MICROSCOPE Vol 56:2 p.87-93 (2008)

Extreme Microchemistry[1]

Sebastian B. Sparenga
McCrone Research Institute*

KEYWORDS

Microchemistry, Pyrex®, soda lime glass, boron, turmeric, borosilicate glass.

ABSTRACT

These days, people use the word *extreme* to describe just about everything, including extreme-Frisbee, extreme-fishing, and even extreme-walking. Why not microchemistry?! A case example involving the microchemical analysis of glass samples to test for differences between original Pyrex kitchenware and new Pyrex kitchenware will be discussed. The microchemical portion of the procedure requires much practice and patience, hence warranting the "extreme" title.

INTRODUCTION

In 2006, McCrone Research Institute was involved with a project investigating whether there is a difference between the original Pyrex kitchenware and new Pyrex kitchenware. The main question was whether the new Pyrex kitchenware was composed of borosilicate glass. This concern stemmed from consumers' complaints stating that their new Pyrex kitchenware was shattering and in many cases causing injuries when it was supposedly being used properly for cooking and baking purposes.

Having been around Pyrex labware for many years, I always assumed that the Pyrex brand name was synonymous with borosilicate glass; I never considered that the kitchenware would be any different. Nonetheless, it seemed to be an interesting project and it was decided that refractive index analysis and scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS) would be used to analyze the samples. The only problem was that SEM/EDS is not able to detect boron because it only detects elements heavier than carbon. The presence of boron would have to be detected by another method, and microchemistry immediately came to mind.

The *Handbook of Chemical Microscopy* has a microchemical test for detecting boron (1). However, it requires getting the glass into solution and the idea of using hydrofluoric acid to do so was not very appealing. After discussing this issue with several colleagues, I was offered advice on how to use a flux to proceed with a microchemical test for boron in glass (2). I was instructed on a way to free the boron from the glass by using this flux, which is also described in *The Particle Atlas* (3). After much practice and patience, I was able to obtain positive results for boron in known borosilicate glass.

Research into the current manufacturing of Pyrex brings up interesting information. Although Pyrex has been manufactured since 1915, its current production in North America has been done by World Kitchen since 1998. World Kitchen openly states on their Web site that "PYREX® glass products are made using a tempered soda-lime glass composite" (4). This answered the question of whether the old and new Pyrex kitchenware is the same, however, we were asked to proceed with our analysis.

Three samples were received for analysis. One sample was a 20+ year old used Pyrex baking dish, another sample was a square-cut portion of a baking dish purchased earlier that week, and the last sample consisted of three fragments from a shattered baking dish (also recently purchased). These were all to be analyzed to determine whether any were composed of borosilicate glass.

---

1 Presented at Inter/Micro 2007, Chicago, IL
* 2820 South Michigan Ave, Chicago, IL 60616

MATERIALS AND METHODS

A small piece of glass (<1mm) was chipped away from each of the three glass samples using a carbide scribe. Each sample was tested by the following methods: refractive index analysis, SEM/EDS, and microchemical testing for the presence of boron.

Refractive index was measured using an Olympus BH-2 polarized light microscope (PLM). The Becke Line method was followed to determine the refractive index by immersing the sample in successive refractive index of known liquids until the particle showed no contrast under monochromatic sodium D light.

After refractive index determination, another portion of each sample was mounted to an SEM stub with carbon tape for EDS elemental analysis using an Amray 1810 SEM equipped with an EDAX detector.

A microchemical test for boron was the final step in the analysis of the glass samples. The boron must first be freed from the glass, which was done in the following manner. A portion of the glass was ground with a mortar and pestle until it was the consistency of a fine powder. Next, sodium carbonate was used as a flux to "open" the material. The sodium carbonate was gathered on a platinum wire loop and heated until molten to create a bead in the loop. A portion (about $1/20^{th}$ of the amount of sodium carbonate) of the glass that had been pulverized into a fine powder was added to the sodium carbonate bead. The combination was heated over an alcohol lamp and kept molten hot for approximately 5 minutes (Figure 1).

The bead was then cooled for approximately 1 minute before being placed into a 1:1 dilution of hydrochloric acid in a spot plate (Figure 2).

A drop of the resulting solution was placed on a slide, and one piece of a turmeric-impregnated fiber was placed into the drop. The turmeric fibers were obtained from the Cargille Chemical Microscopy Set II, but the fibers can be made if this set is not available (1). The fiber was placed into the drop in one of the following ways: by hanging it into the drop by way of a piece of molded clay, by placing it in the drop so that half of it is in the drop and the other half is out of the drop, or by completely submerging it in the drop.

The fiber remained in the drop until the drop had gone to complete dryness. Once the drop was dry, the fiber was removed and transferred to a new slide, and a coverslip was added. As a preliminary indication of the presence of boron, the yellow fiber showed a red color where it had been immersed in the now-evaporated drop (Figure 3a and b).

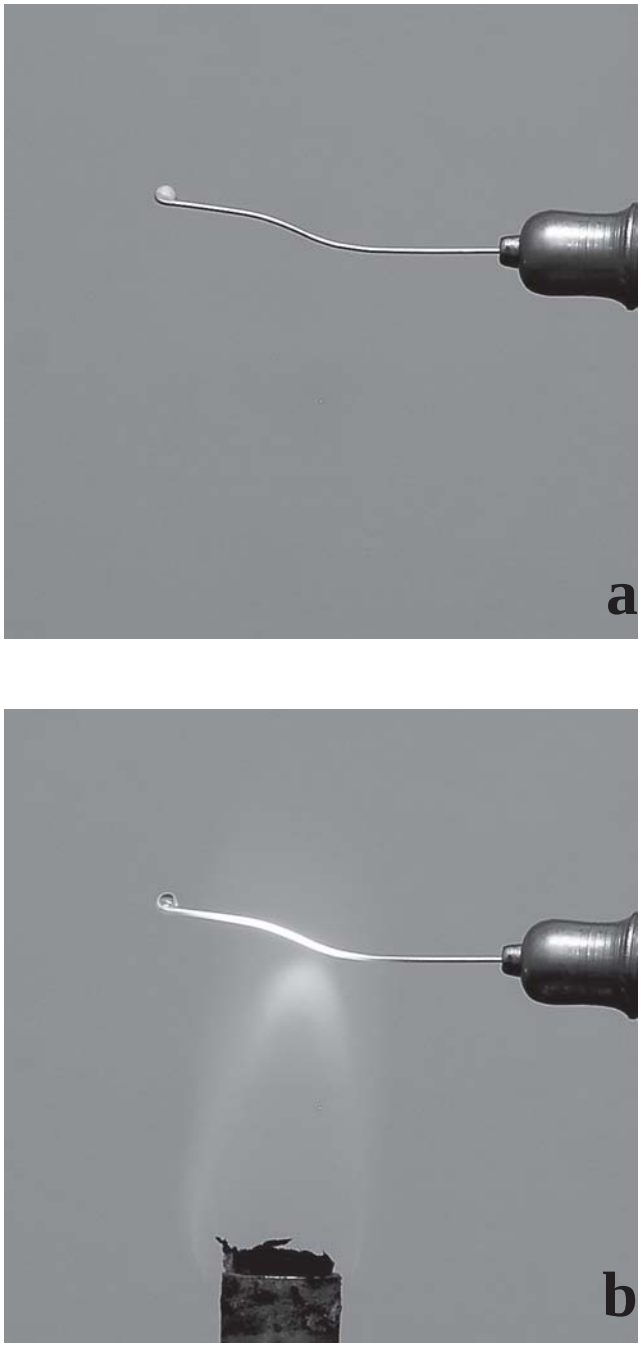

Figure 1: a) Image showing sodium carbonate/pulverized glass combination held in platinum wire loop, b) image showing the loop being heated over alcohol lamp.

Figure 2: Image of platinum wire loop with bead of sodium carbonate and pulverized glass being submerged into 1:1 dilution of hydrochloric acid.

To confirm the presence of boron, a dilute base (1% sodium hydroxide) was added to the edge of the coverslip and allowed to run under by capillary action. If boron was present, the red portion of the fiber turned blue on addition of the base (Figure 4c).

All macroscopic images were taken with Nikon D50 and D200 digital cameras equipped with a Nikon 50mm prime lens and a Nikon 18-200mm zoom lens. Photomicrographs were taken using an Olympus DP70 digital camera coupled to an Olympus BH-2 PLM.

RESULTS AND DISCUSSION

Sample 1

Sample 1 was an entire, used baking dish with a form label including the word "Pyrex®" molded into the pan (Figure 4).

It was prepared for refractive index measurement and gave a value of 1.474 ± 0.001 corrected for 25°C at the sodium D line. The refractive index of the glass from this sample is too low in index for soda lime glass and indicates a borosilicate glass (e.g., Corning code 7740).

Sample 1 was prepared for analysis by SEM/EDS to determine its qualitative elemental composition. Figure 5 is the SEM/EDS spectrum taken from this

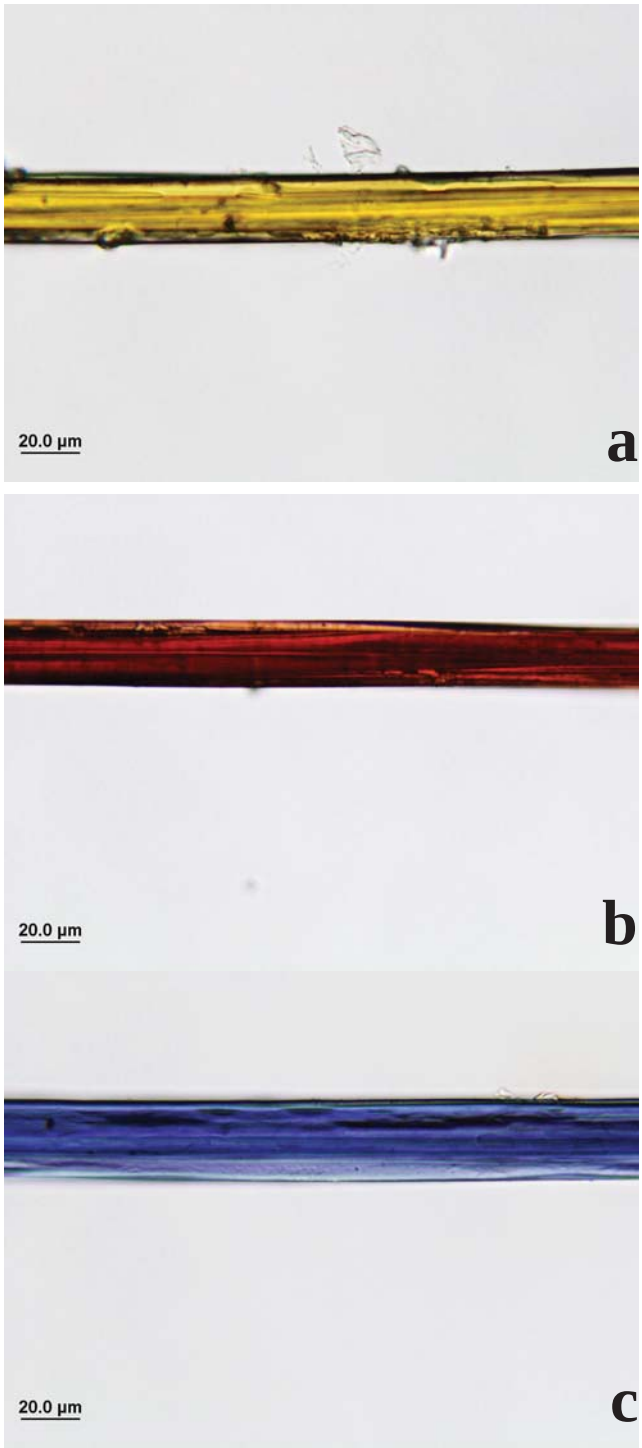

Figure 3: a) Plane polarized light image of yellow turmeric fiber, b) plane polarized light image of turmeric fiber after being submerged in test drop and showing red color, indicating possible presence of boron, plane polarized light of fiber in "b" after being treated with 1% sodium hydroxide, c) turning previously red fiber blue as confirmation of the presence of boron.

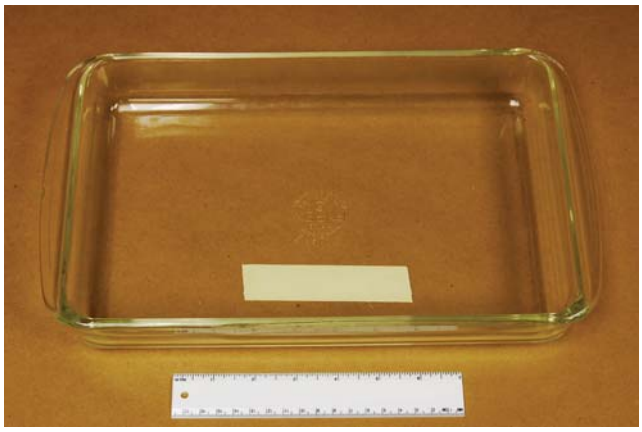

Figure 4: Pyrex baking pan as received for analysis (Sample 1).

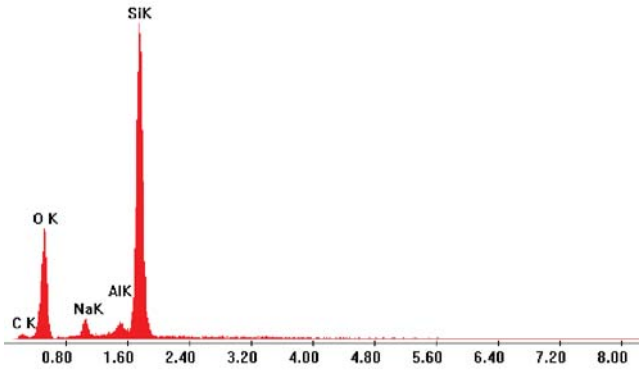

Figure 5: SEM/EDS spectrum showing a composition of primarily oxygen, sodium, aluminum, and silicon (Sample 1).

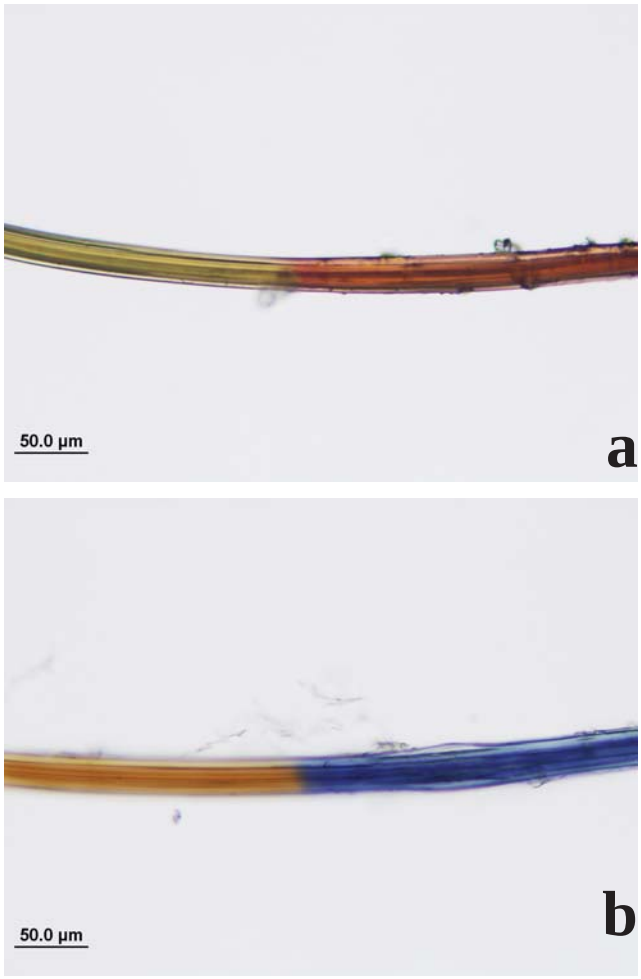

Figure 6: Chemical test for boron (Sample 1). a) Plane polarized light image of the test fiber before addition of sodium hydroxide solution, b) same test fiber after addition of sodium hydroxide solution.

sample. It confirms the refractive index indication that the sample is not composed of soda lime glass. Soda lime glass typically contains sodium (Na) and calcium (Ca) in addition to silicon (Si) and oxygen (O) (5). Sample 1 contains no detectable calcium.

The SEM/EDS only detects elements heavier than carbon (atomic number = 6), so a sensitive chemical test for boron (atomic number = 5) was performed to determine its presence or absence in this sample.

Glass from this sample was prepared for microchemical analysis by freeing and converting boron in the glass to boric acid. The fiber was wetted in the test solution, dried, and viewed under the microscope (Figure 6a). A sodium hydroxide solution was then added to the fiber, which was viewed again under the microscope (Figure 6b). The test confirmed that this sample was positive for boric acid; the fiber developed a red color before and a blue color after addition of the sodium hydroxide. Sample 1 was positive for boron.

Sample 2

Sample 2 was a square-cut piece of glass (Figure 7). Its refractive index was measured to be 1.515 ± 0.001 corrected for 25°C at the sodium D line. This refractive index indicates a soda lime glass.

Sample 2 was then prepared for analysis by SEM/EDS and the spectrum obtained confirms that the sample is a soda lime glass (Figure 8).

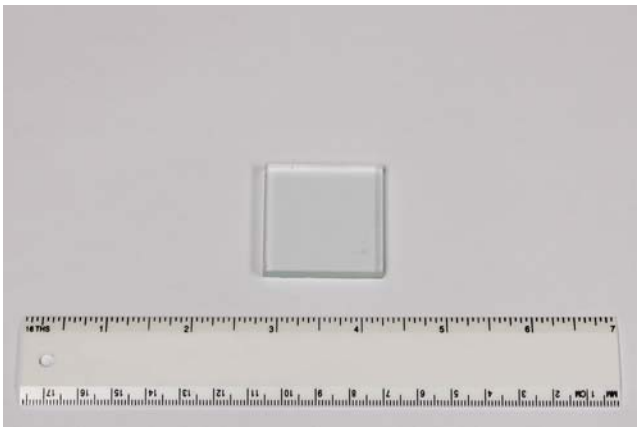

Figure 7: Square-cut piece of glass as received for analysis (Sample 2).

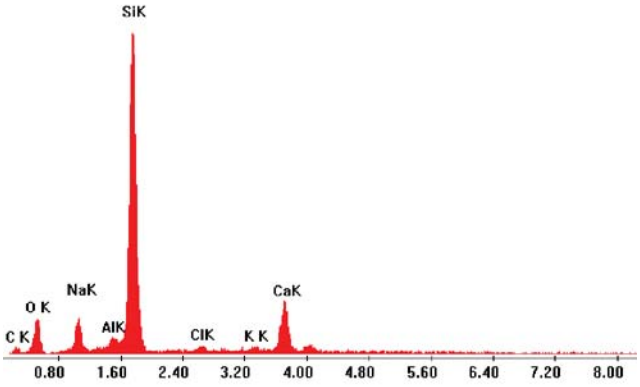

Figure 8: SEM/EDS spectrum showing a composition of primarily oxygen, sodium, aluminum, silicon, chlorine, and calcium (Sample 2).

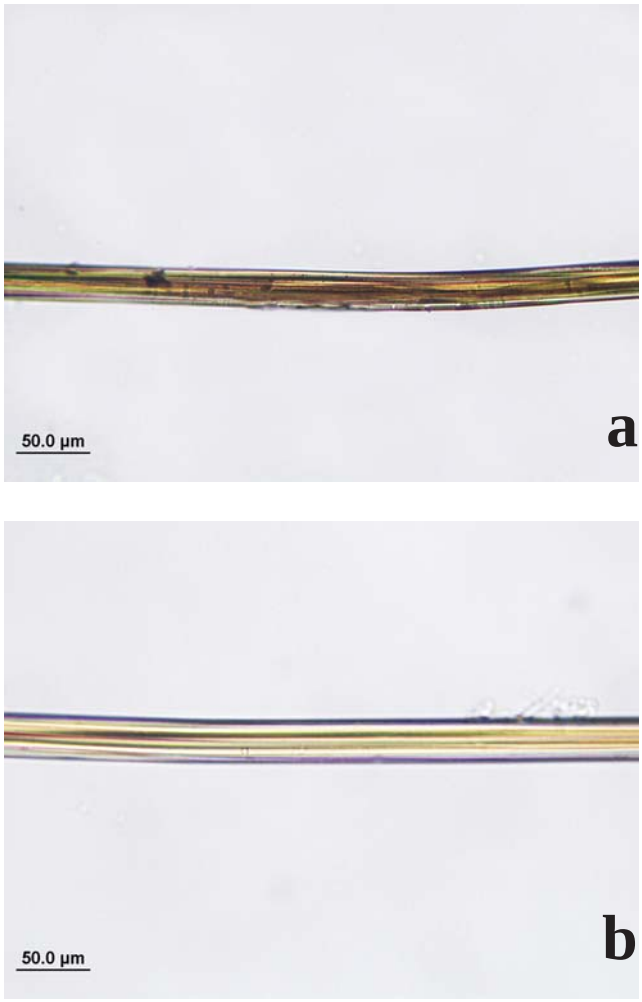

Figure 9: Chemical test for boron (Sample 2). a) Plane polarized light image of the test fiber before addition of sodium hydroxide solution, b) same test fiber after addition of sodium hydroxide solution.

The microchemical test for boron was also performed on Sample 2. Figure 9 shows the results of the test confirming that this sample was negative for boric acid; the fiber developed no red color before and no blue color after addition of the sodium hydroxide. Sample 2 was negative for boron.

Sample 3

Sample 3 consisted of three fragments of clear, colorless broken glass (Figure 10). Refractive index measurement gave a value of 1.517 ± 0.001 corrected for 25°C at the sodium D line. This refractive index indicates a soda lime glass.

The SEM/EDS analysis of Sample 3 confirms that the sample is a soda lime glass (Figure 11).

As with Sample 2, the microchemical test for boron confirmed that this sample was negative for boric acid; the fiber developed no red color before and no blue color after addition of the sodium hydroxide (Figure 12). Sample 3 was negative for boron.

Figure 10: Broken glass fragments as received for analysis (Sample 3).

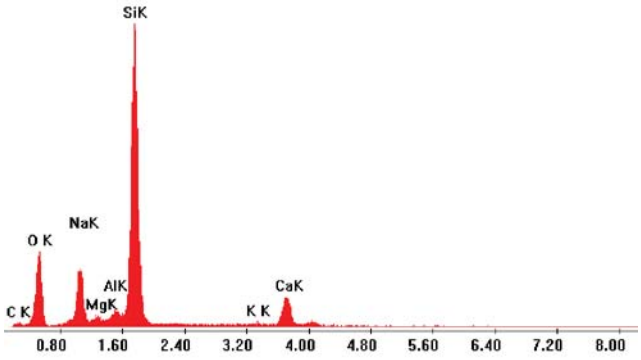

Figure 11: SEM/EDS spectrum showing a composition of primarily oxygen, sodium, magnesium, aluminum, silicon, and calcium (Sample 3).

CONCLUSIONS

The refractive index and composition of the three samples were not identical. Sample 1 was a borosilicate glass; it had a much lower refractive index than soda lime glass, its chemical composition was not like that of soda lime glass (e.g., no calcium), and it tested positive for boron. Samples 2 and 3 were soda lime glass with slightly different refractive indices as well as chemical compositions, and both tested negative for boron. Table 1 contains a summary of the results for the analysis of the three samples analyzed.

The analysis suggests that original Pyrex kitchenware (pre-1998) is borosilicate glass, whereas new Pyrex kitchenware (post-1998) is soda lime glass.

As a side note, we were contacted almost a year later to analyze another Pyrex baking dish that was purchased from Europe (ARC International) a week prior. It turned out to be a borosilicate glass. This information is also openly stated on the ARC International Web site under the heading, "What is Pyrex glass made of? (6)."

ACKNOWLEDGMENTS

I would like to thank Dr. Gary Laughlin for his guidance and assistance with this project as well as Skip Palenik for his help with the boron microchemical test.

TABLE 1

Sample #	Refractive Index	Qualitative Elemental Composition	Chemical Test for Boron
1	1.474	O, Na, Al, Si	Positive
2	1.515	O, Na, Al, Si, Cl, Ca	Negative
3	1.517	O, Na, Mg, Al, Si, Ca	Negative

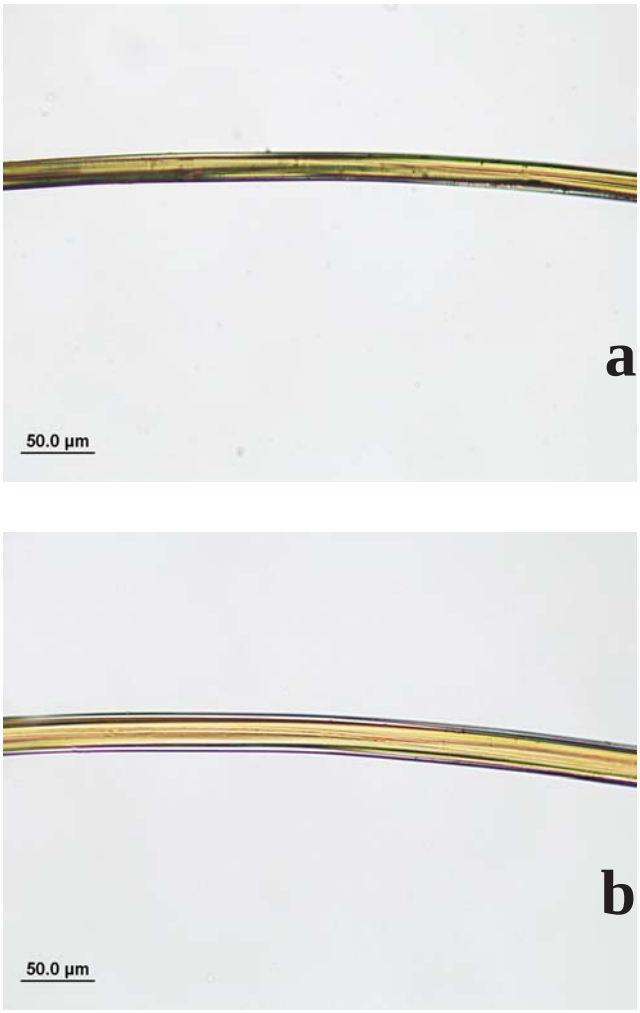

Figure 12: Chemical test for boron (Sample 3). a) Plane polarized light image of the test fiber before addition of sodium hydroxide solution, b) same test fiber after addition of sodium hydroxide solution.

REFERENCES

1. Chamot, E., Mason, C. *Handbook of Chemical Microscopy, Volume II*; republished by McCrone Research Institute, Chicago, Illinois, 1989.

2. Palenik, S., Microtrace LLC, personal communication, 2006.

3. McCrone, W.C., Delly, J.G., and Palenik, S. *The Particle Atlas*, *Edition Two, Volume V*; Ann Arbor Science, Ann Arbor, Michigan, 1973.

4. Manufacturing History. World Kitchen, LLC. http://www.pyrexware.com/thetruthaboutpyrex/manu.htm. Accessed November 2006.

5. Koons, D., Buscaglia, J., Bottrell, M., Miller, E. T. Forensic glass comparison. In: Saferstein, R. *Forensic Science Handbook*. Pearson Education Inc., Upper Saddle River, New Jersey, pp. 161-213, 2002.

6. Glass Ovenware. Pyrex. http://www.arc-international-cookware.com/en_Glass_Ovenware.html. Accessed November 2007.