	1.4	BDL	785.0	36.9	0.01	0.22	0.01	0.60	BDL	BDL
SP3 (Milky brownish blue)	2.5	1.4	271.7	1.4	BDL	832.5	41.5	0.02	0.56	0.01	1.31	BDL	BDL
SP4 (Milky brownish blue)	1.1	1.0	262.8	1.4	BQL	828.8	40.4	0.04	0.72	0.01	1.52	BQL	BDL
SP5 (Milky brownish blue)	2.0	2.2	256.4	1.3	BQL	777.7	38.0	0.04	0.52	0.01	1.25	BDL	0.00
SP6 (Milky brownish blue)	2.0	1.7	298.6	1.5	BQL	766.8	37.4	0.03	0.76	0.01	1.58	BDL	0.00
SP7 (Milky brownish blue)	BDL	1.1	266.6	1.7	BDL	890.9	42.4	0.04	0.48	0.01	1.18	BQL	0.00
SP8 (Milky brownish blue)	0.9	1.3	237.2	1.4	BDL	774.1	37.1	0.03	0.40	0.00	0.93	BQL	0.00
SP9 (Milky brownish blue)	0.9	1.5	228.3	1.4	BQL	817.9	38.6	0.04	0.37	0.01	0.84	BQL	BDL
SP10 (Milky brownish blue)	1.1	1.6	267.5	1.6	BQL	788.7	37.7	0.05	0.56	0.01	1.15	BQL	0.00
SP11 (Milky brownish blue)	1.2	1.2	248.7	1.4	BQL	777.7	36.9	QDL	0.43	0.01	0.97	BDL	BDL
SP12 (Milky brownish blue)	1.1	1.1	247.0	1.5	BQL	792.3	35.1	0.03	0.41	0.01	0.84	BDL	0.00
Detection Limits	0.1	0.3	0.8	0.1	0.3	0.9	0.0	0.00	0.00	0.00	0.00	0.00	0.00

Sample 100309934811 has a similar iron and gallium content to that encountered in sample 100309934812. The iron content is quite low compared to that commonly observed in basalt related sapphires from other sources. However, in this sample the titanium (Ti) content is significantly higher (150 to 300 ppma) when compared to the 10 to 20 ppma levels usually encountered in basalt related sapphires from other sources. This was expected as sample 100309934811 possesses a milky appearance due to the presence of numerous minute exsolved particles, while sample 100309934812 lacks such inclusions. In conclusion it seems logical to believe that these particles are mainly composed of titanium oxide. More importantly, sample 100309934811 was found to contain low, but still noticeable, concentrations of magnesium (Mg), vanadium (V), beryllium (Be), zirconium (Zr), niobium (Nb), hafnium (Hf), tantalum (Ta), and thorium (Th). These interesting elements are probably associated with titanium in the minute particles as noted in sapphires from other deposits like Laos (Wathanakul 2004), Tasmania (Mc Gee 2005), Madagascar (Pardieu 2007; Shen 2012) and Afghanistan (Pardieu 2011).

Discussion about the data collected from GIA reference sample 100309934811 and comparison with sample 100309934812:

The UV-Vis-NIR data collected from this sample is dominated by absorption features that result from particles scattering light, and by the presence of a broad band centered between 800 and 1100 nm. The later is a classic feature of basalt related sapphires.

The combined data collected using magnification and Raman spectroscopy show that sample 100309934812 and sample 100309934811 mainly differ via the presence of numerous minute particles in sample 100309934811. The study of the data collected from LA-ICP-MS indicates that sample 100309934811 hosts a high amount of titanium (about 200 ppma versus about 20 ppma in the other sample), and some trace elements usually associated with Ti in nanoparticles (Shen 2011; Shen 2012). This means titanium is likely present in sample 100309934811 as titanium rich particles either in the form of rutile (no match for rutile was possible when using Raman) or more likely as TiO₂ with a PbO₂ structure as suggested by the presence of beryllium, niobium, tin and tantalum (Shen 2012). Another possibility would be ilmenite, since quite a good match with ilmenite was obtained when using Raman and the RRuff database as a reference. Examination via the microscope using darkfield and fiber-optic illumination clearly revealed minute particles as bands running in specific directions. Furthermore, it seems as though the particles are orientated since they reflect light differently depending on the direction of the light beam. Based on this observation we can deduce that the particles are probably not spherical but are more likely quite flat and orientated, thus their apparent size

might change depending on the direction of view (down the c-axis or perpendicular to it). These particles, probably of different sizes, are obviously responsible for the Rayleigh and/or Tyndall scattering effect that produce the blue color. These effects produce blue coloration in a transparent material when particles present are smaller (Rayleigh) or larger (Tyndall) when compared to the interacting wavelengths. As scattering takes place, less light reaches the detector. This creates an apparent absorbance. As the effect is inversely proportional to the square of the wavelength, for the Tyndall effect, and to the fourth power of the wavelength, for the Rayleigh, then short wavelengths are affected more than longer ones (Owen 1994).. This explain why the milky sapphire looks blue when observed perpendicular to the direction of the light (i.e. in darkfield or fiber-optic illumination when using the microscope or in one's hand) and brownish yellow when observed in transmitted light (i.e. in brightfield illumination when using the microscope or looking through it using one's hand).

To complete the discussion we can end with something of interest. When milky sample 100309934811 was selected it was because it was a sample of the "geuda" sapphire. This would undoubtedly mean that traders would probably heat treat such a sample to try and improve its color and clarity. After studying these two samples in detail it is now apparent that the fine blue color seen in sample 100309934812 is produced by an average of only 17 ppma of titanium associated with iron in an Intervalence Charge Transfer (IVCT) involving Fe^{2+} and Ti^{4+} pairs, whereas Sample 100309934811 hosts an average of more than 200 ppma of titanium with only about 2 ppma of combined magnesium or beryllium. This means that the combination of beryllium and magnesium ions will only be able to block a maximum of 2 ppma of Ti from taking part in any IVCT process with Fe^{2+} (Emmett, Scarratt *et al.* 2003). Thus, if all the titanium atoms, currently mostly present within the particle inclusions, are internally diffused by heat treatment in the sapphire (Emmett 2011), then potentially a concentration of Fe^{2+} and Ti^{4+} pairs about 20 times higher than what we have in sample 100309934812 will exist in the heated stone. With only 20 ppma of titanium the sample would show a fine blue, thus the stone resulting from the heat treatment of sample 100309934811 would probably appear a very dark blue or black. As a consequence the stone might not really be a perfect candidate for heat treatment as it would require some very skillful and careful heat treatment techniques to permit the right amount of Ti^{4+} ions to enter the sapphire in order to produce a fine blue color and not darken it too much.

Part 2: The internal world of sapphires from the Mambilla Plateau:

A microscopic study of the internal features seen in 5 GIA reference specimens reportedly from the new sapphire mining site near Guroji, and 10 samples from the old mining area was performed at the GIA Laboratory Bangkok. Overall the inclusions seen in these sapphires were very similar to those usually seen in basalt related sapphires from other deposits such as Pailin in Cambodia (Sutherland, Giuliani *et al.* 2009), Australia (Hughes 1997) or South Vietnam (Smith 1995), as the main inclusions seen in sapphires from the Mambilla Plateau area are bands of minute particles that scatter light, as well as feldspar, uranopyrochlore and zircon crystals.

Sapphires from the new deposit north of Guroji:

The most common inclusion feature seen in the 5 stones, were bands of minute particles that appear whitish (via darkfield or fiber-optic lighting) and colorless to yellow or brownish (via brightfield illumination) as is often the case with unheated sapphires from either basalt related or metamorphic type deposits. Some mineral inclusions such as feldspar crystals were identified using Raman spectroscopy in some of the sapphires.

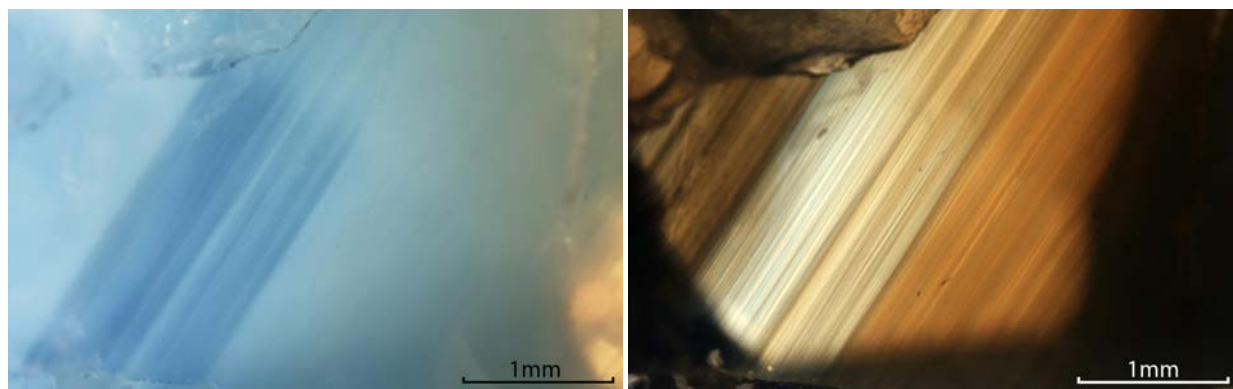


Figure 24: GIA reference sample 100309934811, seen in the direction of the c-axis after fabrication, magnification 30x (field of view 4.1 mm) Left: Bands of whitish particles are visible scattering the light and giving the stone a milky bluish appearance (Tyndall effect) when viewed with fiber-optic lighting. Right: Under brightfield light the blue component of the white light is scattered away by particles, resulting in either yellow or brown milky bands depending of the particle concentration. Photos: Jonathan Muyal © GIA.

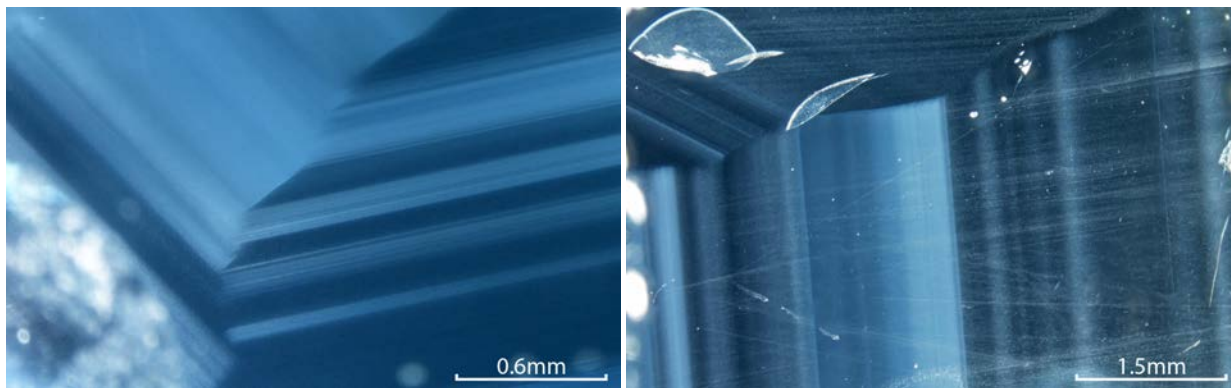


Figure 25: Left: GIA reference sample 100309934813, fiber-optic light illumination, magnification 50x (field of view 2.5 mm), showing bands of particles following a hexagonal growth structure. Right: GIA reference sample 100309934814, darkfield illumination, magnification 20x (field of view 6.3 mm), showing some bands of minute particles following a hexagonal growth structure with some other particles usually present as low density stringers more or less perpendicular to the bands. Photos: Jonathan Moyal © GIA.

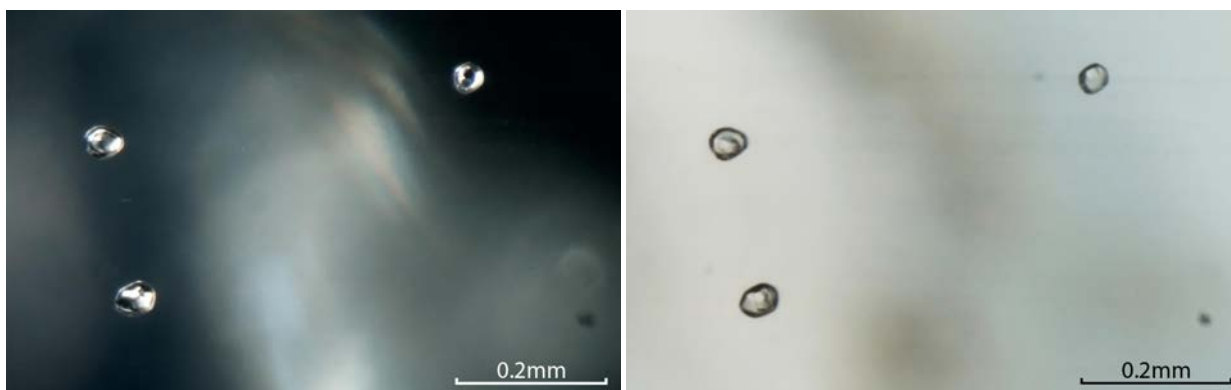


Figure 26: GIA reference sample 100309934814, left: darkfield, right: diffused brightfield illumination, magnification 150x, (field of view 0.8 mm) showing a group of transparent, rather rounded crystal inclusions identified by Raman as feldspar crystals. Photos: Jonathan Moyal © GIA.

Sapphires from the old diggings on the Mambilla Plateau:

The inclusion scene found in sapphires reportedly from the old diggings from the Mambilla Plateau was overall very similar to those found in the samples from the new diggings. Bands of minute particles and feldspar crystals were the most frequent encountered inclusions in both cases. Nevertheless since we possessed more samples from the old diggings and they were more included a greater variety of inclusions were observed during their preliminary study.

Twinning was found in several samples and in one case it was associated with negative crystals (Figure 27, Figure 28). Some very strong hexagonal growth structures were often seen and in one sample the growth structures reflected the light in a similar way to some twinning planes (Figure 29). Partially healed fissures were also present, whether associated with crystals or not. In some cases, when associated with crystals, the healed fissures looked very similar to the

classic discoid features found in heated sapphires. This goes to show that separating heated and unheated stones, particularly in the case of basalt related stones, as all gemologists know, can be quite a challenge. When attempting to separate heated and unheated stones, a gemologist's best guide may be bands of minute particles, when present, such as was the case in many of the stones examined in this study. In unheated sapphires the particle rich milky areas seen in darkfield lighting are associated with colorless to brown areas in transmitted light (depending on the concentration of the particles). This feature is called "chromophore cannibalization" by John Koivula. The difference in heated sapphires is that the particles are associated with blue areas as a result of internal diffusion. However, in one sample studied in this research most of the bands of particles seen in darkfield lighting were associated with colorless to brown areas in brightfield lighting as is commonly the case in unheated sapphires, yet at the same time several specific particle rich bands were also associated with blue areas (Figure 32). The authors have never seen such features in any sapphire that is known to be unheated and as a result some concerns about the true nature of the stone exist. Unfortunately this is sometimes the case with F type parcels obtained from secondary sources. It will be interesting to see whether other samples like this atypical one can be found in future parcels that are known to be unheated.

The bands of particles observed were usually composed of minute milky particles while in some other specimens they were more coarse and formed some flake or lathe-like structures (Figure 36, Figure 37) such as those seen in basalt related sapphires from Vietnam (Smith 1995). In some samples brown clouds of particles (Figure 35) resembling those Sri Lankan gem merchants call "chocolate" were seen in association with some whitish clouds. In some cases these "chocolate" clouds were extremely dense (Figure 38). Other much coarser particles were seen in comet tail like structures usually associated with feldspar crystals (Figure 37, Figure 41). The different appearance of the particles can most likely be explained by their size. Minute particles scatter the light and look milky, while coarser opaque particles will look like dark brown clouds. Another possibility is that they may be composed of different minerals.

The mineral inclusions present consisted mainly of crystals with feldspar being the most common. They appeared either as rounded crystals and/or as flattened and unevenly shaped colorless feldspar inclusions (Figure 38, Figure 39), often in association with brownish particles like comet tails (Figure 41). Other minerals were seen and identified including rounded uranopyrochlore crystals (Figure 39); fine euhedral (Figure 40) and rounded metamict zircon crystals associated with tension fissures (Figure 39); and also dark brownish opaque euhedral ferrotantalite mineral inclusions (Figure 41). Finally several black opaque flat hexagonal shaped crystals (Figure 30) that could not be identified by Raman spectroscopy were observed.

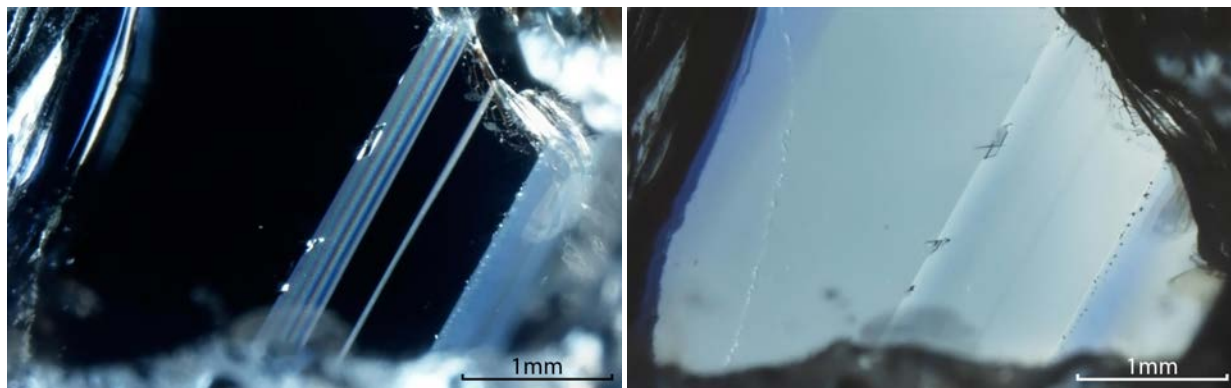


Figure 27: GIA reference sample 100309934765 magnification 30x (field of view 4.1 mm) in the same position in the two photos but viewed under darkfield (left) and under cross polars brightfield illumination (right). In both cases, the presence of lamellar twinning can clearly be seen. Photos: Jonathan Muyal © GIA.

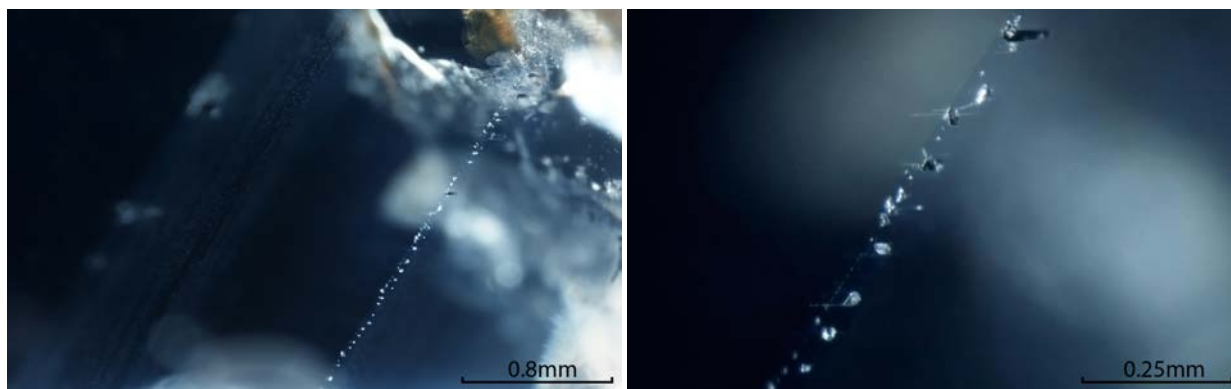


Figure 28: Detailed views of GIA reference sample 100309934765, darkfield illumination. Left, magnification 40x (field of view: 3.1 mm). Right: magnification 128x (field of view: 1 mm). One of the twinning planes hosts negative crystals, some of them associated with parallel tension fissures running perpendicular to the c-axis. Low density bands of minute particles are also visible between the twinning planes. Photos: Jonathan Muyal © GIA.

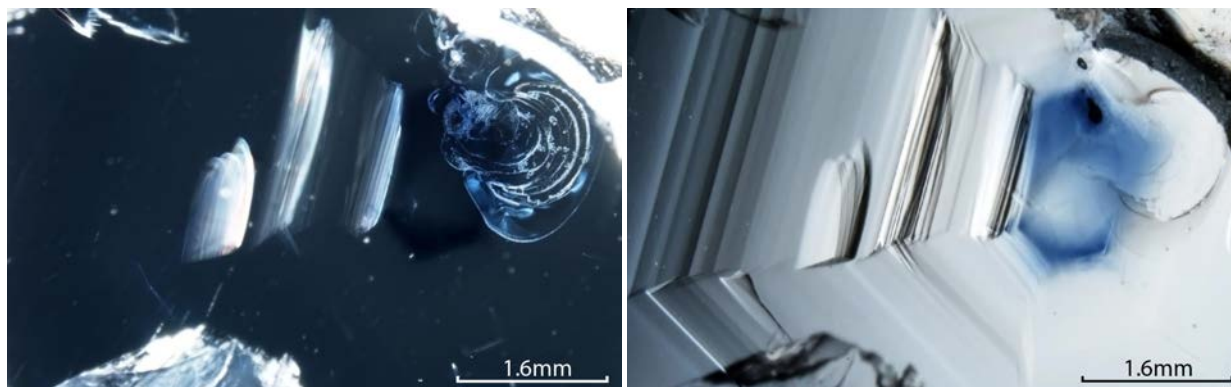


Figure 29: Views of GIA reference sample 100309934758 taken in the same position magnification 20x (field of view 6.3 mm). Left, via darkfield lighting some reflective planes associated with the hexagonal structure are visible. Near the blue core a crystal is associated with some partially healed fissures with a discoid appearance. Some low-density milky bands are visible at different intervals from the dark blue area. Right, view of the same area via brightfield illumination. The growth structure following the hexagonal core is much clearer with its interesting reflective planes. The bands of particles are clearly associated with the colorless area, while the blue central core is outside the particle rich area. This confirms the unheated nature of this stone. Photos: Jonathan Muyal © GIA.

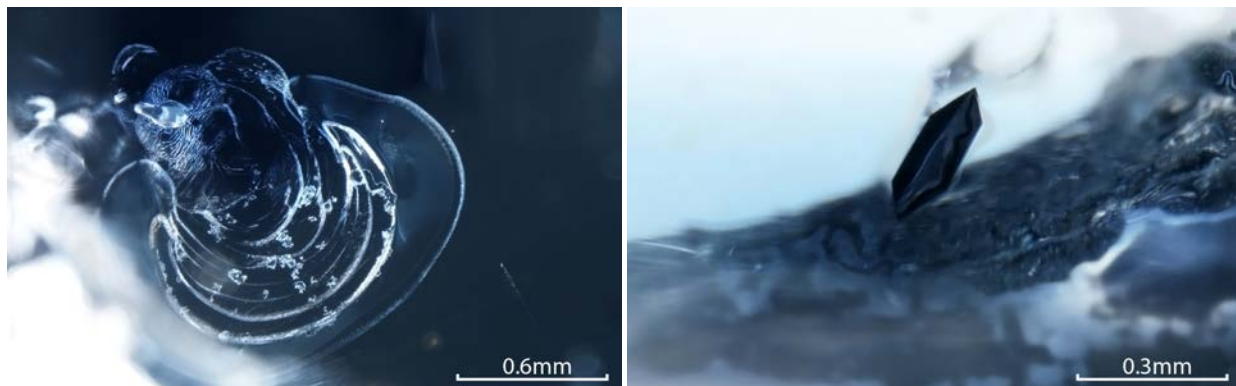


Figure 30: GIA reference sample 100309934758, Left: Darkfield illumination, magnification 50x (field of view 2.5 mm). Detailed view of the (unidentified) crystal associated with partially healed fissures that are themselves showing a discoid appearance and frosty looking fringes. Such inclusions are often seen in heated sapphires and unheated basalt related sapphires. Right: Diffused brightfield + fiber-optic light illumination, magnification 100x (field of view 1.25 mm). Detail of a black, opaque, flat, hexagonal looking (unidentified) crystal also present in the stone. Photos: Jonathan Muyal © GIA.

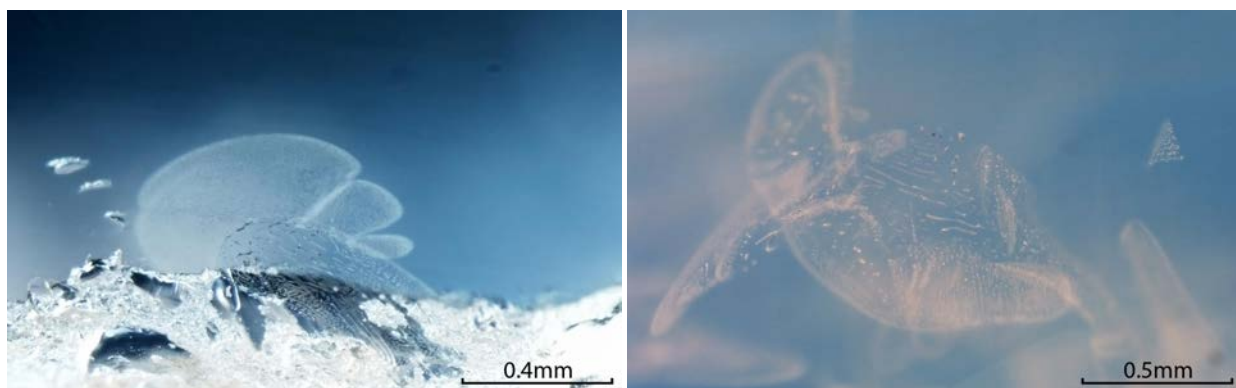


Figure 31: Left: Healed fissures seen in GIA reference sample 100309934765 via darkfield illumination magnification 80x (field of view 1.5 mm) Photo: Jonathan Muyal © GIA. Right: Healed fissures seen in GIA reference sample 100309934761 via darkfield illumination, magnified 60x (field of view 2 mm). Photo: Victoria Raynaud © GIA.

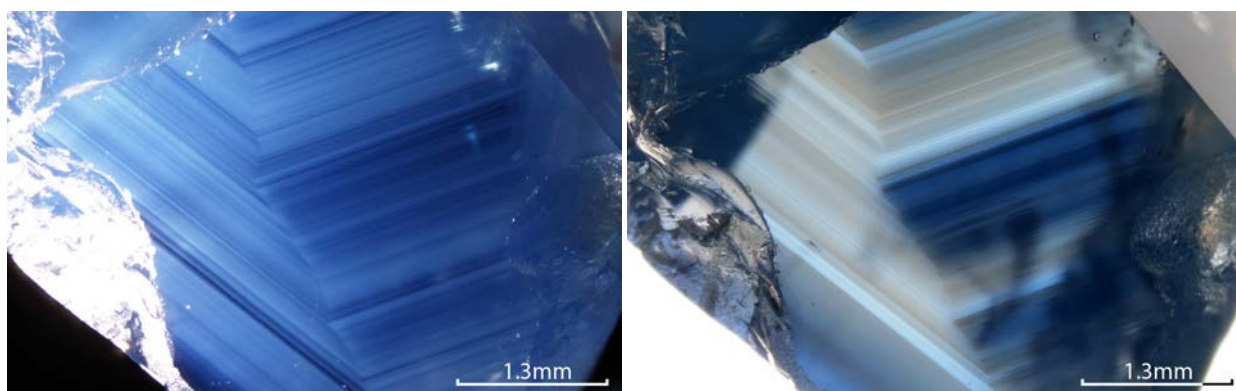


Figure 32: Two views of GIA reference sample 100309934763, magnification 25x (field of view 5.2 mm), on the left via fiber-optic lighting and on the right using diffused brightfield illumination. The bands of minute particles are clearly associated with the hexagonal structure in the left image. Interestingly while most of the bands of particles seen on the left are associated with brownish areas, some specific bands of particles are associated with blue areas as can be seen on the right. Photos: Jonathan Muyal © GIA.

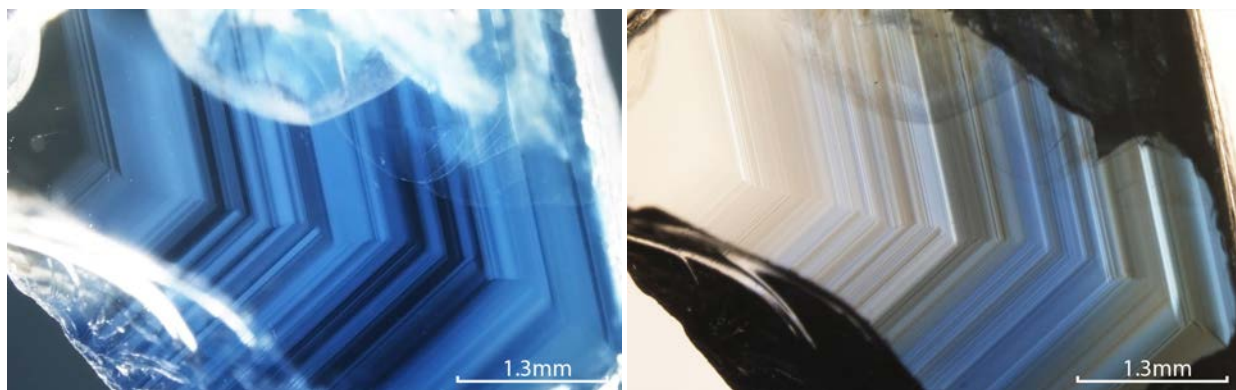


Figure 33: Two views of GIA reference sample 100309934757, magnification 25x (field of view 5.2 mm), on the left via fiber-optic lighting and on the right using brightfield illumination. The bands of minute particles are associated with the hexagonal structure. On studying the bands carefully it can be seen that the milky bands of particles on the left are associated with brownish areas on the right, while the light to deep blue areas are particle free. Photo: Jonathan Moyal © GIA.

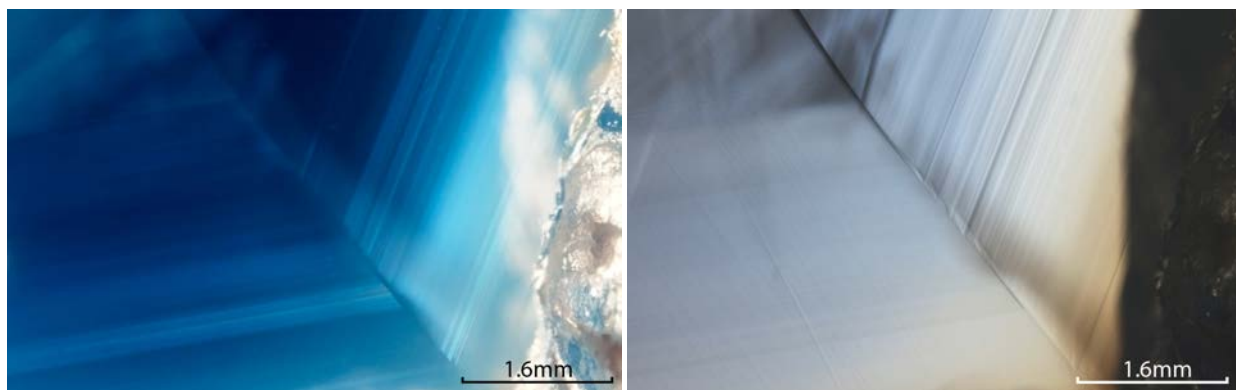


Figure 34: Two views of GIA reference sample 100309934759, magnification 20x (field of view 6.3 mm). Left: Fiber-optic illumination reveals fine whitish bands of particles. Using brightfield lighting (right) reveals that some of these bands now appear as dark brownish clouds of particles in the central area of the growth structure. Photos: Jonathan Moyal © GIA.

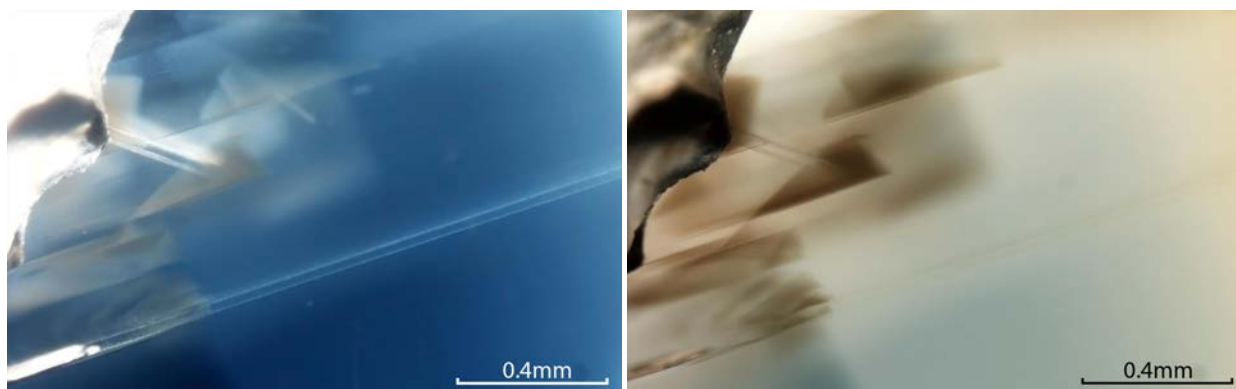


Figure 35: Two detailed views of GIA reference sample 100309934759, magnification 50x (field of view 2.5 mm), left using fiber-optic illumination, right using brightfield illumination. Some whitish and some brown particle rich areas following the stone's growth structure outside the blue colored areas are clearly visible. Photos: Jonathan Moyal © GIA.

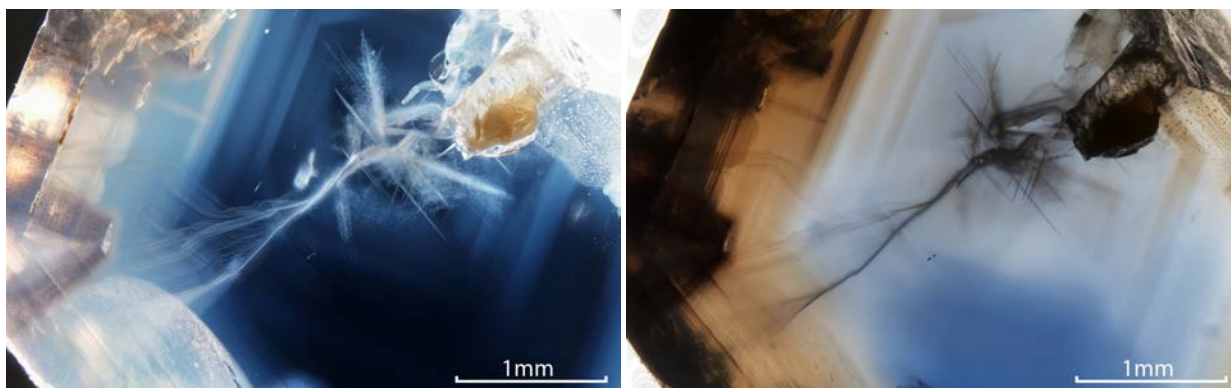


Figure 36: Two views of GIA reference sample 100309934762, magnification: 30x (field of view 4.1 mm), left using fiber-optic illumination, and right using diffused brightfield illumination. Bands of minute particles clearly follow the hexagonal structure. A group of coarse particles forming flake or lathe like structures are also visible in the center of the stone. On the outer area of the stone some dark brown particles are also present in comet tail like structures associated with crystals. Photos: Jonathan Moyal © GIA.

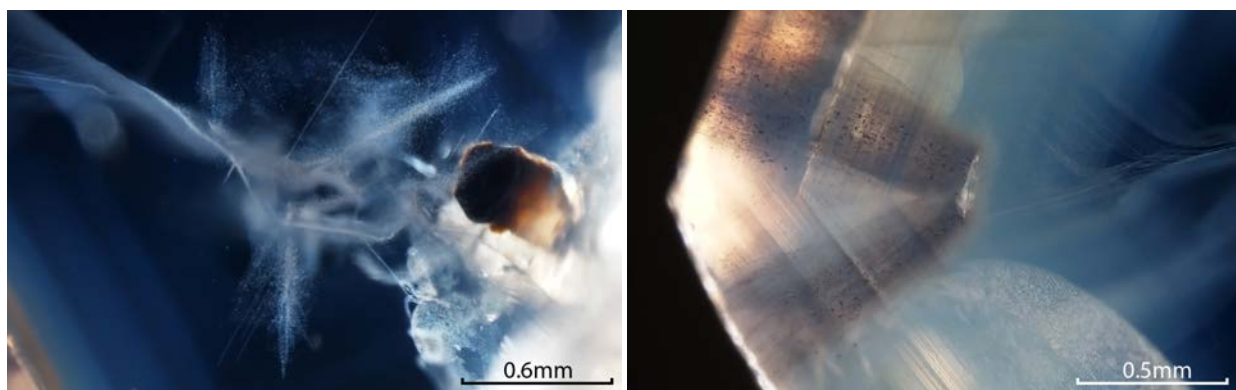


Figure 37: Two detailed views of some specific inclusions in GIA reference sample 100309934762, via darkfield illumination. On the left using 50x magnification (field of view 2.5mm), details of the flake or lathe like structures seen at lower magnification in figure 35 are shown. On the right using 60x magnification (field of view 2 mm) a transparent crystal inclusion, identified as feldspar using Raman (referenced via the RRuff database), is visible in association with a comet tail like structure composed of dark brownish particles. Photos: Jonathan Moyal © GIA.

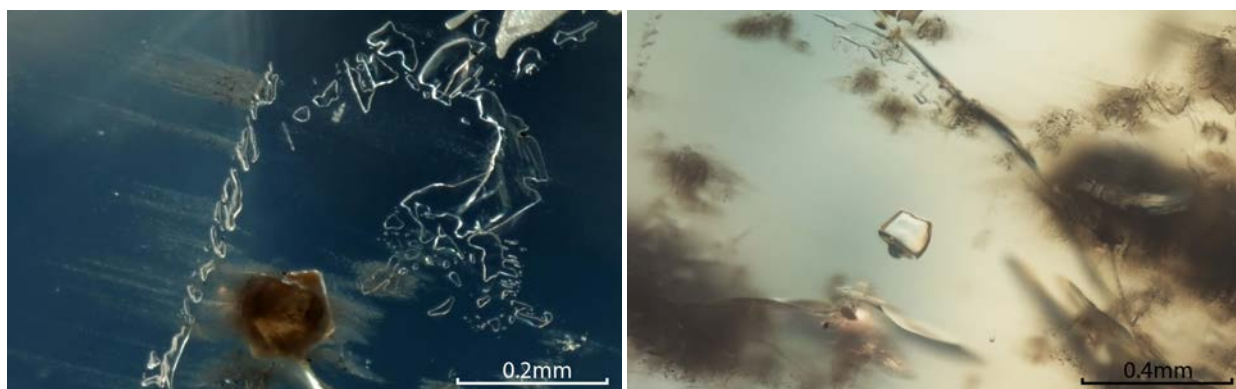


Figure 38: Two detailed views of feldspar inclusions, identified in both cases with Raman (referenced via the RRuff database), in GIA reference sample 100309934762. Left, using darkfield illumination and 150x magnification (field of view 0.85 mm) groups of colorless feldspar crystals, some associated with short comet tail structures, are clearly apparent. On the right, using diffuse brightfield illumination, magnification 80x (field of view 1.5 mm), a single rounded, colorless feldspar crystal associated with brownish clouds of particles, stands out markedly. Photos: Jonathan Moyal © GIA.

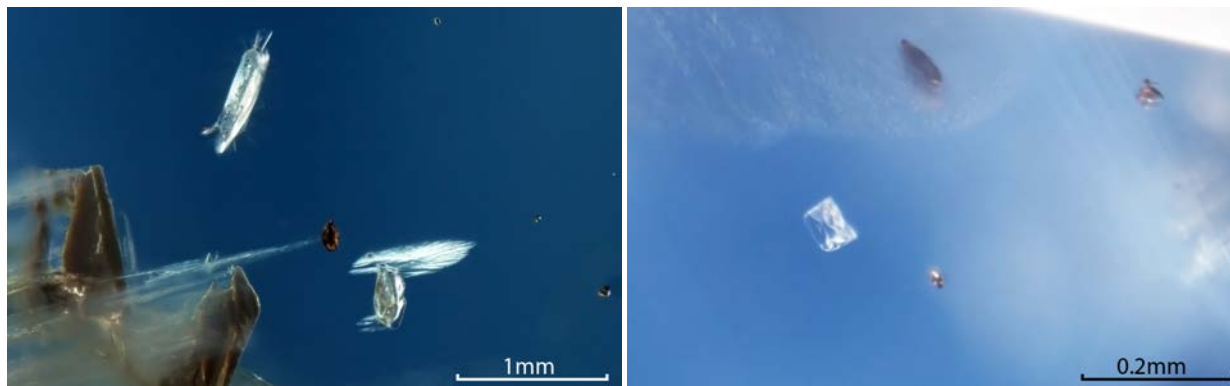


Figure 39: Two detailed views of GIA reference sample 100309934763 using darkfield illumination. On the left with magnification 70x (field of view 1.7 mm) two metamict zircons with associated tension fissures, and an orange-brown rounded uranopyrochlore crystal that is itself associated with a comet tail like structure command attention. On the right using magnification 180x (field of view 0.7 mm) 3 small uranopyrochlore like rounded crystals and a colorless feldspar crystal vie for recognition. All the minerals were identified with Raman (referenced via the RRuff database). Photos: Jonathan Muyal © GIA.

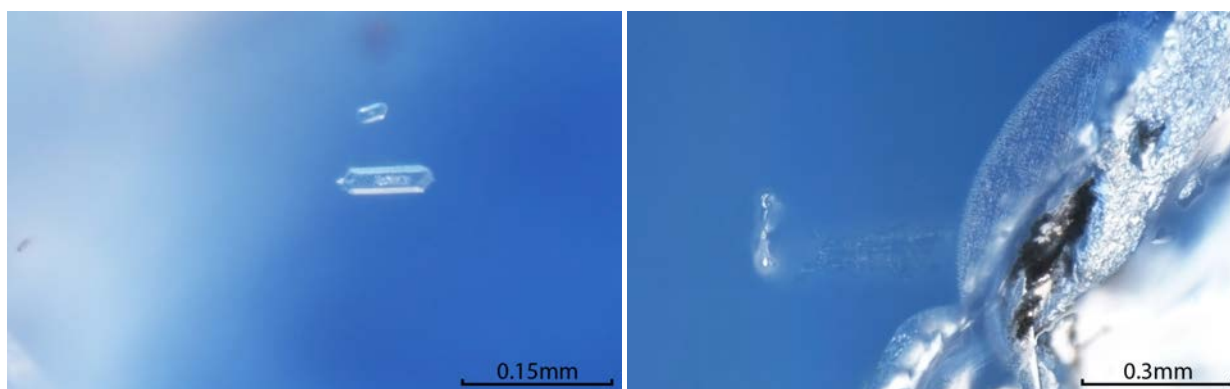


Figure 40: Two more detailed views of GIA reference sample 100309934763 using darkfield illumination. Left, with magnification 200x (field of view 0.65 mm) two small, elongated euhedral zircon crystals, identified by Raman (referenced via the RRuff database) “float” together. Right, with magnification 112x (field of view 1.1 mm) a small group of colorless crystals (unidentified) associated with a comet tail like structure look as though they are being pulled towards the group of partially healed fissures. Photos: Jonathan Muyal © GIA.

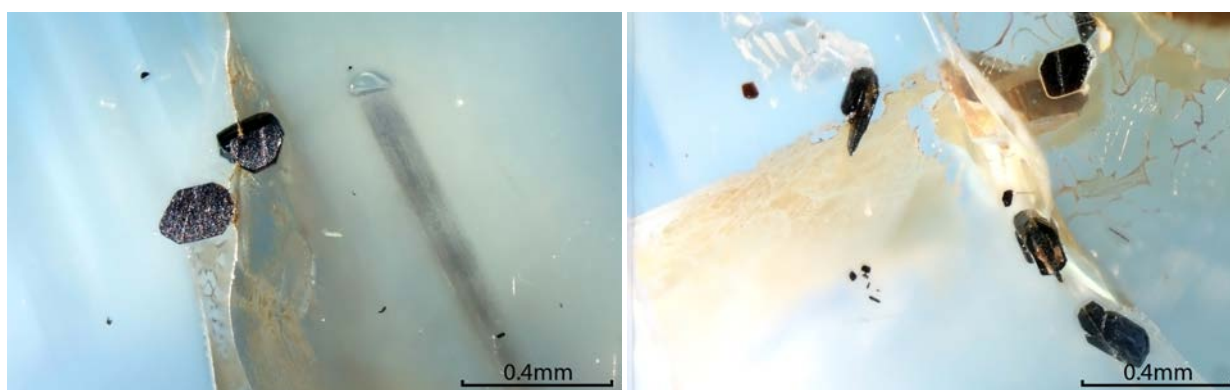


Figure 41: Two detailed views of GIA reference sample 100309934755 using darkfield + fiber-optic illumination at magnification 70x (field of view 1.7 mm). Left: two dark brownish opaque euhedral ferrotantalite crystals reach the surface. Nearby a transparent rounded feldspar crystal is associated with a comet tail structure composed of dark brown particles. Right: Several dark brownish, opaque, euhedral ferrotantalite crystals associated with healed fissures vie for position. In all cases the mineral inclusions were identified by Raman (referenced via the RRuff database). Photos: Jonathan Muyal © GIA.

Part 3: Discussions about sapphires from Mambilla and the challenges presented by the determination of their geographical origin:

Trace element comparison between sapphires from the new and old diggings on the Mambilla Plateau:

Four stones, reportedly from the new deposit on the Mambilla Plateau, and seven stones, reportedly from the old diggings, were studied using LA-ICP-MS. We studied the data collected and compared it with data collected from sapphires from other basalt related deposits in Thailand, Cambodia and Australia.

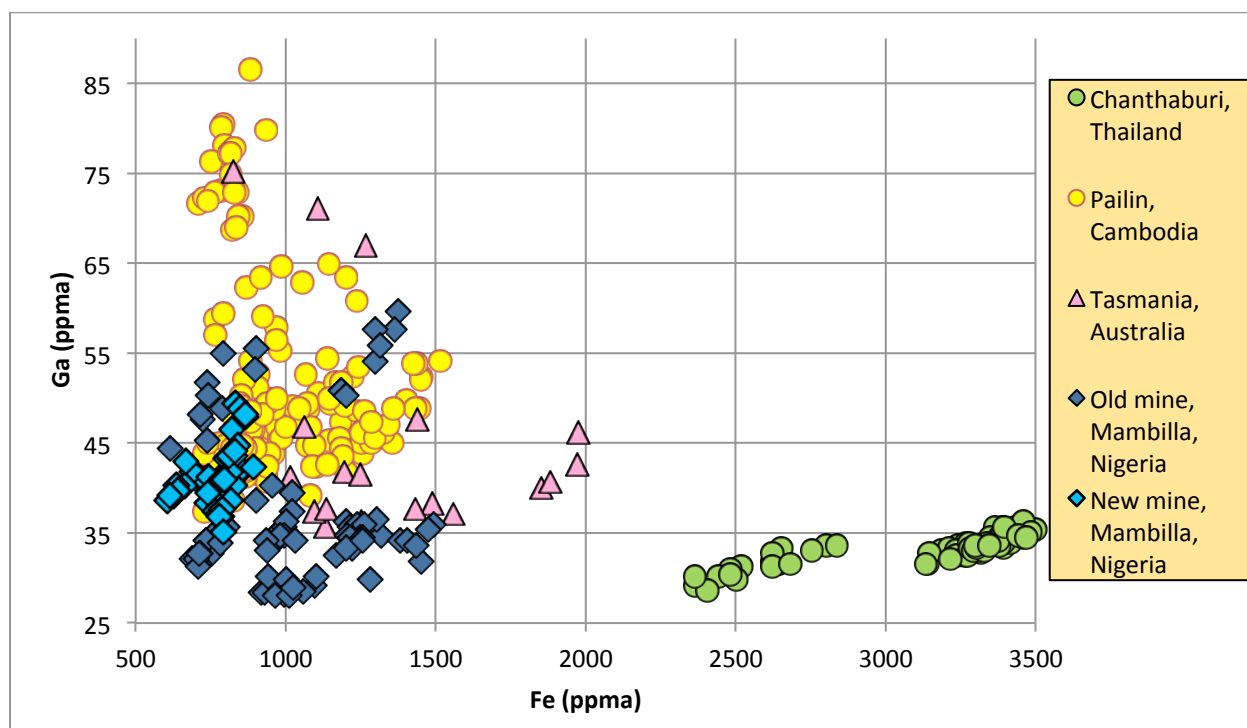


Figure 42: Fe v. Ga population field comparing sapphires from the new deposit near Mambilla Plateau with stones from other deposits in Nigeria, Thailand/Cambodia and Australia. It is readily apparent that the stones from the new deposit near Mambilla possess lower iron and gallium content compared to most other sapphires. Based on this population field, chemistry is an efficient tool to separate these sapphires from their cousins from Tasmania or Chanthaburi. Nevertheless there is a significant overlap with a part of the population from Pailin. © GIA.

Outside particle rich areas, these eleven stones were to all intents and purposes “chemically clean” with only a significant iron content and traces of gallium and titanium. This is quite remarkable as basalt related sapphires from other known deposits like Thailand, Cambodia, and Australia usually have some minor magnesium and vanadium content. With such a limited number of trace elements to depend on, the population field that makes most sense to use is iron versus gallium (see Figure 42).

The stones from the new deposit were found to have a lower iron content when compared to their cousins from the old diggings on the Mambilla Plateau. This is not surprising when the color of the stones from these 2 deposits is taken into account. The stones from the old diggings are darker and present a stronger blue/green dichroism overall and such sapphires usually have higher iron content than lighter colored stones.

Furthermore this population field indicates that, while it may be easy to separate the basalt related sapphires from the new deposit near Mambilla from other basalt related sapphires such as those mined near Chanthaburi in Thailand, based on the latter's higher iron content, relatively low gallium content and the fact that sapphires from Chanthaburi also have some significant levels of magnesium and vanadium, matters are more challenging when it comes to sapphires from Tasmania in Australia.

It was interesting to see that in all the stones from the Mambilla Plateau, whether from the old or the new diggings, the milky Geuda type samples were consistently found to have:

- 1) High titanium content, as expected, when compared to the clear samples.
- 2) Traces of magnesium (Mg), vanadium (V), beryllium (Be), zirconium (Zr), niobium (Nb), hafnium (Hf), tantalum (Ta), and thorium (Th) that were not found in the clear stones. While the presence of niobium, tantalum, zirconium is not a surprise for basalt related sapphires since they have been noted by geologists in many samples from other deposits in Cambodia, Laos, Australia, Madagascar, Vietnam and China, (Jingfeng Guo, O'Reilly et al. 1996; Sutherland 1998; Khin Zaw, Sutherland et al. 2006; Graham 2008), the presence of beryllium is more notable as it was not described by these authors or seen as negligible.

Before the early 2000's, the presence of beryllium in corundum was not studied in any detail. It became a subject of interest after the arrival of a very efficient treatment involving beryllium diffusion (Scarratt 2002; Emmett, Scarratt *et al.* 2003).

Natural sapphires with some beryllium content have then been discovered regularly since 2004 (Wathanakul 2004; Mc Gee 2005; Pardieu 2007; Pardieu 2008; Pardieu 2011; Pardieu 2013). The presence of accompanying trace elements such as niobium (Nb), hafnium (Hf), tantalum (Ta), thorium (Th) or tungsten (W) in sapphires with a natural beryllium content enables gemologists to separate sapphires treated with beryllium from those naturally containing the element. But the interest in these additional trace elements does not end there, since some significant differences in the chemical composition of the trace elements associated with these

sapphires from different deposits exist. Blue sapphires from the Sar-e-Sang deposit in Afghanistan are rich in tungsten (Pardieu 2011) while no significant amounts of tungsten were detected in the sapphires from the Mambilla Plateau in Nigeria. On studying the trace element content in several particle rich samples from the new mining site on the Mambilla Plateau (Figure 43), a very good correlation between beryllium, tantalum, niobium and hafnium was found. Similar trace element correlation was observed in sapphires containing natural beryllium from other deposits (Pardieu 2007; Shen 2007; Pardieu 2008). However, the zirconium and titanium content don't look as well correlated as the beryllium, niobium, tantalum and hafnium content. This suggests the existence of several types of titanium rich particles, some associated with Be, Nb, Ta, Hf in TiO_2 with PbO_2 structure, as observed by Shen (Shen 2012), while other Ti rich particles could not be associated with these trace elements.

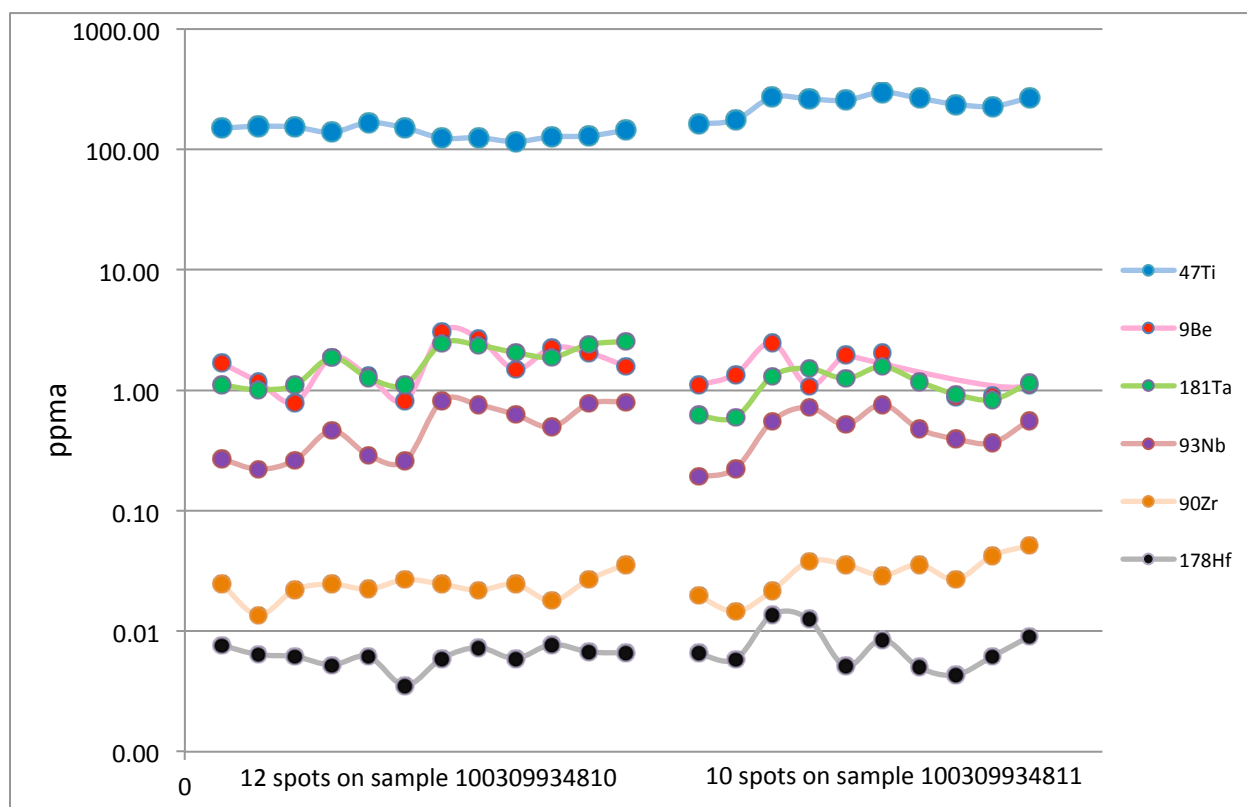


Figure 43: Graph showing the correlation between several trace elements in particle rich areas of 2 milky stones from the new deposit on the Mambilla Plateau: GIA reference sample 100309934810 (12 spots) and 100309934811 (10 spots). Overall, a very good correlation between the Nb, Ta, Be and Hf content is apparent while it is less obvious for Ti and Zr.

Further systematic and comparative studies on the chemistry of natural sapphire samples containing clouds of minute particles from different deposits would be useful to complete our understanding of these gems and see if it would assist in the origin determination of blue sapphires.

Discussion on the challenges presented by the determination of the origin of basalt related sapphires such as those from Mambilla Plateau, Nigeria.

Determining the geographical origin of basalt related sapphires is known to be challenging despite the fact that such stones occur in many different localities on different continents and share many similarities. This is not entirely surprising since they form under similar geological events (Saul 2014), but it is still quite fascinating to the authors. This preliminary study confirms the difficulty, nevertheless some techniques to aid in the identification of certain sources appear to be promising.

Trace element chemistry:

As observed in the previous chapter, chemistry is a powerful tool but it also has its limitations. This is particularly true in particle free areas in stones such as those from the Mambilla Plateau as there are very few trace elements available to study using LA-ICP-MS other than Fe, Ga and small amounts of Ti. But as with similar particle free areas in sapphires from other locations, other trace elements such as Mg and V may be detected and this is already an interesting result. The limited number of trace elements present in basalt related sapphires from different deposits is an issue, as it is easier to find differences between stones from several deposits when there are multiple trace elements to compare. This is why areas rich in particle (dust) sized inclusions are particularly interesting to gemologists since the particles are often found to be rich in trace elements such as Be, Ti, V, Zr, Nb, Sn, Hf, Ta, Th and sometimes W. So instead of studying 3 to 6 trace elements in pure corundum, the potential 10 additional trace elements present in particle rich areas may prove invaluable when attempting to determine the source of basaltic sapphires. Since many of these elements looks to be correlated the chances of finding some diagnostic chemical differences between different deposits increases since the nature of the particles may differ between one deposit and another.

When comparing the chemical data from particle rich areas in sapphires from the Mambilla Plateau with the data collected from particle rich areas in sapphires from other deposits, interesting differences were indeed noted. Such observations, if confirmed by a more detailed comparative study of particle rich areas in sapphires from different origins, could provide a useful tool for gemologists who work on the origin determination of sapphires to use over the coming years.

UV-Vis-NIR Spectroscopy:

The use of UV-Vis-NIR spectroscopy is a convenient and very useful way to separate basalt related sapphires as they show a broad absorption band around 850 to 900 nm,

(the Mambilla Plateau samples are no exception) from metamorphic sapphires which (usually) do not show this feature. Besides this very useful data, the use of the instrument is limited in regards to origin determination as the data provided is only scientifically valid when collected from stones with no or few particles. Indeed, particles will scatter the light, and the validity of the data on UV-Vis spectra dominated by scattering issues becomes questionable. The apparent absorption edge is significantly higher in particle rich samples than in the clear samples. The data only confirms that the stone is full of particles, a fact that is readily apparent with a loupe or microscope, and therefore such a feature might not be so relevant when determining the geographic origin of sapphires.

Infrared Absorption (FTIR) Spectroscopy:

The presence of distinct features at 3309, 3232 and 3186 cm^{-1} is a common observation in unheated alkali basalt related sapphires from numerous sources (Smith 1995; Beran 2006). Such features were observed when analyzing sapphires from the Mambilla Plateau area through both clear and milky areas. In most cases the 3309 cm^{-1} feature was found to be dominant, but in 2 cases (one from the old digging and one from the new deposit) the feature at 3232 cm^{-1} was found to be higher than the one at 3309 cm^{-1} .

Microscopic examination and Raman spectroscopy:

As seen in the inclusion study, the inclusion scene in these stones is very similar to that observed in basalt related sapphires of the 'milky type' from at least two other deposits, e.g., Pailin in Cambodia (Gübelin and Koivula 1986), and Vietnam (Smith 1995). Overall the similarity of the inclusion scene in basalt related sapphires from deposits located on two different continents is noteworthy and already limits the usefulness of microscopy for the determination of the geographic origin of basalt related sapphires. Nevertheless careful examination of the nature and appearance of the inclusions (if present) show some small differences. A more detailed study on a larger number of reference samples will be necessary to characterize the deposit and confirm the usefulness of the study of mineral inclusions as a reliable tool to identify the geographic origin of basalt related sapphires.

On the whole it is useful to note that while particle rich areas can provide a lot of information to the gemologist when studying trace element chemistry, the same particles present limitations to gemologists when UV-Vis-NIR spectroscopy is applied owing to light scattering issues. A parallel can be drawn with the use of a microscope and its usefulness in regards to origin determination when a sapphire hosts tell-tale inclusions, and the lack of help provided by it when the stone is clean. These observations prove that there is no "magic wand" type of instrument in gemology.

Origin determination relies on data collection from numerous classical and advanced gemological techniques in order to obtain the “full picture”, and a good understanding of a given gem is also a prerequisite. The comparison of the collected data from an unknown sample with other reliable data carefully collected from trusted reference stones is now necessary for gemologists to be able to provide an expert opinion on the geographical origin of any gem based on facts rather than on experience and “feelings”.

Summary:

The arrival of fine gemstones from a new deposit is always very exciting. This has been the case with the new Nigerian material, which shows an iron content significantly lower than that usually expected from other basalt related deposits around the world. This element in itself is interesting for gemologists, but equally so for traders as the lower iron content means that stones are less likely to develop a greenish and grayish appearance after fashioning which usually correlates to a higher market value. This is particularly true for large, potentially valuable blue stones. This point alone explains why such material is generating a lot of interest within the trade.

Sadly the opportunity to visit the deposit and collect samples on site has not materialized yet. Nevertheless this preliminary study on several samples collected from secondary sources is a good starting point and has produced some useful results.

The new deposit near Guroji on the Mambilla Plateau seems to be producing some transparent to milky blue sapphires that appear to be colored by 2 different process. While transparent blue sapphires owe their blue color to IVCT (Intervalence Charge Transfer) the milky blue stones owe their milky blue appearance to a Tyndall effect associated with light scattering from particles. The latter particle rich type of sapphire is usually heated before reaching the market as faceted material.

Overall, microscopic, spectroscopic and chemistry results are very similar to clean or milky sapphires from other basalt related sapphire deposits such as Pailin in Cambodia, South Vietnam and some Australian deposits. The geographic separation of these sapphires will probably prove challenging as is commonly the case when considering the source of basalt related sapphires. Nevertheless careful examination of the inclusions, and the study of particle rich areas in these Nigerian stones using LA-ICP-MS, seem to be the two most promising tools available to gemologists wishing to identify the origin of such sapphires.

Special Thanks:

To Jean Claude Michelou, for the numerous discussions we had about Nigeria, to Martial Curti who helped to put VP in contact with some key West African traders in Bangkok and in particular with Mr. Saoud Kouyate and Mrs. Hauwa Ibrahim from “Genduste Investment” in Nigeria who shared a lot of useful information about the interesting new deposit. To Mr. Brume Jeroh from Jos, Nigeria. To Sheryl Elen from the GIA Library in Carlsbad for sending us all the publications we requested, and last but not least to Mr. Kenneth Scarratt, Director of GIA Bangkok, Dr. John Emmett, Tim Thomas and Sudarat Saeseaw from for their support and useful advice.

Annex A: GIA Field Gemology cataloguing System

This system was developed at the GIA Field Gemology department to precisely document the way in which a given research sample was collected.

A conditions: Mined/collected by the field-gemologist.

A1: Collected in-situ from a primary deposit by the field-gemologist.

A2: Collected on the floor/mine waste at the mine (primary/secondary) by the field-gemologist.

A3: Collected after digging in a secondary deposit by the field-gemologist.

B conditions: Field-gemologist witnessed the mining.

B1: Collected on site in jig/sieve from the mine in a secondary type deposit by field-gemologist.

B2: Collected on site in miner's bottle from mine in a secondary type deposit by mines or by field-gemologist.

C conditions: Field-gemologist collected from miners at the mine but without witnessing the mining process.

C1: Collected at mine from mine owner (not mined that day in front of field-gemologist's eyes).

C2: Collected at mine from miner on site (not mined that day in front of field-gemologist's eyes).

D conditions: Field-gemologist collected the stones from miner but not at the mines.

D1: Collected from mine owner (at HQ near the mines).

D2: Collected from miner (near the mines).

D3: Collected from person claiming to be a miner (mine not visited).

D4: Collected from miner in regional/international market.

E conditions: Field-gemologist collected the stones from a secondary source close to the mines.

E1: Bought from trusted secondary source (gemologist/dealer/broker) at local market (close to source).

E2: Bought from trusted secondary source (gemologist/dealer/broker) at regional market (close to source).

E3: Bought from unknown secondary source at local gem market (close to source).

E4: Bought from unknown secondary source at regional gem market (close to source).

F conditions: Field-gemologist collected the stones from a secondary source in international market.

F1: Bought from unknown dealer at international gem market (gem show...).

F2: Bought from trusted source (gemologist/collector/dealer...) in an international market.

F3: Bought from a lab client after the stone was submitted to the GIA Laboratory.

Z conditions: No information about how the stone was collected.

Z1: Lost information.

Z2: No information.

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