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The Forensic Characterization of Polyethylene Films By Elemental Analysis Using Total-Reflection X-ray Fluorescence (TXRF) Spectrometry

by

JoAnn Buscaglia

A dissertation submitted to the Graduate Faculty in Criminal Justice in partial fulfillment of the requirements for the degree of Doctor of Philosophy, The City University of New York.

1999

This manuscript has been read and accepted for the Graduate Faculty in Criminal Justice in satisfaction of the dissertation requirement for the degree of Doctor of Philosophy.

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Abstract

The Forensic Characterization of Polyethylene Films

By Elemental Analysis Using Total-Reflection X-ray Fluorescence (TXRF) Spectrometry

by

JoAnn Buscaglia

Advisor: Professor Peter R. De Forest

Plastic trash bags are encountered as physical evidentiary materials in the investigation and adjudication of homicides, narcotics trafficking, and dumping of hazardous waste. Currently, forensic examinations of plastic bags consist of comparisons of physical patterns arising from random processes during manufacture. The discrimination capability of this approach is limited by sample size, opacity, thermal or mechanical distortion of the bag, and the need for consecutively or nearly consecutively manufactured bags for comparison. Conversely, the inorganic elemental composition of a polymeric material is stable, even after thermal or physical alteration, or exposure to harsh environmental conditions. Further, there should be limited variation in elemental concentrations during a production run. Differences in elemental profiles among plastic bags are expected, because a variety of inorganic constituents are introduced intentionally and accidentally into plastic bags during the manufacturing process.

In this study, the concentrations of selected elements in commercially available polyethylene trash bags were determined using total-reflection X-ray fluorescence (TXRF) spectrometry. Samples of polyethylene bags were cut, cleaned, and weighed,

followed by removal of the polymer matrix by oxygen plasma ashing. The ash residue was quantitatively transferred to the TXRF quartz sample support and an internal standard was added. Removal of the polymer matrix yielded better detection limits for all elements of interest than a solid sample preparation method.

Replicate samples from individual bags from three manufacturers were analyzed in order to determine the variability in element concentrations within each bag. The compositional variation was also determined within and among individual bags, boxes, production lots, and manufacturers. Of the 18 elements readily determinable in most specimens, titanium (Ti), iron (Fe), zinc (Zn), calcium (Ca), strontium (Sr) and barium (Ba) are the most useful for discrimination among manufacturing sources. These element concentrations vary greatly among manufacturers and production runs, but are consistent within individual bags and among bags from a single box. Results of this research indicate that bags produced from the same manufacturer and product line in different plants, and at different times in the same plant, are analytically distinguishable.

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CHAPTER I. INTRODUCTION

This research project concerns the application of a relatively new analytical technique, total-reflection X-ray fluorescence (TXRF) spectrometry, to the forensic characterization of plastic film trash bags. TXRF spectrometry, a micro-analytical technique with good accuracy and precision, offers the potential for enhancing classification and discrimination of manufactured plastic bags using their trace elemental profiles. The ability to demonstrate an association of two compared plastic bags based on their elemental compositions is likely to be highly significant. This project develops and evaluates a method to apply TXRF technology to plastic bags, in an effort to improve the ability of the forensic scientist to compare polyethylene evidentiary materials in the forensic science laboratory.

A. Trace Evidence Considerations

Forensic examinations of physical evidentiary materials typically involve the comparison between a crime scene sample and a sample associated with a subject. These comparisons should employ highly discriminating analytical techniques, ideally, capable of detecting any significant difference between the crime scene and comparison samples, if one exists. When no difference is detected, the known or comparison sample can not be excluded as the source of the crime scene sample. The significance of this association is a function of the discrimination potential of the analytical method employed. However, with the exception of physical matches, pattern comparisons and possibly DNA typing of some biological materials, it is more the rarity than the norm that individualization, or the association of a material with its source to the exclusion of all others, is attained with

physical evidence. Further, when the products are mass-produced with strict quality control procedures in place, as is the situation with polyethylene bags, differences may be subtle and, consequently, extremely difficult to detect. However, as a general proposition, by utilizing analytical procedures with high sensitivity and discrimination capability, an association can be attributed increased significance when the two samples are determined to be indistinguishable.

Trace evidence examiners are presented with a diversity of manufactured materials for comparison and analysis. Mass-produced plastic articles are ubiquitous, as shown by the worldwide usage of 215.4 billion pounds of plastic in 1996. Of this, polyethylene ranked highest as the world's most widely used commodity plastic, accounting for 41% of the usage that year (Reisch, 1997). As a result of their large production volume and the frequent use as packaging materials in criminal activities including drug-related offenses, homicides, and illegal dumping activities, polyethylene bags and wraps are commonly encountered as physical evidence. For example, household polyethylene trash bags are frequently used as a means to conceal or dispose of a victim's body, clothing, or other personal effects in homicide cases. A variety of plastic bags and wraps is also used for wrapping both small and large quantities of illegal drugs for transportation, distribution and sale. Polyethylene trash bags are also found in cases of improper disposal of hazardous materials. Regardless of the offense, laboratory comparison of a plastic bag recovered from a crime scene with known-source bags, generally from a box of bags in a subject's possession or from a possibly related crime, can assist in both the investigative and adjudicative processes.

B. Manufacture of Polyethylene Bags

The interpretation of the results of a forensic comparison of plastic bags also requires knowledge of the manufacturing process. Blown film polyethylene bags are produced by blending organic and inorganic pigments, fillers, stabilizers and various reaction control additives into a melt of polyethylene. This melt is then extruded through an annular die to form a continuous cylinder of plastic film. Subsequently, the film thickness of the plastic tube and the circumference of the cylinder are adjusted by blower motors. After cooling, the tube is collapsed, flattened, and pulled through rollers, followed by sealing and cutting of the edges, heat sealing of the hem, and perforating, separating, and packaging of the bags. This abbreviated, general description of the process varies with manufacturer, size, shape, purpose and design of the product, including special handles and seals. Quality control processes are implemented in the manufacturing plants to minimize film defects and insure product uniformity, including film thickness and color, within specified limits.

C. Forensic Examinations of Polyethylene Bags

Forensic examinations of plastic bags commonly involve comparison of physical characteristics imparted during the manufacturing process, as well as the identification and comparison of the base polymer. In the case of polyethylene bags, the latter generally offers little discrimination among sources. On the other hand, physical manufacturing characteristics, such as patterns and pigment distributions, can be an excellent point of comparison when comparison is made between consecutively or nearly consecutively manufactured bags. The discrimination capability of the application of the manufacturing

characteristics to the forensic association of two or more bags is affected by the sample size, opacity, temporal production distance between compared bags, and thermal or mechanical distortion of the plastic material.

Compositional analysis can be performed for both organic and inorganic components. The determination of the major organic components of plastic materials will identify the particular polymer type, whereas minor organic components may be useful for source discrimination. A limitation to organic compositional analysis of polymers is that organic components may degrade with time and harsh environmental conditions. In contrast, the elemental composition of materials is less prone to change over time and therefore, a valid laboratory analysis may still be accomplished even if the samples have been physically or thermally altered or exposed to various environmental factors for a substantial period of time. However, the analysis of the inorganic constituents in polymeric materials has received little attention, despite the fact that classification and discrimination of many other manufactured products has been accomplished by measurement of their trace elemental profiles.

Inorganic additives, which generally account for less than 1% of the weight of the final product in plastics, are used to improve the processing and performance of plastics (Greek, 1988). Reaction control catalysts and processing additives, such as lubricants and stabilizers, are metal oxides, metal salts, and organometallic compounds. Stabilizers are added to minimize the degradation of the final product by air, light, and heat. Performance additives often contribute the largest amount of inorganic material to the plastic. These additives include fillers and reinforcements, such as clays and glass fibers, inorganic

pigments that impart color, flame retardants, and coupling agents. In addition to the inorganic materials that are intentionally added to plastics during fabrication, trace elements from the raw materials and contaminants from the manufacturing process may be incorporated into the finished product. Current recycling practices on the remixing of scrap plastic, although controlled by manufacturers' quality assurance (QA) policies, may also cause additional variations in the trace elemental profiles over time in the production run. As a result of the random nature of incorporation of these trace elements and limited manufacturing control over certain inorganic additives, the potential exists for enhanced classification and discrimination of polymeric materials using their trace elemental profiles. Unfortunately, this potential has not yet been fulfilled with regard to trace evidentiary materials because of sample size limitations and the destructive nature of some instrumental methods of analysis for the determination of element concentrations. With respect to polymers, the low level of inorganic constituents in a troublesome organic matrix further complicates the analysis.

D. Elemental Analysis of Polyethylene Bags

Recent advances in analytical instrumentation, such as introduction of total-reflection X-ray fluorescence (TXRF) spectrometry, suggest the potential for the elemental analysis of plastics. TXRF spectrometry has been shown to have diverse applications for the accurate quantitation of trace elements in a variety of materials of forensic interest. TXRF spectrometry is well suited for the microanalysis of trace evidence, as it allows for simultaneous multi-element determinations in microgram and sub-microgram samples with

instrument detection levels in the picogram range. Semi-quantitative analysis with minimal, nondestructive sample preparation is also possible using TXRF.

The application of a sensitive analytical technique such as TXRF to the investigation of the compositional variations within and among manufacturing batches of plastic bags could offer increased discrimination among these products. Ideally, individual manufacturing batches or limited segment of production runs could be differentiated. The identification of manufacturer and product line can generally be made based on physical parameters. As a result, methods such as infrared (IR) spectroscopy that do not discriminate at the production run level are not helpful. An association of two compared plastic bags based on their elemental profiles is likely to be highly significant, particularly when trace elements which are randomly incorporated into the material during its production are considered. In such cases, as the number of matching independent variables increases and, if a wide range of elemental compositions between products exists, the probability of a false association diminishes. Therefore, increased weight may be attributed to such evidence when it is presented in court.

CHAPTER II. REVIEW OF THE LITERATURE

A. Elemental Analysis of Physical Evidence

The elemental analysis of physical evidentiary materials has been performed with varying degrees of success for over 50 years. Throughout the last two decades, many articles have been published in the scientific literature regarding the application of a variety of instrumental methods to trace evidence samples and the increased discrimination potential that may be realized by the incorporation of elemental composition into an analytical scheme. Most of these studies used inorganic materials such as glass (Buscaglia, 1994; Koons *et al.*, 1991) and metals, including lead projectiles (Peele, 1991) and aluminum foil (Koons *et al.*, 1993). Fewer studies have been reported concerning the use of elemental composition to discriminate among sources of polymeric materials.

Classification and characterization of a material of unknown origin is facilitated through the use of elemental analysis. This capability was used in the forensic analysis of small glass fragments, where product-use classification is often possible by examination of the elemental profiles of the fragments (Hickman, 1981; Ryland, 1986; Koons *et al.*, 1988). However, the elemental data necessary for product-use classification differs from that for discrimination in the selection of elements for evaluation and the levels of accuracy and precision required. Elements useful for classification, or grouping, are those which occur at consistent concentrations within a product type. On the other hand, discrimination requires detection of very slight differences in element concentrations,

which demands both the use of elements with a wide concentration range within a product type and high precision of analytical measurements.

B. Forensic Examination of Polyethylene Products

Forensic examinations of polyethylene film products have been reported in the scientific literature for approximately 20 years. These examinations have included comparison of physical characteristics, organic analysis, and more recently, elemental analysis of the inorganic components of these products.

1. Physical Comparisons

To date, comparison of physical manufacturing characteristics has received the most attention from the forensic science community with regard to the discrimination among polyethylene bags. Crispino and De Forest (1978) first presented the individualization of polyethylene bags via comparisons of pigment inhomogeneities. Von Bremen and Blunt (1983) subsequently reported on the ability to discriminate among sources of polyethylene garbage and sandwich bags through the physical comparison of the cut ends of sequentially produced bags. Similarly, Stone (1986) used physical markings such as cut ends, perforations and marks imparted by home heat sealers to positively link questioned sealed plastic films to particular devices and manufacturing sources.

In 1992, Stanko and Attenberger presented a concise description of the manufacturing processes that contribute to the physical characteristics used in the comparison of polyethylene bags, along with some guidance on the interpretative value of such markings. Class characteristics considered during the preliminary comparison

include the color, size, shape, embossed code, and design and construction of handles and ties, and the location and construction of the seams and the hem. Additionally, physical markings imparted during manufacture, including pigment bands and die lines (extrusion striae), are useful in the comparison of consecutive bags by the physical alignment of these markings. Pigment bands, which are formed by the inadequate mixing of the dyes and pigments in the polymer melt, generally run in the direction of film production. Die lines are caused by a contaminated or damaged die. Individual characteristics, which include film imperfections, such as fisheyes and arrowheads, may occur as a result of random particulate contamination entering the film as it is being blown. Examination of the heat seal of the hem may also yield individual characteristics, including the incorporation of foreign particles within the heat seal and patterns caused by mechanical imperfections such as nicks on the metal sealing bars. These individual characteristics are useful in establishing a common origin of two consecutively produced polyethylene bags provided that there are a sufficient number of random imperfections and that they run across both bags.

More recently, Ryland (1999) presented data from a case study involving the comparison of extrusion die striae on nonconsecutively produced plastic trash bags. By studying the manufacturing process of the product encountered in a specific case, Ryland calculated the longest persistence of the predominant extrusion die striae and the maximum number of bags that could have been produced in that time frame on a particular extruder. With this approach, Ryland reports that it was possible to

substantially limit the number of possible source boxes from which the crime scene bag could have originated.

2. Organic Analysis

Cleverly (1979) used infrared (IR) analysis for the determination and comparison of the organic constituents of polyethylene bags. The basis of comparison was the frequency, shape and relative intensity of the IR absorption bands, most of which were rather weak. Although some discrimination was achieved, sample preparation requirements and spectral interferences of pigments and dyes limited this examination. Gilburt *et al.* (1991) and Griffin *et al.* (1996) also reported that using IR spectrophotometry and/or Fourier transform infrared (FTIR) microspectrophotometry aided in the discrimination of cling films. However, the majority of the samples in these two studies were composed of poly(vinyl) chloride (PVC) and the limited number of polyethylene samples analyzed failed to exhibit any absorption in the region of the IR that was found to be most discriminating for PVC samples (1450-1650 cm^{-1}). The authors did not draw any conclusions about the discrimination potential of IR for polyethylene films.

3. Elemental Analysis

A number of different instrumental analytical methods have been applied to the determination of inorganic constituents in plastics. These include energy dispersive X-ray fluorescence spectrometry (XRF), neutron activation analysis (NAA), atomic absorption spectrophotometry (AAS), inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS), including solid sampling with laser ablation, and total-reflection X-ray fluorescence

spectrometry (TXRF). While a variety of analytical methods have been employed to this end, the body of literature regarding the elemental analysis of polyethylene is relatively small. Most of the applications for the elemental analysis of polyethylene reported in the scientific literature involve quality control for the plastics manufacturing or food packaging industries. Analytical considerations in these studies including sample preparation, target elements, detection levels, etc., are not always applicable to forensic investigations. However, much insight can be gained from review of such literature, particularly with regard to expected concentrations of various elements in polyethylene and potential problems with sample preparation methods and analytical interferences.

Sample preparation of plastics for elemental analysis by instrumental methods can be difficult. Certain polymers, including polyethylene, are difficult to digest, even with strong acids, and their solubility in organic solvents, although possible, requires the use of toxic chemicals, is time-consuming and dissolution may be incomplete. Many instrumental techniques require that the sample be introduced in liquid form, with low tolerance for even small amounts of undissolved solids. Further, the incompatibility of organic solvents with some of these methods precludes their use in sample digestion. Recent advances in instrumentation and related sampling accessories have addressed some of these concerns. The introduction of more sensitive, matrix-independent techniques such as TXRF, and sampling accessories, such as high solids nebulizers and laser ablation sample introduction systems, have facilitated the elemental analysis of difficult samples such as plastics.

Electrothermal atomization (ET)-AAS, NAA and XRF have been applied to the determination of stainless steel constituents in plastics (Hoffmann *et al.*, 1991). The aim of this study was to identify wear at the metal surfaces of processing equipment by testing the molding compounds for metallic contaminants. While the authors noted the potential advantages of using ICP-AES, namely its simultaneous, multi-element capabilities, they opted not to employ this technique, since it necessitates the use of a digestion procedure. In this study, ET-AAS provided some good analytical information for chromium (Cr), manganese (Mn) and iron (Fe). However, it is not an ideal method for this application, because it requires sample dissolution and is time-consuming. ET-AAS is a single element technique and requires multiple measurements of backgrounds and samples for adequate precision and contamination control. NAA is useful in the measurement of Mn in polyethylene; however, the other elements of concern in this study (Hoffmann *et al.*, 1991) could not be determined by the NAA procedure presented there.

There have been a number of additional studies in recent years applying NAA to the quantitation of trace elements in plastics. NAA is a sensitive, nondestructive, simultaneous multi-element technique, which is particularly suitable for the analysis of polymer materials. The organic matrix, which is primarily carbon, hydrogen and oxygen, will not activate, resulting in a low background in the gamma-ray spectrum and therefore, good detection limits for trace elements (Thompson *et al.*, 1995). NAA has been used for the characterization of trace elements as contaminants in food packaging (Thompson *et al.*, 1994, 1995, 1996), for microelectronic applications (Haas *et al.*, 1993), and for environmental pollution concerns (Bode *et al.*, 1990; Bode, 1993; Landsberger and

Chichester, 1995). Haas *et al.* (1993) report detection of ten elements of interest to the microelectronics industry in a polyethylene sample in the part per billion (ppb) range. Thompson *et al.* (1995) analyzed raw materials and retail food packaging products, with detection limits less than 1 part per million (ppm) for over 50 elements in raw materials. They found higher concentrations of most elements in retail packaging materials than in the raw materials, as well as additional elements, which are introduced as additives in the manufacturing process. The elements detected in polyethylene retail materials reported by Thompson *et al.* (1995) are sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), sulfur (S), chlorine (Cl), potassium (K), calcium (Ca), titanium (Ti), Mn, cobalt (Co), copper (Cu), zinc (Zn), arsenic (As), strontium (Sr), antimony (Sb) and barium (Ba).

The works of Bode *et al.* (1990), Bode (1993), and Landsberger and Chichester (1995) focus on the application of NAA to the characterization of heavy metal components of household plastics as environmental pollutants. Bode *et al.* (1990) used NAA to measure the levels of cadmium (Cd), Cr, Fe, nickel (Ni), Zn, As, selenium (Se), bromine (Br), Sr, Sb, Ba, lanthanum (La), gold (Au) and mercury (Hg) in commercially available plastic products, including polyethylene bags. Landsberger and Chichester (1995), using NAA, report that concentrations of 32 elements in the plastic samples analyzed range from a few ppb to several percent. The authors note the large compositional variation among samples; however, this is expected since sample types in their study reflect a broad range of consumer products, with twelve different colors and at least seven different polymer types.

NAA offers many advantages for the quantitation of trace elements in polyethylene samples. Unfortunately, the difficulty in gaining access to a nuclear reactor or a neutron source and the safety issue of handling radioactive substances limits the application of NAA in routine forensic casework. However, NAA may be useful as an accurate, independent technique for validation of results of other methods applied to the elemental analysis of polyethylene.

Trace elements in food contact polymers were also determined by ICP-MS as part of a research project designed to investigate the catalyst residues in polymers and their potential migration into food (Fordham *et al.*, 1995). In this study, five different polymer types, including polyethylene samples, were digested by a microwave technique using sulfuric acid (H_2SO_4) and nitric acid (HNO_3) prior to analysis by ICP-MS. The use of sulfuric acid, which is required for an effective digestion, yields polyatomic interferences, in addition to the spectral interferences and polyatomics normally encountered with ICP-MS. Because of these interferences, reliable data could not be obtained for Ti, Ca, Fe and Cr, all of which are potentially useful elements for discrimination among polyethylene sources.

An additional drawback to the application of ICP-MS to the elemental analysis of polymers is the need for matrix-matched standards. Certified reference standards for trace elements in polymers are unavailable commercially (Dobney *et al.*, 1997); therefore, in-house polymer reference materials were produced and verified by NAA (Fordham *et al.*, 1995). Matrix-matched standards are also recommended for the

analysis of polymers by ET-AAS and NAA and are essential for analyses involving sampling by laser ablation (LA).

The use of LA-ICP-MS for direct analysis of solids has been investigated for the determination of trace elements in plastic materials (Marshall *et al.*, 1991). Marshall *et al.* (1991) used a neodymium:yttrium aluminum garnet (Nd:YAG) laser operated at IR wavelengths to ablate the samples, followed by quantitative analysis of the ablated matter by ICP-MS. This multi-element technique offers good analytical sensitivity and data for phosphorus (P) and Ni in pigmented polyethylene agreed well with results obtained using an XRF technique. The limitation of the LA-ICP-MS method used in this study is its precision, reported to be on the order of 10% relative standard deviation (RSD). Hemmerlin and Mermet (1996) emphasize the importance of optimizing the operating conditions for the sample type when using laser ablation for solid sample analysis. They also used a Nd:YAG laser for ablation under various operating conditions; however, quantitative analysis was performed with ICP-AES. They concluded that operating the laser at the shorter UV wavelengths provides more efficient, reproducible ablation for polymers. More recent work (Wolf *et al.*, 1998) with LA-ICP-MS reports that, for a filled polymer film, results for ten elements compare well (± 30%) with solution ICP-AES results.

Forensic investigations involving the elemental analysis of polyethylene have been limited to X-ray methods. Energy dispersive X-ray fluorescence spectrometry (XRF) is nondestructive, which is perhaps its major advantage for forensic applications. This not only allows for subsequent analysis of the samples by other techniques, but also

permits verification of the original analytical results, as may be required by a laboratory QA plan or court mandate. Although XRF has a broad dynamic range, it is fairly insensitive for the lower atomic number elements and at best, levels of detection are in the low ppm range. Quantitation and detection limits are matrix-dependent and full quantitative analysis requires the use of matrix-matched standards.

Nir-El (1994) conducted a study of the forensic discrimination of colored polyethylene bags using XRF. In this study, a variety of colored polyethylene bags from local stores in Israel were examined by XRF. The author performed statistical analyses of the XRF elemental profiles obtained for two batches of yellow bags (six bags per batch) and concluded that with this method, a bag will be assigned to its batch with a very high probability, approaching 1. While this study demonstrates the utility of XRF in performing elemental analysis of polyethylene, the size of the sample population studied is extremely small and insufficient to support the probability numbers reported. A larger sample size, including more batches of a similar color, produced by the same and different manufacturers is necessary for a more meaningful statistical interpretation of these data. Additionally, the batches of bags sampled in the Israeli study appear to be small, individual production batches (25 kilograms). Greater differences in elemental concentration between such batches are more likely to occur than with large-scale, continuous manufacturing operations, as is the current practice for the manufacture of polyethylene bags in the United States.

Ryland (1999) also performed a case-related study of the evidential value of elemental analysis of plastic trash bags using XRF. Ryland reports that the Ti to Fe

elemental peak ratios for samples from a single plant on different days and within a single day vary. He found that this variation in elemental composition is independent of extrusion die striae patterns.

TXRF has also been applied to the elemental analysis of polyethylene in forensic science applications (Düwel *et al.*, 1996; Simmross *et al.*, 1997). The results of these preliminary studies look promising, as TXRF offers increased sensitivity over conventional XRF and is free of matrix-related problems and many of the interferences associated with the other instrumental methods described previously. These studies will be discussed in further detail in the next section, so that the reader may fully appreciate the potential of this application, after first having been introduced to the theory of TXRF.

C. TXRF

The foundation of X-ray spectral analysis lies in the discovery of X-rays in 1895 by Röntgen and in the establishment of Moseley's relationship between the wavelengths of the characteristic X-ray lines and the atomic number of the element in 1913. Since then, X-ray fluorescence spectrometry has become a powerful tool for elemental analysis, particularly for solid samples, due to its nondestructive nature and ease of sample preparation. However, its application to trace analysis has been limited by matrix effects and relatively high detection limits.

These matrix effects have been virtually eliminated and detection limits greatly improved by TXRF. Although the phenomenon of the total reflection of X-rays was discovered by Compton in 1923, its application to X-ray spectral analysis was not realized until 1971. Yoneda and Hiriuchi (1971) first presented the concept of using an

optically flat sample support and low incident beam angle to totally reflect the primary X-ray beam, thereby reducing the background and lowering detection limits for energy dispersive X-ray analysis to the nanogram range. Their work was quickly followed by a number of publications detailing fundamental experiments and theoretical predictions regarding the refraction and reflection of X-rays. The introduction of a commercial instrument in 1980 (EXTRA II, Rich. Seifert & Co.), and subsequent improvements in instrumental design (EXTRA IIA, ATOMIKA Instruments, GmbH) have greatly advanced the development of TXRF.

Within the last decade, TXRF has proven to be a reliable analytical method for micro and trace analysis. TXRF has found numerous applications in a multitude of disciplines including semiconductor and surface analysis (Knoth *et al.*, 1989; Penka and Hub, 1989), environmental (Klockenkämper and von Bohlen, 1996) and geological (Muia and van Grieken, 1991) chemistry, marine science (Koopmann and Prange, 1991; Battistion *et al.*, 1993; Haarich *et al.*, 1993), forensic science (Ninomiya *et al.*, 1995; Prange *et al.*, 1995), art conservation (Klockenkämper *et al.*, 1993; Moens *et al.*, 1994; Devos *et al.*, 1995; Necemer *et al.*, 1996), petrochemistry (Reus, 1991; Ojeda *et al.*, 1993; Schirmacher *et al.*, 1993; Pasti *et al.*, 1996), biology (Ayala *et al.*, 1991), materials analysis (Hoffmann *et al.*, 1990; Hein *et al.*, 1992) and pharmaceuticals (Nomura *et al.*, 1992; Wagner *et al.*, 1996; Wagner *et al.*, 1997).

1. Theoretical Principles

TXRF differs from conventional energy dispersive XRF in that TXRF utilizes an incident X-ray beam at a grazing angle of less than 0.1°, which is less than the

critical angle for total reflection. Since excitation is carried out without significant penetration into the sample substrate, this geometry greatly improves the signal-to-noise ratio and thereby yields greater analytical sensitivity.

A full presentation of the theoretical principles and formulae associated with the phenomenon of the total reflection of X-rays is beyond the scope of this research project and the interested reader is referred to the literature (Klockenkämper *et al.*, 1992; Schwenke and Knoth, 1993; Aiginger and Streli, 1997; Klockenkämper, 1997). However, the following discussion is presented to acquaint the reader with the fundamental knowledge necessary to apply this technique to the investigation of trace evidentiary materials.

a) Reflection and Refraction

When a beam of electromagnetic radiation strikes an interface between two different dielectric materials at an oblique angle, it is both reflected and refracted. The degree to which each of these phenomena occurs depends upon the index of refraction of the two media, the wavelength of the radiation, and the angle of incidence. Simply, radiation which travels from a medium of high refractive index into a medium of lower refractive index will be refracted or bent away from the normal; that is, toward the boundary plane. This is the situation for X-rays penetrating into solid samples from vacuum or air. As the angle between the incident X-ray beam and the surface approaches zero, the refracted beam will emerge from the second medium tangential to the boundary surface. The angle above which refraction occurs is known as the critical angle. With glancing incident angles that are lower than the critical angle, there is, ideally, no primary beam penetration into the substrate and the boundary acts like a perfect mirror. The incident

radiation is totally reflected back into the first medium, at an angle equal to that of the glancing angle of the incident beam.

In reality, the totally reflected X-rays do penetrate into the medium (the sample support), but only very slightly. Sample supports used for TXRF are highly reflective and optically flat, such as highly polished quartz, glassy carbon and Plexiglas™, and must be chemically inert and free of contamination. The reflectivity of the support, or the ratio of the intensities of the reflected and incident beams, and the penetration depth, both of which are related to the angle of incidence, can and have been calculated for a number of sample supports using Fresnel equations. For highly reflective media, reflectivity drops and penetration depth increases dramatically as the incident beam passes through the critical angle. Therefore, by utilizing an angle of incidence which is just below the critical angle, which for most applications is about 0.1°, conditions for total reflection are optimized.

b) Standing Waves

The unique capabilities of TXRF are based on the formation of standing waves above surfaces and within the near-surface layers, on the order of a few nanometers. In the simplest case, the standing waves are created above the reflective support surface by the coherent interference of the incident and reflected beams. For incident angles well below the critical angle, the interference zone, or standing wave field, which exists above the interface between the incident and reflected waves, will show strong nodes and anti-nodes (minima and maxima), typically with a period of 10 to 100

nanometers. The intensity of the penetrating X-rays decreases exponentially with increasing depth below the interface.

2. Sample Preparation

Considering the important role of the formation of standing waves, proper sample preparation is critical for TXRF analysis. To insure accurate quantitative analytical results, the sample should be prepared as a homogeneous, thin film on a highly reflective sample support whenever possible. While direct solid analysis can be performed, care must be taken to ensure that the sample is sufficiently thin and that the conditions for total reflection have been satisfied. For thin, homogenous residues, the standing wave nodes and anti-nodes generally occur within the height of the sample, therefore potential fluorescent intensity differences which may occur due to differences in sample height will likely be minimal. The use of an internal standard, homogeneously mixed with the sample, is further recommended, as the ratio of fluorescence intensities will improve analytical accuracy and precision.

When conditions for total reflection of X-rays have been satisfied, the twofold excitation from the incident and reflected beams of a sample residue on a suitable support doubles the intensity of fluorescence radiation. Due to the high reflectivity of the substrate and low incident angle of the incident X-rays, contribution by the substrate to the fluorescence spectrum is negligible in comparison to that from the sample. Both the increased sample fluorescence and decreased background contribute to the notably low detection limits in TXRF.

3. Instrumentation

TXRF instrumentation employs basic components that are common to conventional XRF, including an X-ray source, Si(Li) detector, multi-channel analyzer, and a computer system for data processing. In the TXRF spectrometer, the primary beam is generated by an X-ray source, which passes through an appropriate filter to effectively suppress the low energy part of the exciting radiation. The beam is then spatially defined by a diaphragm or edge and directed onto a quartz reflector mirror, which acts as a low-pass filter to eliminate the high-energy *Bremsstrahlung* radiation. This is accomplished through careful geometric design such that only X-rays that are totally reflected by the quartz mirror will reach the sample plane. The primary beam is further defined by another diaphragm or edge upon exiting the quartz mirror, so that a ribbon-shaped beam arrives at the sample. In the instrument used for the present study, the use of two X-ray sources, molybdenum (Mo) and tungsten (W) anodes, with three operating conditions, allows for good sensitivity for elements in the range of sodium (Na) through uranium (U). The fluorescence radiation emitted from the sample is detected by a large Si(Li) detector, whose face is oriented parallel to the sample surface. With this configuration, none of the primary radiation reaches the detector. In addition, the distance between the sample and the detector is very small, on the order of one millimeter, providing a large acceptance angle for the X-ray fluorescence radiation.

4. Application to Forensic Samples

TXRF has been shown to be suitable for the trace elemental analysis of a variety of materials of forensic interest (Kubic *et al.*, 1995; Ninomiya *et al.*, 1995). The

analytical merits of TXRF, including good accuracy and precision, have been demonstrated with forensic samples including glass, brass, aluminum, and gunshot residue (GSR), and through validation studies using NIST standards and blind proficiency testing (Kubic and Buscaglia, 1996). The results of the preliminary application of this method to the characterization and discrimination of forensic samples of a variety of polymer types, including polyethylene bags, polypropylene tapes, poly(vinyl) chloride (PVC) tapes, wire insulation, and synthetic fibers (Buscaglia and Koons, 1998) have also been presented.

Additional survey and feasibility studies of the application of TXRF to the elemental analysis of plastic products of forensic interest have been conducted. Ninomiya *et al.* (1995) described the application of Glancing Incidence X-ray Fluorescence (GIXF), operated in the TXRF mode, to the analysis of vinyl tapes in forensic casework. Recently, Simmross *et al.* (1997) published a validation study using TXRF to determine various amounts of cadmium (Cd) in polyethylene certified reference materials. Solid samples in the 20 to 100 microgram range were prepared as thin films using a specially designed micro-press. The authors reported the accuracy of the method to be 3%, with a stated precision of 6%, as characterized by a confidence interval.

In 1996, Düwel *et al.* presented some preliminary results of the value of TXRF for the discrimination of thermoplastic remains. Their research work compared the discrimination potential of a number of analytical methods with that of TXRF for the analysis of polyethylene. In evaluating the discrimination capability of TXRF, these researchers looked at both the discrimination between packages and that between two bags from within the same package. The samples studied were taken from four packages

of green garbage bags from the same manufacturer purchased at different times over a period of approximately two years. Sample preparation was carried out in the same manner as that reported by Simmross *et al.* (1997). By using linear discriminant analysis, the authors concluded that TXRF was able to discriminate among all bags tested, including bags from within the same box.

There are a number of limitations with this study. First, only a small number of boxes were tested and the number of bags analyzed from within the same package was very small as well. In addition, in criminal investigations involving polyethylene bags, very often the question arises whether or not the crime scene sample can be associated with a particular recovered box of bags. If all bags from within a package are analytically distinguishable, as Simmross *et al.* assert, this question could not be answered by their method of data interpretation. One solution would be to change the definition of "distinguishable" to avoid eliminating other bags from the same production run. It is also possible, however, that the intra-package discrimination reported in this study reflects a sampling error. In the manufacturing process, it is possible that small batches of bags produced on different extruders are incorporated into a single box in the packaging stage. A larger intra-package sampling would have detected this variability, if it exists. Further, since samples were obtained from only one manufacturer, elemental compositions of polyethylene bags produced by other manufacturers and the discrimination capability of this technique among multiple sources are unknown. Finally, as no discussion of manufacturing processes or controls was provided, it is difficult to determine if these samples represent small, individual batches or a continuous process.

CHAPTER III. METHODOLOGY

A. Hypotheses

The aim of this study is to determine the compositional variations that exist within and among polyethylene bags. This information is required for proper evidentiary interpretation of an association between unknown or questioned and known polymeric materials based on trace element compositional analysis. This project addresses these issues and hypotheses listed here are general. More specific study details are contained in the Research Design section below.

Hypothesis 1: The concentrations of key elements can be determined in polyethylene bags with precision needed to provide discrimination of sources.

Hypothesis 2: Sample preparation of polyethylene bags, appropriate for TXRF, is feasible.

Hypothesis 3: TXRF will provide accurate, reproducible quantitative results of the key elements for the low levels of inorganic components that are present in polyethylene bags.

Hypothesis 4: The range of elemental compositions (or variability) increases in the order:

bag → box → production run → manufacturer

B. Scope and limitations

The scope of this research project is limited to the elemental analysis of black, polyethylene garbage bags. Under certain circumstances, discrimination among polyethylene bags sources may be attained through the examination of physical patterns

arising during extrusion. However, the limitations of such examinations include small sample size, as with a recovered fragment of plastic, physical damage or distortion of the bag, and the need for the next sequential bag in the box for comparison. This research project does not include the comparison of physical markings and individual characteristics of the bags analyzed beyond product line differences. However, gross construction characteristics of the bags (e.g., size, shape, handle construction, etc.) were noted and considered when evaluating the increased discrimination afforded by the incorporation of elemental analysis into the analytical scheme. As expected, manufacturers and product lines can be identified by physical measurements.

C. Research Design

This research project was designed in three phases, including method development, sample collection and analysis, and data interpretation.

1. Method Development

a) Instrument Calibration

Initial instrument calibration was performed on installation and a set of calibration factors was supplied for each analytical condition. These factors, which allow for quantitation using a single element internal standard, are matrix-independent and therefore, valid for any sample that meets the preconditions for TXRF. Once initial calibration was established, it was checked using certified multi-element standards and adjusted as needed for accuracy.

Analytical figures of merit, such as detection limits, accuracy, and precision for the TXRF instrument were determined. Replicate sample support preparations

were produced from the same liquid standard sample solutions and analyzed with Mo-K and W-*brems* excitation conditions, for varying assay times. These test samples were used to determine the accuracy and precision of the TXRF, as well as the precision as a function of analysis time. Samples rotated in the beam test the reproducibility of quantitative results with sample positioning. The effects of sample drop size on the accuracy and precision of the analytical measurements was evaluated by preparing five solutions at different concentrations of a given element, such that varying the sample volume resulted in the same final weight of the element on the sample support. All sample volumes tested were small enough, on drying, to be contained within the X-ray beam diameter.

b) Sample preparation

A sample preparation technique was developed and used throughout the course of the data collection phase of the project. While analyzing solid samples is possible, some sample preparation is necessary for good results. Although polyethylene bags may be commonly thought of as thin films, the film thickness of an average trash bag (22 μm) is, at a minimum, 200 times greater than the ideal film thickness for TXRF (10 – 100 nm). Additionally, removal of the organic matrix prior to TXRF analysis improves detection limits for quantitation of the inorganic constituents.

Sample cleaning prior to preparation was employed to reduce the contribution of extrinsic surface elements to the elemental profile. A number of different sample preparation techniques, including direct solid sample analysis, digestion procedures (acids and organic solvents), and high temperature ashing and low temperature oxygen plasma ashing to remove the organic matrix were tested. These sample preparation

techniques were evaluated for suitability based on safety, the extent of sample handling required, availability of ultrapure grade of reagent, destructive nature, completeness of matrix removal, potential loss of volatile elements, reproducibility, and the ability to transfer the product as a thin film onto the sample support. Precision and accuracy of the analytical results are the most important factor for selecting the method of sample preparation. The issue of contamination, always an important consideration in trace elemental analyses, is of particular concern with TXRF due to the sensitivity of the method. Contamination concerns affected the selection of a sample preparation method as well.

Elements such as Fe, Ca, Ti, and Zn may be suitable for discrimination purposes; however, these elements are major components of environmental contamination, so sample collection, handling, and preparation affect the accuracy of measured concentrations. This does not preclude their use for discrimination purposes, particularly when contaminant levels are extremely low and intrinsic levels are comparably high. The selection of sample preparation method and elements used for discrimination was made with allowance for the effects of contaminant elements, in addition to intra- and inter-sample variations.

Contamination was minimized by scrupulous cleaning and acid washing of sample supports and glassware, pre-screening of sample supports, reagents and internal standards for contamination prior to sample preparation, and handling and preparation of samples in a positive pressure, Class 100 clean bench. Good laboratory practice was employed to prevent cross-contamination between samples at the appropriate stages during sample preparation.

Once the sample preparation technique was established, it was necessary to introduce the sample to the TXRF as a thin film on a highly reflective sample support. Ideally, the internal standard can be homogeneously mixed with the sample prior to film formation. This preparation of sample on the support, as well as the selection of the appropriate sample support, constituted the final part of the sample preparation stage.

c) Analytical conditions

The first phase of this project concluded with the determination of the instrumental conditions to be used for analysis of the prepared samples. The choice of operating conditions, including exciting radiation, assay times, and selection and concentration of internal standard, depended upon the sample preparation technique used and the identity and quantity of the elements of interest in the samples. Ideally, one set of analytical operating conditions would be sufficient to quantitate all of the elements in polyethylene. However, these studies showed that, because of spectral overlaps of certain elements present in these samples, it was necessary to analyze each sample with at least two different radiation sources.

2. Sample Collection and Analysis

Sample collection and analysis of polyethylene bags from various sources was performed to test the hypotheses listed above. Measurement of the compositional variations within and among bags, boxes, and product lines was accomplished using a nested approach detailed below. Analytical results from each stage were assessed in order to determine the requisite number of samples for the subsequent stages of the research.

a) Compositional variation within single bags

Determination of the variation of element concentrations within an item of evidence is necessary to establish the homogeneity of the manufactured product. Generally, in order to discriminate among sources, the intra-sample variability of one or more measured parameters must be lower than the variability across sample sources. In accordance with hypothesis #4, it was anticipated that the variability of elemental concentrations within a plastic bag is small, based on manufacturing information and preliminary analytical data.

This study examined a representative number of bags from each manufacturer source, sampled at multiple locations across the bag using a grid pattern. The initial sampling included six samples from each side of the bag, for a total of 12 samples per bag. The size of the sub-samples used was varied to test for the presence of any micro or localized inhomogeneities in the bag, which are sample-size dependent. Data were evaluated with respect to sample position to detect any trend across the length or width of the bag and any variation in composition with bag side. This sampling was repeated on a minimum three bags from three manufacturers, distributors, or product lines so that a statistically significant conclusion may be drawn as to the presence of any inhomogeneity in the distribution of elements throughout bags. Since sample position did not have a significant effect on the elemental concentrations, the remaining intra-sample studies used a total of four samples per bag, two from each side of the bag.

b) Compositional variation within boxes

The variation of elemental compositions for plastic bags from within the same box was also explored. A box is the unit of sale that is available to the consumer. The concentration range of elements among samples that are contained within the same box is important in establishing an association between a crime scene bag and a recovered box. This variation is greatly influenced by packaging operations at the manufacturing plant; therefore, it may differ between manufacturers, plants, production dates, and product lines. Further, the number of bags contained with a box may influence this variation as well, as different box sizes are likely packaged separately. Larger box sizes may incorporate groups of bags that were produced during different manufacturing runs. Some of this information may be obtained from manufacturers, provided that they have the information and do not consider it proprietary. However, in many case situations, analysis of the remaining bags in the recovered box provides the necessary intra-box information as to the range of elemental concentrations, or groups of bags, contained therein. This approach has been used for quite some time with regard to trace elemental analysis in the comparison of lead projectiles (Peele *et al.*, 1991).

Measurement of the intra-box variation in this project began by selecting a box of 10 garbage bags and analyzing each bag within the box by TXRF to determine its elemental composition. Four samples per bag were analyzed, such that the intra-bag variation from this box could also be established. Intra-box and intra-bag data were evaluated to determine the magnitude of these two variations for the concentrations of each element. Results were also evaluated to determine whether bags from within the box

are a single compositional group or constitute multiple groups. This study was repeated using a larger sized box of bags (20; 13 bags analyzed) of the same manufacturer and product line to test whether intra-box variability is affected by box size. Intra-bag variation was tested within this larger box to confirm that it is consistent with that previously measured for smaller boxes of the same product. The order of the folded bags within a box was recorded and compared to the analytical results to test whether interleaving of the products of two or more bag-producing lines occurs during filling of boxes. These data were compared with manufacturer production information where it was available.

c) Compositional variation within manufacturing production runs

The elemental variation within manufacturing production runs is significant in the forensic interpretation of a compositional association. Compositional variation within a single production run or a short period of production time for a continuous process was determined to assess the potential for the association of a questioned product to its production source. Variability within production runs of manufactured products may differ between plants, product lines, and manufacturers. Additionally, the definition of a "lot" for polyethylene trash bags requires additional information from manufacturers on product packaging and may vary between manufacturers. Cooperation from the manufacturers with regard to this definition was sought, so that analytical results from samples of boxes with the same lot number could be properly evaluated.

The determination of the compositional variation within manufacturing production runs may be accomplished in two ways: the analysis of samples

from boxes with the same lot number which are commercially available, and the collection and analysis of manufacturing plant samples taken from the same production line at various points in time. The choice and scope of the latter method depended on the degree of cooperation that could be elicited from manufacturers. The use of purchased products with the same packaging codes was preferred, as sample selection and collection is facilitated. Additionally, the resultant data is more readily applicable to case situations, since bags that are encountered in an investigation were probably purchased in a box from a local retail store. Finally, the use of commercially available packages allows the assessment of within-lot number variability on any selected manufacturer, not simply those that are willing to cooperate, and any number of boxes or lots. Sample selection consisted of a minimum of four analytical samples per bag from each bag analyzed taken from at least three boxes with the same packaging code. The number of bags analyzed per box was based on the results of the within-box variation study described previously. This sampling was repeated for three different packaging codes from a manufacturer. This portion of the study constituted preparation and analysis of 108 samples per analytical condition, requiring approximately 125 hours of spectral acquisition time.

d) Compositional variation across manufacturing production runs

Inter-lot compositional variation was evaluated using the packages of plastic bags acquired from retail stores for the within-lot study above. Comparison of the analytical data collected of samples from packages with different packaging codes was conducted to determine the compositional variation across manufacturing runs. The number of samples per bag and the number of bags per box analyzed in this study were

determined by the product homogeneity, as identified in the earlier studies. When manufacturer-supplied information permitted, bag-producing machines were identified from the packaging code and the variation across production runs from the same and different bag-producing machines was evaluated.

e) Compositional variation across manufacturers

Since gross physical characteristics differentiate manufacturers, the determination of compositional variations across brands is not necessary. Therefore, no additional studies will be undertaken here. Rather, data from other previous studies simply were reevaluated vis-à-vis this hypothesis.

3. Data Evaluation

To a great extent, data evaluation was completed for each study prior to the start of the next stage. However, subsequent to the completion of each study of compositional variation detailed above, the data were reviewed to determine whether the sample size chosen was adequate, or if additional data were necessary for a valid interpretation. After the completion of all studies in the data collection phase, the data set of elemental composition of polyethylene bags was reviewed and the forensic significance of a compositional association was addressed.

Quantitative data collected from the elemental profiles of the various polyethylene samples and sources was used to assess the range of variability within and among manufactured products using standard statistical methods of analysis, including descriptive statistics and analysis of variance (ANOVA). Relationships among plastic bag sources were explored using pairwise mean comparison and range overlap methods

of the element concentration results. Star plots (Haswell and Walmsley, 1998) were constructed to graphically display the compositional differences between manufacturers.

CHAPTER IV. ANALYTICAL METHOD DEVELOPMENT

A. Reagents and Contamination Control

Because TXRF is capable of detecting most elements in the picogram range, contamination control is essential to acquire meaningful quantitative data. Potential sources of contamination include the reagents, glassware and plasticware, pipettes, sample handling, and the laboratory environment, including the room air, bench surfaces, sample preparation equipment, and analyst. If not handled properly, the samples themselves may also contribute contamination to the laboratory environment. Contamination was minimized throughout this research through the use of high purity reagents, cleaning procedures, and a class 100 clean hood, all of which are discussed below.

All reagents used for sample and standard preparation in this research were of the highest quality. Ultrapure water of 18-megaohm resistivity was used for washing, rinsing, diluting, and standard solution preparation. High purity HNO_3 (Optima™ grade, Fisher Scientific, Pittsburgh, PA), certified by the supplier to contain element concentrations of parts per trillion (ppt) or less, was used for sample and standard preparation. All analytical glassware used for standard solution preparation was leached in 40% (v/v) HNO_3 (reagent grade) for two weeks and triple rinsed with ultrapure water before initial use. Details concerning the cleaning procedure for sample supports and glassware used for plastic bag sample preparation are given in the appropriate sections below.

All standard solutions and high purity reagents were transferred to polypropylene or Teflon® containers for storage following preparation. Reagents and internal standards

were screened for contamination by TXRF prior to use in sample preparation. An example TXRF spectrum of high purity 10% (v/v) HNO_3 used in this project is shown in Figure 1. As indicated in the figure, even under the cleanest conditions possible in our laboratory, there are still picogram amounts of elemental contamination such as Ca, Fe, and Zn, detectable by TXRF. Fortunately, the levels of these elements are extremely low compared with the levels characteristic of plastic bags. Therefore, their presence in the reagent does not prohibit its use for this application, but does necessitate that reagent blanks be prepared and analyzed with each set of samples.

Sample handling and preparation was carried out in a positive pressure, class 100 clean hood to reduce contamination. This is a laminar flow, HEPA-filtered hood, which is designed to minimize the level of particulate material in the working environment. This is accomplished by blowing HEPA-filtered room air downward, through the hood, across the hood work area and out toward the user. Foreign matter is prevented from entering the hood and contaminating the sample by the flow of the air stream. Use of the clean hood was essential for reducing contamination, particularly Zn, from our laboratory air supply and particulate matter from the sample preparer.

B. Sample Supports

Sample supports for use with TXRF must have high reflectivity, and be chemically inert, optically flat and contamination-free. There are a number of materials that make suitable sample supports, but high purity quartz is the most commonly used for chemical analysis by TXRF, and was used in this project. Quartz sample supports are polished to be optically flat by the manufacturer and must be cleaned prior to use to

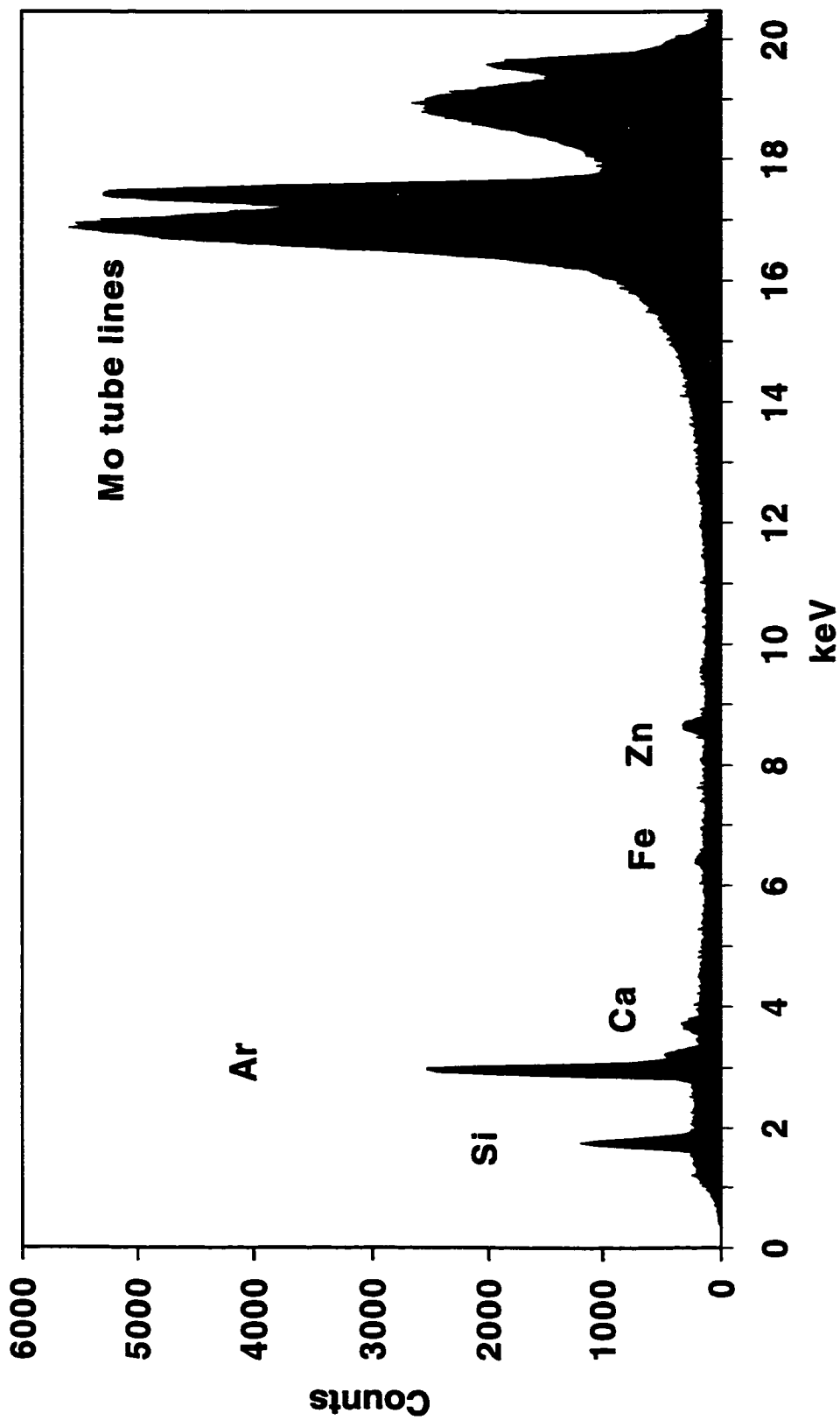


FIGURE 1: TXRF Mo-K excitation spectrum of 20 µL of 10% (v/v) ultrapure HNO_3 used for plastic bag sample preparation.

remove any remaining contamination from the polishing process. Since sample supports were reused in this project, they were also cleaned between samples with soap, ultrapure water, and nitric acid, and the cleanliness of each plate was verified prior to its reuse. After cleaning and drying, the sample supports are silicone treated by applying 10 µL of silicone solution in isopropanol (Serva GmbH & Co., Heidelberg, Germany) to each support. Manually tilting and rotating the support evenly disperses the silicone solution. The sample supports are then dried in an oven at 100°C for one hour. The silicone treatment after cleaning is used to make the quartz support more hydrophobic and aid in droplet formation during sample preparation. The cleaning procedure is further detailed in Figure 2. Example spectra of a new quartz sample support, prior to and after cleaning, are shown in Figures 3a and 3b, respectively. As shown, the contaminants Ca, Ce, Fe, Cu, Zn, and Sr are effectively removed by this cleaning procedure.

All quartz sample supports were scanned by TXRF prior to each use to verify their cleanliness. Any support which showed minor or trace amounts of impurities or which had an unacceptably high silicon peak was either recleaned or used for another application. The acceptability of supports was judged by examining spectra visually for contamination peaks above background and by using manufacturer recommended criteria. To quantitate the contamination level in the spectrum of a blank sample support, the argon (Ar) K_α X-ray peak, which is present in all spectra, was used. Because the sample chamber is not purged, atmospheric Ar remains in the space between the sample and detector and is excited to fluorescence. This virtual mass of Ar in the spectrum is constant at a given X-ray tube power and was calculated by acquiring multiple spectra of

FIGURE 2: Procedure for cleaning quartz sample supports.

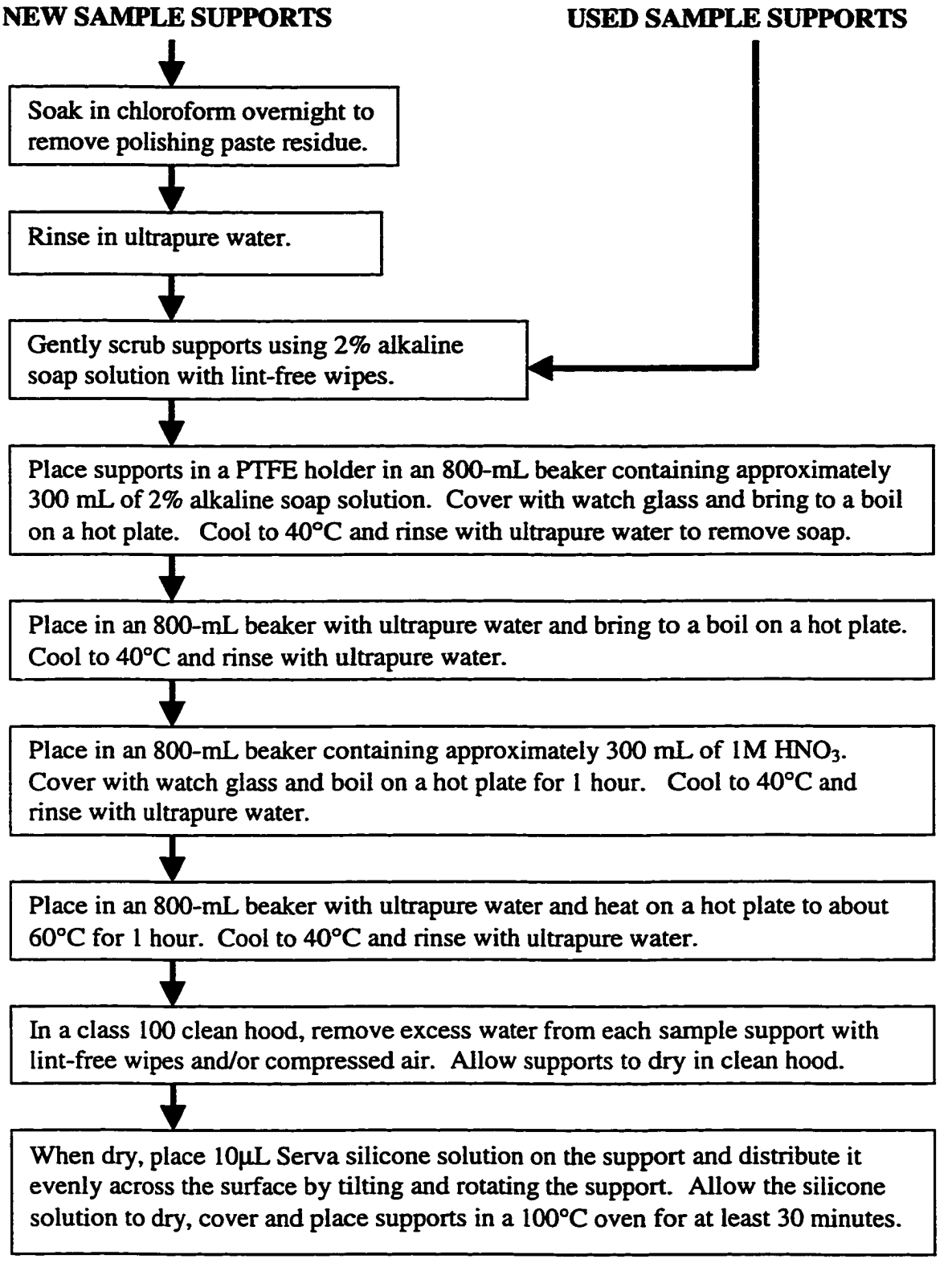
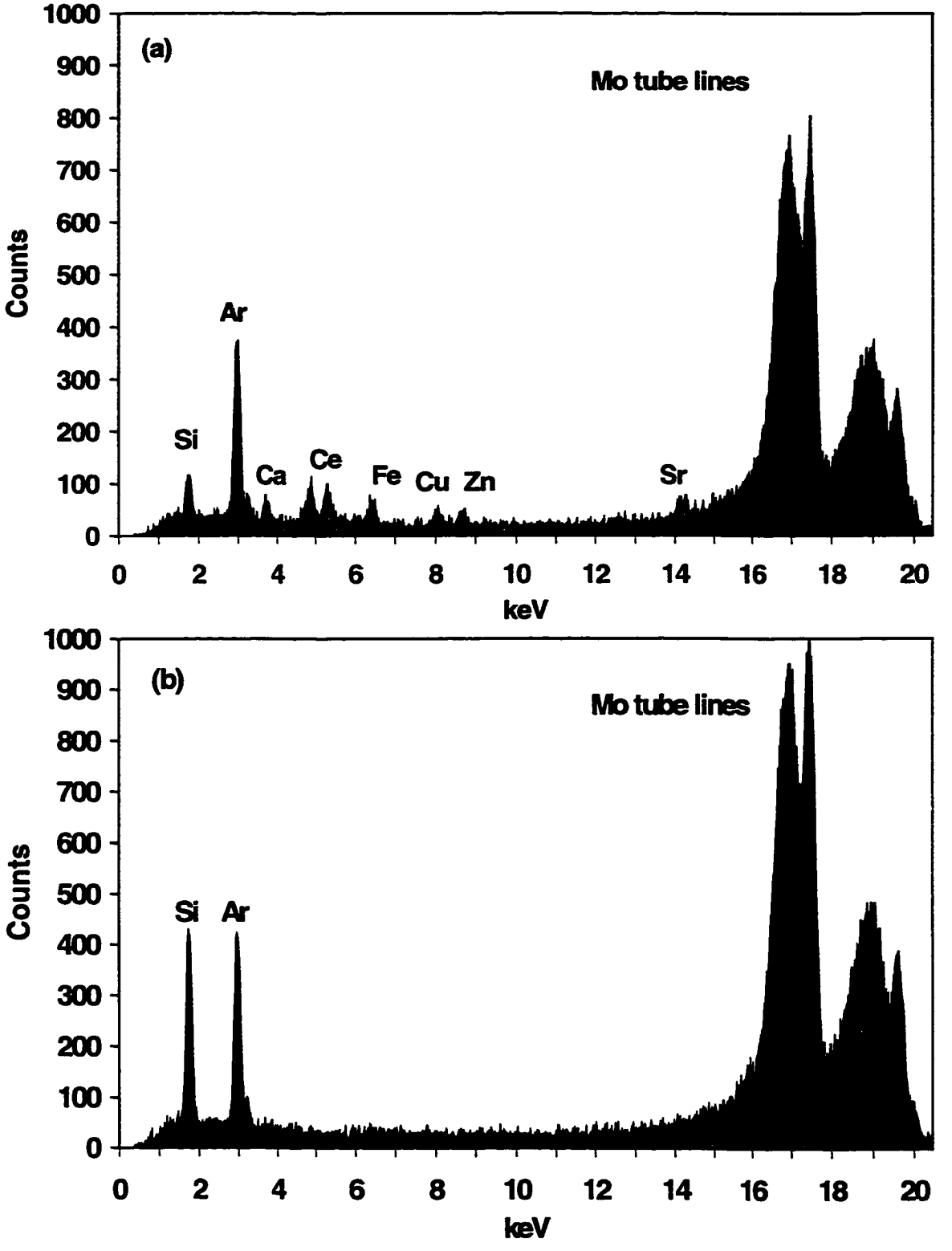


FIGURE 3: TXRF Mo-K excitation spectra of a new quartz sample support (a) before cleaning and (b) after cleaning and siliconizing.



a 1 ng Ni sample with Mo-K excitation, using Ni as the internal standard. Once the virtual mass of Ar was calculated to be 10.6 ng for this instrument, the amounts of contamination on blank sample supports and silicon in the spectrum were calculated using Ar as the internal standard.

The amount of silicon in a spectrum from each clean sample support was used to assess the quality of the surface of the sample support. As per manufacturer specifications, a silicon mass of greater than 1000 ng in the spectrum of a clean sample support indicates surface problems, such as surface scratches or silicone buildup from siliconizing. The use of an alkaline soap in the cleaning procedure removed most of the silicone from the support surface between uses. Sample supports with high Si due to silicone buildup were placed in a low temperature plasma asher for 1 hour at 100 watts RF and 300 cm^3/min oxygen flow to remove the silicone layers. Another method for removing silicone buildup is to immerse the support in 1M hydrofluoric acid (HF) for about 10 minutes, followed by rinsing in ultrapure water. This latter method worked well, but was discontinued when O_2 plasma ashing yielded satisfactory results without requiring HF handling, generating additional acid waste, or exposing plates to etching by HF. Sample supports that continue to show high Si are most likely scratched and need to be repolished before use.

C. Instrument Calibration

The TXRF spectrometer used in this research project is the EXTRA IIA (ATOMIKA Instruments GmbH, Oberschleissheim, Germany). Initial instrument calibration was performed by the manufacturer prior to installation and a set of calibration

factors was supplied for each analytical condition. These factors, which are based on relative fluorescence intensities, are used to obtain quantitative results for all measured elements with a single internal standard element. Calibration factors are used to calculate the mass of an analyte from its X-ray count rate by the following equation:

$$c_x = (Z_x / Z_i) \times (CF_x / CF_i) \times c_i \qquad \text{[Equation 1]}$$

where c_x = concentration of analyte; Z_x = net counts of analyte; Z_i = net counts of internal standard; CF_x = calibration factor of analyte; CF_i = calibration factor of internal standard; c_i = concentration of internal standard.

Once initial calibration was established, it was checked using a certified, NIST-traceable 19-element quality control standard. TXRF spectra from this certified multi-element standard, obtained using Mo-K and W-*brems* excitation, are shown in Figure 4a and 4b, respectively. The elements in this multi-element standard detectable by TXRF are printed on the spectra in Figure 4. Of the 19 elements certified in this standard, only 18 were detected using W-*brems* excitation and 17 using Mo-K excitation. Beryllium, which is present in the standard, can not be detected by TXRF under any excitation condition, and Mo can not be detected with Mo-K excitation. The peaks in the spectra in Figure 4 correspond to a concentration of 1 ng of each element. A smooth trend of increasing peak heights of X-ray K-lines with atomic number can be seen in these spectra. The peak intensities of L-lines also vary predictably with atomic number. This trend is not readily apparent from examination of the spectra, since the only elements whose L-lines are easily resolved visually from K-lines in the spectra in Figure 4 are Tl and Pb.

FIGURE 4a: TXRF Mo-K excitation spectrum of a certified, NIST-traceable multi-element standard. Sample is prepared on a quartz sample support, at a concentration of 1 ng of each element present. Assay time is 1,000 live seconds.

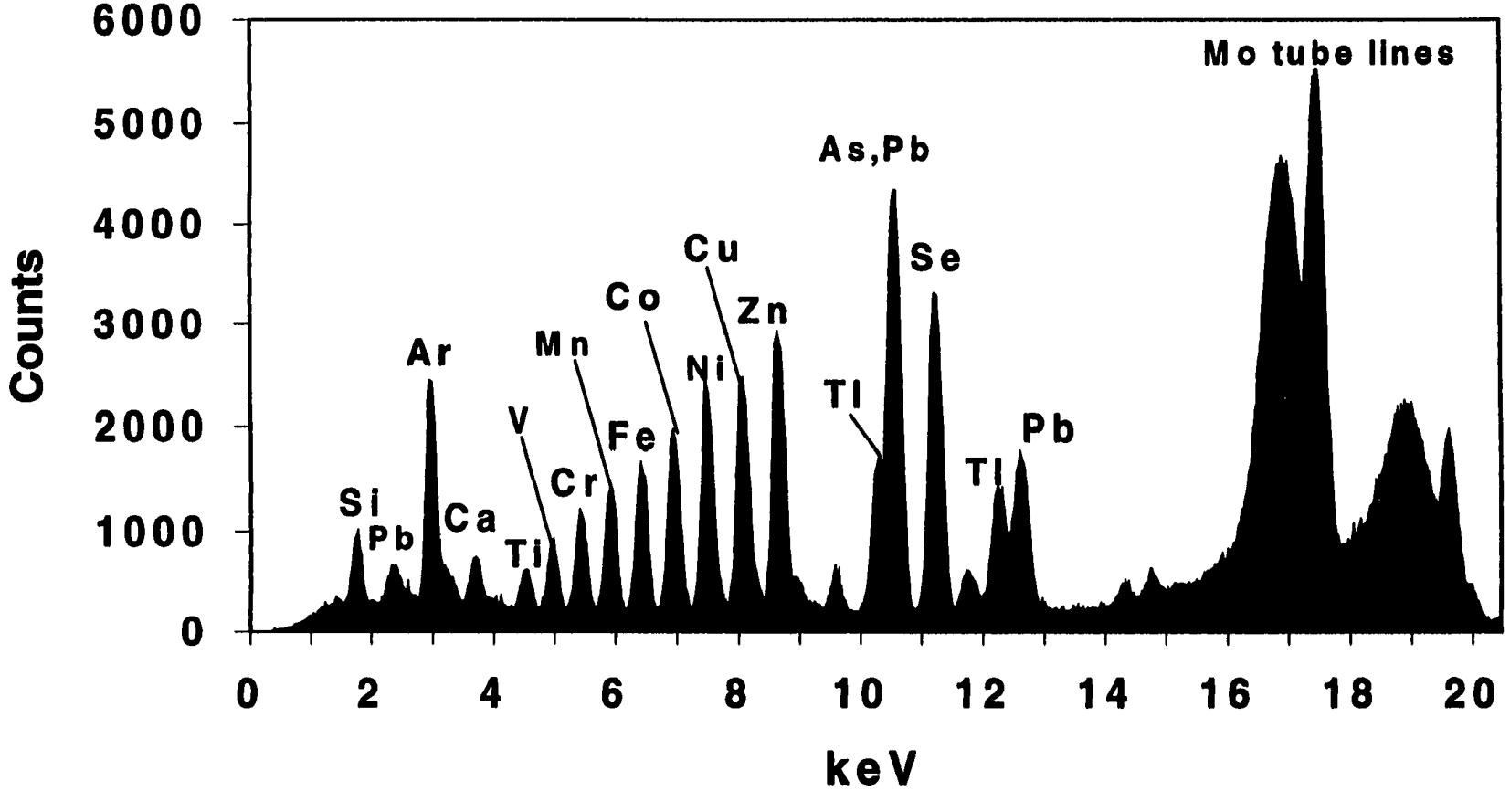
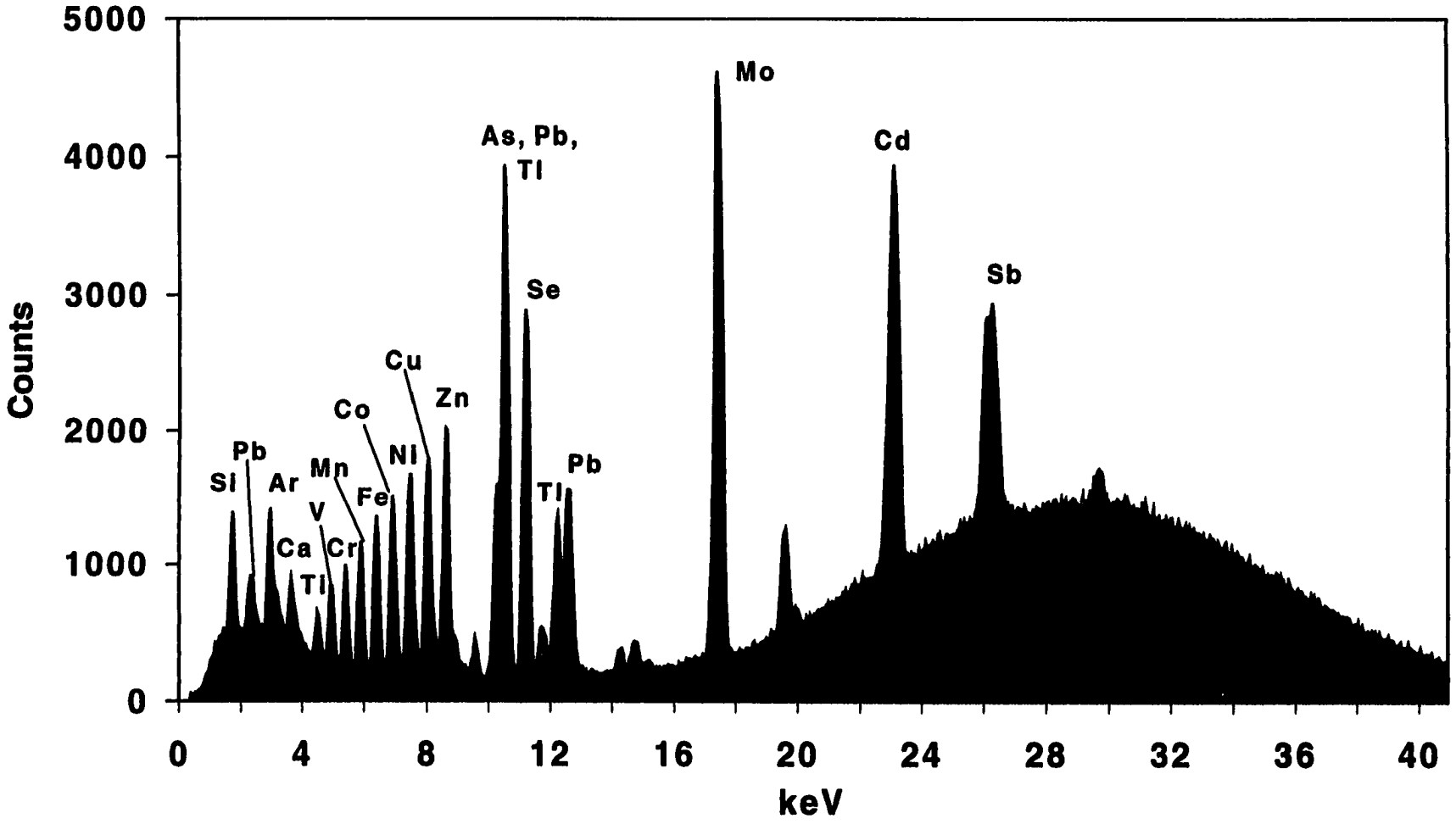


FIGURE 4b: TXRF W-*brems* excitation spectrum of a certified, NIST-traceable multi-element standard. Sample is prepared on a quartz sample support, at a concentration of 1 ng of each element present. Assay time is 1,000 live seconds.



This change in peak height reflects the difference in relative sensitivity with atomic number, which is the basis for use of calibration factors.

Poor accuracy for some elements in the multi-element standard and problems with the optical alignment of the instrument required the calibration factors supplied by the manufacturer to be recalculated. To calculate calibration factors, fluorescence intensities for elements relative to a fixed element are needed for each mode of excitation. This was achieved by the preparation and analysis of multi-element solutions, using Co as the internal standard for Mo-K excitation and Cd as the internal standard for W-*brems* excitation. The concentrations of the elements in the solutions were adjusted so that the preparation of a TXRF sample support yielded X-ray photo-peaks with approximately equal sensitivity and good precision based on X-ray counting statistics. Calibration solutions for Mo-K and W-*brems* excitation were custom-made and certified by High Purity Standards (Charleston, SC). The elements and their concentrations in each calibration solution are listed in Tables 1a and 1b. A custom-made, multi-element "plastic bag" standard (High Purity Standards, Charleston, SC) was used to calculate the calibration factors for K-lines with W-L excitation, using with Ti as the internal standard. This "plastic bag" standard was also used to check the accuracy of the TXRF during data acquisition, which will be addressed later.

Triplicate TXRF sample supports were prepared per calibration solution using 5 – 10 μL, as needed to keep the dead time less than or equal to 40%. Spectra were collected with Mo-K and W-*brems* excitation, and calibration factors were calculated for K and L X-ray lines, by rearranging Equation 1 as follows:

TABLE 1a: Standard solutions for calibration of TXRF relative fluorescence intensities using Mo-K excitation.

Solution	Element	Conc. (mg/L)	Solution	Element	Conc. (mg/L)
M 1	Bismuth	10	M 2	Arsenic	10
(5% HNO_3)	Cesium	80	(5% HNO_3)	Barium	80
	Chromium	30		Calcium	50
	Cobalt	20		Cobalt	20
	Europium	30		Copper	20
	Potassium	50		Lead	10
	Sulfur	100		Manganese	30
	Thulium	20		Praseodymium	50
	Yttrium	10		Strontium	10
	Zinc	10			

Solution	Element	Conc. (mg/L)	Solution	Element	Conc. (mg/L)
W 3	Cobalt	20	W 4	Antimony	100
(5% HNO_3)	Gallium	10	(5% HNO_3	Cerium	50
	Iron	20	+ Trace HF	Cobalt	20
	Neodymium	50	+ Trace HCl)	Gadolinium	30
	Palladium	200		Gold	10
	Rubidium	10		Hafnium	20
	Scandium	40		Holmium	20
	Selenium	10		Nickel	20
	Thallium	10		Rhodium	200
	Ytterbium	20		Uranium	20
				Vanadium	30

Solution	Element	Conc. (mg/L)	Solution	Element	Conc. (mg/L)
W 5	Cadmium	200	W 6	Cobalt	20
(5% HNO_3	Cobalt	20	(5% NH_4OH)	Molybdenum	300
+ Trace HF	Germanium	10		Tungsten	10
+ Trace HCl)	Iridium	10		Bromide	10
	Tantalum	10		Iodide	100
	Titanium	40			
	Zirconium	10			

TABLE 1a: (Continued)

Solution	Element	Conc. (mg/L)
W 7 (10% HCl)	Cobalt	20
	Lanthanum	50
	Lutetium	20
	Platinum	10
	Rhenium	10
	Samarium	50
	Terbium	30
	Thorium	20
	Tin	200

TABLE 1b: Standard solutions for calibration of TXRF relative fluorescence intensities using W-*brems* excitation.

Solution	Element	Conc. (mg/L)	Solution	Element	Conc. (mg/L)
W 1	Bismuth	20	W 2	Arsenic	30
(1M HNO_3)	Cadmium	10	(2M HNO_3)	Barium	40
	Cesium	20		Cadmium	10
	Chromium	100		Calcium	500
	Dysprosium	80		Cobalt	80
	Europium	100		Copper	50
	Potassium	500		Erbium	50
	Thulium	50		Lead	20
	Yttrium	10		Manganese	100
	Zinc	40		Praseodymium	100
				Strontium	20

Solution	Element	Conc. (mg/L)	Solution	Element	Conc. (mg/L)
W 3	Cadmium	10	W 4	Antimony	20
(1M HNO_3)	Gallium	30	(1M HNO_3	Cadmium	10
	Iron	80	+ Trace HF	Cerium	80
	Neodymium	100	+ Trace HCl)	Gadolinium	100
	Palladium	10		Gold	30
	Rubidium	20		Hafnium	50
	Scandium	300		Holmium	80
	Selenium	20		Nickel	50
	Thallium	30		Rhodium	10
	Ytterbium	50		Uranium	20
				Vanadium	200

Solution	Element	Conc. (mg/L)	Solution	Element	Conc. (mg/L)
W 5	Cadmium	10	W 6	Cadmium	10
(2M HNO_3	Germanium	30	(5% NH_3)	Molybdenum	10
+ 1M HF)	Indium	10		Tellurium	10
	Iridium	30		Tungsten	50
	Mercury	30		Bromide	20
	Niobium	10		Iodide	20
	Tantalum	50			
	Titanium	200			
	Zirconium	20			

TABLE 1b: Standard solutions for calibration of TXRF relative fluorescence intensities using W-*brems* excitation.

Solution	Element	Conc. (mg/L)
W 7	Cadmium	10
(2M HCl)	Lanthanum	50
	Lutetium	50
	Platinum	30
	Rhenium	30
	Ruthenium	10
	Samarium	100
	Terbium	80
	Thorium	20
	Tin	10

$$CF_x = (Z_i / Z_x) \times (c_x / c_i) \times CF_i \qquad \text{[Equation 2]}$$

Calibration factor data for all excitation conditions were fit with beta spline functions using TableCurve™ 2D (Jandel Scientific, San Rafael, CA) and calibration factors for elements not analyzed were interpolated from the fitted curve. Calibration factors used for this research for Mo-K, W-*brems*, and W-L excitation conditions are plotted versus atomic number in Figure 5.

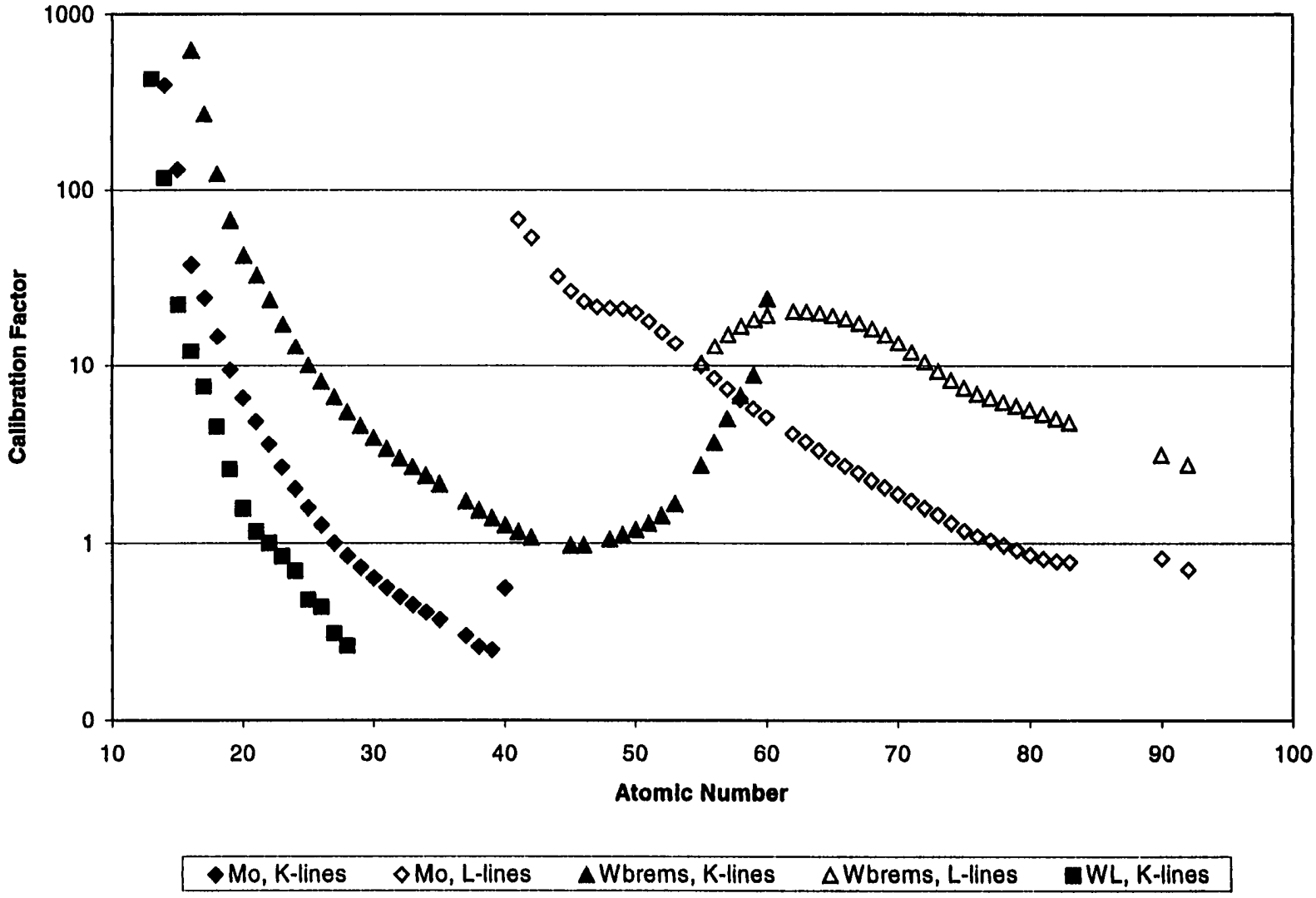
1. Detection Limits

For simple matrices, the detection limits for TXRF spectrometry are on the order of 5 to 15 pg, depending on atomic number. The detection limits of the EXTRA IIA TXRF spectrometer were tested using standard liquid samples, pipetted onto quartz sample supports and dried on a hot plate at 70°C. The final TXRF sample used for the determination of detection limits contained 1 ng Ni, 1 ng V, and 1 ng Mo, for testing Mo-K, W-L, and W-*brems* excitation conditions, respectfully. In order to determine the detection limits, the sample was analyzed for 1,000 seconds of live time (live seconds) at each excitation condition. The net (background subtracted) counts in the X-ray photo-peak of interest were compared with the background counts associated with that peak according to the following equation:

$$N_{DL} = N_{Ref} \times (3 \times \sqrt{S_{BG}} / S_{Ref}) \qquad \text{[Equation 3]}$$

where N_{Ref} is the concentration of the analyte, S_{BG} is the background counts for analyte, and S_{Ref} is the net counts for the analyte. Using this equation, detection limits determined for this instrument are 4.1 pg for Ni, 2.9 pg for V and 5.5 pg for Mo.

FIGURE 5: Calibration factors for quantitation by TXRF using three excitation conditions.



2. Accuracy and Precision

The effects of sample drop size on accuracy and precision of the analytical measurements were evaluated. Five solutions Cr, Co, and Ga at different concentrations were prepared such that varying the sample volume resulted in a final weight of 10 ng of each element on the sample support. Sample volumes tested were 2 µL, 5 µL, 10 µL, 20 µL and 40 µL, all of which are small enough to be contained within the 8 millimeter wide X-ray irradiation area. Quantitative data for Cr and Ga were determined using Co as the internal standard. The results of this study are listed in Table 2a and 2b. The error of the quantitative results for Cr and Ga is 10% or less, depending on the element and excitation condition. These data indicate that there is no significant difference in accuracy between the data acquired for the different sample volumes tested. The replicate precisions for all sample volumes are good, most having a relative standard deviation (RSD) among the replicates of 1.5% or less. As expected, slightly higher RSDs were obtained using the 2 µL volume, which is most likely due to the imprecision of the pipettor (Eppendorf Series 2000, Brinkman Instruments, Inc., Westbury, NY) at this delivery volume. Based on the results of this study and considerations in digestion and dissolution of plastic samples, samples in this research were prepared using drop sizes between 10 µL and 40 µL.

A study was undertaken to determine the optimum assay time for this research project. The precision of X-ray peak counts improves with longer assay times, by the square root of the assay time. In this study, three replicate sample supports were prepared, each containing 1 ng of Co and Ga, and were analyzed for various assay times with Mo-K excitation. The analyte mass was reduced from 10 ng to 1 ng for this study so

TABLE 2a: Results for the study of the effect of sample drop size on analytical precision. Sample preparations contain 10 ng each of Cr, Co, and Ga. Co is the internal standard used for quantitation. Excitation is by Mo-K with a thin filter. Assay time is 200 live seconds.

Results in ng of Cr

	Sample Volume				
Sample Preparation #	2 µL	5 µL	10 µL	20 µL	40 µL
1	10.00	10.32	10.10	10.06	10.37
2	10.15	10.27	10.15	10.13	10.34
3	10.29	10.07	10.26	10.36	10.29
Mean =	10.15	10.22	10.17	10.18	10.33
Standard Deviation =	0.144	0.133	0.080	0.158	0.036
% RSD =	1.41%	1.30%	0.79%	1.55%	0.35%

Results in ng for Ga

	Sample Volume				
Sample Preparation #	2 µL	5 µL	10 µL	20 µL	40 µL
1	9.68	10.12	9.94	10.04	10.25
2	9.79	9.94	9.92	10.14	10.03
3	10.14	9.93	10.06	10.08	10.15
Mean =	9.87	10.00	9.97	10.09	10.14
Standard Deviation =	0.242	0.111	0.074	0.055	0.110
% RSD =	2.45%	1.11%	0.74%	0.54%	1.09%

TABLE 2b: Results for the study of the effect of sample drop size on analytical precision. Sample preparations contain 10 ng each of Cr, Co, and Ga. Co is the internal standard used for quantitation. Excitation is by W-*brems* with a thin filter. Assay time is 200 live seconds.

Results in ng of Cr

	Sample Volume				
Sample Preparation #	2 µL	5 µL	10 µL	20 µL	40 µL
1	10.57	10.98	11.00	11.22	11.00
2	10.72	11.13	10.84	10.84	10.97
3	11.22	10.88	10.88	10.84	10.99
Mean =	10.84	11.00	10.91	10.96	10.99
Standard Deviation =	0.343	0.126	0.085	0.218	0.015
% RSD =	3.16%	1.15%	0.78%	1.99%	0.13%

Results in ng of Ga

	Sample Volume				
Sample Preparation #	2 µL	5 µL	10 µL	20 µL	40 µL
1	9.61	10.10	9.76	9.96	9.94
2	9.65	9.98	9.83	10.00	10.18
3	10.04	9.91	9.80	9.86	10.00
Mean =	9.77	10.00	9.80	9.94	10.04
Standard Deviation =	0.236	0.096	0.034	0.068	0.125
% RSD =	2.42%	0.96%	0.35%	0.69%	1.25%

that precision would be poorer than in the sample size study, and the effect of assay time on precision would be more evident. Spectra were obtained using five assay times ranging from 200 to 10,000 live seconds. Quantitative results at various assay times for Ga were calculated using Co as the internal standard. The RSDs of the 1 ng Ga replicates about their means for each assay time are listed in Table 3, along with the RSDs by X-ray counting statistics. The results for Ga indicate an improvement in precision occurs between 500 and 1,500 live seconds. Based on X-ray counting statistics, the improvement in the precision for Ga is about an order of magnitude, from 1.8% (200 live seconds) to 0.22% (10,000 live seconds). The precision of the quantitative data among the replicate preparations is higher than the X-ray counting uncertainty. This occurs because quantitative data reflect the uncertainty caused by sample preparation and positioning, in addition to the X-ray counting uncertainty. An assay time of 1,000 live seconds was selected for use in the survey portion of this research project. The selected assay time is a compromise between the best precision and a reasonable sample turnaround time, and is expected to yield suitable precisions for discrimination purposes, even at the low nanogram levels. Additionally, increasing the assay time to improve the precision for the analysis of plastic bags would be fruitless, because the heterogeneity of the bags and the variability in sample preparation is greater than the X-ray counting uncertainty.

The accuracy and precision for the TXRF instrument was also determined using standard solutions containing 1 ppm each of Cr, Co, and Ga. Five replicate sample support were prepared from the same sample solutions by pipetting 10 μL of the 1 ppm Cr, Co, Ga solution onto separate supports and drying on a 70°C hot plate. These replicate

TABLE 3: Results for the study of the effect of assay time on analytical precision. Sample preparations contain 1 ng each of Co and Ga. Co is the internal standard used for quantitation. Excitation is by W-*brems* with a thin filter.

Assay time (live seconds)	ng Ga		% RSD based on	
	Mean	Standard Deviation	Replicates	X-ray Counting Statistics
200	1.038	0.023	2.22 %	1.75 %
500	1.008	0.023	2.28 %	1.10 %
1,500	1.001	0.013	1.30 %	0.59 %
4,500	1.002	0.017	1.70 %	0.33 %
10,000	1.004	0.014	1.40 %	0.22 %

TABLE 4: Results for the study of analytical precision among replicate sample preparations containing 10 ng each of Cr, Co, and Ga. Co is the internal standard used for quantitation. Assay time is 200 live seconds.

	Mo-K Excitation		W-*brems* Excitation	
Sample Preparation #	ng Cr	ng Ga	ng Cr	ng Ga
1	10.180	9.965	11.022	9.615
2	10.286	10.013	11.273	9.876
3	10.114	9.974	10.971	9.743
4	10.119	9.951	11.021	9.847
5	10.114	9.909	10.788	9.718
Mean =	10.163	9.962	11.015	9.760
% Error* =	1.6%	-0.4%	10.2%	-2.4%
Standard Deviation =	0.074	0.038	0.173	0.105
% RSD =	0.73%	0.38%	1.57%	1.08%

* % Error = [(M – T) / T] x 100% ; where M is the mean of the measured values and T is the true value.

preparations, which have a total mass of 10 ng each of Cr, Co, and Ga per sample support, were analyzed with Mo-K and W-*brems* excitation conditions. The 10 ng Co served as the internal standard to determine the concentrations of Cr and Ga on each preparation. The results of these tests are shown in Table 4. The accuracy, expressed as the percent error, is between 1 and 2% for Mo-K excitation, with a precision between sample preparations of better than 0.8%. Using W-*brems* excitation, the percent error is between 3 and 10%, with a precision better than 1.6%. The accuracy and reproducibility of the TXRF are good and suitable for source comparison.

These 10 ng Cr, Co, Ga replicate sample preparations were also rotated in the X-ray beam to test the reproducibility of quantitative results with sample positioning. Samples supports were rotated to four sample orientations, 90° apart, and TXRF spectra were collected at each position for 200 live seconds using Mo-K excitation. The quantitative data obtained from this study of the effect of sample position on precision are in Table 5. The precision of the results of a sample rotated to 4 positions is indistinguishable from the precision of the results of five replicates analyzed in one position. These data confirm that, provided the sample is contained within the analytical area on the sample support and positioned to the appropriate height, the orientation of the sample in the X-ray beam has no significant effect on the accuracy or precision. In agreement with the previous study, the precisions of all replicates in this study for 10 ng levels of analyte elements and Mo-K excitation are 0.8%.

TABLE 5: Results of the study of the effect of sample rotation on analytical precision. Sample preparations contain 10 ng each of Cr, Co, and Ga. Co is the internal standard used for quantitation. Excitation is by Mo-K with a thin filter for 200 live seconds. (a) Results are in ng of Cr and (b) results are in ng Ga.

(a)

	Orientation of sample						
Sample Preparation #	0 °	90 °	180 °	270 °	Mean	Std. Dev.	% RSD
1	10.180	10.142	10.143	10.028	10.123	0.066	0.65%
2	10.286	10.095	10.146	10.256	10.196	0.090	0.88%
3	10.114	10.116	10.322	10.151	10.176	0.099	0.97%
4	10.119	10.019	10.215	10.130	10.121	0.080	0.79%
5	10.114	9.984	10.101	9.997	10.049	0.068	0.65%
Mean =	10.163	10.071	10.185	10.112	10.133		
Standard Deviation =	0.074	0.067	0.086	0.104	0.090		
% RSD =	0.73%	0.66%	0.85%	1.02%	0.88%		

(b)

	Orientation of sample						
Sample Preparation #	0 °	90 °	180 °	270 °	Mean	Std. Dev.	% RSD
1	9.965	9.847	9.848	9.997	9.914	0.078	0.79%
2	10.013	9.840	9.957	10.101	9.978	0.109	1.09%
3	9.974	9.931	10.062	9.876	9.961	0.079	0.79%
4	9.951	9.881	9.972	10.040	9.961	0.065	0.65%
5	9.909	9.793	9.961	9.841	9.876	0.074	0.75%
Mean =	9.962	9.858	9.960	9.971	9.938		
Standard Deviation =	0.038	0.051	0.076	0.110	0.083		
% RSD =	0.38%	0.52%	0.76%	1.10%	0.83%		

D. Sample Preparation Methods

Samples were excised from the polyethylene bag and cleaned to remove environmental contamination from the surface. The cleaning procedure consisted of washing the samples in a 2% alkaline soap solution with ultrasonic agitation, followed by triple rinsing in ultrapure water, rinsing in acetone and air drying in a class 100 HEPA-filtered laminar flow hood. Sample preparation methods studied for use in the elemental analysis of polyethylene blown film by TXRF are described below. TXRF spectra are shown for samples prepared by various methods. All spectra included below in the discussion of sample preparation method development are of samples taken from the same Glad® black, polyethylene trash bag to facilitate comparisons of preparation methods.

1. Direct analysis of solid samples

Direct analysis of solid samples would be the ideal choice for forensic samples if suitable analytical results could be obtained with TXRF. The direct analysis of solid samples does not compromise the integrity of the original specimen, is less prone to contamination, and, because it is nondestructive, allows additional or confirmatory testing or reanalysis of the evidentiary material. A number of methods of preparation for the direct analysis of solid samples by TXRF were explored and are discussed below. While some of the techniques for the direct analysis of solid samples tested offer good elemental information for preliminary comparison purposes, the low number of elements detected above the high polymer matrix background does not offer much discrimination potential among sources.

Düwel *et al.* (1996) describe a method for the preparation of thermoplastic remains for analysis by TXRF using a heated micropress to produce a thin polymer film directly on a quartz sample support. A similar approach was attempted by placing a small piece of plastic (approximately 1.5 mm^2) between two sample supports, warming on a hot plate and applying pressure to the upper support until a thin film was produced. The supports were allowed to cool and were then separated manually, resulting in a thin film on one of the supports. Analysis of the preparation yielded a TXRF spectrum with detectable peaks above a high background. However, while stereomicroscopic examination showed no evidence of the presence of plastic on the second sample support used to produce the thin film, analysis of this support showed considerable amounts of contamination from the plastic. This sample preparation technique was not selected because of (1) the potential sample loss caused by contact with the other sample support during thin film formation; (2) the inappropriateness of placing the internal standard for calibration on the surface of the thin film; (3) concerns of inhomogeneities and insufficient mass of elements for quantitation due to the small sample size; and (4) incomplete or irreproducible adhesion of the film to the quartz support, resulting in deviations from optimum TXRF geometry.

Sample thickness and proper geometry for total reflection of the incident X-ray beam are the two of the major problems with direct solids analysis of the plastic film. Since the angle of the incident X-ray beam can not be altered easily to accommodate different sample positions with the EXTRA IIA, a direct solid analysis was attempted using a large disc of the sample plastic bag, equal in size to the sample support

(30 mm diameter). This large disc of plastic was placed directly on top of a sample support and glued lightly at the edges to prevent it from dislodging in the autosampler. With this method, the plastic sample was positioned so that the X-ray beam would reach the sample surface at the proper angle for total reflection. The plastic sample acts as both the sample and reflector, and the X-ray fluorescence originates from only the surface. Figures 6a and 6b show example spectra of a black, polyethylene trash bag prepared in this manner. This method is not ideal for quantitative measurements, because of nonrepresentative sampling of multilayer laminates, the inability to use an appropriate internal standard, and the fact that the sample surface is not perfectly flat. As with the other direct solid methods tested, detection limits are poor compared with the results of digested samples (discussed below), and only a few elements are reliably detected over the high background of the polymer matrix. However, this preparation method is fast, simple, and works well as a screening method for the comparison of samples prior to matrix reduction techniques. Both sides of the plastic sample disc should be analyzed and orientation considered when making comparisons between plastic bags using this method. Differences in the elemental ratios for the major elements present in the polymer outside the expected or measured within-source variations can quickly discriminate among some plastic bag sources. Analysis of large sheet samples of plastic bags by TXRF offers no advantage over the similar results obtained using XRF, a method that is currently available in many forensic science laboratories.

Another solid sample preparation technique that was attempted was placing a small piece of plastic (approximately 1 mm x 3 mm) directly onto a sample

FIGURE 6a: TXRF Mo-K excitation spectrum of a 30 mm diameter sample of Glad® black polyethylene trash bag on a sample support. Assay time is 200 live seconds.

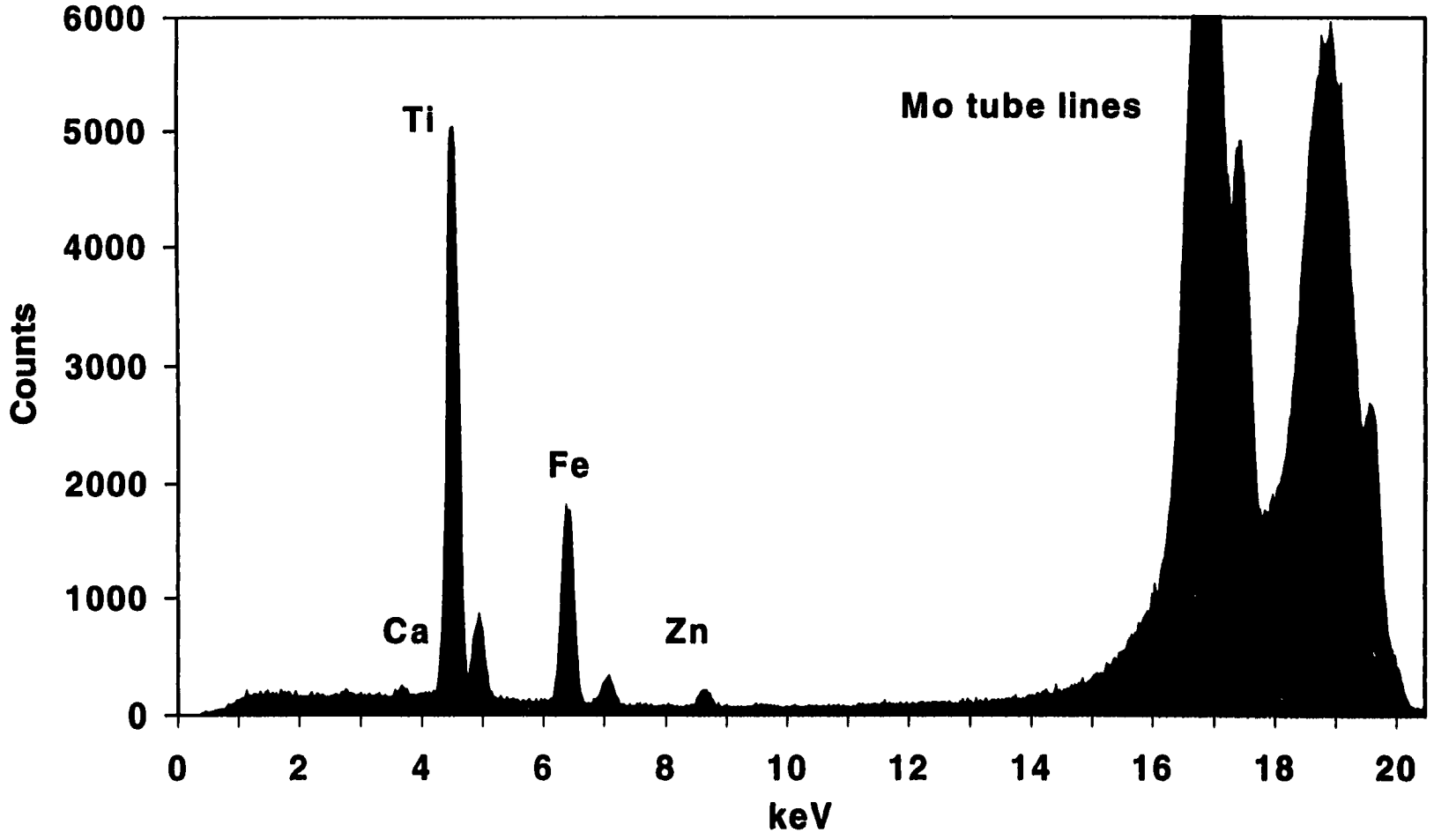
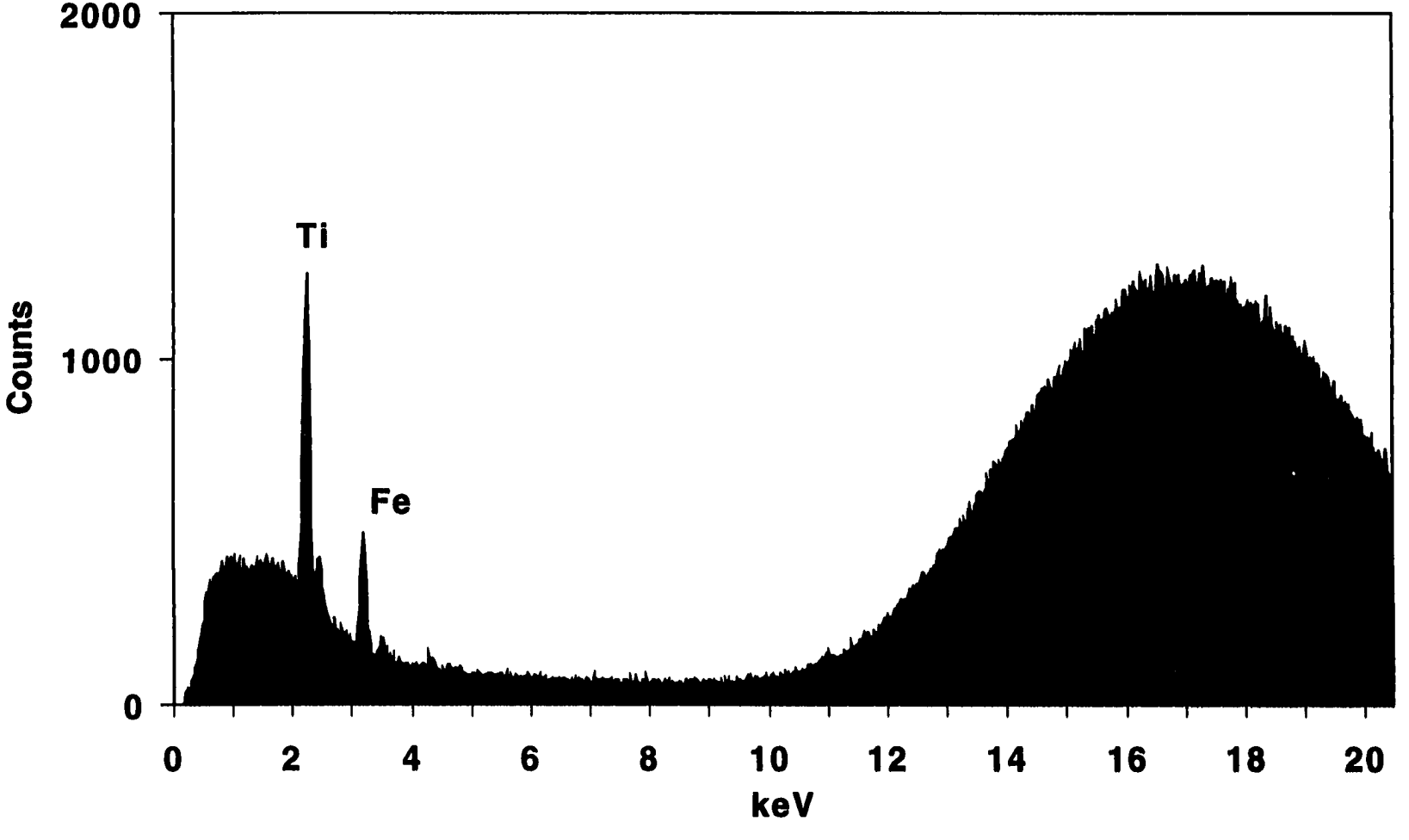


FIGURE 6b: TXRF W-*brems* excitation spectrum of a 30 mm diameter sample of Glad® black polyethylene trash bag on a sample support. Assay time is 200 live seconds.



support. Samples were oriented so that the long axis of the sample was parallel to the W-*brems* X-ray direction. The samples were held flat against the quartz support by their static charge. Example TXRF spectra using this sample preparation technique are shown in Figures 7a (Mo-K excitation) and 7b (W-*brems* excitation). Using a small sample does not offer significantly better results than a large piece of plastic bag placed directly on the support (Compare Figures 6a and 7a). The small sample approach overcomes the problem of sample loss to a second support, as in the hot-press preparation, but the sample is still too thick for TXRF. The resulting spectra display low peak-to-background ratios and only the major constituents Ti, Fe, and Zn were detected. Concerns previously discussed for the hot-press preparation regarding small sample size and the appropriateness of the internal standard apply to this technique as well.

2. Low temperature ashing in place

Low temperature plasma ashing in an oxygen environment is a suitable means of gently removing organic materials from polymers, leaving a residue rich in inorganic constituents. Low temperature ashing involves the generation of an oxygen gas plasma by radiofrequency (RF) electromagnetic radiation, in which free radicals and reactive species are formed. The free radicals and reactive species degrade organic material at low temperatures by using the energy of the plasma electrons rather than thermal energy to breakdown molecular oxygen. The oxidation reactions themselves are exothermic, which results in some heating of the samples. One advantage of low temperature plasma ashing is that most inorganics are not volatile at these low temperatures (on the order of 120°C). Further, since there is no combustion of the

FIGURE 7a: TXRF Mo-K excitation spectrum of a 3 mm^2 piece of Glad® black polyethylene trash bag on a quartz sample support. Assay time is 200 live seconds.

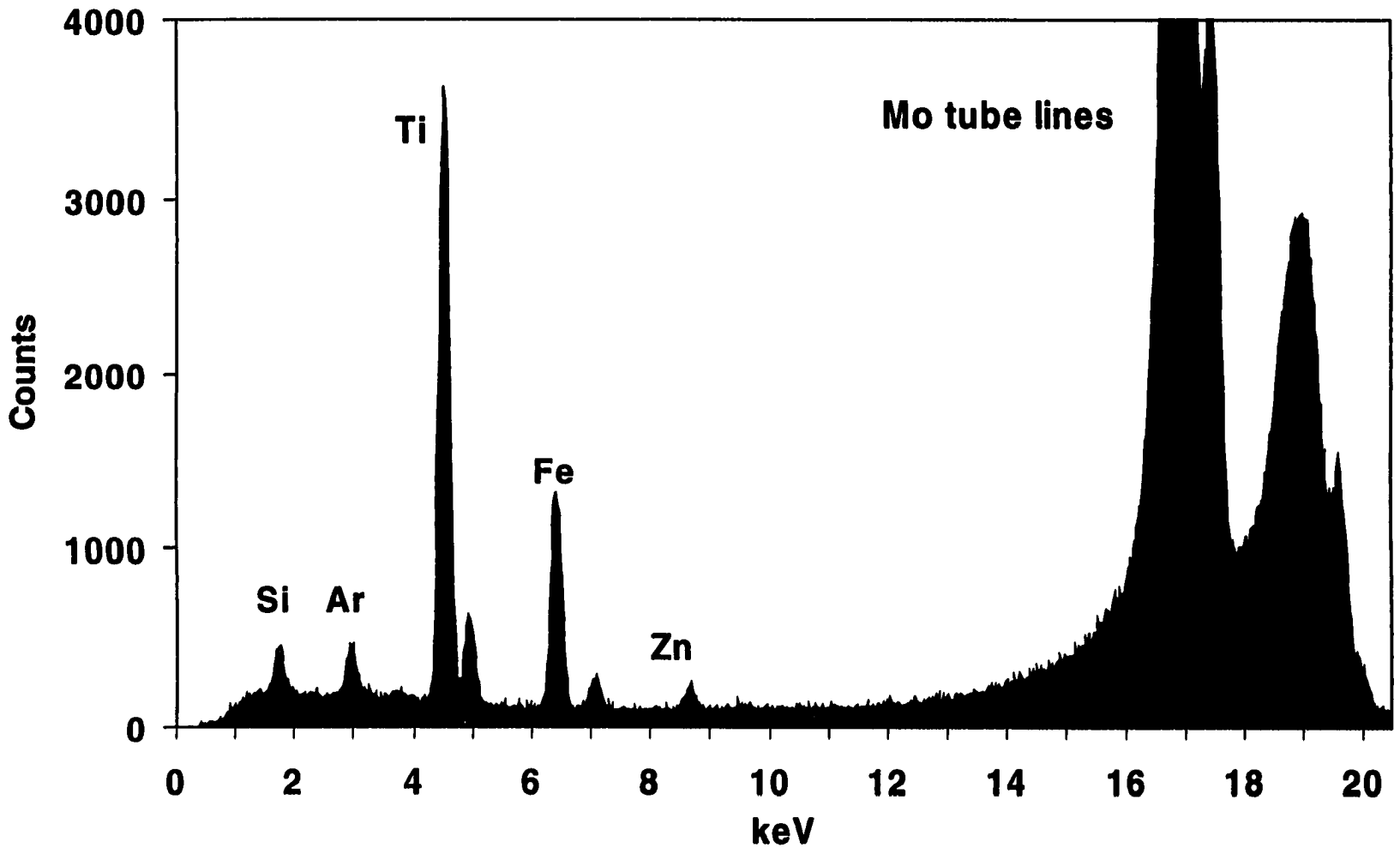
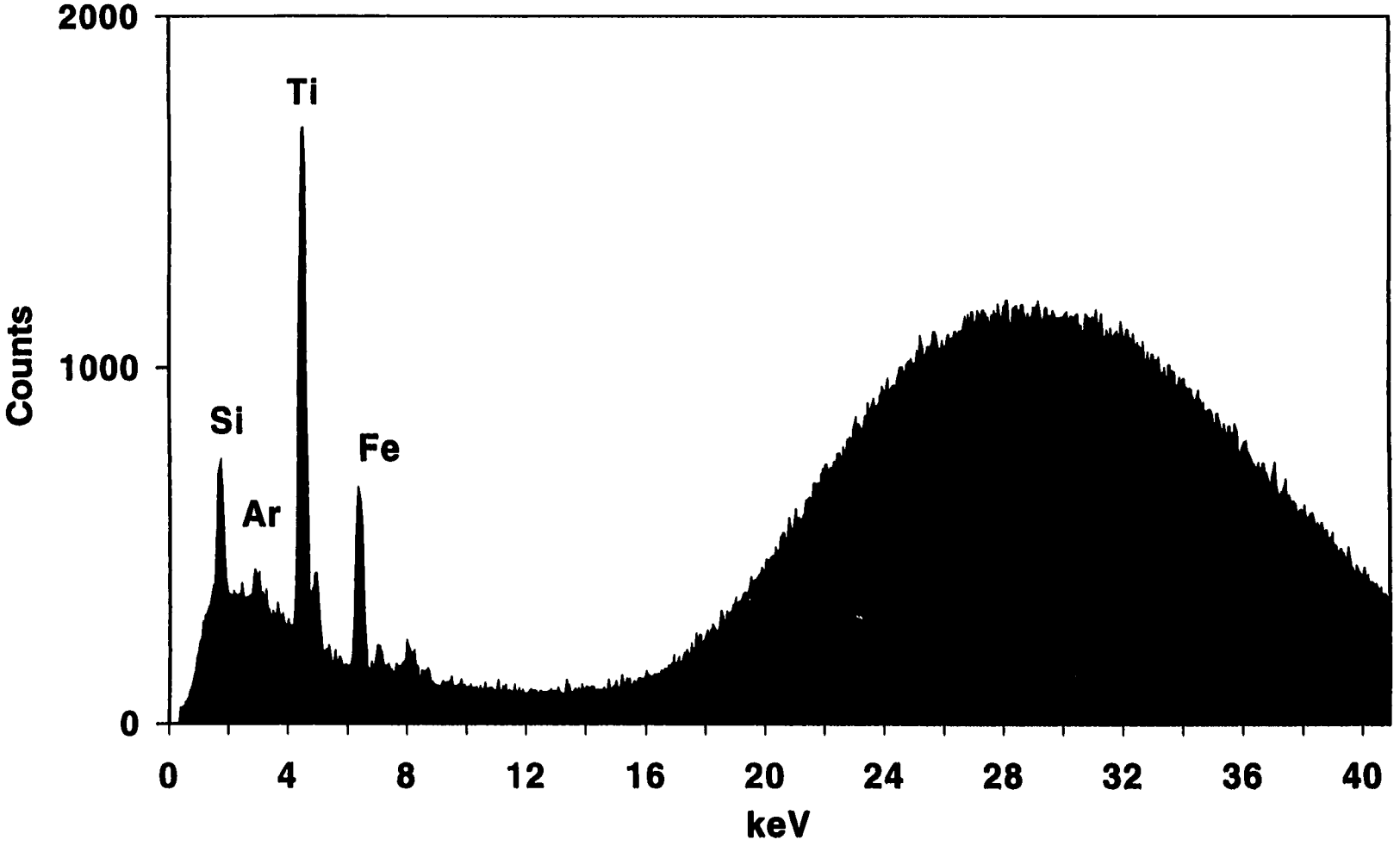


FIGURE 7b: TXRF W-*brems* excitation spectrum of a 3 mm^2 piece of Glad® black polyethylene trash bag on a quartz sample support. Assay time is 200 live seconds.



sample, cross contamination by particulate transfer is not a problem, provided care is taken to bring the samples back to atmospheric pressure slowly and to reduce static charges during transportation and handling of the sample ash.

In an attempt to reduce the sample thickness and increase sensitivity, a 3 mm^2 sample was placed directly on the sample support was subjected to oxygen plasma ashing. Since most of the plastic bag sample can be oxidized and removed as a gas, the total sample mass on the support is greatly reduced by this procedure. Prior to oxygen plasma ashing, 0.5 ng Y as an internal standard in 10% (v/v) HNO_3 was added on top of the plastic, followed by drying on a 70°C hot plate. Examples of the resulting TXRF spectra are shown in Figures 8a (Mo-K excitation) and 8b (W-*brems* excitation). The resulting TXRF spectra exhibit a lower background than either the hot-press or direct analysis of solid samples methods. This is due to the change in sample geometry and, in the case of the hot-press method, sample mass. Comparisons of spectra in Figures 7a with 8a, and Figures 7b with 8b, reveal the better peak-to-background ratios and lower X-ray scattering for the same sample mass with plasma ashing than without. However, in addition to concerns about the small sample size, problems with internal standard addition were encountered with this procedure. Addition of the internal standard prior to plasma ashing likely will not result in a homogeneous mixing of the internal standard and the resulting sample ash. Post-ashing internal standard addition was not suitable because the plasma ashing process removes the silicone layer treatment from the quartz surface. The absence of a silicone layer results in unacceptably large droplet spreading outside of the analytical area on the sample support.

FIGURE 8a: TXRF Mo-K excitation spectrum of a 3 mm^2 piece of Glad® black polyethylene trash bag plasma ashed directly on a quartz sample support. Assay time is 200 live seconds. Internal standard is 1 ng Y.

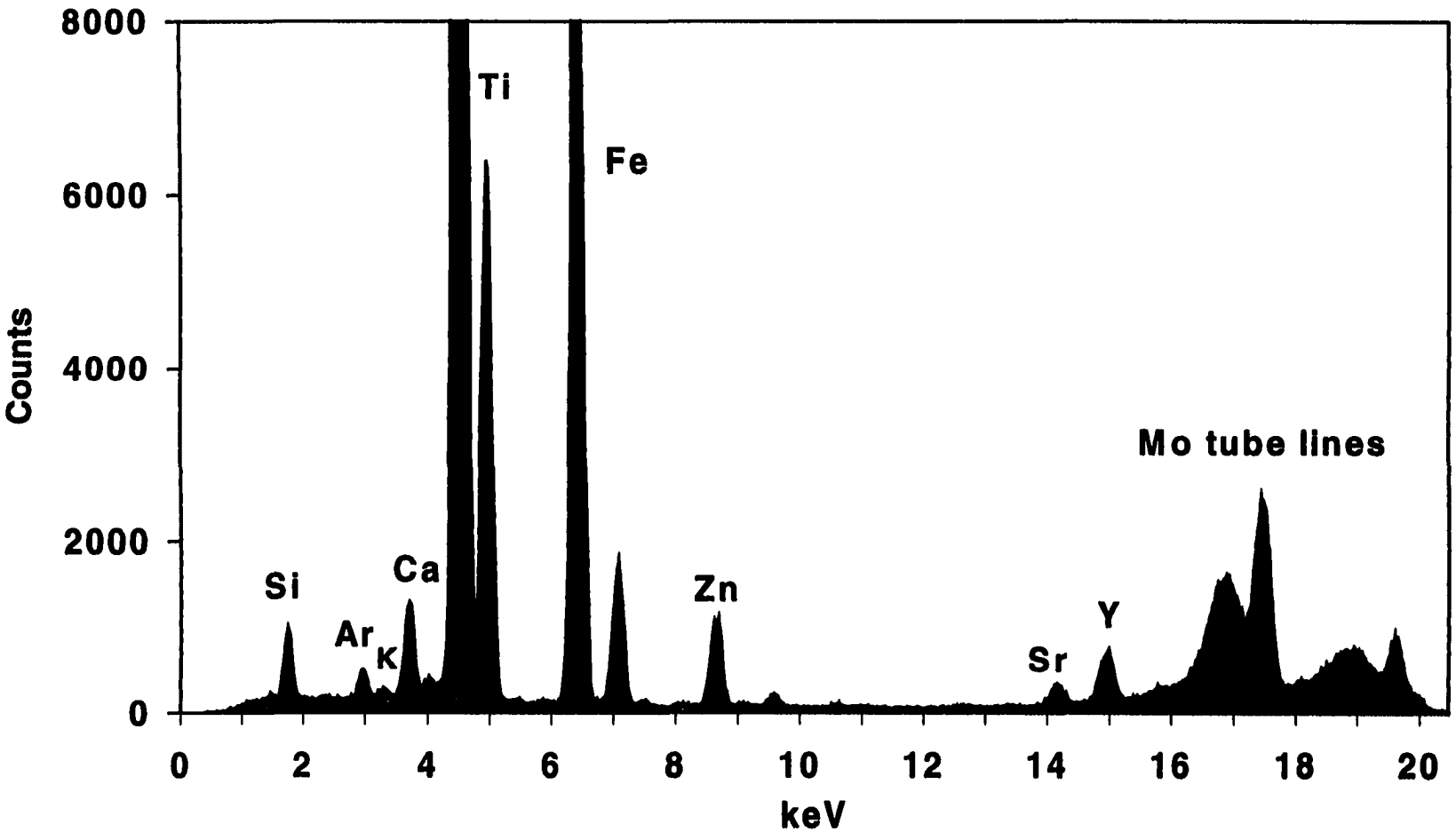
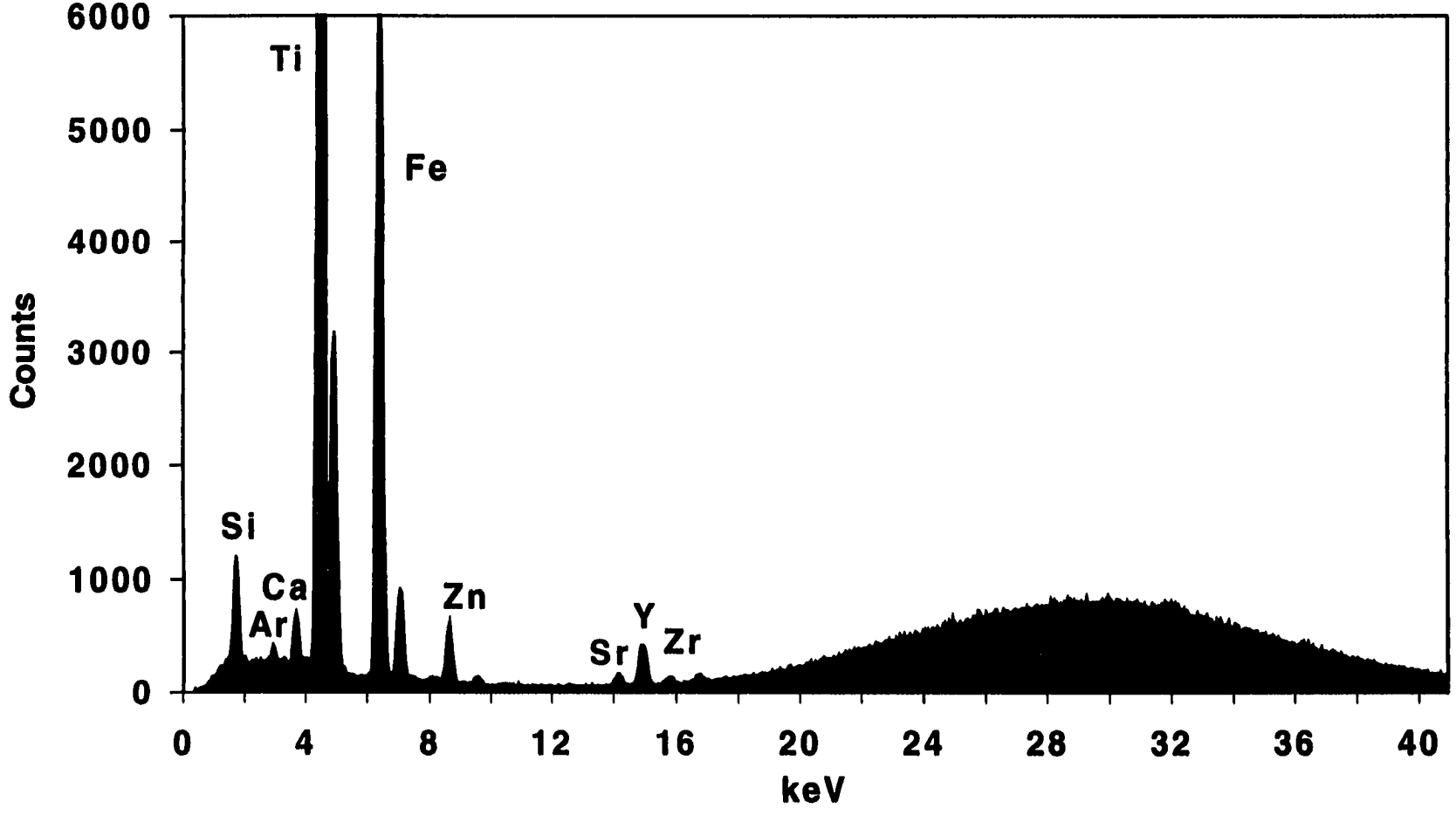


FIGURE 8b: TXRF W-*brems* excitation spectrum of a 3 mm^2 piece of Glad® black polyethylene trash bag plasma ashed directly on a quartz sample support. Assay time is 200 live seconds. Internal standard is 1 ng Y.



3. Organic solvents

Dissolution of a small polyethylene sample in several different organic solvents was attempted, including petroleum ether, chloroform, and methylene chloride, with no success. The selection of organic solvents was limited because of toxicity and the need to work in a laminar flow hood to minimize sample contamination. Polyethylene dissolved easily in hot xylene, but transfer of an aliquot of the dissolved sample to the quartz support was problematic. Polypropylene pipette tips, which were used rather than glass to minimize contamination of the sample from the pipette tip, tended to soften in the hot xylene. Further, when an aliquot of sample solution was withdrawn from the hot solution, it quickly began to gel in the pipette tip on cooling. Finally, the difficulties associated with obtaining and adding an appropriate internal standard disallowed the use of organic solvents for sample dissolution.

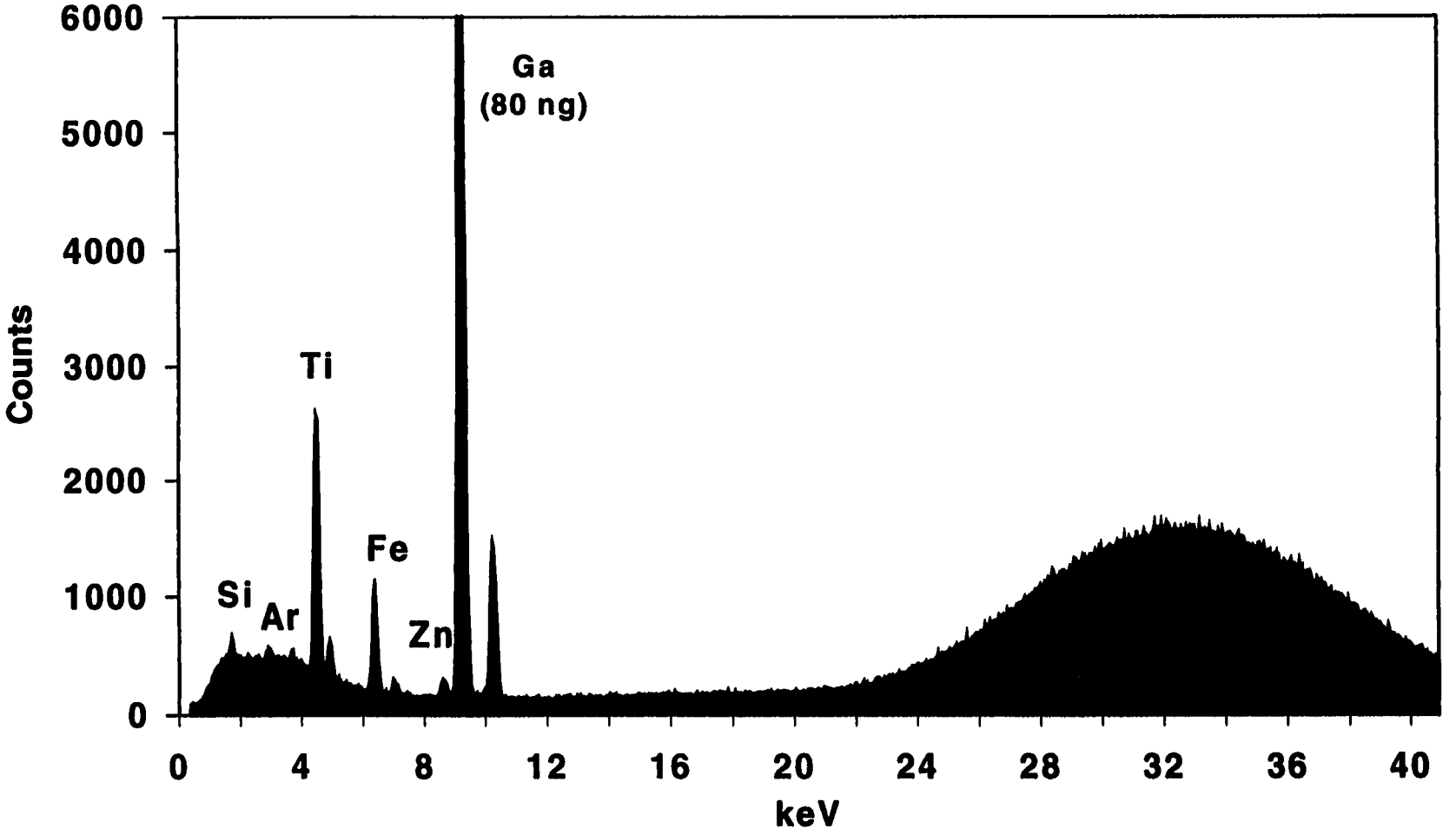
4. Acid Digestion

Although destructive in nature, digestion of the polymer sample to form an aqueous solution is desirable, as it facilitates internal standard addition and preparation of homogeneous samples for TXRF analysis. Several acid digestion methods were tried, including hot plate digestion with ultrapure concentrated HNO_3 and a combination of ultrapure concentrated HNO_3 and peroxide. The use of concentrated sulfuric acid, followed by concentrated HNO_3, is recommended in several microwave digestion procedures for polymers. This method was not attempted because it is difficult to obtain a dry sample, a requirement for TXRF, when using sulfuric acid. While it was possible to dissolve the polyethylene with concentrated HNO_3 by hot plate digestion, a 2 mg sample

required about 3 mL of concentrated HNO_3, added in 1 mL aliquots, for digestion. The resulting digestate was light brown in color and viscous. After the addition of an internal standard, a 20 μL aliquot was transferred onto a quartz TXRF sample support and dried on a hot plate at 70°C. The resulting TXRF spectrum exhibited unacceptably poor sensitivity, with small peaks on a very high background. This is mainly due to the large amount of organic components in the dried film and the aliquot size, which resulted in a low mass of inorganic material on the TXRF sample support. For this to be a suitable technique for TXRF sample preparation of polyethylene, a more concentrated solution is needed to increase the mass of inorganic materials on the sample support. In addition, a means of removing the organic material from the TXRF thin film is needed.

A microwave acid digestion procedure using HNO_3 was also attempted. The microwave procedure was capable of digesting a 2.5 mg sample of polyethylene in 2 mL of concentrated HNO_3, and produced a clear, colorless solution. While this procedure was quicker and required less HNO_3 than the hot plate method, the large volume of digest solution produced and small solution volume required by TXRF allow only a small percentage of the original sample to be analyzed. A TXRF spectrum for the plastic bag sample prepared in this manner is shown in Figure 9. The spectrum exhibits the same problems as those obtained by the hot plate methods, namely, the analyte mass is too low and the background is too high. However, this sample preparation method may be suitable for ICP-MS analysis of polyethylene samples.

FIGURE 9: TXRF W-*brems* excitation spectrum of a 10 µL aliquot of microwave digestate from a 2.5 mg plastic bag digested in 2 mL. Assay time is 1,000 live seconds. Internal standard is 80 ng Ga.



5. High temperature ashing

High temperature ashing in a muffle furnace was evaluated as a means to eliminate the polymer matrix prior to TXRF analysis. Samples of polyethylene bags of approximately 1 mg were cut, cleaned and placed into separate, acid-washed fused silica crucibles in a muffle furnace at 500°C for 6 hours. This method was not selected for further testing because the high temperature resulted in combustion of the polymer. This yielded cross contamination of samples within the furnace, most likely due to transfer of particulates by smoke. It is also suspected that the high temperature required to ash the polymer would result in loss of volatile inorganic components.

6. Low temperature plasma ashing

Low temperature plasma ashing in place on the sample support showed this to be a suitable means of reducing the sample mass. In an effort to produce a more homogeneous TXRF preparation with a larger sample size, studies using low temperature plasma ashing of 10 mg samples in 10 mL fused silica crucibles were conducted. The plasma asher was operated at an RF power of 100 watts, with an oxygen gas flow of 300 cm^3/min. The crucibles containing the ash residues were rinsed with 100 µL of 10% (v/v) ultrapure HNO_3, which contained 0.1 ppm yttrium (Y) as an internal standard. TXRF sample supports were prepared using 10 to 20 µL aliquots of this ash solution. This approach had several limitations. The large (10 mg) sample size was chosen to minimize the effects of local inhomogeneities that might occur in the blown plastic film. However, the large sample size required very long ashing times (6 hours minimum) to insure complete degradation of the polyethylene. Faster ashing times were achieved by

increasing the RF power and gas flow of the plasma asher, but this also increased the temperature. Higher temperatures may result in the loss of some analytes of interest. The most significant drawback, however, is that the ash residue is not completely soluble in 10% HNO_3, resulting in large variations in quantitative results among replicate sample preparations using aliquots of the same solution. Various means to dissolve the ash residue were investigated, including the use of concentrated, 50%, and 10% hydrochloric acid (HCl), different concentrations of HNO_3, and warming the nitric acid and ash residue in the crucible on a hot plate. The use of smaller sample sizes, followed by a quantitative transfer of the entire ash residue to the TXRF sample support, was investigated as a means of assuring that the TXRF preparation accurately represented the original sample. Preliminary studies indicated that this was a viable method for sample preparation of polyethylene for analysis by TXRF.

Several noteworthy revisions in the sample preparation method using low temperature oxygen plasma ashing occurred between the initial studies described above and the final method used in the survey portion of this research. Plasma ashing conditions were altered by increasing the RF power and gas flow slightly, which provided quicker ashing without the additional loss of analytes. Soda-lime-silica microbeakers (Cargille Laboratories, Inc., Cedar Grove, New Jersey) were used instead of fused silica crucibles for plasma ashing. These microbeakers are disposable and hold a total volume of 500 μL. Compared with the 10 mL volume of the crucibles, the small microbeakers permit the use of a smaller rinse volume, which allows nearly complete sample recovery on transfer. Finally, gallium (Ga) was chosen to replace Y as the internal standard used

for TXRF quantitation. The Ga K_α peak-to-background ratio is good for both Mo-K and W-*brems* excitation conditions, whereas Y K_α X-ray photo-peak lies on a rising background with Mo-K excitation.

TXRF spectra of a 15 mm^2 polyethylene film sample prepared in this manner using a spectral acquisition time of 1,000 live seconds are shown in Figures 10a and 10b. Use of the microbeaker for plasma ashing allows for a larger sample size than when plasma ashing is conducted directly on the sample support. The peak-to-background ratios in the TXRF spectra obtained were generally orders of magnitude better than previously explored sample preparation methods. Therefore, a study was undertaken to determine the optimum sample size for TXRF analysis using this preparation method. The sample preparation method that was used for this sample size study, and subsequent analyses throughout this project, is detailed in Figure 11.

7. Sample Size

A single black Glad® Quick-Tie™ polyethylene trash bag was selected for preparation and analysis to determine the optimum sample size for TXRF analysis using low temperature plasma ashing, followed by quantitative transfer of the ash residue to a TXRF sample support. Twelve samples of the bag, six from each side of the assembled bag, were prepared for each of four sample sizes. For each sample size category, samples were cut from the bag to approximately the same dimensions, cleaned, weighed and prepared according to the procedure in Figure 11. The ideal sample size is the largest sample size that gives good quality TXRF spectra. A large sample size minimizes the effects of local inhomogeneities and also increases the mass of analyte on the TXRF

FIGURE 10a: TXRF Mo-K excitation spectrum of a 15 mm^2 piece of Glad® black polyethylene trash bag plasma ashed in a microbeaker and transferred to a quartz sample support. Assay time is 1,000 live seconds. Internal standard is 10 ng Ga.

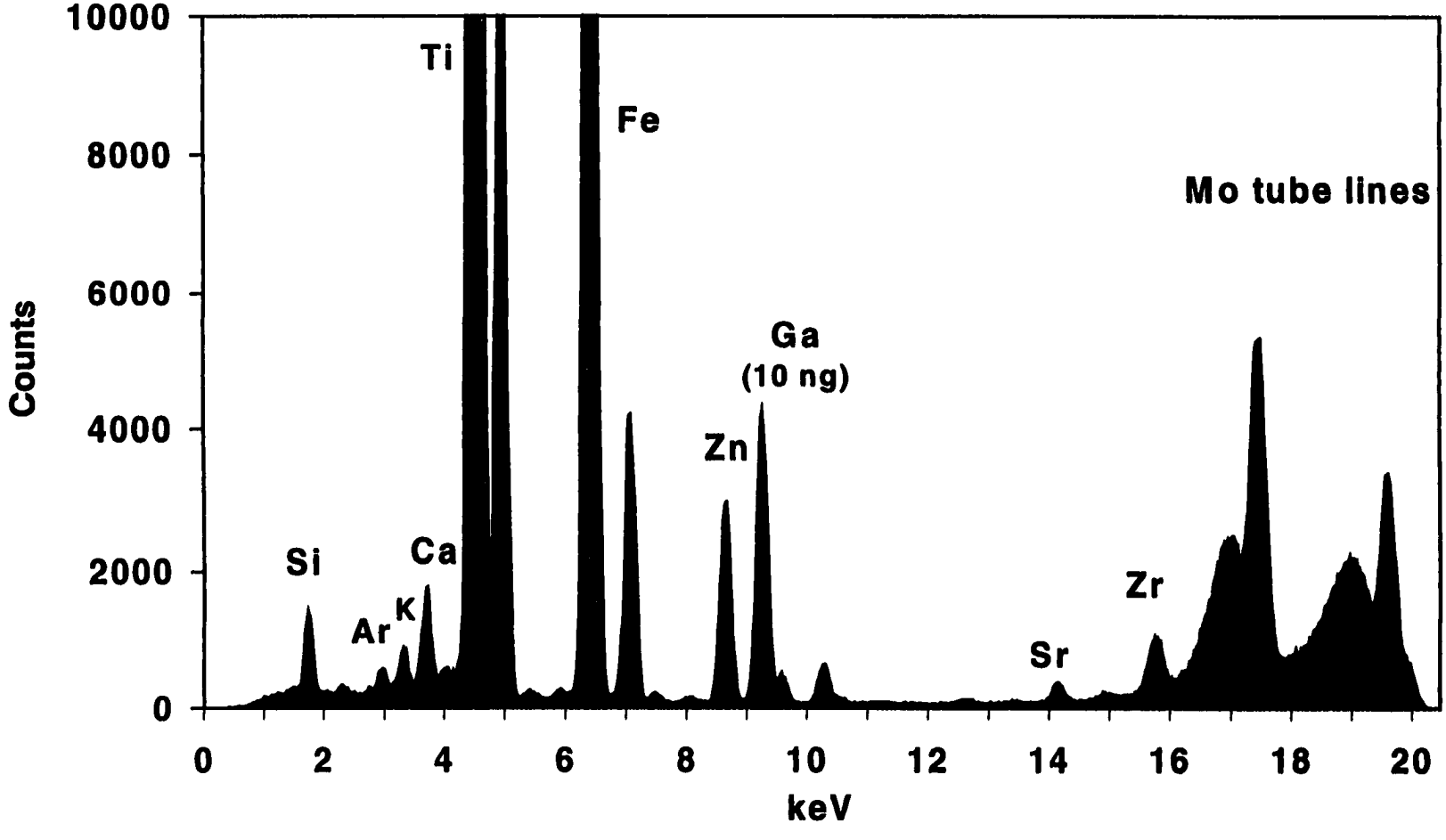


FIGURE 10b: TXRF W-*brems* excitation spectrum of a 15 mm^2 piece of Glad® black polyethylene trash bag plasma ashed in a microbeaker and transferred to a quartz sample support. Assay time is 1,000 live seconds. Internal standard is 10 ng Ga.

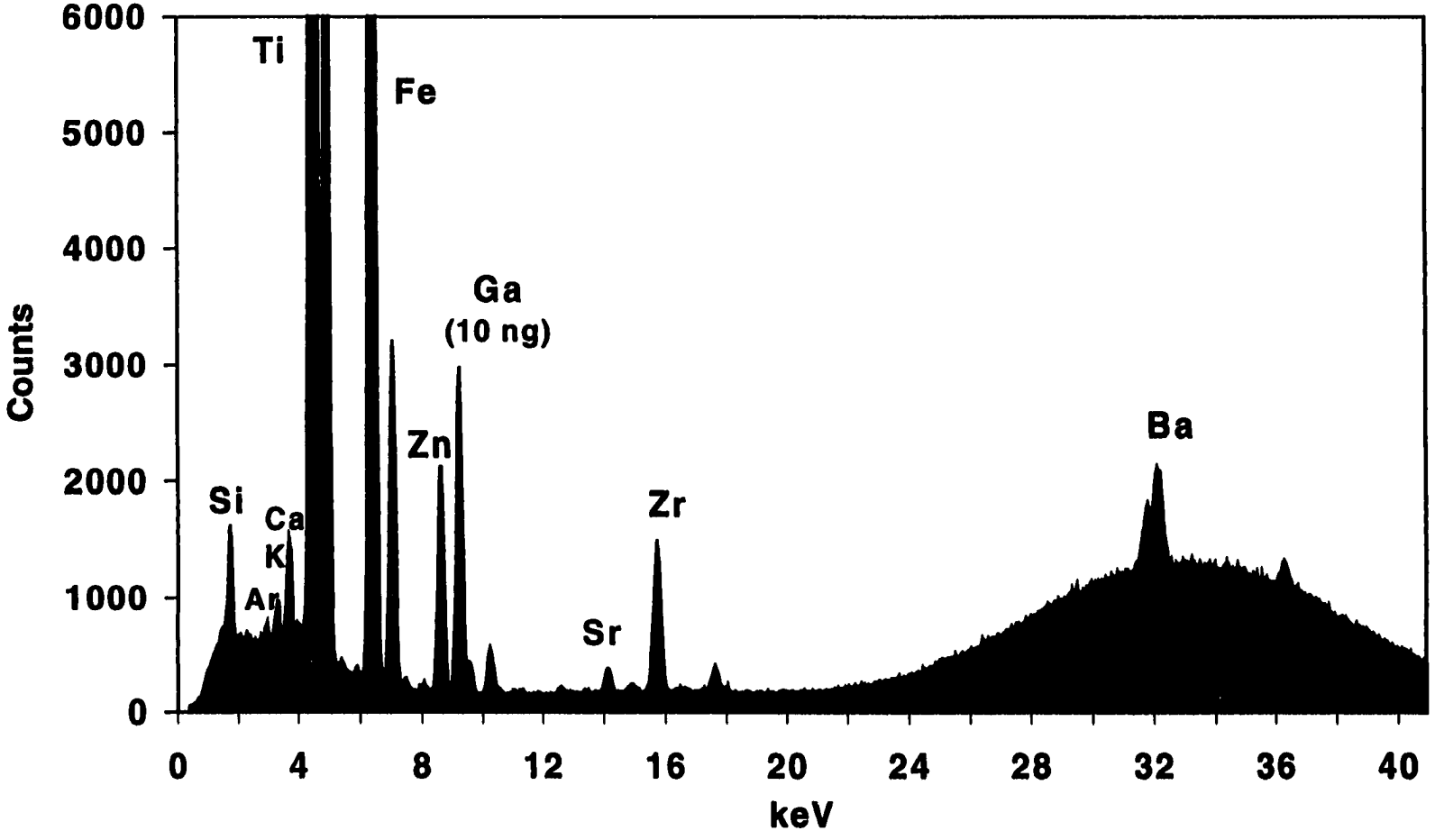
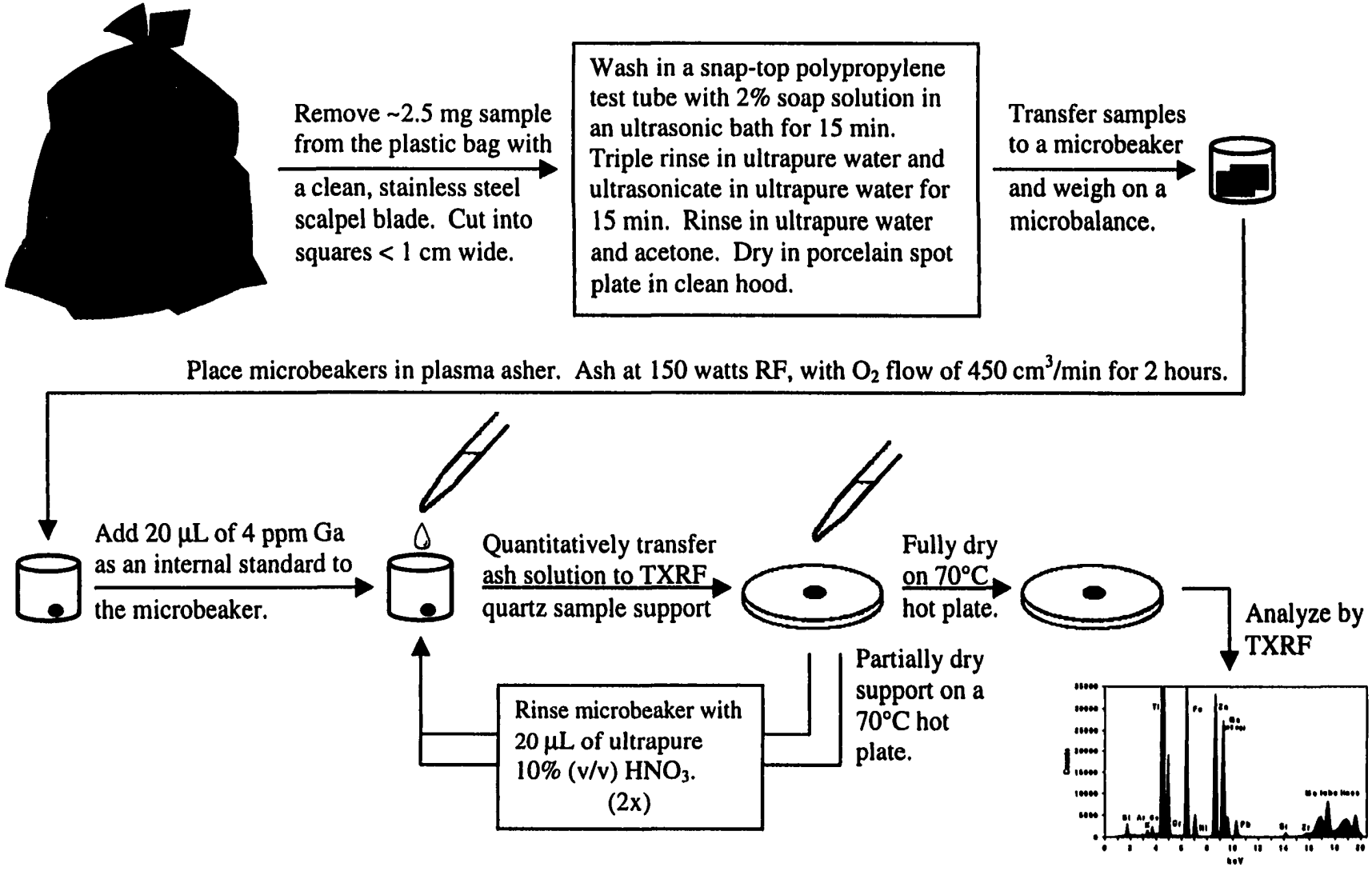


FIGURE 11: Preparation of plastic bag samples for analysis by TXRF.



support, yielding better X-ray counting statistics and quantitative data. However, if the original sample is too large, the TXRF preparation will be too thick, the thin film conditions for TXRF will not be satisfied and the quantitative calculations based on a single internal standard will be invalid. Further, the sample preparations must yield suitable X-ray count rates (≤40% dead time), preferably using a thick X-ray filter, since the background profile is better with the thick filter than with a thin one.

Results of the sample size study using W-*brems* excitation are given in Table 6. Results that are less than the level in the blank or not detected are indicated by "nd" in the table. Results that are above the limit of detection (LOD), as determined by the TXRF software using X-ray counting statistics for each element in each sample, are included in Table 6. These element results have a high percent RSD between the replicate samples, regardless of the sample size. Summary results are also provided in the table for all analyte elements in each sample size category. Determination of the optimum sample size was based on the quality of the resulting TXRF spectra, sample preparation thickness, X-ray count rates, and analytical precision. This evaluation was performed using the summary data in Table 6 for the analyte elements having good peak-to-background ratios. Figure 12 displays the analytical precision for six elements and one element ratio as a function of average sample size for each size group studied with W-*brems* excitation. The RSDs for all elements in general, and for Ca in particular, are unacceptably high for samples weighing less than 1 mg. For most elements, precision is less than 10% at the 2 mg sample size and, with the exception of Zn, continues to improve with sample size until the optimum precision is reached at 3.3 mg or greater.

TABLE 6: TXRF analytical results for four sizes of samples taken from the same plastic bag. Excitation mode is W-*brems*. Assay time is 1,000 live seconds. Results are in µg/g. Sample sizes are (a) 15 mm^2, average weight of 0.34 mg; (b) 50 mm^2, average weight of 0.90 mg; (c) 100 mm^2, average weight of 2.0 mg; and (d) 150 mm^2, average weight of 3.3 mg.

(a)

Sample #	S	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu
1	161.243	88.666	210.472	4637.733	nd	nd	546.812	0.374	1.952
2	nd	51.470	69.589	4848.972	nd	nd	564.624	1.722	0.425
3	281.352	37.831	291.533	4944.531	5.751	0.503	1439.862	0.735	0.475
4	377.607	45.270	401.806	4824.879	nd	nd	576.803	0.594	0.175
5	177.112	50.680	261.417	4412.563	nd	0.083	529.041	0.615	0.146
6	864.289	41.830	1368.338	4878.858	nd	0.062	601.012	0.669	0.348
7	271.368	31.930	341.692	5029.418	nd	nd	599.219	0.601	0.696
8	16.652	43.736	98.607	5097.609	nd	nd	598.062	0.760	0.255
9	nd	45.671	76.490	4956.267	nd	nd	586.469	0.665	0.439
10	28.507	39.503	109.336	4087.868	nd	nd	490.639	0.693	0.264
11	101.951	78.078	103.293	4288.852	nd	nd	513.131	0.549	0.479
12	604.908	35.190	572.670	5185.711	nd	nd	625.304	0.855	0.583
Mean =	240.416	49.155	325.437	4766.105	0.479	0.054	639.248	0.736	0.520
Std. Dev. =	266.893	17.132	362.850	340.531	1.660	0.144	255.285	0.333	0.479
% RSD =	111%	35%	111%	7.1%	346%	267%	40%	45%	92%

(b)

Sample #	S	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu
1	nd	36.440	80.186	5160.096	5.511	1.343	644.834	1.343	9.153
2	nd	30.123	57.761	5188.329	7.953	1.301	659.387	0.370	0.636
3	nd	33.124	34.539	5156.893	5.701	1.147	638.389	0.769	0.690
4	137.689	50.036	416.852	6024.738	7.092	2.485	778.756	0.323	0.832
5	nd	27.865	nd	3949.500	4.217	1.478	502.224	0.447	0.924
6	193.778	27.511	261.707	3431.656	3.342	1.395	435.348	0.353	0.317
7	nd	35.012	44.231	4771.969	3.688	0.944	582.363	0.744	0.553
8	nd	34.405	17.619	5200.740	7.532	1.486	667.944	0.746	0.611
9	nd	41.798	44.748	4769.081	7.223	1.401	612.814	1.275	97.796
10	34.339	33.576	168.026	3652.590	1.348	0.811	450.411	0.627	0.566
11	nd	33.406	2.898	4494.711	5.873	0.745	567.352	0.534	0.971
12	nd	32.365	33.396	4655.547	4.847	1.109	584.780	0.598	0.692
Mean =	30.484	34.638	96.830	4704.654	5.361	1.304	593.717	0.678	9.478
Std. Dev. =	65.043	6.171	125.889	737.773	1.974	0.450	97.200	0.334	27.920
% RSD =	213%	18%	130%	16%	37%	34%	16%	49%	295%

TABLE 6: (Continued)

(a)

Sample #	Zn	Rb	Sr	Zr	Pb	Ba	Sn	Ag	Y	Ti/Fe
1	34.324	0.235	1.672	4.440	1.154	14.189	0.116	0.051	0.076	8.481
2	23.012	0.247	1.290	5.793	1.173	13.810	0.051	nd	0.370	8.588
3	29.320	0.180	1.306	2.283	1.265	12.918	0.211	0.159	0.098	3.434
4	27.713	0.181	1.320	1.139	0.965	13.916	0.189	0.100	0.107	8.365
5	31.757	0.141	1.243	7.608	0.911	13.026	0.126	0.071	0.336	8.341
6	42.212	0.084	1.968	3.382	1.263	14.874	0.037	0.042	0.179	8.118
7	27.858	0.225	1.425	1.400	1.121	15.106	nd	0.004	0.164	8.393
8	22.358	0.216	1.266	1.767	1.155	14.230	0.172	0.093	0.095	8.524
9	23.513	0.179	1.331	2.770	1.040	15.416	0.253	nd	0.067	8.451
10	24.212	0.104	1.284	4.861	0.998	14.103	0.462	0.040	0.250	8.332
11	26.651	0.167	1.039	2.621	1.126	13.592	1.090	0.016	0.100	8.358
12	35.147	0.152	1.616	2.374	0.758	17.069	0.004	0.133	0.119	8.293
Mean =	29.006	0.176	1.397	3.370	1.077	14.354	0.226	0.059	0.164	7.973
Std. Dev. =	5.954	0.051	0.245	1.948	0.149	1.141	0.301	0.053	0.103	1.435
% RSD =	21%	29%	18%	58%	14%	8.0%	133%	90%	63%	18%

(b)

Sample #	Zn	Rb	Sr	Zr	Pb	Ba	Sn	Ag	Y	Ti/Fe
1	33.882	0.194	1.419	1.215	1.147	16.974	1.263	0.162	0.131	8.002
2	28.958	0.125	1.230	1.344	0.945	16.086	0.070	0.079	0.160	7.868
3	31.675	0.113	1.984	1.171	1.078	16.544	0.323	0.096	0.154	8.078
4	43.124	0.150	2.977	1.713	1.235	21.101	0.218	0.090	0.205	7.736
5	24.547	0.112	1.035	0.824	1.164	13.776	0.033	nd	0.093	7.864
6	26.862	0.130	1.015	0.899	1.006	13.484	0.129	0.002	0.078	7.883
7	30.981	0.162	4.020	0.866	1.214	16.959	nd	0.046	0.099	8.194
8	28.576	0.158	1.381	1.410	1.133	18.078	0.024	0.047	0.150	7.786
9	26.846	0.188	1.193	0.958	1.247	16.603	0.183	0.057	0.112	7.782
10	30.794	0.094	1.051	0.778	0.786	12.882	0.050	0.040	0.058	8.109
11	27.006	0.126	1.206	1.080	0.926	15.949	0.133	0.047	0.106	7.922
12	27.776	0.086	1.184	1.686	1.076	16.370	0.144	0.074	0.133	7.961
Mean =	30.086	0.137	1.641	1.162	1.080	16.234	0.214	0.062	0.123	7.932
Std. Dev. =	4.845	0.034	0.932	0.322	0.141	2.202	0.343	0.044	0.040	0.142
% RSD =	16%	25%	57%	28%	13%	14%	160%	71%	33%	1.8%

TABLE 6: (Continued)

(c)

Sample #	S	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu
2	9.572	21.006	73.309	3773.379	5.932	1.328	482.211	1.906	0.383
3	42.057	27.667	106.931	4581.558	5.836	1.537	580.963	1.412	0.715
6	29.644	23.461	80.733	4377.987	4.467	2.001	559.921	0.614	0.428
7	28.451	29.926	81.355	4113.740	5.659	1.558	533.569	0.564	0.536
8	26.765	40.487	73.245	4017.051	6.112	1.668	528.671	0.661	0.804
9	3.351	21.289	79.600	3957.251	6.486	0.947	517.276	0.665	0.574
10	nd	32.158	76.121	4262.304	7.570	1.366	562.512	0.997	0.959
11	111.948	35.121	124.846	4901.351	5.223	1.500	617.921	1.160	0.702
12	24.590	17.386	64.381	3898.598	8.545	0.994	515.314	0.354	1.288
Mean =	30.709	27.611	84.502	4209.246	6.203	1.433	544.262	0.926	0.710
Std. Dev. =	33.403	7.530	19.059	361.414	1.221	0.327	40.505	0.494	0.283
% RSD =	109%	27%	23%	8.6%	20%	23%	7.4%	53%	40%

(d)

Sample #	S	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu
1	7.043	33.896	113.201	4996.206	5.038	1.888	629.291	0.684	0.775
2	40.043	31.728	134.812	4920.709	7.013	0.705	631.672	0.671	0.941
3	29.294	24.849	92.057	4728.946	6.535	1.608	611.042	0.329	0.454
4	81.120	26.768	125.278	4484.772	6.378	1.273	588.856	0.424	0.380
5	nd	21.232	74.471	4457.523	7.588	1.493	584.217	0.164	0.505
6	nd	26.113	82.476	4890.166	6.870	0.916	629.763	0.244	0.655
7	8.143	24.968	87.963	4781.486	4.410	1.828	616.720	0.628	0.592
8	63.132	21.682	93.260	4760.048	5.652	1.343	610.268	0.376	0.683
9	nd	27.964	85.472	4900.381	5.691	1.540	625.638	0.392	0.682
10	24.182	20.978	123.072	4267.727	7.085	1.416	569.481	0.259	0.735
11	43.201	30.094	81.893	4543.147	4.705	1.381	585.880	0.251	0.449
12	13.015	22.646	94.298	4748.056	7.288	1.118	615.457	0.184	0.540
Mean =	25.764	26.076	99.021	4706.597	6.188	1.376	608.191	0.384	0.616
Std. Dev. =	26.626	4.227	19.839	222.019	1.066	0.344	20.996	0.185	0.161
% RSD =	103%	16%	20%	4.7%	17%	25%	3.5%	48%	26%

TABLE 6: (Continued)

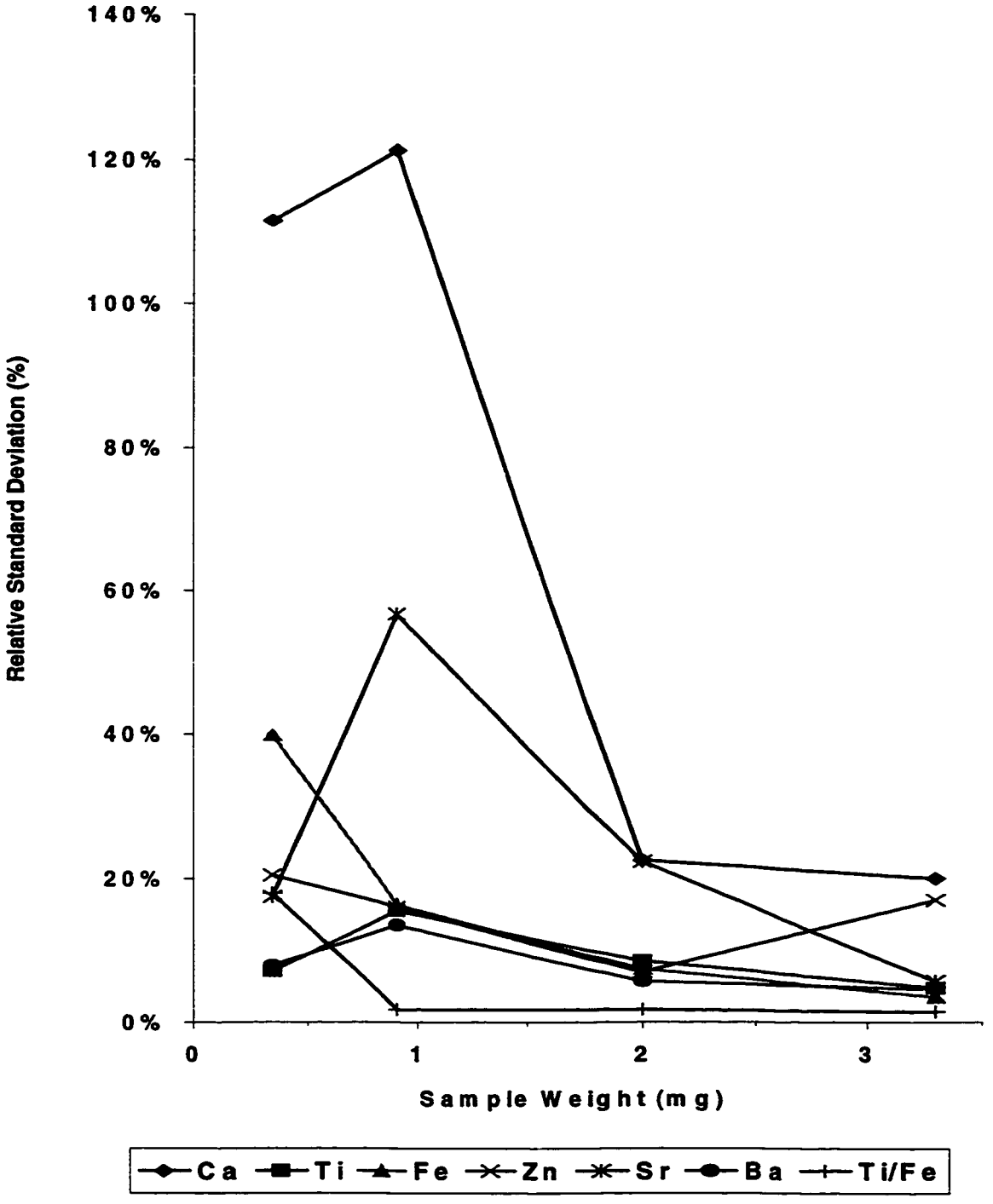
(c)

Sample #	Zn	Rb	Sr	Zr	Pb	Ba	Sn	Ag	Y	Ti/Fe
2	23.662	0.075	0.942	9.521	0.937	15.611	0.974	0.085	0.662	7.825
3	26.936	0.129	1.155	7.020	1.085	17.260	0.453	0.065	0.444	7.886
6	23.010	0.167	1.120	2.963	0.983	17.052	0.151	0.067	0.234	7.819
7	23.818	0.127	1.168	1.774	0.992	17.303	nd	0.039	0.301	7.710
8	24.581	0.169	1.775	1.362	0.942	17.427	0.072	0.067	0.053	7.700
9	23.806	0.087	1.064	3.013	0.980	15.772	0.187	0.056	0.279	7.650
10	23.446	0.133	1.014	1.729	0.879	17.082	0.143	0.062	0.111	7.577
11	27.604	0.113	1.221	2.005	1.181	17.832	0.308	0.105	0.181	7.932
12	23.094	0.081	0.897	1.595	0.879	15.174	0.076	0.074	0.075	7.565
Mean =	24.440	0.120	1.151	3.443	0.984	16.724	0.262	0.069	0.260	7.741
Std. Dev. =	1.677	0.035	0.257	2.866	0.097	0.944	0.299	0.018	0.195	0.132
% RSD =	6.9%	29%	22%	83%	9.8%	5.6%	114%	27%	75%	1.7%

(d)

Sample #	Zn	Rb	Sr	Zr	Pb	Ba	Sn	Ag	Y	Ti/Fe
1	41.684	0.099	1.279	1.154	1.039	18.387	0.175	0.106	0.129	7.939
2	30.140	0.170	1.303	1.039	1.004	18.453	0.185	0.092	0.108	7.790
3	25.737	0.099	1.264	0.855	0.939	16.062	0.108	0.055	0.139	7.739
4	27.609	0.147	1.220	0.971	1.070	18.403	0.051	0.030	0.121	7.616
5	23.447	0.095	1.145	0.849	0.887	17.401	nd	0.014	0.102	7.630
6	25.911	0.129	1.237	0.969	0.991	18.991	nd	0.069	0.105	7.765
7	27.082	0.142	1.263	1.070	1.051	19.117	0.098	0.012	0.147	7.753
8	25.850	0.166	1.205	0.953	1.013	18.979	nd	0.060	0.074	7.800
9	28.558	0.161	1.216	2.494	0.958	18.402	0.195	0.118	0.116	7.833
10	25.126	0.143	1.437	1.070	0.971	18.313	0.083	0.057	0.091	7.494
11	25.567	0.116	1.222	0.893	0.963	17.741	0.022	0.039	0.115	7.754
12	26.182	0.179	1.276	0.960	1.088	18.309	nd	0.063	0.104	7.715
Mean =	27.741	0.137	1.256	1.106	0.998	18.213	0.076	0.060	0.113	7.736
Std. Dev. =	4.716	0.030	0.071	0.447	0.058	0.836	0.076	0.034	0.020	0.114
% RSD =	17%	22%	5.7%	40%	5.9%	4.6%	100%	56%	18%	1.5%

FIGURE 12: Graph of the precision of plastic bag analytical results for six elements and one elemental ratio by TXRF W-*brems* excitation as a function of sample size. Samples of various sizes were taken from locations across a single, Glad® black polyethylene trash bag.



Practical factors, including the desire to transfer the entire analytical sample to the support, potential ash residue loss on the more heavily loaded preparations, and analyte mass limitations to achieve dead times less than or equal to 40%, resulted in selection of a sample size of 2.5 to 3 mg for the remainder of the project.

E. Analytical Conditions

Based on the results of the above studies, a set of analytical conditions was established and used for the sample preparation and analysis for the plastic bag survey portion of this research. These analytical conditions are specified in Table 7. TXRF spectra for a single Glad® Quick-Tie™ trash bag analyzed with these analytical conditions are in Figures 13a (Mo-K excitation), 13b (W-*brems* excitation), and 13c (W-L excitation).

TABLE 7: Analytical Conditions for TXRF Analysis of Plastic Bags

Sample size:	2.5 – 3 mg
Sample container:	Soda-lime-silica microbeaker (0.5 mL)
Plasma Ashing Conditions:	Branson/IPC S3003 Automatic Plasma System RF Power = 150 watts Gas Flow = 450 cm^3/min O_2 Ash time = 2 hours

TXRF support preparation conditions:

Sample support:	Suprasil quartz, 30 mm diameter
Internal standard:	80 ng Ga (20 µL of 4 ppm Ga in 10% (v/v) HNO_3)
Rinse / transfer solution:	40 µL 10% (v/v) HNO_3 in 2, 20 µL aliquots
Sample drying:	Hot plate at 70°C

TXRF Analytical Conditions:

	Mo-K_α	*W-brems*	W-L_α
Energy Range:	0 - 20 keV	0 – 40 keV	0 – 10 keV
Voltage:	50 kV	50 kV	25 kV
Current (maximum)*:	38 mA	38 mA	20 mA
Filter (thick):	150 µm Mo	100 µm Ni	30 µm Cu
Detector:	80 mm^2 Si(Li) area		
Detector resolution:	168 eV FWHM for 5.989 keV (Mn)		
Assay Time:	1,000 live seconds		

* The current was altered by the instrument software automatically to achieve a dead time ≤40% for each analysis.

FIGURE 13a: TXRF Mo-K excitation spectrum of a 2.5 mg sample of Glad® black polyethylene trash bag plasma ashed in a microbeaker and transferred to a quartz sample support. Assay time is 1,000 live seconds. Internal standard is 80 ng Ga.

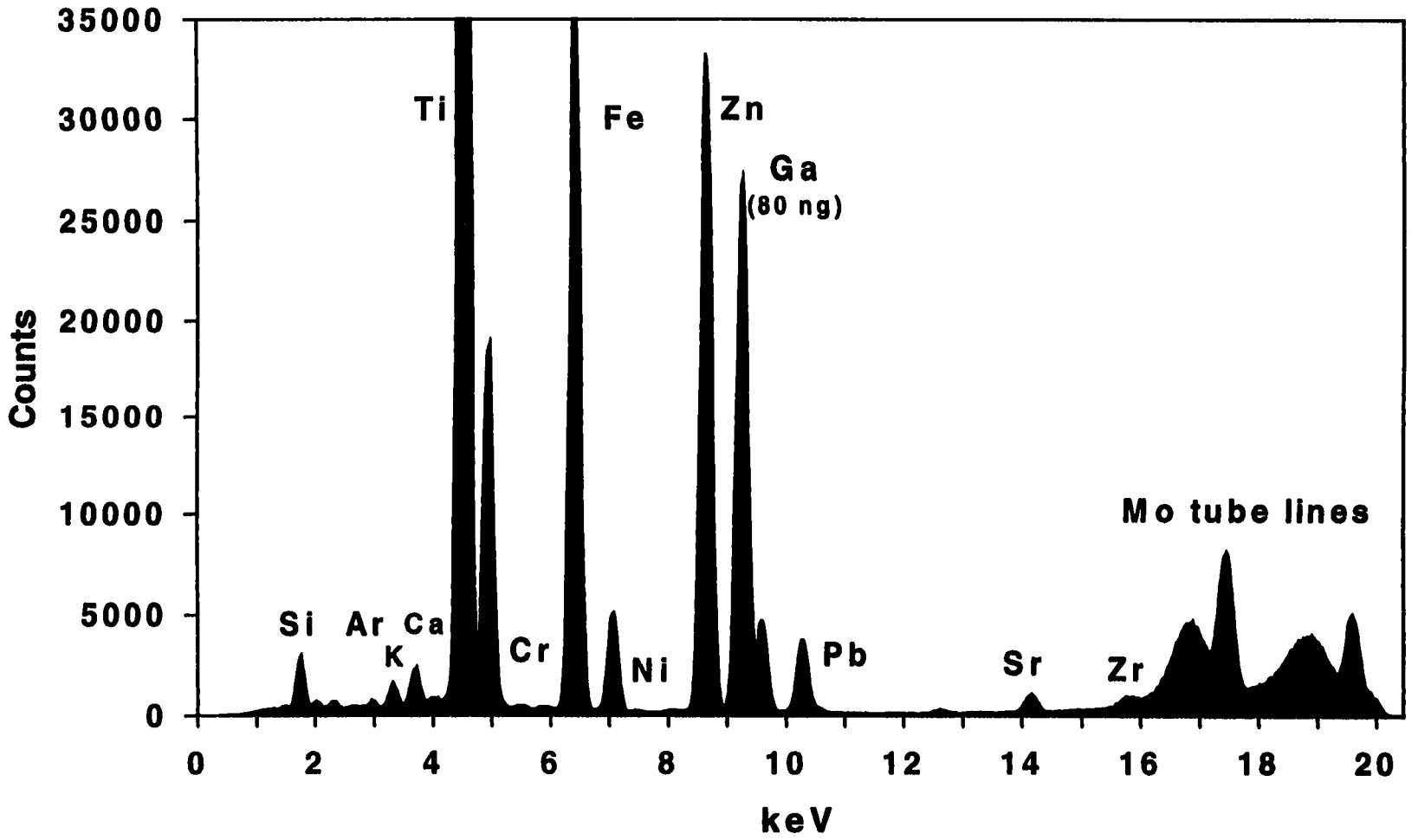


FIGURE 13b: TXRF W-*brems* excitation spectrum of a 2.5 mg sample of Glad® black polyethylene trash bag plasma ashed in a microbeaker and transferred to a quartz sample support. Assay time is 1,000 live seconds. Internal standard is 80 ng Ga.

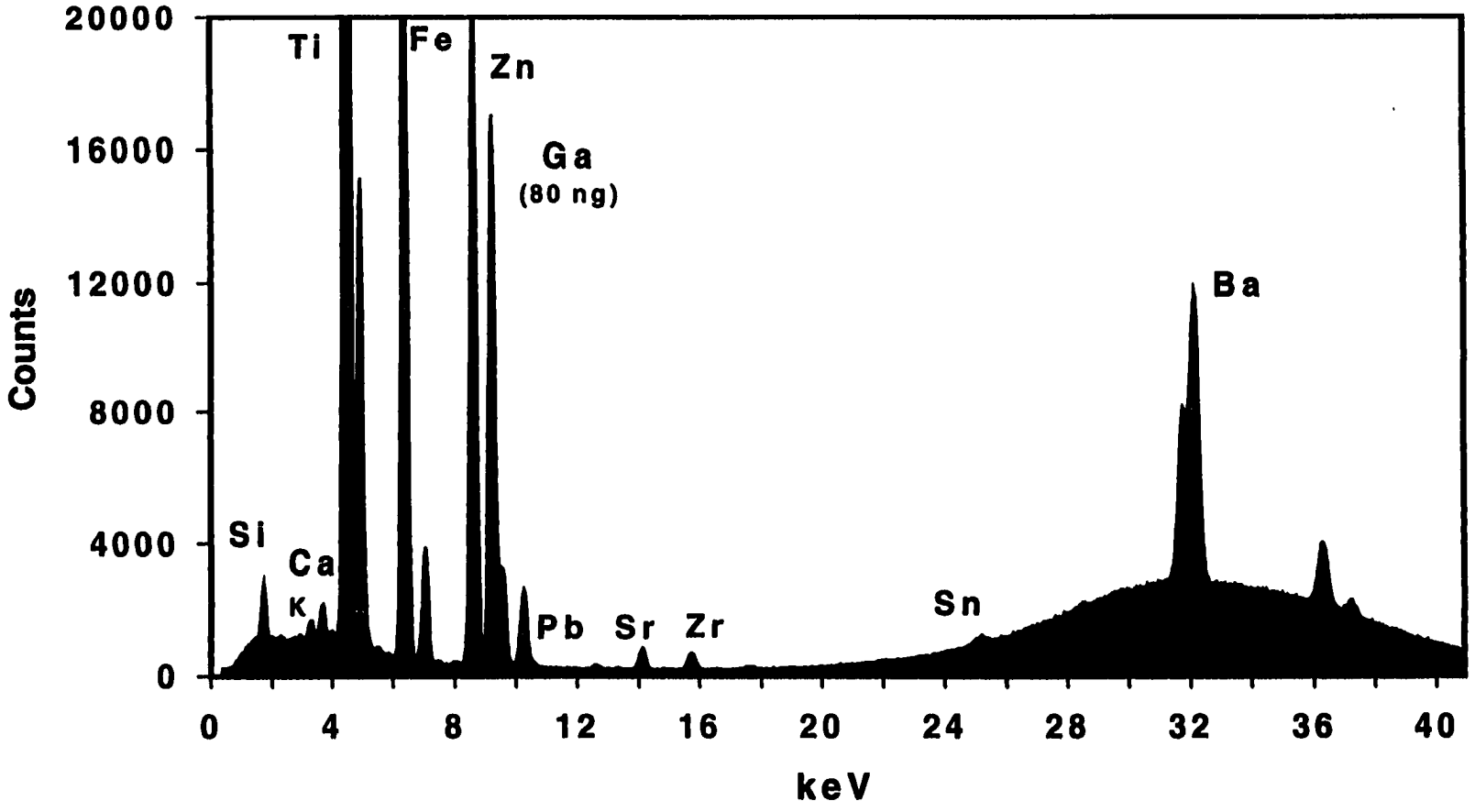
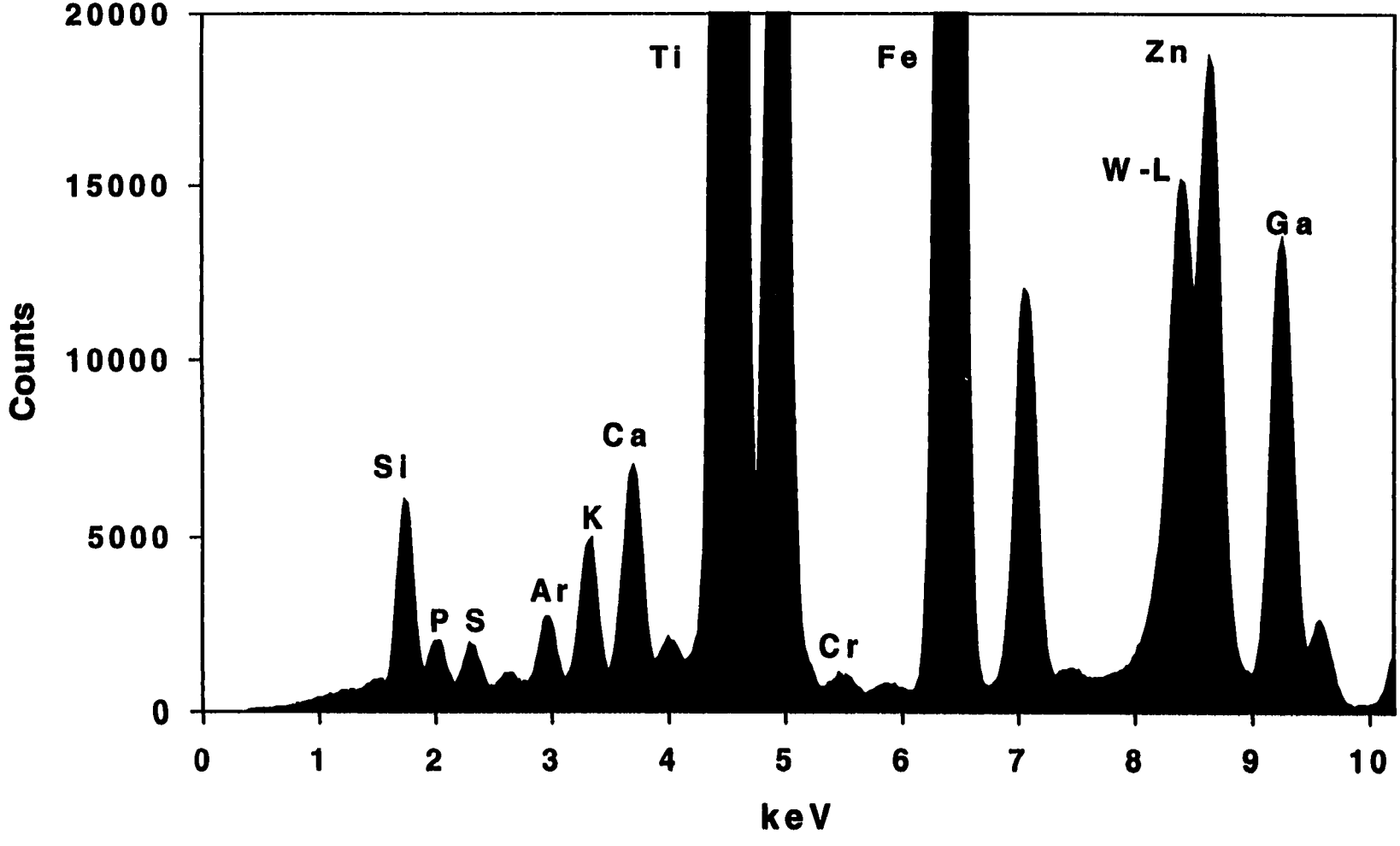


FIGURE 13c: TXRF W-L excitation spectrum of a 2.5 mg sample of Glad® black polyethylene trash bag plasma ashed in a microbeaker and transferred to a quartz sample support. Assay time is 1,000 live seconds. Internal standard is 80 ng Ga.



CHAPTER V. PLASTIC BAG SURVEY SAMPLE COLLECTION AND IDENTIFICATION

The plastic bags analyzed in this study are black, 30-gallon trash bags, which were purchased from local supermarkets. Plastic bags sampled and analyzed in the method development study previously discussed were purchased in June, 1997. These bags were also included in the survey study. The majority of the bags or boxes of bags in the survey portion of this study were purchased on the same day in June 1998. All of the boxes with identical packaging codes from the same manufacturer were purchased together from the same store. The packages of bags purchased were from three different manufacturers, First Brands Corporation (Glad®), Tenneco Packaging, Inc. (Hefty®), and Carlisle Plastics, Inc. (Ruffies™). The product lines sampled include Glad® Quick-Tie™, Hefty® EasyFlaps™, and Ruffies™Pro Trash Bags. Table 8 lists the identity of each package of plastic bags that were analyzed as part of this study.

A system of sample codes was used to simplify sample identification during this research project. The sample identification codes are described in Table 9. These code numbers are used later in the tables of analytical data to identify sample sources. Prior to sampling, the packaging code for each box of bags purchased was recorded and assigned a lot number identifier (A, B, C, etc.) for use in the sample identification code. The sample numbers corresponding to the locations of the analytical samples taken from each bag are identified in Figure 14. An example of the sample identification code is "GB16-1", which represents Glad® Quick-Tie™ bags, lot B, box #1, bag #6, and an analytical sample from region #1.

TABLE 8: Identification of the sources of plastic bags analyzed.

Manufacturer	Product Line	Lot Number	Package Size	Bag thickness	Purchase Date	Packaging Date	Source Code
First Brands	Glad® Quick-Tie™ Flaps	C8C63041	10	0.85 mil	June 1997	October 31, 1996	GA1
First Brands	Glad® Quick-Tie™ Flaps	108C7322	10	0.85 mil	June 1998	November 18, 1997	GB1
First Brands	Glad® Quick-Tie™ Flaps	108C7322	10	0.85 mil	June 1998	November 18, 1997	GB2
First Brands	Glad® Quick-Tie™ Flaps	108C7322	10	0.85 mil	June 1998	November 18, 1997	GB3
First Brands	Glad® Quick-Tie™ Flaps	208C8084	10	0.85 mil	June 1998	March 25, 1998	GC1
First Brands	Glad® Quick-Tie™ Flaps	208C8084	10	0.85 mil	June 1998	March 25, 1998	GC2
First Brands	Glad® Quick-Tie™ Flaps	208C8084	10	0.85 mil	June 1998	March 25, 1998	GC3
First Brands	Glad® Quick-Tie™ Flaps	308C8054	10	0.85 mil	June 1998	February 23, 1998	GD1
First Brands	Glad® Quick-Tie™ Flaps	308C8054	10	0.85 mil	June 1998	February 23, 1998	GD2
First Brands	Glad® Quick-Tie™ Flaps	308C8054	10	0.85 mil	June 1998	February 23, 1998	GD3
First Brands	Glad® Quick-Tie™ Flaps	25A81091	20	0.85 mil	June 1998	April 19, 1998	GE1
Tenneco	Hefty® EasyFlaps™	038042217	20	0.85 mil	June 1998	N/A	HA1
Carlisle Plastics	Ruffies™Pro	N/A	100	0.60 mil	July 1998	N/A	RA1

N/A = Not available.

TABLE 9: Analytical Sample Identification Codes

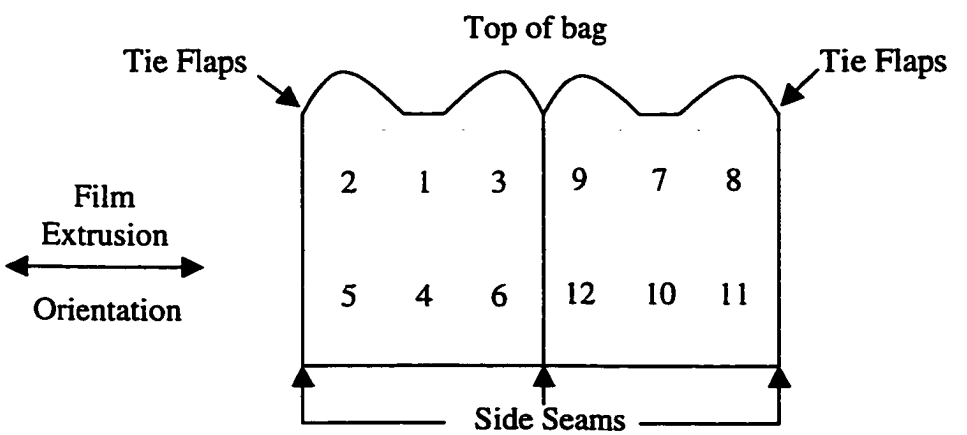
Position	Character	Description	
First	Letter	Manufacturer:	G= Glad® ; H= Hefty® ; R= Ruffies™
Second	Letter	Lot Number:	A, B, C, etc. represent different lot numbers, or packaging codes. These are repeated for each manufacturer.
Third	Number	Box Number:	Multiple boxes with the same packaging code were numbered 1 to 3 prior to opening.
Fourth	Number	Bag Number:	Boxes were opened and bags were numbered consecutively from 1 to the highest number in the box. This number may be 2 digits.

There is a hyphen between the fourth and fifth character in the sample code.

Fifth	Number	Sample Number:	Bags were divided into 12 regions – six on each side using a grid pattern (see Figure 14).

FIGURE 14: Analytical sample locations and identification numbers

View of plastic trash bag split on one side seam and opened flat. The numbers on the diagram below denote the sample number in the sample code corresponding to the analytical sample location.



After assigning the lot identifier letter designation, each box was then opened at the end flap and bags were removed from the box as a group, so as to maintain the bag sequence of the package. The batch of bags was unfolded and each bag was consecutively labeled with a bag number from one to the total number of bags in the box (in most cases, 10). Analytical samples were excised from the bag with a clean stainless steel scalpel blade and the sampling locations were recorded, as shown in Figure 14. The number of analytical samples taken per bag and the number of bags sampled per box were as needed for each study previously described in Chapter III. In some instances, problems encountered during sample preparation including partial loss of ash residue, poor droplet and TXRF film formation, and extrinsic contamination resulted in a difference in the number of reported results among the various studies.

CHAPTER VI. ANALYTICAL RESULTS

The accuracy of the TXRF results was checked periodically using a certified, multi-element standard during the analysis of plastic bags. This "plastic bag" standard is a multi-element solution containing most of the elements encountered in polyethylene bags at concentrations comparable to the levels in the bags. Not all of the elements that were detected in the survey bags are in the standard, because the standard was ordered during method development and prior to the detection of some elements. This standard is a solution and therefore, does not test the recovery efficiency of the plastic bag sample preparation process. However, a certified polyethylene standard for inorganic elements, with the exception of Cd, is not available. While the standard solution used here does not test for inaccuracy that may result from sample preparation, it is suitable to check the accuracy of the instrumental procedure.

Summary results of the TXRF replicate analyses of the "plastic bag" standard for three excitation conditions are shown in Table 10, along with the certified value for each element. These results are based on repeat analysis of a single sample preparation for 1,000 live seconds at each excitation condition. The sample was prepared using 20 μL of "plastic bag" standard and 20 μL of 4 ppm Ga in 10% (v/v) HNO_3 as an internal standard. Since Ga is not a suitable internal standard for W-L excitation, Ti in the "plastic bag" standard was used as the internal standard for these results instead. Results for Ca were blank corrected to account for the concentration of Ca determined in the internal standard solution. Uncertainties based on both X-ray counting statistics and on replicate analyses are included in Table 10. Both the accuracy and precision of repeat measurements of the

TABLE 10: High Purity "FBI Plastic Bag Standard". Results are in μg/g.

	Mo-K			W-*brems*			W-L			
Element	**Mean (n=7)**	**Repeated Measures % RSD**	**Mean X-ray Counting Statistics %RSD**	**Mean (n=5)**	**Repeated Measures % RSD**	**Mean X-ray Counting Statistics %RSD**	**Mean (n=2)**	**Repeated Measures % RSD**	**Mean X-ray Counting Statistics %RSD**	**Certified Value (± 0.5%)**
Barium	----	----	----	46.35	8.1 %	0.9 %	----	----	----	50
Calcium	100.4	2.5 %	2.0 %	103.3	1.9 %	3.3 %	91.23	0.8 %	0.8 %	100
Chromium	8.532	1.7 %	6.1 %	68.18	6.3 %	15.5 %	10.07	6.5 %	1.9 %	10
Copper	0.982	7.2 %	11.5 %	0.707	17.6 %	35.0 %	----	----	----	1
Iron	188.3	0.5 %	0.2 %	188.9	0.5 %	0.2 %	195.9	0.6 %	0.04 %	200
Lead	1.013	6.1 %	21.0 %	1.210	5.4 %	24.9 %	----	----	----	1
Manganese	1.752	7.7 %	24.9 %	1.687	14.5	42.7 %	2.001	12.0 %	9.0 %	2
Nickel	1.294	8.0 %	10.4 %	1.369	7.2 %	17.2 %	1.022	5.6 %		1
Potassium	53.58	3.2 %	4.6 %	47.77	7.2 %	12.0 %	50.19	2.0 %	2.2 %	50
Strontium	0.995	2.6 %	4.4 %	0.985	2.3 %	7.3 %	----	----	----	1
Sulfur	296.4	4.0 %	2.1 %	613.2	3.9 %	8.2 %	304.7	5.4 %	1.0 %	300
Titanium	2006	4.8 %	0.1 %	2012	0.8 %	0.1 %	2000*	0.00 %	0.04 %	2000
Zinc	50.01	0.9 %	0.2 %	50.53	0.8 %	0.4 %	----	----	----	50
Zirconium	0.981	7.5 %	19.5 %	0.989	1.8 %	6.9 %	----	----	----	1

* Ti was used as the internal standard for quantitation by W-L excitation.

---- Excitation condition not suitable for quantitation. Data not available.

"plastic bag" standard are good. As expected, the precision of repeated measurements is better than the X-ray counting uncertainty for elements that are at the low ppm levels.

A new microbeaker was included in the plasma asher with each set of plastic bags and was used as the preparation blank. A TXRF quartz sample support was prepared for each preparation blank in the same manner as the plastic bag samples. Additionally, with each set of samples, a reagent blank was prepared on a TXRF support by pipetting 20 µL of 4 ppm Ga internal standard, and 2-20 µL aliquots of the 10% (v/v) HNO_3 from the same source as that used in preparation of the plastic bag samples. The purpose of the reagent blank was to check the quality of the reagents before use in plastic bag sample preparation or to help identify a source of contamination after preparation. Reagent blanks differed from the preparation blanks in that no microbeakers were used and reagent blanks were not exposed to plasma ashing. Reagent blanks contained lower concentrations of contaminant elements than preparation blanks. Concentration results for the survey samples were blank corrected using results from an average preparation blank. To obtain preparation blank averages, preparation blanks were grouped based on the batches of microbeakers, acid, and internal standard used in their preparation. The average blank was subsequently subtracted from the results for all samples prepared with these groups of blanks. The concentrations of most elements in the blanks are negligible compared to the levels in the bag samples, with the exception of Si, which originates from the quartz sample support. Depending on the concentration of Si in the sample, the blank subtraction for Si in this study was generally on the order of 20% or less.

The TXRF results in μg/g for all elements detected using the three excitation conditions (Table 7) in the survey studies are shown in Tables 11 through 14. The ratio of the quantitative results of Ti and Fe is also included in the tables as indicated. Samples are grouped in Tables 11 through 14 based on the purpose of each study and the excitation condition. All results listed are rounded to three decimal places. A "*" next to a result indicates that it is an outlier based on statistical evaluation (see Chapter VII). Results that are less than the level in the blank or for which no X-ray fluorescence peak was detected are indicated by "nd" in the tables. Results that are above the LOD are included in the tables, even if they are below the limit of quantitation (LOQ). This approach was chosen mainly to provide reasonable estimates of these data for the statistical evaluation of the data. Using zeroes in place of results that are below LOQ underestimates the means, while using the LOQ for these results overestimates these values. Similarly, the precisions of the results are also adversely affected by both of these methods. The approach taken here does not bias the means or standard deviations. Element quantities that are below the LOQ show a high RSD among the replicate sample results, a fact that is taken into account in the statistical evaluation of the grouping of plastic bags.

Compositional results of the TXRF analyses of twelve samples from within single bags from three different manufacturers are shown in Tables 11a through 11c, for three excitation conditions. TXRF results for the study of the compositional variation within a box, analyzing all bags contained within a box of ten Glad® Quick-Tie™ trash bags, are shown in Tables 12a through 12c, for three excitation conditions. Multiple bags from a

larger box of 20 trash bags were also analyzed and the results are included in Tables 13a through 13c. Tables 14a through 14c contain the results of TXRF analyses of replicate samples from within and among boxes of Glad® Quick-Tie™ trash bags for three excitation conditions analyzed to determine the compositional variation within and among manufacturing production runs. The sources for this study are three boxes from each of three different production runs from the same manufacturer.

Because of excitation overlaps between X-ray tube sources, several elements were detected and quantitated with more than one excitation mode. TXRF quantitative results for the same element analyzed under different excitation conditions compare well and are within X-ray counting uncertainties or two standard deviations of the means of replicate sample preparations.

TABLE 11a: Analytical results of the within-bag study using Mo-K excitation. Assay time is 1,000 live seconds. Element concentration results are in µg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB11-1	934.042	2316.638	138.922	40.970	nd	15.893	28.322
GB11-2	792.950	1662.766	132.949	39.763	nd	14.217	19.447
GB11-4	1054.816	2385.067	139.199	36.335	nd	12.774	22.253
GB11-5	964.100	2446.361	133.907	32.037	nd	12.573	22.792
GB11-6	739.159	1786.951	124.400	32.365	nd	17.049	17.701
GB11-7	826.459	2118.743	132.831	31.302	nd	12.674	20.140
GB11-11	939.930	2126.157	131.539	30.803	nd	15.312	18.573
GB11-12	1089.392	2723.256	153.092	44.155	nd	16.674	23.384
HA11-1	1393.221	5030.786	120.356	84.008	nd	20.049	297.484
HA11-3	1446.168	4938.555	124.442	83.842	nd	31.444	296.269
HA11-5	1575.474	4907.812	121.960	86.827	4.211	17.212	289.187
HA11-8	1743.895	5798.387	122.042	92.575	nd	21.532	286.787
HA11-9	1401.633	4848.974	112.021	74.184	nd	16.860	277.511
HA11-10	1546.091	4987.966	116.548	87.817	nd	22.196	289.009
HA11-11	1154.709	4204.438	100.524	74.312	nd	23.359	270.232
HA11-12	1040.861	4020.191	99.086	83.583	nd	19.956	275.559
RA11-1	503.941	1560.459	86.886	87.650	8.832	16.463	129.415
RA11-2	657.853	1987.495	100.654	78.118	7.605	16.717	132.347
RA11-3	626.288	2102.821	100.783	89.524	9.218	16.043	131.482
RA11-4	465.326	1514.875	80.902	63.145	5.520	12.776	116.847
RA11-5	513.537	1667.275	86.157	82.436	12.644	17.171	121.135
RA11-6	525.095	1597.386	90.439	71.702	5.668	15.035	120.520
RA11-7	745.735	2319.795	91.263	79.212	5.760	17.562	124.719
RA11-8	537.521	1672.733	83.106	72.925	5.728	14.502	122.275
RA11-9	438.245	1597.515	82.572	75.649	6.768	17.101	128.942
RA11-10	558.839	1880.337	81.431	73.945	5.439	15.946	129.797
RA11-11	526.440	1639.850	88.972	74.685	5.648	16.629	119.647
RA11-12	524.252	1617.912	89.557	109.205	7.626	23.508	155.188

nd = element not detected.
* = outlier.

TABLE 11a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GB11-1	940.252	nd	0.131	96.311	0.151	0.396	46.131
GB11-2	710.143	nd	0.134	109.430	0.152	0.484	44.951
GB11-4	1054.572	nd	0.149	104.319	0.162	0.263	45.535
GB11-5	1038.052	0.003	0.282	101.517	0.120	0.304	44.975
GB11-6	838.133	0.943	0.142	88.700	0.218	0.374	44.388
GB11-7	899.924	nd	0.147	90.430	0.156	0.363	42.955
GB11-11	943.450	nd	0.178	99.282	0.209	1.308	43.948
GB11-12	1144.232	nd	0.310	113.293	0.192	2.494	46.813
HA11-1	1117.718	0.226	0.589	77.789	0.253	0.271	15.419
HA11-3	1220.642	0.450	0.653	79.217	0.475	0.135	17.720
HA11-5	1160.482	0.397	0.704	81.304	0.260	0.153	12.380
HA11-8	1297.621	0.329	0.908	92.462	0.433	0.392	11.976
HA11-9	1045.627	0.241	0.721	74.945	0.253	0.107	14.816
HA11-10	1143.877	0.246	0.737	79.261	0.266	0.218	13.344
HA11-11	1006.794	0.224	0.658	69.046	0.382	1.051	15.395
HA11-12	1005.560	0.221	0.706	66.248	0.270	0.231	10.580
RA11-1	69.210	0.671	0.170	26.498	0.309	0.300	130.806
RA11-2	90.669	1.590	0.295	37.712	0.742	0.267	125.832
RA11-3	86.103	0.471	0.265	31.882	0.349	0.445	130.700
RA11-4	72.502	0.235	0.218	27.244	0.198	0.088	107.077
RA11-5	79.996	0.218	0.158	26.947	0.269	0.792	132.255
RA11-6	73.406	0.245	0.210	31.275	0.193	0.236	113.852
RA11-7	85.439	0.485	0.206	37.877	0.354	0.276	113.259
RA11-8	72.259	1.379	0.208	32.067	0.688	0.095	109.227
RA11-9	73.046	0.299	0.181	27.279	0.268	0.396	115.590
RA11-10	75.587	0.266	0.207	31.352	0.256	0.339	117.387
RA11-11	70.023	0.237	0.146	29.266	0.302	0.423	118.954
RA11-12	72.167	0.232	0.282	28.080	0.750	1.611	116.705

nd = element not detected.
* = outlier.

TABLE 11a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GB11-1	0.011	0.582	0.274	0.488	9.763
GB11-2	0.008	0.583	0.235	0.489	6.489
GB11-4	0.010	0.614	0.243	0.537	10.109
GB11-5	0.007	0.542	0.192	0.544	10.225
GB11-6	0.006	0.457	0.751	0.440	9.449
GB11-7	0.004	0.576	0.437	0.466	9.952
GB11-11	0.011	0.497	0.261	0.539	9.503
GB11-12	nd	0.669	0.418	0.587	10.100
HA11-1	nd	0.262	0.297	0.216	14.369
HA11-3	0.011	0.340	0.113	0.151	15.409
HA11-5	nd	0.286	0.084	0.170	14.273
HA11-8	0.036	0.243	0.115	0.203	14.034
HA11-9	nd	0.270	0.075	0.176	13.952
HA11-10	0.004	0.250	0.103	0.277	14.432
HA11-11	0.014	0.306	0.074	0.228	14.581
HA11-12	0.008	0.225	0.023	0.283	15.179
RA11-1	0.016	0.073	0.752	0.074	2.612
RA11-2	0.016	0.089	0.876	0.088	2.404
RA11-3	nd	0.140	2.279	0.092	2.701
RA11-4	0.012	0.069	0.647	0.078	2.661
RA11-5	0.008	0.067	0.196	0.098	2.969
RA11-6	0.014	0.074	1.729	0.116	2.347
RA11-7	0.050	0.099	0.614	0.154	2.256
RA11-8	0.019	0.081	0.498	0.079	2.253
RA11-9	0.010	0.079	0.253	0.116	2.678
RA11-10	0.016	0.087	0.626	0.103	2.411
RA11-11	0.013	0.077	0.458	0.168	2.393
RA11-12	0.021	0.115	0.978	0.157	2.570

nd = element not detected.
* = outlier.

TABLE 11b: Analytical results of the within-bag study using W-*brems* excitation. Assay time is 1,000 live seconds. Element concentration results are in µg/g.

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GB11-1	66.852	13.095	27.940	1005.069	0.939	0.044	99.715
GB11-2	44.835	12.613	19.513	909.268	0.501	0.052	120.967
GB11-4	67.147	10.934	19.952	1020.858	1.239	0.127	102.051
GB11-5	40.127	10.287	23.705	1025.297	0.874	0.160	97.542
GB11-6	58.985	14.805	16.791	977.428	1.888	0.247	98.987
GB11-7	53.680	9.856	21.744	999.293	0.955	0.351	97.954
GB11-11	46.452	12.061	18.838	996.626	0.639	0.200	100.672
GB11-12	65.422	7.682	21.006	1072.940	0.958	0.284	102.459
HA11-1	118.801	11.059	281.982	1323.434	5.528	1.152	93.559
HA11-2	137.795	10.937	280.027	1552.650	4.175	0.805	101.928
HA11-3	129.894	23.511	288.973	1249.168	4.972	0.824	84.490
HA11-5	121.941	12.968	276.159	1391.913	4.510	0.701	92.913
HA11-6	113.760	12.342	265.303	1615.732	4.983	1.000	108.987
HA11-8	148.393	16.416	283.854	1450.944	4.645	0.839	101.949
HA11-9	122.450	10.183	277.739	1304.528	4.998	0.819	90.423
HA11-10	127.904	17.856	286.462	1455.507	3.937	0.593	97.413
HA11-11	124.749	15.550	272.127	1238.011	5.101	0.612	87.244
HA11-12	132.800	14.987	273.045	1118.224	4.174	0.607	75.333
RA11-1	102.855	6.585	128.042	80.768	0.499	0.240	25.006
RA11-2	108.576	6.899	132.642	90.868	1.474	0.175	32.744
RA11-3	114.018	8.468	123.510	86.075	0.374	0.229	28.395
RA11-4	74.434	4.878	123.174	80.971	0.276	0.207	26.179
RA11-5	124.272	8.986	123.594	88.074	0.177	0.298	25.841
RA11-6	96.774	7.205	125.077	78.174	0.192	0.123	28.465
RA11-7	109.813	6.494	125.552	82.845	0.292	0.184	30.347
RA11-8	79.047	4.481	126.001	75.825	1.127	0.241	27.552
RA11-9	99.671	7.811	133.785	77.201	0.351	0.256	24.406
RA11-10	115.303	8.231	131.769	79.532	0.288	0.162	26.678
RA11-11	87.760	7.376	124.284	77.829	0.124	0.223	29.037
RA11-12	169.972	13.345	152.482	76.082	0.231	0.310	26.018

nd = element not detected.
* = outlier.

TABLE 11b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GB11-1	0.107	0.367	49.219	nd	0.527	0.011	0.287
GB11-2	0.205	0.410	46.897	0.023	0.550	0.013	0.270
GB11-4	0.152	0.358	47.892	0.014	0.541	0.104	0.290
GB11-5	0.172	0.237	47.151	0.012	0.514	0.023	0.265
GB11-6	0.164	0.360	47.420	0.022	0.459	0.037	0.738
GB11-7	0.153	0.402	45.585	0.032	0.550	0.013	0.373
GB11-11	0.181	1.238	46.428	0.030	0.494	0.036	0.275
GB11-12	0.188	2.411	47.753	0.017	0.587	0.018	0.376
HA11-1	0.233	0.288	17.723	0.013	0.274	nd	0.368
HA11-2	0.429	nd	13.103	0.021	0.237	0.031	0.571
HA11-3	0.336	0.118	20.243	0.036	0.305	0.001	0.239
HA11-5	0.187	0.082	13.955	0.003	0.257	0.033	0.323
HA11-6	0.341	0.622	13.078	0.029	0.223	nd	0.329
HA11-8	0.261	0.311	13.441	nd	0.218	nd	0.325
HA11-9	0.114	0.172	17.414	0.030	0.274	0.006	0.294
HA11-10	0.311	0.229	14.946	0.041	0.281	nd	0.356
HA11-11	0.291	1.021	18.194	0.018	0.261	0.023	0.303
HA11-12	0.230	0.300	12.637	0.009	0.215	nd	0.212
RA11-1	0.359	0.094	130.146	nd	0.053	0.025	0.675
RA11-2	0.642	0.161	128.073	0.028	0.086	0.036	0.655
RA11-3	0.396	0.387	137.171	nd	0.110	0.114	1.772
RA11-4	0.154	nd	111.626	0.004	0.072	0.025	0.608
RA11-5	0.398	0.548	133.334	0.020	0.078	0.003	0.257
RA11-6	0.214	0.023	118.486	nd	0.058	0.084	1.417
RA11-7	0.356	0.154	114.927	0.032	0.077	0.025	0.433
RA11-8	0.600	nd	111.275	0.006	0.072	0.033	0.372
RA11-9	0.238	0.256	117.390	0.030	0.073	0.014	0.236
RA11-10	0.281	0.227	119.453	0.020	0.071	0.031	0.527
RA11-11	0.348	0.229	123.161	0.002	0.084	0.020	0.437
RA11-12	0.771	1.469	121.942	0.010	0.086	0.057	0.778

nd = element not detected.
* = outlier.

TABLE 11b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GB11-1	0.534	28.771	0.171	0.026	10.079
GB11-2	0.536	23.707	0.171	0.032	7.517
GB11-4	0.492	29.094	0.189	0.033	10.003
GB11-5	0.587	28.481	0.209	0.026	10.511
GB11-6	0.440	25.569	0.345	0.023	9.874
GB11-7	0.464	28.993	0.264	0.012	10.202
GB11-11	0.619	26.720	0.866	0.067	9.900
GB11-12	0.573	30.921	2.194	3.557	10.472
HA11-1	0.278	nd	0.108	0.060	14.146
HA11-2	0.351	0.418	0.063	0.030	15.233
HA11-3	0.178	nd	0.027	0.020	14.785
HA11-5	0.135	0.177	0.077	0.059	14.981
HA11-6	0.355	0.076	0.321	1.228	14.825
HA11-8	0.277	0.277	0.226	0.041	14.232
HA11-9	0.156	nd	nd	0.061	14.427
HA11-10	0.258	0.147	0.035	0.045	14.942
HA11-11	0.252	0.671	0.469	0.032	14.190
HA11-12	0.245	0.268	0.034	0.078	14.844
RA11-1	0.134	nd	0.082	0.008	3.230
RA11-2	0.104	nd	0.228	0.007	2.775
RA11-3	0.122	0.220	0.203	0.101	3.031
RA11-4	0.118	0.295	0.038	0.006	3.093
RA11-5	0.133	0.029	0.038	0.012	3.408
RA11-6	0.213	0.401	0.059	0.012	2.746
RA11-7	0.161	0.172	0.073	0.025	2.730
RA11-8	0.142	0.185	0.018	0.043	2.752
RA11-9	0.156	0.212	0.174	0.010	3.163
RA11-10	0.172	nd	0.296	0.019	2.981
RA11-11	0.210	0.302	0.446	0.022	2.680
RA11-12	0.250	0.220	1.236	0.021	2.924

nd = element not detected.
* = outlier.

TABLE 11c: Analytical results of the within-bag study using W-L excitation. Assay time is 1,000 live seconds. Element concentration results are in μg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB11-1	1107.632	2946.116	148.850	105.050	1.862	35.662	49.768
GB11-2	700.439	1638.994	104.018	69.415	nd	24.463	23.525
GB11-4	1011.601	2483.911	110.561	71.216	0.748	24.574	28.680
GB11-5	1007.968	2657.889	116.774	74.161	nd	38.516	32.016
GB11-6	880.321	2344.793	125.225	71.158	1.635	25.720	35.201
GB11-7	883.860	2453.674	99.545	87.929	12.686	nd	49.826
GB11-11	818.394	2295.508	67.573	25.377	nd	12.359	14.116
GB11-12	1279.724	3770.977	123.146	81.905	0.078	30.355	31.203
HA11-1	1440.366	5379.436	93.504	195.001	3.540	26.451	429.536
HA11-3	1327.749	5058.811	100.888	221.999	5.009	44.595	466.271
HA11-5	1355.855	5334.793	105.365	242.064	19.037	32.855	500.623
HA11-8	1368.891	5708.070	62.080	121.739	nd	15.741	254.407
HA11-9	1366.042	4801.731	98.040	313.684	4.342	34.398	610.559
HA11-10	1341.386	5189.633	88.302	244.780	1.489	40.458	485.131
HA11-11	1151.302	4025.086	74.990	190.931	0.054	36.474	435.748
HA11-12	1124.814	4232.175	77.139	241.114	0.495	33.462	472.256
RA11-1	444.337	2053.341	74.820	158.312	18.029	20.535	231.678
RA11-2	480.055	2326.795	73.258	113.328	3.479	20.789	192.259
RA11-3	471.188	2562.396	73.088	139.080	16.128	18.034	199.987
RA11-4	318.392	1650.135	47.895	60.790	2.987	9.174	133.820
RA11-5	390.347	2014.444	56.813	114.160	37.934	17.164	154.412
RA11-6	341.549	1731.091	60.007	88.961	4.068	12.745	163.460
RA11-7	562.048	2874.090	73.402	124.552	4.873	24.761	205.534
RA11-8	377.425	1964.734	58.803	90.949	2.179	12.953	166.702
RA11-9	411.506	2009.351	60.274	102.703	3.407	18.754	182.414
RA11-10	388.262	1990.411	51.945	87.005	1.669	14.371	158.682
RA11-11	399.415	1898.685	61.334	105.145	2.283	16.509	178.249
RA11-12	423.507	1966.280	72.413	214.673	6.938	39.243	282.965

nd = element not detected.
* = outlier.

TABLE 11c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GB11-1	939.946	0.289	0.442	137.630	0.332	6.830
GB11-2	709.770	0.192	0.361	139.780	0.184	5.078
GB11-4	1054.302	0.341	0.416	129.181	0.276	8.161
GB11-5	1037.653	3.087	0.329	145.970	0.449	7.109
GB11-6	837.852	0.082	0.317	123.110	0.244	6.806
GB11-7	899.652	0.214	0.359	124.344	0.432	7.235
GB11-11	943.117	0.116	0.340	113.191	0.187	8.332
GB11-12	1143.925	0.252	0.492	156.311	0.255	7.318
HA11-1	1117.766	0.557	0.875	100.571	0.432	11.114
HA11-3	1220.691	0.834	0.966	97.302	0.593	12.545
HA11-5	1160.535	1.213	0.998	103.996	0.412	11.159
HA11-8	1297.676	0.448	1.021	104.525	0.359	12.415
HA11-9	1045.672	0.816	0.903	101.185	0.445	10.334
HA11-10	1143.926	0.428	0.993	100.595	0.522	11.372
HA11-11	1006.842	0.563	0.920	83.850	0.458	12.008
HA11-12	1005.604	0.648	0.898	84.317	0.402	11.927
RA11-1	68.842	1.169	0.491	40.629	0.492	1.694
RA11-2	90.355	2.850	0.501	54.964	0.855	1.644
RA11-3	85.790	0.699	0.473	47.479	0.458	1.807
RA11-4	72.172	0.309	0.328	33.312	0.202	2.167
RA11-5	79.623	3.896	0.646	46.003	2.192	1.731
RA11-6	73.057	0.391	0.374	41.814	0.241	1.747
RA11-7	85.055	0.780	0.568	61.913	0.446	1.374
RA11-8	71.878	2.908	0.444	46.402	0.895	1.549
RA11-9	72.698	0.486	0.431	40.186	0.325	1.809
RA11-10	75.278	0.344	0.408	40.537	0.276	1.857
RA11-11	69.637	0.440	0.370	44.280	0.346	1.573
RA11-12	71.837	0.470	0.508	44.518	1.092	1.614

nd = element not detected.
* = outlier.

TABLE 12a: Analytical results of the within-box GB1 study using Mo-K excitation. Assay time is 1,000 live seconds. Element concentration results are in μg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB11-3	623.638	1303.449	117.784	12.507	nd	7.969	15.767
GB11-6	681.398	1455.626	111.873	10.831	nd	9.037	19.504
GB11-7	564.102	1075.561	115.525	8.498	nd	4.667	11.557
GB11-12	540.212	1277.544	112.376	7.236	nd	8.515	11.747
GB12-3	614.605	1393.832	110.181	18.204	nd	10.472	72.876*
GB12-4	707.301	1605.663	102.240	13.644	nd	12.597	13.441
GB12-11	846.755	1979.661	111.954	35.236*	1.302	26.983*	25.033
GB12-12	743.373	1575.349	107.849	13.631	nd	13.825	15.217
GB13-4	855.293	2023.430	128.610	21.029	nd	4.838	16.164
GB13-5	794.689	1912.527	122.060	21.762	nd	7.129	20.431
GB13-7	750.140	2113.152	112.785	11.694	nd	8.364	15.618
GB13-12	726.955	1782.699	116.787	18.703	nd	3.888	11.701
GB14-2	861.240	1975.847	118.554	23.828	3.177	4.226	20.545
GB14-6	861.697	2270.621	131.309	18.994	nd	5.072	14.201
GB14-7	784.221	2019.143	117.059	16.221	nd	4.899	15.550
GB14-11	739.925	1838.988	113.209	12.762	nd	6.096	14.677
GB15-1	781.844	1954.396	125.108	16.818	nd	10.775	15.702
GB15-5	736.004	1853.252	127.480	18.217	0.402	7.687	14.992
GB15-9	754.868	1796.583	110.310	18.826	nd	7.312	12.724
GB15-11	683.417	1755.497	114.170	17.401	5.138	14.909	20.050
GB16-2	599.399	1511.046	119.207	24.451	1.656	19.319	21.364
GB16-3	499.505	1099.759	116.980	13.775	nd	7.401	14.045
GB16-7	626.170	1440.219	130.539	12.457	nd	10.036	13.086
GB16-8	600.948	1155.307	117.778	12.252	nd	10.316	14.502
GB17-3	628.526	1485.005	112.820	12.285	nd	8.554	15.344
GB17-5	743.542	1680.426	116.663	17.003	nd	8.637	17.750
GB17-8	670.193	1376.959	125.536	32.913*	nd	17.813	16.380
GB17-11	639.381	1394.799	116.801	10.232	nd	10.834	16.214

nd = element not detected.
* = outlier.

TABLE 12a: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB18-3	1141.334*	2466.557*	145.529*	23.567	nd	17.686	18.463
GB18-5	688.122	1987.814	126.606	12.060	nd	4.519	16.420
GB18-8	642.901	1553.278	122.633	9.127	nd	4.057	11.212
GB18-9	699.670	1821.253	115.512	16.841	nd	7.717	15.342
GB19-2	656.232	1319.432	125.520	10.703	nd	9.333	16.415
GB19-5	662.442	1336.837	107.727	4.510	nd	9.245	14.706
GB19-10	586.666	1386.313	118.253	14.718	nd	7.278	12.405
GB19-12	599.185	1490.563	120.564	16.273	nd	11.068	12.815
GB110-5	470.143	964.618	111.447	13.368	nd	7.728	11.656
GB110-6	750.096*	1656.433	124.157	12.557	nd	9.441	15.469
GB110-10	616.158	1607.898	120.750	13.343	nd	15.346	13.781
GB110-11	554.538	1309.962	114.169	8.874	nd	8.826	12.724

nd = element not detected.
* = outlier.

TABLE 12a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GB11-3	697.510	nd	0.204	80.643	0.110	0.038	43.273
GB11-6	790.134	nd	0.149	82.984	0.197	0.118	41.325
GB11-7	820.681	nd	0.179	85.650	0.110	0.035	40.387
GB11-12	576.929	nd	0.114	69.032	0.189	0.044	41.560
GB12-3	641.201	nd	0.147	73.475	0.258	0.189	40.387
GB12-4	834.155	nd	0.127	86.923	0.309	0.303	40.581
GB12-11	898.782	0.805	0.289	92.537	1.009*	0.353	38.425
GB12-12	741.419	0.008	0.119	80.921	0.581	0.171	41.003
GB13-4	834.019	0.109	0.156	89.210	0.103	0.231	40.669
GB13-5	811.943	nd	0.234	85.553	0.109	0.370	36.597
GB13-7	718.159	nd	0.157	81.909	0.093	0.179	41.638
GB13-12	818.970	0.078	0.308	86.456	0.177	0.169	40.396
GB14-2	792.938	0.054	0.113	85.040	0.124	0.047	36.559
GB14-6	849.601	0.174	0.137	89.453	0.161	0.148	40.917
GB14-7	824.224	0.467	0.178	87.787	0.133	0.124	40.104
GB14-11	903.803	nd	0.144	89.679	0.140	0.228	36.597
GB15-1	759.281	0.108	0.165	83.465	0.133	0.329	40.801
GB15-5	754.780	nd	0.199	81.768	0.171	0.249	39.715
GB15-9	787.685	0.090	0.112	80.876	0.117	0.116	35.126
GB15-11	778.493	nd	0.196	82.477	0.173	0.219	40.401
GB16-2	791.202	nd	0.165	78.814	0.170	0.249	39.847
GB16-3	635.946	nd	0.112	71.122	0.119	0.079	40.511
GB16-7	801.974	nd	0.153	85.312	0.110	0.075	39.184
GB16-8	733.089	nd	0.120	77.648	0.068	0.053	41.600
GB17-3	757.803	nd	0.203	83.504	0.101	0.249	41.643
GB17-5	841.161	nd	0.204	87.169	0.034	0.342	44.329
GB17-8	708.024	nd	0.145	77.388	0.074	0.367	46.611
GB17-11	700.428	nd	0.160	76.689	0.058	0.940*	43.231

nd = element not detected.
* = outlier.

TABLE 12a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GB18-3	966.727	0.238	0.106	102.861	0.111	0.402	40.928
GB18-5	766.375	0.101	0.140	81.328	0.134	0.113	42.471
GB18-8	761.319	nd	0.128	81.458	0.118	0.086	40.106
GB18-9	890.347	0.046	0.156	90.334	0.077	0.123	41.061
GB19-2	637.295	nd	0.116	73.340	0.088	0.064	40.984
GB19-5	791.533	nd	0.221	83.586	0.074	0.145	39.605
GB19-10	662.386	nd	0.198	74.865	0.040	0.194	40.535
GB19-12	692.061	nd	0.175	81.060	0.049	0.093	41.346
GB110-5	657.960	nd	0.182	72.521	0.047	0.033	39.328
GB110-6	726.808	nd	0.221	81.318	0.090	0.090	41.344
GB110-10	776.752	nd	0.095	83.841	0.102	0.146	42.312
GB110-11	607.153	nd	0.120	71.763	0.080	0.068	40.072

nd = element not detected.
* = outlier.

TABLE 12a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GB11-3	nd	0.428	0.719	0.461	8.649
GB11-6	0.005	0.489	0.530	0.472	9.522
GB11-7	0.003	0.466	0.130	0.447	9.582
GB11-12	0.005	0.453	0.539	0.474	8.357
GB12-3	0.012	0.523	9.371	0.518	8.727
GB12-4	0.008	0.416	27.376	0.537	9.597
GB12-11	0.004	0.479	46.816	0.577	9.713
GB12-12	nd	0.423	16.145	0.436	9.162
GB13-4	0.005	0.492	1.970	0.514	9.349
GB13-5	0.009	0.481	0.384	0.497	9.491
GB13-7	0.004	0.505	0.253	0.542	8.768
GB13-12	nd	0.435	6.125	0.559	9.473
GB14-2	nd	0.486	1.762	0.523	9.324
GB14-6	0.012	0.482	1.790	0.528	9.498
GB14-7	0.003	0.462	0.834	0.510	9.389
GB14-11	nd	0.455	0.453	0.521	10.078
GB15-1	0.018	0.498	0.480	0.509	9.097
GB15-5	nd	0.482	0.982	0.495	9.231
GB15-9	nd	0.433	1.934	0.455	9.739
GB15-11	0.004	0.454	0.649	0.534	9.439
GB16-2	0.017	0.474	3.722	0.662	10.039
GB16-3	0.019	0.416	3.188	0.518	8.942
GB16-7	0.012	0.425	1.309	0.464	9.400
GB16-8	0.001	0.467	1.656	0.524	9.441
GB17-3	nd	0.470	1.370	0.509	9.075
GB17-5	nd	0.420	1.201	0.713	9.650
GB17-8	0.000	0.411	1.180	1.111	9.149
GB17-11	0.002	0.426	1.140	0.527	9.133

nd = element not detected.
* = outlier.

TABLE 12a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GB18-3	0.014	0.504	0.462	0.551	9.398
GB18-5	nd	0.445	0.373	0.481	9.423
GB18-8	0.007	0.482	0.256	0.529	9.346
GB18-9	nd	0.493	0.519	0.503	9.856
GB19-2	0.001	0.429	0.601	0.493	8.690
GB19-5	0.003	0.426	0.624	0.491	9.470
GB19-10	nd	0.426	0.422	0.483	8.848
GB19-12	0.001	0.451	0.560	0.483	8.538
GB110-5	0.008	0.413	0.597	0.468	9.073
GB110-6	0.001	0.493	0.262	0.520	8.938
GB110-10	0.011	0.429	0.359	0.453	9.265
GB110-11	0.000	0.435	0.614	0.442	8.461

nd = element not detected.
* = outlier.

TABLE 12b: Analytical results of the within-box GB1 study using W-*brems* excitation. Assay time is 1,000 live seconds. Element concentration results are in μg/g.

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GB11-3	15.435	7.608	17.350	689.320	0.578	0.294	83.594
GB11-6	21.881	10.316	21.057	811.547	0.973	0.215	84.981
GB11-7	5.311	3.494	14.030	826.892	nd	0.023	85.477
GB11-12	1.091	8.956	14.143	637.309	0.467	0.101	72.058
GB12-3	7.092	11.495	79.018	806.426	1.015	0.166	85.900
GB12-4	nd	11.074	12.797	874.077	1.297	0.311	93.014
GB12-11	nd	21.321	23.736	825.604	2.086	0.462	90.531
GB12-12	nd	10.792	15.658	866.153	1.597	0.328	92.083
GB13-4	1.078	3.550	17.403	1005.717	0.719	0.449	101.754
GB13-5	15.874	7.600	19.705	850.834	1.280	0.176	88.997
GB13-7	6.563	5.450	10.579	797.942	3.134*	0.432	95.491
GB13-12	nd	4.019	11.369	921.599	1.922	0.383	97.538
GB14-2	21.957	3.752	22.075	934.117	1.440	0.069	94.683
GB14-7	15.911	4.252	15.379	879.919	1.871	0.305	94.029
GB14-11	2.839	4.438	14.693	1017.273	1.300	0.125	98.712
GB15-5	18.080	6.486	16.357	830.876	1.391	0.170	88.971
GB15-9	22.239	6.546	14.008	843.473	1.102	0.120	86.277
GB15-11	2.755	13.551	20.697	856.498	1.152	0.249	91.378
GB16-3	16.502	7.336	14.658	634.998	nd	0.096	70.396
GB16-7	4.891	10.612	15.555	823.248	0.473	0.277	87.161
GB16-8	1.632	10.505	16.396	723.707	0.252	0.046	77.197
GB17-3	1.893	8.573	16.026	952.122	1.134	0.057	99.568
GB17-5	17.375	10.658	19.346	935.072	0.312	0.336	93.278
GB17-8	52.500	17.622	18.880	653.045	0.601	0.066	75.485
GB17-11	16.458	9.882	19.125	684.988	0.127	0.068	76.784

nd = element not detected.
* = outlier.

TABLE 12b: (Continued)

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GB18-3	33.820	15.928	18.899	1139.504	1.962	0.149	117.987*
GB18-5	13.884	3.384	15.557	910.778	1.708	0.381	96.059
GB18-8	16.004	3.269	11.992	850.191	1.703	0.167	91.949
GB18-9	3.413	7.337	16.069	872.436	1.694	0.123	94.046
GB19-2	0.006	9.342	17.963	787.766	0.407	0.171	83.751
GB19-5	12.967	8.794	17.217	760.529	0.434	0.194	81.462
GB19-10	14.903	8.273	13.822	770.599	0.206	0.108	81.507
GB19-12	nd	9.147	15.044	739.214	0.465	0.256	83.481
GB110-5	2.647	8.985	13.597	615.972	nd	0.179	70.056
GB110-6	17.055	6.667	17.292	795.094	1.212	0.144	87.757
GB110-10	5.449	13.550	15.127	762.953	1.366	0.183	84.741
GB110-11	nd	10.591	13.825	756.239	0.717	0.439	81.595

nd = element not detected.
* = outlier.

TABLE 12b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GB11-3	0.100	0.085	45.419	nd	0.441	0.013	0.747
GB11-6	0.195	0.117	44.965	nd	0.448	0.020	0.512
GB11-7	0.072	nd	42.055	nd	0.458	0.012	0.202
GB11-12	0.136	0.019	43.173	nd	0.449	0.033	0.536
GB12-3	0.254	0.220	42.886	0.006	0.539	0.052	9.548
GB12-4	0.199	0.273	43.423	nd	0.412	0.077	26.341
GB12-11	0.786	0.412	40.661	0.003	0.467	0.102	36.007
GB12-12	0.580	0.253	43.614	0.013	0.436	0.054	14.843
GB13-4	0.025	0.237	43.392	nd	0.499	0.022	1.973
GB13-5	0.059	0.370	39.401	nd	0.434	0.019	0.344
GB13-7	0.057	0.317	46.046	0.003	0.500	0.016	0.301
GB13-12	0.099	0.170	43.753	nd	0.419	0.029	5.842
GB14-2	0.109	0.044	39.119	nd	0.473	0.001	1.785
GB14-7	0.049	0.080	42.986	nd	0.442	0.005	0.764
GB14-11	0.157	0.237	39.308	0.010	0.490	nd	0.417
GB15-5	0.107	0.256	42.414	nd	0.442	0.015	1.033
GB15-9	0.150	0.086	37.615	nd	0.384	0.017	1.819
GB15-11	0.085	0.230	42.687	0.019	0.390	0.018	0.643
GB16-3	0.140	0.072	41.315	nd	0.417	0.017	2.660
GB16-7	0.070	nd	41.338	nd	0.419	0.017	1.289
GB16-8	0.043	0.025	43.477	nd	0.461	0.004	1.484
GB17-3	0.096	0.338	44.905	0.020	0.462	0.020	1.543
GB17-5	0.113	0.276	47.433	nd	0.424	0.023	1.169
GB17-8	0.069	0.347	48.420	nd	0.400	0.016	1.040
GB17-11	0.006	0.853	45.034	0.016	0.430	0.044	1.084

nd = element not detected.
* = outlier.

TABLE 12b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GB18-3	0.137	0.309	44.139	0.021	0.500	0.003	0.477
GB18-5	0.046	0.156	45.962	0.005	0.445	nd	0.409
GB18-8	0.013	0.108	43.862	nd	0.453	0.019	0.300
GB18-9	0.034	0.097	44.384	0.039	0.479	0.002	0.534
GB19-2	0.054	0.032	42.686	0.005	0.443	0.008	0.618
GB19-5	0.073	0.069	41.654	0.001	0.420	0.019	0.578
GB19-10	nd	0.149	42.957	nd	0.415	nd	0.480
GB19-12	0.032	0.006	42.707	nd	0.428	0.011	0.559
GB110-5	0.059	nd	41.717	0.003	0.419	0.012	0.544
GB110-6	0.058	0.088	43.408	0.006	0.434	0.006	0.309
GB110-10	0.073	0.151	44.192	nd	0.438	0.013	0.404
GB110-11	0.064	0.080	42.496	0.009	0.447	0.011	0.557

nd = element not detected.
* = outlier.

TABLE 12b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GB11-3	0.452	21.169	0.145	0.023	8.246
GB11-6	0.389	21.660	0.148	0.018	9.550
GB11-7	0.369	24.251	0.125	0.006	9.674
GB11-12	0.475	20.280	0.107	nd	8.844
GB12-3	0.522	23.886	0.264	0.007	9.388
GB12-4	0.464	21.824	0.251	0.038	9.397
GB12-11	0.489	22.907	0.340	0.051	9.120
GB12-12	0.438	21.820	2.345*	0.026	9.406
GB13-4	0.454	25.081	0.213	0.036	9.884
GB13-5	0.495	22.432	0.341	0.009	9.560
GB13-7	0.513	24.460	0.200	0.011	8.356*
GB13-12	0.480	22.933	0.191	nd	9.449
GB14-2	0.447	23.351	0.202	0.040	9.866
GB14-7	0.487	21.237	0.229	0.046	9.358
GB14-11	0.439	23.800	0.186	0.026	10.305
GB15-5	0.473	21.312	0.177	0.021	9.339
GB15-9	0.420	19.794	0.166	0.038	9.776
GB15-11	0.462	18.945	0.151	0.029	9.373
GB16-3	0.502	20.705	0.117	0.021	9.020
GB16-7	0.453	22.046	0.244	0.039	9.445
GB16-8	0.438	21.427	0.210	0.006	9.375
GB17-3	0.517	22.601	0.214	0.021	9.563
GB17-5	0.597	22.450	0.265	0.026	10.025
GB17-8	0.987*	20.335	0.161	0.028	8.651
GB17-11	0.470	20.906	0.182	0.022	8.921

nd = element not detected.
* = outlier.

TABLE 12b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GB18-3	0.521	27.516*	0.262	0.009	9.658
GB18-5	0.473	22.587	0.172	0.038	9.481
GB18-8	0.541	20.843	0.185	0.024	9.246
GB18-9	0.455	20.356	0.198	0.042	9.277
GB19-2	0.442	22.695	0.144	0.025	9.406
GB19-5	0.409	21.538	0.172	nd	9.336
GB19-10	0.452	25.659	0.194	0.040	9.454
GB19-12	0.426	22.453	0.151	0.039	8.855
GB110-5	0.460	21.572	0.161	0.027	8.793
GB110-6	0.432	22.316	0.195	nd	9.060
GB110-10	0.394	21.648	0.151	0.024	9.003
GB110-11	0.442	22.184	0.149	0.038	9.268

nd = element not detected.
* = outlier.

TABLE 12c: Analytical results of the within-box GB1 study using W-L excitation. Assay time is 1,000 live seconds. Element concentration results are in µg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB11-3	470.076	1197.322	58.820	11.553	nd	6.095	11.918
GB11-6	690.176	1554.775	107.917	29.985	5.786	20.347	27.115
GB11-7	484.729	1036.050	65.060	12.862	1.317	6.814	13.143
GB11-12	594.089	1360.878	98.964	29.867	8.639	19.861	18.969
GB12-3	773.143	1648.344	122.677	54.470	9.618	27.050	119.438
GB12-4	735.929	1632.255	103.864	35.032	3.266	25.775	21.087
GB12-11	925.583	2097.906	89.782	45.953	7.587	34.247	28.211
GB12-12	731.756	1688.250	99.891	32.371	5.705	27.348	23.269
GB13-4	915.939	2290.031	137.796	68.490	1.979	23.550	35.747
GB13-5	810.490	2136.950	109.331	48.993	2.004	19.978	34.237
GB13-7	1043.185	3311.665	131.133	51.769	1.897	25.602	29.027
GB13-12	896.679	2149.080	131.971	55.117	4.805	18.400	22.582
GB14-2	937.082	2180.184	138.415	63.210	10.972	21.555	40.354
GB14-6	973.648	2627.178	125.664	49.285	2.723	20.598	25.012
GB14-7	888.967	2331.734	122.944	51.551	3.455	20.704	27.780
GB14-11	786.700	1972.936	101.428	42.938	4.577	17.327	22.479
GB15-1	851.341	2306.001	121.089	54.463	3.421	28.010	27.754
GB15-5	755.672	1890.322	114.528	43.301	4.723	20.681	23.057
GB15-9	799.223	1986.862	116.150	57.278	2.069	21.975	22.573
GB15-11	826.054	2076.619	130.674	59.773	17.441	40.303	39.820
GB16-2	645.651	1584.517	105.265	47.851	16.466	37.012	32.180
GB16-3	462.848	847.063	70.816	18.086	nd	8.551	14.440
GB16-7	576.659	1451.813	89.653	25.292	1.831	15.270	17.257
GB16-8	512.930	1088.489	73.699	20.797	10.014	14.916	16.306

nd = element not detected.
* = outlier.

TABLE 12c: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB17-3	668.784	1586.094	102.259	38.328	3.752	20.682	25.002
GB17-5	967.408	2112.732	149.058	64.435	10.806	24.959	37.565
GB17-8	513.419	1112.100	75.128	36.050	nd	19.203	16.738
GB17-11	527.253	1174.515	70.939	14.612	nd	8.707	13.982
GB18-3	1203.339	3288.871	169.790	84.466	1.930	57.397	46.062
GB18-5	780.735	2112.111	121.921	44.980	nd	17.759	28.577
GB18-8	720.371	1742.174	111.062	36.149	nd	13.708	19.265
GB18-9	883.768	2035.132	124.539	46.671	nd	21.998	29.723
GB19-2	631.629	1285.839	114.602	35.465	6.497	26.284	25.768
GB19-5	593.255	1248.418	74.246	16.648	nd	13.052	17.214
GB19-10	610.883	1517.513	102.237	32.618	6.914	16.676	18.749
GB19-12	649.643	1543.660	118.261	40.202	5.967	28.233	23.405
GB110-5	514.699	951.816	82.141	19.286	nd	10.667	12.162
GB110-6	784.460	1807.145	124.719	43.429	6.010	27.437	29.812
GB110-10	657.574	1734.162	87.992	19.529	3.306	23.072	16.491
GB110-11	599.179	1309.591	122.208	35.635	3.450	26.761	25.460

nd = element not detected.
* = outlier.

TABLE 12c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GB11-3	697.598	0.017	0.241	86.489	0.131	8.066
GB11-6	790.227	0.026	0.239	116.619	0.251	6.776
GB11-7	820.789	nd	0.307	102.242	0.150	8.028
GB11-12	577.037	0.029	0.199	95.767	0.254	6.025
GB12-3	641.303	0.273	0.251	109.389	0.452	5.863
GB12-4	834.256	0.190	0.302	114.944	0.320	7.258
GB12-11	898.878	1.599	0.470	110.747	1.162	8.117
GB12-12	741.516	0.293	0.231	108.535	0.788	6.832
GB13-4	834.103	0.794	0.308	130.948	0.218	6.370
GB13-5	812.026	0.566	0.270	121.550	0.186	6.681
GB13-7	718.239	0.688	0.257	118.854	0.248	6.043
GB13-12	819.062	0.823	0.332	127.607	0.291	6.419
GB14-2	793.033	1.070	0.364	132.517	0.257	5.984
GB14-6	849.689	0.764	0.389	131.821	0.316	6.446
GB14-7	824.320	1.533	0.372	132.188	0.231	6.236
GB14-11	903.896	0.195	0.295	125.672	0.233	7.192
GB15-1	759.375	0.965	0.314	122.572	0.258	6.195
GB15-5	754.872	0.623	0.286	113.793	0.249	6.634
GB15-9	787.772	0.477	0.307	120.573	0.264	6.534
GB15-11	778.592	0.232	0.359	121.425	0.317	6.412
GB16-2	791.295	0.096	0.302	110.079	0.184	7.188
GB16-3	636.047	nd	0.216	80.676	0.110	7.884
GB16-7	802.072	0.116	0.272	104.032	0.158	7.710
GB16-8	733.187	nd	0.167	90.253	0.094	8.124

nd = element not detected.
* = outlier.

TABLE 12c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GB17-3	757.897	0.067	0.253	116.118	0.159	6.527
GB17-5	841.259	nd	0.400	137.774	0.155	6.106
GB17-8	708.114	0.037	0.198	85.130	0.111	8.318
GB17-11	700.532	0.071	0.203	84.930	0.103	8.248
GB18-3	966.808	1.044	0.437	155.854	0.282	6.203
GB18-5	766.463	0.806	0.283	116.020	0.234	6.606
GB18-8	761.404	0.361	0.242	114.380	0.203	6.657
GB18-9	890.431	0.463	0.338	131.379	0.242	6.778
GB19-2	637.392	nd	0.237	102.684	0.157	6.207
GB19-5	791.632	nd	0.204	100.279	0.122	7.894
GB19-10	662.479	0.025	0.309	102.065	0.109	6.491
GB19-12	692.159	0.168	0.286	125.933	0.173	5.496
GB110-5	658.059	nd	0.187	86.195	0.098	7.634
GB110-6	726.910	0.021	0.241	123.990	0.250	5.863
GB110-10	776.839	0.028	0.265	103.301	0.200	7.520
GB110-11	607.243	0.178	0.267	114.644	0.200	5.297

nd = element not detected.
* = outlier.

TABLE 13a: Analytical results of the within-box GE1 study using Mo-K excitation. Assay time is 1,000 live seconds. Element concentration results are in μg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GE11-1	803.666	2482.191*	131.079	19.155	nd	5.958	13.916
GE11-3	692.576	1587.893	116.854	nd	nd	nd	40.167
GE11-10	605.535	1350.982	103.923	20.864	nd	8.767	35.120
GE11-12	747.880	1522.537	122.935	14.213	nd	10.077	21.515
GE12-1	617.831	1269.920	126.692	22.364	nd	11.242	13.562
GE12-6	730.116	1488.537	130.663	23.551	nd	5.864	12.047
GE12-8	598.929	1425.954	123.430	19.899	nd	8.068	13.178
GE12-9	660.133	1358.605	122.794	nd	nd	nd	13.995
GE13-1	659.842	1597.339	115.649	nd	nd	nd	15.906
GE13-3	568.392	1437.860	126.544	37.215	1.474	16.833	25.919
GE13-11	705.549	1629.329	121.421	23.620	nd	14.552	15.223
GE14-1	597.795	1597.259	123.734	nd	nd	nd	14.473
GE14-6	927.860*	1930.850	146.958	48.298	nd	19.769	12.547
GE14-10	753.640	1766.078	116.567	0.415	nd	0.755	40.634
GE14-12	705.356	1522.100	113.724	21.282	nd	9.328	17.900
GE15-1	762.679	1696.013	130.203	7.383	nd	nd	72.231*
GE15-3	642.444	1408.762	115.161	18.023	nd	8.066	12.466
GE15-9	597.099	1364.379	125.484	17.124	nd	9.782	13.745
GE15-11	593.344	1367.134	117.913	15.120	nd	20.543	15.785
GE16-1	689.742	1472.085	124.126	41.670	nd	10.044	92.357*
GE16-2	780.975	1843.734	129.557	nd	nd	nd	14.053
GE16-9	659.178	1415.503	123.158	18.879	nd	10.779	13.240
GE16-11	756.819	1645.977	125.720	14.762	nd	7.831	17.576
GE17-1	747.916	1733.505	117.185	nd	nd	nd	26.529
GE17-6	753.542	1558.299	139.657	16.634	nd	8.482	14.942
GE17-9	643.471	1541.860	127.596	16.381	nd	10.556	12.616
GE17-10	671.017	1462.031	122.019	34.299	nd	20.296	12.812

nd = element not detected.
* = outlier.

TABLE 13a: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GE18-1	627.613	1436.285	117.760	nd	nd	nd	35.917
GE18-3	745.403	1591.063	111.969	nd	nd	nd	16.388
GE18-7	658.212	1528.384	107.831	nd	nd	nd	23.995
GE19-1	756.156	1545.516	111.107	6.555	nd	8.832	16.914
GE19-2	779.461	1559.284	97.654	nd	nd	nd	16.670
GE19-7	741.282	1613.637	101.087	nd	nd	nd	28.368
GE19-8	753.394	1908.742	126.815	29.137	nd	14.765	20.346
GE110-4	790.766	1934.100	140.744	32.144	nd	24.987	19.795
GE110-5	709.918	1500.374	112.159	nd	nd	nd	28.703
GE110-8	771.675	1795.307	109.390	nd	nd	nd	15.371
GE111-3	628.155	1388.673	116.195	16.432	nd	7.567	12.696
GE111-4	604.060	1278.367	112.949	36.056	nd	18.953	13.882
GE111-7	682.425	1513.494	119.228	nd	nd	nd	19.677
GE112-4	670.361	1459.283	114.418	14.861	nd	6.353	10.850
GE112-8	641.142	1424.759	113.107	18.959	nd	13.813	14.391
GE112-9	714.199	1492.041	118.346	nd	nd	nd	21.251
GE113-5	673.344	1542.042	121.931	nd	nd	1.569	13.121
GE113-7	688.505	1501.599	125.564	nd	nd	nd	34.993
GE113-11	686.488	1463.210	111.910	nd	nd	2.557	16.843

nd = element not detected.
* = outlier.

TABLE 13a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GE11-1	897.221	nd	0.272	21.140	0.152	1.079	45.653
GE11-3	926.732	nd	0.285	20.104	0.269	1.057	53.302
GE11-10	849.451	nd	0.331	18.177	0.214	1.116	50.511
GE11-12	883.314	0.304	0.322	19.761	0.181	1.485	50.279
GE12-1	849.813	nd	0.299	19.063	0.123	1.090	48.455
GE12-6	803.177	nd	0.246	19.120	0.042	0.967	47.714
GE12-8	862.878	nd	0.264	19.842	0.086	1.062	47.584
GE12-9	840.547	nd	0.240	17.597	0.050	1.167	49.450
GE13-1	935.477	1.896	0.381	25.973	1.592	2.123	49.563
GE13-3	903.488	nd	0.264	19.495	nd	1.112	48.686
GE13-11	953.140	nd	0.265	21.741	0.039	1.203	48.181
GE14-1	919.791	nd	0.218	19.163	nd	1.227	49.329
GE14-6	968.586	nd	0.316	22.221	0.071	1.122	45.671
GE14-10	1034.136	nd	0.251	21.440	0.066	1.173	48.935
GE14-12	975.389	nd	0.271	22.316	nd	1.181	45.124
GE15-1	955.488	nd	0.282	22.435	0.070	1.144	73.096
GE15-3	813.613	nd	0.194	17.774	nd	1.089	46.072
GE15-9	848.566	nd	0.257	19.144	0.027	1.175	46.709
GE15-11	882.491	nd	0.265	19.102	0.016	1.238	47.415
GE16-1	797.821	nd	0.204	19.519	0.032	1.404	50.230
GE16-2	1015.776	nd	0.326	24.120	0.004	1.706	46.600
GE16-9	914.873	nd	0.273	18.788	0.009	1.217	46.372
GE16-11	920.996	nd	0.265	19.654	0.012	1.233	46.843
GE17-1	1003.257	nd	0.273	20.200	0.001	1.254	51.227
GE17-6	881.425	nd	0.208	19.226	nd	4.510*	49.556
GE17-9	848.224	nd	0.245	17.170	0.052	1.146	46.886
GE17-10	826.799	nd	0.371	18.203	0.046	1.345	48.859

nd = element not detected.
* = outlier.

TABLE 13a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GE18-1	788.138	nd	0.312	18.166	nd	1.038	54.588
GE18-3	908.951	nd	0.293	18.757	nd	1.126	47.793
GE18-7	908.283	nd	0.237	20.465	nd	1.258	49.375
GE19-1	879.337	nd	0.422	30.010	0.257	1.147	47.548
GE19-2	891.727	nd	0.237	19.998	nd	1.157	45.883
GE19-7	1038.780	nd	0.302	21.607	0.022	1.125	45.993
GE19-8	1079.542	nd	0.349	25.844	0.011	1.470	50.385
GE110-4	1016.257	nd	0.388	26.856	0.022	1.120	55.806
GE110-5	831.347	nd	0.348	18.851	nd	1.144	50.982
GE110-8	877.179	nd	0.304	26.327	nd	1.118	46.761
GE111-3	855.377	nd	0.281	19.005	nd	1.129	45.106
GE111-4	744.186	nd	0.279	16.908	nd	0.978	47.409
GE111-7	812.998	nd	0.223	18.255	nd	1.088	46.266
GE112-4	923.463	nd	0.250	19.004	nd	1.001	46.820
GE112-8	752.260	nd	0.293	17.233	nd	1.268	47.691
GE112-9	853.313	nd	0.225	19.580	nd	1.053	46.896
GE113-5	844.099	nd	0.341	19.270	0.023	1.120	45.909
GE113-7	843.753	nd	0.222	19.063	nd	1.192	49.595
GE113-11	784.541	nd	0.246	17.649	0.069	1.064	46.501

nd = element not detected.
* = outlier.

TABLE 13a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GE11-1	nd	0.608	11.807	0.130	42.443
GE11-3	nd	0.631	22.922	0.082	46.096
GE11-10	nd	0.610	12.921	0.106	46.732
GE11-12	nd	0.590	8.099	0.072	44.700
GE12-1	nd	0.586	12.277	0.037	44.580
GE12-6	nd	0.577	15.974	0.068	42.007
GE12-8	nd	0.585	7.938	0.084	43.487
GE12-9	nd	0.626	7.508	0.099	47.766
GE13-1	nd	0.548	4.048	0.111	36.017
GE13-3	nd	0.573	1.651	0.085	46.345
GE13-11	nd	0.610	3.978	0.103	43.840
GE14-1	nd	0.611	1.546	0.100	47.998
GE14-6	nd	0.599	1.243	0.073	43.590
GE14-10	nd	0.654	2.680	0.114	48.234
GE14-12	nd	0.607	3.568	0.070	43.709
GE15-1	nd	0.702	0.899	0.058	42.590
GE15-3	nd	0.590	0.681	0.056	45.776
GE15-9	nd	0.582	3.358	0.533	44.327
GE15-11	nd	0.571	0.384	0.085	46.200
GE16-1	nd	0.625	1.812	0.073	40.873
GE16-2	nd	0.616	0.573	0.039	42.114
GE16-9	nd	0.636	0.585	0.075	48.695
GE16-11	nd	0.640	0.408	0.121	46.861
GE17-1	nd	0.611	0.441	0.109	49.665
GE17-6	nd	0.562	0.473	0.090	45.846
GE17-9	nd	0.535	0.405	0.139	49.402
GE17-10	0.004	0.650	0.342	0.079	45.422

nd = element not detected.
* = outlier.

TABLE 13a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GE18-1	nd	0.623	0.829	0.108	43.385
GE18-3	nd	0.599	0.462	0.054	48.460
GE18-7	nd	0.649	0.971	0.081	44.381
GE19-1	nd	0.600	0.425	0.077	29.301
GE19-2	nd	0.595	0.512	0.080	44.590
GE19-7	nd	0.655	1.001	0.255	48.077
GE19-8	nd	0.656	1.540	0.071	41.771
GE110-4	nd	0.624	0.408	0.063	37.841
GE110-5	nd	0.653	0.485	0.039	44.100
GE110-8	nd	0.672	0.576	0.084	33.319
GE111-3	nd	0.616	0.657	0.052	45.008
GE111-4	0.004	0.595	0.278	0.040	44.013
GE111-7	nd	0.595	0.330	0.130	44.534
GE112-4	nd	0.572	2.763	0.085	48.594
GE112-8	nd	0.600	0.654	0.067	43.653
GE112-9	nd	0.607	0.122	0.077	43.581
GE113-5	nd	0.629	0.112	0.073	43.805
GE113-7	nd	0.647	0.125	0.070	44.262
GE113-11	nd	0.622	0.256	0.087	44.452

nd = element not detected.
* = outlier.

TABLE 13b: Analytical results of the within-box GE1 study using W-*brems* excitation. Assay time is 1,000 live seconds. Element concentration results are in µg/g.

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GE11-1	nd	8.448	15.288	1056.972	1.477	nd	24.097
GE11-3	nd	0.031	41.261	1069.683	1.552	0.287	22.808
GE11-10	nd	11.622	37.008	1060.551	2.275	0.027	22.370
GE11-12	nd	11.974	24.289	1110.177	1.826	0.074	22.604
GE12-1	nd	12.941	16.323	1020.867	1.072	0.266	21.597
GE12-6	nd	9.212	9.771	1052.142	1.315	0.104	23.414
GE12-8	5.257	12.179	13.629	1093.265	1.429	0.126	24.034
GE12-9	nd	1.899	13.309	1096.553	1.646	nd	20.822
GE13-1	nd	nd	16.115	1020.807	4.649*	nd	28.814
GE13-3	65.546	20.281	26.931	1142.199	1.130	0.088	22.889
GE13-11	37.357	18.826	15.282	1127.248	1.538	nd	24.114
GE14-1	nd	0.945	14.343	1063.824	0.840	nd	21.187
GE14-6	69.508	24.197	13.646	1066.220	0.688	0.138	22.600
GE14-10	nd	2.337	43.444	1124.945	0.740	0.078	22.078
GE14-12	23.396	14.115	20.866	1104.685	1.042	0.250	25.254
GE15-1	nd	nd	73.712	1072.582	0.857	nd	24.283
GE15-3	23.445	11.839	13.096	1077.457	0.917	0.051	21.790
GE15-9	nd	13.565	13.871	1008.187	1.493	0.010	21.953
GE15-11	32.083	21.569	18.883	1079.241	0.953	0.108	21.723
GE16-1	70.383	13.233	90.505	1093.586	0.929	nd	24.614
GE16-2	nd	0.055	15.540	1122.536	1.100	0.137	23.722
GE16-9	31.659	15.714	14.984	1149.366	0.855	0.123	22.828
GE16-11	26.322	9.862	18.762	1008.218	1.097	0.248	21.036
GE17-1	nd	nd	28.287	1068.269	1.328	0.132	21.811
GE17-6	23.170	7.880	16.641	958.705	0.991	0.117	21.309
GE17-9	20.197	14.912	16.572	1058.935	0.979	nd	20.380
GE17-10	45.782	23.468	13.012	1086.037	0.661	0.357	22.015

nd = element not detected.
* = outlier.

TABLE 13b: (Continued)

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GE18-1	nd	nd	37.231	957.767	1.102	0.180	21.067
GE18-3	nd	nd	16.346	1102.406	1.101	0.131	22.093
GE18-7	nd	nd	23.943	1025.279	1.488	0.175	22.441
GE19-1	16.960	9.885	18.399	1030.302	1.100	0.227	32.466
GE19-2	nd	nd	17.234	1068.349	1.036	nd	21.503
GE19-7	nd	2.544	28.468	1081.455	1.150	0.114	22.542
GE19-8	49.344	14.786	16.807	1111.108	1.810	0.153	26.214
GE110-4	34.781	27.549	20.288	1063.918	1.029	0.260	27.706
GE110-5	nd	0.118	29.514	1031.248	nd	0.153	21.626
GE110-8	nd	1.806	15.378	1081.852	0.569	0.206	28.287
GE111-3	12.382	9.855	11.454	1030.105	1.105	0.041	21.889
GE111-4	78.189	22.540	14.527	957.360	1.252	0.136	20.700
GE111-7	nd	nd	21.212	991.509	1.043	0.028	21.578
GE112-4	25.067	10.481	11.367	996.641	0.917	nd	20.749
GE112-8	12.754	15.876	15.886	998.356	1.916	nd	21.306
GE112-9	nd	nd	19.685	1034.944	1.390	0.041	22.898
GE113-5	nd	4.953	14.829	1007.339	0.938	nd	21.454
GE113-7	nd	0.395	35.278	991.573	0.805	nd	20.481
GE113-11	nd	5.998	16.239	1095.895	0.895	0.104	22.067

nd = element not detected.
* = outlier.

TABLE 13b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GE11-1	0.176	1.113	49.262	0.021	0.550	0.027	12.207
GE11-3	0.232	1.106	56.990	nd	0.630	0.039	21.751
GE11-10	0.230	1.211	54.954	nd	0.555	0.040	13.920
GE11-12	0.142	1.594	54.266	0.019	0.527	0.015	8.353
GE12-1	0.165	1.252	51.301	nd	0.556	0.028	11.997
GE12-6	0.089	1.087	52.592	0.011	0.549	0.021	15.326
GE12-8	0.116	1.170	52.268	0.008	0.532	0.030	7.907
GE12-9	0.057	1.347	54.709	nd	0.543	0.008	7.246
GE13-1	1.766	2.219	53.737	nd	0.544	0.011	4.116
GE13-3	0.013	1.183	53.021	nd	0.559	0.013	1.924
GE13-11	0.019	1.296	51.846	nd	0.552	0.025	3.982
GE14-1	0.059	1.278	52.892	nd	0.541	nd	1.518
GE14-6	0.051	1.132	49.337	0.016	0.532	nd	1.193
GE14-10	0.095	1.269	52.563	nd	0.614	0.013	2.588
GE14-12	0.026	1.236	49.652	0.013	0.506	0.005	3.122
GE15-1	0.068	1.266	77.815*	nd	0.734	0.003	0.921
GE15-3	0.012	1.178	50.803	nd	0.569	0.012	0.847
GE15-9	0.051	1.231	50.599	nd	0.594	0.017	3.296
GE15-11	0.075	1.162	50.279	nd	0.546	0.010	0.534
GE16-1	0.013	1.431	54.580	nd	0.576	0.013	1.838
GE16-2	0.069	1.678	49.722	nd	0.569	0.004	0.556
GE16-9	0.007	1.342	50.183	nd	0.606	0.025	0.663
GE16-11	0.036	1.397	50.645	nd	0.587	0.010	0.461
GE17-1	0.031	1.347	54.446	nd	0.604	0.008	0.487
GE17-6	0.109	4.770	53.769	nd	0.566	nd	0.522
GE17-9	0.055	1.296	51.208	0.001	0.522	nd	0.519
GE17-10	0.133	1.362	52.044	0.017	0.607	0.003	0.456

nd = element not detected.
* = outlier.

TABLE 13b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GE18-1	nd	1.182	60.150	nd	0.554	0.020	0.933
GE18-3	nd	1.254	52.691	nd	0.555	0.006	0.552
GE18-7	0.015	1.426	53.971	nd	0.560	0.001	1.030
GE19-1	0.216	1.053	50.987	0.013	0.580	0.011	0.506
GE19-2	0.061	1.252	49.097	0.003	0.558	0.008	0.564
GE19-7	0.062	1.234	49.734	nd	0.575	0.006	0.936
GE19-8	0.037	1.565	53.920	0.008	0.626	0.011	1.524
GE110-4	0.017	1.279	58.776	nd	0.607	nd	0.458
GE110-5	nd	1.105	54.318	nd	0.609	nd	0.530
GE110-8	nd	1.181	49.612	nd	0.578	0.001	0.524
GE111-3	0.025	1.280	50.040	nd	0.527	0.007	0.785
GE111-4	nd	1.100	52.111	0.008	0.569	nd	0.398
GE111-7	nd	1.068	50.941	nd	0.564	0.002	0.450
GE112-4	0.035	1.133	50.926	0.003	0.532	0.016	2.587
GE112-8	nd	1.502	52.472	nd	0.587	0.005	0.716
GE112-9	0.004	1.155	51.228	nd	0.560	0.004	0.212
GE113-5	0.034	1.229	50.330	nd	0.544	0.007	0.216
GE113-7	0.003	1.229	53.134	nd	0.599	nd	0.205
GE113-11	0.021	1.163	47.006	nd	0.543	0.002	0.319

nd = element not detected.
* = outlier.

TABLE 13b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GE11-1	0.165	36.300	0.031	0.030	43.863
GE11-3	0.105	36.343	0.030	0.061	46.899
GE11-10	0.175	34.127	0.032	0.038	47.409
GE11-12	0.110	32.295	0.046	0.059	49.114
GE12-1	0.140	33.686	0.062	0.028	47.268
GE12-6	0.093	34.732	0.050	0.045	44.937
GE12-8	0.090	33.051	0.021	0.037	45.489
GE12-9	0.165	32.971	0.057	0.028	52.663
GE13-1	0.098	33.667	0.869	0.245	35.428
GE13-3	0.159	33.109	nd	0.034	49.902
GE13-11	0.147	33.439	0.008	0.013	46.746
GE14-1	0.174	35.289	0.064	0.003	50.212
GE14-6	0.120	35.176	0.009	0.017	47.177
GE14-10	0.117	35.616	0.061	0.032	50.954
GE14-12	0.127	32.916	0.020	nd	43.743
GE15-1	0.105	37.494	0.012	0.019	44.170
GE15-3	0.166	34.603	0.022	0.032	49.447
GE15-9	0.618*	35.367	0.140	0.011	45.925
GE15-11	0.197	32.312	0.010	0.003	49.681
GE16-1	0.159	33.233	0.152	0.056	44.429
GE16-2	0.063	35.962	0.443	0.038	47.320
GE16-9	0.163	34.863	0.022	0.049	50.348
GE16-11	0.183	34.693	0.010	0.024	47.928
GE17-1	0.155	34.696	0.060	0.011	48.978
GE17-6	0.130	34.280	2.826	0.203	44.990
GE17-9	0.195	31.643	0.026	0.013	51.960
GE17-10	0.043	35.771	0.112	0.002	49.332

nd = element not detected.
* = outlier.

TABLE 13b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GE18-1	0.125	34.343	0.786	0.010	45.463
GE18-3	0.068	38.438	0.300	0.049	49.899
GE18-7	0.139	37.482	0.309	2.109	45.689
GE19-1	0.190	36.409	0.197	0.013	31.735
GE19-2	0.140	35.973	0.082	0.008	49.683
GE19-7	0.288	38.173	0.066	0.018	47.974
GE19-8	0.081	40.192	0.142	0.877	42.387
GE110-4	0.166	37.638	0.138	0.065	38.400
GE110-5	0.079	36.366	0.058	0.006	47.686
GE110-8	0.118	36.433	0.067	nd	38.246
GE111-3	0.146	35.611	0.067	0.031	47.060
GE111-4	0.057	35.740	0.067	0.046	46.249
GE111-7	0.208	37.351	3.064	0.012	45.950
GE112-4	0.139	34.311	0.262	0.015	48.033
GE112-8	0.112	36.478	0.334	0.042	46.858
GE112-9	0.157	35.089	0.020	0.003	45.198
GE113-5	0.142	37.754	0.074	0.016	46.953
GE113-7	0.121	37.397	0.131	0.029	48.415
GE113-11	0.108	36.143	0.034	0.061	49.662

nd = element not detected.
* = outlier.

TABLE 13c: Analytical results of the within-box GE1 study using W-L excitation. Assay time is 1000 live seconds. Element concentration results are in µg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GE11-1	665.221	1617.447	134.806	68.630	3.573	23.241	31.175
GE11-3	677.286	1492.040	120.996	25.231	2.053	8.442	70.267
GE11-10	657.449	1440.838	134.293	71.549	3.777	29.496	72.144
GE11-12	597.358	1467.306	148.415	74.548	7.958	35.922	51.598
GE12-1	525.362	1131.860	132.504	73.423	0.993	32.917	31.980
GE12-6	571.821	1333.975	129.048	73.073	3.138	25.019	29.172
GE12-8	631.186	1430.429	132.422	61.380	1.022	19.950	29.316
GE12-9	597.316	1384.002	140.017	17.547	4.196	15.249	35.153
GE13-1	593.670	1397.428	129.043	11.179	nd	11.779	35.392
GE13-3	589.270	1506.279	129.002	102.354	7.795	40.978	53.814
GE13-11	686.580	1715.960	141.656	86.908	6.107	41.463	38.500
GE14-1	569.226	1505.897	113.815	7.339	0.335	9.275	26.310
GE14-6	733.329	2042.438	134.356	107.076	1.078	43.101	28.802
GE14-10	674.552	1711.310	152.094	36.832	4.051	23.039	90.681
GE14-12	569.828	1266.435	95.456	49.486	nd	19.093	30.740
GE15-1	765.605	1678.231	144.396	53.939	4.188	12.086	147.762
GE15-3	558.515	1330.422	131.157	67.118	0.649	26.036	30.101
GE15-9	668.746	1496.967	141.956	82.776	3.540	44.999	43.687
GE15-11	567.152	1323.914	107.991	53.136	2.738	42.210	31.535
GE16-1	657.039	1522.304	137.902	126.769	2.765	33.912	179.801
GE16-2	765.476	1880.354	131.054	12.662	3.753	12.129	31.408
GE16-9	604.824	1415.310	130.301	73.305	1.774	33.842	34.197
GE16-11	721.137	1745.185	155.114	88.933	1.320	43.111	51.413
GE17-1	712.466	1765.694	142.537	17.971	0.796	12.939	61.197
GE17-6	625.112	1343.833	139.576	68.358	3.397	31.469	36.570
GE17-9	587.530	1518.614	134.035	60.052	3.241	35.649	30.771
GE17-10	621.476	1511.482	130.702	98.652	nd	49.900	31.051

nd = element not detected.
* = outlier.

TABLE 13c: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GE18-1	569.788	1391.801	114.219	17.086	nd	3.541	63.920
GE18-3	632.365	1483.463	96.311	2.438	nd	2.427	26.651
GE18-7	693.793	1641.054	125.014	19.070	nd	13.118	49.928
GE19-1	595.381	1495.756	129.105	60.861	4.547	33.854	45.759
GE19-2	678.920	1611.156	113.655	22.258	nd	13.708	48.559
GE19-7	623.876	1529.214	100.684	10.012	nd	8.985	43.868
GE19-8	867.029	2042.709	159.297	121.725	6.319	57.627	60.079
GE110-4	833.434	2024.469	148.779	101.450	6.056	71.991	53.154
GE110-5	673.657	1651.560	133.089	29.418	nd	14.206	70.078
GE110-8	699.340	1870.395	121.628	16.114	nd	12.255	36.397
GE111-3	549.585	1368.744	122.687	67.123	1.738	23.694	32.529
GE111-4	551.142	1231.565	115.657	102.244	1.619	42.062	31.971
GE111-7	598.165	1420.670	110.814	13.120	nd	8.776	40.913
GE112-4	607.851	1484.829	110.582	48.678	nd	19.022	24.224
GE112-8	601.590	1550.719	134.376	76.192	2.849	42.795	37.776
GE112-9	574.044	1418.425	125.223	9.160	nd	8.670	42.901
GE113-5	620.356	1418.350	126.949	20.610	nd	16.457	33.987
GE113-7	582.555	1295.124	128.012	23.490	nd	12.651	70.608
GE113-11	609.386	1580.090	113.483	14.093	nd	19.204	31.903

nd = element not detected.
* = outlier.

TABLE 13c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GE11-1	897.266	0.632	0.304	30.669	0.386	29.256
GE11-3	926.777	0.529	0.259	27.348	0.444	33.888
GE11-10	849.495	0.503	0.225	26.054	0.379	32.605
GE11-12	883.357	1.968	0.343	27.614	0.420	31.989
GE12-1	849.856	0.609	0.227	25.602	0.343	33.195
GE12-6	803.222	0.148	0.233	24.713	0.249	32.502
GE12-8	862.923	0.368	0.238	27.170	0.307	31.761
GE12-9	840.592	0.111	0.228	25.760	0.239	32.632
GE13-1	935.520	2.886	0.221	30.979	1.463	30.199
GE13-3	903.533	0.393	0.193	26.129	0.217	34.580
GE13-11	953.187	0.794	0.311	32.610	0.175	29.230
GE14-1	919.838	0.136	0.198	25.516	0.184	36.050
GE14-6	968.631	0.037	0.345	33.548	0.275	28.873
GE14-10	1034.183	0.375	0.251	28.922	0.289	35.757
GE14-12	975.437	0.088	0.273	33.314	0.136	29.280
GE15-1	955.536	0.498	0.368	34.784	0.298	27.471
GE15-3	813.660	0.016	0.274	22.998	0.182	35.380
GE15-9	848.614	0.456	0.302	31.227	0.294	27.175
GE15-11	882.537	0.111	0.265	25.234	0.212	34.975
GE16-1	797.869	0.317	0.190	30.023	0.330	26.575
GE16-2	1015.819	0.236	0.340	33.743	0.224	30.104
GE16-9	914.919	0.200	0.234	26.310	0.232	34.774
GE16-11	921.039	0.337	0.350	32.013	0.337	28.770
GE17-1	1003.305	0.130	0.243	29.047	0.260	34.541
GE17-6	881.475	0.058	0.257	24.296	0.221	36.281
GE17-9	848.267	0.079	0.226	23.617	0.208	35.918
GE17-10	826.846	0.323	0.193	25.000	0.269	33.074

nd = element not detected.
* = outlier.

TABLE 13c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GE18-1	788.184	0.192	0.203	24.652	0.169	31.973
GE18-3	908.996	0.003	0.174	24.306	0.112	37.398
GE18-7	908.328	0.248	0.244	27.728	0.241	32.759
GE19-1	879.384	1.012	0.410	54.355	0.654	16.178
GE19-2	891.773	0.229	0.256	29.764	0.278	29.961
GE19-7	1038.825	0.048	0.196	27.033	0.122	38.428
GE19-8	1079.587	0.626	0.370	39.222	0.397	27.525
GE110-4	1016.306	0.527	0.376	45.504	0.318	22.335
GE110-5	831.398	0.369	0.279	28.757	0.270	28.911
GE110-8	877.224	0.343	0.352	45.318	0.156	19.357
GE111-3	855.422	0.269	0.233	27.479	0.217	31.131
GE111-4	744.232	0.319	0.156	22.801	0.183	32.641
GE111-7	813.045	0.163	0.237	25.176	0.158	32.295
GE112-4	923.510	0.034	0.212	25.042	0.159	36.879
GE112-8	752.308	0.434	0.199	24.695	0.224	30.465
GE112-9	853.359	0.508	0.182	26.363	0.198	32.369
GE113-5	844.143	0.060	0.247	25.625	0.202	32.942
GE113-7	843.799	0.331	0.171	23.521	0.222	35.874
GE113-11	784.583	0.131	0.164	24.046	0.258	32.628

nd = element not detected.
* = outlier.

TABLE 14a: Analytical results of the within-lot study using Mo-K excitation. Assay time is 1,000 live seconds. Element concentration results are in μg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB12-3	614.605	1393.832	110.181	18.204	nd	10.472	72.876
GB12-4	707.301	1605.663	102.240	13.644	nd	12.597	13.441
GB12-11	846.755	1979.661	111.954	35.236	1.302	26.983	25.033
GB12-12	743.373	1575.349	107.849	13.631	nd	13.825	15.217
GB13-4	855.293	2023.430	128.610	21.029	nd	4.838	16.164
GB13-5	794.689	1912.527	122.060	21.762	nd	7.129	20.431
GB13-7	750.140	2113.152	112.785	11.694	nd	8.364	15.618
GB13-12	726.955	1782.699	116.787	18.703	nd	3.888	11.701
GB17-3	628.526	1485.005	112.820	12.285	nd	8.554	15.344
GB17-5	743.542	1680.426	116.663	17.003	nd	8.637	17.750
GB17-8	670.193	1376.959	125.536	32.913	nd	17.813	16.380
GB17-11	639.381	1394.799	116.801	10.232	nd	10.834	16.214
GB23-4	659.324	1644.663	117.392	14.400	9.481	12.716	14.649
GB23-6	576.315	1285.945	98.841	15.676	nd	5.357	11.990
GB23-9	571.536	1526.917	107.618	6.781	nd	5.521	14.183
GB23-11	551.964	1637.395	110.780	15.676	5.229	6.877	12.795
GB25-2	658.636	1618.349	100.930	15.873	nd	5.860	13.913
GB25-4	640.907	1610.387	109.138	14.450	nd	6.835	14.901
GB25-11	679.823	1658.220	109.044	18.736	4.517	7.190	12.838
GB25-12	591.493	1454.542	102.107	17.259	6.201	6.814	12.324
GB27-1	644.243	1447.847	104.194	11.203	nd	4.656	12.882
GB27-12	600.817	1528.477	115.576	14.561	4.218	5.761	13.582
GB31-3	657.775	1572.371	117.178	22.708	nd	7.175	10.554
GB31-6	608.585	1446.702	110.721	19.423	nd	4.670	10.980
GB31-7	620.345	1481.996	102.464	19.850	nd	8.499	14.958
GB31-12	639.598	1552.977	104.526	16.534	nd	4.159	11.489
GB32-1	640.118	1510.151	99.483	21.521	nd	8.623	9.613
GB32-6	660.062	1619.907	117.539	23.783	nd	8.274	11.515
GB32-7	565.427	1490.230	109.228	16.751	nd	7.360	11.418
GB32-8	575.024	1433.237	108.885	23.952	nd	8.425	12.194
GB38-3	633.900	1554.009	114.397	12.540	nd	6.752	10.803
GB38-4	577.350	1420.188	104.526	15.336	nd	4.463	11.697
GB38-7	587.500	1495.578	110.673	20.974	nd	5.477	13.806
GB38-9	560.214	1444.331	102.890	18.976	nd	6.126	12.977

nd = element not detected.

* = outlier.

TABLE 14a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GB12-3	641.201	nd	0.147	73.475	0.258	0.189	40.387
GB12-4	834.155	nd	0.127	86.923	0.309	0.303	40.581
GB12-11	898.782	0.805	0.289	92.537	1.009	0.353	38.425
GB12-12	741.419	0.008	0.119	80.921	0.581	0.171	41.003
GB13-4	834.019	0.109	0.156	89.210	0.103	0.231	40.669
GB13-5	811.943	nd	0.234	85.553	0.109	0.370	36.597
GB13-7	718.159	nd	0.157	81.909	0.093	0.179	41.638
GB13-12	818.970	0.078	0.308	86.456	0.177	0.169	40.396
GB17-3	757.803	nd	0.203	83.504	0.101	0.249	41.643
GB17-5	841.161	nd	0.204	87.169	0.034	0.342	44.329
GB17-8	708.024	nd	0.145	77.388	0.074	0.367	46.611
GB17-11	700.428	nd	0.160	76.689	0.058	0.940	43.231
GB23-4	739.177	0.127	0.086	79.343	0.084	0.030	40.260
GB23-6	717.719	0.129	0.178	74.392	0.025	0.059	38.185
GB23-9	738.771	nd	0.138	78.484	0.162	0.030	39.425
GB23-11	716.539	0.264	0.166	76.324	0.089	0.102	43.424
GB25-2	748.830	nd	0.220	80.383	0.084	0.003	38.457
GB25-4	676.273	0.204	0.111	73.223	0.063	nd	37.519
GB25-11	785.700	0.202	0.160	84.774	0.125	0.096	40.141
GB25-12	687.223	0.170	0.115	73.347	0.043	0.060	41.054
GB27-1	641.986	0.033	0.152	70.365	0.052	0.137	39.307
GB27-12	710.487	0.073	0.103	77.759	0.075	0.274	41.608
GB31-3	713.027	nd	0.259	74.510	0.299	0.080	38.600
GB31-6	698.301	0.100	0.202	82.654	0.505	0.044	39.426
GB31-7	679.738	0.037	0.264	72.765	0.374	0.005	39.229
GB31-12	729.037	0.224	0.179	78.280	0.336	0.008	37.950
GB32-1	727.495	0.217	0.167	77.374	0.616	0.061	37.273
GB32-6	764.219	0.029	0.206	78.622	0.364	0.025	39.427
GB32-7	653.394	0.267	0.230	74.953	0.586	0.028	33.696
GB32-8	678.399	0.134	0.238	83.712	0.403	0.167	37.878
GB38-3	693.598	0.107	0.250	75.185	0.407	nd	37.183
GB38-4	649.305	0.224	0.246	72.359	0.556	nd	34.781
GB38-7	683.189	0.065	0.170	74.848	0.382	nd	37.307
GB38-9	657.357	0.174	0.220	72.648	0.377	0.008	36.514

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GB12-3	0.012	0.523	9.371	0.518	8.727
GB12-4	0.008	0.416	27.376	0.537	9.597
GB12-11	0.004	0.479	46.816	0.577	9.713
GB12-12	nd	0.423	16.145	0.436	9.162
GB13-4	0.005	0.492	1.970	0.514	9.349
GB13-5	0.009	0.481	0.384	0.497	9.491
GB13-7	0.004	0.505	0.253	0.542	8.768
GB13-12	nd	0.435	6.125	0.559	9.473
GB17-3	nd	0.470	1.370	0.509	9.075
GB17-5	nd	0.420	1.201	0.713	9.650
GB17-8	0.000	0.411	1.180	1.111	9.149
GB17-11	0.002	0.426	1.140	0.527	9.133
GB23-4	0.007	0.446	0.293	0.367	9.316
GB23-6	nd	0.466	0.122	0.381	9.648
GB23-9	nd	0.488	0.106	0.355	9.413
GB23-11	nd	0.458	0.228	0.471	9.388
GB25-2	nd	0.487	0.141	0.414	9.316
GB25-4	0.003	0.466	0.125	0.370	9.236
GB25-11	0.005	0.464	0.211	0.359	9.268
GB25-12	nd	0.435	0.232	0.362	9.369
GB27-1	nd	0.440	0.172	0.383	9.124
GB27-12	nd	0.461	0.115	0.386	9.137
GB31-3	nd	0.466	0.355	0.356	9.570
GB31-6	nd	0.432	0.298	0.363	8.449
GB31-7	0.018	0.449	0.366	0.351	9.342
GB31-12	nd	0.486	0.199	0.384	9.313
GB32-1	nd	0.449	0.429	0.340	9.402
GB32-6	0.002	0.472	0.199	0.394	9.720
GB32-7	nd	0.453	0.236	0.365	8.717
GB32-8	nd	0.515	0.268	0.402	8.104
GB38-3	nd	0.483	0.262	0.384	9.225
GB38-4	nd	0.460	0.226	0.418	8.973
GB38-7	nd	0.512	0.225	0.388	9.128
GB38-9	0.005	0.459	0.305	0.411	9.048

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GC13-2	1288.923	3125.825	119.126	5.518	nd	6.001	25.296
GC13-5	1307.102	2907.098	125.403	nd	nd	8.991	29.580
GC13-8	1347.737	3167.044	126.813	nd	nd	5.374	24.722
GC16-4	1288.340	3279.087	134.730	nd	nd	4.841	26.581
GC16-6	905.758	2328.099	89.386	nd	nd	1.509	17.870
GC16-11	1231.514	2763.528	107.819	nd	nd	5.211	22.368
GC16-12	1239.731	2659.731	115.016	nd	nd	5.484	18.112
GC19-5	1289.727	3369.704	118.635	nd	nd	12.359	20.819
GC19-6	1275.813	3546.586	103.504	8.290	nd	6.737	51.086
GC19-7	1154.491	2847.769	119.452	10.697	nd	6.607	67.597
GC19-11	1213.777	3008.710	102.872	5.048	nd	6.832	28.262
GC21-2	1261.928	2957.282	110.307	nd	nd	7.085	28.743
GC21-3	953.502	2345.368	95.204	nd	nd	2.641	23.645
GC21-8	1458.123	3547.774	119.209	0.330	nd	5.860	34.805
GC21-11	1153.045	2572.678	106.580	2.391	nd	7.577	42.333
GC26-5	1189.022	2894.551	107.646	nd	nd	5.676	37.113
GC26-6	1141.641	2794.932	111.727	10.654	nd	6.055	75.733
GC26-7	1307.981	3402.982	128.271	nd	nd	11.106	37.621
GC26-10	1080.293	2621.986	101.731	16.909	nd	nd	84.182
GC27-5	1100.057	2870.948	112.579	0.417	nd	5.655	38.330
GC27-6	1039.654	2638.909	120.833	nd	nd	8.114	19.327
GC27-7	1205.334	2820.708	108.688	nd	nd	14.329	50.073
GC27-9	1227.196	3115.791	125.242	nd	nd	5.289	44.566
GC34-2	1290.564	2965.872	97.569	30.763	nd	20.846	43.821
GC34-5	1222.663	2893.828	82.081	42.019	nd	22.588	49.390
GC34-8	1338.756	3273.460	125.441	39.375	nd	23.878	32.206
GC34-11	1082.010	2675.314	84.740	47.504	3.639	19.156	71.848
GC37-1	1148.287	2874.534	84.120	33.385	nd	21.199	36.176
GC37-2	1111.693	2658.801	112.812	53.004	nd	28.021	55.927
GC37-7	1180.182	2747.114	113.704	60.533	1.408	32.076	31.858
GC37-11	1125.372	2880.880	124.552	36.914	nd	20.827	67.305
GC38-3	1119.445	3076.631	104.261	45.238	nd	23.899	44.958
GC38-5	1149.321	3046.477	111.248	39.070	3.419	22.263	34.878
GC38-8	1084.695	3090.110	97.681	34.991	nd	25.251	29.066
GC38-12	1107.768	2814.908	102.420	44.168	nd	27.328	34.850

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GC13-2	1613.776	0.447	0.397	97.284	0.671	0.051	39.515
GC13-5	1711.962	0.716	0.290	99.455	0.395	0.170	39.632
GC13-8	1634.763	0.569	0.372	98.538	0.301	0.221	38.830
GC16-4	1628.953	0.875	0.368	98.952	0.605	0.029	39.112
GC16-6	1283.968	0.694	0.254	74.202	0.237	0.097	28.840
GC16-11	1639.681	0.557	0.321	95.837	0.302	0.867*	37.712
GC16-12	1525.443	2.382	0.291	101.322	11.371*	0.082	36.515
GC19-5	1671.186	0.657	0.372	104.310	0.639	0.202	39.172
GC19-6	1843.641	1.419	0.257	112.585	1.119	0.353	44.017
GC19-7	1650.429	0.596	0.291	96.885	0.272	0.238	41.093
GC19-11	1611.742	0.841	0.278	97.756	0.300	0.228	37.346
GC21-2	1654.739	0.183	0.227	97.957	0.269	0.199	35.896
GC21-3	1504.741	nd	0.322	84.200	0.153	0.171	38.546
GC21-8	1914.565	0.842	0.487	115.884	1.131	0.258	38.816
GC21-11	1427.811	0.714	0.242	90.150	0.296	0.250	42.705
GC26-5	1446.867	0.651	0.315	91.732	0.266	0.253	38.704
GC26-6	1477.527	0.843	0.381	93.229	0.312	0.214	38.903
GC26-7	1739.288	2.074	0.278	124.970	0.549	0.261	37.551
GC26-10	1713.354	nd	0.359	93.972	0.074	0.136	41.745
GC27-5	1577.022	0.643	0.355	93.349	0.262	0.178	38.679
GC27-6	1600.712	0.471	0.393	91.613	0.275	0.279	39.246
GC27-7	1777.312	0.056	0.289	96.837	0.097	0.336	39.453
GC27-9	1650.682	0.166	0.378	98.109	0.270	0.268	38.170
GC34-2	1597.111	0.764	0.325	93.330	0.680	0.097	39.713
GC34-5	1541.308	0.735	0.366	96.146	0.684	0.421	43.024
GC34-8	1597.256	1.233	0.393	98.767	0.830	0.706*	40.340
GC34-11	1632.729	0.836	0.439	94.535	0.759	0.109	47.196
GC37-1	1555.674	1.032	0.319	94.461	0.711	0.237	38.149
GC37-2	1469.494	1.009	0.312	88.139	0.712	0.250	41.211
GC37-7	1561.872	0.626	0.260	92.931	0.516	0.170	40.582
GC37-11	1625.751	0.826	0.354	95.474	0.735	0.245	41.994
GC38-3	1795.711	1.156	0.385	100.187	0.704	0.235	42.425
GC38-5	1562.927	0.366	0.244	94.515	0.423	0.216	39.804
GC38-8	1798.667	0.650	0.348	100.507	0.415	0.287	38.424
GC38-12	1572.961	0.392	0.311	92.288	0.487	0.322	39.735

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GC13-2	0.048	0.809	34.443	0.446	16.588
GC13-5	0.058	0.865	13.654	0.824	17.213
GC13-8	0.061	0.813	13.832	0.487	16.590
GC16-4	0.074	0.918	24.830	0.436	16.462
GC16-6	0.037	0.582	12.427	0.383	17.304
GC16-11	0.046	0.877	11.018	0.462	17.109
GC16-12	0.033	0.808	5.102	0.477	15.055
GC19-5	0.071	0.812	11.145	0.560	16.021
GC19-6	0.078	0.913	65.297	0.566	16.376
GC19-7	0.035	0.777	23.747	0.662	17.035
GC19-11	0.048	0.927	28.590	0.463	16.487
GC21-2	0.046	0.873	5.741	0.461	16.893
GC21-3	0.028	0.752	2.961	0.401	17.871
GC21-8	0.068	0.887	17.693	0.387	16.521
GC21-11	0.036	0.845	9.822	0.473	15.838
GC26-5	0.060	0.760	16.809	0.452	15.773
GC26-6	0.067	0.867	7.851	0.453	15.848
GC26-7	0.072	0.843	5.144	0.427	13.918
GC26-10	0.012	0.801	3.518	0.464	18.233
GC27-5	0.056	0.828	9.928	0.425	16.894
GC27-6	0.030	0.704	6.308	0.420	17.473
GC27-7	0.032	0.719	8.958	0.421	18.354
GC27-9	0.051	0.820	8.856	0.520	16.825
GC34-2	0.044	0.831	34.540	0.454	17.113
GC34-5	0.064	0.809	40.669	0.444	16.031
GC34-8	0.092	0.860	37.950	0.446	16.172
GC34-11	0.075	0.826	38.236	0.402	17.271
GC37-1	0.058	0.977	48.735	0.488	16.469
GC37-2	0.084	0.890	45.286	0.412	16.672
GC37-7	0.063	0.798	22.863	0.454	16.807
GC37-11	0.045	0.939	14.954	0.426	17.028
GC38-3	0.065	0.787	48.216	0.632	17.924
GC38-5	0.063	0.970	13.502	0.467	16.536
GC38-8	0.079	0.767	23.427	0.458	17.896
GC38-12	0.066	0.848	23.766	0.454	17.044

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GD15-1	857.383	2143.179	88.784	nd	nd	nd	27.400
GD15-3	910.700	2013.015	85.240	nd	nd	2.866	23.513
GD15-7	862.819	2178.888	87.640	nd	nd	nd	25.024
GD15-11	895.857	2203.500	96.573	nd	nd	1.940	65.266*
GD16-3	842.542	1963.847	77.916	nd	nd	1.634	18.550
GD16-4	940.015	2061.389	92.080	nd	nd	3.239	18.104
GD16-11	795.258	1956.784	78.793	nd	nd	7.059	22.000
GD16-12	820.014	2048.415	84.218	nd	nd	3.741	38.178
GD110-2	784.597	2030.736	86.434	nd	nd	2.814	22.907
GD110-5	816.864	2058.349	91.792	nd	nd	4.811	25.012
GD110-11	916.364	2138.599	89.788	nd	nd	12.023	20.100
GD110-12	871.930	2257.644	94.983	nd	nd	4.505	37.641
GD22-4	1009.193	2275.253	83.359	nd	nd	4.755	29.468
GD22-6	766.274	1782.009	85.864	nd	nd	1.360	24.140
GD22-9	920.334	2293.943	83.179	nd	nd	7.711	28.955
GD24-4	854.110	2063.165	85.474	nd	nd	0.122	31.172
GD24-5	868.060	2212.046	100.987	22.494	nd	13.850	19.701
GD24-9	701.757	2098.051	100.753	28.630	nd	20.146	17.706
GD24-11	833.394	2047.097	88.166	nd	nd	2.504	18.583
GD29-1	1057.073	2273.051	85.248	nd	nd	1.770	22.005
GD29-6	962.417	2226.440	100.947	28.360	nd	12.644	17.141
GD29-9	920.461	2140.134	101.636	20.683	nd	16.027	22.588
GD32-8	872.051	2114.144	90.694	nd	nd	4.089	24.674
GD32-9	1029.648	2571.067	86.772	nd	nd	3.684	21.610
GD33-5	928.510	2227.088	93.718	14.351	nd	17.725	21.240
GD33-6	1004.350	2372.068	104.643	27.395	nd	16.726	20.524
GD33-7	774.909	2028.117	88.850	nd	nd	4.200	40.034
GD33-9	722.697	2006.029	74.546	nd	0.093	3.748	46.273
GD36-2	872.028	2281.667	99.940	27.930	nd	14.007	18.355
GD36-3	1121.888	2626.210	118.342	nd	nd	1.934	41.986
GD36-11	1007.289	2391.899	69.216	nd	nd	2.831	22.163

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Cu	Zn
GD15-1	1489.403	0.399	0.250	205.800	1.644	0.134	28.880
GD15-3	1579.026	0.564	0.312	210.618	1.439	0.122	26.105
GD15-7	1474.408	0.245	0.216	202.018	1.886	0.154	26.723
GD15-11	1400.595	0.541	0.194	200.835	1.201	0.170	37.244
GD16-3	1811.378	0.301	0.342	233.965	1.303	0.101	26.627
GD16-4	1617.953	0.389	0.228	216.332	1.364	0.143	26.949
GD16-11	1841.940	0.421	0.347	238.149	1.599	0.231	26.831
GD16-12	1640.757	0.428	0.332	220.059	1.321	0.170	27.891
GD110-2	1733.036	0.324	0.370	228.906	1.387	0.144	27.230
GD110-5	1506.972	0.489	0.311	214.568	1.637	0.225	26.190
GD110-11	1695.242	0.385	0.360	223.817	2.173	0.252	25.023
GD110-12	1376.908	0.511	0.322	197.872	1.826	0.180	27.515
GD22-4	1616.084	0.394	0.293	217.622	1.537	0.301	28.113
GD22-6	1415.256	0.548	0.159	156.555	1.582	0.207	25.674
GD22-9	1570.695	0.857	0.298	210.584	1.345	0.383	28.520
GD24-4	1562.309	0.848	0.236	211.644	2.782	0.114	27.710
GD24-5	1475.098	0.271	0.335	205.943	1.291	0.144	25.717
GD24-9	1833.284	0.587	0.441	238.477	1.446	0.224	27.486
GD24-11	1465.560	0.539	0.359	201.892	1.620	0.187	28.703
GD29-1	1798.770	0.207	0.283	234.234	1.100	0.218	28.903
GD29-6	1536.577	0.443	0.246	211.113	1.414	0.358	27.876
GD29-9	1491.207	0.544	0.268	208.150	1.329	0.153	28.216
GD32-8	1683.896	0.486	0.271	224.073	6.220	0.207	26.548
GD32-9	1687.048	0.226	0.396	224.417	1.416	0.345	28.339
GD33-5	1525.141	0.855	0.403	213.982	2.757	0.170	26.051
GD33-6	1553.337	0.542	0.329	216.051	1.753	0.207	28.588
GD33-7	1555.518	0.507	0.302	217.692	1.838	0.179	32.960
GD33-9	1449.578	0.883	0.172	199.514	3.165	0.172	34.803
GD36-2	1589.902	0.346	0.322	214.858	1.403	0.120	28.094
GD36-3	1839.707	0.464	0.432	241.047	1.466	0.128	33.613
GD36-11	1556.449	0.363	0.291	211.328	1.272	0.121	25.356

nd = element not detected.
* = outlier.

TABLE 14a: (Continued)

Sample ID #	Rb	Sr	Zr	Pb	Ti/Fe
GD15-1	0.005	0.625	3.007	0.977	7.237
GD15-3	0.009	0.601	1.745	0.976	7.497
GD15-7	nd	0.574	2.880	1.020	7.298
GD15-11	nd	0.674	3.575	1.001	6.974
GD16-3	nd	0.651	1.047	1.011	7.742
GD16-4	0.009	0.753	1.032	1.007	7.479
GD16-11	nd	0.661	6.196	1.028	7.734
GD16-12	0.028	0.621	2.980	1.038	7.456
GD110-2	0.004	0.713	1.592	0.970	7.571
GD110-5	0.015	0.629	1.347	1.006	7.023
GD110-11	0.011	0.636	1.224	0.938	7.574
GD110-12	0.021	0.687	0.808	1.050	6.959
GD22-4	0.012	0.727	9.780	1.051	7.426
GD22-6	0.016	0.597	18.420	0.964	9.040
GD22-9	0.022	0.601	26.592	0.940	7.459
GD24-4	0.012	0.604	4.126	0.966	7.382
GD24-5	0.019	0.609	2.978	0.956	7.163
GD24-9	0.032	0.593	11.209	0.949	7.687
GD24-11	0.027	0.592	7.725	0.995	7.259
GD29-1	0.018	0.629	5.975	1.039	7.679
GD29-6	0.037	0.566	4.757	0.963	7.278
GD29-9	0.027	0.664	4.432	0.987	7.164
GD32-8	0.025	0.622	3.420	1.043	7.515
GD32-9	0.018	0.617	6.001	1.120	7.517
GD33-5	0.032	0.586	10.763	1.003	7.127
GD33-6	0.033	0.626	9.056	1.179	7.190
GD33-7	0.005	0.683	2.784	1.037	7.146
GD33-9	0.018	0.646	10.544	0.991	7.266
GD36-2	0.021	0.612	9.717	0.937	7.400
GD36-3	0.008	0.721	23.902	1.067	7.632
GD36-11	0.009	0.718	30.998	0.981	7.365

nd = element not detected.
* = outlier.

TABLE 14b: Analytical results of the within-lot study using W-*brems* excitation. Assay time is 1,000 live seconds. Element concentration results are in μg/g.

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GB12-3	7.092	11.495	79.018	806.426	1.015	0.166	85.900
GB12-4	nd	11.074	12.797	874.077	1.297	0.311	93.014
GB12-11	nd	21.321	23.736	825.604	2.086	0.462	90.531
GB12-12	nd	10.792	15.658	866.153	1.597	0.328	92.083
GB13-4	1.078	3.550	17.403	1005.717	0.719	0.449	101.754
GB13-5	15.874	7.600	19.705	850.834	1.280	0.176	88.997
GB13-7	6.563	5.450	10.579	797.942	3.134	0.432	95.491
GB13-12	nd	4.019	11.369	921.599	1.922	0.383	97.538
GB17-3	1.893	8.573	16.026	952.122	1.134	0.057	99.568
GB17-5	17.375	10.658	19.346	935.072	0.312	0.336	93.278
GB17-8	52.500	17.622	18.880	653.045	0.601	0.066	75.485
GB17-11	16.458	9.882	19.125	684.988	0.127	0.068	76.784
GB23-4	14.633	12.054	16.415	896.738	1.939	0.266	94.122
GB23-6	19.548	4.476	13.818	891.580	1.030	0.236	87.667
GB23-9	nd	4.626	15.376	834.850	1.465	0.140	87.846
GB23-11	nd	6.850	13.645	826.255	2.486	0.149	89.836
GB25-2	nd	4.771	15.759	975.803	1.216	0.148	99.608
GB25-4	12.338	5.631	16.260	889.839	1.322	0.174	90.310
GB25-11	15.744	4.920	13.648	957.267	1.174	0.218	97.441
GB25-12	7.272	4.285	13.877	869.690	0.972	0.055	90.968
GB27-1	nd	3.742	14.930	858.161	0.926	0.114	87.014
GB27-12	4.929	2.643	12.663	907.646	1.239	0.208	95.779
GB31-3	24.975	10.404	12.613	898.679	0.241	0.374	87.786
GB31-6	32.861	7.451	11.317	849.518	1.191	0.155	96.295
GB31-7	43.470	10.065	15.404	833.921	0.905	0.176	86.157
GB31-12	39.403	5.393	12.381	838.260	1.442	0.163	88.056
GB32-1	22.561	12.598	12.112	890.603	0.963	0.189	88.669
GB32-6	46.665	10.589	12.144	864.006	0.877	0.211	86.814
GB32-7	3.940	11.214	13.685	813.430	1.281	0.253	86.306
GB32-8	43.606	11.058	14.341	856.305	0.848	0.134	97.155
GB38-3	16.004	8.093	12.262	854.043	0.954	0.193	87.691
GB38-4	28.194	8.770	12.077	858.617	1.159	0.318	87.829
GB38-7	21.538	8.215	14.618	857.341	0.820	0.156	88.842
GB38-9	17.469	8.717	14.364	833.752	0.798	0.374	86.653

nd = element not detected.

* = outlier.

TABLE 14b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GB12-3	0.254	0.220	42.886	0.006	0.539	0.007	0.052
GB12-4	0.199	0.273	43.423	nd	0.412	0.038	0.077
GB12-11	0.786	0.412	40.661	0.003	0.467	0.051	0.102
GB12-12	0.580	0.253	43.614	0.013	0.436	0.026	0.054
GB13-4	0.025	0.237	43.392	nd	0.499	0.036	0.022
GB13-5	0.059	0.370	39.401	nd	0.434	0.009	0.019
GB13-7	0.057	0.317	46.046	0.003	0.500	0.011	0.016
GB13-12	0.099	0.170	43.753	nd	0.419	nd	0.029
GB17-3	0.096	0.338	44.905	0.020	0.462	0.021	0.020
GB17-5	0.113	0.276	47.433	nd	0.424	0.026	0.023
GB17-8	0.069	0.347	48.420	nd	0.400	0.028	0.016
GB17-11	0.006	0.853	45.034	0.016	0.430	0.022	0.044
GB23-4	0.101	0.003	44.136	0.035	0.498	0.026	0.344
GB23-6	0.057	0.034	40.737	0.003	0.418	0.000	0.171
GB23-9	0.138	0.013	42.313	nd	0.472	0.004	0.168
GB23-11	0.022	0.101	46.647	nd	0.427	0.000	0.322
GB25-2	0.086	0.051	41.948	0.012	0.520	0.011	0.213
GB25-4	nd	nd	40.466	nd	0.441	0.015	0.157
GB25-11	0.109	0.095	42.692	nd	0.495	0.015	0.264
GB25-12	0.091	0.046	43.580	nd	0.432	0.020	0.284
GB27-1	0.072	0.109	42.026	nd	0.438	0.028	0.196
GB27-12	0.055	0.324	43.783	0.006	0.448	0.018	0.245
GB31-3	0.423	0.059	41.191	0.006	0.437	0.034	0.373
GB31-6	0.447	0.061	42.155	0.017	0.406	0.013	0.337
GB31-7	0.293	nd	42.074	0.008	0.421	0.019	0.340
GB31-12	0.337	nd	40.997	nd	0.449	0.022	0.258
GB32-1	0.709	nd	40.084	nd	0.436	0.015	0.405
GB32-6	0.343	0.023	41.901	nd	0.450	0.021	0.238
GB32-7	0.555	nd	36.452	nd	0.447	0.009	0.251
GB32-8	0.341	0.219	41.003	nd	0.449	0.019	0.299
GB38-3	0.424	nd	39.844	0.031	0.450	0.010	0.299
GB38-4	0.536	nd	37.961	0.011	0.464	0.006	0.243
GB38-7	0.332	nd	41.016	0.004	0.466	0.000	0.276
GB38-9	0.345	nd	40.062	nd	0.439	0.000	0.341

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GB12-3	9.548	0.522	23.886	0.264	9.388
GB12-4	26.341	0.464	21.824	0.251	9.397
GB12-11	36.007	0.489	22.907	0.340	9.120
GB12-12	14.843	0.438	21.820	2.345	9.406
GB13-4	1.973	0.454	25.081	0.213	9.884
GB13-5	0.344	0.495	22.432	0.341	9.560
GB13-7	0.301	0.513	24.460	0.200	8.356
GB13-12	5.842	0.480	22.933	0.191	9.449
GB17-3	1.543	0.517	22.601	0.214	9.563
GB17-5	1.169	0.597	22.450	0.265	10.025
GB17-8	1.040	0.987	20.335	0.161	8.651
GB17-11	1.084	0.470	20.906	0.182	8.921
GB23-4	0.411	20.920	0.187	0.040	9.527
GB23-6	0.519	20.127	0.178	0.041	10.170
GB23-9	0.502	21.312	0.127	0.036	9.504
GB23-11	0.457	22.073	0.127	0.036	9.197
GB25-2	0.462	21.518	0.175	0.038	9.796
GB25-4	0.465	21.605	0.196	0.052	9.853
GB25-11	0.507	23.574	0.194	nd	9.824
GB25-12	0.451	21.276	0.179	0.021	9.560
GB27-1	0.423	20.689	0.181	0.028	9.862
GB27-12	0.441	22.279	0.340	0.048	9.476
GB31-3	0.461	19.694	0.173	0.041	10.237
GB31-6	0.435	19.629	0.268	0.056	8.822
GB31-7	0.388	18.973	0.171	0.032	9.679
GB31-12	0.461	20.358	0.163	0.012	9.520
GB32-1	0.478	19.993	0.133	0.000	10.044
GB32-6	0.430	20.099	0.143	0.000	9.952
GB32-7	0.422	19.591	0.133	0.040	9.425
GB32-8	0.414	19.816	0.139	0.003	8.814
GB38-3	0.406	20.257	0.194	0.033	9.739
GB38-4	0.470	19.626	0.168	0.000	9.776
GB38-7	0.402	19.974	0.139	0.024	9.650
GB38-9	0.431	18.298	0.184	0.036	9.622

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GC13-2	nd	3.890	18.351	1983.115	4.741	0.000	112.979
GC13-5	nd	6.797	24.224	2055.094	5.687	0.013	115.678
GC13-8	nd	4.220	17.528	1964.610	4.440	0.405	111.573
GC16-4	nd	5.445	22.218	2021.834	4.935	0.249	112.825
GC16-6	nd	1.623	17.178	1437.930	3.637	0.000	81.344
GC16-11	nd	3.873	17.526	1869.208	4.474	0.000	106.483
GC16-12	nd	5.328	12.375	1852.309	7.697	0.000	116.603
GC19-5	nd	7.008	14.433	2016.014	5.046	0.000	116.242
GC19-6	nd	6.576	43.678	2248.367	6.152	0.000	127.272
GC19-7	nd	2.431	58.357	1973.942	5.178	0.000	112.686
GC19-11	nd	6.464	22.617	2036.580	5.182	0.000	114.144
GC21-2	nd	7.600	25.393	2127.099	4.685	0.071	118.839
GC21-3	nd	4.468	21.423	1846.139	3.429	0.137	102.376
GC21-8	nd	3.863	29.633	2271.035	5.372	0.081	128.569
GC21-11	nd	9.444	39.370	1841.102	4.262	0.329	106.645
GC26-5	nd	6.788	31.471	1822.738	4.898	0.282	106.597
GC26-6	nd	3.438	70.635	1840.203	5.340	0.078	110.843
GC26-7	nd	9.468	30.183	1818.504	6.998	0.000	124.693
GC26-10	nd	nd	83.435	1984.045	2.507	0.000	103.952
GC27-5	nd	6.454	34.674	1858.964	5.046	0.000	107.495
GC27-6	nd	8.670	13.738	1956.453	5.043	0.131	109.679
GC27-7	nd	15.843	46.554	2049.148	4.211	0.000	110.642
GC27-9	nd	4.128	38.896	2033.974	4.162	0.000	113.232
GC34-2	nd	4.083	28.817	1926.881	4.262	0.000	106.649
GC34-5	nd	6.842	35.785	1959.432	4.472	0.000	111.007
GC34-8	nd	8.451	20.295	1959.231	3.870	0.000	109.499
GC34-11	nd	1.131	42.455	1596.312	2.935	0.035	86.485
GC37-1	nd	3.300	20.954	1828.814	3.990	0.187	104.508
GC37-2	nd	11.499	41.929	1764.085	4.663	0.126	99.132
GC37-7	0.751	14.678	14.958	1946.157	5.127	0.000	110.005
GC37-11	nd	3.665	51.515	1974.577	5.679	0.000	113.406
GC38-3	nd	8.776	30.819	2165.513	5.060	0.000	116.248
GC38-5	nd	5.307	20.423	1966.493	3.986	0.000	108.546
GC38-8	nd	11.864	15.925	1982.117	4.021	0.000	109.560
GC38-12	nd	8.954	19.207	1918.446	4.346	0.071	107.693

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GC13-2	0.586	0.181	43.452	0.033	0.733	0.098	31.739
GC13-5	0.293	0.270	43.485	0.054	0.763	0.094	13.193
GC13-8	0.318	0.256	43.840	0.058	0.726	0.068	12.488
GC16-4	0.506	0.162	43.234	0.030	0.838	0.108	21.786
GC16-6	0.296	0.126	31.101	0.037	0.509	0.070	10.824
GC16-11	0.435	0.961	42.597	0.035	0.791	0.073	9.262
GC16-12	10.934	nd	40.359	0.028	0.707	0.064	4.825
GC19-5	0.472	0.349	42.958	0.039	0.759	0.069	10.417
GC19-6	0.982	0.301	48.422	0.051	0.774	0.220	55.196
GC19-7	0.342	0.334	45.691	0.051	0.778	0.126	22.319
GC19-11	0.282	0.238	42.055	0.067	0.746	0.155	26.810
GC21-2	0.299	0.229	40.135	0.058	0.819	0.081	6.142
GC21-3	0.034	0.282	41.820	0.080	0.699	0.049	3.395
GC21-8	1.071	0.255	42.736	0.054	0.772	0.082	16.681
GC21-11	0.352	0.238	47.228	0.021	0.760	0.040	9.371
GC26-5	0.243	0.216	43.383	0.017	0.701	0.108	17.504
GC26-6	0.255	0.306	44.244	0.052	0.814	0.073	7.896
GC26-7	0.456	0.285	40.903	0.056	0.767	0.073	4.110
GC26-10	0.180	0.150	44.709	0.026	0.690	0.053	3.552
GC27-5	0.269	0.329	43.115	0.030	0.736	0.050	9.935
GC27-6	0.235	0.474	42.032	0.066	0.651	0.051	6.912
GC27-7	0.187	0.372	42.051	0.041	0.659	0.096	9.001
GC27-9	0.136	0.428	42.000	0.047	0.753	0.088	7.671
GC34-2	0.349	0.052	42.471	0.049	0.678	0.113	34.752
GC34-5	0.389	0.332	46.115	0.050	0.720	0.110	40.663
GC34-8	0.444	0.572	43.546	0.031	0.773	0.091	33.169
GC34-11	0.252	nd	39.131	0.059	0.577	0.108	31.920
GC37-1	0.441	nd	40.541	0.034	0.859	0.157	44.413
GC37-2	0.501	0.142	44.243	0.084	0.834	0.111	42.746
GC37-7	0.224	0.122	43.214	0.049	0.734	0.105	21.347
GC37-11	0.387	0.190	46.112	0.052	0.834	0.081	13.778
GC38-3	0.431	0.137	44.787	0.045	0.736	0.218	44.334
GC38-5	0.182	0.150	43.113	0.057	0.828	0.097	12.931
GC38-8	0.156	0.203	39.896	0.066	0.715	0.087	22.764
GC38-12	0.225	0.212	43.234	0.048	0.715	0.117	22.988

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GC13-2	0.593	20.186	0.135	0.080	17.553
GC13-5	0.863	21.928	0.297	0.037	17.766
GC13-8	0.568	21.835	0.205	0.028	17.608
GC16-4	0.583	21.322	0.148	0.051	17.920
GC16-6	0.471	14.364	0.111	0.000	17.677
GC16-11	0.586	19.899	0.127	0.000	17.554
GC16-12	0.517	21.055	0.174	0.025	15.886
GC19-5	0.590	20.910	0.193	0.046	17.343
GC19-6	0.640	22.745	0.235	0.065	17.666
GC19-7	0.724	19.508	0.196	0.050	17.517
GC19-11	0.466	22.066	0.169	0.086	17.842
GC21-2	0.491	20.897	0.286	0.019	17.899
GC21-3	0.495	23.081	0.151	0.032	18.033
GC21-8	0.544	24.028	0.228	0.055	17.664
GC21-11	0.535	19.928	0.160	0.043	17.264
GC26-5	0.459	20.467	0.165	0.034	17.099
GC26-6	0.486	21.557	0.204	0.052	16.602
GC26-7	0.506	19.414	0.214	0.055	14.584
GC26-10	0.615	19.221	0.196	0.028	19.086
GC27-5	0.534	20.019	0.219	0.040	17.293
GC27-6	0.477	19.854	0.229	0.032	17.838
GC27-7	0.592	20.082	0.260	0.005	18.521
GC27-9	0.519	20.231	0.231	0.068	17.963
GC34-2	0.416	20.522	0.156	0.027	18.068
GC34-5	0.478	20.697	0.173	0.087	17.651
GC34-8	0.530	22.682	0.142	0.034	17.893
GC34-11	0.298	18.879	0.126	0.038	18.458
GC37-1	0.449	20.849	0.174	0.077	17.499
GC37-2	0.538	20.381	0.153	0.110	17.795
GC37-7	0.540	20.480	0.136	0.251	17.691
GC37-11	0.620	18.879	0.152	0.000	17.412
GC38-3	0.761	21.690	0.176	0.063	18.628
GC38-5	0.463	21.021	0.149	0.041	18.117
GC38-8	0.499	20.887	0.169	0.053	18.092
GC38-12	0.534	20.423	0.123	0.039	17.814

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	S	K	Ca	Ti	Cr	Mn	Fe
GD15-1	nd	0.193	25.475	1994.529	1.637	0.229	258.288
GD15-7	nd	nd	24.916	1853.375	3.164	0.167	246.758
GD15-11	nd	2.722	62.945	1791.709	3.108	0.195	247.551
GD16-3	nd	3.465	14.787	1980.701	3.428	0.000	257.623
GD16-4	nd	1.570	14.997	1856.159	3.071	0.394	247.338
GD16-11	nd	6.878	20.042	1976.044	3.312	0.274	260.482
GD16-12	nd	4.542	37.822	2048.798	3.220	0.507	271.878
GD110-2	nd	2.706	19.097	1999.205	3.237	0.163	265.258
GD110-5	nd	2.942	22.322	1971.073	4.048	0.109	273.004
GD110-11	nd	11.089	16.397	1992.971	4.594	0.209	270.223
GD110-12	nd	4.528	35.777	1791.107	3.298	0.128	245.820
GD22-4	nd	4.778	26.348	1953.855	3.171	0.202	261.290
GD22-6	nd	3.053	21.708	1874.597	2.997	0.401	244.670
GD22-9	nd	9.099	26.808	1977.207	3.844	0.023	261.853
GD24-4	nd	1.055	26.370	1898.998	3.492	0.270	253.664
GD24-5	47.006	14.436	16.335	1827.103	4.034	0.119	251.103
GD24-9	33.211	18.855	10.250	1929.160	4.147	0.294	261.017
GD24-11	nd	5.250	18.011	2042.111	3.560	0.147	270.894
GD29-1	nd	nd	11.185	2027.004	4.266	0.034	272.843
GD29-6	23.924	12.211	14.841	1969.091	3.474	0.375	262.927
GD29-9	26.337	17.659	21.262	1953.285	3.480	0.333	267.000
GD29-10	nd	10.039	22.353	1953.263	4.708	0.184	268.029
GD32-4	nd	1.692	13.156	1706.787	2.933	0.173	228.606
GD32-8	nd	5.836	19.154	1928.931	4.220	0.419	265.115
GD32-9	nd	3.582	13.879	1642.245	3.144	0.323	218.802
GD33-5	0.775	17.136	17.892	1881.758	3.634	0.240	251.808
GD33-6	24.918	14.534	11.432	1726.617	3.848	0.214	234.667
GD33-7	nd	4.340	40.663	2003.763	4.063	0.142	270.946
GD33-9	nd	4.207	43.071	1919.467	4.481	0.282	257.629
GD36-2	29.825	13.541	15.623	1922.159	3.686	0.163	253.618
GD36-3	nd	1.963	38.818	2097.531	3.914	0.122	275.771
GD36-11	nd	0.839	12.636	1611.469	2.940	0.350	217.953
GD36-12	nd	18.751	18.056	2014.406	3.324	0.340	267.053

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	Ni	Cu	Zn	Rb	Sr	Y	Zr
GD15-1	1.834	0.046	30.969	0.024	0.588	0.018	3.011
GD15-7	1.872	0.184	28.958	nd	0.572	0.020	2.559
GD15-11	1.250	0.124	40.781	0.008	0.623	0.023	3.258
GD16-3	1.335	0.168	28.721	nd	0.589	0.047	1.161
GD16-4	1.204	0.134	28.999	0.014	0.687	0.025	1.085
GD16-11	1.401	0.204	28.706	0.033	0.594	0.029	5.897
GD16-12	1.441	0.194	30.970	0.038	0.576	0.046	3.088
GD110-2	1.418	0.243	29.426	0.028	0.694	0.025	1.593
GD110-5	1.509	0.208	28.868	nd	0.556	0.025	1.572
GD110-11	2.260	0.229	28.281	0.003	0.587	0.038	1.383
GD110-12	1.626	0.051	29.464	nd	0.596	0.034	0.924
GD22-4	1.438	0.268	30.428	0.009	0.633	0.040	8.625
GD22-6	1.368	0.118	27.881	0.023	0.536	0.070	16.427
GD22-9	1.219	0.290	31.083	0.019	0.531	0.059	24.432
GD24-4	2.623	0.030	30.111	0.033	0.562	0.033	4.039
GD24-5	1.240	0.096	28.066	0.014	0.562	0.031	2.857
GD24-9	1.449	0.258	30.496	0.030	0.589	0.009	10.298
GD24-11	1.630	0.177	32.201	0.013	0.532	0.066	8.506
GD29-1	1.139	0.305	31.935	0.046	0.555	0.019	5.709
GD29-6	1.286	0.298	30.781	0.004	0.598	0.061	4.612
GD29-9	1.303	0.175	30.948	0.030	0.637	0.050	4.682
GD29-10	1.132	0.304	31.388	0.032	0.588	0.062	3.601
GD32-4	1.153	0.094	25.875	0.005	0.510	0.039	5.256
GD32-8	6.122	0.150	29.305	0.028	0.560	0.038	3.479
GD32-9	1.073	0.286	25.614	0.007	0.453	0.044	4.378
GD33-5	2.584	0.017	26.768	0.016	0.571	0.025	10.472
GD33-6	1.246	0.115	26.671	0.047	0.473	0.031	8.057
GD33-7	1.901	0.192	36.460	0.011	0.609	0.025	3.072
GD33-9	3.203	0.068	38.559	nd	0.597	0.047	11.085
GD36-2	1.337	0.023	29.406	0.018	0.529	0.042	9.371
GD36-3	1.335	0.039	36.207	0.028	0.618	0.087	23.191
GD36-11	0.976	0.062	23.584	0.019	0.540	0.052	22.351
GD36-12	1.469	0.201	30.875	0.005	0.606	0.044	16.313

nd = element not detected.
* = outlier.

TABLE 14b: (Continued)

Sample ID #	Pb	Ba	Sn	Ag	Ti/Fe
GD15-1	1.085	19.783	0.502	0.043	7.722
GD15-7	1.016	17.037	0.470	0.044	7.511
GD15-11	0.968	16.764	0.566	0.049	7.238
GD16-3	1.124	19.715	0.509	0.042	7.688
GD16-4	1.111	17.496	0.486	0.028	7.505
GD16-11	1.023	18.054	0.493	0.042	7.586
GD16-12	1.121	19.024	0.572	0.000	7.536
GD110-2	1.117	18.886	0.539	0.014	7.537
GD110-5	1.108	18.843	1.772	0.001	7.220
GD110-11	1.136	18.668	5.685	0.054	7.375
GD110-12	1.095	18.430	0.499	0.054	7.286
GD22-4	1.183	17.631	0.584	0.043	7.478
GD22-6	1.048	17.062	0.549	0.059	7.662
GD22-9	1.118	18.233	0.544	0.068	7.551
GD24-4	1.030	17.440	0.580	0.067	7.486
GD24-5	1.075	19.127	0.542	0.054	7.276
GD24-9	1.001	18.430	0.577	0.044	7.391
GD24-11	1.053	18.877	0.525	0.006	7.538
GD29-1	1.119	17.900	0.560	0.057	7.429
GD29-6	1.041	17.775	0.866	0.051	7.489
GD29-9	1.095	18.733	0.584	0.022	7.316
GD29-10	1.118	17.982	0.571	0.036	7.288
GD32-4	0.946	16.374	0.460	0.022	7.466
GD32-8	1.031	18.740	0.564	0.040	7.276
GD32-9	1.003	16.101	0.502	0.035	7.506
GD33-5	1.058	18.756	0.558	0.045	7.473
GD33-6	1.166	16.110	0.516	0.032	7.358
GD33-7	1.134	20.083	0.572	0.027	7.395
GD33-9	1.163	16.864	0.598	0.075	7.451
GD36-2	1.050	17.498	0.555	0.070	7.579
GD36-3	1.250	21.636	0.643	0.074	7.606
GD36-11	0.976	15.344	0.452	0.057	7.394
GD36-12	1.213	19.739	0.658	0.078	7.543

nd = element not detected.
* = outlier.

TABLE 14c: Analytical results of the within-lot study using W-L excitation. Assay time is 1,000 live seconds. Element concentration results are in µg/g.

Sample ID #	Al	Si	P	S	Cl	K	Ca
GB12-3	773.143	1648.344	122.677	54.470	9.618	27.050	119.438
GB12-4	735.929	1632.255	103.864	35.032	3.266	25.775	21.087
GB12-11	925.583	2097.906	89.782	45.953	7.587	34.247	28.211
GB12-12	731.756	1688.250	99.891	32.371	5.705	27.348	23.269
GB13-4	915.939	2290.031	137.796	68.490	1.979	23.550	35.747
GB13-5	810.490	2136.950	109.331	48.993	2.004	19.978	34.237
GB13-7	1043.185	3311.665	131.133	51.769	1.897	25.602	29.027
GB13-12	896.679	2149.080	131.971	55.117	4.805	18.400	22.582
GB17-3	668.784	1586.094	102.259	38.328	3.752	20.682	25.002
GB17-5	967.408	2112.732	149.058	64.435	10.806	24.959	37.565
GB17-8	513.419	1112.100	75.128	36.050	nd	19.203	16.738
GB17-11	527.253	1174.515	70.939	14.612	nd	8.707	13.982
GB23-4	732.503	1975.853	115.133	42.944	35.205	37.580	27.450
GB23-6	551.637	1232.242	93.050	34.568	nd	14.202	19.467
GB23-9	593.962	1527.432	103.973	21.215	4.457	15.937	24.962
GB23-11	637.005	1649.783	116.749	39.547	14.702	22.379	18.986
GB25-2	711.804	1800.408	104.207	35.071	4.741	18.121	22.049
GB25-4	621.026	1690.676	109.893	36.648	1.746	21.264	26.472
GB25-11	699.877	1817.609	119.956	56.224	20.242	26.618	28.602
GB25-12	640.125	1565.420	108.342	42.864	13.961	19.397	24.521
GB27-1	552.817	1370.166	88.522	28.001	nd	14.029	21.051
GB27-12	635.213	1574.921	103.689	37.886	14.481	16.822	21.740
GB31-3	646.579	1690.948	117.491	103.675	4.198	33.520	33.328
GB31-6	690.756	1726.513	118.946	96.423	4.721	30.333	33.829
GB31-7	727.683	1700.687	130.456	111.285	5.585	45.982	48.177
GB31-12	706.958	1724.483	134.432	109.898	5.223	36.271	42.561
GB32-1	727.864	1694.171	113.539	121.208	6.108	48.475	40.559
GB32-6	767.926	1761.853	144.805	128.147	4.558	42.431	41.487
GB32-7	702.422	1626.616	140.894	120.450	7.041	46.373	45.026
GB32-8	695.327	1762.922	140.274	124.944	4.742	42.571	43.677
GB38-3	700.445	1830.135	144.557	106.516	7.686	41.610	42.244
GB38-4	667.570	1579.834	123.613	101.591	6.709	36.079	42.348
GB38-7	660.042	1572.622	118.354	100.888	4.801	33.440	43.084
GB38-9	683.632	1825.733	129.837	115.833	6.285	42.091	46.314

nd = element not detected.

* = outlier.

TABLE 14c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GB12-3	641.303	0.273	0.251	109.389	0.452	5.863
GB12-4	834.256	0.190	0.302	114.944	0.320	7.258
GB12-11	898.878	1.599	0.470	110.747	1.162	8.117
GB12-12	741.516	0.293	0.231	108.535	0.788	6.832
GB13-4	834.103	0.794	0.308	130.948	0.218	6.370
GB13-5	812.026	0.566	0.270	121.550	0.186	6.681
GB13-7	718.239	0.688	0.257	118.854	0.248	6.043
GB13-12	819.062	0.823	0.332	127.607	0.291	6.419
GB17-3	757.897	0.067	0.253	116.118	0.159	6.527
GB17-5	841.259	nd	0.400	137.774	0.155	6.106
GB17-8	708.114	0.037	0.198	85.130	0.111	8.318
GB17-11	700.532	0.071	0.203	84.930	0.103	8.248
GB23-4	739.256	0.770	0.264	106.357	0.195	6.951
GB23-6	717.804	0.623	0.168	94.725	0.124	7.578
GB23-9	738.849	0.626	0.249	100.510	0.266	7.351
GB23-11	716.617	1.026	0.233	101.364	0.151	7.070
GB25-2	748.908	0.617	0.246	111.500	0.163	6.717
GB25-4	676.358	0.823	0.292	103.714	0.148	6.521
GB25-11	785.786	1.188	0.329	121.620	0.260	6.461
GB25-12	687.300	0.936	0.241	96.478	0.133	7.124
GB27-1	642.071	0.494	0.214	87.375	0.165	7.348
GB27-12	710.565	0.706	0.190	99.700	0.173	7.127
GB31-3	713.141	1.299	0.371	107.417	0.858	6.639
GB31-6	698.411	1.865	0.389	125.901	1.022	5.547
GB31-7	679.847	1.590	0.393	107.139	1.063	6.345
GB31-12	729.137	1.854	0.419	117.571	1.030	6.202
GB32-1	727.621	2.412	0.542	124.753	1.411	5.832
GB32-6	764.328	1.668	0.475	115.998	1.019	6.589
GB32-7	653.509	2.614	0.484	131.289	1.489	4.978
GB32-8	678.512	2.149	0.545	132.943	1.006	5.104
GB38-3	693.712	1.720	0.309	114.788	1.090	6.043
GB38-4	649.412	2.043	0.556	116.686	1.372	5.565
GB38-7	683.300	1.367	0.412	106.481	0.925	6.417
GB38-9	657.468	1.902	0.487	109.419	1.067	6.009

nd = element not detected.
* = outlier.

TABLE 14c: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GC13-2	1244.392	3141.592	104.070	28.900	nd	19.948	46.290
GC13-5	1333.782	3075.546	133.450	38.232	nd	26.792	63.978
GC13-8	1305.227	3264.207	111.147	29.810	nd	23.051	49.266
GC16-4	1449.092	3517.240	117.929	31.432	6.435	24.880	55.035
GC16-6	990.107	2390.752	83.703	14.883	nd	14.441	36.510
GC16-11	1169.863	2855.044	110.041	28.856	nd	21.811	47.947
GC16-12	1129.697	2626.824	108.146	31.141	nd	22.633	38.060
GC19-5	1448.882	3620.242	116.130	23.599	4.341	34.712	40.991
GC19-6	1622.016	3645.460	108.417	52.247	9.962	26.134	98.638
GC19-7	1265.945	2882.285	117.736	50.486	2.338	22.994	130.203
GC19-11	1371.493	3293.804	120.138	37.823	5.555	25.372	54.940
GC21-2	1435.344	3514.382	122.124	26.604	nd	29.023	57.537
GC21-3	1049.157	2478.257	111.718	28.454	2.402	18.967	49.124
GC21-8	1614.442	4050.142	120.849	26.403	nd	25.289	63.092
GC21-11	1211.432	2738.788	108.893	34.049	nd	24.070	74.841
GC26-5	1309.852	3272.511	124.422	30.528	nd	27.484	76.581
GC26-6	1357.914	3182.968	129.175	59.533	nd	30.713	173.156
GC26-7	1371.559	3387.875	110.259	26.692	1.511	32.774	66.996
GC26-10	1120.313	2633.693	94.947	46.516	nd	8.906	129.026
GC27-5	1135.928	2899.770	107.119	32.416	0.410	23.229	75.849
GC27-6	1011.638	2283.987	96.369	20.293	3.483	19.641	32.574
GC27-7	1170.319	3074.595	98.318	21.972	nd	30.426	71.284
GC27-9	1317.301	3215.769	111.815	27.100	nd	19.371	75.273
GC34-2	1099.354	2884.091	94.779	28.783	nd	18.491	61.781
GC34-5	1029.697	2512.787	81.805	45.645	nd	21.587	62.243
GC34-8	1318.237	3250.049	127.971	52.704	nd	29.274	53.309
GC34-11	1252.170	2739.657	102.786	56.117	7.733	13.573	110.141
GC37-1	1241.454	2856.444	83.019	32.373	nd	20.127	50.957
GC37-2	1144.359	2533.198	106.938	68.661	nd	35.097	82.589
GC37-7	981.175	2342.567	96.046	87.574	nd	35.077	41.753
GC37-11	1241.980	3055.906	113.500	50.238	nd	19.870	100.360
GC38-3	1111.128	2828.905	82.626	37.749	nd	19.027	46.535
GC38-5	1131.699	2717.714	86.605	28.424	2.258	17.106	37.461
GC38-8	1179.870	2605.400	83.758	37.562	3.656	29.840	35.336
GC38-12	1034.798	2479.929	74.331	54.031	14.229	28.876	42.137

nd = element not detected.
* = outlier.

TABLE 14c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GC13-2	1613.818	1.842	0.458	132.903	1.016	12.143
GC13-5	1712.003	2.482	0.474	141.215	0.708	12.123
GC13-8	1634.810	2.226	0.465	139.302	0.605	11.736
GC16-4	1629.006	3.032	0.654	138.557	1.031	11.757
GC16-6	1284.016	1.739	0.352	103.332	0.523	12.426
GC16-11	1639.724	1.912	0.546	138.225	0.691	11.863
GC16-12	1525.489	6.002	0.652	146.753	16.437	10.395
GC19-5	1671.235	1.951	0.488	145.746	1.024	11.467
GC19-6	1843.686	2.908	0.566	162.075	1.708	11.375
GC19-7	1650.474	1.440	0.454	136.281	0.535	12.111
GC19-11	1611.785	1.713	0.512	137.896	0.556	11.688
GC21-2	1654.785	1.641	0.467	143.517	0.526	11.530
GC21-3	1504.787	0.465	0.463	119.681	0.451	12.573
GC21-8	1914.617	2.974	0.425	168.875	1.740	11.337
GC21-11	1427.855	2.042	0.401	126.761	0.557	11.264
GC26-5	1446.917	2.527	0.390	136.144	0.640	10.628
GC26-6	1477.570	3.109	0.570	146.860	0.905	10.061
GC26-7	1739.332	5.332	0.661	183.705	1.166	9.468
GC26-10	1713.404	0.488	0.424	121.436	0.277	14.110
GC27-5	1577.065	1.511	0.581	131.815	0.622	11.964
GC27-6	1600.756	1.108	0.630	119.248	0.457	13.424
GC27-7	1777.359	0.648	0.420	127.412	0.406	13.950
GC27-9	1650.730	0.988	0.358	136.813	0.507	12.066
GC34-2	1597.040	1.955	0.290	128.910	0.663	12.389
GC34-5	1541.241	1.436	0.280	129.858	0.479	11.869
GC34-8	1597.189	2.696	0.422	140.013	0.883	11.407
GC34-11	1632.650	1.308	0.287	130.408	0.774	12.520
GC37-1	1555.605	1.649	0.505	133.525	0.742	11.650
GC37-2	1469.429	1.413	0.422	117.808	0.657	12.473
GC37-7	1561.803	1.155	0.448	123.535	0.551	12.643
GC37-11	1625.684	1.466	0.503	138.457	0.889	11.741
GC38-3	1795.646	1.380	0.453	126.620	0.520	14.181
GC38-5	1562.855	0.201	0.316	117.375	0.333	13.315
GC38-8	1798.604	0.601	0.343	128.872	0.461	13.957
GC38-12	1572.893	0.724	0.320	118.916	0.328	13.227

nd = element not detected.
* = outlier.

TABLE 14c: (Continued)

Sample ID #	Al	Si	P	S	Cl	K	Ca
GD15-1	1030.432	2371.247	107.149	13.950	4.525	16.560	57.029
GD15-3	1005.197	2134.515	92.270	13.406	nd	20.800	42.178
GD15-7	873.206	2144.809	81.355	nd	nd	12.085	43.924
GD15-11	951.240	2446.271	90.433	26.480	1.987	15.552	115.295
GD16-3	920.315	2121.640	92.553	8.200	0.251	19.471	37.207
GD16-4	1001.074	2226.192	87.471	7.299	0.166	17.066	31.412
GD16-11	919.598	2101.733	83.998	11.171	nd	20.234	34.776
GD16-12	874.190	2379.914	83.726	4.713	0.802	19.041	63.272
GD110-2	945.474	2252.921	111.416	13.845	2.429	20.638	46.412
GD110-5	767.895	1977.883	82.794	8.729	3.120	15.623	36.309
GD110-11	920.562	2127.315	92.110	9.773	nd	35.302	38.652
GD110-12	895.945	2307.667	99.715	21.016	2.680	20.956	64.429
GD22-4	1092.155	2663.401	93.955	18.971	nd	26.877	57.268
GD22-6	861.333	1685.392	62.954	nd	nd	5.925	29.370
GD22-9	902.639	2406.110	78.480	10.009	nd	22.925	45.514
GD24-4	874.758	1976.338	78.020	6.504	nd	11.781	48.895
GD24-5	1051.571	2642.242	115.459	90.926	8.113	47.379	46.124
GD24-9	1073.775	2562.059	105.877	91.458	nd	51.235	38.937
GD24-11	892.270	2139.392	89.645	10.303	nd	20.861	38.447
GD29-1	1083.696	2435.468	88.958	3.548	4.729	18.202	40.114
GD29-6	911.486	2218.841	103.579	81.637	0.564	33.927	30.542
GD29-9	949.611	2386.230	109.146	63.975	2.508	45.199	45.934
GD32-8	1005.771	2203.934	73.399	5.192	nd	26.227	39.492
GD32-9	1015.117	2516.251	86.945	17.758	nd	22.751	46.835
GD33-5	920.530	2362.946	85.129	43.552	1.443	36.610	36.818
GD33-6	1002.959	2778.866	124.443	85.640	3.346	51.084	41.704
GD33-7	873.198	2079.524	93.631	14.107	4.710	20.739	65.817
GD33-9	853.973	2402.045	89.416	20.118	15.515	20.985	84.033
GD36-2	1034.806	2468.508	118.521	80.055	2.508	40.362	38.320
GD36-3	1103.316	2862.167	120.198	35.879	12.217	22.937	95.898
GD36-11	957.328	2326.007	67.312	9.948	1.997	15.640	36.541

nd = element not detected.
* = outlier.

TABLE 14c: (Continued)

Sample ID #	Ti	Cr	Mn	Fe	Ni	Ti/Fe
GD15-1	1489.453	1.456	0.433	299.008	2.766	4.981
GD15-3	1579.073	1.378	0.429	289.342	2.355	5.457
GD15-7	1474.464	1.107	0.393	265.717	2.811	5.549
GD15-11	1400.640	1.569	0.393	282.768	1.833	4.953
GD16-3	1811.425	1.305	0.507	324.959	2.334	5.574
GD16-4	1618.000	1.172	0.410	280.449	1.976	5.769
GD16-11	1841.982	1.210	0.420	307.539	2.079	5.989
GD16-12	1640.808	1.439	0.414	297.747	1.901	5.511
GD110-2	1733.081	1.613	0.444	329.140	2.296	5.265
GD110-5	1507.013	1.271	0.441	286.165	2.111	5.266
GD110-11	1695.286	1.781	0.374	307.030	3.244	5.522
GD110-12	1376.948	1.562	0.460	278.586	2.884	4.943
GD22-4	1616.130	1.272	0.568	302.756	2.528	5.338
GD22-6	1415.306	0.996	0.276	234.348	1.619	6.039
GD22-9	1570.741	1.876	0.395	269.000	1.749	5.839
GD24-4	1562.354	1.845	0.501	280.133	3.682	5.577
GD24-5	1475.141	1.678	0.466	302.808	2.359	4.872
GD24-9	1833.332	1.705	0.540	333.865	2.185	5.491
GD24-11	1465.603	1.567	0.472	276.131	2.546	5.308
GD29-1	1798.822	1.078	0.364	316.158	1.704	5.690
GD29-6	1536.625	1.469	0.402	291.925	2.094	5.264
GD29-9	1491.249	1.560	0.516	286.148	2.231	5.211
GD32-8	1683.940	1.377	0.460	289.320	8.599	5.820
GD32-9	1687.096	1.194	0.470	308.147	2.256	5.475
GD33-5	1525.188	1.836	0.384	283.339	3.799	5.383
GD33-6	1553.386	1.865	0.543	315.375	2.673	4.926
GD33-7	1555.561	1.164	0.484	302.585	2.701	5.141
GD33-9	1449.621	2.639	0.495	272.718	4.851	5.315
GD36-2	1589.945	1.398	0.464	303.307	2.196	5.242
GD36-3	1839.752	1.827	0.660	339.866	2.475	5.413
GD36-11	1556.497	1.079	0.435	269.331	1.779	5.779

nd = element not detected.
* = outlier.

CHAPTER VII. DISCUSSION OF RESULTS

Descriptive statistics were calculated for all element concentration results at each excitation condition for each study. Means, standard deviations, and relative standard deviations within bags, within boxes, within and among manufacturing runs, and within manufacturers are shown in Tables 15 through 19. Means and standard deviations reported in these tables were rounded based on significant figures. Percent RSDs were rounded to whole percents for results greater than 10%, and to tenths of a percent for those less than 10%. A comparison was made of the RSDs for each element that was determined by more than one excitation condition. This process was done to make a single list of elements based on the best available data for use in comparison among samples. For each element, results from the excitation condition that yielded the lowest overall RSD were selected for use in the statistical evaluation. In cases where there was no significant difference between the results for two or more excitation conditions, the result from the excitation condition that either had better accuracy for the element in question or theoretically had better excitation efficiency for it was used. The excitation modes selected by this process for the elements in the single compositional profile are listed in Table 20. Results obtained using W-L excitation were not used, since Mo-K excitation provided results for the same elements with equivalent or better accuracy and precision. An additional advantage to this selection for further application of this study is the one-third reduction in analysis time.

TABLE 15a: Descriptive statistics for the study of compositional variation within bags. Excitation mode is Mo-K.

		Al	Si	P	S	Cl
GLAD	Mean	920	2200	136	36	nd
GB11	Standard Dev	120	300	8	5	
N=8	% RSD	13%	16%	6.2%	14%	
HEFTY	Mean	1400	4800	115	83	0.5
HA11	Standard Dev	200	500	10	6	1.5
N=8	% RSD	16%	11%	8.7%	7.6%	283%
RUFFIES	Mean	600	1800	89	80	7
RA11	Standard Dev	90	300	7	12	2
N=12	% RSD	16%	14%	7.6%	15%	30%
GLAD	Mean	2300	4800	140	45	nd
GA11	Standard Dev	500	900	30	19	
N=12	% RSD	22%	20%	25%	43%	

		K	Ca	Ti	Cr	Mn
GLAD	Mean	14.6	22	950	0.1	0.18
GB11	Standard Dev	1.8	3	140	0.3	0.07
N=8	% RSD	13%	16%	14%	282%	38%
HEFTY	Mean	22	285	1120	0.29	0.71
HA11	Standard Dev	5	10	100	0.09	0.09
N=8	% RSD	21%	3.5%	9.2%	31%	13%
RUFFIES	Mean	17	128	77	0.5	0.21
RA11	Standard Dev	3	10	7	0.5	0.05
N=12	% RSD	15%	7.9%	9.3%	89%	22%
GLAD	Mean	37	93	4100	1.0	1.54
GA11	Standard Dev	11	19	600	0.4	0.19
N=12	% RSD	29%	21%	14%	43%	12%

TABLE 15a (Continued)

		Fe	Ni	Cu	Zn	Rb
GLAD	Mean	100	0.17	0.7	45.0	0.007
GB11	Standard Dev	9	0.03	0.8	1.2	0.004
N=8	% RSD	8.6%	20%	105%	2.7%	55%
HEFTY	Mean	78	0.32	0.3	14	0.009
HA11	Standard Dev	8	0.09	0.3	2	0.012
N=8	% RSD	10%	28%	97%	17%	132%
RUFFIES	Mean	30	0.4	0.4	119	0.016
RA11	Standard Dev	4	0.2	0.4	9	0.012
N=12	% RSD	13%	54%	94%	7.2%	74%
GLAD	Mean	530	1.0	0.8	23.3	0.19
GA11	Standard Dev	70	0.6	0.3	1.9	0.04
N=12	% RSD	13%	54%	34%	8.1%	21%

		Sr	Zr	Pb	Ti/Fe
GLAD	Mean	0.57	0.35	0.51	9.4
GB11	Standard Dev	0.07	0.18	0.05	1.2
N=8	% RSD	12%	52%	9.5%	13%
HEFTY	Mean	0.27	0.11	0.21	14.5
HA11	Standard Dev	0.04	0.08	0.05	0.5
N=8	% RSD	14%	73%	23%	3.6%
RUFFIES	Mean	0.09	0.8	0.11	2.5
RA11	Standard Dev	0.02	0.6	0.03	0.2
N=12	% RSD	24%	73%	30%	8.5%
GLAD	Mean	1.4	3.9	0.88	7.81
GA11	Standard Dev	0.3	3.6	0.10	0.12
N=12	% RSD	25%	91%	11%	1.5%

TABLE 15b: Descriptive statistics for the study of compositional variation within bags. Excitation mode is W-*brems*.

		S	K	Ca	Ti	Cr
GLAD	Mean	55	11	21	1000	1.0
GB11	Standard Dev	11	2	3	50	0.4
N=8	% RSD	19%	19%	16%	4.7%	42%
HEFTY	Mean	128	15	279	1370	4.7
HA11	Standard Dev	10	4	7	150	0.5
N=8	% RSD	7.8%	28%	2.6%	11%	11%
RUFFIES	Mean	110	8	129	81	0.5
RA11	Standard Dev	20	2	8	5	0.4
N=12	% RSD	23%	30%	6.4%	6.0%	92%
GLAD	Mean	31	28	85	4200	6.2
GA11	Standard Dev	33	8	19	400	1.2
N=12	% RSD	109%	27%	23%	8.6%	20%

		Mn	Fe	Ni	Cu	Zn
GLAD	Mean	0.18	103	0.17	0.7	47.3
GB11	Standard Dev	0.11	8	0.03	0.8	1.1
N=8	% RSD	59%	7.5%	18%	104%	2.3%
HEFTY	Mean	0.80	93	0.27	0.3	15
HA11	Standard Dev	0.18	10	0.09	0.3	3
N=8	% RSD	23%	10%	33%	96%	17%
RUFFIES	Mean	0.22	28	0.40	0.3	122
RA11	Standard Dev	0.06	2	0.19	0.4	8
N=12	% RSD	25%	8.7%	47%	136%	6.9%
GLAD	Mean	1.4	540	0.9	.7	24.4
GA11	Standard Dev	0.3	40	0.5	.3	1.7
N=12	% RSD	23%	7.4%	53%	40%	6.9%

TABLE 15b (Continued)

		Rb	Sr	Y	Zr	Pb
GLAD	Mean	0.019	0.53	0.03	0.36	0.53
GB11	Standard Dev	0.010	0.04	0.03	0.16	0.06
N=8	% RSD	55%	7.4%	98%	45%	12%
HEFTY	Mean	0.020	0.26	0.009	0.33	0.25
HA11	Standard Dev	0.014	0.03	0.014	0.10	0.08
N=8	% RSD	70%	12%	148%	29%	30%
RUFFIES	Mean	0.013	0.077	0.04	0.7	0.16
RA11	Standard Dev	0.013	0.015	0.03	0.5	0.04
N=12	% RSD	99%	19%	81%	68%	28%
GLAD	Mean	0.12	1.2	0.26	3	0.98
GA11	Standard Dev	0.04	0.3	0.20	3	0.10
N=12	% RSD	29%	22%	75%	83%	9.8%

		Ba	Sn	Ag	Ti/Fe
GLAD	Mean	28	0.5	0.5	9.8
GB11	Standard Dev	2	0.7	1.2	1.0
N=8	% RSD	8.3%	128%	264%	9.8%
HEFTY	Mean	0.2	0.14	0.17	14.7
HA11	Standard Dev	0.2	0.15	0.37	0.4
N=8	% RSD	106%	113%	226%	2.6%
RUFFIES	Mean	0.17	0.2	0.02	3.0
RA11	Standard Dev	0.14	0.3	0.03	0.2
N=12	% RSD	80%	141%	111%	7.9%
GLAD	Mean	16.7	0.3	0.069	7.74
GA11	Standard Dev	0.9	0.3	0.018	0.13
N=12	% RSD	5.6%	114%	27%	1.7%

TABLE 15c: Descriptive statistics for the study of compositional variation within bags. Excitation mode is W-L.

		Al	Si	P	S	Cl
GLAD	Mean	960	2600	110	70	2
GB11	Standard Dev	180	600	20	20	4
N=8	% RSD	19%	24%	21%	31%	204%
HEFTY	Mean	1310	5000	88	220	4
HA11	Standard Dev	110	600	15	60	6
N=8	% RSD	8.5%	12%	17%	25%	148%
RUFFIES	Mean	420	2100	64	120	9
RA11	Standard Dev	70	300	9	40	11
N=12	% RSD	16%	17%	15%	34%	123%

		K	Ca	Ti	Cr	Mn
GLAD	Mean	24	33	950	0.6	0.38
GB11	Standard Dev	13	12	140	1.0	0.06
N=8	% RSD	52%	37%	14%	179%	16%
HEFTY	Mean	33	460	1120	0.7	0.95
HA11	Standard Dev	9	100	100	0.3	0.06
N=8	% RSD	27%	22%	9.2%	38%	5.8%
RUFFIES	Mean	19	190	76	1.2	0.46
RA11	Standard Dev	8	40	7	1.2	0.09
N=12	% RSD	41%	21%	9.3%	102%	19%

		Fe	Ni	Ti/Fe
GLAD	Mean	134	0.30	7.1
GB11	Standard Dev	14	0.10	1.0
N=8	% RSD	10%	35%	14%
HEFTY	Mean	97	0.45	11.6
HA11	Standard Dev	8	0.07	0.7
N=8	% RSD	8.6%	16%	6.4%
RUFFIES	Mean	45	0.7	1.71
RA11	Standard Dev	7	0.6	0.20
N=12	% RSD	16%	86%	11%

TABLE 16a: Descriptive statistics for each bag in box GB1 and for the entire box. Excitation mode is Mo-K.

		Al	Si	P	S	K
Bag #1	Mean	600	1280	114	10	7.5
	Standard Dev	60	160	3	2	2.0
	% RSD	11%	12%	2.4%	24%	26%
Bag #2	Mean	730	1600	108	15	12.3
	Standard Dev	100	200	4	3	1.7
	% RSD	13%	15%	3.9%	17%	14%
Bag #3	Mean	780	1960	120	18	6
	Standard Dev	60	140	7	5	2
	% RSD	7.2%	7.3%	5.7%	25%	34%
Bag #4	Mean	810	2030	120	18	5.1
	Standard Dev	60	180	8	5	0.8
	% RSD	7.4%	8.9%	6.5%	26%	15%
Bag #5	Mean	740	1840	119	17.8	10
	Standard Dev	40	90	8	0.9	4
	% RSD	5.6%	4.7%	7.0%	5.0%	35%
Bag #6	Mean	580	1300	121	16	12
	Standard Dev	60	200	6	6	5
	% RSD	9.6%	16%	5.2%	37%	44%
Bag #7	Mean	670	1480	118	13	11
	Standard Dev	50	140	5	3	4
	% RSD	7.7%	9.4%	4.6%	26%	38%
Bag #8	Mean	680	1800	122	15	8
	Standard Dev	30	200	6	6	6
	% RSD	4.4%	12%	4.6%	41%	75%
Bag #9	Mean	630	1380	118	12	9.2
	Standard Dev	40	80	7	5	1.5
	% RSD	6.2%	5.6%	6.4%	45%	17%
Bag #10	Mean	550	1400	118	12	10
	Standard Dev	70	300	6	2	3
	% RSD	13%	23%	5.0%	18%	33%
Box	Mean	680	1600	118	15	9
	Standard Dev	100	300	7	5	4
	% RSD	15%	20%	5.7%	32%	43%

TABLE 16a (Continued)

		Ca	Ti	Cr	Mn	Fe
Bag #1	Mean	15	720	nd	0.16	80
	Standard Dev	4	110		0.04	7
	% RSD	26%	15%		24%	9.2%
Bag #2	Mean	18	780	0.2	0.17	83
	Standard Dev	6	110	0.4	0.08	8
	% RSD	35%	14%	198%	47%	9.8%
Bag #3	Mean	16	800	0.05	0.21	86
	Standard Dev	4	50	0.06	0.07	3
	% RSD	22%	6.6%	119%	34%	3.5%
Bag #4	Mean	16	840	0.2	0.14	88
	Standard Dev	3	50	0.2	0.03	2
	% RSD	18%	5.6%	120%	19%	2.4%
Bag #5	Mean	16	770	0.05	0.17	82.1
	Standard Dev	3	16	0.06	0.04	1.1
	% RSD	19%	2.0%	116%	24%	1.3%
Bag #6	Mean	16	740	nd	0.14	78
	Standard Dev	4	80		0.03	6
	% RSD	24%	10%		19%	7.4%
Bag #7	Mean	16.4	750	nd	0.18	81
	Standard Dev	1.0	60		0.03	5
	% RSD	6.1%	8.6%		17%	6.2%
Bag #8	Mean	15	850	0.10	0.13	89
	Standard Dev	3	100	0.10	0.02	10
	% RSD	20%	12%	108%	16%	11%
Bag #9	Mean	14.1	700	nd	0.18	78
	Standard Dev	1.8	70		0.05	5
	% RSD	13%	9.7%		25%	6.3%
Bag #10	Mean	13.4	690	nd	0.16	77
	Standard Dev	1.6	70		0.06	6
	% RSD	12%	11%		37%	7.9%
Box	Mean	16	760	0.06	0.16	82
	Standard Dev	3	90	0.15	0.05	7
	% RSD	20%	11%	263%	29%	8.0%

TABLE 16a (Continued)

		Ni	Cu	Zn	Rb	Sr
Bag #1	Mean	0.15	0.06	41.6	0.003	0.46
	Standard Dev	0.05	0.04	1.2	0.002	0.03
	% RSD	32%	67%	2.9%	71%	5.5%
Bag #2	Mean	0.38	0.25	40.1	0.006	0.46
	Standard Dev	0.17	0.09	1.1	0.005	0.05
	% RSD	45%	35%	2.9%	84%	11%
Bag #3	Mean	0.12	0.24	40	0.005	0.48
	Standard Dev	0.04	0.09	2	0.004	0.03
	% RSD	32%	39%	5.6%	79%	6.4%
Bag #4	Mean	0.140	0.14	39	0.004	0.471
	Standard Dev	0.016	0.08	2	0.006	0.015
	% RSD	11%	55%	6.0%	155%	3.2%
Bag #5	Mean	0.15	0.23	39	0.006	0.47
	Standard Dev	0.03	0.09	3	0.009	0.03
	% RSD	19%	39%	6.7%	154%	6.2%
Bag #6	Mean	0.12	0.11	40.3	0.012	0.45
	Standard Dev	0.04	0.09	1.0	0.008	0.03
	% RSD	36%	79%	2.6%	65%	6.6%
Bag #7	Mean	0.07	0.32	44	0.001	0.43
	Standard Dev	0.03	0.06	2	0.001	0.03
	% RSD	42%	20%	4.7%	182%	6.1%
Bag #8	Mean	0.11	0.18	41.1	0.005	0.48
	Standard Dev	0.02	0.15	1.0	0.007	0.03
	% RSD	22%	82%	2.4%	127%	5.3%
Bag #9	Mean	0.063	0.12	40.6	0.001	0.433
	Standard Dev	0.022	0.06	0.8	0.001	0.012
	% RSD	35%	46%	1.9%	92%	2.8%
Bag #10	Mean	0.08	0.08	40.7	0.005	0.44
	Standard Dev	0.02	0.05	1.3	0.005	0.04
	% RSD	30%	56%	3.3%	104%	7.9%
Box	Mean	0.13	0.17	41	0.005	0.46
	Standard Dev	0.09	0.11	2	0.006	0.03
	% RSD	71%	63%	5.1%	120%	6.8%

TABLE 16a (Continued)

		Zr	Pb	Ti/Fe
Bag #1	Mean	0.48	0.463	9.0
	Standard Dev	0.25	0.012	0.6
	% RSD	52%	2.7%	6.8%
Bag #2	Mean	25	0.52	9.3
	Standard Dev	16	0.06	0.4
	% RSD	66%	12%	4.8%
Bag #3	Mean	2.2	0.53	9.3
	Standard Dev	2.8	0.03	0.3
	% RSD	126%	5.2%	3.7%
Bag #4	Mean	1.2	0.520	9.6
	Standard Dev	0.7	0.007	0.3
	% RSD	56%	1.4%	3.6%
Bag #5	Mean	1.0	0.50	9.4
	Standard Dev	0.7	0.03	0.3
	% RSD	64%	6.6%	3.0%
Bag #6	Mean	2.5	0.54	9.5
	Standard Dev	1.2	0.09	0.5
	% RSD	47%	16%	4.8%
Bag #7	Mean	1.22	0.7	9.3
	Standard Dev	0.10	0.3	0.3
	% RSD	8.3%	39%	2.9%
Bag #8	Mean	0.40	0.52	9.5
	Standard Dev	0.12	0.03	0.2
	% RSD	29%	5.9%	2.5%
Bag #9	Mean	0.55	0.487	8.9
	Standard Dev	0.09	0.005	0.4
	% RSD	16%	1.0%	4.6%
Bag #10	Mean	0.46	0.47	8.9
	Standard Dev	0.18	0.04	0.3
	% RSD	38%	7.3%	3.8%
Box	Mean	3	0.53	9.3
	Standard Dev	9	0.11	0.4
	% RSD	247%	21%	4.4%

TABLE 16b: Descriptive statistics for each bag in box GB1 and for the entire box. Excitation mode is W-*brems*.

		S	K	Ca	Ti	Cr
Bag #1	Mean	11	8	17	740	0.5
	Standard Dev	9	3	3	90	0.4
	% RSD	87%	39%	20%	13%	79%
Bag #2	Mean	2	14	33	840	1.5
	Standard Dev	4	5	31	30	0.5
	% RSD	200%	37%	95%	3.8%	31%
Bag #3	Mean	6	5.2	15	890	1.3
	Standard Dev	7	1.8	4	90	0.6
	% RSD	123%	35%	30%	10%	46%
Bag #4	Mean	14	4.1	17	940	1.5
	Standard Dev	10	0.4	4	70	0.3
	% RSD	72%	8.5%	24%	7.3%	19%
Bag #5	Mean	14	9	17	844	1.22
	Standard Dev	10	4	3	13	0.16
	% RSD	72%	46%	20%	1.5%	13%
Bag #6	Mean	8	9.5	15.5	730	0.2
	Standard Dev	8	1.9	0.9	90	0.2
	% RSD	102%	20%	5.6%	13%	98%
Bag #7	Mean	22	12	18.3	810	0.5
	Standard Dev	21	4	1.6	160	0.4
	% RSD	98%	35%	8.5%	20%	81%
Bag #8	Mean	17	7	16	940	1.77
	Standard Dev	13	6	3	130	0.13
	% RSD	75%	79%	18%	14%	7.4%
Bag #9	Mean	7	8.9	16.0	760	0.38
	Standard Dev	8	0.5	1.9	20	0.12
	% RSD	116%	5.3%	12%	2.7%	31%
Bag #10	Mean	6	10	15.0	730	0.8
	Standard Dev	8	3	1.7	80	0.6
	% RSD	120%	29%	11%	11%	75%
Box	Mean	11	9	18	820	1.0
	Standard Dev	11	4	11	110	0.6
	% RSD	106%	47%	60%	14%	65%

TABLE 16b (Continued)

		Mn	Fe	Ni	Cu	Zn
Bag #1	Mean	0.16	82	0.13	0.06	43.9
	Standard Dev	0.12	6	0.05	0.06	1.6
	% RSD	76%	7.8%	42%	100%	3.6%
Bag #2	Mean	0.32	90	0.5	0.29	42.6
	Standard Dev	0.12	3	0.3	0.08	1.4
	% RSD	38%	3.5%	61%	29%	3.2%
Bag #3	Mean	0.36	96	0.06	0.27	43
	Standard Dev	0.13	5	0.03	0.09	3
	% RSD	35%	5.5%	51%	32%	6.4%
Bag #4	Mean	0.17	96	0.11	0.12	40
	Standard Dev	0.12	3	0.05	0.10	2
	% RSD	74%	2.6%	52%	85%	5.4%
Bag #5	Mean	0.18	89	0.11	0.19	41
	Standard Dev	0.07	3	0.03	0.09	3
	% RSD	36%	2.9%	29%	48%	7.0%
Bag #6	Mean	0.14	78	0.08	0.03	42.0
	Standard Dev	0.12	8	0.05	0.04	1.2
	% RSD	87%	11%	59%	114%	3.0%
Bag #7	Mean	0.13	86	0.07	0.5	46.4
	Standard Dev	0.14	12	0.05	0.3	1.8
	% RSD	104%	14%	66%	59%	3.8%
Bag #8	Mean	0.21	94	0.06	0.17	44.6
	Standard Dev	0.12	2	0.06	0.10	0.9
	% RSD	58%	2.2%	94%	58%	2.1%
Bag #9	Mean	0.18	82.6	0.04	0.06	42.5
	Standard Dev	0.06	1.2	0.03	0.06	0.6
	% RSD	34%	1.5%	79%	98%	1.4%
Bag #10	Mean	0.24	81	0.064	0.08	43.0
	Standard Dev	0.14	8	0.007	0.06	1.1
	% RSD	58%	9.6%	11%	78%	2.5%
Box	Mean	0.21	88	0.12	0.18	43
	Standard Dev	0.13	8	0.15	0.17	2
	% RSD	60%	9.4%	126%	93%	5.2%

TABLE 16b (Continued)

		Rb	Sr	Y	Zr	Pb
Bag #1	Mean	nd	0.449	0.020	0.5	0.42
	Standard Dev		0.007	0.010	0.2	0.05
	% RSD		1.6%	50%	45%	12%
Bag #2	Mean	0.005	0.46	0.07	22	0.48
	Standard Dev	0.006	0.06	0.02	12	0.04
	% RSD	106%	119%	33%	55%	7.5%
Bag #3	Mean	0.001	0.46	0.021	2	0.49
	Standard Dev	0.001	0.04	0.005	3	0.03
	% RSD	200%	9.2%	25%	123%	5.2%
Bag #4	Mean	0.003	0.47	0.002	1.0	0.46
	Standard Dev	0.006	0.03	0.003	0.7	0.03
	% RSD	173%	5.3%	119%	72%	5.6%
Bag #5	Mean	0.006	0.41	0.017	1.2	0.45
	Standard Dev	0.011	0.03	0.002	0.6	0.03
	% RSD	173%	8.0%	111%	51%	6.1%
Bag #6	Mean	nd	0.43	0.013	1.8	0.46
	Standard Dev		0.03	0.008	0.7	0.03
	% RSD		5.7%	61%	41%	7.2%
Bag #7	Mean	0.009	0.43	0.026	1.2	0.53
	Standard Dev	0.011	0.03	0.012	0.2	0.06
	% RSD	117%	6.0%	48%	19%	12%
Bag #8	Mean	0.016	0.47	0.006	0.43	0.50
	Standard Dev	0.018	0.03	0.009	0.10	0.04
	% RSD	109%	5.3%	154%	23%	8.0%
Bag #9	Mean	0.001	0.427	0.010	0.56	0.432
	Standard Dev	0.002	0.012	0.008	0.06	0.019
	% RSD	172%	2.9%	82%	10%	4.4%
Bag #10	Mean	0.005	0.435	0.010	0.45	0.43
	Standard Dev	0.004	0.012	0.003	0.12	0.03
	% RSD	83%	2.7%	31%	26%	6.5%
Box	Mean	0.005	0.45	0.02	3	0.46
	Standard Dev	0.009	0.03	0.02	7	0.05
	% RSD	177%	7.4%	105%	229%	9.7%

TABLE 16b (Continued)

		Ba	Sn	Ag	Ti/Fe
Bag #1	Mean	21.8	0.131	0.012	9.1
	Standard Dev	1.7	0.019	0.011	0.7
	% RSD	7.8%	15%	89%	7.3%
Bag #2	Mean	22.6	0.29	0.031	9.33
	Standard Dev	1.0	0.05	0.019	0.14
	% RSD	4.4%	17%	62%	1.5%
Bag #3	Mean	23.7	0.24	0.014	9.6
	Standard Dev	1.2	0.07	0.016	0.2
	% RSD	5.3%	30%	111%	2.3%
Bag #4	Mean	22.8	0.21	0.037	9.8
	Standard Dev	1.4	0.02	0.010	0.5
	% RSD	6.0%	10%	27%	4.8%
Bag #5	Mean	20.0	0.165	0.029	9.5
	Standard Dev	1.2	0.013	0.009	0.2
	% RSD	6.0%	7.9%	29%	2.6%
Bag #6	Mean	21.4	0.19	0.022	9.3
	Standard Dev	0.7	0.07	0.016	0.2
	% RSD	3.1%	35%	74%	2.5%
Bag #7	Mean	21.6	0.21	0.024	9.3
	Standard Dev	1.1	0.05	0.003	0.6
	% RSD	5.2%	22%	12%	6.7%
Bag #8	Mean	21.3	0.20	0.028	9.42
	Standard Dev	1.2	0.04	0.015	0.19
	% RSD	5.5%	20%	53%	2.0%
Bag #9	Mean	23.1	0.17	0.026	9.3
	Standard Dev	1.8	0.02	0.019	0.3
	% RSD	7.7%	14%	72%	3.0%
Bag #10	Mean	21.9	0.16	0.022	9.03
	Standard Dev	0.4	0.02	0.016	0.20
	% RSD	1.7%	13%	72%	2.2%
Box	Mean	22.1	0.19	0.024	9.3
	Standard Dev	1.5	0.05	0.014	0.4
	% RSD	6.6%	28%	59%	4.3%

TABLE 16c: Descriptive statistics for each bag in box GB1 and for the entire box. Excitation mode is W-L.

		Al	Si	P	S	K
Bag #1	Mean	560	1300	80	21	13
	Standard Dev	100	200	20	10	8
	% RSD	18%	17%	30%	49%	59%
Bag #2	Mean	790	1800	104	42	29
	Standard Dev	90	200	14	10	4
	% RSD	12%	13%	13%	24%	13%
Bag #3	Mean	920	2500	128	56	22
	Standard Dev	100	600	13	9	3
	% RSD	11%	23%	9.8%	15%	15%
Bag #4	Mean	900	2300	122	52	20.0
	Standard Dev	80	300	15	8	1.9
	% RSD	9.0%	12%	13%	16%	9.3%
Bag #5	Mean	810	2060	121	54	28
	Standard Dev	40	180	7	7	9
	% RSD	5.1%	8.6%	6.0%	14%	32%
Bag #6	Mean	550	1200	85	28	19
	Standard Dev	80	300	16	14	12
	% RSD	14%	27%	19%	48%	66%
Bag #7	Mean	700	1500	100	38	18
	Standard Dev	200	500	40	20	7
	% RSD	32%	31%	36%	53%	38%
Bag #8	Mean	900	2300	130	50	28
	Standard Dev	200	700	30	20	20
	% RSD	24%	30%	20%	40%	72%
Bag #9	Mean	620	1400	102	31	21
	Standard Dev	20	150	20	10	7
	% RSD	3.9%	11%	20%	33%	35%
Bag #10	Mean	640	1500	100	29	22
	Standard Dev	110	400	20	12	8
	% RSD	18%	28%	21%	41%	35%
Box	Mean	730	1800	110	41	22
	Standard Dev	170	600	20	17	10
	% RSD	24%	32%	23%	41%	43%

TABLE 16c (Continued)

		Ca	Ti	Cr	Mn	Fe
Bag #1	Mean	18	720	0.018	0.25	100
	Standard Dev	7	110	0.013	0.05	13
	% RSD	39%	15%	72%	18%	13%
Bag #2	Mean	48	780	0.6	0.31	111
	Standard Dev	48	110	0.7	0.11	3
	% RSD	99%	14%	115%	35%	2.6%
Bag #3	Mean	30	800	0.72	0.29	125
	Standard Dev	6	50	0.12	0.04	6
	% RSD	20%	6.6%	16%	12%	4.4%
Bag #4	Mean	29	840	0.9	0.35	130
	Standard Dev	8	50	0.6	0.04	3
	% RSD	27%	5.6%	63%	12%	2.5%
Bag #5	Mean	28	770	0.6	0.32	120
	Standard Dev	8	16	0.3	0.03	4
	% RSD	28%	2.0%	53%	9.7%	3.3%
Bag #6	Mean	20	740	0.05	0.24	98
	Standard Dev	8	80	0.06	0.06	13
	% RSD	41%	10%	117%	25%	14%
Bag #7	Mean	23	750	0.04	0.26	110
	Standard Dev	11	60	0.03	0.09	30
	% RSD	45%	8.6%	75%	36%	24%
Bag #8	Mean	31	850	0.7	0.33	129
	Standard Dev	11	100	0.3	0.08	19
	% RSD	36%	12%	47%	26%	15%
Bag #9	Mean	21	700	0.05	0.26	108
	Standard Dev	4	70	0.08	0.05	12
	% RSD	19%	9.7%	168%	18%	11%
Bag #10	Mean	21	690	0.06	0.24	107
	Standard Dev	8	70	0.08	0.04	16
	% RSD	39%	11%	145%	16%	15%
Box	Mean	27	760	0.4	0.29	113
	Standard Dev	17	90	0.4	0.07	17
	% RSD	63%	11%	119%	24%	15%

TABLE 16c (Continued)

		Ni	Ti/Fe
Bag #1	Mean	0.20	7.2
	Standard Dev	0.07	1.0
	% RSD	33%	14%
Bag #2	Mean	0.7	7.0
	Standard Dev	0.4	0.9
	% RSD	55%	13%
Bag #3	Mean	0.24	6.4
	Standard Dev	0.05	0.3
	% RSD	19%	4.1%
Bag #4	Mean	0.26	6.5
	Standard Dev	0.04	0.5
	% RSD	15%	8.1%
Bag #5	Mean	0.27	6.44
	Standard Dev	0.03	0.19
	% RSD	11%	2.9%
Bag #6	Mean	0.14	7.7
	Standard Dev	0.04	0.4
	% RSD	31%	5.1%
Bag #7	Mean	0.13	7.3
	Standard Dev	0.03	1.1
	% RSD	22%	16%
Bag #8	Mean	0.24	6.6
	Standard Dev	0.03	0.2
	% RSD	14%	3.8%
Bag #9	Mean	0.14	6.5
	Standard Dev	0.03	1.0
	% RSD	21%	15%
Bag #10	Mean	0.19	6.6
	Standard Dev	0.06	1.2
	% RSD	34%	18%
Box	Mean	0.25	6.8
	Standard Dev	0.19	0.8
	% RSD	77%	12%

TABLE 17a: Descriptive statistics for each bag in box GE1 and for the entire box. Excitation mode is Mo-K.

		Al	Si	P	S	K
Bag #1	Mean	710	1490	119	14	6
	Standard Dev	80	120	11	9	4
	% RSD	12%	8.2%	9.6%	70%	72%
Bag #2	Mean	650	1390	126	16	6
	Standard Dev	60	90	4	11	5
	% RSD	8.9%	6.8%	2.9%	67%	75%
Bag #3	Mean	640	1550	121	20	10
	Standard Dev	70	100	5	19	9
	% RSD	11%	6.6%	4.5%	93%	87%
Bag #4	Mean	690	1700	125	17	7
	Standard Dev	80	180	15	23	9
	% RSD	12%	11%	12%	130%	124%
Bag #5	Mean	650	1460	122	14	10
	Standard Dev	80	160	7	5	8
	% RSD	12%	11%	5.6%	34%	88%
Bag #6	Mean	720	1590	126	18	7
	Standard Dev	60	190	3	17	5
	% RSD	7.9%	12%	2.2%	92%	69%
Bag #7	Mean	700	1570	127	17	10
	Standard Dev	60	110	10	14	8
	% RSD	7.8%	7.3%	7.6%	83%	85%
Bag #8	Mean	680	1520	113	nd	nd
	Standard Dev	60	80	5		
	% RSD	9.0%	5.1%	4.4%		
Bag #9	Mean	758	1660	109	9	6
	Standard Dev	16	170	13	14	7
	% RSD	2.1%	10%	12%	155%	123%
Bag #10	Mean	760	1700	121	11	8
	Standard Dev	40	200	17	19	14
	% RSD	5.6%	13%	14%	173%	173%
Bag #11	Mean	640	1390	116	17	9
	Standard Dev	40	120	3	18	10
	% RSD	6.3%	8.4%	2.7%	103%	108%
Bag #12	Mean	680	1460	115	11	7
	Standard Dev	40	30	3	10	7
	% RSD	5.4%	2.3%	2.4%	89%	103%
Bag #13	Mean	683	1500	120	nd	1.4
	Standard Dev	8	40	7		1.3
	% RSD	1.2%	2.6%	5.9%		94%
Box	Mean	690	1540	120	13	7
	Standard Dev	60	160	10	14	7
	% RSD	9.2%	11%	8.1%	103%	103%

TABLE 17a (Continued)

		Ca	Ti	Cr	Mn	Fe
Bag #1	Mean	28	890	0.08	0.30	19.8
	Standard Dev	12	30	0.15	0.03	1.2
	% RSD	44%	3.6%	200%	9.5%	6.2%
Bag #2	Mean	13.2	840	nd	0.26	18.9
	Standard Dev	0.8	30		0.03	0.9
	% RSD	6.3%	3.1%		10%	5.0%
Bag #3	Mean	19	930	0.6	0.30	22
	Standard Dev	6	30	1.1	0.07	3
	% RSD	32%	2.7%	173%	22%	15%
Bag #4	Mean	21	970	nd	0.26	21.3
	Standard Dev	13	50		0.04	1.5
	% RSD	61%	4.8%		16%	6.9%
Bag #5	Mean	14.0	870	nd	0.25	19.6
	Standard Dev	1.7	60		0.04	2.0
	% RSD	12%	6.9%		16%	10%
Bag #6	Mean	15	910	nd	0.27	21
	Standard Dev	2	90		0.05	2
	% RSD	15%	9.8%		19%	12%
Bag #7	Mean	17	890	nd	0.27	18.7
	Standard Dev	7	80		0.07	1.3
	% RSD	40%	8.9%		26%	7.0%
Bag #8	Mean	25	870	nd	0.28	19.1
	Standard Dev	10	70		0.04	1.2
	% RSD	39%	8.0%		14%	6.2%
Bag #9	Mean	21	970	nd	0.33	24
	Standard Dev	5	100		0.08	4
	% RSD	27%	11%		24%	19%
Bag #10	Mean	22	910	nd	0.35	24
	Standard Dev	7	100		0.04	4
	% RSD	32%	11%		12%	19%
Bag #11	Mean	15	800	nd	0.26	18.1
	Standard Dev	4	60		0.03	1.1
	% RSD	24%	7.0%		13%	5.9%
Bag #12	Mean	15	840	nd	0.26	18.6
	Standard Dev	5	90		0.03	1.2
	% RSD	34%	10%		13%	6.6%
Bag #13	Mean	22	820	nd	0.27	18.7
	Standard Dev	12	30		0.06	0.9
	% RSD	54%	4.2%		23%	4.7%
Box	Mean	19	890	0.05	0.28	20
	Standard Dev	8	80	0.28	0.05	3
	% RSD	42%	8.7%	590%	18%	14%

TABLE 17a (Continued)

		Ni	Cu	Zn	Rb	Sr
Bag #1	Mean	0.20	1.18	50	nd	0.610
	Standard Dev	0.05	0.20	3		0.017
	% RSD	25%	17%	6.3%		2.8%
Bag #2	Mean	0.08	1.07	48.3	nd	0.59
	Standard Dev	0.04	0.08	0.9		0.02
	% RSD	50%	7.7%	1.8%		3.7%
Bag #3	Mean	0.5	1.5	48.8	nd	0.58
	Standard Dev	0.9	0.6	0.7		0.03
	% RSD	167%	38%	1.4%		5.4%
Bag #4	Mean	0.03	1.18	47	nd	0.62
	Standard Dev	0.04	0.04	2		0.03
	% RSD	116%	3.6%	4.6%		4.0%
Bag #5	Mean	0.03	1.16	53	nd	0.61
	Standard Dev	0.03	0.06	13		0.06
	% RSD	106%	5.4%	25%		10%
Bag #6	Mean	0.015	1.4	47.5	nd	0.629
	Standard Dev	0.012	0.2	1.8		0.011
	% RSD	85%	16%	3.8%		1.8%
Bag #7	Mean	0.03	1.25	49.1	0.001	0.59
	Standard Dev	0.03	0.10	1.8	0.002	0.05
	% RSD	114%	8.0%	3.7%	200%	8.6%
Bag #8	Mean	nd	1.14	51	nd	0.62
	Standard Dev		0.11	4		0.03
	% RSD		9.7%	7.0%		4.0%
Bag #9	Mean	0.07	1.23	47	nd	0.63
	Standard Dev	0.12	0.16	2		0.03
	% RSD	170%	13%	4.4%		5.3%
Bag #10	Mean	0.007	1.127	51	nd	0.65
	Standard Dev	0.013	0.015	5		0.02
	% RSD	173%	1.3%	8.8%		3.7%
Bag #11	Mean	nd	1.07	46.3	0.001	0.602
	Standard Dev		0.08	1.2	0.002	0.013
	% RSD		7.3%	2.5%	173%	2.1%
Bag #12	Mean	nd	1.10	47.1	nd	0.593
	Standard Dev		0.14	0.5		0.019
	% RSD		13%	1.0%		3.1%
Bag #13	Mean	0.03	1.13	47.3	nd	0.633
	Standard Dev	0.04	0.06	2.0		0.013
	% RSD	115%	5.7%	4.2%		2.0%
Box	Mean	0.08	1.19	49	0.000	0.61
	Standard Dev	0.24	0.20	4	0.001	0.03
	% RSD	308%	17%	8.9%	475%	%

TABLE 17a (Continued)

		Zr	Pb	Ti/Fe
Bag #1	Mean	14	0.10	45.0
	Standard Dev	6	0.03	1.9
	% RSD	46%	27%	4.2%
Bag #2	Mean	11	0.07	44
	Standard Dev	4	0.03	2
	% RSD	37%	37%	5.5%
Bag #3	Mean	3.2	0.100	42
	Standard Dev	1.4	0.013	5
	% RSD	42%	13%	13%
Bag #4	Mean	2.3	0.09	46
	Standard Dev	1.1	0.02	3
	% RSD	47%	24%	5.6%
Bag #5	Mean	1.3	0.2	44.7
	Standard Dev	1.4	0.2	1.6
	% RSD	103%	128%	3.7%
Bag #6	Mean	0.8	0.07	45
	Standard Dev	0.7	0.03	4
	% RSD	77%	44%	8.4%
Bag #7	Mean	0.42	0.10	47
	Standard Dev	0.06	0.03	2
	% RSD	14%	25%	4.8%
Bag #8	Mean	0.8	0.08	45
	Standard Dev	0.3	0.03	3
	% RSD	35%	34%	5.9%
Bag #9	Mean	0.9	0.12	41
	Standard Dev	0.5	0.09	8
	% RSD	59%	75%	20%
Bag #10	Mean	0.49	0.06	38
	Standard Dev	0.08	0.02	5
	% RSD	17%	36%	14%
Bag #11	Mean	0.4	0.07	44.5
	Standard Dev	0.2	0.05	0.5
	% RSD	49%	66%	1.1%
Bag #12	Mean	1.2	0.076	45
	Standard Dev	1.4	0.009	3
	% RSD	118%	12%	6.3%
Bag #13	Mean	0.16	0.077	44.2
	Standard Dev	0.08	0.009	0.3
	% RSD	49%	12%	0.8%
Box	Mean	3	0.10	44
	Standard Dev	5	0.08	4
	% RSD	160%	79%	8.9%

TABLE 17b: Descriptive statistics for each bag in box GE1 and for the entire box. Excitation mode is W-*brems*.

		S	K	Ca	Ti	Cr
Bag #1	Mean	nd	8.0	29	1070	1.8
	Standard Dev		5.6	12	20	0.4
	% RSD		69%	40%	2.3%	20%
Bag #2	Mean	1	9	13	1070	1.4
	Standard Dev	3	5	3	40	0.2
	% RSD	200%	56%	20%	3.4%	18%
Bag #3	Mean	34	13	19	1100	1.3
	Standard Dev	33	11	6	70	0.3
	% RSD	96%	87%	33%	6.0%	22%
Bag #4	Mean	20	10	23	1090	0.83
	Standard Dev	30	11	14	30	0.16
	% RSD	141%	105%	61%	2.7%	19%
Bag #5	Mean	14	12	30	1060	1.1
	Standard Dev	16	9	29	30	0.3
	% RSD	118%	76%	98%	3.2%	28%
Bag #6	Mean	32	10	35	1090	1.00
	Standard Dev	29	7	37	60	0.12
	% RSD	91%	71%	106%	5.6%	12%
Bag #7	Mean	22	12	19	1040	1.0
	Standard Dev	19	10	7	60	0.3
	% RSD	84%	87%	36%	5.5%	28%
Bag #8	Mean	nd	nd	26	1030	1.2
	Standard Dev			11	70	0.2
	% RSD			41%	7.0%	18%
Bag #9	Mean	17	7	20	1070	1.3
	Standard Dev	23	7	6	30	0.4
	% RSD	140%	100%	27%	3.1%	28%
Bag #10	Mean	12	10	22	1060	0.5
	Standard Dev	20	15	7	30	0.5
	% RSD	173%	157%	33%	2.4%	97%
Bag #11	Mean	30	11	16	990	1.13
	Standard Dev	40	11	5	40	0.11
	% RSD	139%	105%	32%	3.7%	9.5%
Bag #12	Mean	13	9	16	1010	1.4
	Standard Dev	13	8	4	20	0.5
	% RSD	99%	92%	27%	2.1%	36%
Bag #13	Mean	nd	4	22	1030	0.88
	Standard Dev		3	11	60	0.07
	% RSD		79%	52%	5.5%	7.7%
Box	Mean	15	9	23	1060	1.1
	Standard Dev	22	8	15	50	0.4
	% RSD	147%	91%	68%	4.6%	35%

TABLE 17b (Continued)

		Mn	Fe	Ni	Cu	Zn
Bag #1	Mean	0.10	23.0	0.20	1.3	54
	Standard Dev	0.13	0.8	0.04	0.2	3
	% RSD	135%	3.4%	22%	18%	6.1%
Bag #2	Mean	0.12	22.5	0.11	1.21	52.7
	Standard Dev	0.11	1.5	0.05	0.11	1.4
	% RSD	88%	6.7%	43%	9.2%	2.7%
Bag #3	Mean	0.03	25	0.6	1.6	52.9
	Standard Dev	0.05	3	1.0	0.6	1.0
	% RSD	173%	12%	169%	36%	1.8%
Bag #4	Mean	0.12	22.8	0.06	1.23	51.1
	Standard Dev	0.11	1.8	0.03	0.07	1.9
	% RSD	90%	7.7%	50%	5.5%	3.7%
Bag #5	Mean	0.04	22.4	0.05	1.21	50.6
	Standard Dev	0.05	1.2	0.03	0.05	0.3
	% RSD	116%	5.5%	55%	4.0%	0.5%
Bag #6	Mean	0.13	23.0	0.03	1.46	52
	Standard Dev	0.10	1.5	0.03	0.15	2
	% RSD	80%	6.6%	90%	10%	4.3%
Bag #7	Mean	0.15	21.4	0.08	2.2	52.9
	Standard Dev	0.15	0.7	0.05	1.7	1.5
	% RSD	98%	3.4%	58%	78%	2.8%
Bag #8	Mean	0.16	21.9	0.005	1.29	56
	Standard Dev	0.03	0.7	0.009	0.13	4
	% RSD	17%	3.3%	173%	9.7%	7.2%
Bag #9	Mean	0.12	23	0.09	1.3	51
	Standard Dev	0.10	2	0.08	0.2	2
	% RSD	77%	11%	87%	17%	4.2%
Bag #10	Mean	0.21	26	0.006	1.19	54
	Standard Dev	0.05	4	0.010	0.09	5
	% RSD	26%	14%	173%	7.3%	8.4%
Bag #11	Mean	0.07	21.4	0.008	1.15	51.0
	Standard Dev	0.06	0.6	0.014	0.11	1.0
	% RSD	86%	2.9%	173%	9.9%	2.0%
Bag #12	Mean	0.01	21.7	0.013	1.3	51.5
	Standard Dev	0.02	1.1	0.019	0.2	0.8
	% RSD	173%	5.2%	145%	16%	1.6%
Bag #13	Mean	0.04	21.3	0.019	1.21	50
	Standard Dev	0.06	0.8	0.016	0.04	3
	% RSD	173%	3.7%	82%	3.2%	6.1%
Box	Mean	0.10	22.7	0.1	1.4	52
	Standard Dev	0.10	2.0	0.3	0.6	3
	% RSD	94%	8.7%	270%	41%	4.9%

TABLE 17b (Continued)

		Rb	Sr	Y	Zr	Pb
Bag #1	Mean	0.010	0.57	0.031	14	0.14
	Standard Dev	0.012	0.05	0.012	6	0.04
	% RSD	116%	7.9%	39%	40%	26%
Bag #2	Mean	0.005	0.545	0.022	11	0.12
	Standard Dev	0.006	0.010	0.010	4	0.04
	% RSD	119%	1.9%	46%	36%	30%
Bag #3	Mean	nd	0.552	0.016	3.3	0.14
	Standard Dev		0.008	0.008	1.2	0.03
	% RSD		1.4%	47%	37%	24%
Bag #4	Mean	0.007	0.55	0.005	2.1	0.13
	Standard Dev	0.009	0.05	0.006	0.9	0.03
	% RSD	117%	8.5%	135%	43%	20%
Bag #5	Mean	nd	0.61	0.011	1.4	0.16
	Standard Dev		0.09	0.006	1.3	0.05
	% RSD		14%	55%	91%	30%
Bag #6	Mean	nd	0.584	0.013	0.9	0.14
	Standard Dev		0.016	0.009	0.6	0.05
	% RSD		2.7%	69%	73%	38%
Bag #7	Mean	0.004	0.58	0.003	0.50	0.13
	Standard Dev	0.008	0.04	0.004	0.03	0.06
	% RSD	189%	7.0%	135%	6.3%	49%
Bag #8	Mean	nd	0.556	0.009	0.8	0.11
	Standard Dev		0.003	0.010	0.3	0.04
	% RSD		0.6%	105%	30%	34%
Bag #9	Mean	0.006	0.58	0.009	0.9	0.18
	Standard Dev	0.006	0.03	0.002	0.5	0.09
	% RSD	93%	5.0%	26%	53%	50%
Bag #10	Mean	nd	0.598	0.000	0.50	0.12
	Standard Dev		0.018	0.001	0.04	0.04
	% RSD		2.9%	173%	7.9%	36%
Bag #11	Mean	0.003	0.55	0.003	0.5	0.14
	Standard Dev	0.005	0.02	0.004	0.2	0.08
	% RSD	173%	4.1%	116%	39%	56%
Bag #12	Mean	0.001	0.56	0.008	1.2	0.14
	Standard Dev	0.001	0.03	0.007	1.3	0.02
	% RSD	173%	5.0%	79%	107%	17%
Bag #13	Mean	nd	0.56	0.003	0.25	0.124
	Standard Dev		0.03	0.004	0.06	0.017
	% RSD		5.7%	118%	26%	14%
Box	Mean	0.003	0.57	0.011	3	0.14
	Standard Dev	0.006	0.04	0.011	4	0.05
	% RSD	195%	6.7%	99%	156%	34%

TABLE 17b (Continued)

		Ba	Sn	Ag	Ti/Fe
Bag #1	Mean	34.8	0.035	0.047	47
	Standard Dev	1.9	0.008	0.015	2
	% RSD	5.6%	22%	33%	4.7%
Bag #2	Mean	33.6	0.047	0.034	48
	Standard Dev	0.8	0.018	0.008	4
	% RSD	2.4%	39%	24%	7.4%
Bag #3	Mean	33.4	0.3	0.10	44
	Standard Dev	0.3	0.5	0.13	8
	% RSD	0.8%	171%	132%	17%
Bag #4	Mean	34.8	0.04	0.013	48
	Standard Dev	1.2	0.03	0.015	3
	% RSD	3.6%	73%	115%	6.8%
Bag #5	Mean	35	0.05	0.016	48
	Standard Dev	2	0.06	0.012	3
	% RSD	6.1%	137%	75%	5.7%
Bag #6	Mean	34.7	0.16	0.042	48
	Standard Dev	1.1	0.20	0.014	2
	% RSD	3.2%	129%	33%	5.1%
Bag #7	Mean	34.1	0.8	0.06	49
	Standard Dev	1.8	1.4	0.10	3
	% RSD	5.1%	183%	170%	5.9%
Bag #8	Mean	37	0.5	0.7	47
	Standard Dev	2	0.3	1.2	2
	% RSD	5.8%	60%	166%	5.3%
Bag #9	Mean	37.7	0.12	0.2	43
	Standard Dev	1.9	0.06	0.4	8
	% RSD	5.1%	49%	188%	19%
Bag #10	Mean	36.8	0.08	0.02	41
	Standard Dev	0.7	0.04	0.04	5
	% RSD	1.9%	50%	153%	13%
Bag #11	Mean	36.2	1.1	0.030	46.4
	Standard Dev	1.0	1.7	0.017	0.6
	% RSD	2.7%	162%	58%	1.2%
Bag #12	Mean	35.3	0.21	0.02	46.7
	Standard Dev	1.1	0.17	0.02	1.4
	% RSD	3.1%	80%	98%	3.0%
Bag #13	Mean	37.1	0.08	0.04	48.3
	Standard Dev	0.8	0.05	0.02	1.4
	% RSD	2.3%	61%	66%	2.8%
Box	Mean	35.3	0.2	0.1	46
	Standard Dev	1.8	0.6	0.3	4
	% RSD	5.2%	246%	335%	8.7%

TABLE 17c: Descriptive statistics for each bag in box GE1 and for the entire box. Excitation mode is W-L.

		Al	Si	P	S	K
Bag #1	Mean	650	1500	135	60	24
	Standard Dev	40	80	11	20	12
	% RSD	5.5%	5.2%	8.3%	39%	48%
Bag #2	Mean	580	1320	133	60	23
	Standard Dev	40	130	5	30	8
	% RSD	7.7%	10%	3.5%	47%	33%
Bag #3	Mean	620	1540	133	70	31
	Standard Dev	50	160	7	50	17
	% RSD	8.8%	11%	5.5%	73%	54%
Bag #4	Mean	640	1600	120	50	23
	Standard Dev	80	300	20	40	14
	% RSD	13%	20%	20%	83%	60%
Bag #5	Mean	640	1460	131	64	31
	Standard Dev	100	170	17	14	15
	% RSD	15%	12%	13%	22%	49%
Bag #6	Mean	690	1600	139	80	31
	Standard Dev	70	200	12	50	13
	% RSD	10%	13%	8.3%	63%	43%
Bag #7	Mean	640	1530	137	60	32
	Standard Dev	50	170	5	30	15
	% RSD	8.4%	11%	3.9%	54%	47%
Bag #8	Mean	630	1510	112	13	6
	Standard Dev	60	130	14	9	6
	% RSD	9.8%	8.4%	13%	71%	92%
Bag #9	Mean	690	1700	130	50	30
	Standard Dev	120	300	30	50	20
	% RSD	18%	15%	20%	94%	78%
Bag #10	Mean	740	1850	134	50	30
	Standard Dev	90	190	14	50	30
	% RSD	12%	10%	10%	94%	103%
Bag #11	Mean	570	1340	116	60	25
	Standard Dev	30	100	6	40	17
	% RSD	4.9%	7.3%	5.1%	74%	67%
Bag #12	Mean	594	1480	123	40	23
	Standard Dev	18	70	12	30	17
	% RSD	3.0%	4.5%	9.7%	75%	75%
Bag #13	Mean	604	1430	123	19	16
	Standard Dev	19	140	8	5	3
	% RSD	3.2%	10%	6.6%	25%	20%
Box	Mean	640	1500	129	50	26
	Standard Dev	70	200	15	30	16
	% RSD	12%	14%	11%	66%	61%

TABLE 17c (Continued)

		Ca	Ti	Cr	Mn	Fe
Bag #1	Mean	56	890	0.9	0.28	27.9
	Standard Dev	19	30	0.7	0.05	2.0
	% RSD	34%	3.6%	78%	18%	7.0%
Bag #2	Mean	31	840	0.3	0.231	25.8
	Standard Dev	3	30	0.2	0.005	1.0
	% RSD	9.0%	3.1%	74%	2.2%	3.9%
Bag #3	Mean	43	930	1.4	0.24	30
	Standard Dev	10	30	1.3	0.06	3
	% RSD	23%	2.7%	99%	25%	11%
Bag #4	Mean	40	970	0.16	0.27	30
	Standard Dev	30	50	0.15	0.06	4
	% RSD	70%	4.8%	94%	23%	13%
Bag #5	Mean	60	880	0.3	0.30	29
	Standard Dev	60	60	0.2	0.05	5
	% RSD	90%	6.9%	90%	15%	19%
Bag #6	Mean	70	910	0.27	0.28	31
	Standard Dev	70	90	0.07	0.08	3
	% RSD	96%	9.8%	24%	28%	11%
Bag #7	Mean	40	890	0.15	0.23	25
	Standard Dev	14	80	0.12	0.03	2
	% RSD	36%	8.9%	82%	12%	9.6%
Bag #8	Mean	47	870	0.15	0.21	25.6
	Standard Dev	19	70	0.13	0.04	1.9
	% RSD	40%	8.0%	87%	17%	7.4%
Bag #9	Mean	50	970	0.5	0.31	38
	Standard Dev	7	100	0.4	0.10	12
	% RSD	15%	11%	90%	32%	33%
Bag #10	Mean	53	910	0.41	0.34	40
	Standard Dev	17	100	0.10	0.05	10
	% RSD	32%	11%	24%	15%	24%
Bag #11	Mean	35	800	0.25	0.21	25
	Standard Dev	5	60	0.08	0.05	2
	% RSD	14%	7.0%	32%	22%	9.3%
Bag #12	Mean	35	840	0.3	0.198	25.4
	Standard Dev	10	90	0.3	0.015	0.9
	% RSD	28%	10%	79%	7.5%	3.5%
Bag #13	Mean	50	820	0.17	0.19	24.4
	Standard Dev	20	30	0.14	0.05	1.1
	% RSD	48%	4.2%	81%	24%	4.5%
Box	Mean	50	890	0.4	0.26	29
	Standard Dev	30	80	0.5	0.06	6
	% RSD	61%	8.7%	126%	25%	22%

TABLE 17c (Continued)

		Ni	Ti/Fe
Bag #1	Mean	0.41	31.9
	Standard Dev	0.03	2.0
	% RSD	7.4%	6.1%
Bag #2	Mean	0.28	32.5
	Standard Dev	0.05	0.6
	% RSD	17%	1.8%
Bag #3	Mean	0.6	31
	Standard Dev	0.7	3
	% RSD	118%	9.1%
Bag #4	Mean	0.22	32
	Standard Dev	0.07	4
	% RSD	33%	12%
Bag #5	Mean	0.25	31
	Standard Dev	0.06	5
	% RSD	24%	15%
Bag #6	Mean	0.28	30
	Standard Dev	0.06	3
	% RSD	22%	12%
Bag #7	Mean	0.24	35.0
	Standard Dev	0.03	1.5
	% RSD	13%	4.2%
Bag #8	Mean	0.17	34
	Standard Dev	0.06	3
	% RSD	37%	8.6%
Bag #9	Mean	0.4	28
	Standard Dev	0.2	9
	% RSD	62%	33%
Bag #10	Mean	0.25	24
	Standard Dev	0.08	5
	% RSD	34%	21%
Bag #11	Mean	0.19	32.0
	Standard Dev	0.03	0.8
	% RSD	16%	2.5%
Bag #12	Mean	0.19	33
	Standard Dev	0.03	3
	% RSD	17%	9.9%
Bag #13	Mean	0.23	33.8
	Standard Dev	0.03	1.8
	% RSD	12%	5.3%
Box	Mean	0.29	31
	Standard Dev	0.20	4
	% RSD	71%	14%

TABLE 18a: Descriptive statistics for each bag and box in a lot and for the entire lot. Excitation mode is Mo-K.

(1) Glad Lot B

		Al	Si	P	S	K
Bag #2	Mean	730	1600	108	15	12.3
	Standard Dev	100	300	4	3	1.7
	% RSD	13%	15%	3.9%	17%	14%
Bag #3	Mean	780	1960	120	18	6
	Standard Dev	60	140	7	5	2
	% RSD	7.2%	7.3%	5.7%	25%	34%
Bag #7	Mean	670	1480	118	13	11
	Standard Dev	50	140	5	3	4
	% RSD	7.7%	9.4%	4.6%	26%	38%
Box #1	Mean	730	1700	115	16	10
	Standard Dev	80	300	7	4	4
	% RSD	11%	16%	6.5%	26%	41%
Bag #3	Mean	590	1520	109	13	8
	Standard Dev	50	170	8	4	3
	% RSD	8.1%	11%	7.1%	33%	46%
Bag #5	Mean	640	1590	105	16.6	6.7
	Standard Dev	40	90	4	1.8	0.6
	% RSD	5.9%	5.7%	4.2%	11%	8.5%
Bag #7	Mean	620	1490	110	13	5.2
	Standard Dev	30	60	8	2	0.8
	% RSD	4.9%	3.8%	7.3%	18%	15%
Box #2	Mean	620	1540	108	14	7
	Standard Dev	40	120	6	3	2
	% RSD	7.2%	7.7%	5.7%	23%	33%
Bag #1	Mean	630	1510	109	20	6
	Standard Dev	20	60	7	3	2
	% RSD	3.4%	3.9%	6.1%	13%	34%
Bag #2	Mean	610	1510	109	22	8.2
	Standard Dev	50	80	7	3	0.6
	% RSD	7.7%	5.2%	6.8%	16%	6.8%
Bag #8	Mean	590	1480	108	17	5.7
	Standard Dev	30	60	5	4	1.0
	% RSD	5.3%	4.0%	5.0%	22%	17%
Box #3	Mean	610	1500	109	19	6.7
	Standard Dev	40	60	6	4	1.7
	% RSD	5.9%	4.1%	5.4%	18%	25%
Lot B	Mean	650	1580	111	17	8
	Standard Dev	80	190	7	4	3
	% RSD	12%	12%	6.6%	25%	40%

TABLE 18a: (Continued)

(1) Glad Lot B

		Ca	Ti	Cr	Mn	Fe
Bag #2	Mean	14.3	780	0.2	0.17	83
	Standard Dev	1.3	110	0.4	0.08	8
	% RSD	8.8%	14%	197%	47%	9.8%
Bag #3	Mean	16	800	0.05	0.21	86
	Standard Dev	4	50	0.06	0.07	3
	% RSD	22%	6.6%	119%	34%	3.5%
Bag #7	Mean	16.4	750	nd	0.18	81
	Standard Dev	1.0	60		0.03	5
	% RSD	6.1%	8.6%		17%	6.2%
Box #1	Mean	16	780	0.08	0.19	83
	Standard Dev	2	80	0.23	0.06	6
	% RSD	15%	9.7%	276%	33%	6.7%
Bag #3	Mean	13.4	728	0.13	0.14	77
	Standard Dev	1.2	13	0.11	0.04	2
	% RSD	9.2%	1.7%	83%	29%	2.9%
Bag #5	Mean	13.5	720	0.14	0.15	78
	Standard Dev	1.1	50	0.10	0.05	6
	% RSD	8.5%	7.2%	68%	34%	7.3%
Bag #7	Mean	13.2	680	0.05	0.13	74
	Standard Dev	0.5	50	0.03	0.04	5
	% RSD	3.7%	7.2%	53%	27%	7.1%
Box #2	Mean	13.4	720	0.12	0.14	77
	Standard Dev	1.0	40	0.09	0.04	4
	% RSD	7.4%	5.7%	76%	28%	5.5%
Bag #1	Mean	12.0	710	0.09	0.23	77
	Standard Dev	2.0	20	0.10	0.04	4
	% RSD	17%	3.0%	109%	19%	5.7%
Bag #2	Mean	11.1	710	0.16	0.21	79
	Standard Dev	1.1	50	0.10	0.03	4
	% RSD	9.9%	7.0%	64%	15%	4.7%
Bag #8	Mean	12.3	670	0.14	0.22	73.8
	Standard Dev	1.3	20	0.07	0.04	1.5
	% RSD	11%	3.1%	50%	17%	2.0%
Box #3	Mean	11.8	690	0.13	0.22	76
	Standard Dev	1.5	30	0.09	0.03	4
	% RSD	12%	5.0%	68%	16%	4.9%
Lot B	Mean	14	730	0.11	0.19	79
	Standard Dev	2	60	0.15	0.06	6
	% RSD	17%	8.7%	137%	30%	7.0%

TABLE 18a: (Continued)

(1) Glad Lot B

		Ni	Cu	Zn	Rb	Sr
Bag #2	Mean	0.38	0.25	40.1	0.006	0.45
	Standard Dev	0.17	0.09	1.1	0.005	0.05
	% RSD	45%	35%	2.9%	84%	11%
Bag #3	Mean	0.12	0.24	40	0.005	0.48
	Standard Dev	0.04	0.09	2	0.004	0.03
	% RSD	32%	39%	5.6%	79%	6.4%
Bag #7	Mean	0.07	0.5	44	0.001	0.43
	Standard Dev	0.03	0.3	2	0.001	0.03
	% RSD	42%	66%	4.7%	182%	6.1%
Box #1	Mean	0.17	0.3	41	0.004	0.46
	Standard Dev	0.16	0.2	3	0.004	0.04
	% RSD	93%	65%	6.3%	110%	8.6%
Bag #3	Mean	0.09	0.06	40	0.002	0.465
	Standard Dev	0.06	0.03	2	0.003	0.018
	% RSD	63%	62%	5.5%	200%	3.8%
Bag #5	Mean	0.08	0.04	39.2	0.002	0.46
	Standard Dev	0.04	0.05	1.6	0.002	0.02
	% RSD	45%	117%	4.1%	118%	4.6%
Bag #7	Mean	0.064	0.21	40.5	nd	0.451
	Standard Dev	0.017	0.10	1.6		0.015
	% RSD	26%	47%	4.0%		3.2%
Box #2	Mean	0.08	0.08	39.9	0.001	0.461
	Standard Dev	0.04	0.08	1.8	0.002	0.018
	% RSD	50%	103%	4.4%	169%	3.8%
Bag #1	Mean	0.38	0.03	38.8	0.004	0.46
	Standard Dev	0.09	0.04	0.7	0.009	0.02
	% RSD	24%	103%	1.7%	200%	5.1%
Bag #2	Mean	0.49	0.07	37	0.000	0.47
	Standard Dev	0.13	0.07	2	0.001	0.03
	% RSD	26%	95%	6.5%	200%	6.4%
Bag #8	Mean	0.43	0.002	36.4	0.001	0.48
	Standard Dev	0.08	0.004	1.2	0.003	0.03
	% RSD	20%	200%	3.2%	200%	5.2%
Box #3	Mean	0.43	0.04	37.4	0.002	0.47
	Standard Dev	0.10	0.05	1.8	0.005	0.03
	% RSD	24%	138%	4.8%	252%	5.4%
Lot B	Mean	0.24	0.15	40	0.002	0.46
	Standard Dev	0.19	0.19	3	0.004	0.03
	% RSD	79%	124%	6.7%	168%	6.3%

TABLE 18a: (Continued)

(1) Glad Lot B

		Zr	Pb	Ti/Fe
Bag #2	Mean	25	0.52	9.3
	Standard Dev	16	0.06	0.4
	% RSD	66%	12%	4.8%
Bag #3	Mean	2.2	0.53	9.3
	Standard Dev	2.7	0.03	0.3
	% RSD	126%	5.2%	3.7%
Bag #7	Mean	1.22	0.7	9.3
	Standard Dev	0.10	0.3	0.3
	% RSD	8.3%	39%	2.9%
Box #1	Mean	9	0.59	9.3
	Standard Dev	14	0.18	0.3
	% RSD	152%	30%	3.5%
Bag #3	Mean	0.19	0.39	9.44
	Standard Dev	0.09	0.05	0.14
	% RSD	47%	13%	1.5%
Bag #5	Mean	0.18	0.38	9.30
	Standard Dev	0.05	0.03	0.06
	% RSD	29%	6.8%	0.6%
Bag #7	Mean	0.14	0.385	9.130
	Standard Dev	0.04	0.002	0.009
	% RSD	28%	0.5%	0.1%
Box #2	Mean	0.18	0.39	9.32
	Standard Dev	0.06	0.04	0.15
	% RSD	36%	9.0%	1.6%
Bag #1	Mean	0.31	0.364	9.2
	Standard Dev	0.08	0.015	0.5
	% RSD	25%	4.0%	5.4%
Bag #2	Mean	0.28	0.38	9.0
	Standard Dev	0.10	0.03	0.7
	% RSD	36%	7.6%	8.0%
Bag #8	Mean	0.25	0.400	9.09
	Standard Dev	0.04	0.017	0.11
	% RSD	15%	4.2%	1.2%
Box #3	Mean	0.28	0.38	9.1
	Standard Dev	0.07	0.03	0.5
	% RSD	26%	6.5%	5.1%
Lot B	Mean	3	0.45	9.2
	Standard Dev	9	0.15	0.4
	% RSD	270%	32%	3.8%

TABLE 18a: (Continued)

(2) Glad Lot C

		Al	Si	P	S	K
Bag #3	Mean	1310	3070	124	2	6.8
	Standard Dev	30	140	4	3	1.9
	% RSD	2.3%	4.6%	3.3%	173%	29%
Bag #6	Mean	1170	2800	112	nd	4.3
	Standard Dev	180	400	19		1.9
	% RSD	15%	14%	17%		44%
Bag #9	Mean	1230	3200	111	6	8
	Standard Dev	60	300	9	5	3
	% RSD	5.0%	10%	8.2%	77%	35%
Box #1	Mean	1230	3000	115	3	6
	Standard Dev	120	300	13	4	3
	% RSD	9.7%	12%	11%	149%	42%
Bag #1	Mean	1200	2900	108	0.7	6
	Standard Dev	200	500	10	1.2	2
	% RSD	18%	18%	9.2%	169%	38%
Bag #6	Mean	1180	2900	112	7	6
	Standard Dev	100	300	11	8	5
	% RSD	8.2%	12%	10%	121%	80%
Bag #7	Mean	1140	2860	117	0.1	8
	Standard Dev	90	200	8	0.2	4
	% RSD	7.7%	6.9%	6.5%	200%	50%
Box #2	Mean	1180	2900	112	3	7
	Standard Dev	130	300	10	5	4
	% RSD	11%	12%	8.6%	213%	55%
Bag #4	Mean	1230	3000	97	40	22
	Standard Dev	110	200	20	7	2
	% RSD	9.1%	8.4%	20%	18%	9.5%
Bag #7	Mean	1140	2790	109	46	26
	Standard Dev	30	110	17	13	5
	% RSD	2.6%	3.8%	16%	28%	21%
Bag #8	Mean	1120	3010	104	41	25
	Standard Dev	30	130	6	5	2
	% RSD	2.4%	4.3%	5.4%	12%	8.7%
Box #3	Mean	1160	2920	103	42	24
	Standard Dev	80	180	15	9	4
	% RSD	7.0%	6.3%	14%	20%	15%
Lot C	Mean	1190	2900	110	16	12
	Standard Dev	110	300	13	20	9
	% RSD	9.6%	10%	12%	124%	72%

TABLE 18a: (Continued)

(2) Glad Lot C

		Ca	Ti	Cr	Mn	Fe
Bag #3	Mean	27	1650	0.58	0.35	98.4
	Standard Dev	3	50	0.14	0.06	1.1
	% RSD	10%	3.1%	23%	16%	1.1%
Bag #6	Mean	21	1520	1.1	0.31	93
	Standard Dev	4	170	0.8	0.05	12
	% RSD	19%	11%	75%	16%	14%
Bag #9	Mean	40	1690	0.9	0.30	103
	Standard Dev	20	100	0.4	0.05	7
	% RSD	51%	6.1%	43%	17%	7.1%
Box #1	Mean	30	1620	0.9	0.32	98
	Standard Dev	15	140	0.6	0.05	9
	% RSD	51%	8.4%	63%	16%	9.4%
Bag #1	Mean	32	1600	0.4	0.32	97
	Standard Dev	8	200	0.4	0.12	14
	% RSD	25%	13%	94%	37%	14%
Bag #6	Mean	60	1590	0.9	0.33	101
	Standard Dev	20	150	0.9	0.05	16
	% RSD	42%	9.6%	97%	14%	16%
Bag #7	Mean	38	1650	0.3	0.35	95
	Standard Dev	13	90	0.3	0.05	3
	% RSD	35%	5.4%	81%	13%	3.2%
Box #2	Mean	43	1620	0.6	0.34	98
	Standard Dev	19	150	0.6	0.07	11
	% RSD	45%	9.1%	105%	22%	12%
Bag #4	Mean	49	1590	0.9	0.38	96
	Standard Dev	17	40	0.2	0.05	2
	% RSD	34%	2.4%	26%	13%	2.5%
Bag #7	Mean	48	1550	0.87	0.31	93
	Standard Dev	17	60	0.19	0.04	3
	% RSD	35%	4.1%	22%	13%	3.5%
Bag #8	Mean	36	1680	0.6	0.32	97
	Standard Dev	7	130	0.4	0.06	4
	% RSD	18%	7.9%	57%	19%	4.2%
Box #3	Mean	44	1610	0.8	0.34	95
	Standard Dev	14	100	0.3	0.06	4
	% RSD	32%	6.1%	34%	16%	3.7%
Lot C	Mean	39	1620	0.7	0.33	97
	Standard Dev	17	120	0.5	0.06	9
	% RSD	44%	7.7%	67%	18%	8.8%

TABLE 18a: (Continued)

(2) Glad Lot C

		Ni	Cu	Zn	Rb	Sr
Bag #3	Mean	0.46	0.15	39.3	0.056	0.83
	Standard Dev	0.19	0.09	0.4	0.007	0.03
	% RSD	42%	59%	1.1%	12%	3.8%
Bag #6	Mean	0.38	0.07	36	0.048	0.80
	Standard Dev	0.20	0.04	5	0.018	0.15
	% RSD	51%	51%	13%	38%	19%
Bag #9	Mean	0.6	0.26	40	0.058	0.86
	Standard Dev	0.4	0.07	3	0.020	0.07
	% RSD	68%	26%	7.1%	34%	8.6%
Box #1	Mean	0.5	0.17	38	0.054	0.83
	Standard Dev	0.3	0.10	4	0.016	0.10
	% RSD	57%	61%	9.7%	30%	12%
Bag #1	Mean	0.5	0.22	39	0.045	0.84
	Standard Dev	0.5	0.04	3	0.017	0.06
	% RSD	98%	19%	7.2%	39%	7.2%
Bag #6	Mean	0.30	0.22	39.2	0.05	0.81
	Standard Dev	0.20	0.06	1.8	0.03	0.05
	% RSD	65%	26%	4.5%	53%	5.7%
Bag #7	Mean	0.23	0.27	38.9	0.042	0.77
	Standard Dev	0.09	0.07	0.6	0.013	0.07
	% RSD	38%	25%	1.5%	31%	8.5%
Box #2	Mean	0.3	0.23	39.0	0.046	0.81
	Standard Dev	0.3	0.06	1.8	0.019	0.06
	% RSD	85%	24%	4.5%	41%	7.6%
Bag #4	Mean	0.74	0.21	43	0.069	0.83
	Standard Dev	0.07	0.18	3	0.020	0.02
	% RSD	9.7%	88%	8.0%	30%	2.5%
Bag #7	Mean	0.67	0.23	40.5	0.063	0.90
	Standard Dev	0.10	0.04	1.7	0.016	0.08
	% RSD	15%	17%	4.1%	26%	8.6%
Bag #8	Mean	0.51	0.27	40.1	0.069	0.84
	Standard Dev	0.14	0.05	1.7	0.007	0.09
	% RSD	27%	18%	4.2%	11%	11%
Box #3	Mean	0.64	0.24	41	0.067	0.86
	Standard Dev	0.14	0.09	2	0.014	0.07
	% RSD	22%	39%	5.9%	22%	8.3%
Lot C	Mean	0.5	0.21	40	0.056	0.83
	Standard Dev	0.3	0.09	3	0.018	0.08
	% RSD	55%	41%	7.3%	33%	9.3%

TABLE 18a: (Continued)

(2) Glad Lot C

		Zr	Pb	Ti/Fe
Bag #3	Mean	21	0.6	16.8
	Standard Dev	12	0.2	0.4
	% RSD	58%	35%	2.1%
Bag #6	Mean	13	0.44	16.5
	Standard Dev	8	0.04	1.0
	% RSD	62%	9.4%	6.2%
Bag #9	Mean	30	0.56	16.5
	Standard Dev	20	0.08	0.4
	% RSD	72%	15%	2.5%
Box #1	Mean	22	0.52	16.6
	Standard Dev	17	0.13	0.6
	% RSD	76%	24%	3.9%
Bag #1	Mean	9	0.43	16.8
	Standard Dev	6	0.04	0.8
	% RSD	71%	10%	5.1%
Bag #6	Mean	8	0.449	15.9
	Standard Dev	6	0.016	1.8
	% RSD	71%	3.5%	11%
Bag #7	Mean	8.5	0.45	17.4
	Standard Dev	1.5	0.05	0.7
	% RSD	18%	11%	4.1%
Box #2	Mean	9	0.44	16.7
	Standard Dev	5	0.04	1.3
	% RSD	54%	8.2%	7.5%
Bag #4	Mean	38	0.44	16.6
	Standard Dev	3	0.02	0.6
	% RSD	6.7%	5.3%	3.8%
Bag #7	Mean	33	0.45	16.7
	Standard Dev	17	0.03	0.2
	% RSD	50%	7.5%	1.4%
Bag #8	Mean	27	0.50	17.4
	Standard Dev	15	0.09	0.7
	% RSD	54%	17%	3.9%
Box #3	Mean	33	0.46	16.9
	Standard Dev	13	0.06	0.6
	% RSD	38%	13%	3.5%
Lot C	Mean	21	0.47	16.7
	Standard Dev	16	0.09	0.9
	% RSD	74%	18%	5.2%

TABLE 18a: (Continued)

(3) Glad Lot D

		Al	Si	P	S	K
Bag #5	Mean	880	2130	90	nd	1.2
	Standard Dev	30	80	5		1.4
	% RSD	2.9%	4.0%	5.5%		120%
Bag #6	Mean	850	2010	83	nd	4
	Standard Dev	60	50	7		2
	% RSD	7.5%	2.7%	7.8%		58%
Bag #10	Mean	850	2120	91	nd	6
	Standard Dev	60	100	4		4
	% RSD	6.9%	4.8%	4.0%		68%
Box #1	Mean	860	2090	88	nd	4
	Standard Dev	50	100	6		3
	% RSD	5.8%	4.6%	6.6%		88%
Bag #2	Mean	900	2100	84.1	nd	5
	Standard Dev	120	300	1.5		3
	% RSD	14%	14%	1.8%		69%
Bag #4	Mean	810	2110	94	13	9
	Standard Dev	80	70	8	15	9
	% RSD	9.4%	3.5%	8.7%	117%	103%
Bag #9	Mean	980	2210	96	16	10
	Standard Dev	70	70	9	15	7
	% RSD	7.1%	3.0%	9.7%	90%	73%
Box #2	Mean	890	2140	92	10	8
	Standard Dev	110	160	8	13	7
	% RSD	12%	7.2%	9.1%	131%	88%
Bag #2	Mean	950	2300	89	nd	3.9
	Standard Dev	110	300	3		0.3
	% RSD	12%	14%	3.1%		7.4%
Bag #5	Mean	860	2160	90	10	11
	Standard Dev	130	170	12	13	8
	% RSD	15%	8.1%	14%	126%	72%
Bag #6	Mean	1000	2430	100	9	6
	Standard Dev	130	180	20	16	7
	% RSD	13%	7.2%	26%	173%	108%
Box #3	Mean	930	2300	92	8	8
	Standard Dev	130	200	15	12	6
	% RSD	14%	9.7%	16%	158%	85%
Lot D	Mean	890	2160	90	5	6
	Standard Dev	100	180	10	11	6
	% RSD	11%	8.1%	11%	193%	94%

TABLE 18a: (Continued)

(3) Glad Lot D

		Ca	Ti	Cr	Mn	Fe
Bag #5	Mean	25.3	1490	0.44	0.24	205
	Standard Dev	2.0	70	0.15	0.05	4
	% RSD	7.7%	4.9%	34%	21%	2.2%
Bag #6	Mean	24	1730	0.38	0.31	227
	Standard Dev	9	110	0.06	0.06	11
	% RSD	39%	6.7%	15%	18%	4.7%
Bag #10	Mean	26	1580	0.43	0.34	216
	Standard Dev	8	170	0.09	0.03	14
	% RSD	29%	11%	21%	8.3%	6.3%
Box #1	Mean	25	1600	0.42	0.30	216
	Standard Dev	7	150	0.10	0.06	13
	% RSD	27%	9.6%	24%	20%	6.2%
Bag #2	Mean	28	1530	0.6	0.25	190
	Standard Dev	3	110	0.2	0.08	30
	% RSD	11%	6.9%	39%	31%	17%
Bag #4	Mean	22	1580	0.6	0.34	214
	Standard Dev	6	170	0.2	0.08	16
	% RSD	29%	11%	42%	25%	7.7%
Bag #9	Mean	21	1610	0.40	0.266	218
	Standard Dev	3	170	0.17	0.018	14
	% RSD	15%	10%	43%	6.9%	6.6%
Box #2	Mean	23	1580	0.5	0.29	210
	Standard Dev	5	140	0.2	0.08	20
	% RSD	22%	8.8%	41%	26%	11%
Bag #2	Mean	23	1685	0.36	0.33	224.2
	Standard Dev	2	2	0.18	0.09	0.2
	% RSD	9.4%	0.1%	52%	26%	0.1%
Bag #5	Mean	32	1520	0.7	0.30	212
	Standard Dev	13	50	0.2	0.10	8
	% RSD	41%	3.3%	29%	32%	3.9%
Bag #6	Mean	28	1660	0.39	0.35	222
	Standard Dev	13	150	0.06	0.07	16
	% RSD	46%	9.3%	16%	21%	7.3%
Box #3	Mean	29	1600	0.5	0.32	218
	Standard Dev	11	120	0.2	0.08	11
	% RSD	38%	7.2%	43%	25%	5.2%
Lot D	Mean	26	1590	0.48	0.30	215
	Standard Dev	8	130	0.18	0.07	16
	% RSD	30%	8.5%	38%	23%	7.5%

TABLE 18a: (Continued)

(3) Glad Lot D

		Ni	Cu	Zn	Rb	Sr
Bag #5	Mean	1.5	0.15	30	0.004	0.62
	Standard Dev	0.3	0.02	5	0.004	0.04
	% RSD	19%	14%	17%	124%	6.9%
Bag #6	Mean	1.40	0.16	27.1	0.009	0.67
	Standard Dev	0.14	0.06	0.6	0.013	0.06
	% RSD	9.8%	34%	2.1%	143%	8.5%
Bag #10	Mean	1.8	0.20	26.5	0.013	0.66
	Standard Dev	0.3	0.05	1.1	0.007	0.04
	% RSD	19%	24%	4.3%	53%	6.1%
Box #1	Mean	1.6	0.17	28	0.008	0.65
	Standard Dev	0.3	0.05	3	0.009	0.05
	% RSD	18%	27%	11%	105%	7.6%
Bag #2	Mean	1.49	0.30	27.4	0.017	0.64
	Standard Dev	0.13	0.09	1.5	0.005	0.07
	% RSD	8.5%	30%	5.6%	32%	12%
Bag #4	Mean	1.8	0.17	27.4	0.023	0.600
	Standard Dev	0.7	0.05	1.2	0.009	0.008
	% RSD	38%	29%	4.5%	39%	1.4%
Bag #9	Mean	1.28	0.24	28.3	0.027	0.62
	Standard Dev	0.16	0.11	0.5	0.009	0.05
	% RSD	13%	43%	1.8%	34%	8.1%
Box #2	Mean	1.5	0.23	27.7	0.022	0.62
	Standard Dev	0.5	0.09	1.1	0.008	0.05
	% RSD	30%	40%	4.1%	38%	7.5%
Bag #2	Mean	1.4	0.28	27.4	0.021	0.620
	Standard Dev	1.4	0.10	1.3	0.005	0.003
	% RSD	100%	36%	4.6%	24%	0.6%
Bag #5	Mean	2.4	0.182	31	0.022	0.64
	Standard Dev	0.7	0.017	4	0.013	0.04
	% RSD	29%	9.5%	13%	59%	6.4%
Bag #6	Mean	1.38	0.123	29	0.012	0.68
	Standard Dev	0.10	0.004	4	0.007	0.06
	% RSD	7.2%	3.5%	15%	58%	9.1%
Box #3	Mean	1.9	0.18	29	0.019	0.65
	Standard Dev	0.7	0.07	4	0.010	0.05
	% RSD	37%	38%	12%	54%	7.5%
Lot D	Mean	1.6	0.19	28	0.016	0.64
	Standard Dev	0.5	0.07	3	0.011	0.05
	% RSD	30%	38%	9.9%	68%	7.6%

TABLE 18a: (Continued)

(3) Glad Lot D

		Zr	Pb	Ti/Fe
Bag #5	Mean	2.8	0.99	7.3
	Standard Dev	0.8	0.02	0.2
	% RSD	27%	2.1%	3.0%
Bag #6	Mean	3	1.021	7.60
	Standard Dev	2	0.015	0.16
	% RSD	87%	1.4%	2.1%
Bag #10	Mean	1.2	0.99	7.3
	Standard Dev	0.3	0.05	0.3
	% RSD	26%	4.9%	4.6%
Box #1	Mean	2.2	1.00	7.4
	Standard Dev	1.5	0.03	0.3
	% RSD	68%	3.2%	3.8%
Bag #2	Mean	18	0.99	8.0
	Standard Dev	8	0.06	0.9
	% RSD	46%	5.9%	12%
Bag #4	Mean	7	0.97	7.3
	Standard Dev	4	0.02	0.2
	% RSD	57%	2.1%	3.1%
Bag #9	Mean	5.1	1.00	7.4
	Standard Dev	0.8	0.04	0.3
	% RSD	16%	3.9%	3.7%
Box #2	Mean	10	0.98	7.6
	Standard Dev	8	0.04	0.6
	% RSD	78%	3.8%	7.3%
Bag #2	Mean	4.7	1.08	7.516
	Standard Dev	1.8	0.05	0.002
	% RSD	39%	5.0%	0.0%
Bag #5	Mean	8	1.05	7.18
	Standard Dev	4	0.09	0.06
	% RSD	45%	8.2%	0.9%
Bag #6	Mean	22	1.00	7.47
	Standard Dev	10	0.07	0.15
	% RSD	50%	6.7%	1.9%
Box #3	Mean	12	1.04	7.35
	Standard Dev	9	0.07	0.18
	% RSD	79%	7.2%	2.5%
Lot D	Mean	7	1.01	7.4
	Standard Dev	8	0.05	0.4
	% RSD	104%	5.3%	5.0%

TABLE 18b: Descriptive statistics for each bag and box in a lot and for the entire lot. Excitation mode is W-*brems*.

(1) Glad Lot B

		S	K	Ca	Ti	Cr
Bag #2	Mean	2	14	30	840	1.5
	Standard Dev	4	5	30	30	0.5
	% RSD	200%	37%	95%	3.8%	31%
Bag #3	Mean	6	5.2	15	890	1.8
	Standard Dev	7	1.8	4	90	1.0
	% RSD	123%	35%	30%	10%	59%
Bag #7	Mean	20	12	18.3	810	0.5
	Standard Dev	20	4	1.6	160	0.4
	% RSD	98%	35%	8.5%	20%	81%
Box #1	Mean	10	10	22	850	1.3
	Standard Dev	15	5	18	100	0.8
	% RSD	152%	51%	84%	12%	66%
Bag #3	Mean	9	7	14.8	860	1.7
	Standard Dev	10	4	1.3	40	0.6
	% RSD	118%	51%	8.9%	4.3%	36%
Bag #5	Mean	9	4.9	14.9	920	1.17
	Standard Dev	7	0.6	1.3	50	0.15
	% RSD	77%	11%	8.8%	5.6%	13%
Bag #7	Mean	2	3.2	13.8	880	1.1
	Standard Dev	3	0.8	1.6	30	0.2
	% RSD	141%	24%	12%	4.0%	21%
Box #2	Mean	7	5	14.6	890	1.4
	Standard Dev	8	3	1.3	50	0.5
	% RSD	102%	48%	8.8%	5.4%	35%
Bag #1	Mean	35	8	12.9	850	0.9
	Standard Dev	8	2	1.7	30	0.5
	% RSD	23%	28%	14%	3.5%	55%
Bag #2	Mean	30	11.4	13.1	860	0.99
	Standard Dev	20	0.9	1.1	30	0.20
	% RSD	68%	7.6%	8.6%	3.7%	20%
Bag #8	Mean	20	8.4	13.3	850	0.93
	Standard Dev	5	0.3	1.3	12	0.17
	% RSD	26%	4.1%	10%	1.4%	18%
Box #3	Mean	28	9.4	13.1	850	1.0
	Standard Dev	13	2.0	1.3	20	0.3
	% RSD	46%	21%	9.9%	2.8%	32%
Lot B	Mean	16	8	17	860	1.2
	Standard Dev	16	4	11	70	0.6
	% RSD	99%	48%	68%	8.0%	51%

TABLE 18b: (Continued)

(1) Glad Lot B

		Mn	Fe	Ni	Cu	Zn
Bag #2	Mean	0.32	90	0.5	0.29	42.6
	Standard Dev	0.12	3	0.3	0.08	1.4
	% RSD	38%	3.5%	61%	29%	3.2%
Bag #3	Mean	0.36	96	0.06	0.27	43
	Standard Dev	0.13	5	0.03	0.09	3
	% RSD	35%	5.5%	51%	32%	6.4%
Bag #7	Mean	0.13	86	0.07	0.5	46.4
	Standard Dev	0.14	12	0.05	0.3	1.8
	% RSD	104%	14%	66%	59%	3.8%
Box #1	Mean	0.27	91	0.2	0.34	44
	Standard Dev	0.16	8	0.2	0.18	3
	% RSD	58%	9.0%	124%	52%	5.8%
Bag #3	Mean	0.20	90	0.08	0.04	43
	Standard Dev	0.06	3	0.05	0.04	3
	% RSD	32%	3.3%	63%	116%	5.8%
Bag #5	Mean	0.15	95	0.07	0.05	42.2
	Standard Dev	0.07	5	0.05	0.04	1.3
	% RSD	46%	4.9%	68%	81%	3.1%
Bag #7	Mean	0.16	91	0.063	0.22	42.9
	Standard Dev	0.07	6	0.012	0.15	1.2
	% RSD	41%	6.8%	19%	71%	2.9%
Box #2	Mean	0.17	92	0.07	0.08	42.8
	Standard Dev	0.06	4	0.04	0.10	1.8
	% RSD	37%	4.8%	56%	123%	4.2%
Bag #1	Mean	0.22	90	0.38	0.03	41.6
	Standard Dev	0.11	5	0.07	0.04	0.6
	% RSD	49%	5.1%	19%	116%	1.4%
Bag #2	Mean	0.20	90	0.49	0.06	40
	Standard Dev	0.05	5	0.18	0.11	2
	% RSD	25%	5.6%	37%	176%	6.0%
Bag #8	Mean	0.26	87.8	0.41	nd	39.7
	Standard Dev	0.10	0.9	0.09		1.3
	% RSD	40%	1.0%	23%		3.2%
Box #3	Mean	0.23	89	0.42	0.03	40.4
	Standard Dev	0.09	4	0.12	0.06	1.7
	% RSD	38%	4.2%	29%	212%	4.2%
Lot B	Mean	0.23	91	0.2	0.15	42
	Standard Dev	0.12	6	0.2	0.18	3
	% RSD	51%	6.4%	90%	120%	6.0%

TABLE 18b: (Continued)

(1) Glad Lot B

		Rb	Sr	Y	Zr	Pb
Bag #2	Mean	0.005	0.46	0.07	22	0.48
	Standard Dev	0.006	0.06	0.02	12	0.04
	% RSD	106%	12%	33%	55%	7.5%
Bag #3	Mean	0.001	0.46	0.021	2	0.49
	Standard Dev	0.001	0.04	0.005	3	0.03
	% RSD	200%	9.2%	25%	123%	5.2%
Bag #7	Mean	0.009	0.43	0.026	1.2	0.6
	Standard Dev	0.011	0.03	0.012	0.2	0.2
	% RSD	117%	6.0%	48%	19%	37%
Box #1	Mean	0.005	0.45	0.04	8	0.54
	Standard Dev	0.007	0.04	0.03	12	0.15
	% RSD	144%	9.4%	70%	141%	28%
Bag #3	Mean	0.009	0.45	0.007	0.25	0.47
	Standard Dev	0.017	0.04	0.012	0.10	0.05
	% RSD	179%	8.3%	164%	38%	10%
Bag #5	Mean	0.003	0.47	0.015	0.23	0.47
	Standard Dev	0.006	0.04	0.004	0.06	0.03
	% RSD	200%	9.0%	25%	25%	5.2%
Bag #7	Mean	0.003	0.443	0.023	0.22	0.432
	Standard Dev	0.004	0.007	0.007	0.04	0.012
	% RSD	141%	1.7%	32%	16%	2.9%
Box #2	Mean	0.006	0.46	0.014	0.24	0.46
	Standard Dev	0.011	0.04	0.010	0.07	0.04
	% RSD	196%	7.6%	72%	28%	7.7%
Bag #1	Mean	0.008	0.43	0.022	0.33	0.44
	Standard Dev	0.007	0.02	0.009	0.05	0.04
	% RSD	90%	4.4%	40%	15%	8.0%
Bag #2	Mean	nd	0.445	0.016	0.30	0.44
	Standard Dev		0.006	0.006	0.08	0.03
	% RSD		1.4%	35%	25%	6.6%
Bag #8	Mean	0.012	0.455	0.004	0.29	0.43
	Standard Dev	0.014	0.013	0.005	0.04	0.03
	% RSD	120%	2.8%	120%	14%	7.3%
Box #3	Mean	0.006	0.443	0.014	0.31	0.43
	Standard Dev	0.010	0.017	0.010	0.05	0.03
	% RSD	148%	3.8%	70%	18%	6.7%
Lot B	Mean	0.006	0.45	0.023	3	0.48
	Standard Dev	0.009	0.03	0.022	8	0.10
	% RSD	158%	7.2%	94%	251%	21%

TABLE 18b: (Continued)

(1) Glad Lot B

		Ba	Sn	Ag	Ti/Fe
Bag #2	Mean	22.6	0.8	0.031	9.33
	Standard Dev	1.0	1.0	0.019	0.14
	% RSD	4.4%	129%	62%	1.5%
Bag #3	Mean	23.7	0.24	0.014	9.3
	Standard Dev	1.2	0.07	0.016	0.7
	% RSD	5.3%	30%	111%	7.1%
Bag #7	Mean	21.6	0.21	0.024	9.3
	Standard Dev	1.1	0.05	0.003	0.6
	% RSD	5.2%	22%	12%	6.7%
Box #1	Mean	22.6	0.4	0.023	9.3
	Standard Dev	1.4	0.6	0.015	0.5
	% RSD	6.1%	148%	64%	5.2%
Bag #3	Mean	21.1	0.16	0.038	9.6
	Standard Dev	0.8	0.03	0.003	0.4
	% RSD	3.8%	21%	7.0%	4.3%
Bag #5	Mean	22.0	0.186	0.03	9.76
	Standard Dev	1.1	0.011	0.02	0.13
	% RSD	4.8%	5.8%	81%	1.4%
Bag #7	Mean	21.5	0.26	0.038	9.7
	Standard Dev	1.1	0.11	0.014	0.3
	% RSD	5.2%	43%	36%	2.8%
Box #2	Mean	21.5	0.19	0.034	9.7
	Standard Dev	1.0	0.06	0.015	0.3
	% RSD	4.4%	31%	44%	2.8%
Bag #1	Mean	19.7	0.19	0.035	9.6
	Standard Dev	0.6	0.05	0.018	0.6
	% RSD	2.9%	26%	52%	6.1%
Bag #2	Mean	19.9	0.137	0.011	9.6
	Standard Dev	0.2	0.005	0.019	0.6
	% RSD	1.1%	3.6%	180%	5.9%
Bag #8	Mean	19.5	0.17	0.023	9.70
	Standard Dev	0.9	0.02	0.016	0.07
	% RSD	4.4%	14%	70%	0.8%
Box #3	Mean	19.7	0.17	0.023	9.6
	Standard Dev	0.6	0.04	0.019	0.4
	% RSD	2.9%	23%	84%	4.5%
Lot B	Mean	21.3	0.3	0.026	9.5
	Standard Dev	1.6	0.4	0.017	0.4
	% RSD	7.6%	143%	64%	4.5%

TABLE 18b: (Continued)

(2) Glad Lot C

		S	K	Ca	Ti	Cr
Bag #3	Mean	nd	5.0	20	2000	5.0
	Standard Dev		1.6	4	50	0.7
	% RSD		32%	18%	2.4%	13%
Bag #6	Mean	nd	4.1	17	1800	5.2
	Standard Dev		1.8	4	300	1.8
	% RSD		44%	23%	14%	34%
Bag #9	Mean	nd	6	30	2070	5.4
	Standard Dev		2	20	120	0.5
	% RSD		38%	57%	5.9%	9.5%
Box #1	Mean	nd	4.9	24	2000	5.2
	Standard Dev		1.8	14	200	1.1
	% RSD		37%	57%	10%	20%
Bag #1	Mean	nd	6	29	2000	4.4
	Standard Dev		3	8	200	0.8
	% RSD		42%	27%	11%	18%
Bag #6	Mean	nd	5	50	1870	4.9
	Standard Dev		4	30	80	1.9
	% RSD		83%	50%	4.2%	38%
Bag #7	Mean	nd	9	33	1970	4.6
	Standard Dev		5	14	90	0.5
	% RSD		58%	42%	4.4%	11%
Box #2	Mean	nd	7	40	1950	4.6
	Standard Dev		4	20	140	1.1
	% RSD		60%	52%	7.4%	24%
Bag #4	Mean	nd	5	32	1860	3.9
	Standard Dev		3	9	180	0.7
	% RSD		63%	30%	9.5%	18%
Bag #7	Mean	0.19	8	32	1880	4.9
	Standard Dev	0.38	6	17	100	0.7
	% RSD	200%	69%	53%	5.3%	15%
Bag #8	Mean	nd	9	22	2010	4.4
	Standard Dev		3	6	110	0.5
	% RSD		31%	30%	5.4%	11%
Box #3	Mean	0.06	7	29	1920	4.4
	Standard Dev	0.22	4	12	140	0.7
	% RSD	346%	55%	42%	7.2%	16%
Lot C	Mean	0.02	6	31	1940	4.7
	Standard Dev	0.13	4	16	160	1.0
	% RSD	592%	56%	54%	8.1%	21%

TABLE 18b: (Continued)

(2) Glad Lot C

		Mn	Fe	Ni	Cu	Zn
Bag #3	Mean	0.14	113	0.40	0.24	43.5
	Standard Dev	0.23	2	0.16	0.05	0.2
	% RSD	165%	1.8%	41%	20%	0.5%
Bag #6	Mean	0.06	104	3	0.3	39
	Standard Dev	0.13	16	5	0.4	6
	% RSD	200%	15%	173%	140%	14%
Bag #9	Mean	nd	118	0.5	0.31	45
	Standard Dev		7	0.3	0.05	3
	% RSD		5.6%	61%	16%	6.4%
Box #1	Mean	0.06	112	1.4	0.3	42
	Standard Dev	0.14	11	3.2	0.2	4
	% RSD	225%	10%	226%	85%	10%
Bag #1	Mean	0.16	114	0.4	0.25	43
	Standard Dev	0.12	12	0.4	0.02	3
	% RSD	78%	10%	101%	9.3%	7.0%
Bag #6	Mean	0.09	112	0.28	0.24	43.3
	Standard Dev	0.13	9	0.12	0.07	1.7
	% RSD	148%	8.3%	42%	30%	3.9%
Bag #7	Mean	0.03	110	0.21	0.40	42.3
	Standard Dev	0.07	2	0.06	0.06	0.5
	% RSD	200%	2.2%	28%	16%	1.3%
Box #2	Mean	0.09	112	0.3	0.30	42.9
	Standard Dev	0.11	8	0.3	0.09	1.9
	% RSD	122%	7.3%	85%	31%	4.4%
Bag #4	Mean	0.009	103	0.36	0.2	43
	Standard Dev	1.017	11	0.08	0.3	3
	% RSD	200%	11%	23%	111%	6.8%
Bag #7	Mean	0.08	107	0.39	0.11	44
	Standard Dev	0.09	6	0.12	0.08	2
	% RSD	120%	5.9%	31%	71%	5.3%
Bag #8	Mean	0.018	111	0.25	0.18	43
	Standard Dev	0.035	4	0.13	0.04	2
	% RSD	200%	3.5%	50%	21%	4.8%
Box #3	Mean	0.04	107	0.33	0.18	43
	Standard Dev	0.06	8	0.12	0.16	2
	% RSD	178%	7.2%	36%	89%	5.2%
Lot C	Mean	0.06	110	0.7	0.25	43
	Standard Dev	0.11	9	1.8	0.18	3
	% RSD	170%	8.3%	272%	70%	6.7%

TABLE 18b: (Continued)

(2) Glad Lot C

		Rb	Sr	Y	Zr	Pb
Bag #3	Mean	0.048	0.74	0.087	19	0.68
	Standard Dev	0.013	0.02	0.016	11	0.16
	% RSD	27%	2.6%	19%	57%	24%
Bag #6	Mean	0.032	0.71	0.08	12	0.54
	Standard Dev	0.004	0.15	0.02	7	0.06
	% RSD	13%	20%	26%	62%	10%
Bag #9	Mean	0.052	0.764	0.14	29	0.61
	Standard Dev	0.012	0.015	0.06	19	0.11
	% RSD	22%	2.0%	44%	66%	18%
Box #1	Mean	0.044	0.74	0.10	19	0.60
	Standard Dev	0.013	0.08	0.05	14	0.11
	% RSD	29%	11%	46%	72%	19%
Bag #1	Mean	0.05	0.76	0.06	9	0.52
	Standard Dev	0.02	0.05	0.02	6	0.03
	% RSD	46%	6.5%	34%	65%	5.3%
Bag #6	Mean	0.038	0.74	0.08	8	0.52
	Standard Dev	0.019	0.06	0.02	6	0.07
	% RSD	51%	7.8%	30%	78%	13%
Bag #7	Mean	0.046	0.70	0.07	8.4	0.53
	Standard Dev	0.015	0.05	0.02	1.4	0.05
	% RSD	32%	7.5%	34%	16%	9.0%
Box #2	Mean	0.046	0.74	0.07	9	0.52
	Standard Dev	0.019	0.06	0.02	5	0.05
	% RSD	42%	7.6%	31%	54%	8.9%
Bag #4	Mean	0.047	0.69	0.106	35	0.43
	Standard Dev	0.011	0.08	0.010	4	0.10
	% RSD	24%	12%	9.2%	11%	23%
Bag #7	Mean	0.06	0.82	0.11	31	0.54
	Standard Dev	0.02	0.06	0.03	15	0.07
	% RSD	38%	6.8%	28%	50%	13%
Bag #8	Mean	0.054	0.75	0.13	26	0.56
	Standard Dev	0.010	0.05	0.06	13	0.13
	% RSD	18%	7.2%	46%	51%	24%
Box #3	Mean	0.052	0.75	0.12	30	0.51
	Standard Dev	0.014	0.08	0.04	11	0.11
	% RSD	27%	11%	32%	38%	22%
Lot C	Mean	0.047	0.74	0.10	20	0.54
	Standard Dev	0.016	0.07	0.04	14	0.10
	% RSD	33%	9.7%	42%	71%	19%

TABLE 18b: (Continued)

(2) Glad Lot C

		Ba	Sn	Ag	Ti/Fe
Bag #3	Mean	21.3	0.21	0.05	17.64
	Standard Dev	1.0	0.08	0.03	0.11
	% RSD	4.6%	38%	58%	0.6%
Bag #6	Mean	19	0.14	0.019	17.3
	Standard Dev	3	0.03	0.024	0.9
	% RSD	17%	19%	128%	5.4%
Bag #9	Mean	21.3	0.20	0.062	17.6
	Standard Dev	1.4	0.03	0.018	0.2
	% RSD	6.7%	14%	29%	1.2%
Box #1	Mean	21	0.18	0.04	17.5
	Standard Dev	2	0.05	0.03	0.6
	% RSD	11%	30%	67%	3.2%
Bag #1	Mean	22.0	0.21	0.037	17.7
	Standard Dev	1.9	0.06	0.015	0.3
	% RSD	8.6%	31%	41%	1.9%
Bag #6	Mean	20.2	0.20	0.042	16.8
	Standard Dev	1.1	0.02	0.013	1.8
	% RSD	5.3%	11%	31%	11%
Bag #7	Mean	20.05	0.235	0.04	17.9
	Standard Dev	0.16	0.018	0.03	0.5
	% RSD	0.8%	7.5%	72%	2.8%
Box #2	Mean	20.71	0.21	0.038	17.5
	Standard Dev	1.5	0.04	0.017	1.1
	% RSD	7.1%	19%	45%	6.4%
Bag #4	Mean	20.7	0.15	0.05	18.0
	Standard Dev	1.6	0.02	0.03	0.3
	% RSD	7.5%	14%	60%	1.9%
Bag #7	Mean	20.1	0.154	0.11	17.6
	Standard Dev	0.9	0.016	0.11	0.2
	% RSD	4.3%	10%	96%	1.0%
Bag #8	Mean	21.0	0.15	0.049	18.2
	Standard Dev	0.5	0.02	0.011	0.3
	% RSD	2.5%	15%	23%	1.9%
Box #3	Mean	20.6	0.152	0.07	17.9
	Standard Dev	1.0	0.018	0.07	0.4
	% RSD	5.0%	12%	95%	2.0%
Lot C	Mean	20.6	0.18	0.05	17.6
	Standard Dev	1.6	0.05	0.04	0.8
	% RSD	7.8%	25%	87	4.3

TABLE 18b: (Continued)

(3) Glad Lot D

		S	K	Ca	Ti	Cr
Bag #5	Mean	nd	1.6	30	1910	2.8
	Standard Dev		1.8	20	100	0.8
	% RSD		110%	66%	5.2%	29%
Bag #6	Mean	nd	4	24	1960	3.20
	Standard Dev		3	12	100	0.12
	% RSD		61%	49%	5.0%	3.8%
Bag #10	Mean	nd	5	23	1940	3.8
	Standard Dev		4	9	100	0.6
	% RSD		74%	37%	5.1%	17%
Box #1	Mean	nd	4	27	1930	3.3
	Standard Dev		3	14	90	0.7
	% RSD		85%	53%	4.7%	22%
Bag #2	Mean	nd	6	25	1940	3.3
	Standard Dev		3	3	50	0.4
	% RSD		55%	11%	2.8%	13%
Bag #4	Mean	20	10	18	1920	3.8
	Standard Dev	20	8	7	90	0.3
	% RSD	119%	83%	38%	4.6%	8.7%
Bag #9	Mean	13	10	17	1980	4.0
	Standard Dev	15	7	5	40	0.6
	% RSD	116%	74%	31%	1.8%	15%
Box #2	Mean	12	9	20	1950	3.7
	Standard Dev	17	7	6	60	0.5
	% RSD	147%	74%	30%	3.2%	14%
Bag #2	Mean	nd	4	15	1760	3.4
	Standard Dev		2	3	150	0.7
	% RSD		56%	21%	8.5%	20%
Bag #3	Mean	6	10	28	1880	4.0
	Standard Dev	12	7	16	120	0.4
	% RSD	192%	67%	57%	6.2%	9.0%
Bag #6	Mean	7	9	21	1900	3.5
	Standard Dev	15	9	12	200	0.4
	% RSD	200%	100%	56%	11%	12%
Box #3	Mean	5	8	22	1860	3.7
	Standard Dev	11	7	12	160	0.5
	% RSD	220%	86%	55%	8.7%	14%
Lot D	Mean	6	7	23	1920	3.6
	Standard Dev	13	6	11	120	0.6
	% RSD	223%	88%	50%	6.1%	17%

TABLE 18b: (Continued)

(3) Glad Lot D

		Mn	Fe	Ni	Cu	Zn
Bag #5	Mean	0.15	253	1.6	0.13	32
	Standard Dev	0.10	6	0.3	0.06	6
	% RSD	69%	2.5%	21%	47%	18%
Bag #6	Mean	0.37	260	1.35	0.18	29.6
	Standard Dev	0.12	12	0.13	0.04	1.2
	% RSD	30%	4.7%	9.4%	21%	4.2%
Bag #10	Mean	0.15	264	1.7	0.18	29.0
	Standard Dev	0.04	12	0.4	0.09	0.6
	% RSD	29%	4.7%	22%	49%	1.9%
Box #1	Mean	0.22	259	1.6	0.16	30
	Standard Dev	0.14	11	0.3	0.07	4
	% RSD	64%	4.1%	20%	41%	12%
Bag #2	Mean	0.21	256	1.34	0.23	29.8
	Standard Dev	0.19	10	0.11	0.09	1.7
	% RSD	91%	3.8%	8.3%	42%	5.7%
Bag #4	Mean	0.21	259	1.7	0.14	30.2
	Standard Dev	0.09	9	0.6	0.10	1.7
	% RSD	42%	3.4%	35%	71%	5.6%
Bag #9	Mean	0.23	268	1.22	0.27	31.3
	Standard Dev	0.16	4	0.09	0.06	0.5
	% RSD	67%	1.5%	7.6%	24%	1.7%
Box #2	Mean	0.22	261	1.4	0.21	30.5
	Standard Dev	0.13	9	0.4	0.10	1.4
	% RSD	60%	3.3%	29%	46%	4.6%
Bag #2	Mean	0.31	238	3	0.18	27
	Standard Dev	0.12	24	3	0.10	2
	% RSD	41%	10%	104%	56%	7.6%
Bag #3	Mean	0.22	254	2.2	0.10	32
	Standard Dev	0.06	15	0.8	0.07	6
	% RSD	27%	5.9%	38%	76%	20%
Bag #6	Mean	0.24	250	1.3	0.08	30
	Standard Dev	0.12	30	0.2	0.08	5
	% RSD	49%	10%	17%	100%	17%
Box #3	Mean	0.25	250	2.0	0.11	30
	Standard Dev	0.10	20	1.5	0.09	5
	% RSD	39%	8.4%	75%	75%	17%
Lot D	Mean	0.23	256	1.6	0.16	30
	Standard Dev	0.12	15	0.9	0.09	4
	% RSD	53%	5.8%	56%	56%	12%

TABLE 18b: (Continued)

(3) Glad Lot D

		Rb	Sr	Y	Zr	Pb
Bag #5	Mean	0.008	0.59	0.027	2.5	1.05
	Standard Dev	0.012	0.02	0.014	0.9	0.07
	% RSD	143%	3.6%	51%	38%	6.7%
Bag #6	Mean	0.028	0.62	0.033	3	1.09
	Standard Dev	0.013	0.06	0.011	2	0.05
	% RSD	46%	9.6%	32%	72%	5.0%
Bag #10	Mean	0.008	0.61	0.031	1.4	1.114
	Standard Dev	0.013	0.06	0.007	0.3	0.017
	% RSD	175%	9.8%	22%	23%	1.5%
Box #1	Mean	0.013	0.61	0.030	2.3	1.08
	Standard Dev	0.015	0.05	0.010	1.5	0.06
	% RSD	110%	7.4%	33%	63%	5.1%
Bag #2	Mean	0.017	0.57	0.057	16	1.12
	Standard Dev	0.007	0.06	0.015	8	0.07
	% RSD	40%	10%	27%	48%	6.0%
Bag #4	Mean	0.022	0.56	0.04	6	1.04
	Standard Dev	0.011	0.02	0.02	4	0.03
	% RSD	47%	4.2%	69%	55%	3.0%
Bag #9	Mean	0.028	0.60	0.05	4.7	1.09
	Standard Dev	0.017	0.03	0.02	0.9	0.04
	% RSD	62%	5.6%	42%	19%	3.3%
Box #2	Mean	0.023	0.58	0.05	9	1.08
	Standard Dev	0.012	0.04	0.02	7	0.05
	% RSD	54%	6.6%	45%	77%	4.8%
Bag #2	Mean	0.013	0.51	0.040	4.4	0.99
	Standard Dev	0.013	0.05	0.003	0.9	0.04
	% RSD	95%	11%	8.5%	20%	4.3%
Bag #3	Mean	0.02	0.56	0.032	8	1.13
	Standard Dev	0.02	0.06	0.010	4	0.05
	% RSD	109%	11%	32%	45%	4.4%
Bag #6	Mean	0.017	0.57	0.06	18	1.12
	Standard Dev	0.010	0.05	0.02	6	0.13
	% RSD	56%	7.9%	37%	36%	12%
Box #3	Mean	0.017	0.55	0.043	11	1.09
	Standard Dev	0.014	0.06	0.017	7	0.10
	% RSD	82%	10%	39%	67%	9.2%
Lot D	Mean	0.018	0.58	0.040	7	1.08
	Standard Dev	0.014	0.05	0.017	7	0.07
	% RSD	78%	8.8%	44%	92%	6.5%

TABLE 18b: (Continued)

(3) Glad Lot D

		Ba	Sn	Ag	Ti/Fe
Bag #5	Mean	18.3	0.51	0.044	7.5
	Standard Dev	1.6	0.04	0.003	0.2
	% RSD	9.0%	7.8%	7.1%	2.9%
Bag #6	Mean	18.2	0.52	0.02	7.54
	Standard Dev	0.8	0.05	0.02	0.04
	% RSD	4.3%	9.3%	91%	0.5%
Bag #10	Mean	18.7	2	0.03	7.36
	Standard Dev	0.2	2	0.03	0.14
	% RSD	1.1%	115%	90%	1.9%
Box #1	Mean	18.4	1.1	0.03	7.47
	Standard Dev	1.0	1.6	0.02	0.17
	% RSD	5.4%	143%	60%	2.3%
Bag #2	Mean	17.6	0.56	0.057	7.56
	Standard Dev	0.6	0.02	0.013	0.09
	% RSD	3.3%	3.9%	22%	1.2%
Bag #4	Mean	18.5	0.56	0.04	7.42
	Standard Dev	0.7	0.03	0.03	0.12
	% RSD	4.0%	4.8%	62%	1.6%
Bag #9	Mean	18.1	0.65	0.042	7.38
	Standard Dev	0.4	0.15	0.016	0.10
	% RSD	2.4%	23%	38%	1.3%
Box #2	Mean	18.1	0.59	0.046	7.45
	Standard Dev	0.6	0.09	0.019	0.12
	% RSD	3.5%	16%	41%	1.6%
Bag #2	Mean	17.1	0.51	0.032	7.42
	Standard Dev	1.5	0.05	0.009	0.12
	% RSD	8.5%	10%	29%	1.7%
Bag #3	Mean	18.0	0.56	0.05	7.42
	Standard Dev	1.8	0.03	0.02	0.05
	% RSD	10%	6.1%	48%	0.7%
Bag #6	Mean	19	0.58	0.070	7.53
	Standard Dev	3	0.10	0.009	0.10
	% RSD	15%	16%	13%	1.3%
Box #3	Mean	18	0.55	0.05	7.46
	Standard Dev	2	0.07	0.02	0.10
	% RSD	11%	12%	42%	1.3%
Lot D	Mean	18.2	0.7	0.04	7.46
	Standard Dev	1.3	0.9	0.02	0.13
	% RSD	7.3%	122%	48%	1.7%

TABLE 18c: Descriptive statistics for each bag and box in a lot and for the entire lot. Excitation mode is W-L.

(1) Glad Lot B

		Al	Si	P	S	K
Bag #2	Mean	800	1800	104	42	29
	Standard Dev	90	200	14	10	4
	% RSD	12%	13%	13%	24%	13%
Bag #3	Mean	900	2500	128	56	22
	Standard Dev	100	600	13	9	3
	% RSD	11%	23%	9.8%	15%	15%
Bag #7	Mean	700	1500	100	40	18
	Standard Dev	200	500	40	20	7
	% RSD	32%	31%	36%	53%	38%
Box #1	Mean	800	1900	110	45	23
	Standard Dev	170	600	20	15	6
	% RSD	21%	31%	22%	33%	27%
Bag #3	Mean	600	1600	107	35	23
	Standard Dev	80	300	11	10	11
	% RSD	12%	19%	10%	28%	47%
Bag #5	Mean	700	1720	111	43	21
	Standard Dev	40	120	7	10	4
	% RSD	6.6%	6.8%	6.0%	23%	18%
Bag #7	Mean	600	1470	96	33	15
	Standard Dev	60	140	11	7	2
	% RSD	9.8%	9.8%	11%	21%	13%
Box #2	Mean	640	1600	106	37	21
	Standard Dev	60	200	10	9	7
	% RSD	9.8%	14%	9.4%	25%	35%
Bag #1	Mean	700	1711	125	105	37
	Standard Dev	30	18	8	7	7
	% RSD	5.0%	1.0%	6.7%	6.4%	19%
Bag #2	Mean	720	1710	135	124	45
	Standard Dev	30	70	14	4	3
	% RSD	4.5%	3.8%	11%	2.9%	6.6%
Bag #8	Mean	678	1700	130	106	38
	Standard Dev	18	150	11	7	4
	% RSD	2.6%	8.5%	8.8%	6.5%	11%
Box #3	Mean	700	1710	130	112	40
	Standard Dev	30	80	11	10	6
	% RSD	4.7%	4.9%	8.7%	9.3%	15%
Lot B	Mean	710	1800	116	70	28
	Standard Dev	120	400	20	40	11
	% RSD	17%	22%	17%	54%	38%

TABLE 18c: (Continued)

(1) Glad Lot B

		Ca	Ti	Cr	Mn	Fe
Bag #2	Mean	50	780	0.6	0.3	111
	Standard Dev	50	110	0.7	0.1	3
	% RSD	100%	14%	115%	35%	2.6%
Bag #3	Mean	30	800	0.7	0.29	125
	Standard Dev	6	50	0.1	0.04	6
	% RSD	20%	6.6%	16%	12%	4.4%
Bag #7	Mean	20	750	0.04	0.26	110
	Standard Dev	11	60	0.03	0.09	30
	% RSD	45%	8.6%	75%	36%	24%
Box #1	Mean	30	780	0.5	0.29	114
	Standard Dev	30	80	0.5	0.08	16
	% RSD	82%	9.7%	105%	28%	14%
Bag #3	Mean	23	728	0.76	0.23	101
	Standard Dev	4	13	0.19	0.04	5
	% RSD	18%	1.7%	25%	19%	4.7%
Bag #5	Mean	25	720	0.9	0.28	108
	Standard Dev	3	50	0.2	0.04	11
	% RSD	11%	7.2%	27%	15%	9.9%
Bag #7	Mean	21.4	680	0.60	0.202	94
	Standard Dev	0.5	50	0.15	0.017	9
	% RSD	2.3%	7.2%	25%	8.6%	9.3%
Box #2	Mean	24	720	0.8	0.24	102
	Standard Dev	3	40	0.2	0.05	9
	% RSD	14%	5.7%	28%	19%	9.2%
Bag #1	Mean	39	710	1.7	0.39	115
	Standard Dev	7	20	0.3	0.02	9
	% RSD	18%	3.0%	16%	5.0%	7.9%
Bag #2	Mean	43	710	2.2	0.51	126
	Standard Dev	2	50	0.4	0.04	8
	% RSD	4.8%	7.0%	19%	7.3%	6.1%
Bag #8	Mean	43.5	670	1.8	0.44	112
	Standard Dev	1.9	20	0.3	0.11	5
	% RSD	4.4%	3.1%	17%	24%	4.2%
Box #3	Mean	42	690	1.9	0.45	118
	Standard Dev	4	30	0.4	0.08	9
	% RSD	11%	5.0%	21%	18%	7.9%
Lot B	Mean	34	730	1.1	0.33	112
	Standard Dev	18	60	0.7	0.11	13
	% RSD	54%	8.7%	70%	34%	12%

TABLE 18c: (Continued)

(1) Glad Lot B

		Ni	Ti/Fe
Bag #2	Mean	0.7	7.0
	Standard Dev	0.4	0.9
	% RSD	55%	13%
Bag #3	Mean	0.24	6.4
	Standard Dev	0.05	0.3
	% RSD	19%	4.1%
Bag #7	Mean	0.13	7.3
	Standard Dev	0.03	1.1
	% RSD	22%	16%
Box #1	Mean	0.3	6.9
	Standard Dev	0.3	0.9
	% RSD	91%	13%
Bag #3	Mean	0.18	7.2
	Standard Dev	0.06	0.3
	% RSD	34%	3.9%
Bag #5	Mean	0.18	6.7
	Standard Dev	0.06	0.3
	% RSD	33%	4.5%
Bag #7	Mean	0.169	7.24
	Standard Dev	0.006	0.16
	% RSD	3.4%	2.2%
Box #2	Mean	0.18	7.0
	Standard Dev	0.05	0.4
	% RSD	28%	5.2%
Bag #1	Mean	0.99	6.2
	Standard Dev	0.09	0.5
	% RSD	9.3%	7.5%
Bag #2	Mean	1.2	5.6
	Standard Dev	0.3	0.7
	% RSD	21%	13%
Bag #8	Mean	1.11	6.0
	Standard Dev	0.19	0.3
	% RSD	17%	5.8%
Box #3	Mean	1.1	5.9
	Standard Dev	0.2	0.5
	% RSD	18%	9.2%
Lot B	Mean	0.6	6.6
	Standard Dev	0.5	0.8
	% RSD	82%	12.2%

TABLE 18c: (Continued)

(2) Glad Lot C

		Al	Si	P	S	K
Bag #3	Mean	1290	3200	120	32	23
	Standard Dev	50	100	15	5	3
	% RSD	3.5%	3.0%	13%	16%	15%
Bag #6	Mean	1180	2800	105	27	21
	Standard Dev	190	500	15	8	5
	% RSD	16%	17%	14%	30%	22%
Bag #9	Mean	1430	3400	116	40	27
	Standard Dev	150	400	5	13	5
	% RSD	11%	11%	4.4%	32%	19%
Box #1	Mean	1300	3100	112	33	24
	Standard Dev	170	400	12	11	5
	% RSD	13%	13%	11%	33%	21%
Bag #1	Mean	1300	3200	116	29	24
	Standard Dev	200	700	7	4	4
	% RSD	19%	23%	5.7%	12%	17%
Bag #6	Mean	1290	3100	115	41	25
	Standard Dev	120	300	15	15	11
	% RSD	9.0%	11%	13%	37%	44%
Bag #7	Mean	1160	2900	103	25	23
	Standard Dev	130	400	7	5	5
	% RSD	11%	14%	7.1%	22%	22%
Box #2	Mean	1260	3100	111	32	24
	Standard Dev	170	500	11	11	7
	% RSD	14%	16%	10%	35%	28%
Bag #4	Mean	1180	2800	102	46	21
	Standard Dev	130	300	19	12	7
	% RSD	11%	11%	19%	27%	32%
Bag #7	Mean	1150	2700	100	60	28
	Standard Dev	120	300	13	20	9
	% RSD	11%	12%	13%	40%	32%
Bag #8	Mean	1110	2660	82	39	24
	Standard Dev	60	150	5	11	7
	% RSD	5.4%	5.6%	6.4%	27%	28%
Box #3	Mean	1150	2700	95	48	24
	Standard Dev	100	300	16	17	7
	% RSD	9.0%	9.5%	17%	36%	30%
Lot C	Mean	1230	3000	106	38	24
	Standard Dev	160	400	15	15	6
	% RSD	13%	14%	14%	40%	26%

TABLE 18c: (Continued)

(2) Glad Lot C

		Ca	Ti	Cr	Mn	Fe
Bag #3	Mean	53	1650	2.2	0.466	138
	Standard Dev	9	50	0.3	0.008	4
	% RSD	18%	3.1%	15%	1.8%	3.2%
Bag #6	Mean	44	1520	3	0.55	132
	Standard Dev	9	170	2	0.14	19
	% RSD	20%	11%	62%	26%	15%
Bag #9	Mean	80	1690	2.0	0.51	150
	Standard Dev	40	103	0.6	0.05	12
	% RSD	50%	6.1%	32%	9.3%	8.1%
Box #1	Mean	60	1620	2.5	0.51	138
	Standard Dev	30	140	1.3	0.09	14
	% RSD	48%	8.4%	51%	18%	10%
Bag #1	Mean	61	1600	1.8	0.44	140
	Standard Dev	11	200	1.0	0.03	20
	% RSD	18%	13%	58%	7.3%	16%
Bag #6	Mean	110	1590	3	0.51	150
	Standard Dev	50	150	2	0.13	30
	% RSD	44%	9.6%	70%	25%	18%
Bag #7	Mean	60	1650	1.06	0.50	129
	Standard Dev	20	90	0.4	0.13	7
	% RSD	33%	5.4%	34%	26%	5.8%
Box #2	Mean	80	1620	1.9	0.48	140
	Standard Dev	40	150	1.4	0.10	20
	% RSD	48%	9.1%	75%	21%	14%
Bag #4	Mean	70	1590	1.8	0.32	132
	Standard Dev	30	40	0.6	0.07	5
	% RSD	36%	2.4%	34%	21%	3.9%
Bag #7	Mean	70	1550	1.4	0.47	128
	Standard Dev	30	60	0.2	0.04	9
	% RSD	40%	4.1%	14%	8.8%	7.3%
Bag #8	Mean	40	1680	0.7	0.36	123
	Standard Dev	5	130	0.5	0.06	6
	% RSD	12%	7.9%	67%	18%	4.6%
Box #3	Mean	60	1610	1.3	0.38	128
	Standard Dev	20	100	0.6	0.09	7
	% RSD	41%	6.1%	49%	22%	5.9%
Lot C	Mean	70	1620	1.9	0.46	135
	Standard Dev	30	120	1.2	0.11	15
	% RSD	47%	7.7%	65%	23%	11%

TABLE 18c: (Continued)

(2) Glad Lot C

		Ni	Ti/Fe
Bag #3	Mean	0.8	12.0
	Standard Dev	0.2	0.2
	% RSD	28%	1.9%
Bag #6	Mean	5	11.6
	Standard Dev	8	0.9
	% RSD	168%	7.4%
Bag #9	Mean	1.0	11.7
	Standard Dev	0.6	0.3
	% RSD	58%	2.8%
Box #1	Mean	2	11.7
	Standard Dev	5	0.5
	% RSD	209%	4.6%
Bag #1	Mean	0.8	11.7
	Standard Dev	0.6	0.6
	% RSD	75%	5.2%
Bag #6	Mean	0.7	11.0
	Standard Dev	0.4	2.1
	% RSD	51%	19%
Bag #7	Mean	0.50	12.9
	Standard Dev	0.09	1.0
	% RSD	19%	7.7%
Box #2	Mean	0.7	11.9
	Standard Dev	0.4	1.5
	% RSD	59%	12%
Bag #4	Mean	0.70	12.0
	Standard Dev	0.17	0.5
	% RSD	25%	4.2%
Bag #7	Mean	0.71	12.1
	Standard Dev	0.14	0.5
	% RSD	20%	4.2%
Bag #8	Mean	0.41	13.7
	Standard Dev	0.10	0.5
	% RSD	23%	3.4%
Box #3	Mean	0.61	12.6
	Standard Dev	0.19	0.9
	% RSD	32%	7.1%
Lot C	Mean	1.2	12.1
	Standard Dev	2.7	1.1
	% RSD	232%	9.1%

TABLE 18c: (Continued)

(3) Glad Lot D

		Al	Si	P	S	K
Bag #5	Mean	970	2270	93	13	16
	Standard Dev	70	160	11	11	4
	% RSD	7.2%	7.0%	12%	80%	22%
Bag #6	Mean	930	2210	87	8	19.0
	Standard Dev	50	130	4	3	1.4
	% RSD	5.7%	5.8%	4.7%	34%	7.1%
Bag #10	Mean	880	2170	97	13	23
	Standard Dev	80	150	12	6	8
	% RSD	9.0%	6.8%	13%	42%	37%
Box #1	Mean	930	2220	92	12	19
	Standard Dev	70	140	10	7	6
	% RSD	7.7%	6.3%	11%	61%	29%
Bag #2	Mean	950	2300	78	10	19
	Standard Dev	120	500	16	9	11
	% RSD	13%	23%	20%	98%	60%
Bag #4	Mean	970	2300	97	50	33
	Standard Dev	100	300	17	50	19
	% RSD	11%	14%	17%	96%	59%
Bag #9	Mean	980	2350	100	50	32
	Standard Dev	90	110	10	40	14
	% RSD	9.2%	4.8%	10%	82%	42%
Box #2	Mean	970	2300	93	40	28
	Standard Dev	90	300	16	40	16
	% RSD	9.8%	13%	18%	104%	55%
Bag #2	Mean	1010	2400	80	11	24
	Standard Dev	7	200	10	9	2
	% RSD	0.7%	9.4%	12%	77%	10%
Bag #5	Mean	910	2400	98	40	32
	Standard Dev	70	300	17	30	15
	% RSD	7.3%	12%	18%	79%	45%
Bag #6	Mean	1030	2600	100	40	26
	Standard Dev	70	300	30	40	13
	% RSD	7.1%	11%	30%	85%	48%
Box #3	Mean	970	2400	100	30	29
	Standard Dev	80	300	20	30	12
	% RSD	8.2%	10%	22%	86%	40%
Lot D	Mean	950	2300	93	30	25
	Standard Dev	80	200	15	30	12
	% RSD	8.6%	11%	16%	110%	47%

TABLE 18c: (Continued)

(3) Glad Lot D

		Ca	Ti	Cr	Mn	Fe
Bag #5	Mean	60	1490	1.4	0.41	284
	Standard Dev	30	70	0.2	0.02	14
	% RSD	53%	4.9%	14%	5.4%	4.9%
Bag #6	Mean	42	1730	1.28	0.44	303
	Standard Dev	15	110	0.12	0.05	19
	% RSD	35%	6.7%	9.3%	11%	6.1%
Bag #10	Mean	46	1580	1.6	0.43	300
	Standard Dev	13	170	0.2	0.04	20
	% RSD	27%	11%	14%	8.8%	7.6%
Box #1	Mean	50	1600	1.4	0.43	296
	Standard Dev	20	150	0.2	0.04	19
	% RSD	45%	9.6%	14%	8.3%	6.4%
Bag #2	Mean	44	1530	1.4	0.41	270
	Standard Dev	14	110	0.5	0.15	30
	% RSD	32%	6.9%	33%	36%	13%
Bag #4	Mean	43	1580	1.70	0.50	300
	Standard Dev	5	170	0.11	0.03	30
	% RSD	12%	11%	6.7%	6.8%	8.9%
Bag #9	Mean	39	1610	1.4	0.43	298
	Standard Dev	8	170	0.3	0.08	16
	% RSD	20%	10%	19%	19%	5.3%
Box #2	Mean	42	1580	1.5	0.45	290
	Standard Dev	8	140	0.3	0.09	30
	% RSD	20%	8.8%	20%	20%	9.5%
Bag #2	Mean	43	1686	1.29	0.465	299
	Standard Dev	5	2	0.13	0.007	13
	% RSD	12%	0.1%	10%	1.5%	4.5%
Bag #5	Mean	60	1520	1.9	0.48	294
	Standard Dev	20	50	0.6	0.07	19
	% RSD	39%	3.3%	32%	14%	6.5%
Bag #6	Mean	60	1660	1.4	0.52	300
	Standard Dev	30	150	0.4	0.12	40
	% RSD	59%	9.3%	26%	24%	12%
Box #3	Mean	50	1600	1.6	0.49	300
	Standard Dev	20	120	0.5	0.08	20
	% RSD	42%	7.2%	31%	16%	7.5%
Lot D	Mean	49	1590	1.5	0.45	290
	Standard Dev	19	130	0.3	0.07	20
	% RSD	40%	8.5%	23%	16%	7.6%

TABLE 18c: (Continued)

(3) Glad Lot D

		Ni	Ti/Fe
Bag #5	Mean	2.4	5.2
	Standard Dev	0.5	0.3
	% RSD	19%	6.0%
Bag #6	Mean	2.1	5.7
	Standard Dev	0.2	0.2
	% RSD	9.1%	3.8%
Bag #10	Mean	2.6	5.2
	Standard Dev	0.5	0.2
	% RSD	20%	4.5%
Box #1	Mean	2.4	5.4
	Standard Dev	0.4	0.3
	% RSD	19%	6.1%
Bag #2	Mean	2.0	5.7
	Standard Dev	0.5	0.4
	% RSD	25%	6.3%
Bag #4	Mean	2.7	5.3
	Standard Dev	0.7	0.3
	% RSD	25%	5.9%
Bag #9	Mean	2.0	5.4
	Standard Dev	0.3	0.3
	% RSD	14%	4.9%
Box #2	Mean	2.3	5.5
	Standard Dev	0.6	0.3
	% RSD	26%	6.2%
Bag #2	Mean	5	5.6
	Standard Dev	4	0.2
	% RSD	83%	4.3%
Bag #5	Mean	3.5	5.2
	Standard Dev	1.0	0.2
	% RSD	30%	3.9%
Bag #6	Mean	2.2	5.5
	Standard Dev	0.4	0.3
	% RSD	16%	5.0%
Box #3	Mean	3	5.4
	Standard Dev	2	0.3
	% RSD	61%	5.3%
Lot D	Mean	2.7	5.4
	Standard Dev	1.3	0.3
	% RSD	49%	5.7%

TABLE 19a: Descriptive statistics for the study of compositional variation within manufacturer. Excitation mode is Mo-K.

		Al	Si	P	S	K
LOT B	Mean	650	1580	111	17	8
	Standard Dev	80	190	7	4	3
	% RSD	12%	12%	6.6%	25%	40%
LOT C	Mean	1190	2900	110	16	12
	Standard Dev	110	300	13	20	9
	% RSD	9.6%	10%	12%	124%	72%
LOT D	Mean	890	2160	90	5	6
	Standard Dev	100	180	10	11	6
	% RSD	11%	8.1%	11%	193%	94%

		Ca	Ti	Cr	Mn	Fe
LOT B	Mean	14	730	0.11	0.19	79
	Standard Dev	2	60	0.15	0.06	6
	% RSD	17%	8.7%	137%	30%	7.0%
LOT C	Mean	39	1620	0.7	0.33	97
	Standard Dev	17	120	0.5	0.06	9
	% RSD	44%	7.7%	67%	18%	8.8%
LOT D	Mean	26	1590	0.48	0.30	215
	Standard Dev	8	130	0.18	0.07	16
	% RSD	30%	8.5%	38%	23%	7.5%

		Ni	Cu	Zn	Rb	Sr
LOT B	Mean	0.24	0.15	40	0.002	0.46
	Standard Dev	0.19	0.19	3	0.004	0.03
	% RSD	79%	124%	6.7%	168%	6.3%
LOT C	Mean	0.5	0.21	40	0.056	0.83
	Standard Dev	0.3	0.09	3	0.018	0.08
	% RSD	55%	41%	7.3%	33%	9.3%
LOT D	Mean	1.6	0.19	28	0.016	0.64
	Standard Dev	0.5	0.07	3	0.011	0.05
	% RSD	30%	38%	9.9%	68%	7.6%

TABLE 19a: (Continued)

		Zr	Pb	Ti/Fe
LOT B	Mean	3	0.45	9.2
	Standard Dev	9	0.15	0.4
	% RSD	270%	32%	3.8%
LOT C	Mean	21	0.47	16.7
	Standard Dev	16	0.09	0.9
	% RSD	74%	18%	5.2%
LOT D	Mean	7	1.01	7.4
	Standard Dev	8	0.05	0.4
	% RSD	104%	5.3%	5.0%

TABLE 19b: Descriptive statistics for the study of compositional variation within manufacturer. Excitation mode is W-*brems*.

		S	K	Ca	Ti	Cr
LOT B	Mean	16	8	17	860	1.2
	Standard Dev	16	4	11	70	0.6
	% RSD	99%	48%	68%	8.0%	51%
LOT C	Mean	0.02	6	31	1940	4.7
	Standard Dev	0.13	4	16	160	1.0
	% RSD	592%	56%	54%	8.1%	21%
LOT D	Mean	6	7	23	1920	3.6
	Standard Dev	13	6	11	120	0.6
	% RSD	223%	88%	50%	6.1%	17%

		Mn	Fe	Ni	Cu	Zn
LOT B	Mean	0.23	91	0.2	0.15	42
	Standard Dev	0.12	6	0.2	0.18	3
	% RSD	51%	6.4%	90%	120%	6.0%
LOT C	Mean	0.06	110	0.7	0.25	43
	Standard Dev	0.11	9	1.8	0.18	3
	% RSD	170%	8.3%	272%	70%	6.7%
LOT D	Mean	0.23	256	1.6	0.16	30
	Standard Dev	0.12	15	0.9	0.09	4
	% RSD	53%	5.8%	56%	56%	12%

		Rb	Sr	Y	Zr	Pb
LOT B	Mean	0.006	0.45	0.023	3	0.48
	Standard Dev	0.009	0.03	0.022	8	0.10
	% RSD	158%	7.2%	94%	251%	21%
LOT C	Mean	0.047	0.74	0.10	20	0.54
	Standard Dev	0.016	0.07	0.04	14	0.10
	% RSD	33%	9.7%	42%	71%	19%
LOT D	Mean	0.018	0.58	0.040	7	1.08
	Standard Dev	0.014	0.05	0.017	7	0.07
	% RSD	78%	8.8%	44%	92%	6.5%

TABLE 19b: (Continued)

		Ba	Sn	Ag	Ti/Fe
LOT B	Mean	21.3	0.3	0.026	9.5
	Standard Dev	1.6	0.4	0.017	0.4
	% RSD	7.6%	143%	64%	4.5%
LOT C	Mean	20.6	0.18	0.05	17.6
	Standard Dev	1.6	0.05	0.04	0.8
	% RSD	7.8%	25%	87	4.3
LOT D	Mean	18.2	0.7	0.04	7.46
	Standard Dev	1.3	0.9	0.02	0.13
	% RSD	7.3%	122%	48%	1.7%

TABLE 19c: Descriptive statistics for the study of compositional variation within manufacturer. Excitation mode is W-L.

		Al	Si	P	S	K
LOT B	Mean	710	1800	116	70	28
	Standard Dev	120	400	20	40	11
	% RSD	17%	22%	17%	54%	38%
LOT C	Mean	1230	3000	106	38	24
	Standard Dev	160	400	15	15	6
	% RSD	13%	14%	14%	40%	26%
LOT D	Mean	950	2300	93	30	25
	Standard Dev	80	200	15	30	12
	% RSD	8.6%	11%	16%	110%	47%

		Ca	Ti	Cr	Mn	Fe
LOT B	Mean	34	730	1.1	0.33	112
	Standard Dev	18	60	0.7	0.11	13
	% RSD	54%	8.7%	70%	34%	12%
LOT C	Mean	70	1620	1.9	0.46	135
	Standard Dev	30	120	1.2	0.11	15
	% RSD	47%	7.7%	65%	23%	11%
LOT D	Mean	49	1590	1.5	0.45	290
	Standard Dev	19	130	0.3	0.07	20
	% RSD	40%	8.5%	23%	16%	7.6%

		Ni	Ti/Fe
LOT B	Mean	0.6	6.6
	Standard Dev	0.5	0.8
	% RSD	82%	12.2%
LOT C	Mean	1.2	12.1
	Standard Dev	2.7	1.1
	% RSD	232%	9.1%
LOT D	Mean	2.7	5.4
	Standard Dev	1.3	0.3
	% RSD	49%	5.7%

TABLE 20: Elements and excitation modes selected for comparison among sources

Elements and X-ray lines by Mo-K_α excitation	Elements and X-ray lines by W-*brems* excitation	
Al K_α	Ti K_α	Y K_α
Si K_α	Cr K_α	Zr K_α
P K_α	Mn K_α	Pb L_α
S K_α	Fe K_α	Ba K_α
K K_α	Zn K_α	Sn K_α
Ca K_α	Rb K_α	Ag K_α
Ni K_α	Sr K_α	
Cu K_α		

A. Compositional Variation Within Bags

The variations of element concentrations within plastic bags were determined for four bags from separate production runs from three different manufacturers. This information is necessary to define the degree of variability within the manufactured product. The effect of sample size (2.5 to 3 mg) on the variability of results was minimized by using the optimum sample size determined previously (Chapter IV). Each bag was sampled in twelve locations, as per the grid pattern in Figure 14. Summary results for the variations of element concentrations within these bags are shown in Table 15. Descriptive statistics for Cl are only included in Tables 15a and 15c, because Cl was only reliably detected above the level in the blank in the Ruffies™ plastic bag. Within-bag precisions for Ti, Ca, Fe, Zn, and Ba are generally better than 10% RSD, and may be below 5% when elements are present at levels well above the LOQ. The within-bag precisions of other elements, such as Al, Si, P, S, K, Ca, and Sr, are generally better than 20% RSD. Elements Zr, Sn, and Ag, when present at measurable levels, may be highly

variable within a single bag. These within-bag studies were performed early in the research project, and the precisions of the element concentrations within bags determined in later studies are generally better than these by a factor of 2 or more. The improvement in precision over this study resulted from improved ability to transfer samples completely and eliminate contamination. Variations across boxes, lots and manufacturers, which were obtained in later studies, were required to evaluate the value of all elements, and further discussions follow. The differences in within-bag compositional variations among the manufacturers in this study result primarily from the differences in element concentration levels among the products. That is, regardless of manufacturer, for any given element the RSD decreases as concentrations increase into a good analytical range. The effect of sample position with respect to the X-ray beam on the element concentrations was examined. Differences in the element concentrations were not detected between the two sides of the assembled bag or across the length or width of the bag. The compositional variation within single bags was found to be small compared with the variation among manufacturing sources (Tables 16-19). Based on the results of this study, four replicate samples per bag chosen from randomly selected locations were analyzed in the subsequent studies.

B. Compositional Variation Within Boxes

The within-box compositional variations of Glad® Quick-Tie™ plastic trash bags were determined for boxes of ten and twenty bags. The concentration range of elements among bags contained within a single box is important for the determination of an association between a crime scene bag and a known-source box. Four replicates were

sampled from each bag. These served as measures of the within-bag variability for this box. In addition, the repeated analysis of replicate samples from within bags throughout the course of this research project increased the experience base for within-bag compositional variations.

All of the bags in a box of ten bags (GB1) were analyzed. Descriptive statistics, including means, standard deviations, and RSDs, within each of the bags and within the box are shown in Tables 16a through 16c. The within-bag RSDs of the element concentrations determined for bags from within the same box are better than those previously measured in the within-bag study. The element concentrations of Al, Si, Ca, and Pb have within-bag RSDs that are generally between 10% and 30%. The mean within-bag RSDs for the concentrations of P, Zn, Sr, and Ba are 5% or less. The mean RSDs for the concentrations of Ti and Fe are about 10% or less. The mean RSD for Ti/Fe, which is less than 3%, is better than the RSD of either element concentration alone. The graphs in Figures 15 and 16 demonstrate the precision of the concentrations of Ti, Fe, Zn, Sr, Ba, and Zr within this box. The error bars plotted in the graphs are ± 2 standard deviation around the bag means. Visual comparison of the overlap of the error bars in Figures 15 and 16 indicates that some bag-to-bag differences exist within this box. For this box, the within-bag and within-box RSDs for Zr are large compared to the RSDs for the other elements measured.

Analysis of variance (ANOVA) was performed for each element to test for a significant difference between the within-bag and within-box (between bag) means. SYSTAT® version 8.03 software (SPSS, Inc., Chicago, IL) was used to perform the

FIGURE 15: Graph of the within-bag precisions among plastic bags contained in box GB1 for three element concentrations. Results are for data acquired by TXRF W-*brems* excitation. Error bars represent ± 2 standard deviations around the bag means.

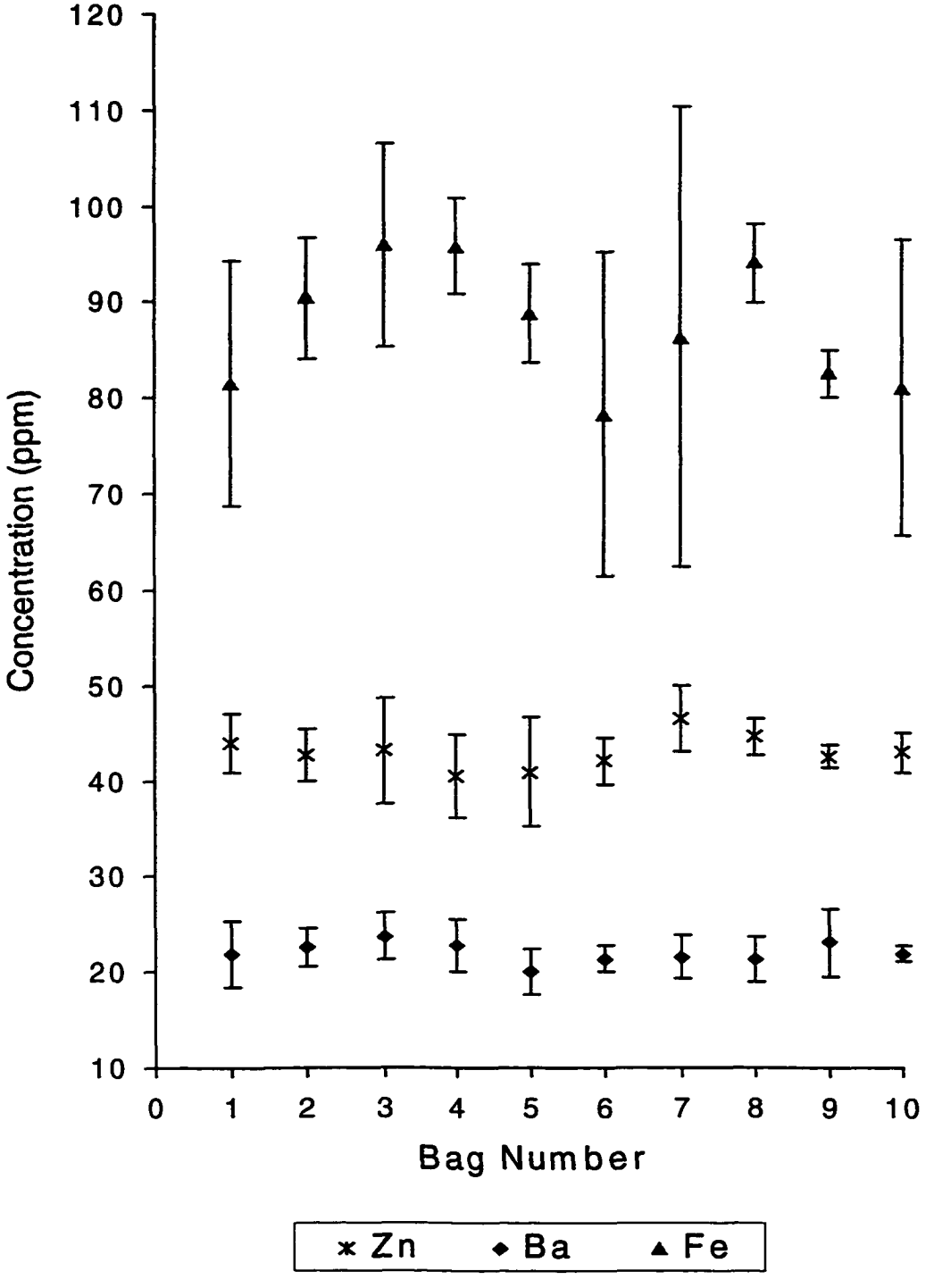
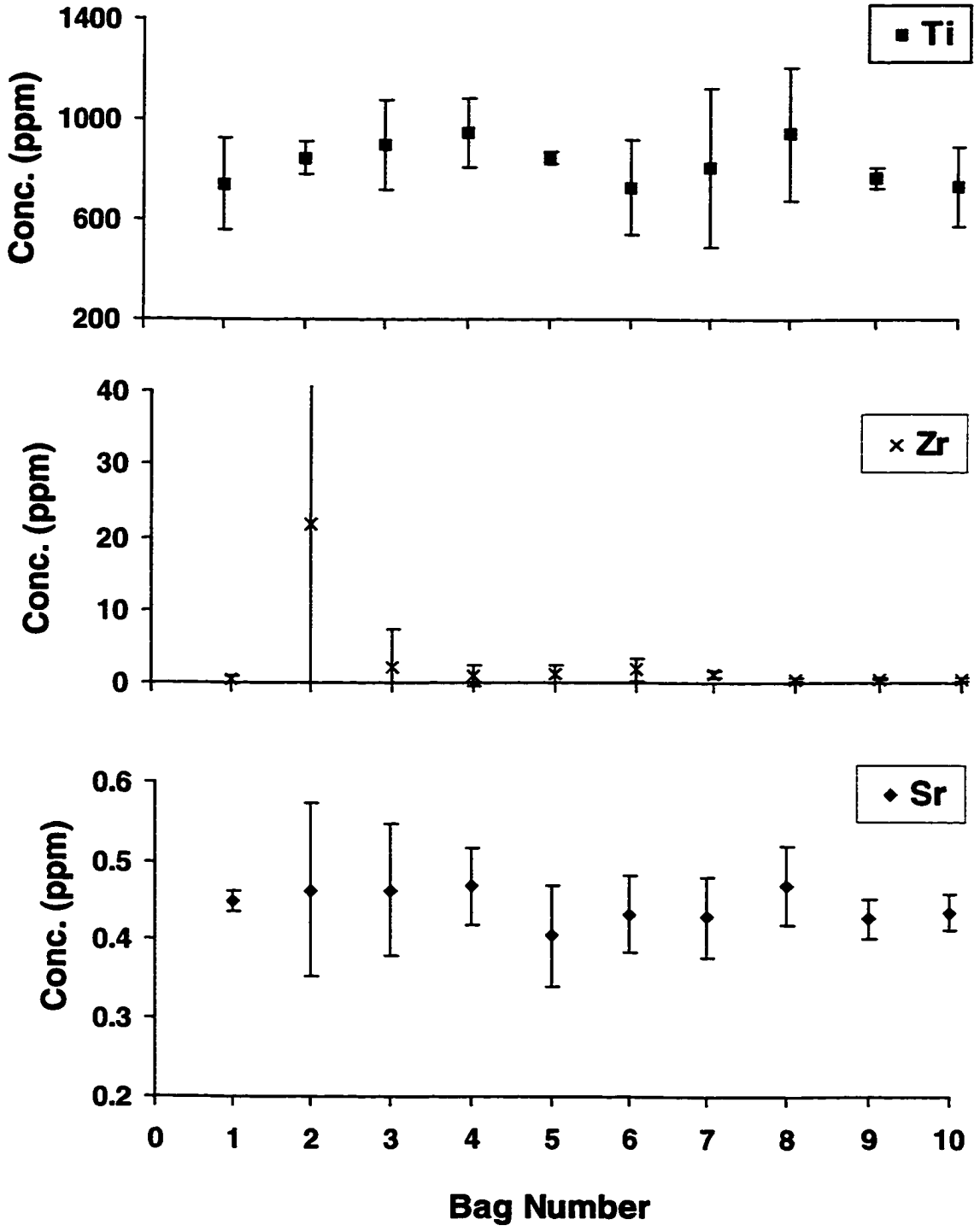


FIGURE 16: Graph of the within-bag precisions among plastic bags contained in box GB1 for three element concentrations. Results are for data acquired by TXRF W-*brems* excitation. Error bars represent ± 2 standard deviations around the bag means.



statistical evaluation of the data. Outlier element concentrations were identified by SYSTAT® software using the studentized residual during the ANOVA calculations. These outliers were evaluated manually and, where appropriate, were removed from the data set and the ANOVA was recalculated. The outliers that were removed from the data set are indicated with an "*" in Tables 11 through 14. ANOVA indicates whether at least one mean significantly differs from the others in the group, but it does not identify which pairs of means are different. Tukey pairwise mean comparisons of the bag means were performed to identify the pairs of bags that were significantly different from one another and the element(s) that discriminated between the bags. This multiple comparisons method was used instead of *t* tests because it protects against increased Type I errors, which result from multiple individual comparisons each performed at a significance level (α). The Tukey method was chosen instead of the Bonferroni adjustment because it is more sensitive in detecting differences when the number of pairwise comparisons is large (greater than 5).

Of the 21 element concentrations measured, the ANOVAs failed to detect a significant difference between the element concentrations of P, S, K, Ca, Ti, Mn, Rb, Sr, Ag and Ti/Fe among the bags within this box. These element concentrations are appropriate for grouping or classifying these bags as belonging to this box, since the within-box variation of the concentration of these elements is relatively small. In order for these element concentrations to be useful for discrimination among sources, the range of concentrations of elements among boxes, lots, or manufacturers must be large compared to the within-box variations.

The pairs of bags from within box GB1 that are distinguishable using the pairwise comparisons are identified in Table 21, along with the elements that differentiate each pair of bags. An empty cell in the table indicates that the pairs of bags are analytically indistinguishable by all elements measured. Tukey pairwise mean comparison results indicate that 29 out of the 45 pairs of bags compared (64%) were significantly different in one or more element concentration. Most of the element concentrations that are different between bags within the same box are present at very low levels, such as Cr, Ni, and Y, or are highly variable both within and between bags, such as Zr and Sn. Replicate data for element concentrations among pairs of bags found to be significantly different by the Tukey comparisons were evaluated using a range overlap method. If the element concentrations for the replicates between two pairs overlapped, the bags were considered indistinguishable. This is a more conservative method and was selected to avoid overstating the discrimination capability of the analytical method. It is also more appropriate when the variances are unequal, which is the case with highly variable data such as that of Zr and Sn. It is interesting to note that the range overlap method resulted in 10 pairs of bags being declared indistinguishable for a given element when Tukey comparisons resulted in a significant difference. However, because of the number of element concentrations that differ between pairs, only one bag pair that was considered distinguishable by Tukey was indistinguishable based on the range overlap results.

The compositional variation within a larger box of plastic bags (GE1) was studied by analyzing 13 out of 20 bags contained within the box. Descriptive statistics for these results are shown in Table 17. The within-bag precisions of the element concentrations

TABLE 21: Results of the element concentration pairwise comparisons of all bags contained within a single box (GB1). Elements listed for the bag pair are those whose concentrations were significantly different by both the Tukey pairwise mean comparison and the range overlap method.

Bag No. → / ↓	1	2	3	4	5	6	7	8	9	10
1										
2	Ni, Cr, Y, Zr, Sn									
3	Al, Si, Sn	Ni, Y, Zr								
4	Al, Si, Cr	Ni, Y, Zr								
5	Si	Ni, Y, Zr, Sn	Ba							
6		Ni, Cr, Y, Zr	Al	Al, Cr	Al, Si					
7	Cu	Ni, Y, Zr	Si	Si, Zn, Y	Zn					
8	Si, Cr	Ni, Y, Zr				Cr				
9		Ni, Cr, Y, Zr, Sn	Cr	Al, Cr			Cu			
10		Ni, Y, Zr, Sn	Al	Al	Al		Cu			

are generally equal to or better than those measured for the plastic bags in the box GB1. As previously discussed, this is likely related to the improved precision in sample preparation gained from experience during the course of this research project. ANOVA and Tukey pairwise mean comparisons of the concentration results for all elements determined were compared in the same manner as for box GB1 described above. The only element concentrations that have a significant difference in means among bags in this box are Cr, Y, and Zr. Twenty-two out of 78 pairs compared (28%) were significantly different on the basis of one or more of the concentrations of these three elements using the Tukey and range overlap methods (Table 22). Bag #1 and bag #2 from this box are analytically indistinguishable from each other, but are different from all other bags tested from this box, accounting for all 22 differing pairs. Bags #1 and #2 may be considered as a separate compositional group within this box.

The results of the study of the compositional variation within boxes indicate that quantifiable differences in elemental concentrations do occur among bags contained within the same box at least for the elements Cr, Ni, Y, Zr, and Sn. Other elements are present at analytically indistinguishable levels among the bags within a box. The ability to detect within-box differences is not an advantage for most forensic plastic bag examinations, since comparisons are generally made between a crime-scene bag and a known-source box. Within-box variability for each box should be measured whenever possible in casework applications and taken into account in setting the criteria for the determination of the compared samples as analytically indistinguishable. Otherwise, one-on-one bag comparisons could result in incorrect source elimination.

TABLE 21: Results of the element concentration pairwise comparisons of all bags contained within a single box (GB1). Elements listed for the bag pair are those whose concentrations were significantly different by both the Tukey pairwise mean comparison and the range overlap method.

Bag No. → / ↓	1	2	3	4	5	6	7	8	9	10
1										
2	Ni, Cr, Y, Zr, Sn									
3	Al, Si, Sn	Ni, Y, Zr								
4	Al, Si, Cr	Ni, Y, Zr								
5	Si	Ni, Y, Zr, Sn	Ba							
6		Ni, Cr, Y, Zr	Al	Al, Cr	Al, Si					
7	Cu	Ni, Y, Zr	Si	Si, Zn, Y	Zn					
8	Si, Cr	Ni, Y, Zr				Cr				
9		Ni, Cr, Y, Zr, Sn	Cr	Al, Cr			Cu			
10		Ni, Y, Zr, Sn	Al	Al	Al		Cu			

C. Compositional Variation Within Manufacturing Production Runs

The variability of the element concentrations within a production source is important in determining the ability of the analytical method to discriminate among manufactured items from a given production source. In other words, for plastic bags, the variability among boxes produced at the same time in the same manufacturing plant is helpful in comparing it to the variations observed over time and manufacturer. This information is valuable for the interpretation of the significance of a comparison of two plastic bags in a case situation. For the purposes of this study, the manufacturer's packaging code imprinted on the box was used to indicate a particular production source. The term "lot" is often used in this discussion and in the tables for simplicity and should be considered synonymous with "packaging code".

Three boxes each for three different packaging codes were analyzed by TXRF under the analytical conditions specified in Table 7. Descriptive statistics for the element concentrations within bags, within boxes, and within lots analyzed in this study are shown in Tables 17a through 17c. Results of the element concentrations for four replicates from each of three bags per box were grouped as representative of the within-box variability and evaluated using ANOVA, Tukey pairwise mean comparison, and range overlap methods as described previously.

The ANOVA tests identified at least one within-lot difference between the means of boxes for each lot. For Lot B, ANOVA detected at least one significant difference in the mean concentrations of Al, Si, P, S, K, Ca, Ni, Cu, Zn, Y, Zr, Pb, and Ba between the boxes in the lot. ANOVA tests of the means of the element concentrations for boxes

within Lots C and D showed fewer significant differences than for Lot B. For Lot C, significant differences among boxes within the lot were identified by ANOVA for the concentrations of S, K, Ni, Y, Zr, Pb, and Sn. ANOVA only detected a significant difference among the boxes in Lot D for the means of the element concentrations of Si, Sr, and Zr.

Using the Tukey method, most element concentrations are indistinguishable between the three boxes within each lot. However, with the exception of one pair (box #2 vs. box #3 from Lot D), at least one element concentration was different enough to distinguish all boxes within each lot. Some of the measured element concentrations, such as Zr and Sn, have high and unequal variances, which result in the finding of significant differences using the Tukey method. The results of the pairwise comparisons between pairs of boxes in each of three lots, using the range overlap method differed substantially from those by the Tukey method, with fewer significant differences between pairs. Pairs of boxes within lots that were distinguishable by both the Tukey and range overlap methods are shown in Table 23. Of the nine pairs compared, four pairs of boxes are analytically distinguishable. Two-thirds of the pairs of boxes for each of Lots B and C are compositionally different; however, only a maximum of two element concentrations differ for each pair. Element concentrations that discriminated among these boxes were S, K, Ni, and Cu. None of the pairs of boxes from Lot D were analytically distinguishable.

Based on information obtained from the manufacturer of the product tested in this study and Ryland (1999), boxes with the same packaging codes do not necessarily contain

TABLE 23: Element concentration differences between pairs of boxes with the same packaging code.

Lot B	Box #1	Box #2	Box #3
Box #1			
Box #2			
Box #3	Cu	Ni	

Lot C	Box #1	Box #2	Box #3
Box #1			
Box #2			
Box #3	S, K	S, K	

Lot D	Box #1	Box #2	Box #3
Box #1			
Box #2			
Box #3			

plastic bags that were extruded from the same polymer source. The reason is that the packaging codes reflect the date, time, and place that the final bags were cut and packaged. However, the code does not indicate when the polymer film used to produce the individual bags in the box was extruded. As part of the QA program, the manufacturer does keep records of the identity of the source rolls that supply the bag-producing machines for a particular packaging code. In the manufacturing process for this product line, the sinusoidal tie flaps are cut into the extruded collapsed film, which is then rolled into four "jumbo" roll sets (approximately 12,000 bag equivalents per roll). Rolls are selected as needed to supply bag-producing machines, by product type and the age of the roll. All boxes of bags of the same product line that are cut and packaged on a particular line during an eight-hour shift in the manufacturing plant are assigned the same packaging code, regardless of the roll from which they were produced. Since bag machines produce this product line at a rate of approximately 220 bags per minute, over an eight hour shift, a minimum of eight rolls will be used to produce bags with the same packaging code. It is likely that the element concentrations across a single roll of 12,000 bag equivalents and between rolls used in the same packaging code differ. Therefore, compositional differences between boxes with the same packaging codes were expected.

In this study, only a small number of differences in element concentrations between boxes from the same lot was detected. This may be due in part to the shipping and distribution practices of the manufacturer, since these boxes were purchased from the same store. Boxes containing bags that originate from the same rolls, produced over a short time period, may be shipped together. In case applications, information on the

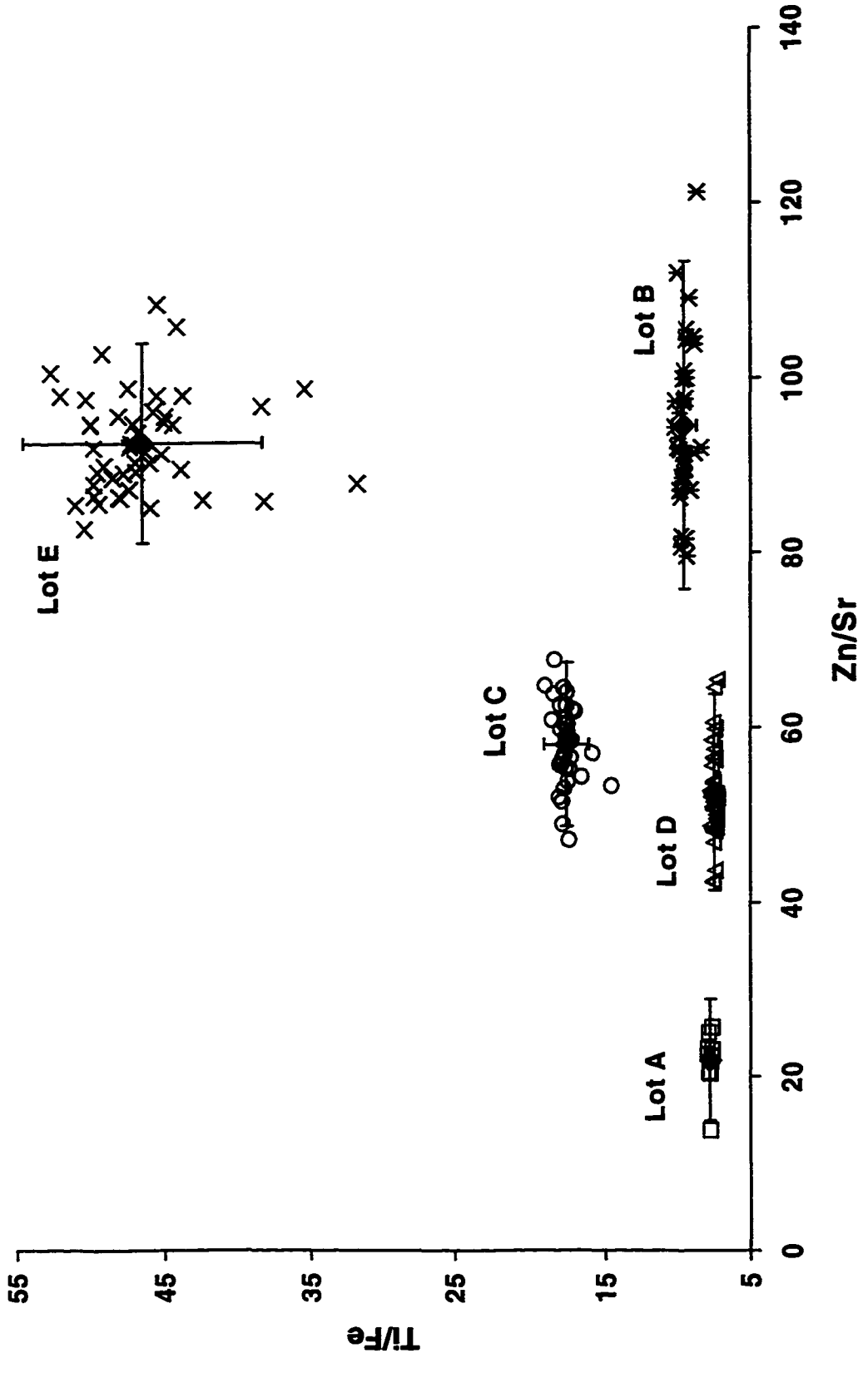
shipment and distribution of boxes and lots is needed to assist in the evaluation of the significance of compositionally indistinguishable items.

D. Compositional Variation Among Manufacturing Production Runs

Results from the study of the compositional variation within manufacturing runs were reevaluated for differences in element concentrations that exist among the different sources or packaging codes. Descriptive statistics for the element concentrations among the three different lots are shown in Tables 19a through 19c. Within-lot element concentration RSDs are small compared to the variations across lots to the degree that individual lots may be distinguished without the use of statistical comparisons.

The summary data in Tables 19a through 19c indicate that lots B, C, and D are easily separated based on concentrations of Ti and Fe alone. Samples from two additional sources of this product line, lots A and E, were previously analyzed for the determination of optimum sample size and within-box variation, respectively. For this reason, the numbers of samples differ from those for lots B, C, and D. Analytical results and descriptive statistics for samples analyzed from lot A are shown in Table 6, and those for lot E are shown in Tables 12b and 17b. Assuming that the samples analyzed are representative of their sources, lots A and E can also be differentiated from each other, and from lots B, C, and D. The discrimination of lots A and E from each other and from all other lots tested can be accomplished by comparison of the concentrations of Ti, Fe, Zn, and Sr. The discrimination among lots A through E using Ti/Fe and Zn/Sr is demonstrated in Figure 17. Mean results for each lot are indicated in the graph by a diamond. The error bars plotted around the mean are ± 2 standard deviations for all

FIGURE 17: Discrimination among lots of Glad® Quick-Tie™ plastic bags by element ratios Ti/Fe and Zn/Sr.



samples results from each lot. Results from different boxes within each lot are included as part of each lot in the graph. Although only two variables (using four element concentrations) were needed to separate these five lots into distinct compositional groups, more differences in element concentrations exist among these groups. This can be discerned by examination of the descriptive statistics among lots, which are shown in Tables 19a through 19c. In particular, Lots B, C, and D can be separated on the basis of Ti/Fe alone. Additional differences in element concentrations occur among these lots, including the concentrations of Al, Si, S, Ca, Ti, Fe, Ni, Pb, and Sn. Lots A and E also differ in concentrations of Sr and Zn, as shown in Figure 17.

Based on packaging code information from the manufacturer, the plant location and dates of packaging of these lots was determined and is indicated in Table 8. The bags in the boxes from lots B, C, and D, which are analytically distinguishable across lots, were all produced on the same bag-producing line and packaged in the same plant over a four-month period. The bags in the box from lot A were produced on the same bag-producing line in the same plant as those from lots B, C, and D, but almost one year earlier than lot B. The bags in the box from Lot E were produced and packaged at a separate plant location from the other lots in the study.

E. Compositional Variation Among Manufacturers

Element concentration results from the within-bag studies, which included plastic bags from three different manufacturers, were compared to determine the compositional differences among manufacturers. In the forensic science context, this is generally not needed because identification of the manufacturer and product line of a specific bag can

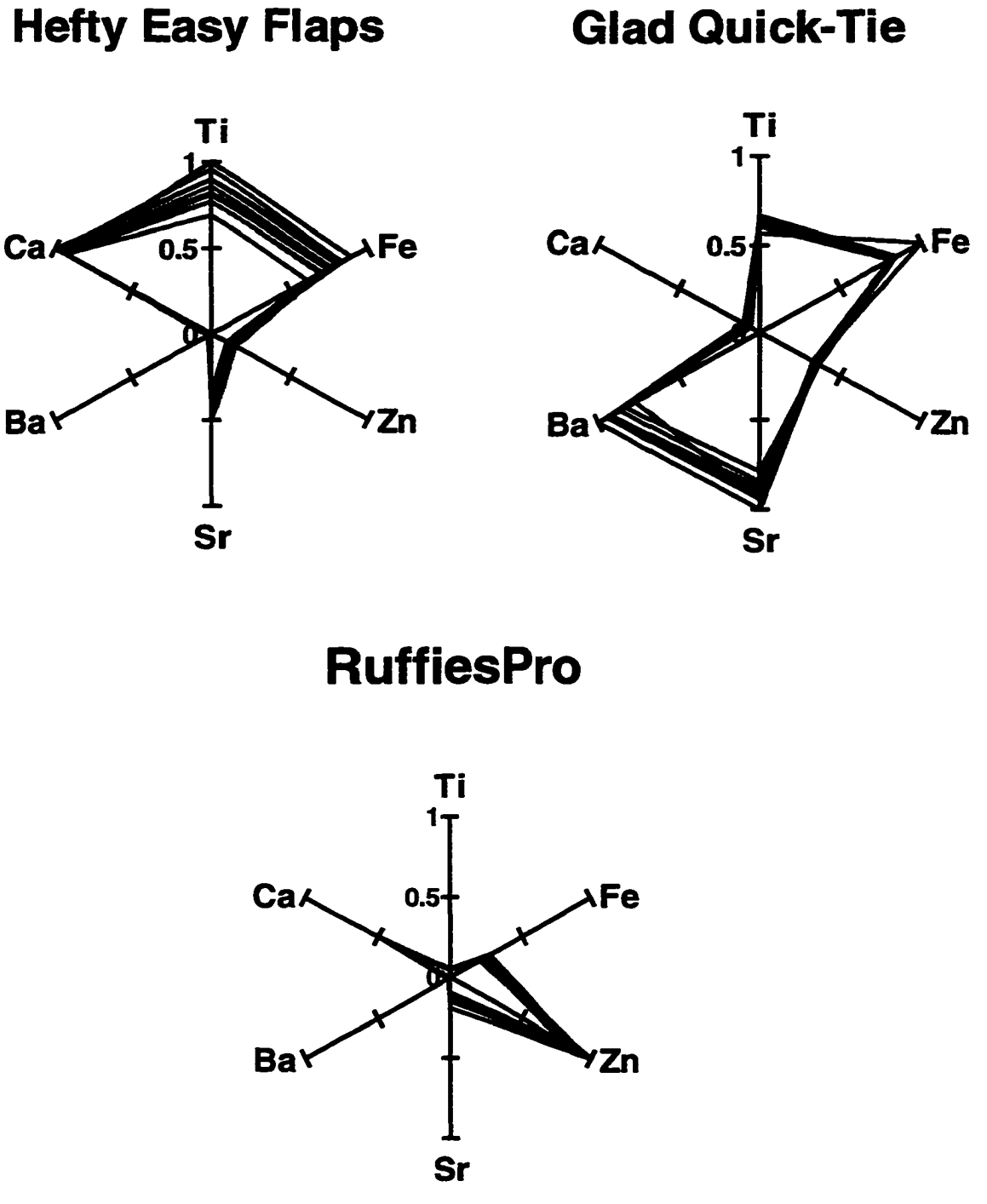
generally be achieved by the comparison of physical construction characteristics. Comparison of the TXRF spectra from the three different manufacturers shows large differences in the element concentrations among manufacturers. In particular, among the limited plastic bags analyzed from different manufacturers in this survey, Ba was only reliably detected and quantitated in the Glad® bags. The plastic bag from Ruffies™ contains low Ti and high Zn concentrations compared to the levels in the Hefty® bag or any of the Glad® bags analyzed. The Hefty® bag analyzed contains higher levels of Ca and Cr than the Ruffies™ or Glad® bags analyzed. Visual observation of the data shows that the bags analyzed from each manufacturer have readily identifiable elemental differences. A larger sampling of bags from each manufacturer would be necessary to identify a manufacturer-specific elemental profile, if one exists.

To facilitate the demonstration of the magnitude of these differences in multivariate data, star plots were constructed for each manufacturer using six of the 21 element concentrations determined. Star plots use axes radiating from a center point to display multivariate data in two dimensions. Each axis represents a variable and the distance of each point from the center indicates the value of the corresponding element variable. Star plots were constructed by scaling all of the concentration results among manufacturers for each element to the highest result obtained for each element among all sources. This process results in axes on the star plots ranging from 0 to 1 and allows for easy visual comparisons among plots.

The within-bag results for three different manufacturers plotted on star plots are shown in Figure 18. Replicate sample results are plotted on the same graph to

demonstrate that the within-bag precision is small compared to the variations among manufacturers. Figure 18 demonstrates that plastic bags from different manufacturers are readily differentiated by elemental composition. The graphical display of elemental composition data using star plots greatly simplifies the comparison process.

FIGURE 18: Star plots of compositional differences in plastic bags among manufacturers using six elements. Data are from a single bag from each manufacturer. Within-bag replicate samples are superimposed in each star plot.



CHAPTER VIII. CONCLUSIONS

This research evaluated the suitability of TXRF spectrometry for the analysis of and discrimination among polyethylene film bags sources based on trace elemental compositions. A procedure was developed for the preparation of polymeric materials for analysis by TXRF. Matrix reduction using oxygen plasma ashing was determined to be an effective means of removing the organic components of the polyethylene bag. This procedure greatly increases the mass of inorganic constituents that can be deposited on the TXRF sample support, yet remains within the size requirements of TXRF, and enhances the analytical sensitivity. The preparation procedure and analytical protocol developed and used in this project are suitable for application to a wide range of polymeric materials which are encountered as evidence, including tapes, wire insulation, and synthetic fibers.

Using the sample preparation method and analytical protocol developed in this research, TXRF spectrometry was shown to provide quantitative results with good precisions for those elements that are useful for classification of source and discrimination among similar sources present in polyethylene bags. Twenty-one element concentrations were determined in 2.5 to 3 mg samples of polyethylene bags using TXRF, 18 of which are readily determinable in most specimens. The precisions of the element concentration results for the low levels of inorganic components that are present in plastic bags are suitable for comparison among sources. The variability of elemental compositions within bags was determined to be equal to or below the variability within a box of bags for all measured elements. For most elements, the range of trace element concentrations within a box was determined to be equivalent to the range across boxes from within the same

manufacturing production run. The few elements with different concentrations among boxes within a lot are present at low concentrations and the differences among boxes are small. The variability of element concentrations across manufacturing production runs is significantly greater than the variability within a single production run. The ranges of element concentrations across manufacturers are larger than the range of elements concentrations within a single manufacturer.

Of the 18 elements readily determinable in most specimens, titanium (Ti), iron (Fe), zinc (Zn), calcium (Ca), strontium (Sr) and barium (Ba) are the most useful for discrimination among manufacturing sources. These element concentrations vary greatly among manufacturers and production runs, but are consistent within individual bags and among bags from a single box. Ti levels typically range from the low ppm level to 0.5%, with measured variations on the order of 5% RSD within each individual bag and among boxes produced at the same time. Fe, Zn, and Ba levels range from below the detection limits to the low ppm range, where the measured variations are typically less than 10% RSD. Results of this study indicate that bags produced from the same manufacturer and product line in different plants, and at different times in the same plant, are analytically distinguishable. Some results also suggest that discrimination among boxes from the same production run is possible.

Discrimination among plastic bag sources by elemental composition using large data sets were easily accomplished by graphical display using star plots. Star plots display a large amount of data in two dimensions by forming profiles with readily distinguishable shapes. Graphical display methods such as star plots can be useful in the

forensic sciences when attempting to exhibit differences among complex data in a readily recognizable format. Although data is easily represented by graphical display methods, care must be taken in their use for the presentation of comparisons. The variability within the data set per plot must be included in each plot for a valid comparison between plots.

CHAPTER IX. CONTRIBUTION TO KNOWLEDGE

This research contributes to the body of knowledge regarding the application of elemental profiles to the characterization and discrimination of trace evidentiary materials. The discrimination among batches of mass-produced polyethylene films by comparison of their trace elemental profiles is possible. By narrowing the range of potential sources to particular manufacturing lots or batches, increased significance can be assigned to the interpretation of indistinguishable trace evidentiary materials encountered in forensic casework. Further, the incorporation of compositional analysis into the analytical scheme offers the additional opportunity to detect differences that truly exist between very similar items, thereby reducing the risk of false associations. Elemental analysis of plastic bags by TXRF, or any other method, can be conducted on two or more samples that were found to be indistinguishable by conventional means.

Additionally, this research can provide the appropriate validation necessary to satisfy the *Daubert*, *Frye* or similar admissibility standard (Thornton, 1994) and therefore, may also assist juries in understanding the significance of a trace evidence "match". With some modifications, the procedures developed in this research could be applied to any number of polymeric materials received as trace evidence in the crime laboratory.

Inorganic contaminants in plastics, particularly toxic metals, are also of concern in the food packaging and pharmaceuticals industries, as well as in the control of environmental pollution from waste disposal. The development of a sensitive and

efficient instrumental method for the elemental analysis of plastics offers a significant contribution to these industries as well.

CHAPTER X. FURTHER CONSIDERATIONS

The discrimination potential of the comparison of plastic bags using an 18-element compositional profile is high. In order to determine the discrimination power of the method, compositional data from additional plastic bag sources must be acquired to determine more representative elemental concentration ranges across plastic bag sources. Data from an industry-wide survey of plastic bags is required to identify the number of distinguishable element concentrations in plastic bags using TXRF. This information, in turn, is needed to determine the magnitude of the discrimination potential of the method.

An additional area of research not addressed by this project is the comparison of TXRF results to data acquired by an alternative analytical technique, for example, ICP-MS or NAA. This would be useful in testing the accuracy of the TXRF results for the plastic bag samples. In order to analyze plastic bag samples by ICP-MS, further research is required to develop an appropriate sample digestion method or a method using laser ablation for direct solid sampling of the polymer film. The development of a method for the analysis of plastic bags by ICP- MS is feasible and worthy of further consideration. ICP-MS is widely available in many environmental laboratories and may be more readily accessible to the forensic science community than TXRF.

Direct elemental analysis of solid polymeric samples is worth further study. NAA and LA-ICP-MS are suitable techniques for the direct analysis of solid samples. Results recently reported in the literature have demonstrated that NAA followed by radiochemistry is a very sensitive technique for direct analysis of solid polymeric materials. In addition to its use as an independent test of the accuracy of TXRF results, NAA could be used as an

alternate analytical method for discrimination among sources. An potential advantage of NAA over TXRF it that NAA may provide good quantitative results for the rare earth elements that may be present in plastics, which were not detected by TXRF in this study. NAA with gamma-ray spectrometry can be applied to larger samples sizes than TXRF, thereby further minimizing the effects of local within-source heterogeneity on precision. Unfortunately, the unavailability of a neutron source for activation and safety concerns in handling and transporting radioactive materials has limited the use of NAA in forensic science laboratories.

A logical extension of this research would be the application of compositional analysis by TXRF to other polymer types. With slight modifications, the methods developed in this project have been applied successfully to the compositional analysis of other polymer types and plastic materials, including tapes, wire insulation, and synthetic fibers (Buscaglia and Koons, 1998). Analysis of a larger number of samples per product type is necessary to identify the additional discrimination offered by elemental analysis for each particular commodity.

Finally, graphical means for displaying multivariate data warrants further investigation. Star plots have been used successfully to demonstrate differences among compared variables. Additional graphical methods, such as Chernoff's faces, should also be considered. In constructing Chernoff's faces, each variable is plotted as a different facial feature. With multivariate data that is highly variable, the variability between the compared data sets is readily noticed because the resulting faces look different. Differences in facial appearance are easily identified, since this process of sorting is

performed by each person every day. These presentation techniques could assist the forensic expert in classifying or grouping products, and demonstrating the degree of discrimination offered by compositional analysis or any other multivariate data.

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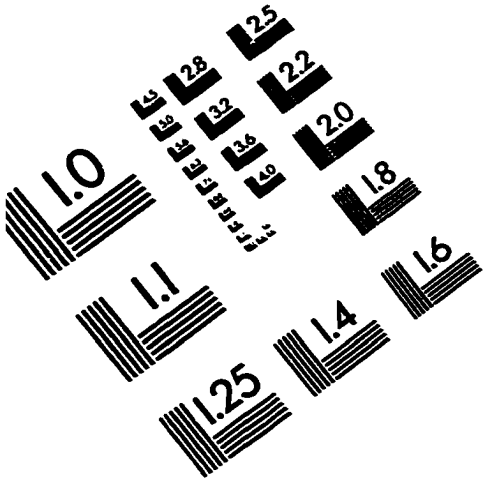
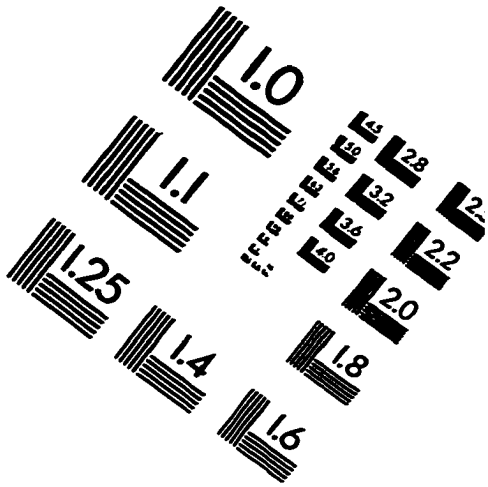
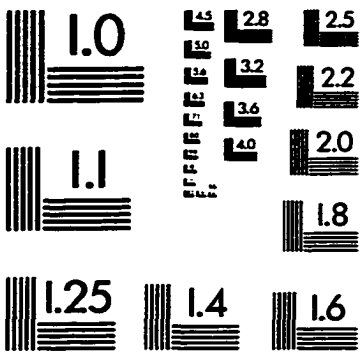
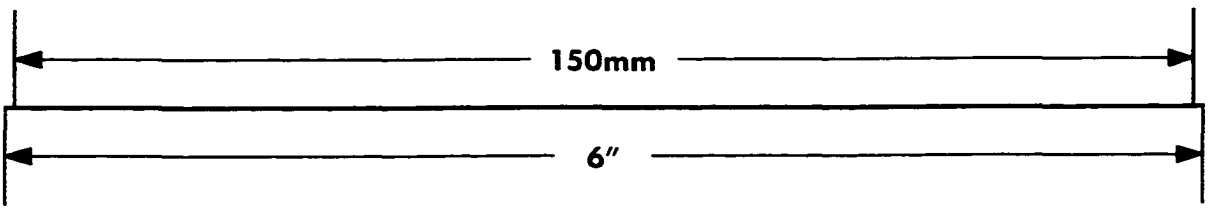
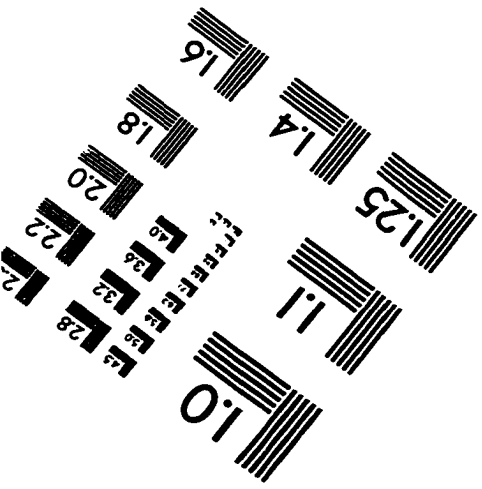
IMAGE EVALUATION TEST TARGET (QA-3)

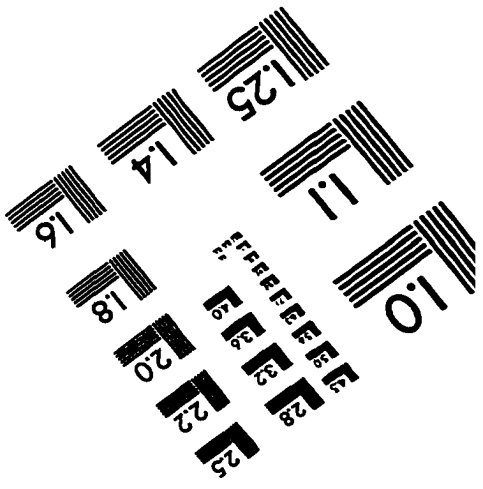












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