It’s Been 25 Years  By Irene Brierley

Dear Friends, yet another exciting year is behind us. 2013 was successful for PIKE and we hope for all of you too. It was also a year of personal changes for me. It was the year I retired from daily operations at PIKE, and concentrated on spending more time with my family. My two daughters and their families live in the Madison area and I have been blessed with three beautiful grandchildren – Jacob, 8, Zander, 5, and Kemba, 2. And another one to join us in March of this year!!!!! I am now able to be a constant in their lives and time spent with them is precious. I am also planning to visit family in England more regularly. Of course my love and commitment to my PIKE family and customers is still as strong as ever and I plan to still be involved in long-term company plans and growth. This decision was made a little easier for me because Elizabeth joined us six years ago and has been gradually working to make me redundant!!!!!

Liz (still Elizabeth to me) is my youngest daughter and has been part of PIKE since its creation. She started by helping her dad assemble accessories when she was 9, and has always felt a strong connection to the Company he loved. After her college years out in Boston at B.U. and Northeastern, she returned to Madison to further her education in Business and came to take her place at PIKE. Over the last few years we have worked together with the PIKE team to bring exciting new technology and growth whilst maintaining the original PIKE goal of employee appreciation, exceptional customer support and good quality products. I know that with her taking the title of Vice President this will continue. I cannot possibly give enough thanks to those of you who have given me so much love and support during PIKE’s journey. After Phil passed away in 1999, we were a small group – but we were a group dedicated to PIKE’s success. Loyal customers are a treasure and we certainly have them!!!! It would be impossible for me to finish without saying thank you to a special person. Kent Gundlach, PIKE’s other Vice President, took the responsibility of our Engineering Department fifteen years ago and has been instrumental in PIKE’s growth. I am grateful for his hard work, and especially for the loyalty he has shown to PIKE and to me personally.

2014 will mark a milestone year for PIKE, as we celebrate 25 years of business. In the innovation and “new ideas” area, we are continuing expansion of our engineering and manufacturing capabilities with the addition of new state-of-the-art optical component equipment and metrology tools. This will allow us to grow our accessory portfolio, improve product quality and engage in new and unique designs. Therefore, please stay tuned for new products and new developments. I certainly will, as I am confident and excited to see PIKE’s future. Thank you for a wonderful 25 years and good luck to you all.  Irene

Liz Brierley, Vice President, PIKE Technologies, Inc.

The Frontline

With last year’s increase in business and the anticipated growth in 2014, we decided to strengthen the customer-facing part of our organization. Moving forward, Sarah Henke and Kristina Zins will be responsible for the daily management of your inquiries, quotations and order entry. Although most of you already know them from your previous contacts with PIKE (Sarah has been a long time employee of PIKE and Kristina joined us over two years ago), this announcement clarifies their new roles and should allow you to put their names and faces together. Sarah and Kristina bring enthusiasm, dedication and wealth of product and logistical experience to the table. Together they form a powerful, customer-oriented team. Please congratulate Sarah and Kristina on this new assignment and do not hesitate to contact either one with your questions.
Postage Stamp Analysis with MIRacle, Single Reflection ATR

Composition of Ink and Paper of the 1861 One-Cent Benjamin Franklin Stamp

By Harry G. Brittain, Ph.D., FRSC

The onset of the American Civil War caused the Federal government to demonetize all supplies of existing postage stamps (primarily from the series of 1851 and 1857), and to issue a set of newly designed stamps to be used in the Union states. In 1861, the Post Office Department awarded a contract to the National Bank Note Company, and the new stamps were issued on August 17, 1861. One of the stamps in this series was a one-cent stamp bearing the image of Benjamin Franklin (identified to collectors as Scott-63) (Figure 1).

Since not much is known about the composition of inks used to print stamps in the 19th century, infrared absorption spectroscopy coupled with attenuated total reflectance sampling (ATR) was used to study the stamp surfaces in an attempt to identify the major components in the printing ink. Since 19th century stamps have been the focus of many counterfeits, such analytical information would be highly valuable in establishing the genuineness of a given stamp.

Experimental Details
Fourier-transform infrared absorption spectra were obtained at a resolution of 4 cm⁻¹ and 40 scans. The data were acquired using the ATR sampling mode, where the samples were clamped against the zinc selenide (ZnSe) crystal of a PIKE MIRacle™ single reflection horizontal ATR accessory (Figure 2).

Study of the Paper Used in Printing of the US-63 Stamps
In the first half of the 19th century, linen and cotton rags were the source of papermaking fibers. Rags were cleaned, cut into small pieces, and heated in kettles with chemicals that destroyed any coloring. This process also rendered the pieces into pulverized fibers that consisted entirely of cellulose. A very similar process is used today to produce microcrystalline cellulose (MCC), which is extensively used as a non-active ingredient in pharmaceutical tablet formulations. To make MCC, one starts with purified cotton and through chemical treatment and heating to eventually yield a dry powdered form of cellulose.

Figure 3 contrasts the FTIR spectrum of pharmaceutical grade MCC with the FTIR spectrum of the unprinted region between the stamps of Figure 1. The spectra are dominated by the broad band at 1025 cm⁻¹ (due to the overlapping bending mode vibrations of the cellulosic hydroxyl vibrational modes), the broad absorbance at 3330 cm⁻¹ (due to the stretching mode vibrations of the cellulosic hydroxyl groups), and the weaker feature at 2900 cm⁻¹ (due to the stretching mode vibrations of the cellulosic hydrocarbon groups).

Since MCC is a good model compound for the paper of the Scott-63 stamps, one can simply blend a reference compound into MCC and compare the resulting FTIR spectrum with that of the FTIR spectrum obtained from the stamp surface. In this manner, the identity of the 1648 cm⁻¹ band in the spectrum of the stamp paper is shown in Figure 3 as being due to the presence of rosin in the paper.

Figure 1. Scott-63 stamp (right).
Figure 2. Experimental configuration used to obtain FTIR spectra (above).
Figure 3. Spectra of MCC, 10% physical blend of powdered cherry rosin in MCC, and non-printed paper region of the Scott-63 strip. The position of the diagnostic rosin peak is marked.
Studies of the Inked Scott-63 Stamp Surfaces

While the exact details of ink compositions that were used to print postage stamps of the 19th century are not absolutely known, a sufficient body of knowledge does exist and a general discussion of the inks used in the intaglio printing of that time is available. Mineral pigments were used almost exclusively, and were typically mixed with a drying oil (usually linseed oil), a surfactant (typically a soft soap), and a solvent. This mixture was homogenized through the use of a ball-milling process to produce a colloidal dispersion of ink pigments in the vehicle.

Figure 4 contains the FTIR spectrum of the blue ink portion on the face of the stamp in Figure 1, along with the FTIR spectrum obtained from the unprinted paper area. It is evident from this figure that the ink used to print the stamps contained several absorption bands not present in the FTIR spectrum of the paper. The origin of the peak observed at an energy of approximately 2090 cm⁻¹ (due to the symmetric stretching mode of a cyano –CN group) is extremely easy to identify (Figure 4) as being due to the presence of the blue pigment Prussian blue. This compound has a chemical formula which can be written as Fe₅K₂[Fe(CN)₆]₅, and is known to exhibit an intense blue color. Similarly, the peaks observed at 872 and 1410 cm⁻¹ were assigned to the presence of calcium carbonate (Figure 4) in the ink layer on the stamp surface.

While the presence of these components was easily established, the peaks at 1396, 1456, and 1537 cm⁻¹ were not readily identifiable as a known compound. Initial guidance to the identity of these FTIR peaks was obtained through the use of X-ray fluorescence analysis. This study demonstrated that among other elements, the surface of the Scott-63 stamp contained large amounts of a zinc compound, which in turn suggested that the ink formulators used zinc oxide as a whitener. However, the situation must be more complicated than that, since zinc oxide itself does not exhibit any significant absorption bands in its FTIR spectrum.

Since zinc oxide is a basic metal oxide, it would tend to react with surfactants in the ink composition to produce a metal soap product. For example, basic lead oxide has been shown to react with components in linseed oil to yield a lead soap product. This suggested that the zinc oxide component in the ink might undergo a similar reaction with the soap in the oil component of the ink formulation. To evaluate this possibility, zinc soap was synthesized by the reaction of hydrated zinc oxide with a water dispersion of Ivory soap. The resulting product was blended into microcrystalline cellulose, and the FTIR spectrum of this blend is shown in Figure 4. It is very clear from the figure that the peaks at 1396, 1456, and 1537 cm⁻¹ in the FTIR spectrum of the Scott-63 pair have corresponding peaks in the FTIR spectrum of zinc soap. In addition, the FTIR spectrum of the zinc soap blend also exhibits strong hydrocarbon absorption bands at the same 2849 and 2916 cm⁻¹ as observed in the FTIR spectrum of the Scott-63 pair.

Conclusion

The forensic analysis of one-cent Benjamin Franklin stamps of the 1861 series has revealed that:

- The paper on which the Scott-63 stamps were printed consisted primarily of cellulose that would have been derived from cotton fibers, and contained rosin as a sizing agent.
- The blue pigment in the ink used to print the images on the stamps consisted solely of Prussian blue. The intense blue color of this pigment was softened by the inclusion of two white pigments; primarily zinc oxide, but with a small amount of calcium carbonate.
- The surfactant in the processing oil reacted with some of the zinc oxide, producing a zinc soap product.

References

7. The X-ray Fluorescence Data were Obtained by Thomas M. Lera, Winton M. Blount Chair in Research, at the Smithsonian National Postal Museum.
Sealed Clamp for MIRacle ATR

We just introduced a Sealed Sample Chamber for the MIRacle Single Reflection ATR accessory. The high pressure Sealed Sample Chamber press attaches to MIRacle diamond, ZnSe or Ge crystal plates allowing the complete assembly to be moved from the spectrometer to a protective environment for sample loading and handling. Typical applications include studies of toxic or chemically aggressive solids and powders.

The chamber is made of stainless steel and is sealed against the crystal plate with a chemically resistant O-ring. The chamber contains an internal, spring-loaded anvil that compresses the sample against the ATR crystal at a pre-set, clutch-controlled setting.

In the standard configuration, the clamp is equipped with the Sealed Chamber attachment shown at right.

The clamp can be also used with other MIRacle sampling attachments – including flat, swivel and concave tips, flow-through, and liquid retainer/volatiles cover set.

For running toxic or sensitive samples, move the clamp assembly into the safe environment, raise the sealed chamber and place your sample on top of the ATR crystal. Lower the sealed chamber and keep turning the clamp pressure knob until three clicks. Move the loaded assembly back and attach it to the MIRacle optical module for measurement.

PIKE Events Calendar

Pittcon 2014, Booth 1344
March 2–6, Chicago, IL

ACS Spring 2014, Booth 523
March 16–20, Dallas, TX

Analytica, Hall A2 Booth 424
April 1–4, Munich, Germany

ACS Fall 2014, Booth 325
August 10–14, San Francisco, CA

Please stop and see us at one of these locations!

New Challenge – New Kindle Fire

Answer this question correctly to be entered into a drawing for a Kindle Fire™ Tablet...

What can be done to convert this polymer spectrum collected with a 30 degree specular reflection accessory (a) to absorption like spectrum (b)?

Please submit your answer through the contest link on www.piketech.com or via e-mail at info@piketech.com. Entries must be received by 5/31/2014.

©2014 PIKE Technologies, Inc. Kindle Fire is a trademark of Amazon.com, Inc. or its affiliates. All other trademarks are the property of PIKE Technologies.

Previous Winner
Rebecca Michelsen, Ashland, VA