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EVALUATION OF DRY-SOLIDS-BLEND MATERIAL SOURCE FOR GROUTS CONTAINING 106-AN WASTE: FINAL REPORT

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PREFACE

This report meets requirements for Milestone 3.2, ''Final Report Establishing Specifications for Essential Materials Used in the 106-AN Formulation,'' as described in Statement of Work TMG-SOW-H-91, rev. 0, in support of the Westinghouse Hanford Grout Disposal Program.

EXECUTIVE SUMMARY

Stabilization/solidification technology is one of the most widely used techniques for the treatment and ultimate disposal of both radioactive and chemically hazardous wastes. Cement-based products, commonly referred to as grouts, are the predominant materials of choice because of their low associated processing costs, compatibility with a wide variety of disposal scenarios, and ability to meet stringent processing and performance requirements.

Such technology is being utilized in a Grout Treatment Facility (GTF) by the Westinghouse Hanford Company (WHC) for the disposal of various wastes, including 106-AN wastes, located on the Hanford Reservation. The WHC personnel have developed a grout formula for 106-AN disposal that is designed to meet stringent performance requirements. This formula consists of a dry-solids blend containing 40 wt % limestone, 28 wt % granulated blast furnace slag (BFS), 28 wt % American Society for Testing and Materials (ASTM) Class F fly ash, and 4 wt % Type I-II-LA Portland cement. This blend is mixed with 106-AN at a mix ratio of 9 lb of dry-solids blend per gallon of waste. This report documents the final results of efforts at Oak Ridge National Laboratory in support of WHC's Grout Technology Program to assess the effects of the source of the dry-solids-blend materials on the resulting grout formula.

The limestone, fly ash, and cement were obtained in two different grades or from two different sources; the BFSs, from ten different grades and/or sources. Grouts were made with various combinations of these materials and synthetic 106-AN waste. The composition of the grout formula was varied in a manner to produce a matrix of grouts for each component combination designed to represent the population of variations expected during operation of the GTF. On each grout formulation the following characteristics were determined: critical velocity, frictional pressure drop, 10-min gel strength, fluid density, 28-d unconfined compressive strength, and 28-d freestanding liquid. A comparison of the average characteristics of the reference formulation and the matrix population showed a statistically significant difference in all measured grout characteristics that is dependent on the source of the dry-solids-blend components.

Significantly, all grouts evaluated in this report met GTF performance requirements with regard to critical velocity, frictional pressure drop, 10-min gel strength, and 28-d unconfined compressive strength. Thus, although the source of material affects these grout properties, the impact is not significant relative to the desired or expected performance based on these properties. However, this conclusion does not apply to 28-d freestanding liquid. Average values for the reference formulations and the matrices routinely exceeded the performance

criterion of ≤ 5 vol % freestanding liquid. Statistically, most of the average values for the matrix reference grouts were insignificantly different from ≤ 5 vol % (i.e., the null hypothesis that the reference grout failed the 28-d freestanding-liquid criterion for most of the matrices was accepted).

The data support the contention that the freestanding liquid was affected by the particle size of the dry-blend materials. Smaller particles settle slower and react faster. The constraint of the reference grout formulation may proscribe specifying finer cement or fly ash, but specifying a higher Blaine fineness for the BFS and the finer limestone may help (although not guarantee) that the freestanding-liquid criterion is met. Assuming that control of freestanding liquid is due partly to the sorption or wetting of the limestone, then, logically, the use of limestone flour with its finer grind, smaller particle size, and, hence, larger surface area (as compared with the ground limestone) would prove beneficial. The same argument holds for the other materials, with the added factor that the gelling reaction rate increases as the particle size decreases and, consequently, may help control the freestanding liquid. Unfortunately, the limestone supplier no longer offers the limestone flour. Fortunately, however, the single ''new grind'' offered is close to the particle-size distribution of the old limestone flour. It would be preferable to use an ASTM Class S limestone rather than the Class T currently supplied. The highest Blaine fineness that is practical should be specified for the BFS.

This suspected sensitivity of freestanding liquid with respect to the surface area of the dry-blend materials has potentially serious implications to both plant operation and material purchase specifications. Requiring a finer grind of materials beyond that consistent with ASTM guidelines will most certainly increase the cost. In addition, without significant development, neither the acceptable particle-size distribution, which is used as a purchase specification, nor a correlation between particle-size distribution and freestanding liquid can be established. Unfortunately, such a development effort is not consistent with budget and schedule constraints. Thus, the plant will likely experience variations in freestanding liquid from batch to batch of dry-blend materials because of differences in particle-size distribution. At present, the size of these variations is unknown. If the purchase specifications for the dry-blend materials are made consistent with the material used in this study, then the freestanding-liquid criterion will probably not be met during field operations; thus, to meet this criterion, purchase specifications must be greater than those used in this study.

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ABSTRACT

This report documents the evaluation of dry-solids-blend material source and the subsequent impact on waste-form performance criteria. The evaluation provides documentation in support of purchase specifications for individual dry-solids-blend components.

1. INTRODUCTION

Stabilization/solidification (S/S) technology is one of the most widely used techniques for the treatment and ultimate disposal of both radioactive and chemically hazardous wastes. Cement-based products, commonly referred to as grouts, are the predominant materials of choice because of their low associated processing costs, compatibility with a wide variety of disposal scenarios, and ability to meet stringent processing and performance requirements.

Such technology is being utilized in a Grout Treatment Facility (GTF) by the Westinghouse Hanford Company (WHC) for the disposal of various wastes, including 106-AN wastes, located on the Hanford Reservation. The WHC personnel have developed a grout formula for 106-AN disposal that is designed to meet the stringent performance requirements. This formula consists of a dry-solids blend containing 40 wt % limestone, 28 wt % granulated blast furnace slag (BFS), 28 wt % American Society for Testing and Materials (ASTM) Class F fly ash, and 4 wt % Type I-II-LA Portland cement. This blend is mixed with 106-AN at a mix ratio of 9-lb dry-solids blend per gallon of waste. This report documents the efforts at Oak Ridge National Laboratory (ORNL) in support of WHC's Grout Technology Program to assess the effects of the source of the dry-solids-blend materials on the resulting grout formula. This report finalizes the work reported on in an earlier progress report.[1] Much of the data generated during this project was reported in ref. 1 and will not be duplicated in this report. Data not listed in ref. 1 will be listed in this report, either in the text or an appendix.

2. BACKGROUND

The grout is to be used in WHC's GTF where the dry-solids-blend materials are combined with specified volumes of waste in equipment located adjacent to the waste storage tanks. The resulting fresh grout is then pumped to a permanent disposal site where it hardens and forms an engineered barrier protecting against the intrusion of groundwater and subsequent release of the waste constituents of concern. As such, the grout must meet stringent process and product performance criteria as encompassed in this project.

It is essential that the dry-solids-blend components be of sufficient quality to meet these requirements when combined to form the grout, and it is economically desirable that these materials be available from as many commercial sources as possible. The four dry-solids-blend components (i.e., limestone, fly ash, granulated BFS, and cement) are generic materials produced by numerous commercial vendors, and each can be characterized by well-established ASTM standards.

2.1 PERFORMANCE CRITERIA AND CONSTRAINTS

The target performance criteria for the laboratory studies follow:

Product Performance Criteria

Unconfined compressive strength >60 psi after 28 d at 50°C
Freestanding liquid ≤5 vol % after 28 d at 50°C

Process Performance Criteria (based on nominal 2-in. Schedule 80 pipe)

Frictional pressure drop <14 psi/100 ft
10-min gel strength <100 $lb_f/100\ ft^2$
Critical velocity <60 gal/min

The project was also conducted under the following constraints:

1. The grout formulation was fixed at the specified dry-blend composition and mix ratio.
2. The simulated waste was to be heated to 50°C before mixing with the dry blend.

The idea was that a given set of dry-materials sources should meet the target criteria without altering the reference grout formula. As will be seen, meeting the freestanding liquid criterion was a problem regardless of the source of the dry materials.

2.2 LIMESTONE

Limestone is added to the grout as an inert material to minimize the heat liberated during the curing process. As such, it adds little to the final strength or durability of the grout product. However, even though an inert material, it may alter the fluidity and density of freshly prepared grouts at elevated temperatures. Limestone is an agricultural liming material whose calcium and magnesium compounds are capable of neutralizing soil acidity. As such, it can be characterized by ASTM C 602-69, "Standard Specification for AGRICULTURAL LIMING MATERIALS." As an essentially chemically pure material ($CaCO_3$), the standard specifies that limestone be classified according to its size distribution. Principal classification specifications are as shown in Table 1. In addition, the limestone shall have a calcium carbonate equivalent of not less than 80%.

Table 1. Principal classification specifications for limestone

Class designation	Passing No. 8 sieve (minute %)	Passing No. 60 sieve (minute %)
S	100	100
T	99	75
O	95	55
N	90	40
E	80	25

2.3 GRANULATED BLAST-FURNACE SLAG

BFS is added to the grout primarily due to its redox potential and its corresponding ability to reduce Tc(VII) to Tc(IV), which is significantly less mobile. In addition, BFS has cementitious properties and may result in a product with a finer microstructure than that exhibited by simple cement paste. The molten material forms a glassy, granular material on being rapidly chilled, as by immersion in water. It can be used as an additive for construction-grade concrete and can be characterized by ASTM C 989-88, "Standard Specification for GROUND GRANULATED BLAST-FURNACE SLAG FOR USE IN CONCRETE AND MORTARS." As described in ASTM C 989-88, the principal characteristics of granulated BFS are listed in Tables 2 and 3.

Table 2. General properties of granulated blast furnace slag

Property	Amount (maximum %)
Amount retained when wet screened on a 45-μm (No. 325) sieve	20.0
Air content of slag mortar	12.0
Sulfide sulfur	2.5
Sulfate ion reported as SO_3	4.0

Table 3. Grade-specific properties of granulated blast furnace slag

	Slag Activity Index (minimum %)	
	Average of last five consecutive samples	Any individual sample
7-d index		
Grade 80	NR[a]	NR[a]
Grade 100	75	70
Grade 120	95	90
28-d index		
Grade 80	75	70
Grade 100	95	90
Grade 120	115	110

[a] No requirement specified.

In effect, granulated BFS, hereafter referred to as BFS, is also classified according to size distribution or grade—the higher the grade classification, the smaller the particle size.

2.4 FLY ASH

Fly ash is added to the grout for a variety of reasons. It minimizes the NaOH and $Ca(OH)_2$ content of the final grout product, reduces the heat liberated during curing (by substitution for BFS and cement), undergoes cementitious reactions when chemically or thermally activated, increases the fluidity of fresh grouts, and improves the final strength and microstructure of the product. Fly ash is used routinely as a mineral admixture in concrete for structural applications. As such, it can be characterized by ASTM C 618-85, "Standard

Specification for FLY ASH AND RAW OR CALCINED NATURAL POZZOLAN FOR USE AS A MINERAL ADMIXTURE IN PORTLAND CEMENT CONCRETE.'' Fly ash is classified primarily by gross chemical composition as shown in Table 4.

Table 4. Fly ash classification

Characteristics	Classification		
	N	F	C
	Minimum %		
Silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3)	70.0	70.0	50.0
	Maximum %		
Sulfur trioxide (SO_3)	4.0	5.0	5.0
Moisture content	3.0	3.0	3.0
Loss on ignition	10.0	6.0	6.0

ASTM C 618-85 specifications indirectly dictate allowable CaO content. In general, CaO is the primary constituent in fly ash beyond those specified in the standard. As such, Class F fly ash would have a significantly lower CaO content than does Class C fly ash; however, since the CaO content is not specified directly, it can vary within any individual fly-ash class.

2.5 CEMENT

Cement and BFS are the primary binder materials in the grout that produce a monolithic product. Quite often, cement is used as the activating agent for BFS. It is a common construction material and can be characterized by ASTM C 150-84, ''Standard Specification for PORTLAND CEMENT.'' This standard classifies cement primarily by its chemical composition as illustrated in Table 5.

2.6 IMPACT ON MATERIAL-SOURCE SELECTION

It is envisioned that a minimum purchase specification for each of the blend components will be consistent with basic applicable ASTM standards for the material (i.e., ASTM C 602-69, 989-88, 618-85, and 150-84). As seen in Subsects. 2.2 through 2.5, these basic

Table 5. Cement classification

Chemical composition	Cement type	
	I-LA	II-LA
	Minimum %	
Silicon dioxide (SiO_2)		20.0
	Maximum %	
Aluminum oxide (Al_2O_3)		6.0
Ferric oxide (Fe_2O_3)		6.0
Magnesium oxide (MgO)	6.0	6.0
Sulfur trioxide (SO_3)	3.0	3.0
When C_3A is $\geq 8\%$	3.5	
When C_3A is $< 8\%$		
Loss on ignition	3.0	3.0
Insoluble residue	0.75	0.75
Tricalcium aluminate (C_3A)		8.0
Sum of tricalcium silicate and tricalcium aluminate		58.0[a]
Alkalies ($Na_2O + 0.65\ K_2O$)	0.60	0.60

[a]Moderate heat-of-hydration option.

specifications allow some variability in the characteristics of the material, particularly with respect to those materials that are by-products (i.e., fly ash and BFS). Therefore, in selecting materials for evaluation, it was desirable to identify sufficient sources of materials so as to address variability allowed by the basic ASTM standard. Specifically, this variability included:

1. variations in particle-size distribution for limestone within a single-class designation,
2. variations in BFS grade from a single source, and
3. variation in CaO content for fly ash meeting Class F specifications.

The basic ASTM cement specifications have historically proven to be adequate. Consequently, a large number of cement sources was not needed.

3. SELECTION OF DRY-SOLIDS-BLEND MATERIAL SOURCE

Known suppliers of the dry-solids-blend materials were contacted, and samples were requested for evaluation in this study. To limit the number of materials evaluated to meet budget and schedule commitments, the general operation of the GTF and its location were described to each vendor, and the following constraints were placed on material source:

1. The vendor must be able to supply material for two 1,000,000-gal disposal campaigns per year. (Current plans are for four 1,000,000-gal campaigns per year, but each vendor was only asked about two 1,000,000-gal campaigns.)
2. The material must meet applicable ASTM standards as described in Sect. 2.
3. If chosen as a supplier, the vendor must be willing to have its quality assurance program/procedures audited periodically by WHC personnel.
4. Considering the nature or use of the material (waste disposal) and the location of the GTF (Hanford Reservation), the vendor must believe that his material is a viable candidate for eventual use.

It was the last constraint that provided the limiting factor for the materials to be evaluated. As generic bulk materials, the greatest contributor to their ultimate cost is shipping/transportation. Typically, the associated transportation costs limit the source of these materials to a radius of approximately 500 miles from their final destination (Hanford). Because ''local'' sources of cement, fly ash, and limestone are available, vendors from other parts of the country did not feel that they would be economically competitive. However, because no ''local'' source of BFS exists, numerous vendors were willing to participate. Significantly, no vendor expressed concern over the fact that the materials would be used for waste disposal.

These constraints, believed to be realistic with respect to operation of the GTF, allowed the desires detailed in Subsect. 2.5 to be met—the materials evaluated encompassed variations allowed by the basic ASTM standards. Specifically, the materials received included:

1. a single-class designation of limestone with different particle-size distributions,
2. at least one case of two different grades of BFS from a single source, and
3. ASTM Class F fly ash with different CaO contents.

The sources of materials used in this study are documented in the following subsections. Characterization data were given in ref. 1, and additional characterization data not provided in ref. 1 are shown in Appendix B.

3.1 CEMENT

Type I-II-LA Portland cement was supplied by the following vendors:

1. Ash Grove Cement West, Inc.
   3801 East Marginal Way, South
   Seattle, WA 98134, and

2. Lafarge Corporation
   N. 209 Havana Street
   P.O. Box 13189
   Spokane, WA 99213-3189

Two separate shipments of the same material were supplied by the first source and are referred to as C-88 and C-90 throughout the remainder of this report. C-88 was characterized, but C-90 was used to make some samples. These two—C-88 and C-90—were considered to be the same material and, hence, interchangeable. One shipment was supplied by the second source and is referred to as C-91 throughout the remainder of this report.

3.2 FLY ASH

ASTM Class F fly ash was supplied by Pozzolanic International, 7525 SE 24th Street, Suite 630, Mercer Island, WA 98040.

Two separate shipments of fly ash were supplied from each of two plants:

1. Jim Bridger, hereafter referred to as P-56 and P-61, and
2. Centralia, hereafter referred to as P-55 and P-62.

The CaO content of the Jim Bridger ash was on the order of 6 wt %, while the Centralia fly ash had a CaO content on the order of 12 wt %.

3.3 LIMESTONE

Limestone was supplied by Ash Grove Cement West, Inc., P. O. Box 83007, St. Johns Station, Portland, Oregon 97283-0007.

A total of three grinds of limestone was received from this vendor:

1. ground limestone, hereafter referred to as P-58;
2. limestone flour, hereafter referred to as P-59 and P-60; and

3. new grind limestone, hereafter referred to as P-65.

All of these materials are classified as Class T in accordance with ASTM C 602-69.

Originally, only the first two grinds of material were received from the vendor. Toward the end of the project, this source quit producing these two grinds and, instead, was producing a single grind of material (No. 3 above) that did not have the same sieve classification as the original two grinds but was closer to the limestone flour. Most results were generated with the original two grinds, but as these materials were depleted, some results were generated from a shipment of new grind limestone. The source did not have a name for this grind at the time the shipment was obtained, so it will be referred to in this report as the ''new grind.''

This new material (P-65) was not received until late in the project; thus it does not appear in any of the matrix blends. P-65 was used only in the screening tests of 2-h freestanding liquid reported in the appendix.

3.4 GRANULATED BLAST FURNACE SLAG

As indicated previously, numerous potential sources of BFS were identified. Two slags—S-12 and S-15—were not able to be included in a matrix for the evaluation of grout properties, but some properties and later tests did include these two. The suppliers and their internal labels are listed in Table 6.

4. CHARACTERIZATION OF DRY-SOLIDS-BLEND MATERIALS

Some characterization data were supplied by the vendor source of the material, and some characterization measurements were made during this project. Much of the detailed data was previously reported in ref. 1 and will not be duplicated in this report. Detailed data not reported in ref. 1 are listed in Appendix B. The measurements made during the course of this project were density, hydraulic activity, Blaine fineness, slag activity, scanning electron microscope analysis, X-ray diffraction analysis, and microscope particle-size analysis. The density, hydraulic activity, Blaine fineness, and slag activity were measured using ASTM procedures. These procedures are summarized in detail in Appendix A. The results of these measurements are summarized in the remainder of Sect. 4.

Table 6. Sources for blast furnace slag

Label	Description	Supplier
S-6 and S-18	5000 Blaine	The Standard Slag Company 1200 Stambaugh Building P.O. Box 1378 Youngstown, Ohio 44501
S-7 and S-19	6000 Blaine	The Standard Slag Company 1200 Stambaugh Building P.O. Box 1378 Youngstown, Ohio 44501
S-8 and S-16		Ash Grove Cement West, Inc. 3801 East Marginal Way, South Seattle, WA 98134
S-9 and S-22	Grade 120	Blue Circle Atlantic, Inc. P.O. Box 3 Ravena, New York 12143
S-10 and S-20	Grade 100	Blue Circle Atlantic, Inc. P.O. Box 3 Ravena, New York 12143
S-11 and S-23	3720 Blaine	C. T. Takahashi & Co., Inc. Bay Vista Building, Suite 230 2815 Second Avenue Seattle, WA 98121
S-12 and S-21	5060 Blaine	C. T. Takahashi & Co., Inc. Bay Vista Building, Suite 230 2815 Second Avenue Seattle, WA 98121
S-13 and S-17	High Blaine	Standard Slag Cement 360 Jones Road Fruitland, Ontario LOR 1LO
S-14	3910 Blaine	Standard Slag Cement 360 Jones Road Fruitland, Ontario LOR 1LO
S-15		Reiss Lime Co. of Canada Ltd. Box 1690, Hwy 17E Blind River, Ontario POR 1BO

4.1 DENSITY

The densities measured using ASTM C 188-84, "Standard Test Method for DENSITY OF HYDRAULIC CEMENT," are listed in Table 7 along with values supplied by the vendor sources with their material. Some vendor values may have been measured on the material actually supplied for this project, while others are typical values for their product (sometimes reported as a range).

Table 7. Dry-material densities

Material	Measured[a] (g/cm^3)	Reported by vendor (g/cm^3)
	Cement	
C-88	3.18	—
C-91	3.17	—
	Fly ash	
P-55	2.18	2.07-2.37
P-62	2.21	2.07-2.37
P-56	2.34	2.30-2.37
P-61	2.30	2.30-2.37
	Blast furnace slag	
S-6	2.89	2.90-2.95
S-7	2.89	2.90-2.95
S-8	2.88	—
S-9	2.91	—
S-10	2.91	—
S-11	2.90	2.90
S-12	2.90	2.90
S-13	2.89	—
S-14	2.91	—
S-15	2.93	2.92

[a] Measured by ASTM C 188-84.

4.2 HYDRAULIC ACTIVITY

The hydraulic activities of the BFS measured using ASTM C 1073-85, "Standard Test Method for HYDRAULIC ACTIVITY OF GROUND SLAG BY REACTION WITH ALKALI," are listed in Table 8. The S-15 was received late in the project; some of its properties were measured and some were not. Hydraulic activity was one of the properties not measured for S-15.

Table 8. Hydraulic activity of the blast furnace slag

Material	Measured[a] (psi)
S-6	1537
S-7	1831
S-8	2251
S-9	2355
S-10	1491
S-11	2541
S-12	2514
S-13	1892
S-14	1319

[a] Measured by ASTM C 1073-85.

4.3 BLAINE FINENESSES

The Blaine finenesses measured by ASTM C 204-84, "Standard Test Method for FINENESS OF PORTLAND CEMENT BY AIR PERMEABILITY," are listed in Table 9 along with values supplied by the vendor sources.

4.4 SLAG ACTIVITY

The standard procedure for measuring slag activity is Sect. 10 of ASTM C 989-88, "Standard Specification for GROUND GRANULATED BLAST-FURNACE SLAG FOR USE

Table 9. Blaine fineness of the dry materials

Material	Measured[a] (cm^2/g)	Reported by vendor (cm^2/g)
	Cement	
C-88	3788	3585
C-91	3830	3860
	Blast furnace slag	
S-6	5178	5000 (4000-5500)
S-7	6273	6000 (4000-5500)
S-8	5672	5990
S-9	5657	5410
S-10	4019	4170
S-11	4280	3720
S-12	5040	5060
S-13	5920	5570
S-14	4190	3910
S-15	5920	—

[a] Measured by ASTM C 204-84.

IN CONCRETE AND MORTARS.'' The water content could not be determined as specified (via flow table) in the standard procedure, and this procedure was modified in its selection of water content in making the compressive-strength samples for this project. The standard procedure states that one should ''prepare the mortars in accordance with Test Method C 109, except that sufficient water shall be used in each batch to produce a flow of 110 ± 5%.''

The water content for neither the cement nor the slag mortars could be determined according to these instructions. Either the flow was >115%, or the mortar crumbled during the flow table test (which implies too little water). The procedure was modified by using the amount of water specified in ASTM Test Method C 109 for the cement mortar and picking a water content in the area of the critical water content based on the flow table results. The critical water content is that range of a few milliliters of water content in the recipe that went from >115% flow to crumbling. A single-water content was selected from this critical range and used to make the mortars for all the BFSs in determining their activities. The values measured using this modified procedure are listed in Table 10 along with the values reported by the vendors. It is not clear what effect this modification would have on the numbers reported by the vendors, but Table 10 does demonstrate that the values measured using this modification were comparable to those reported by the vendors.

Table 10. Slag activities

		Slag activity index			
Material	Time	Measured[a]		Reported by vendor	
		(%)	Grade	(%)	Grade
S-6	7 d	92	100	91	100
	28 d	131	120	124	120
S-7	7 d	84	100	91	100
	28 d	110	120	124	120
S-8	7 d	90	100	94[b]	120
	28 d	118	120	123[b]	120
S-9	7 d	97	120	97	120
	28 d	122	120	120	120
S-10	7 d	89	100	79	100
	28 d	115	120	104	100
S-11	7 d	88	100	—	—
	28 d	119	120	—	—
S-12	7 d	101	120	—	—
	28 d	141	120	—	—
S-13	7 d	108	120	—	—
	28 d	137	120	—	—
S-14	7 d	73	80	—	—
	28 d	91	80	—	—
S-15	7 d	91	100	—	—
	28 d	121	120	—	—

[a] Measured by modified ASTM C 989-88. Average of six.

[b] Single value.

4.5 MICROTECHNIQUES

The microtechniques basically consisted of evaluating the morphology and composition of the dry materials utilizing scanning electron microscopy (SEM) and X-ray diffraction (XRD). The K-25 Technical Division performed these microanalyses, and their report is given in Appendix C. The photomicrographs in Figs. 1 through 8 illustrate the typical morphology for each type of material by SEM. These analyses resulted in two semiquantitative sets [i.e., energy dispersive spectroscopy (EDS) and XRD] and one quantitative set (i.e., number particle-size distribution) of results. These results are discussed in more detail in the following sections and Appendix C.

4.5.1 Energy Dispersive Spectroscopy

The elements (above a given atomic number) making up the material being studied by EDS give off distinctive peaks, which allows qualitative identification of much of the elemental composition. These peaks are analyzed to give the amounts of the elements relative to each other. This method works quite well for quantitative determination of the relative amounts but cannot match the quantitative accuracy of bulk analytical techniques, such as inductively coupled plasma spectroscopy. Figures 9 through 13 illustrate the elemental compositions estimated by EDS for the cements, the limestones, the fly ashes, the series one BFS (i.e., S-7, S-8, S-10, and S-13), and the nonseries one BFS (i.e., S-6, S-9, S-11, S-12, S-14, and S-15) respectively. The cement C-92 in Fig. 9 was a Type I/II Portland cement obtained from Dixie Cement Company, in Knoxville, Tennessee, for the slag activity test (ASTM C-989-88). The cements used for making the grouts could not be used because their alkali content was lower than that specified in the ASTM Standard.

4.5.2 X-ray Diffraction

X-ray diffraction measures the crystalline composition of the dry-blend materials. The technique identifies the crystalline phases (qualitative) and the relative abundance of a given crystalline phase among all of the crystalline phases observed (but only crystalline phases, hence, the technique is only semiquantitative). Table 11 lists the crystalline phases identified and their relative abundance among the observed crystalline phases. The phase identified in the

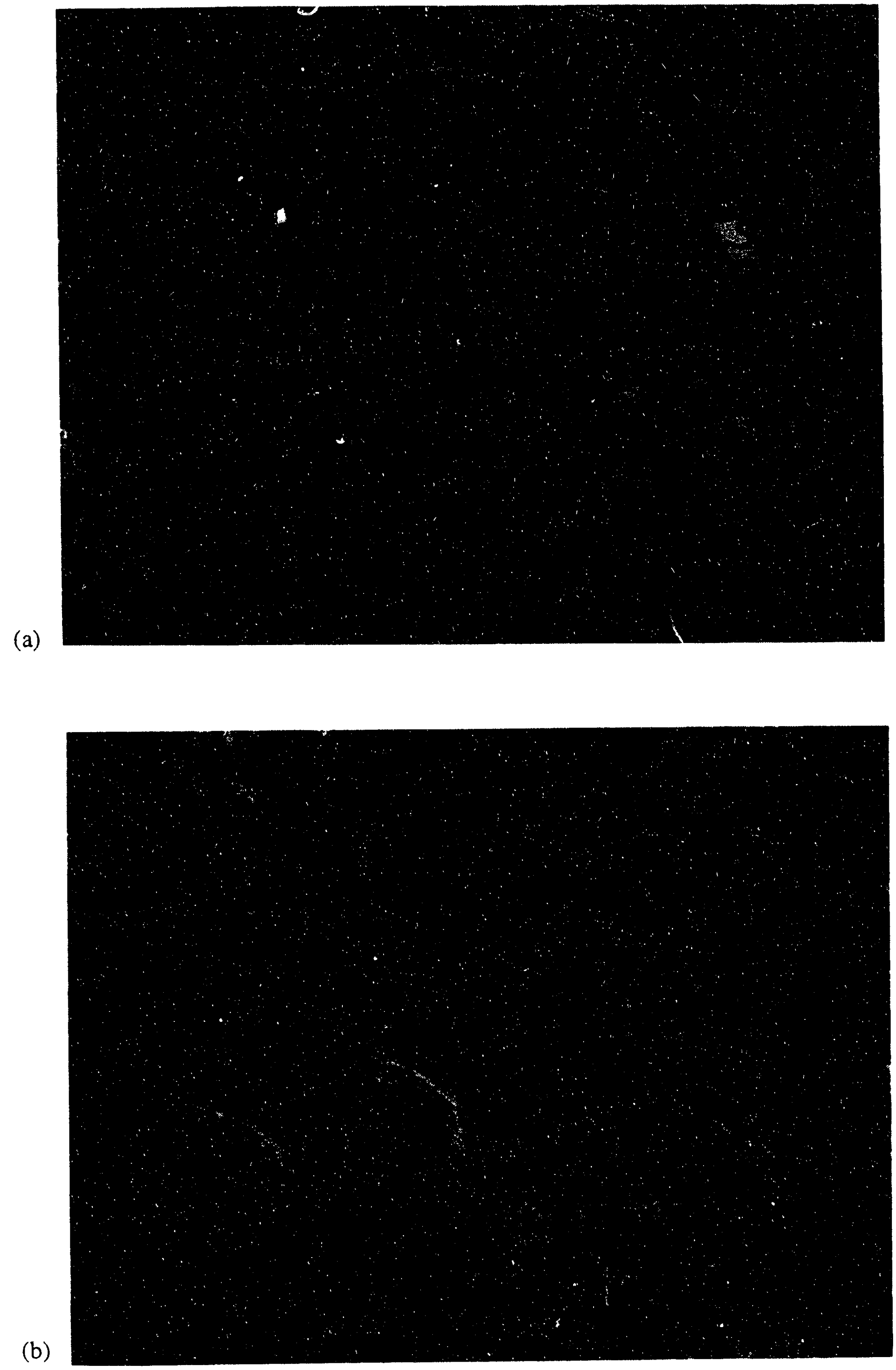

Fig. 1. Photomicrographs of Type II Portland cement (C-88) at (a) 500× and (b) 1000×.

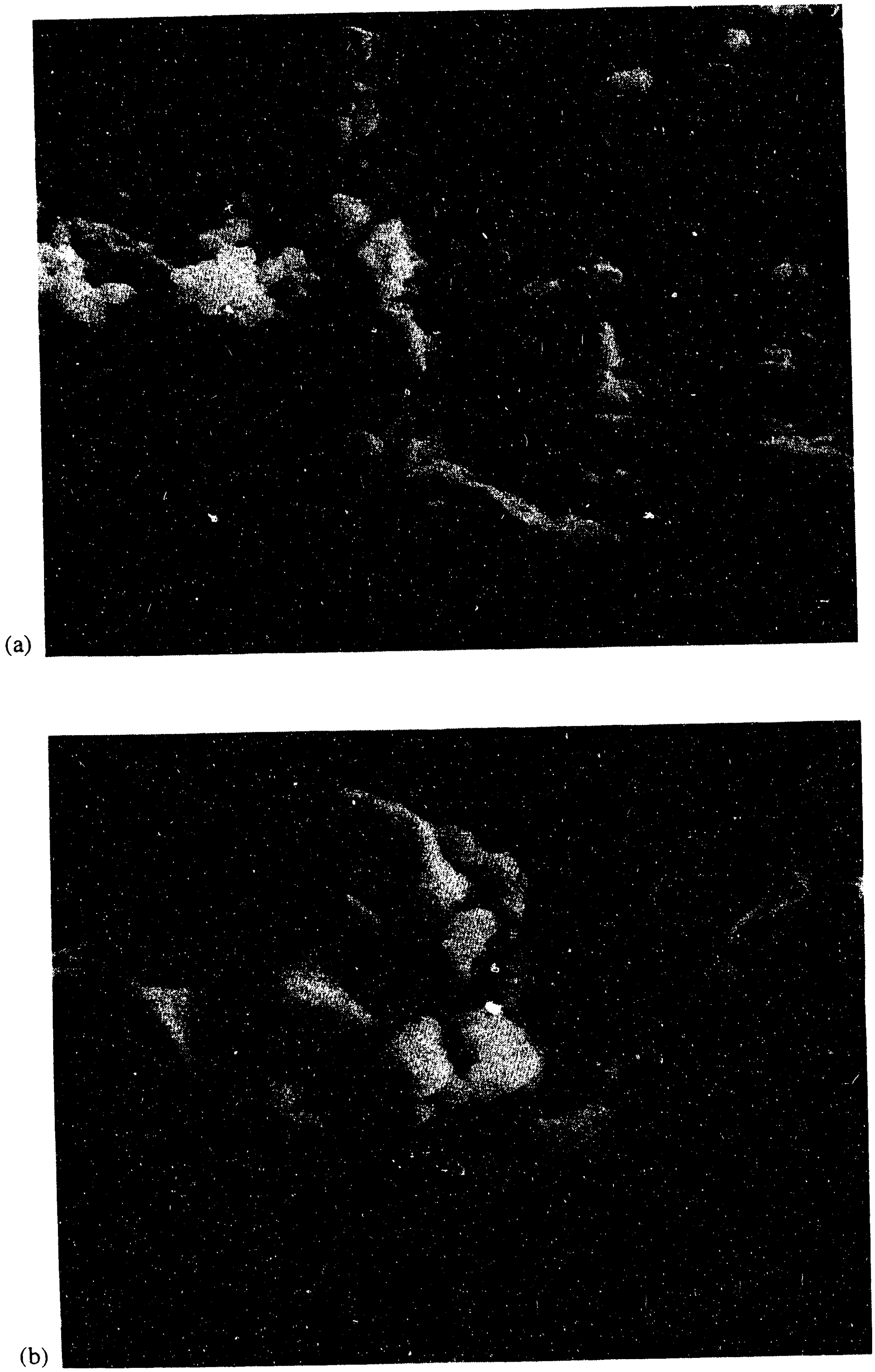

Fig. 2. Photomicrographs of Type II Portland cement (C-88) at (a) 2000× and (b) 3000×.

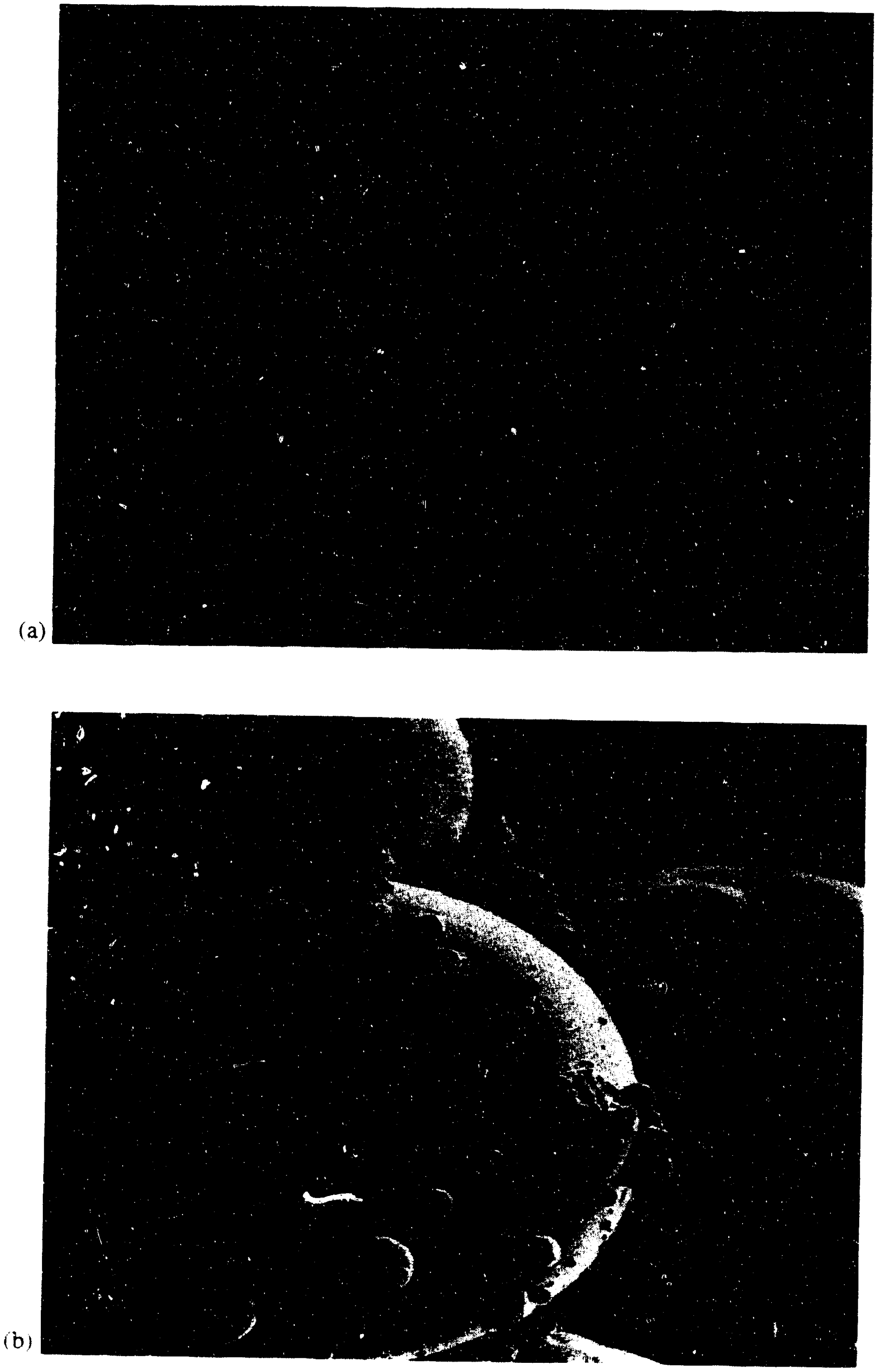

Fig. 3. Photomicrographs of fly ash (P-62) at (a) 500× and (b) 1000×.

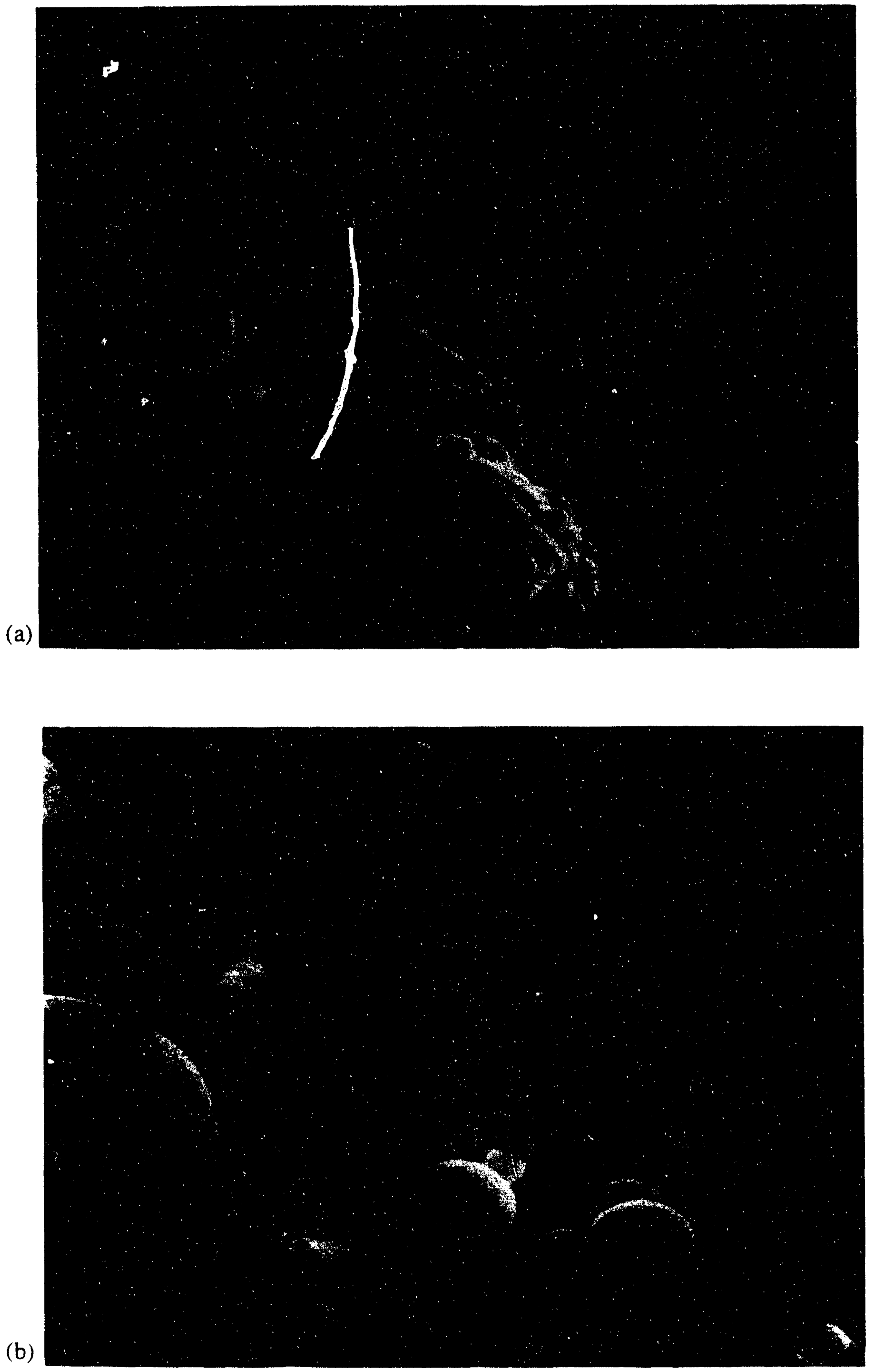

Fig. 4. Photomicrographs of fly ash (P-62) at (a) 2000× and (b) 3000×.

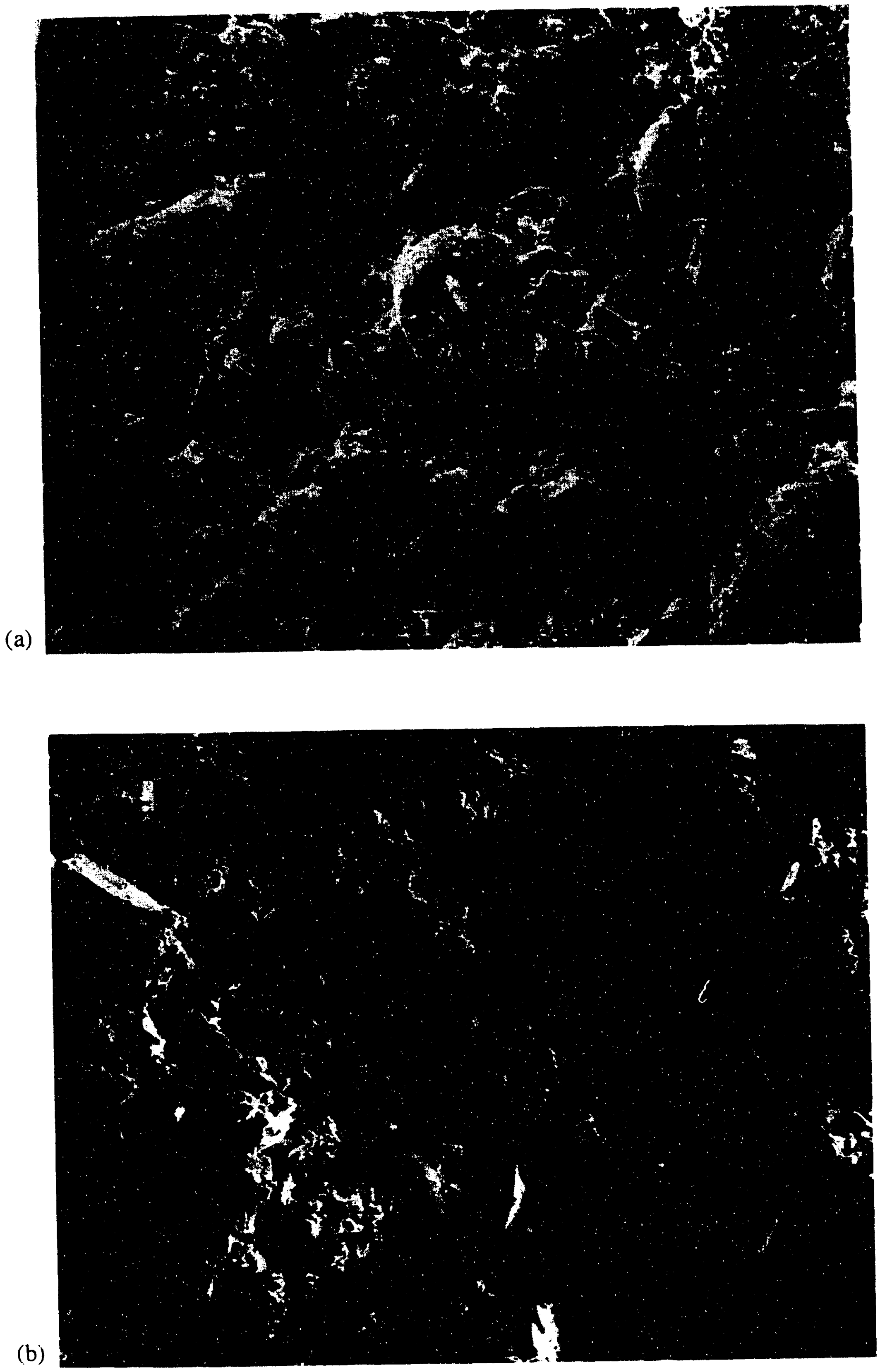

Fig. 5. Photomicrographs of blast furnace slag (S-8) at (a) 500× and (b) 1000×.

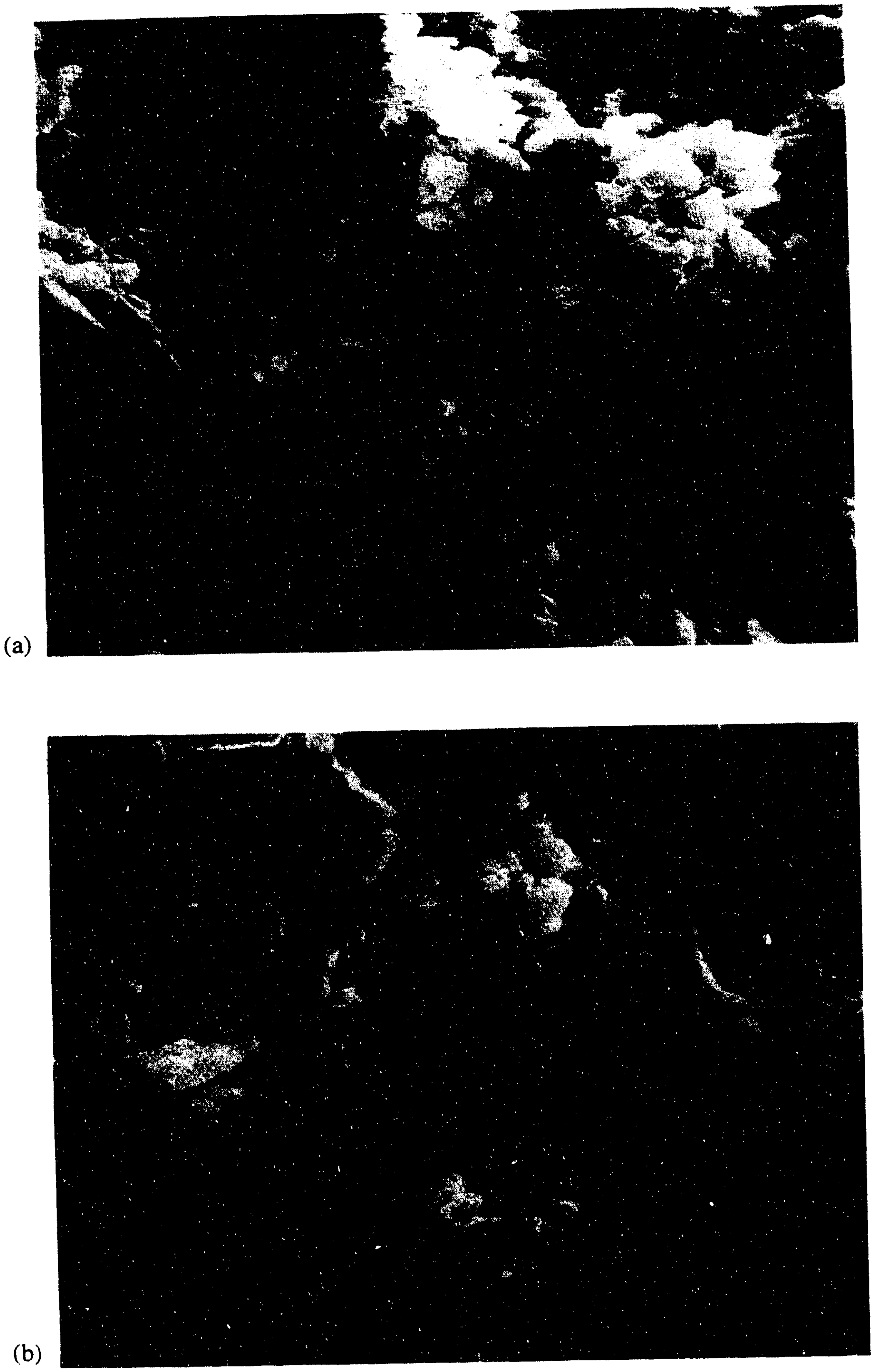

Fig. 6. Photomicrographs of blast furnace slag (S-8) at (a) 2000× and (b) 3000×.

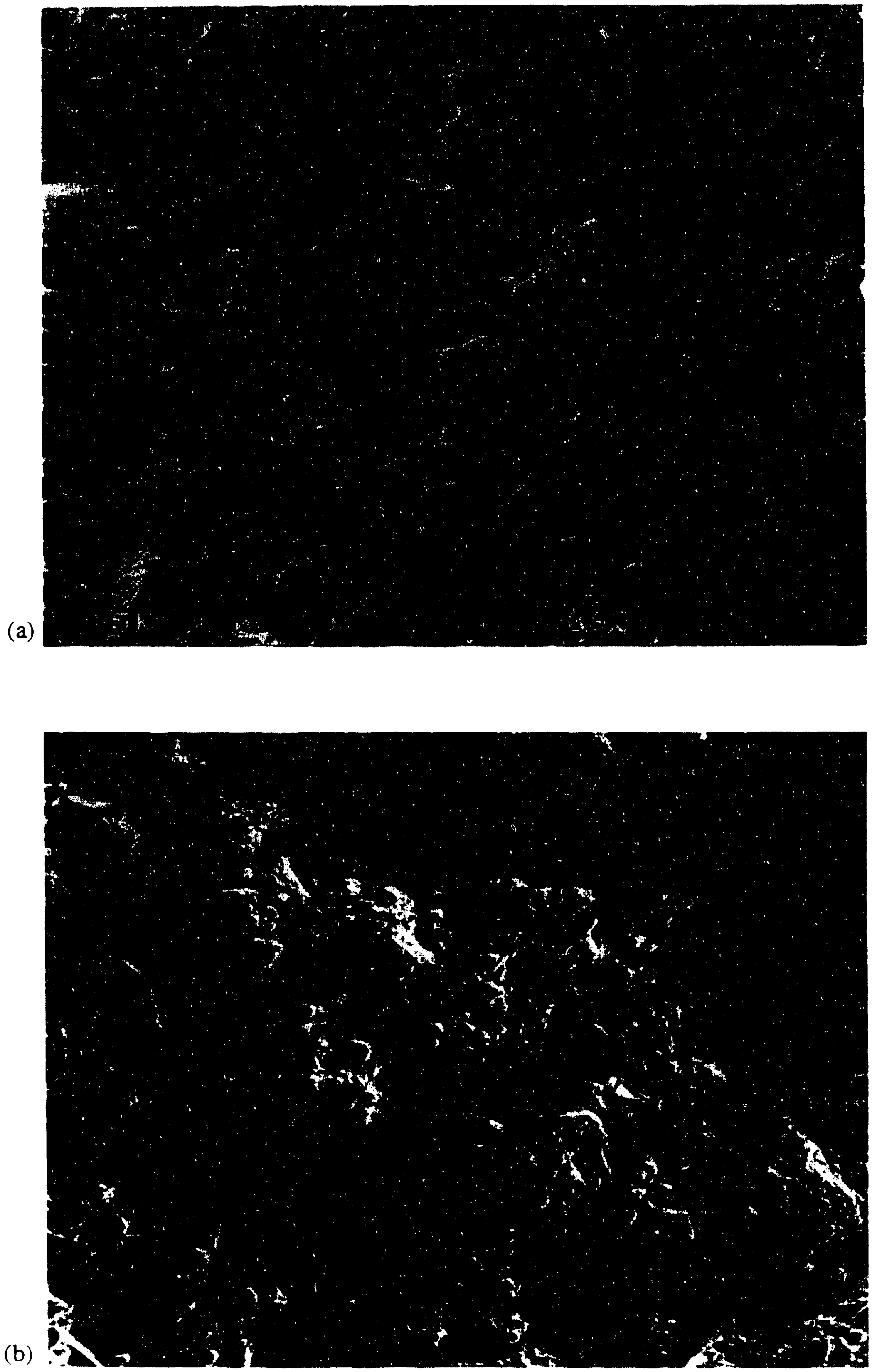

Fig. 7. Photomicrographs of limestone (P-65) at (a) 500× and (b) 1000×.

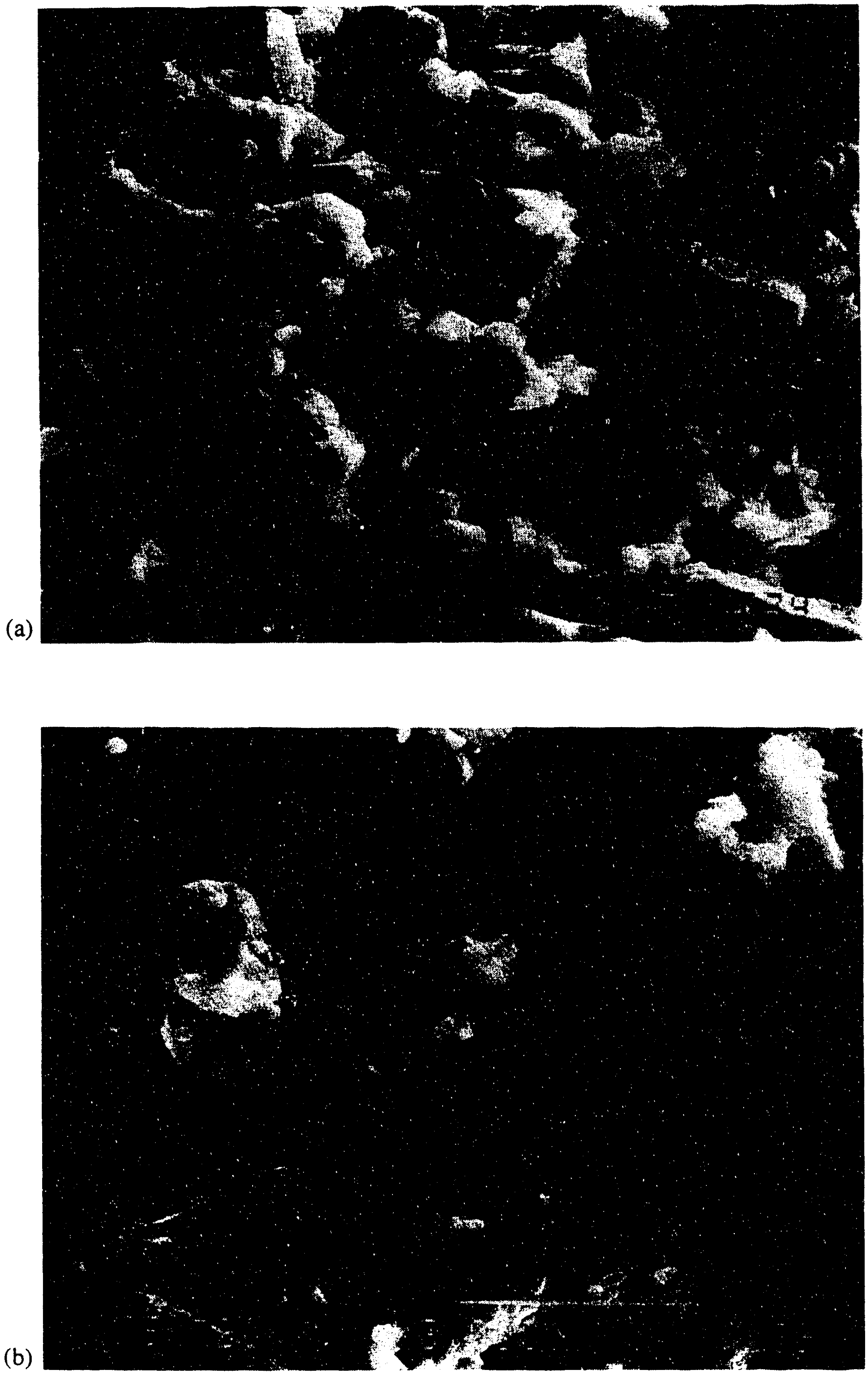

Fig. 8. Photomicrographs of limestone (P-65) at (a) 2000× and (b) 3000×.

ORNL DWG 93A-650

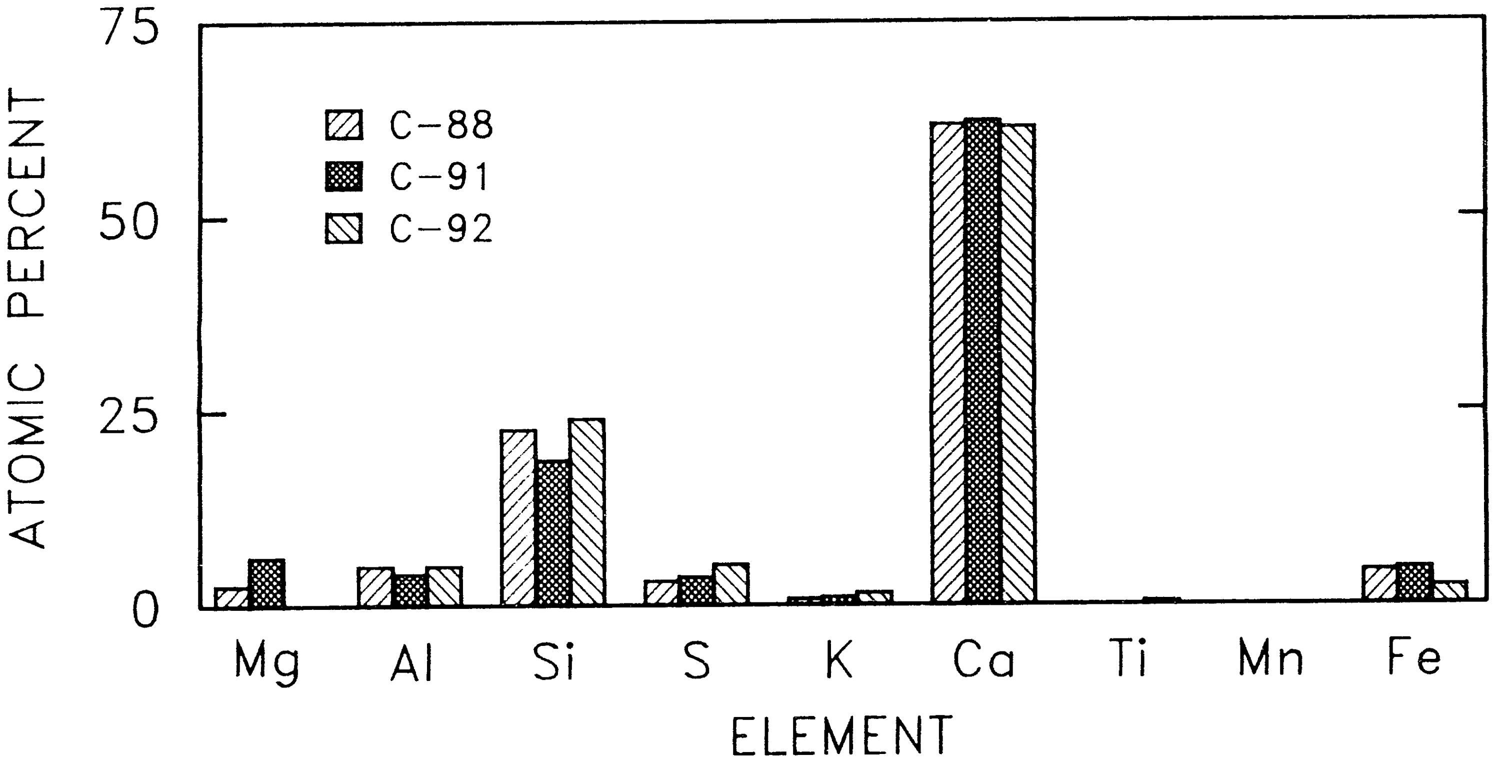

Fig. 9. Elemental distribution of cements by energy dispersive spectroscopy.

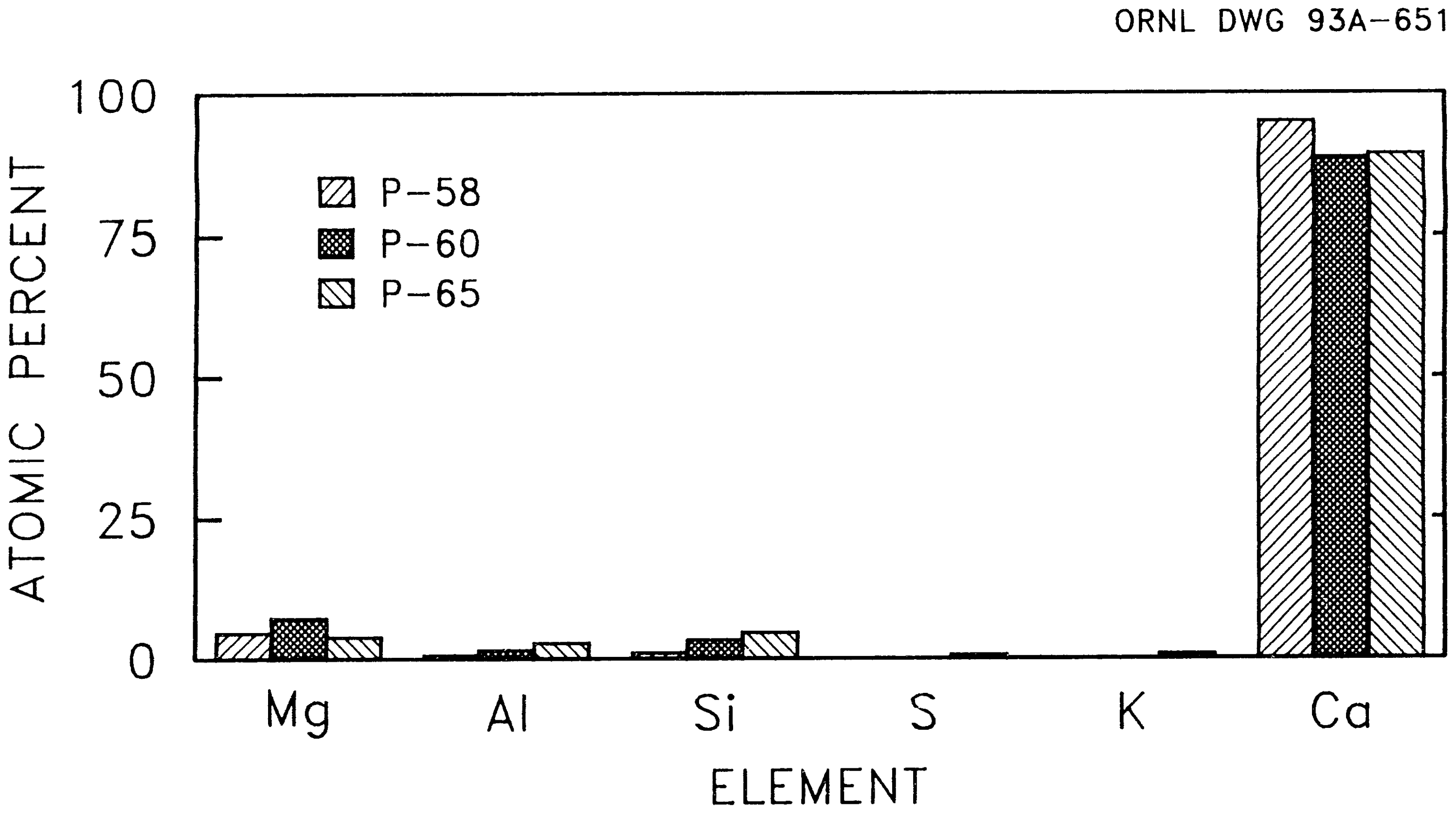

Fig. 10. Elemental distribution of limestones by energy dispersive spectroscopy.

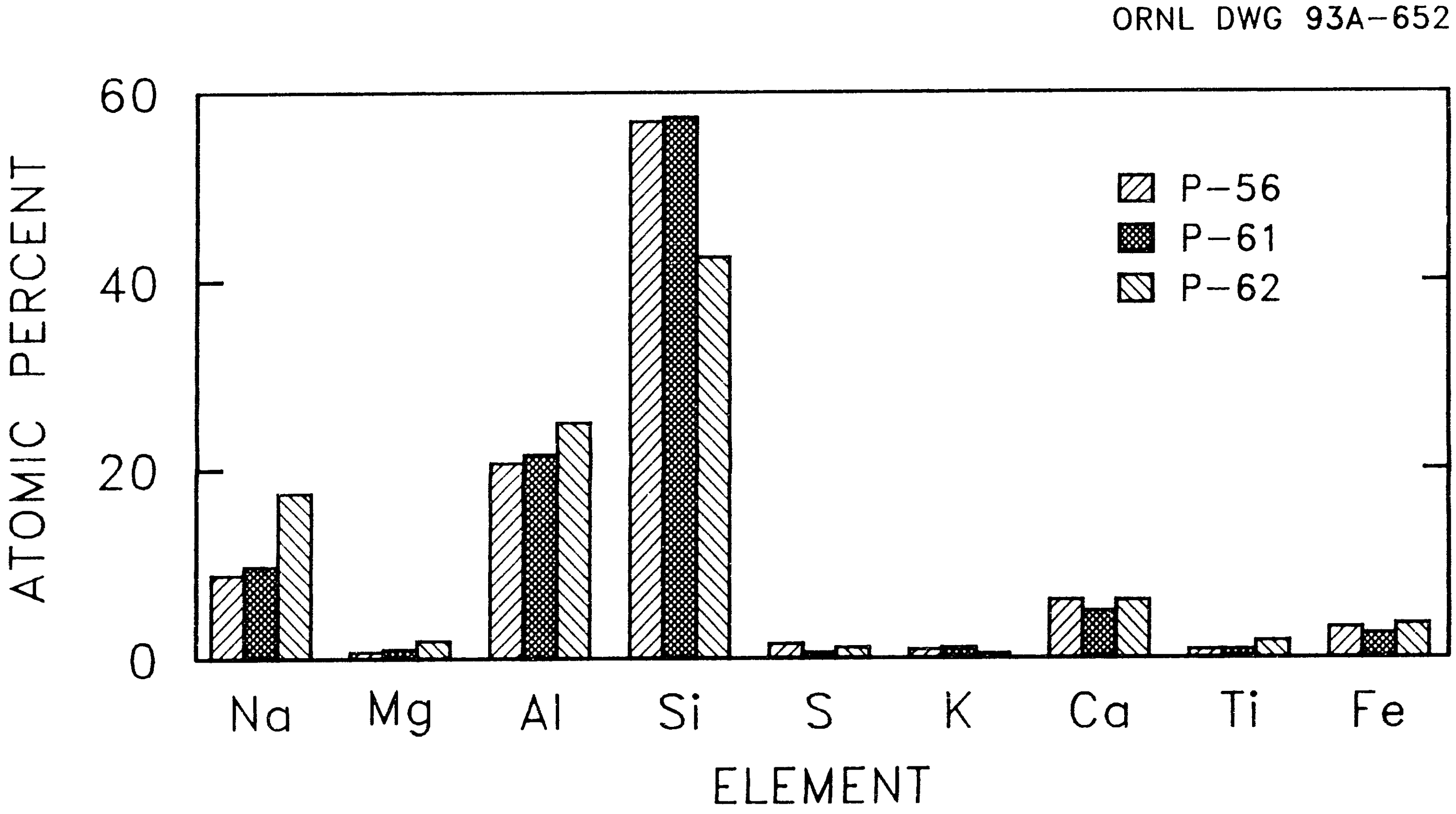

Fig. 11. Elemental distribution of fly ashes by energy dispersive spectroscopy.

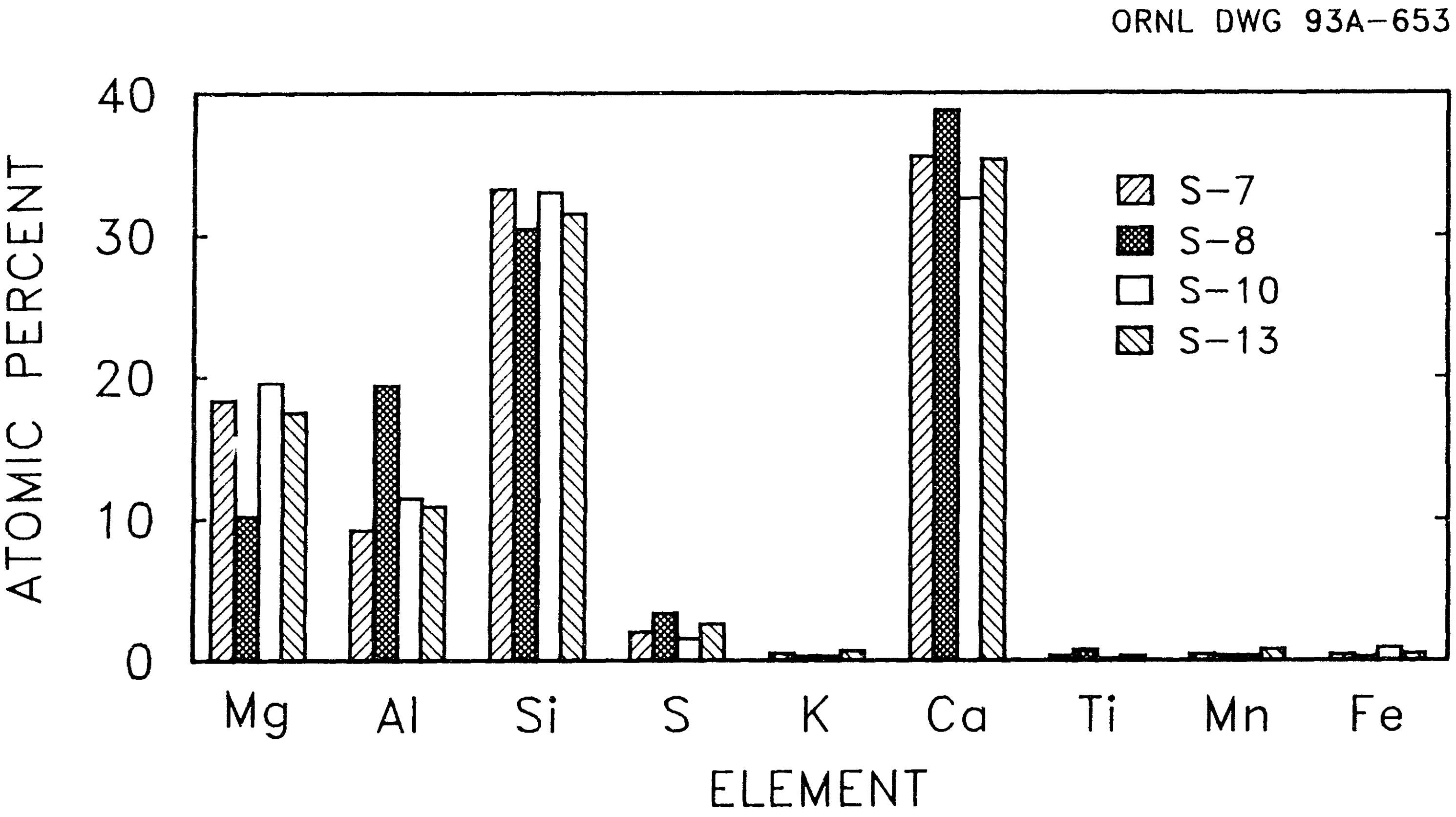

Fig. 12. Elemental distribution of series one blast furnace slag by energy dispersive spectroscopy.

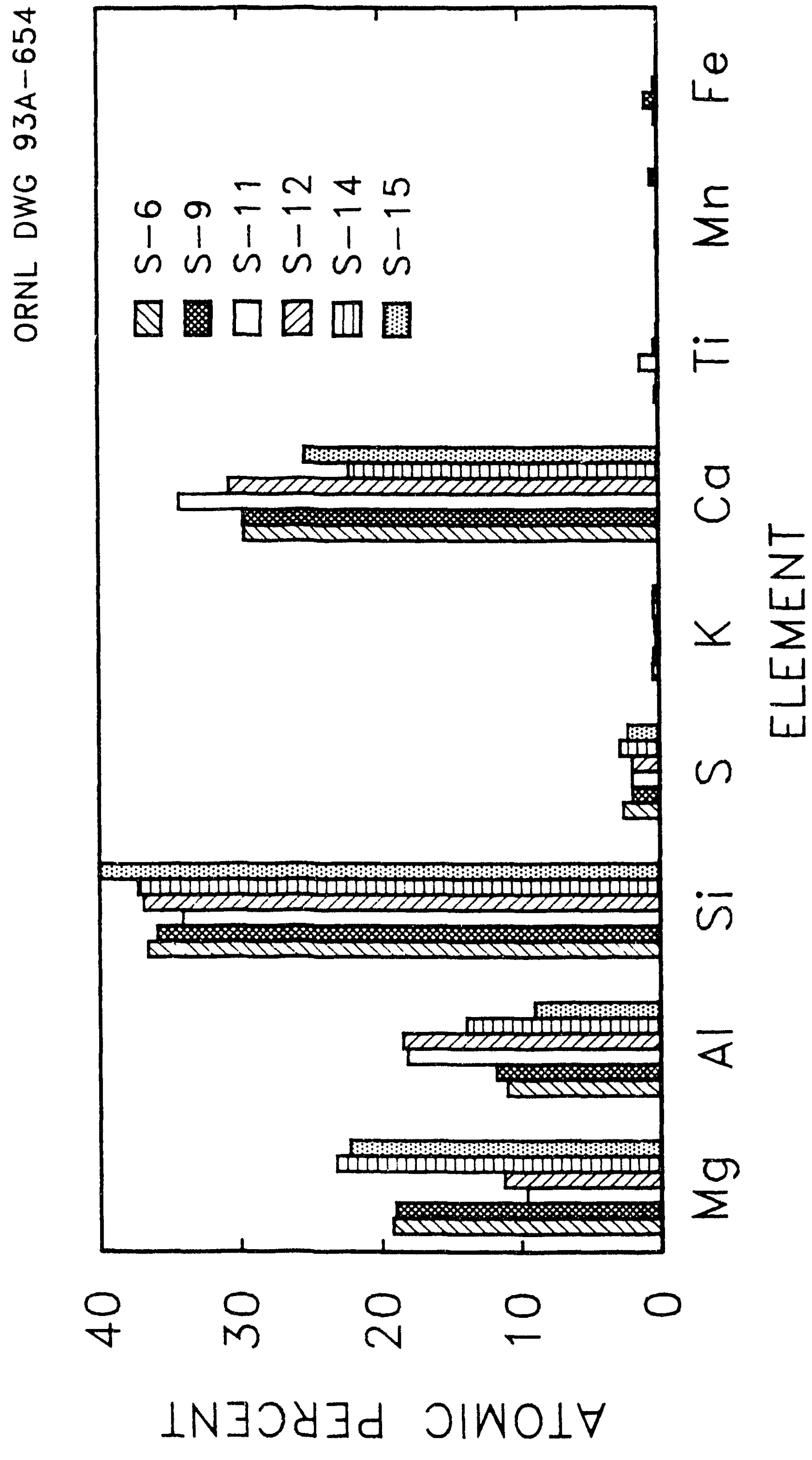

Fig. 13. Elemental distribution of nonseries one blast furnace slag by energy dispersive spectroscopy.

Table 11. Crystalline phases identified by X-ray diffraction

Material	Phase	Relative abundance (%)
	Cement	
C-88	$Ca_{54}MgAl_2Si_{16}O_{90}$	85
	Unidentified	15
C-91	$Ca_{54}MgAl_2Si_{16}O_{90}$	87
	Unidentified	13
C-92	$Ca_{54}MgAl_2Si_{16}O_{90}$	84
	Unidentified	16
	Limestone	
P-58	$CaCO_3$ (calcite)	100
P-60	$CaCO_3$ (calcite)	100
P-65	$CaCO_3$ (calcite)	100
	Fly ash	
P-56	SiO_2 (quartz)	92
	Fe_2O_3 (hematite)	8
P-61	SiO_2 (quartz)	100
P-62	SiO_2 (quartz)	73
	Fe_2O_3 (hematite)	27
	Blast furnace slag	
S-6	$Ca_3Mg(SiO_4)_2$ (merwinite)	76
	Unidentified	24
S-7	$Ca_3Mg(SiO_4)_2$ (merwinite)	82
	Unidentified	18
S-8	Unidentified	100
S-9	Unidentified	100
S-10	Unidentified	100
S-11	Unidentified	100
S-12	Unidentified	100
S-13	$Ca_3Mg(SiO_4)_2$ (merwinite)	62
	Unidentified	38
S-14	$Ca_3Mg(SiO_4)_2$ (merwinite)	75
	Unidentified	25
S-15	Unidentified	100

cements is a solid-solution combination of the oxides (i.e., 54 CaO, 16 SiO_2, Al_2O_3, MgO) that occurs in Portland cement clinker and basic slags.

In addition to the information presented in Table 11, the quality and intensity of the diffraction patterns were a qualitative indication of the relative amounts of crystalline and amorphous content of the materials. The patterns of the cements indicated that the majority of these materials were crystalline in nature. All patterns of the limestones indicated a pure crystalline phase of calcite. The fly ashes had relatively weak diffraction responses, suggesting that these materials were not purely crystalline in nature. The diffraction pattern for P-62 was the weakest for the three fly ashes. The XRD patterns collected for the BFS indicated that these materials were primarily amorphous. Thus, the crystalline phases identified in Table 11 were not expected to be a major constituent of the overall composition of the BFS.

4.5.3 Particle-Size Analysis

The particle-size distribution of the dry-blend materials was estimated by computer analysis of SEM images, image analysis (i.e., the size range of a large population of particles of a given material was subdivided with the number of particles within each subdivision counted and reported as a percentage of the total number of particles). Consequently, the resulting distribution was based on the number of particles at a given size (i.e., number particle-size distribution) as opposed to a mass or volume particle-size distribution (e.g., the mass particle-size distribution by dry sieving reported for the limestones by the vendor source). The image analysis uses the measured distribution to estimate a smoothed number particle-size distribution curve. Plots of the measured distributions and these smoothed curves are included in Appendix C for each material. Figures 14 through 18 illustrate these smoothed distributions for the cements, the limestones, the fly ashes, and the BFS respectively. All of these materials achieve a maximum peak height in their smoothed number particle-size distribution for a particle size of 2.0–2.5 μm. The differences lie in the details of the distribution (e.g., the maximum peak height and minor bimodal tendencies). In addition to the particle-size distribution, the image analysis also reported the aspect ratio (i.e., an estimate of the length to width ratio) for each of the materials. Table 12 lists the maximum smoothed peak height and aspect ratio for each material.

All of the aspect ratios were between 1.3 and 1.7. The ground materials—cement, limestone, and BFS—had aspect ratios >1.5. The fly ashes had aspect ratios around 1.4, however, which was significantly different from the ground materials and reflects the different

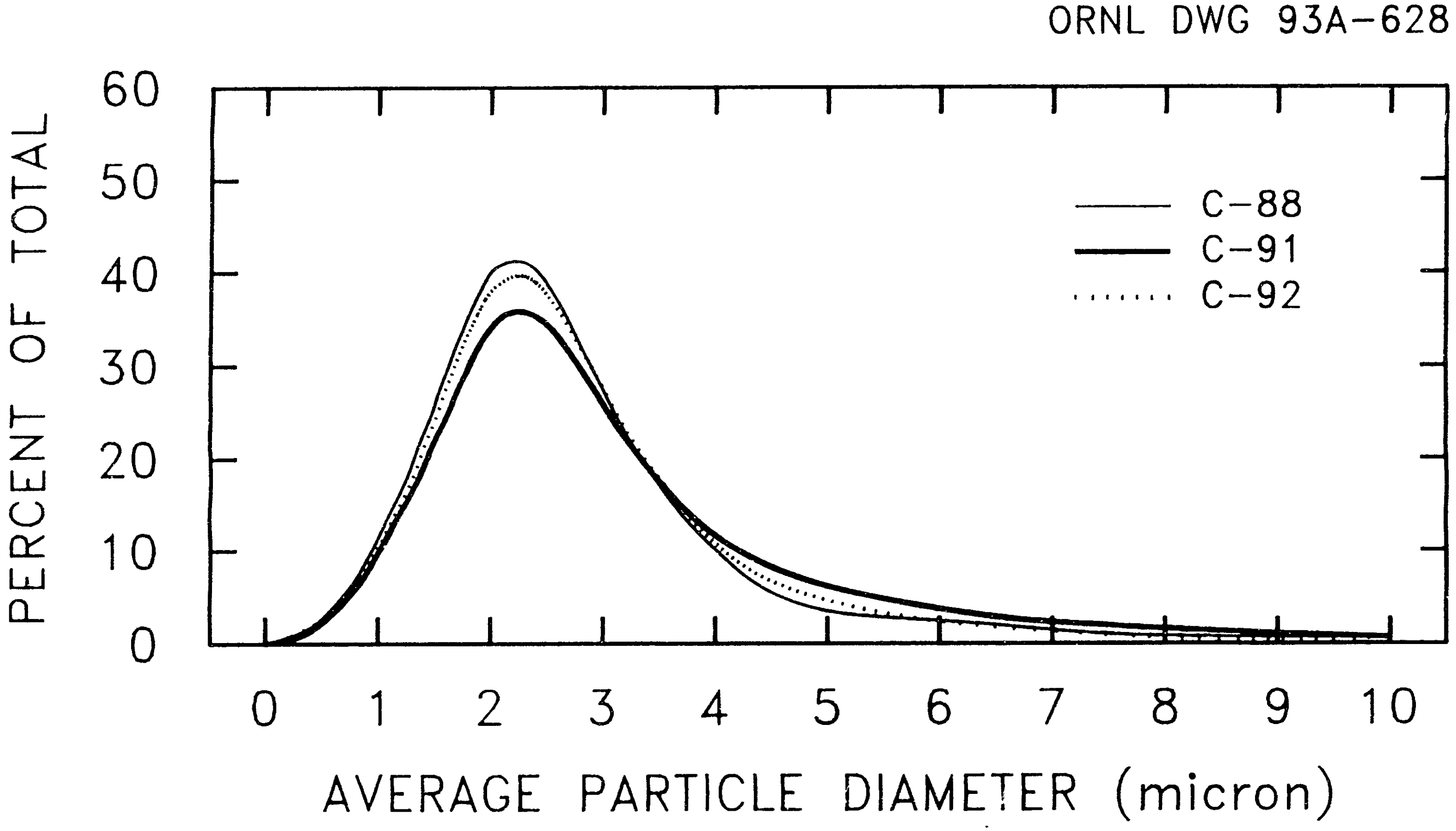

Fig. 14. Particle-size distribution for Portland cement.

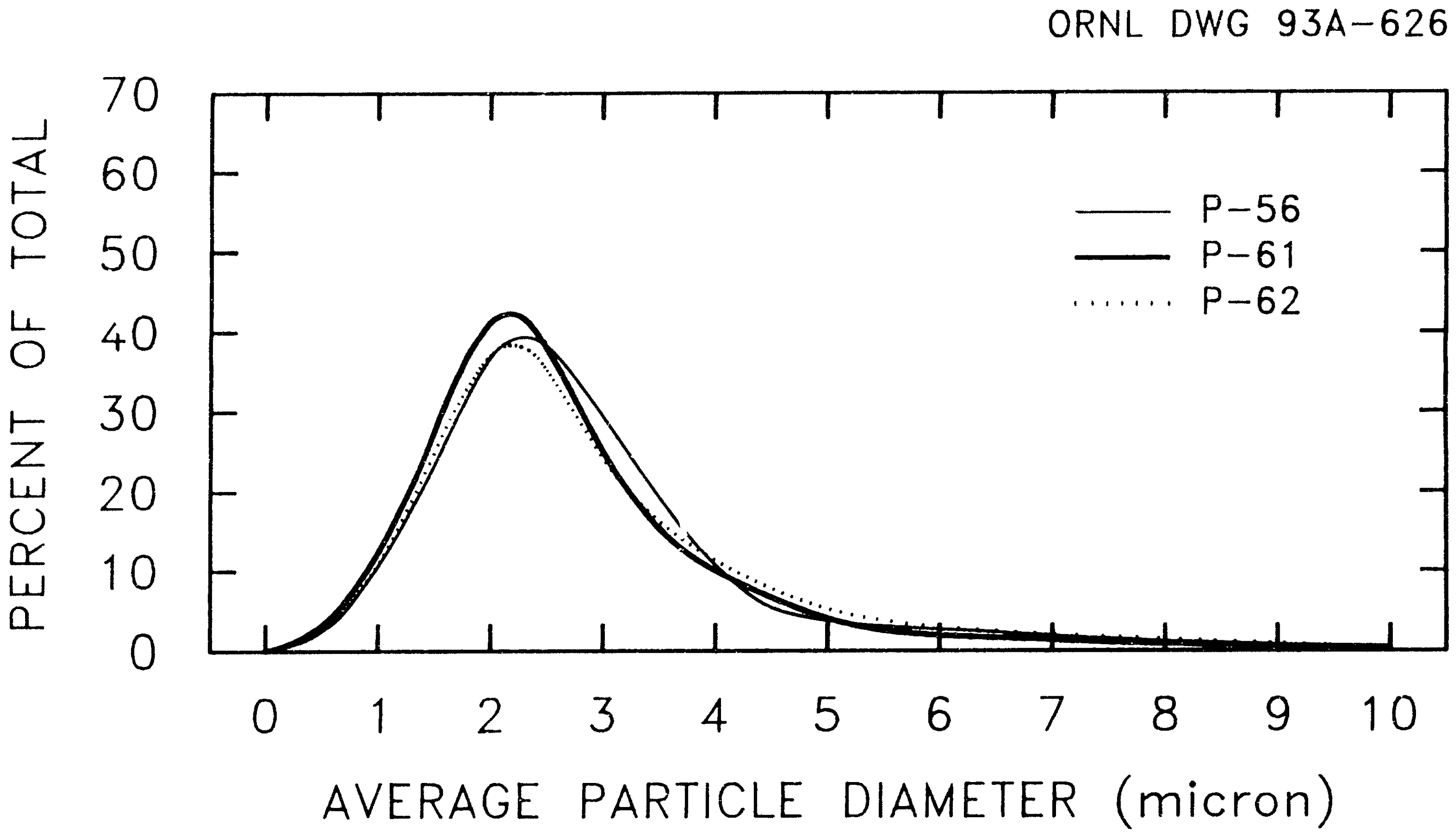

Fig. 15. Particle-size distribution for fly ash.

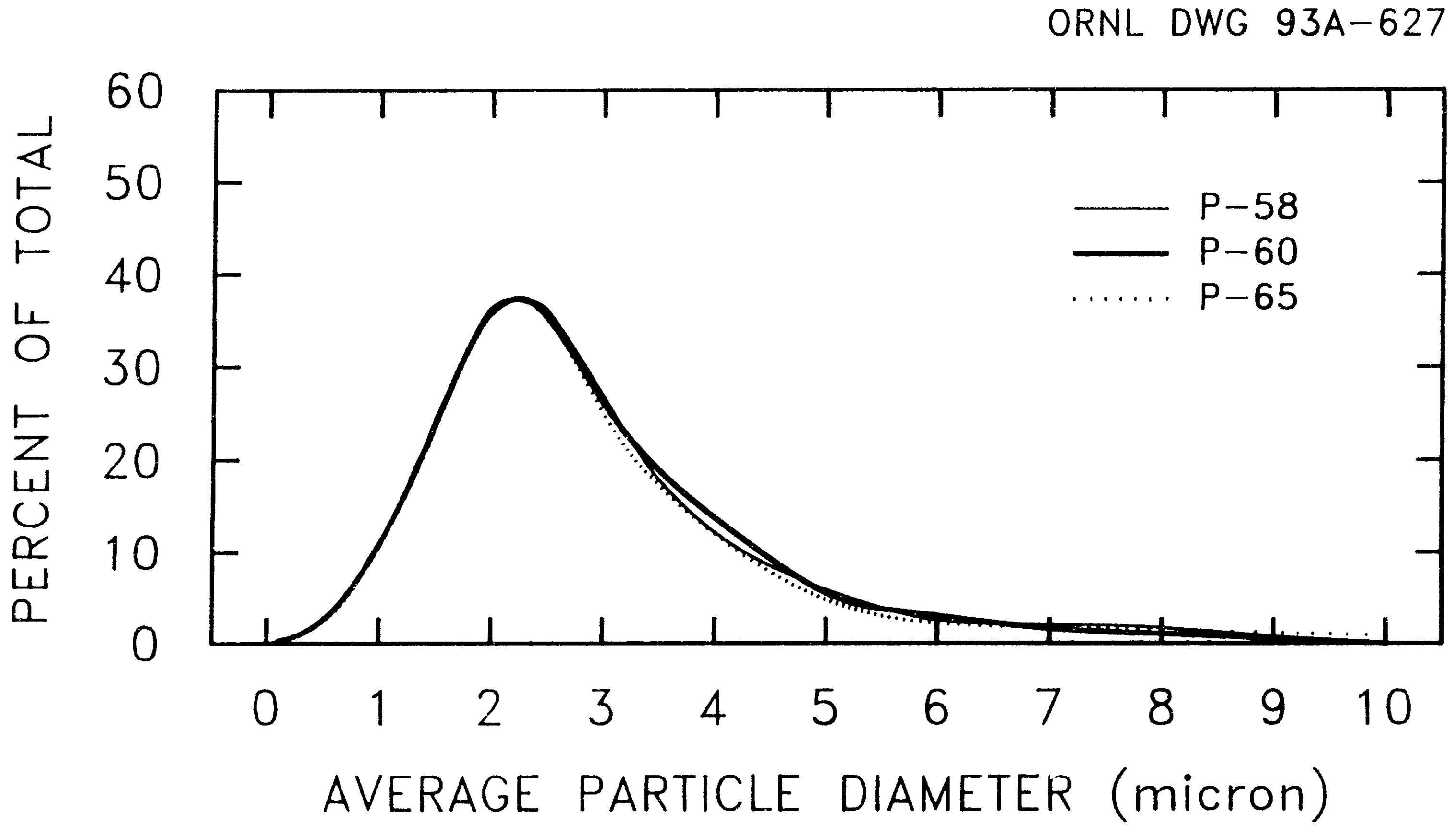

Fig. 16. Particle-size distribution for limestone.

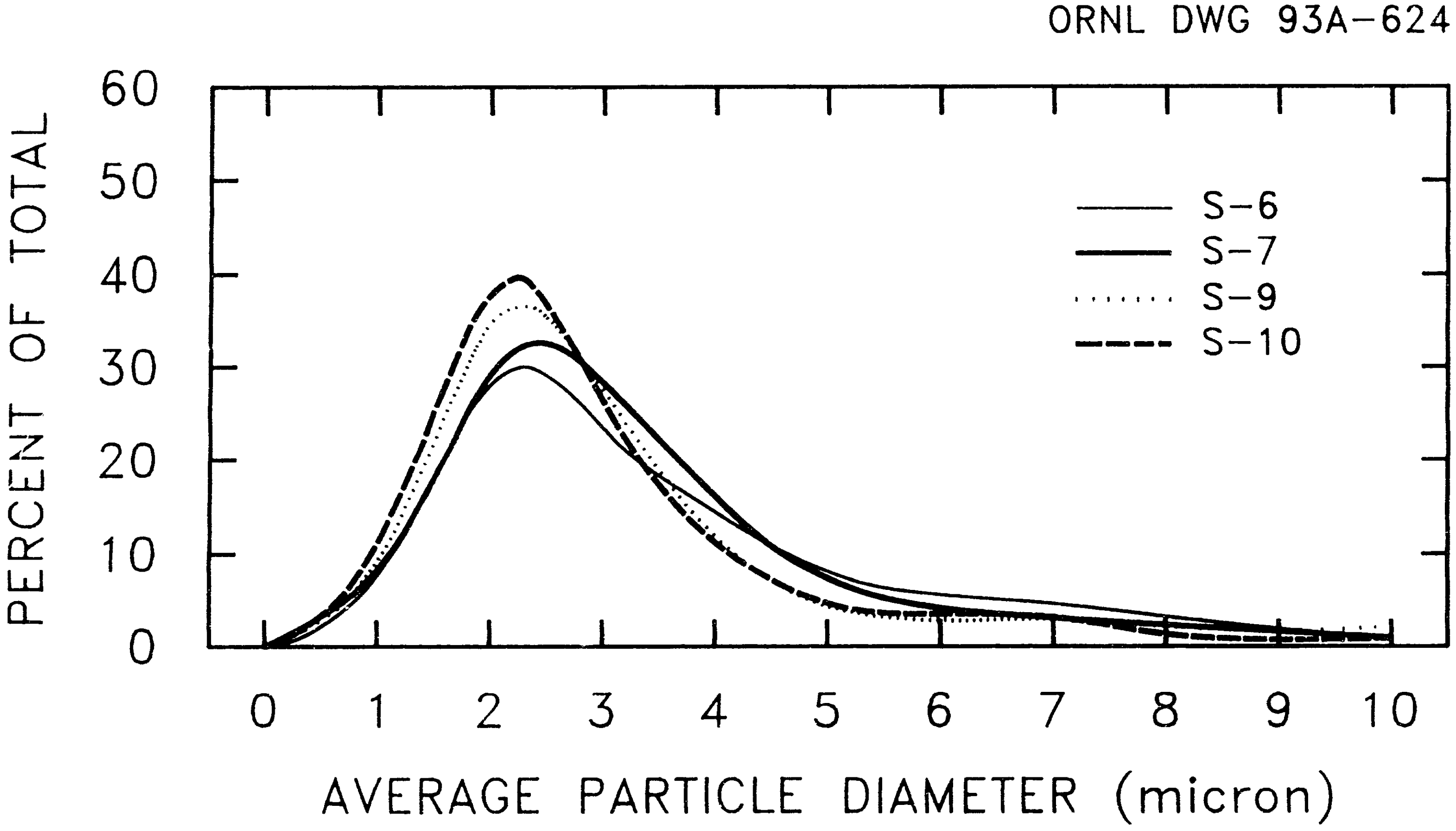

Fig. 17. Particle-size distribution for blast furnace slags.

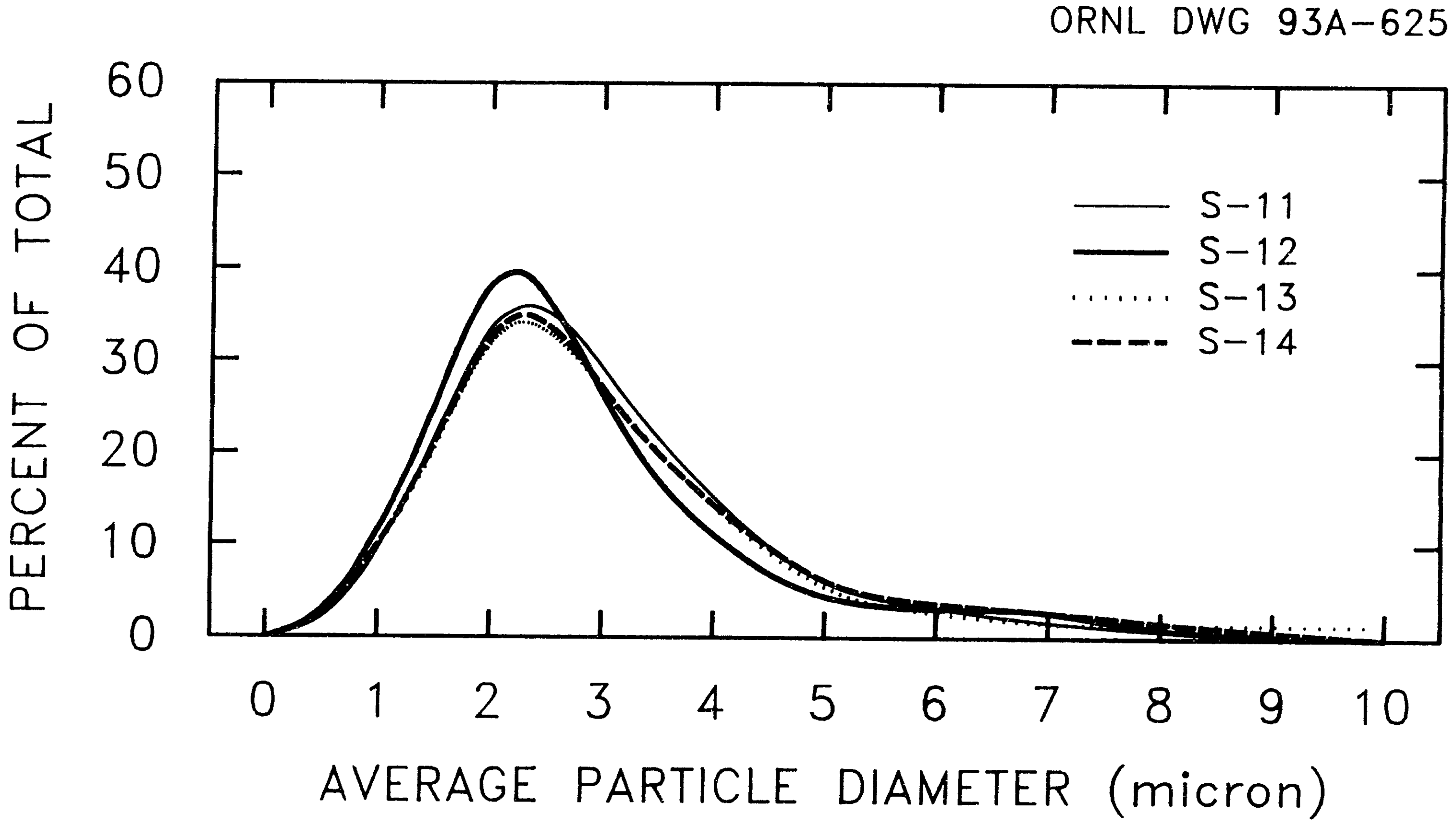

Fig. 18. Particle-size distribution for blast furnace slags.

Table 12. Maximum percentage in the smoothed number particle-size distribution

Material	Aspect ratio	Maximum No. (%)
	Cement	
C-88	1.56	
C-91	1.64	36
C-92	1.57	40
	Limestone	
P-58	1.62	38
P-60	1.60	38
P-65	1.59	38
	Fly Ash	
P-56	1.44	
P-61	1.37	42
P-62	1.31	39
	Blast furnace slag	
S-6	1.59	30
S-7	1.60	33
S-8	1.60	42
S-9	1.56	37
S-10	1.58	39
S-11	1.62	36
S-12	1.66	39
S-13	1.59	27
S-14	1.57	34
S-15	1.61	34

way in which such materials were made. The photomicrographs in Figs. 1 through 8 also illustrate the morphological differences between the angular structure of the ground materials and the spherical structure of the condensed and solidified particles of fly ash.

5. METHODOLOGY FOR EVALUATION OF GROUT PROPERTIES

Ultimately, it is the effect of the material source on subsequent properties of the grout prepared using that material which is of interest. To address this interest, it is desirable to have an experimental matrix that encompasses the expected compositional variations of the material in the grout. Each matrix would be comprised of grouts made from materials from one source (per component). Data obtained from this matrix would be averaged; the average would be representative of the population (or material composition variations) encompassed by the matrix. This matrix would then be repeated using different material sources, and the average values would be compared. Similar averages between matrices would indicate no significant differences in grout properties due to the material source. On the other hand, significant differences between the averages would indicate a dependency on material source. The same could be said by comparing identical observations between matrices. Data obtained, as described in these subsections, are presented in ref. 1.

5.1 MATRIX SELECTION

The matrix of interest must encompass the compositional variations associated with the dry-solids-blend materials that could be expected to occur during routine operation of the GTF. The range of interest that needs to be encompassed by the matrix is the range in composition expected during the normal blending and mixing of limestone (A), fly ash (B), granulated BFS (C), cement (D), and mix ratio (E). The basic matrix chosen to evaluate these materials is a quarter of the full factorial for five variables. The basic matrix (Table 13) is as follows. In this matrix, variables A through D reflect blend composition, while variable E reflects mix ratio. Typically, these possess different units. Blend composition is routinely thought of as wt %, while mix ratio is typically expressed in pounds per gallon. In reality, the GTF will measure all of these variables based on weight. Thus, if the variables are selected based on 1 gal of waste, then all will be in units of pounds.

Based on 1 gal of waste, the reference blend composition (R) in units of pounds is limestone flour, 3.6; fly ash, 2.52; granulated BFS, 2.52; cement, 0.36; and mix ratio 9. During routine operation of the GTF, the weight of individual blend components can be controlled ±5%, while the mix ratio can be controlled ±0.5 lb (on a per gallon basis). Using this degree of control as a guideline, the plus values in the block for variables A, B, C, and D are 110% of the reference value, while the minus values are 90% of the reference value. For variable E,

Table 13. Basic matrix used for comparison of grouts prepared from various sources of material

Observation No.	Variable				
	A	B	C	D	E
1	–[a]	–	–	+[b]	–[c]
2	–	–	+	–	+[d]
3	–	+	–	–	+
4	–	+	+	+	–
5	+	–	–	+	+
6	+	–	+	–	–
7	+	+	–	–	–
8	+	+	+	+	+
9	R[e]	R	R	R	R

[a] The minus indicates 90% of the reference value for A through D.

[b] The plus indicates 110% of the reference value for A through D.

[c] The minus indicates 8.5 lb/gal for variable E.

[d] The plus indicates 9.5 lb/gal for variable E.

[e] R indicates reference blend composition.

the plus value is 9.5, while the minus value is 8.5. Note that the variability of the blend components is greater than that expected during routine operation of the GTF.

For the reader's convenience, Table 14 presents the blend compositions (from Table 13) in more typical units. Table 15 presents the blend materials used in each matrix.

Another way of looking at this problem is the variation in the grout composition expected to be produced during routine operation. The mass fractions of each of the components (i.e., limestone, fly ash, BFS, cement, and waste) are viewed as a dimension (i.e., x_1, x_2, x_3, x_4, and x_5 respectively) in a multidimensional volume. Note that only four dimensions are truly independent (i.e., the sum of $x_{1\text{-}5}$ always equals one). In this case the reference grout formula can be represented as a point at the coordinates (i.e., 0.189, 0.132, 0.132, 0.019, 0.528). Routine operation can be represented by the multidimensional volume encompassed by the following points:

Point	Coordinates
1	(0.204, 0.129, 0.129, 0.0185, 0.519)
2	(0.174, 0.135, 0.135, 0.0193, 0.537)
3	(0.185, 0.143, 0.129, 0.0185, 0.525)
4	(0.193, 0.122, 0.135, 0.0193, 0.531)
5	(0.185, 0.129, 0.143, 0.0185, 0.525)
6	(0.193, 0.135, 0.122, 0.0193, 0.531)
7	(0.184, 0.129, 0.129, 0.0204, 0.538)
8	(0.193, 0.135, 0.135, 0.0175, 0.519)
9	(0.173, 0.121, 0.121, 0.0173, 0.567)
10	(0.204, 0.143, 0.143, 0.0204, 0.490)

This multidimensional volume falls well within the volume representing the grout compositions produced from the matrices.

Table 14. Basic matrix (converted to typical units) used for comparison of grouts prepared from various sources of material

Observation No.	Blend composition (weight %)				Mix ratio (lb/gal)
	Limestone	Fly ash	BFS	Cement	
1	39.63	27.76	27.76	4.84	8.5
2	37.66	26.38	32.19	3.77	9.5
3	37.66	32.19	26.38	3.77	9.5
4	35.31	30.19	30.19	4.32	8.5
5	44.51	25.52	25.52	4.45	9.5
6	42.47	24.35	29.71	3.47	8.5
7	42.47	29.71	24.35	3.47	8.5
8	40.02	27.99	27.99	4.0	9.5
9	40.0	28.0	28.0	4.0	9.0

5.2 SAMPLE-PREPARATION PROCEDURE

Prior to sample preparation for each matrix, the sources of material were selected. These materials were then used for all blends in a particular matrix. The runs in each matrix were performed in a random order. The run number used in this report and ref. 1 represents the order in which the experiment was performed. The observation number relates the formulation used to prepare Tables 14 and 15.

Table 15. Dry-solids-blend components used in each matrix[a]

Matrix No.	Limestone	Fly ash	Cement	BFS
1	P-59 P-60	P-55 P-62	C-88 C-90	S-8 S-16
2	P-59 P-60	P-55 P-62	C-88 C-90	S-10 S-20
2R	P-59 P-60	P-55 P-62	C-88 C-90	S-10 S-20
3	P-59 P-60	P-55 P-62	C-88 C-90	S-9 S-22
4	P-59 P-60	P-55 P-62	C-88 C-90	S-6 S-18
5	P-59 P-60	P-55 P-62	C-88 C-90	S-7 S-19
6	P-59 P-60	P-55 P-62	C-88 C-90	S-13
7	P-59 P-60	P-56 P-61	C-88 C-90	S-13
8	P-59 P-60	P-56 P-61	C-88 C-90	S-7 S-19
9	P-59 P-60	P-56 P-61	C-88 C-90	S-14 S-17
10	P-58	P-55 P-62	C-88 C-90	S-6 S-18
11	P-58	P-56 P-61	C-88 C-90	S-14 S-17
12	P-59 P-60	P-55 P-62	C-91	S-8 S-16
13	P-59 P-60	P-55 P-62	C-91	S-11 S-23

[a] Only 13 matrices were used. More than one batch of a given material was obtained from most vendors. The batches of the same material were given different numbers but were used interchangeably in the matrices.

5.2.1 Dry-Solids-Blend Preparation

Predetermined weights of each blend component were added to a 3-ft^3, Patterson-Kelly, twin-shell V-blender in sufficient quantity to accommodate sample requirements for all runs in a matrix. The materials were then tumbled for 23 h. The resulting blended material became the dry-solids blend to be added to the waste in the grout preparation step.

5.2.2 Waste Preparation

Synthetic 106-AN waste of the composition shown in Table 16 was prepared in sufficient quantity to accommodate all sample requirements for all runs in a matrix. The waste was maintained at 40°C and continuously stirred for introduction to the grout preparation step.

Table 16. Composition of synthetic 106-AN waste

Component	Molarity
$NaAl(OH)_4$	0.421
Na_2SO_4	0.031
NaCl	0.15
NaF	0.0081
$Ca(NO_3)_2 4(H_2O)$	0.002
NaOH[a]	0.675
$NaNO_3$	1.29
$NaNO_2$	0.758
Na_2CO_3	0.382
HEDTA	0.019
Na_4EDTA	0.0044
Hydroxyacetic acid[b]	0.042
$Na_3PO_4 12(H_2O)$	0.155
$Na_3C_6H_5O_7 2(H_2O)$[c]	0.03

[a] Added as a 50 wt % aqueous solution.
[b] Added as a 70 wt % glycolic acid solution.
[c] Sodium citrate.

5.2.3 Grout Preparation

A predetermined volume of the waste was added to a Model N-50 Hobart Mixer. The mixer was set to a low setting (~140 rpm), and a predetermined weight of dry-solids blend was added over a 10- to 15-s period and mixed for a total of 30 s at this setting. The mixer was then set to medium (~285 rpm), and mixing was continued for an additional 30 s. The volume of grout prepared was sufficient for the following:

1 sample for determination of rheological properties,
1 sample for determination of freestanding liquid,
1 sample for determination of grout density,
3 samples for determination of compressive strength, and
3 samples for determination of leachability.

Grout preparation and subsequent data acquisition were repeated three times for each blend in the matrix.

5.3 DETERMINATION OF RHEOLOGICAL PROPERTIES

A grout volume of 350 mL was placed in a Fann viscometer. Shear stress data were obtained as a function of shear rate at shear-rate settings of 600, 300, 200, 100, 181, 90, 60, and 30 rpm.

The resulting data are represented by the power-law model as follows:

$$S_s = K' \, (S_r)^{n'} \, , \tag{1}$$

where

S_s = shear stress, $\mathrm{lb_f/ft^2}$;
k' = fluid consistency index, $\mathrm{lb_f{\cdot}s^{\eta}/ft^2}$;
S_r = shear rate, $\mathrm{s^{-1}}$;
η' = flow behavior index ($0 < \eta' < 1.0$), dimensionless.

The Reynolds number as defined for non-Newtonian fluids is derived from Eq. (1) to be

$$N_{Re} = \frac{1.86V^{(2-n')}\rho}{k'(96/d_i)^{n'}} , \tag{2}$$

where

N_{Re} = Reynolds number, dimensionless;

V = fluid velocity, ft/s;

d_i = pipe inside diam, in.;

ρ = fluid density, lb/gal.

Critical velocity is determined from Eq. (2) assuming a Reynolds number of 2100 and a schedule 80 pipe of nominal 2-in. diam. Grout density was determined using an NL Baroid mud balance.

Additional fluid properties are calculated as follows:

$$P_f = \frac{0.039L\rho V^2 f}{d_i} , \tag{3}$$

where

P_f = frictional pressure drop through a straight pipe, psi;

L = pipe length, ft;

f = fanning friction factor (0.008), dimensionless;

and

$$P_H = \frac{G \cdot A_w}{(1.44 \times 10^4)A_p} , \tag{4}$$

where

P_H = pump head pressure necessary to overcome gel strength, psi;

G = gel strength, $lb_f/100\ ft^2$;

A_W = pipe inside surface area, in.^{2};

A_p = inside pipe cross-sectional area, in.^{2}.

For calculations presented in this report, the measured gel strength of the fresh grout is the maximum deflection taken from the Fann viscometer at 3 rpm after the grout has remained static for 10 min.

5.4 DETERMINATION OF SOLID-GROUT PROPERTIES

5.4.1 Unconfined Compressive Strength

Freshly prepared grout was poured into three 2-in. cube molds conforming to ASTM C 109-80 specifications and then placed on a Model VP5101 Syntron vibrating table at a setting of 6.5 for 20 s. The samples were then stored in a humidity cabinet maintained at 50°C and relative humidity >95%. After curing 28 d, the cured grouts were removed, and the unconfined compressive strength was determined using a Tinius Olsen Super L Universal Testing Machine in accordance with ASTM C 109-80.

5.4.2 Freestanding Liquid

Freshly prepared grout (250 mL) was placed in a polymethylpentene graduated cylinder, which was sealed below the pour spout with an inverted No. 7 stopper. The cylinder was then stored in a cabinet maintained at 50°C. Freestanding liquid was observed at 2 h, 1 d, 7 d, 14 d, 21 d, and 28 d after placing the grout in the cylinders.

5.4.3 Nitrate Leachability

Freshly prepared grout was placed into cylindrical molds (2.50-cm ID × 4.65-cm high) and cured at 50°C and >95% humidity for 28 d. After curing, any freestanding liquid was removed and saved for later addition to the rinse water. (The amount of nitrate in this rinse was measured and subtracted from the original nitrate inventory of the cylinder to estimate the leachability index. Thus, the nitrate in the freestanding liquid and easily rinsed off the cylinder was instantaneously released, and the remainder was leached in a manner represented by the leachability index.) Three cured cylinders were weighed, measured, and leached using a modified ANSI/ANS-16.1-1986 procedure. The leachant was double distilled water prepared by distilling house distilled water in a Corning Mega-Pure™ MP-11A quartz still system. The quantity of leachant used in each step was equal to 10 mL for each cm^2 of geometric surface area of the sample cylinders. The cylinder heights were inevitably less than the mold height; therefore the surface area and leachant quantity were calculated from the measured dimensions of each cylinder. Consequently, the leachant volumes were different from sample to sample.

Each cylinder was suspended sequentially in eight separate volumes of leachant (the same quantity for each volume) contained in polyethylene bottles. The cylinders were held for different time periods in the separate volumes of leachant (representing the different leach intervals for each sample and labeled the zeroth to the seventh leach interval). The zeroth leach interval involved dipping the sample for only 30 s and was also known as the rinse step. This step was intended to wash off any loose material or salt precipitated on the surface and was not counted in the total leaching time. The remaining leach intervals used were 2, 5, 17, 24, 24, 24, and 72 h for total leaching times after each interval of 0.083, 0.292, 1, 2, 3, 4, and 7 d.

The freestanding liquid previously collected for each sample was added to the rinse, and the rinse and the seven leachates were analyzed for their nitrate concentration. The nitrate concentrations were measured using a Wescan Ion Chromatograph system with a Wescan Anion/R chromatography column.

6. MATRIX GROUT PERFORMANCE RESULTS

The results for the performance criteria tests—frictional pressure drop, 10-min gel strength, critical velocity, unconfined compressive strength, and freestanding liquid—were discussed in detail in ref. 1 and will only be summarized in this report. The nitrate leachability results were not reported in ref. 1 and are presented in this report. The results were analyzed in ref. 1 statistically using the analysis of variance and Tukey's paired comparison techniques.[1,2] Briefly, these statistical techniques demonstrated that:

1. changing the source of dry-solids-blend material did result in significantly different grout properties (i.e., unconfined compressive strength, freestanding liquid, frictional pressure drop, 10-min gel strength, and critical velocity);
2. changing the source of dry-solids-blend material and the variation in grout composition expected from routine operation of the GTF did not result in failure of the criteria for unconfined compressive strength, frictional pressure drop, 10-min gel strength, and critical velocity; and

3. the freestanding liquid results were mixed with some matrix grouts meeting this criterion and others failing this criterion.

These results made the freestanding-liquid criterion the focus of the project and implied that the reference grout formulation needed to be changed to meet the criterion, a need that was beyond the scope of this project. To the extent that the data would allow, an attempt was made to identify what dry-solids-materials-blend properties affected the freestanding liquid. Standard ASTM specifications for these materials were apparently satisfactory for all but the freestanding-liquid criterion. Qualitative guidance suggests that improvement should be realized by using finer particle sizes. Freestanding-liquid performance is discussed in more detail in Subsect. 6.1.

The performance of the reference matrix grouts reported in ref. 1 are summarized in Tables 17 through 20 for the critical velocity, 10-min gel strength, 28-d freestanding liquid, and 28-d unconfined compressive strength. The same statistical information can be obtained from the critical velocity as from the frictional pressure drop; thus a table for frictional pressure drop was not included. (The interdependencies of some of the data were discussed in ref. 1.) The criteria for each of these properties are given at the top of the table. Each table lists the mean, standard deviation, and the probability that the mean will meet the stated criteria, using the hypothesis t-test. Obviously, one has a high level of confidence (>99.9%) that the reference grout will meet the criteria for critical velocity, 10-min gel strength, and 28-d unconfined compressive strength regardless of the sources of the dry solids. This is not the case for the 28-d freestanding liquid. Four matrix grouts gave a >97% probability of meeting this criterion, but the others gave a low confidence level. Some grouts, in fact, gave a >99% probability of *not* meeting this criterion.

6.1 POTENTIALLY SPURIOUS SURROGATE FOR THE FIRST THREE MATRICES

The surrogate 106-AN prepared for the first three matrices (i.e., MX 1, 2, and 3) had a different appearance from subsequent surrogate preparations. While the typical 106-AN surrogate solution was a translucent, yellowish-green solution with a slightly hazy appearance, this first preparation was extremely cloudy (i.e., almost completely opaque) and contained suspended, undissolved solids. The second matrix (i.e., MX 2R) was repeated using some of the later preparations of the 106-AN surrogate. The results for MX 2R were different enough from MX 2 to imply that some unknown effect in preparing the surrogate solution was affecting the results and could compromise efforts to evaluate the effect of varying the source

Table 17. Hypothesis t-test cf the critical velocity obtained from data on the reference formulation[a]

Matrix No.	Critical velocity[b] (gal/min)		Probability mean less than criteria (%)
	Mean	Standard deviation	
1-8	14.2	0.8	99.996
2-3	13.9	0.6	99.998
2R-3	14.1	0.5	99.999
3-2	14.1	0.4	99.9996
4-1	14.7	0.2	99.999996
5-8	16.2	0.2	99.999995
6-7	16.1	0.1	99.9999999995
7-3	18.2	0.3	99.9997
8-4	16.6	0.5	99.999
9-5	14.8	0.5	99.999
10-1	15.2	0.8	99.995
11-1	15.1	0.4	99.9996
12-4	15.7	0.2	99.999996
13-9	14.8	0.2	99.999996

[a] Reference formulation corresponds to observation No. 9 in each matrix. Criterion is <60 gal/min.

[b] Average value and one standard deviation obtained from three replicates.

of dry-solids materials (e.g., this cloudy precipitate appeared to improve freestanding-liquid performance). For this reason the first three matrices—MX 1, 2, and 3—were not used in evaluating the effect of the source of dry-solids materials; MX 2R was used instead.

The cause of cloudy precipitate in this first preparation is not known, but speculation centers on the age of the chemicals used and their exposure to the atmosphere. Although the chemicals had not exceeded their shelf life, they were older than the chemicals later used, and one chemical (i.e., sodium aluminate) was known to have been exposed to room air for prolonged periods. This technical grade sodium aluminate contains significant amounts of sodium hydroxide. In addition to picking up moisture from the air, the sodium hydroxide will also react with carbon dioxide to form sodium carbonate. Thus, the cloudy precipitate may

Table 18. Hypothesis t-test of the 10-min gel strength obtained from data on the reference formulation[a]

Matrix No.	10-min gel strength[b] (lb_f/100/ft^2)		Probability mean less than criteria (%)
	Mean	Standard deviation	
1-8	6.2	0.4	99.999997
2-3	10.8	2.5	99.99
2R-3	3.5	0.0	99.99999[c]
3-2	13.0	1.0	99.999
4-1	6.3	0.6	99.99993
5-8	6.5	0.5	99.99998
6-7	4.7	0.3	99.99999992
7-3	5.5	0.5	99.99998
8-4	4.3	0.3	99.99999993
9-5	4.2	0.3	99.99999993
10-1	3.5	0.5	99.99999
11-1	4.2	0.3	99.99999993
12-4	5.8	0.3	99.99999991
13-9	4.7	0.3	99.99999992

[a] Reference formulation corresponds to observation No. 9 in each matrix. Criterion is <100 lb_f/100 ft^2.

[b] Average value and one standard deviation obtained from three replicates.

[c] Calculated based on standard deviation of 0.5.

have been sodium carbonate, calcium carbonate, or a combination. This premise is only speculation. Such a minor change in the 106-AN recipe could have significant effects on the grout properties. After all, the 106-AN waste has been in storage for decades, will be sparged with air (or some gas), and is known to have significant variations in composition and properties.

Table 19. Hypothesis t-test of the freestanding liquid obtained from data on the reference formulation[a]

Matrix No.	28-d freestanding liquid[b] (vol %)		Probability mean less than criteria (%)
	Mean	Standard deviation	
1-8	2.8	0.8	97.8
2-5	15.3	1.2	0.24
2R-5	8.4	0.0	0.93
3-2	8.8	3.3	9.5
4-1	7.9	3.0	13.0
5-8	3.1	0.8	97.2
6-7	4.7	0.8	70.0
7-3	3.5	0.6	97.5
8-4	5.3	0.5	22.0
9-5	6.9	0.5	1.2
10-1	7.7	0.8	1.6
11-1	8.3	0.5	0.41
12-4	4.0	0.4	97.5
13-9	6.7	0.5	1.6

[a] Reference formulation corresponds to observation No. 9 in each matrix. Criterion is <5 vol %.

[b] Average value and one standard deviation obtained from three replicates.

6.2 FREESTANDING LIQUID

In general, the freestanding liquid achieved its maximum value within 24 h and declined thereafter. In addition, the error in the measurements increased with time as the sample was held under the harsh conditions of 50°C inside ovens. The freestanding liquid observed at 24 h may be more representative of the true freestanding liquid for these products than the value observed at 28 d, although this contention cannot be conclusively proved with the existing data. If true, this means that the data listed in Table 19 are nonconservative and that the true values are even in less compliance than these values.

Table 20. **Hypothesis t-test of the 28-d unconfined compressive strength obtained from data on the reference formulation**[a]

Matrix No.	28-d unconfined compressive strength[b] (psi)		Probability mean greater than criteria (%)
	Mean	Standard deviation	
1-8	489	49	99.99998
2-3	346	21	99.9999998
2R-3	379	30	99.999997
3-2	306	33	99.99994
4-1	492	69	99.9998
5-8	380	23	99.9999998
6-7	481	30	99.9999999
7-3	383	30	99.999997
8-4	392	48	99.99991
9-5	367	32	99.999992
10-1	377	55	99.9997
11-1	510	75	99.9998
12-4	491	35	99.9999993
13-9	436	41	99.99999

[a] Reference formulation corresponds to observation No. 9 in each matrix. Criterion is >60 psi.

[b] Average value and one standard deviation obtained from three replicates.

The freestanding liquid at 2 h was observed to be representative of that at 24 h and usually greater than that observed at 28 d. Thus, a 2-h freestanding liquid test provides an excellent screening test for the 28-d freestanding-liquid performance of the grouts and was used during the later stages of this project (see Appendix E). Also, the 1-d freestanding liquid was used as the main basis of comparison in this section of the report. Table 21 lists the average, standard deviation, and 95 % confidence limits for the 1-d freestanding liquid generated for the reference grout formulation for each matrix.

In order to determine what properties of the dry-blend materials affect the freestanding liquid, it is necessary to try to understand what causes the differences in freestanding-liquid performance among the matrices. The surmise of Dr. Ryan Lokken of Battelle Pacific

Table 21. The average, standard deviation, and 95% confidence limits for the 1-d freestanding liquid using the reference grout formulation data[a]

Matrix No.	Average (vol %)	Standard deviation (vol %)	95% confidence limits (± vol %)
1-8	3.9	0.4	0.9
2-3	20.4	0.6	1.4
2R-3	14.0	0.6	1.4
3-2	11.2	2.4	5.9
4-1	8.9	2.4	5.9
5-8	5.1	1.0	2.5
6-7	6.4	0.7	1.6
7-3	4.4	0.7	1.6
8-4	6.5	0.2	0.5
9-5	8.5	0.4	0.9
10-1	9.7	0.2	0.5
11-1	9.6	0.3	0.8
12-4	5.6	0.2	0.5
13-9	8.3	0.2	0.5

[a]Based on three observations for each matrix.

Northwest Laboratory about the high freestanding liquids observed for the reference grout formulation with simulated 106-AN waste may be the key to understanding the differences observed in this project.[3] During measurements of the adiabatic temperature rise of this grout, Dr. Lokken observed that the temperature remained constant for a period of around 24 h before the temperature started increasing. He surmised that grout set was retarded by some constituent in the simulated waste. This would result in settling and segregation of this soupy grout over a period of about 24 h prior to set, leading to excessive freestanding liquid. In addition, higher temperatures would lead to accelerated set and lower freestanding liquid, as he indeed observed. This surmise cannot be proven conclusively by either Dr. Lokken's data or the data from this project, but it was consistent with his observations. According to Dr. Lokken, the grouts produced during routine operation of the GTF should have even more freestanding liquid than those observed during this project; because, although the temperature of the grout during routine operation will climb well above 50°C (the curing temperature used in this project), the temperature remains at about 40°C during the critical first 24 h when the settling determines the amount of freestanding liquid.

A potential set retarder in the waste solution is not the only possible explanation that fits the facts. Granulated BFS must be activated before it will begin hydrating as a cement. This activation undoubtedly causes a time delay in the start of hydration. In fact, later results from Dr. Lokken clearly demonstrate that hydration begins immediately for cements in 106-AN and is delayed for several hours (as long as 24 h or more) for ground granulated blast furnace slag (GGBFS). Another factor might be the presence of the ground limestone. Cement is ground with gypsum because the gypsum coats the cement particles, delaying set for a few hours and allowing processing and emplacement of the plastic cement mass. Limestone is being considered as a replacement for gypsum, implying that ground limestone will have a similar effect. As noted before, controlled cement hydration begins almost immediately in 106-AN; so it is not clear that the long delay in hydration of the GGBFS can be blamed on ground limestone. The solids settle in only a few hours; therefore it may be moot whether there is a long delay. This settling does lead to a top layer enriched in limestone, supporting the WHC concern about full-scale pouring of this formulation and desire for a pilot-scale pour.

Assuming that the freestanding liquid was controlled by free settling during a retarded (or delayed) set, then the performance could be improved by accelerating the set or by slowing the rate of settling. Theoretically, this could be accomplished within the constraints of this project by specifying a minimum concentration of some constituent in the dry-blend-materials composition that accelerates the set or specifying the smallest particle sizes that can be commercially achieved for each of the products, although neither of these approaches can guarantee meeting the freestanding-liquid criterion. Identifying a chemical specie in the makeup of the dry-blend materials that would control set in a predictable manner proved to be beyond the scope of this project, but some of the dry-blend material characterization tests were definitely a measure of the particle size, or fineness, of the materials. The hydraulic activity, Blaine fineness, slag activity, and particle-size distribution may be, or are, a function of the particle size of the materials.

The results from the matrix data set do support this hypothesis. There were four sources of BFS that supplied two separate slags of different Blaine fineness or grade (i.e., S-6 and S-7, S-9 and S-10, S-11 and S-12, and S-13 and S-14). S-12 was not included in the matrix data set, but the other three combinations resulted in three matrix combinations—MX 2 with MX 3, MX 4 with MX 5, and MX 7 with MX 9—where the only difference in grout composition was a variation in slag fineness. Table 22 lists the slag properties and 1-d freestanding liquid for the reference grout formulations of these six matrices. Table 22 clearly demonstrates that the finer slag, as indicated by the hydraulic activity and Blaine fineness, resulted in less freestanding liquid. The trend for S-6 and S-7 was opposite to the other two combinations for the slag activity index and maximum peak height for the number particle-size distribution. This implies that ordering a higher grade of slag is no guarantee of achieving the desired effect and that the Blaine fineness or hydraulic activity should be specified or measured.

Table 22. Performance of the 1-d freestanding liquid as a function of the blast furnace slag properties

Matrix No.	BFS	D[a] (g/cm^3)	HA[b] (psi)	BF[c] (cm^2/g)	SA[d] (%)	PH[e] (no. %)	1-d FSL[f] (vol %)
2-3	S-10	2.91	1491	4019	115	39	20.4
3-2	S-9	2.91	2355	5657	122	37	11.2
4-1	S-6	2.89	1537	5178	131	30	8.9
5-8	S-7	2.89	1831	6273	110	33	5.1
9-5	S-14	2.91	1319	4190	91	34	8.5
7-3	S-13	2.89	1892	5920	137	27	4.4

[a] Density of BFS.
[b] Hydraulic activity of BFS.
[c] Blaine fineness of BFS.
[d] 28-d slag activity index of BFS.
[e] Maximum peak height in number particle-size distribution of BFS.
[f] 1-d freestanding liquid of the reference grout for this matrix.

Although the Blaine fineness affected the freestanding-liquid performance, there was no reliable way (with the current data base) of predicting what Blaine fineness would be needed to pass the freestanding-liquid criterion confidently, or even whether specification of a given Blaine fineness alone would guarantee passing this criterion. The matrix 1-d freestanding liquid for the reference grout formulation versus the BFS Blaine fineness is plotted in Fig. 19 along with the regression lines for two models.

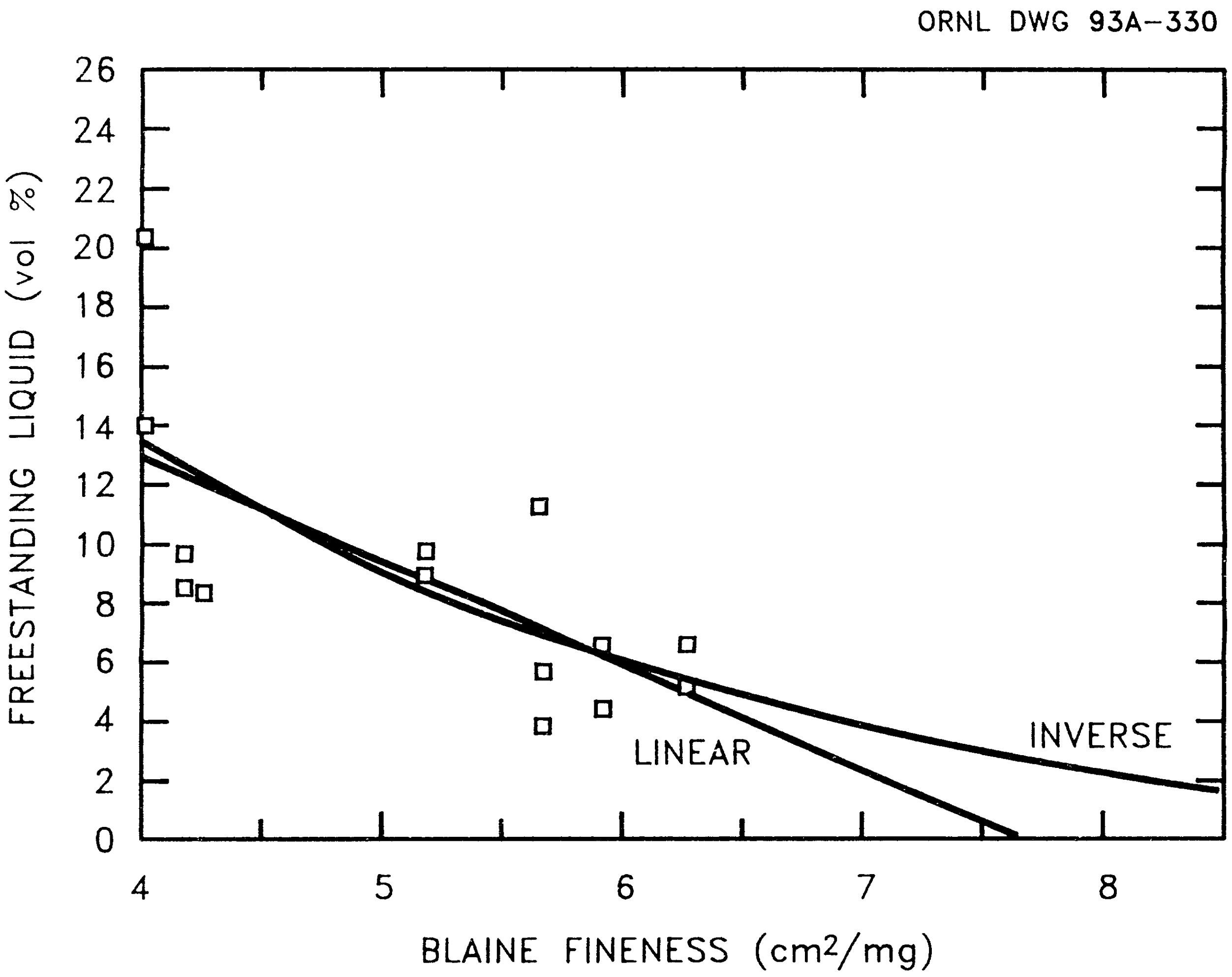

Fig. 19. 1-d freestanding liquid as a function of Blaine fineness.

The linear relationship is

$$y = A + B\ x; \tag{5}$$

the inverse relationship,

$$y = A + B/x,$$

where

y = 1-d freestanding liquid, vol %;

x = Blaine fineness of BFS, cm^2/mg;

A, B = regression constants of appropriate units.

The scatter in these data was obvious, and the linear regression fits were poor (i.e., correlation coefficients on the order of 0.5). These regression fits were extrapolated to get a handle on what Blaine fineness might be needed, based on the observed general trend, to pass a freestanding-liquid test of 1-d freestanding liquid of 1.6 vol %, the criterion value minus the Q-value. (The Q-value provided a comfortable safety margin from 5 vol %. Extrapolation makes these estimates even more unreliable than the predicted interpolated values.) The linear model and inverse model predicted that a Blaine fineness of >7200 and >8400 cm^2/g, respectively, would be required. (The confidence in these regression fits was quite low; for example, the standard deviation for a given y-estimate from both regressions was 3.2 vol %, and confidence in extrapolated y-estimates was worse.) The situation is further complicated by the fact that the grout contains three other fine granular materials besides BFS, and even less data were available on the effect of their particle finenesses. It is also not clear whether the vendor sources can supply material with a Blaine fineness >8000 or whether the cost would be too high even if they could.

In general, the highest Blaine fineness that can be economically attained commercially should be specified for each of the four materials. Negotiations with the vendors can establish the practical upper limit for Blaine fineness in their materials and the price of such ''ultrafine'' material. It is likely that the materials used in this project represent the upper limit of the readily available Blaine fineness of these materials. (One vendor indicated that cements were limited to a Blaine fineness of about 5500 cm^2/g and that the BFS Blaine fineness limit would be higher but in the same ballpark. This limitation was based on the flow properties of such a fine powder because it would ''stick'' in the ball mill and not flow out. The 5500 Blaine fineness cement this vendor produced was marketed as Type III cement, which is a change in the grout formulation that is not allowed by the constraints of this project. This implies that specifying Type I/II-LA Portland cement might implicitly specify the Blaine fineness for the cement and that an extra specification for a higher Blaine fineness is mutually exclusive with the earlier specifications. Negotiations with cement vendor sources should be done to establish the Blaine fineness upper limit that can be specified for Type I/II-LA Portland cement.) Nevertheless, it is possible that one or more vendor sources might be able to produce finer material, with the notable exception of the fly ash, which is not ground. Unfortunately, even if ''ultrafine'' materials can be obtained, meeting the freestanding-liquid criterion is still not certain with any confidence, using the data base generated from this project. (*Caution*: Even if the vendors can supply ''ultrafine'' material, the existing dry-solids handling equipment in the GTF may not be able to handle material this fine.)

Basically, samples of ''ultrafine'' materials, if available, should be obtained and tested for effectiveness in meeting the criteria prior to making a major effort to obtain such materials, just to make sure the effort is worthwhile. The constraints of this project and the data generated during the project forced the recommendation of specifying the highest Blaine fineness obtainable. In summary, the following is recommended:

1. If the vendors have an upper limit on the Blaine fineness and if the GTF can handle material that fine, then the upper limit of the vendor Blaine fineness should be specified in the purchase specifications with appropriate accommodation for normal variability (i.e., specify that the Blaine fineness be greater than a value that is below the lower 95% confidence limit of the maximum Blaine fineness that the vendor can supply or specify the maximum Blaine fineness with a specified 95% confidence limit).
2. If the vendor can supply a Blaine fineness greater than the GTF can handle, then the purchase specification should specify a Blaine fineness close to the GTF upper limit, allowing for a contingency factor to prevent exceeding this upper limit in the normal product variation.
3. If neither the vendor nor the GTF equipment has limits on the Blaine fineness, then the maximum Blaine fineness that can be economically negotiated, or the Blaine fineness that has been conclusively demonstrated to pass the criteria (this project's data were not conclusive), should be specified.

Blaine fineness was a difficult test to perform and would likely require a trained person dedicated to just this task and special facilities (i.e., environmental room with temperature and humidity control) to use this test as a QA/QC tool. Blaine fineness is measured on cements and BFS but not fly ash and limestone. Limestone is a ground material, and specifying a finer ground is desired (e.g., class designation S rather than the T for the limestones used during this project, according to ASTM C 602-69). (The vendor source for the limestone currently only markets one ''grind'' of limestone, but they might be amenable in negotiations to provide a higher ''grind'' to GTF for the right price.) The fly ash particle size probably cannot be controlled, since they are the byproduct of coal combustion; so one would have to resort to sieving this byproduct to limit the particle size, assuming such a service is available.

6.3 NITRATE LEACHABILITY

The nitrate leachability was measured for the reference grout formulations of each matrix combination, plus a grout made using S-12 and a grout made using S-15. These leach samples were made separately from the grouts used for the other performance tests, although the same compositions were used. Even though only reference formulations were made, the same numbering system was used; MX S12 and MX S15 were used for the two BFSs not previously included in the matrices.

Because fresh simulated 106-AN waste was used to prepare the leach samples, the problems encountered in Matrices 1 through 3 in the earlier performance tests did not apply for the leach samples, the results for these three could be compared to the other matrices, and there was no need for a MX 2R matrix grout. Three samples of each grout were leached after curing for 28 d. These triplicate sets of data were analyzed by a computer program (NEWBOX) developed by ORNL for estimating leaching parameters by least squares analysis to get the best estimate of the effective diffusion coefficient.[4]

The nitrate available for leaching was estimated by subtracting the ''measured washoff'' from the total nitrate originally mixed in the grout sample. The ''measured washoff'' was defined as the nitrate in the freestanding liquid plus that rinsed off the sample (the freestanding liquid and rinse were combined and the nitrate measured, hence, the term ''measured''). NEWBOX calculated a nonzero intercept, the ''calculated washoff'' for the diffusion-controlled leaching. The ''total washoff'' was defined as the sum of the ''measured washoff'' and ''calculated washoff.'' The leachability index is defined as the negative logarithm of the effective diffusion coefficient (cm^2/s), that is

$$L = -\log(D) , \tag{7}$$

where

L = leachability index,

D = effective diffusion coefficient, cm^2/s.

NEWBOX estimated the calculated washoff, D, and the standard deviations from the leaching data. The standard deviation for D was used to calculate its 95% confidence limits. By taking the negative logarithm of these upper and lower limits of D, the 95% upper and lower limits of L were calculated. The upper 95% confidence interval was not exactly equal to the lower 95% confidence interval, but they were close enough to be rounded off to approximately the same value. Table 23 lists the amount of nitrate measured in the rinse as a percentage of the total nitrate left in the sample *after* the rinse (rinse wt %), the calculated washoff (and its standard deviation) as a percentage of the total nitrate left in the sample *after* the rinse (calculated washoff wt %), the sum of these first two (total washoff wt %), and the leachability index (and its rounded off 95% confidence interval) for each sample. (The total nitrate *after* the rinse was used as a basis in Table 23 because this was the same basis used in NEWBOX, and the amount leached in the NEWBOX model approaches this value.) Appendix D gives the detailed results of these nitrate leaching tests.

The leachability indices varied from 7.7 to 8.9. The ratio of the mean squares (F-test ratio) was 26.3. The F-value at a 0.1% level of significance is <4 for the (14, 31) degrees of freedom of these leachability results, meaning that the level of significance for the observed F-ratio was well below 0.1% or a better than 99.9% confidence of a significant difference in the leachability indices among the grouts from the different matrices. Thus, the null hypothesis that no difference exists in the leachability indices among the different matrix grouts is rejected. In other words, the different dry-blend sources are concluded to result in different nitrate leaching behavior. The Q-value for the leachability index was calculated to be ±0.79 for the 95% confidence limits. The nitrate leachability indices for any two matrices were significantly different for a given level of confidence if the difference between the average of their measured leachability indices exceeds the corresponding Q-value. The average leachability indices varied from 7.77 to 8.85, a difference of 1.08. Thus, some significant differences were found at the 95% level of confidence. There was no criterion for the nitrate

Table 23. Nitrate leachability index and washoff for 106-AN grouts

Matrix No.	Rinse (wt %)[b]	Calculated washoff[a] (wt %)	Total washoff (wt %)[b]	Leachability index (95% confidence limit)
1-8 A	3.4	1.2 (0.3)	4.6	8.80 (±0.02)
B	3.2	2.0 (0.3)	5.2	8.82 (±0.02)
C	3.4	1.3 (0.2)	4.7	8.77 (±0.02)
2-3 A	7.9	1.3 (0.4)	9.2	8.20 (±0.02)
B	7.5	2.7 (0.3)	10.2	8.60 (±0.02)
C	7.7	1.5 (0.2)	9.2	8.58 (±0.01)
3-2 A	3.8	2.1 (0.2)	5.9	8.68 (±0.02)
B	7.9	3.1 (0.4)	11.0	8.38 (±0.02)
C	4.7	0.8 (0.3)	5.5	8.64 (±0.03)
4-1 A	3.7	1.6 (0.2)	5.3	8.90 (±0.02)
B	5.1	1.5 (0.3)	6.6	8.88 (±0.03)
C	5.4	1.9 (0.3)	7.3	8.77 (±0.02)
5-8 A	11.7	1.9 (0.5)	13.6	8.54 (±0.04)
B	9.2	1.4 (0.5)	10.6	8.65 (±0.03)
C	13.5	1.9 (0.1)	15.4	8.77 (±0.01)
6-7 A	5.6	1.5 (0.3)	7.1	8.76 (±0.02)
B	8.4	1.8 (0.2)	10.2	8.75 (±0.02)
C	8.0	1.2 (0.1)	9.2	8.84 (±0.01)
7-3 A	3.9	0.3 (0.2)	4.2	8.35 (±0.01)
B	4.6	0.1 (0.1)	4.7	8.27 (±0.01)
C	3.7	0.0 (0.1)	3.7	8.29 (±0.01)
8-4 A	3.2	0.1 (0.2)	3.3	8.37 (±0.01)
B	3.1	0.6 (0.1)	3.7	8.44 (±0.01)
C	1.4	0.2 (0.2)	1.6	8.52 (±0.01)
9-5 A	2.7	2.4 (0.8)	5.1	7.76 (±0.02)
B	3.4	3.0 (1.0)	6.4	7.86 (±0.03)
C	0.3	3.1 (1.4)	3.4	7.69 (±0.03)
10-1 A	6.5	0.6 (0.1)	7.1	8.44 (±0.01)
B	7.0	0.8 (0.4)	7.8	8.28 (±0.02)
C	4.2	0.8 (0.3)	5.0	8.30 (±0.02)
11-1 A	3.9	0.0 (0.4)	3.9	7.87 (±0.01)
B	2.5	0.8 (0.4)	3.3	7.89 (±0.01)
C	2.4	0.4 (0.3)	2.8	7.97 (±0.01)
12-4 A	1.6	0.6 (0.1)	2.2	8.38 (±0.01)
B	1.8	0.6 (0.1)	2.4	8.35 (±0.01)
C	1.5	0.5 (0.2)	2.0	8.30 (±0.01)

Table 23 (continued)

Matrix No.		Rinse (wt %)[b]	Calculated washoff[a] (wt %)	Total washoff (wt %)[b]	Leachability index (95% confidence limit)
13-9	A	0.3	1.2 (0.2)	1.5	8.22 (±0.01)
	B	0.3	1.2 (0.3)	1.5	8.22 (±0.01)
	C	0.3	1.0 (0.3)	1.3	8.30 (±0.01)
S12[c]	A	6.3	1.2 (0.5)	7.5	8.91 (±0.05)
	B	6.2	0.7 (0.2)	6.9	8.77 (±0.02)
	C	5.3	0.8 (0.2)	6.1	8.70 (±0.02)
S15[c]	A	7.0	2.1 (0.3)	9.1	8.33 (±0.02)
	B	9.2	1.5 (0.3)	10.7	8.68 (±0.02)
	C	9.8	1.8 (0.2)	11.6	8.60 (±0.01)

[a] The standard deviation for this parameter is given in parentheses.

[b] Wt % of the total nitrate in the sample *after* the rinse. Thus, the sum of the calculated washoff wt %, amount leached wt %, and amount remaining in sample wt % always equals 100 wt %. The total is >100 wt % when the rinse wt % is added because of this basis.

[c] The dry blend with the two additional BFS was 40 wt % P-60, 28 wt % P-62, 4 wt % C-88, and 28 wt % of the indicated BFS (S-12 or S-15). This blend was mixed with simulated 106-AN at a mix ratio of 9 lb/gal.

leachability index except perhaps the value of six required by the Nuclear Regulatory Commission for radioisotopes.

The scatter in correlation between the nitrate leachability index and BFS blaine fineness was even worse than with the 1-d freestanding liquid. Only two grouts—MX 9-5 and MX 11-1—had leachability indices less than eight. Both of these grouts had C-88, P-56, and S-14. The C-88 cement was used in most of the grouts, and the P-56 fly ash was used in two other grouts that had nitrate leachability indices of 8.3 through 8.5. These two grouts were the only ones that used S-14. Although not conclusive, the implication seems to be that S-14 was the cause of these lower leachability indices. The vendor source for S-14 also supplied another BFS, S-13, which was ostensibly the same except for a higher Blaine fineness. Another BFS, S-10, had a Blaine fineness lower than S-14, but the grout using it had leachability indices of 8.2 through 8.6, well above the 7.7 through 7.9 for the grouts made using S-14. This implies that although Blaine fineness may be a factor, it is not the only factor.

7. RECOMMENDATIONS FOR THE PURCHASE SPECIFICATIONS

Based on the observed performance relative to the performance criteria, the specifications currently in use should be sufficient to meet all of the criteria except for freestanding liquid. Summarizing, these specifications are described below.

Limestone

ASTM C 602-69, ''Standard Specification for AGRICULTURAL LIMING MATERIALS,'' can serve as the minimum basis for purchase specifications for this material. The vendor source of this material currently supplies one ''grind'' of this material that would be classed as ''T'' by ASTM C 602-69. Apparently this material also exceeds the calcium carbonate equivalent of not less than 80% as specified in ASTM C 602-69. It may be desirable to specify a Class ''S'' limestone, as opposed to the Class ''T'' tested, because of the freestanding-liquid problem as long as the performance is not adversely affected for the other criteria.

Granulated Blast Furnace Slag

ASTM C 989-88, ''Standard Specification for GROUND GRANULATED BLAST-FURNACE SLAG FOR USE IN CONCRETE AND MORTARS,'' can serve as the minimum basis for purchase specifications for this material. It is recommended that Grade 120, as described in ASTM C 989-88, be specified. It is also recommended that a high Blaine fineness measured according to ASTM C 204-84, ''Standard Test Method for FINENESS OF PORTLAND CEMENT BY AIR PERMEABILITY,'' be specified, allowing for the variation expected during routine operation. Based on the materials supplied by vendor sources during this project, specifying a Blaine fineness of ≥6000 does not appear unreasonable unless the Dry Materials Facility would have trouble handling material this fine.

Fly Ash

ASTM C 618-85, ''Standard Specification for FLY ASH AND RAW CALCINED NATURAL POZZOLAN FOR USE AS A MINERAL ADMIXTURE IN PORTLAND CEMENT CONCRETE,'' can be used as the minimum basis for the purchase specifications for the fly ash. Class F, as defined by ASTM C 618-85, should be specified. It may be desirable to specify presieving of the fly ash to control the particle size of the solids in the grout because of the freestanding-liquid problem as long the performance is not adversely affected for any of the other criteria and if the vendor sources supply such a service.

Cement

ASTM C 150-84, "Standard Specification for PORTLAND CEMENT," can be used as the minimum basis for the purchase specifications for the cement. Type I/II-LA or II-LA Portland cement as defined by ASTM C 150-84 should be specified. It may be desirable to specify a higher Blaine fineness than that measured for the two cements used during this project because of the freestanding-liquid problem; however, a higher Blaine fineness cement must not adversely affect the other performance tests, and the cement should still be Type I/II-LA or Type II-LA Portland cement. (A finer grind may result in accelerated hydration and changing cement into Type III.)

The reference grout formulation cannot meet the freestanding-liquid criterion with a high level of confidence no matter what purchase specifications are used for the dry-blend materials, based on the results observed during this project. A qualitative trend was noted of improving freestanding-liquid performance with the Blaine fineness of the BFS. All other factors being equal, a finer grade of each material should help meet the freestanding-liquid criterion, although the results cannot be guaranteed. The performance should be tested using the finest grades obtainable of each material (1) to ensure that the desired effect is achieved and (2) to specify these finer grades of material in addition to the minimum specifications.

8. SUMMARY AND CONCLUSIONS

1. The reference grout performance was significantly affected by changing the source of the dry-blend materials.
2. Meeting the performance criteria was not significantly affected by the source of the dry-blend materials, except for the freestanding-liquid criterion.
3. The variation in the grout composition from routine operation of the GTF would not significantly affect the grout meeting the performance criteria except for freestanding liquid.

4. The reference grout formulation did not pass the freestanding-liquid criterion with a high degree of confidence for any of the combinations of dry-blend sources, and some combinations failed this criterion with a high degree of confidence.
5. This project was constrained from formulation development to meet the performance criteria and was required to use dry-materials properties that could be used as performance specifications in an attempt to meet the freestanding-liquid criterion.
6. No dry-materials property meeting performance specifications was identified with a high degree of confidence.
7. The best dry-materials property identified for helping control freestanding liquid was the fineness of the dry material, measured as Blaine fineness for the cement and BFS.
8. Specifying high fineness for all of the dry materials may help the reference grout performance approach the freestanding-liquid criterion but cannot be guaranteed to eventually meet this freestanding-liquid criterion.
9. It is recommended that the effectiveness of high fineness materials be tested before a large effort or expense is devoted for this purpose.
10. Other than freestanding liquid, the standard specifications for these materials proved satisfactory.
11. The nitrate leachability indices were also significantly different for different sources of dry-blend materials.
12. Some of the source combinations resulted in nitrate leachability indices approaching nine, fairly high for nitrate, while others were less than eight, more typical of nitrate for such ''soupy'' grouts.
13. The Blaine fineness and slag activity index procedures were difficult to perform and were recommended for QA/QC use by WHC on a routine basis in a special facility only by a person especially trained and dedicated for this purpose. An alternative would be to take samples on a nonroutine basis and submit them to a commercial laboratory familiar with these procedures.
14. The hydraulic activity procedure could be performed by WHC personnel on a nonroutine basis, but it is not clear whether hydraulic activity provides a sensitive enough measure of the fineness, the property of importance for the grout freestanding-liquid performance. In other words, the hydraulic activity could be used to compare different shipments of BFS but not to guarantee a given Blaine fineness.

9. REFERENCES

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2. G.E.P Box, W. G. Hunter, and J. S. Hunter, *Statistics For Experimenters*, Wiley, New York, 1978.
3. Ryan Lokken, Battelle Pacific Northwest Laboratory, November 1990, personal communication to R. D. Spence, Oak Ridge National Laboratory.
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10. ACKNOWLEDGMENTS

The authors wish to thank T. L. Welsh and C. M. Sager, Westinghouse Hanford Company, for their assistance in developing the blend-composition matrix; T. C. Scott, ORNL, for his assistance in statistical evaluation of the results; and C. W. Nestor, Jr., ORNL, for his assistance in upgrading NEWBOX.

APPENDIXES

Appendix A

DRY-SOLIDS-BLEND MATERIALS CHARACTERIZATION PROCEDURES

Appendix A

DRY-SOLIDS-BLEND MATERIALS CHARACTERIZATION PROCEDURES

A major effort of this study was to identify test methods applicable to the characterization of individual dry-solids-blend components. Efforts focused on existing ASTM standard test methods. Methods considered were as follows:

Portland cement
- ASTM C 109-88 Compressive strength
- ASTM C 185-88 Air content
- ASTM C 188-84 Density
- ASTM C 191-82 Time of setting
- ASTM C 204-84 Blaine fineness

Fly ash
- ASTM C 188-84 Density
- ASTM C 311-88 Physical properties
- ASTM C 430-83 Fineness

Granulated blast furnace slag
- ASTM C 204-84 Blaine fineness
- ASTM C 430-83 Fineness
- ASTM C 184-88 Air content
- ASTM C 188-84 Density
- ASTM C 989-88 Slag activity index
- ASTM C 1073-85 Hydraulic activity

The procedures that were used during this project were those for compressive strength, density, Blaine fineness, slag activity index, and hydraulic activity. The details of the hydraulic activity, the density, and the Blaine fineness procedures follow.

A.1 HYDRAULIC ACTIVITY

Hydraulic activity of the BFS samples was evaluated by ASTM C 1073-85, ''Standard Test Method for HYDRAULIC ACTIVITY OF GROUND SLAG BY REACTION WITH ALKALI.'' This test method provides a means for the rapid determination of hydraulic activity by measuring the accelerated strength development of the slag using sodium hydroxide solution as mixing water and curing at elevated temperatures.

As described in the standard, this test method can be used as (1) a quality control test for slag from a single source and (2) an evaluation technique for slag from a specific source with respect to fineness specifications or requirements. Although this test method is intended primarily as a quality control tool, it may also be capable of evaluating the hydraulic activity of slags from different sources.

A.1.1 Experimental Procedure

Samples are prepared in a Hobart Model N-50 mixer in accordance with Step 7 of ASTM C 305-82, ''Standard Method for MECHANICAL MIXING OF HYDRAULIC CEMENT PASTES AND MORTARS OF PLASTIC CONSISTENCY,'' as follows:

1. Place 225 mL of a 20 wt % NaOH solution in the mixing bowl.
2. Add 500 g of the slag sample to the solution; then start the mixer and mix for 30 s on slow speed (~140 rpm).
3. With the mixer still on the slow setting, add 1375 g of Standard natural 20-30 Ottawa sand over a 30-s period.
4. At the end of the 30-s time interval in Step 3, change mixer speed to medium (~285 rpm) and mix for 30 s.
5. At the end of the 30-s time interval in Step 4, stop the mixer and let the mortar stand for 1.5 min. During the first 15 s of the 1.5-min time interval, mortar on the sides of the bowl is scraped into the bottom of the bowl. The bowl is covered with a lid and allowed to set for the remaining time interval.
6. At the end of the 1.5-min time interval in Step 5, the mortar is mixed for an additional minute at a medium setting.
7. The mortar is spooned into 2-in. compressive-strength molds and tamped to ensure complete filling of the molds. The step is carried out on a vibrating table to assist mold filling and remove entrapped air.
8. Excess mortar is removed from the molds by scraping mold top with a spatula.
9. The molds are placed into a plastic container, along with a known volume of water, and then sealed.
10. The plastic containers are placed in a humidity cabinet maintained at 55 ± 2 C and relative humidity of 98%.
11. The samples are cured for 23 ± 0.25 h and then removed. The water contained in the plastic container is measured and compared with the initial amount. If the water loss is greater than 30 wt %, the specimens are to be discarded.

12. The specimens are tested for compressive strength at 24 ± 0.5 h after initiating cure in accordance with ASTM C 109, "Test Method for COMPRESSIVE STRENGTH OF HYDRAULIC CEMENT MORTARS (USING 2-IN OR 50-MM CUBE SPECIMENS)."

A.2 DENSITY

Density of the dry-solids-blend materials was evaluated using ASTM C 188-84, "Standard Test Method for DENSITY OF HYDRAULIC CEMENT." The method utilizes a known weight of material, combined with volume displacement of kerosene, to determine the density.

A.2.1 Experimental Procedure

1. Fill a Le Chatelier flask (constructed in accordance with specifications in ASTM C188-84, see Fig. A.1) with kerosene to a volume between the 0 and 1-mL mark using a long-stem funnel. Care must be taken to ensure the inside flask wall is dry and free of kerosene after filling.
2. Immerse the flask in a Cole-Palmer Model 1268-30 circulating water bath maintained at 20°C and allow 15 min for kerosene temperature to stabilize. (Note: It is critical that the kerosene be at 20°C, and it may take longer than 15 min to stabilize the temperature.)
3. Remove the flask from the water bath and record the level of kerosene. (Note: If the level is above or below the 1 or 0 mark, respectively, adjust the level and repeat Steps 1–3.)
4. Introduce an appropriate weight of sample material (weighed to nearest 0.05 g) using a vibrating pencil. (Note: Care should be exercised to avoid splashing kerosene on the inside flask walls. The appropriate weight of material is ~50 g for fly ash and ~64 g for Portland cement and BFS.)
5. Place a stopper in the flask and gently spin in a horizontal circle until no air bubbles are observed rising to the surface. (Note: If the correct amount of material has been added, the liquid level should be at a position among the upper series of graduations; if not, discard and repeat Steps 1-5.)
6. Place the flask back into the water bath and allow the temperature to stabilize for 15 min.
7. Record the volume of displaced kerosene in milliliters.
8. Determine the density by dividing mass of material from Step 4 by the displaced volume of kerosene from Step 7.
9. Replicate density values should not differ by more than 0.03 g/cm^3.

ORNL DWG 90A-344

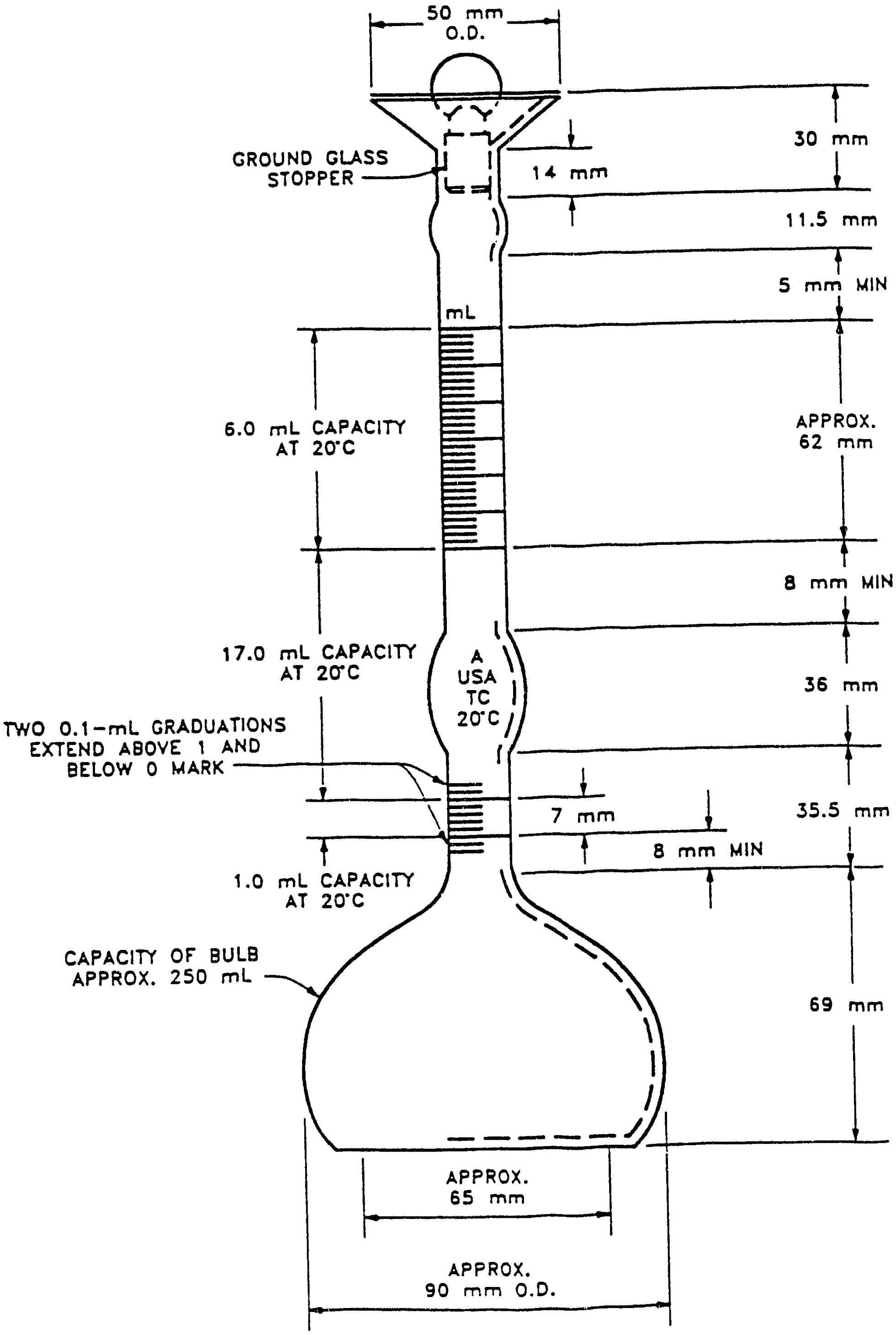

Fig. A.1. Schematic of Le Chatelier flask for the density test as described in ASTM C-188-84.

A.3 BLAINE FINENESS

Tests were performed to determine the fineness of the cement and granulated BFS dry-solids-blend materials using a Blaine air permeability apparatus (Fig. A.2) as described in ASTM C 204-84, "Standard Test Method for FINENESS OF PORTLAND CEMENT BY AIR PERMEABILITY APPARATUS." The Blaine air permeability apparatus essentially consists of a means of drawing a definite quantity of air through a prepared bed of material of definite porosity. The number and size of the pores in a prepared bed of definite porosity is a function of the size of the particles and determines the rate of air flow through the bed. In this method Blaine fineness or total surface area in square centimeters per gram (or square meters per kilogram) of a material is reported relative to a standard material—National Bureau of Standards (NBS) Reference Material No. 114 in this case.

A.3.1 Calibration Procedure for Blaine Air Permeability Apparatus

Determining the bulk volume of a compacted bed of cement:

1. Seat the perforated metal disk in the permeability cell.
2. Place two filter paper disks [Type I, Grade B, as prescribed in Federal Specification for Paper; Filtering (UU-P-236)] in the cell, pressing down edges until the paper disks are flat against the metal disk.
3. Fill the cell with ACS reagent grade mercury, removing any air bubbles that may try to adhere to the cell wall.
4. Level the mercury in the cell with the cell top by placing a piece of plate glass against the mercury surface until the glass is flush with the mercury and rim of the cell.
5. Remove the mercury from the cell and weigh and record the weight.
6. Remove one of the paper disks from the cell.
7. Place 2.80 g (± 0.001 g) of cement sample C-88 into the cell.
8. Tap the cell lightly on the sides to level the cement bed and place another paper disk on top of the cement bed.
9. Compress the cement by pushing the plunger into the cell until it contacts the cell top. (Note: Do not use more than thumb pressure to compress the cement.)
10. Slowly lift the plunger off the cement bed, rotate 90°, and repress. Withdraw the plunger.

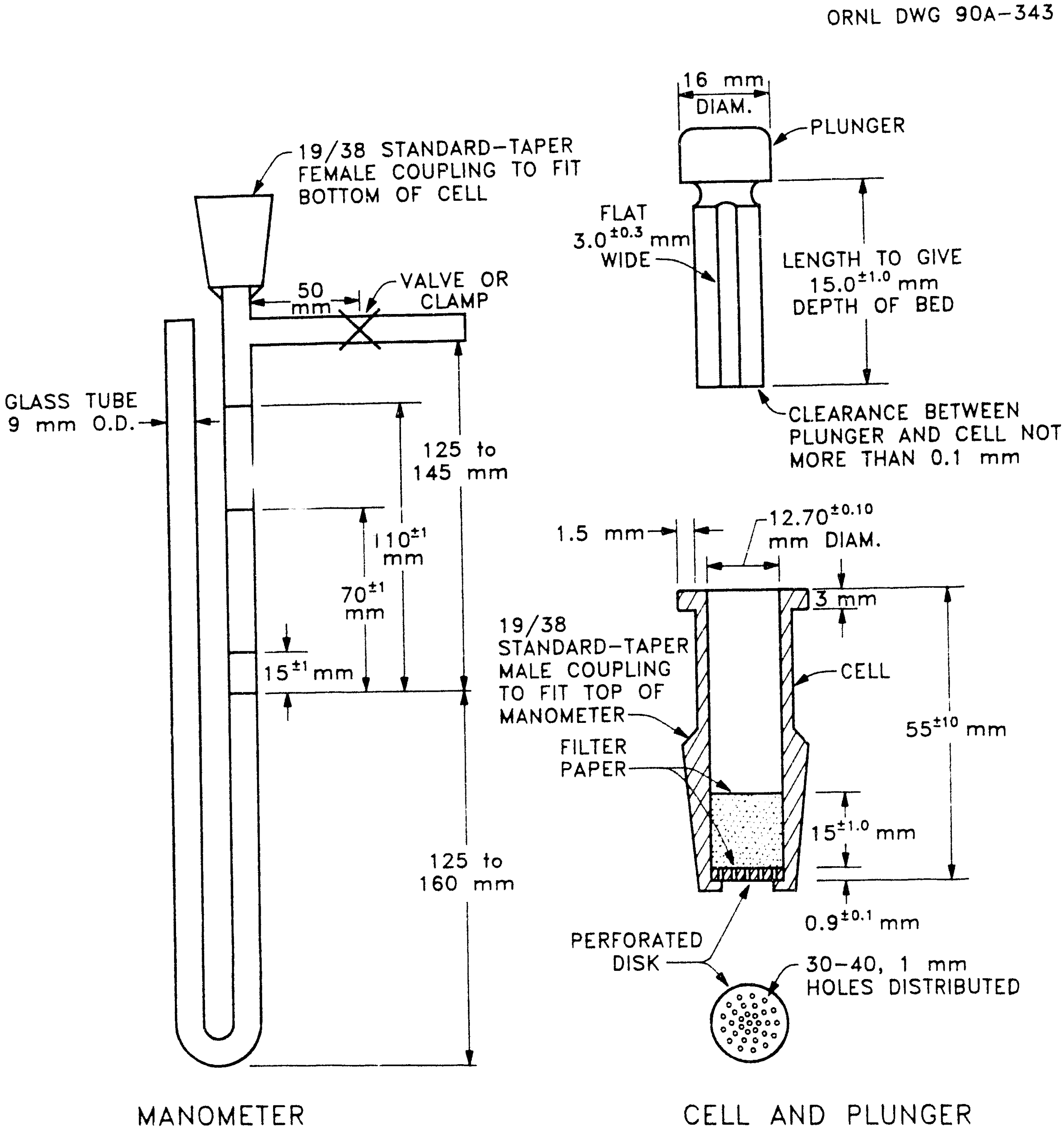

Fig. A.2. Schematic of Blaine air permeability apparatus as described in ASTM C-204-84.

11. Fill the dead space above the compacted cement bed with mercury and level as in Step 4.
12. Remove mercury from cell and record weight. Also, record ambient temperature.
13. Calculate the bulk volume occupied by the cement as follows:

$$V = (WA - WB)/D \ , \qquad (A.1)$$

where

V = bulk volume, cm^3;

WA = grams of mercury recorded from Step 5;

WB = grams of mercury recorded from Step 12; and

D = density of mercury at temperature recorded in Step 12, g/cm^3.

14. A minimum of two determinations of bulk volume of cement is required. The bulk volume value used in subsequent calculations will be the average of two values agreeing within ± 0.005 cm^3. Determinations are repeated until desired agreement has been obtained.
15. Thoroughly clean and gas-dry the cell.

Calibration of the permeability cell

16. Empty the contents of one vial of the NBS Standard Reference Cement into a ~4-oz. jar and shake vigorously for two minutes to fluff cement and break up lumps.
17. Let the jar stand unopened for an additional 2 min, then open the jar and stir gently to evenly distribute fines throughout the sample.
18. Calculate the weight of the standard reference cement to be used in calibration using the following equation:

$$W = \rho V(1-e) \ , \qquad (A.2)$$

where

W = grams of material needed;

ρ = density of test sample (3.15), g/cm^3;

V = bulk volume of bed determined in Steps 13 and 14, cm^3;

e = desired bed porosity (0.5).

19. Using the cement weight determined in Step 18 (weighed to nearest 0.001 g), prepare the bed of cement in the same manner as in Steps 7 through 10.
20. Apply a small amount of stopcock grease to the standard taper on the cell and insert the cell into the coupling on the manometer tube, making sure the connection is airtight.
21. Verify that there is no leakage by stoppering the cell and partially evacuating the manometer tube, then close the stopcock. Any continuous drop in the liquid level indicates a leak in the system. Investigate and repair the leak, if any, before proceeding. Unstopper cell and open stopcock.
22. Using the bulb attachment, slowly evacuate air from the manometer tube until the liquid level reaches the top mark on the tube, then close the stopcock valve tightly.
23. Start the timer when the bottom of the meniscus of the manometer liquid reaches the second (next to the top) mark and stop the timer when the bottom of the meniscus reaches the third (next to the bottom) mark.
24. Record the elapsed time interval in seconds.
25. Record the ambient temperature in degrees (°C).
26. Make one determination of the time of flow on each of three separately prepared beds of the standard reference cement. (Note: A sample may be refluffed and reused provided it has been kept dry and all tests are completed within 4 h of opening the sample vial.)
27. The average time of flow (from at least three determinations) defines the variable, T_s, used in subsequent Blaine fineness calculations (see Sects. A.3.2 and A.3.3).

A.3.2 Blaine Fineness of Cement Samples

1. Obtain a small subsample (~4 oz.) of the cement material.
2. The weight of sample to be used in subsequent steps is the same weight (to the nearest 0.001 g) as that used for the reference cement used in the calibration procedure (see Step 18 in Sect. A.3.1)
3. Assemble the permeability cell (i.e., inset metal disk and one filter disk).
4. Place a sample of weight determined in Step 3 (± 0.001 g) into the cell.
5. Tap the cell lightly on the sides to level the cement bed and place another paper disk on top of the cement bed.

6. Compress the cement by pushing the plunger into the cell until it contacts the cell top. (Note: Do not use more than thumb pressure to compress the cement.)
7. Slowly lift the plunger off the cement bed, rotate 90°, repress, and then withdraw the plunger.
8. Apply a small amount of stopcock grease to the standard taper on the cell and insert the cell into the coupling on the manometer tube, making sure the connection is airtight.
9. Verify that there is no leakage by stoppering the cell and partially evacuating the manometer tube, then close the stopcock. Any continuous drop in the liquid level indicates a leak in the system. Investigate and repair the leak, if any, before proceeding. Unstopper cell and open stopcock.
10. Using the bulb attachment, slowly evacuate air from the manometer tube until the liquid level reaches the top mark on the tube, then close the stopcock valve tightly.
11. Start the timer when the bottom of the meniscus of the manometer liquid reaches the second (next to the top) mark and stop the timer when the bottom of the meniscus reaches the third (next to the bottom) mark.
12. Record the elapsed time interval in seconds.
13. Record the ambient temperature in degrees (°C).
14. Make determinations of the time of flow on each of three separately prepared beds of the cement being tested.
15. The time of flow for each determination defines the variable, T, used in subsequent Blaine fineness calculations.
16. Calculate Blaine fineness of each sample bed using:

$$S = \frac{S_s(T)^{1/2}}{(T_s)^{1/2}}, \qquad \text{(A.3)}$$

where

S = Blaine fineness of test sample, cm^3/g;

S_s = Blaine fineness of reference cement (3460), cm^3/g.

A.3.3 Blaine Fineness Determination for Granulated BFS Samples

Bulk volume determination of a compacted bed of BFS

1. Place two filter paper disks [Type I, Grade B, as prescribed in Federal Specification for Paper; filtering (UU-P-236)] in the cell, pressing down edges until the paper disks are flat against the metal disk.
2. Fill the cell with ACS reagent grade mercury, removing any air bubbles that may try to adhere to the cell wall.
3. Level the mercury in the cell with the cell top by placing a piece of plate glass against the mercury surface until the glass is flush with the mercury and rim of the cell.
4. Remove the mercury from the cell and weigh. Record the weight.
5. Remove one of the paper disks from the cell.
6. Place 2.80 g (±0.001 g) of slag sample into the cell.
7. Tap the cell lightly on the sides to level the slag bed and place another paper disk on top of the slag bed.
8. Compress the slag by pushing the plunger into the cell until it contacts the cell top. Note: Do not use more than thumb pressure to compress the slag.
9. Slowly lift the plunger off the slag bed, rotate 90°, repress, and withdraw the plunger.
10. Fill the dead space above the compacted slag bed with mercury and level as in Step 3.
11. Remove mercury from cell and record weight. Also, record the ambient temperature.
12. Calculate the bulk volume occupied by the slag using Eq. A.1.
13. A minimum of two determinations of bulk volume of slag is required. The bulk volume value used in subsequent calculations will be the average of two values agreeing within ±0.005 cm^3. Determinations are repeated until desired agreement has been obtained.
14. Thoroughly clean and gas-dry the cell and reassemble. Fineness determination for granulated BFS.
15. Obtain a subsample of the slag to be tested (~4 oz.).
16. Calculate the weight of sample to be used in subsequent steps with Eq. A.2 using the following variables: Density (g/cm^3) determined as described in Sect. A.2 and volume (cm^3) as determined in Step 13 e of 0.53 ± 0.005.
17. Place a sample of weight determined in Step 16 (± 0.001 g) into the cell.
18. Tap the cell lightly on the sides to level the slag bed and place another paper disk on top of the slag bed.

19. Compress the slag by pushing the plunger into the cell until it contacts the cell top. (Note: Do not use more than thumb pressure to compress the slag.)
20. Slowly lift the plunger off the slag bed, rotate 90°, repress, and withdraw the plunger.
21. Apply a small amount of stopcock grease to the standard taper on the cell and insert the cell into the coupling on the manometer tube, making sure the connection is airtight.
22. Verify that there is no leakage by stoppering the cell and partially evacuating the manometer tube, then close the stopcock. Any continuous drop in the liquid level indicates a leak in the system. Investigate and repair the leak, if any, before proceeding. Unstopper cell and open stopcock.
23. Using the bulb attachment, slowly evacuate air from the manometer tube until the liquid level reaches the top mark on the tube, then close the stopcock valve tightly.
24. Start the timer when the bottom of the meniscus of the manometer liquid reaches the second (next to the top) mark and stop the timer when the bottom of the meniscus reaches the third (next to the bottom) mark.
25. Record the elapsed time interval in seconds.
26. Record the ambient temperature in degrees (°C).
27. Make determinations of the time of flow on each of three separately prepared beds of the slag being tested. Bulk volume values used in subsequent calculations is the average of **two** values agreeing within ± 0.005 cm^3. It is possible to obtain this precision by testing only two beds of compacted slag.
28. The time of flow for each determination defines the variable, T, used in subsequent Blaine fineness calculations.
29. Calculate Blaine fineness of each sample bed using:

$$S = \frac{S_s \rho_s (b-e_s)(e^3)^{1/2}(T)^{1/2}}{(b-e)(e_s^3)^{1/2}(T_s)^{1/2}} , \qquad (A.4)$$

where

S = Blaine fineness of test sample, cm^3/g;

S_s = specific surface of the standard sample used in apparatus calibration, cm^2/g;

T = measured time interval, seconds, of manometer fluid drop for test sample;

T_s = measured time interval, seconds, of manometer fluid drop for standard sample used in apparatus calibration;

e = porosity of prepared bed of test sample;

e_s = porosity of prepared bed of standard reference cement sample (0.5);

ρ_s = density of test sample, g/cm^3;

s = density of standard reference cement sample (3.15), g/cm^3;

b = constant.

The constant, b, is determined on no less than three samples of the slag being tested. Each slag sample is tested at a minimum of four porosities over a porosity range of at least 0.06. Porosity is varied by selecting a desired porosity and substituting this porosity value along with the volume value determined in Step 14 into Eq. A.2. The resulting calculated weight of material is then used to determine the time of flow as described in this procedure. For each test specimen, b is determined as the intercept a linear fit of $(e^3T)^{1/2}$ vs e on each test specimen. The correlation coefficient for the linear fit of the data for each sample tested must be at least 0.9970. The average value of b obtained from the three test specimens is then used in Eq. A.4. Note that the bed porosities for determining an individual b value are based on the anticipated bulk volume that the compacted bed at the selected porosity would occupy in the permeability cell. Too little material in the cell does not compact uniformly between replicate runs, and the resulting data will not fall within the precision requirements of this test method.

Appendix B

DRY-SOLIDS-BLEND-MATERIALS CHARACTERIZATION DATA

Appendix B

DRY-SOLIDS-BLEND-MATERIALS CHARACTERIZATION DATA

B.1 DATA SUPPLIED BY VENDOR SOURCES

Reference 1 listed this information for the original two limestones (P-58 and P-59), one cement (C-88), both fly ashes (P-55 and P-56), and nine out of the ten BFSs (S-6, S-7, S-8, S-9, S-10, S-11, S-12, S-13, and S-14). This information will not be duplicated here and is the same for second batches supplied of some of these materials (C-90 for C-88, P-60 for P-59, P-61 for P-56, P-62 for P-55, S-18 for S-6, S-19 for S-7, S-16 for S-8, S-22 for S-9, S-20 for S-10, S-23 for S-11, S-21 for S-12, and S-17 for S-14). The information not included in ref. 1 follows (i.e., for P-65, C-91, and S-15).

$CaCO_3$

Source: Ash Grove Cement West, Inc.
P.O. Box 83007
St. Johns Station
Portland, Oregon 97283-0007

Label: P-58 (Ground), P-59 and P-60 (Flour), and P-65 (New)

Mesh (sieve opening, mm)	Ground limestone (P-58) wt % passing	Limestone flour (P-59 & P-60) wt % passing	New grind (P-65) wt % passing
10 (2.000)	99	100	100
20 (0.841)	97	99	99.5
40 (0.420)	90	98	97.5
60 (0.250)	80	94	--
100 (0.149)	70	90	88.5
200 (0.074)	--	--	75.5

Cement, Type I/II-LA

Source: Lafarge Corporation
N. 209 Havana St.
P.O. Box 13189
Spokane, Washington 99213-3189

Label: C-91

Origination: Exshaw, Alberta, Canada

CHEMICAL COMPOSITION			wt %
SiO_2			21.14
Al_2O_3			3.59
Fe_2O_3			5.18
CaO total			61.55
MgO			4.32
SO_3			2.42
Loss on ignition			0.94
Insoluble residue			0.20
Alkalies (Na_2O equivalent)	max 0.59%	min 0.57%	0.58
C_3S	max 52.34%	min 49.35%	51.13
C_3A	max 1.03	min 0.76	0.89
C_2S			22.03
C_4AF			15.77

PHYSICAL TESTS	
Blaine	3860 cm^2/g
Setting time: Vicat	
Initial	115 min
Final	195 min
Autoclave expansion	0.10%
Air entrainment	7.4%
Compressive strength	
3 d	3065 psi
7 d	3875 psi

Granulated Blast Furnace Slag

Source: Reiss Lime Company of Canada Limited
P.O. Box 1690
Blind River, Ontario POR 1B0

Label: S-15

TYPICAL SLAG CHEMISTRY	wt %
MgO	16.0
CaO	32.5
SiO_2	38.0
Al_2O_3	8.5
K_2O	0.6
MnO	1.4
Fe_2O_3	0.3
S	1.2
Other	1.5

PHYSICAL DATA	
Product identifier	SLAG CEMENT
Physical state	grey powder
Odour	slight sulphur odor
Appearance	pale grey powder
Specific gravity	2.92
pH (approximately)	13

B.2 DATA MEASURED DURING THIS PROJECT

Most of the density and Blaine fineness, and all of the hydraulic activity, results were reported in ref. 1. The density and Blaine fineness results not reported in ref. 1 and the slag activity index results were reported here. For the sake of completeness and comparison, the density and Blaine fineness results reported in ref. 1 were also reported here. (Thus, only the hydraulic activity results were reported in ref. 1, but not here.) Some measurements were also reported on cement C-92. This was a Type I/II cement acquired locally (Dixie Cement Co., Knoxville, TN) for measuring the slag activity index, which requires a cement of a certain specified chemical composition, including an alkali content higher than the LA cements used in the Hanford grout formulation.

Table B.2.1. Density of matrix materials[a]

Material	Density (g/cm^3)			
	Replicate measurements			Average
		Cement		
C-88	3.176	3.184		3.18
C-91	3.168	3.168		3.17
C-92	3.153	3.138		3.15
		Fly ash		
P-55	2.183	2.178		2.18
P-56	2.336	2.341		2.34
P-61	2.304	2.294		2.30
P-62	2.222	2.208		2.21
		Blast furnace slag		
S-6	2.896	2.889		2.89
S-7	2.896	2.889		2.89
S-8	2.869	2.896	2.889	2.88
S-9	2.909	2.909		2.91
S-10	2.903	2.909		2.91
S-11	2.896	2.909		2.90
S-12	2.896	2.896		2.90
S-13	2.883	2.889		2.89
S-14	2.916	2.896		2.91
S-15	2.929	2.923		2.93

[a] Measured according to ASTM C 188-84.

Table B.2.2. Blaine Fineness measurements[a]

Material	Blaine fineness (cm^2/g)				
	Replicate measurements			Average	Standard deviation
		Cement			
C-88	3779.6	3791.6	3791.6	3788	6
C-91	3875.8	3818.3	3779.4	3825	40
C-92	3338.3	3367.3		3353	15
		Blast furnace slag			
S-6	5170.0	5194.9	5170.0	5178	14
S-7	6296.0	6263.0	6260.9	6273	20
S-8	5684.0	5659.0		5672	18
S-9	5411.0	5668.0	5646.0	5657	16
S-10	3922.0	4083.0	3955.0	4019	91
S-11	4271.1	4289.2		4280	13
S-12	5045.4	5035.2		5040	7
S-13	5925.7	5832.2	5907.5	5888	40
S-14	4188.7	4198.3		4194	5
S-15	5922.5	5911.7		5917	5

[a] Measured according to ASTM C 204-84.

Table B.2.3 Slag activity index measurements[a]

Test length	Unconfined compressive strength (psi)							Slag activity index (%)
	Replicate measurements						Average	
C-92								N/A
7 d	4700	4740	4770	4370	4400	3940	4450	
	4660	4500	4910	4300	4740	4380		
	3880	4140	4320					
28 d	3860	3430	3640	3930	4690	3480	3883	
	3900	4240	4210	3960	3910	3500		
	3730	3940	3830					
S-6								
7 d	3730	4280	3900	4210	4260	4310	4120	92
28 d	5070	4250	5880	5620	5020	4780	5103	131
S-7								
7 d	4130	3920	4020	3230	3280	3770	3730	84
28 d	5190	4260	4490	3630	4050	4130	4291	110
S-8								
7 d	4150	4190	4220	4240	3240	3860	3980	90
28 d	4730	5240	4540	4270	4490	4340	4601	118
S-9								
7 d	3730	3300	4150	4890	4820	4950	4310	97
28 d	5080	4050	4430	5070	4570	5280	4746	122
S-10								
7 d	3790	3840	3720	4510	4240	3710	3970	89
28 d	3950	4300	4270	4640	5320	4420	4483	115
S-11								
7 d	4620	3950	4170	3640	3810	3590	3960	88
28 d	4500	4590	4590	4830	4890	4340	4623	119
S-12								
7 d	4230	4340	3700	5350	4830	4710	4526	101
28 d	4690	5240	5500	6330	5900	5260	5486	141
S-13								
7 d	5130	5150	4940	4300	4740	4780	4840	108
28 d	5280	5680	5640	4740	5570	5020	5321	137
S-14								
7 d	3010	3420	3380	3180	3450	3140	3263	73
28 d	3410	3190	3250	4050	3630	3690	3536	91
S-15								
7 d	3630	4170	4290	4230	4080	3950	4058	91
28 d	4280	4790	4530	5710	4860	4090	4710	121

[a] Measured by a modified ASTM C 989-88 procedure.

Appendix C

DRY-SOLIDS-BLEND-MATERIALS CHARACTERIZATION BY MICROTECHNIQUES

Appendix C

DRY-SOLIDS-BLEND-MATERIALS CHARACTERIZATION BY MICROTECHNIQUES

Samples of the dry-blend materials from different sources were submitted to the Technical Services Division of K-25 for evaluation by scanning electron microscope (SEM) and X-ray diffraction (XRD). The report on this evaluation is included in this appendix in its entirety except for Appendix I, which is a copy of the project outline, and the numerous photomicrographs referred to in the report. At present, these photomicrographs are contained in a looseleaf binder under the control of R. D. Spence and are stored in Room A-26, Building 4500N.

Characterization of Individual Grout Components: Westinghouse Hanford Company Material

17 January 1991

Prepared for
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Dianna Vail

Glossary of Terms:

BEI (Backscattered Electron Imagining) — The use of backscattered electrons, which results from the interaction of the primary electron beam with the sample, to image the specimen. The contrast of BEI is proportional to the average atomic number (high Z, bright image).

EDS (Energy Dispersive X-ray Spectroscopy) — An analytical technique which measures the energy of x-rays emitted from a sample which has been excited by a beam of high energy electrons. The x-ray energy distribution and intensities provides qualitative and quantitative elemental information.

IA (Image Analysis) — A wide variety of software tools that allows data manipulation of a digital (stored) electron micrograph. Calculations to determine particle size, pore size distribution, chemical speciation are a few of the possible applications.

SEI (Secondary Electron Imaging) — The use of secondary electrons, which are produced by the impingement of primary electrons upon a sample, to image the specimen.

SEM (Scanning Electron Microscopy) — An instrumental technique which utilizes a finely focused beam of high energy electrons to image the surface of a sample over a dynamic range of magnifications (~10× to 500,000×).

XRD (X-ray Diffraction) — An analytical technique which measures the angle and intensity of x-ray diffraction from a polycrystalline sample. The resulting diffraction pattern supplies qualitative and quantitative chemical phase information.

Introduction

Nineteen samples of raw cementitious materials used for grouting waste were received for characterization. Samples of Portland cement-type I,II, fly ash-class F, limestone, and blast furnace slag (BFS) were obtained from Chemical Technology Division personnel. These samples were representative of materials used in previous grouting studies. The results of these studies had revealed that certain combinations of the raw materials responded better to waste immobilization criteria than others. The morphology, particle-size distribution and crystalline phases of the materials were to be analyzed to reveal differences which may be related to the differences in the waste immobilization performance criteria. A copy of the project outline is listed as Appendix I.

Table I lists the code used for the samples and their general class of material. Note: an H was added as a prefix to all sample names during the course of the project (see appendix I) and will be seen associated with the data. The names listed in Table I are consistent with those used by the Chemical Technology Division. Samples were supplied in triplicate, by random grabs samples, from the same container presently being used in the laboratory. The triplicate samples were used to check homogeneity of the material on a localized population. The triplicate sample were denoted by a -A, -B, or -C suffix onto the code listed in Table I.

Characterization was split into two levels; primary and secondary tests. The primary tests consisted of XRD and low magnification (~500×) SEM analysis of all 57 samples. The secondary tests were not performed in triplicate and were only performed on selected samples (as denoted by an asterisk, *, in Table I). This secondary test characterization consisted of EDS analysis, SEM morphology analysis, particle-size distribution (IA), and cross section analysis. The exception to this is that EDS analysis was performed on all 19 of the samples. Due to the differences in the nature of the tests the results for the primary test will be presented on the basis of the individual tests. The results of the secondary test will be presented based upon the nature of the material.

Objective

This investigation is to serve two main purposes:

1) Document the chemical, phase, and morphological nature of the raw grout materials.
2) Determine if any of the measure parameters (crystalline phases, elemental distributions, particle size, or morphology) can be correlated to the indexes used to evaluate the grout/waste mixture.

Results: Primary Tests

XRD Analysis:

Table II lists XRD results for the 19 materials. No significant differences were found among the triplicate samples and hence the average results from the three samples is listed. The relative intensity is a semi-quantitative number which was calculated by determining the percent contribution to the total intensity based upon the major diffraction line from each phase. No quantitative meaning is implied by this number and only semi-quantitative relationships should be concluded.

Portland Cement The XRD patterns collected for the three Portland cements were similar (Table I). The patterns indicated that the majority of the material was crystalline in nature. The interpretation of these patterns is still being performed. The initial phase identification for all three cements is a calcium magnesium aluminum silicate phase. This is a solid solution material which is a combination of the various oxides (54 CaO, 16 SiO_2, Al_2O_3, MgO). The JCPDS file (#11-593 & 13-272) comments stated that this occurs in Portland cement clinker and in basic slags. This was the same crystalline phase which was identified in an earlier report (K/QT-203 "Blast furnace slags-Cement blends for the immobilization of Tc-Containing waste") There are still unidentified peaks present in the patterns. The percent phase contribution is based upon the assumption that the unidentified peaks represent a single phase.

Limestone The XRD patterns of the limestone material were essentially identical. All patterns indicated a pure crystalline phase of calcite ($CaCO_3$).

Fly Ash The XRD patterns of the fly ash materials were different in the total phase contribution but similar in the relative weak diffraction response. This suggests that the fly ash material is not purely crystalline in nature. From the results in Table I it can be seen that quartz is the major constituent in all three materials. The difference between the three materials is the extent of crystalline iron oxide detected. The iron oxide phase ranges from 0% for the P-61 material to 27% for the P-62 material. It should be noted that the diffraction patterns collected for the P-62 material was the weakest of the three materials.

Blast Furnace Slag The XRD patterns collected for the BFS material indicated that the material was primarily amorphous. Very few diffraction lines were detected. Listed in Table I for the BFS material, beside the phase information, is a ratio of the signal intensity to the background intensity (S/B) for the most intense peak. It can clearly be seen that the majority of the BFS materials had values less than one. As a comparison the value for one of the limestone materials (P-58) was also calculated and is listed in Table I. It can be seen that there are several orders of magnitude difference between the signal intensity of the limestone and the BFS. This indicates that the blast furnace slag material is not crystalline in nature. For several of the BFS samples (S-6, S-7, S-13, S-14) a calcium magnesium orthosilicate phase (Merwinite) was found to account for the majority of the maximum phase intensity. But for most of the BFS material the crystalline phase was not identifiable (UID).

SEI Analysis:

Figure 1 shows the collection of electron micrographs collected for the 57 samples. Each page consists of electron micrographs from the three replicate samples for a specific material. The materials identification and negative number of each micrograph is given in the key in the lower right hand corner of the page. With the exception of fly ash there is striking similarity to the morphology of the remaining three materials. The distinctive spherical nature of the fly ash is clearly unique among these materials. The particle-size distribution will be favored by the smaller particles. This can clearly be seen by looking at the number of larger particles compared to the number of smaller particles that the smaller particles will dominate the number based size distribution.

Series One:

General

Figure 2 (which constitutes the final 2/3 of the notebook) shows a representative EDS spectrum, the standardless semi-quantitative analysis results, the particle-size distribution, and high resolution electron micrographs for each of the series one materials. Note, the EDS analysis and particle-size distribution was performed on all samples and is included, in order, with these results. It should be noted that the results of the semi-quantitative analysis only represents the elemental distributions for elements with atomic number greater than $Z \geq 11$ (Na). The samples which were analyzed were not polished and no correction for surface roughness has been attempted. The collection method was kept consistent from sample to sample and hence the results have a high level of confidence when used to compare trends between samples but the exact magnitude of the result does not imply an exact quantitative figure.

The key to the micrographs is shown in the header sheet for ''Fig. 2''. The micrograph in the upper left hand corner was taken at low accelerating voltage (5 kV) at a 1000x. This will be the most surface sensitive micrograph. The remaining three micrographs were taken at 20 Kv accelerating voltages at 1000x, 2000x, and 3000x as shown in the key. All micrographs show the structure of the materials in very good detail.

Portland Cement

The EDS results for the three Portland cement samples show that the elemental profiles for the materials are relatively constant. There are some minor differences as can be seen in the Table but it is difficult to estimate the importance of differences in the minor components. The electron micrographs illustrate a wide range of particle sizes and illustrate the similarity in the morphology between the materials. The particle-size distribution for the cement samples are also shown.

Limestone

The EDS results are in agreement with the XRD results in that the material is essentially a pure $CaCO_3$ phase. All three samples show small contributions of impurities. The electron micrographs shows that the morphology of the three limestone samples are representative. The particle-size distribution of the limestone material is shown.

Fly Ash

The elemental distribution for the fly ash samples show the P-56 and P-61 materials are similar in elemental nature but that the P-62 sample has a higher Na content and is diminished in Si. No morphological difference were seen in the electron micrographs. The particle-size distribution of the fly ash materials are shown.

Blast Furnace Slag

The results of the EDS analysis are split into two categories; the series one samples and the non-series one samples. In comparison of the series one samples the main discrepancy can be seen to be in the Mg to Al ratio. The S-7, S-10 and S-13 samples have a higher Mg level where as the S-8 sample has a higher Al level. This same discrepancy can be seen for the non-series one samples where the S-6, S-9, S-14, and S-15 samples have higher Mg levels and the remainder have higher Al levels. Some discrepancies in the Ca level can be seen for this series of material. No discrepancies in morphologies were noticed for the BFS material. The particle-size distributions of the slags are shown.

Discussion/Conclusions:

This study is the cumulative effort of many people. This study has learned a lot about characterization methods of cementitious materials and the abilities of several of the instrumental techniques have been shown. In analysis of the results there does not appear to be a readily apparent trend between any of the parameters measured in this study and the results of the freestanding liquid or compressive strength tests.

In addition to the individual data tables also include in the collection of tables is a summary of the particle-size data for all of the Hanford material. Also shown in this table is the aspect ratio. The aspect ratio is a measure of the width to length of the particles and hence is influenced by average particle morphology. By looking at the aspect ratios it is clear that the blast furnace slags, limestones, and cements have similar morphologies and the fly ash is different. This is in agreement with the SEM results.

The last table added shows the compilation of the EDS results. Also shown on this table is the expected elemental profile for the grout matrix based upon the combination of the various components. Also show are the average and standard deviation (SD) for the elemental profiles. It can be seen that the elemental profiles are consistent regardless of their components.

Table I

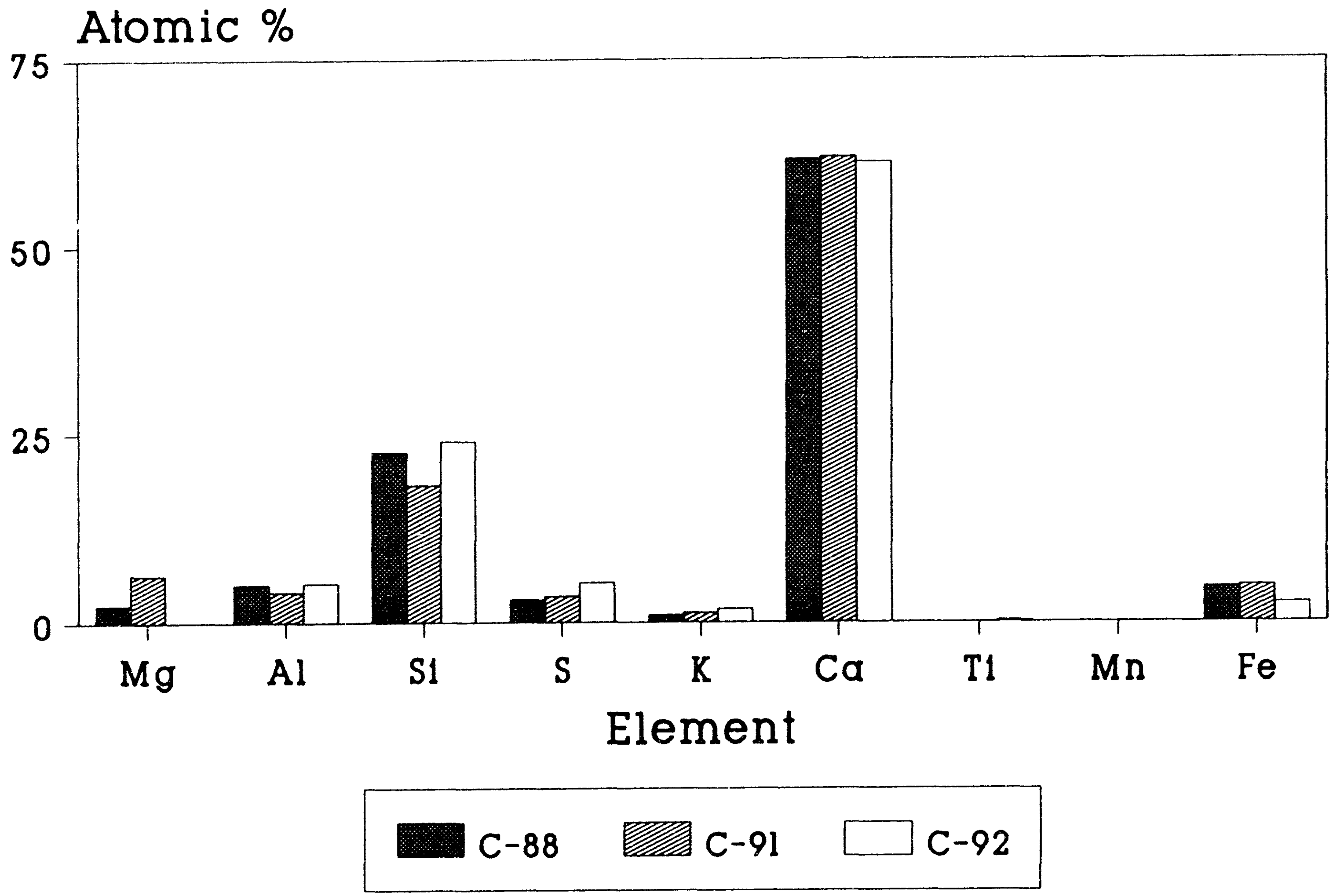
Nomenclature used for Sample Identification

SERIES-1	Sample	Description
*	C-88	Portland Cement, Type I,II
*	C-91	Portland Cement, Type I,II
	C-92	Portland Cement, Type I,II
*	P-58	Limestone
*	P-60	Limestone
*	P-65	Limestone (added after project initiation)
	P-56	Fly Ash, Class F
*	P-61	Fly Ash, Class F
*	P-62	Fly Ash, Class F
	S-6	Blast Furnace Slag
*	S-7	Blast Furnace Slag
*	S-8	Blast Furnace Slag
	S-9	Blast Furnace Slag
*	S-10	Blast Furnace Slag
	S-11	Blast Furnace Slag
	S-12	Blast Furnace Slag
*	S-13	Blast Furnace Slag
	S-14	Blast Furnace Slag
	S-15	Blast Furnace Slag

Table II

XRD results averaged for the triplicate samples

Sample	Phase (Relative Intensity)	
Portland Cement:		
C-88	$Ca_{54}MgAl_2Si_{16}O_{90}$ (85%), UID (15%)	
C-91	$Ca_{54}MgAl_2Si_{16}O_{90}$ (87%), UID (13%)	
C-92	$Ca_{54}MgAl_2Si_{16}O_{90}$ (84%), UID (16%)	
Limestone:		
P-58	Calcite/$CaCO_3$ (100%)	S/B= 256
P-60	Calcite/$CaCO_3$ (100%)	
P-65	Calcite/$CaCO_3$ (100%)	
Fly Ash:		
P-56	Quartz/SiO_2 (92%), Hematite/Fe_2O_3 (8%)	
P-61	Quartz/SiO_2 (100%)	
P-62	Quartz/SiO_2 (73%), Hematite/Fe_2O_3 (27%)	
Blast Furnace Slag:		
S-6	Merwinite/$Ca_3Mg(SiO_4)_2$ (76%), UID (24%),	S/B= 1.8
S-7	Merwinite/$Ca_3Mg(SiO_4)_2$ (82%), UID (18%),	S/B= 2.7
S-8	UID (100%),	S/B= 0.8
S-9	UID (100%),	S/B= 0.3
S-10	UID (100%),	S/B= 0.3
S-11	UID (100%),	S/B= 0.3
S-12	UID (100%),	S/B= 0.5
S-13	Merwinite/$Ca_3Mg(SiO_4)_2$ (62%), UID (38%),	S/B= 0.7
S-14	Merwinite/$Ca_3Mg(SiO_4)_2$ (75%), UID (25%),	S/B= 0.4
S-15	UID (100%),	S/B= 0.4

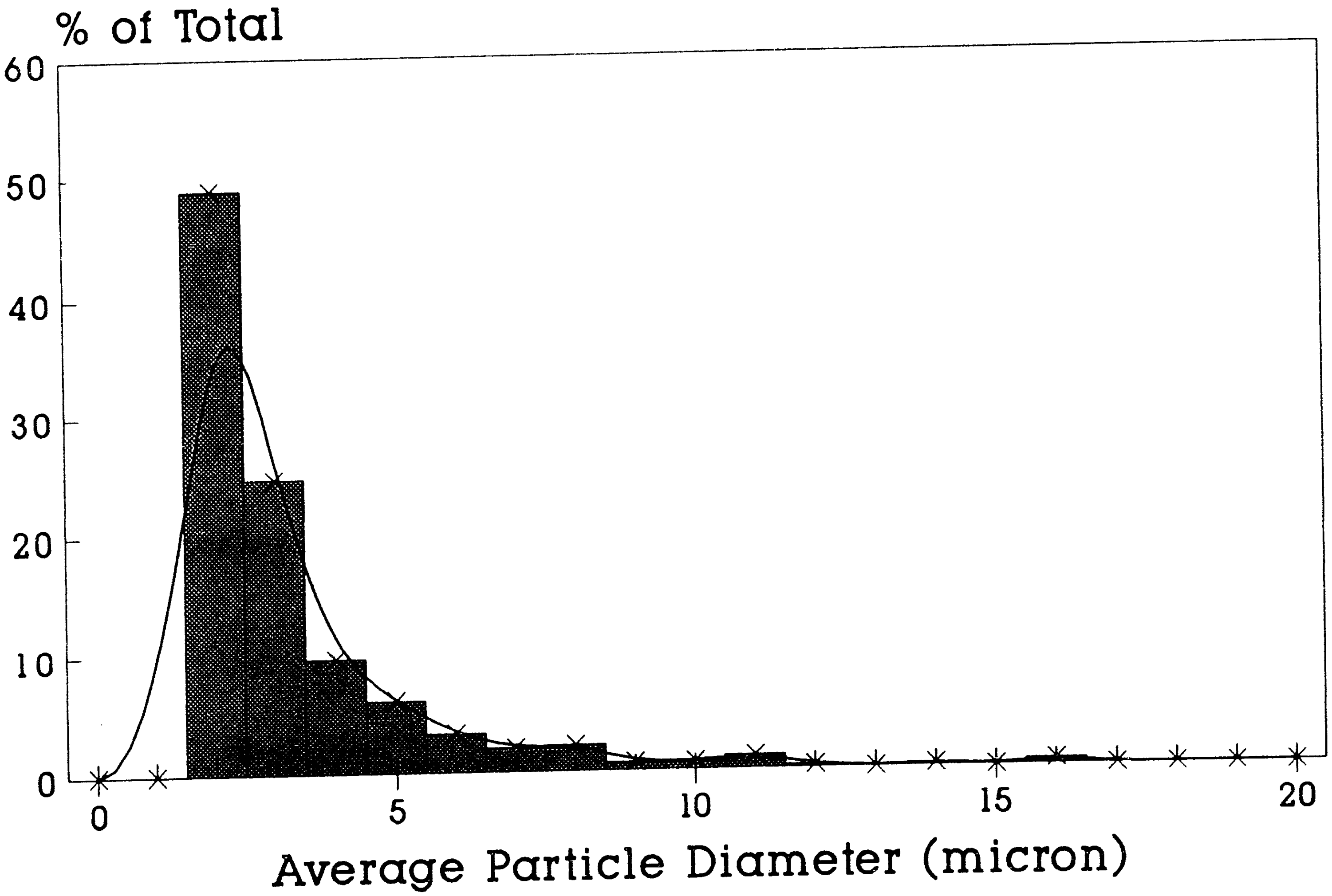


Elemental distributions for Portland cement.

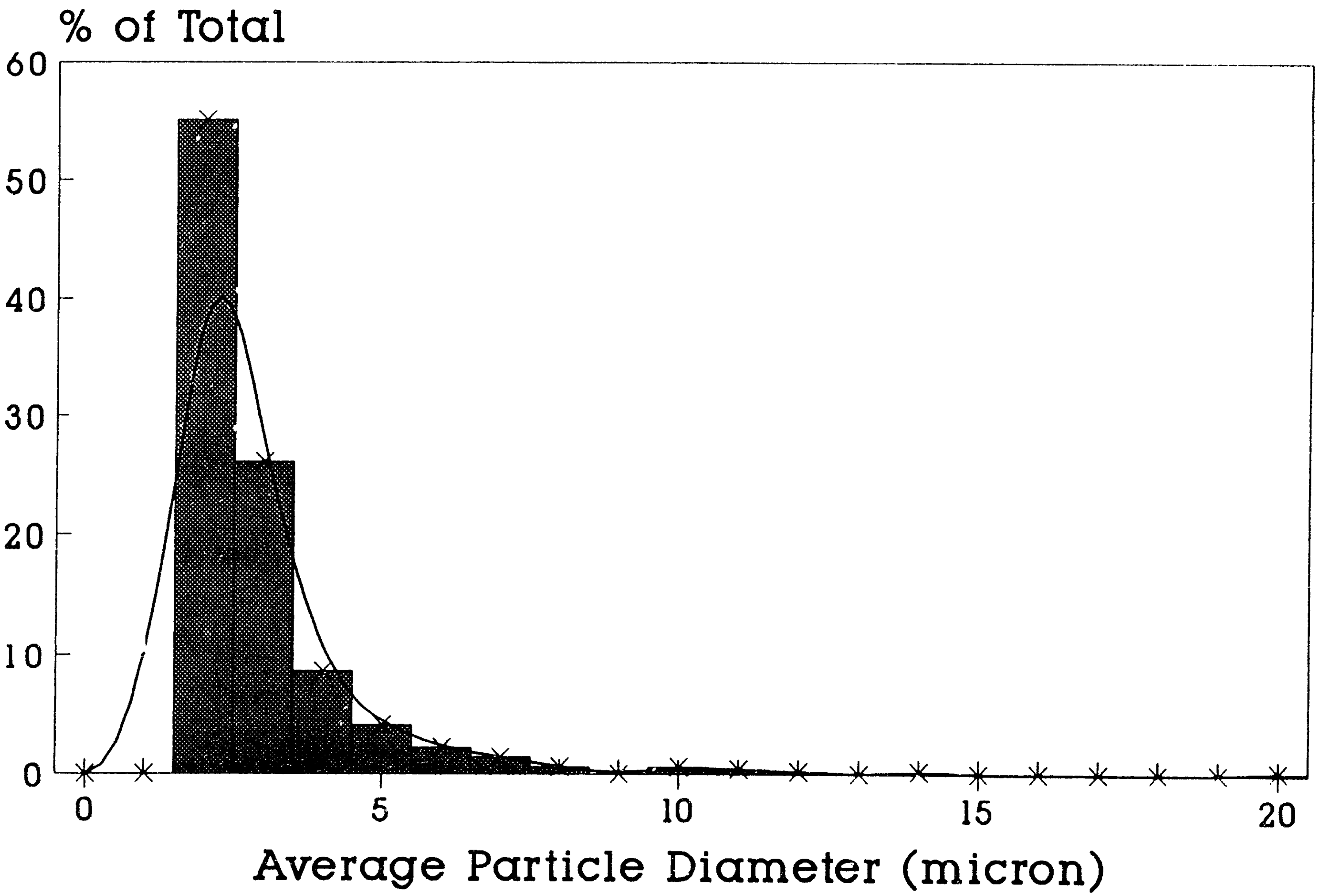
Standardless Semi-quantitative EDS Analysis

Portland Cement

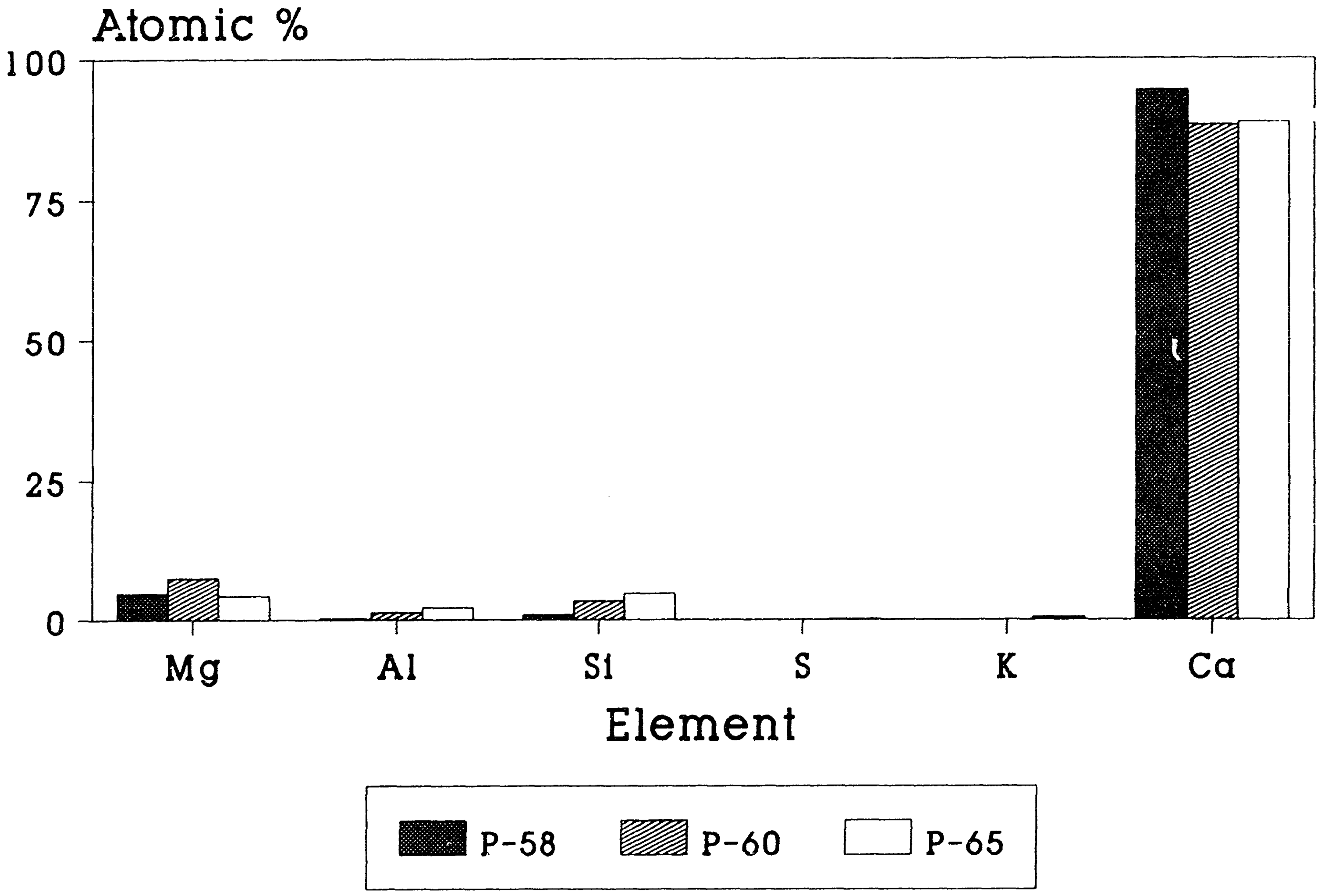
Element	C-88	C-91	C-92
Mg	2.3	6.3	0.0
Al	5.0	4.0	5.2
Si	22.5	18.3	24.0
S	2.9	3.4	5.2
K	0.9	1.3	1.7
Ca	61.8	62.0	61.3
Ti	0.0	0.0	0.1
Fe	84.6	4.8	2.5



Particle size distribution for C-91 Portland cement.



Particle size distribution for C-92 Portland cement.

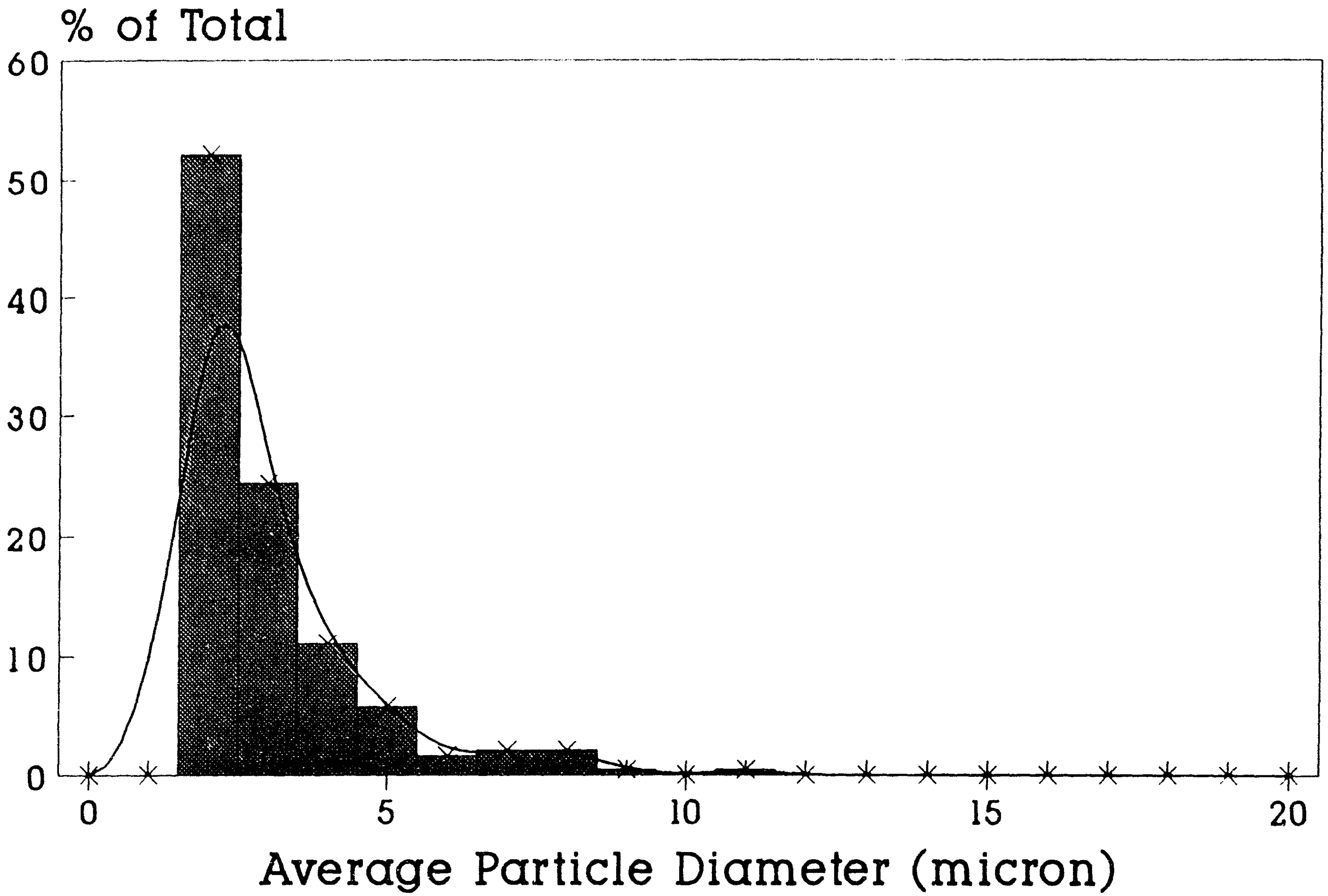


Elemental distributions for limestone.

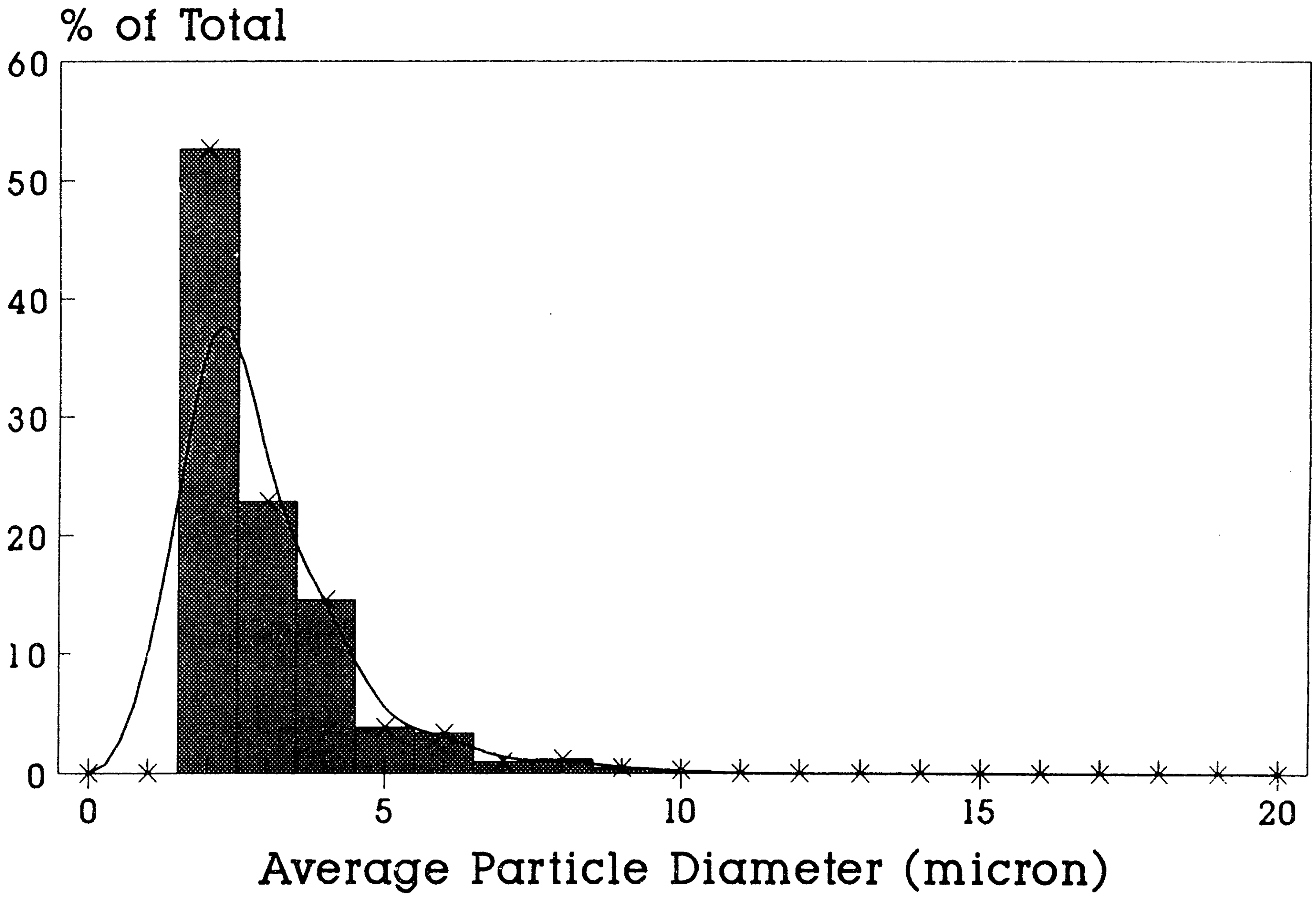
Standardless Semi-quantitative EDS Analysis

Limestone

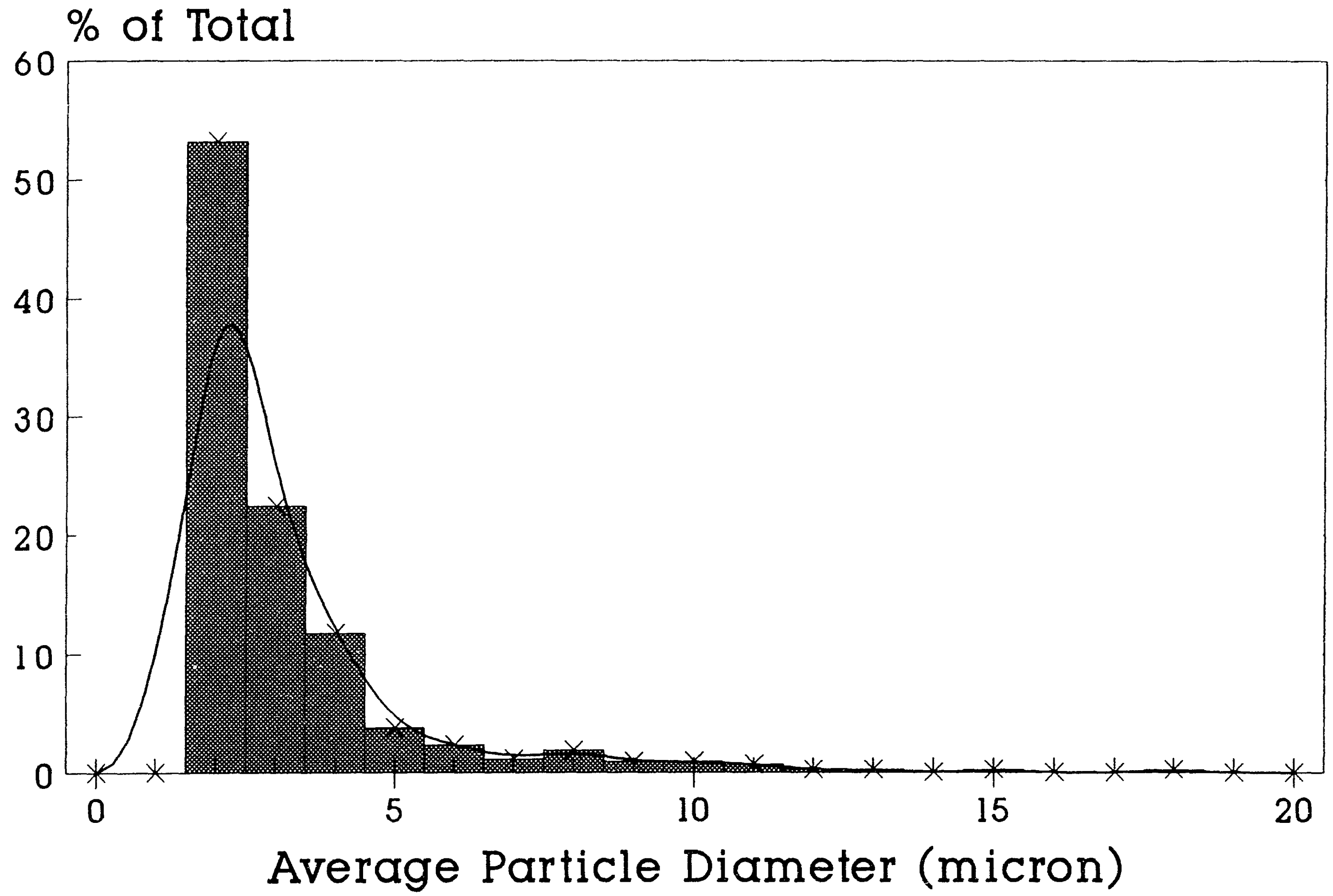
Element	P-58	P-60	P-65
Mg	4.6	7.4	4.2
Al	0.2	1.2	2.0
Si	0.8	3.2	4.6
S	0.0	0.0	0.1
K	0.0	0.0	0.4
Ca	94.4	88.2	88.8



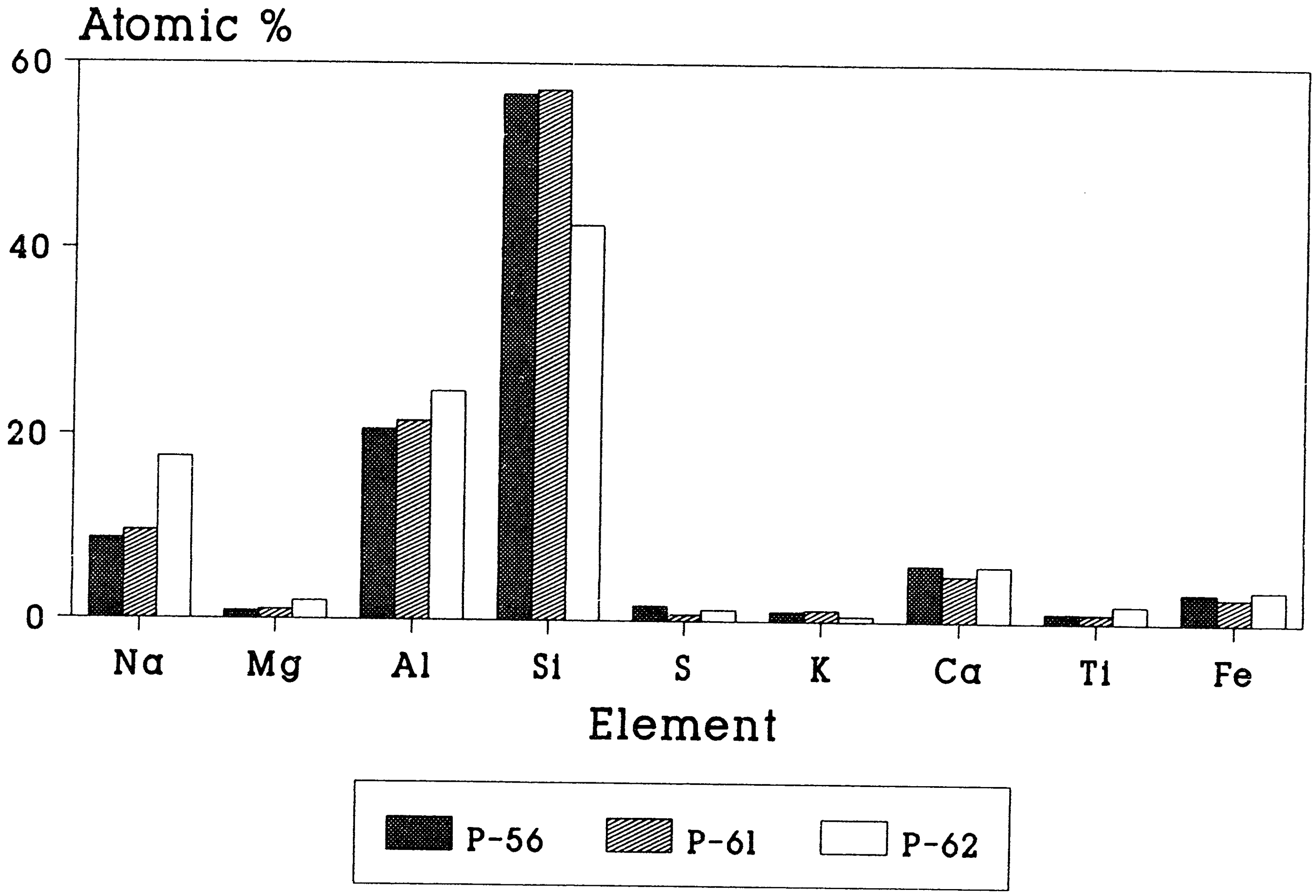
Particle size distribution for P-58 limestone.



Particle size distribution for P-60 limestone.



Particle size distribution for P-65 limestone.

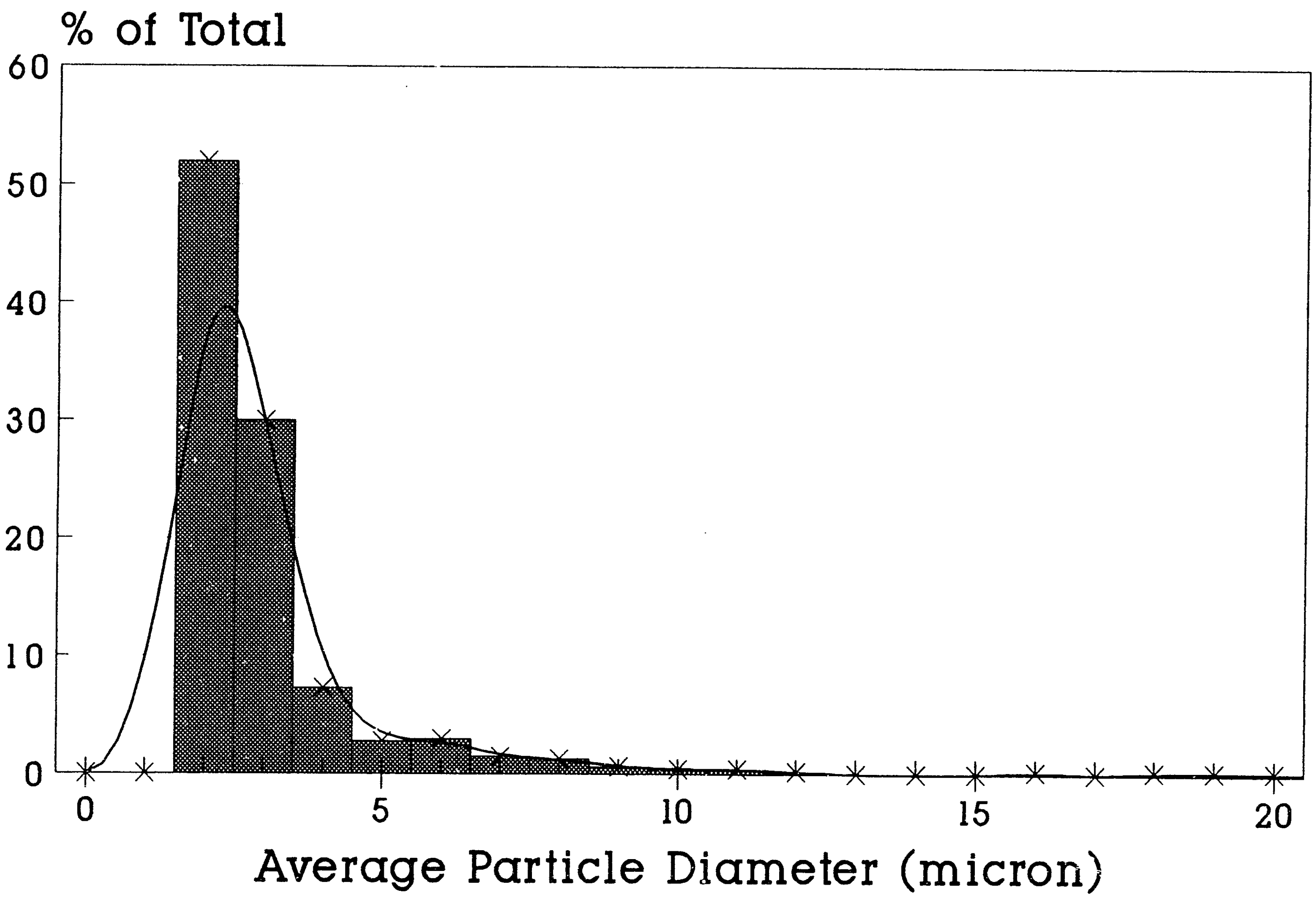


Elemental distributions for fly ash.

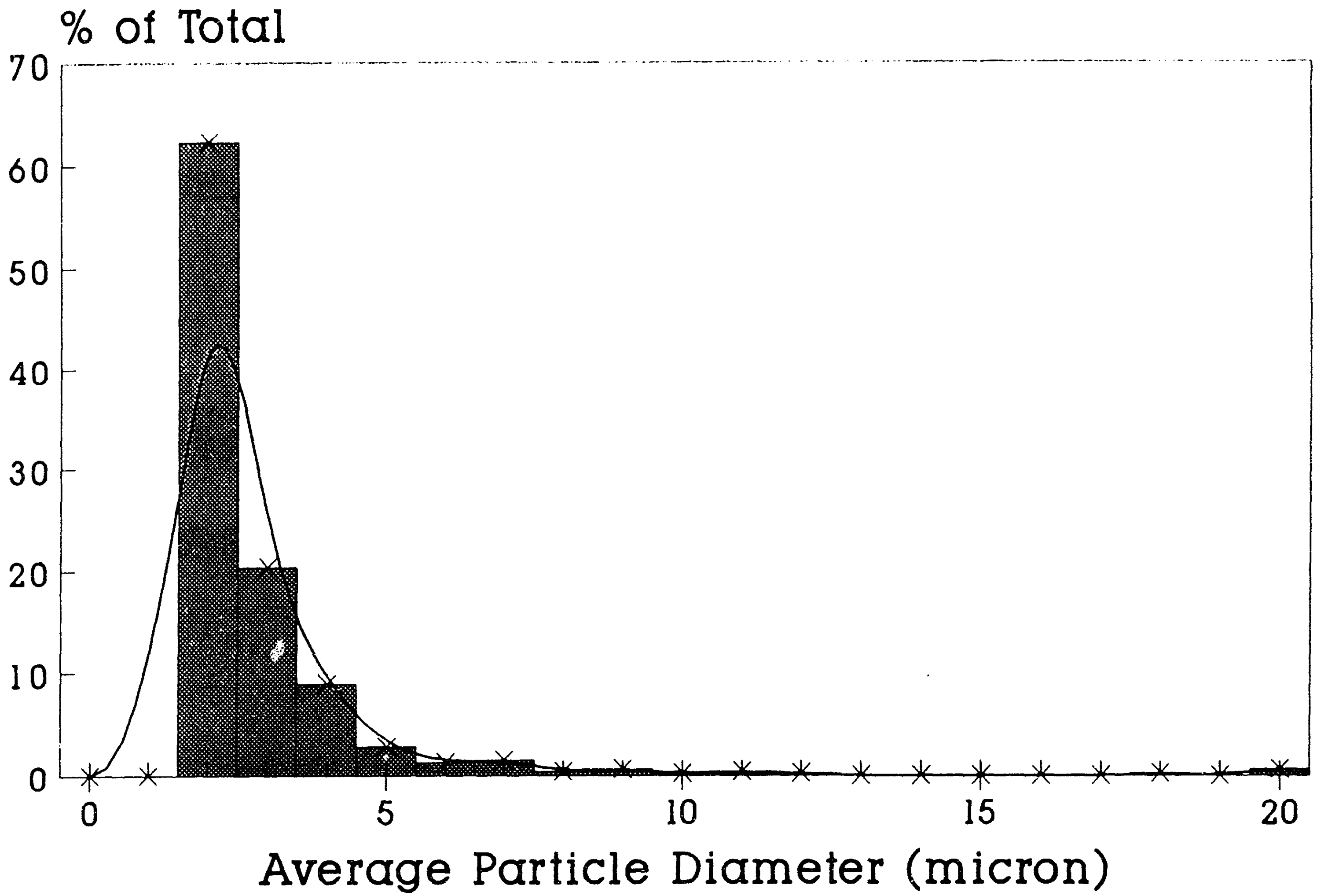
Standardless Semi-quantitative EDS Analysis:

Fly Ash

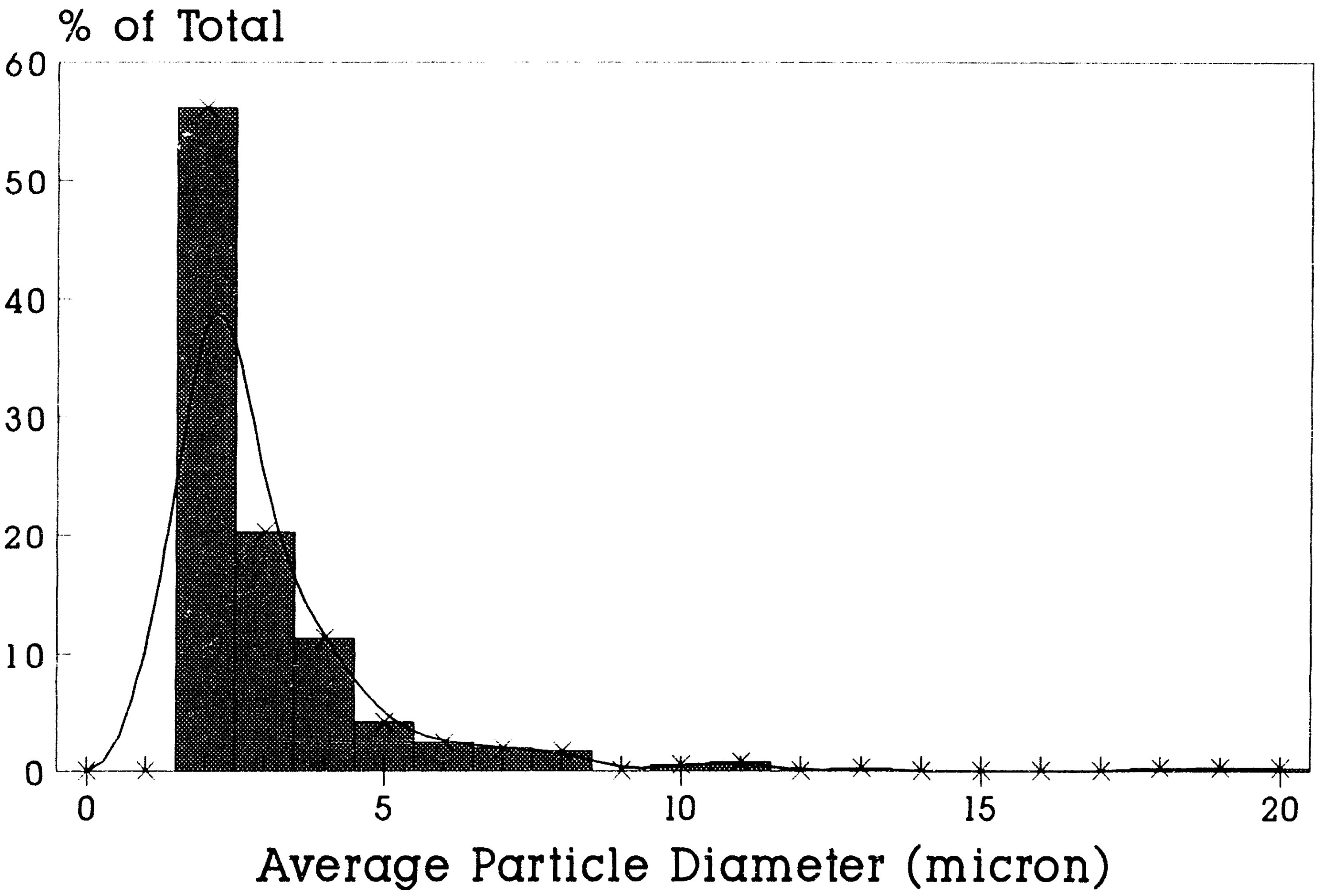
Element	P-56	P-61	P-62
Na	8.8	9.7	17.4
Mg	0.8	1.0	2.0
Al	20.6	21.5	24.7
Si	56.8	57.3	42.6
S	1.6	0.7	1.2
K	1.0	1.1	0.6
Ca	6.1	5.0	6.0
Ti	1.0	0.9	1.8
Fe	3.3	2.7	3.6



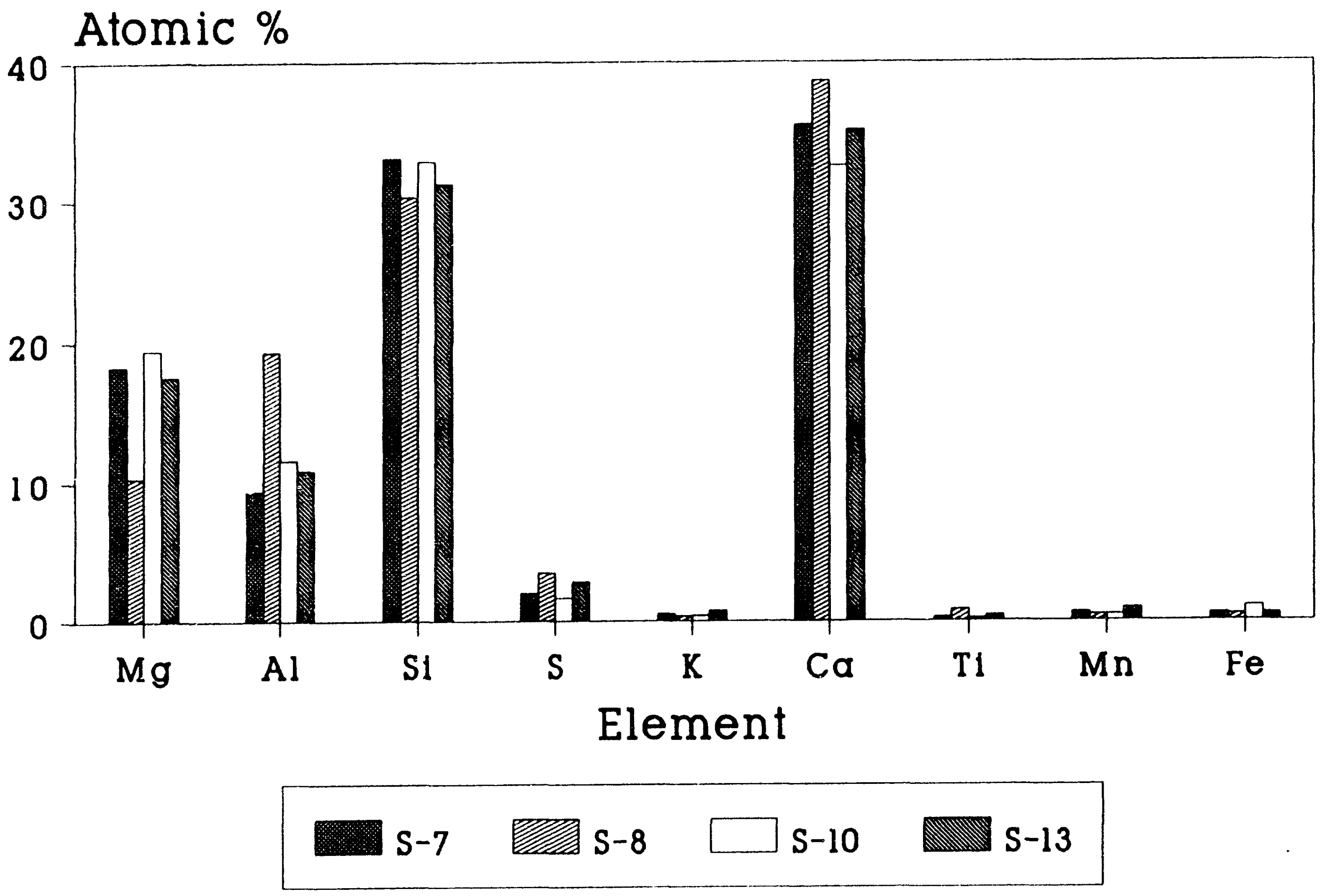
Particle size distribution for P-56 fly ash.



Particle size distribution for P-61 fly ash.



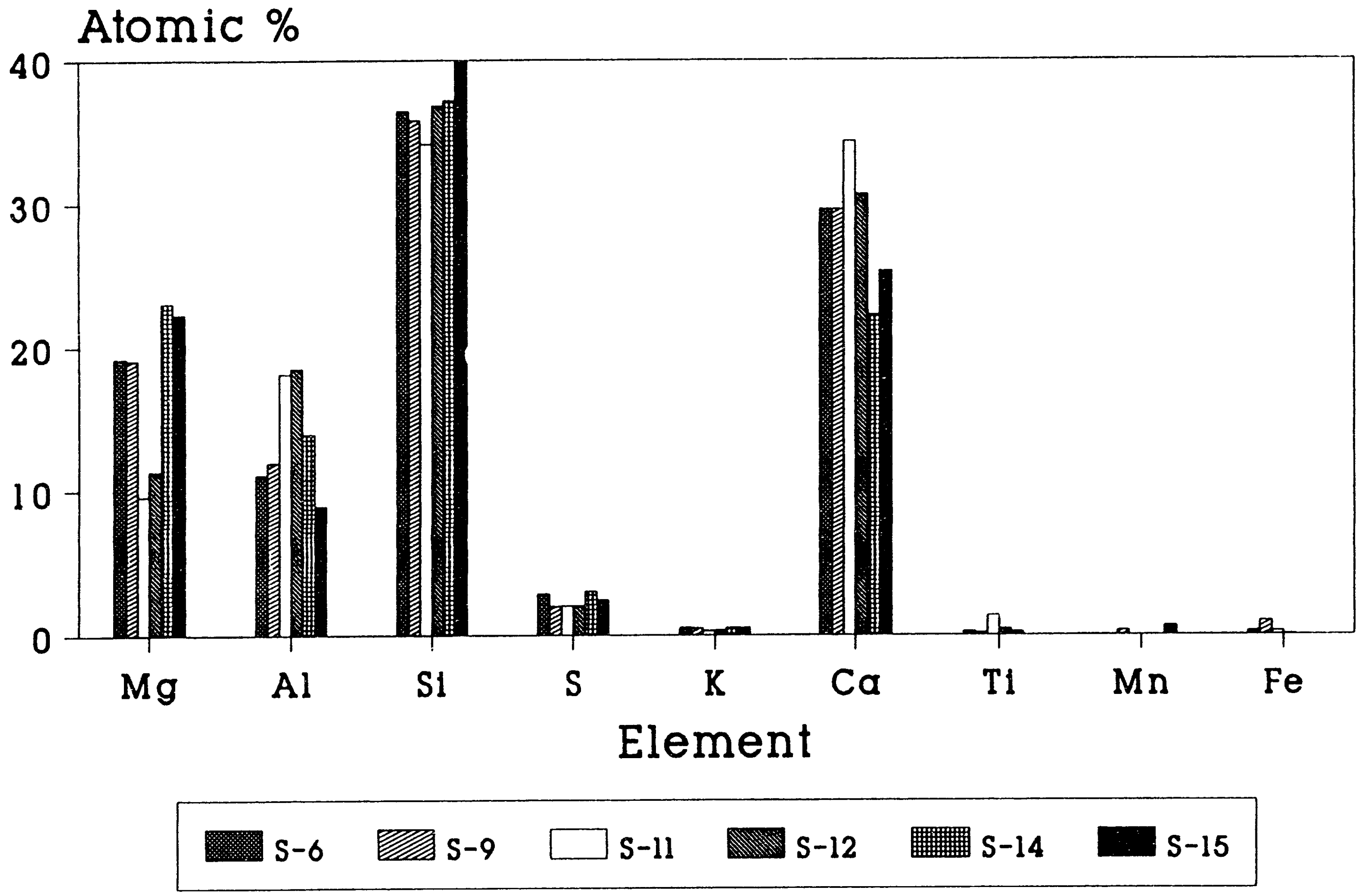
Particle size distribution for P-62 fly ash.



Elemental distributions for series one blast furnace slag.

Standardless Semi-quantitative EDS Analysis:
Blast Furnace Slag (Series One)

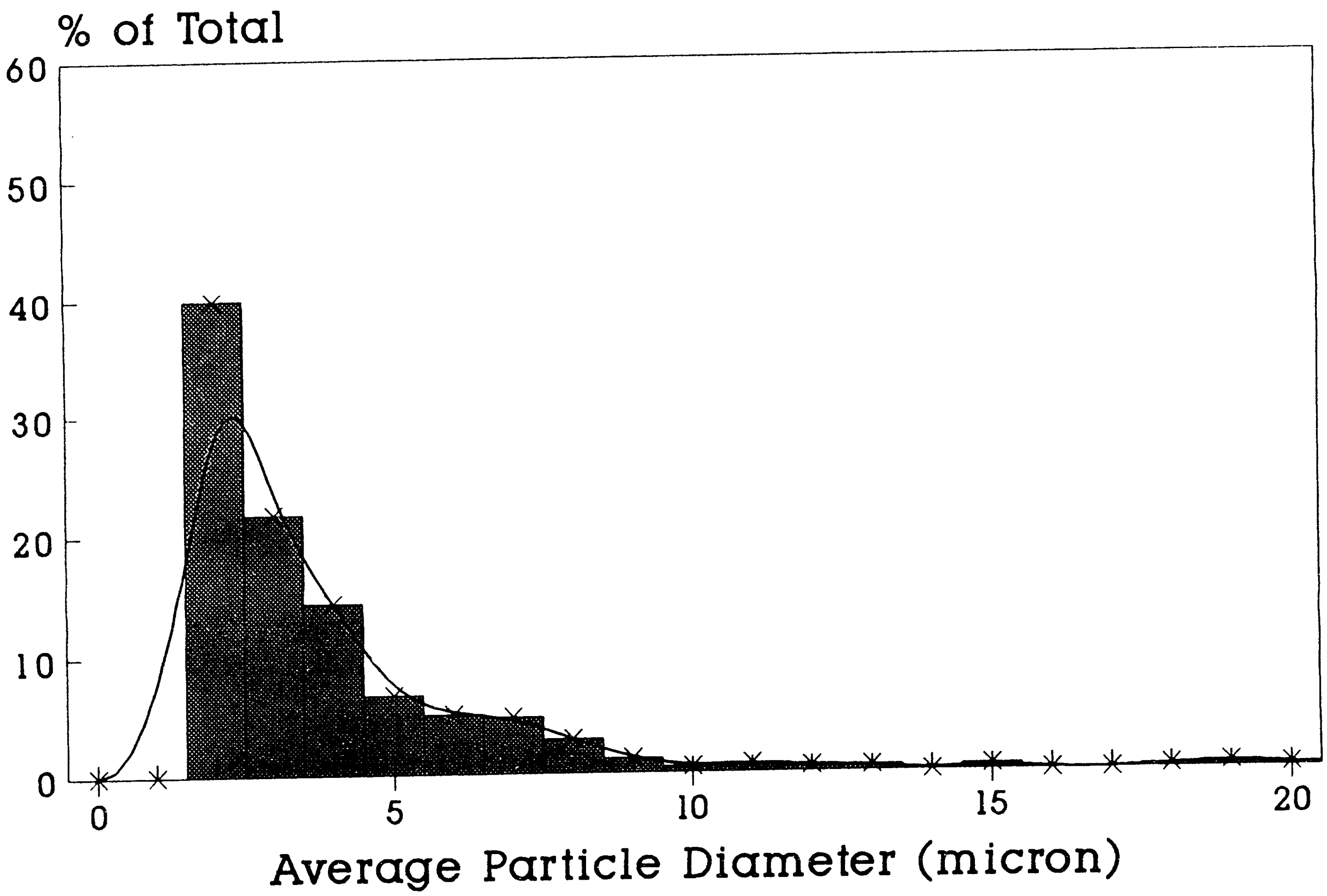
Element	S-7	S-8	S-10	S-13
Mg	18.2	10.3	19.4	22.2
Al	9.4	15.3	11.6	12.4
Si	33.1	30.4	32.9	34.2
S	2.0	3.5	1.6	2.7
K	0.5	0.3	0.4	0.5
Ca	35.5	38.6	32.6	27.0
Ti	0.2	0.8	0.2	0.2
Mn	0.6	0.4	0.4	0.5
Fe	0.5	0.4	1.0	0.3



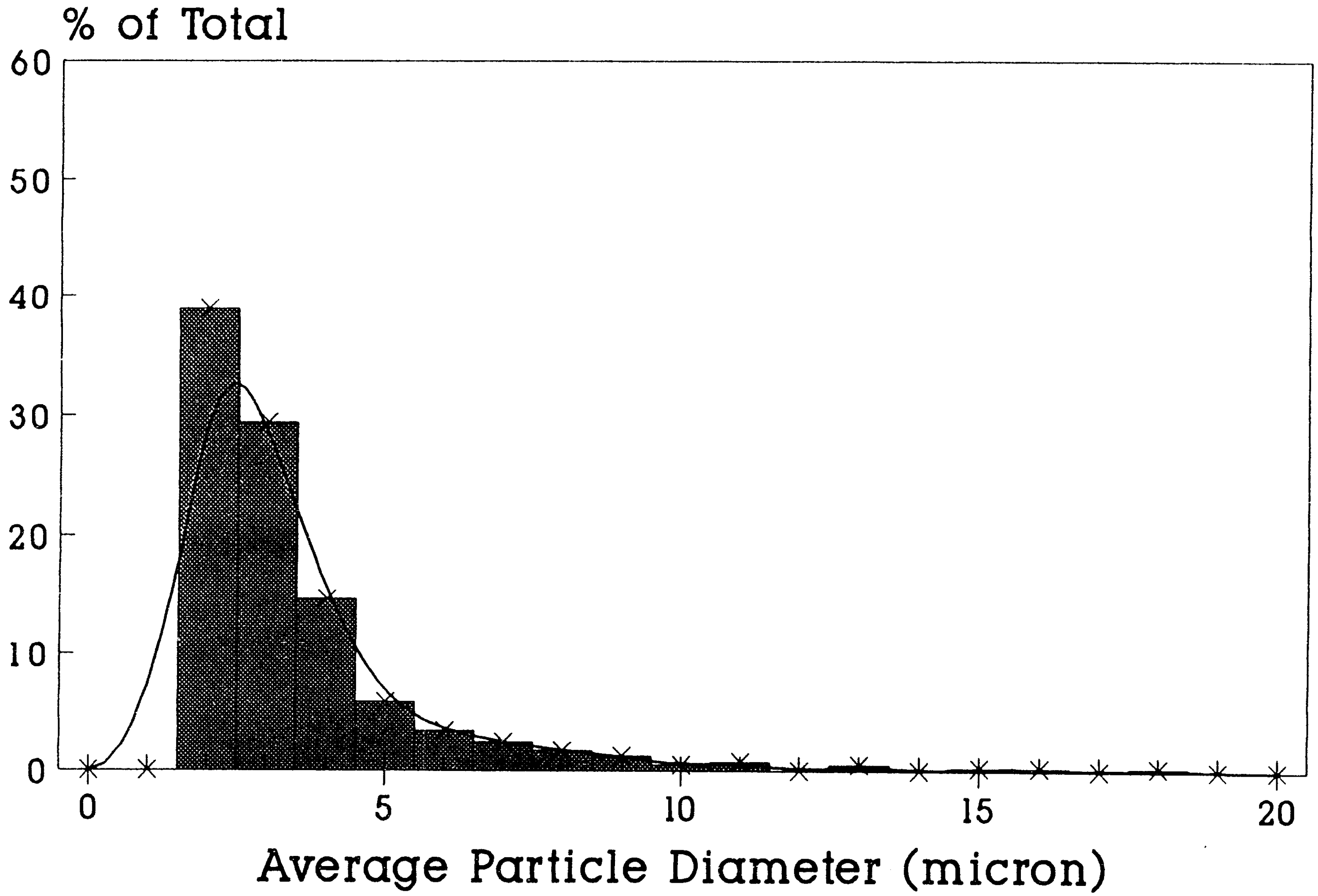
Elemental distributions for nonseries one blast furnace slag.

Standardless Semi-quantitative EDS Analysis:
Blast Furnace Slag (Non-series One)

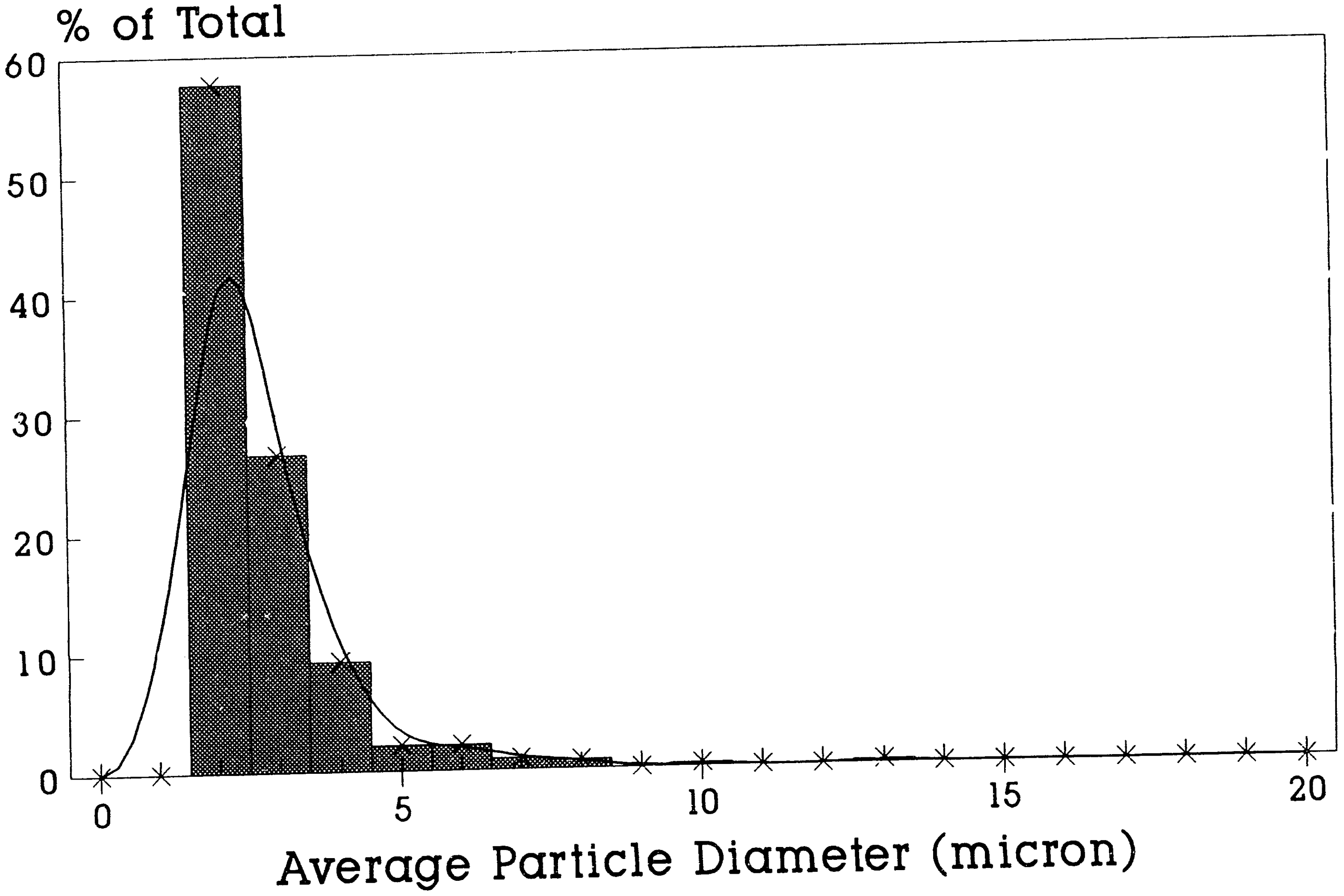
Element	S-6	S-9	S-11	S-12	S-14	S-15
Mg	19.1	19.0	9.6	11.3	23.1	22.3
Al	11.0	11.9	18.1	18.5	13.8	8.9
Si	36.5	35.8	34.2	36.8	37.2	40.0
S	2.8	1.9	2.0	2.0	3.0	2.4
K	0.5	0.4	0.3	0.3	0.5	0.5
Ca	29.6	29.6	34.4	30.6	22.2	25.3
Ti	0.2	0.1	1.3	0.4	0.2	0.0
Mn	0.0	0.3	0.0	0.0	0.0	0.6
Fe	0.2	0.9	0.2	0.1	0.0	0.0



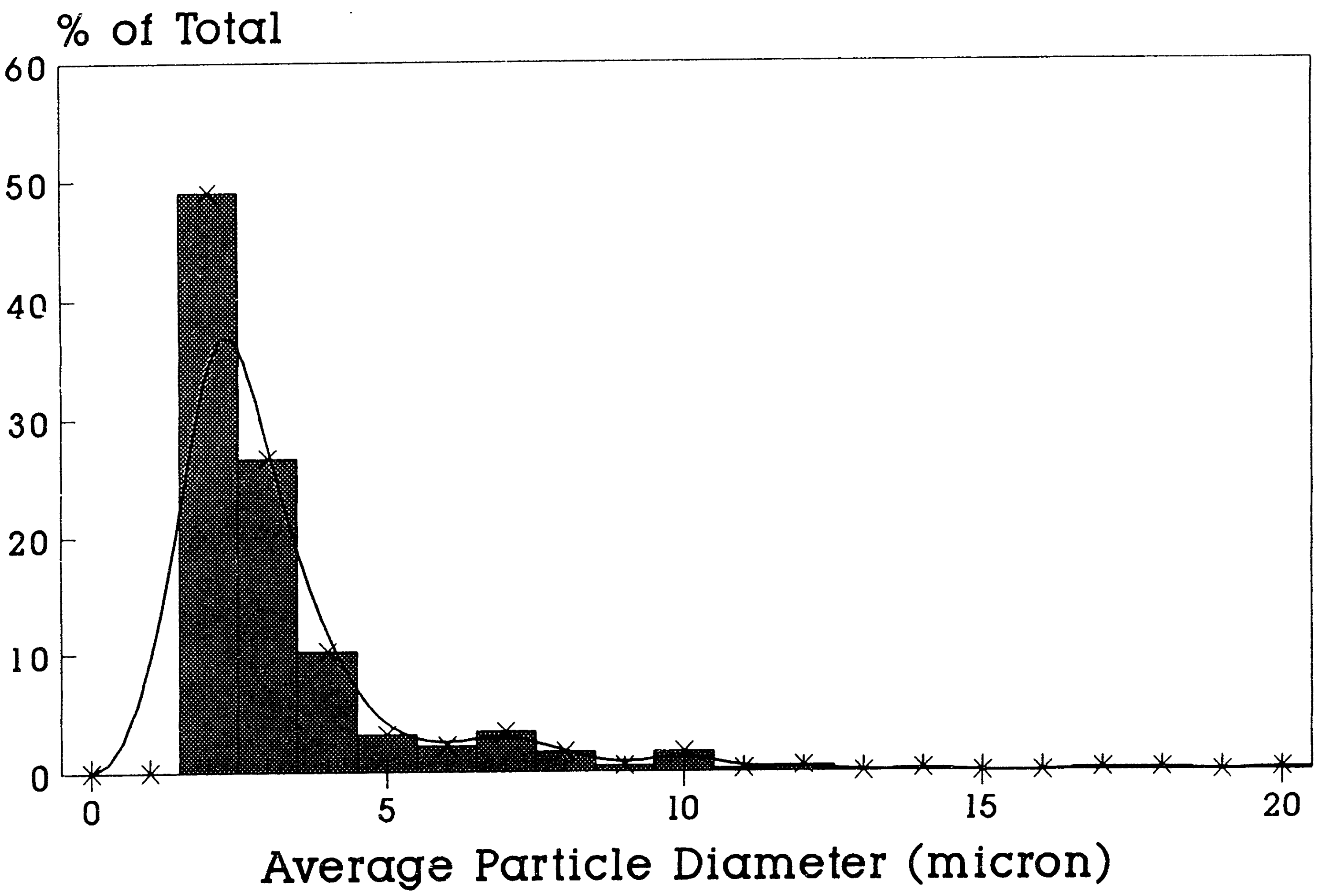
Particle size distribution for S-6 blast furnace slag.



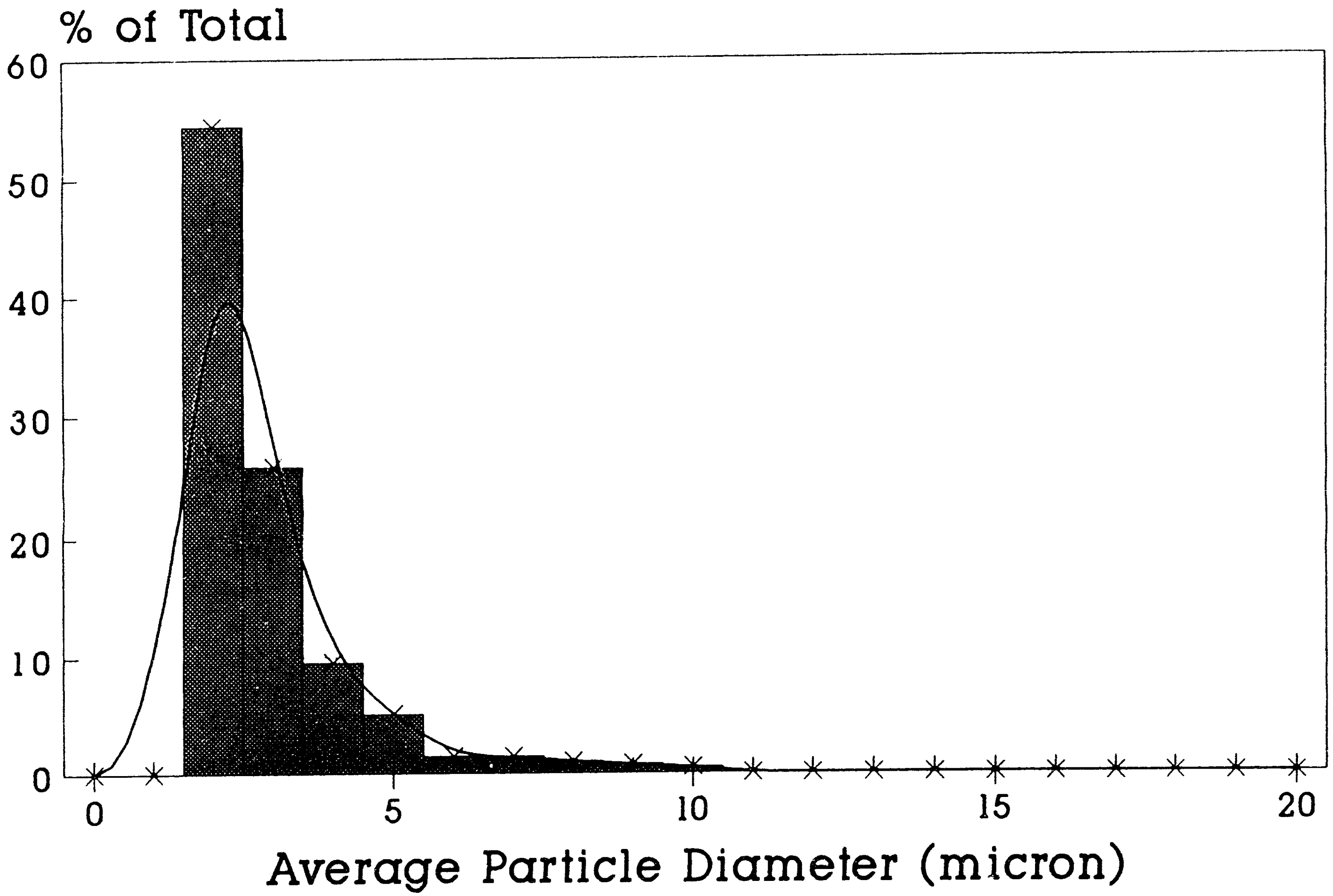
Particle size distribution for S-7 blast furnace slag.



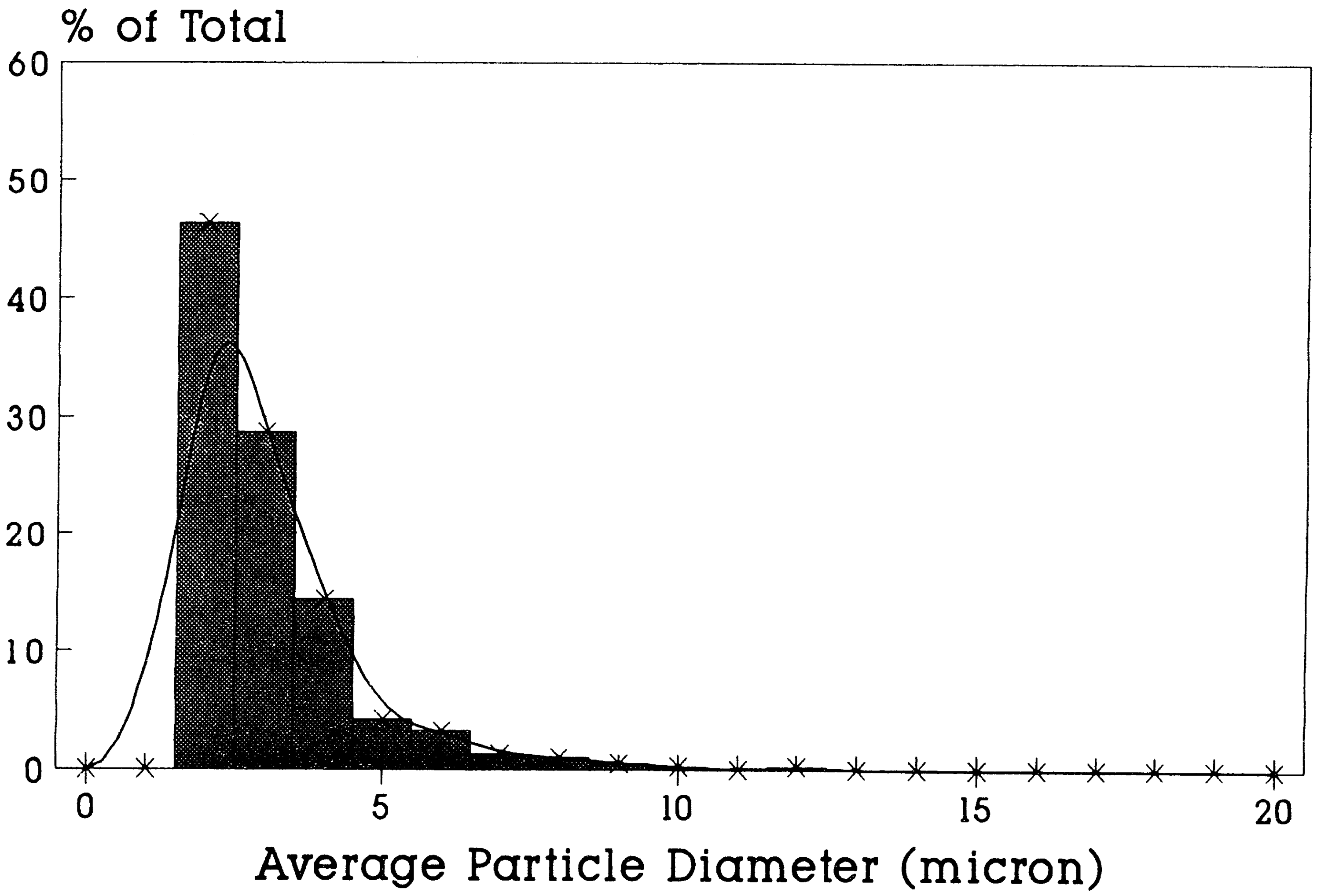
Particle size distribution for S-8 blast furnace slag.



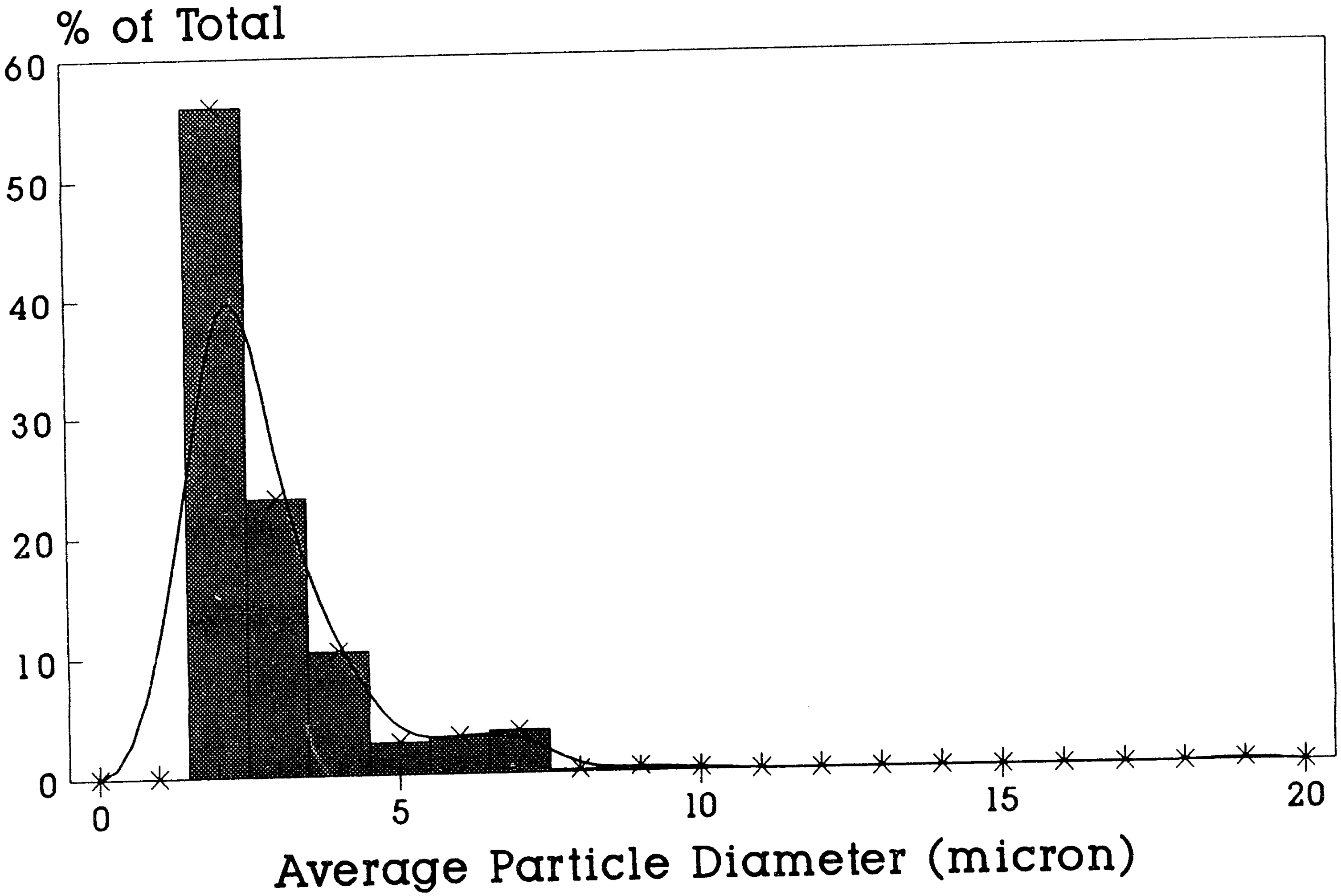
Particle size distribution for S-9 blast furnace slag.



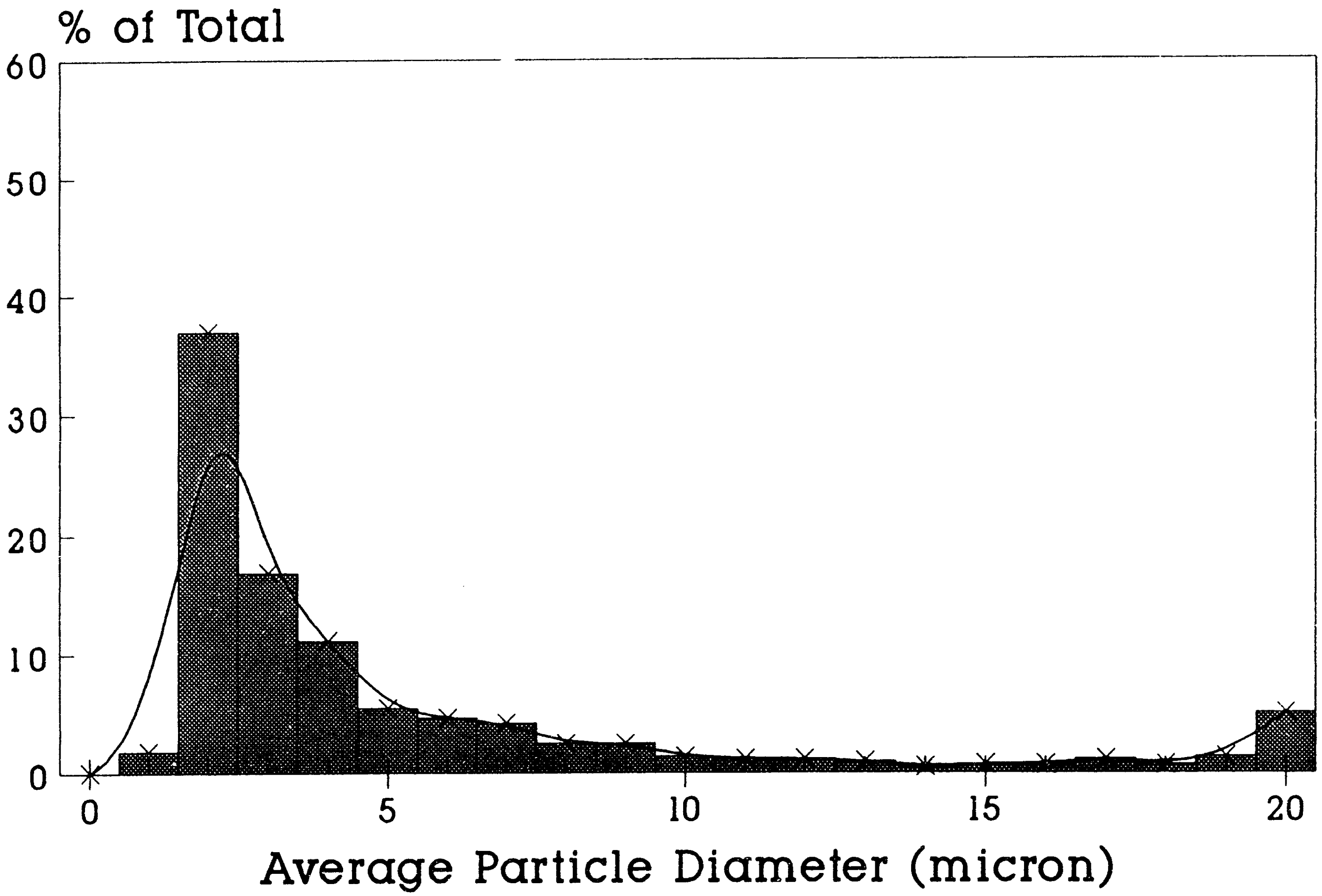
Particle size distribution for S-10 blast furnace slag.



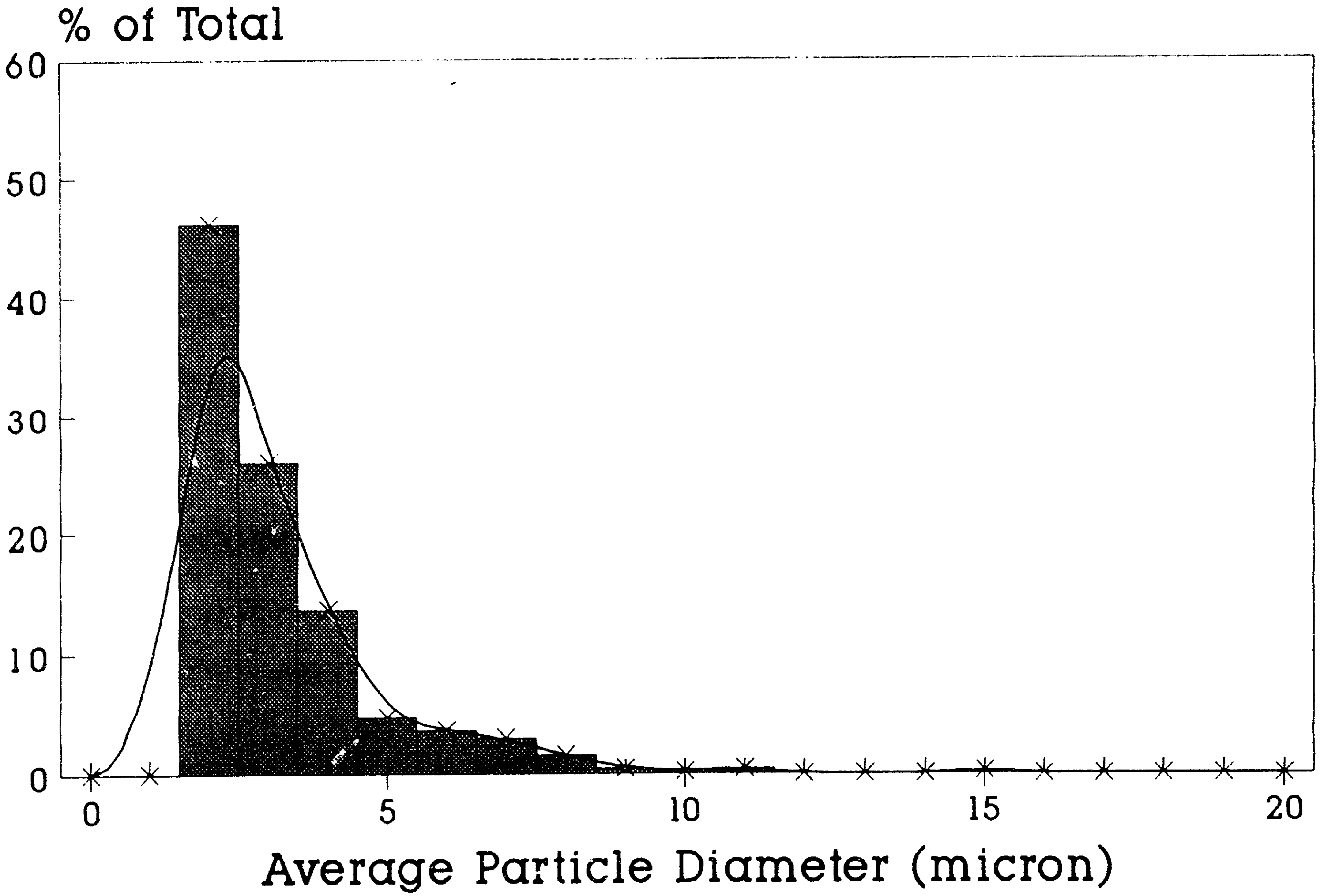
Particle size distribution for S-11 blast furnace slag.



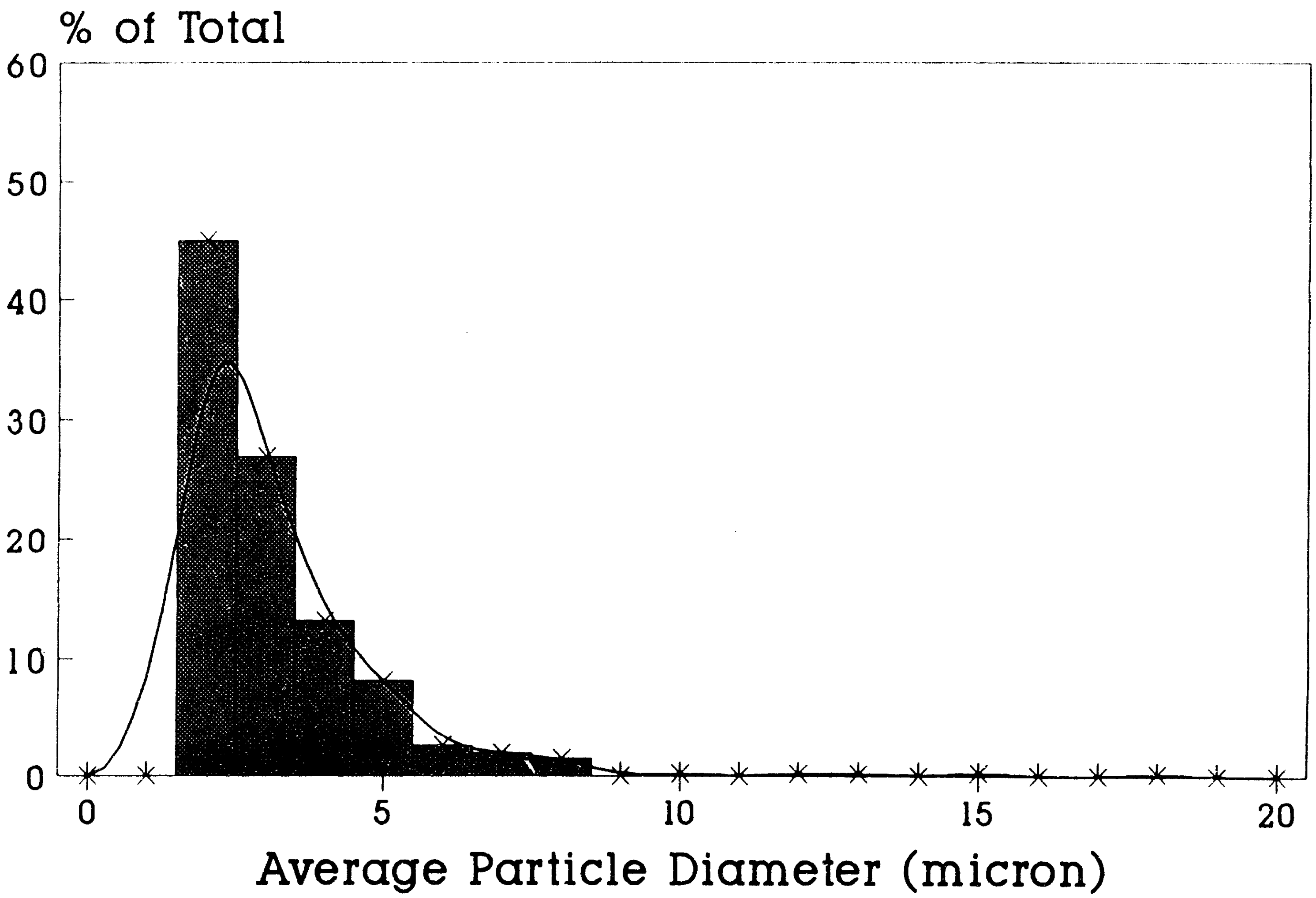
Particle size distribution for S-12 blast furnace slag.



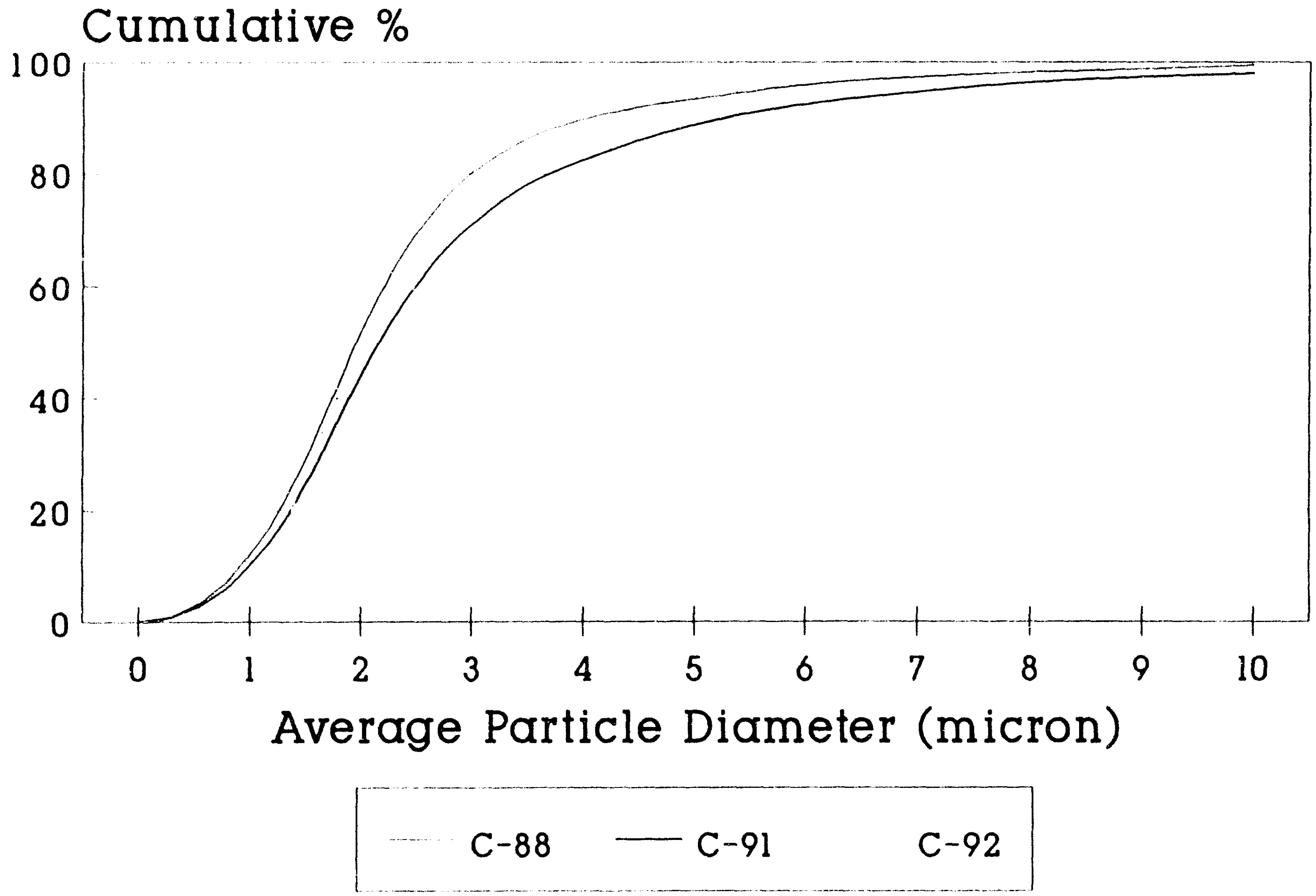
Particle size distribution for S-13 blast furnace slag.



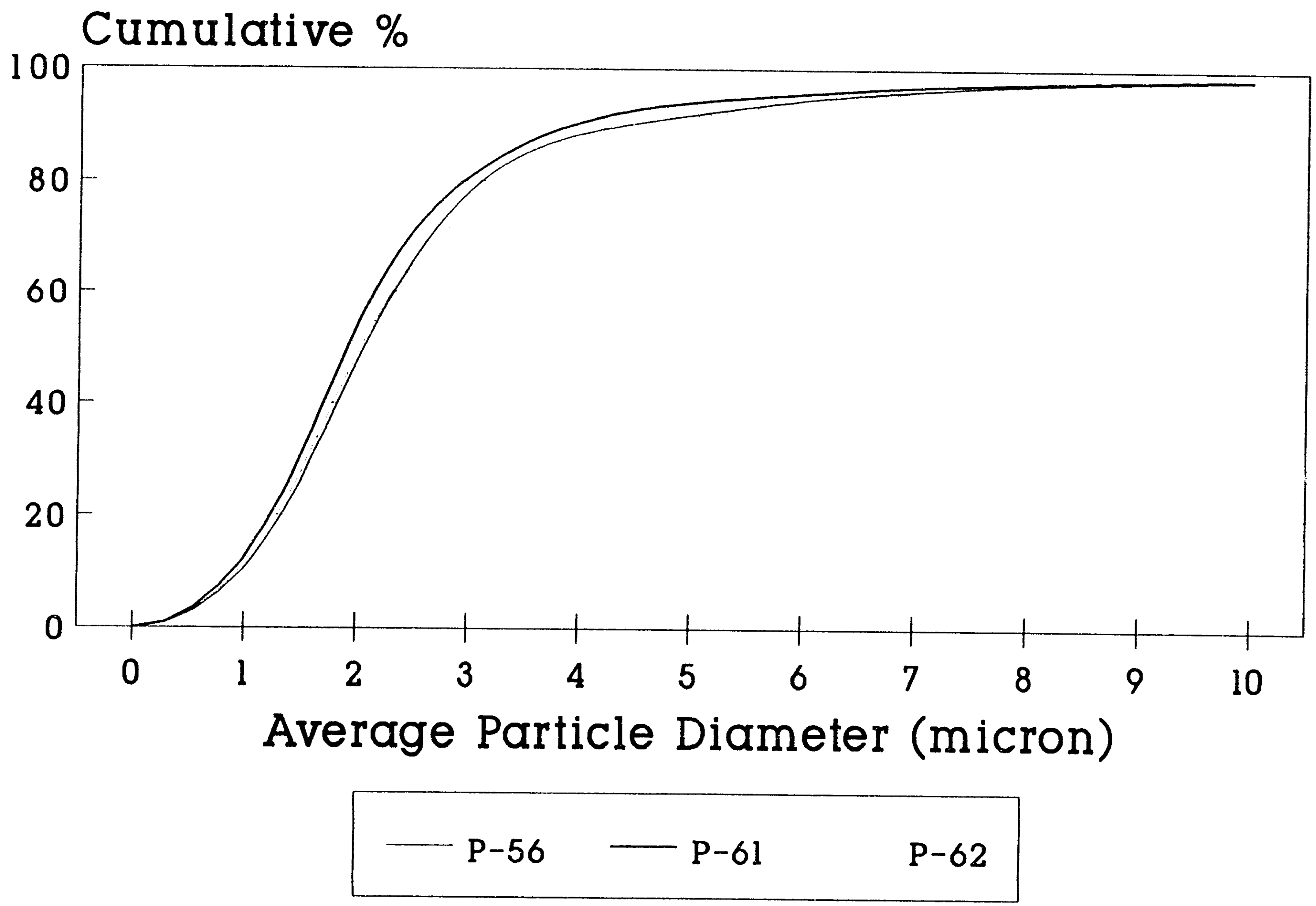
Particle size distribution for S-14 blast furnace slag.



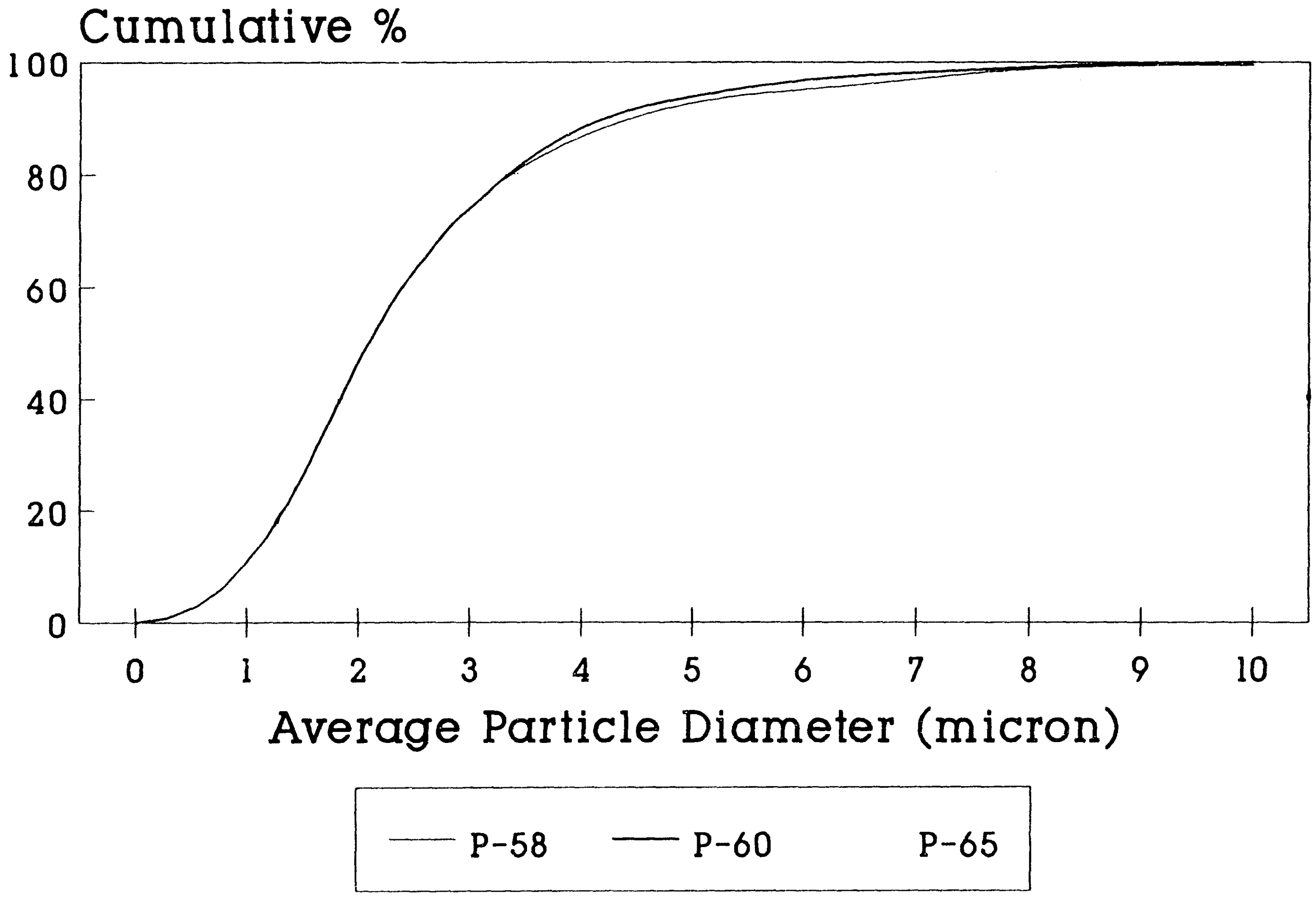
Particle size distribution for S-15 blast furnace slag.



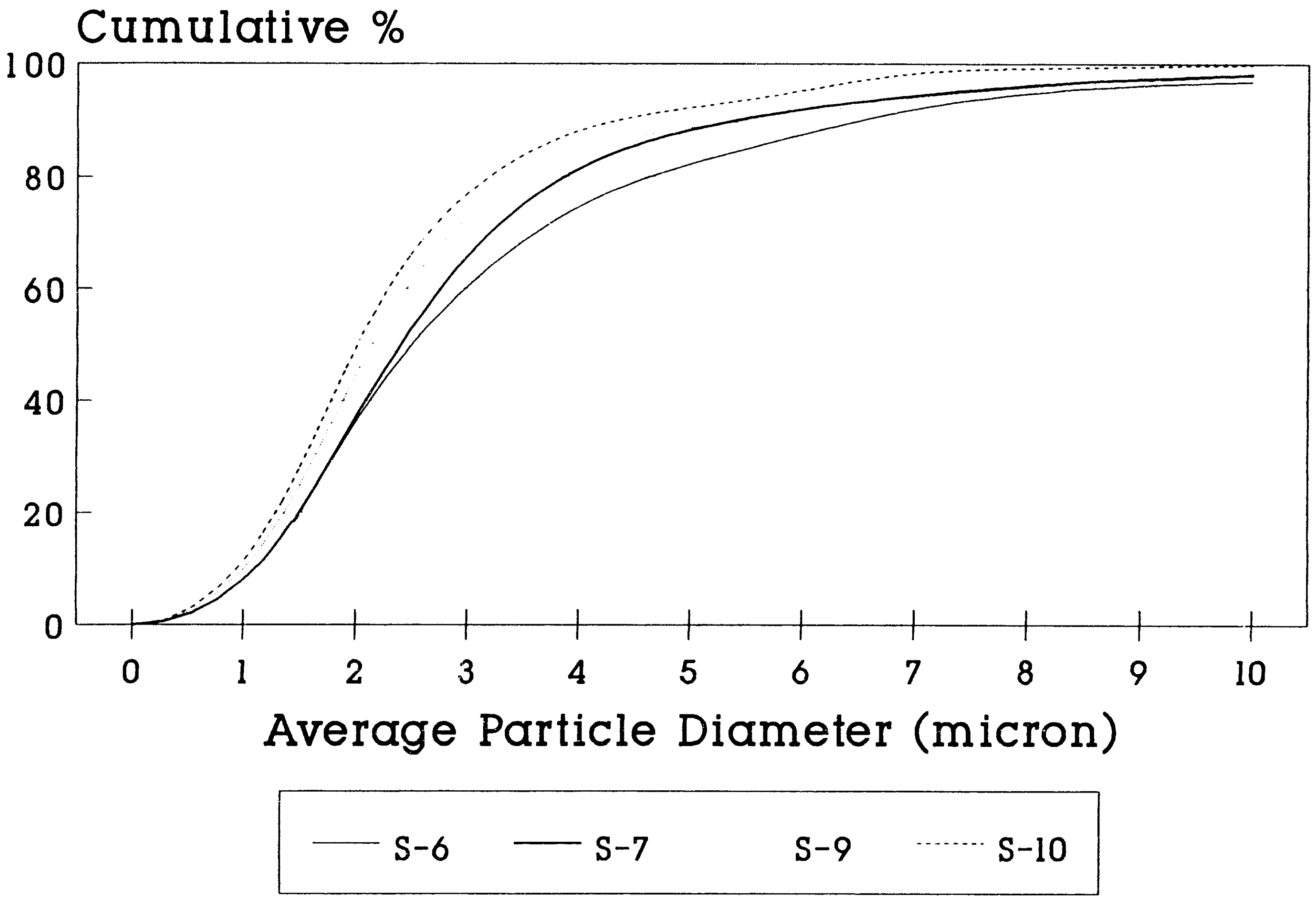
Particle size distribution for Portland cement.



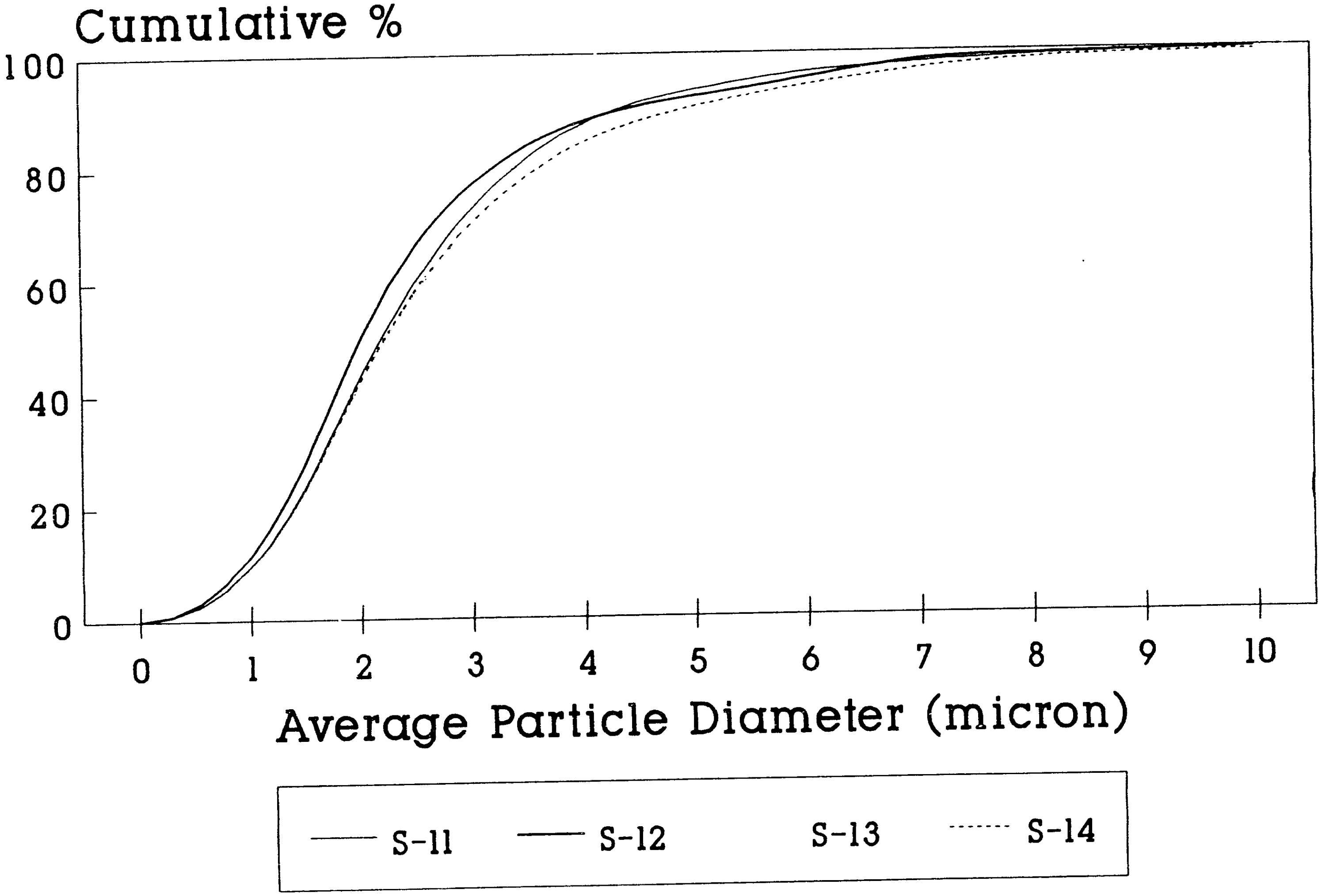
Particle size distribution for fly ash.



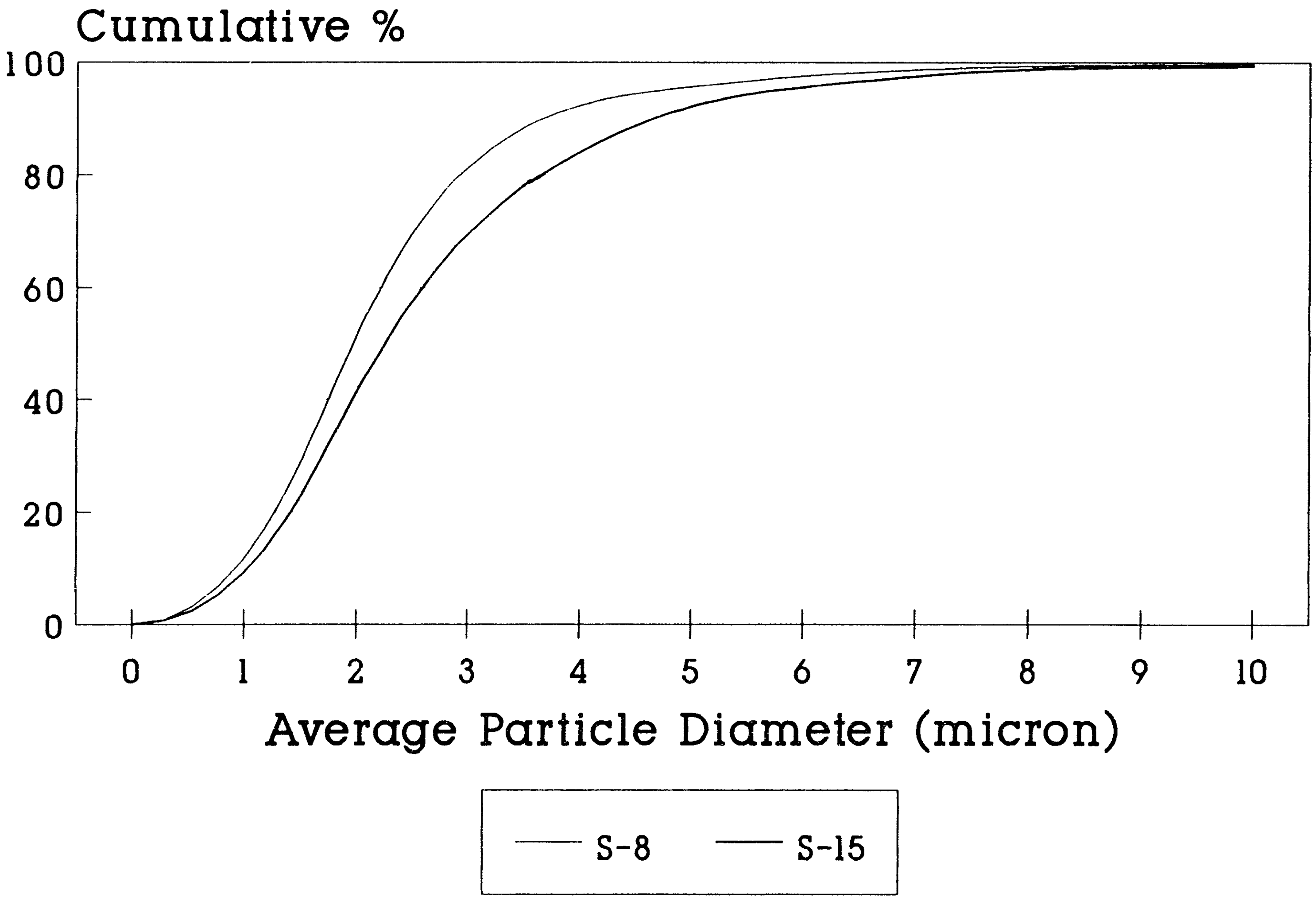
Particle size distribution for limestone.



Particle size distribution for blast furnace slags (S-6, S-7, S-9, S-10).



Particle size distribution for blast furnace slags (S-11, S-12, S-13, S-14).



Particle size distribution for blast furnace slags (S-8, S-15).

Particle-size data for Hanford material

Sample	Number of particles	Aspect ratio	Average area (± range) (microns)
S-6	419	1.59	10.2 (21.3)
S-7	409	1.60	7.9 (16.9)
S-8	413	1.60	3.8 (7.0)
S-9	414	1.56	8.2 (26.6)
S-10	410	1.58	4.6 (7.1)
S-11	404	1.62	4.9 (6.8)
S-12	473	1.66	4.7 (8.8)
S-13	457	1.59	7.5 (17.8)
S-14	446	1.57	6.1 (10.1)
S-15	465	1.61	5.9 (11.3)
P-58	434	1.62	5.1 (7.8)
P-60	420	1.60	4.7 (6.6)
P-65	427	1.59	6.7 (14.9)
C-88	422	1.56	4.6 (7.5)
C-91	458	1.64	7.0 (13.3)
C-92	479	1.57	5.6 (20.1)
P-56	481	1.44	8.0 (43.7)
P-61	462	1.37	9.2 (56.3)
P-62	410	1.31	8.2 (31.3)

EDS Analysis: Individual Components and Combined Matrix

	C-88	C-91	P-58	P-60	P-61	P-62	S-6	S-7	S-8	S-9	S-10	S-11	S-12	S-13	S-14	S-15
Na	0.0	0.0	0.0	0.0	9.7	17.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Mg	2.3	6.3	4.6	7.4	1.0	2.0	19.1	18.2	10.3	19.0	19.4	9.6	11.3	22.2	23.1	22.3
Al	5.0	4.0	0.2	1.2	21.5	24.7	11.0	9.4	15.3	11.9	11.6	18.1	18.5	12.4	13.8	8.9
Si	22.5	18.3	0.8	3.2	57.3	42.6	36.5	33.1	30.4	35.8	32.9	34.2	36.8	34.2	37.2	40.0
S	2.9	3.4	0.0	0.0	0.7	1.2	2.8	2.0	3.5	1.9	1.6	2.0	2.0	2.7	3.0	2.4
K	0.9	1.3	0.0	0.0	1.1	0.6	0.5	0.5	0.3	0.4	0.4	0.3	0.3	0.5	0.5	0.5
Ca	61.8	62.0	94.4	88.2	5.0	6.0	29.6	35.5	38.6	29.6	32.6	34.4	30.6	27.0	22.2	25.3
Ti	0.0	0.0	0.0	0.0	0.9	1.8	0.2	0.2	0.8	0.1	0.2	1.3	0.4	0.2	0.2	0.0
Mn	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.6	0.4	0.3	0.4	0.0	0.0	0.5	0.0	0.6
Fe	4.6	4.8	0.0	0.0	2.7	3.6	0.2	0.5	0.4	0.9	1.0	0.2	0.1	0.3	0.0	0.0

	MX1	MX2	MX3	MX4	MX5	MX6	MX7	MX8	MX9	MX10	MX11	MX12	MX13	AVG	SD
Na	4.9	4.9	4.9	4.9	4.9	4.9	2.7	2.7	2.7	4.9	2.7	4.9	4.9	4.2	1.0
Mg	6.5	9.0	8.9	9.0	8.7	9.8	9.5	8.4	9.8	7.8	8.7	6.7	6.5	8.4	1.2
Al	11.9	10.8	10.9	10.7	10.2	11.1	10.2	9.3	10.6	10.3	10.2	11.8	12.6	10.8	0.8
Si	22.6	23.3	24.1	24.3	23.4	23.7	27.8	27.5	28.6	23.4	27.7	22.5	23.5	24.8	2.1
S	1.4	0.9	1.0	1.2	1.0	1.2	1.1	0.9	1.2	1.2	1.2	1.5	1.0	1.1	0.2
K	0.3	0.3	0.3	0.3	0.3	0.3	0.5	0.5	0.5	0.3	0.5	0.3	0.3	0.4	0.1
Ca	50.2	48.6	47.7	47.7	49.4	47.0	46.7	49.1	45.4	50.2	47.8	50.2	49.1	48.4	1.4
Ti	0.7	0.6	0.5	0.6	0.6	0.6	0.3	0.3	0.3	0.6	0.3	0.7	0.9	0.5	0.2
Mn	0.1	0.1	0.1	0.0	0.2	0.1	0.1	0.2	0.0	0.0	0.0	0.1	0.0	0.1	0.1
Fe	1.3	1.5	1.4	1.2	1.3	1.3	1.0	1.1	0.9	1.2	0.9	1.3	1.3	1.2	0.2

Appendix D

NITRATE LEACHING DATA

Nitrate Leaching Data
Matrix Run No.: MX1-8

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.81	3.84	3.76
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	42	39	42
Cylinder at start of leaching, Ao =	1234	1238	1234

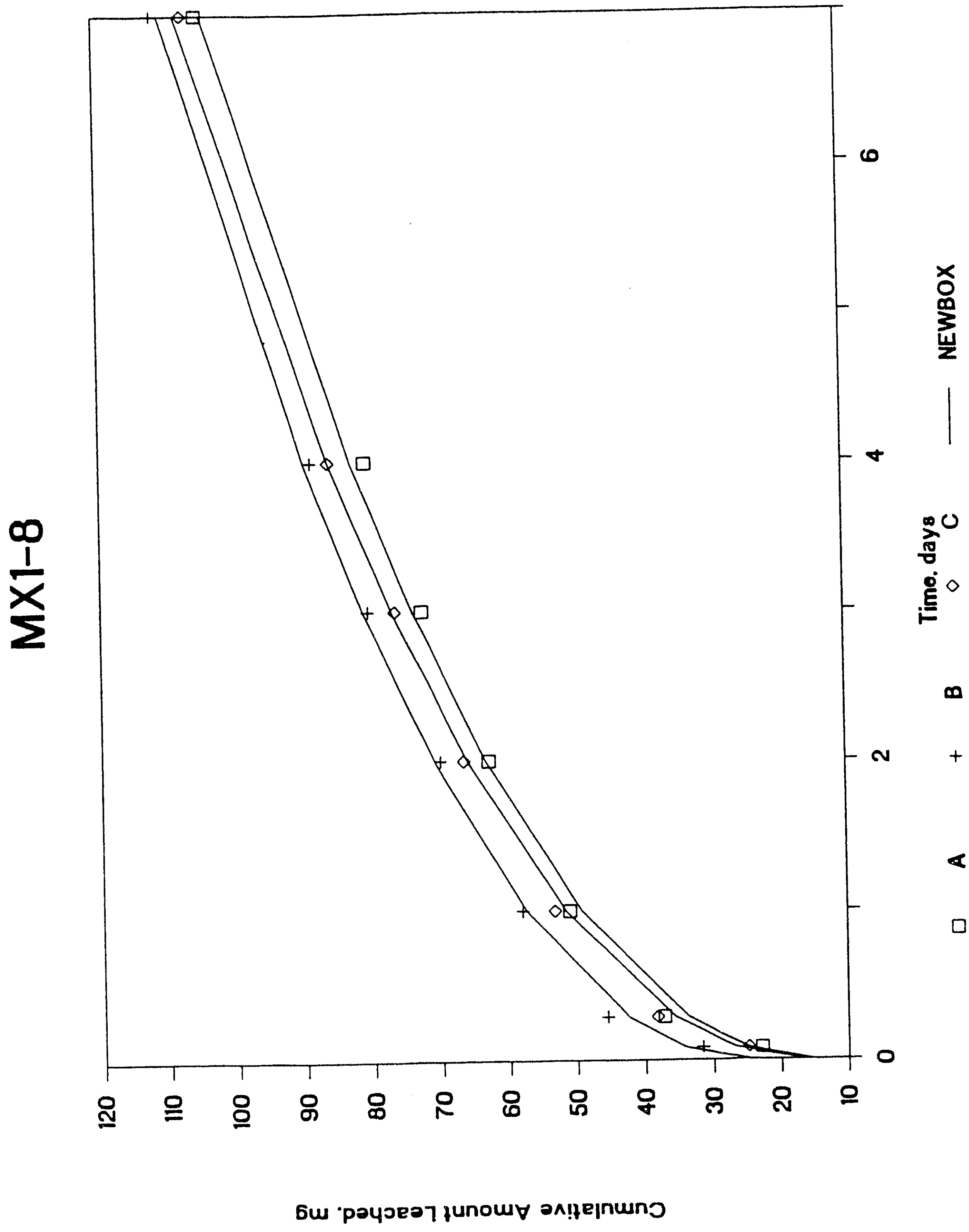
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	23	32	25
2	0.292	37	46	38
3	1	51	58	53
4	2	63	70	66
5	3	72	80	76
6	4	81	89	86
7	7	105	111	107

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	15	25	17
Std. Dev., mg =	3	3	2
Effective Diff. Coeff., sq. cm/s =	1.59E-09	1.50E-09	1.68E-09
Std. Dev., sq. cm/s =	2.44E-10	2.20E-10	1.84E-10
Leachability Index =	8.797	8.823	8.774
Upper 95% Conf. Int. =	0.025	0.024	0.018
Lower 95% Conf. Int. =	0.024	0.023	0.017

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.034	1.031	1.034
Rinse =	0.034	0.031	0.034
Ao =	1.000	1.000	1.000
Washout =	0.012	0.020	0.013
Rinse + Washout =	0.046	0.051	0.048
Leaching by diffusion control =	0.988	0.980	0.987

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.019	0.020	0.026	0.028	0.020	0.022
0.292	0.030	0.027	0.037	0.034	0.031	0.029
1	0.041	0.040	0.047	0.046	0.043	0.042
2	0.051	0.051	0.057	0.057	0.053	0.053
3	0.058	0.060	0.065	0.065	0.062	0.062
4	0.066	0.067	0.072	0.072	0.070	0.070
7	0.085	0.084	0.090	0.089	0.087	0.087


MX1-8
Cumulative Amount Leached, mg
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX2-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	94	89	91
Cylinder at start of leaching, Ao =	1183	1188	1186

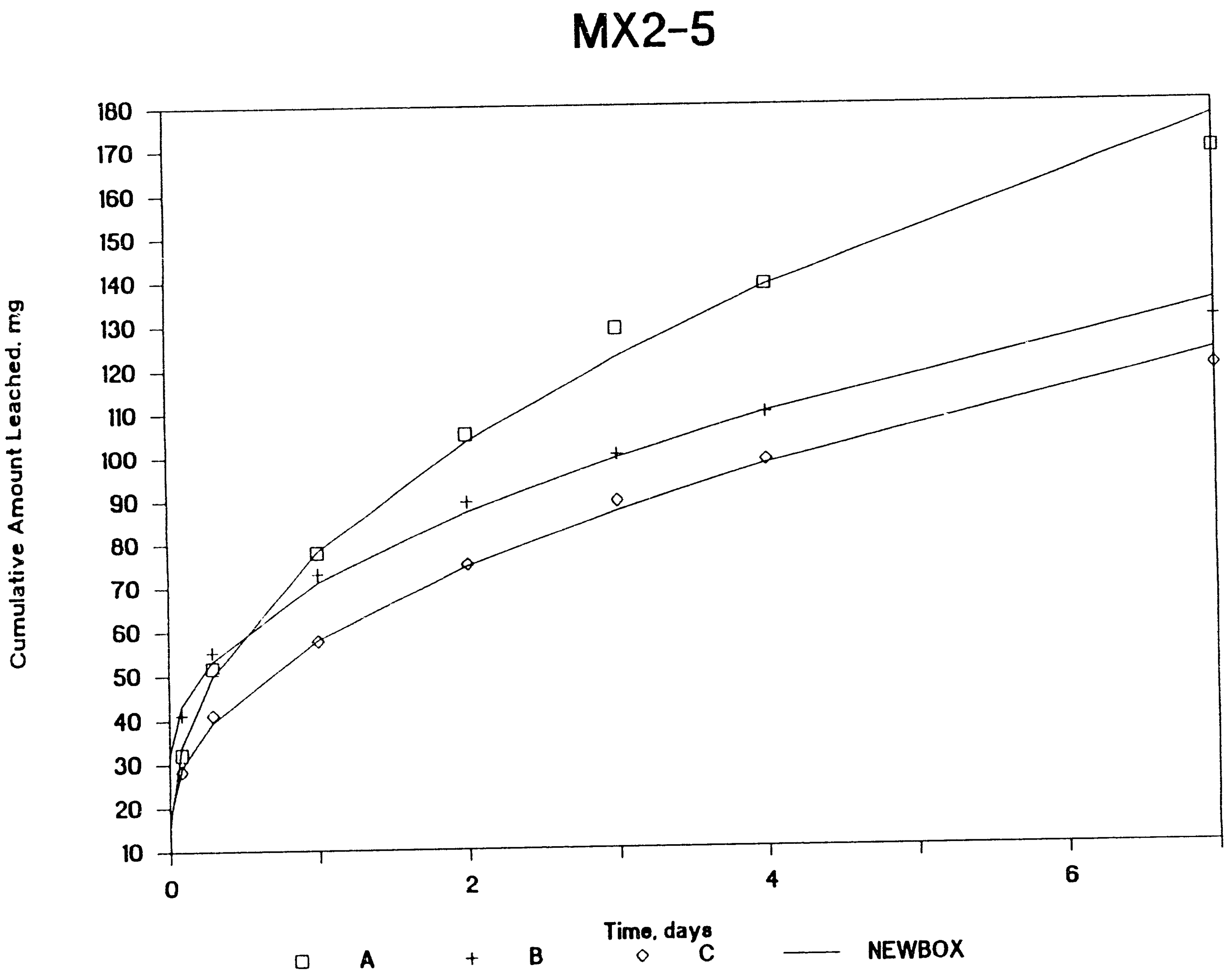
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	32	41	28
2	0.292	52	55	41
3	1	78	73	58
4	2	105	89	75
5	3	129	100	89
6	4	139	110	99
7	7	169	131	120

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	16	32	17
Std. Dev., mg =	5	3	2
Effective Diff. Coeff., sq. cm/s =	6.31E-09	2.52E-09	2.65E-09
Std. Dev., sq. cm/s =	7.88E-10	2.95E-10	2.26E-10
Leachability Index =	8.200	8.599	8.578
Upper 90% Conf. Int. =	0.020	0.019	0.014
Lower 90% Conf. Int. =	0.019	0.018	0.013

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.079	1.075	1.077
Rinse =	0.079	0.075	0.077
Ao =	1.000	1.000	1.000
Washout =	0.013	0.027	0.015
Rinse + Washout =	0.093	0.102	0.091
Leaching by diffusion control =	0.987	0.973	0.985

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.027	0.029	0.035	0.036	0.024	0.025
0.292	0.044	0.042	0.046	0.045	0.035	0.033
1	0.066	0.066	0.061	0.060	0.049	0.049
2	0.089	0.087	0.075	0.073	0.063	0.063
3	0.109	0.104	0.084	0.084	0.075	0.074
4	0.117	0.117	0.093	0.092	0.083	0.083
7	0.143	0.149	0.110	0.113	0.101	0.104


MX2-5
Cumulative Amount Leached, mg
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX3-2

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.70	3.84	3.75
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	47	94	57
Cylinder at start of leaching, Ao =	1230	1183	1220

Cumulative Amount Leached, mg nitrate

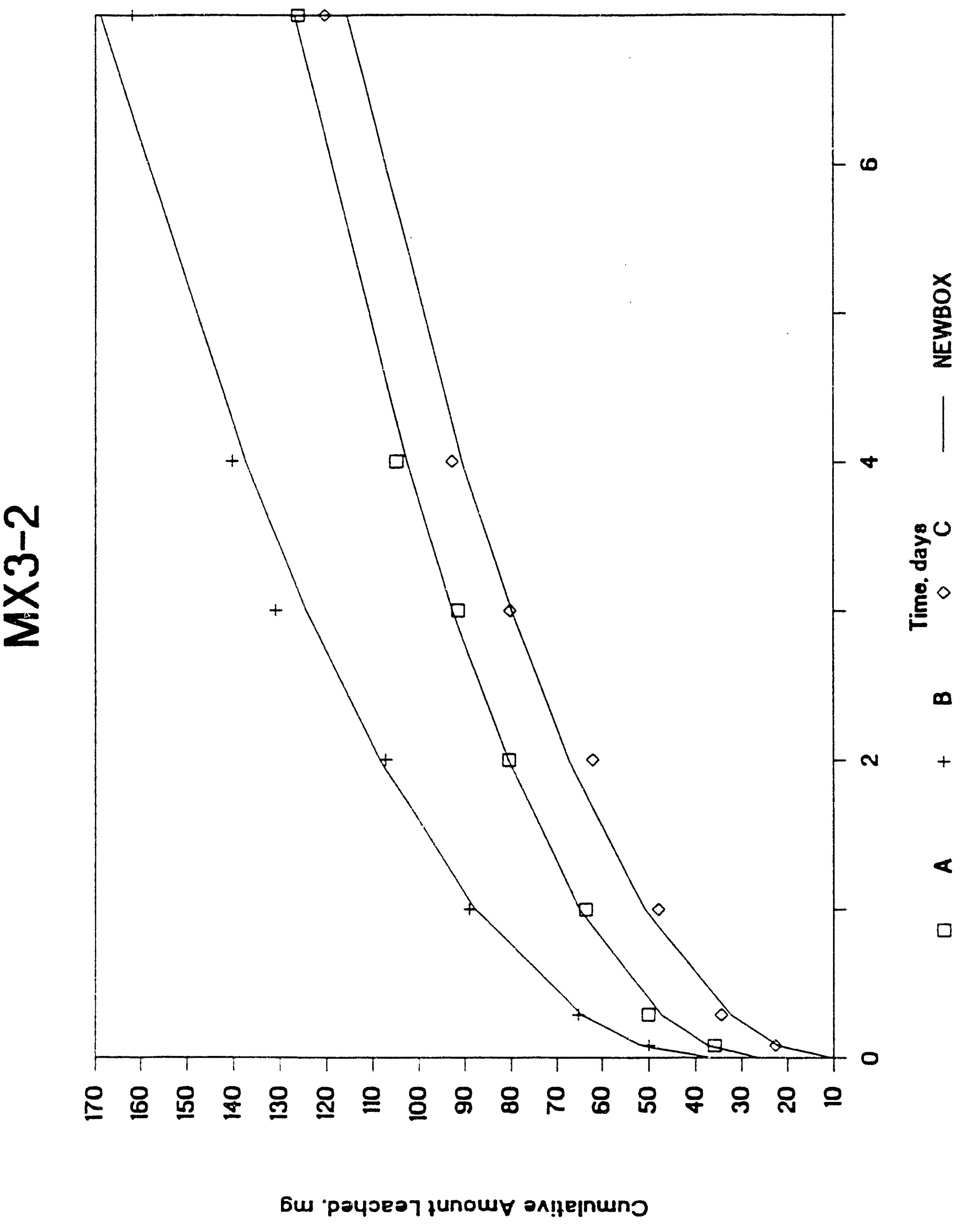
Interval	Time days	A	B	C
1	0.083	36	50	23
2	0.292	50	65	34
3	1	64	89	48
4	2	81	107	62
5	3	91	131	80
6	4	105	140	93
7	7	126	162	120

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	26	37	10
Std. Dev., mg =	3	5	4
Effective Diff. Coeff., sq. cm/s =	2.09E-09	4.12E-09	2.28E-09
Std. Dev., sq. cm/s =	2.27E-10	6.20E-10	3.76E-10
Leachability Index =	8.680	8.385	8.642
Upper 95% Conf. Int. =	0.018	0.025	0.027
Lower 95% Conf. Int. =	0.017	0.023	0.026

Cumulative Fraction Leached Based on Ao (CFL)

Initially in fresh grout =	1.038	1.079	1.046
Rinse =	0.038	0.079	0.046
Ao =	1.000	1.000	1.000
Washout =	0.021	0.031	0.009
Rinse + Washout =	0.059	0.110	0.055
Leaching by diffusion control =	0.979	0.969	0.991

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.029	0.030	0.042	0.044	0.019	0.018
0.292	0.041	0.038	0.055	0.055	0.028	0.027
1	0.052	0.053	0.075	0.074	0.039	0.042
2	0.066	0.066	0.090	0.092	0.051	0.055
3	0.074	0.075	0.111	0.105	0.066	0.066
4	0.085	0.084	0.118	0.116	0.076	0.074
7	0.102	0.103	0.137	0.143	0.098	0.095


MX3-2
Cumulative Amount Leached, mg
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX4-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.69	3.61	3.63
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	46	62	65
Cylinder at start of leaching, Ao =	1230	1214	1212

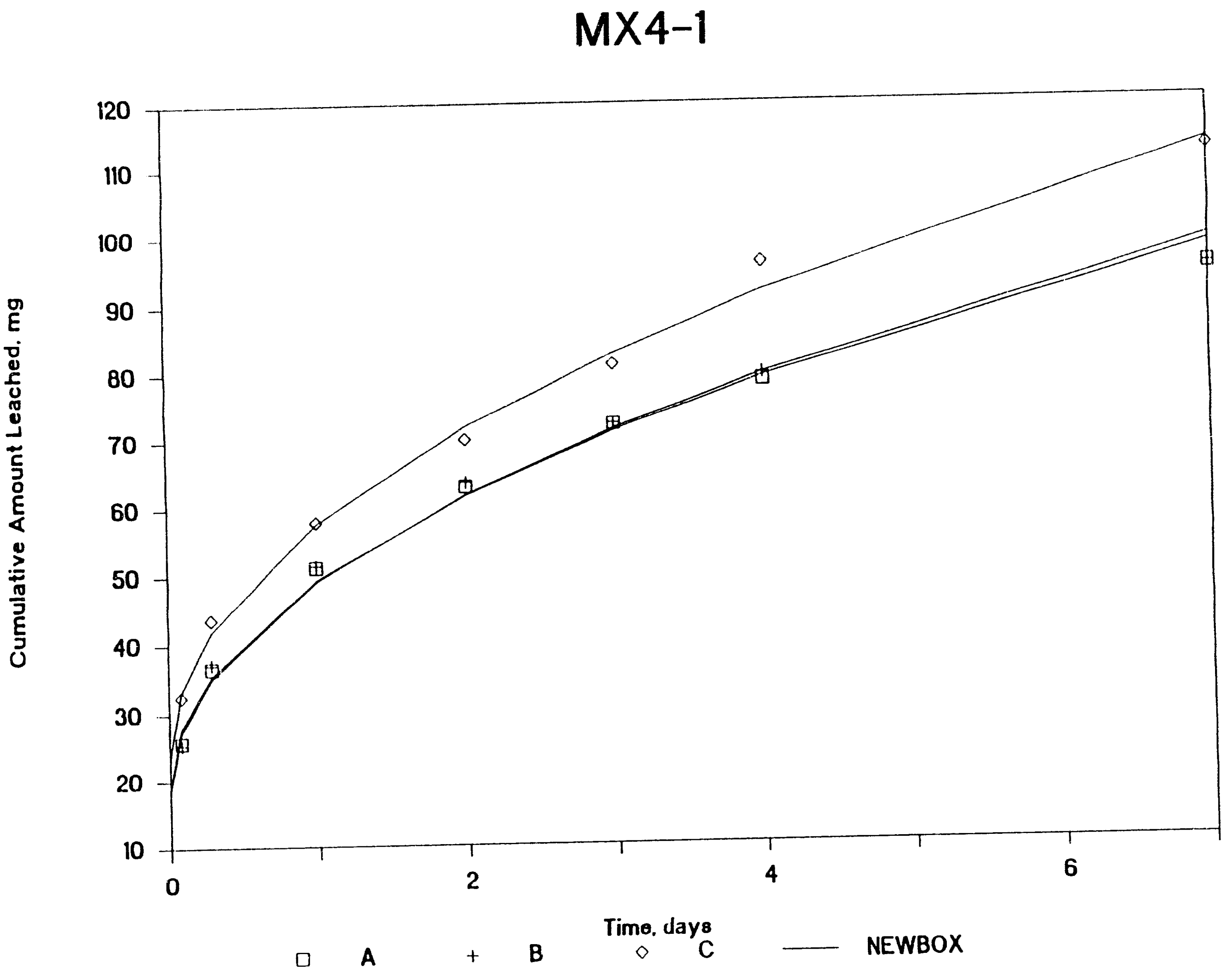
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	26	25	32
2	0.292	37	37	44
3	1	51	51	58
4	2	63	64	70
5	3	72	72	81
6	4	79	80	96
7	7	95	95	112

NEWBOY Estimate of Parameters	A	B	C
Washoff, mg =	19	18	23
Std. Dev., mg =	3	3	3
Effective Diff. Coeff., sq. cm/s =	1.25E-09	1.32E-09	1.70E-09
Std. Dev., sq. cm/s =	1.67E-10	2.18E-10	2.30E-10
Leachability Index =	8.904	8.878	8.770
Upper 95% Conf. Int. =	0.022	0.027	0.022
Lower 95% Conf. Int. =	0.021	0.026	0.021

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.038	1.051	1.054
Rinse =	0.038	0.051	0.054
Ao =	1.000	1.000	1.000
Washout =	0.016	0.015	0.019
Rinse + Washout =	0.053	0.067	0.073
Leaching by diffusion control =	0.984	0.985	0.981

Time days	CFL of A		CFL of B		CFL of C	
	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.021	0.023	0.021	0.022	0.026	0.027
0.292	0.030	0.029	0.030	0.029	0.036	0.035
1	0.041	0.040	0.042	0.041	0.048	0.048
2	0.051	0.050	0.053	0.051	0.058	0.059
3	0.059	0.058	0.059	0.059	0.067	0.068
4	0.064	0.064	0.066	0.066	0.079	0.076
7	0.077	0.080	0.078	0.081	0.092	0.094


MX4-1
Cumulative Amount Leached, mg
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX5-8

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	134	108	88
Cylinder at start of leaching, Ao =	1142	1169	1189

Cumulative Amount Leached, mg nitrate

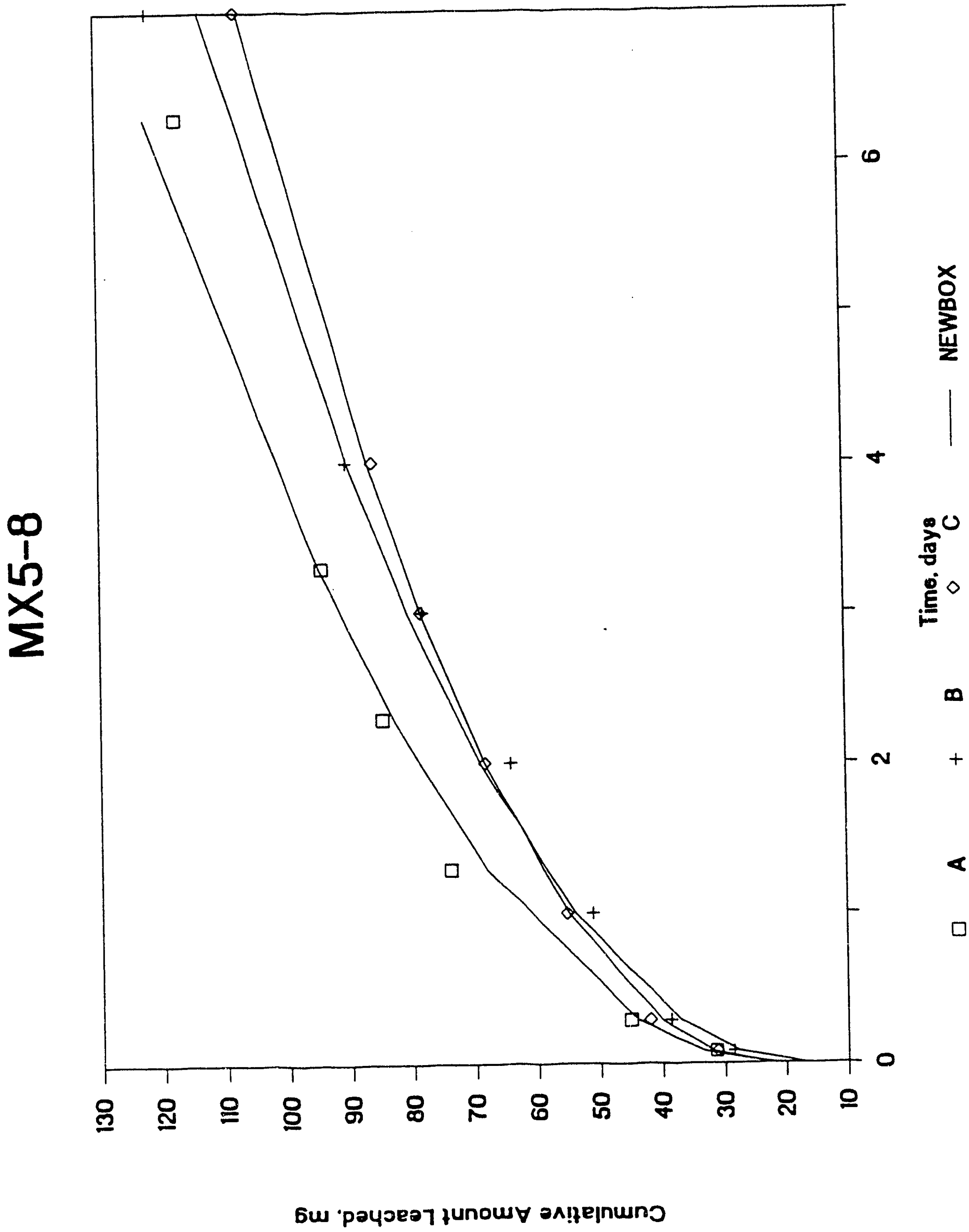
Interval	Time days	A	B	C
1	0.083	31	29	31
2	0.292	45	39	42
3	1.000		51	55
	1.292	74		
4	2.000		64	68
	2.292	85		
5	3.000		78	78
	3.292	94		
6	4.000		90	86
	6.292	117		
7	7.000		122	108

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	22	17	23
Std. Dev., mg =	5	5	2
Effective Diff. Coeff., sq. cm/s =	2.88E-09	2.24E-09	1.68E-09
Std. Dev., sq. cm/s =	6.40E-10	5.10E-10	1.30E-10
Leachability Index =	8.541	8.650	8.774
Upper 90% Conf. Int. =	0.041	0.036	0.012
Lower 90% Conf. Int. =	0.038	0.033	0.012

Cumulative Fraction Leached Based on Ao (CFL)

Initially in fresh grout =	1.117	1.092	1.074
Rinse =	0.117	0.092	0.074
Ao =	1.000	1.000	1.000
Washout =	0.019	0.014	0.019
Rinse + Washout =	0.136	0.106	0.093
Leaching by diffusion control =	0.981	0.986	0.981

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.027	0.029	0.025	0.024	0.026	0.027
0.292	0.039	0.038	0.033	0.032	0.035	0.034
1.000			0.044	0.046	0.046	0.046
1.292	0.065	0.059				
2.000			0.055	0.059	0.057	0.057
2.292	0.074	0.073				
3.000			0.067	0.069	0.066	0.066
3.292	0.082	0.083				
4.000			0.077	0.077	0.072	0.073
6.292	0.102	0.107				
7.000			0.104	0.097	0.091	0.090


MX5-8
Cumulative Amount Leached, mg
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX6-7

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.71	3.70	3.59
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	68	99	95
Cylinder at start of leaching, Ao =	1208	1178	1181

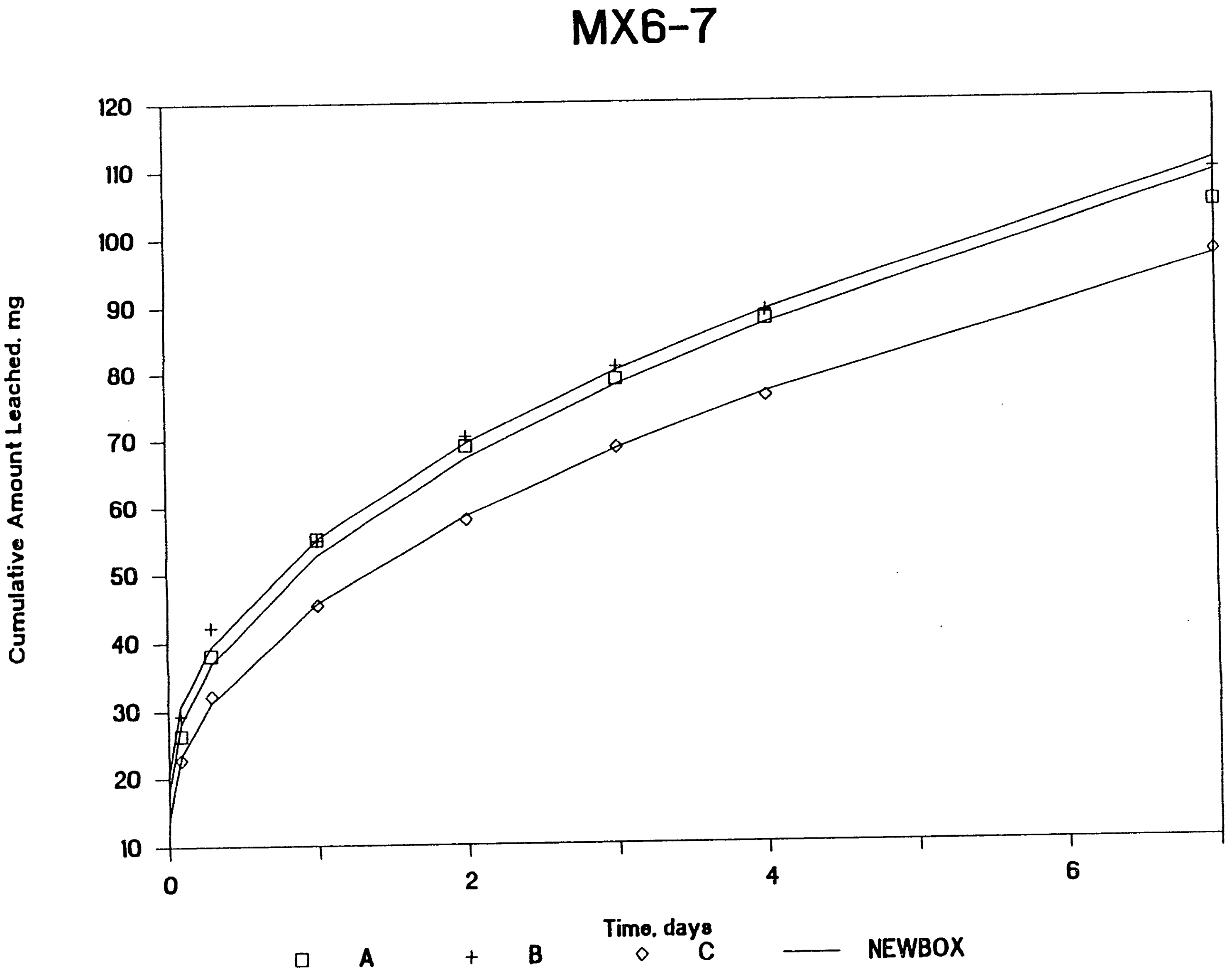
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	26	29	23
2	0.292	38	42	32
3	1	55	55	45
4	2	69	70	58
5	3	79	81	69
6	4	88	89	76
7	7	104	109	97

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	18	21	14
Std. Dev., mg =	3	2	1
Effective Diff. Coeff., sq. cm/s =	1.74E-09	1.80E-09	1.45E-09
Std. Dev., sq. cm/s =	2.57E-10	1.98E-10	6.58E-11
Leachability Index =	8.760	8.745	8.839
Upper 95% Conf. Int. =	0.024	0.018	0.007
Lower 95% Conf. Int. =	0.023	0.017	0.007

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.056	1.084	1.081
Rinse =	0.056	0.084	0.081
Ao =	1.000	1.000	1.000
Washout =	0.015	0.018	0.012
Rinse + Washout =	0.071	0.101	0.093
Leaching by diffusion control =	0.985	0.982	0.988

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.022	0.023	0.025	0.026	0.019	0.020
0.292	0.031	0.030	0.036	0.033	0.027	0.026
1	0.046	0.044	0.047	0.047	0.038	0.039
2	0.057	0.056	0.059	0.059	0.049	0.050
3	0.065	0.065	0.069	0.068	0.058	0.058
4	0.073	0.072	0.076	0.076	0.064	0.065
7	0.086	0.090	0.093	0.094	0.082	0.082


MX6-7
Cumulative Amount Leached. mg
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time. days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX7-3

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.41	4.41	4.42
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	48	56	46
Cylinder at start of leaching, Ao =	1229	1221	1230

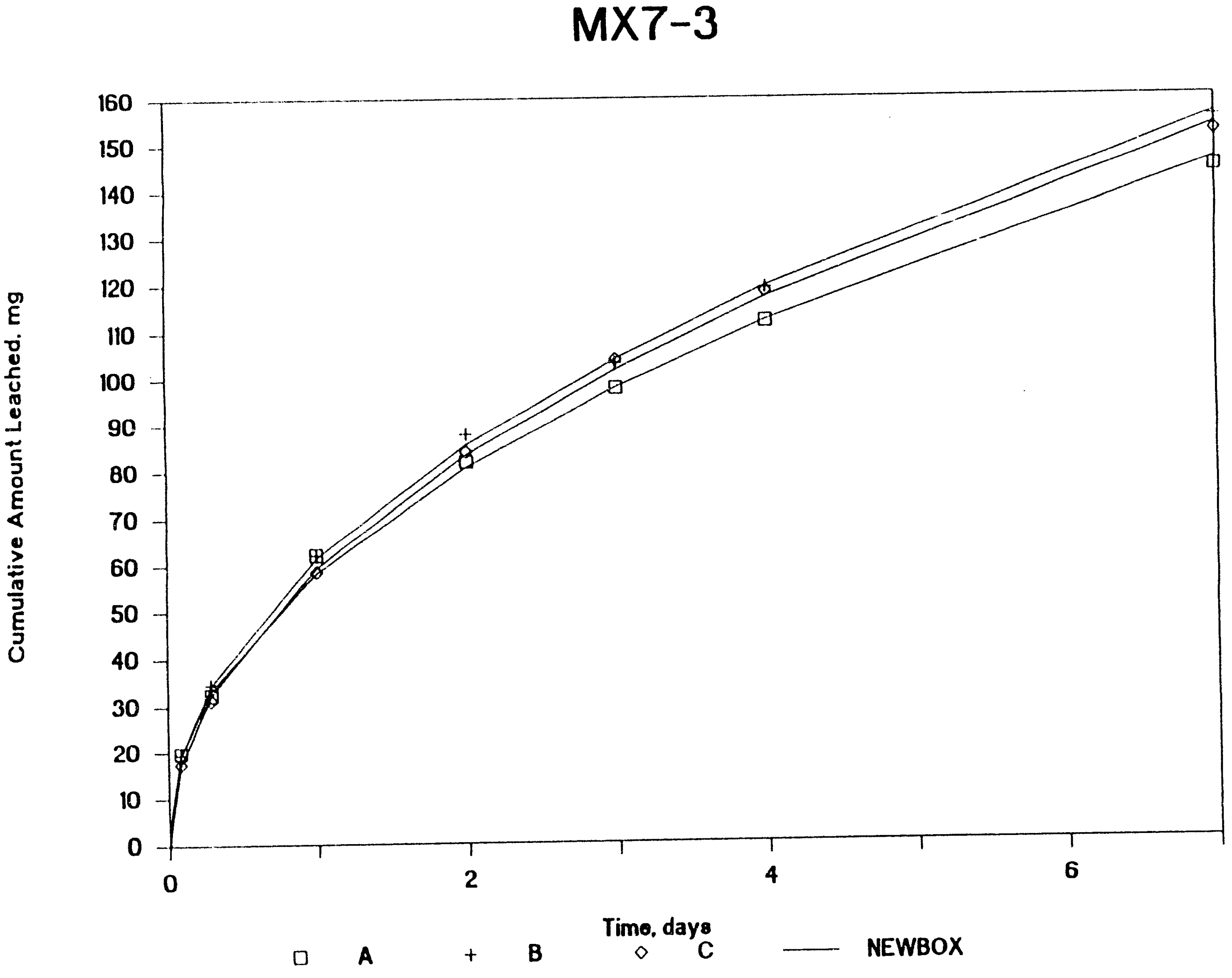
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	20	20	17
2	0.292	32	35	31
3	1	62	62	59
4	2	82	88	84
5	3	98	103	104
6	4	112	119	118
7	7	144	155	152

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	3	2	0
Std. Dev., mg =	2	1	1
Effective Diff. Coeff., sq. cm/s =	4.50E-09	5.32E-09	5.16E-09
Std. Dev., sq. cm/s =	3.21E-10	1.90E-10	2.25E-10
Leachability Index =	8.347	8.274	8.287
Upper 95% Conf. Int. =	0.010	0.005	0.006
Lower 95% Conf. Int. =	0.010	0.005	0.006

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.039	1.046	1.037
Rinse =	0.039	0.046	0.037
Ao =	1.000	1.000	1.000
Washout =	0.003	0.001	0.000
Rinse + Washout =	0.041	0.047	0.037
Leaching by diffusion control =	0.997	0.999	1.000

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.015	0.016	0.016	0.016	0.014	0.014
0.292	0.026	0.027	0.028	0.028	0.025	0.026
1	0.050	0.048	0.051	0.050	0.048	0.048
2	0.067	0.066	0.072	0.070	0.068	0.068
3	0.079	0.080	0.084	0.085	0.084	0.083
4	0.091	0.091	0.097	0.098	0.096	0.095
7	0.117	0.119	0.127	0.128	0.123	0.125


MX7-3
Cumulative Amount Leached, mg
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
0
2
4
6
Time, days
□ A
+ B
◇ C
— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX8-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.43	4.45	4.44
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	39	38	18
Cylinder at start of leaching, Ao =	1237	1239	1259

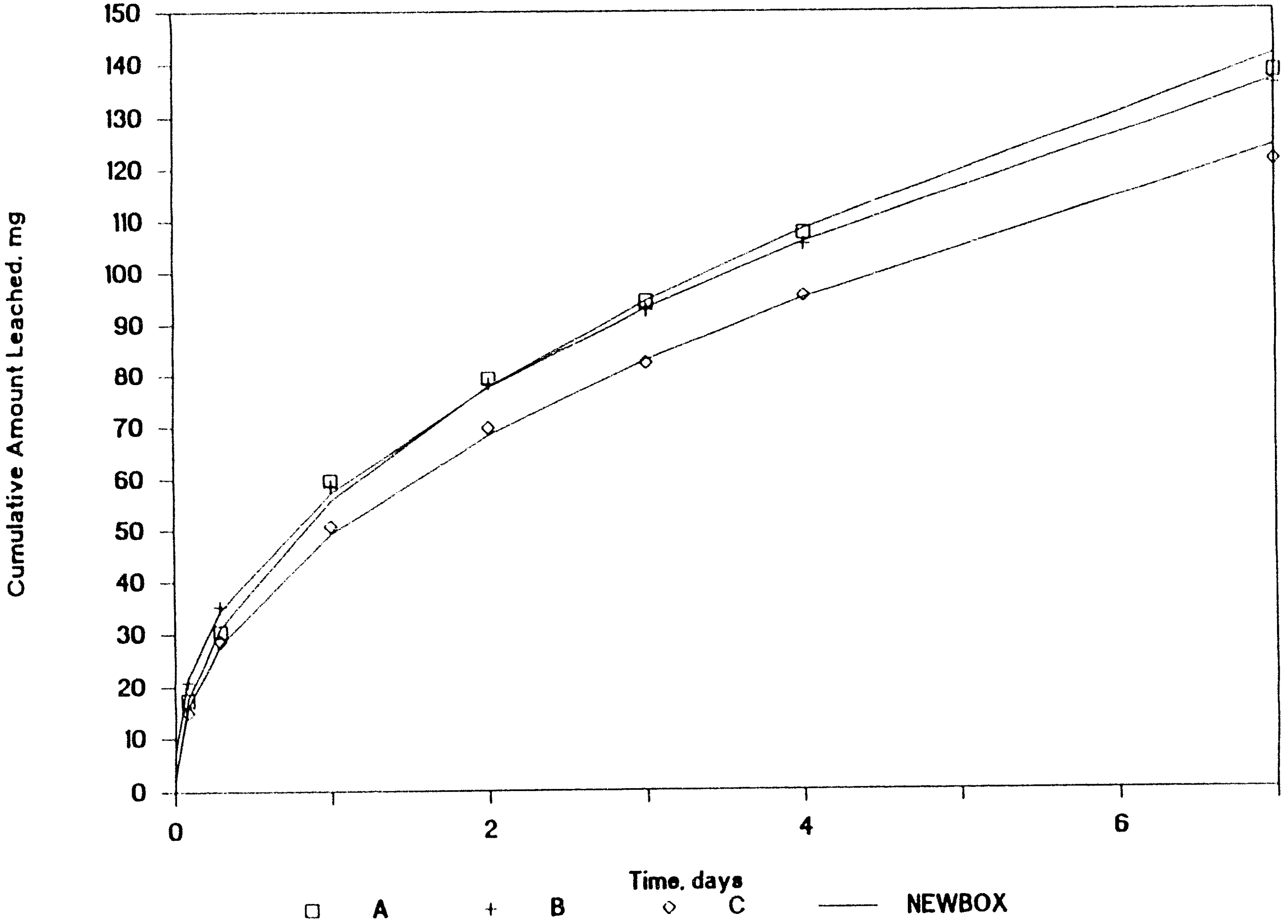
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	17	21	15
2	0.292	30	35	29
3	1	60	59	51
4	2	79	78	70
5	3	94	92	82
6	4	108	105	95
7	7	138	136	121

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	2	7	2
Std. Dev., mg =	3	1	2
Effective Diff. Coeff., sq. cm/s =	4.22E-09	3.63E-09	3.05E-09
Std. Dev., sq. cm/s =	3.68E-10	1.76E-10	2.30E-10
Leachability Index =	8.374	8.440	8.515
Upper 95% Conf. Int. =	0.012	0.007	0.011
Lower 95% Conf. Int. =	0.012	0.007	0.010

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.032	1.031	1.014
Rinse =	0.032	0.031	0.014
Ao =	1.000	1.000	1.000
Washout =	0.001	0.006	0.002
Rinse + Washout =	0.033	0.036	0.016
Leaching by diffusion control =	0.999	0.994	0.998

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.014	0.014	0.017	0.018	0.012	0.013
0.292	0.024	0.025	0.028	0.028	0.023	0.022
1	0.049	0.045	0.048	0.046	0.041	0.039
2	0.064	0.063	0.063	0.062	0.056	0.054
3	0.076	0.076	0.074	0.075	0.065	0.066
4	0.087	0.087	0.085	0.085	0.075	0.075
7	0.112	0.114	0.110	0.110	0.096	0.098



MX8-4

150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Cumulative Amount Leached, mg
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

```
Nitrate Leaching Data
Matrix Run No.:   MX9-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm  Nitrate mass =   1276.5 mg

Cured Cylinder =                          A        B        C
Length, cm =                              4.33     4.30     4.35
Diameter, cm =                            2.50     2.50     2.50

Nitate Mass, mg
Rinse =                                     34       42        4
Cylinder at start of leaching, Ao =       1242     1234     1273

Cumulative Amount Leached, mg nitrate

                        Interval   Time       A        B        C
                                   days

                               1   0.083      56       57       63
                               2   0.292      93       95      114
                               3       1     141      143      175
                               4       2     178      173      214
                               5       3     222      199      240
                               6       4     242      221      265
                               7       7     283      262      315

NEWBOX Estimate of Parameters             A        B        C

Washoff, mg =                               30       37       39
        Std. Dev., mg =                     10       12       18
Effective Diff. Coeff., sq. cm/s =  1.75E-08 1.38E-08 2.06E-08
        Std. Dev., sq. cm/s =       2.83E-09 2.85E-09 5.22E-09
Leachability Index =                     7.756    7.860    7.685
        Upper 95% Conf. Int. =           0.023    0.030    0.037
        Lower 95% Conf. Int. =           0.022    0.028    0.034

Cumulative Fraction Leached Based on Ao (CFL)
Initially in fresh grout =               1.028    1.034    1.003
Rinse =                                  0.028    0.034    0.003
Ao =                                     1.000    1.000    1.000
Washout =                                0.024    0.030    0.031
Rinse + Washout =                        0.051    0.064    0.034
Leaching by diffusion control =          0.976    0.970    0.969

   Time      CFL of A          CFL of B          CFL of C
   days   Obsv'd   Calc'd   Obsv'd   Calc'd   Obsv'd   Calc'd

  0.083   0.045    0.049    0.046    0.052    0.049    0.058
  0.292   0.075    0.071    0.077    0.072    0.090    0.081
      1   0.114    0.110    0.116    0.106    0.137    0.123
      2   0.143    0.144    0.140    0.137    0.168    0.160
      3   0.179    0.170    0.161    0.160    0.189    0.188
      4   0.195    0.191    0.179    0.179    0.208    0.210
      7   0.228    0.242    0.212    0.224    0.247    0.264
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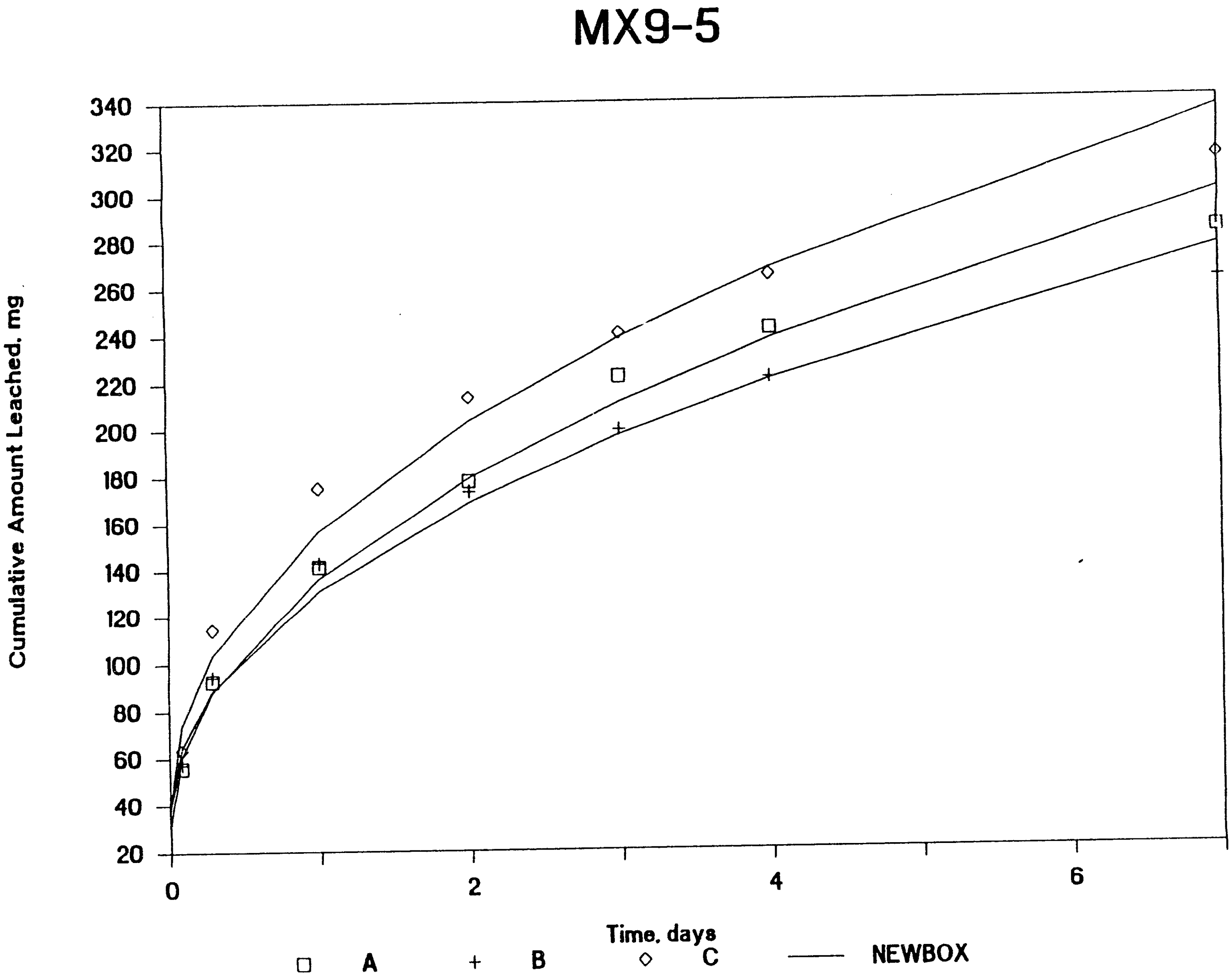

MX9-5
Cumulative Amount Leached, mg
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX10-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.14	4.17	4.14
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	78	83	51
Cylinder at start of leaching, Ao =	1199	1193	1226

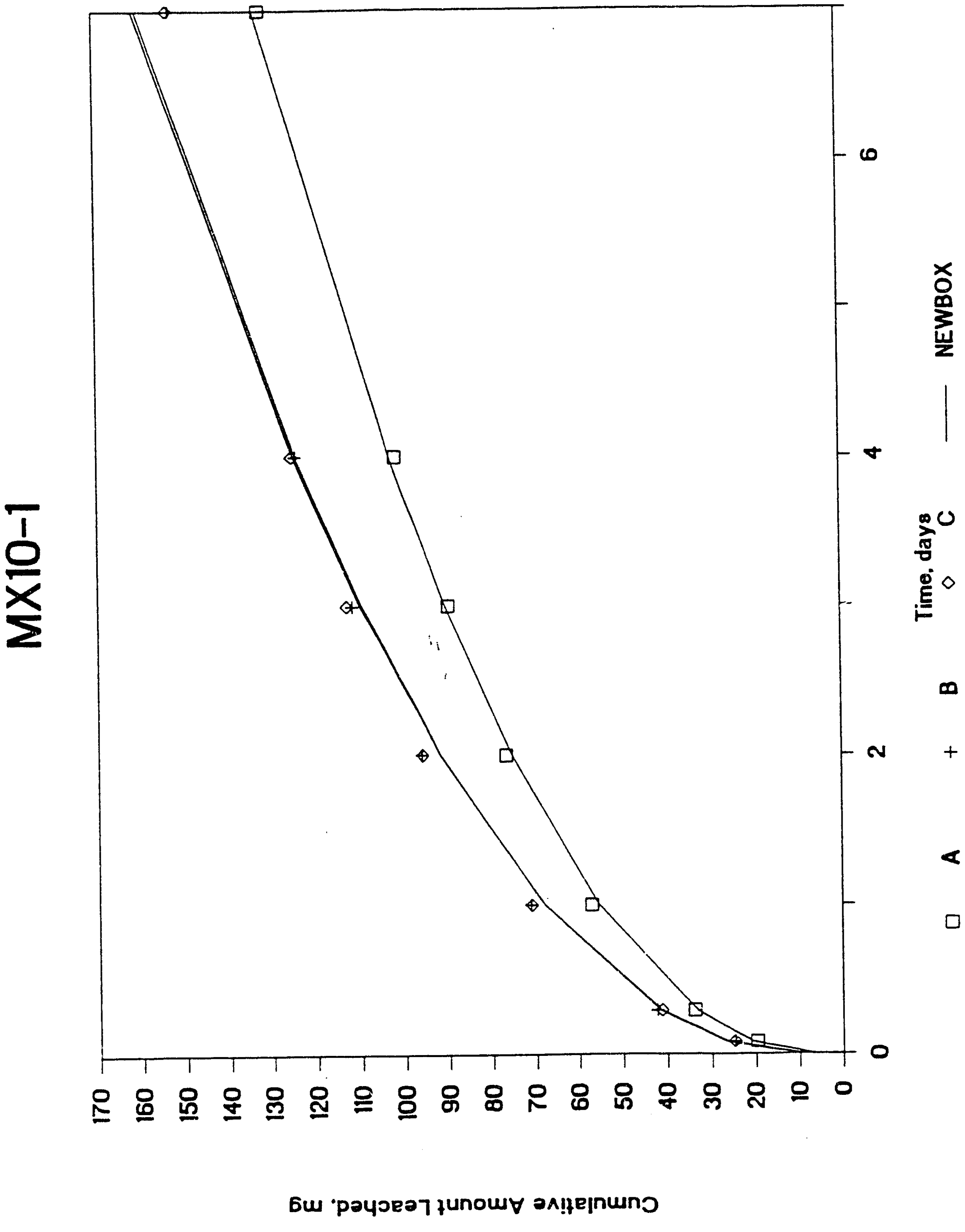
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	20	25	25
2	0.292	34	42	41
3	1	57	71	71
4	2	77	96	96
5	3	90	112	113
6	4	102	125	125
7	7	132	152	153

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	7	10	9
Std. Dev., mg =	2	4	4
Effective Diff. Coeff., sq. cm/s =	3.60E-09	5.24E-09	5.02E-09
Std. Dev., sq. cm/s =	2.02E-10	6.65E-10	6.12E-10
Leachability Index =	8.444	8.281	8.300
Upper 95% Conf. Int. =	0.008	0.018	0.017
Lower 95% Conf. Int. =	0.008	0.017	0.017

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.065	1.070	1.041
Rinse =	0.065	0.070	0.041
Ao =	1.000	1.000	1.000
Washout =	0.006	0.008	0.008
Rinse + Washout =	0.071	0.078	0.049
Leaching by diffusion control =	0.994	0.992	0.992

Time days	CFL of A Obsv'd	CFL of A Calc'd	CFL of B Obsv'd	CFL of B Calc'd	CFL of C Obsv'd	CFL of C Calc'd
0.083	0.017	0.017	0.021	0.022	0.020	0.022
0.292	0.028	0.028	0.035	0.035	0.033	0.034
1	0.048	0.046	0.060	0.057	0.058	0.055
2	0.064	0.063	0.080	0.077	0.078	0.075
3	0.075	0.075	0.094	0.092	0.092	0.090
4	0.085	0.086	0.105	0.104	0.102	0.102
7	0.110	0.111	0.127	0.134	0.125	0.131


MX10-1
Cumulative Amount Leached, mg
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX11-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.28	4.24	4.11
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	48	31	30
Cylinder at start of leaching, Ao =	1229	1245	1247

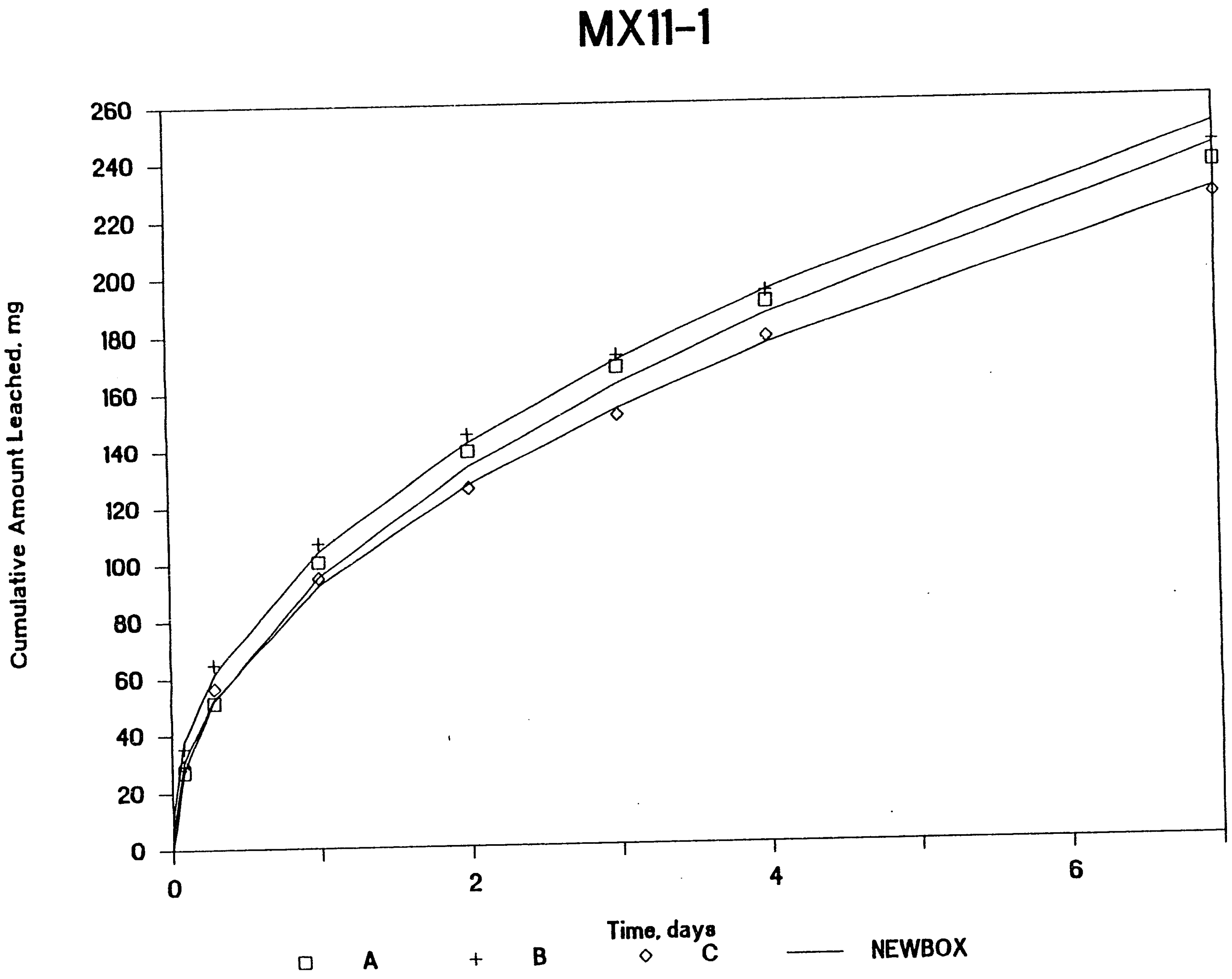
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	27	35	29
2	0.292	51	65	56
3	1	101	107	95
4	2	139	145	126
5	3	168	172	151
6	4	190	194	178
7	7	236	243	225

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	0	10	5
Std. Dev., mg =	5	5	3
Effective Diff. Coeff., sq. cm/s =	1.34E-08	1.29E-08	1.07E-08
Std. Dev., sq. cm/s =	1.43E-09	1.11E-09	7.63E-10
Leachability Index =	7.874	7.889	7.972
Upper 95% Conf. Int. =	0.015	0.012	0.010
Lower 95% Conf. Int. =	0.015	0.012	0.010

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.039	1.025	1.024
Rinse =	0.039	0.025	0.024
Ao =	1.000	1.000	1.000
Washout =	0.000	0.008	0.004
Rinse + Washout =	0.039	0.033	0.028
Leaching by diffusion control =	1.000	0.992	0.996

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.020	0.023	0.028	0.030	0.023	0.025
0.292	0.041	0.042	0.052	0.049	0.045	0.042
1	0.082	0.077	0.086	0.084	0.076	0.074
2	0.105	0.108	0.116	0.114	0.101	0.102
3	0.137	0.132	0.138	0.137	0.121	0.123
4	0.155	0.151	0.156	0.156	0.143	0.141
7	0.192	0.197	0.195	0.201	0.180	0.182


MX11-1
Cumulative Amount Leached, mg
0
20
40
60
80
100
120
140
160
180
200
220
240
260
Time, days
0
2
4
6
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX12-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.42	4.43
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	20	22	19
Cylinder at start of leaching, Ao =	1257	1254	1257

Cumulative Amount Leached, mg nitrate

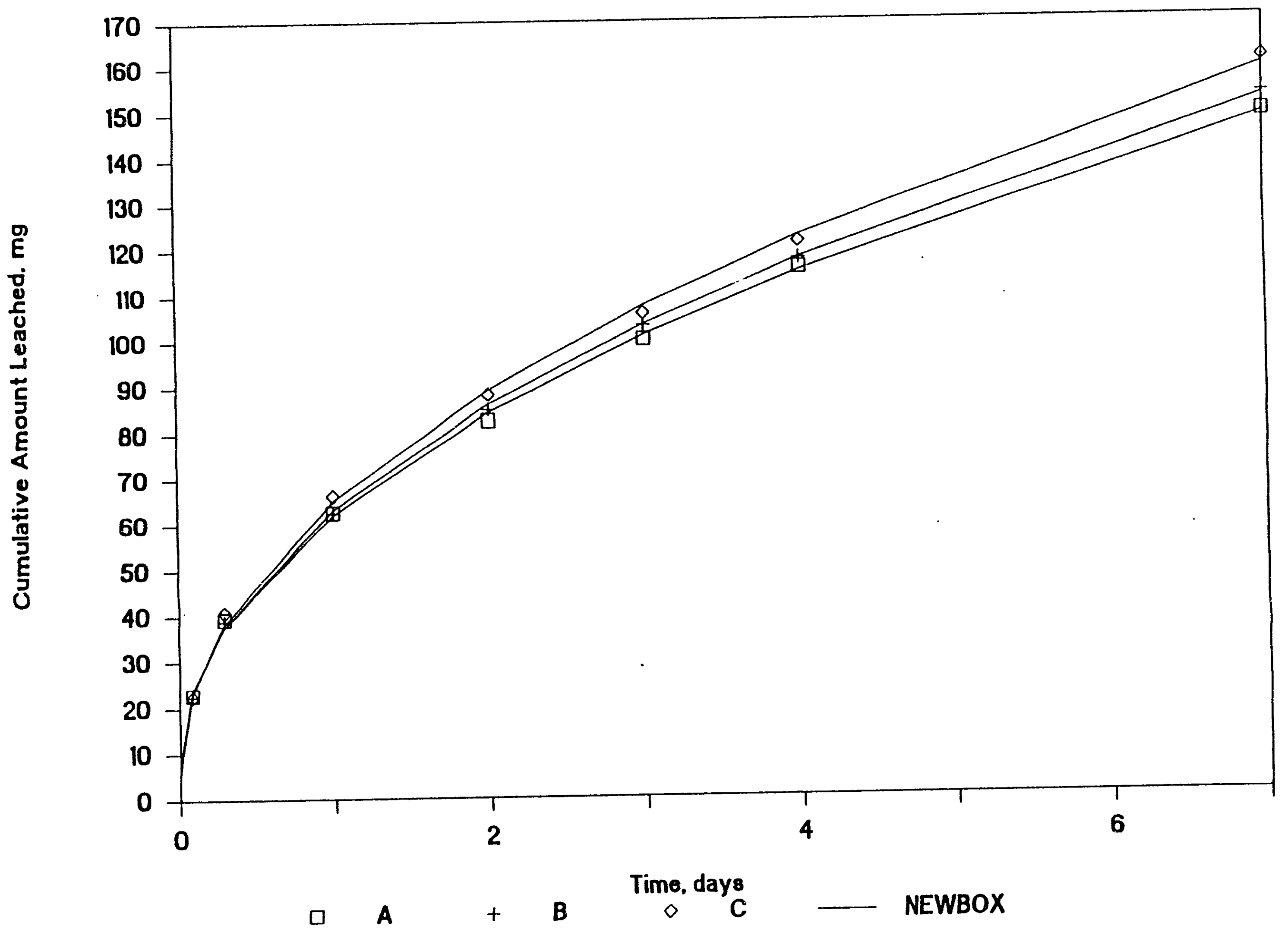
Interval	Time days	A	B	C
1	0.083	23	23	22
2	0.292	39	39	41
3	1	63	63	66
4	2	82	85	88
5	3	100	103	106
6	4	116	118	121
7	7	149	153	161

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	8	7	6
Std. Dev., mg =	2	1	3
Effective Diff. Coeff., sq. cm/s =	4.19E-09	4.48E-09	4.97E-09
Std. Dev., sq. cm/s =	2.39E-10	1.62E-10	3.75E-10
Leachability Index =	8.378	8.348	8.304
Upper 95% Conf. Int. =	0.008	0.005	0.011
Lower 95% Conf. Int. =	0.008	0.005	0.010

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.016	1.018	1.015
Rinse =	0.016	0.018	0.015
Ao =	1.000	1.000	1.000
Washout =	0.006	0.006	0.005
Rinse + Washout =	0.022	0.024	0.020
Leaching by diffusion control =	0.994	0.994	0.995

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.018	0.019	0.018	0.019	0.018	0.018
0.292	0.031	0.030	0.031	0.030	0.033	0.030
1	0.050	0.049	0.050	0.050	0.053	0.052
2	0.065	0.067	0.068	0.069	0.070	0.071
3	0.080	0.080	0.082	0.082	0.084	0.085
4	0.092	0.091	0.094	0.094	0.096	0.098
7	0.119	0.118	0.122	0.121	0.128	0.126

MX12-4


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Cumulative Amount Leached, mg
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX13-9

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.49	4.49
Diameter, cm =	2.50	2.50	2.50

Nitate Mass, mg			
Rinse =	4	4	4
Cylinder at start of leaching, Ao =	1272	1273	1272

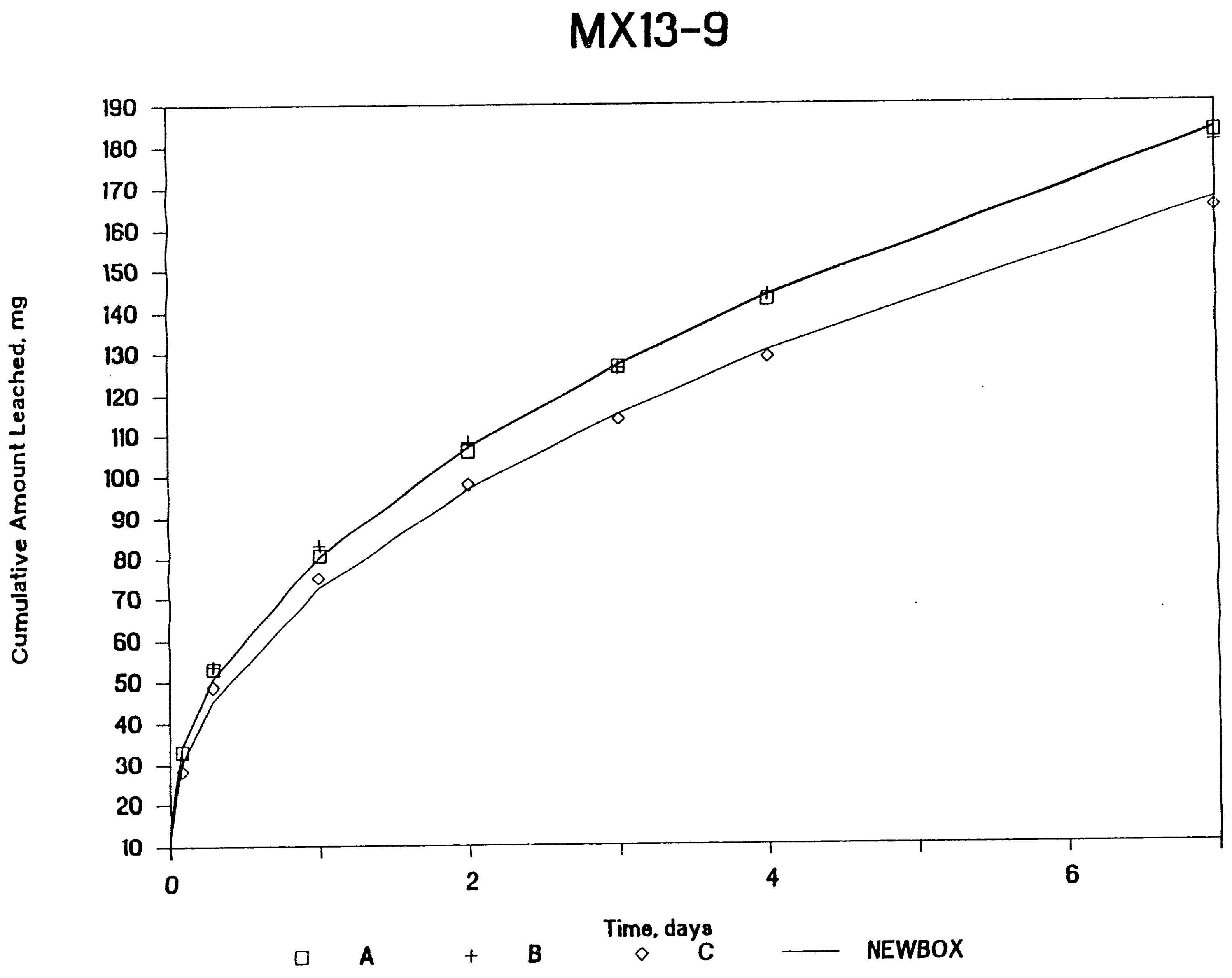
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	33	32	28
2	0.292	53	54	49
3	1	81	83	75
4	2	106	108	98
5	3	127	126	114
6	4	143	144	129
7	7	183	181	165

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	15	15	13
Std. Dev., mg =	2	3	4
Effective Diff. Coeff., sq. cm/s =	6.00E-09	6.04E-09	4.98E-09
Std. Dev., sq. cm/s =	3.07E-10	5.36E-10	5.15E-10
Leachability Index =	8.222	8.219	8.302
Upper 95% Conf. Int. =	0.007	0.013	0.015
Lower 95% Conf. Int. =	0.007	0.012	0.014

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.003	1.003	1.003
Rinse =	0.003	0.003	0.003
Ao =	1.000	1.000	1.000
Washout =	0.012	0.012	0.010
Rinse + Washout =	0.015	0.015	0.014
Leaching by diffusion control =	0.988	0.988	0.990

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.026	0.027	0.025	0.027	0.022	0.024
0.292	0.042	0.040	0.042	0.040	0.039	0.036
1	0.064	0.063	0.065	0.063	0.059	0.057
2	0.083	0.084	0.085	0.084	0.077	0.076
3	0.100	0.100	0.099	0.100	0.090	0.090
4	0.112	0.113	0.113	0.113	0.101	0.103
7	0.144	0.144	0.142	0.144	0.130	0.131


MX13-9
Cumulative Amount Leached, mg
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX-S-12

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

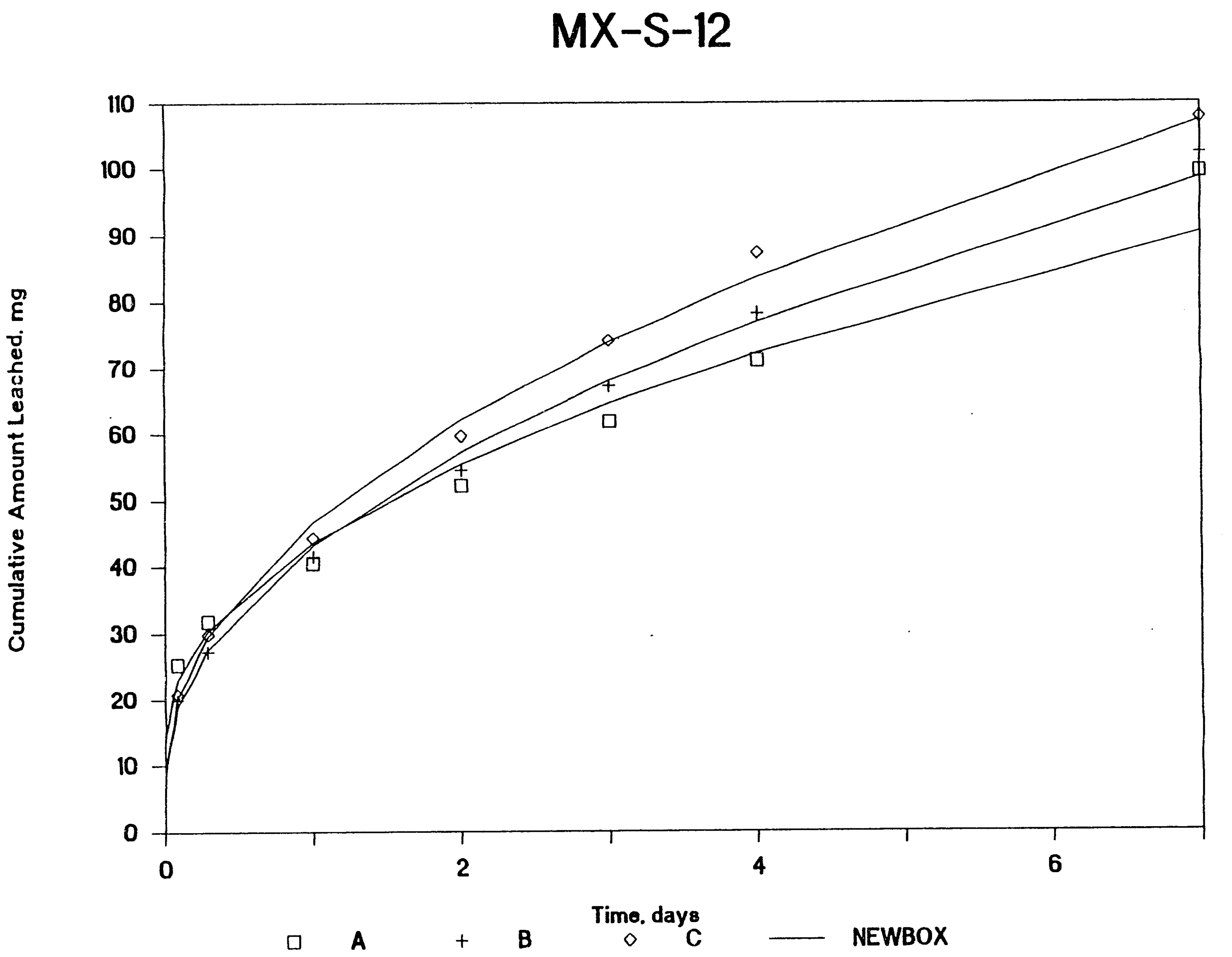
Cured Cylinder =	A	B	C
Length, cm =	3.91	3.76	3.85
Diameter, cm =	2.50	2.50	2.50
Nitrate Mass, mg			
Rinse =	76	75	64
Cylinder at start of leaching, Ao =	1200	1201	1213

Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	25	20	21
2	0.292	32	27	30
3	1	40	42	44
4	2	52	55	60
5	3	62	67	74
6	4	71	78	87
7	7	100	102	108

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	15	9	9
Std. Dev., mg =	6	2	3
Effective Diff. Coeff., sq. cm/s =	1.24E-09	1.70E-09	2.01E-09
Std. Dev., sq. cm/s =	3.70E-10	1.84E-10	2.28E-10
Leachability Index =	8.908	8.770	8.697
Upper 95% Conf. Int. =	0.051	0.018	0.018
Lower 95% Conf. Int. =	0.045	0.017	0.018
Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.064	1.063	1.052
Rinse =	0.064	0.063	0.052
Ao =	1.000	1.000	1.000
Washout =	0.012	0.007	0.008
Rinse + Washout =	0.076	0.070	0.060
Leaching by diffusion control =	0.988	0.993	0.992

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.021	0.019	0.017	0.016	0.017	0.017
0.292	0.027	0.025	0.022	0.023	0.025	0.024
1	0.033	0.036	0.035	0.036	0.036	0.039
2	0.043	0.046	0.046	0.048	0.049	0.051
3	0.052	0.054	0.056	0.057	0.061	0.061
4	0.059	0.060	0.065	0.064	0.072	0.069
7	0.083	0.075	0.085	0.082	0.089	0.088


MX-S-12
Cumulative Amount Leached, mg
0
10
20
30
40
50
60
70
80
90
100
110
0
2
4
6
Time, days
□ A
+ B
◇ C
NEWBOX

Nitrate Leaching Data
Matrix Run No.: MX-S-15

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrate mass = 1276.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.81	3.72	3.73
Diameter, cm =	2.50	2.50	2.50

Nitrate Mass, mg			
Rinse =	83	107	114
Cylinder at start of leaching, Ao =	1194	1169	1163

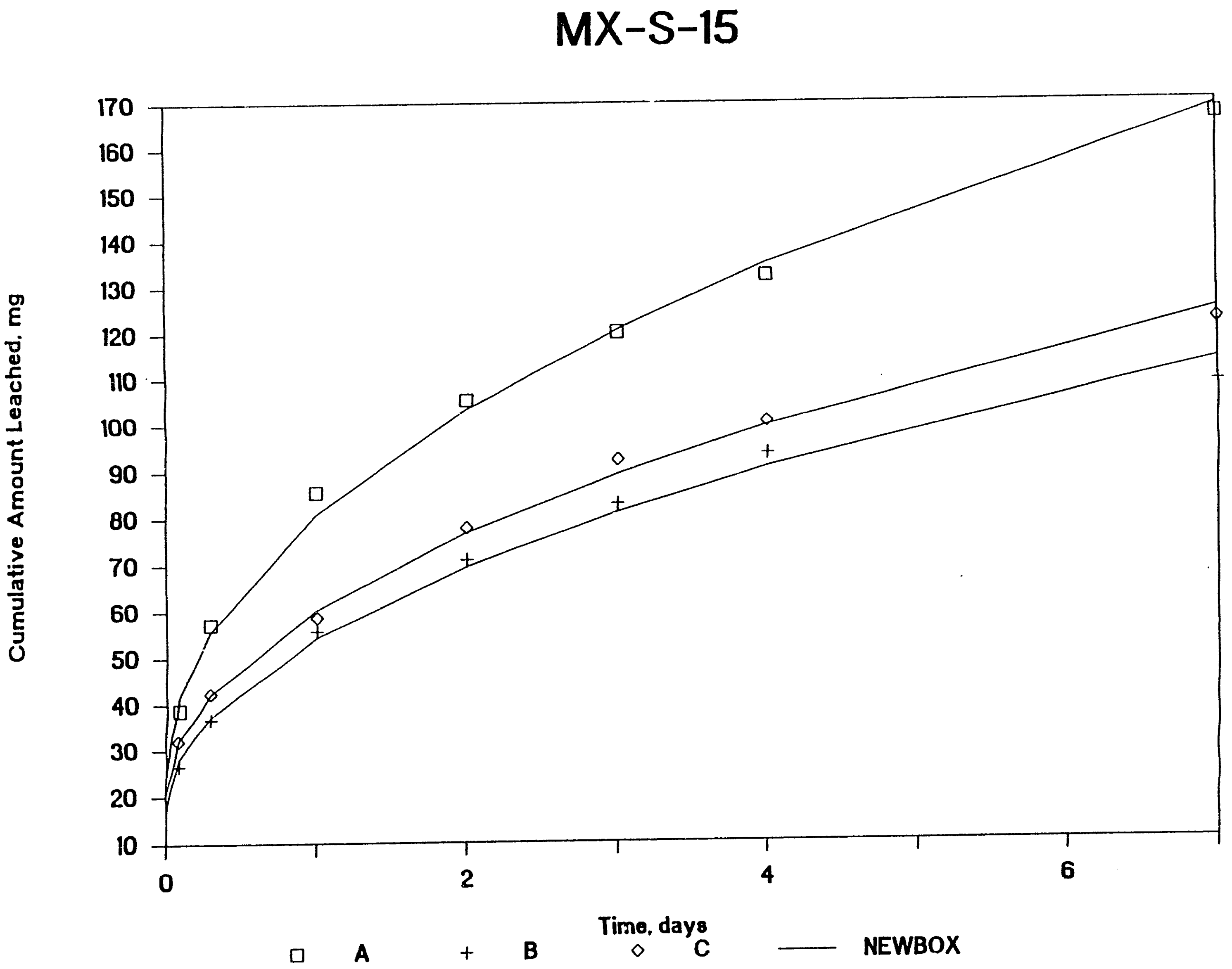
Cumulative Amount Leached, mg nitrate

Interval	Time days	A	B	C
1	0.083	39	27	32
2	0.292	57	37	42
3	1	86	56	59
4	2	105	71	78
5	3	120	83	92
6	4	132	93	100
7	7	167	108	122

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	25	17	20
Std. Dev., mg =	4	3	2
Effective Diff. Coeff., sq. cm/s =	4.68E-09	2.10E-09	2.50E-09
Std. Dev., sq. cm/s =	5.51E-10	2.85E-10	2.00E-10
Leachability Index =	8.330	8.678	8.602
Upper 95% Conf. Int. =	0.019	0.022	0.013
Lower 95% Conf. Int. =	0.018	0.021	0.013

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.069	1.092	1.098
Rinse =	0.069	0.092	0.098
Ao =	1.000	1.000	1.000
Washout =	0.021	0.015	0.018
Rinse + Washout =	0.090	0.107	0.115
Leaching by diffusion control =	0.979	0.985	0.982

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.033	0.035	0.023	0.024	0.028	0.028
0.292	0.048	0.047	0.032	0.032	0.036	0.036
1	0.072	0.068	0.048	0.046	0.051	0.052
2	0.088	0.087	0.061	0.059	0.067	0.066
3	0.101	0.101	0.071	0.069	0.079	0.077
4	0.111	0.113	0.080	0.078	0.086	0.086
7	0.140	0.141	0.092	0.097	0.105	0.107


MX-S-15
Cumulative Amount Leached, mg
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Appendix E

FREESTANDING-LIQUID SCREENING TESTS

Appendix E
FREESTANDING-LIQUID SCREENING TESTS

E.1: SIMULATED ADIABATIC TEMPERATURE PROFILE

Meeting the freestanding-liquid criterion was a matter of some concern, as was made clear in the text of this report. Dr. Ryan Lokken indicated that the freestanding liquid experienced during field operations may be worse than that observed for laboratory measurements at 50°C. The basis for this concern was that estimates indicate that at least 90% of each grout monolith will approach the temperature profile of adiabatic curing. The ultimate temperature achieved adiabatically is well above 50°C, and higher isothermal temperatures lead to lower freestanding liquids. Unfortunately, most of the freestanding liquid is generated during the initial 24 h, and, during this critical time period, the grout generated in the field is expected to be about 40°C. The fact that several days are required to pour one monolith will complicate the actual observed field freestanding liquid in unknown ways, but the high freestanding liquids observed at these lower temperatures in the laboratory causes concern. Because of these concerns, a set of freestanding liquids was measured in the laboratory at temperatures approximating those of an adiabatic-temperature profile supplied by Dr. Lokken. The samples were matrix reference grouts left over from making leach samples, and there was only enough grout for one 250-mL freestanding liquid sample. Without replicates, these data must be considered as screening tests, but they give an idea of what the freestanding liquid performance will be for these grouts cured adiabatically. While not conclusive, these results did support Dr. Lokken's theory of retarded set and physical segregation.

Figure E.1 illustrates the measured temperature profile inside the oven housing these freestanding liquid samples. The temperatures were intended to be a series of step changes emulating the smooth rise in temperature after 24 h of the adiabatic temperature profile supplied by Dr. Lokken. As can be seen in Fig. E.1, the oven temperature tended to drift, requiring an occasional manual reset, during the 28 d of the test.

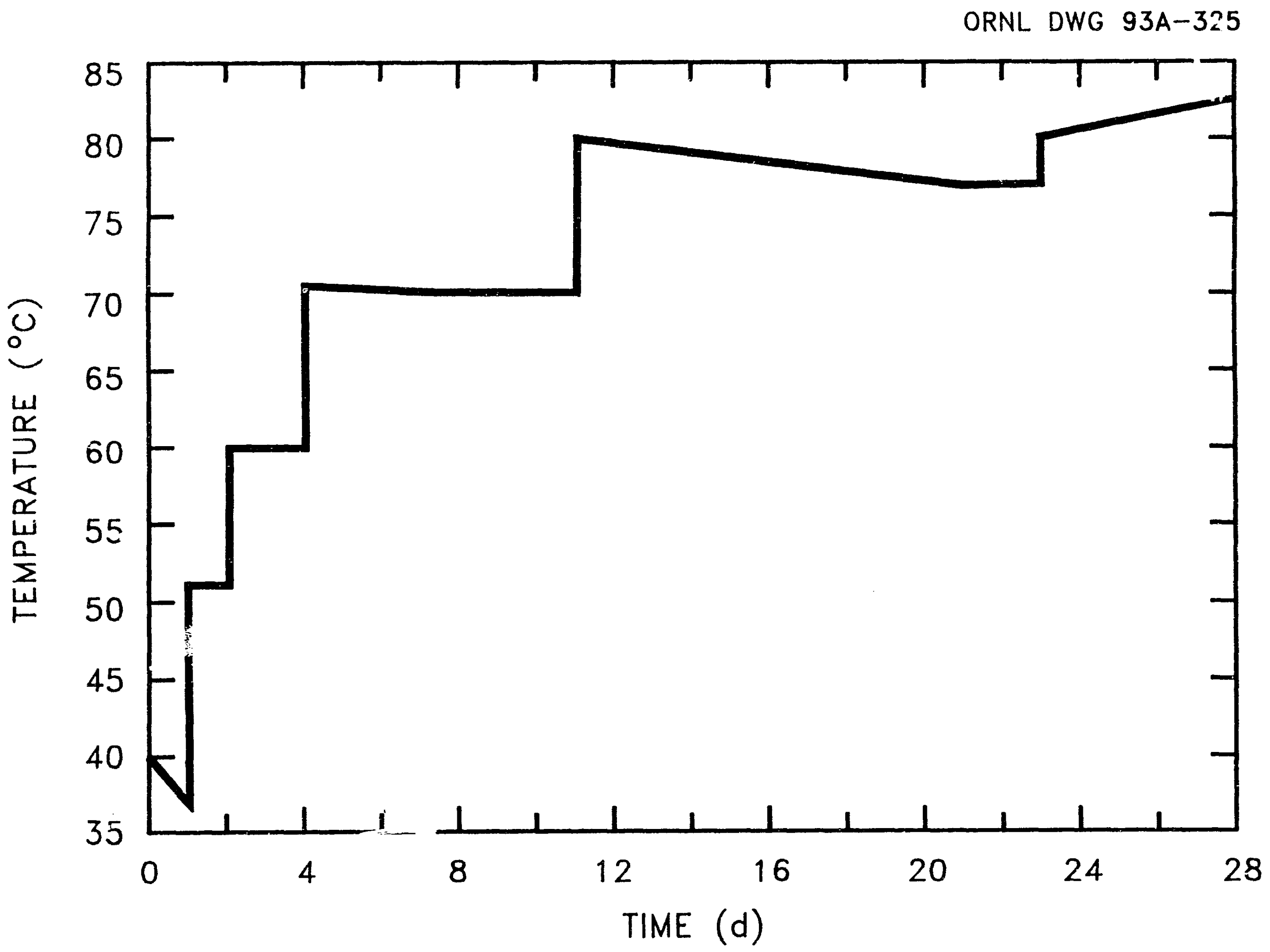


Fig. E.1. Measured temperature profile simulating adiabatic curing.

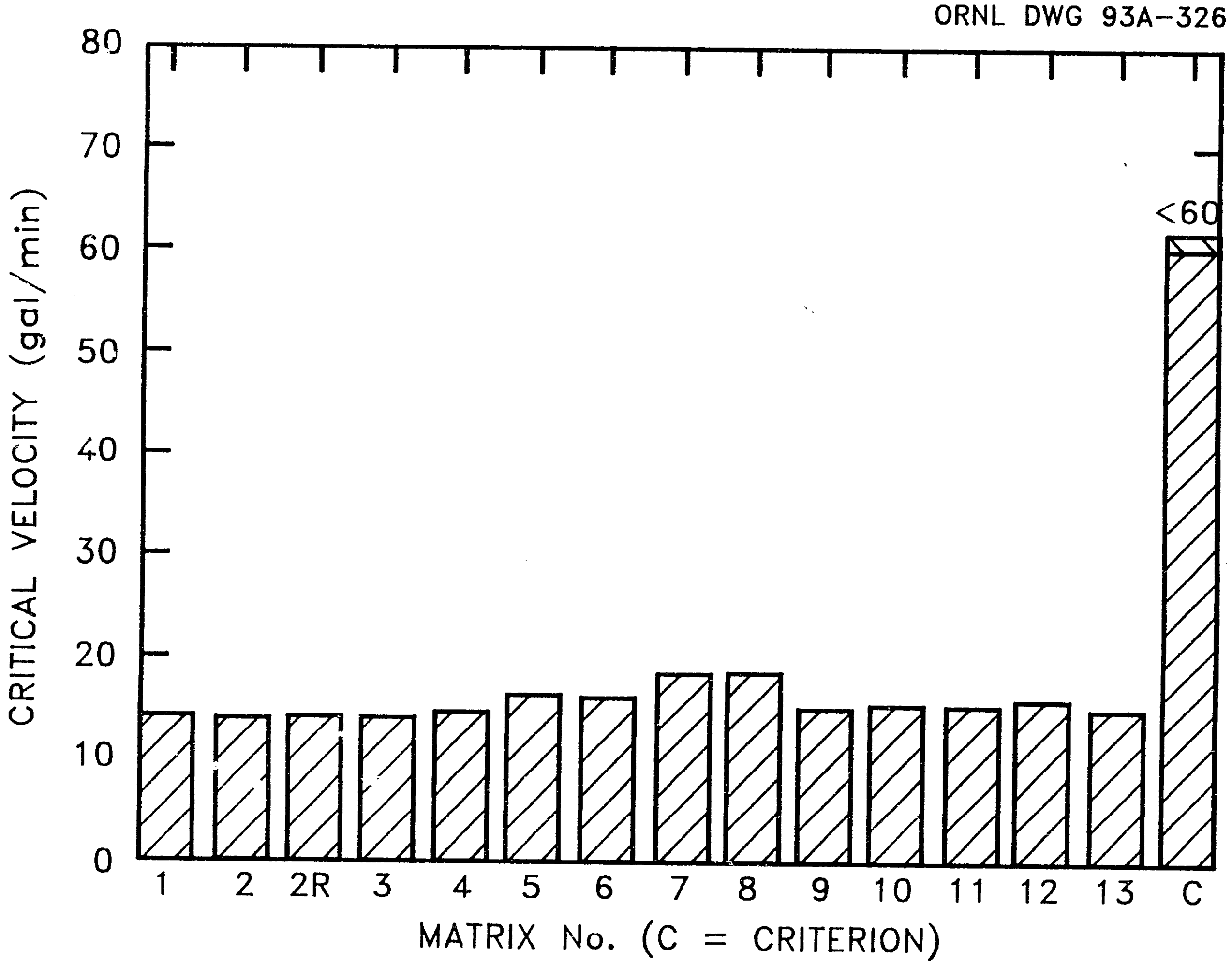


Fig. E.2. Average critical velocity for each matrix using the reference grout formulation.

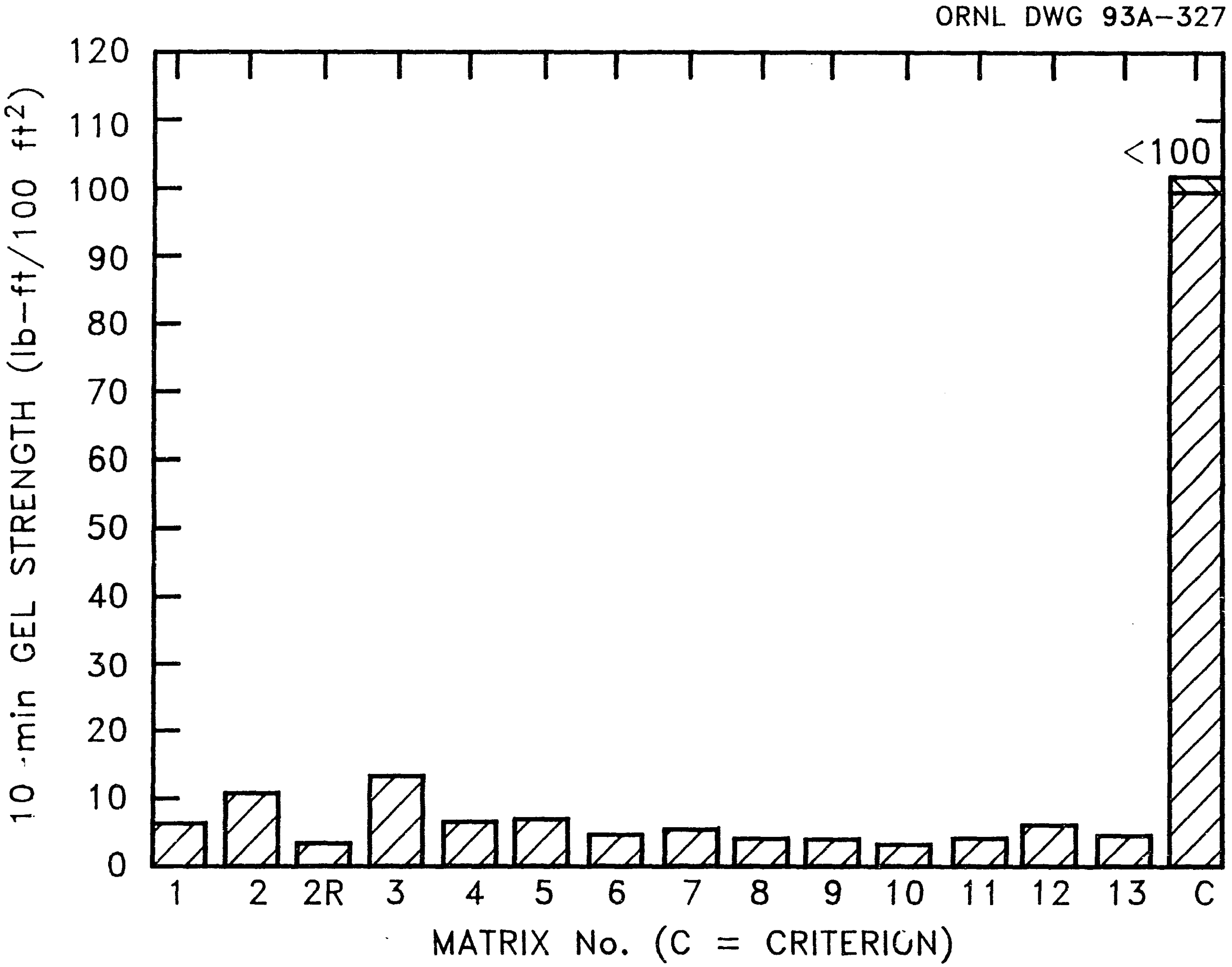


Fig. E.3. Average 10-min gel strength for each matrix using the reference grout formulation.

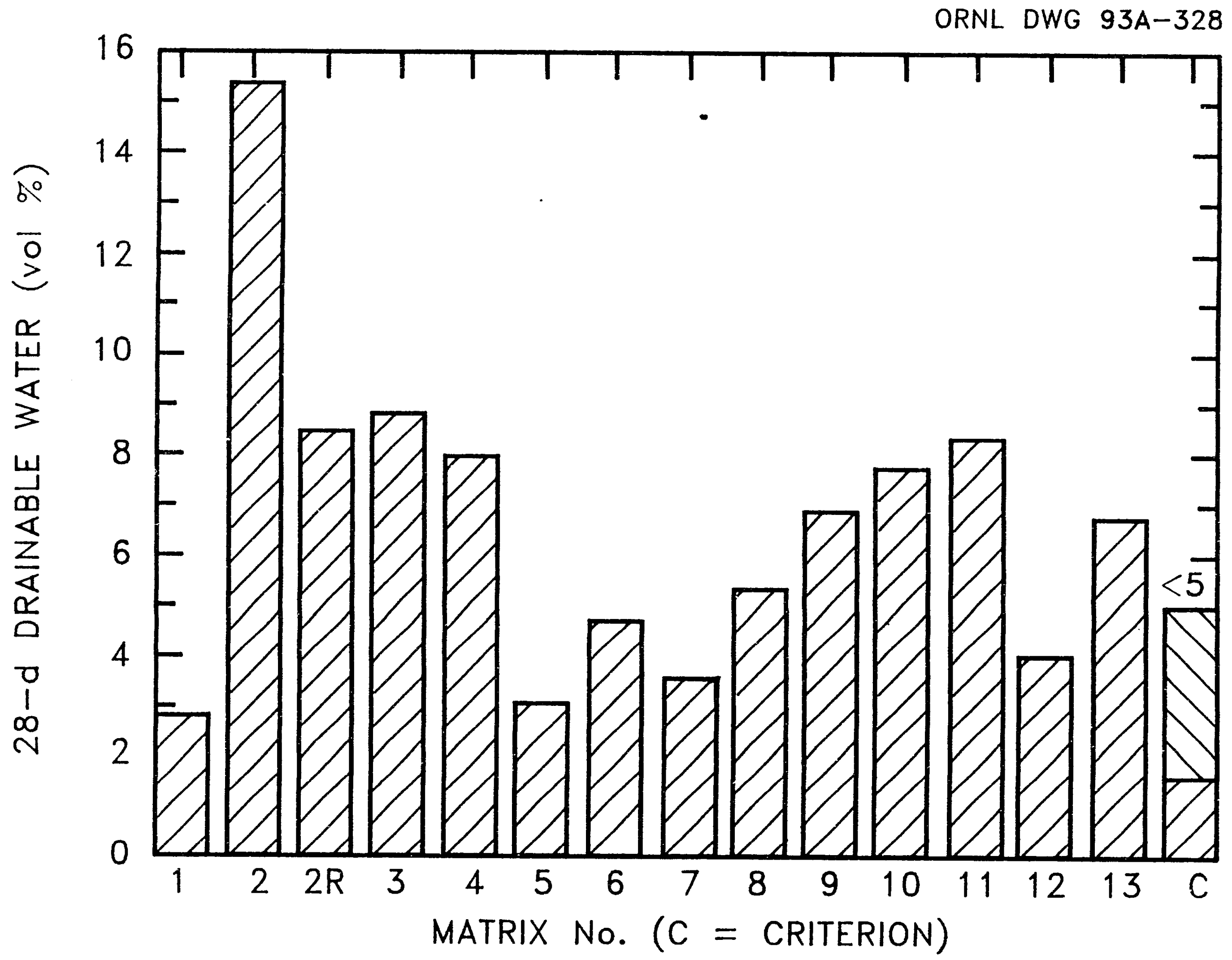


Fig. E.4. Average 28-d freestanding water for each matrix using the reference grout formulation.

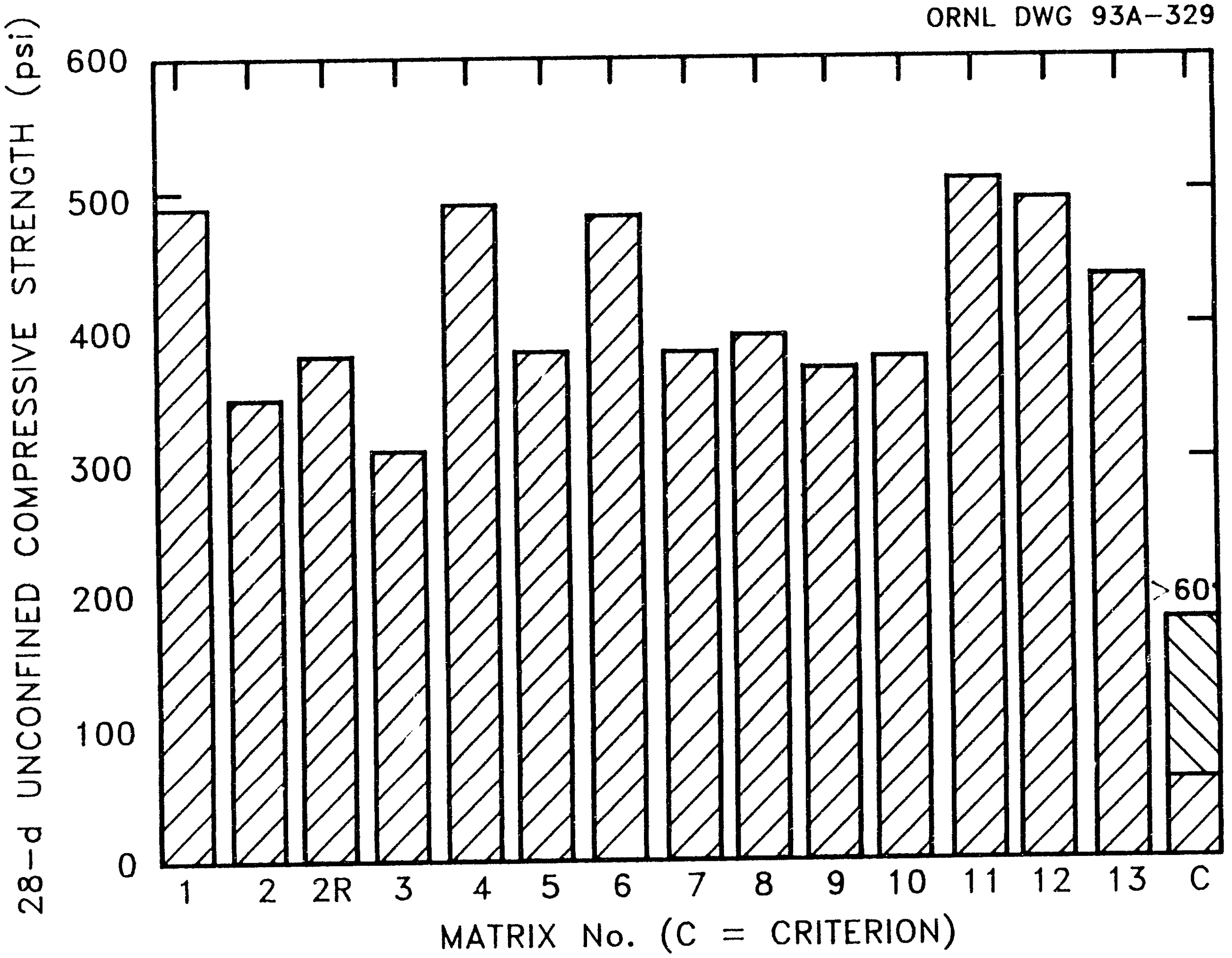


Fig. E.5. Average 28-d unconfined compressive strength for each matrix using the reference grout formulation.

Table E.1. Freestanding liquid of reference grouts cured at the simulated adiabatic temperature

Matrix No.	Freestanding liquid (vol %)					
	2 h	1 d	7 d	14 d	21 d	28 d
1-8	7.8	8.8	7.6	8.9	5.5[a]	11.2
2-3	11.2	12.6	13.1	15.4	14.1	18.5
3-2	8.8	9.8	10.1	11.2	12.0	12.1
4-1	11.4	12.8	12.2	15.1	16.4	15.7
5-8	10.4	11.0	10.2	11.3	12.0	11.8
6-7	10.8	12.4	12.1	13.1	12.6	13.2
S12	10.1	11.0	10.8	11.3	11.9	12.9
S15	10.6	12.0	11.6	12.3	12.5	13.2

[a] The discrepancy from the earlier reading was noted at the time of this reading and carefully reexamined prior to recording. The grout inside the graduated cylinder was in the process of breaking near the top and separating, leading to speculation that the "missing" liquid was hidden from view. Some of the other samples had similar water pockets at the bottom or in-between that were included in the observed vol % of freestanding liquid. This was the only sample that had an obvious discrepancy among its readings.

E.2 TWO-HOUR FREESTANDING LIQUID TESTS

The 2-h freestanding liquid was observed to be a good measure of the maximum observed freestanding liquid and 28-d freestanding liquid throughout this project. Thus, 2-h freestanding liquid tests provide an excellent screening tool for quickly evaluating the effect on freestanding liquid of changing the grout or its curing conditions. This appendix contains the 2-d freestanding liquid results for two such screening tests: (1) the effect of ball milling a given BFS and (2) generating a data base for different temperatures. Of course, any new wrinkle expected to have a delayed time effect will not be observed in a 2-h freestanding liquid test.

E.2.1 Ball milling of S-20

The suggestion that higher Blaine fineness improved the freestanding liquid behavior led to trying to improve the performance of one of the BFS via ball milling in the laboratory. The BFS with the lowest Blaine fineness was selected for this test. Table E.3 indicates that S-10 was the BFS with the lowest Blaine fineness. Since almost all of S-10 was used in the earlier tests, S-20, the new batch equivalent to S-10, was substituted. Different samples of S-20 were milled in a laboratory ball mill with ceramic balls for 1, 3, and 4 d. The fine slag caked on the container walls, so it is not clear how effective the milling was. The Blaine fineness of these materials was measured. The 2-h freestanding liquid was measured on dry blends—40 wt % P-65, 28 wt % P-62, 4 wt % C-88, and 28 wt % S-20—made up for each ball milling time interval (including the "as received" S-20), mixed with fresh simulated 106-AN preheated to 40°C, and placed in an oven at 50°C. Table E.2 lists the results. Results indicate ball milling was effective at increasing the Blaine fineness, but the effect on the 2-h freestanding liquid was mixed and not conclusive. The freestanding liquid was higher after 1 d of ball milling but lower than the original material after 3 d and 4 d of ball milling. The average for 4 d was slightly (but not significantly) less than for 3 d.

Table E.2. Effects of ball milling on the 2-h freestanding liquid of S-20

Milling time (d)	Blaine fineness (cm^2/g)	2-h freestanding liquid (vol %)
0	4490	10.6
		10.0
		11.2
1	4530	13.8
		13.6
		13.8
3	5420	8.4
		9.2
		8.8
4	5110	8.8
		8.8
		8.0

E.2.2 Isothermal database

The 2-h freestanding liquid was measured for the reference matrix grouts using C-90, P-65, P-62, and the various BFSs at different temperatures. (The BFSs used were S-18, S-19, S-16, S-22, S-20, S-23, S-21, S-13, S17, and S-15.) Table E.3 lists the results.

Table E.3. 2-h freestanding liquid at different temperatures

BFS	2-h freestanding liquid (vol %)						
	40°C	50°C	60°C	70°C	80°C	90°C	95°C
S-18	5.6	5.2	5.2	5.6	6.4	5.1	6.1
	4.0	4.4	4.0	4.8	4.8	5.9	6.3
	5.6	4.8	4.8	4.8	4.8	6.6	5.4
S-19	3.6[a]	5.6	4.8	4.0	4.8	6.6	7.0
	6.0	5.2	4.8	4.0	5.1	6.2	6.9
	5.6	5.6	4.8	4.4	4.4	5.1	7.8
S-16	5.6	4.8	4.4	4.0	4.3	5.1	7.3
	2.0[a]	5.6	4.8	4.8	4.4	5.1	8.1
	3.2[a]	4.8	4.8	4.4	4.7	6.3	7.0
S-22	4.8	6.0	5.6	5.2	4.4	7.3	6.6
	6.0	6.4	6.0	5.2	4.8	5.6	8.1
	6.8	6.0	6.0	5.2	5.2	6.3	6.2
S-20	7.1	6.0	6.8	6.0	4.8	6.2	8.8
	7.0	8.1	6.8	5.6	5.2	5.9	10.3
	7.4	7.6	7.2	6.4	5.2	8.6	5.1
S-23	7.2	6.8	8.8	5.2	5.2	9.8	7.7
	8.8	8.0	8.0	4.8	5.2	8.7	9.2
	7.7	7.2	6.8	5.6	5.2	10.2	7.0
S-21	5.2	4.0	1.2	5.6	4.0	4.7	7.6
	4.0	4.0	4.0	5.2	4.0	5.1	6.2
	4.4	4.0	3.6	5.2	4.4	6.3	6.6
S-13	5.6	4.8	4.8	4.8	4.4	7.0	5.1
	6.0	4.4	4.0	4.8	4.3	6.3	7.3
	4.8	4.4	5.0	5.2	4.4	6.3	6.6
S-17	6.4	5.6	5.6	5.6	6.0	5.6	8.1
	6.4	6.0	6.0	6.4	5.1	6.7	8.1
	6.4	6.4	5.2	5.6	5.2	6.8	8.0
S-15	5.6	6.0	5.6	4.4	4.4	5.5	7.3
	5.6	5.6	4.0	4.4	4.8	5.9	7.3
	6.0	5.6	5.2	3.6	5.6	5.9	6.3

[a] Hidden liquid was found in some samples at 40°C. These abnormally low values may result from unobserved hidden voids and liquid.

Appendix F

LEACHING DATA FOR NITRITE, SULFATE, AND THE TOTAL OF NITRATE AND NITRITE

Appendix F

LEACHING DATA FOR NITRITE, SULFATE, AND THE TOTAL OF NITRATE AND NITRITE

All of the leachate concentrations were measured by ion chromatography. The chromatograph was only calibrated for nitrate because only the nitrate leachability index measurements had been requested, but the chromatograms generated in measuring the nitrate concentrations contained at least two other peaks—nitrite and sulfate. After completing the nitrate analyses, the nitrite and sulfate concentrations were estimated by comparing the areas under these peaks with the areas generated by known concentrations of these two anions. This procedure is similar to how the chromatograph is calibrated, but the calibration for nitrite and sulfate was performed after all leachate analyses had been done and not before and during the analyses as the nitrate calibration was done. In other words, several nitrate calibrations were performed during the course of the analyses, and nitrate calibration checks were performed for every 10 to 20 analyses, but a generic calibration for nitrite and sulfate was performed for the entire batch of leachates after all the leachate chromatograms had been generated. Thus, the nitrite and sulfate data contain more error than the nitrate data. The total of nitrate and nitrite was obtained by simply adding the masses of these two anions. The total of these two is of interest because one is chemically convertible into the other, depending on the oxygen potential of the environment, and there was no way of telling whether the initial mass of either anion stayed as that anion or converted into the other anion.

In general, the estimated sulfate leachability indexes were lower than that of nitrate or nitrite. The estimated sulfate leachability indexes are likely too low because the dry blend is known to contribute some sulfate to the grout that was not added to the 106-AN sulfate in the process of estimating the leachability index. In other words, the sulfate driving force was higher than reported and the fraction leached at each interval lower. The amount of leachable sulfate added by the dry blend was unknown, but the estimated sulfate leachability index can be considered a conservative estimate; the true sulfate leachability index is an unknown amount higher than that reported here.

Sulfate concentrations were not obtained for all the leachates, and one set—MX 13-9—did not have quantifiable or detectable concentrations in all of the leachates. Since NEWBOX requires the cumulative amount leached, the sulfate data for MX 13-9 was analyzed using the differential technique of ANSI/ANS-16.1-1986 rather than the integral technique of NEWBOX. In other words, the sulfate leachability index of MX 13-9 was not estimated using NEWBOX, as all the other leachability indexes were but was estimated using the technique recommended by the standard ANSI/ANS-16.1-1986 procedure.

Nitrite Leaching Data
Matrix Run No.: MX1-8

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.81	3.84	3.76
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	23	20	22
Cylinder at start of leaching, Ao =	531	535	533

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	12.0	15.6	10.9
2	0.292	19.2	22.5	16.2
3	1	25.5	27.6	22.4
4	2	30.0	32.2	27.5
5	3	34.2	36.2	31.3
6	4	37.5	39.3	34.9
7	7	54.0	48.3	43.2

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	8	14	8
Std. Dev., mg =	3	2	1
Effective Diff. Coeff., sq. cm/s =	1.97E-09	1.30E-09	1.40E-09
Std. Dev., sq. cm/s =	5.14E-10	3.08E-10	1.66E-10
Leachability Index =	8.706	8.886	8.854
Upper 95% Conf. Int. =	0.127	0.114	0.053
Lower 95% Conf. Int. =	0.101	0.092	0.049

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.043	1.037	1.041
Rinse =	0.043	0.037	0.041
Ao =	1.000	1.000	1.000
Washoff =	0.015	0.026	0.015
Rinse + Washoff =	0.059	0.063	0.055
Leaching by diffusion control =	0.985	0.974	0.985

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.023	0.024	0.029	0.033	0.020	0.022
0.292	0.036	0.032	0.042	0.039	0.030	0.029
1	0.048	0.046	0.052	0.050	0.042	0.040
2	0.056	0.058	0.060	0.060	0.052	0.051
3	0.064	0.068	0.068	0.068	0.059	0.059
4	0.071	0.076	0.074	0.074	0.065	0.066
7	0.102	0.095	0.090	0.090	0.081	0.082

```
Nitrate + Nitrite Leaching Data
Matrix Run No.:   MX1-8

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm  NO3 + NO2 mass     1831 mg

Cured Cylinder =                          A          B          C
Length, cm =                             3.81       3.84       3.76
Diameter, cm =                           2.50       2.50       2.50

Nitrate + Nitrite Mass, mg
Rinse =                                    65         58         64
Cylinder at start of leaching, Ao =      1766       1773       1767

Cumulative Amount Leached, mg (nitrate + nitrite)

                    Interval   Time         A          B          C
                               days

                           1   0.083       34.9       47.3       35.8
                           2   0.292       56.4       68.1       54.4
                           3       1       76.5       85.6       75.7
                           4       2       92.7      102.1       93.9
                           5       3      106.7      116.6      107.7
                           6       4      118.1      127.9      120.9
                           7       7      158.7      159.7      150.0

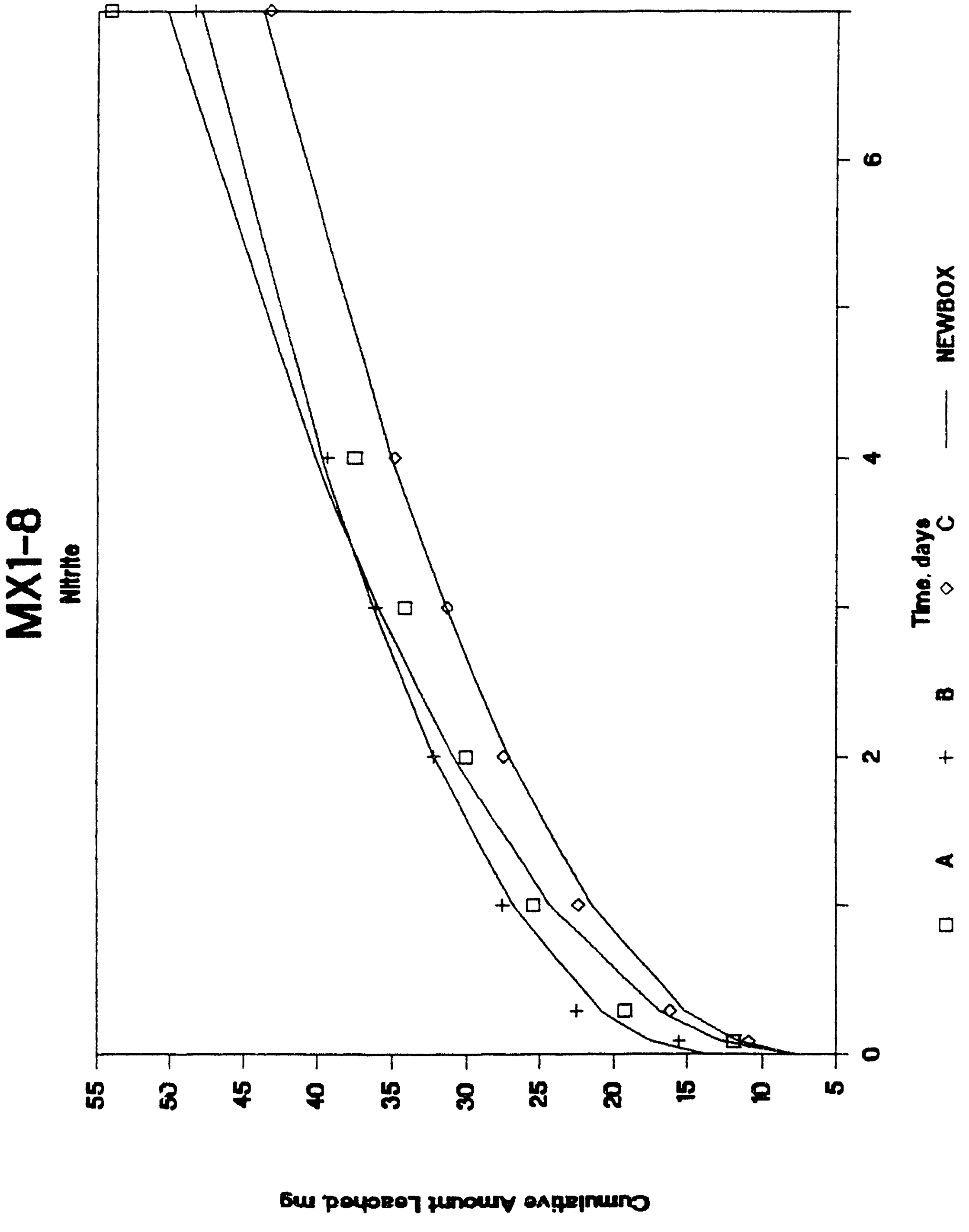
NEWBOX Estimate of Parameters             A          B          C

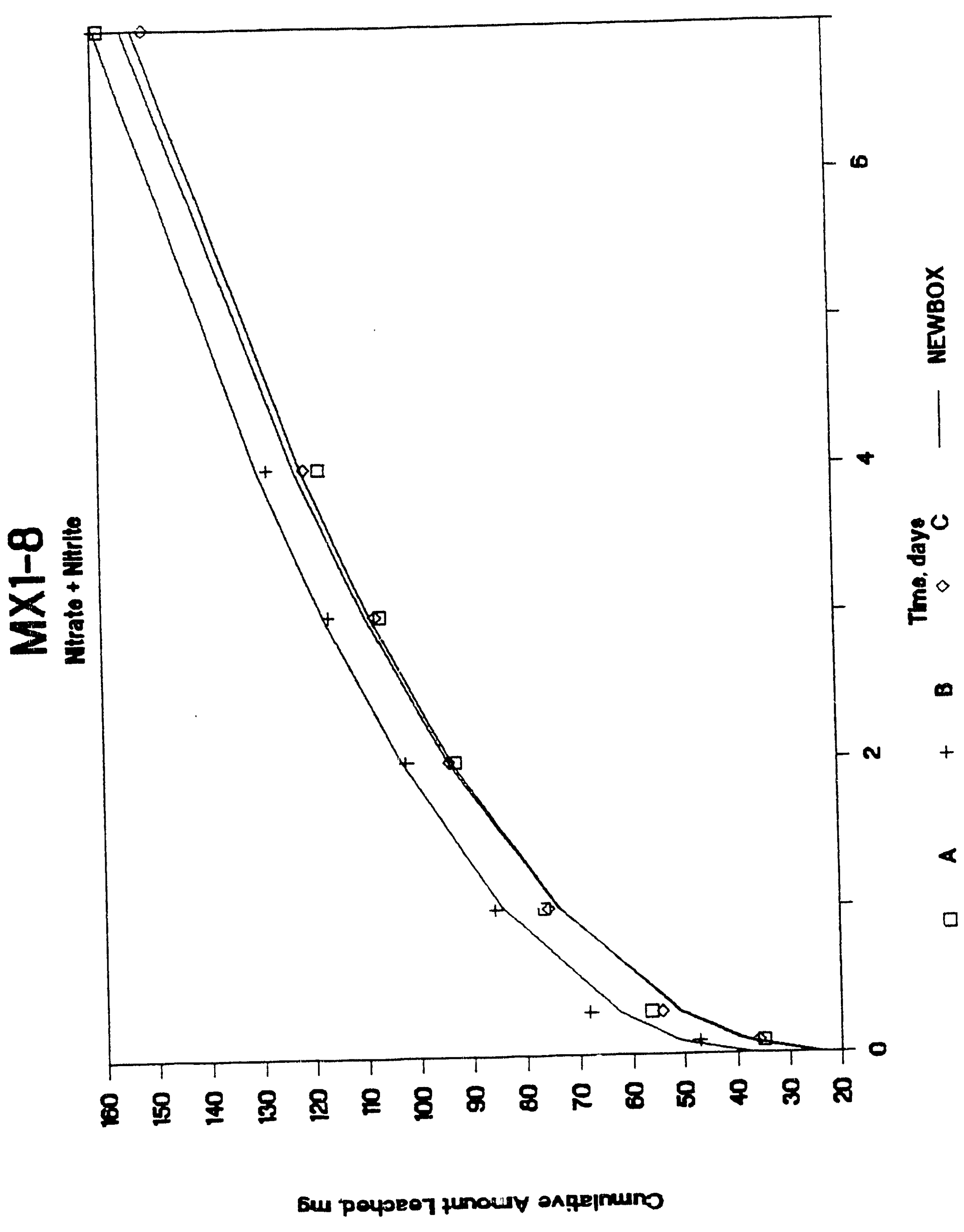
Washoff, mg =                              23         37         25
          Std. Dev., mg =                   6          5          4
Effective Diff. Coeff., sq. cm/s =  1.71E-09  1.51E-09  1.61E-09
          Std. Dev., sq. cm/s =     3.07E-10  2.50E-10  1.84E-10
Leachability Index =                    8.767      8.821      8.793
          Upper 95% Conf. Int. =        0.083      0.076      0.051
          Lower 95% Conf. Int. =        0.072      0.067      0.047

Cumulative Fraction Leached Based on Ao (CFL)
Initially in fresh grout =              1.037      1.033      1.036
Rinse =                                 0.037      0.033      0.036
Ao =                                    1.000      1.000      1.000
Washoff =                               0.013      0.021      0.014
Rinse + Washoff =                       0.050      0.054      0.050
Leaching by diffusion control =         0.987      0.979      0.986

    Time       CFL of A            CFL of B            CFL of C
    days    Obsv'd    Calc'd    Obsv'd    Calc'd    Obsv'd    Calc'd

   0.083     0.020     0.022     0.027     0.029     0.020     0.022
   0.292     0.032     0.029     0.038     0.035     0.031     0.029
       1     0.043     0.042     0.048     0.047     0.043     0.042
       2     0.053     0.053     0.058     0.058     0.053     0.053
       3     0.060     0.062     0.066     0.066     0.061     0.062
       4     0.067     0.070     0.072     0.073     0.068     0.069
       7     0.090     0.087     0.090     0.090     0.085     0.086
```


MX1-8
Nitrite
Cumulative Amount Leached, mg
55
50
45
40
35
30
25
20
15
10
5
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX1-8
Nitrate + Nitrite
Cumulative Amount Leached, mg
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX2-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	89	72	79
Cylinder at start of leaching, Ao =	465	483	476

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	27.7	32.4	17.5
2	0.292	38.3	38.3	23.5
3	1	52.2	45.9	31.6
4	2	66.6	60.3	41.5
5	3	80.1	65.3	50.3
6	4	84.5	69.7	57.5
7	7	101.8	82.3	69.8

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	19	26	11
Std. Dev., mg =	2	2	1
Effective Diff. Coeff., sq. cm/s =	1.29E-08	5.34E-09	5.35E-09
Std. Dev., sq. cm/s =	1.44E-09	8.01E-10	4.90E-10
Leachability Index =	7.889	8.272	8.272
Upper 95% Conf. Int. =	0.050	0.068	0.040
Lower 95% Conf. Int. =	0.046	0.061	0.038

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.192	1.149	1.166
Rinse =	0.192	0.149	0.166
Ao =	1.000	1.000	1.000
Washoff =	0.041	0.053	0.022
Rinse + Washoff =	0.233	0.202	0.188
Leaching by diffusion control =	0.959	0.947	0.978

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.060	0.062	0.067	0.067	0.037	0.036
0.292	0.082	0.080	0.079	0.078	0.049	0.049
1	0.112	0.113	0.095	0.100	0.066	0.070
2	0.143	0.142	0.125	0.118	0.087	0.090
3	0.172	0.164	0.135	0.133	0.106	0.105
4	0.182	0.182	0.144	0.145	0.121	0.117
7	0.219	0.225	0.171	0.173	0.147	0.146

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX2-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	183	161	170
Cylinder at start of leaching, Ao =	1648	1670	1661

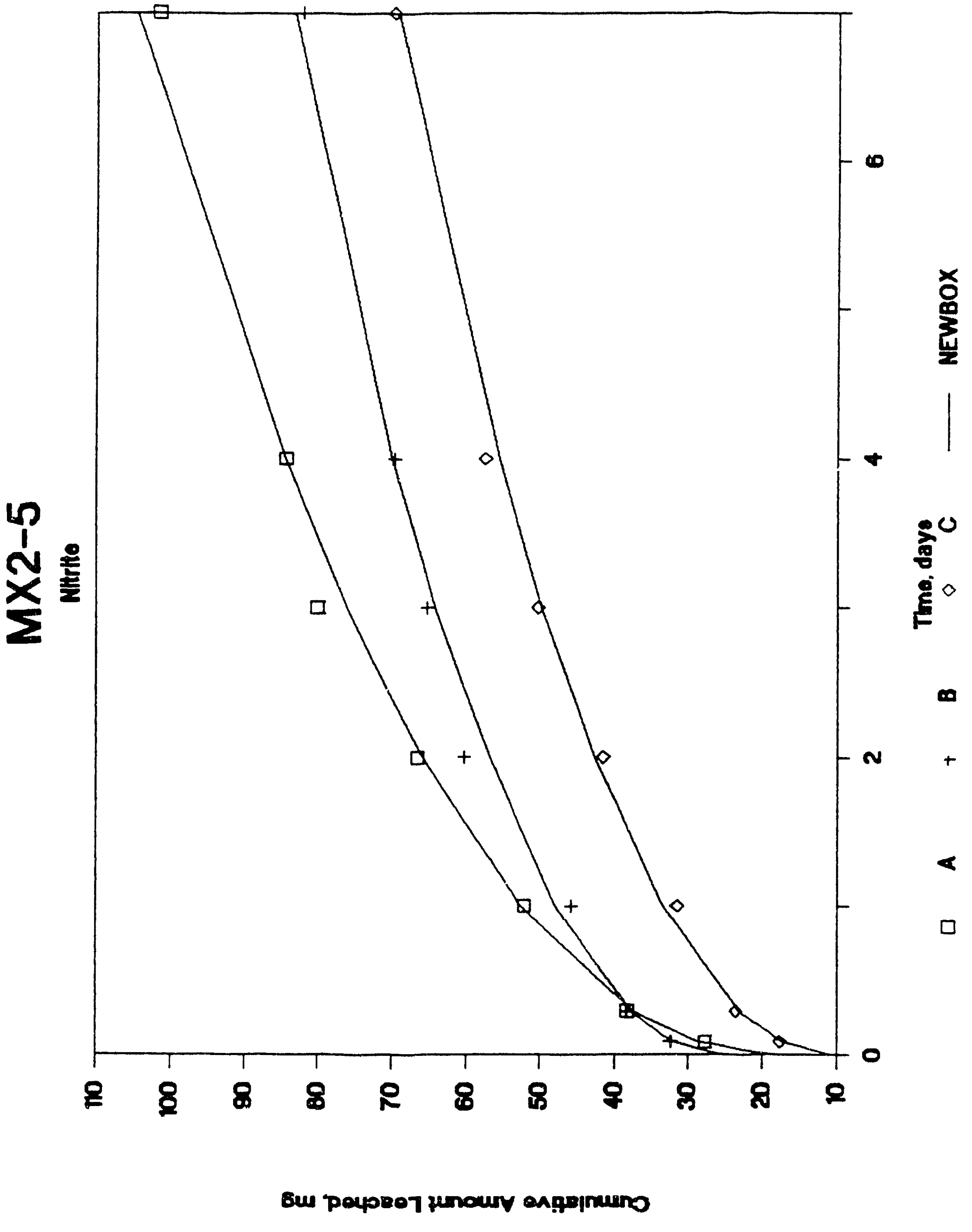
Cumulative Amount Leached, mg (nitrate + nitrite)

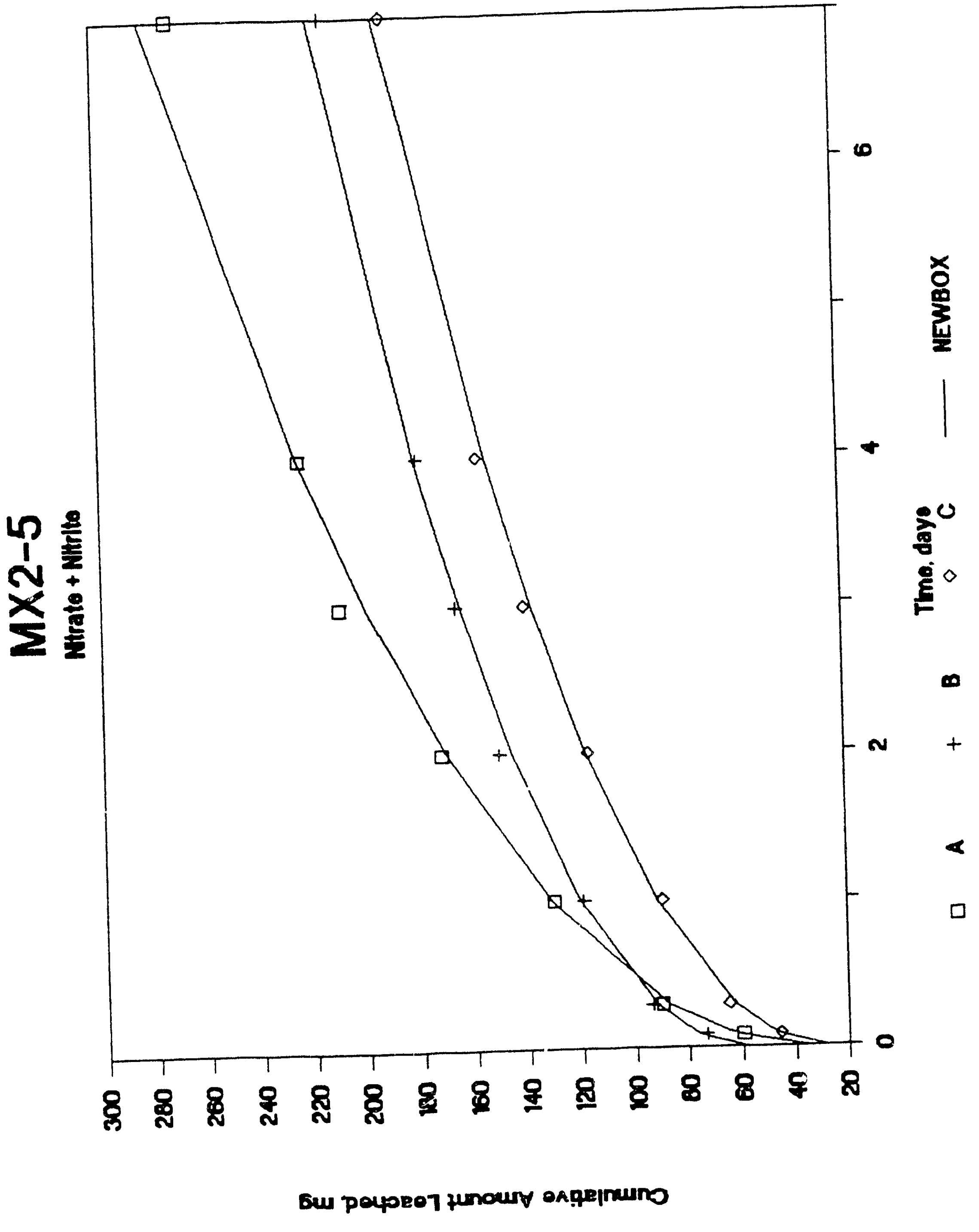
Interval	Time days	A	B	C
1	0.083	59.8	73.6	45.8
2	0.292	90.0	93.7	64.6
3	1	130.1	118.9	89.4
4	2	171.6	149.7	116.5
5	3	209.2	165.6	139.7
6	4	223.5	179.3	156.2
7	7	270.7	212.8	189.4

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	35	59	29
Std. Dev., mg =	7	5	3
Effective Diff. Coeff., sq. cm/s =	7.89E-09	3.16E-09	3.29E-09
Std. Dev., sq. cm/s =	9.43E-10	3.51E-10	2.80E-10
Leachability Index =	8.103	8.500	8.483
Upper 95% Conf. Int. =	0.054	0.050	0.037
Lower 95% Conf. Int. =	0.049	0.046	0.035

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.111	1.096	1.102
Rinse =	0.111	0.096	0.102
Ao =	1.000	1.000	1.000
Washoff =	0.021	0.035	0.017
Rinse + Washoff =	0.133	0.131	0.119
Leaching by diffusion control =	0.979	0.965	0.983

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.036	0.038	0.044	0.046	0.028	0.028
0.292	0.055	0.053	0.056	0.055	0.039	0.038
1	0.079	0.079	0.071	0.072	0.054	0.055
2	0.104	0.103	0.090	0.087	0.070	0.071
3	0.127	0.121	0.099	0.098	0.084	0.082
4	0.136	0.136	0.107	0.108	0.094	0.092
7	0.164	0.171	0.127	0.130	0.114	0.116


MX2-5
Nitrite
Cumulative Amount Leached, mg
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
☐ A
+ B
◇ C
—— NEWBOX


MX2-5
Nitrate + Nitrite
Cumulative Amount Leached, mg
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX3-2

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.70	3.84	3.75
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	41	76	47
Cylinder at start of leaching, Ao =	514	479	507

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	31.9	35.2	12.7
2	0.292	39.7	43.7	17.5
3	1	45.2	55.8	23.2
4	2	54.2	65.4	29.0
5	3	58.6	77.3	38.3
6	4	65.8	81.2	45.3
7	7	75.6	90.7	60.6

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	28	29	6
Std. Dev., mg =	2	3	4
Effective Diff. Coeff., sq. cm/s =	3.05E-09	6.90E-09	2.93E-09
Std. Dev., sq. cm/s =	5.00E-10	1.35E-09	9.35E-10
Leachability Index =	8.516	8.161	8.533
Upper 95% Conf. Int. =	0.075	0.092	0.161
Lower 95% Conf. Int. =	0.066	0.078	0.120

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.080	1.158	1.093
Rinse =	0.080	0.158	0.093
Ao =	1.000	1.000	1.000
Washoff =	0.054	0.061	0.013
Rinse + Washoff =	0.133	0.219	0.106
Leaching by diffusion control =	0.946	0.939	0.987

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.062	0.064	0.074	0.077	0.025	0.024
0.292	0.077	0.073	0.091	0.090	0.035	0.033
1	0.088	0.090	0.116	0.115	0.046	0.050
2	0.106	0.105	0.137	0.136	0.057	0.065
3	0.114	0.116	0.162	0.153	0.076	0.077
4	0.128	0.126	0.170	0.167	0.089	0.087
7	0.147	0.148	0.189	0.199	0.120	0.110

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX3-2

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	3.70	3.84	3.75
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	88	170	104
Cylinder at start of leaching, Ao =	1743	1661	1727

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	67.8	85.2	35.3
2	0.292	89.9	109.0	51.9
3	1	108.8	144.7	71.1
4	2	134.7	172.6	91.1
5	3	150.0	208.3	118.6
6	4	170.7	221.5	138.1
7	7	201.7	252.5	181.0

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	54	66	16
Std. Dev., mg =	5	9	7
Effective Diff. Coeff., sq. cm/s =	2.35E-09	4.86E-09	2.52E-09
Std. Dev., sq. cm/s =	2.97E-10	8.35E-10	5.14E-10
Leachability Index =	8.629	8.313	8.599
Upper 95% Conf. Int. =	0.057	0.079	0.096
Lower 95% Conf. Int. =	0.052	0.069	0.081

Cumulative Fraction Leached Based on Ao (CFL)	A	B	C
Initially in fresh grout =	1.050	1.102	1.060
Rinse =	0.050	0.102	0.060
Ao =	1.000	1.000	1.000
Washoff =	0.031	0.040	0.009
Rinse + Washoff =	0.081	0.142	0.070
Leaching by diffusion control =	0.969	0.960	0.991

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.039	0.040	0.051	0.053	0.020	0.019
0.292	0.052	0.049	0.066	0.065	0.030	0.028
1	0.062	0.064	0.087	0.086	0.041	0.044
2	0.077	0.077	0.104	0.105	0.053	0.058
3	0.086	0.088	0.125	0.119	0.069	0.069
4	0.098	0.096	0.133	0.131	0.080	0.078
7	0.116	0.117	0.152	0.159	0.105	0.100

Sulfate Leaching Data
Matrix Run No.: MX3-2

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	3.70	3.84	3.75
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	2	4	3
Cylinder at start of leaching, Ao =	46	43	44

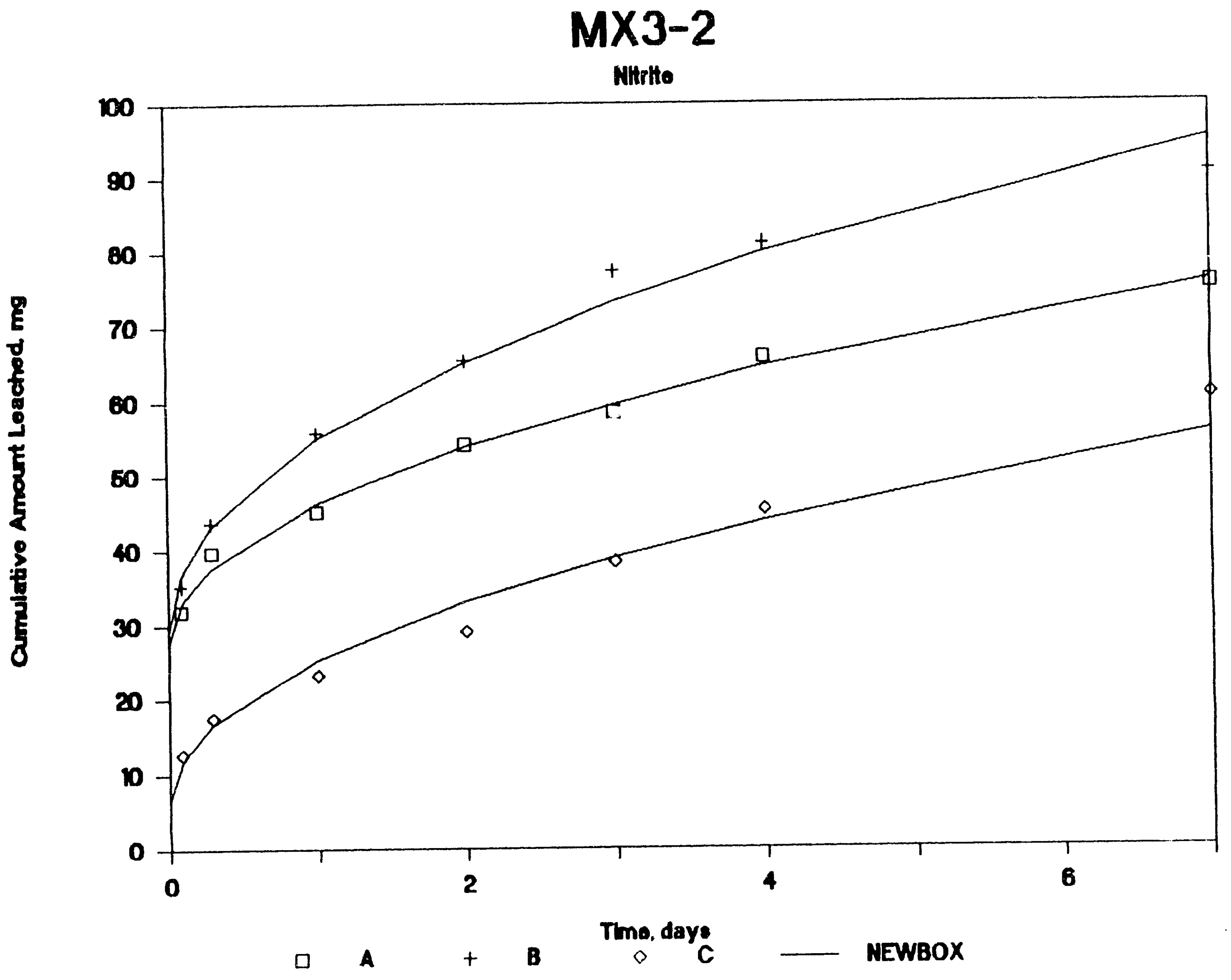
Cumulative Amount Leached, mg sulfate

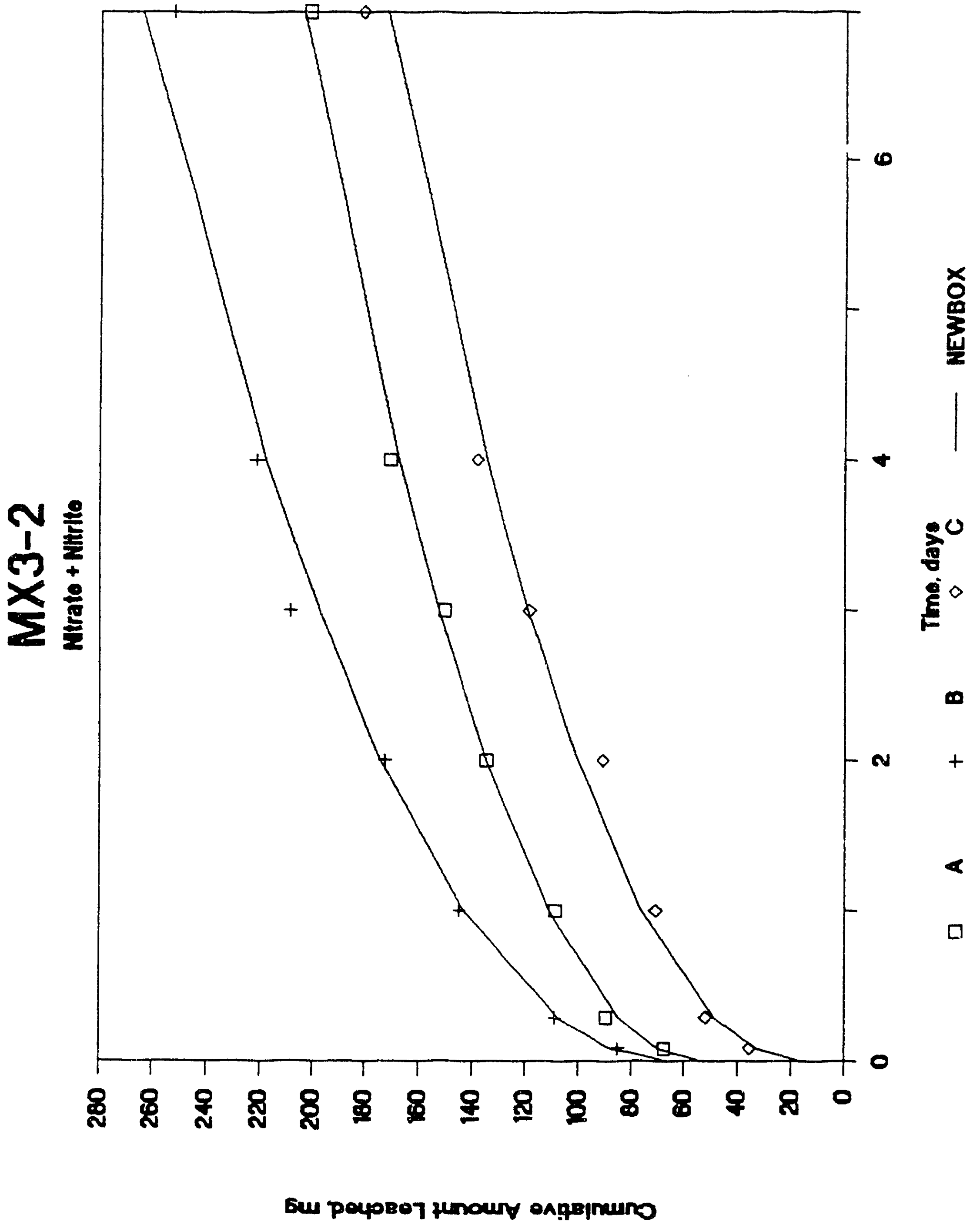
Interval	Time days	A	B	C
1	0.083	0.4	1.6	1.9
2	0.292	0.6	2.0	2.6
3	1	1.6	2.9	3.7
4	2	2.4	3.8	4.8
5	3	2.8	4.2	5.6
6	4	3.2	5.2	6.1
7	7	4.3	6.2	7.3

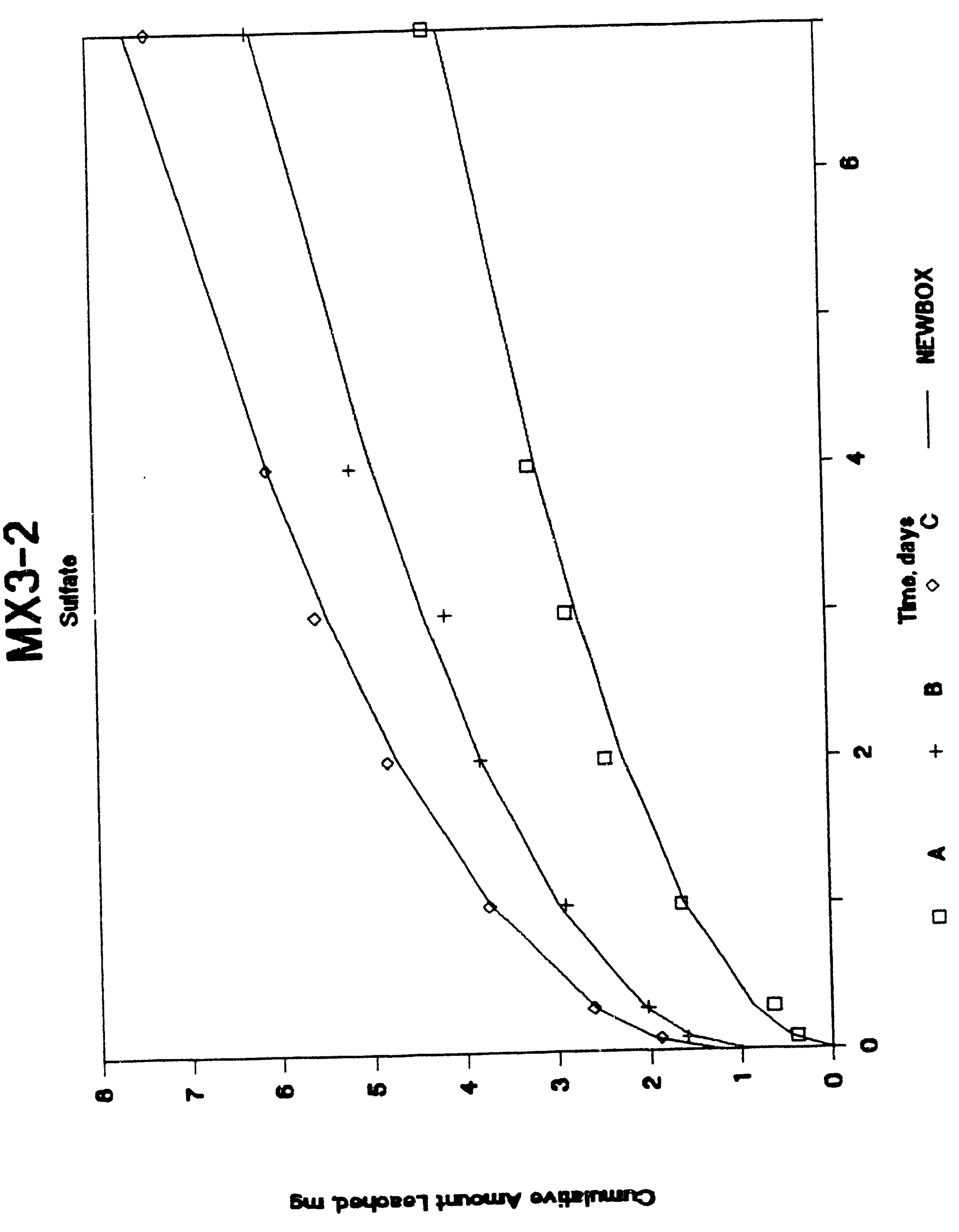
NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	0	1	1
Std. Dev., mg =	2	0	0
Effective Diff. Coeff., sq. cm/s =	2.45E-09	4.81E-09	6.86E-09
Std. Dev., sq. cm/s =	6.75E-10	6.73E-10	6.75E-10
Leachability Index =	8.611	8.318	8.164
Upper 95% Conf. Int. =	0.135	0.063	0.044
Lower 95% Conf. Int. =	0.106	0.057	0.041

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.033	1.096	1.076
Rinse =	0.033	0.096	0.076
Ao =	1.000	1.000	1.000
Washoff =	0.000	0.022	0.028
Rinse + Washoff =	0.033	0.117	0.104
Leaching by diffusion control =	1.000	0.978	0.972

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.009	0.010	0.037	0.036	0.043	0.045
0.292	0.014	0.019	0.047	0.047	0.059	0.059
1	0.036	0.035	0.067	0.069	0.085	0.084
2	0.053	0.049	0.088	0.088	0.110	0.107
3	0.062	0.060	0.097	0.102	0.127	0.124
4	0.071	0.069	0.120	0.114	0.138	0.138
7	0.094	0.090	0.144	0.143	0.167	0.172


MX3-2
Nitrite
Cumulative Amount Leached, mg
100
90
80
70
60
50
40
30
20
10
0
0
2
4
6
Time, days
□ A
+ B
◇ C
NEWBOX


MX3-2
Nitrate + Nitrite
Cumulative Amount Leached, mg
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
0
2
4
6
Time, days
☐ A
+ B
◇ C
—— NEWBOX


MX3-2
Sulfate
Cumulative Amount Leached, mg
0
1
2
3
4
5
6
7
8
Time, days
0
2
4
6
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX4-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.69	3.61	3.63
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	28	58	65
Cylinder at start of leaching, Ao =	526	497	490

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	14.4	14.6	32.6
2	0.292	18.9	19.2	37.2
3	1	25.2	24.9	43.1
4	2	30.0	31.4	47.9
5	3	33.8	35.0	54.5
6	4	36.4	38.0	61.9
7	7	43.0	44.4	68.7

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	11	12	28
Std. Dev., mg =	1	1	2
Effective Diff. Coeff., sq. cm/s =	1.22E-09	1.44E-09	2.34E-09
Std. Dev., sq. cm/s =	2.15E-10	1.92E-10	4.80E-10
Leachability Index =	8.914	8.842	8.631
Upper 95% Conf. Int. =	0.082	0.060	0.097
Lower 95% Conf. Int. =	0.070	0.054	0.081

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.053	1.117	1.132
Rinse =	0.053	0.117	0.132
Ao =	1.000	1.000	1.000
Washoff =	0.022	0.024	0.057
Rinse + Washoff =	0.075	0.141	0.189
Leaching by diffusion control =	0.978	0.976	0.943

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.027	0.029	0.029	0.031	0.066	0.066
0.292	0.036	0.035	0.039	0.038	0.076	0.074
1	0.048	0.046	0.050	0.050	0.088	0.089
2	0.057	0.056	0.063	0.061	0.098	0.102
3	0.064	0.063	0.070	0.069	0.111	0.112
4	0.069	0.069	0.077	0.076	0.126	0.121
7	0.082	0.085	0.089	0.092	0.140	0.141

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX4-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	3.69	3.61	3.63
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	74	120	130
Cylinder at start of leaching, Ao =	1757	1711	1701

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	40.0	39.9	65.0
2	0.292	55.5	56.4	80.9
3	1	76.4	76.4	101.0
4	2	93.0	95.0	117.9
5	3	106.0	107.2	135.5
6	4	114.9	117.6	157.8
7	7	137.8	139.2	181.1

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	31	30	51
Std. Dev., mg =	4	4	5
Effective Diff. Coeff., sq. cm/s =	1.24E-09	1.36E-09	1.88E-09
Std. Dev., sq. cm/s =	1.81E-10	2.05E-10	2.69E-10
Leachability Index =	8.907	8.866	8.726
Upper 95% Conf. Int. =	0.066	0.069	0.065
Lower 95% Conf. Int. =	0.059	0.061	0.058

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.042	1.070	1.076
Rinse =	0.042	0.070	0.076
Ao =	1.000	1.000	1.000
Washoff =	0.017	0.018	0.030
Rinse + Washoff =	0.060	0.088	0.106
Leaching by diffusion control =	0.983	0.982	0.970

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.023	0.025	0.023	0.025	0.038	0.039
0.292	0.032	0.031	0.033	0.032	0.048	0.046
1	0.043	0.042	0.045	0.043	0.059	0.060
2	0.053	0.052	0.056	0.054	0.069	0.072
3	0.060	0.059	0.063	0.062	0.080	0.081
4	0.065	0.066	0.069	0.069	0.093	0.089
7	0.078	0.081	0.081	0.085	0.106	0.107

Sulfate Leaching Data
Matrix Run No.: MX4-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	3.69	3.61	3.63
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	4	6	4
Cylinder at start of leaching, Ao =	44	41	43

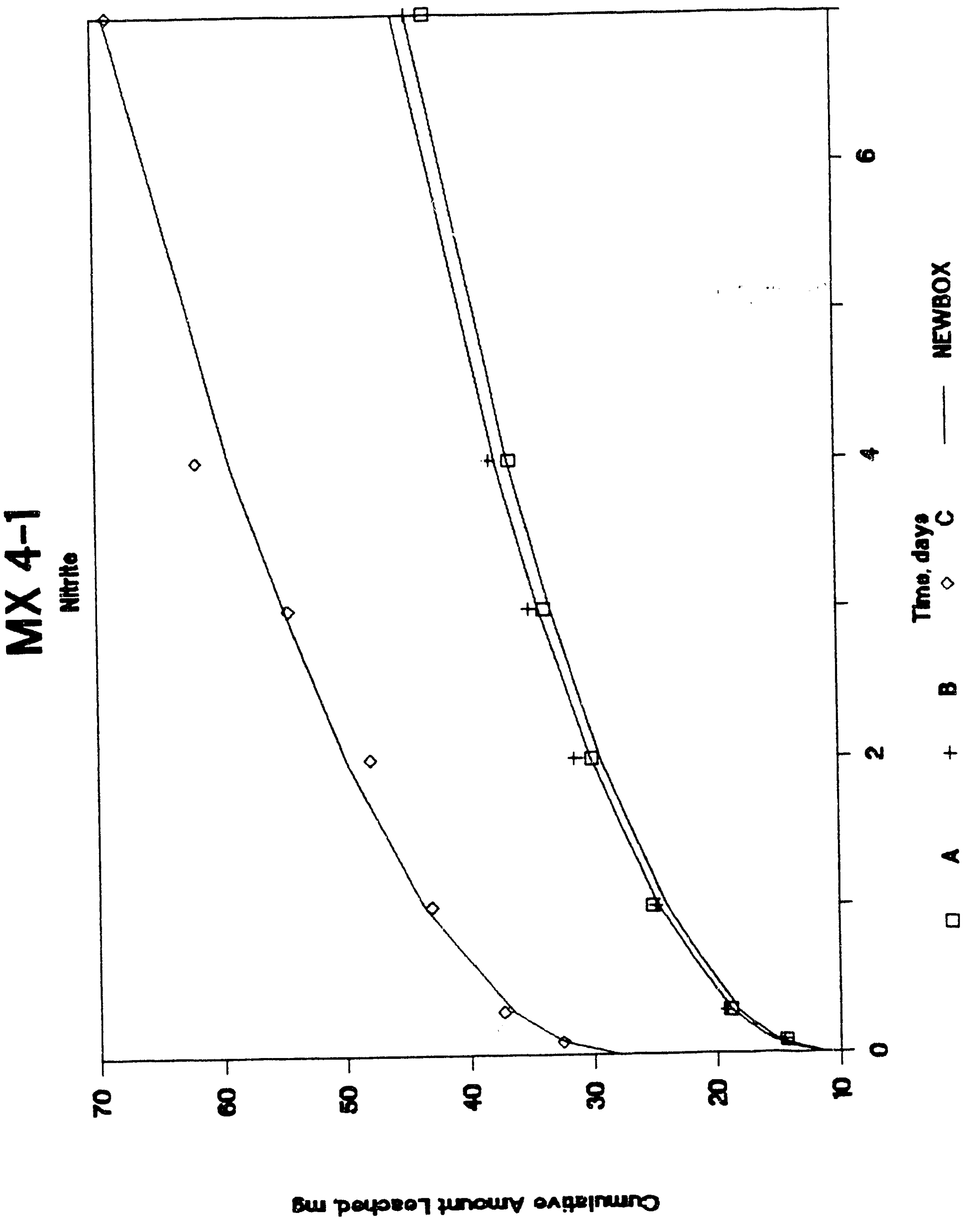
Cumulative Amount Leached, mg sulfate

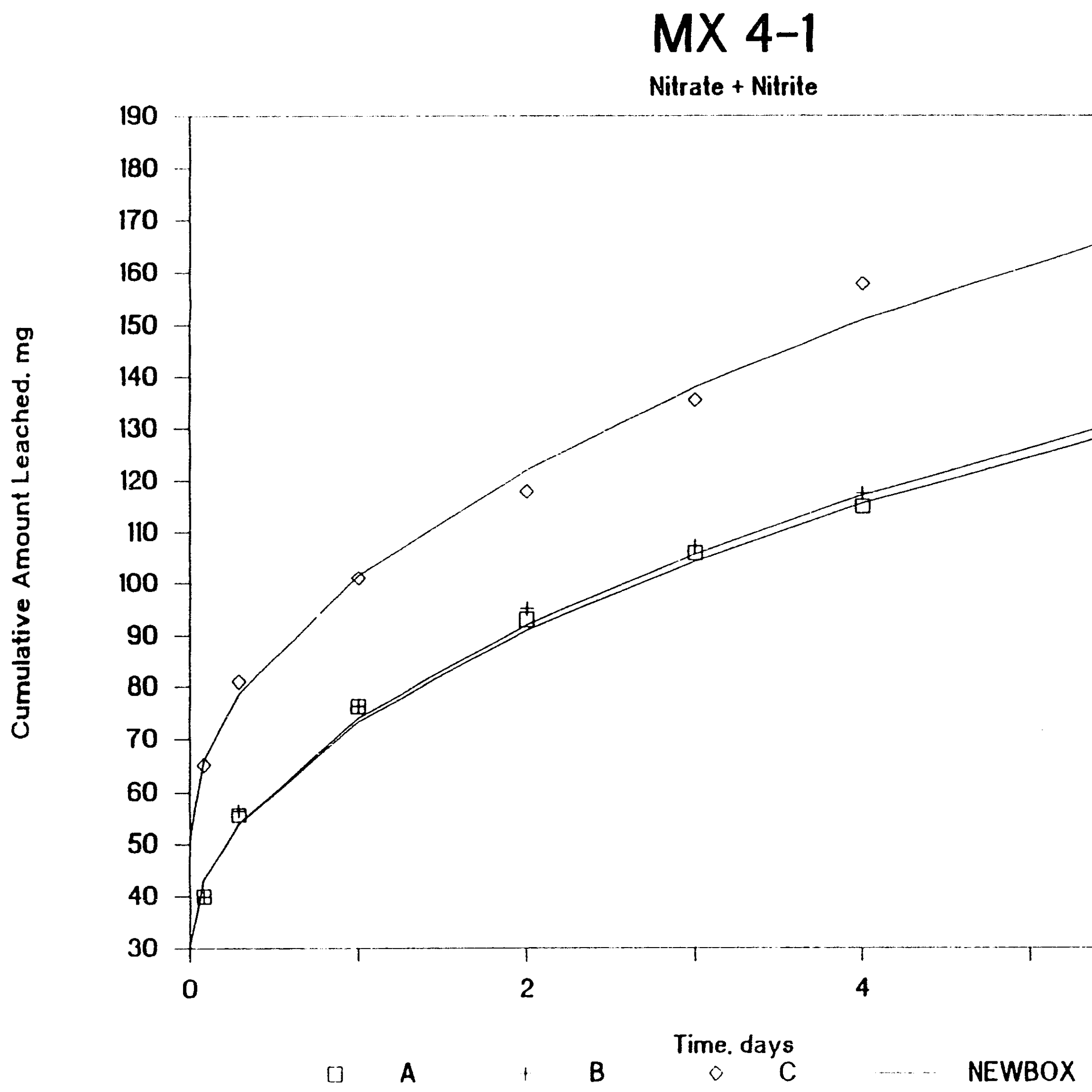
Interval	Time days	A	B	C
1	0.083	2.5	2.6	1.4
2	0.292	3.8	3.5	2.1
3	1	4.4	5.5	3.2
4	2	5.6	7.3	4.5
5	3	6.1	7.9	5.0
6	4	6.8	8.4	5.4
7	7	7.5	9.0	6.6

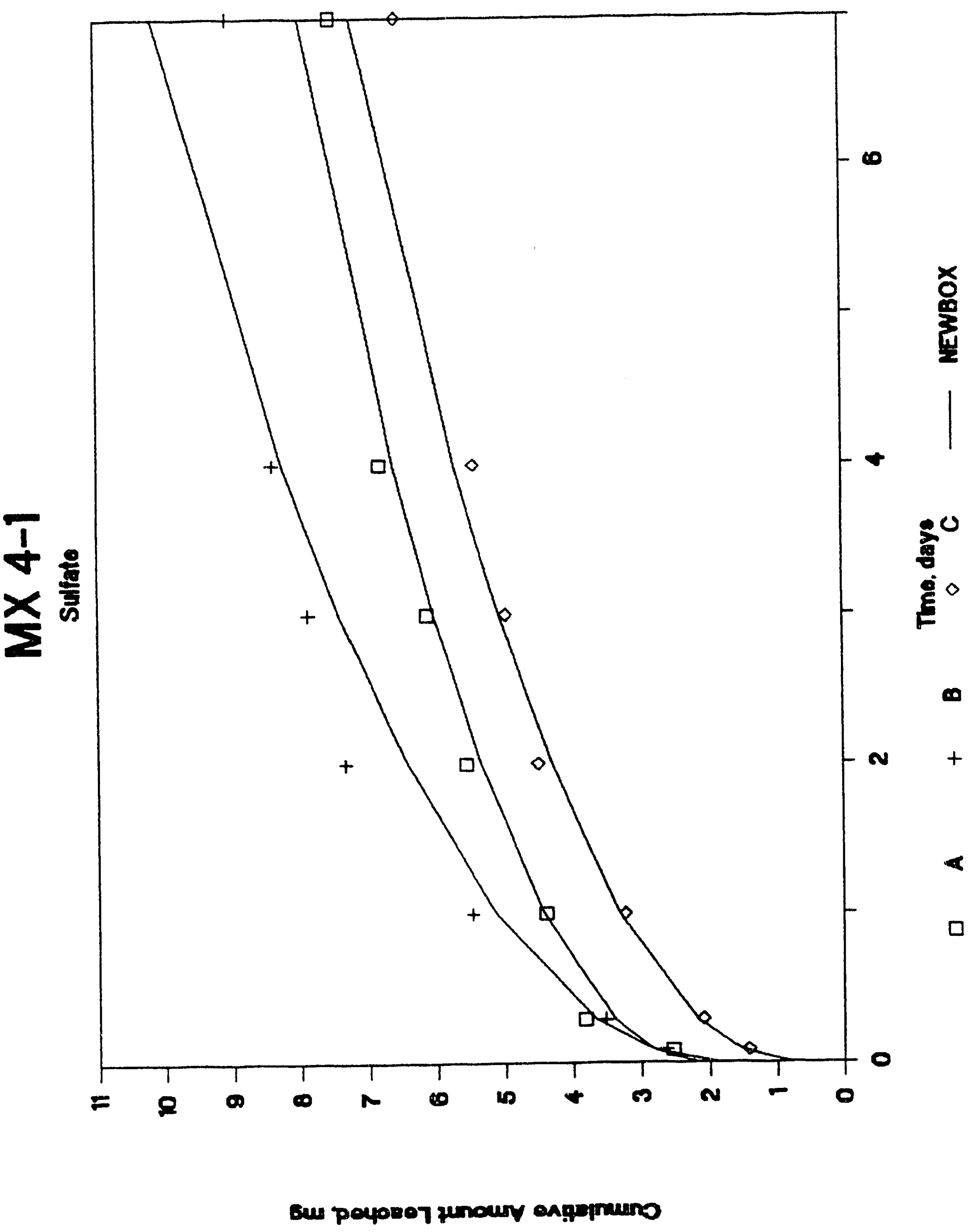
NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	2	2	1
Std. Dev., mg =	0	1	0
Effective Diff. Coeff., sq. cm/s =	6.07E-09	1.43E-08	7.14E-09
Std. Dev., sq. cm/s =	1.91E-09	5.27E-09	1.15E-09
Leachability Index =	8.217	7.845	8.146
Upper 95% Conf. Int. =	0.158	0.193	0.074
Lower 95% Conf. Int. =	0.119	0.136	0.065

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.082	1.148	1.102
Rinse =	0.082	0.148	0.102
Ao =	1.000	1.000	1.000
Washoff =	0.049	0.045	0.018
Rinse + Washoff =	0.131	0.193	0.120
Leaching by diffusion control =	0.951	0.955	0.982

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.058	0.064	0.064	0.069	0.033	0.036
0.292	0.087	0.077	0.085	0.089	0.049	0.051
1	0.100	0.101	0.133	0.124	0.075	0.077
2	0.127	0.122	0.178	0.156	0.105	0.100
3	0.140	0.138	0.191	0.180	0.116	0.118
4	0.156	0.151	0.204	0.200	0.126	0.133
7	0.172	0.182	0.219	0.246	0.153	0.168


MX 4-1
Nitrite
Cumulative Amount Leached, mg
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
— NEWBOX


MX 4-1
Nitrate + Nitrite
Cumulative Amount Leached, mg
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
0
2
4
6
Time, days
□ A
+ B
◇ C
NEWBOX


MX 4-1
Sulfate
Cumulative Amount Leached, mg
0
1
2
3
4
5
6
7
8
9
10
11
Time, days
0
2
4
6
□ A
+ B
◇ C
—— NEWBOX

```
Nitrite Leaching Data
Matrix Run No.:   MX5-8

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm  Nitrite mass =    554.5 mg

Cured Cylinder =                             A        B        C
Length, cm =                              4.33     4.30     4.35
Diameter, cm =                            2.50     2.50     2.50

Nitrite Mass, mg
Rinse =                                     61       62       59
Cylinder at start of leaching, Ao =        493      493      496

Cumulative Amount Leached, mg nitrite

                  Interval   Time          A        B        C
                             days

                         1 0.083333 16.62471 14.78136 12.74980
                         2 0.291666 21.88431 19.07934 17.08623
                         3        1          24.47578 22.58373
                           1.291666 36.99252
                         4        2          30.18171 27.90091
                           2.291666 41.64726
                         5        3          35.72200 32.08341
                           3.291666 45.81111
                         6        4          40.42100 35.56222
                           6.291666 56.90448
                         7        7          54.64878 52.22185
NEWBOX Estimate of Parameters                A        B        C

Washoff, mg =                               12       10        8
         Std. Dev., mg =                     3        3        3
Effective Diff. Coeff., sq. cm/s =  3.48E-09 2.34E-09 2.05E-09
         Std. Dev., sq. cm/s =      7.63E-10 5.75E-10 5.50E-10
Leachability Index =                     8.458    8.631    8.688
         Upper 95% Conf. Int. =          0.114    0.118    0.131
         Lower 95% Conf. Int. =          0.090    0.093    0.101

Cumulative Fraction Leached Based on Ao (CFL)
Initially in fresh grout =               1.124    1.125    1.119
Rinse =                                  0.124    0.125    0.119
Ao =                                     1.000    1.000    1.000
Washoff =                                0.025    0.020    0.017
Rinse + Washoff =                        0.149    0.145    0.135
Leaching by diffusion control =          0.975    0.980    0.983

    Time         CFL of A           CFL of B           CFL of C
    days     Obsv'd   Calc'd    Obsv'd   Calc'd    Obsv'd   Calc'd

0.083333     0.034    0.036     0.030    0.029     0.026    0.025
0.291666     0.044    0.046     0.039    0.037     0.034    0.033
       1                        0.050    0.052     0.046    0.047
1.291666     0.075    0.069
       2                        0.061    0.065     0.056    0.059
2.291666     0.084    0.083
       3                        0.072    0.075     0.065    0.068
3.291666     0.093    0.094
       4                        0.082    0.083     0.072    0.076
6.291666     0.115    0.120
       7                        0.111    0.103     0.105    0.095
```

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX5-8

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3+NO2 mass = 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50
Nitrite Mass, mg			
Rinse =	195	169	147
Cylinder at start of leaching, Ao =	1636	1662	1684

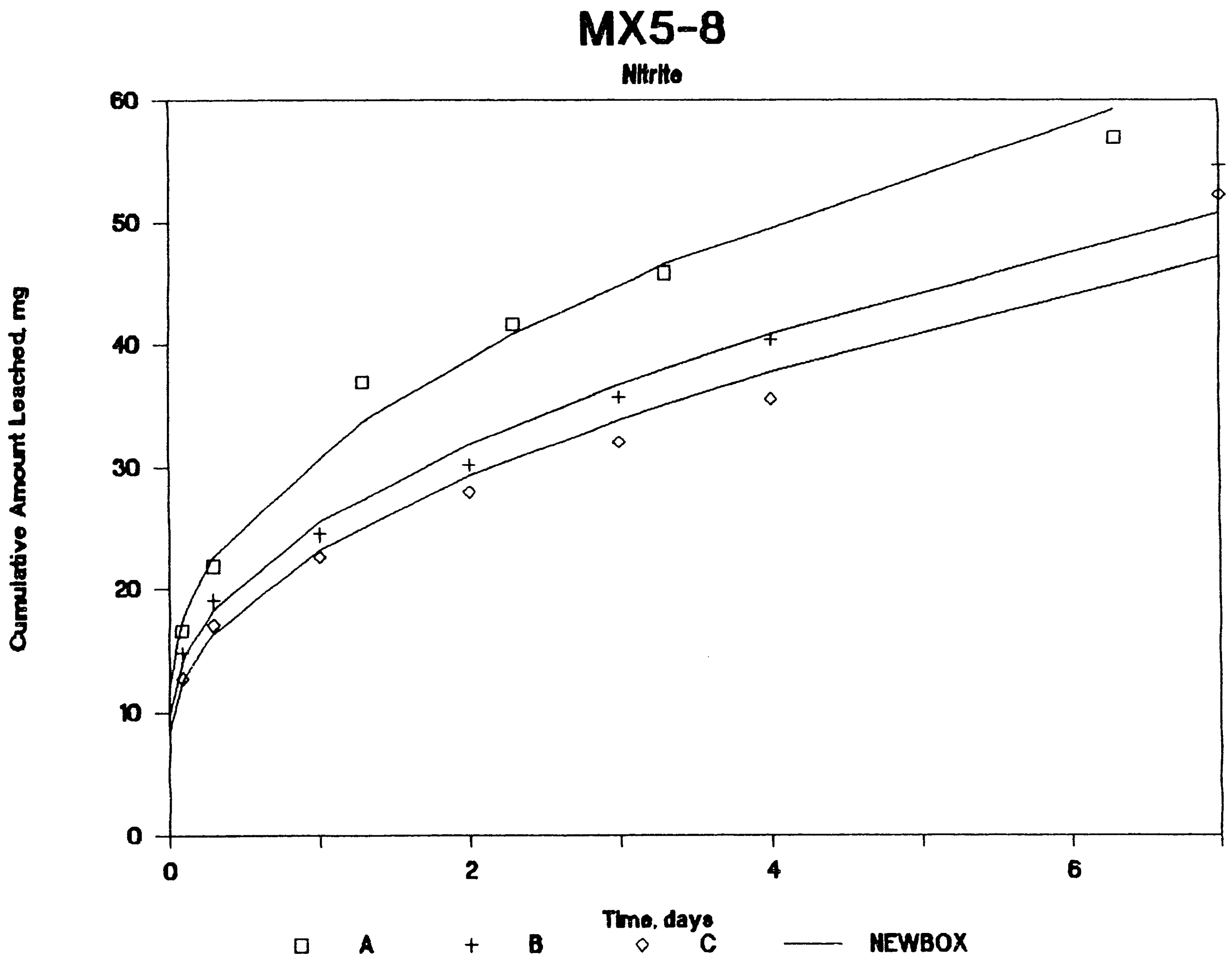
Cumulative Amount Leached, mg nitrite

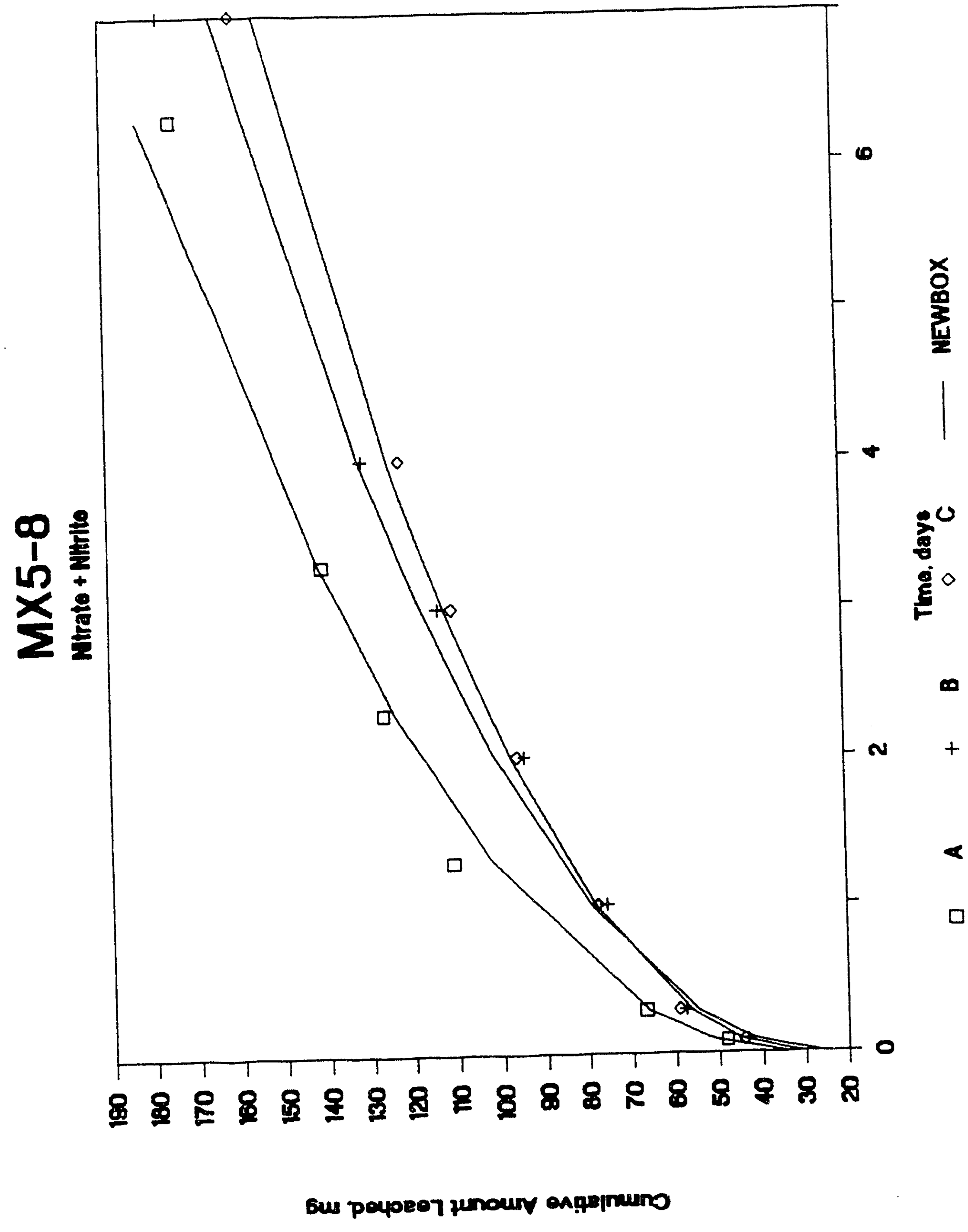
Interval	Time days	A	B	C
1	0.083333	48.07712	43.32846	43.97560
2	0.291666	66.96785	57.73931	59.13550
3	1		75.63300	77.80062
	1.291666	110.7496		
4	2		94.29388	96.06991
	2.291666	126.3925		
5	3		113.7263	110.4161
	3.291666	140.2910		
6	4		130.7743	121.6706
	6.291666	173.9612		
7	7		176.2169	159.8057

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	34	26	31
Std. Dev., mg =	8	7	4
Effective Diff. Coeff., sq. cm/s =	3.04E-09	2.31E-09	1.80E-09
Std. Dev., sq. cm/s =	6.53E-10	4.85E-10	2.12E-10
Leachability Index =	8.517	8.636	8.745
Upper 95% Conf. Int. =	0.111	0.099	0.053
Lower 95% Conf. Int. =	0.088	0.081	0.047

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.119	1.102	1.087
Rinse =	0.119	0.102	0.087
Ao =	1.000	1.000	1.000
Washoff =	0.021	0.016	0.018
Rinse + Washoff =	0.140	0.118	0.105
Leaching by diffusion control =	0.979	0.984	0.982

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083333	0.029	0.031	0.026	0.025	0.026	0.027
0.291666	0.041	0.041	0.035	0.033	0.035	0.034
1			0.046	0.048	0.046	0.046
1.291666	0.068	0.062				
2			0.057	0.061	0.057	0.058
2.291666	0.077	0.076				
3			0.068	0.071	0.066	0.067
3.291666	0.086	0.086				
4			0.079	0.079	0.072	0.074
6.291666	0.106	0.111				
7			0.106	0.099	0.095	0.092


MX5-8
Nitrite
Cumulative Amount Leached, mg
60
50
40
30
20
10
0
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX5-8
Nitrate + Nitrite
Cumulative Amount Leached, mg
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX6-7

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.71	3.70	3.59
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	40	85	101
Cylinder at start of leaching, Ao =	515	470	453

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	12.7	15.9	9.4
2	0.292	17.9	21.6	13.7
3	1	25.1	26.9	19.6
4	2	31.2	33.9	25.1
5	3	35.7	37.9	29.5
6	4	39.0	40.6	32.0
7	7	48.3	51.3	42.4

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	9	13	6
Std. Dev., mg =	1	1	1
Effective Diff. Coeff., sq. cm/s =	1.84E-09	2.11E-09	1.90E-09
Std. Dev., sq. cm/s =	1.69E-10	2.88E-10	2.22E-10
Leachability Index =	8.735	8.676	8.721
Upper 95% Conf. Int. =	0.041	0.062	0.052
Lower 95% Conf. Int. =	0.038	0.056	0.048

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.077	1.181	1.223
Rinse =	0.077	0.181	0.223
Ao =	1.000	1.000	1.000
Washoff =	0.018	0.027	0.012
Rinse + Washoff =	0.095	0.207	0.235
Leaching by diffusion control =	0.982	0.973	0.988

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.025	0.027	0.034	0.036	0.021	0.021
0.292	0.035	0.034	0.046	0.044	0.030	0.029
1	0.049	0.048	0.057	0.058	0.043	0.043
2	0.061	0.060	0.072	0.071	0.055	0.055
3	0.069	0.069	0.081	0.081	0.065	0.065
4	0.076	0.077	0.086	0.089	0.071	0.073
7	0.094	0.095	0.109	0.108	0.094	0.092

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX6-7

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	3.71	3.70	3.59
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	108	184	196
Cylinder at start of leaching, Ao =	1723	1647	1635

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	39.0	45.2	32.1
2	0.292	56.0	63.8	45.8
3	1	80.3	81.9	65.0
4	2	100.1	104.1	82.9
5	3	114.5	118.6	98.0
6	4	126.8	129.6	108.1
7	7	152.7	160.7	139.5

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	27	33	20
Std. Dev., mg =	4	4	2
Effective Diff. Coeff., sq. cm/s =	1.78E-09	1.91E-09	1.57E-09
Std. Dev., sq. cm/s =	2.11E-10	2.15E-10	9.15E-11
Leachability Index =	8.750	8.719	8.804
Upper 95% Conf. Int. =	0.053	0.050	0.025
Lower 95% Conf. Int. =	0.049	0.046	0.025

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.063	1.111	1.120
Rinse =	0.063	0.111	0.120
Ao =	1.000	1.000	1.000
Washoff =	0.016	0.020	0.012
Rinse + Washoff =	0.078	0.132	0.132
Leaching by diffusion control =	0.984	0.980	0.988

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.023	0.024	0.027	0.029	0.020	0.020
0.292	0.032	0.032	0.039	0.036	0.028	0.027
1	0.047	0.045	0.050	0.050	0.040	0.040
2	0.058	0.057	0.063	0.063	0.051	0.051
3	0.066	0.066	0.072	0.072	0.060	0.060
4	0.074	0.073	0.079	0.080	0.066	0.067
7	0.089	0.092	0.098	0.098	0.085	0.084

```
Sulfate Leaching Data
Matrix Run No.:    MX6-7

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm  Sulfate mass =     47.4 mg

Cured Cylinder =                          A         B         C
Length, cm =                              3.71      3.70      3.59
Diameter, cm =                            2.50      2.50      2.50

Sulfate Mass, mg
Rinse =                                      0         5         0
Cylinder at start of leaching, Ao =         47        42        47

Cumulative Amount Leached, mg sulfate

                  Interval    Time        A         B         C
                              days

                         1    0.083      0.0       0.7       0.0
                         2    0.292      0.0       1.0       0.0
                         3        1      0.0       1.5       0.0
                         4        2      0.0       1.8       0.0
                         5        3      0.0       2.5       0.0
                         6        4      0.0       3.3       0.0
                         7        7      0.0       4.1       0.0

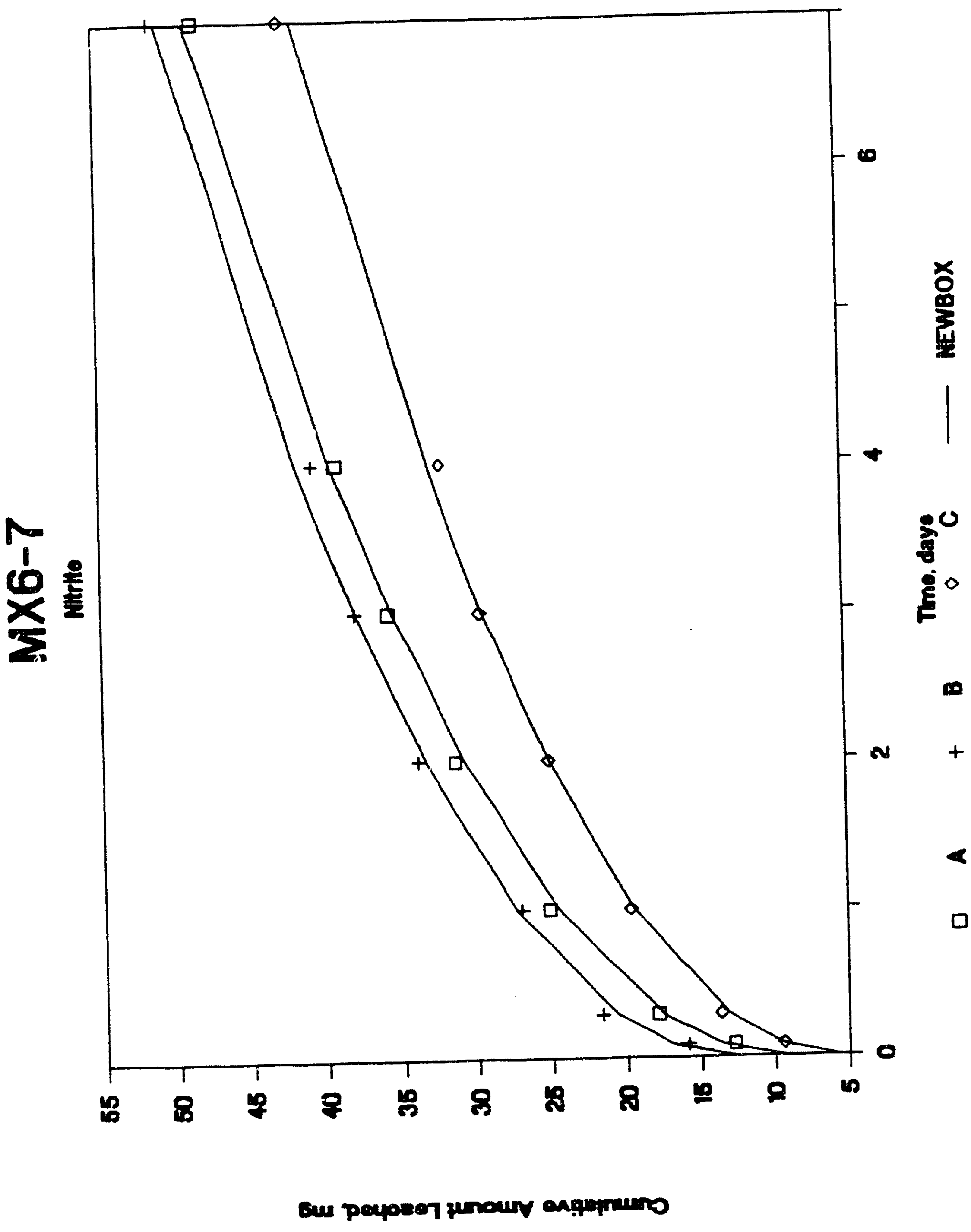
NEWBOX Estimate of Parameters             A         B         C

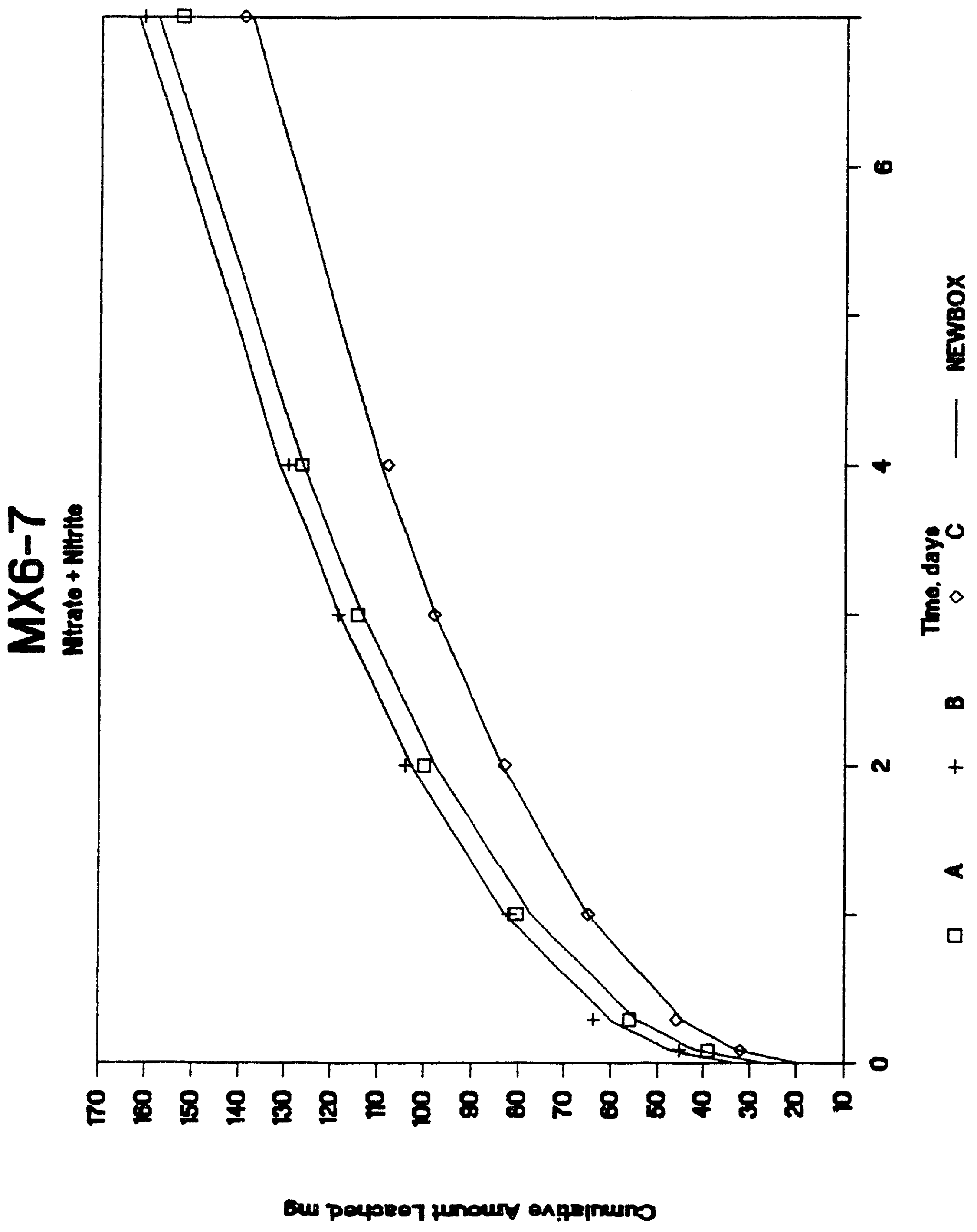
Washoff, mg =                                0         0         0
         Std. Dev., mg =                     0         0         0
Effective Diff. Coeff., sq. cm/s =   0.00E+00  2.24E-09  0.00E+00
         Std. Dev., sq. cm/s =       0.00E+00  7.63E-10  0.00E+00
Leachability Index =                       ERR     8.650       ERR
         Upper 95% Conf. Int. =            ERR     0.175       ERR
         Lower 95% Conf. Int. =            ERR     0.127       ERR

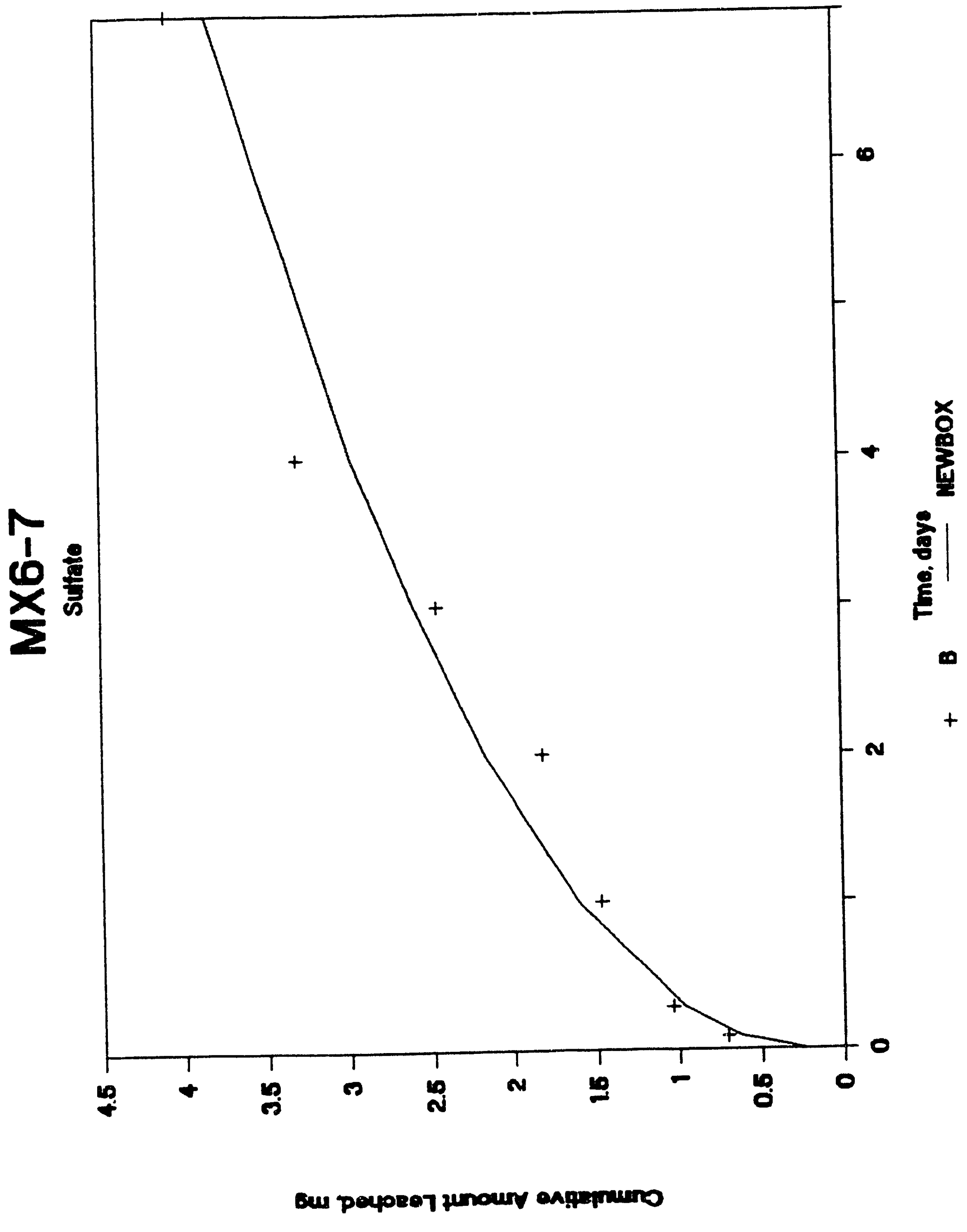
Cumulative Fraction Leached Based on Ao (CFL)
Initially in fresh grout =               1.000     1.127     1.000
Rinse =                                  0.000     0.127     0.000
Ao =                                     1.000     1.000     1.000
Washoff =                                0.000     0.005     0.000
Rinse + Washoff =                        0.000     0.132     0.000
Leaching by diffusion control =          1.000     0.995     1.000

    Time       CFL of A            CFL of B            CFL of C
    days    Obsv'd    Calc'd    Obsv'd    Calc'd    Obsv'd    Calc'd

   0.083     0.000     0.000     0.017     0.015     0.000     0.000
   0.292     0.000     0.000     0.025     0.023     0.000     0.000
       1     0.000     0.000     0.035     0.038     0.000     0.000
       2     0.000     0.000     0.043     0.052     0.000     0.000
       3     0.000     0.000     0.059     0.062     0.000     0.000
       4     0.000     0.000     0.078     0.071     0.000     0.000
       7     0.000     0.000     0.097     0.091     0.000     0.000
```


MX6-7
Nitrite
Cumulative Amount Leached, mg
55
50
45
40
35
30
25
20
15
10
5
Time, days
0
2
4
6
□ A
+ B
◇ C
— NEWBOX


MX6-7
Nitrate + Nitrite
Cumulative Amount Leached, mg
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX6-7
Sulfate
Cumulative Amount Leached, mg
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0
2
4
6
Time, days
+ B
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX7-3

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.41	4.41	4.42
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	23	22	22
Cylinder at start of leaching, Ao =	532	533	533

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	8.9	8.4	7.5
2	0.292	15.0	13.8	13.7
3	1	28.6	23.6	27.7
4	2	34.9	33.7	40.2
5	3	41.8	38.6	48.6
6	4	47.5	43.8	53.1
7	7	64.8	60.3	69.7

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	2	2	0
Std. Dev., mg =	2	1	2
Effective Diff. Coeff., sq. cm/s =	4.49E-09	3.86E-09	5.93E-09
Std. Dev., sq. cm/s =	5.65E-10	2.74E-10	6.32E-10
Leachability Index =	8.348	8.413	8.227
Upper 95% Conf. Int. =	0.057	0.031	0.047
Lower 95% Conf. Int. =	0.051	0.030	0.044

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.043	1.040	1.041
Rinse =	0.043	0.040	0.041
Ao =	1.000	1.000	1.000
Washoff =	0.004	0.003	0.000
Rinse + Washoff =	0.047	0.044	0.041
Leaching by diffusion control =	0.996	0.997	1.000

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.017	0.017	0.016	0.015	0.014	0.015
0.292	0.028	0.029	0.026	0.026	0.026	0.028
1	0.054	0.049	0.044	0.045	0.052	0.052
2	0.066	0.067	0.063	0.062	0.075	0.072
3	0.079	0.081	0.073	0.075	0.091	0.088
4	0.089	0.093	0.082	0.085	0.100	0.101
7	0.122	0.120	0.113	0.111	0.131	0.133

```
Nitrate + Nitrite Leaching Data
Matrix Run No.:   MX7-3

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm  NO3 + NO2 mass     1831 mg

Cured Cylinder =                         A        B        C
Length, cm =                           4.41     4.41     4.42
Diameter, cm =                         2.50     2.50     2.50

Nitrate + Nitrite Mass, mg
Rinse =                                  70       77       68
Cylinder at start of leaching, Ao =    1761     1754     1763

Cumulative Amount Leached, mg (nitrate + nitrite)

                  Interval   Time        A        B        C
                             days

                         1   0.083     28.7     28.0     24.8
                         2   0.292     47.4     48.4     45.0
                         3       1     90.8     85.7     86.2
                         4       2    116.8    121.6    124.2
                         5       3    139.6    141.8    152.6
                         6       4    159.5    162.8    171.3
                         7       7    209.2    215.4    221.6

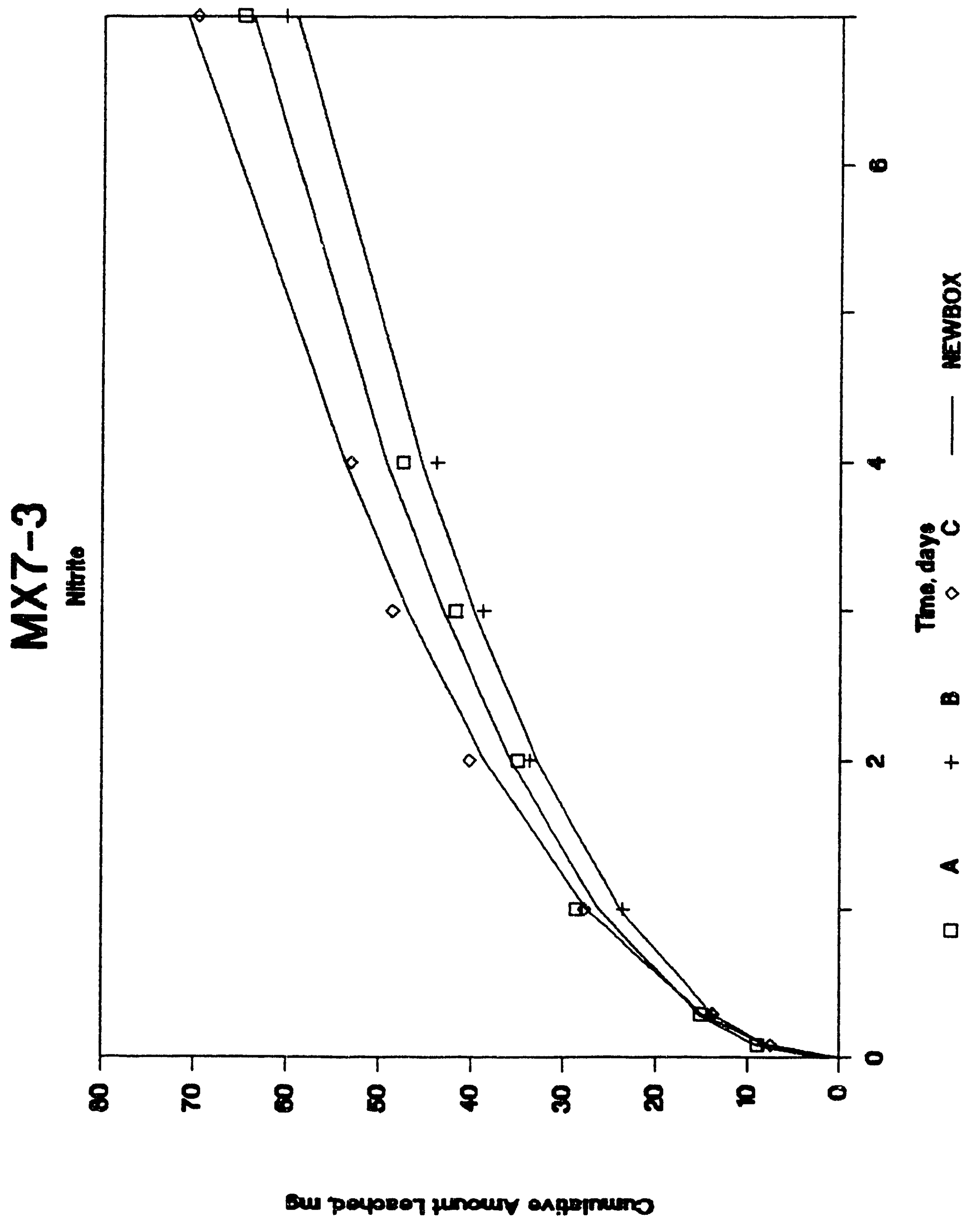
NEWBOX Estimate of Parameters            A        B        C

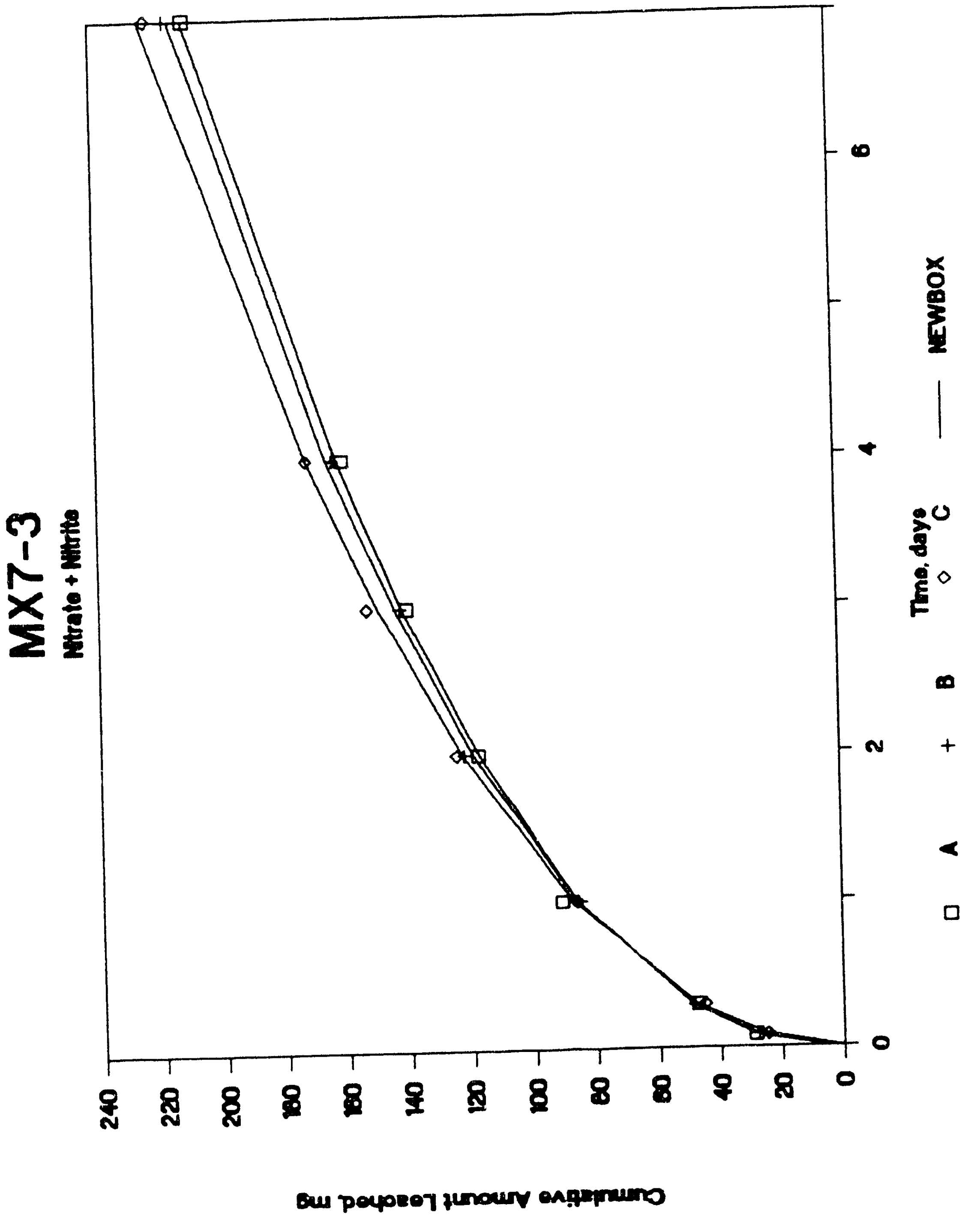
Washoff, mg =                              6        4        0
         Std. Dev., mg =                   3        2        2
Effective Diff. Coeff., sq. cm/s =  4.48E-09 4.82E-09 5.43E-09
         Std. Dev., sq. cm/s =      3.51E-10 1.90E-10 2.90E-10
Leachability Index =                   8.349    8.317    8.265
         Upper 95% Conf. Int. =        0.034    0.017    0.023
         Lower 95% Conf. Int. =        0.033    0.017    0.023

Cumulative Fraction Leached Based on Ao (CFL)
Initially in fresh grout =             1.040    1.044    1.039
Rinse =                                0.040    0.044    0.039
Ao =                                   1.000    1.000    1.000
Washoff =                              0.004    0.002    0.000
Rinse + Washoff =                      0.044    0.047    0.039
Leaching by diffusion control =        0.996    0.998    1.000

    Time      CFL of A          CFL of B          CFL of C
    days   Obsv'd   Calc'd   Obsv'd   Calc'd   Obsv'd   Calc'd

   0.083    0.016    0.017    0.016    0.016    0.014    0.014
   0.292    0.027    0.028    0.028    0.028    0.026    0.027
       1    0.052    0.049    0.049    0.049    0.049    0.049
       2    0.066    0.066    0.069    0.068    0.070    0.069
       3    0.079    0.080    0.081    0.082    0.087    0.084
       4    0.091    0.092    0.093    0.094    0.097    0.097
       7    0.119    0.119    0.123    0.122    0.126    0.127
```


MX7-3
Nitrite
Cumulative Amount Leached, mg
0
10
20
30
40
50
60
70
80
Time, days
0
2
4
6
□ A
+ B
◇ C
—— NEWBOX


MX7-3
Nitrate + Nitrite
Cumulative Amount Leached, mg
240
220
200
180
160
140
120
100
80
60
40
20
0
Time, days
0
2
4
6
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX8-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.43	4.45	4.44
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	19	18	6
Cylinder at start of leaching, Ao =	536	536	548

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	7.8	6.8	5.0
2	0.292	13.7	12.2	9.0
3	1	25.1	20.0	17.9
4	2	33.3	27.1	25.7
5	3	38.9	31.8	31.6
6	4	44.8	37.4	37.2
7	7	59.6	52.6	45.5

NEWBOY Estimate of Parameters	A	B	C
Washoff, mg =	2	2	0
Std. Dev., mg =	1	1	1
Effective Diff. Coeff., sq. cm/s =	3.85E-09	2.67E-09	2.39E-09
Std. Dev., sq. cm/s =	2.31E-10	3.47E-10	2.59E-10
Leachability Index =	8.415	8.573	8.622
Upper 95% Conf. Int. =	0.026	0.059	0.048
Lower 95% Conf. Int. =	0.025	0.053	0.045

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.035	1.034	1.011
Rinse =	0.035	0.034	0.011
Ao =	1.000	1.000	1.000
Washoff =	0.003	0.003	0.000
Rinse + Washoff =	0.038	0.037	0.011
Leaching by diffusion control =	0.997	0.997	1.000

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.015	0.015	0.013	0.013	0.009	0.010
0.292	0.026	0.026	0.023	0.022	0.016	0.018
1	0.047	0.045	0.037	0.038	0.033	0.033
2	0.062	0.062	0.051	0.052	0.047	0.046
3	0.073	0.074	0.059	0.062	0.058	0.057
4	0.084	0.085	0.070	0.071	0.068	0.065
7	0.111	0.111	0.098	0.093	0.083	0.086

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX8-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.43	4.45	4.44
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	58	56	24
Cylinder at start of leaching, Ao =	1773	1775	1807

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	25.3	27.7	20.0
2	0.292	44.1	47.5	37.5
3	1	84.7	78.5	68.7
4	2	112.5	105.3	95.4
5	3	133.1	124.3	113.8
6	4	152.3	142.7	132.4
7	7	197.7	188.3	166.6

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	4	9	2
Std. Dev., mg =	3	2	2
Effective Diff. Coeff., sq. cm/s =	4.12E-09	3.34E-09	2.89E-09
Std. Dev., sq. cm/s =	3.12E-10	1.51E-10	1.63E-10
Leachability Index =	8.385	8.476	8.539
Upper 95% Conf. Int. =	0.033	0.020	0.024
Lower 95% Conf. Int. =	0.032	0.019	0.024

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.033	1.032	1.013
Rinse =	0.033	0.032	0.013
Ao =	1.000	1.000	1.000
Washoff =	0.002	0.005	0.001
Rinse + Washoff =	0.035	0.036	0.014
Leaching by diffusion control =	0.998	0.995	0.999

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.014	0.015	0.016	0.016	0.011	0.012
0.292	0.025	0.025	0.027	0.026	0.021	0.021
1	0.048	0.045	0.044	0.044	0.038	0.037
2	0.063	0.062	0.059	0.059	0.053	0.052
3	0.075	0.076	0.070	0.071	0.063	0.063
4	0.086	0.087	0.080	0.081	0.073	0.072
7	0.112	0.113	0.106	0.105	0.092	0.095

Sulfate Leaching Data
Matrix Run No.: MX8-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	4.43	4.45	4.44
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	1	5	1
Cylinder at start of leaching, Ao =	46	43	46

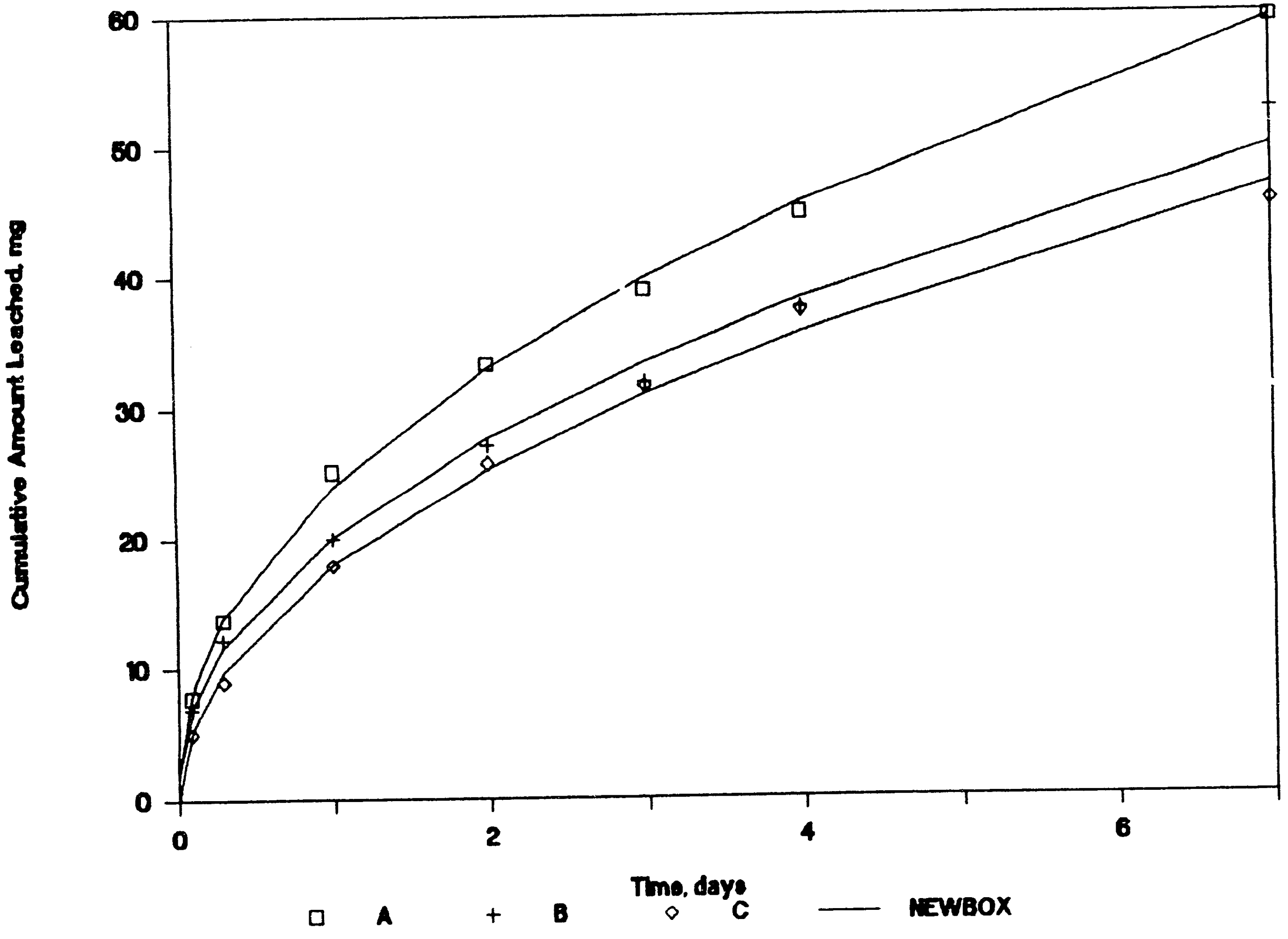
Cumulative Amount Leached, mg sulfate

Interval	Time days	A	B	C
1	0.083	0.7	1.1	1.1
2	0.292	1.7	3.9	2.6
3	1	3.7	5.6	4.5
4	2	5.1	7.4	5.7
5	3	7.5	7.5	7.5
6	4	8.5	8.8	9.0
7	7	10.5	11.0	11.0

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	0	0	0
Std. Dev., mg =	1	1	0
Effective Diff. Coeff., sq. cm/s =	1.66E-08	2.76E-08	2.11E-08
Std. Dev., sq. cm/s =	6.15E-09	1.06E-08	3.07E-09
Leachability Index =	7.780	7.559	7.676
Upper 95% Conf. Int. =	0.194	0.203	0.066
Lower 95% Conf. Int. =	0.137	0.141	0.059

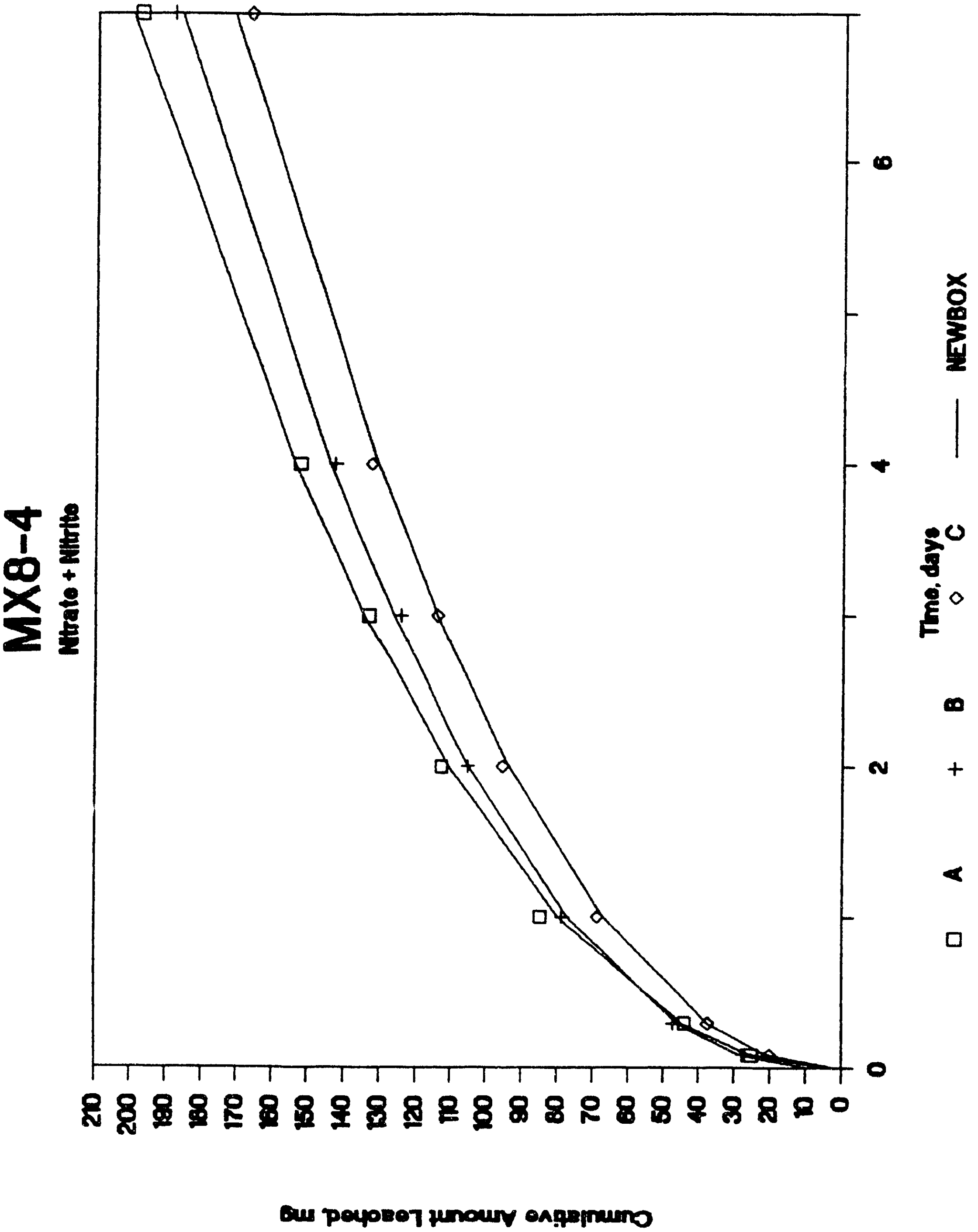
Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.029	1.106	1.032
Rinse =	0.029	0.106	0.032
Ao =	1.000	1.000	1.000
Washoff =	0.000	0.004	0.000
Rinse + Washoff =	0.029	0.109	0.032
Leaching by diffusion control =	1.000	0.996	1.000

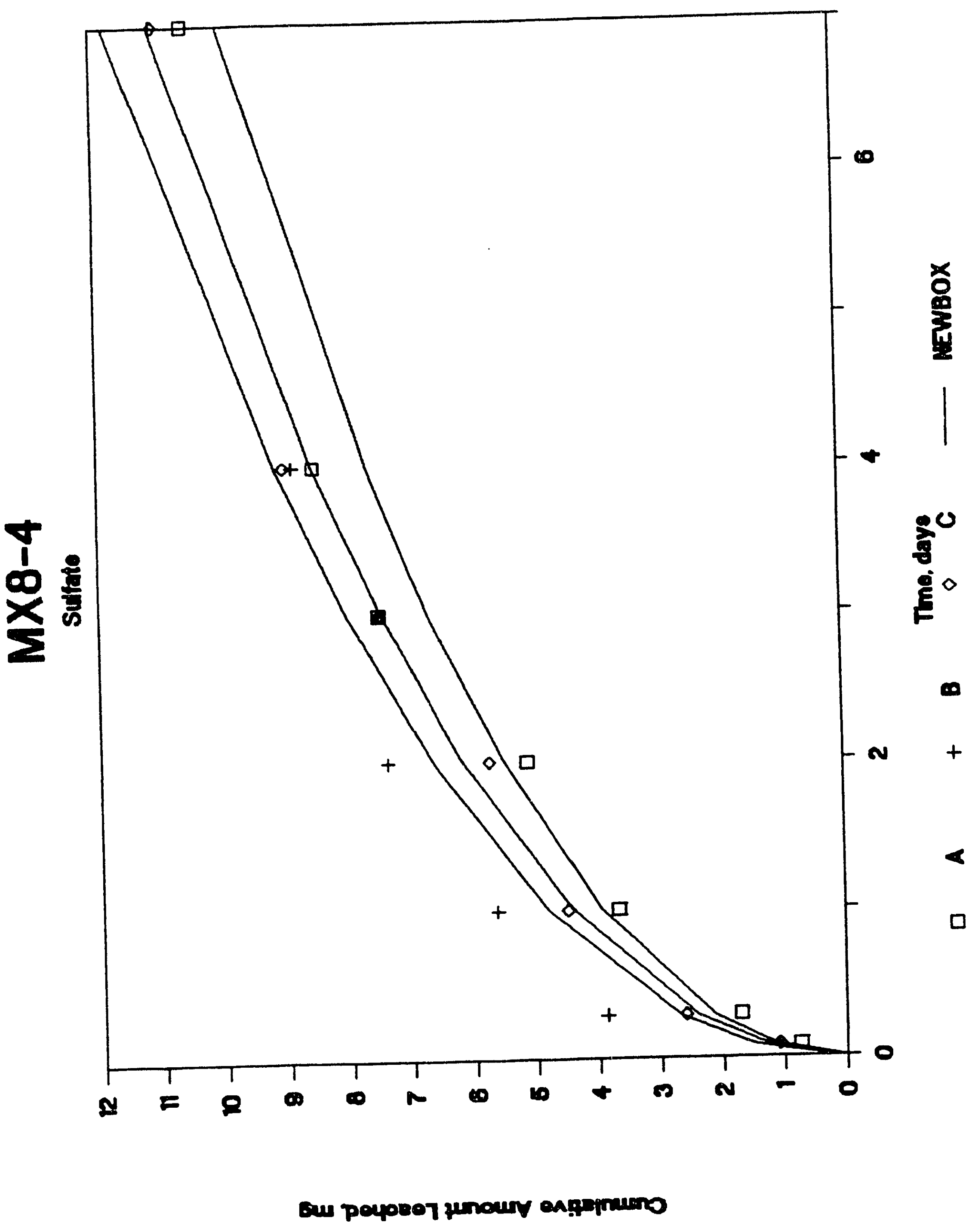
Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.016	0.025	0.026	0.036	0.024	0.028
0.292	0.037	0.047	0.090	0.063	0.057	0.052
1	0.079	0.085	0.131	0.112	0.097	0.096
2	0.111	0.119	0.172	0.155	0.125	0.134
3	0.162	0.145	0.175	0.187	0.163	0.162
4	0.185	0.166	0.207	0.213	0.196	0.186
7	0.228	0.216	0.257	0.275	0.240	0.241



MX8-4

Nitrite


MX8-4
Nitrate + Nitrite
Cumulative Amount Leached, mg
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX8-4
Sulfate
Cumulative Amount Leached, mg
0
1
2
3
4
5
6
7
8
9
10
11
12
Time, days
0
2
4
6
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX9-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50
Nitrite Mass, mg			
Rinse =	15	19	2
Cylinder at start of leaching, Ao =	539	535	553

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	27.2	30.5	33.0
2	0.292	40.3	45.8	56.7
3	1	61.2	68.4	87.0
4	2	79.0	79.7	103.0
5	3	93.1	92.6	112.4
6	4	99.4	107.1	124.4
7	7	114.2	126.0	149.0

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	17	20	23
Std. Dev., mg =	5	4	10
Effective Diff. Coeff., sq. cm/s =	1.44E-08	1.61E-08	2.33E-08
Std. Dev., sq. cm/s =	2.93E-09	2.57E-09	6.59E-09
Leachability Index =	7.842	7.793	7.633
Upper 95% Conf. Int. =	0.096	0.073	0.140
Lower 95% Conf. Int. =	0.080	0.064	0.108

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.028	1.036	1.003
Rinse =	0.028	0.036	0.003
Ao =	1.000	1.000	1.000
Washoff =	0.031	0.037	0.041
Rinse + Washoff =	0.060	0.073	0.044
Leaching by diffusion control =	0.969	0.963	0.959

Time days	CFL of A Obsv'd	CFL of A Calc'd	CFL of B Obsv'd	CFL of B Calc'd	CFL of C Obsv'd	CFL of C Calc'd
0.083	0.050	0.054	0.057	0.061	0.060	0.070
0.292	0.075	0.074	0.086	0.082	0.103	0.094
1	0.113	0.109	0.128	0.119	0.157	0.138
2	0.147	0.140	0.149	0.151	0.186	0.176
3	0.173	0.163	0.173	0.175	0.203	0.205
4	0.184	0.182	0.200	0.196	0.225	0.229
7	0.212	0.228	0.235	0.243	0.270	0.284

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX9-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	50	62	5
Cylinder at start of leaching, Ao =	1781	1769	1826

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	82.9	87.9	96.4
2	0.292	132.9	140.4	171.1
3	1	202.5	211.3	262.2
4	2	256.9	253.1	316.7
5	3	315.4	292.0	352.8
6	4	341.4	328.4	389.3
7	7	397.6	388.2	463.7

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	46	57	62
Std. Dev., mg =	15	16	28
Effective Diff. Coeff., sq. cm/s =	1.67E-08	1.45E-08	2.16E-08
Std. Dev., sq. cm/s =	2.78E-09	2.62E-09	5.63E-09
Leachability Index =	7.777	7.839	7.666
Upper 95% Conf. Int. =	0.077	0.084	0.127
Lower 95% Conf. Int. =	0.067	0.072	0.101

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.028	1.035	1.003
Rinse =	0.028	0.035	0.003
Ao =	1.000	1.000	1.000
Washoff =	0.026	0.032	0.034
Rinse + Washoff =	0.054	0.067	0.037
Leaching by diffusion control =	0.974	0.968	0.966

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.047	0.051	0.050	0.055	0.053	0.062
0.292	0.075	0.072	0.079	0.075	0.094	0.085
1	0.114	0.110	0.119	0.110	0.144	0.128
2	0.144	0.143	0.143	0.141	0.173	0.165
3	0.177	0.168	0.165	0.164	0.193	0.193
4	0.192	0.189	0.186	0.184	0.213	0.216
7	0.223	0.238	0.219	0.230	0.254	0.270

Sulfate Leaching Data
Matrix Run No.: MX9-5

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	4.33	4.30	4.35
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	1	2	1
Cylinder at start of leaching, Ao =	46	45	46

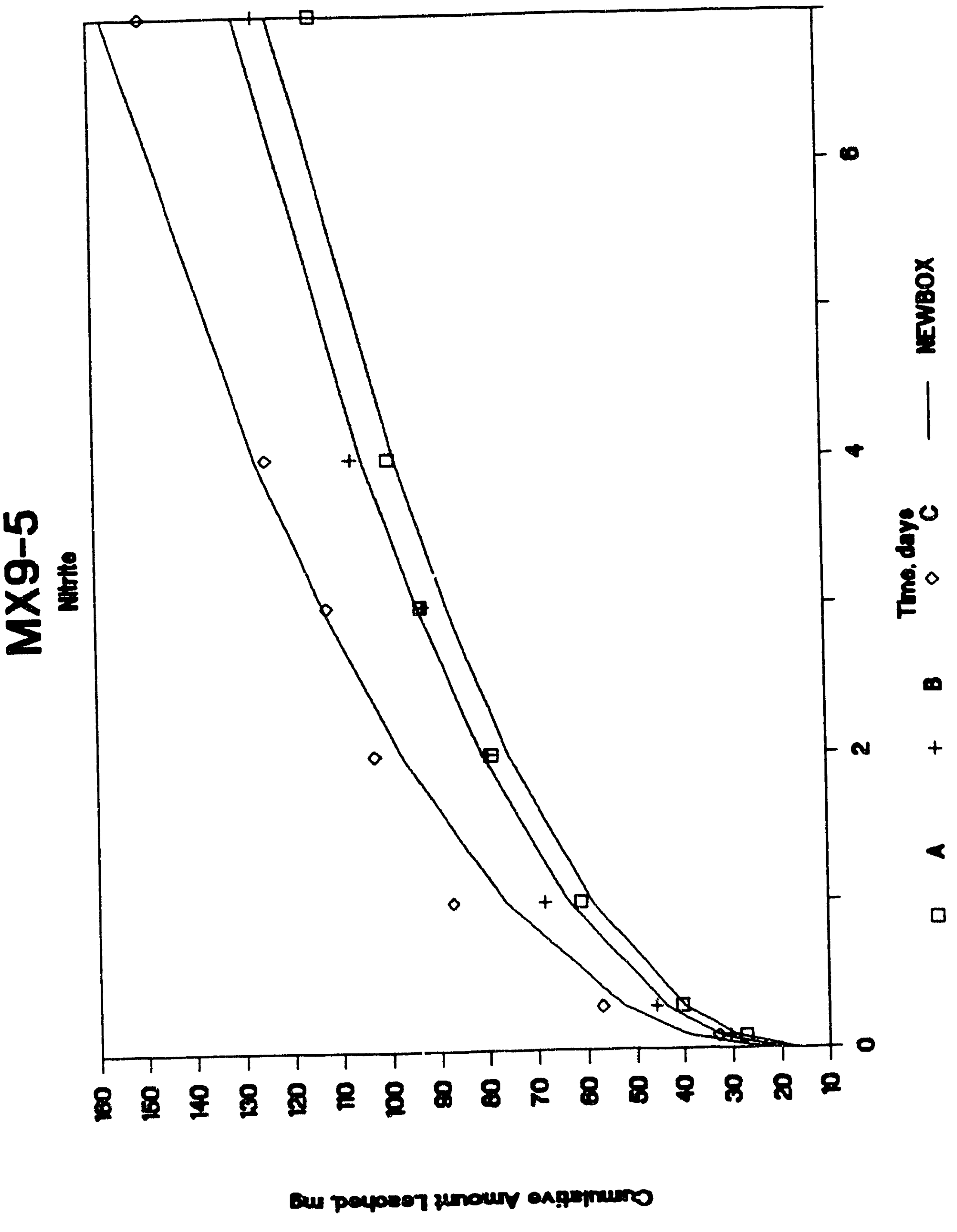
Cumulative Amount Leached, mg sulfate

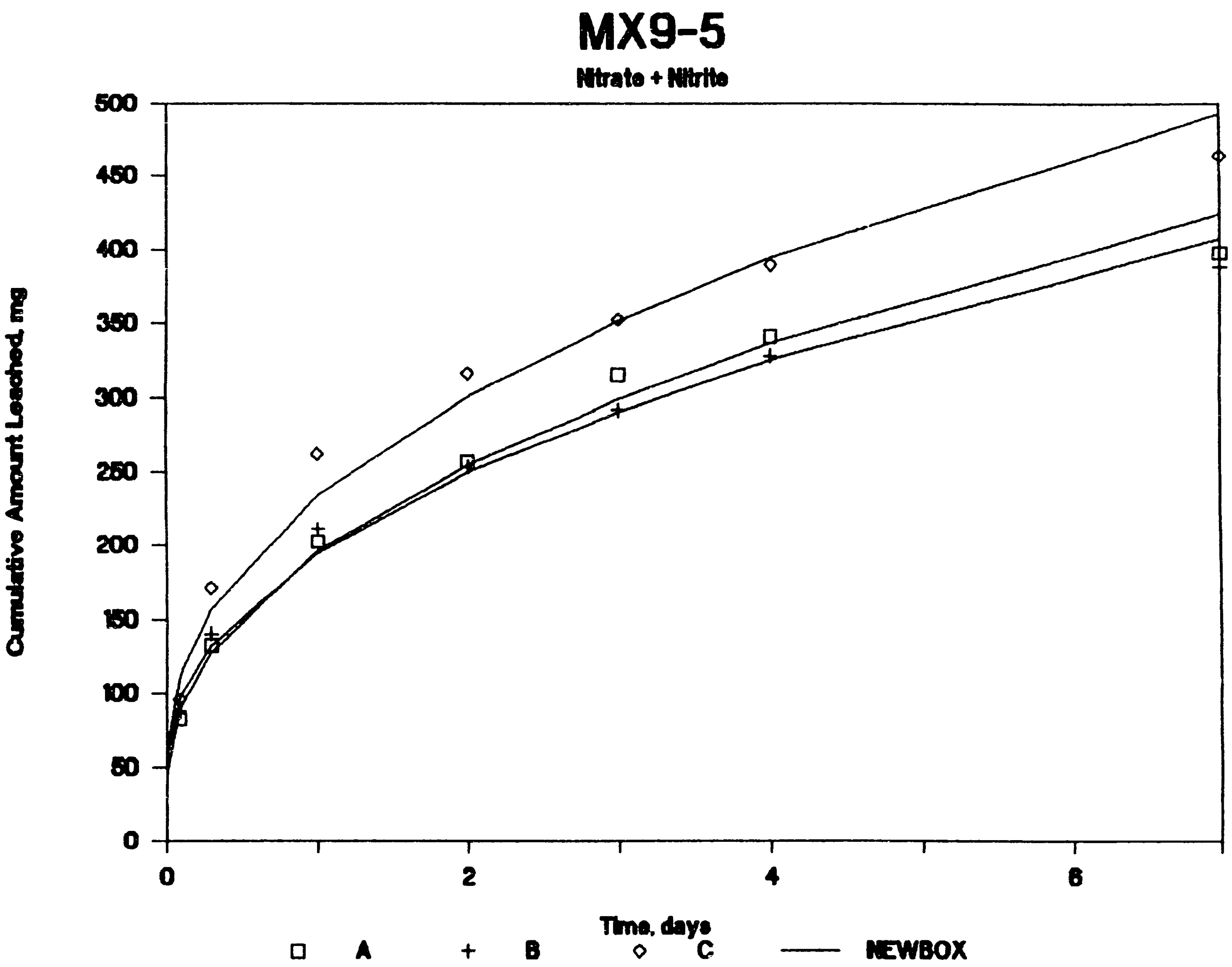
Interval	Time days	A	B	C
1	0.083	4.8	8.2	4.7
2	0.292	6.1	9.8	8.2
3	1	9.1	12.1	10.4
4	2	9.4	13.7	13.9
5	3	11.4	14.8	16.3
6	4	13.1	15.6	18.1
7	7	15.8	15.9	22.7

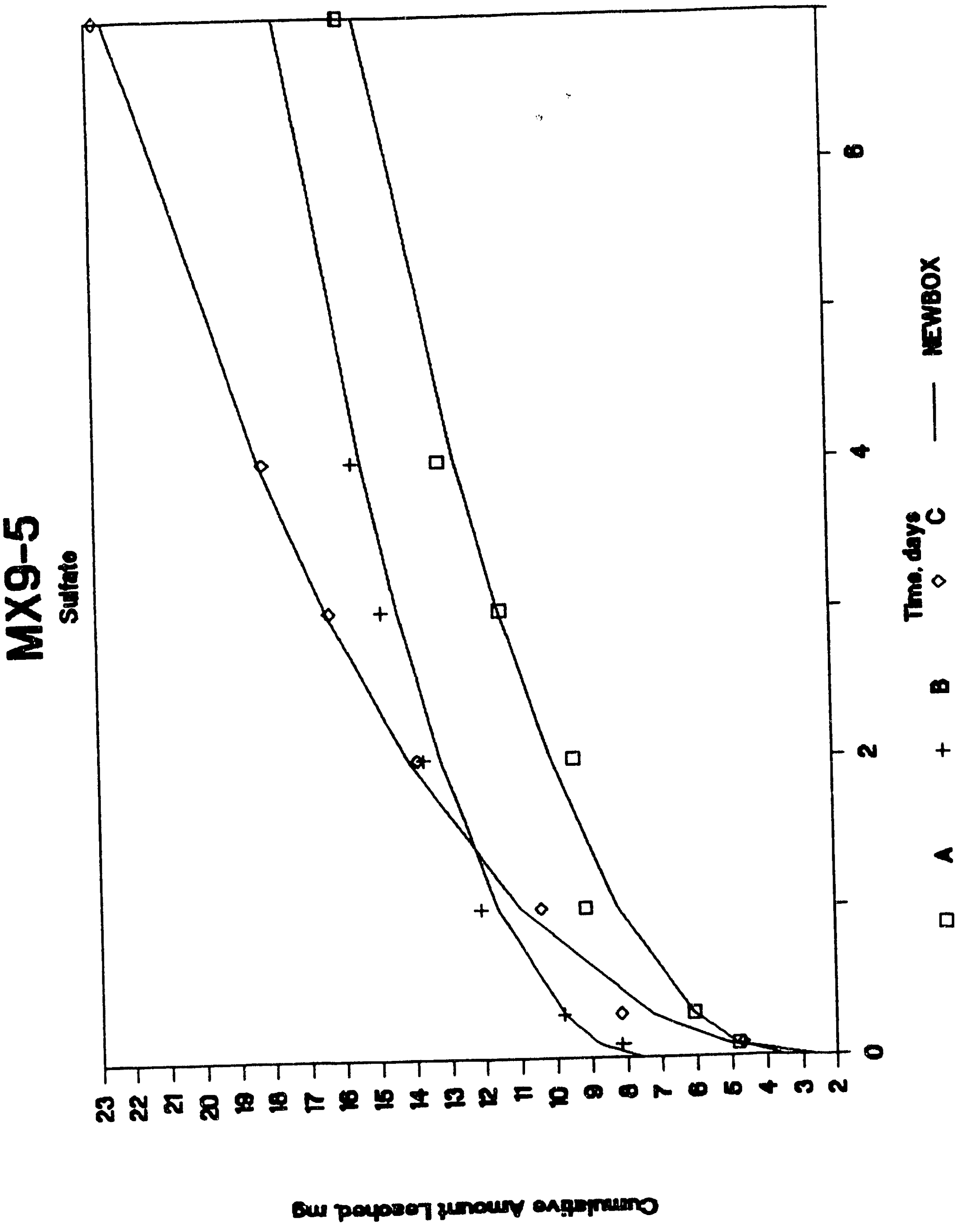
NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	3	8	3
Std. Dev., mg =	1	1	1
Effective Diff. Coeff., sq. cm/s =	2.85E-08	2.64E-08	8.52E-08
Std. Dev., sq. cm/s =	8.52E-09	1.23E-08	1.48E-08
Leachability Index =	7.545	7.578	7.070
Upper 95% Conf. Int. =	0.149	0.262	0.080
Lower 95% Conf. Int. =	0.114	0.166	0.070

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.030	1.053	1.019
Rinse =	0.030	0.053	0.019
Ao =	1.000	1.000	1.000
Washoff =	0.076	0.168	0.057
Rinse + Washoff =	0.106	0.221	0.076
Leaching by diffusion control =	0.924	0.832	0.943

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.104	0.106	0.181	0.195	0.100	0.111
0.292	0.132	0.132	0.218	0.217	0.176	0.157
1	0.198	0.179	0.270	0.258	0.224	0.236
2	0.205	0.219	0.304	0.293	0.298	0.304
3	0.249	0.250	0.329	0.319	0.351	0.353
4	0.285	0.275	0.347	0.341	0.391	0.393
7	0.343	0.333	0.353	0.392	0.490	0.484


MX9-5
Nitrite
Cumulative Amount Leached, mg
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX9-5
Nitrate + Nitrite
Cumulative Amount Leached, mg
0
50
100
150
200
250
300
350
400
450
500
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX9-5
Sulfate
Cumulative Amount Leached, mg
23
22
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
0
2
4
6
Time, days
□ A
+ B
◇ C
— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX10-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm   Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.14	4.17	4.14
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	47	50	27
Cylinder at start of leaching, Ao =	507	504	528

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	8.0	13.3	7.6
2	0.292	13.1	19.6	15.7
3	1	16.4	30.0	28.4
4	2	25.9	39.5	38.7
5	3	31.6	46.3	46.3
6	4	36.7	52.3	51.8
7	7	49.0	63.1	61.7

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	3	7	2
Std. Dev., mg =	3	1	2
Effective Diff. Coeff., sq. cm/s =	2.44E-09	4.39E-09	4.96E-09
Std. Dev., sq. cm/s =	6.24E-10	3.83E-10	7.71E-10
Leachability Index =	8.613	8.358	8.305
Upper 95% Conf. Int. =	0.124	0.038	0.071
Lower 95% Conf. Int. =	0.099	0.036	0.063

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.093	1.100	1.050
Rinse =	0.093	0.100	0.050
Ao =	1.000	1.000	1.000
Washoff =	0.005	0.014	0.003
Rinse + Washoff =	0.098	0.114	0.053
Leaching by diffusion control =	0.995	0.986	0.997

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.016	0.015	0.026	0.027	0.014	0.017
0.292	0.026	0.023	0.039	0.038	0.030	0.029
1	0.032	0.039	0.060	0.059	0.054	0.051
2	0.051	0.052	0.078	0.077	0.073	0.070
3	0.062	0.063	0.092	0.090	0.088	0.085
4	0.072	0.071	0.104	0.102	0.098	0.097
7	0.097	0.092	0.125	0.129	0.117	0.126

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX10-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.14	4.17	4.14
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	125	134	77
Cylinder at start of leaching, Ao =	1706	1697	1754

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	27.7	38.1	32.5
2	0.292	47.0	61.9	57.0
3	1	73.5	101.2	99.3
4	2	102.6	135.6	134.6
5	3	121.4	158.1	159.4
6	4	138.5	176.9	177.2
7	7	180.9	215.5	214.6

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	9	17	10
Std. Dev., mg =	2	5	7
Effective Diff. Coeff., sq. cm/s =	3.23E-09	5.04E-09	5.08E-09
Std. Dev., sq. cm/s =	1.68E-10	5.66E-10	6.90E-10
Leachability Index =	8.491	8.298	8.294
Upper 95% Conf. Int. =	0.023	0.050	0.061
Lower 95% Conf. Int. =	0.022	0.046	0.055

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.073	1.079	1.044
Rinse =	0.073	0.079	0.044
Ao =	1.000	1.000	1.000
Washoff =	0.006	0.010	0.006
Rinse + Washoff =	0.079	0.089	0.050
Leaching by diffusion control =	0.994	0.990	0.994

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.016	0.017	0.022	0.024	0.019	0.020
0.292	0.028	0.026	0.036	0.036	0.033	0.032
1	0.043	0.044	0.060	0.058	0.057	0.054
2	0.060	0.060	0.080	0.077	0.077	0.073
3	0.071	0.072	0.093	0.092	0.091	0.088
4	0.081	0.082	0.104	0.104	0.101	0.101
7	0.106	0.105	0.127	0.133	0.122	0.130

Sulfate Leaching Data
Matrix Run No.: MX10-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	4.14	4.17	4.14
Diameter, cm =	2.50	2.50	2.50
Sulfate Mass, mg			
Rinse =	10	6	3
Cylinder at start of leaching, Ao =	38	41	44

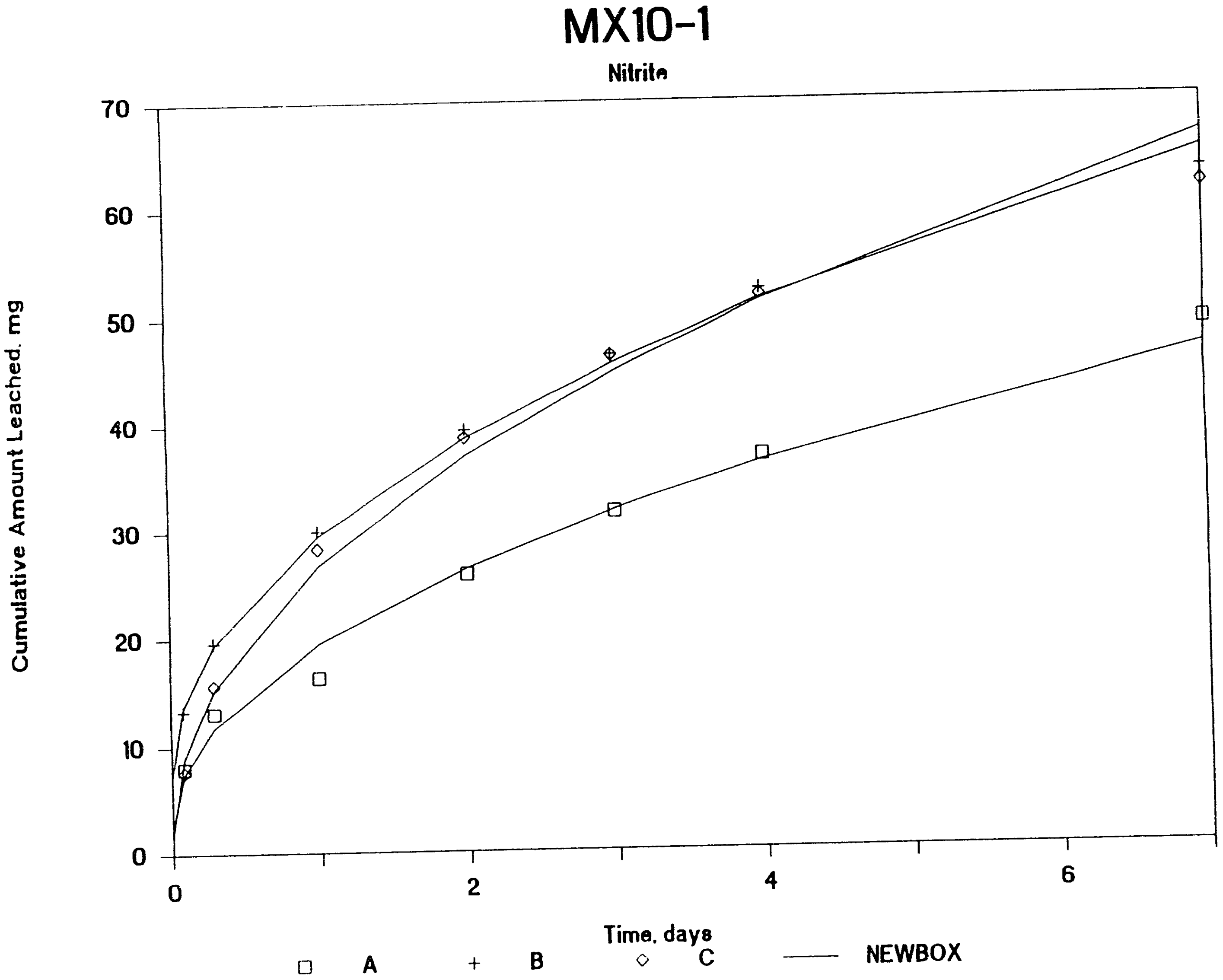
Cumulative Amount Leached, mg sulfate

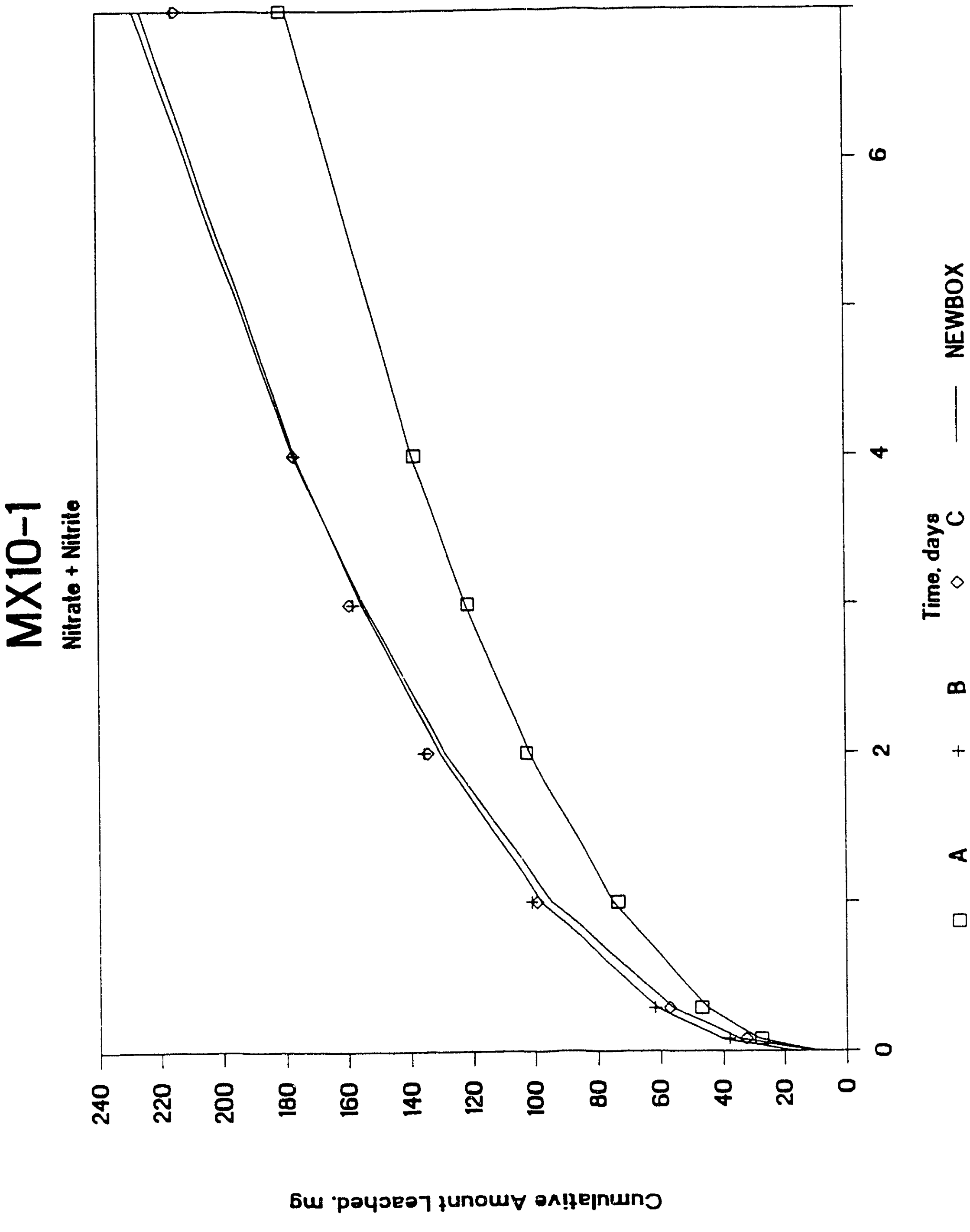
Interval	Time days	A	B	C
1	0.083	3.7	2.3	1.6
2	0.292	6.9	7.8	2.2
3	1	7.3	10.8	3.8
4	2	7.8	13.5	5.1
5	3	8.9	16.0	5.8
6	4	11.8	16.4	5.9
7	7	14.2	17.9	10.5

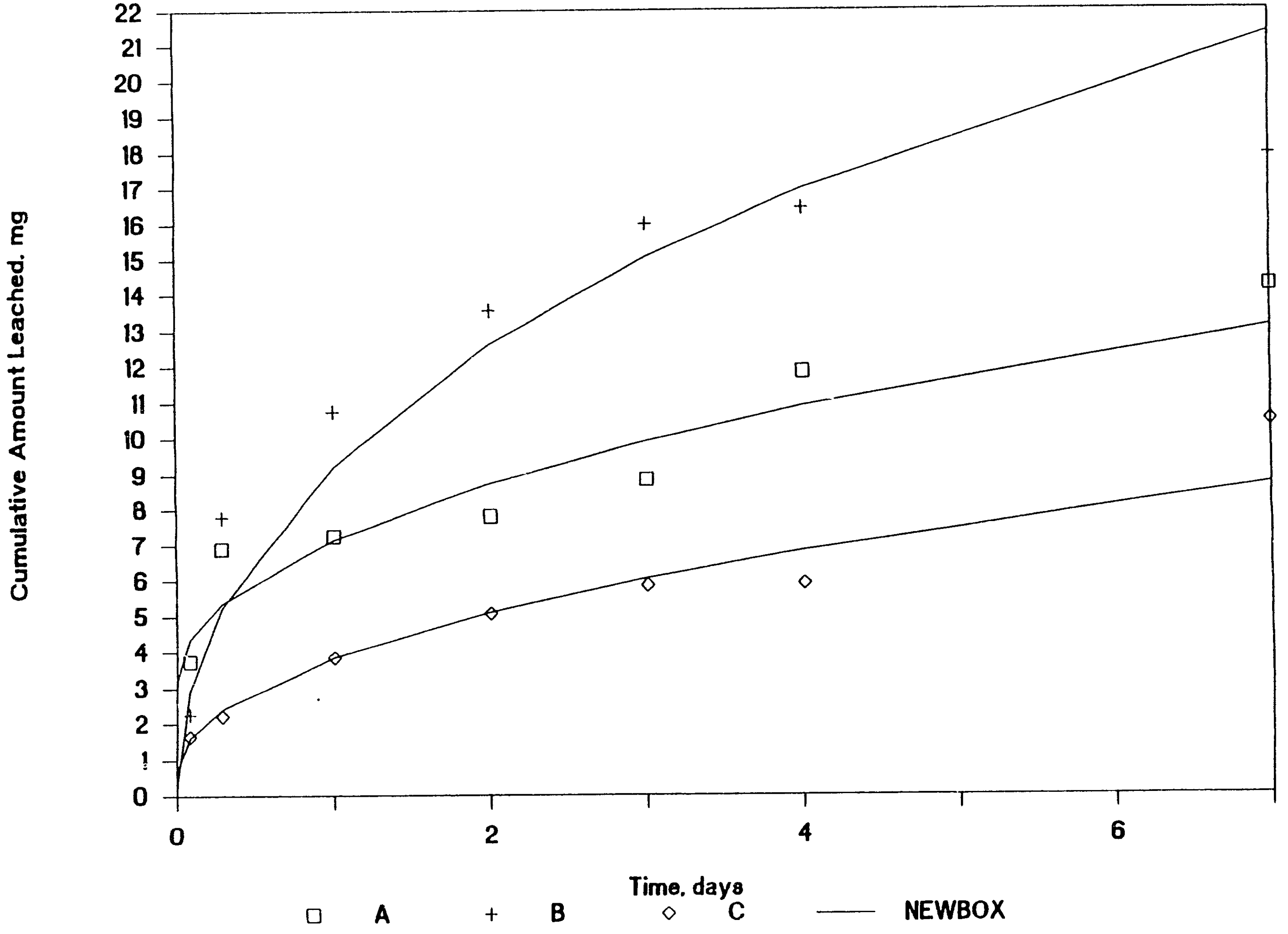
NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	3	0	1
Std. Dev., mg =	1	2	1
Effective Diff. Coeff., sq. cm/s =	3.05E-08	1.19E-07	1.16E-08
Std. Dev., sq. cm/s =	1.66E-08	6.02E-08	4.73E-09
Leachability Index =	7.516	6.924	7.936
Upper 95% Conf. Int. =	0.327	0.294	0.219
Lower 95% Conf. Int. =	0.189	0.178	0.149

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.262	1.149	1.073
Rinse =	0.262	0.149	0.073
Ao =	1.000	1.000	1.000
Washoff =	0.086	0.005	0.016
Rinse + Washoff =	0.347	0.153	0.089
Leaching by diffusion control =	0.914	0.995	0.984

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.099	0.116	0.055	0.071	0.037	0.037
0.292	0.184	0.143	0.189	0.127	0.050	0.055
1	0.194	0.191	0.261	0.224	0.087	0.087
2	0.208	0.233	0.329	0.306	0.115	0.116
3	0.236	0.264	0.388	0.365	0.132	0.137
4	0.315	0.290	0.398	0.412	0.134	0.155
7	0.379	0.349	0.435	0.518	0.237	0.198


MX10-1
Nitrite
Cumulative Amount Leached. mg
70
60
50
40
30
20
10
0
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX


MX10-1
Nitrate + Nitrite
Cumulative Amount Leached, mg
0
20
40
60
80
100
120
140
160
180
200
220
240
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX



MX10-1

Sulfate

Nitrite Leaching Data
Matrix Run No.: MX11-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.28	4.24	4.11
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	24	12	13
Cylinder at start of leaching, Ao =	530	542	542

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	12.1	8.5	11.8
2	0.292	23.0	19.1	22.9
3	1	41.1	37.5	40.6
4	2	58.2	52.4	55.2
5	3	68.3	62.6	65.6
6	4	77.7	71.4	74.8
7	7	100.4	94.6	95.9

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	1	0	2
Std. Dev., mg =	1	3	1
Effective Diff. Coeff., sq. cm/s =	1.25E-08	1.00E-08	1.06E-08
Std. Dev., sq. cm/s =	6.37E-10	1.53E-09	6.90E-10
Leachability Index =	7.903	8.000	7.975
Upper 95% Conf. Int. =	0.022	0.070	0.028
Lower 95% Conf. Int. =	0.022	0.062	0.027

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.045	1.023	1.024
Rinse =	0.045	0.023	0.024
Ao =	1.000	1.000	1.000
Washoff =	0.002	0.000	0.003
Rinse + Washoff =	0.047	0.023	0.027
Leaching by diffusion control =	0.998	1.000	0.997

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.023	0.024	0.016	0.020	0.022	0.024
0.292	0.043	0.043	0.035	0.037	0.042	0.041
1	0.077	0.076	0.069	0.067	0.075	0.073
2	0.110	0.106	0.097	0.094	0.102	0.100
3	0.129	0.129	0.116	0.115	0.121	0.122
4	0.147	0.148	0.132	0.132	0.138	0.139
7	0.189	0.192	0.174	0.172	0.177	0.181

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX11-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.28	4.24	4.11
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	72	44	43
Cylinder at start of leaching, Ao =	1759	1787	1788

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	39.3	43.7	41.3
2	0.292	74.2	83.7	79.2
3	1	141.6	144.6	135.3
4	2	197.0	197.5	181.0
5	3	236.4	234.9	216.6
6	4	267.9	265.3	252.7
7	7	336.9	337.9	321.1

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	0	8	7
Std. Dev., mg =	5	5	4
Effective Diff. Coeff., sq. cm/s =	1.36E-08	1.23E-08	1.07E-08
Std. Dev., sq. cm/s =	9.48E-10	8.74E-10	6.71E-10
Leachability Index =	7.866	7.910	7.971
Upper 95% Conf. Int. =	0.030	0.031	0.027
Lower 95% Conf. Int. =	0.029	0.030	0.026

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.041	1.024	1.024
Rinse =	0.041	0.024	0.024
Ao =	1.000	1.000	1.000
Washoff =	0.000	0.005	0.004
Rinse + Washoff =	0.041	0.029	0.028
Leaching by diffusion control =	1.000	0.995	0.996

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.022	0.023	0.024	0.026	0.023	0.024
0.292	0.042	0.043	0.047	0.045	0.044	0.042
1	0.080	0.078	0.081	0.079	0.076	0.074
2	0.112	0.109	0.110	0.108	0.101	0.102
3	0.134	0.133	0.131	0.131	0.121	0.123
4	0.152	0.152	0.148	0.149	0.141	0.140
7	0.191	0.198	0.189	0.193	0.180	0.182

Sulfate Leaching Data
Matrix Run No.: MX11-1

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm   Diameter = 2.50 cm   Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	4.28	4.24	4.11
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	7	5	6
Cylinder at start of leaching, Ao =	40	42	42

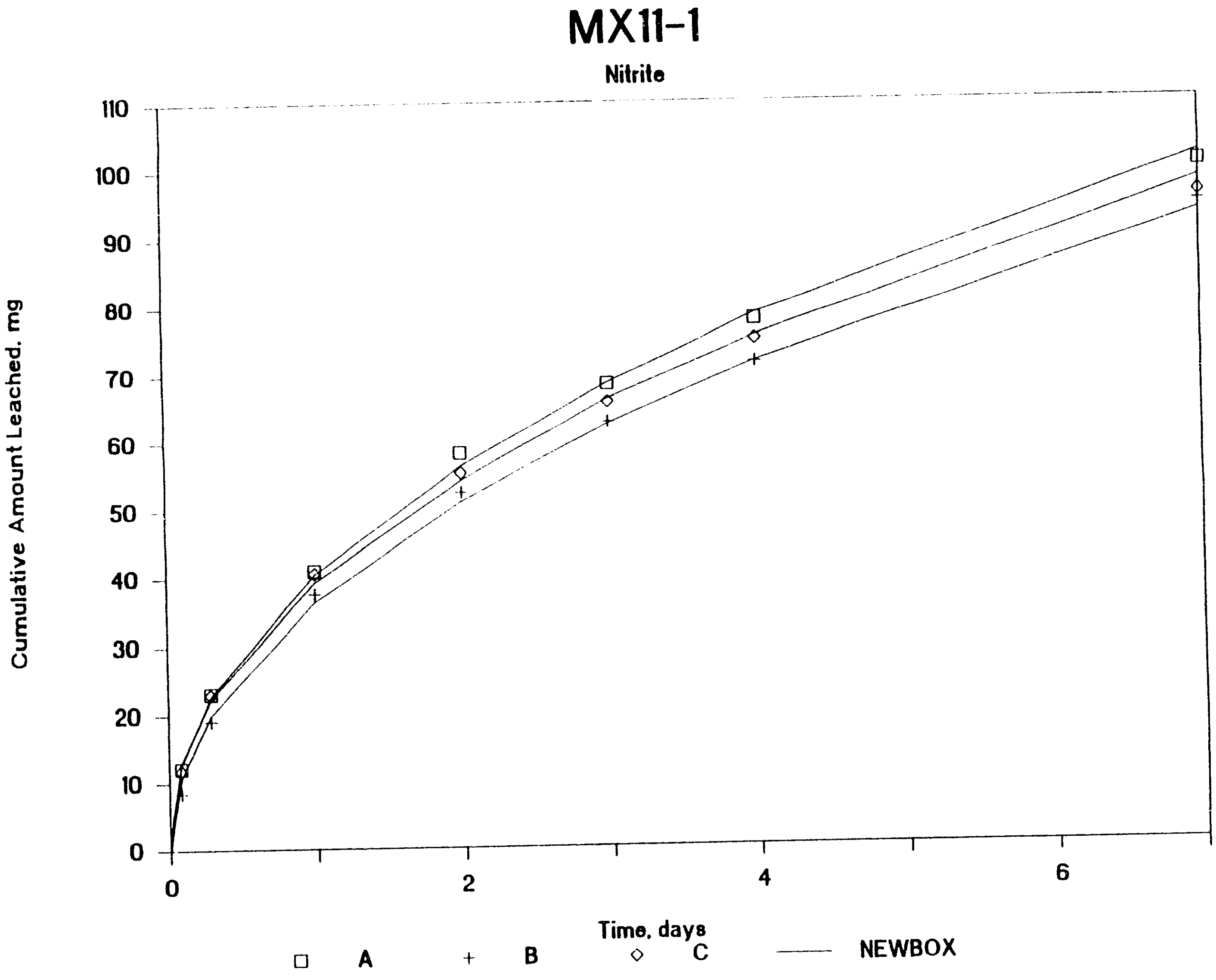
Cumulative Amount Leached, mg sulfate

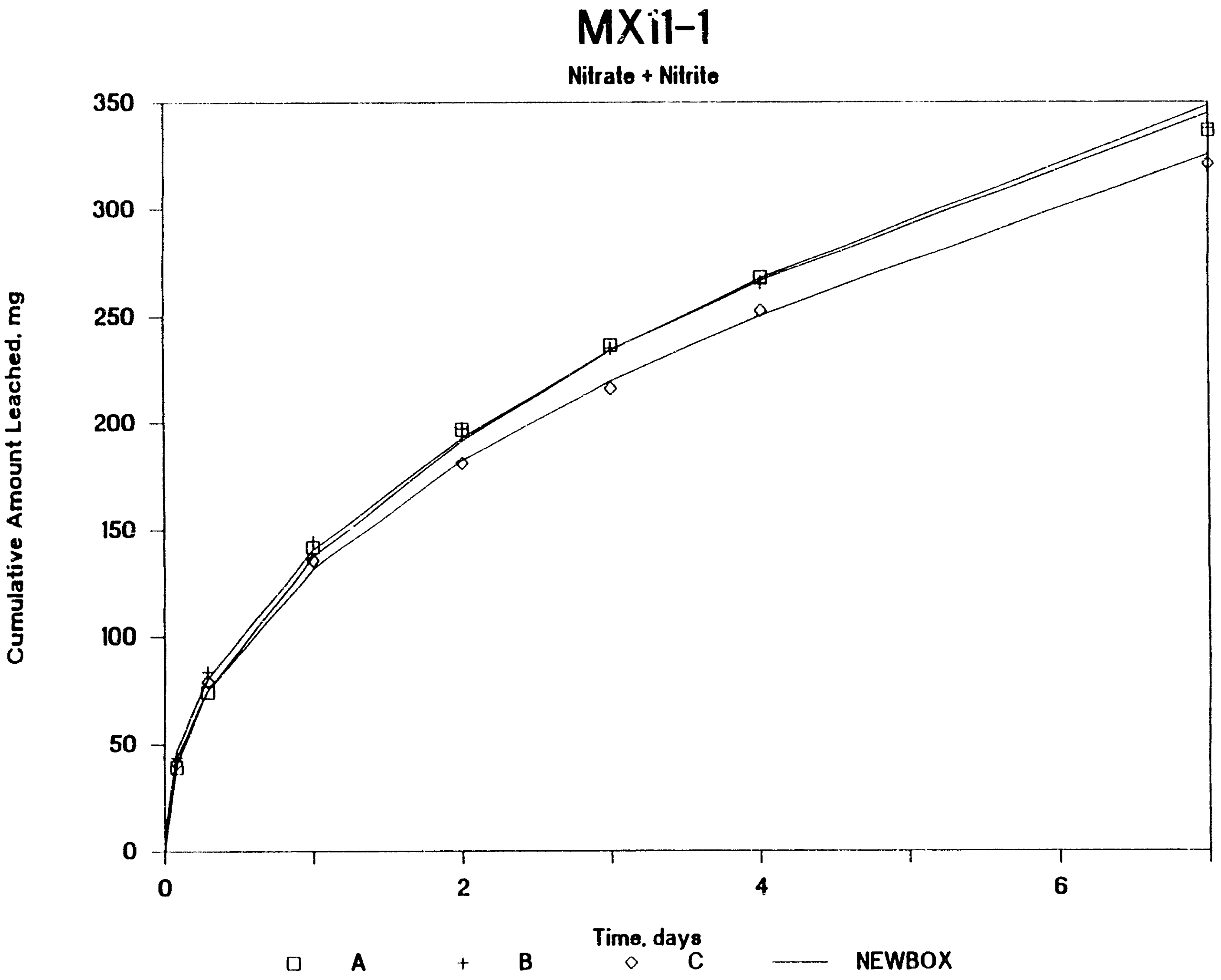
Interval	Time days	A	B	C
1	0.083	3.5	3.6	4.3
2	0.292	5.9	5.3	8.3
3	1	9.7	6.8	9.9
4	2	12.6	9.2	11.9
5	3	17.1	11.9	14.9
6	4	20.6	14.6	16.7
7	7	27.5	21.4	22.4

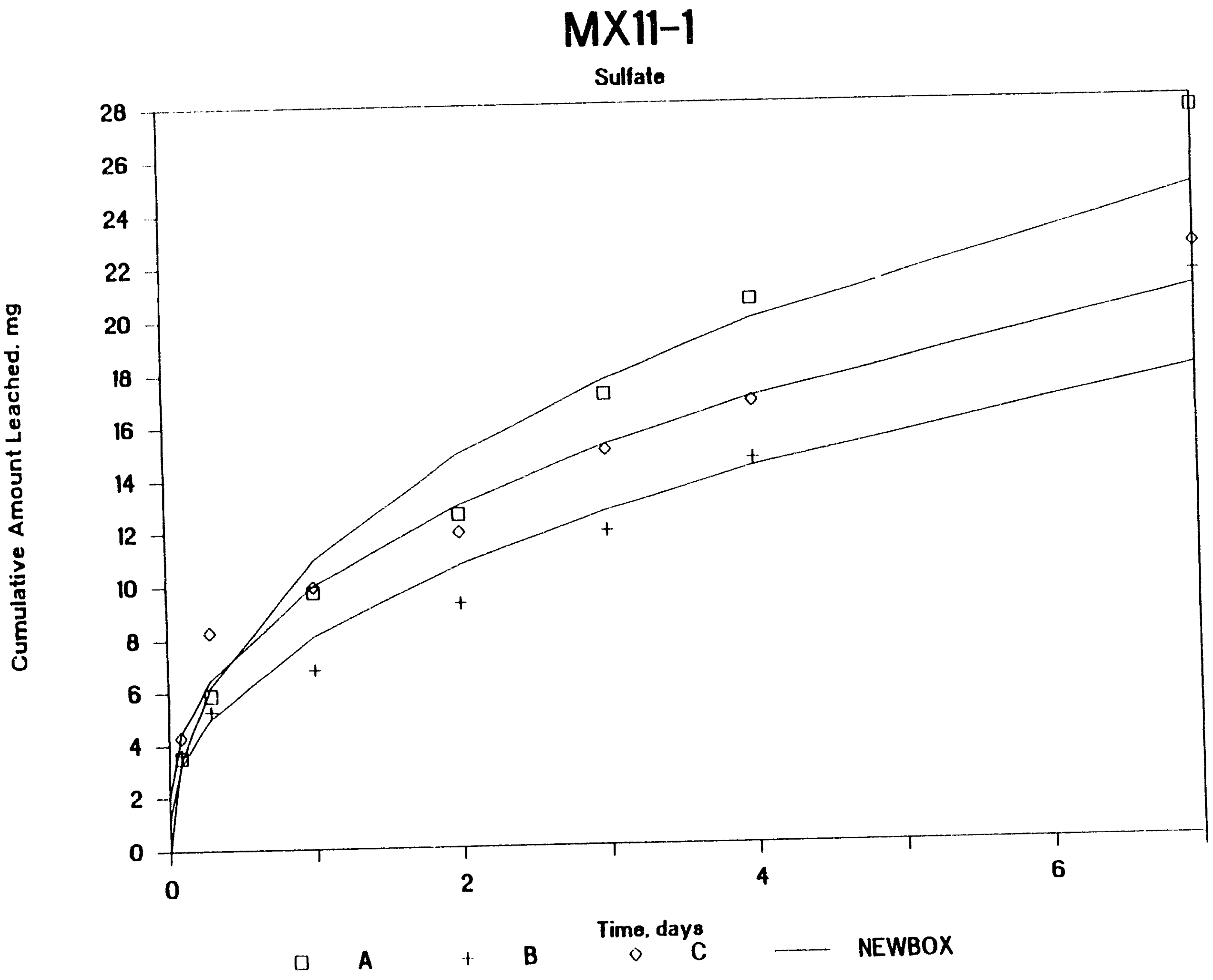
NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	0	1	2
Std. Dev., mg =	2	2	1
Effective Diff. Coeff., sq. cm/s =	1.89E-07	6.71E-08	9.65E-08
Std. Dev., sq. cm/s =	5.89E-08	3.07E-08	2.58E-08
Leachability Index =	6.724	7.173	7.015
Upper 95% Conf. Int. =	0.157	0.255	0.131
Lower 95% Conf. Int. =	0.118	0.164	0.103

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.184	1.123	1.137
Rinse =	0.184	0.123	0.137
Ao =	1.000	1.000	1.000
Washoff =	0.002	0.028	0.049
Rinse + Washoff =	0.186	0.151	0.186
Leaching by diffusion control =	0.998	0.972	0.951

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.089	0.085	0.086	0.077	0.103	0.106
0.292	0.147	0.155	0.125	0.119	0.199	0.155
1	0.243	0.274	0.161	0.192	0.238	0.240
2	0.315	0.372	0.219	0.254	0.286	0.311
3	0.426	0.441	0.282	0.300	0.359	0.364
4	0.515	0.496	0.345	0.338	0.402	0.406
7	0.689	0.616	0.507	0.424	0.539	0.501


MX11-1
Nitrite
Cumulative Amount Leached, mg
0
10
20
30
40
50
60
70
80
90
100
110
0
2
4
6
Time, days
□ A
+ B
◇ C
NEWBOX


MXi1-1
Nitrate + Nitrite
Cumulative Amount Leached, mg
350
300
250
200
150
100
50
0
0
2
4
6
Time, days
□ A
+ B
◇ C
NEWBOX


MX11-1
Sulfate
Cumulative Amount Leached, mg
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX12-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.42	4.43
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	8	9	8
Cylinder at start of leaching, Ao =	546	545	546

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	9.3	9.5	9.6
2	0.292	15.9	16.4	17.8
3	1	24.7	27.3	28.3
4	2	33.1	36.9	37.4
5	3	39.6	44.4	44.7
6	4	47.3	50.5	51.1
7	7	61.5	65.6	62.7

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	3	2	4
Std. Dev., mg =	1	0	1
Effective Diff. Coeff., sq. cm/s =	3.75E-09	4.64E-09	4.20E-09
Std. Dev., sq. cm/s =	3.38E-10	1.56E-10	4.19E-10
Leachability Index =	8.426	8.333	8.377
Upper 95% Conf. Int. =	0.040	0.014	0.044
Lower 95% Conf. Int. =	0.037	0.014	0.041

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.015	1.017	1.015
Rinse =	0.015	0.017	0.015
Ao =	1.000	1.000	1.000
Washoff =	0.005	0.004	0.007
Rinse + Washoff =	0.020	0.020	0.022
Leaching by diffusion control =	0.995	0.996	0.993

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.017	0.017	0.017	0.017	0.017	0.020
0.292	0.029	0.027	0.030	0.029	0.032	0.031
1	0.045	0.046	0.050	0.049	0.052	0.050
2	0.061	0.063	0.068	0.068	0.068	0.068
3	0.072	0.075	0.081	0.082	0.082	0.081
4	0.087	0.086	0.093	0.093	0.094	0.092
7	0.113	0.111	0.120	0.121	0.115	0.119

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX12-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.42	4.43
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	28	31	27
Cylinder at start of leaching, Ao =	1803	1800	1804

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	32.0	32.0	32.0
2	0.292	55.2	55.3	58.6
3	1	87.4	89.8	94.5
4	2	115.5	121.7	125.6
5	3	139.6	147.5	150.5
6	4	163.0	168.3	172.4
7	7	210.3	218.5	223.4

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	11	9	10
Std. Dev., mg =	3	1	4
Effective Diff. Coeff., sq. cm/s =	4.06E-09	4.50E-09	4.73E-09
Std. Dev., sq. cm/s =	2.32E-10	1.46E-10	3.59E-10
Leachability Index =	8.391	8.347	8.325
Upper 95% Conf. Int. =	0.025	0.014	0.033
Lower 95% Conf. Int. =	0.024	0.014	0.032

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.016	1.017	1.015
Rinse =	0.016	0.017	0.015
Ao =	1.000	1.000	1.000
Washoff =	0.006	0.005	0.006
Rinse + Washoff =	0.022	0.023	0.021
Leaching by diffusion control =	0.994	0.995	0.994

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.018	0.018	0.018	0.018	0.018	0.019
0.292	0.031	0.029	0.031	0.030	0.032	0.031
1	0.048	0.048	0.050	0.050	0.052	0.051
2	0.064	0.066	0.068	0.068	0.070	0.070
3	0.077	0.079	0.082	0.082	0.083	0.084
4	0.090	0.090	0.093	0.094	0.096	0.096
7	0.117	0.116	0.121	0.121	0.124	0.124

Sulfate Leaching Data
Matrix Run No.: MX12-4

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.42	4.43
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	3	4	3
Cylinder at start of leaching, Ao =	44	43	45

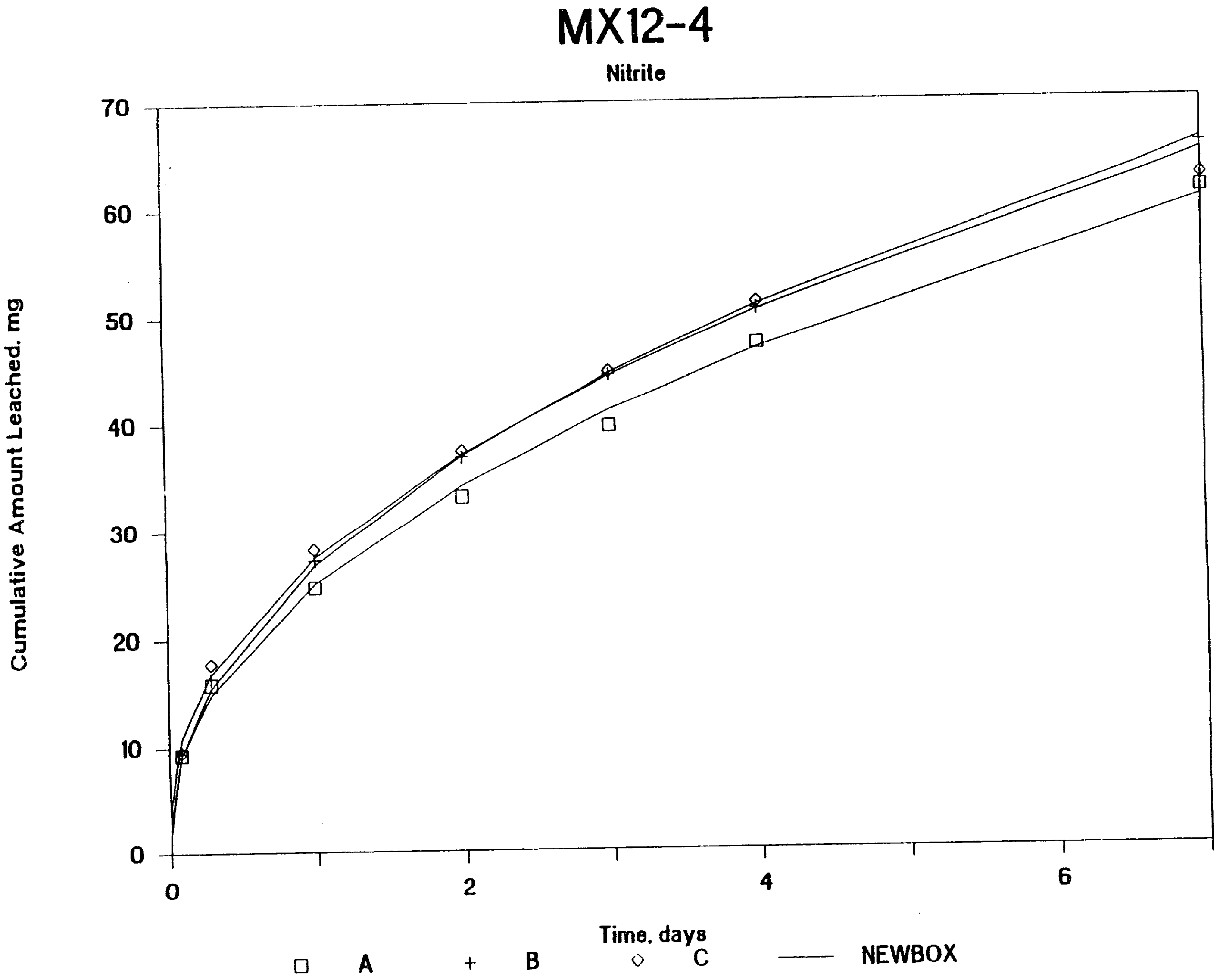
Cumulative Amount Leached, mg sulfate

Interval	Time days	A	B	C
1	0.083	3.8	3.3	2.6
2	0.292	5.4	5.1	5.3
3	1	8.6	8.7	9.6
4	2	11.8	11.0	13.2
5	3	14.8	13.7	15.7
6	4	17.9	16.3	18.7
7	7	24.7	21.0	24.9

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	0	0	0
Std. Dev., mg =	2	1	1
Effective Diff. Coeff., sq. cm/s =	1.12E-07	9.31E-08	1.23E-07
Std. Dev., sq. cm/s =	3.28E-08	1.09E-08	1.88E-08
Leachability Index =	6.951	7.031	6.910
Upper 95% Conf. Int. =	0.145	0.052	0.070
Lower 95% Conf. Int. =	0.112	0.048	0.062

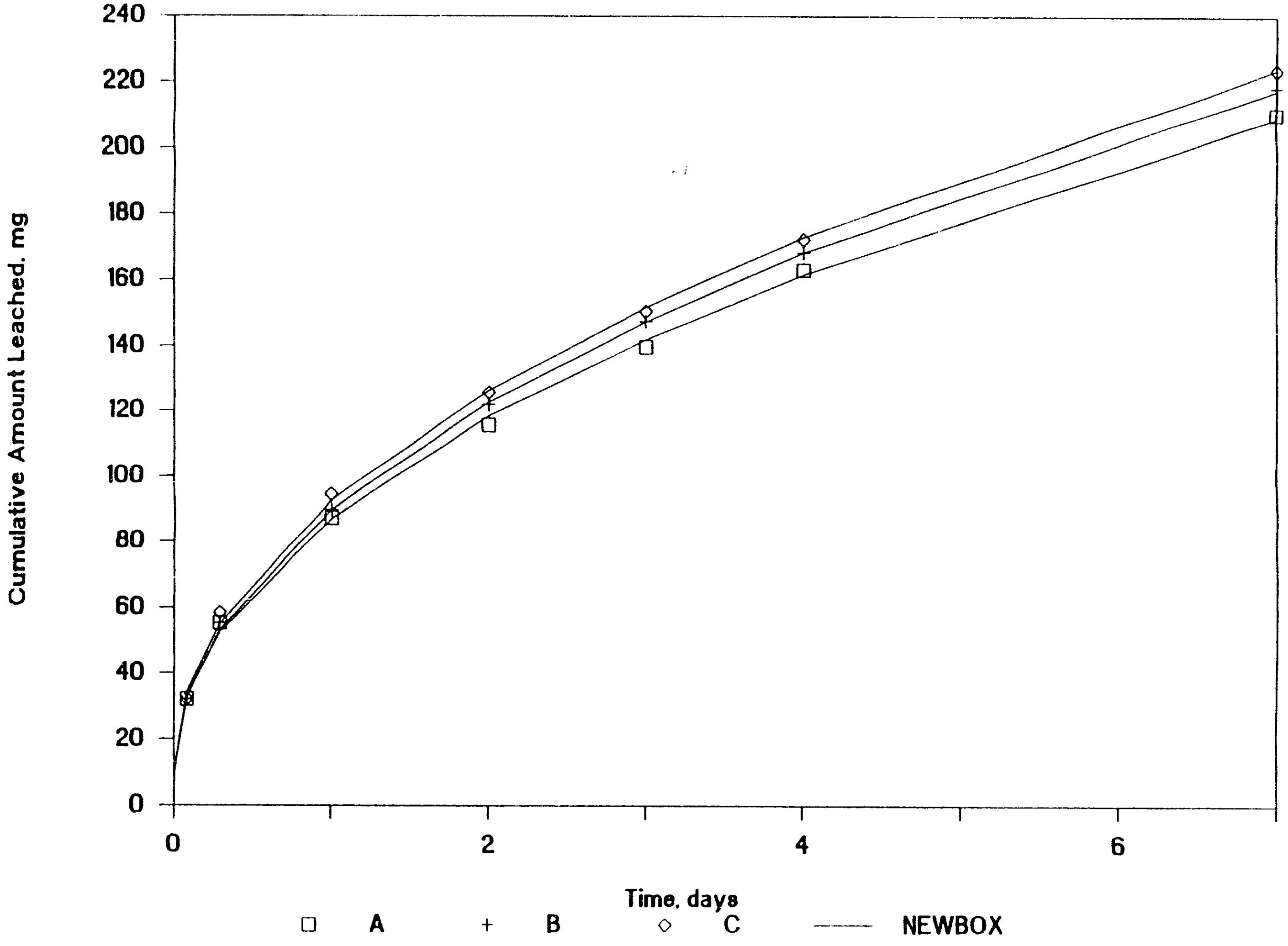
Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.072	1.091	1.059
Rinse =	0.072	0.091	0.059
Ao =	1.000	1.000	1.000
Washoff =	0.010	0.010	0.000
Rinse + Washoff =	0.082	0.101	0.059
Leaching by diffusion control =	0.990	0.990	1.000

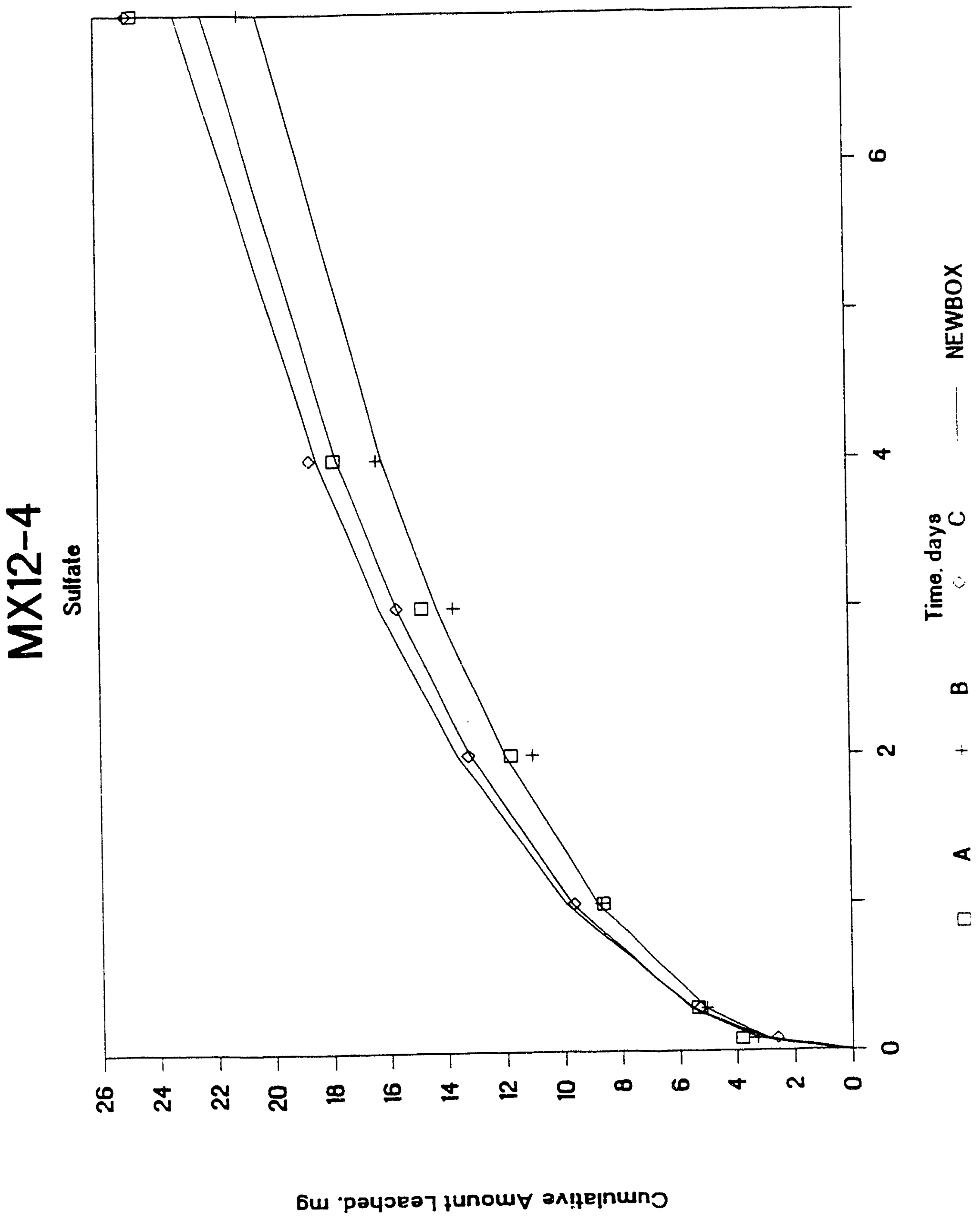
Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.086	0.073	0.076	0.068	0.058	0.067
0.292	0.121	0.127	0.117	0.117	0.118	0.124
1	0.195	0.220	0.200	0.203	0.216	0.222
2	0.267	0.298	0.254	0.276	0.296	0.304
3	0.335	0.355	0.316	0.329	0.350	0.364
4	0.404	0.401	0.377	0.372	0.418	0.412
7	0.560	0.504	0.484	0.469	0.556	0.519


MX12-4
Nitrite
Cumulative Amount Leached, mg
70
60
50
40
30
20
10
0
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

MX12-4

Nitrate + Nitrite




MX12-4
Sulfate
Cumulative Amount Leached, mg
0
2
4
6
8
10
12
14
16
18
20
22
24
26
Time, days
0
2
4
6
□ A
+ B
◇ C
— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX13-9

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.49	4.49
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	2	2	2
Cylinder at start of leaching, Ao =	553	553	553

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	13.3	12.9	12.1
2	0.292	21.0	21.8	20.4
3	1	32.1	33.6	31.2
4	2	42.5	43.7	40.3
5	3	50.6	51.4	47.5
6	4	56.9	58.0	53.9
7	7	70.9	75.1	69.3

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	6	6	6
Std. Dev., mg =	1	1	1
Effective Diff. Coeff., sq. cm/s =	4.92E-09	5.36E-09	4.58E-09
Std. Dev., sq. cm/s =	3.44E-10	4.94E-10	3.31E-10
Leachability Index =	8.308	8.271	8.339
Upper 95% Conf. Int. =	0.031	0.041	0.032
Lower 95% Conf. Int. =	0.029	0.038	0.030

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.003	1.003	1.004
Rinse =	0.003	0.003	0.004
Ao =	1.000	1.000	1.000
Washoff =	0.011	0.011	0.010
Rinse + Washoff =	0.014	0.014	0.013
Leaching by diffusion control =	0.989	0.989	0.990

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.024	0.025	0.023	0.025	0.022	0.023
0.292	0.038	0.036	0.039	0.037	0.037	0.034
1	0.058	0.058	0.061	0.059	0.056	0.055
2	0.077	0.076	0.079	0.079	0.073	0.073
3	0.092	0.091	0.093	0.094	0.086	0.087
4	0.103	0.103	0.105	0.106	0.097	0.098
7	0.128	0.131	0.136	0.136	0.125	0.126

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX13-9

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.49	4.49
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	6	5	6
Cylinder at start of leaching, Ao =	1825	1826	1825

Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	46.4	44.5	40.4
2	0.292	74.1	75.3	69.1
3	1	112.8	116.7	106.3
4	2	148.5	151.7	138.1
5	3	177.2	177.7	161.2
6	4	199.6	201.6	182.6
7	7	253.8	255.6	233.9

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	22	20	19
Std. Dev., mg =	3	5	5
Effective Diff. Coeff., sq. cm/s =	5.67E-09	5.95E-09	4.86E-09
Std. Dev., sq. cm/s =	3.00E-10	5.28E-10	4.60E-10
Leachability Index =	8.246	8.225	8.313
Upper 95% Conf. Int. =	0.023	0.039	0.042
Lower 95% Conf. Int. =	0.022	0.037	0.039

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.003	1.003	1.003
Rinse =	0.003	0.003	0.003
Ao =	1.000	1.000	1.000
Washoff =	0.012	0.011	0.010
Rinse + Washoff =	0.015	0.014	0.014
Leaching by diffusion control =	0.988	0.989	0.990

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.025	0.026	0.024	0.026	0.022	0.024
0.292	0.041	0.039	0.041	0.039	0.038	0.035
1	0.062	0.061	0.064	0.062	0.058	0.056
2	0.081	0.082	0.083	0.083	0.076	0.075
3	0.097	0.097	0.097	0.098	0.088	0.089
4	0.109	0.110	0.110	0.111	0.100	0.101
7	0.139	0.140	0.140	0.142	0.128	0.129

Sulfate Leaching Data
Matrix Run No.: MX13-9

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Sulfate mass = 47.4 mg

Cured Cylinder =	A	B	C
Length, cm =	4.46	4.49	4.49
Diameter, cm =	2.50	2.50	2.50

Sulfate Mass, mg			
Rinse =	0	0	0
Cylinder at start of leaching, Ao =	47	47	47

Amount Leached in Each Interval, mg sulfate

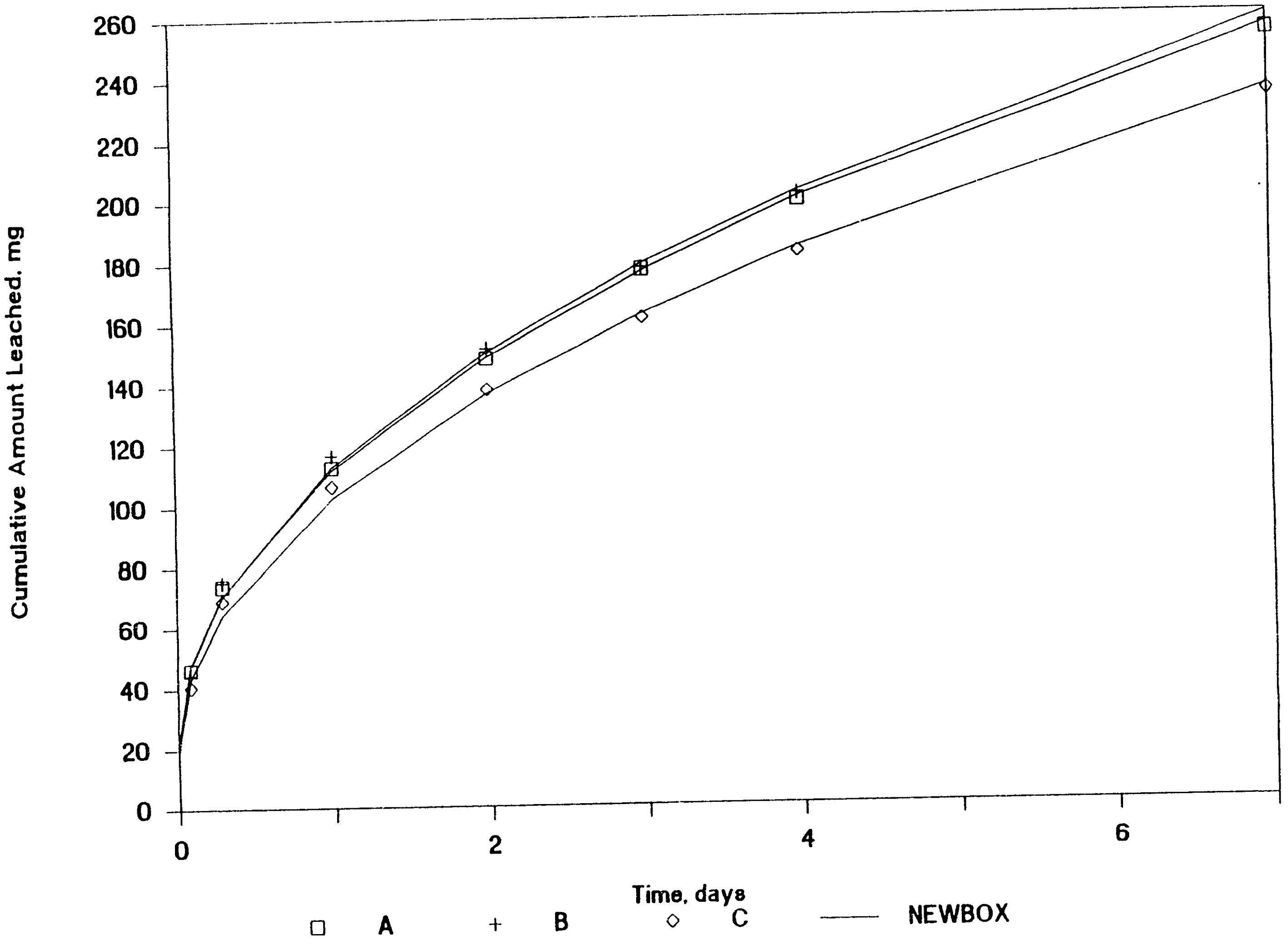
Interval	Time days	A	B	C
1	0.083	0.75	1.92	5.59
2	0.292	1.99	0.95	3.67
3	1	2.19	3.89	3.36
4	2	1.15	2.03	2.16
5	3		2.52	1.43
6	4			2.18
7	7	2.71	3.92	3.58

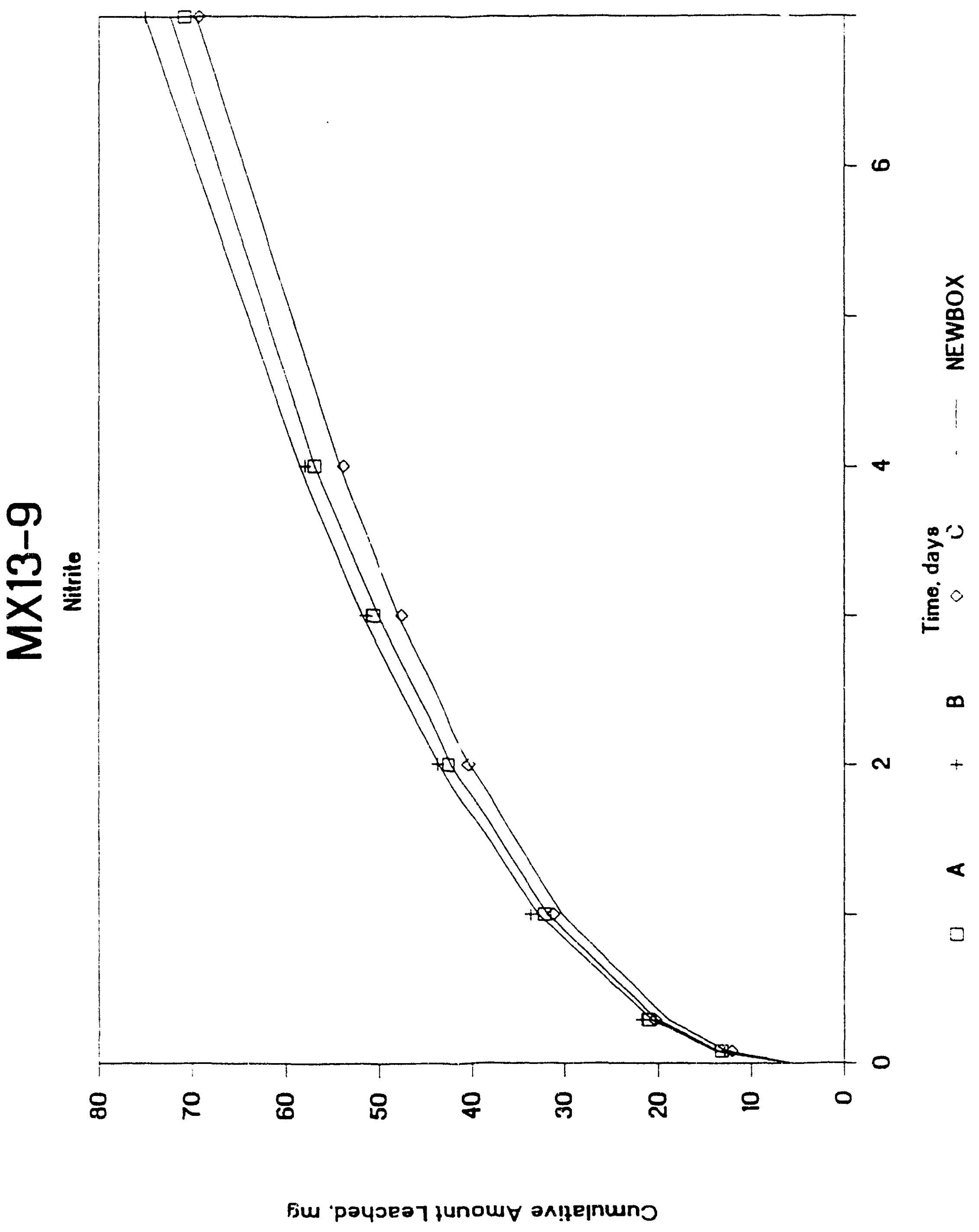
Differential Estimate of D, sq cm/s		A	B	C
1	0.083333	6.50E-09	4.29E-08	3.63E-07
2	0.291666	6.04E-08	1.39E-08	2.06E-07
3	1	2.20E-08	6.93E-08	1.88E-07
4	2	7.48E-09	2.32E-08	1.28E-07
5	3		4.85E-08	1.05E-07
6	4			1.08E-07
7	7	1.71E-08	3.57E-08	9.09E-08

Leachability Index		A	B	C
1	0.083333	8.187	7.368	6.440
2	0.291666	7.219	7.858	6.685
3	1	7.658	7.159	6.726
4	2	8.126	7.634	6.894
5	3		7.315	6.979
6	4			6.968
7	7	7.768	7.448	7.041
	Average	7.792	7.463	6.819
	Std Dev	0.350	0.227	0.197
	95% Conf. limits	0.435	0.238	0.183

MX13-9

Nitrate + Nitrite




MX13-9
Nitrite
Cumulative Amount Leached, mg
0
10
20
30
40
50
60
70
80
0
2
4
6
Time, days
□ A
+ B
◇ C
NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX7-S12

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.91	3.76	3.85
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	28	46	28
Cylinder at start of leaching, Ao =	526	508	527

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	8.5	7.9	7.9
2	0.292	11.4	10.9	11.4
3	1	16.2	17.0	16.8
4	2	21.1	22.0	22.2
5	3	25.1	26.7	27.4
6	4	29.1	30.7	32.1
7	7	38.2	39.4	47.9

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	4	4	3
Std. Dev., mg =	1	1	3
Effective Diff. Coeff., sq. cm/s =	1.19E-09	1.44E-09	1.73E-09
Std. Dev., sq. cm/s =	1.23E-10	9.38E-11	5.01E-10
Leachability Index =	8.924	8.842	8.762
Upper 95% Conf. Int. =	0.046	0.028	0.144
Lower 95% Conf. Int. =	0.043	0.027	0.110

Cumulative Fraction Leached Based on Ao (CFL)	1.054	1.091	1.053
Initially in fresh grout =	0.054	0.091	0.053
Rinse =	1.000	1.000	1.000
Ao =	0.008	0.008	0.005
Washoff =	0.062	0.099	0.058
Rinse + Washoff =	0.992	0.992	0.995
Leaching by diffusion control =			

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.016	0.015	0.016	0.015	0.015	0.014
0.292	0.022	0.021	0.021	0.022	0.022	0.021
1	0.031	0.032	0.034	0.034	0.032	0.034
2	0.040	0.041	0.043	0.045	0.042	0.046
3	0.048	0.049	0.053	0.053	0.052	0.055
4	0.055	0.055	0.060	0.060	0.061	0.062
7	0.073	0.070	0.078	0.076	0.091	0.080

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX7-S12

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	3.91	3.76	3.85
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	105	122	91
Cylinder at start of leaching, Ao =	1726	1709	1740

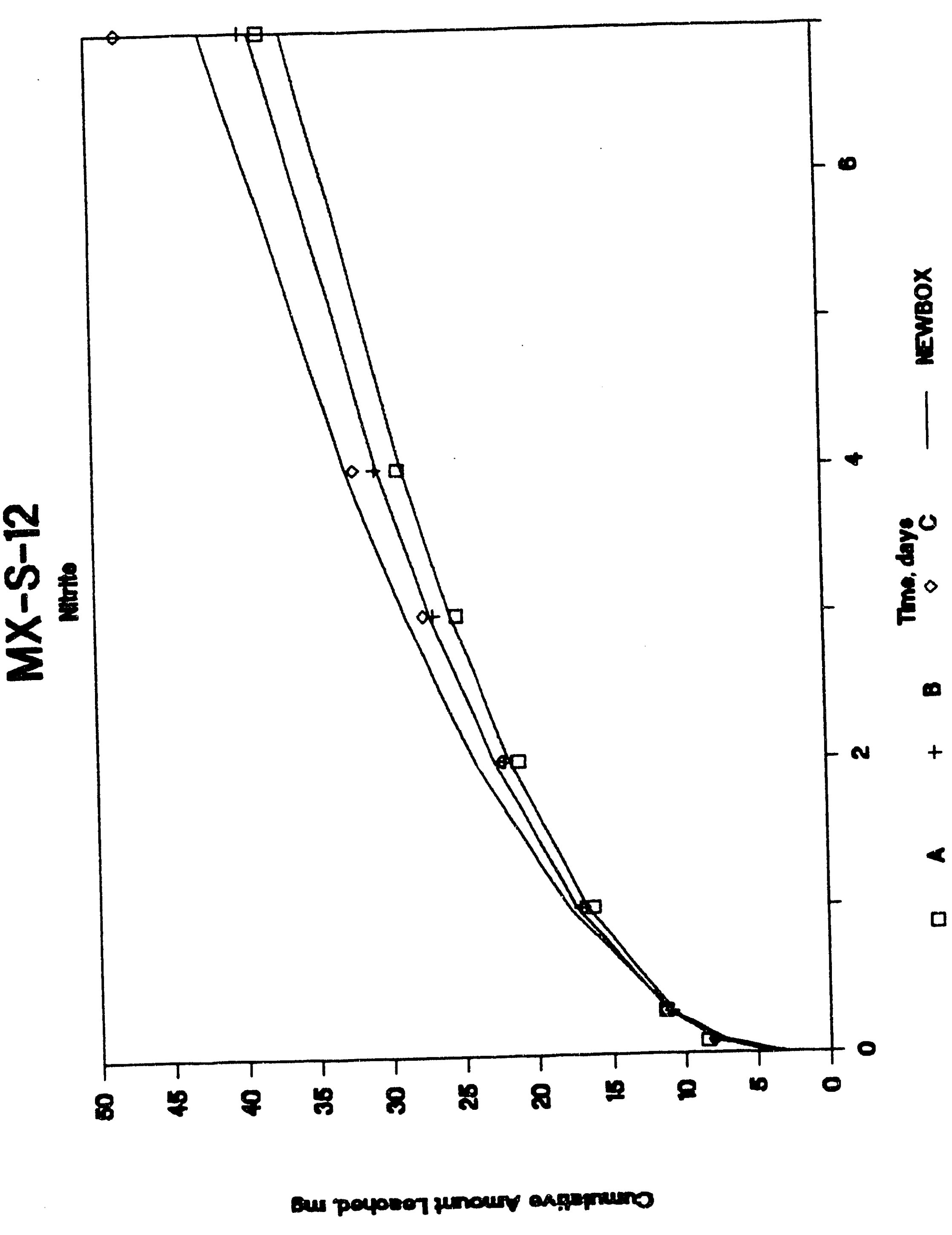
Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	33.8	27.8	28.8
2	0.292	43.2	38.2	41.2
3	1	56.7	58.5	61.2
4	2	73.3	76.6	81.8
5	3	87.0	94.0	101.6
6	4	100.3	109.0	119.5
7	7	137.7	141.8	155.5

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	20	13	12
Std. Dev., mg =	6	3	4
Effective Diff. Coeff., sq. cm/s =	1.20E-09	1.64E-09	1.95E-09
Std. Dev., sq. cm/s =	2.90E-10	1.64E-10	2.65E-10
Leachability Index =	8.921	8.785	8.710
Upper 95% Conf. Int. =	0.116	0.044	0.062
Lower 95% Conf. Int. =	0.094	0.041	0.055

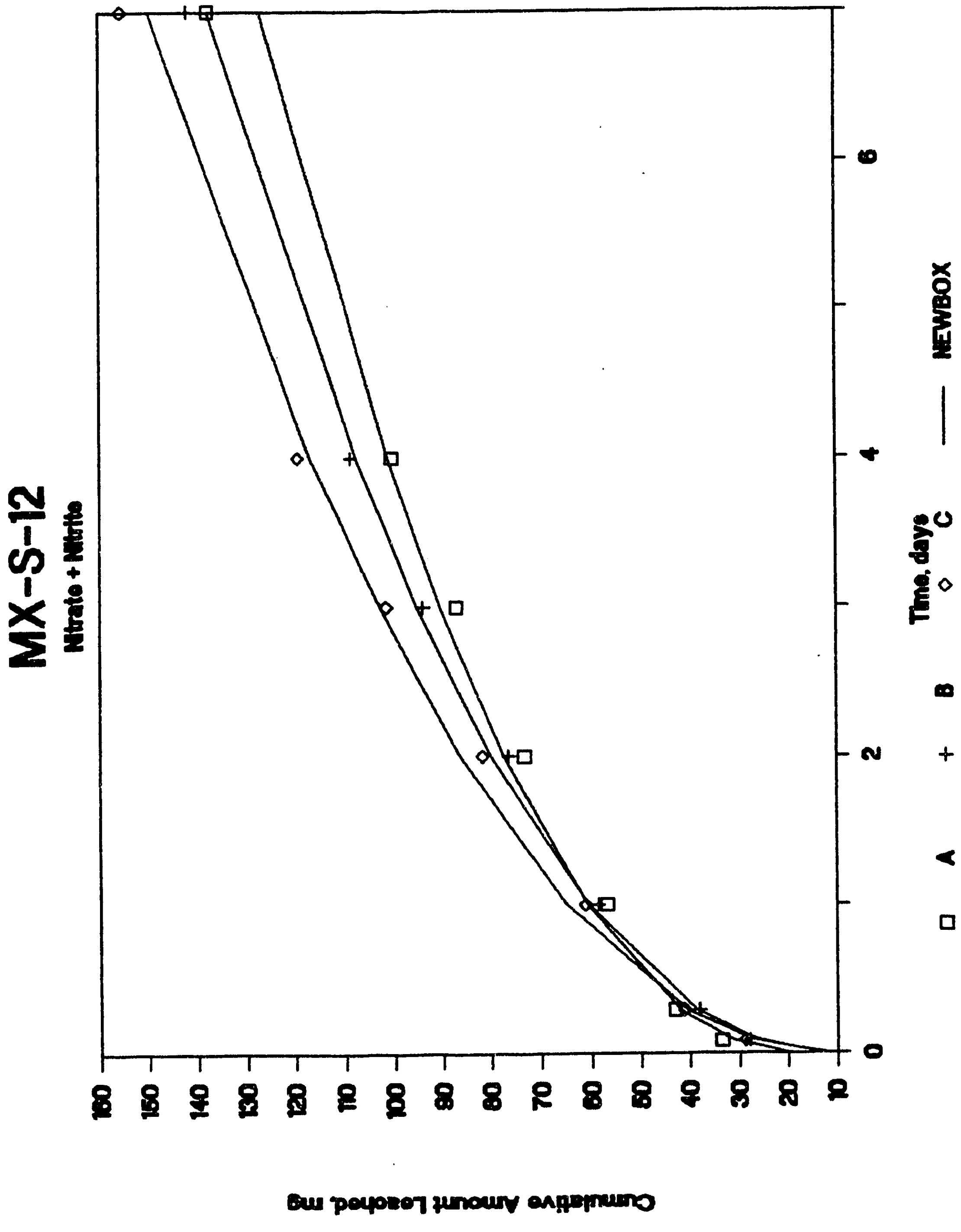
Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.061	1.071	1.053
Rinse =	0.061	0.071	0.053
Ao =	1.000	1.000	1.000
Washoff =	0.011	0.007	0.007
Rinse + Washoff =	0.072	0.079	0.059
Leaching by diffusion control =	0.989	0.993	0.993

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.020	0.018	0.016	0.016	0.017	0.016
0.292	0.025	0.024	0.022	0.023	0.024	0.023
1	0.033	0.035	0.034	0.035	0.035	0.037
2	0.042	0.045	0.045	0.047	0.047	0.050
3	0.050	0.052	0.055	0.056	0.058	0.059
4	0.058	0.059	0.064	0.063	0.069	0.067
7	0.080	0.074	0.083	0.081	0.089	0.086



MX-S-12

Nitrite


MX-S-12
Nitrate + Nitrite
Cumulative Amount Leached, mg
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
0
2
4
6
Time, days
□ A
+ B
◇ C
—— NEWBOX

Nitrite Leaching Data
Matrix Run No.: MX8-S15

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm Nitrite mass = 554.5 mg

Cured Cylinder =	A	B	C
Length, cm =	3.81	3.72	3.73
Diameter, cm =	2.50	2.50	2.50

Nitrite Mass, mg			
Rinse =	53	82	87
Cylinder at start of leaching, Ao =	501	473	467

Cumulative Amount Leached, mg nitrite

Interval	Time days	A	B	C
1	0.083	18.7	10.3	12.0
2	0.292	27.8	14.6	16.7
3	1	41.5	21.8	23.4
4	2	50.1	28.5	31.8
5	3	56.0	33.6	37.8
6	4	61.1	37.7	41.4
7	7	73.3	48.5	55.1

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	14	5	7
Std. Dev., mg =	3	1	1
Effective Diff. Coeff., sq. cm/s =	5.17E-09	2.57E-09	3.15E-09
Std. Dev., sq. cm/s =	1.01E-09	1.60E-10	3.93E-10
Leachability Index =	8.287	8.590	8.502
Upper 95% Conf. Int. =	0.091	0.027	0.056
Lower 95% Conf. Int. =	0.077	0.026	0.051

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.106	1.173	1.186
Rinse =	0.106	0.173	0.186
Ao =	1.000	1.000	1.000
Washoff =	0.028	0.011	0.014
Rinse + Washoff =	0.134	0.184	0.201
Leaching by diffusion control =	0.972	0.989	0.986

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.037	0.042	0.022	0.022	0.026	0.025
0.292	0.056	0.054	0.031	0.031	0.036	0.035
1	0.083	0.076	0.046	0.047	0.050	0.053
2	0.100	0.096	0.060	0.061	0.068	0.068
3	0.112	0.111	0.071	0.072	0.081	0.080
4	0.122	0.123	0.080	0.081	0.089	0.090
7	0.146	0.152	0.103	0.103	0.118	0.114

Nitrate + Nitrite Leaching Data
Matrix Run No.: MX8-S15

Initial grout cylinder prior to phase separation and curing
Length = 4.65 cm Diameter = 2.50 cm NO3 + NO2 mass 1831 mg

Cured Cylinder =	A	B	C
Length, cm =	3.81	3.72	3.73
Diameter, cm =	2.50	2.50	2.50

Nitrate + Nitrite Mass, mg			
Rinse =	136	189	201
Cylinder at start of leaching, Ao =	1695	1642	1630

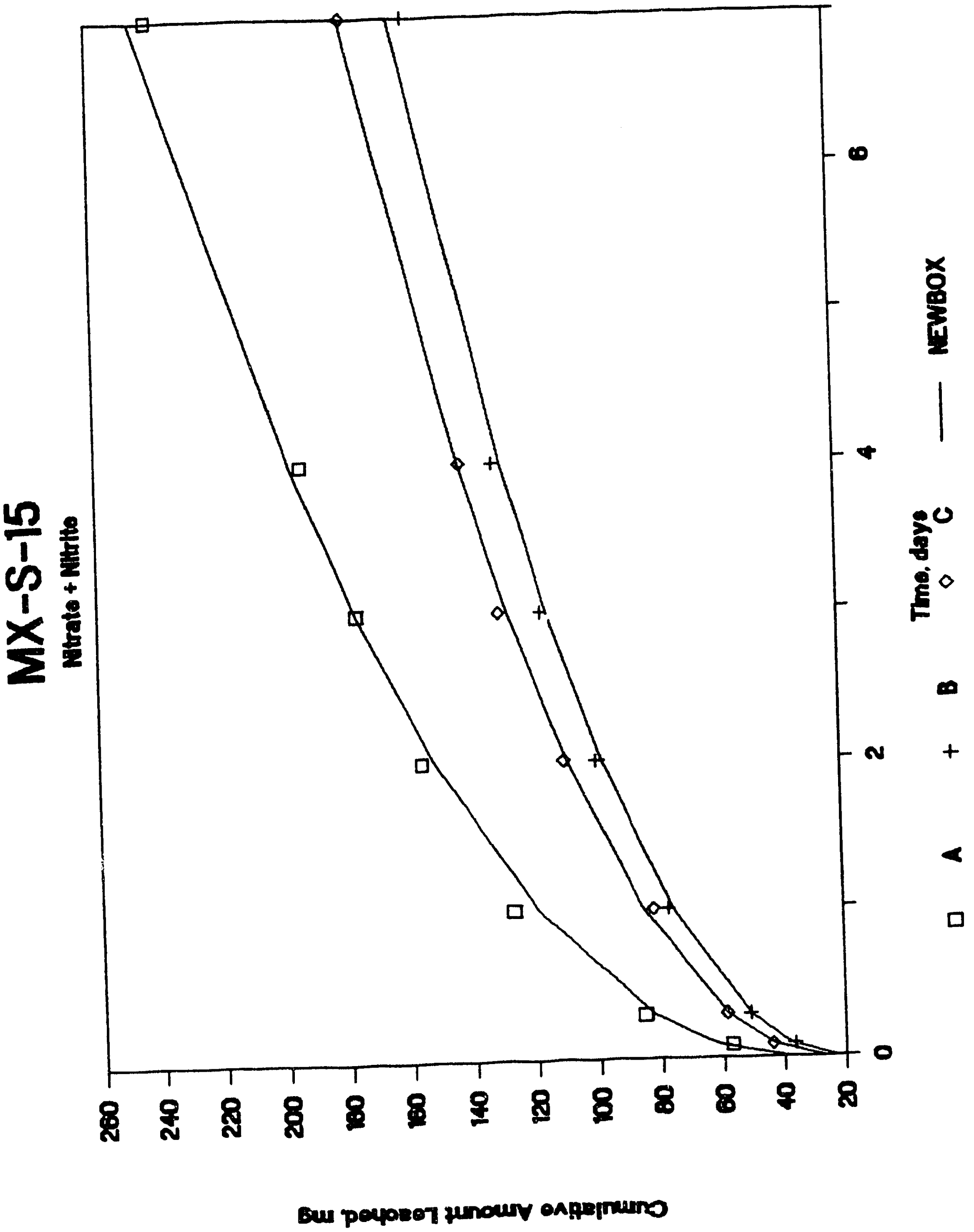
Cumulative Amount Leached, mg (nitrate + nitrite)

Interval	Time days	A	B	C
1	0.083	57.5	36.9	44.1
2	0.292	85.1	51.4	58.9
3	1	127.1	77.3	82.0
4	2	155.5	99.4	109.7
5	3	176.0	116.5	129.9
6	4	193.1	131.1	141.8
7	7	240.1	156.9	177.2

NEWBOX Estimate of Parameters	A	B	C
Washoff, mg =	38	22	27
Std. Dev., mg =	7	3	3
Effective Diff. Coeff., sq. cm/s =	4.90E-09	2.25E-09	2.71E-09
Std. Dev., sq. cm/s =	7.15E-10	1.77E-10	1.87E-10
Leachability Index =	8.310	8.648	8.567
Upper 95% Conf. Int. =	0.066	0.035	0.030
Lower 95% Conf. Int. =	0.059	0.033	0.029

Cumulative Fraction Leached Based on Ao (CFL)			
Initially in fresh grout =	1.080	1.115	1.123
Rinse =	0.080	0.115	0.123
Ao =	1.000	1.000	1.000
Washoff =	0.023	0.014	0.016
Rinse + Washoff =	0.103	0.129	0.140
Leaching by diffusion control =	0.977	0.986	0.984

Time	CFL of A		CFL of B		CFL of C	
days	Obsv'd	Calc'd	Obsv'd	Calc'd	Obsv'd	Calc'd
0.083	0.034	0.036	0.022	0.023	0.027	0.027
0.292	0.050	0.048	0.031	0.031	0.036	0.036
1	0.075	0.070	0.047	0.046	0.050	0.052
2	0.092	0.089	0.061	0.060	0.067	0.067
3	0.104	0.104	0.071	0.070	0.080	0.078
4	0.114	0.116	0.080	0.078	0.087	0.087
7	0.142	0.145	0.096	0.099	0.109	0.109


MX-S-15
Nitrate + Nitrite
Cumulative Amount Leached, mg
260
240
220
200
180
160
140
120
100
80
60
40
20
Time, days
0
2
4
6
□ A
+ B
◇ C
— NEWBOX

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