Natural and synthetic vanadium-bearing chrysoberyl

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Abstract: Mineralogical and gemmological properties of natural and synthetic V-bearing chrysoberyls are described. The natural samples originate from four sources (Tunduru in Tanzania, Ilakaka in Madagascar, Sri Lanka and Mogok in Myanmar) and the synthetic material was produced by Kyocera Corporation in Japan. The natural crystals show tabular habit with one of the pinacoids **a** {100} or **b** {010} as dominant crystal forms. Their morphology is consistent with internal growth structures determined in the immersion microscope. A few samples contain mineral inclusions of apatite, feldspar, or calcite. Dominant colour-causing trace elements are either V (Tunduru and Ilakaka) or a combination of V and Cr (Sri Lanka and Myanmar). Iron is present in samples from Tunduru, Ilakaka and Sri Lanka, but not from Mogok. Other trace elements such as Ga and Sn were detected in most of the natural samples and were absent from the synthetics.

UV-Vis spectra show superimposed V^{3+} and Cr^{3+} absorptions, as well as minor Fe^{3+} bands. According to the specific V:Cr ratio of the samples, the major ν_1 absorption band in the visible range is shifted from the red-orange (~607 nm) for Cr-free or almost Cr-free non-phenomenal chrysoberyls to the orange range (~589 nm) for a sample with the greatest Cr contents (0.24 wt.% Cr_2O_3). In comparison, colour-change Cr-dominant alexandrites show this major absorption band in the greenish yellow range (about 576–573 nm).

Diagnostic features of V-bearing natural chrysoberyl are compared with their synthetic Kyoc–era counterparts and with samples produced in Russia by flux growth and the horizontally oriented crystallization (HOC) technique. A distinction of natural from synthetic samples is possible by evaluating a combination of chemical, spectroscopic and microscopic features.

Keywords: chromium, colorimetry, crystal habit, inclusions, Madagascar, microprobe analyses, Myanmar, pleochroism, Sri Lanka, Tanzania, UV-Vis spectra, vanadium, X-ray fluorescence analysis

In the mid-1990s, V-bearing synthetic chrysoberyl was grown by Kyocera Corporation as a synthetic counterpart to material discovered at Tunduru, Tanzania. Shown here are a 1.53 ct V-bearing chrysoberyl from Tunduru (top) and a 1.06 ct synthetic chrysoberyl grown by Kyocera (bottom, 7.0×5.0 mm). Photo by K. Schmetzer.

Figure 1: (a) Vanadium-bearing chrysoberyl was first discovered at Tunduru, Tanzania, which is the source of this 1.53 ct oval cut (7.8×6.2 mm). (b) Its synthetic counterpart was grown by Kyocera Corporation (here, 1.12 ct or 7.6×5.7 mm). (c) The Ilakaka area in Madagascar is also a source of V-bearing chrysoberyl; this sample weighs 2.09 ct and measures 9.1×7.2 mm. Composite photo by M. S. Krzemnicki (not to scale).

Introduction

In the mid-1990s, some new bright green chrysoberyls (e.g., *Figure 1a*) found their way to gemmological laboratories worldwide (Johnson and Koivula, 1996; Bank *et al.,* 1997; McClure, 1998; also see Hänni, 2010, who also gives a summary of the different varieties of chrysoberyl). The first faceted stones were extremely clean and thus a synthetic origin was considered possible. Mainly trace-element analysis was applied to establish criteria to distinguish these samples from synthetic counterparts known at that time. The natural samples reportedly originated from the large alluvial mining area of Tunduru in southern Tanzania. Faceted samples of this 'mint' green chrysoberyl variety are considered rare and valuable gems (Mayerson, 2003). Their coloration was found to be due to vanadium and they contained very little or no chromium. Furthermore, no distinct colour change was observed between daylight and incandescent light, as it is commonly seen for Cr-bearing chrysoberyl (alexandrite).

Most alexandrites from various natural sources with a clear colour change (i.e., from green or bluish green in daylight to a colour ranging from violet-purple and reddish purple to purplish red in incandescent light) show distinctly higher Cr than V contents (Schmetzer and Malsy, 2011). Chrysoberyl with almost equal amounts of V and Cr (e.g., from Orissa or Andhra Pradesh, India) also appears green in daylight, but changes in incandescent light only to a pale greyish green (Schmetzer and Bosshart, 2010).

The first synthetic alexandrite, grown by Creative Crystals and marketed since the early 1970s, contained Cr and Fe as the only significant colour-causing trace elements (Cline and Patterson, 1975; Schmetzer *et al.,* 2012). To improve the colour and grow samples that were "comparable to natural alexandrite from the Ural mountain region", distinct amounts of V were added, in addition to various quantities of Cr (Machida and Yoshibara, 1980, 1981). Distinct V contents were also found in some flux-grown synthetic alexandrite trillings produced in Novosibirsk, USSR, and some flux-grown single crystals with V>Cr also were reported (Schmetzer *et al.,* 1996). Furthermore, distinct amounts of V have been recorded in some samples of Russian synthetic alexandrite grown from the melt by the Czochralski technique, as well as by the HOC method (horizontally oriented crystallization, a horizontal floating zone technique; see Schmetzer and Bosshart, 2010; Malsy and Armbruster, 2012; Schmetzer *et al.,* 2013a).

In the patent documents by Machida and Yoshibara (1980, 1981) mentioned previously, which were assigned to Kyoto Ceramic Corporation (Kyocera) from Kyoto, Japan, the growth of almost Cr-free, V-bearing synthetic chrysoberyl was also reported. This material is comparable to the non-phenomenal V-bearing green synthetic chrysoberyl that was grown at the same time by the Czochralski method in Novosibirsk (Bukin *et al.,* 1980). Another type of non-phenomenal green synthetic chrysoberyl was grown some years later by Tairus in Novosibirsk using the HOC method (Koivula *et al.,* 1994).

Subsequent to the discovery of V-bearing gem chrysoberyl in Tunduru in the mid-1990s (see above), another

Figure 2: Rough and cut V-bearing synthetic chrysoberyl grown by Kyocera Corporation. The top-right piece weighs 10.5 g and measures 15.5×15.5×14.4 mm; the faceted samples weigh 0.49–1.33 ct. Photo by K. Schmetzer.

patent application was filed by Kyocera in Japan that described the production of the V-bearing counterpart of this gem material (Nishigaki and Mochizuki, 1998). This material was probably of the same type as the samples described briefly by Krzemnicki and Kiefert (1999; see also *Figure 1b*), and also those donated to the Bavarian State Collection for Mineralogy in Munich, Germany *(Figure 2)*.

To the knowledge of the present authors, only one polarized spectrum of a Czochralski-grown V-bearing chrysoberyl is published and it appears in an article that is difficult to access (Bukin *et al.,* 1980). Furthermore, pleochroism and colour were not mentioned in that paper, and descriptions of natural V-bearing chrysoberyl are often vague. This is due to the difficulty in orienting faceted chrysoberyl properly for spectroscopy in relation to the crystallographic axes. There is also a lack of published data on V-bearing non-phenomenal chrysoberyl from sources other than Tunduru, such as from Ilakaka, Madagascar *(Figure 1c)*. The present paper investigates the coloration and other gemmological and mineralogical properties of non-phenomenal natural and synthetic V-bearing chrysoberyl, and aims to fill some of the gaps mentioned above.

Table I. Trace element contents, colour, pleochroism, and spectroscopic properties of V-bearing synthetic chrysoberyl grown by Kyocera Corp. in Japan.

Colour and pleochroism[a]

Orientation	X||a	Y||b	Z||c
Daylight	Yellowish green	Green	Bluish green
Incandescent	Yellowish green	Green	Bluish green

Spectroscopic properties

Property	Polarization, position (nm)			Assignment in all three directions of polarization
Orientation	X||a	Y||b	Z||c	
Maxima	607	618	608	V^{3+} first absorption band (ν_1)
	408	411	418	V^{3+} first absorption band (ν_2)
Minima	510	518	498	

Chemical properties (wt.%)

Sample	Ky1	Ky2	Ky3	Ky4	Ky5	Ky6	Ky7
No. analyses[b]	10	10	10	10	1	1	1
TiO_2	<0.01	<0.01	<0.01	<0.01	0.002	0.001	0.002
V_2O_3	0.11	0.12	0.13	0.12	0.115	0.119	0.081
Cr_2O_3	<0.01	<0.01	<0.01	<0.01	nd[c]	0.004	0.006
MnO	<0.01	<0.01	<0.01	<0.01	nd	nd	nd
Fe_2O_3	<0.01	<0.01	<0.01	<0.01	0.002	0.001	0.002

[a] Based on a morphological cell with a = 4.42, b = 9.39 and c = 5.47 Å.
[b] One analysis = EDXRF, 10 analyses (averaged) = electron microprobe.
[c] nd = not detected.

Samples

For the synthetics, we examined three pieces of rough and 12 faceted samples from Kyocera. All the rough and nine of the cut stones had been donated by Kyocera Germany to the Bavarian State Collection for Mineralogy in Munich. The other faceted samples were donated by Kyocera Corp. to the Swiss Gemmological Institute SSEF, Basel, Switzerland.

The natural samples consisted of 27 rough and faceted chrysoberyls with V>Cr. They originated from the large placers at Tunduru (14 samples) and Ilakaka (6), as well as from secondary deposits in Sri Lanka (5) and unspecified sources in Mogok (2). Many of the samples were obtained from the H.A. Hänni gemstone collection, which is housed as a reference collection at SSEF. Four rough pieces from Tunduru were obtained in Tanzania by S. Pfenninger for her diploma thesis (2000). Several samples were also obtained from museums or private collections and from the trade. The two chrysoberyls from Mogok were loaned from public or private collections in England where they had been housed since the 1970s (for further details, see Schmetzer *et al.,* 2013b).

In addition to these chrysoberyls from known sources, we also examined five faceted samples from private collections and from the trade with unspecified origins. The data from these samples, which revealed V>Cr by microprobe analysis, are not specifically included in the present study, but notably their chemical and spectroscopic properties all fell within the ranges determined for the larger group of 27 chrysoberyls from known localities. Furthermore, data from 12 light green samples that were found to contain Cr>V (e.g., from Ilakaka and Sri Lanka) are not presented in this study, since such material has already been described elsewhere (see, e.g., Schmetzer *et al.,* 2002).

Instrumentation and methods

Gemmological and microscopic properties were determined for all the samples using standard instrumentation. The determination of growth structures and crystal morphology was detailed by Schmetzer (2011). Six samples that contained inclusions of measurable size were studied with a Renishaw InVia Raman microspectrometer, using an argon laser (514 nm) in confocal mode coupled with an Olympus microscope.

Quantitative chemical data for all samples were obtained by electron microprobe (JEOL JXA-8600 and Cameca Camebax SX 50 instruments) or energy-dispersive X-ray fluorescence (EDXRF) spectroscopy using Spectrace 5000 Tracor X-ray and ThermoScientific Quant'X instruments. In addition to the trace elements given in *Tables I* and *II*, aluminium contents were also measured as normal and used as a control value for the quality of data (since BeO cannot be measured reliably by electron microprobe and EDXRF analyses). Furthermore for all samples semi-quantitative data for Ga and Sn were obtained by one of the analytical techniques mentioned above.

Absorption spectra were recorded with a CCD-type Czerny-Turner spectrometer in combination with an integrating sphere (for further details, see Schmetzer *et al.,* 2013a). Non-polarized spectra were obtained for 12 synthetic and 25 natural samples from known sources as well as for five chrysoberyls from unknown localities. In addition, polarized spectra were performed for about half of these samples. Colorimetric data were obtained in transmission mode for 15 samples with a Zeiss MCS 311 multichannel colour spectrometer (see Schmetzer *et al.,* 2009; Schmetzer and Bosshart, 2010).

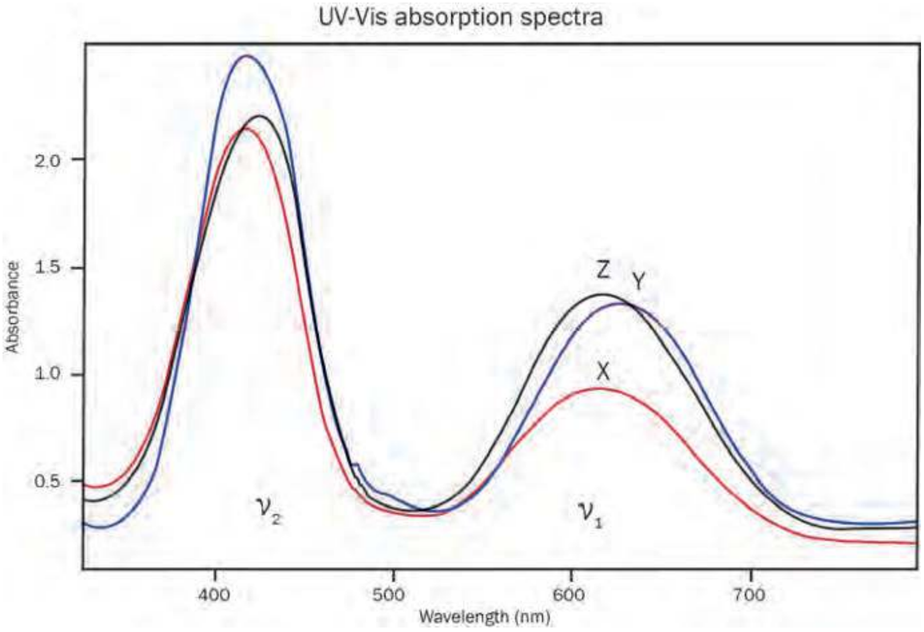


Figure 3: Polarized absorption spectrum of a V-bearing synthetic chrysoberyl grown by Kyocera Corp., with X || a, Y || b and Z || c. The dominant absorption bands are designated ν_1 and ν_2 (see Table I).

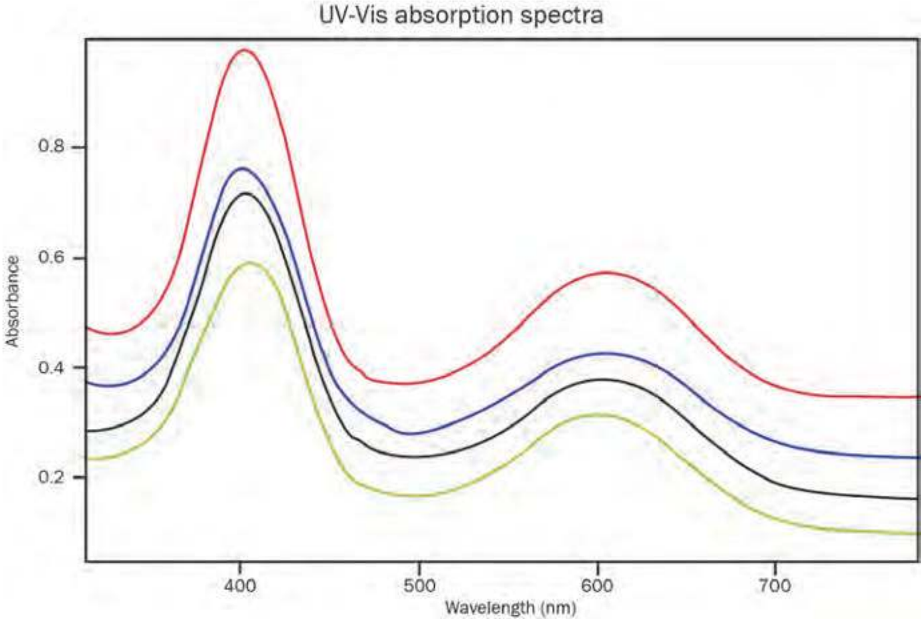


Figure 4: Absorption spectra of four faceted V-bearing synthetic chrysoberyls in random orientation. The top three spectra are displaced vertically for clarity.

Kyocera synthetic V-bearing chrysoberyl

The rough samples consisted of two somewhat irregular pseudo-hexagonal cylinders and one pseudo-hexagonal pyramid *(Figure 2)*. They had been sawn from Czochralski-grown crystals, and their upper and lower bases as well as the side faces were roughly polished to clean the surfaces and to give a better visual impression. The faceted gems were cut without specific orientation.

The overall colour of the samples was bright green in both daylight and incandescent light. Pleochroism was weak in the faceted samples but clearly observed in the rough pieces, with X = yellowish green, Y = green and Z = bluish green, independent of lighting.

The dominant colour-causing trace element in this group of samples is vanadium (0.08–0.13 wt.% V_2O_3; *Table I*). Other trace elements were, in general, below the detection limits of the electron microprobe, with small traces of Cr and Fe observed occasionally by EDXRF (see *Table I*).

Polarized absorption spectra of the rough samples showed two strong absorption bands in the red-orange range (~610 nm, designated as first strong absorption band ν_1) and in the violet area (~415 nm, designated as second strong absorption band ν_2) for X, Y and Z, with some variation in maxima positions and intensities *(Table I, Figure 3)*. These absorption bands in the visible range are assigned to V^{3+} which replaces Al^{3+} in the chrysoberyl structure. These absorption features are similar to those depicted by Bukin *et al.* (1980). Also, the non-polarized spectra from the faceted synthetics which are mostly randomly orientated *(Figure 4)* are consistent with these data.

The ν_1 absorption bands for Y and Z have approximately the same intensity, but due to the different position of their absorption maxima (618 and 608 nm, respectively), the minimum for Y at 518 nm is in the green range of the spectrum, while the minimum for Z at 498 nm is in the bluish green range. Thus, the different colours of Y and Z are understandable. The ν_1 absorption band in the X spectrum

Table II. Trace-element contents (wt.%), colour, and pleochroism of V-bearing chrysoberyl.

Source	Tunduru, Tanzania								
Colour	Pale to moderate intense green (Type 1)					Intense to very intense green (Type 2)			
Pleochroism[a]									
X	Not observed or very weak					Yellowish green			
Y						Green			
Z						Bluish green			
Sample	Tu1	Tu2	Tu3	Tu4	Tu5	Tu6	Tu7	Tu8	Tu9
No. of analyses[b]	10	16	8	10	1	1	7	20	15
TiO_2	0.01	0.08	0.01	0.01	0.001	0.007	0.15	0.11	0.01
V_2O_3	0.04	0.06	0.06	0.06	0.103	0.166	0.18	0.21	0.30
Cr_2O_3	0.01	0.01	0.01	0.01	0.004	0.038	0.03	0.03	0.04
MnO	nd[c]	nd	nd	0.01	nd	nd	nd	nd	nd
Fe_2O_3	0.17	0.13	0.10	0.24	0.317	0.227	0.08	0.09	0.20

Source	Ilakaka, Madagascar					Sri Lanka				Mogok, Myanmar
Colour	Pale to moderate intense green				Intense green	Moderate yellowish green				Intense bluish green
Pleochroism[a]										
X	Not observed or very weak				Yellowish green	Not observed or very weak		Yellowish green		Greyish violet
Y					Green			Green		Yellowish green
Z					Bluish green			Green		Intense blue-green
Sample	Il1	Il2	Il3	Il4	Il5	SL1	SL2	SL3	SL4	M1
No. of analyses[b]	8	10	10	9	10	10	10	10	10	12
TiO_2	0.01	0.05	0.02	0.01	0.01	0.05	0.01	0.11	0.10	0.08
V_2O_3	0.03	0.04	0.06	0.08	0.15	0.05	0.07	0.08	0.11	0.38
Cr_2O_3	<0.01	<0.01	<0.01	<0.01	<0.01	0.03	0.02	0.05	0.10	0.24
MnO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	nd
Fe_2O_3	0.55	0.11	0.23	0.50	0.17	0.90	0.47	0.82	0.65	<0.01

[a] Daylight, based on a morphological cell with a = 4.42, b = 9.39, c = 5.47 Å, and X||a, Y||b, Z||c.
[b] One analysis = EDXRF, 7–20 analyses (averaged) = electron microprobe.
[c] nd = not detected.

is weaker than in Y and Z, which explains the somewhat more yellowish green colour of X.

Microscopic examination of faceted samples revealed no inclusions, growth striations, or clearly visible gas bubbles. Furthermore, gas bubbles were easily observed in only one of the rough pieces *(Figure 5)*.

Natural V-bearing chrysoberyl

Morphology of the rough and internal growth structures

Due to their origin from secondary deposits, most of the rough samples from Tunduru, Ilakaka and Sri Lanka were heavily waterworn and broken. Thus, we could determine the complete morphology only for a few samples *(Figure 6)* by goniometric measurements in combination with the examination of internal growth structures. *Figure 7* shows some examples of internal growth patterns seen in a crystal from Ilakaka, a faceted stone from Tunduru, a crystal from Mogok and a twinned crystal from Sri Lanka.

The crystal forms noted in our samples are well-known and seen frequently in natural chrysoberyl: the pinacoids **a** {100}, **b** {010} and **c** {001}; the prisms **i** {011}, **k** {021}, **m** {110}, **s** {120}, **r** {130} and **x** {101}; and the dipyramids **o** {111}, **n** {121} and **w** {122}. (For further details on chrysoberyl morphology and identification of crystal faces, see Schmetzer, 2011.) Idealized crystal drawings representing all four localities, for which a complete habit determination was possible, are presented

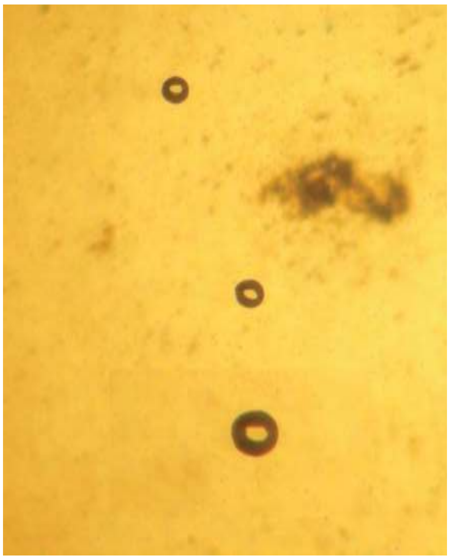


Figure 5: Gas bubbles are rarely seen in synthetic V-bearing chrysoberyl from Kyocera. Immersion, field of view 2.0×2.7 mm. Photo by K. Schmetzer.

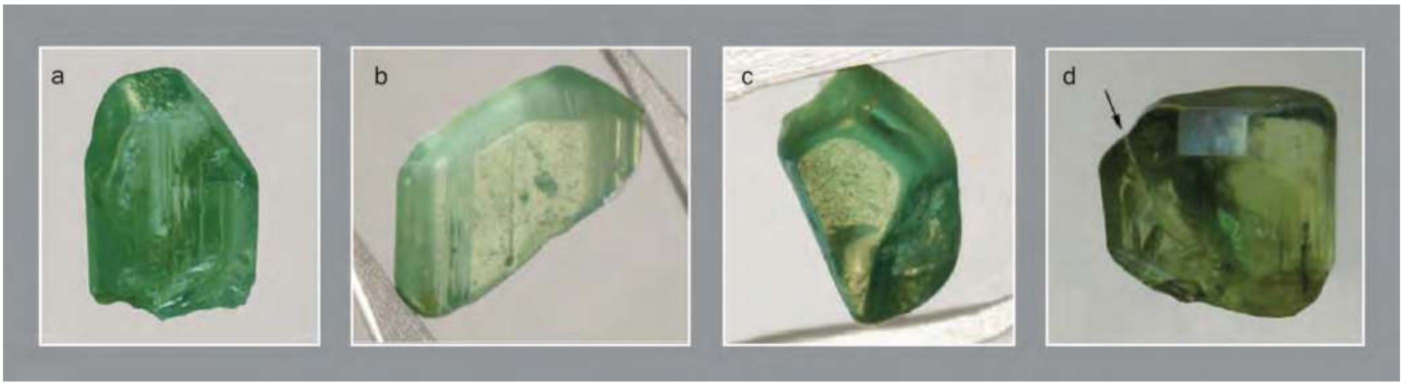


*Figure 6: Morphology of V-bearing chrysoberyl crystals. (a) Ilakaka, 12.7×8.4 mm, tabular parallel to **b** {010}; (b) Ilakaka, 7.4×6.5 mm, tabular parallel to **a** {100}; (c) Tunduru, 5.2×3.1 mm, tabular parallel to **a** {100}; (d) twinned crystal from Sri Lanka, 6.9×6.1 mm, the largest face is **a** (100), and the arrow indicates the twin boundary. Photos by K. Schmetzer.*

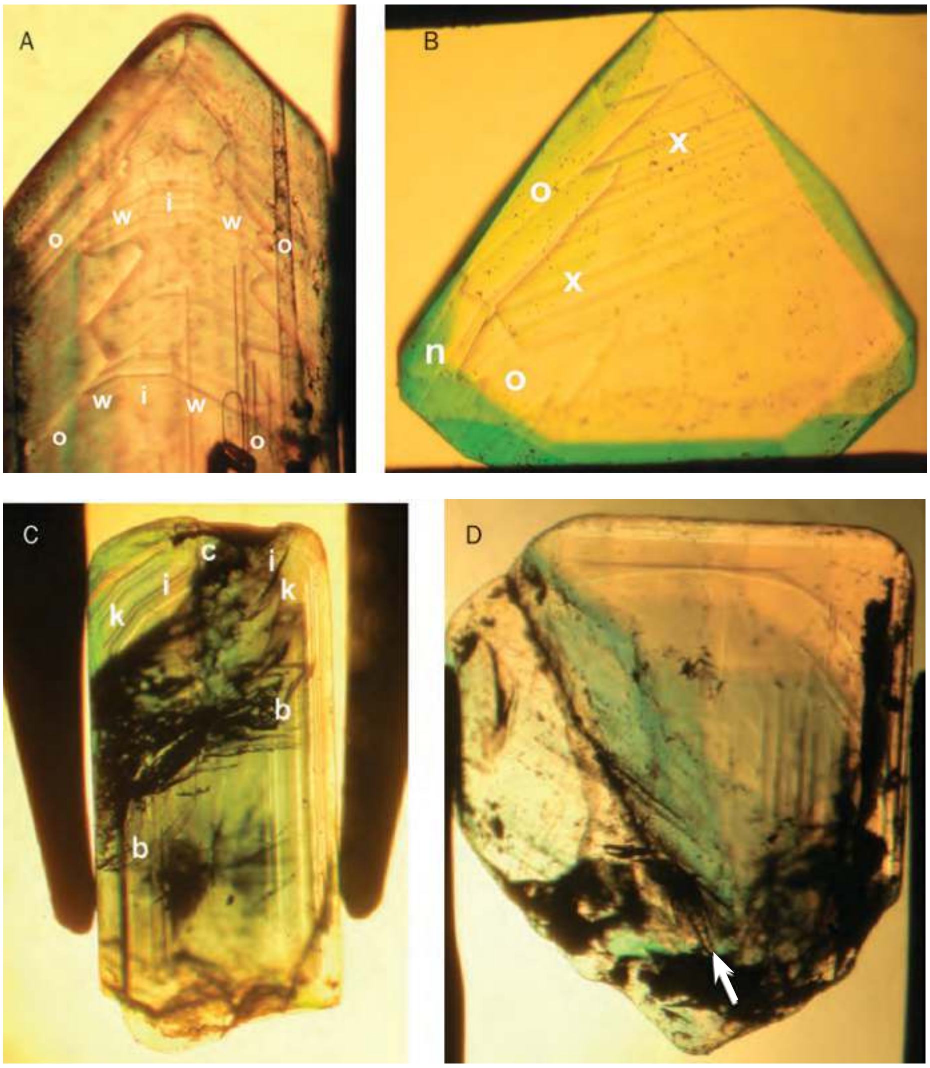


Figure 7: Growth structures in V-bearing chrysoberyl. (A) Crystal from Ilakaka (see Figure 6b*), tabular parallel to **a** {100}; view inclined to the c-axis, showing growth faces parallel to the prism **i** and to the dipyramids **w** and **o**; field of view 2.4×3.2 mm. (B) Cut stone from Tunduru, showing growth zoning parallel to the prism **x** and to the dipyramids **n** and **o**; field of view 5.1×4.7 mm. (C) Crystal from Mogok; view parallel to the a-axis, showing growth faces associated with colour zoning parallel to the pinacoids **b** and **c** and to the prism faces **i** and **k**; field of view 5.3×7.1 mm. (D) Crystal from Sri Lanka (see* Figure 6d*); the largest face is **a** (100) and the arrow points at the twin boundary; field of view 6.7×7.7 mm. Photos in immersion by K. Schmetzer.*

in *Figure 8*. Most samples consisted of untwinned single crystals with variable morphology, but two specific habits were frequently seen, both of which were elongated along the c-axis: tabular with a dominant **b** pinacoid (*Figures 6a* and *8A,D*), or tabular with a dominant **a** pinacoid (*Figures 6b,c* and *8B,C*).

Although twinning was rarely observed, *Figures 6d* and *7D* depict a sample from Sri Lanka that consists of a dominant crystal with a smaller second crystal in twin position. The morphology of the larger crystal is depicted in *Figure 8E*.

Colour, pleochroism and colour-causing trace elements

An overview of the chemical properties and coloration of selected samples from all four sources examined is given in *Table II*, and *Figure 9* provides a comparison of the coloration of the faceted natural and synthetic samples. Vanadium-bearing chrysoberyl from Ilakaka *(Figure 10a)* and Tunduru *(Figure 10b)* ranged from pale to very intense green, in very light to medium-light tones. In lighter green chrysoberyl from both sources, pleochroism was not observed or was very weak. All intense or very intense green stones showed identical pleochroic colours with X = yellowish green, Y = green and Z = bluish green, comparable to the pleochroism observed in the synthetic rough from Kyocera (*Tables I* and *II*). Colour intensity is dependent on and directly correlated with V concentration, which was found to vary between 0.03 and 0.15 wt.% V_2O_3 in chrysoberyl from Ilakaka and between 0.04 and 0.30 wt.% V_2O_3 from Tunduru. Chromium contents in the Ilakaka samples were <0.01 wt.% Cr_2O_3. The pale to moderately intense green samples from Tunduru (designated Type 1 in this study) also showed very low Cr contents in the range of 0.01 wt.% Cr_2O_3, while the more intensely coloured samples with higher V contents (designated Type 2) had somewhat greater Cr contents in the range of 0.03–0.04 wt.% Cr_2O_3 *(Table II)*. Iron contents were between 0.08 and 0.32 wt.% Fe_2O_3 for samples from Tunduru, with somewhat higher amounts in chrysoberyls from Ilakaka (0.11–0.55 wt.% Fe_2O_3).

Vanadium-bearing chrysoberyls from Sri Lanka *(Figure 10c)* were yellowish green with moderate saturation. The more intensely coloured samples showed pleochroism with X = yellowish green, Y = green and Z = green. Vanadium contents ranged from 0.05 to 0.11 wt.% V_2O_3, and Cr varied between 0.02 and 0.10 wt.% Cr_2O_3 (i.e., somewhat higher Cr than samples from Ilakaka and Tunduru). Iron ranged from 0.47 to 0.90 wt.% Fe_2O_3.

The intense bluish green samples from Myanmar (e.g., *Figure 10d*) showed strong pleochroism with X = greyish violet, Y = yellowish green and Z = intense blue green. The sample analysed by microprobe had the highest V and Cr contents of our chrysoberyls (0.38 wt.% V_2O_3 and 0.24 wt.% Cr_2O_3), with Fe below the detection limit.

The chemical properties of V-bearing natural and synthetic chrysoberyls given in *Tables I* and *II* are plotted in *Figure*

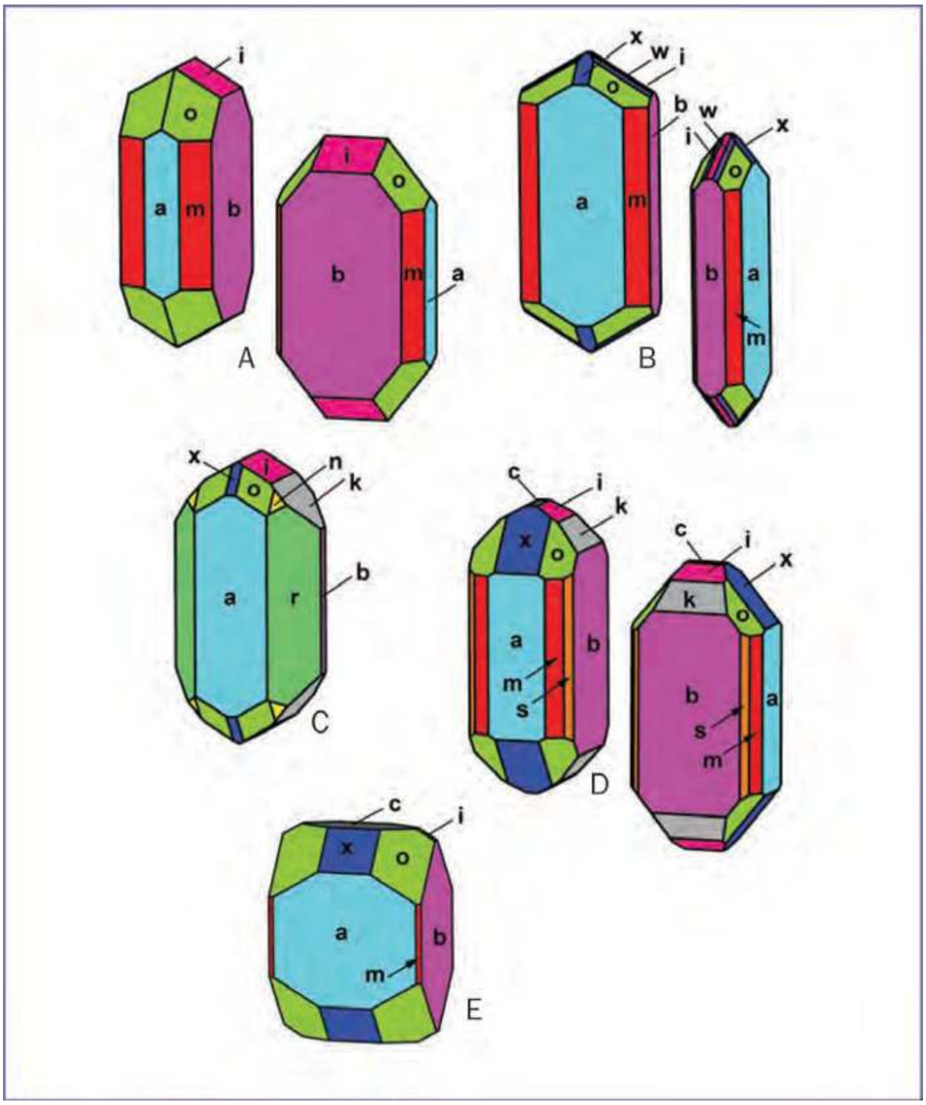


Figure 8: Idealized drawings (clinographic projections) showing the morphology of V-bearing chrysoberyl crystals from various localities; all equivalent crystal faces have the same colour. (A) Ilakaka, views parallel to the a-axis (left) and b-axis (right); the sample (see Figure 6a*) is tabular parallel to* ***b*** *{010}; (B) Ilakaka, views parallel to the a-axis (left) and b-axis (right); the sample (see* Figures 6b *and* 7A*) is tabular parallel to* ***a*** *{100}; (C) Tunduru, view parallel to the a-axis, the sample (see* Figure 6c*) is tabular parallel to* ***a*** *{100}; (D) Mogok, views parallel to the a-axis (left) and b-axis (right); the sample (see* Figure 7C*) is tabular parallel to* ***b*** *{010}; (E) Sri Lanka, view parallel to the a-axis; the drawing represents the larger part of the twin depicted in* Figures 6d *and* 7D*, and the largest face is* ***a*** *(100). Crystal drawings by K. Schmetzer.*

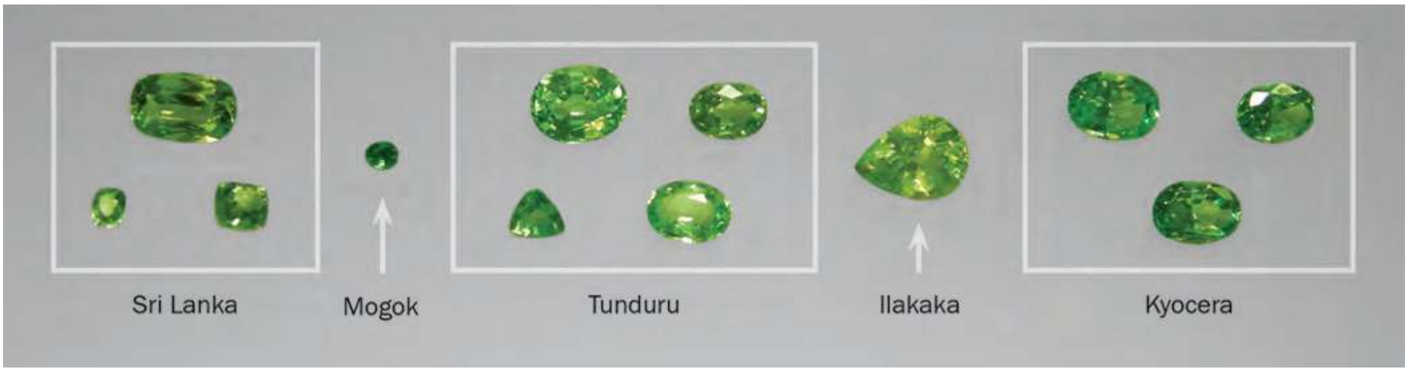


Figure 9: Colour comparison of faceted V-bearing chrysoberyl from various localities and synthetic chrysoberyl grown by Kyocera Corp. The chrysoberyl from Ilakaka weighs 2.09 ct and measures 9.1×7.2 mm. Photo by K. Schmetzer.

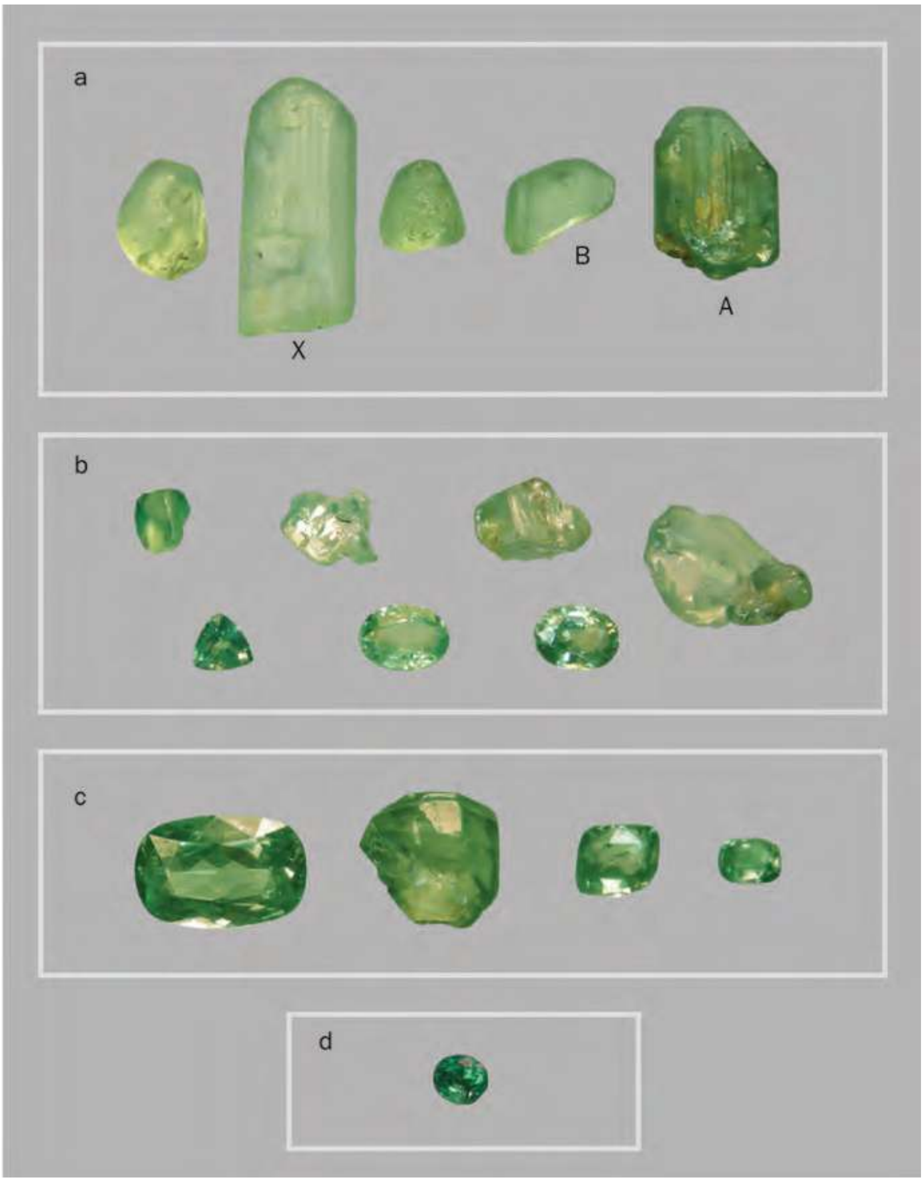


Figure 10: Vanadium-bearing chrysoberyl from various localities. (a) Ilakaka: for the three labelled single crystals, the external morphologies are: sample X (17.9×7.6 mm), tabular parallel to ***b*** *{010}; sample B (7.4×6.5 mm), tabular parallel to* ***a*** *{100}; and sample A (12.7×8.4 mm), tabular parallel to* ***b*** *{010}. (b) Tunduru: the faceted samples weigh 0.38 ct (4.3×4.2 mm) to 0.89 ct. (c) Sri Lanka: the cut stones weigh 1.44 ct (8.4×5.6 mm), 0.53 ct and 0.13 ct. (d) Mogok: the sample weighs 0.08 ct and measures 2.6×2.3 mm. Photos by K. Schmetzer (*Figures a–d *not to scale).*

11. The V and Cr contents are similar in the synthetic chrysoberyl from Kyocera and the natural samples from Ilakaka and Tunduru Type 1. In contrast, the more intense green Tunduru Type 2 samples revealed higher V and Cr contents, and the stones from Sri Lanka showed greater Cr and Fe. The bright bluish green stone from Mogok was unique according to its highly enriched amounts of both colour-causing trace elements, V and Cr *(Figure 11a)*. Iron contents were variable: the synthetic material from Kyocera and the Mogok sample were Fe-free, while moderate Fe contents (0.05–0.35 wt.% Fe_2O_3) were found in chrysoberyl from Tunduru and in some of the Ilakaka samples. Greater Fe values were found in the stones from Sri Lanka and in some of the Ilakaka material *(Figure 11b)*.

Gallium and tin as trace elements

Gallium and tin are common trace elements in natural alexandrite from various sources (Ottemann, 1965; Ottemann *et al.*, 1978). It was no surprise that quantitative or semi-quantitative chemical analyses showed significant amounts of Ga in all samples from the four sources examined in this study. The characteristic X-ray lines of Sn were seen in all of the chrysoberyls from Ilakaka and Sri Lanka, and in most samples from Tunduru. No Sn lines were observed in two of the Tunduru Type 2 gems or in the chrysoberyls from Mogok.

Spectroscopic properties and colour variation under different light sources

Polarized absorption spectra were recorded for rough crystals with tabular habit showing dominant **a** (100) or **b** (010) pinacoids (see *Figures 6, 8* and *10*) and for a few faceted samples that were cut with the tables more or less parallel to one of those pinacoids. None of the faceted stones had the table facet oriented parallel to **c** (001). Due to the morphology of the crystals and the table orientations of the faceted stones, we normally could measure only two of the three possible polarized spectra X, Y and Z for each sample. Therefore, we recorded polarized spectra for X and Z (beam parallel to the b-axis) or for Y and Z (beam parallel to the a-axis); example spectra are depicted in *Figure 12*.

Even with these restrictions, it was evident that the positions of absorption maxima and intensity ratios of absorption bands of V^{3+} (in Cr-free or almost Cr-free samples) are consistent with those observed in the Kyocera synthetics (see *Table I*). In most natural samples, the V^{3+}-related features are superimposed on the known Fe^{3+} spectrum of chrysoberyl. The main absorption maxima assigned to Fe^{3+} are located at 365, 375–376 and 439 nm, with somewhat weaker absorption bands or shoulders at 357, 381 and 430 nm. Only the main absorption band showed a slight polarization dependency, with the maximum at 375 nm for X and Z, and at 376 nm for Y. The Fe^{3+} absorption bands we recorded are consistent for chrysoberyl in the literature (Farrell and Newnham, 1965).

A comparison of non-polarized spectra from the four localities *(Figure 13)* shows

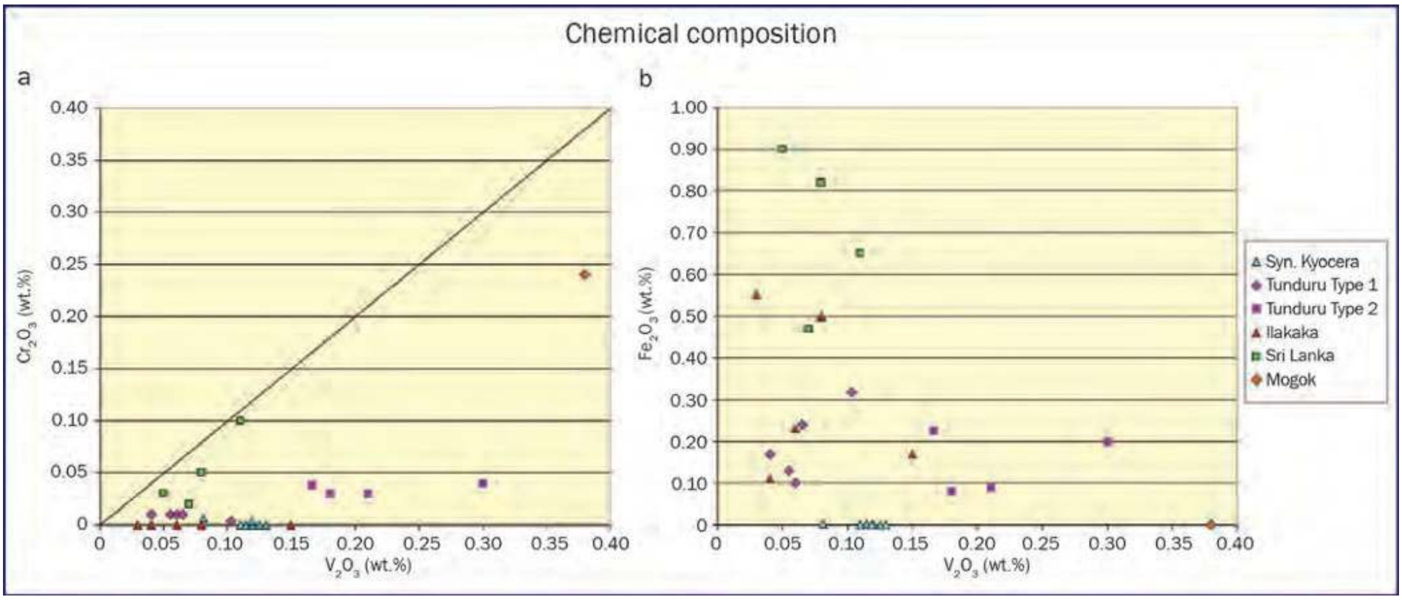


Figure 11: Chemical plots of synthetic chrysoberyl from Kyocera and natural samples from various sources. (a) V_2O_3 versus Cr_2O_3: All points are below the diagonal, as the gems contain more V than Cr. Trace-element contents in the Tunduru Type 1 and Ilakaka stones are similar to the Kyocera material. The Tunduru Type 2 samples reveal higher V and Cr contents, and the chrysoberyls from Sri Lanka also have greater Cr. The sample from Mogok shows highly enriched contents of both trace elements. (b) V_2O_3 versus Fe_2O_3: The Kyocera synthetics and the bright green stone from Mogok are Fe-free, while samples from Tunduru, Ilakaka and Sri Lanka show variable Fe.

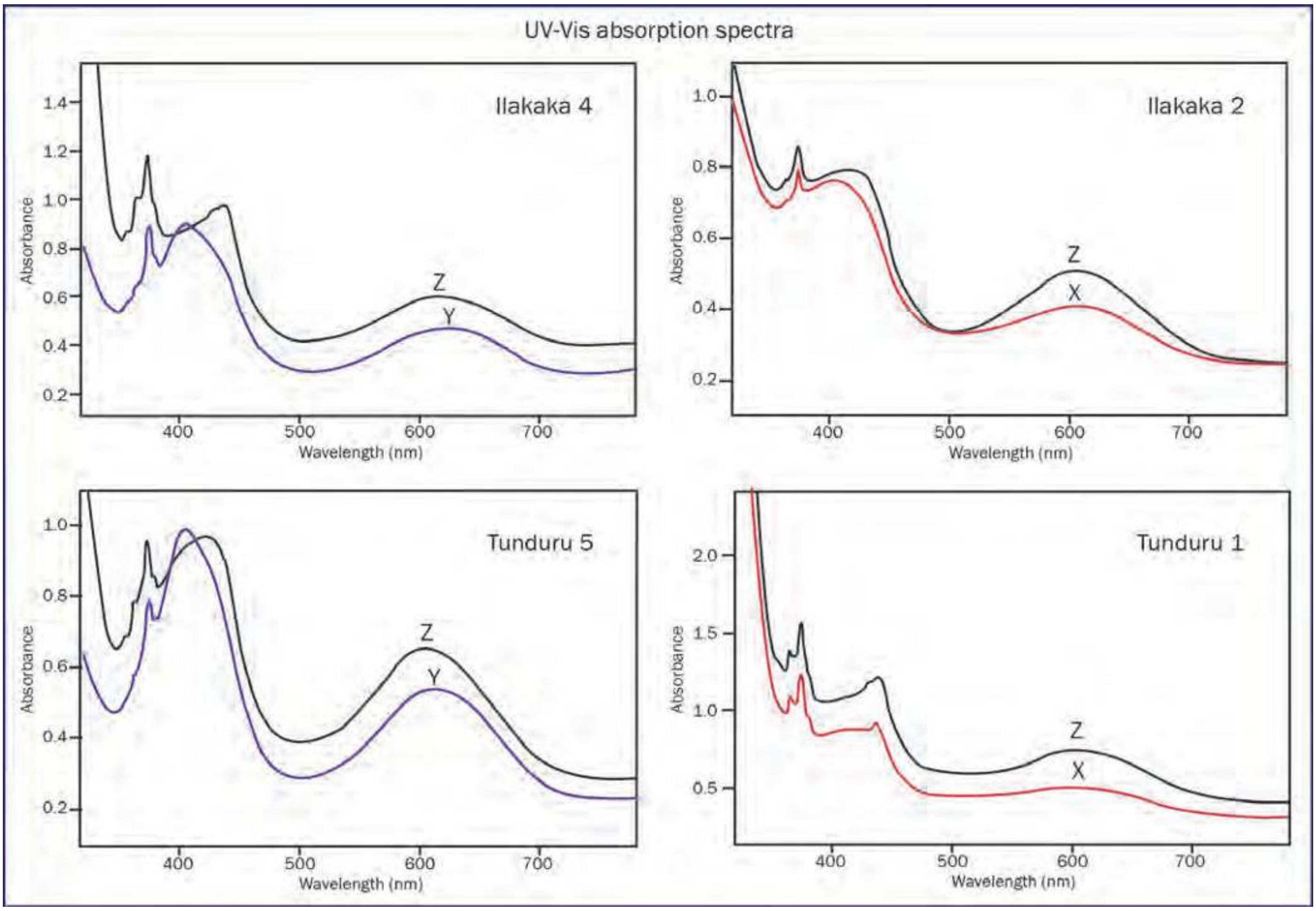


Figure 12: Polarized UV-Vis absorption spectra of one faceted sample and three V-bearing chrysoberyl crystals from Ilakaka and Tunduru, with X || a, Y || b and Z || c. According to the morphology of the samples, polarized spectra were recorded either with the beam parallel to the a-axis (Y and Z polarizations) or parallel to the b-axis (X and Z polarizations). Thickness of samples: Ilakaka 4 = 2.3 mm; Ilakaka 2 = 3.2 mm; Tunduru 5 (faceted) = 3.1 mm; Tunduru 1 = 3.8 mm.

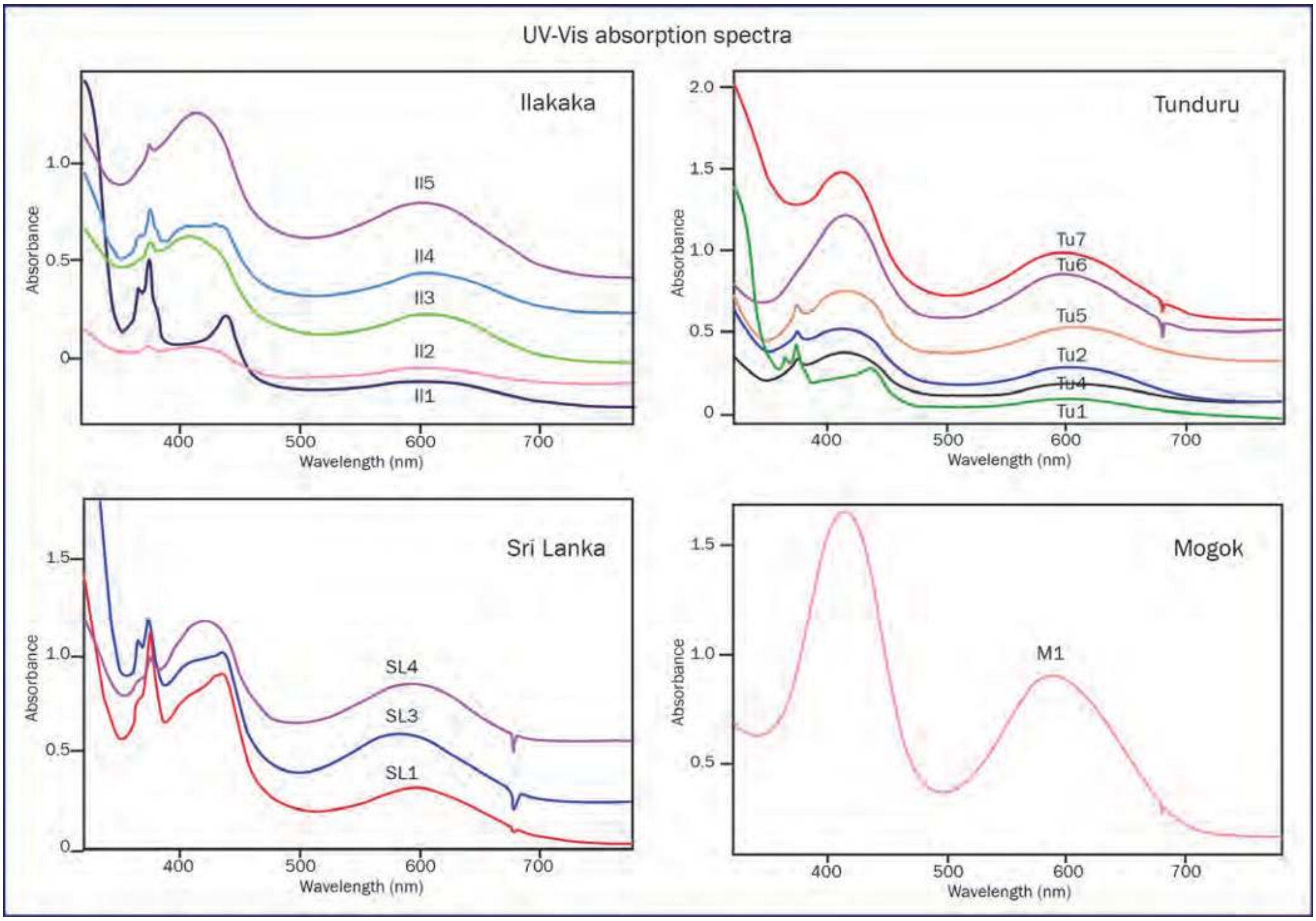


Figure 13: Non-polarized UV-Vis spectra of V-bearing chrysoberyl show absorption bands of V^{3+} and Fe^{3+} (Ilakaka and Tunduru), of V^{3+} and Cr^{3+} (Mogok), and of V^{3+}, Cr^{3+}, and Fe^{3+} (Sri Lanka). Sharp Cr^{3+} lines at ~ 680 nm are recorded for some of the samples only (Tunduru Type 2 [Tu6, Tu7], Sri Lanka and Mogok). The spectra of samples Il2–Il5, Tu2–Tu7, and SL3–SL4 are displaced vertically for clarity; the chemical properties of the samples are given in Table II.

that samples from Ilakaka and some of those from Tunduru do not show the two sharp Cr^{3+} lines at 680 and 678 nm. The Cr contents of these samples are low (<0.01 wt.% Cr_2O_3). In contrast, Cr^{3+} lines were observed in higher-Cr Tunduru samples and in those from Sri Lanka and Myanmar. Due to the experimental conditions, these Cr^{3+} lines are shown in the spectra as 'negative' luminescence peaks (see details in Schmetzer *et al.,* 2013a). Depending on the iron contents of the individual samples, the Fe^{3+} bands described above are also seen with variable intensity in the samples from Ilakaka, Tunduru and Sri Lanka.

In non-polarized spectra (see again *Figure 13*), the position of the first strong V^{3+} absorption band (ν_1) was recorded in the red-orange range at 608–606 nm for Cr-free or almost Cr-free chrysoberyls. Although there is some influence of sample orientation, it is clear that this strong absorption band shifts towards shorter wavelengths with greater Cr contents and with increasing Cr:V ratios *(Figure 14)*. For both samples from Myanmar (Cr:V ratio near 1:1.6), the position of this strong absorption band was recorded in the orange region at 589 nm. For Cr-dominant natural and synthetic alexandrites, this absorption band was shifted further to the greenish yellow range at about 576–573 nm *(Figure 14, Table III)*.

To compare polarized spectra of V-bearing, Cr-free synthetic chrysoberyl with V-free, Cr-bearing synthetic alexandrite, we selected samples grown by Kyocera Corp. (this study) and Creative Crystals (Schmetzer *et al.,* 2012), respectively. As already mentioned for V-bearing synthetic chrysoberyl, the polarized spectra for both trace elements, Cr and V, consist of two strong absorption maxima which are located in the greenish yellow to red-orange range (first maximum, ν_1) and in the blue-violet to violet range (second maximum, ν_2). In *Figure 15* these absorption maxima are shown in all three polarizations. For ν_1, in all three polarization directions the absorption bands for V^{3+} are located at higher wavelengths than for Cr^{3+} *(Table III)*. For this discussion, the Fe^{3+} bands of the synthetic alexandrite are neglected because they are all below 500 nm and therefore not within the range of the ν_1 band. In contrast, the ν_2 bands are much closer to one another in all three polarization directions for both V^{3+} and Cr^{3+}.

For all V- and Cr-bearing chrysoberyls, the V^{3+} and Cr^{3+} absorption bands are superimposed and, in general, no separation in absorption maxima are recorded for these two chromophores. Depending on the Cr:V ratio of an individual sample, the position of ν_1 is shifted from the red-orange at about 608–606 nm (for Cr-free or almost Cr-free samples) towards lower wavelengths (for Cr-bearing samples, see *Table III*). For V-free or almost V-free natural alexandrite, the maximum of ν_1 is observed in the greenish yellow range at 576–573 nm *(Figure 14)*. This shift in the position of the ν_1 band is responsible for the variable coloration of V-dominant chrysoberyl from different sources. It is also responsible for the colour change seen in Cr-dominant alexandrite.

Colorimetric measurements confirm the visual impressions. For V-bearing samples without Cr (e.g., Kyocera synthetic chrysoberyl), a slight variation from green to bluish green is measured from daylight to incandescent light, and for V- and Cr-bearing chrysoberyls with distinct Fe contents (e.g., from Sri Lanka), a small variation from yellowish green to yellow-green is detected *(Figure 16)*. This different colour behaviour is due to the greater Cr as well as the somewhat higher Fe content (see again *Figure 11b*), which in general increases the yellowness of chrysoberyls. Samples with somewhat higher Cr than V (e.g., from Ilakaka) have been described as yellowish green or green (Schmetzer *et al.,* 2002).

These colour variations within V-bearing samples from various sources are quite different from the characteristically distinct colour change seen in Cr-bearing chrysoberyl (alexandrite) between daylight and incandescent light (see, e.g., Schmetzer *et al.,* 2012, 2013a). In alexandrite, the colour change is normally observed in two of the three polarization directions, X and Y. The third direction, Z, remains green or bluish green under both illumination sources. These observations can be explained by the different positions of the ν_1 absorption maxima (see again *Table III*), with X at 570 (greenish yellow) and Y at 564 nm (yellow-green), while Z is at somewhat higher wavelengths (~586 nm) in the orange range. For vanadium all three maxima are above 600 nm in the red-orange range and therefore none of the polarization directions shows a distinct colour change (rather, only a slight colour variation). Consequently, V-bearing chrysoberyl does not show distinct colour change behaviour (for further details on V- and Cr-bearing samples, especially from Myanmar, see Schmetzer *et al.,* 2013b).

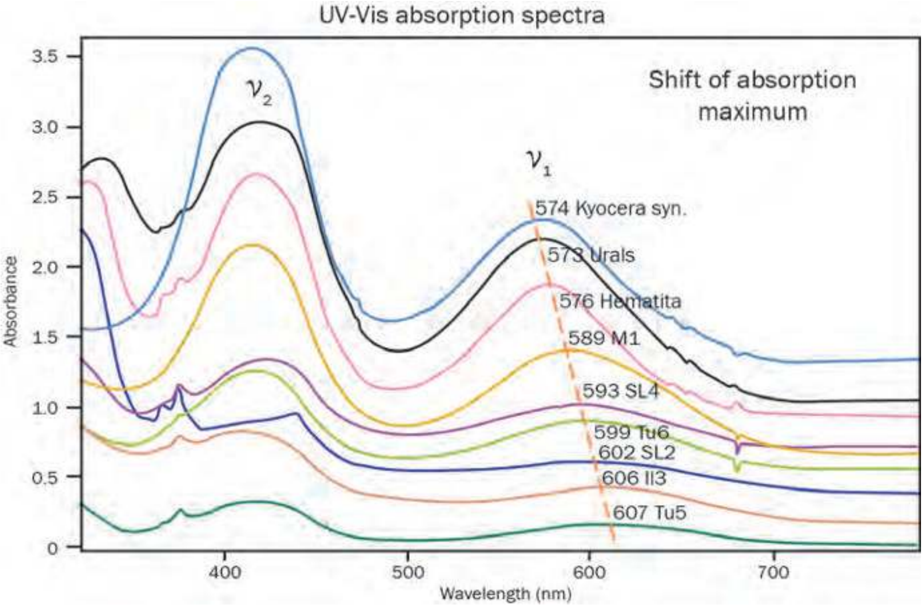


Figure 14: Non-polarized UV-Vis absorption spectra of V-bearing chrysoberyl from various sources (Il = Ilakaka; Tu = Tunduru; SL = Sri Lanka and M = Mogok), together with Cr-bearing alexandrite from Hematita (Brazil) and the Ural Mountains (Russia), as well as synthetic alexandrite grown by Kyocera. Increasing Cr contents cause a shift of the ν_1 absorption maxima towards lower wavelengths. With the exception of sample Tu5, the spectra are displaced vertically for clarity.

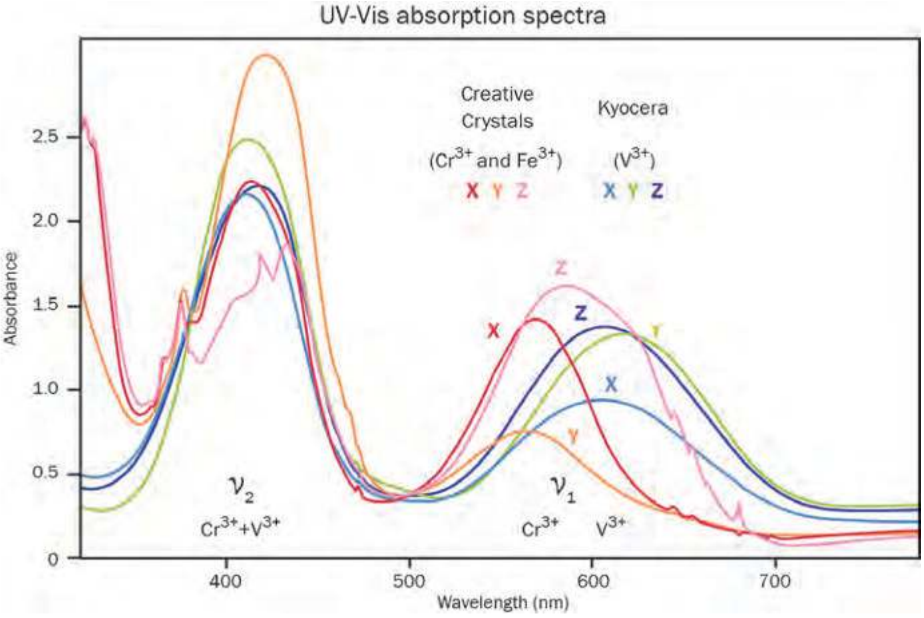


Figure 15: A plot of the polarized UV-Vis absorption spectra of V^{3+} in synthetic chrysoberyl from Kyocera (this paper) and Cr^{3+} in synthetic alexandrite from Creative Crystals (taken from Schmetzer et al., *2012) shows the position of absorption maxima for all three polarization directions (X, Y and Z). The positions of the absorption bands of Cr^{3+} and V^{3+} are close to one another, which explains why separate bands for these ions are not observed in visible-range spectra for samples containing both V and Cr.*

Inclusions

Consistent with previous descriptions of V-bearing natural chrysoberyl from Tunduru, most of our samples from this locality were clean and did not show any mineral inclusions. However, in two Type

1 chrysoberyls we identified numerous tiny apatite crystals *(Figure 17a)* that were partly associated with cavities or growth tubes oriented parallel to the c-axis *(Figure 17b,c)*. Raman analysis of the fluid phase within these elongated cavities showed characteristic peaks of CO_2. A third Type 1 chrysoberyl contained isolated tubes running parallel to the c-axis *(Figure 17d)* and a tiny negative crystal that also was found to contain liquid CO_2. In addition, small partially healed fractures were observed in some samples *(Figure 17e)*. In contrast to this inclusion pattern, one Tunduru Type 2 stone contained numerous minute inclusions identified as feldspar, with Raman spectra indicative of orthoclase (K-feldspar).

A light green chrysoberyl from Ilakaka showed an inclusion pattern consistent with that seen in the Tunduru Type 1 samples. It showed numerous tiny, birefringent, isolated crystals *(Figure 17f)* that were identified as apatite. Some of these apatite inclusions were connected to elongated tubes running parallel to the c-axis of the host. The other samples from Ilakaka did not contain any mineral inclusions.

Table III. Absorption maxima (nm) of the first absorption band (ν_1) in V- and Cr-bearing chrysoberyl and alexandrite.

Property	Polarization			Without polarization
	X || a	Y || b	Z || c	
V-bearing, Cr-free synthetic chrysoberyl from Kyocera	607	618	608	608–606
V- and Cr-bearing chrysoberyl, Mogok[a]	570[b]	599	593	589
Cr-bearing, V-free synthetic alexandrite from Creative Crystals[c]	570[b]	564[b]	586	576–573[b]

[a] Cr:V = 1:1.6.

[b] These directions show a colour change between daylight and incandescent light.

[c] From Schmetzer *et al.* (2012).

In the faceted chrysoberyl from Mogok we identified several small mineral inclusions as calcite. Some of these calcites were part of two- or multi-phase inclusions with a fluid component also showing the Raman lines of CO_2.

In the V-bearing chrysoberyl from Sri Lanka we were unable to identify any characteristic mineral inclusions by Raman spectroscopy.

Characteristic features and distinction of synthetic and natural V-bearing chrysoberyls

Two types of V-bearing synthetic chrysoberyl were produced in the 1990s, by Kyocera in Japan and at various institutes in Novosibirsk, USSR (Academy of Science and/or Tairus). The Kyocera material was characterized for this report and its properties are compared to natural V-bearing chrysoberyl in *Table IV*. The Russian synthetics were examined by H. A. Hänni and K. Schmetzer (Johnson and Koivula, 1996, 1997; see *Figure 18a*) and found to contain extremely high

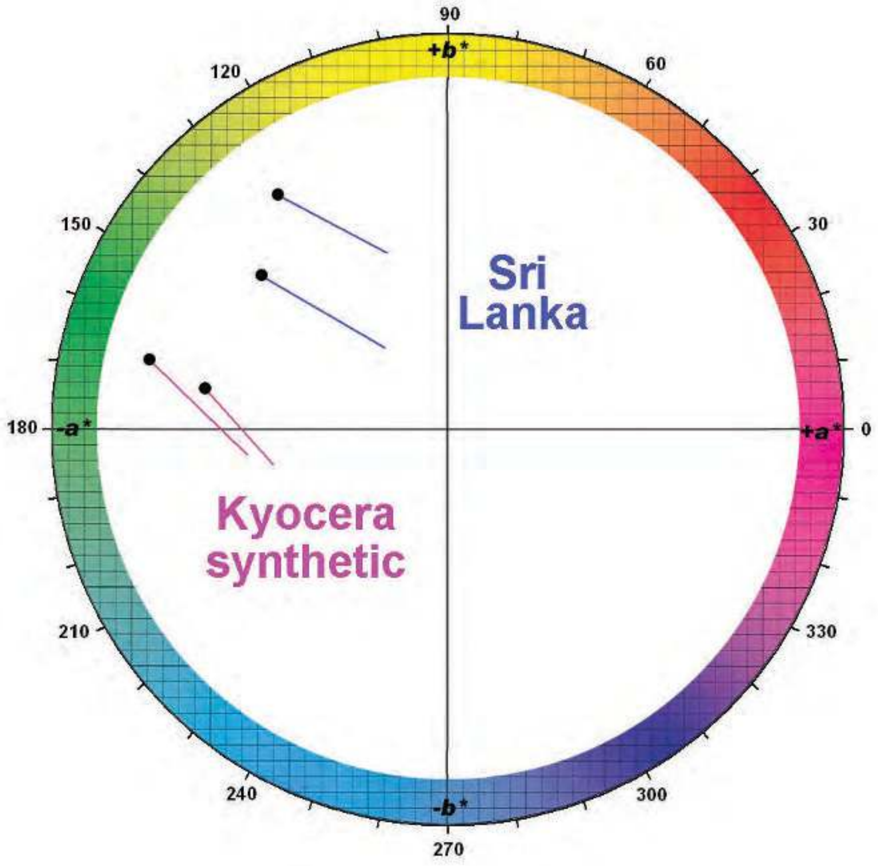
Table IV. Diagnostic properties of some natural and synthetic V-bearing chrysoberyl[a].

Feature	Characteristic	Kyocera synthetic	Tunduru		Ilakaka	Sri Lanka	Mogok[b]
			Type 1	Type 2			
Chemical	Chromophores	V	V, Fe	V>>Cr, Fe	V, Fe	V>Cr, Fe	V>Cr
	Other trace elements	Not observed	Ga, Sn	Ga or Ga, Sn	Ga, Sn	Ga, Sn	Ga
Microscopic	Growth structures	Not observed	Growth patterns in various directions	Growth patterns in various directions	Growth patterns in various directions	Growth patterns in various directions	Growth patterns in various directions
	Mineral inclusions	Not observed	Apatite, occasionally associated with growth tubes	K-feldspar	Apatite, occasionally associated with growth tubes	Not observed	Calcite
	Other inclusions	Gas bubbles	Negative crystals with CO_2, isolated growth tubes		Isolated growth tubes		Fluid inclusions with CO_2
Spectroscopic	Absorption bands	V	V, Fe	V, Fe	V, Fe	V, Cr, Fe	V, Cr
	Sharp Cr lines	Not observed	Not observed	Present	Not observed	Present	Present

[a] For diagnostic properties of V- and Cr-bearing synthetic chrysoberyl and alexandrite grown by the floating zone method (HOC), see Johnson and Koivula (1996, 1997) and Schmetzer *et al.* (2013a); for diagnostic properties of V- and Cr-bearing synthetic chrysoberyl and alexandrite grown by the flux method, see Schmetzer *et al.* (1996, 2012).

[b] For further details, see Schmetzer *et al.* (2013b).

*Figure 16: Colorimetric parameters for two V- and Cr-bearing, Fe-rich chrysoberyls from Sri Lanka (with V>Cr), and V-bearing, Fe- and Cr-free synthetic chrysoberyl from Kyocera are plotted for daylight and incandescent light in the CIELAB colour circle. The neutral point (white point) is in the centre of the a*b* coordinate system and the outer circle represents a chroma of 40. The black circles plot the colour coordinates in daylight D_{65} and the other ends of the differently coloured bars represent the coordinates of the same samples in tungsten light A. The V-, Cr- and Fe-bearing chrysoberyls from Sri Lanka show a small colour variation from yellow green to greenish yellow, whilst the V-bearing, Fe- and Cr-free synthetic chrysoberyls from Kyocera shift slightly from green to bluish green.*



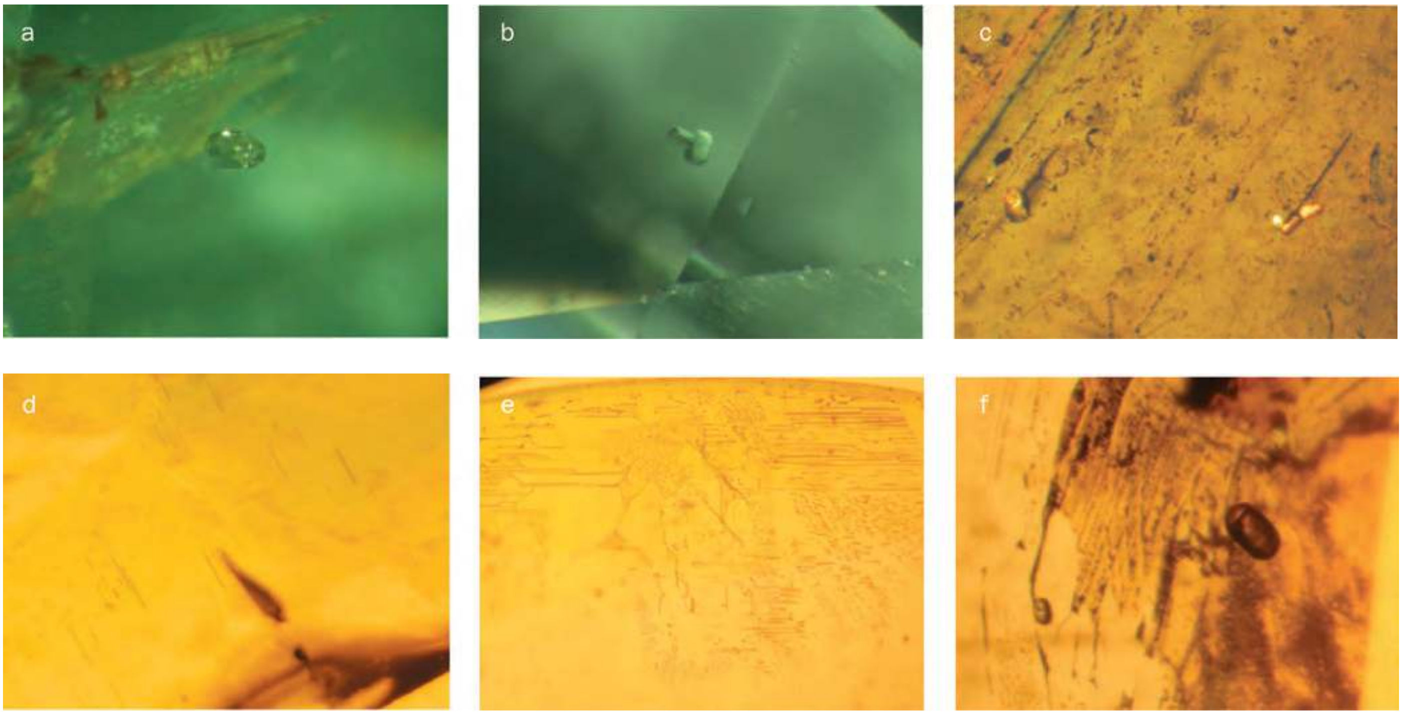


Figure 17: Inclusions seen in V-bearing chrysoberyl from Tunduru (a–e) and Ilakaka (f). Shown here are (a) apatite, (b) apatite next to a cavity, (c) apatite crystals associated with growth tubes oriented parallel to the c-axis, (d) growth tubes running parallel to the c-axis, (e) a partially healed fracture, and (f) apatite. (a,b) Fibre-optic illumination, field of view 1.6×1.2 mm; (c) immersion, crossed polarizers, 3.1×2.3 mm; (d,e,f) immersion, 2.3×1.8 mm, 2.8×2.1 mm, 2.0×1.5 mm, respectively. Photos by M.S. Krzemnicki (a,b) and K. Schmetzer (c–f).

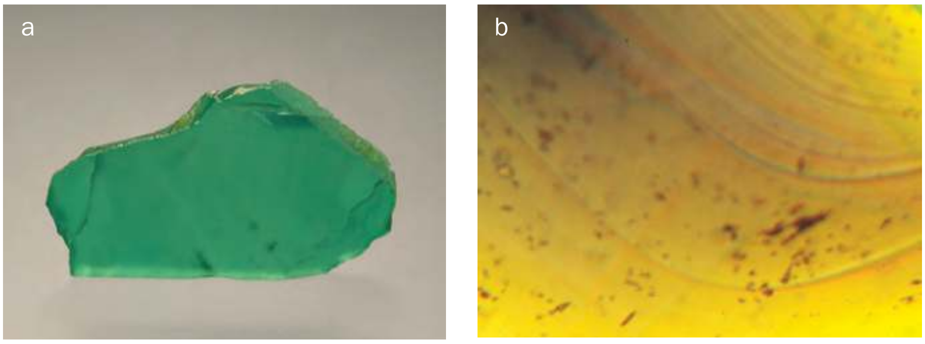


Figure 18: Vanadium-bearing synthetic chrysoberyl grown by the HOC technique in Novosibirsk, Russia. (a) Slice of 1.5 mm thickness, 19.3×10.8 mm; (b) irregularly curved growth striations; immersion, field of view 2.0×1.5 mm. Photos by K. Schmetzer

V contents (1.8 wt.% V_2O_3). Irregularly curved growth striations *(Figure 18b)* suggested this material was produced by the HOC technique. Recently, it was confirmed that a limited quantity of V-bearing synthetic chrysoberyl was grown by this method in Novosibirsk in the 1990s and released into the gem trade (V.V. Gurov, pers. comm., 2012).

Schmetzer *et al.* (1996) examined numerous flux-grown Russian synthetic alexandrites and a few samples were analysed that showed traces of V (up to 0.29 wt.% V_2O_3) in addition to Cr. One such sample even contained more V than Cr. All of the samples with relatively high V contents showed the same twinning, growth structures and inclusions (mostly various forms of residual flux) as observed in the Cr-dominant samples with lower V contents. Extremely high amounts of germanium (up to several wt.% GeO_2) are a characteristic chemical property that can be used to identify this type of synthetic alexandrite and V-bearing chrysoberyl.

Figure 19: Light yellowish green or greenish yellow chrysoberyls such as this 2.44 ct gem (9.7×5.3 mm) typically contain — in addition to some Fe — small amounts of V and/or Cr (in this instance, microprobe analyses showed 0.77 wt.% Fe_2O_3, 0.02 wt.% V_2O_3, and 0.01 wt.% Cr_2O_3). Photo by K. Schmetzer.

Chemical properties of samples from Tunduru reflect two different types: Type 1 is almost Cr-free and contains Sn, while Type 2 contains somewhat greater amounts of Cr (with V>>Cr) but no Sn was detected in some of those samples. These data are consistent with the literature (Johnson and Koivula, 1996; Mayerson, 2003). The trace-element pattern of V-bearing chrysoberyl from Ilakaka is similar to Type 1 Tunduru material. The few samples examined from Sri Lanka showed traces of both V and Cr, as well as unambiguous X-ray emissions of Sn. The samples from Mogok showed relatively high V and Cr contents, but no Sn. All natural samples with the exception of chrysoberyl from Mogok showed distinct Fe and characteristic X-ray lines of Ga were detected in stones from all localities. In contrast, the Kyocera synthetics contained little or no Cr and Fe, and neither Ga nor Sn was detected.

The majority of the natural V-bearing chrysoberyl displayed diagnostic growth patterns that were clearly observable in immersion. Some of the natural samples also contained characteristic inclusions such as apatite (occasionally associated with cavities or growth tubes), K-feldspar, calcite and negative crystals. The Kyocera synthetics showed no growth structures or mineral inclusions.

The absorption spectra of V-bearing chrysoberyl from three localities show dominant maxima due to V^{3+} (Tunduru and Ilakaka) or mixed V^{3+} and Cr^{3+} (Sri Lanka), as well as the characteristic absorption lines of Fe^{3+}, which were not present in the synthetic chrysoberyl grown by Kyocera.

Consequently, a combination of microscopic, spectroscopic and chemical features provide a clear distinction of natural V-bearing chrysoberyl from its known synthetic counterparts.

Conclusions

Since its discovery in Tunduru, Tanzania, in the mid-1990s, 'mint' green V-bearing chrysoberyl has remained a rare gem material. Despite the demand for this attractive gem, only small quantities have been produced from the known deposits at Tunduru, Ilakaka, Sri Lanka and Mogok.

Until now, the more yellowish green material from Sri Lanka containing relatively higher Fe contents and V>Cr have not been separated from green chrysoberyl from Sri Lanka with Cr>V. This also applies to chrysoberyl from other sources containing both V and Cr in variable but low amounts. Several chrysoberyls from various sources, typically with high Fe contents and low V and/or Cr (<0.04 wt.% oxide) have been analysed by the present authors. Such stones (see, e.g., *Figure 19*) are consistently light yellowish green or greenish yellow, and no separation of these gemstones according to colour or colour cause (i.e., V:Cr ratio) is presently done by the trade.

The two intense bluish green samples examined by the present authors came from Mogok to collections in the UK in the 1970s, and additional samples produced during this time may exist in other private or museum collections. The rediscovery of the original source in the Mogok area of these magnificent, bright gems would certainly cause excitement.

The separation of natural from synthetic V-bearing chrysoberyl can be successfully accomplished by a combination of microscopic, spectroscopic and chemical criteria, provided the gemmologist has an awareness of the different types of natural and synthetic materials produced in the past. Characteristic microscopic features that may help identify natural materials are inclusions and growth structures, while the synthetics contain no inclusions or may reveal curved growth structures, gas bubbles or residual flux inclusions (depending on the growth technique used). Spectroscopic and chemical data are useful for indicating the presence and ratio of various colour-causing trace elements (V, Cr and Fe). Natural chrysoberyls contain traces of Ga and usually Sn, which are both absent from synthetic material.

Acknowledgements

Most of the V-bearing chrysoberyl samples examined in this study were obtained from the H.A. Hänni collection at SSEF. For the loan of additional samples, the authors are grateful to Dr R. Hochleitner (Bavarian State Collection for Mineralogy, Munich), P. Tandy (Department of Earth Sciences at the Natural History Museum, London) and Dr E. Fritsch (University of Nantes, France). In addition, natural V-bearing chrysoberyls were kindly supplied for the present study by C. Cavey (London), D. Gravier (Poncin, France) and S. Hanken (Waldkraiburg, Germany), from their collections or company stock. Thanks go also to Dr W. Balmer (Unterseen, Switzerland) for performing colorimetric measurements. Dr V.V. Gurov (Institute of Geology and Mineralogy, Russian Academy of Sciences, Novosibirsk) provided information about the Russian production of synthetic chrysoberyl and alexandrite in the past.

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