A microscopy-based screening system to identify natural and treated sapphires in the yellow to reddish-orange colour range

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Abstract: *Features visible under a microscope of untreated, heat-treated and beryllium-diffusion-treated yellow, orange and reddish-orange sapphires, including padparadschas, are presented. A microscopy-based screening system for recognition and distinction of untreated, heat-treated and diffusion-treated sapphires combines structural features such as growth structures, colour zoning and inhomogeneous colour distribution patterns with the visual appearance of inclusions. Spectra and chemical compositions may be added to these microscopic characteristics and applied to an evaluation of samples of unknown origin and unknown treatment history. These properties combine to form a type of locality-specific data set, and are considered in the light of present knowledge about treatment techniques. The screening system allows the recognition of most untreated or heat-treated samples from Sri Lanka and Montana, U.S.A., and their distinction from beryllium-diffusion-treated samples from Sri Lanka, Montana, U.S.A., Ilakaka, Madagascar, and Songea, Tanzania.*

Keywords: *absorption spectroscopy, beryllium diffusion, colour zoning, growth structures, heat-treatment, inclusions, padparadscha, sapphire, screening system, trace-element chemistry*

In a previous paper (Schmetzer and Schwarz, 2004) we summarised the knowledge of causes of colour in untreated, heat-treated and diffusion-treated orange and pinkish-orange sapphires (*Figure 1a, b, c, d*). The present paper deals with methods for the recognition of beryllium-diffusion-treated sapphires in the yellow, orange or reddish-orange colour range and for distinction of these sapphires from different types of untreated or heat-treated samples. The paper consists of three parts:

Part I: Introduction; Materials and methods; Alteration of inclusions
Part II: Growth structures; Colour zoning and inclusions; Chromium and iron contents; Spectra of sapphires from Sri Lanka, Montana, Ilakaka and Songea
Part III: A microscopy-based screening system; Case studies; Conclusions

BOX A: Alteration of inclusions upon heat treatment and diffusion treatment of corundum

Because low oxygen pressure inhibits development of the orange colour centre, Be-diffusion treatment of sapphires is generally performed in oxidizing atmospheres (Emmett *et al.*, 2003). In the 1990s, special furnaces with Kanthal Super 1900 molybdenum disilicide heating elements allowed heat treatment of corundum for longer periods at temperatures up to 1820°C in an oxygen-bearing environment. Special designs allowed treatment up to this temperature under pressures between 5 and 100 atmospheres, especially to reduce the times necessary to develop the desired colour change (*Figure 4*). With Zircothal (yttrium-stabilised zirconium oxide) heating elements, heat treatment of corundum in oxidising atmospheres at temperatures up to 2000°C can be performed. Furnaces of the types mentioned above, i.e. with both types of heating elements with or without high pressure design, have been available in Thailand since before 1995 (H. Linn, pers. com., 2002, 2003). These furnaces were used for several years for 'simple' or 'normal' heat treatment of ruby and sapphire before Be-diffusion treatment started commercially, probably in 2001 (see Coldham, 2002).

Figure 4: High temperature furnace 'ruby star' that allows commercial treatment of corundum in oxidising environment at temperatures up to 1820°C and pressures up to 100 bars; courtesy of Linn High Therm GmbH, Eschenfelden, Germany.

Other furnaces used for commercial heat treatment of corundum at temperatures up to at least 1850°C are fired by gas (T. Häger, pers. com., 2003).

Dissolution of rutile needles or larger crystals and diffusion of titanium within the corundum lattice takes place above 1550°C. Thus, the observation of residues of rutile needles or diffusion haloes around rutile crystals only indicates heat treatment above about 1550°C. Most silicate inclusions and apatite (see Adolfsson *et al.*, 1999) decompose or melt at temperatures below 1500°C, but zircon, monazite and xenotime commonly survive. Melting temperatures of 1995°C for xenotime and between 2045 and 2072°C for natural and synthetic