Analysis of Postage Stamps by Proton-Induced X-Ray Emission Spectrometry

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ABSTRACT. Subtle variations in ink color and paper of postage stamps can affect their value and desirability to collectors and investors. Proton-induced X-ray emission spectrometry (PIXE) is a generally nondestructive forensic analysis tool that reveals the concentrations of chemical elements in a sample. PIXE has been used for more than 30 years, sometimes in conjunction with other analytical methods, to differentiate philatelic printing techniques; to quantify differences between different shades, varieties, and reprints of stamps; and to separate genuine stamps from forgeries. This paper reviews the history of philatelic paper and ink analysis by PIXE through a focus on case studies from the 1980s to the present. For example, PIXE has been used to show distinct chemical signatures between genuine stamps from China, Czechoslovakia, Eastern Silesia, South Africa, South-West Africa, and Taiwan and forgeries thereof. PIXE analysis showed how variations in ink chemistry control color shades of stamps from Mexico and reveal the origin (country of printing) of Iranian stamps, suggesting early (pre-twentieth century) utilization of organic inks on European-printed stamps from both nations. The technique was utilized to determine variations in paper types on stamps from China and Taiwan and how original People’s Republic of China stamps from the early 1950s can be separated from modern reprints. The paper concludes with a PIXE investigation of Mexico’s 1895–1898 “Multitas” stamps. The analysis reveals that unlike previous Mexican issues, the Multitas were printed with organic-based inks on largely clay-free papers. Differing amounts and/or formulations of metal (zinc, lead, and titanium) oxide whiteners apparently controlled the present-day color shade of the stamps, either directly or indirectly, with bluish inks having relatively more zinc and lead and greenish inks having relatively more titanium.

INTRODUCTION

Since their introduction in 1840, postage stamps have been acquired as collectibles and investments and studied to reveal information on the history of printing and the chemical and material properties of papers and inks. Potentially subtle variations in ink and/or paper of stamps with otherwise identical designs may affect their value and desirability to philatelists and investors or reveal them to be forgeries or reprints. Because of its ability to nondestructively reveal elemental compositions, proton-induced X-ray emission spectrometry (PIXE) has long been an applicable technique for analysis of a variety of historical inks and papers (Cahill et al., 1981; Johanson, 1989; Demortier, 1991; Hassanazadeh et al., 1999) and has been used for analysis of postage stamps from the technique’s early days to the present (e.g., Cahill et al., 1981; Malmqvist, 1986; Almquist, 1985, 1986, 1988; Johanson et al., 1986; Hall and Lee, 1990; Johanson, 1992; Cheng et al., 1996, 1998; Pillay, 2000; Gill, 2007; Oliai et al., 2009; Voltr and Nejedly, 2012).
PROTON-INDUCED X-RAY EMISSION SPECTROMETRY

Proton-induced X-ray emission spectrometry was developed in the 1970s as a multielemental analysis technique that is generally nondestructive; requires minimal or no sample preparation; and is quantitative, fast, and sensitive (Pillay, 2000). It is conceptually similar to X-ray fluorescence spectrometry (XRF), which has also been used for analysis of stamps (Preiss and Robie, 1982; Cesareo and Brunetti, 2008; Jelovica Badovinac et al., 2010), but with PIXE a beam of protons, generally with energy of millions of electron volts (MeV), is used to bombard a sample. A particle accelerator, usually a Van de Graaff accelerator, cyclotron, or similar machine, is used to create the proton beam; thus, PIXE facilities generally exist only at specialized academic or national nuclear laboratories (Johanson, 1992) and are less ubiquitous and much more costly than XRF. The high-energy proton beam ionizes some of the atoms in the sample. Some of the electrons in the inner shells orbiting the sample’s atomic nuclei are knocked away, creating a “hole.” In response, other electrons “drop down” from outer orbitals of the atom to “fill the hole” and take the place of the ejected electrons. In the process, X-rays with a constant and unique energy for each such transition (characteristic X-rays) will be emitted (Johanson, 1989). Each chemical element will typically have several unique, characteristic X-ray emission energies. This discovery, made by Moseley 100 years ago, led him to suggest that emitted X-ray spectra could be used for chemical analysis (Moseley, 1913). These X-rays are measured by a detector and converted to electronic signals, and software is used to calculate the concentrations of the individual elements within the analyzed part of the sample from the number of X-rays detected with each characteristic energy. The higher energy of the proton beam used in PIXE, as compared to the X-ray beam in XRF or electron beam from an electron microprobe, allows for higher sensitivity than these other techniques, detecting smaller concentrations of a material’s constituent elements more quickly.

Although all detectable elements are quantified simultaneously, the PIXE technique allows detection only of elements sodium and heavier, so the lightest elements such as carbon and oxygen cannot be analyzed directly by PIXE. PIXE successfully detects a number of relatively light elements such as aluminum (used as a sizing agent for some philatelic papers) (Lera et al., this volume) and magnesium (also found in some philatelic papers) (Almquist, 1988) not determined by X-ray fluorescence. The technique works best for quantifying elements with atomic numbers between 20 and 30 (Johanson, 1992), including calcium and the “transition metals” such as iron and zinc (common components of printing inks and papers), where concentrations as low as a part per million or less can be routinely detected (Johanson, 1992). Samples that are thin and flat, such as postage stamps, provide ideal surfaces to be analyzed by PIXE. The actual detection limit (minimum concentration of an element detectable in the sample) will depend on the energy of the beam and the length of time the sample is exposed to the beam. Samples may be analyzed in a vacuum; in a chamber filled with helium, argon, or another gas; or in air (the “external beam” technique often used for philatelic applications [Almquist, 1985; Hall and Lee, 1990; Voltr and Nejedly, 2012]). The proton beam generally penetrates only an ultrathin layer on the surface of the sample. It will not pass through the paper of a postage stamp or envelope, thus revealing the composition of the printing ink or, when the beam is placed on a portion of the stamp clear of the design, the paper. The size of the beam determines which part of the sample is analyzed at any given moment. Most reported philatelic analyses have been of spots on the stamp several millimeters in diameter (“proton millprobe”), although the beam can be focused down to pinpoint tiny areas on the order of a micrometer in size (“proton microprobe”) or moved across the sample to provide its overall composition. X-ray spectrometric techniques can also often be useful to determine what elements are not present in an ink or paper (beyond a minimum concentration), in addition to what elements are present (Allen and Lera, this volume). This is especially true for PIXE, with its relatively low minimum detection limits for many elements.

A potential disadvantage of PIXE is that it is purely an elemental analysis technique. PIXE cannot reveal the crystal structure or molecular species in which an element resides, which has led to controversies in its application for analysis of historical documents such as the titanium contained in the Vinland map (Skelton et al., 1995). With regard to the detection of fakes and forgeries, PIXE data can only show that the elemental concentrations and ratios are consistent with a known ink or paper, not always conclusively proving the (in)authenticity of a given item. PIXE relies on proton beams of high energy, typically in the millions of electron volts. This is an advantage in that it can detect lower concentrations of elements than other techniques such as X-ray fluorescence. However, it also represents a disadvantage in that prolonged exposure to the beam, longer than a few minutes, could burn or otherwise damage fragile materials such as paper (Cahill et al., 1986; Hall and Lee, 1990; Xeng et al., 1990; Demortier, 1991; Sánchez, 2006). Hall and Lee (1990) sandwiched the stamps they analyzed between thin layers of Kapton film and still observed some damage after five minutes of exposure to the proton beam. Voltr and Nejedly (2012) examined stamps by PIXE at reduced pressure in an argon atmosphere to decrease the potential radiation damage compared to analysis in a vacuum, while still allowing a quantitative analysis. When used carefully, however, PIXE is a nondestructive method of testing. The experiences of multiple investigations (as described throughout this paper) show that a thorough and ample PIXE analysis should be obtained within a few minutes of irradiation, which would not risk damage to the stamp.

Although PIXE has been used most widely in materials science, environmental studies, and biological and geological investigations, it was recognized early on as a powerful tool for historic and forensic analyses of inks and papers (Cahill et al., 1981; Thompson, 1983; Kusko, 1988), especially after it was...
used to reveal the printing methods and materials of the “Gutenberg Bible” (Schwab et al., 1983). Since then, reviews of this technique inevitably mention its application for analysis of historical printed documents (Johanson, 1989, 1992; Demortier, 1991; Pillay, 2000; Vijayan et al., 2003; Vodopivec et al., 2005).

**PHILATELIC APPLICATIONS OF PIXE: A REVIEW**

The first published report on the application of PIXE in philately was written by the late chemist and philatelist Herbert J. Almquist, appearing in the *American Philatelist* in 1985 (Almquist, 1985). Almquist worked with physicists and document experts at the Crocker Nuclear Laboratory of the University of California-Davis, utilizing PIXE to analyze variations in color from blue-green to dull green of ledger lines on the paper of the Mexican “Large Numerals” stamps printed in 1887 (Scott 3195–211) (Figure 1).

No significant variations were found in the elemental chemistry of the different colored lines, and Almquist (1985:242) concluded “the grey-green color is derived from the turquoise-green by aging and sulfurization over a period of many years.” The PIXE analyses did, however, shed light on the compositions of the paper and the ledger line ink, suggesting that clay and a calcium compound were added to the paper and that the ink was an “iron blue” compound with zinc oxide added as a whitener.

Additional analyses of the papers, as well as the ink used to print the Mexico 1-centavo green stamp (Scott 201) (Figure 2), were reported a year later in *Mexicana*, the quarterly journal of the Mexico-Elmhurst Philatelic Society International (Almquist, 1986). Almquist (1986:207) concluded that “the amounts of clay corresponding to these elements could be several percents of the paper,” that the green ink and the green lines on the paper were derived from different materials, that the green color of the ink could have been derived in part from green chromic oxide, and that zinc and lead oxides were used as whiteners in the inks. A final set of PIXE analyses reported by Almquist (1988) examined the compositions of the paper used for the “Numerals” issue and three other watermarked papers used for contemporary Mexican stamps (Scott Mexico watermark numbers 152–154). The presence of relatively high amounts of magnesium, silicon, and aluminum in the Numerals paper, but not the watermarked papers, suggested to Almquist that the ledger-lined papers of the Numerals were grass based with clay fillers, while the watermarked papers were derived from wood fiber.

The first reports in the scientific literature referring to PIXE analysis of stamps were published by Johansen et al. (1986) in a report also providing “proof of concept” of PIXE’s application to the forensic analysis of art objects, jewelry, and artifacts. Various examples of the Swedish “6-ore ringtyp” stamps issued in 1872 (Scott 20) (Figure 3), which exist in various shades of violet, lilac, and mauve, were analyzed in a Swedish nuclear research center, with the inked paper in the center of the stamp...
evaluated separately from the inkless edges of the stamps near the perforations.

The analysis of the edges provided an assumed contribution from the paper alone. The authors determined that the inks appeared to be made from iron-and-lead- or mercury-based pigments, with zinc oxide added as a whitener. Johansen et al. (1986:48) concluded that “different proportions of the pigments give rise to many nuances, towards red and blue, and a visual classification is not easy. An objective classification can be obtained by measuring the metal content and plotting the results in a suitable way.” In their summary, Johansen et al. (1986:45) reported “for one group of stamps clustering of the visually determined nuances occurred in a two-dimensional plot of lead versus zinc, while for another group a plot of mercury versus zinc showed no such clustering.”

Peisach (1986), a South African scientist, in the same journal issue, was the first investigator to apply PIXE to investigate differences between genuine stamps and forgeries. Known forgeries were compared with genuine stamps from the nineteenth-century South African Republic and South-West Africa, and on the basis of multielemental analytical data, the forgeries were shown to have different compositions from the genuine stamps.

Hall and Lee (1990) published the first scientific paper specifically limited to PIXE analysis of postage stamps, in which the different chemical compositions of paper and ink were compared. In their work, several stamps from the short-lived Taiwanese republic of 1895 (Stanley Gibbons numbers 4–6) (Jeffries, 2011) were irradiated in a 4.0 MeV external proton beam. Hall and Lee (1990:573) summarized their results by stating,

the paper and printing inks were compared between stamps made from different dies. The elemental analysis revealed that the 4 dies of stamps were printed on only 3 different types of paper. A majority of the elements observed in the stamps were printed on only 3 different types of paper. A majority of the elements observed in the stamps were due to contributions from the paper except for the red stamps which were due to cinnabar (HgS). In addition, the elemental concentrations were different in counterfeit stamps as compared to the genuine stamps.

PIXE was utilized in China by Cheng et al. (1996, 1998) to determine the ink composition of modern Chinese stamps and their reprints. They compared three stamps issued in 1950 by the People’s Republic of China (Scott 57–59) with their official reprints (Scott 57r–59r), in the same color, design, and printing technique, stated by the authors to have been issued in 1995 (Figure 4).

Although the colors are quite similar to the original issues, the 1995 reprints were shown to have much different elemental concentrations and/or compositions. For example, the brown stamp (Scott numbers 57 and 57r) contains lead and zinc in the original printing, which was not present in the 1995 reprints. The relative amount of calcium was eight times greater in the reprints than in the early printings. Cheng et al. (1998:900) concluded that “we still do not know the reason for modifying the printing ink for the new edition. Maybe the original type of printing ink was no longer available due to technical improvements. However, the PIXE method can be used to distinguish between the (original printing) and (reprints) of stamps.”

More recently, a group of Iranian scientists (Oliaiy et al., 2009) used (in conjunction with other techniques) a PIXE microscope (which uses a micrometer-scale proton beam) scanned over a square with dimensions of 2.5 mm on each side to analyze various areas of ink and paper on eighteen Iranian stamps issued between 1881 and 1915 and printed in Austria, France, the Netherlands, and Iran. The micro-PIXE was able to indicate the characteristic elements used for inks of different colors, as well as differences in the formulas for philatelic inks of the same colors utilized in different countries. Oliaiy et al. (2009) showed a titanium compound had been added to the paper used in the Austrian-printed stamps—iron and sulfur contents of green ink were different between the Austrian-printed and French-printed stamps. There were differences in chromium and lead levels in orange inks of the Iranian-printed and Netherlands-printed examples, as well as other differences in brown, black, grey, violet, and silver inks of stamps printed in different countries, suggesting (from lack of detected metal in some colors) that certain inks were organic in nature.

Voltr and Nejedly (2012) used PIXE to analyze two early spurious Czechoslovakian stamps with double overprints on Austrian stamps from 1919 depicting Emperor Franz Joseph (Figure 5) and a 1920 Eastern Silesia stamp (Scott 5) (Figure 6), an overprint on a 20-heller green Prague Castle stamp from Czechoslovakia, to determine if genuine overprints could be distinguished from forgeries. In this case, the two Czechoslovakian stamps carried one genuine and one forged overprint on the same stamp, while two examples of the 1920 Eastern Silesia stamp were analyzed, one each with genuine and forged overprints. Voltr and Nejedly (2012:238) reported, “In all cases,
we found barium as the most prominent element, with concentrations one or two orders higher than that of other elements” and also discovered “a specific and interesting variation of lead concentrations.”

In the Czechoslovakian double overprints, lead and iron levels “were several times higher on genuine overprint than on other places” (Voltr and Nejedly, 2012:238). For the Eastern Silesia stamp, there was a significant difference in the quantity of iron between the genuine and forged overprints, some variation in the concentrations of lead, and a much higher quantity of potassium in the genuine overprint. Voltr and Nejedly (2012:239) concluded,

We are proposing a hypothesis that the stamping dies used shortly after the World War I were made from a material different from the one later used for falsification and that traces of the material remained in the overprints. Falsifier aimed to emulate the properties of the ink but failed to match the composition of the stamping die. As another hypothesis, we attribute the difference in potassium to the distinct printing houses—stamps A and B were originally overprinted in the printing house A. Haase in Prague in 1919, and stamp C was overprinted in Unie Praha later on.

Even with careful precautions made in Voltr and Nejedly’s experimental setup, the stamps began to show radiation damage (discoloration of the irradiated areas) after analysis, forestalling any opportunity for further examination.

**CASE STUDY:**

**PIXE ANALYSIS OF COLOR VARIATIONS IN THE 15-CENTAVO MEXICAN “MULITAS” STAMPS**

The late philatelist Herbert J. Almquist had noted that color variations were known, ranging from bluish green (believed to be from the earlier printings) to greenish blue (believed to be from later printings), on the 15-centavo “Mulitas” stamp (so named because it depicts a stagecoach drawn by mules) of the Mail Transportation stamp set printed by Mexico from 1895 to 1898 (Scott numbers 251, 263, 275, and 286) (Figure 7) (Almquist, 1985).
Almquist expected that the ink used to print these stamps was the same one used to print the 1-centavo green Numeral stamp (Almquist, 1988) (Figure 2). Some investigations had been made of variations in the paper of the Mail Transportation stamps (Schimmer and Bulak, 1972) but not of their ink. PIXE was an ideal technique to determine whether these variations were due to chemical differences.

Approximately 100 “Mulitas” stamps for sale from commercial stamp dealers were examined. Four stamps, each displaying the most distinctly bluish and greenish color shades, were chosen for testing. Proton-induced X-ray emission analyses were performed at the Crocker Nuclear Laboratory at the University of California-Davis (UCD) with an external beam (in air) PIXE proton milliprobe, as had been done in Almquist’s analyses of the “Numerals” stamps (Almquist, 1985). This external beam PIXE analytical setup had been used for a large number of archeometric analyses of papers and inks, including Gutenberg’s Bibles (Schwab et al., 1983) and the Vinland map (Cahill et al., 1987), as well as the philatelic works of Almquist (1985, 1986, 1988). The 193-cm isochronous cyclotron at UCD produced a beam of protons with energy of 4.5 MeV, which was taken into approximately 3 cm of air, passed into the stamps generating characteristic X-rays, and collected in a Faraday cup. The beam size was a rectangle 1.0 x 0.5 mm for these analyses and was aligned by a laser to irradiate specific spots on the sample for 100 seconds. Prior analyses of historical papers and inks analyzed in this laboratory under the same setup had not been reported to cause any noticeable damage to samples, and no discoloration or other observable damage to the stamps was caused by these analyses. A lithium-drifted silicon energy–dispersive detector (SiLi detector) was placed several centimeters from the sample, presenting a large solid angle for X-ray detection and measurement. X-rays from the sample were counted at rates of approximately 700–1500 per second for the samples analyzed here. A thin film multielemental standard material from the National Institute of Standards and Technology was also analyzed during the run to ensure that reported concentrations were accurate and traceable. A dark area of solid ink near the top frame of each stamp of the four greenish and bluish stamps (away from any cancellation mark) was analyzed, as well as a portion of the unprinted, uncanceled paper in the top margin of all eight stamps.

Only eight chemical elements were detected in the ink and paper of the stamps. Three elements were present at relatively high densities, exceeding a microgram per square centimeter: zinc, silicon, and sulfur. Five “trace” elements were detected at lower concentrations: lead, calcium, iron, titanium, and chlorine. Distinct differences in elemental composition between bluish and greenish inks of the “Mulitas” were noted, as were differences between the “Mulitas” inks and papers and those of the “numerals” analyzed by Almquist (1986, 1988) using the same machine and experimental setup.

Unlike the “Numerals” stamps (Figure 1) previously analyzed by Almquist (1985, 1986, 1988), which were apparently printed with potassium- and iron-based, chromium-bearing inks on papers with clay (magnesium, silicon, aluminum, and calcium) coatings or fillers, the absence or much lower concentrations of these elements in the “Mulitas” stamps suggests they may have been printed with organic-based inks on papers largely devoid of clay fillers. This and the presence of sulfur suggests that Mexican stamps could have been printed with aniline (sulfone-derived organic) pigments earlier than 1899, as had been previously thought (Schimmer and Bulak, 1972). The “Mulitas” paper is harder and stronger than the “Numerals” paper, which was easily torn, perhaps due to the latter’s clay- and calcium-based fillers. The “Mulitas” paper is noticeably lower in calcium than other eighteenth-century papers investigated in prior PIXE analyses (Cahill et al., 1981).

Oxides or hydroxides of zinc, lead, and/or titanium were often added to Mexican (Almquist, 1985, 1986) and other (Johanson et al., 1986; Hall and Lee, 1990; Voltz and Nejedly, 2012) postage stamp inks of the late nineteenth century as brighteners, lighteners, or extenders. The presence of these elements in the “Mulitas” inks, along with their relative scarcity in the paper, suggests they were modulated color shades of the “Mulitas” stamps, with bluish inks having more zinc and lead and less titanium and greenish inks having less zinc and lead and more titanium. These elements could have either directly influenced the shade of the ink as printed or secondarily protected against a postprinting color change of the stamps potentially caused by the sulfur in the inks.

CONCLUSIONS

Proton-induced X-ray emission spectrometry has been a useful technique for the analysis of philatelic inks and papers for more than 30 years. Although the machines required to perform a PIXE analysis are relatively few and not commercially available, when this method can be utilized, its ability to determine low concentrations of metals, for example, in organic-based inks and papers, keeps it relevant. PIXE analysis has shown that a widely used practice controlling color shades of nineteenth-century stamp inks was the addition of metal oxides as whiteners. In addition, the papers used to print stamps of that era from many entities contained clay mineral elements as fillers. PIXE also has shown utility as a forensic tool, through its ability to discriminate chemical differences between original, genuine stamps and forgeries or reprints.

The case study of the 1895–1898 Mexican “Mulitas” stamps indicated that they, unlike the previous Mexican issues, were printed with organic-based inks on largely clay-free papers. Mexican stamps appear to have been printed with organic inks several years earlier than previously believed. Differing amounts and/or formulations of metal (Zn, Pb, and Ti) oxide whiteners apparently controlled the present-day color shade of the stamps, either directly or indirectly, with bluish inks having relatively more Zn and Pb and greenish inks having relatively more Ti.

PIXE is one of many tools described at the First International Symposium on Analytical Methods in Philately that have
been used for philatelic analyses. Since PIXE is an elemental analysis technique, it may be combined with other techniques determining the actual chemical compounds or molecules for a more complete nondestructive analysis. In conjunction with PIXE or XRF elemental analyses, other methods that have been successfully applied to stamps, such as Fourier transform infrared spectroscopy (Poslusny and Daugherty, 1988; Ferrer and Vila, 2006), Raman spectroscopy (Castro et al., 2008; Jelovica Badovinac et al., 2010; Withnall et al., 2012), and/or X-ray diffraction (Foner and Adan, 1983; Liston, 2003; Vila et al., 2007; Lera et al., this volume; Brittain, this volume) should be able to discern the specific component compounds of the ink and paper.

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NOTES

1. When the Vinland map, supposedly a fifteenth-century map of the Earth, was analyzed by PIXE, titanium was found to be a component of the ink; other analytical techniques suggested that the titanium was in the form of the compound anatase, a material not synthesized in such a form until the twentieth century.

2. The Crocker Nuclear Laboratory at the University of California-Davis, one of the first institutes where PIXE was used for analysis of historical inks and papers, was named after University of California Regent William Henry Crocker (1861–1937), who not only funded one of the first cyclotrons in the university system but also was a philatelist and one of the 100 founding members of the Collectors Club of New York.

3. All references herein to “Scott” refer to the catalogue numbers and, where specified, watermark numbers, assigned in the Scott Standard Postage Stamp Catalogues, published every year by Scott Publishing Co., Sidney, Ohio.

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