BINDER MODIFICATION AND DEVELOPMENT FOR BRIQUETTING STEEL MILL RESIDUES

EERC Final Report

for the period June 2, 1997, through June 30, 1998

Prepared for:

Federal Energy Technology Center
AAD Document Control
U.S. Department of Energy
PO Box 10940, MS 921-143
Pittsburgh, PA 15236

Cooperative Agreement No. DE-FC21-93MC30098 - -53
Performance Monitor: Bob Patton

Prepared by:

Edwin S. Olson

Energy & Environmental Research Center
University of North Dakota
PO Box 9018
Grand Forks, ND 58202-9018

TABLE OF CONTENTS

LIST OF FIGURES	ii
LIST OF TABLES	iii
EXECUTIVE SUMMARY	v
INTRODUCTION	1
BACKGROUND	2
OBJECTIVES	2
ACCOMPLISHMENTS	3
Method Development	3
Hand Press – Standard Molasses–OPC Binder	3
Briquetting – Komarek Press	3
Briquette Evaluation	5
Sand Rammer	5
Odor Tests	6
Binder Studies	6
PVA Binders	6
Other Polymer Binders	8
Clay Binders	9
Fly Ash Binders	10
Clay–Ash Composite Binders	13
Silicate Binders	16
Aluminosilicate Binders	16
Iron Oxide Binders	18
Molasses Additives	18
Ferric Nitrate Additive	18
Aluminum Nitrate Additives	20
Additional Binder Compositions	20
Briquetting	25
Matrix 1	25
Matrix 2	26
Matrix 3	26
Pyrolysis Odor Tests	32
SEM Morphology	32
Molasses Binder Pellet Sample 980429	32
Molasses–Aluminum Nitrate Binder Pellet Sample 980428	32
Molasses–Ferric Nitrate Binder Pellet Sample 980427	36
Molasses–Ferric Nitrate Binder Briquette Sample 980426	36
Molasses–Ferrous Sulfate Binder Pellet Sample 980430	40
REFERENCES	42
SEM DATA SHEETS	Appendix A

LIST OF FIGURES

1	Molasses binder pellet – Image 1	34
2	Molasses binder pellet – Image 2	34
3	Molasses binder pellet – Image 3	35
4	Molasses–aluminum nitrate binder pellet – Image 1	35
5	Molasses–aluminum nitrate binder pellet – Image 2	37
6	Molasses–aluminum nitrate binder pellet – Image 3	37
7	Molasses–ferric nitrate binder pellet – Image 1	38
8	Molasses–ferric nitrate binder – Image 2	38
9	Molasses–ferric nitrate binder briquette – Image 1	39
10	Molasses–ferric nitrate binder briquette – Image 2	39
11	Molasses–ferric nitrate binder briquette – Image 3	40
12	Molasses–ferrous sulfate binder pellet – Image 1	41
13	Molasses–ferrous sulfate binder pellet – Image 2	41
14	Molasses–ferrous sulfate binder pellet – Image 3	42

LIST OF TABLES

1	Fabrication and Strength-Testing Conditions for Steel Mill Residue Pellets	4
2	PVA Binders	6
3	Effect of Water Content on PVA Pellets	7
4	Effect of Binder Concentration	8
5	Other Macromolecular Binders	8
6	Results with Cheto Clay Binders	10
7	Clay-PVA Binders	10
8	Bentonite-Lime Pellets	11
9	Fly Ash-Lime Binders	12
10	Results with Class C Fly Ash Binders	13
11	Results from Ash-Clay Composite Binders	14
12	Effect of Time Delay on Pellet Strength	15
13	Pelletization of Premixed Steel Waste-Binder Mixture	15
14	Silicate Binders	16
15	Results with Aluminosilicate Binder	17
16	Results from Metal Salt Additives to Molasses Binders	19
17	Effect of Water Concentration on Molasses-Metal Salt Binders	19
18	Effect of Metal Salts and Water Concentrations on Molasses-OPC and Molasses-Lime Binders	21
19	Effect of Molasses-Metal Salt Binder Concentrations	22
20	Effect of Aluminum Nitrate and Molasses Concentrations	22
21	Results of Tests with Molasses-Salt Binders of Various Compositions	23

22	Compositions and Conditions for Briquetting Steel Mill Waste	27
23	Drop-Shatter and Compressive Strength Results	29
24	Results of Pyrolysis Odor Tests	33

BINDER MODIFICATION AND DEVELOPMENT FOR BRIQUETTING STEEL MILL RESIDUES

EXECUTIVE SUMMARY

Alternatives to the molasses-ordinary portland cement (OPC) binder as a binder for steel mill residues were extensively investigated. A large number of binder modifications were tested using a hand pelleting apparatus to identify effective compositions. The more effective compositions were evaluated in a series of three briquetting matrices using the Komarek press. Evaluations consisted of drop tests, compression tests of green and cured pellets, and tumble tests of cured pellets. Pyrolysis odor tests were performed to determine the effects of certain compositions on the disagreeable "wet campfire" odor produced during heating of the molasses-OPC briquettes. Several scanning electron microscope (SEM) morphology studies were conducted to examine the nature of structures formed during pelleting or briquetting.

Several tacky polymers were investigated as alternatives to the molasses compositions. A water-soluble form of polyvinyl alcohol (PVA) gave high-strength pellets and briquettes at low concentrations of binder (1%). At 0.5% PVA, the pellets failed the drop test, but compressive strengths were adequate for 0.75% PVA. Water was added to dilute the PVA and allow better coating of the steel residues. Maximum strength was achieved at 4% water. Variation in the OPC and lime concentrations had little effect on the green strength, but increased the cured strength at higher concentrations (8%). Briquettes made with the PVA binder were of comparably high strength. Other macromolecular binders were less successful. Pyrolysis odor tests showed that the PVA pellets also generate a wet campfire odor. Cost of the PVA would be high; thus, further work with alternative organic binders was not pursued.

Clay binders were investigated using two types of clays (montmorillonite and bentonite) in various compositions, excluding molasses. The montmorillonite compositions gave poor green strengths and failed drop tests. Cured strengths were also relatively poor. Sodium bentonite-lime compositions passed the drop tests but gave poor cured compressive strengths. A clay-PVA binder composition gave a much stronger pellet.

Binders were prepared from two types of fly ashes. A fly ash obtained from Heckett Multiserve was tested with and without an alkali pretreatment. The fly ash-lime binders all failed the drop test and gave poor cured strengths. A Class C fly ash (Corrette) was also tested in mixtures with lime, but again all pellets failed the drop test. Cured strengths were higher but not comparable to those for the molasses-lime binders. Clays were combined with the two ashes and the composite binders tested. These all passed the drop test and developed moderate cured strengths. When the Class C ash was pretreated with sodium hydroxide and mixed with lime and clay, the resulting pellets were substantially stronger, but still not comparable to those formed with molasses-lime binders.

Several other inorganic binders were investigated in combination with lime and OPC. Silicate binders were unsuccessful. Aluminosilicate binders gave better results, with survival of

the drop test and moderate compressive strengths for cured pellets. However, the results were inferior to those from pelleting with molasses binders, and the cost of the aluminosilicate binders may be prohibitive.

The addition of salts of trivalent ions, such as aluminum and iron, to the molasses results in a dramatic decrease in the viscosity of the molasses. Consequently, the binder is able to coat or wet the steel waste much more effectively and much stronger binding occurs. Pellets produced using aluminum or ferric nitrate were about 30% stronger. Less molasses-salt binder can be used to produce the same strength as molasses alone. Other salts, such as sulfates and chlorides, were also effective in increasing the binder strength of the molasses.

Pyrolysis odor tests demonstrated that eliminating the calcium from the binder changes the wet campfire odor to be more like burned sugar and burned wood. Further addition of nitrate salts reduces the odor significantly at lower temperatures, presumably by oxidizing the odor-producing constituents.

Many kinds of component particles were identified in the SEM images of the modified molasses pellets and briquettes. Characterization of the bonding interactions were not conclusive, however, since no definite crystalline structures formed between the particles. Instead, the molasses appears to be evenly distributed around the particles. Subsequent testing will determine whether these binder systems generate noxious odors during devolatilization in the kind of reducing atmosphere that is found in the top section of a blast furnace.

Briquetting tests with the Komarek press showed that the fly ash, hydrated lime, and sodium silicate binder systems are ineffective at providing tumble resistance that approaches that of the baseline (OPC, molasses, water). Further, without the aid of a significant level of moisture, none of these binder systems provide the necessary green drop-shatter resistance that the molasses imparts. However, the molasses and aluminum/iron nitrate/sulfate salt binder systems evaluated in Matrix 3 are clearly superior to the baseline binder.

The modified molasses-salt binders represent a significant advance in binder technology in increasing the strength and abrasion resistance of briquettes. The potential for odor control needs further investigation. Additional benefits that can be obtained from these binder systems are 1) reduction in slag owing to elimination of inerts such as portland cement or hydrated lime, 2) reduction in furnace fuel requirements owing to lower heat burden for melting inert binder components, 3) simplification of the briquetting process, and 4) reduction of material handling. The cost of the additives has not been evaluated, but utilizing a small unit to convert steel waste to iron nitrate or sulfate might be considered.

BINDER MODIFICATION AND DEVELOPMENT FOR BRIQUETTING STEEL MILL RESIDUES

INTRODUCTION

Blast furnace operations for steel production generate a diverse array of residues. The metallic residues include blast furnace dust, mill scale, blast oxygen furnace (BOF) precipitator dust, BOF metallic fines (metallic recovery from slag), and kish metallics (desulfurization waste materials). Cost recovery, energy efficiency, and environmental regulations are current driving factors for companies in developed countries to deal with steel mill residues acceptably.

The diverse steel mill residues need to be agglomerated before being recycled to the blast furnace. Disc pelletizers, pelletizer or extrusion die machines, and briquetting roll presses have been used. Using a double-roll briquetting press with and without binder to agglomerate the steel mill residues is currently an economically and environmentally sound practice in developed countries. In the absence of a binder, very high temperatures are used (e.g., 1470°–1650°F). Several papers attest to the substantial interest in agglomeration technology, particularly briquetting with binders via roll presses (Aumônier, 1995) and extrusion with binders (Steele, 1993) for recycling steel residues.

Commonly used binders for briquetting steel mill residues include molasses–hydrated lime (portlandite [$\text{Ca}(\text{OH})_2$]), lignosulfonate– $\text{Ca}(\text{OH})_2$, bentonite–cement, sodium silicate–cement, and cement–molasses, to name a few. Good green strength is required from the selected binder for the briquettes to survive initial handling and transport. In addition, the binder needs to provide subsequent high-temperature strength in order for the briquettes to withstand the elevated temperatures in the upper section of the blast furnace. The binder selected must be cost-effective, be applied at relatively low concentrations, exhibit acceptable blending characteristics, have benign qualities during handling and after application, and be in ready supply.

Several new binders or binder mixtures for use with iron/steel mill residues have appeared in the patent literature over the last 25 years. These include polyvinyl pyrrolidone, phenolaldehyde resin, starch plus polyvinyl alcohol (or with an acrylic or a gum), urethane (or epoxy resin), metal phosphate, and silica/clay. A major disincentive in using most of these binders is the cost of application or, in the case of the organic binders, their failure at high temperatures. For aggregating steel mill residues, Lambert (1995) has delineated the key problems of some past and currently used binders as well as the criteria for briquette strength relating to steel mill operations.

Fly ash and other waste products from combustion of fossil fuel are a potential resource for binder materials, owing to their content of aluminum and silicon and known pozzolanic activity. They interact strongly with cement and can replace a portion of cement, and they can provide a basis for their own cementitious reactions. Methods for accelerating these reactions require further study.

The search for the optimum binder for briquetting steel mill residues continues, motivated by cost and environmental pollution factors. Obnoxious odors ("wet campfire" smell) result from the use of effective molasses—ordinary portland cement (OPC) binders in the current practice. Increasing competition, more stringent regulations, and the drive for improved financial return on all aspects of operating a steel mill have focused attention on the need for an improved means of agglomerating waste by-products. Briquetting waste materials is an effective and efficient means of producing lump products from fine solid feedstocks which otherwise could contaminate the environment.

BACKGROUND

Heckett MultiServ has expressed a keen interest in the development and application of a modified or new binder in place of its existing binder for briquetting BOF and allied residues into pillow-shaped briquettes with dimensions of $3 \times 2.5 \times 1.25$ in. The existing binder consists of molasses (8 wt%)—portland cement (8 wt%) or molasses (4 wt%)—whey (4 wt%)—portland cement (8 wt%). The major problems of these two binder mixtures are the dustiness of the briquettes, the insufficient strength of the briquettes at high temperatures (e.g., the molasses—whey—cement-based briquettes break up at about $570^{\circ}\text{--}750^{\circ}\text{F}$), and the noxious odor emanating from the cooling towers following the addition of the briquettes to the blast furnace. It is believed that this odor is associated with the molasses component of the binder. The lack of binder strength is due to the weakness of the bond between the inorganic and organic components at temperatures above about 570°F .

Heckett MultiServ in its many plants worldwide currently handles hundreds of thousands of tons of blast furnace residues. The economic potential for modifying or developing a suitable binder is critical, as are the environmental implications. The goal is to produce an acceptable binder with a cost of US\$12–\$15 per metric ton of briquetted product, i.e., no more than the cost of adding molasses, whey, and cement. The briquetted steel mill residue must have a suitable green strength and a high-temperature strength, say, at $700^{\circ}\text{--}800^{\circ}\text{F}$, of about 50% of its cured ambient strength. No odor should result from use of the binder after transfer of the briquettes to the blast furnace. An understanding of the binder mechanism is also required for improving the binder and the conditions of application.

OBJECTIVES

The objectives were to investigate the following:

- Alternative components to molasses and or whey blended with portland cement as a binder for a mixture of steel mill residues. The modified binder is to yield briquettes of an acceptable green strength to withstand a 2-ft drop, durability to moisture, adequate strength at high temperatures ($700^{\circ}\text{--}800^{\circ}\text{F}$) (i.e., 50% of the cured strength at ambient temperatures). In addition, the modified binder must not generate any noxious odors as

a result of briquette use. The cost of the binder components should not exceed US\$15 per metric ton of briquetted product.

- A new noncement binder meeting the above criteria.
- The mechanism of binder action.

ACCOMPLISHMENTS

Method Development

Hand Press – Standard Molasses–OPC Binder

Tests were undertaken to develop a “standard” pellet based on the supplied blended steel mill residue and molasses–cement binder. Pellets were made by blending steel mill residues, 1.6 g of sugarcane molasses, and 1.6 g of OPC in a Simplimet 11 (Buehler Ltd.) mixer and pressing the mixture at about 7000 psi to generate pellets 1.25 in. in diameter and about 1 in. long. The pellets were tested for green as well as cured strength by dropping them in air from a height of 42 in. onto a steel plate and by determining compressive strengths.

Seven conditions of mixing were tested, from room temperature to 140°F. Various pressing conditions were applied to the mixture, again from room temperature to 140°F, and pressing immediately, after 1 minute, or after 3 minutes. Conditions and test results are presented in Table 1.

Satisfactory pellets were produced at all temperatures using the pelleting press, but the heated mixture and mold gave somewhat stronger pellets. Generally, the drop tests resulted in little damage or cracking to the standard binder pellets.

Briquetting – Komarek Press

A series of three briquetting matrices was performed to allow evaluation of the most promising binders identified during bench-scale pelleting tests. A total of 35 briquetting tests was performed, with the conditions as specified in the tables in the Briquetting section. Binder concentrations are presented in the tables on a steel mill waste basis.

Some preliminary tests were performed using the polyvinyl alcohol (PVA)–OPC binder. This binder gave briquettes with acceptable drop test results, and compressive strengths were comparable to those obtained using the molasses–OPC binder. However, the briquettes were not fully evaluated with tumble tests, since the PVA binder gave poor results in the pyrolysis odor test.

TABLE 1

Fabrication and Strength-Testing Conditions for Steel Mill Residue Pellets

Mixing Conditions	Pressing Conditions	Drop Test	Compressive Strength (psi)	
			Green	Cured
Residue and Molasses at Room Temperature	Mold at 75°F	All pellets passed.	32	242
			29	248
			32	242
			Avg. = 31	Avg. = 244
Residue and OPC at 75°F – Hot Molasses (100°F) Added and Mixed Thoroughly	Mold at 100°F	All pellets passed.	38	286
			39	273
			38	273
			Avg. = 38	Avg. = 277

Briquetting Tests 1 to 15 were performed between October 3 and 7, 1997; Tests 16 to 24 were performed on December 17, 1997; and Tests 25 to 35 were performed between May 13 and 22, 1998. Tests 1 to 15 and 25 to 35 were performed using -16-mesh steel mill waste, and Tests 16 to 24 used - $\frac{1}{8}$ -in. steel mill waste. Briquettes were produced using a Komarek Model B100R cantilever roll press equipped with 5-in.-diameter by 2-in.-wide rolls. The briquetter rolls contain 18 pockets and produce briquettes with nominal dimensions of $1\frac{1}{4} \times \frac{7}{8} \times \frac{1}{2}$ in. The briquetter is equipped with a steel chute to collect briquettes directly at the roll discharge.

Briefly, for each test, between 3000 and 4000 g of steel mill waste was used. The procedures for binder addition differed between matrices. For Matrices 1 (Tests 1 to 15) and 2 (Tests 16 to 24), the dry binder components (Corrette fly ash, hydrated lime, sodium bentonite, Heckett Multiserv fly ash, and portland cement) were added first to the dry steel mill waste, with water and American Crystal Sugar beet molasses (preheated to 135°F) than added. For Matrix 3 (Tests 25 to 35), the dry binder components (ferric nitrate, aluminum nitrate, ferrous sulfate, and ferric sulfate) were added to the Heckett Multiserv molasses. The molasses was preheated to 100°F prior to addition to the steel mill waste. Mixing of the steel mill waste and binder components was achieved using a hand-held kitchen-type blender. All wet binder components were incrementally added to facilitate distribution on the steel mill waste.

Tests 1 to 3 functioned as shakedown tests to determine suitable operating conditions with the B100R briquetter. For subsequent tests, a roll speed of 3 rpm (25% of maximum speed) and a roll separation force of 90 kN (90% of maximum) were used. To attain uniformity of briquetting conditions from test to test, an attempt was made to achieve a nominal roll torque of 950 700 ft-lb_r. This was accomplished by adjusting the screw feed rate. During shakedown, it was determined that the roll timing was off slightly, but this was considered to be insignificant for the purpose of comparative briquette evaluations.

For each test, eight or nine briquettes were collected from the discharge chute of the briquetter and were then used for green strength determinations. The remaining briquettes were collected in a 5-gal plastic pail after a vertical drop of about 16 in. The collected briquettes were screened with ¼-in. sieve to remove flashing and then spread in a single layer on a flat surface to dry at the prevailing conditions of the laboratory.

Briquette Evaluation

Briquette evaluations included drop-shatter, compressive strength, and tumble (abrasion resistance) testing. For drop-shatter testing, five or six briquettes were sequentially dropped through a 34-in.-long, 1.5-in.-ID steel pipe, oriented with the bottom opening 8 in. from the concrete floor. The briquettes were given a pass or fail ranking. A pass rating was given to briquettes that largely remained intact with only minor chipping or deformation. A fail rating was given to briquettes that broke into several substantially smaller fragments or disintegrated.

For compressive strength testing, a briquette was stressed between a stationary block and a moveable platen on a spring-type load tester. A dial indicator measured the deflection of the calibrated spring on the load tester. The briquette was stressed until deflection of the spring stopped. This usually indicated the point at which the briquette fractured or catastrophically failed. The maximum deflection, as measured by the dial indicator, was converted to a force using a calibration curve for the spring. A normalized compressive strength was obtained by dividing the force value by the thickness of the briquette as measured by a dial caliper. The average compressive strength value for the three briquettes is reported.

Tumble testing was performed in a 7.8-in.-long × 7.8-in.-diameter stainless steel vessel with a wall thickness of ¼ in. The tumbling vessel was outfitted with three 1-in.-wide lifters to increase tumbling intensity. For this procedure, six briquettes were weighed and then charged to the tumbling vessel. The vessel was turned on rollers at 40 rpm for 20 minutes. The proper tumbling conditions were determined from shakedown tests performed using briquettes from Test 14 of the first matrix. The contents of the tumbling vessel was screened with a ¼-in. sieve and the material retained on the screen weighed to determine the percent recovery.

Drop-shatter and compressive strength testing were performed on green briquettes, typically between 1 and 3 minutes after the briquette was produced. Compressive strength and tumble testing were performed on briquettes that had been air-dried for 2 and 7 days.

Sand Rammer

Testing with the sand rammer using the molasses-OPC binder for the steel waste mixture gave sporadic results. Nearly all the large pellets exhibited cracks or deformed shapes. It was difficult to judge exactly how much water to add, and limited testing with other binders gave similarly poor results. Although Heckett Multiserv has utilized this test method, we found it difficult to evaluate the results of our tests. The pellets pressed in the hand press and briquettes were superior in quality, and evaluation of the results could be done by compression testing, which gives a value for the strength.

Odor Tests

Pyrolysis odor tests were conducted with the molasses binder to determine the basis for potential environmental problems resulting from use of various binders. A 7-g sample of a cured pellet prepared with molasses-OPC was heated in a tube furnace from ambient temperature to 600°C. At about 300°C, the wet campfire odor that is characteristic of the molasses briquette pyrolysis was considerable. The evolving gases were bubbled through water and a water sample stored in a freezer. The water sample continued to evolve the odor for some time, but after a day the pellet sample emitted a strong freshly poured cement odor.

Binder Studies

PVA Binders

After several unsuccessful attempts to dissolve PVA in water, the more soluble hydrolyzed PVA (Elmer's glue) was used for the macromolecular binder. The binder was prepared by diluting 4 g of the glue (moisture content = 59 wt%) with 8 g of water.

The steel residue (20 g), diluted glue (1.2 or 1.6 g), and OPC (1.2 or 1.6 g) and, on occasion, Ca(OH)_2 , were thoroughly mixed and the mixture pressed in the Simplimet hand press. As previously, the pressure was about 7000 psi. The 1.25-in.-diameter pellets were tested for green strength by dropping them down a tube in air from a height of 42 in. onto a steel plate. The results are given in Table 2.

TABLE 2

PVA Binders

PVA (wt%)	OPC (wt%)	Ca(OH)_2 (wt%)	H_2O (wt%)	Drop Test	Compressive Strength (psi)	
					Green	Cured
1	4	4	6.4	Passed	45	172
1	8	0	6.4	Passed	51	204
1	4	0	6.4	Passed	41	159
1	0	4	6.4	Passed	44	178
1	0	8	6.4	Passed	51	204
1	4	4 (CaO)	6.4	Passed	48	210

Pellets prepared with 4% and 8% Ca(OH)_2 and with 4% and 8% OPC were nearly comparable in strength. Use of CaO gave a high-strength pellet.

Comparison of the data in Tables 1 and 2 shows that the green strength of the pellets produced with Elmer's glue and cement was markedly greater than the green strength of the standard molasses-OPC pellets. The Elmer's glue-produced pellets also exhibited a very good appearance.

The effect of water content on the strength of the pellets was investigated by using a different dilution of the PVA solution. The results are given in Table 3. The 2% water content was too low to adequately wet the particles, and not much strength developed during curing. Maximum strength was obtained at the 4% water content.

Since the 1% PVA binder concentration used in the previous tests would likely result in costs exceeding \$20/ton of product, lower concentrations of the binder were investigated. All of the pellets prepared with 0.75% PVA passed the drop test and developed acceptable cured strengths. When the concentration was reduced to 0.5% PVA, the pellets exhibited cracks in the drop test (Table 4). Green strength was not evaluated for this concentration.

A pyrolysis odor test was conducted to determine whether an environmental problem is likely to occur as it does with the molasses binder. A 7-g sample of a cured pellet prepared with PVA and Ca(OH)_2 was heated in a tube furnace from ambient temperature to 500°C. At about 300°C, the wet campfire odor that is characteristic of the molasses briquette pyrolysis was considerable. The evolving gases were bubbled through water and a water sample stored in a freezer. Water continued to evolve the odor for some time, but after a day, no odor was observed. The frozen sample was thawed, and the odor had changed to a slight aldehyde odor and later to a strong freshly poured cement odor.

TABLE 3

Effect of Water Content on PVA Pellets

PVA (wt%)	OPC (wt%)	Ca(OH)_2 (wt%)	H_2O (wt%)	Drop Test	Compressive Strength (psi)	
					Green	Cured
1	8	0	6.4	Passed	51	204
1	0	8	6.4	Passed	51	204
1	8	0	4	Passed	54	217
1	0	8	4	Passed	67	229
1	8	0	2	Failed	50	89
1	0	8	2	Failed	51	95

TABLE 4

Effect of Binder Concentration

PVA (wt%)	OPC (wt%)	Ca(OH) ₂ (wt%)	H ₂ O (wt%)	Drop Test	Compressive Strength (psi)	
					Green	Cured
0.75	4	4	5	P	43	165
0.75	8	0	5	P	48	172
0.75	0	8	4	P	57	185
0.75	0	4	4	P	54	178
0.5	0	8	4	Cracked	NA ¹	NA

¹ Not applicable*Other Polymer Binders*

Other macromolecular binders were investigated (Table 5). These include polyvinylpyrrolidone-co-polyvinyl acetate (PVPcoPVA), polyethylene oxide, polyacrylamide, and a water-soluble low-sulfur lignin preparation, all at 1 wt% concentrations in the pellets. The PVPcoPVA binder with Ca(OH)₂ and with OPC gave pellets that passed the drop test, but did not develop significant cured strength. A second series of tests did not pass the drop test. Other binders (polyethylene oxide [PEO], polyacrylamide, and lignin) with 8% Ca(OH)₂ failed the drop test and were not further investigated with OPC.

TABLE 5

Other Macromolecular Binders

Organic Binder (wt%)		Cheto Clay (wt%)	OPC (wt%)	Ca(OH) ₂ (wt%)	H ₂ O (wt%)	Drop Test	Compressive Strength (psi)	
							Green	Cured
PVPcoPVA	1	0	8	0	5	P	25	89
PVPcoPVA	1	0	0	8	5	P	27	95
PVPcoPVA	1	0	0	8	5	F	NA	NA
PVPcoPVA	1	0	8	0	5	F	NA	NA
PEO	1	0	0	8	5	F	NA	NA
Polyacrylamide	1	0	0	8	5	F	NA	NA
Lignin	1	0	0	8	5	F	NA	NA

Clay Binders

Clay minerals can be divided into two general categories, the swelling clays and the nonswelling clays. Swelling clays are often referred to as smectites, which is a general term for all the swelling clays. Of the smectites, montmorillonite is probably the most common form. Nonswelling clays do not have a general group name but include kaolinite, illite, and chlorite as commonly occurring forms. The swelling-versus-nonswelling mechanism involves water, which in the smectites is able to move in and out of the sheetlike layers without destroying the clay mineral structure. Nonswelling clays collapse upon dehydration, which is not a reversible reaction with the exception of a few species that hold water in columns rather than between their sheetlike layers.

Three properties exhibited by smectite clays allow these materials to be considered as a potential binding medium. The first has to do with the "balling," or ability of smectites to stick to each other, which is evident to anyone who has ever stepped in mud. The ability of self-adherence has to do partly with the size and shape of the particles, which are extremely small ($<2\ \mu\text{m}$), platelike crystals. The platelike crystals end up in somewhat random orientation and form a meshlike grid. The second property is related to a slight electrical imbalance often exhibited on the surface of the crystals. This helps particles to adhere to each other as well as to form weak bonds to the materials to be bonded. The third property has to do with the chemistry of the clay minerals, which contain mostly SiO_2 and Al_2O_3 . These components become available to react with other compounds at or about a pH of 9. When $\text{Ca}(\text{OH})_2$ is added to swelling clays, the SiO_2 and Al_2O_3 react with the $\text{Ca}(\text{OH})_2$ and form calcium aluminum silicate hydrates, which are often the same or very similar to the cementitious phases found in portland cement concrete and are known to have good long-term binding properties.

Cheto Clay Binders

Cheto (montmorillonite) clay was tested as a binder for steel waste pellets. The cheto clay is a very sticky clay, and it was hoped that it would provide some green strength to the briquettes. However, this work established that the clay by itself or with sodium aluminate (NaAlO_2) was not an effective binder (Table 6). With addition of $\text{Ca}(\text{OH})_2$, the pellets passed the drop test and developed some strength on curing, but were far inferior to those formed with the molasses binder.

Mixed Clay-PVA Binders

When the PVA binder was utilized (1%) with a montmorillonite (cheto) clay (8%), the pellet passed the drop test (Table 7) and developed considerable cured strength (178 psi). This binder-clay combination represents an alternative to the patented binder mixtures of PVA with $\text{Ca}(\text{OH})_2$ and with OPC. The mechanism of bond formation may involve reaction of the PVA alcohol groups with ions and layers of the clay structure similar to those reactions occurring with the calcium in OPC or $\text{Ca}(\text{OH})_2$.

TABLE 6

Results with Cheto Clay Binders

Cheto Clay (wt%)	OPC (wt%)	NaAlO ₂ (wt%)	Ca(OH) ₂ (wt%)	H ₂ O (wt%)	Drop Test	Compressive Strength (psi)	
						Green	Cured
8	0	0	0	4	Failed	—	—
6.3	0	0	6.30	8	Passed	13	89
6.1	0	0	6.1	11	Passed	32	105
6.2	0	0	3.1	12	Passed	—	108
5.4	0	1.8	0	3.5	Failed (d)	—	—
3.5	3.5	1.8	0	3.5	Failed (t)	—	—
3.5	0	1.8	3.5	3.5	Failed (d)	—	—

d = duplicate; t = triplicate.

TABLE 7

Clay-PVA Binders

Organic Binder (wt%)		Cheto Clay (wt%)	OPC (wt%)	Ca(OH) ₂ (wt%)	H ₂ O (wt%)	Drop Test	Compressive Strength (psi)	
							Green	Cured
PVA	0.75	8	0	0	4	P	89	178
PVA	0	8	0	0	4	F	—	—

Sodium Bentonite Binders

Another clay (bentonite) was tested in the sodium form as a binder for steel waste pellets. The sodium bentonite was ground to -60-mesh size and pelleted and tested as described above. Results are given in Table 8. Some success was achieved in the drop tests, but none of the pellets exhibited cured strengths comparable to that of pellets made with the molasses binders.

*Fly Ash Binders**Fly Ash-Lime Binders*

A fly ash sample (HM ash) was obtained from Heckett Multiserv that would be representative of locally obtained fly ash. The analysis showed 21.3% Al₂O₃, 6.08% CaO, and 0.70% Na₂O. Ash was mixed with steel waste and Ca(OH)₂ and minimal water, hand pelletized,

TABLE 8

Bentonite-Lime Pellets				
Additive (g)	Binder (g)	Water (g)	Drop Test	Cured Compressive Strength (psi)
Lime (0.8)	Sod. Bentonite (1.6)	3	Passed	—
Lime (0.8)	Sod. Bentonite (1.6)	3	Passed	89
Lime (0.8)	Sod. Bentonite (0.8)	3	Passed	—
Lime (0.8)	Sod. Bentonite (0.8)	3	Passed	115
Lime (0.4)	Sod. Bentonite (0.8)	3	Passed	—
Lime (0.4)	Sod. Bentonite (0.8)	3	Passed	76
Lime (0.4)	Sod. Bentonite (0.8)	2	Passed	—
Lime (0.4)	Sod. Bentonite (0.8)	2	Passed	89
OPC (0.8)	Sod. Bentonite (0.4)	2	Passed	—
OPC (0.8)	Sod. Bentonite (0.4)	2	Passed	96

1.6 g = 8 wt%, 0.8 g = 4 wt%, 0.4 g = 2 wt%.

and tested as described above (Table 9). The pellets from the unprocessed ash failed the drop test.

U.S. Patent 5,565,028 describes the addition of alkali to fly ash to improve setting times of cementitious ash. Sodium hydroxide (NaOH) was added to the HM ash-steel waste mixture to help solubilize silicate for reaction with $\text{Ca}(\text{OH})_2$. These pellets also failed the drop test. Some strength did develop on curing these pellets.

Pretreatment of the HM ash with NaOH was attempted to solubilize some of the aluminate and silicate, so that on mixture with the $\text{Ca}(\text{OH})_2$, a cementitious reaction could take place more quickly. Thus, a binder was prepared by mixing 20 g of HM ash with 14 g of NaOH in 20 g of water and heating to 95°C overnight (Binder 1). The binder paste was then mixed with steel

TABLE 9

Fly Ash–Lime Binders

Additive (g)	Binder (g)	Water (g)	Drop Test	Cured Compressive Strength (psi)
None	HM Ash (1.6)	3	Failed (duplicate)	—
Lime (0.8)	HM Ash (1.6)	3	Failed ¹	—
Lime (0.8)	HM Ash (1.6)	3	Failed ¹	82
Lime (0.8)	HM Ash (1.6)	10% NaOH 3	Failed ¹	—
Lime (0.8)	HM Ash (1.6)	10% NaOH 3	Failed ¹	89
Lime (0.8)	Binder I (1.6)	3	Failed ¹	—
Lime (0.8)	Binder I (1.6)	3	Failed ¹	89
Lime (0.8)	Binder II (1.6)	3	Passed	—
Lime (0.8)	Binder II (1.6)	3	Failed ¹	—
None	Binder III (1.6)	—	Failed (duplicate)	—
Water (0.3)	Binder III (1.6)	0.3	Failed (duplicate)	—

1.6 g = 8 wt%, 0.8 g = 4 wt%, 0.4 g = 2 wt%.

¹ Pellet broke into two pieces.

waste and the mixture pressed into pellets and tested. However, the pellets again failed the drop test. Binder II was prepared by mixing 8 g of fly ash with 10 g of 20% NaOH and heating at 100°F for 1 hour. Binder III was prepared by heating 14 g of ash with 2 g of Ca(OH)₂ and 6.4 g of 20% NaOH in a digestion bomb at 100°C for 1 hour. None of these binders gave pellets with acceptable green strengths.

Class C Fly Ash Binders

A Class C fly ash (Corrette) was also ineffective by itself. An alkali-activated binder (Binder AI) was prepared by mixing 20 g of class C ash with a solution of 14 g NaOH in 20 g water. The paste was heated at 95°C overnight. The residue was heated at 450°C for 3 hours. The residue was ground to -60 mesh size and pelleted with steel waste as described above. Results are shown in Table 10.

When mixed with water and $\text{Ca}(\text{OH})_2$, the calcined fly ash mixture (Binder AI) rapidly sets as a result of cementitious reactions. The results were compared with results for the same fly ash without calcining. The results from the two tests were similar. The pellets did not disintegrate on impact, but cracked in half. Considerable strength developed on curing the uncracked pellets.

TABLE 10

Results with Class C Fly Ash Binders

Binder AI (wt%)	Class C Fly Ash (wt%)	$\text{Ca}(\text{OH})_2$ (wt%)	H_2O (wt%)	Drop Test	Cured Compressive Strength (psi)
8	0	4	2	Failed (d)	—
8	0	4	3	Failed (d)	—
8	0	4	2	—	146
6.5	0	3.3	8	Cracked (d)	—
6.3	0	3.2	11	Cracked (t)	146
0	6.3	6.3	8	Cracked (d)	—
0	6.1	6.1	11	Cracked (d)	—
0	6.2	3.1	12	—	153
0	6.2	3.1	12 (10% NaOH)	—	159

d = duplicate; t = triplicate.

Clay-Ash Composite Binders

Clay-Ash mixtures

Sodium bentonite and cheto clay were ground to -60-mesh size and used as a binder for steel waste. Corrette (Class C) ash was used as received. Pellets were prepared and tested as above.

Activated Ash-Clay Binders

Two batches of a binder were prepared by mixing 24 g of Class C ash with a solution of 20 g of 1N NaOH in water. The slurry was thoroughly mixed to form a paste. Upon stirring, the control slurry batch (not mixed with steel waste) thickened and became solid after overnight standing, retaining 83% of the added water. Results are given in Table 11. Strengths were not comparable with those for the molasses binders.

TABLE 11

Results from Ash-Clay Composite Binders

Clay (g)	Ash (g)	Water (g)	Drop Test	Cured Compressive Strength (psi)
Sodium Bentonite (0.4) + Lime (0.8)	Class C ash (1.6)	(3)	Passed	—
Sodium Bentonite (0.4) + Lime (0.8)	Class C ash (1.6)	(3)	Passed	146
Sodium Bentonite (0.4) + Lime (0.8)	Class C ash (1.6)	(2)	Passed	—
Sodium Bentonite (0.4) + Lime (0.8)	Class C ash (1.6)	(2)	Passed	153
Cheto clay (0.4) + Lime(0.8)	Class C ash (1.6)	(2)	Passed	115
Cheto clay (0.4) + Lime (0.8)	Class C ash (1.6)	(2)	Passed	—

1.6 g = 8 wt%, 0.8 g = 4 wt%, 0.4 g = 2 wt%.

The second batch was mixed after certain time intervals with steel waste to determine the effect of pretreatment time on the strength. As-received steel waste was sized to obtain particles less than $\frac{1}{8}$ in. in size. To 20 g of steel waste fines, 0.8 g of Ca(OH)_2 and 0.6 g of sodium bentonite were added and thoroughly mixed. To the mixture, 4.4 g of the binder paste was added and thoroughly mixed after 1-hour time intervals (Table 12). Pellet strength decreased with pretreatment time in this experiment. This implies that the cementitious reactions may occur fairly rapidly when NaOH is added to the ash to solubilize silicate.

TABLE 12

Effect of Time Delay on Pellet Strength

Time (hr)	Steel Waste (g)	Ca(OH)_2 (g)	Sodium Bentonite (g)	Binder ¹ (g)	Compressive Strength (psi)
1	20	0.8	0.6	4.4	227
2	20	0.8	0.6	4.4	204
3	20	0.8	0.6	4.4	191
4	20	0.8	0.6	4.4	159

1.6 g = 8 wt%, 0.8 g = 4 wt%, 0.4 g = 2 wt%.

¹ Paste of Class C ash in 1N NaOH solution.

Earlier briquetting results indicated that steel waste–binder mixtures that were not briquetted immediately seemed to give strong briquettes after standing. To further test this hypothesis, the binder paste was added to thoroughly mixed steel waste (200 g), Ca(OH)_2 (8 g), and sodium bentonite (6 g), and the mixture was pelletized after several time intervals to determine the effect of hold time on the eventual strength (Table 13). Hold time did not seem to have much effect in this test.

TABLE 13

Pelletization of Premixed Steel Waste–Binder Mixture

Time (hr)	Mixture (g)	Compressive Strength (psi)
1	25.8	204
2	25.8	197
3	25.8	210
16	25.8	216

Silicate Binders

Silicate binder was prepared by mixing 94 g of silicic acid (85.5% SiO₂, 14.5% water) with 120 g of water and blending at top speed for 15 minutes (Binder S1). The results were compared with pellets prepared using purchased sodium silicate solution and silicic acid (Table 14).

TABLE 14

Silicate Binders

Additive (g)	Binder (g)	Water (g)	Drop Test
Lime (1.6)	Sodium Silicate (0.5)	—	Failed
Lime (1.6)	Sodium Silicate (0.5)	—	Failed
Lime (1.5)	Sodium Silicate (0.5)	0.5	Failed
Lime (1.5)	Sodium Silicate (0.5)	0.5	Failed
Lime (1.6)	Silicic acid (0.8)	2	Failed ¹
Lime (1.6)	Silicic acid (0.8)	2	Failed ₁
Lime (1.6)	Binder S1 (2.14)	—	Failed ¹
Lime (1.6)	Binder S1 (2.14)	—	Failed ¹

Sodium Silicate used is 14 wt% NaOH and 27 wt% SiO₂.

¹ Pellet broke into two pieces.

Aluminosilicate Binders

A series of pellets was prepared using variations of an aluminosilicate gel as the binder. This material was known from our previous catalyst work to give a very hard material when dried, which is suitable for binding zeolites and clays. The gel is prepared from aluminum chloride and sodium silicate. The dried gel was mixed with Ca(OH)₂, water, and steel waste. The

Ca(OH)_2 is needed to cement the gel and steel waste together. The pellets survived the drop test, but had a mediocre compressive strength compared to the molasses-OPC standard.

When the wet aluminosilicate gel (paste with a solids dry weight content of 10.7%) was used with Ca(OH)_2 and steel waste and water was removed to bring water content down to 6 wt% in the mixture, the resulting pellets were stronger (Table 15), but not in the class of the molasses standard. OPC mixed with the aluminosilicate gave less strong pellets, indicating that the Ca(OH)_2 may be undergoing some cementitious reactions with the aluminosilicates.

When the wet gel and Ca(OH)_2 were premixed and dried to lower water content, then mixed with the steel waste, again pellets with mediocre strength were obtained (Table 15). All these pellets had good immediate green strength.

TABLE 15

Results with Aluminosilicate Binder

Aluminosilicate Gel (g)	Additive (g)	Steel Waste (g)	Method	Compressive Strength (psi)
1.6	Lime (1.6)	20	Add dry gel, then 6 wt% H_2O	165
1.6	Lime (1.6)	20	Add dry gel, then 6 wt% H_2O	156
1.6	Lime (1.6)	20	Add wet gel containing 8 wt% H_2O , then remove H_2O down to 6 wt%	188
1.6	Lime (1.6)	20	Add wet gel containing 8 wt% H_2O , then remove H_2O down to 6 wt%	194
1.6	OPC (1.6)	20	Add wet gel containing 8 wt% H_2O , then remove H_2O down to 6 wt%	169
1.6	OPC (1.6)	20	Add wet gel containing 8 wt% H_2O , then remove H_2O down to 6 wt%	173
1.6	Lime (1.6)	20	Mix wet gel and Ca(OH)_2 , dry to remove excess H_2O , mix with waste	182
1.6	Lime (1.6)	20	Mix wet gel and Ca(OH)_2 , dry to remove excess H_2O , mix with waste	178

Iron Oxide Binders

Iron hydroxyoxides or hydroxide hydrates resulting from reactions of iron(II) and (III) compounds convert rapidly into other iron oxide forms. Initial studies with combinations of Ca(OH)_2 and NaOH with iron salts (nitrate and oxalate) set up to hard iron oxide mixtures. It was postulated that these mixtures could be used as a binder for the steel wastes, with the iron salts being generated by dissolution of steel waste in acid. Preliminary work has shown that a binder consisting of an iron hydroxyoxide (no cement) gave a pellet from steel mill waste that passed the drop test.

However, all combinations of the steel waste with the iron nitrate and Ca(OH)_2 or NaOH failed to generate pellets that passed the drop test. Some of the pellets developed cured strengths in the 170-psi range, but the green strengths were poor. No further briquetting studies were done with the iron oxide binders; however, the iron salts proved to be extremely effective with molasses, as described in the following section.

Molasses Additives

The preparation of steel waste pellets with molasses binder was investigated to determine the effects of various additives to the molasses on binding strength. Initially the strength of pellets made with and without cement and Ca(OH)_2 in the hand press were evaluated. These tests did not demonstrate any positive effect of the added cement and Ca(OH)_2 . Pyrolysis odor tests indicated that the odor produced from the heated pellets was more objectionable when the cement and Ca(OH)_2 were present. Thus the calcium appeared to contribute substantially to the wet campfire odor.

To determine the effects of other metal cations on the molasses binding strengths and odors produced during heating, a series of salts was added to the molasses at 100°F. On mixing the salt, a change in viscosity of the molasses was noted for several of the salts. In the case of aluminum and iron salts, the viscosity of the molasses-salt mixture was dramatically reduced. We have not found any explanation for this viscosity phenomenon. The highly fluid molasses-salt mixture was then mixed with the steel waste at 100°F, and the pellets were formed in the hand press. Owing to the decreased viscosity, the waste material was wetted much more effectively than with straight, highly viscous molasses. Drop tests and compressive strengths determined for the pellets using molasses-salt binders are reported in Table 16.

Ferric Nitrate Additive

Compression tests of the cured pellets showed that addition of ferric nitrate (1%, dry weight basis) to molasses (8%, wet weight basis) produced a superior pellet (avg. 396) compared with molasses alone (295). Increasing the amount of ferric nitrate to 3% gave much poorer results. This decrease in strength may be attributed to the increase in water added as the hydrate (see below). When calcium nitrate (1%, dry weight basis) plus a small amount of water equivalent to that added as the hydrate in the iron nitrate salt were added to the molasses, the resulting cured pellet exhibited a much lower compressive strength.

TABLE 16

Results from Metal Salt Additives to Molasses Binders

Molasses (g)	OPC (g)	Lime (g)	Fe(NO ₃) ₃ · 9H ₂ O (g)	Ca(NO ₃) ₂ · 4H ₂ O (g)	Water (g)	Drop Test	Compressive Strength (psi)	
							Cured	Mean
1.6	—	—	—	—	—	Passed	299	
1.6	—	—	—	—	—	Passed	291	295
1.6	—	—	0.34	—	—	Passed	387	
1.6	—	—	0.34	—	—	Passed	405	396
1.6	—	—	1.36	—	—	Passed	236	
1.6	—	—	1.36	—	—	Passed	242	239
1.6	—	—	—	1.16	0.2	Passed	211	
1.6	—	—	—	1.16	0.2	Passed	208	210

The effect of water concentration on the cured pellet strengths was investigated for a few molasses-salt binder mixtures. In this series of tests (Table 17), the ferric nitrate with no added water was very strong (compressive strength 468 psi) compared with the same salt and a small amount of added water (312 psi). These results are consistent with many earlier studies showing that too much water is deleterious.

TABLE 17

Effect of Water Concentration on Molasses-Metal Salt Binders

Molasses (g)	OPC (g)	Lime (g)	Fe(NO ₃) ₃ · 9H ₂ O (g)	Ca(NO ₃) ₂ · 4H ₂ O (g)	Water (g)	Drop Test	Compressive Strength (psi)	
							Cured	Mean
1.6	—	—	0.34	—	0	Passed	468	
1.6	—	—	0.34	—	0	Passed	469	468
1.6	—	—	0.34	—	0.46	Passed	315	
1.6	—	—	0.34	—	0.46	Passed	308	312

The compressive strengths of pellets made by adding metal salts to molasses-OPC and molasses- Ca(OH)_2 binders were also investigated. These tests were also done using several water concentrations. The results (Table 18) show an effect similar to that observed with molasses-salt binders. Thus, the pellet using molasses-OPC with ferric nitrate (1%) and no added water was significantly stronger (362 psi) than the pellet using molasses-OPC (282 psi), but the corresponding tests with added water showed little difference as a result of added salt.

In the case of molasses- Ca(OH)_2 -ferric nitrate binders (no water), the pellets again showed a significant increase in strength (240 psi) compared with the molasses- Ca(OH)_2 pellets (199 psi). The pellets with added water showed a similar increase in strength resulting from the added ferric nitrate.

The addition of calcium nitrate to the molasses- Ca(OH)_2 binder (water added) had a small negative effect on the compressive strengths of the resulting pellets. This effect is similar to that observed with the molasses-calcium salt binder. Addition of water to the molasses- Ca(OH)_2 -calcium pellet showed further substantial decrease in strength.

A series of pellets was studied to determine if smaller amounts of molasses binder could be used as a result of the increased strength of the molasses-metal salt binders (Table 19). When only 4% molasses was used with the iron nitrate additive (no water), the strength was still adequate (avg. 226 psi). Addition of extra water to the 4% molasses-ferric nitrate binder increased the strength somewhat. Compressive strengths of the molasses- Ca(OH)_2 -ferric nitrate binder at the low concentration were relatively low.

Aluminum Nitrate Additives

Aluminum nitrate was also very effective in decreasing the viscosity of molasses and gave very strong pellets. The result of several tests with molasses-aluminum nitrate at various concentrations are shown in Table 20. The molasses-aluminum nitrate pellets were substantially stronger than the molasses pellets. The strength of the 6 wt % molasses-aluminum nitrate binder was comparable to that of the 8 wt % molasses binder, and the 4 wt % binder also gave pellets with adequate strength.

Additional Binder Compositions

A large number of pellets were produced by adding other salts (magnesium, calcium, aluminum, zinc, and ammonium) containing various anions. In this series of tests (Table 21), the aluminum nitrate, aluminum chloride, ferric nitrate, and ferric sulfate were the most effective of the salts. These also decreased the viscosity of the molasses to the greatest degree. Aluminum sulfate also produced the viscosity-reducing effect and gave strong pellets. Magnesium, calcium, and ammonium nitrates had little effect on the viscosity, but gave pellets with adequate strength in these tests. Sodium silicate and sodium carbonate increased the viscosity of the molasses, and had a small effect on the strength. Sodium chloride had the most negative effect on the strength.

TABLE 18

Effect of Metal Salts and Water Concentrations on Molasses–OPC and Molasses–Lime Binders

Molasses (g)	OPC (g)	Lime (g)	Fe(NO ₃) ₃ ·9H ₂ O (g)	Ca(NO ₃) ₂ ·4H ₂ O (g)	Water (g)	Drop Test	Compressive Strength (psi)	
							Cured	Mean
1.6	1.6	—	—	—	—	Passed	286	
1.6	1.6	—	—	—	—	Passed	276	282
1.6	1.6	—	0.34	—	—	Passed	367	
1.6	1.6	—	0.34	—	—	Passed	356	362
1.6	1.6	—	—	—	0.46	Passed	319	
1.6	1.6	—	—	—	0.46	Passed	308	312
1.6	1.6	—	0.34	—	0.46	Passed	319	
1.6	1.6	—	0.34	—	0.46	Passed	312	316
1.6	—	1.6	—	—	—	Passed	196	
1.6	—	1.6	—	—	—	Passed	202	199
1.6	—	1.6	0.34	—	—	Passed	234	
1.6	—	1.6	0.34	—	—	Passed	246	240
1.6	—	1.6	—	—	0.46	Passed	232	
1.6	—	1.6	—	—	0.46	Passed	227	230
1.6	—	1.6	0.34	—	0.46	Passed	107	
1.6	—	1.6	0.34	—	0.46	Passed	105	106
1.6	—	1.6	0.34	—	1.46	Passed	99	
1.6	—	1.6	0.34	—	1.46	Passed	94	97
1.6	—	1.6	—	0.29	0.51	Passed	200	
1.6	—	1.6	—	0.29	0.51	Passed	202	201
1.6	—	1.6	—	0.29	1.6	Passed	101	
1.6	—	1.6	—	0.29	1.6	Passed	97	99

TABLE 19

Effect of Molasses–Metal Salt Binder Concentrations

Molasses (g)	Lime (g)	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (g)	Water (g)	Drop Test	Compressive Strength (psi)	
					Cured	Mean
1.6	—	0.34	0	Passed	468	
1.6	—	0.34	0	Passed	469	468
0.8	—	0.34	0	Passed	234	
0.8	—	0.34	0	Passed	218	226
1.6	—	0.34	0.46	Passed	315	
1.6	—	0.34	0.46	Passed	308	312
0.8	—	0.34	0.46	Passed	233	
0.8	—	0.34	0.46	Passed	257	245
1.6	1.6	0.34	0.46	Passed	107	
1.6	1.6	0.34	0.46	Passed	105	106
0.8	0.8	0.34	0.46	Passed	117	
0.8	0.8	0.34	0.46	Passed	125	121

TABLE 20

Effect of Aluminum Nitrate and Molasses Concentrations

Molasses (g)	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (g)	Water ¹ (g)	Drop Test	Compressive Strength (psi)	
				Cured	Mean
1.6	—	—	Passed	299	
1.6	—	—	Passed	291	295
1.6	0.17	—	Passed	352	
1.6	0.17	—	Passed	346	349
1.2	0.17	—	Passed	299	
1.2	0.17	—	Passed	291	295
0.8	0.17	—	Passed	183	
0.8	0.17	—	Passed	195	189

¹ Water was present in the hydrated salts used.

TABLE 21

Results of Tests with Molasses-Salt Binders of Various Compositions

Molasses (g)	Salt ¹ (g)	Water ² (g)	Drop Test	Compressive Strength (psi)	
				Cured	Mean
1.6	—	—	Passed	315	
1.6	—	—	Passed	315	315
1.6	Aluminum chloride $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (0.34)	—	Passed	362	
1.6	Aluminum chloride $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (0.34)	—	Passed	368	365
1.6	Aluminum nitrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.34)	0	Passed	392	
1.6	Aluminum nitrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.34)	0	Passed	398	395
1.6	Aluminum sulfate $\text{Al}_2(\text{SO}_4)_3$ (0.2)	0.15	Passed	339	
1.6	Aluminum sulfate $\text{Al}_2(\text{SO}_4)_3$ (0.2)	0.15	Passed	330	334
1.6	Ammonium nitrate NH_4NO_3 (0.2)	0.14	Passed	320	
1.6	Ammonium nitrate NH_4NO_3 (0.2)	0.14	Passed	328	324
1.6	Calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (0.29)	0.05	Passed	369	
1.6	Calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (0.29)	0.05	Passed	366	368
1.6	Ferric sulfate $\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$ (0.29)	0.05	Passed	398	
1.6	Ferric sulfate $\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$ (0.29)	0.05	Passed	378	388
1.6	Ferric citrate $\text{FeC}_6\text{H}_5\text{O}_7 \cdot n\text{H}_2\text{O}$ (0.29)	0.05	Passed	312	
1.6	Ferric citrate $\text{FeC}_6\text{H}_5\text{O}_7 \cdot n\text{H}_2\text{O}$ (0.29)	0.05	Passed	323	318
0.8	Ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.34)	0.2	Passed	255	
0.8	Ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.34)	0.2	Passed	245	250
1.6	Ferrous acetate $\text{Fe}(\text{CH}_3\text{COO})_2$ (0.2)	0.14	Passed	307	

Continued . . .

TABLE 21 (continued)

Molasses (g)	Salt ¹ (g)	Water ² (g)	Drop Test	Compressive Strength (psi)	
				Cured	Mean
1.6	Ferrous acetate $\text{Fe}(\text{CH}_3\text{COO})_2$ (0.2)	0.14	Passed	318	313
1.6	Ferrous sulfate $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (0.34)	—	Passed	335	
1.6	Ferrous sulfate $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (0.34)	—	Passed	319	327
1.6	Magnesium sulfate $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.30)	0.04	Passed	366	
1.6	Magnesium sulfate $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.30)	0.04	Passed	369	368
1.6	Sodium acetate $\text{Na}(\text{CH}_3\text{COO})$ (0.2)	0.14	Passed	284	
1.6	Sodium acetate $\text{Na}(\text{CH}_3\text{COO})$ (0.2)	0.14	Passed	288	286
1.6	Sodium carbonate Na_2CO_3 (0.2)	0.14	Passed	329	
1.6	Sodium carbonate Na_2CO_3 (0.2)	0.14	Passed	332	330
1.6	Sodium chloride NaCl (0.2)	0.15	Passed	195	
1.6	Sodium chloride NaCl (0.2)	0.15	Passed	203	199
1.6	Sodium nitrate NaNO_3 (0.2)	0.15	Passed	286	
1.6	Sodium nitrate NaNO_3 (0.2)	0.15	Passed	292	289
1.6	Sodium silicate ¹ (0.5)	0	Passed	298	
1.6	Sodium silicate ¹ (0.5)	0	Passed	316	307
1.6	Sodium sulfate Na_2SO_4 (0.2)	0.14	Passed	345	
1.6	Sodium sulfate Na_2SO_4 (0.2)	0.14	Passed	356	350
1.6	Zinc sulfate $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (0.34)	0	Passed	325	
1.6	Zinc sulfate $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (0.34)	0	Passed	316	320

¹ Dry weight basis.² Water was present in the hydrated salts used.

Briquetting

The compositions and conditions for 35 briquetting tests are reported in Table 22. The results of drop-shatter, compressive strength, and tumble testing are presented for the 35 briquetting tests in Table 23. The purpose of the test program was to develop a binder system that would produce steel mill waste briquettes with strength characteristics similar to briquettes made with the baseline binder system: 8 wt% OPC, 8 wt% molasses, and 4 wt% water. Progress made toward producing a suitable binder system is presented on a matrix-by-matrix basis.

Matrix 1

The binder systems evaluated in Matrix 1 consisted of various combinations of fly ash and Ca(OH)_2 with and without sodium bentonite. The fly ash types evaluated included a Corrette Class C fly ash and a fly ash provided by Heckett Multiserv (Test 11). A single test (No. 12) was performed using a calcined form of the Heckett Multiserv fly ash. Tests 3 and 15 were performed with material recovered from previous briquetting tests that had air-dried for up to 20 hours. Two tests (8A and B) were performed to evaluate water alone as the binder system. All binder systems were compared against the baseline binder system (Test 14), which used sugar beet molasses from American Crystal Sugar (ACS) in East Grand Forks, MN.

Results of briquette evaluation indicated that briquettes made with the baseline binder system (OPC, molasses, water) had the highest tumble resistance by a considerable margin. After 7 days of air-drying (curing) the tumble recovery was approximately 72 wt% for the baseline briquettes. The Test 4 briquettes had the next highest value, approximately 48 wt%, after 7 days. Additionally, the results indicate that the baseline binder system requires a shorter curing period to attain near maximum tumble resistance. After 2 days, the tumble recovery value was almost 69 wt% compared to 72 wt% after 7 days. Results of Tests 10, 11, and 12 show that their respective binder systems need longer than 3 days to achieve maximum tumble resistance. The compressive strength of briquettes produced with the baseline binder was at least 40% higher than values obtained with the other binder systems.

Moisture content appeared to be a critical variable affecting briquetability and strength. It was not possible to achieve a nominal roll torque of 700 ft-lb_f when processing mixtures at 15 wt% water (Tests 5 and 9). The higher-moisture-content briquettes did not consistently release from the roll pockets, appeared to be less compacted, and tended to have a significant amount of fines adhered to their surface. Further, the higher moisture content contributed to significantly reduced tumble resistance and compressive strength relative to binder systems with lower moisture content. Water alone (Test 8B) as the binder system produced briquettes with tumble resistance higher than that of several of the binder systems, although the roll torque was considerably higher than desired.

Results further indicate that tumble resistance may be degraded by reducing sodium bentonite concentration and that the binder systems using Heckett Multiserv fly ash are less effective than those using the Corrette ash.

Matrix 2

The binder systems evaluated in Matrix 2 included Corrette fly ash with Ca(OH)_2 and/or sodium silicate. The fly ash concentrations were higher in Matrix 2 relative to Matrix 1 (10, 12, and 14 wt% versus 8 wt%). Further, lower moisture contents were used (4, 6, and 8 wt% versus 10 and 15 wt%) in Matrix 2 to improve densification. A single briquetting test (No. 16) was performed with the baseline binder system using ACS sugar beet molasses.

The results of briquette evaluation indicated the baseline binder system was superior to the alternative binder systems investigated in Matrix 2. Briquettes made with baseline binder still had the highest tumble resistance, 67 versus 46 wt% (Test 24) after 6 days of air drying. Briquettes made with the baseline binder system also exhibited better drop-shatter resistance. The baseline binder briquettes principally exhibited only slight end deformation, with one briquette losing an estimated 20% of its mass. The other briquette types experienced greater levels of material loss with some individual briquettes (Tests 21, 23, 24), essentially disintegrating upon impact.

The addition of sodium silicate did not appear to impart significant improvement to briquette strength. However, comparison of Tests 18 and 20 suggests that substituting sodium silicate for Ca(OH)_2 at a concentration of 4 wt% shortens the curing time required to achieve maximum tumble resistance.

Tumble resistance of baseline briquettes made with $-\frac{1}{8}$ -in. steel mill waste was similar to that of baseline briquettes made with -16 -mesh steel mill waste.

Matrix 3

The binder systems evaluated in Matrix 3 included various nitrate and sulfate salts and molasses provided by Heckett Multiserv. Water was used only in small concentration (0.375 wt%) to substitute for the hydrate water of low-nitrate and -sulfate salt binder systems. A single briquetting test (No. 35) was performed with the baseline binder system using Heckett Multiserv molasses.

In contrast to Matrices 1 and 2, all binder systems except one (Test 32) produced briquettes with higher tumble resistance than the baseline briquettes produced using Heckett Multiserv molasses. Briquettes from Tests 25, 28, and 29, with molasses concentrations of 8 wt%, exhibited tumble recovery values of approximately 92 wt%. Even briquettes made with molasses alone (at 8 wt%) had a tumble recovery of approximately 83 wt%. These values compare to 74.4 wt% for the baseline briquettes, which also used 8 wt% molasses. Further, only the baseline briquettes exhibited deformation during drop-shatter testing. None of the other briquettes exhibited any degree of chipping.

The results of tumble tests repeated with briquettes from Test 14 (Matrix 1) indicated that ACS and Heckett Multiserv molasses produce briquettes with similar tumble resistance values (77 and 74 wt%, respectively).

The results of tumble testing also indicate that the curing time with the salt binder systems was essentially 2 days, although compressive strength results showed the briquettes increased in strength between 2 and 7 days. A check after completing tumbling tests on briquettes cured for 2 days showed that the rotational speed was slower than required (40 rpm). Adjustments were made to the rotational speed prior to tumble testing at the seventh day. As a consequence, the tumble recovery values obtained after 2 days of air curing were higher than values obtained after 7 days.

TABLE 22

Compositions and Conditions for Briquetting Steel Mill Waste

Test No.	Binders (wt%)			
1	C ash ¹ + Ca(OH) ₂ + water			
	(8)	(4)	(3)	
2	C ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(3)	(10)
3	Fines from Tests 1 and 2. No water added.			
4	C ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(3)	(10)
5	C ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(3)	(15)
6	Fines from Tests 1 to 5 dried in 5-gallon pail overnight.			
7	C ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(2)	(10)
8a	Water			
	(10)			
8b	Water			
	(10)			
9	C ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(3)	(15)
10	C ash + OPC + sodium bentonite + water			
	(8)	(4)	(2)	(10)
11	HM ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(2)	(10)
12	HM calcined ash + Ca(OH) ₂ + sodium bentonite + water			
	(8)	(4)	(2)	(10)
13	Ca(OH) ₂ + sodium bentonite + water			
	(4)		(2)	(10)
14	OPC + ACS molasses + water			
	(8)	(8)	(4)	
15	Fines from Tests 1 to 9 dried on floor overnight.			
16	OPC + ACS molasses + water			
	(8)	(8)	(4)	

Continued . . .

TABLE 22 (continued)

Test No.	Binders (wt%)		
17	C ash + Ca(OH) ₂ + water (12) (4) (8)		
18	C ash + Ca(OH) ₂ + water (12) (4) (6)		
19	C ash + Ca(OH) ₂ + water (12) (4) (4)		
20	C ash + sodium silicate + water (12) (4) (6)		
21	C ash + Ca(OH) ₂ + water (14) (4) (6)		
22	C ash + Ca(OH) ₂ + water (10) (4) (4)		
23	C ash + sodium silicate + water (12) (2) (6)		
24	C ash + sodium silicate + Ca(OH) ₂ + water (12) (2) (2) (6)		
25	Ferric nitrate + HM molasses (1) (8)		
26	Ferric nitrate + HM molasses (1) (4)		
27	HM molasses (8)		
28	Aluminum nitrate + HM molasses + water (0.5) (6) (0.375)		
29	Ferric nitrate + HM molasses + water (0.5) (8) (0.375)		
30	Ferric nitrate + HM molasses (1) (6)		
31	Ferrous sulfate + HM molasses (1) (6)		
32	Ferric sulfate + HM molasses (1) (6)		
33	Aluminum sulfate + HM molasses (1) (8)		
34	Ferrous sulfate + HM molasses + water (0.5) (8) (0.375)		
35	OPC + HM molasses + water (8) (8) (4)		

C = Corette; OPC = ordinary portland cement; HM = Hekett Multiserv;
ACS = American Crystal Sugar.

TABLE 23

Drop-Shatter and Compressive Strength Results

Test No.	Drop Test	Compressive Strength (psi)					Tumble Testing		
		Green	1 day	2 days	7 days	Recovery (wt%)	Cure Time (days)	Recovery (wt%)	Cure Time (days)
1	Pass	221 (after 80 min)	—	236	279	—	—	—	—
2	Pass	87 (after 40 min)	—	260	251	—	—	—	—
3	Pass	154 (after 15 min)	—	244	261	—	—	—	—
4	Pass	53	—	295	296	48.1	7	47.2	12
5	Pass	26	—	133	141	—	—	14.4	9
6	Pass	—	—	260	306	46.9	4	50.1	9
7	Pass	46	—	248	262	34.2	4	38.3	9
8A	Three no loss, two 20% loss	121	—	—	287	—	—	—	—
8B	All chipped, about 15% loss	123	—	267	291	36.6	4	—	—
9	—	—	—	171	172	—	—	—	—
10	End deformed on two, three 15% loss	44	216	238	252	21.9	3	30.0	8
11	Pass	33	230	280	259	24.1	3	33.3	7

Continued . . .

TABLE 23 (continued)

Test No.	Drop Test	Compressive Strength (psi)				Tumble Testing			
		Green	1 day	2 days	7 days	Recovery (wt%)	Cure Time (days)	Recovery (wt%)	Cure Time (days)
12	Minor chipping	47	213	252	249	22.6	3	30.6	8
13	Some chipping	40	189	248	254	—	—	—	—
14	Pass	78	277	493	465	68.6	3	72.3	7
15	Pass	276 (after 4 hr)	275	343	320	—	—	—	—
16	End deformed on four, one 20% loss	27	—	—	—	68.8	2	66.8	6
17	Four 15% to 40% loss	53	—	—	—	25.1	2	38.7	6
18	All 5% to 20% loss	90	—	—	—	22.6	2	36.2	6
19	Two 20% to 25% loss, one 10% loss	148	—	—	—	28.5	2	37.9	6
20	One 40% loss, one 10% loss	161	—	—	—	41.8	2	40.2	6
21	One broke into pieces, one 40% loss	97	—	—	—	36.4	2	41.4	6
22	Three 20% loss	150	—	—	—	19.4	2	35.2	6

Continued ...

TABLE 23 (continued)

Test No.	Drop Test	Green	Compressive Strength (psi)			Tumble Testing			
			1 day	2 days	7 days	Recovery (wt%)	Cure Time (days)	Recovery (wt%)	Cure Time (days)
23	One broke into pieces, two 20% loss	130	—	—	—	30.6	2	42.7	6
24	One broke into pieces, one 30% loss	115	—	—	—	—	—	45.5	6
25	Pass	141	—	329	703	92.6	2	92.1	7
26	Pass	200	—	366	541	83.3	2	80.8	7
27	Pass	139	—	298	502	93.3	2	82.5	7
28	Pass	95	—	279	658	95.6	2	92.8	7
29	Pass	101	—	279	700	96.0	2	91.9	7
30	Pass	180	—	408	685	89.0	2	87.5	7
31	Pass	156	—	368	589	89.3	2	86.4	7
32	Pass	202	—	357	447	77.4	2	70.7	7
33	Pass	154	—	361	600	93.0	2	89.1	7
34	Pass	121	—	—	777	—	—	81.8	7
35	End deformed on all	38	—	—	632	—	—	74.4	7

Pyrolysis Odor Tests

These tests were conducted in order to determine the effects of calcium on the peculiar odor that results from pyrolysis of the molasses-steel waste briquettes and the potential of oxidizers for elimination of odor resulting from the pellets containing molasses as the binder. The cured pellets were crushed to ¼-in. particles. The pieces were placed in a Vycor tube and heated in a tube furnace in a gentle flow of nitrogen. The effluent gas was smell-tested to compare the odors coming from the heated pellet. The heating was carried out in the temperature range of 24°–600°C using a 10°C/min heating rate. The odor of the gas was checked every 50°C.

The results indicated that the iron and aluminum did not contribute to the wet campfire odor as did the calcium (Ca(OH)_2 and OPC) (Table 24). The nitrate salt reduced the burned sugar odor considerably, probably by oxidizing the organic pyrolysis vapors to CO_2 .

SEM Morphology

Three of the pellets prepared with the molasses-metal salt binders were selected for scanning electron microscope (SEM) examination. These three samples and a briquette prepared with molasses-ferric nitrate binder and the molasses with no added salt were coated with carbon film for SEM morphology analysis. SEM images were scanned for interesting features, especially at the boundaries between particles. SEM data sheets are included in Appendix A.

Molasses Binder Pellet Sample 980429

An image (Figure 1) containing an imbedded 30-µm sphere was examined. The white sphere was mainly Fe with small amounts of Ca oxide (1.1). The white lattice boundary (1.2) appears to be mostly Fe and Ca oxides (no evidence for carbohydrate). The grey boundary area (1.3) is similar, but contains some silicate and possibly carbohydrate or carbonate. Another grey area (1.4) contains less Fe but more Al, Mg, Si (slag-clay). The black area (1.5) is mostly carbon (coke). It is likely that the molasses is coating the particles, such that it cannot be found in particles.

A second image (Figure 2) contains numerous small (2–4-µm) spheres of Fe (white) and a coke particle (2.1). The grey boundary areas (2.2 and 2.3) may contain carbohydrate in addition to the Ca and Fe present. The third image (Figure 3) contains various grey areas (3.1–3.5) with varying amounts of Ca, Fe, S, Si, and carbohydrate or carbonate.

It is clear from this and subsequent sets of images that the white areas are mostly Fe. The black areas are mostly a coke type of carbon.

Molasses-Aluminum Nitrate Binder Pellet Sample 980428

The first image (Figure 4) from this sample exhibits a variety of shapes and sizes of particles. A grey area (1.1) contains mainly Mg silicate with substantial carbonate or

TABLE 24

Results of Pyrolysis Odor Tests

Additives (g)	Compressive Strength (psi)	Temperature (°C)	Odor
Lime (1.6)	315	170–225 250 250–500 500 550	Very slight burnt sugar smell Wet campfire smell Campfire smell increased Burnt smell started to decrease Smell changed to burned wood (fireplace) and got stronger
Lime (1.6) + $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.2)	384	170–225 At 250–300 315 340–500 At 550 At 600	Very light smell Slight NO_x smell, no burn odor Sweet garbage smell, white fumes Slight burn smell Light burnt smell Strong burn smell
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.34) + H_2O (0.46) + OPC (1.6)	316	250 300	Slight burning smell Burning garbage smell and white fumes that became strong as the temperature increased to 600°C
			Results similar to Test 2 except that the intensity of the smell was less
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.34) + H_2O (0.46)	340	255 At 250–350 350–425 450	Sweet caramel smell Same sweet caramel smell continued Slight burning sugar smell Onward burning sugar or fireplace smell
$\text{Al}(\text{NO}_3)_3 \cdot \text{NH}_2\text{O}$ (0.34)	395	150 200 250 300–365 425 500	Sweet smell Sweet smell Sweet smell Slight caramel smell Mild caramel smell Mild caramel
NH_4NO_3 (0.2)	324	130 213 300 350 400 500 550	Ammonia smell began and became very strong at 145°C Fumes and burning paper smell was observed Campfire smell although not as strong as with molasses alone Burning garbage smell Smoking and burning garbage smell Smoky and burning garbage smell became intense Less smoky but strong burning garbage smell persists

Figure 1. Molasses binder pellet – Image 1.

Figure 2. Molasses binder pellet – Image 2.

Figure 3. Molasses binder pellet – Image 3.

Figure 4. Molasses-aluminum nitrate binder pellet – Image 1.

carbohydrate. The Al content (2.79%) is higher than that present in most other images of this and other samples. Another grey area (1.2) contains striations that may be from fracturing. The K and Fe contents were high, as was the carbonate or carbohydrate. This area also contains Al (1.89%) and some silicate and Ca. Another grey area (1.3) is high in Fe and contains some Ca and carbonate or carbohydrate. A grey area (1.4) between the small white iron spheres is high in Fe and Ca and contains Al (1.99%) and carbonate or carbohydrate. No evidence was found for N (nitrate) in the composition data.

A second image (Figure 5) has a vesicular slag on a large black coke particle. The slag is mainly composed of Ca and sulfate but with some carbonate. Al is low, so the slag may have been on the coke and not formed in the binding.

A third image (Figure 6) from this sample shows a crevasse filled with radiating needle crystals (3.1) composed of Ca, Al, and silicate. These are identified as ettringite. The light matrix (3.3) is mainly Ca silicate. Since this composite particle was also found in a sample (980427) that was not treated with $\text{Al}(\text{NO}_3)_3$, and ettringite was not found elsewhere in this sample, it is likely that the particle is a component of the steel waste and ettringite was not formed during the binding process.

Molasses–Ferric Nitrate Binder Pellet Sample 980427

The first image (Figure 7) examined contains light triangular particles (1.1–1.2) in a darker grey matrix (1.3). A grey triangular particle (1.1) is mainly Ca and silicate, but contains some Fe and quite a bit of carbon (carbohydrate or carbonate). A lighter triangular particle (1.2) contains more Fe and much less Ca and silicate but similar carbon. The dark grey matrix (1.3) is similar in composition, but contains more Fe. Although the white particles are mostly Fe, it appears that Fe compounds are present in darker particles also. The evidence that Fe in the dark particles may represent added Fe is not at all conclusive because of the high Fe content in the waste particles. It is likely that the Fe from the binder is spread thinly around all the particles in the sample. No evidence was found for N (nitrate) in any of the images in any of the samples.

The second image (Figure 8) contains patches of lighter and darker particles. The darker grey (2.1) particles are high in Fe and contain Ca and C. The lighter area (2.2) is high in Pb, but also contains Fe, Ca, and C. A third image (not shown) has radiating needle crystals of ettringite similar to those found in the molasses– $\text{Al}(\text{NO}_3)_3$ binder sample (980428).

Molasses–Ferric Nitrate Binder Briquette Sample 980426

The first image (Figure 9) of this sample contains a large coke particle (dark area, composition not given). The light grey area (1.1) off the edge of the large particle is mainly Ca, but contains some Fe and carbonate or carbohydrate. Similar grey areas further away from the coke particle (1.2–1.3) have a variety of compositions.

The second image (Figure 10) shows another large coke particle and large iron particles. The grey areas between (2.1–2.2) are mainly Ca, Fe, and carbon. A lighter grey area is Fe,

Figure 5. Molasses-aluminum nitrate binder pellet – Image 2.

Figure 6. Molasses-aluminum nitrate binder pellet – Image 3.

Figure 8. Molasses-ferric nitrate binder – Image 2.

Figure 7. Molasses-ferric nitrate binder pellet – Image 1.

Figure 9. Molasses-ferric nitrate binder briquette – Image 1.

Figure 10. Molasses-ferric nitrate binder briquette – Image 2.

silicate and carbonate or carbohydrate. A third image (Figure 11) shows various grey areas between white Fe spheres. These are all mainly Ca, Fe, silicate, and carbonate or carbohydrate in somewhat different proportions. As in the previous sample, it is impossible to find a definitive layer of binder around the particulates.

Molasses-Ferrous Sulfate Binder Pellet Sample 980430

The first image (Figure 12) shows a grey area (1.2) with a relatively high S content that could correspond to a particle of FeSO_4 . Not all of the FeSO_4 dissolved in the molasses, so this could be a remnant of the added salt. A second area (1.5) also shows a high S content. In the second (Figure 13) and third image (Figure 14), there are many 2- μm white Fe spheres and some appearance of a grey film around the spheres. Analysis of the composition of grey areas indicated higher sulfate content.

Figure 11. Molasses-ferric nitrate binder briquette – Image 3.

Figure 12. Molasses-ferrous sulfate binder pellet – Image 1.

Figure 13. Molasses-ferrous sulfate binder pellet – Image 2.

Figure 14. Molasses-ferrous sulfate binder pellet – Image 3.

REFERENCES

- Aumônier, J. "Experimental Agglomeration of BOF Slurry," *In* Proceedings of The Institute for Briquetting and Agglomeration 24th Biennial Conference; Philadelphia, PA, Oct. 1995; pp 1-11.
- Günter, H. "The Application of Waste Paper as a Binder in Pressure Agglomeration," *In* Proceedings of The Institute for Briquetting and Agglomeration 23rd Biennial Conference; Seattle, WA, Oct. 1993; pp 25-34.
- Lambert, R.C. "Techniques for Agglomerating Coal, Coke and Steel Mill Wastes," *In* Proceedings of The Institute for Briquetting and Agglomeration 24th Biennial Conference; Philadelphia, PA, Oct. 1995; pp 139-147.
- Steele, R.B. "Agglomeration of Steel Mill By-Products via Auger Extrusion," *In* Proceedings of The Institute for Briquetting and Agglomeration 23rd Biennial Conference; Seattle, WA, Oct. 1993; pp 205-217.

APPENDIX A

SEM DATA SHEETS

Wed May 27 10:25:15 1998

980429.1.1

Refit Na-K' Na-K" Mg-K' Mg-K" Al-K' Al-K" Si-K' Si-K" P -K' P -K" Cl

Refit S -K' K -K' K -K" Ca-K' Fe-K' Ti-K

Refit Ba-L

Filter Fit Method

Chi-sqd = 10.84 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00180 +/-	0.00024	113 +/-	15
Mg-K	0.00249 +/-	0.00028	162 +/-	18
Al-K	0.00143 +/-	0.00034	92 +/-	22
Si-K	0.00542 +/-	0.00048	340 +/-	30
P -K	0.00053 +/-	0.00053	33 +/-	33
S -K	0.01103 +/-	0.00080	577 +/-	42
Cl-K	0.00386 +/-	0.00081	173 +/-	36
K -K	0.01107 +/-	0.00117	397 +/-	42
Ca-K	0.11402 +/-	0.00400	3567 +/-	125
Fe-K	0.81863 +/-	0.01495	9473 +/-	173
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.02972 +/-	0.00139	920 +/-	43

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 4

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0017	3.004	1.02	0.51	+/- 0.07	1.577
Mg-K	0.0023	2.124	0.94	0.50	+/- 0.06	1.457
Al-K	0.0013	1.727	0.40	0.23	+/- 0.06	0.611
Si-K	0.0051	1.397	1.17	0.71	+/- 0.06	1.804
P -K	0.0005	1.274	0.09	0.06	+/- 0.06	0.145
S -K	0.0104	1.126	1.68	1.17	+/- 0.09	2.592
Cl-K	0.0036	1.105	0.52	0.40	+/- 0.08	0.805
K -K	0.0104	0.997	1.22	1.04	+/- 0.11	1.888
Ca-K	0.1073	0.956	11.77	10.25	+/- 0.36	18.189
Fe-K	0.7704	1.035	65.67	79.72	+/- 1.46	101.504
Ba-L	0.0000	1.158	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	0.998	0.00	0.00	+/- 0.00	0.000
O -K	0.0280	1.931	15.53	5.40	+/- 0.25	---
TOTAL			100.00	100.00		130.574

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 10:36:45 1998

29
980426.1.2

Refit _Mg-K' _Mg-K" _F -K' _F -K" _K -K' _K -K" _Ba-L' _Ba-L" _Ti-K' _Ti-K"
Refit _Si-K' _Si-K" _S -K' _S -K" _Cl-K' _Cl-K" _Ca-K' _Ti-K
Refit _Al-K"

Filter Fit Method

Chi-sqd = 31.90 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00435 +/-	0.00075	366 +/-	63
Mg-K	0.00256 +/-	0.00035	223 +/-	46
Al-K	0.01909 +/-	0.00064	1652 +/-	55
Si-K	0.02832 +/-	0.00076	2392 +/-	64
P -K	0.00006 +/-	0.00062	5 +/-	54
S -K	0.02165 +/-	0.00089	1528 +/-	63
Cl-K	0.00953 +/-	0.00083	575 +/-	50
K -K	0.00446 +/-	0.00089	217 +/-	43
Ca-K	0.16796 +/-	0.00367	7097 +/-	155
Fe-K	0.50747 +/-	0.01043	7932 +/-	163
Ba-L	0.00017 +/-	0.00230	4 +/-	56
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.23439 +/-	0.00309	9790 +/-	129

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 KV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0032	2.718	1.04	0.87	+/- 0.15	0.400
Mg-K	0.0019	1.961	0.41	0.37	+/- 0.06	0.160
Al-K	0.0141	1.628	2.32	2.29	+/- 0.08	0.895
Si-K	0.0209	1.367	2.77	2.85	+/- 0.08	1.072
P -K	0.0000	1.279	0.01	0.01	+/- 0.06	0.002
S -K	0.0160	1.151	1.56	1.94	+/- 0.08	0.604
Cl-K	0.0070	1.145	0.62	0.80	+/- 0.07	0.239
K -K	0.0033	1.048	0.24	0.34	+/- 0.07	0.093
Ca-K	0.1238	1.018	8.58	12.60	+/- 0.28	3.314
Fe-K	0.3740	1.112	20.33	41.60	+/- 0.85	7.854
Ba-L	0.0001	1.277	0.00	0.02	+/- 0.22	0.001
Ti-K	0.0000	1.084	0.00	0.00	+/- 0.00	0.000
O -K	0.1728	2.108	62.12	36.41	+/- 0.48	---
TOTAL			100.00	100.00		14.634

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 10:45:52 1998

980429.1.3b

Refit Al-K' Al-K" F -K' F -K" Cl-K' Cl-K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K" S -K" K -K' K -K" Ca-K' Ba-L C -K'

Filter Fit Method

Chi-sqd = 1.89 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00882 +/-	0.00097	554 +/-	61
Mg-K	0.01162 +/-	0.00067	759 +/-	44
Al-K	0.00562 +/-	0.00068	364 +/-	44
Si-K	0.02421 +/-	0.00141	1529 +/-	89
P -K	0.00179 +/-	0.00078	111 +/-	48
S -K	0.05865 +/-	0.00138	3094 +/-	73
Cl-K	0.00875 +/-	0.00107	394 +/-	48
K -K	0.05751 +/-	0.00171	2084 +/-	62
Ca-K	0.31481 +/-	0.00370	9948 +/-	180
Fe-K	0.27508 +/-	0.01018	3216 +/-	119
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00056 +/-	0.00139	14 +/-	34
O -K	0.13334 +/-	0.00266	4165 +/-	83
C -K	0.09924 +/-	0.00226	2416 +/-	55

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0054	2.347	1.22	1.28	+/- 0.14	0.724
Mg-K	0.0072	1.755	1.14	1.26	+/- 0.07	0.675
Al-K	0.0035	1.511	0.43	0.52	+/- 0.06	0.253
Si-K	0.0150	1.282	1.50	1.92	+/- 0.11	0.889
P -K	0.0011	1.226	0.10	0.14	+/- 0.06	0.057
S -K	0.0362	1.118	2.78	4.05	+/- 0.10	1.645
Cl-K	0.0054	1.139	0.38	0.62	+/- 0.07	0.226
K -K	0.0355	1.052	2.10	3.74	+/- 0.11	1.244
Ca-K	0.1944	1.056	11.28	20.54	+/- 0.37	6.672
Fe-K	0.1699	1.156	7.74	19.65	+/- 0.73	4.582
Ba-L	0.0000	1.360	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0003	1.175	0.02	0.04	+/- 0.10	0.011
O -K	0.0824	3.580	40.56	29.49	+/- 0.59	---
C -K	0.0613	2.736	30.74	16.77	+/- 0.38	18.187
Total			100.00	100.00		35.165

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 10:39:56 1998

29
980426.1.4

Refit F -K' F -K" Cl-K' Cl-K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Na-K' Al-K" Si-K" K -K" Ca-K" Fe-K' Ba-L
Filter Fit Method

Chi-sqd = 22.37 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00695 +/-	0.00114	317 +/-	52
Mg-K	0.01223 +/-	0.00182	580 +/-	86
Al-K	0.05612 +/-	0.00136	2634 +/-	65
Si-K	0.09644 +/-	0.00155	4420 +/-	71
P -K	0.00249 +/-	0.00096	111 +/-	43
S -K	0.08442 +/-	0.00296	3231 +/-	114
Cl-K	0.01243 +/-	0.00132	407 +/-	43
K -K	0.04731 +/-	0.00194	1243 +/-	51
Ca-K	0.36851 +/-	0.00436	8448 +/-	100
Fe-K	0.13870 +/-	0.01050	1176 +/-	89
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00455 +/-	0.00186	76 +/-	31
O -K	0.16985 +/-	0.00344	3850 +/-	78

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element Wt %	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0045	2.160	1.04	0.97	+/- 0.16	0.431
Mg-K	0.0079	1.639	1.31	1.30	+/- 0.19	0.544
Al-K	0.0363	1.435	4.74	5.20	+/- 0.13	1.969
Si-K	0.0623	1.279	6.98	7.97	+/- 0.13	2.897
P -K	0.0016	1.272	0.16	0.20	+/- 0.08	0.067
S -K	0.0546	1.150	4.81	6.27	+/- 0.22	1.998
Cl-K	0.0080	1.175	0.65	0.94	+/- 0.10	0.272
K -K	0.0306	1.070	2.06	3.27	+/- 0.13	0.654
Ca-K	0.2382	1.070	15.65	25.50	+/- 0.30	6.493
Fe-K	0.0896	1.159	4.58	10.39	+/- 0.79	1.899
Ba-L	0.0000	1.399	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0029	1.199	0.18	0.35	+/- 0.14	0.075
O -K	0.1098	3.427	57.83	37.62	+/- 0.76	---
Total			100.00	100.00		17.493

The number of cation results are based upon 24 Oxygen atoms

980429.2.2

Refit Na-K Refit Mg-K Refit Al-K Refit Si-K Refit S-K Refit Ba-K
 Refit P-K Refit Cl-K Refit Cl-K Refit K-K Refit Ca-K Refit Ca-K Refit Ba-L
 Refit Ti-K
 Filter Fit Method

Chi-sqd = 0.86 Livetime = 30.0 Sec.

Standardless Analysis

Element Relative Error

K-ratio (1-sigma) Counts (1-sigma) Error

Na-K 0.00182 +/- 0.00049

Mg-K 0.00365 +/- 0.00038

Al-K 0.00116 +/- 0.00067

Si-K 0.00449 +/- 0.00091

P-K 0.00390 +/- 0.00151

S-K 0.01283 +/- 0.00154

Cl-K 0.02123 +/- 0.00185

K-K 0.06637 +/- 0.00586

Ca-K 0.36281 +/- 0.00581

Fe-K 0.38646 +/- 0.01937

Ba-L 0.00000 +/- 0.00001

Ti-K 0.00000 +/- 0.00001

O-K 0.4403 +/- 0.00267

C-K 0.05924 +/- 0.00284

Adjustment Factors

Z-Balance:

Shell:

EAF Collection Acc.Volt. = 15 kV Take-off Angle = 43.31 deg
 Number of Iterations = 4

Element	K-ratio	ZAF	Atom %	Element	Wt %	Wt % Error	No. of
Na-K	0.00182	2.496	0.43	Na	0.35	+/- 0.09	Cations
Mg-K	0.00365	1.824	0.60	Mg	0.51	+/- 0.08	
Al-K	0.00365	1.540	0.14	Al	0.14	+/- 0.08	
Si-K	0.00365	1.289	0.45	Si	0.44	+/- 0.09	
P-K	0.00365	1.210	2.87	P	3.14	+/- 0.14	
S-K	0.00365	1.113	0.97	S	1.09	+/- 0.13	
Cl-K	0.00365	1.107	1.42	Cl	1.86	+/- 0.16	
K-K	0.00365	1.017	3.86	K	3.33	+/- 0.46	
Ca-K	0.00365	1.030	20.23	Ca	29.61	+/- 0.46	
Fe-K	0.00365	1.122	16.85	Fe	33.26	+/- 1.66	
Ba-L	0.00365	1.331	0.00	Ba	0.00	+/- 0.00	
Ti-K	0.00365	1.145	0.00	Ti	0.00	+/- 0.00	
O-K	0.00365	3.906	23.33	O	13.17	+/- 0.80	
C-K	0.00365	2.695	28.84	C	12.22	+/- 0.59	
Total			100.00		100.00		

The number of cation results are based upon 24 oxygen atoms

Wed May 27 11:01:16 1998

980429.2.3

Refit Na-K' Na-K" Mg-K' Mg-K" Al-K' Al-K" Si-K' Si-K" S -K' S -K" Ba-
Refit P -K' K -K' Ca-K' Ca-K" Fe-K' Ba-L O -K' C -K'
Filter Fit Method

Chi-sqd = 0.97 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00464 +/-	0.00060	132 +/-	17
Mg-K	0.00453 +/-	0.00068	134 +/-	20
Al-K	0.00265 +/-	0.00075	78 +/-	22
Si-K	0.00756 +/-	0.00099	215 +/-	28
P -K	0.05367 +/-	0.00170	1486 +/-	47
S -K	0.01958 +/-	0.00168	466 +/-	40
Cl-K	0.03326 +/-	0.00350	675 +/-	71
K -K	0.10626 +/-	0.00577	1732 +/-	94
Ca-K	0.43871 +/-	0.00612	6239 +/-	87
Fe-K	0.14924 +/-	0.00894	786 +/-	47
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00405 +/-	0.00232	42 +/-	24
O -K	0.05919 +/-	0.00192	832 +/-	27
C -K	0.11665 +/-	0.00256	1278 +/-	28

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0031	2.181	0.66	0.67	+/- 0.09	0.582
Mg-K	0.0030	1.647	0.46	0.49	+/- 0.07	0.405
Al-K	0.0018	1.429	0.21	0.25	+/- 0.07	0.185
Si-K	0.0050	1.225	0.50	0.61	+/- 0.08	0.436
P -K	0.0356	1.175	3.06	4.18	+/- 0.13	2.689
S -K	0.0130	1.103	1.01	1.43	+/- 0.12	0.891
Cl-K	0.0221	1.109	1.57	2.45	+/- 0.26	1.375
K -K	0.0705	1.034	4.23	7.29	+/- 0.40	3.712
Ca-K	0.2911	1.066	17.56	31.04	+/- 0.43	15.424
Fe-K	0.0990	1.164	4.68	11.53	+/- 0.69	4.112
Ba-L	0.0000	1.412	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0027	1.211	0.15	0.33	+/- 0.19	0.135
O -K	0.0393	4.910	27.33	19.28	+/- 0.63	---
C -K	0.0774	2.640	38.57	20.43	+/- 0.45	33.877
Total			100.00	100.00		63.823

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:12:46 1998

980429.3.1

Refit Na-K' Na-K" Mg-K' Mg-K" P -K' P -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Al-K' Si-K' Si-K" Cl-K' K -K' Ca-K" Fe-K' Fe-K" Ba-L C -K'
Filter Fit Method

Chi-sqd = 1.06 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00291 +/-	0.00059	129 +/-	26
Mg-K	0.00622 +/-	0.00074	288 +/-	34
Al-K	0.01656 +/-	0.00083	758 +/-	38
Si-K	0.01503 +/-	0.00087	671 +/-	39
P -K	0.00074 +/-	0.00087	32 +/-	38
S -K	0.10783 +/-	0.00311	4021 +/-	116
Cl-K	0.02206 +/-	0.00261	702 +/-	83
K -K	0.06218 +/-	0.00367	1593 +/-	94
Ca-K	0.26302 +/-	0.00372	5875 +/-	83
Fe-K	0.09901 +/-	0.00520	818 +/-	43
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00381 +/-	0.00135	63 +/-	22
O -K	0.25390 +/-	0.00412	5606 +/-	91
C -K	0.14674 +/-	0.00331	2525 +/-	57

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-F	0.0015	2.174	0.26	0.32	+/- 0.07	0.121
Mg-K	0.0032	1.639	0.39	0.52	+/- 0.06	0.185
Al-K	0.0084	1.431	0.83	1.21	+/- 0.06	0.387
Si-F	0.0077	1.244	0.63	0.95	+/- 0.06	0.294
P -K	0.0004	1.195	0.03	0.04	+/- 0.05	0.013
S -K	0.0550	1.107	3.50	6.09	+/- 0.18	1.643
Cl-K	0.0113	1.154	0.68	1.30	+/- 0.15	0.317
K -K	0.0317	1.084	1.62	3.44	+/- 0.20	0.760
Ca-K	0.1342	1.084	6.69	11.54	+/- 0.21	3.136
Fe-F	0.0505	1.187	1.98	5.99	+/- 0.32	0.928
Ba-L	0.0000	1.406	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0019	1.202	0.09	0.23	+/- 0.08	0.042
O -K	0.1288	3.429	51.17	44.41	+/- 0.72	---
C -K	0.0749	2.798	32.14	20.94	+/- 0.47	15.073
Total			100.00	100.00		22.898

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:21:04 1998

980429.3.2

Refit Na-K' Na-K" P -K' P -K" K -K' K -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K' Al-K" Si-K' S -K" Cl-K' Cl-K" Ca-K' Ba-L' Ti-K' Ti-K"
Filter Fit Method

Chi-sqd = 3.06 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00236 +/-	0.00057	120 +/-	29
Mg-K	0.01653 +/-	0.00074	876 +/-	39
Al-K	0.01593 +/-	0.00082	838 +/-	43
Si-K	0.02953 +/-	0.00094	1516 +/-	48
P -K	0.00082 +/-	0.00074	41 +/-	37
S -K	0.02331 +/-	0.00114	1000 +/-	49
Cl-K	0.01157 +/-	0.00107	424 +/-	39
K -K	0.00585 +/-	0.00105	172 +/-	31
Ca-K	0.21997 +/-	0.00316	5646 +/-	81
Fe-K	0.37490 +/-	0.01201	3559 +/-	114
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.22257 +/-	0.00398	5648 +/-	101
C -K	0.07667 +/-	0.00268	1517 +/-	53

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg

Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0015	2.498	0.35	0.36	+/- 0.09	0.164
Mg-K	0.0102	1.827	1.68	1.67	+/- 0.08	0.795
Al-K	0.0098	1.566	1.25	1.54	+/- 0.08	0.591
Si-K	0.0182	1.328	1.89	2.42	+/- 0.08	0.893
P -K	0.0005	1.264	0.05	0.06	+/- 0.06	0.021
S -K	0.0144	1.143	1.13	1.65	+/- 0.08	0.532
Cl-K	0.0071	1.146	0.51	0.82	+/- 0.08	0.239
K -K	0.0036	1.059	0.21	0.38	+/- 0.07	0.101
Ca-K	0.1359	1.041	7.74	11.14	+/- 0.20	3.653
Fe-K	0.2316	1.147	10.43	26.56	+/- 0.85	4.924
Ba-L	0.0000	1.310	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.127	0.00	0.00	+/- 0.00	0.000
O -K	0.1375	2.697	50.84	37.09	+/- 0.66	---
C -K	0.0474	2.766	23.92	13.10	+/- 0.46	11.292
Total			100.00	100.00		23.206

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:22:17 1998

980429.3.3

Refit Na-K' Na-K" F -K' F -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K' Mg-K" Al-K' Si-K" S -K' Cl-K' Cl-K" K -K' K -K" Ca-K' Ca-
Refit Ti-K

Filter Fit Method

Chi-sqd = 0.82 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00323 +/-	0.00084	73 +/-	19
Mg-K	0.02930 +/-	0.00132	689 +/-	31
Al-K	0.01755 +/-	0.00131	409 +/-	31
Si-K	0.04778 +/-	0.00172	1087 +/-	39
P -K	0.00176 +/-	0.00136	39 +/-	30
S -K	0.05915 +/-	0.00232	1123 +/-	44
Cl-K	0.03413 +/-	0.00222	553 +/-	36
K -K	0.03406 +/-	0.00246	444 +/-	32
Ca-K	0.39237 +/-	0.00624	4463 +/-	71
Fe-K	0.21018 +/-	0.01664	885 +/-	70
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.12437 +/-	0.00276	1399 +/-	31
C -K	0.04612 +/-	0.00263	405 +/-	23

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg

Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0021	2.251	0.48	0.47	+/- 0.12	0.261
Mg-K	0.0188	1.687	3.08	3.16	+/- 0.14	1.677
Al-K	0.0112	1.490	1.47	1.67	+/- 0.12	0.799
Si-K	0.0306	1.280	3.30	3.91	+/- 0.14	1.796
P -K	0.0011	1.239	0.11	0.14	+/- 0.11	0.058
S -K	0.0378	1.125	3.14	4.26	+/- 0.17	1.711
Cl-K	0.0218	1.144	1.67	2.50	+/- 0.16	0.908
K -K	0.0218	1.054	1.39	2.30	+/- 0.17	0.757
Ca-K	0.2511	1.058	15.68	26.57	+/- 0.42	8.546
Fe-K	0.1345	1.157	6.59	15.56	+/- 1.23	3.591
Ba-L	0.0000	1.385	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.191	0.00	0.00	+/- 0.00	0.000
O -K	0.0796	3.743	44.04	29.79	+/- 0.66	---
C -K	0.0295	3.280	19.06	9.68	+/- 0.55	10.387
Total			100.00	100.00		30.492

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:24:10 1998

980429.3.4

Refit Na-K' Na-K" P -K' P -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K' Al-K" Si-K' S -K' S -K" Cl-K' K -K" Ca-K' Ca-K" Fe-K' Fe-
Refit C -K"

Filter Fit Method

Chi-sqd = 1.06 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00244 +/-	0.00096	76 +/-	30
Mg-K	0.13351 +/-	0.00210	4330 +/-	68
Al-K	0.16728 +/-	0.00240	5379 +/-	77
Si-K	0.06524 +/-	0.00169	2048 +/-	53
P -K	0.00000 +/-	0.00001	1 +/-	6
S -K	0.03898 +/-	0.00172	1023 +/-	45
Cl-K	0.03664 +/-	0.00331	821 +/-	74
K -K	0.01983 +/-	0.00172	357 +/-	31
Ca-K	0.22534 +/-	0.00414	3539 +/-	65
Fe-K	0.06852 +/-	0.00603	399 +/-	35
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.16535 +/-	0.00290	2567 +/-	45
C -K	0.07687 +/-	0.00240	930 +/-	29

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 7

Element	k-ratio (calc.)	ZAF	Atom %	Element Wt %	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0013	1.848	0.21	0.24	+/- 0.10	0.137
Mg-K	0.0721	1.448	8.52	10.45	+/- 0.16	5.558
Al-K	0.0904	1.440	9.57	13.02	+/- 0.19	6.239
Si-K	0.0353	1.372	3.41	4.84	+/- 0.13	2.226
P -K	0.0000	1.345	0.00	0.00	+/- 0.00	0.000
S -K	0.0211	1.189	1.55	2.50	+/- 0.11	1.010
Cl-K	0.0198	1.194	1.32	2.36	+/- 0.21	0.862
K -K	0.0107	1.108	0.60	1.19	+/- 0.10	0.393
Ca-K	0.1218	1.086	6.54	13.23	+/- 0.24	4.268
Fe-K	0.0370	1.175	1.54	4.35	+/- 0.38	1.007
Ba-L	0.0000	1.393	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.197	0.00	0.00	+/- 0.00	0.000
O -K	0.0893	3.324	36.80	29.69	+/- 0.52	---
C -K	0.0415	4.364	29.92	18.13	+/- 0.57	19.514
Total			100.00	100.00		41.216

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:25:24 1998

980429.3.5

Refit Na-K' Na-K' Mg-K' Al-K' F-K' Ba-L' Ti-
Refit Si-K' S-K' Cl-K' K-K' Ca-K' Fe-K' Ba-L' Ti-
Filter Fit Method

Chi-sq = 0.83 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative K-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00452 +/-	0.00066	166 +/-	24
Mg-K	0.00566 +/-	0.00074	216 +/-	28
Al-K	0.00419 +/-	0.00083	158 +/-	31
Si-K	0.11681 +/-	0.00174	4300 +/-	64
P-K	0.00081 +/-	0.00098	29 +/-	36
S-K	0.03230 +/-	0.00150	993 +/-	46
Cl-K	0.02443 +/-	0.00145	641 +/-	38
K-K	0.02796 +/-	0.00171	590 +/-	36
Ca-K	0.41987 +/-	0.00500	7731 +/-	92
Fe-K	0.04669 +/-	0.00485	318 +/-	33
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O-K	0.21136 +/-	0.00291	3947 +/-	53
C-K	0.10539 +/-	0.00310	1495 +/-	44

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt. = 15 kV Take-off Angle = 13.31 deg
Number of Iterations = 6

Element	K-ratio (calc.)	ZAF	Atom %	Element Wt %	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0024	2.123	0.44	0.51	+/- 0.07	0.200
Mg-K	0.0030	1.613	0.39	0.49	+/- 0.06	0.180
Al-K	0.0022	1.411	0.23	0.32	+/- 0.06	0.105
Si-K	0.0527	1.224	5.33	7.67	+/- 0.11	2.416
P-K	0.0004	1.239	0.03	0.03	+/- 0.06	0.016
S-K	0.0173	1.133	1.19	1.96	+/- 0.09	0.548
Cl-K	0.0131	1.145	0.83	1.50	+/- 0.09	0.379
K-K	0.0150	1.061	0.79	1.59	+/- 0.10	0.365
Ca-K	0.2352	1.076	11.79	24.24	+/- 0.29	5.416
Fe-K	0.0250	1.188	1.04	2.98	+/- 0.31	0.477
Ba-L	0.0000	1.439	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.237	0.00	0.00	+/- 0.00	0.000
O-K	0.1134	3.781	52.25	42.87	+/- 0.59	---
C-K	0.0565	2.799	25.69	15.82	+/- 0.47	11.798
Total			100.00	100.00		21.932

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 14:42:08 1998

980428.1.1

Refit _Na-K' _Na-K" _P -K' _P -K" _S -K' _S -K" _Cl-K' _Cl-K" _Ba-L' _Ba-L" _Ti-
Refit _Mg-K" _Al-K" _Ca-K' _Fe-K" _C -K'
Refit _K -K'
Refit _K -K"

Filter Fit Method

Chi-sqd = 1.30 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00269 +/-	0.00060	170 +/-	38
Mg-K	0.13706 +/-	0.00147	9021 +/-	97
Al-K	0.03574 +/-	0.00108	2332 +/-	70
Si-K	0.18181 +/-	0.00273	11575 +/-	174
P -K	0.00139 +/-	0.00068	87 +/-	42
S -K	0.00271 +/-	0.00077	145 +/-	41
Cl-K	0.00959 +/-	0.00090	436 +/-	41
K -K	0.06659 +/-	0.00164	2429 +/-	60
Ca-K	0.03485 +/-	0.00311	1110 +/-	99
Fe-K	0.08287 +/-	0.00450	976 +/-	53
Ba-L	0.00189 +/-	0.00333	34 +/-	60
Ti-K	0.00375 +/-	0.00168	87 +/-	39
O -K	0.36037 +/-	0.00379	11423 +/-	120
C -K	0.07870 +/-	0.00218	1950 +/-	54

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 7

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0014	1.945	0.22	0.27	+/- 0.06	0.100
Mg-K	0.0718	1.510	8.20	10.84	+/- 0.12	3.753
Al-K	0.0187	1.488	1.90	2.79	+/- 0.08	0.869
Si-K	0.0953	1.305	8.13	12.43	+/- 0.19	3.723
P -K	0.0007	1.346	0.06	0.10	+/- 0.05	0.027
S -K	0.0014	1.210	0.10	0.17	+/- 0.05	0.045
Cl-K	0.0050	1.201	0.31	0.60	+/- 0.06	0.143
K -K	0.0349	1.127	1.85	3.93	+/- 0.10	0.846
Ca-K	0.0183	1.098	0.92	2.00	+/- 0.18	0.421
Fe-K	0.0434	1.181	1.69	5.13	+/- 0.28	0.773
Ba-L	0.0010	1.375	0.02	0.14	+/- 0.24	0.008
Ti-K	0.0020	1.181	0.09	0.23	+/- 0.10	0.041
O -K	0.1888	2.417	52.43	45.63	+/- 0.48	---
C -K	0.0412	3.815	24.08	15.73	+/- 0.44	11.022
Total			100.00	100.00		21.771

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 14:44:19 1998

980428.1.2

Refit Na-K' Na-K" P -K' P -K" S -K' S -K" Cl-K' Cl-K" Ba-L' Ba-L" Ti-
Refit Mg-K" Al-K' Al-K" Si-K" Ca-K' Fe-K" O -K" C -K' C -K"

Filter Fit Method

Chi-sqd = 1.12 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00580 +/-	0.00097	73 +/-	12
Mg-K	0.01566 +/-	0.00124	203 +/-	16
Al-K	0.01966 +/-	0.00156	252 +/-	20
Si-K	0.04351 +/-	0.00200	543 +/-	25
P -K	0.00016 +/-	0.00132	3 +/-	23
S -K	0.00633 +/-	0.00182	67 +/-	19
Cl-K	0.01023 +/-	0.00225	91 +/-	20
K -K	0.18962 +/-	0.01133	1357 +/-	81
Ca-K	0.10323 +/-	0.01250	644 +/-	78
Fe-K	0.33644 +/-	0.01951	777 +/-	45
Ba-L	0.00425 +/-	0.01302	16 +/-	49
Ti-K	0.01056 +/-	0.00638	49 +/-	30
O -K	0.16865 +/-	0.00451	1049 +/-	28
C -K	0.08589 +/-	0.00371	418 +/-	18

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0037	2.372	0.85	0.87	+/- 0.14	0.459
Mg-K	0.0099	1.763	1.61	1.74	+/- 0.14	0.871
Al-K	0.0124	1.522	1.57	1.89	+/- 0.15	0.851
Si-K	0.0274	1.303	2.86	3.57	+/- 0.16	1.548
P -K	0.0001	1.254	0.01	0.01	+/- 0.10	0.005
S -K	0.0040	1.132	0.32	0.45	+/- 0.13	0.172
Cl-K	0.0065	1.127	0.46	0.73	+/- 0.16	0.250
K -K	0.1195	1.065	7.33	12.73	+/- 0.76	3.964
Ca-K	0.0651	1.071	3.92	6.97	+/- 0.84	2.117
Fe-K	0.2121	1.143	9.77	24.24	+/- 1.41	5.284
Ba-L	0.0027	1.331	0.06	0.36	+/- 1.09	0.032
Ti-K	0.0067	1.144	0.36	0.76	+/- 0.46	0.194
O -K	0.1063	2.967	44.39	31.54	+/- 0.84	---
C -K	0.0541	2.610	26.49	14.13	+/- 0.61	14.321
Total			100.00	100.00		30.068

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 14:48:05 1998

980428.1.3

Refit _P -K' _P -K" _Cl-K' _Cl-K" _Ba-L' _Ba-L" _Ti-K' _Ti-K"
Refit _Mg-K" _Al-K' _Al-K" _Si-K" _S -K" _K -K' _K -K" _Ca-K" _Ba-L _C -K'
Filter Fit Method

Chi-sqd = 5.20 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01243 +/-	0.00088	1292 +/-	91
Mg-K	0.01619 +/-	0.00058	1752 +/-	63
Al-K	0.00844 +/-	0.00053	905 +/-	57
Si-K	0.01044 +/-	0.00054	1092 +/-	56
P -K	0.00030 +/-	0.00052	32 +/-	54
S -K	0.01212 +/-	0.00070	1059 +/-	61
Cl-K	0.00632 +/-	0.00068	472 +/-	51
K -K	0.00864 +/-	0.00082	519 +/-	49
Ca-K	0.12749 +/-	0.00176	6665 +/-	92
Fe-K	0.42449 +/-	0.00864	8205 +/-	167
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00047 +/-	0.00100	18 +/-	38
O -K	0.25709 +/-	0.00298	13388 +/-	155
C -K	0.11557 +/-	0.00197	4702 +/-	80

Adjustment Factors	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0076	2.521	1.72	1.91	+/- 0.13	0.851
Mg-K	0.0098	1.867	1.57	1.84	+/- 0.07	0.777
Al-K	0.0051	1.593	0.63	0.82	+/- 0.05	0.311
Si-K	0.0063	1.339	0.63	0.85	+/- 0.04	0.311
P -K	0.0002	1.261	0.02	0.02	+/- 0.04	0.008
S -K	0.0074	1.142	0.55	0.84	+/- 0.05	0.269
Cl-K	0.0038	1.144	0.26	0.44	+/- 0.05	0.127
K -K	0.0053	1.064	0.30	0.56	+/- 0.05	0.147
Ca-K	0.0775	1.036	4.17	8.04	+/- 0.11	2.058
Fe-K	0.2582	1.145	11.00	29.57	+/- 0.60	5.436
Ba-L	0.0000	1.275	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0003	1.117	0.01	0.03	+/- 0.07	0.007
O -K	0.1563	2.392	48.57	37.40	+/- 0.43	---
C -K	0.0703	2.516	30.58	17.68	+/- 0.30	15.111
Total			100.00	100.00		25.413

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 14:51:36 1998

980428.1.4

Refit P -K' P -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K" Al-K" Si-K" S -K' S -K" Cl-K" Ca-K" Ba-L C -K'
Filter Fit Method

Chi-sqd = 1.55 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01753 +/-	0.00104	1483 +/-	88
Mg-K	0.01458 +/-	0.00069	1284 +/-	61
Al-K	0.02251 +/-	0.00071	1964 +/-	62
Si-K	0.02848 +/-	0.00079	2424 +/-	67
P -K	0.00269 +/-	0.00066	224 +/-	55
S -K	0.02046 +/-	0.00093	1455 +/-	66
Cl-K	0.02033 +/-	0.00096	1234 +/-	58
K -K	0.02768 +/-	0.00246	1351 +/-	120
Ca-K	0.26831 +/-	0.00280	11415 +/-	119
Fe-K	0.26338 +/-	0.00833	4143 +/-	131
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00203 +/-	0.00113	64 +/-	35
O -K	0.21791 +/-	0.00281	9235 +/-	119
C -K	0.09411 +/-	0.00196	3117 +/-	65

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0102	2.315	2.15	2.36	+/- 0.14	1.035
Mg-K	0.0085	1.755	1.28	1.49	+/- 0.07	0.618
Al-K	0.0131	1.517	1.54	1.99	+/- 0.06	0.742
Si-K	0.0166	1.303	1.61	2.16	+/- 0.06	0.775
P -K	0.0016	1.246	0.13	0.20	+/- 0.05	0.063
S -K	0.0119	1.133	0.88	1.35	+/- 0.06	0.424
Cl-K	0.0118	1.139	0.79	1.35	+/- 0.06	0.383
K -K	0.0161	1.060	0.91	1.71	+/- 0.15	0.440
Ca-K	0.1564	1.054	8.58	16.48	+/- 0.17	4.138
Fe-K	0.1535	1.161	6.65	17.81	+/- 0.56	3.210
Ba-L	0.0000	1.350	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0012	1.167	0.06	0.14	+/- 0.08	0.029
O -K	0.1270	3.005	49.74	38.16	+/- 0.49	---
C -K	0.0548	2.698	25.69	14.80	+/- 0.31	12.396
Total			100.00	100.00		24.254

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 15:33:25 1998

980428.2.1

- vesicular slag on
Coke particle
- photo only - no thermal
print

Refit P -K' P -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K' Al-K' Al-K" Si-K' Si-K" Cl-K' K -K' K -K" Ca-K' Fe-K' Ti-
Filter Fit Method

Chi-sqd = 1.36 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00949 +/-	0.00100	743 +/-	78
Mg-K	0.01741 +/-	0.00070	1417 +/-	57
Al-K	0.00929 +/-	0.00063	749 +/-	50
Si-K	0.02494 +/-	0.00074	1962 +/-	58
P -K	0.00114 +/-	0.00068	88 +/-	52
S -K	0.08581 +/-	0.00226	5628 +/-	148
Cl-K	0.02295 +/-	0.00104	1285 +/-	58
K -K	0.01365 +/-	0.00100	614 +/-	45
Ca-K	0.29672 +/-	0.00296	11639 +/-	116
Fe-K	0.08852 +/-	0.00379	1284 +/-	55
Ba-L	0.00400 +/-	0.00216	90 +/-	48
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.26520 +/-	0.00343	10506 +/-	136
C -K	0.16087 +/-	0.00283	5002 +/-	88

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=45.96 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0048	2.107	0.81	1.02	+/-	0.11	0.384
Mg-K	0.0089	1.616	1.07	1.44	+/-	0.06	0.511
Al-K	0.0047	1.430	0.46	0.68	+/-	0.05	0.217
Si-K	0.0127	1.241	1.02	1.58	+/-	0.05	0.486
P -K	0.0006	1.201	0.04	0.07	+/-	0.04	0.019
S -K	0.0438	1.112	2.76	4.87	+/-	0.13	1.313
Cl-K	0.0117	1.150	0.69	1.35	+/-	0.06	0.329
K -K	0.0070	1.076	0.35	0.75	+/-	0.05	0.166
Ca-K	0.1514	1.075	7.38	16.28	+/-	0.16	3.513
Fe-K	0.0452	1.188	1.75	5.37	+/-	0.23	0.831
Ba-L	0.0020	1.400	0.04	0.29	+/-	0.15	0.018
Ti-K	0.0000	1.209	0.00	0.00	+/-	0.00	0.000
O -K	0.1353	3.281	50.43	44.39	+/-	0.57	---
C -K	0.0821	2.674	33.20	21.94	+/-	0.39	15.800
Total			100.00	100.00			23.587

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 15:49:22 1998

980428.3.1

Refit Na-K' Na-K" Mg-K' Mg-K" Si-K' Si-K" P -K' P -K" Cl-K' Cl-K" K -
Refit Ca-K' Ca-K" Ti-K C -K'

Filter Fit Method

Chi-sqd = 1.23 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00040 +/-	0.00037	37 +/-	34
Mg-K	0.00032 +/-	0.00052	30 +/-	49
Al-K	0.06639 +/-	0.00132	6113 +/-	122
Si-K	0.00458 +/-	0.00059	412 +/-	53
P -K	0.00006 +/-	0.00063	6 +/-	64
S -K	0.16458 +/-	0.00243	12349 +/-	182
Cl-K	0.00139 +/-	0.00075	90 +/-	48
K -K	0.00150 +/-	0.00082	78 +/-	42
Ca-K	0.42162 +/-	0.00330	18937 +/-	148
Fe-K	0.00777 +/-	0.00211	129 +/-	35
Ba-L	0.00362 +/-	0.00197	93 +/-	50
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.26647 +/-	0.00275	11923 +/-	123
C -K	0.06131 +/-	0.00166	2143 +/-	58

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	No. of Cations
Na-K	0.0002	2.098	0.04	0.04	+/- 0.04	0.015
Mg-K	0.0002	1.588	0.02	0.03	+/- 0.04	0.009
Al-K	0.0353	1.393	3.59	4.92	+/- 0.10	1.414
Si-K	0.0024	1.255	0.21	0.31	+/- 0.04	0.084
P -K	0.0000	1.172	0.00	0.00	+/- 0.04	0.001
S -K	0.0875	1.108	5.95	9.69	+/- 0.14	2.346
Cl-K	0.0007	1.171	0.05	0.09	+/- 0.05	0.019
K -K	0.0008	1.073	0.04	0.09	+/- 0.05	0.017
Ca-K	0.2240	1.079	11.88	24.17	+/- 0.19	4.681
Fe-K	0.0041	1.190	0.17	0.49	+/- 0.13	0.068
Ba-L	0.0019	1.424	0.04	0.27	+/- 0.15	0.015
Ti-K	0.0000	1.242	0.00	0.00	+/- 0.00	0.000
O -K	0.1416	3.494	60.91	49.48	+/- 0.51	---
C -K	0.0326	3.200	17.09	10.42	+/- 0.28	6.735
Total			100.00	100.00		15.405

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 15:50:41 1998

980428.3.2

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _P -K' _P -K" _Cl-K' _Cl-K" _K -K' _K -K" _Fe-
Refit _Si-K' _Ba-L
Refit _Ti-K
Filter Fit Method

Chi-sqd = 1.21 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00125 +/-	0.00038	116 +/-	35
Mg-K	0.00038 +/-	0.00051	37 +/-	50
Al-K	0.05329 +/-	0.00130	5099 +/-	125
Si-K	0.00954 +/-	0.00120	892 +/-	112
P -K	0.00130 +/-	0.00060	119 +/-	55
S -K	0.14338 +/-	0.00224	11178 +/-	175
Cl-K	0.00117 +/-	0.00071	79 +/-	47
K -K	0.00428 +/-	0.00082	229 +/-	44
Ca-K	0.39749 +/-	0.00469	18552 +/-	219
Fe-K	0.01426 +/-	0.00220	246 +/-	38
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.29639 +/-	0.00305	13780 +/-	142
C -K	0.07726 +/-	0.00198	2806 +/-	72

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0006	2.118	0.11	0.14	+/- 0.04	0.044
Mg-K	0.0002	1.598	0.02	0.03	+/- 0.04	0.010
Al-K	0.0276	1.401	2.74	3.87	+/- 0.09	1.065
Si-K	0.0049	1.251	0.42	0.62	+/- 0.08	0.164
P -K	0.0007	1.199	0.05	0.08	+/- 0.04	0.019
S -K	0.0742	1.110	4.91	8.24	+/- 0.13	1.911
Cl-K	0.0006	1.165	0.04	0.07	+/- 0.04	0.015
K -K	0.0022	1.072	0.12	0.24	+/- 0.05	0.045
Ca-K	0.2058	1.079	10.59	22.22	+/- 0.26	4.119
Fe-K	0.0074	1.192	0.30	0.88	+/- 0.14	0.117
Ba-L	0.0000	1.444	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.240	0.00	0.00	+/- 0.00	0.000
O -K	0.1535	3.367	61.69	51.67	+/- 0.53	---
C -K	0.0400	2.987	19.01	11.95	+/- 0.31	7.395
Total			100.00	100.00		14.904

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 15:52:17 1998

980428.3.3

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _P -K' _P -K" _Cl-K' _Cl-K" _Fe-K' _Fe-K" _Ba-
Refit _Al-K' _Si-K" _S -K" _K -K' _K -K" _Ti-K
Refit _Al-K"

Filter Fit Method

Chi-sqd = 1.22 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00214 +/-	0.00040	146 +/-	27
Mg-K	0.00290 +/-	0.00050	205 +/-	35
Al-K	0.00966 +/-	0.00067	676 +/-	47
Si-K	0.13337 +/-	0.00139	9110 +/-	95
P -K	0.00155 +/-	0.00074	104 +/-	49
S -K	0.03753 +/-	0.00110	2141 +/-	63
Cl-K	0.00532 +/-	0.00092	259 +/-	45
K -K	0.02032 +/-	0.00130	795 +/-	51
Ca-K	0.60556 +/-	0.00683	20672 +/-	233
Fe-K	0.00784 +/-	0.00285	99 +/-	36
Ba-L	0.00300 +/-	0.00253	59 +/-	50
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.08984 +/-	0.00215	3056 +/-	73
C -K	0.08097 +/-	0.00211	2151 +/-	56

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0013	2.026	0.26	0.27	+/- 0.05	0.162
Mg-K	0.0018	1.552	0.26	0.28	+/- 0.05	0.159
Al-K	0.0061	1.366	0.68	0.83	+/- 0.06	0.420
Si-K	0.0841	1.199	7.93	10.08	+/- 0.11	4.890
P -K	0.0010	1.235	0.09	0.12	+/- 0.06	0.053
S -K	0.0237	1.125	1.83	2.66	+/- 0.08	1.131
Cl-K	0.0034	1.133	0.24	0.38	+/- 0.07	0.146
K -K	0.0128	1.024	0.74	1.31	+/- 0.08	0.457
Ca-K	0.3818	1.065	22.42	40.68	+/- 0.46	13.830
Fe-K	0.0049	1.185	0.23	0.59	+/- 0.21	0.143
Ba-L	0.0019	1.448	0.04	0.27	+/- 0.23	0.027
Ti-K	0.0000	1.260	0.00	0.00	+/- 0.00	0.000
O -K	0.0566	4.975	38.91	28.18	+/- 0.67	---
C -K	0.0511	2.807	26.36	14.33	+/- 0.37	16.257
Total			100.00	100.00		37.675

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 16:33:13 1998

980427.1.1

Refit _Mg-K' _Mg-K" _P -K' _P -K" _S -K' _S -K" _Cl-K' _Cl-K" _K -K' _K -K" _Ba-
Refit _Al-K"

Filter Fit Method

Chi-sqd = 3.66 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01804 +/-	0.00087	1636 +/-	79
Mg-K	0.00440 +/-	0.00060	416 +/-	57
Al-K	0.02584 +/-	0.00073	2414 +/-	69
Si-K	0.13363 +/-	0.00199	12181 +/-	181
P -K	0.00160 +/-	0.00062	142 +/-	55
S -K	0.00463 +/-	0.00067	353 +/-	51
Cl-K	0.00197 +/-	0.00071	128 +/-	46
K -K	0.00494 +/-	0.00088	259 +/-	46
Ca-K	0.31384 +/-	0.00443	14299 +/-	202
Fe-K	0.24659 +/-	0.00784	4154 +/-	132
Ba-L	0.00221 +/-	0.00310	57 +/-	81
Ti-K	0.01479 +/-	0.00163	492 +/-	54
O -K	0.13835 +/-	0.00253	6279 +/-	115
C -K	0.08918 +/-	0.00206	3163 +/-	73

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0110	2.222	2.31	2.43	+/- 0.12	1.426
Mg-K	0.0027	1.702	0.41	0.46	+/- 0.06	0.252
Al-K	0.0157	1.467	1.86	2.30	+/- 0.07	1.149
Si-K	0.0811	1.277	8.03	10.36	+/- 0.15	4.967
P -K	0.0010	1.296	0.09	0.13	+/- 0.05	0.055
S -K	0.0028	1.166	0.22	0.33	+/- 0.05	0.138
Cl-K	0.0012	1.155	0.08	0.14	+/- 0.05	0.053
K -K	0.0030	1.057	0.18	0.32	+/- 0.06	0.109
Ca-K	0.1905	1.050	10.87	20.01	+/- 0.28	6.724
Fe-K	0.1497	1.158	6.76	17.34	+/- 0.55	4.181
Ba-L	0.0013	1.364	0.03	0.18	+/- 0.26	0.018
Ti-K	0.0090	1.171	0.48	1.05	+/- 0.12	0.296
O -K	0.0840	3.394	38.82	28.51	+/- 0.52	---
C -K	0.0541	3.041	29.86	16.46	+/- 0.38	18.464
Total			100.00	100.00		37.831

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 16:35:12 1998

980427.1.2

Refit P -K' P -K" S -K' S -K" Cl -K' Cl -K" Ba -L' Ba -L" Ti -K' Ti -K"
Refit Mg -K' Mg -K" Al -K" K -K' K -K" Ca -K" Fe -K' Ba -L C -K'
Filter Fit Method

Chi-sqd = 1.76 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00928 +/-	0.00076	890 +/-	73
Mg-K	0.01043 +/-	0.00059	1040 +/-	59
Al-K	0.01467 +/-	0.00061	1451 +/-	60
Si-K	0.02803 +/-	0.00135	2704 +/-	130
P -K	0.00245 +/-	0.00061	231 +/-	57
S -K	0.00827 +/-	0.00076	667 +/-	61
Cl-K	0.00580 +/-	0.00079	399 +/-	54
K -K	0.01442 +/-	0.00099	798 +/-	55
Ca-K	0.18438 +/-	0.00222	8891 +/-	107
Fe-K	0.48219 +/-	0.00993	8598 +/-	177
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00091 +/-	0.00117	33 +/-	42
O -K	0.15116 +/-	0.00227	7262 +/-	109
C -K	0.08802 +/-	0.00171	3303 +/-	64

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0061	2.558	1.60	Na-K	1.57	+/- 0.13	0.983
Mg-K	0.0069	1.881	1.25	Mg-K	1.30	+/- 0.07	0.769
Al-K	0.0097	1.592	1.34	Al-K	1.54	+/- 0.06	0.824
Si-K	0.0185	1.341	2.08	Si-K	2.48	+/- 0.12	1.275
P -K	0.0016	1.271	0.16	P -K	0.21	+/- 0.05	0.096
S -K	0.0055	1.145	0.46	S -K	0.63	+/- 0.06	0.281
Cl-K	0.0038	1.138	0.29	Cl-K	0.44	+/- 0.06	0.177
K -K	0.0095	1.049	0.60	K -K	1.00	+/- 0.07	0.368
Ca-K	0.1218	1.026	7.32	Ca-K	12.50	+/- 0.15	4.495
Fe-K	0.3186	1.129	15.12	Fe-K	35.96	+/- 0.74	9.280
Ba-L	0.0000	1.271	0.00	Ba-L	0.00	+/- 0.00	0.000
Ti-K	0.0006	1.113	0.03	Ti-K	0.07	+/- 0.09	0.020
O -K	0.0999	2.668	39.10	O -K	26.64	+/- 0.40	---
C -K	0.0581	2.696	30.65	C -K	15.68	+/- 0.30	18.815
Total			100.00	Total	100.00		37.384

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 16:38:50 1998

980427.1.3

Refit _P -K' _P -K" _S -K' _S -K" _Cl-K' _Cl-K" _Ba-L' _Ba-L" _Ti-K' _Ti-K"
Refit _Mg-K' _Mg-K" _K -K" _Ca-K' _Ba-L _Ti-K _C -K'
Filter Fit Method

Chi-sqd = 2.30 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01045 +/-	0.00076	1051 +/-	76
Mg-K	0.00889 +/-	0.00063	931 +/-	66
Al-K	0.01394 +/-	0.00127	1445 +/-	131
Si-K	0.04033 +/-	0.00144	4081 +/-	146
P -K	0.00272 +/-	0.00059	268 +/-	58
S -K	0.00730 +/-	0.00070	617 +/-	59
Cl-K	0.00667 +/-	0.00075	481 +/-	54
K -K	0.01611 +/-	0.00097	934 +/-	56
Ca-K	0.10544 +/-	0.00312	5332 +/-	158
Fe-K	0.54625 +/-	0.01000	10214 +/-	187
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.14363 +/-	0.00224	7236 +/-	113
C -K	0.09826 +/-	0.00175	3867 +/-	69

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=44.22 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0069	2.591	1.82	1.79	+/- 0.13	1.274
Mg-K	0.0059	1.903	1.07	1.12	+/- 0.08	0.753
Al-K	0.0092	1.603	1.28	1.48	+/- 0.13	0.896
Si-K	0.0266	1.349	2.99	3.59	+/- 0.13	2.095
P -K	0.0018	1.286	0.17	0.23	+/- 0.05	0.122
S -K	0.0048	1.155	0.41	0.56	+/- 0.05	0.285
Cl-K	0.0044	1.146	0.33	0.51	+/- 0.06	0.233
K -K	0.0106	1.058	0.67	1.13	+/- 0.07	0.472
Ca-K	0.0697	1.024	4.16	7.14	+/- 0.21	2.915
Fe-K	0.3610	1.122	16.93	40.50	+/- 0.74	11.874
Ba-L	0.0000	1.236	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.063	0.00	0.00	+/- 0.00	0.000
O -K	0.0949	2.471	34.21	23.45	+/- 0.37	---
C -K	0.0649	2.850	35.96	18.51	+/- 0.33	25.227
Total			100.00	100.00		46.146

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 16:50:44 1998

980427.2.1

Refit _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _K -K' _K -K" _Ba-
Refit _P -K _S -K" _Ca-K" _Ba-L _C -K'

Filter Fit Method

Chi-sqd = 1.93 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01282 +/-	0.00095	759 +/-	56
Mg-K	0.00590 +/-	0.00059	364 +/-	36
Al-K	0.00033 +/-	0.00053	20 +/-	33
Si-K	0.00511 +/-	0.00066	304 +/-	39
P -K	0.00000 +/-	0.00001	0 +/-	0
S -K	0.01519 +/-	0.00117	755 +/-	58
Cl-K	0.02201 +/-	0.00253	933 +/-	107
K -K	0.00746 +/-	0.00123	254 +/-	42
Ca-K	0.22706 +/-	0.00310	6744 +/-	92
Fe-K	0.57013 +/-	0.01375	6260 +/-	151
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00263 +/-	0.00153	57 +/-	33
O -K	0.08319 +/-	0.00221	2480 +/-	66
C -K	0.04817 +/-	0.00167	1125 +/-	39

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=45.10 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0097	2.660	3.25	2.59	+/- 0.19	2.545
Mg-K	0.0045	1.955	1.04	0.88	+/- 0.09	0.814
Al-K	0.0002	1.629	0.04	0.04	+/- 0.07	0.034
Si-K	0.0039	1.344	0.54	0.52	+/- 0.07	0.420
P -K	0.0000	1.240	0.00	0.00	+/- 0.00	0.000
S -K	0.0115	1.121	1.16	1.29	+/- 0.10	0.911
Cl-K	0.0167	1.116	1.52	1.86	+/- 0.21	1.188
K -K	0.0057	1.001	0.42	0.57	+/- 0.09	0.328
Ca-K	0.1725	1.003	12.44	17.29	+/- 0.24	9.743
Fe-K	0.4331	1.100	24.61	47.65	+/- 1.15	19.269
Ba-L	0.0000	1.252	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0020	1.091	0.13	0.22	+/- 0.13	0.103
O -K	0.0632	2.691	30.65	17.00	+/- 0.45	---
C -K	0.0366	2.755	24.20	10.08	+/- 0.35	18.953
Total			100.00	100.00		54.307

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 16:53:52 1998

980427.2.2

Refit _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _S -K' _S -K" _K -
Refit _Cl-K' _Ca-K' _Ba-L _Ti-K _O -K' _Pb-M"

Filter Fit Method

Chi-sqd = 4.63 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01652 +/-	0.00089	1547 +/-	83
Mg-K	0.00290 +/-	0.00052	283 +/-	51
Al-K	0.00130 +/-	0.00047	126 +/-	46
Si-K	0.00305 +/-	0.00062	287 +/-	58
P -K	0.00060 +/-	0.00067	55 +/-	61
S -K	0.01259 +/-	0.00530	988 +/-	416
Cl-K	0.01706 +/-	0.00251	1143 +/-	168
K -K	0.00269 +/-	0.00089	146 +/-	48
Ca-K	0.07248 +/-	0.00285	3404 +/-	134
Fe-K	0.34119 +/-	0.00934	5921 +/-	162
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.11549 +/-	0.00242	5440 +/-	114
C -K	0.06179 +/-	0.00174	2280 +/-	64
Pb-M	0.35234 +/-	0.00803	13999 +/-	319

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=45.10 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0117	2.289	3.72	2.68	+/- 0.14	2.199
Mg-K	0.0021	1.753	0.47	0.36	+/- 0.07	0.279
Al-K	0.0009	1.492	0.16	0.14	+/- 0.05	0.096
Si-K	0.0022	1.252	0.31	0.27	+/- 0.05	0.182
P -K	0.0004	1.168	0.05	0.05	+/- 0.06	0.030
S -K	0.0089	1.068	0.95	0.95	+/- 0.40	0.561
Cl-K	0.0121	1.247	1.36	1.51	+/- 0.22	0.802
K -K	0.0019	1.122	0.17	0.21	+/- 0.07	0.103
Ca-K	0.0513	1.060	4.33	5.43	+/- 0.21	2.563
Fe-K	0.2413	1.048	14.47	25.30	+/- 0.69	8.563
Ba-L	0.0000	1.246	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.071	0.00	0.00	+/- 0.00	0.000
O -K	0.0817	2.487	40.55	20.32	+/- 0.43	---
C -K	0.0437	2.454	28.52	10.73	+/- 0.30	16.878
Pb-M	0.2492	1.286	4.94	32.05	+/- 0.73	2.924
Total			100.00	100.00		35.180

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:09:22 1998

980426.1.1

Refit _Mg-K' _Mg-K" _Al-K' _Al-K" _P -K' _P -K" _S -K' _S -K" _Cl-K' _Cl-K" _Ba-
Refit _Na-K' _K -K' _K -K" _Fe-K' _Ti-K

Filter Fit Method

Chi-sqd = 1.03 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00504 +/-	0.00072	317 +/-	45
Mg-K	0.00787 +/-	0.00055	516 +/-	36
Al-K	0.00510 +/-	0.00063	332 +/-	41
Si-K	0.01768 +/-	0.00139	1122 +/-	88
P -K	0.00172 +/-	0.00081	106 +/-	50
S -K	0.03188 +/-	0.00128	1688 +/-	68
Cl-K	0.01250 +/-	0.00108	566 +/-	49
K -K	0.01780 +/-	0.00138	648 +/-	50
Ca-K	0.54328 +/-	0.00681	17230 +/-	216
Fe-K	0.14024 +/-	0.00887	1645 +/-	104
Ba-L	0.00095 +/-	0.00284	18 +/-	53
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.05777 +/-	0.00204	1812 +/-	64
C -K	0.15324 +/-	0.00274	3744 +/-	67
Pb-M	0.00494 +/-	0.00447	132 +/-	120

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0033	2.209	0.71	Na-K	0.74	+/- 0.10	0.634
Mg-K	0.0052	1.667	0.79	Mg-K	0.87	+/- 0.06	0.707
Al-K	0.0034	1.449	0.40	Al-K	0.49	+/- 0.06	0.359
Si-K	0.0118	1.243	1.14	Si-K	1.46	+/- 0.11	1.026
P -K	0.0011	1.194	0.10	P -K	0.14	+/- 0.06	0.087
S -K	0.0212	1.093	1.58	S -K	2.31	+/- 0.09	1.424
Cl-K	0.0083	1.107	0.57	Cl-K	0.92	+/- 0.08	0.512
K -K	0.0118	1.007	0.67	K -K	1.19	+/- 0.09	0.601
Ca-K	0.3610	1.049	20.73	Ca-K	37.85	+/- 0.47	18.628
Fe-K	0.0932	1.171	4.29	Fe-K	10.91	+/- 0.69	3.854
Ba-L	0.0006	1.417	0.01	Ba-L	0.09	+/- 0.27	0.013
Ti-K	0.0000	1.226	0.00	Ti-K	0.00	+/- 0.00	0.000
O -K	0.0384	5.072	26.71	O -K	19.47	+/- 0.69	---
C -K	0.1018	2.271	42.26	C -K	23.12	+/- 0.41	37.972
Pb-M	0.0033	1.313	0.05	Pb-M	0.43	+/- 0.39	0.041
Total			100.00	Total	100.00		65.857

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:10:48 1998

980426.1.2

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _S -
Refit _Ca-K' _Ca-K" _Ba-L _C -K'

Filter Fit Method

Chi-sqd = 0.92 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00097 +/-	0.00025	47 +/-	12
Mg-K	0.00177 +/-	0.00030	90 +/-	15
Al-K	0.00175 +/-	0.00036	88 +/-	18
Si-K	0.00736 +/-	0.00057	359 +/-	28
P -K	0.00402 +/-	0.00069	192 +/-	33
S -K	0.02911 +/-	0.00130	1188 +/-	53
Cl-K	0.01124 +/-	0.00118	392 +/-	41
K -K	0.01747 +/-	0.00157	489 +/-	44
Ca-K	0.38919 +/-	0.00434	9505 +/-	106
Fe-K	0.47929 +/-	0.01473	4330 +/-	133
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00051 +/-	0.00174	9 +/-	32
O -K	0.02038 +/-	0.00137	493 +/-	33
C -K	0.02599 +/-	0.00133	490 +/-	25
Pb-M	0.01096 +/-	0.00459	226 +/-	94

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 4

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0008	2.629	0.34	0.22	+/- 0.06	0.579
Mg-K	0.0015	1.902	0.43	0.29	+/- 0.05	0.721
Al-K	0.0015	1.585	0.32	0.24	+/- 0.05	0.538
Si-K	0.0064	1.313	1.07	0.84	+/- 0.07	1.796
P -K	0.0035	1.223	0.49	0.43	+/- 0.07	0.828
S -K	0.0252	1.100	3.10	2.77	+/- 0.12	5.216
Cl-K	0.0097	1.106	1.09	1.08	+/- 0.11	1.830
K -K	0.0151	0.997	1.38	1.51	+/- 0.14	2.326
Ca-K	0.3369	0.999	30.14	33.65	+/- 0.38	50.640
Fe-K	0.4149	1.092	29.12	45.30	+/- 1.39	48.928
Ba-L	0.0000	1.303	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0004	1.123	0.04	0.05	+/- 0.17	0.062
O -K	0.0176	3.609	14.29	6.37	+/- 0.43	---
C -K	0.0225	2.670	17.96	6.01	+/- 0.31	30.167
Pb-M	0.0095	1.319	0.22	1.25	+/- 0.52	0.364
Total			100.00	100.00		143.995

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:13:12 1998

980426.1.3

Refit Na-K' Na-K" Mg-K' Mg-K" P -K' P -K" S -K' S -K" Cl-K' Cl-K" Ba-
Refit K -K" Ca-K' Ca-K" Fe-K' O -K"

Filter Fit Method

Chi-sqd = 0.91 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00296 +/-	0.00052	132 +/-	23
Mg-K	0.00367 +/-	0.00080	170 +/-	37
Al-K	0.14725 +/-	0.00321	6723 +/-	147
Si-K	0.20660 +/-	0.00379	9208 +/-	169
P -K	0.00246 +/-	0.00092	108 +/-	40
S -K	0.01037 +/-	0.00118	387 +/-	44
Cl-K	0.00702 +/-	0.00120	223 +/-	38
K -K	0.02618 +/-	0.00172	669 +/-	44
Ca-K	0.16853 +/-	0.00323	3758 +/-	72
Fe-K	0.13944 +/-	0.01091	1151 +/-	90
Ba-L	0.00285 +/-	0.00452	36 +/-	57
Ti-K	0.00129 +/-	0.00228	21 +/-	37
O -K	0.13574 +/-	0.00222	2992 +/-	49
C -K	0.13523 +/-	0.00320	2323 +/-	55
Pb-M	0.01041 +/-	0.00414	195 +/-	78

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 7

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0016	1.923	0.25	0.30	+/- 0.05	0.200
Mg-K	0.0019	1.494	0.23	0.29	+/- 0.06	0.182
Al-K	0.0775	1.340	7.30	10.39	+/- 0.23	5.912
Si-K	0.1088	1.284	9.43	13.97	+/- 0.26	7.632
P -K	0.0013	1.342	0.11	0.17	+/- 0.07	0.086
S -K	0.0055	1.205	0.39	0.66	+/- 0.08	0.315
Cl-K	0.0037	1.202	0.24	0.44	+/- 0.08	0.192
K -K	0.0138	1.111	0.74	1.53	+/- 0.10	0.601
Ca-K	0.0887	1.085	4.56	9.63	+/- 0.18	3.687
Fe-K	0.0734	1.173	2.93	8.62	+/- 0.67	2.368
Ba-L	0.0015	1.377	0.03	0.21	+/- 0.33	0.023
Ti-K	0.0007	1.184	0.03	0.08	+/- 0.14	0.026
O -K	0.0715	3.500	29.65	25.02	+/- 0.41	---
C -K	0.0712	3.919	44.06	27.91	+/- 0.66	35.663
Pb-M	0.0055	1.443	0.07	0.79	+/- 0.31	0.059
Total			100.00	100.00		56.947

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:19:53 1998

980426.2.1

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _S -
Refit _Ca-K' _Ca-K" _Ba-L _Pb-M

Filter Fit Method

Chi-sqd = 1.36 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00006 +/-	0.00025	3 +/-	13
Mg-K	0.00021 +/-	0.00024	8 +/-	9
Al-K	0.00018 +/-	0.00026	7 +/-	11
Si-K	0.00064 +/-	0.00031	24 +/-	11
P -K	0.00034 +/-	0.00043	13 +/-	16
S -K	0.00389 +/-	0.00063	118 +/-	19
Cl-K	0.00202 +/-	0.00078	52 +/-	20
K -K	0.01138 +/-	0.00145	236 +/-	30
Ca-K	0.36847 +/-	0.00483	6642 +/-	87
Fe-K	0.43833 +/-	0.01680	2923 +/-	112
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00236 +/-	0.00190	31 +/-	25
O -K	0.00309 +/-	0.00067	56 +/-	12
C -K	0.16903 +/-	0.00310	2348 +/-	43
Pb-M	0.00000 +/-	0.00001	0 +/-	0

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0000	2.585	0.01	0.01	+/-	0.05	0.180
Mg-K	0.0002	1.871	0.03	0.03	+/-	0.04	0.474
Al-K	0.0001	1.566	0.02	0.02	+/-	0.03	0.300
Si-K	0.0005	1.306	0.06	0.07	+/-	0.03	0.851
P -K	0.0003	1.220	0.03	0.03	+/-	0.04	0.386
S -K	0.0031	1.103	0.27	0.34	+/-	0.06	3.832
Cl-K	0.0016	1.095	0.13	0.18	+/-	0.07	1.791
K -K	0.0090	0.994	0.59	0.90	+/-	0.11	8.282
Ca-K	0.2931	1.008	18.79	29.53	+/-	0.39	265.372
Fe-K	0.3487	1.124	17.89	39.17	+/-	1.50	252.646
Ba-L	0.0000	1.318	0.00	0.00	+/-	0.00	0.000
Ti-K	0.0019	1.139	0.11	0.21	+/-	0.17	1.608
O -K	0.0025	4.342	1.70	1.07	+/-	0.23	---
C -K	0.1345	2.115	60.37	28.43	+/-	0.52	852.661
Pb-M	0.0000	1.314	0.00	0.00	+/-	0.00	0.000
Total			100.00	100.00			1388.383

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:21:58 1998

980426.2.2

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _S -
Refit _K -K" _Ca-K" _Fe-K" _Ba-L _Ti-K _Pb-M

Filter Fit Method

Chi-sqd = 1.37 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00087 +/-	0.00028	44 +/-	14
Mg-K	0.00181 +/-	0.00031	94 +/-	16
Al-K	0.00015 +/-	0.00032	8 +/-	18
Si-K	0.00302 +/-	0.00042	150 +/-	21
P -K	0.00151 +/-	0.00050	73 +/-	24
S -K	0.00700 +/-	0.00072	291 +/-	30
Cl-K	0.00744 +/-	0.00085	264 +/-	30
K -K	0.01434 +/-	0.00123	409 +/-	35
Ca-K	0.33292 +/-	0.00390	8286 +/-	97
Fe-K	0.30884 +/-	0.00836	2844 +/-	77
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.01492 +/-	0.00154	368 +/-	38
C -K	0.30719 +/-	0.00386	5890 +/-	74
Pb-M	0.00000 +/-	0.00001	0 +/-	0

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0006	2.357	0.13	0.14	+/- 0.05	0.471
Mg-K	0.0013	1.742	0.18	0.22	+/- 0.04	0.685
Al-K	0.0001	1.491	0.01	0.02	+/- 0.03	0.044
Si-K	0.0021	1.266	0.19	0.27	+/- 0.04	0.717
P -K	0.0011	1.203	0.08	0.13	+/- 0.04	0.309
S -K	0.0049	1.100	0.34	0.54	+/- 0.06	1.268
Cl-K	0.0052	1.104	0.32	0.58	+/- 0.07	1.223
K -K	0.0101	1.017	0.52	1.02	+/- 0.09	1.968
Ca-K	0.2339	1.035	12.05	24.21	+/- 0.28	45.374
Fe-K	0.2169	1.159	8.98	25.14	+/- 0.68	33.817
Ba-L	0.0000	1.355	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.166	0.00	0.00	+/- 0.00	0.000
O -K	0.0105	4.878	6.38	5.11	+/- 0.53	---
C -K	0.2158	1.975	70.81	42.62	+/- 0.54	266.589
Pb-M	0.0000	1.315	0.00	0.00	+/- 0.00	0.000
Total			100.00	100.00		352.466

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:24:41 1998

980426.2.3

Refit Mg-K' Mg-K" P -K' P -K" S -K' S -K" Cl-K' Cl-K" K -K' K -K" Ba-
Refit Al-K" Ca-K' Ba-L

Filter Fit Method

Chi-sqd = 3.55 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.06933 +/-	0.00116	7714 +/-	129
Mg-K	0.00206 +/-	0.00060	239 +/-	70
Al-K	0.03062 +/-	0.00069	3515 +/-	79
Si-K	0.15720 +/-	0.00188	17614 +/-	211
P -K	0.00098 +/-	0.00056	107 +/-	61
S -K	0.00547 +/-	0.00064	513 +/-	60
Cl-K	0.00648 +/-	0.00065	519 +/-	52
K -K	0.00128 +/-	0.00068	82 +/-	44
Ca-K	0.14130 +/-	0.00291	7922 +/-	163
Fe-K	0.22287 +/-	0.00666	4622 +/-	138
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00020 +/-	0.00086	9 +/-	39
O -K	0.29408 +/-	0.00305	16297 +/-	169
C -K	0.06502 +/-	0.00178	2809 +/-	77
Pb-M	0.00311 +/-	0.00225	147 +/-	106

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 7

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0388	2.149	7.31	8.35	+/-	0.14	3.455
Mg-K	0.0012	1.760	0.17	0.20	+/-	0.06	0.080
Al-K	0.0172	1.500	1.92	2.57	+/-	0.06	0.908
Si-K	0.0881	1.306	8.24	11.50	+/-	0.14	3.898
P -K	0.0005	1.333	0.05	0.07	+/-	0.04	0.022
S -K	0.0031	1.195	0.23	0.37	+/-	0.04	0.109
Cl-K	0.0036	1.188	0.24	0.43	+/-	0.04	0.116
K -K	0.0007	1.097	0.04	0.08	+/-	0.04	0.019
Ca-K	0.0792	1.066	4.24	8.44	+/-	0.17	2.004
Fe-K	0.1249	1.162	5.23	14.51	+/-	0.43	2.473
Ba-L	0.0000	1.336	0.00	0.00	+/-	0.00	0.000
Ti-K	0.0001	1.158	0.01	0.01	+/-	0.06	0.003
O -K	0.1648	2.449	50.75	40.35	+/-	0.42	---
C -K	0.0364	3.532	21.56	12.87	+/-	0.35	10.194
Pb-M	0.0017	1.431	0.02	0.25	+/-	0.18	0.011
Total			100.00	100.00			23.292

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:30:00 1998

980426.3.1

Refit _P -K' _P -K" _S -K' _S -K" _Cl-K' _Cl-K" _K -K' _K -K" _Ba-L' _Ba-L" _Ti-
Refit _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K" _Cl-K _Ti-K _Pb-M
Filter Fit Method

Chi-sqd = 5.25 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01051 +/-	0.00075	1158 +/-	83
Mg-K	0.00541 +/-	0.00049	621 +/-	56
Al-K	0.00588 +/-	0.00050	668 +/-	57
Si-K	0.13718 +/-	0.00110	15208 +/-	122
P -K	0.00139 +/-	0.00054	151 +/-	58
S -K	0.00552 +/-	0.00060	511 +/-	56
Cl-K	0.00000 +/-	0.00001	0 +/-	0
K -K	0.00087 +/-	0.00069	55 +/-	44
Ca-K	0.27418 +/-	0.00368	15209 +/-	204
Fe-K	0.23256 +/-	0.00682	4771 +/-	140
Ba-L	0.00137 +/-	0.00178	43 +/-	56
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.21523 +/-	0.00281	11802 +/-	154
C -K	0.10990 +/-	0.00208	4697 +/-	89
Pb-M	0.00000 +/-	0.00001	0 +/-	0

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0059	2.276	1.18	1.35	+/-	0.10	0.598
Mg-K	0.0031	1.716	0.43	0.52	+/-	0.05	0.220
Al-K	0.0033	1.478	0.37	0.49	+/-	0.04	0.185
Si-K	0.0775	1.269	7.03	9.83	+/-	0.08	3.564
P -K	0.0008	1.291	0.07	0.10	+/-	0.04	0.033
S -K	0.0031	1.166	0.23	0.36	+/-	0.04	0.115
Cl-K	0.0000	1.154	0.00	0.00	+/-	0.00	0.000
K -K	0.0005	1.068	0.03	0.05	+/-	0.04	0.014
Ca-K	0.1549	1.058	8.21	16.39	+/-	0.22	4.162
Fe-K	0.1314	1.168	5.52	15.35	+/-	0.45	2.798
Ba-L	0.0008	1.366	0.02	0.11	+/-	0.14	0.008
Ti-K	0.0000	1.171	0.00	0.00	+/-	0.00	0.000
O -K	0.1216	3.102	47.32	37.72	+/-	0.49	---
C -K	0.0621	2.854	29.61	17.72	+/-	0.34	15.016
Pb-M	0.0000	1.402	0.00	0.00	+/-	0.00	0.000
Total			100.00	100.00			26.715

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:31:36 1998

980426.3.2

Refit Mg-K' Mg-K" P -K' P -K" S -K' S -K" Cl-K' Cl-K" K -K' K -K" Ba-
Refit Al-K" Ti-K

Filter Fit Method

Chi-sqd = 1.41 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.02261 +/-	0.00090	2309 +/-	92
Mg-K	0.00444 +/-	0.00057	472 +/-	61
Al-K	0.02059 +/-	0.00064	2169 +/-	68
Si-K	0.13271 +/-	0.00183	13642 +/-	188
P -K	0.00246 +/-	0.00059	246 +/-	59
S -K	0.01982 +/-	0.00080	1703 +/-	69
Cl-K	0.00179 +/-	0.00061	132 +/-	45
K -K	0.00299 +/-	0.00073	176 +/-	43
Ca-K	0.31310 +/-	0.00406	16103 +/-	209
Fe-K	0.10204 +/-	0.00568	1941 +/-	108
Ba-L	0.00120 +/-	0.00179	35 +/-	52
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.21200 +/-	0.00283	10778 +/-	144
C -K	0.15643 +/-	0.00250	6199 +/-	99
Pb-M	0.00783 +/-	0.00272	339 +/-	118

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg

Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0117	2.098	1.99	2.46	+/-	0.10	1.074
Mg-K	0.0023	1.633	0.29	0.38	+/-	0.05	0.155
Al-K	0.0107	1.425	1.05	1.52	+/-	0.05	0.566
Si-K	0.0689	1.248	5.69	8.59	+/-	0.12	3.068
P -K	0.0013	1.269	0.10	0.16	+/-	0.04	0.052
S -K	0.0103	1.155	0.69	1.19	+/-	0.05	0.372
Cl-K	0.0009	1.164	0.06	0.11	+/-	0.04	0.031
K -K	0.0015	1.076	0.08	0.17	+/-	0.04	0.043
Ca-K	0.1625	1.073	8.09	17.43	+/-	0.23	4.361
Fe-K	0.0530	1.184	2.09	6.27	+/-	0.35	1.126
Ba-L	0.0006	1.397	0.01	0.09	+/-	0.13	0.006
Ti-K	0.0000	1.208	0.00	0.00	+/-	0.00	0.000
O -K	0.1100	3.480	44.53	38.29	+/-	0.51	---
C -K	0.0812	2.805	35.28	22.78	+/-	0.36	19.016
Pb-M	0.0041	1.386	0.05	0.56	+/-	0.20	0.027
Total			100.00	100.00			29.898

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:35:00 1998

980426.3.3

Refit P -K' P -K" S -K' S -K" Cl-K' Cl-K" K -K' K -K" Ba-L' Ba-L" Ti-
Refit Mg-K" Cl-K Ca-K' Ca-K" C -K' Pb-M

Filter Fit Method

Chi-sqd = 1.41 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01379 +/-	0.00088	1347 +/-	86
Mg-K	0.00815 +/-	0.00075	828 +/-	76
Al-K	0.05160 +/-	0.00198	5202 +/-	200
Si-K	0.19867 +/-	0.00236	19547 +/-	232
P -K	0.00063 +/-	0.00058	60 +/-	56
S -K	0.00068 +/-	0.00062	57 +/-	52
Cl-K	0.00000 +/-	0.00001	0 +/-	0
K -K	0.00034 +/-	0.00076	20 +/-	44
Ca-K	0.36537 +/-	0.00293	17987 +/-	144
Fe-K	0.14582 +/-	0.00654	2656 +/-	119
Ba-L	0.00097 +/-	0.00240	27 +/-	68
Ti-K	0.00042 +/-	0.00120	16 +/-	45
O -K	0.16371 +/-	0.00220	7967 +/-	107
C -K	0.04986 +/-	0.00140	1891 +/-	53
Pb-M	0.00000 +/-	0.00001	0 +/-	0

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0083	2.109	1.66	1.74	+/- 0.11	0.864
Mg-K	0.0049	1.624	0.72	0.79	+/- 0.07	0.372
Al-K	0.0309	1.420	3.57	4.39	+/- 0.17	1.854
Si-K	0.1191	1.270	11.81	15.12	+/- 0.18	6.132
P -K	0.0004	1.333	0.04	0.05	+/- 0.05	0.018
S -K	0.0004	1.192	0.03	0.05	+/- 0.04	0.017
Cl-K	0.0000	1.168	0.00	0.00	+/- 0.00	0.000
K -K	0.0002	1.042	0.01	0.02	+/- 0.05	0.006
Ca-K	0.2190	1.064	12.75	23.30	+/- 0.19	6.621
Fe-K	0.0874	1.167	4.01	10.20	+/- 0.46	2.081
Ba-L	0.0006	1.391	0.01	0.08	+/- 0.20	0.007
Ti-K	0.0003	1.196	0.01	0.03	+/- 0.09	0.007
O -K	0.0981	3.436	46.21	33.72	+/- 0.45	---
C -K	0.0299	3.512	19.17	10.50	+/- 0.29	9.954
Pb-M	0.0000	1.431	0.00	0.00	+/- 0.00	0.000
Total			100.00	100.00		27.933

The number of cation results are based upon 24 Oxygen atoms

Thu May 28 10:37:07 1998

980426.3.4

Refit _Mg-K' _Mg-K" _Al-K' _Al-K" _P -K' _P -K" _S -K' _S -K" _Cl-K' _Cl-K" _K
Refit _Si-K" _Ca-K' _Ca-K" _Ba-L _Ti-K _C -K'
Filter Fit Method

Chi-sqd = 1.88 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00382 +/-	0.00054	283 +/-	40
Mg-K	0.00297 +/-	0.00038	229 +/-	29
Al-K	0.00471 +/-	0.00041	360 +/-	32
Si-K	0.02112 +/-	0.00060	1572 +/-	45
P -K	0.00128 +/-	0.00048	94 +/-	35
S -K	0.00576 +/-	0.00069	358 +/-	43
Cl-K	0.00171 +/-	0.00066	92 +/-	35
K -K	0.00408 +/-	0.00089	175 +/-	38
Ca-K	0.19066 +/-	0.00250	7102 +/-	93
Fe-K	0.63970 +/-	0.01241	8813 +/-	171
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.06590 +/-	0.00182	2426 +/-	67
C -K	0.05416 +/-	0.00160	1554 +/-	46
Pb-M	0.00415 +/-	0.00240	130 +/-	75

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=43.31 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0030	2.803	1.09	0.84	+/-	0.12	1.050
Mg-K	0.0023	2.009	0.58	0.47	+/-	0.06	0.554
Al-K	0.0037	1.658	0.68	0.61	+/-	0.05	0.653
Si-K	0.0166	1.366	2.42	2.26	+/-	0.06	2.320
P -K	0.0010	1.276	0.12	0.13	+/-	0.05	0.119
S -K	0.0045	1.139	0.48	0.51	+/-	0.06	0.462
Cl-K	0.0013	1.125	0.13	0.15	+/-	0.06	0.123
K -K	0.0032	1.001	0.25	0.32	+/-	0.07	0.236
Ca-K	0.1494	0.994	11.13	14.86	+/-	0.19	10.679
Fe-K	0.5014	1.089	29.36	54.61	+/-	1.06	28.170
Ba-L	0.0000	1.226	0.00	0.00	+/-	0.00	0.000
Ti-K	0.0000	1.056	0.00	0.00	+/-	0.00	0.000
O -K	0.0516	2.581	25.01	13.33	+/-	0.37	---
C -K	0.0424	2.702	28.68	11.47	+/-	0.34	27.515
Pb-M	0.0033	1.363	0.06	0.44	+/-	0.26	0.062
Total			100.00	100.00			71.943

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:40:37 1998

980430.1.1

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _S -
Refit _Al-K' _Fe-K" _Ti-K
Refit _Ba-L

Filter Fit Method

Chi-sqd = 1.77 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00020 +/-	0.00023	27 +/-	31
Mg-K	0.00074 +/-	0.00024	106 +/-	34
Al-K	0.00000 +/-	0.00001	0 +/-	0
Si-K	0.00094 +/-	0.00025	129 +/-	35
P -K	0.00034 +/-	0.00027	45 +/-	36
S -K	0.00058 +/-	0.00031	68 +/-	36
Cl-K	0.00060 +/-	0.00032	59 +/-	31
K -K	0.00034 +/-	0.00034	26 +/-	26
Ca-K	0.00639 +/-	0.00048	441 +/-	33
Fe-K	0.01183 +/-	0.00125	302 +/-	32
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.00848 +/-	0.00108	575 +/-	73
C -K	0.96957 +/-	0.00437	50992 +/-	230

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element Wt %	Wt % Err. (1-Sigma)	NO. of Cations
Na-K	0.0002	1.711	0.02	0.03	+/- 0.03	0.111
Mg-K	0.0006	1.373	0.04	0.09	+/- 0.03	0.314
Al-K	0.0000	1.251	0.00	0.00	+/- 0.00	0.000
Si-K	0.0008	1.142	0.04	0.09	+/- 0.03	0.287
P-K	0.0003	1.139	0.01	0.03	+/- 0.03	0.093
S-K	0.0005	1.083	0.02	0.05	+/- 0.03	0.148
Cl-K	0.0005	1.121	0.02	0.06	+/- 0.03	0.143
K-K	0.0003	1.104	0.01	0.03	+/- 0.03	0.073
Ca-K	0.0056	1.083	0.19	0.60	+/- 0.05	1.300
Fe-K	0.0103	1.226	0.28	1.77	+/- 0.13	1.955
Ba-L	0.0000	1.367	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.193	0.00	0.00	+/- 0.00	0.000
O-K	0.0074	6.010	3.43	4.45	+/- 0.57	---
C-K	0.8460	1.103	95.93	93.29	+/- 0.42	670.331
Total			100.00	100.00		674.755

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:41:52 1998

980430.1.2

Refit Al-K' Al-K" Si-K' Si-K" P -K' P -K" Ba-L' Ba-L" Ti-K' Ti-K"
Refit Mg-K' Mg-K" S -K' Cl-K' Cl-K" K -K' K -K" Ca-K' Ca-K" Fe-K' Fe-K" Ba-
Filter Fit Method

Chi-sqd = 1.46 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00848 +/-	0.00084	735 +/-	73
Mg-K	0.01003 +/-	0.00059	906 +/-	53
Al-K	0.00205 +/-	0.00051	185 +/-	46
Si-K	0.00823 +/-	0.00059	720 +/-	52
P -K	0.00464 +/-	0.00066	395 +/-	56
S -K	0.06851 +/-	0.00122	5007 +/-	89
Cl-K	0.01699 +/-	0.00091	1060 +/-	57
K -K	0.05739 +/-	0.00257	2882 +/-	129
Ca-K	0.15208 +/-	0.00215	6662 +/-	94
Fe-K	0.10953 +/-	0.00395	1775 +/-	64
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00097 +/-	0.00097	31 +/-	31
O -K	0.08443 +/-	0.00233	3626 +/-	100
C -K	0.47666 +/-	0.00398	15910 +/-	133

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0043	2.006	0.59	0.87	+/- 0.09	0.720
Mg-K	0.0051	1.554	0.51	0.80	+/- 0.05	0.624
Al-K	0.0011	1.384	0.08	0.15	+/- 0.04	0.102
Si-K	0.0042	1.209	0.28	0.51	+/- 0.04	0.345
P -K	0.0024	1.175	0.14	0.28	+/- 0.04	0.171
S -K	0.0351	1.098	1.88	3.86	+/- 0.07	2.283
Cl-K	0.0087	1.141	0.44	0.99	+/- 0.05	0.532
K -K	0.0294	1.089	1.28	3.21	+/- 0.14	1.556
Ca-K	0.0780	1.085	3.29	8.47	+/- 0.12	4.007
Fe-K	0.0562	1.199	1.88	6.73	+/- 0.24	2.287
Ba-L	0.0000	1.394	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0005	1.195	0.02	0.06	+/- 0.06	0.024
O -K	0.0133	4.675	19.72	36.24	+/- 0.56	---
C -K	0.2444	2.202	69.88	53.84	+/- 0.45	85.042
Total			100.00	100.00		97.693

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:44:43 1998

980430.1.3

Refit _Mg-K' _Mg-K' _Al-K' _Al-K' _P -K' _P -K' _S -K' _S -K' _Cl-K' _Cl-K' _K -
Refit _Si-K' _Ca-K' _Ba-L
Refit _Ti-K

Filter Fit Method

Chi-sqd = 11.49 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01174 +/-	0.00070	1482 +/-	88
Mg-K	0.00313 +/-	0.00043	412 +/-	57
Al-K	0.00276 +/-	0.00041	361 +/-	53
Si-K	0.01057 +/-	0.00049	1344 +/-	62
P -K	0.00335 +/-	0.00050	416 +/-	62
S -K	0.00543 +/-	0.00060	577 +/-	64
Cl-K	0.00235 +/-	0.00061	214 +/-	55
K -K	0.00501 +/-	0.00071	367 +/-	52
Ca-K	0.04254 +/-	0.00111	2711 +/-	71
Fe-K	0.60924 +/-	0.00899	14362 +/-	212
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.23086 +/-	0.00280	14418 +/-	175
C -K	0.07302 +/-	0.00173	3545 +/-	64

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

SAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0081	2.830	2.38	2.30	+/- 0.14	1 232
Mg-K	0.0022	2.049	0.43	0.44	+/- 0.06	0.225
Al-K	0.0019	1.686	0.28	0.32	+/- 0.05	0.147
Si-K	0.0073	1.387	0.86	1.01	+/- 0.05	0.445
P -K	0.0023	1.269	0.23	0.30	+/- 0.04	0.119
S -K	0.0038	1.157	0.32	0.43	+/- 0.05	0.167
Cl-K	0.0016	1.146	0.13	0.19	+/- 0.05	0.065
K -K	0.0035	1.058	0.22	0.37	+/- 0.05	0.116
Ca-K	0.0294	1.012	1.77	2.99	+/- 0.08	0.915
Fe-K	0.4214	1.113	20.00	46.89	+/- 0.69	10 341
Ba-L	0.0000	1.192	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.028	0.00	0.00	+/- 0.00	0.000
O -K	0.1597	1.952	46.41	31.17	+/- 0.38	---
C -K	0.0505	2.692	26.97	13.60	+/- 0.32	13.945
Total			100.00	100.00		27.716

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:48:44 1998

980430.1.4

Refit Na-K' Na-K" F -K' F -K" S -K' S -K" Cl-K' Cl-K" K -K' K -K" Ba-
Refit Si-K' Ti-K' O -K' C -K'

Filter Fit Method

Chi-sqd = 1.33 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative K-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00139 +/-	0.00052	123 +/-	46
Mg-K	0.02346 +/-	0.00149	2150 +/-	137
Al-K	0.15305 +/-	0.00259	13917 +/-	235
Si-K	0.14239 +/-	0.00139	12644 +/-	123
P -K	0.00069 +/-	0.00059	60 +/-	51
S -K	0.00144 +/-	0.00063	107 +/-	47
Cl-K	0.00104 +/-	0.00068	66 +/-	43
K -K	0.00218 +/-	0.00084	112 +/-	43
Ca-K	0.43630 +/-	0.00501	19404 +/-	223
Fe-K	0.02692 +/-	0.00468	443 +/-	77
Ba-L	0.00072 +/-	0.00211	18 +/-	53
Ti-K	0.00000 +/-	0.00001	0 +/-	0
C -K	0.14515 +/-	0.00158	6328 +/-	69
C -K	0.06527 +/-	0.00162	2212 +/-	55

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 KV Take-off Angle=42.37 deg
Number of Iterations = 6

Element	K-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	NO. of Cations
Na-K	0.0008	1.953	0.14	0.15	+/- 0.06	0.079
Mg-K	0.0134	1.505	1.74	2.02	+/- 0.13	0.975
Al-K	0.0877	1.362	9.25	11.94	+/- 0.20	5.185
Si-K	0.0816	1.310	7.94	10.68	+/- 0.10	4.456
P -K	0.0004	1.327	0.04	0.05	+/- 0.04	0.020
S -K	0.0008	1.189	0.06	0.10	+/- 0.04	0.036
Cl-K	0.0006	1.174	0.04	0.07	+/- 0.05	0.023
K -K	0.0012	1.067	0.07	0.13	+/- 0.05	0.040
Ca-K	0.2499	1.073	13.99	26.81	+/- 0.31	7.838
Fe-K	0.0154	1.180	0.68	1.82	+/- 0.32	0.382
Ba-L	0.0004	1.416	0.01	0.06	+/- 0.17	0.005
Ti-K	0.0000	1.234	0.00	0.00	+/- 0.00	0.000
C -K	0.0631	3.942	42.75	33.78	+/- 0.36	---
C -K	0.0374	3.577	23.26	13.38	+/- 0.33	13.046
Total			100.00	100.00		32.084

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 11:50:16 1998

980430.1.5

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _Cl-
Refit _K -K' _K -K" _Ca-K' _Ba-L' _O -K' _O -K" _C -K'
Filter Fit Method

Chi-sqd = 1.41 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00022 +/-	0.00020	10 +/-	9
Mg-K	0.00064 +/-	0.00023	30 +/-	11
Al-K	0.00049 +/-	0.00027	24 +/-	13
Si-K	0.00484 +/-	0.00046	221 +/-	21
P -K	0.00133 +/-	0.00059	60 +/-	26
S -K	0.02576 +/-	0.00195	979 +/-	74
Cl-K	0.00861 +/-	0.00108	279 +/-	35
F -K	0.02355 +/-	0.00161	615 +/-	42
Ca-K	0.35138 +/-	0.00426	7994 +/-	97
Fe-K	0.55276 +/-	0.01580	4653 +/-	133
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00296 +/-	0.00181	49 +/-	30
O -K	0.00847 +/-	0.00067	189 +/-	15
C -K	0.01898 +/-	0.00115	330 +/-	20

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 4

Element	k-ratio (calc.)	ZAF	Atom %	Element Wt %	Wt % Err. (1-Sigma)	NO. OF Cations
Na-K	0.0002	1.753	0.10	0.05	+/- 0.05	0.354
Mg-K	0.0006	1.971	0.19	0.12	+/- 0.04	0.690
Al-K	0.0004	1.626	0.11	0.07	+/- 0.04	0.394
Si-K	0.0011	1.335	0.84	0.54	+/- 0.06	3.064
P -K	0.0012	1.234	0.19	0.15	+/- 0.07	0.706
S -K	0.0035	1.103	3.22	2.59	+/- 0.20	11.790
Cl-K	0.0078	1.097	0.97	0.85	+/- 0.11	3.513
F -K	0.0115	0.986	2.16	1.12	+/- 0.14	7.905
Ca-K	0.3205	0.985	31.40	31.58	+/- 0.38	114.960
Fe-K	0.5812	1.078	38.76	51.33	+/- 1.55	141.405
Ba-L	0.0000	1.279	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0027	1.101	0.25	0.30	+/- 0.18	0.904
O -K	0.0077	1.105	5.55	7.53	+/- 0.21	---
C -K	0.0189	1.560	15.27	4.50	+/- 0.28	55.915
Total			100.00	100.00		342.150

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 13:30:19 1998

980430.2.1

Refit _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _Cl-K' _Cl-K" _Ba-
Refit _S -K" _K -K" _Ba-L

Filter Fit Method

Chi-sqd = 1.93 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00362 +/-	0.00049	346 +/-	47
Mg-K	0.00231 +/-	0.00034	230 +/-	34
Al-K	0.00153 +/-	0.00035	151 +/-	34
Si-K	0.00543 +/-	0.00045	521 +/-	43
P -K	0.00205 +/-	0.00050	193 +/-	47
S -K	0.02041 +/-	0.00080	1639 +/-	64
Cl-K	0.00409 +/-	0.00073	280 +/-	50
K -K	0.02640 +/-	0.00107	1456 +/-	59
Ca-K	0.06910 +/-	0.00289	3324 +/-	139
Fe-K	0.70468 +/-	0.01124	12539 +/-	200
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00011 +/-	0.00114	4 +/-	42
O -K	0.09255 +/-	0.00197	4364 +/-	93
C -K	0.06771 +/-	0.00172	2481 +/-	63

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0027	2.911	0.98	0.80	+/-	0.11	0.846
Mg-K	0.0018	2.069	0.42	0.36	+/-	0.05	0.363
Al-K	0.0012	1.695	0.20	0.20	+/-	0.04	0.177
Si-K	0.0041	1.385	0.57	0.57	+/-	0.05	0.493
P -K	0.0016	1.277	0.18	0.20	+/-	0.05	0.156
S -K	0.0155	1.142	1.55	1.77	+/-	0.07	1.340
Cl-K	0.0031	1.135	0.28	0.35	+/-	0.06	0.241
K -K	0.0200	1.039	1.49	2.08	+/-	0.08	1.294
Ca-K	0.0524	1.000	3.67	5.24	+/-	0.22	3.178
Fe-K	0.5346	1.088	29.21	58.16	+/-	0.93	25.310
Ba-L	0.0000	1.183	0.00	0.00	+/-	0.00	0.000
Ti-K	0.0001	1.020	0.01	0.01	+/-	0.09	0.004
O -K	0.0702	2.250	27.70	15.80	+/-	0.34	---
C -K	0.0514	2.814	33.75	14.45	+/-	0.37	29.245
Total			100.00	100.00			62.648

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 13:37:44 1998

980430.2.2

Refit Mg-K' Mg-K" Al-K' Al-K" Si-K' Si-K" P -K' P -K" Ba-L' Ba-L" Ti-
Refit S -K" Cl-K" K -K" Ca-K' Ca-K" Ti-K

Filter Fit Method

Chi-sqd = 1.70 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.01191 +/-	0.00088	803 +/-	59
Mg-K	0.00496 +/-	0.00057	348 +/-	40
Al-K	0.00237 +/-	0.00053	165 +/-	37
Si-K	0.00796 +/-	0.00065	542 +/-	44
P -K	0.00471 +/-	0.00073	312 +/-	48
S -K	0.02903 +/-	0.00111	1649 +/-	63
Cl-K	0.01141 +/-	0.00101	554 +/-	49
K -K	0.02598 +/-	0.00126	1014 +/-	49
Ca-K	0.10630 +/-	0.00215	3618 +/-	73
Fe-K	0.38625 +/-	0.01064	4863 +/-	134
Ba-L	0.00416 +/-	0.00260	80 +/-	50
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.11877 +/-	0.00291	3962 +/-	97
C -K	0.28620 +/-	0.00366	7422 +/-	95

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0067	2.403	1.28	1.61	+/- 0.12	1.198
Mg-K	0.0028	1.796	0.38	0.50	+/- 0.06	0.353
Al-K	0.0013	1.532	0.14	0.20	+/- 0.05	0.129
Si-K	0.0045	1.297	0.38	0.58	+/- 0.05	0.354
P -K	0.0027	1.232	0.19	0.33	+/- 0.05	0.180
S -K	0.0163	1.128	1.05	1.84	+/- 0.07	0.983
Cl-K	0.0064	1.144	0.38	0.73	+/- 0.07	0.354
K -K	0.0146	1.075	0.74	1.57	+/- 0.08	0.687
Ca-K	0.0598	1.050	2.87	6.28	+/- 0.13	2.680
Fe-K	0.2173	1.160	8.27	25.22	+/- 0.69	7.726
Ba-L	0.0023	1.313	0.04	0.31	+/- 0.19	0.038
Ti-K	0.0000	1.110	0.00	0.00	+/- 0.00	0.000
O -K	0.0668	3.358	25.70	22.44	+/- 0.55	---
C -K	0.1610	2.384	58.57	38.39	+/- 0.49	54.685
Total			100.00	100.00		69.370

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 13:49:31 1998

980430.3.1

Refit _Na-K' _Na-K" _Mg-K' _Mg-K" _Al-K' _Al-K" _Si-K' _Si-K" _P -K' _P -K" _S -
Refit _Ca-K' _Ba-L _Ti-K _O -K" _C -K"
Refit _Ca-K"

Filter Fit Method

Chi-sqd = 0.69 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00139 +/-	0.00122	8 +/-	7
Mg-K	0.00667 +/-	0.00167	41 +/-	10
Al-K	0.00096 +/-	0.00192	6 +/-	12
Si-K	0.02393 +/-	0.00275	139 +/-	16
P -K	0.00742 +/-	0.00300	42 +/-	17
S -K	0.03914 +/-	0.00474	190 +/-	23
Cl-K	0.00869 +/-	0.00458	36 +/-	19
K -K	0.04349 +/-	0.00660	146 +/-	22
Ca-K	0.26329 +/-	0.01169	767 +/-	34
Fe-K	0.22016 +/-	0.04366	238 +/-	47
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.13183 +/-	0.00631	376 +/-	18
C -K	0.25304 +/-	0.00857	562 +/-	19

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0008	2.274	0.14	0.18	+/- 0.15	0.102
Mg-K	0.0037	1.694	0.48	0.63	+/- 0.16	0.345
Al-K	0.0005	1.465	0.05	0.08	+/- 0.16	0.039
Si-K	0.0133	1.256	1.09	1.67	+/- 0.19	0.793
P -K	0.0041	1.212	0.30	0.50	+/- 0.20	0.215
S -K	0.0217	1.117	1.39	2.43	+/- 0.29	1.011
Cl-K	0.0048	1.136	0.28	0.55	+/- 0.29	0.206
K -K	0.0241	1.062	1.21	2.56	+/- 0.39	0.876
Ca-K	0.1462	1.065	7.15	15.56	+/- 0.69	5.186
Fe-K	0.1222	1.177	4.74	14.39	+/- 2.85	3.441
Ba-L	0.0000	1.373	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.182	0.00	0.00	+/- 0.00	0.000
O -K	0.0732	3.928	33.07	28.75	+/- 1.38	---
C -K	0.1405	2.328	50.11	32.71	+/- 1.11	36.369
Total			100.00	100.00		48.583

Wed May 27 13:53:05 1998

980430.3.2

Refit Na-K' Na-K" Mg-K' Mg-K" Al-K' Al-K" Si-K' Si-K" P -K' P -K" S -
Refit Ca-K' Ca-K" Fe-K' Fe-K" Ti-K O -K' O -K"
Filter Fit Method

Chi-sqd = 0.87 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00350 +/-	0.00110	35 +/-	11
Mg-K	0.00741 +/-	0.00125	77 +/-	13
Al-K	0.00138 +/-	0.00138	15 +/-	15
Si-K	0.02114 +/-	0.00208	214 +/-	21
P -K	0.00825 +/-	0.00234	82 +/-	23
S -K	0.02494 +/-	0.00333	211 +/-	28
Cl-K	0.02560 +/-	0.00362	184 +/-	26
K -K	0.03044 +/-	0.00450	177 +/-	26
Ca-K	0.18531 +/-	0.00753	935 +/-	38
Fe-K	0.17195 +/-	0.01768	322 +/-	33
Ba-L	0.03785 +/-	0.01121	109 +/-	32
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.12070 +/-	0.00465	597 +/-	23
C -K	0.36152 +/-	0.00962	1390 +/-	37

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Wt %	Err. (1-Sigma)	No. of Cations
Na-K	0.0019	2.185	0.30	0.41	+/-	0.13	0.266
Mg-K	0.0039	1.662	0.46	0.65	+/-	0.11	0.404
Al-K	0.0007	1.456	0.07	0.11	+/-	0.11	0.060
Si-K	0.0112	1.252	0.85	1.41	+/-	0.14	0.753
P -K	0.0044	1.211	0.29	0.53	+/-	0.15	0.258
S -K	0.0133	1.118	0.79	1.48	+/-	0.20	0.695
Cl-K	0.0136	1.137	0.74	1.55	+/-	0.22	0.656
K -K	0.0162	1.075	0.76	1.74	+/-	0.26	0.668
Ca-K	0.0985	1.065	4.46	10.49	+/-	0.43	3.932
Fe-K	0.0914	1.184	3.30	10.81	+/-	1.11	2.910
Ba-L	0.0201	1.369	0.34	2.75	+/-	0.82	0.301
Ti-K	0.0000	1.175	0.00	0.00	+/-	0.00	0.000
O -K	0.0641	3.985	27.24	25.55	+/-	0.98	---
C -K	0.1921	2.214	60.39	42.53	+/-	1.13	53.209
Total			100.00	100.00			64.110

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 13:55:44 1998

980430.3.3

Refit _Na-K' _Na-K" _Al-K' _Al-K" _P -K' _P -K" _Cl-K' _Cl-K" _K -K' _K -K" _Ba-
Refit _Si-K' _Si-K" _S -K' _Ca-K" _Fe-K' _Ba-L _O -K"
Filter Fit Method

Chi-sqd = 1.28 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00221 +/-	0.00062	105 +/-	29
Mg-K	0.06803 +/-	0.00180	3327 +/-	88
Al-K	0.00418 +/-	0.00082	203 +/-	40
Si-K	0.01402 +/-	0.00089	665 +/-	42
P -K	0.00449 +/-	0.00093	207 +/-	43
S -K	0.03585 +/-	0.00250	1419 +/-	99
Cl-K	0.01015 +/-	0.00127	343 +/-	43
K -K	0.01674 +/-	0.00158	456 +/-	43
Ca-K	0.56583 +/-	0.00523	13423 +/-	124
Fe-K	0.12430 +/-	0.00980	1091 +/-	86
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	5
O -K	0.05762 +/-	0.00146	1341 +/-	34
C -K	0.09658 +/-	0.00254	1747 +/-	46

Adjustment Factors

	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0015	2.161	0.32	0.32	+/- 0.09	0.278
Mg-K	0.0455	1.630	7.09	7.42	+/- 0.20	6.094
Al-K	0.0028	1.512	0.36	0.42	+/- 0.08	0.313
Si-K	0.0094	1.281	0.99	1.20	+/- 0.08	0.854
P -K	0.0030	1.218	0.27	0.37	+/- 0.08	0.236
S -K	0.0240	1.110	1.93	2.66	+/- 0.19	1.657
Cl-K	0.0068	1.118	0.50	0.76	+/- 0.10	0.428
K -K	0.0112	1.011	0.67	1.13	+/- 0.11	0.578
Ca-K	0.3787	1.050	23.02	39.74	+/- 0.37	19.786
Fe-K	0.0832	1.169	4.04	9.73	+/- 0.77	3.475
Ba-L	0.0000	1.431	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.232	0.00	0.00	+/- 0.00	0.000
O -K	0.0386	4.991	27.93	19.24	+/- 0.49	---
C -K	0.0646	2.630	32.86	17.00	+/- 0.45	28.238
Total			100.00	100.00		61.936

The number of cation results are based upon 24 Oxygen atoms

Wed May 27 13:58:17 1998

980430.3.4

Refit _P -K' _P -K" _Ba-L' _Ba-L" _Ti-K' _Ti-K"
Refit _Mg-K" _Al-K" _Si-K" _S -K" _Cl-K' _Cl-K" _K -K' _K -K" _Ba-L _Ti-K _C -
Filter Fit Method

Chi-sqd = 1.68 Livetime = 30.0 Sec.

Standardless Analysis

Element	Relative k-ratio	Error (1-Sigma)	Net Counts	Error (1-Sigma)
Na-K	0.00875 +/-	0.00094	639 +/-	69
Mg-K	0.01145 +/-	0.00067	870 +/-	51
Al-K	0.01214 +/-	0.00067	916 +/-	50
Si-K	0.02444 +/-	0.00079	1800 +/-	58
P -K	0.00199 +/-	0.00071	143 +/-	51
S -K	0.06054 +/-	0.00128	3727 +/-	79
Cl-K	0.01490 +/-	0.00103	783 +/-	54
K -K	0.02761 +/-	0.00130	1167 +/-	55
Ca-K	0.29638 +/-	0.00499	10932 +/-	184
Fe-K	0.22649 +/-	0.00879	3092 +/-	120
Ba-L	0.00000 +/-	0.00001	0 +/-	0
Ti-K	0.00000 +/-	0.00001	0 +/-	0
O -K	0.19281 +/-	0.00290	6973 +/-	105
C -K	0.12249 +/-	0.00231	3443 +/-	65

Adjustment Factors	K	L	M
Z-Balance:	0.00000	0.00000	0.00000
Shell:	1.00000	1.00000	1.00000

ZAF Correction Acc.Volt.= 15 kV Take-off Angle=42.37 deg
Number of Iterations = 6

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	No. of Cations
Na-K	0.0049	2.320	1.00	1.14	+/- 0.12	0.517
Mg-K	0.0064	1.737	0.92	1.12	+/- 0.07	0.479
Al-K	0.0068	1.500	0.76	1.02	+/- 0.06	0.395
Si-K	0.0137	1.283	1.26	1.76	+/- 0.06	0.654
P -K	0.0011	1.229	0.09	0.14	+/- 0.05	0.046
S -K	0.0340	1.124	2.40	3.82	+/- 0.08	1.243
Cl-K	0.0084	1.148	0.54	0.96	+/- 0.07	0.283
K -K	0.0155	1.064	0.85	1.65	+/- 0.08	0.440
Ca-K	0.1666	1.062	8.86	17.69	+/- 0.30	4.598
Fe-K	0.1273	1.169	5.35	14.88	+/- 0.58	2.776
Ba-L	0.0000	1.371	0.00	0.00	+/- 0.00	0.000
Ti-K	0.0000	1.180	0.00	0.00	+/- 0.00	0.000
O -K	0.1084	3.401	46.26	36.85	+/- 0.56	---
C -K	0.0688	2.755	31.71	18.96	+/- 0.36	16.449
Total			100.00	100.00		27.880

The number of cation results are based upon 24 Oxygen atoms