LAB UPDATE

Yellow lead-glass-filled sapphire

By GIT-Gem Testing Laboratory

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Introduction

Lead-glass-filled corundum produced by Thai gemstone-heating experts has been circulating in the gem trade over a decade. In 2004, the lead-glass filled ruby was firstly entered the market (Rockwell & Breeding, 2004; Smith et al., 2005, McClure et al., 2006; Milisenda et al., 2006). Later in 2012, the blue cobalt glass-filled sapphire was introduced to the trade (Leelawatanasuk, 2012 and Leelawatanasuk et al., 2013); and in Mid 2014, the green lead-glass-filled sapphire was encountered in the market (Henn et al., 2014; Leelawatanasuk et al., 2015). Recently, GIT-Gem Testing Laboratory (GIT-GTL) received a newly unusual yellow stone that the owner claimed to be a normal heated sapphire and wanted to check for beryllium treatment.

Material and methods

The study sample was a 10.37 ct. yellow faceted stone came in mixed cut and oval shape (Figure 1). Standard gemological properties were collected by using basic gem instruments. The internal features were observed under normal-type microscopes. For advanced testing, FTIR spectrometer (model Nicolet 6700), EDXRF spectrometer (model Eagle III), UV-Vis-NIR spectrometer (model PerkinElmer Lambda 950), and X-radiography (Softex SFX-100) were used to collect the specific spectroscopic data.

Figure 1: The 10.67 ct. yellow sapphire sample submitted to GIT-GTL for identification. Photo by C. Onlaor.

RESULTS

General properties

The standard gemological properties of the stone are consistent with corundum (see Table 1), i.e., R.I. 1.760-1.770, Birefringence 0.010, S.G. 3.99 and Optic Character as Uniaxial Negative. This stone exhibits weak orange fluorescence to long-wave UV radiation and very weak to short-wave UV radiation.

Table 1: Properties of yellow glass-filled sapphire*

Property	Values and Description
Refractive Index	1.760–1.770 (0.010)
Polariscope Reaction	Double Refractive, Uniaxial Negative
Specific Gravity (Hydrostatic)	3.99
UV Fluorescence	LW: weak orange, SW: very weak orange
Inclusion	**Natural features:** fingerprints, repeated twinning **Lead-glass filled features:** orange and blue flash effect, tabular trapped gas bubbles, yellow color concentration along fissures, and glass-filled cavities on the surface

Remark: *Based on the testing of stone weighing 10.67 ct. (as shown in figure 1)

Microscopic features

Microscopic observation reveals several natural inclusions, such as fingerprints, repeated twinning (Figure 2). In addition, the sapphire contains many fissures that obviously show orange and blue flash effect (Figure 3, left). Many tabular trapped gas bubbles (Figures 3, right) and yellow color concentration (Figure 4, left) can be easily observed along fissures in the near-colorless host corundum. Glass-filled cavities are also seen on the surface (Figures 4, right).

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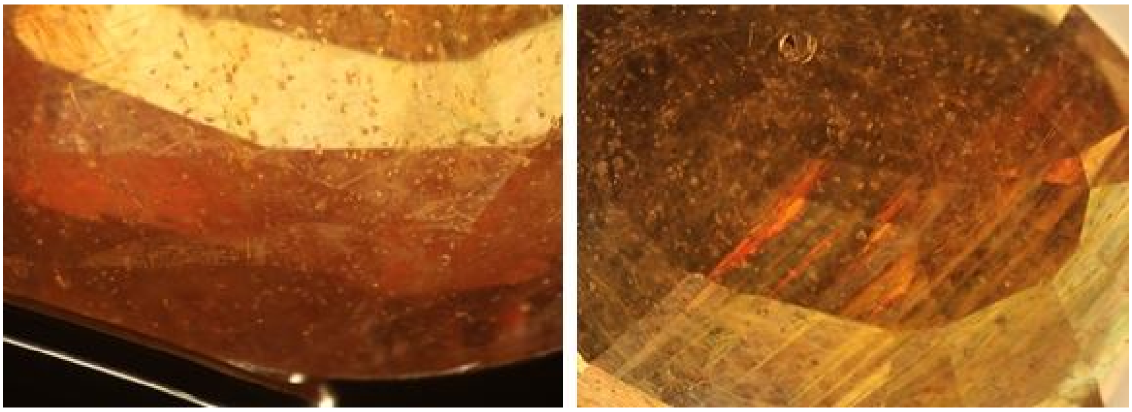

Figure 2: Fingerprint-like inclusions (left) and repeated twinning (right) are natural characteristic features of this yellow glass-filled sapphire, magnified 15x. Photos by P. Ounorn.

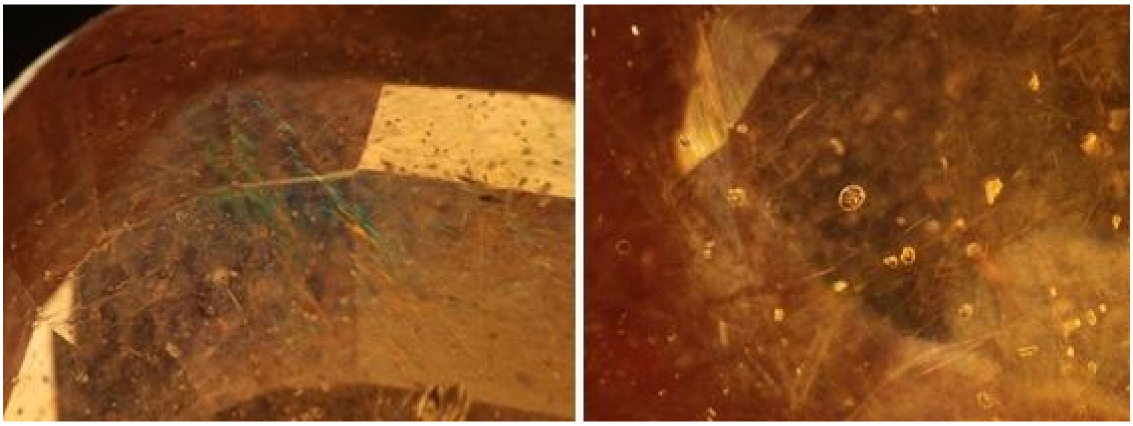

Figure 3: The stone shows orange and blue flash effect (left), magnified 15x; and trapped gas bubbles along fissures (right), magnified 25x. Photos by P. Ounorn

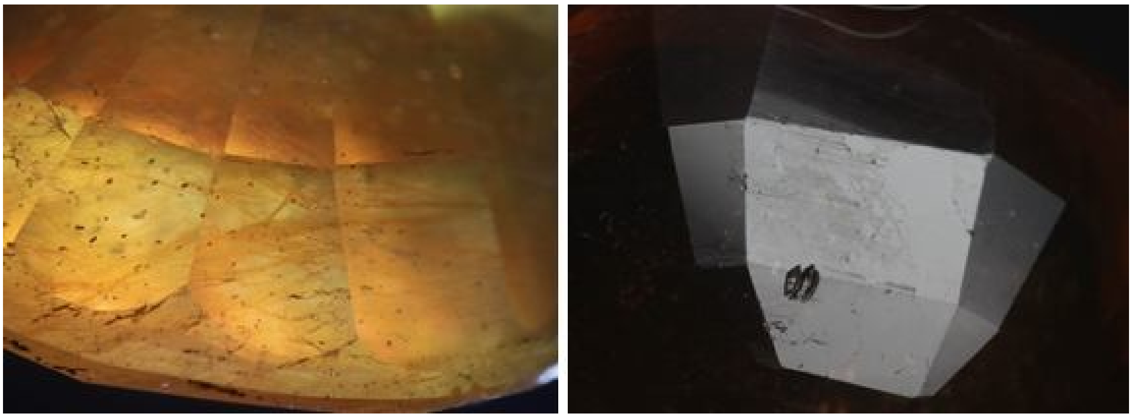

Figure 4: The yellow color concentration along fissures (left), and glass-filled cavities seen on the-surface under reflected light (right), magnified 15x. Photos by P. Ounorn

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ADVANCED TESTING

Spectra

The advanced analytical testing, such as FTIR, UV-Vis-NIR, EDXRF spectrometry, X-radiography, are also helpful to give additional details of the stone. Infrared spectrum of the sample was recorded in the mid-IR with FTIR spectrometer. The spectrum clearly shows strong absorption bands at approximately 3480 and 2618 cm^{-1} that may be related to the glass filler (Figure 5).

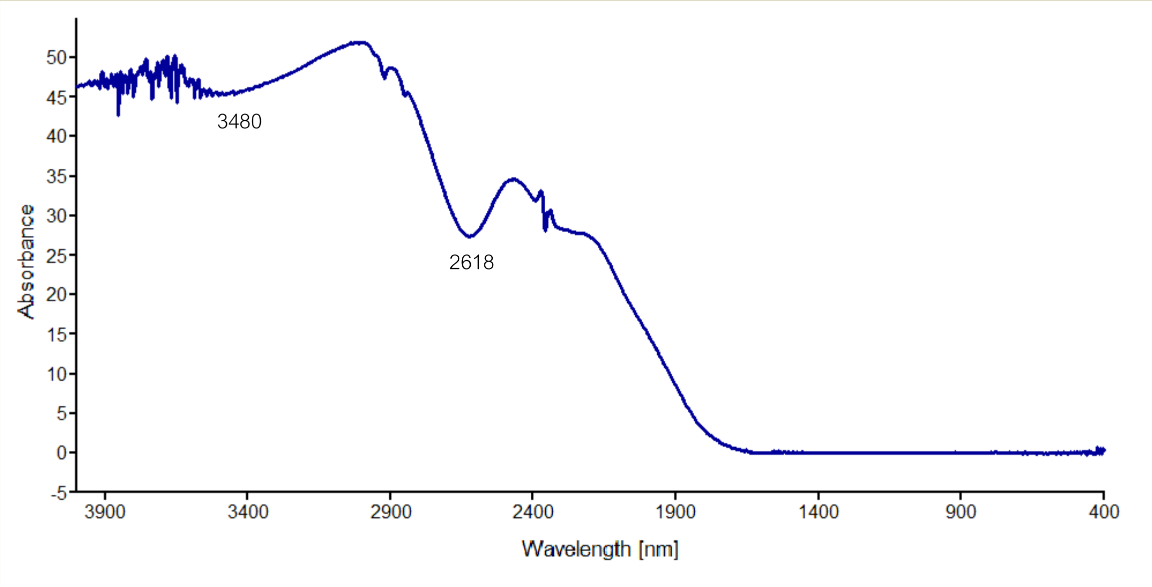

Figure 5: Mid-FTIR spectrum of the yellow glass-filled sapphire showing strong absorption bands at approximately 3480 and 2618 cm^{-1}.

UV-Vis-NIR absorption spectra of the sample were recorded over the 380-800 nm range to confirm the cause of its coloration (Figure 6). The spectra displays an absorption band at ~560 nm and a continuous increase absorption pattern from ~500 nm towards the UV region (causing yellow coloration) that may be related to yellow vanadium-doped glass filler (Abdelghany and Hammad, 2011 and 2014). A small iron-related peak at ~450 nm is also present (Figure 6).

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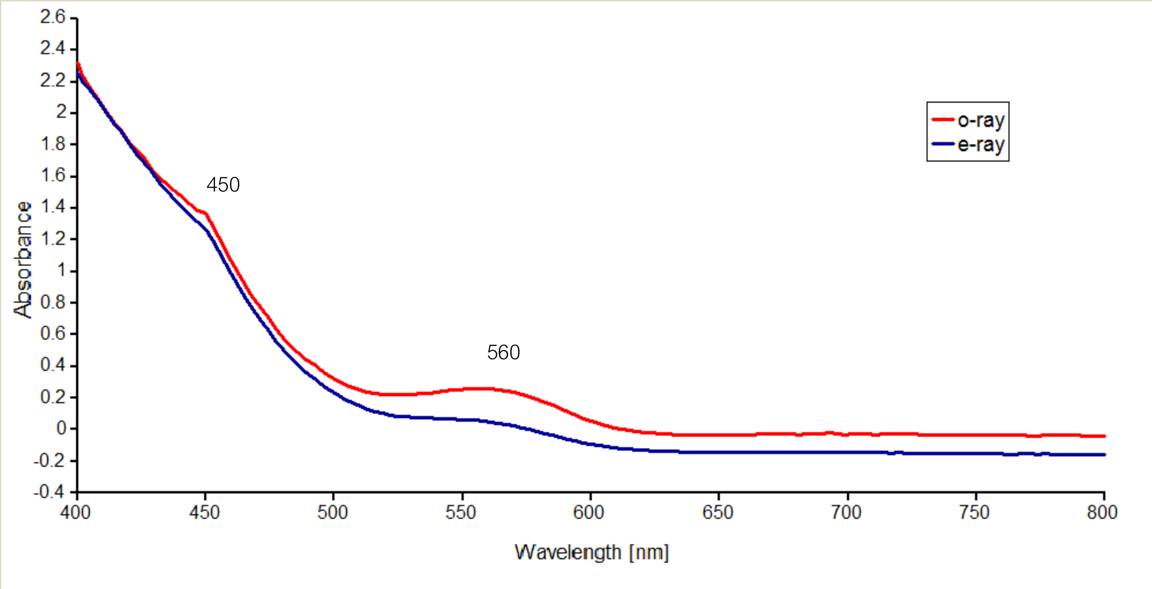

Figure 6: UV-Vis-NIR spectrum of the stone showing an absorption band at ~560 nm and a continuous increase absorption pattern from ~500 nm towards the UV region (probably related to yellow vanadium-doped glass filler) as well as a small iron-related peak at ~450 nm.

Chemistry

The semi-quantitative chemical analysis of host sapphire by EDXRF spectrometer shows a typical glass-filled sapphire with elevated lead content (see Table 2). By contrast, the chemical composition of glassy residue analyzed at stone surface comprises major constituents of lead, silicon and aluminum (probably contributed from the host corundum) with significant content of vanadium and trace amounts of iron, chromium, gallium and titanium. The significant vanadium content present in the glass filler is in good agreement with the observed vanadium absorption band at ~560 nm and the continuous increase absorption pattern from ~500 nm towards the UV region as shown in figure 6.

Table 2: Chemical contents of the yellow sapphire obtained by EDXRF.

Element (wt.% Oxide)	Al_2O_3	SiO_2	TiO_2	V_2O_5	Cr_2O_3	Fe_2O_3	Ga_2O_3	PbO_2
Host sapphire	97.63	0.48	0.005	0.02	0.23	0.41	0.05	1.17
Glassy residue on the surface	20.37	13.49	0.04	1.60	0.15	0.31	0.22	63.85

X-radiography

The X-radiograph of the sample clearly reveals glass-filler areas along fissures and cavities (Figure 7). The filler appears darker than the host sapphire in this positive image; the light and dark patterns correspond to differences in the penetration capability of the X-rays through corundum versus lead glass.

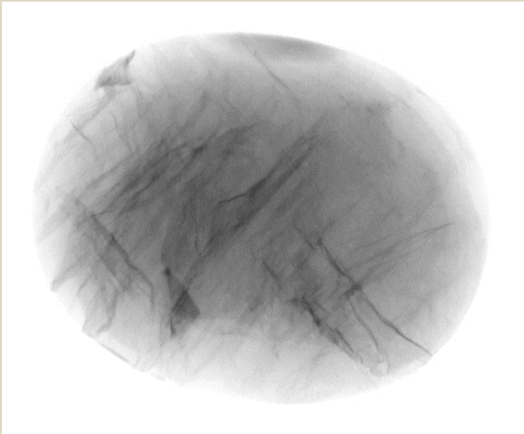

Figure 7: X-radiograph of the yellow sapphire showing distinct opaque areas along the fissures and cavities. Photo by P. Ounorn.

Conclusions

Based on the aforementioned data, the submitted sample is originally a natural near-colorless sapphire that contains many open fissures. Those fissures and cavities were later filled with vanadium (causing yellow coloration) lead glass to modify the stone's color and clarity. Hence this stone should be called "yellow lead-glass-filled sapphire" similar to their predecessors. As such, this yellow glass-filled sapphire can be easily identify by using the same criteria for distinguishing lead-glass type treatments, such as orange and blue flash effect, yellow color concentration and trapped gas bubbles along fissures, as well as glass-filled cavities on the surface (may be difficult to observe by 10x loupe for malee size stone). For advanced testing, FTIR, UV-Vis-NIR, EDXRF spectrometry and X-radiography, are also the essential tools to confirm the result.

Even though the stability test has not been carried out on the yellow lead-glass-filled sapphire yet, this treated stone is likely to pose durability problems similar to those found in the previous lead-glass filled corundum; i.e., during jewelry manufacturing/repair and cleaning, the glass filler could

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be etched by certain acidic or basic solutions, and heat from a jeweler's torch may easily melt the glass (McClure et al., 2006; LMHC, 2012; GIT, 2012; Leelawatanasuk et al., 2013).

It should be noted further that careful observations are strongly recommended for traders and jewelers to avoid any mix-up and/or misidentification of this newly treated material, especially for melee or mounted stones. In addition, it is no doubt that other colored lead-glass-filled sapphires could be available in near future.

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