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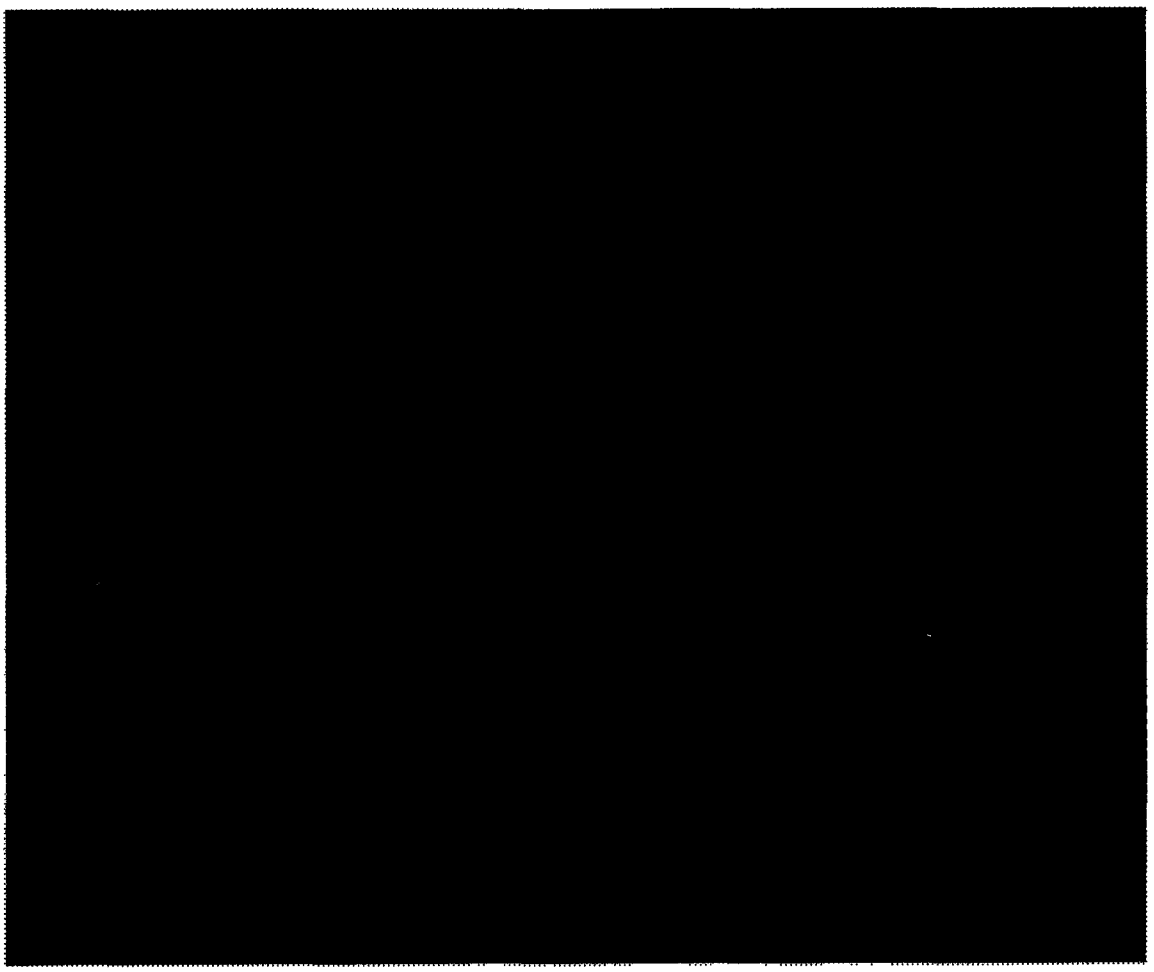
Direct/Delayed Response Project: Quality Assurance Report for Physical and Chemical Analyses of Soils from the Southern Blue Ridge Province of the United States



Aquatic Effects Research Program

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Direct/Delayed Response Project: Quality Assurance Report for Physical and Chemical Analyses of Soils from the Southern Blue Ridge Province of the United States

by

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A Contribution to the
National Acid Precipitation Assessment Program

U.S. Environmental Protection Agency
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Notice

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This document is one volume of a set which fully describes the Direct/Delayed Response Project, Southern Blue Ridge and Northeast soil surveys. The complete document set includes the major data report, quality assurance plan, analytical methods manual, field operations reports, and quality assurance reports. Similar sets are being produced for each Aquatic Effects Research Program component project. Colored covers, artwork, and the use of the project name in the document title serve to identify each companion document set.

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Abstract

The Direct/Delayed Response Project is designed to address the concern over potential acidification of surface waters by atmospheric sulfur deposition within the United States. The Southern Blue Ridge Province Soil Survey was conducted during the summer of 1986 as a synoptic physical and chemical survey to characterize watersheds located in a region of the United States believed to be susceptible to the effects of acidic deposition. This document addresses the quality assurance program and its implementation in the assessment of the verified analytical data base for the Southern Blue Ridge Province Soil Survey. It is addressed primarily to the users of the data base who will be analyzing the data and making various assessments and conclusions relating to the effects of acidic deposition on the soils of the Southern Blue Ridge Province of the United States.

Data quality is assessed by describing the detectability, precision, accuracy (interlaboratory differences), representativeness, completeness, and comparability of the data for the quality assurance samples used throughout the soil survey. The fifty-one parameters in the data base are segregated into nine groups for ease in discussion.

This report is submitted in partial fulfillment of Contract Number 68-03-3249 by Lockheed Engineering & Sciences Company (formerly Lockheed Engineering and Management Services Company), Las Vegas, Nevada, under sponsorship of the U. S. Environmental Protection Agency.

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List of Abbreviations

AA	atomic absorption
AERP	Aquatic Effects Research Program
AC_BACL	barium chloride triethanolamine exchangeable acidity
AC_KCL	potassium chloride exchangeable acidity
AL_AO	acid oxalate extractable aluminum
AL_CD	citrate dithionite extractable aluminum
AL_CL2	extractable aluminum in calcium chloride
AL_KCL	exchangeable aluminum in potassium chloride
AL_PYP	pyrophosphate extractable aluminum
ANOVA	analysis of variance
AS	audit samples
CA_CL	exchangeable calcium in ammonium chloride
CA_CL2	extractable calcium in calcium chloride
CA_OAC	exchangeable calcium in ammonium acetate
CEC	cation exchange capacity
CEC_CL	ammonium chloride cation exchange capacity
CEC_OAC	ammonium acetate cation exchange capacity
CLAY	total clay fraction
CLP	Contract Laboratory Program
CRDL	*contract-required detection limit*
COS	coarse sand fraction
COSI	coarse silt fraction
C_TOT	total carbon
DDRP	Direct/Delayed Response Project
DL	detection limit
DL-QCCS	detection limit quality control check sample
DQO	data quality objective
EGME	ethylene glycol monoethyl ether
EMSL-LV	Environmental Monitoring Systems Laboratory at Las Vegas, Nevada
EPA	U.S. Environmental Protection Agency
ERL-C	Environmental Research Laboratory at Corvallis, Oregon
FD	field duplicate sample
FE_AO	acid oxalate extractable iron
FE_CD	citrate dithionite extractable iron
FE_CL2	extractable iron in calcium chloride
FE_PYP	pyrophosphate extractable iron
FIA	flow injection analysis
FP	flame photometry
FSI	fine silt fraction
FS	fine sand fraction
GIS	geographic information system
IC	*ion chromatography*
ICP	inductively coupled plasma
IDL	instrument detection limit
IFB	invitation for bid
K_CL	exchangeable potassium in ammonium chloride

List of Abbreviations (continued)

K_CL2	extractable potassium in calcium chloride
K_OAC	exchangeable potassium in ammonium acetate
MG_CL	exchangeable magnesium in ammonium chloride
MG_CL2	extractable magnesium in calcium chloride
MG_OAC	exchangeable magnesium in ammonium acetate
MOIST	air-dry soil moisture
MS	medium sand fraction
NA_CL	exchangeable sodium in ammonium chloride
NA_CL2	extractable sodium in calcium chloride
NA_OAC	exchangeable sodium in ammonium acetate
NAPAP	National Acid Precipitation Assessment Program
NCC	National Computer Center
NSWS	National Surface Water Survey
N_TOT	total nitrogen
ORNL	Oak Ridge National Laboratory
PD	preparation duplicate sample
PE	performance evaluation
PH_002M	pH in 0.002M calcium chloride
PH_01M	pH in 0.01M calcium chloride
PH_H2O	pH in water
QA	quality assurance
QC	quality control
QCCS	quality control check sample
RS	routine samples
RSD	relative standard deviation
SAND	total sand fraction
SAS	Statistical Analysis Systems, Inc.
SBRP	Southern Blue Ridge Province
SCS	Soil Conservation Service
SD	standard deviation
SDL	system detection limit
S/H	sampling class/horizon
SILT	total silt fraction
SO4_0	zero mg S/L sulfate isotherm parameter
SO4_2	two mg S/L sulfate isotherm parameter
SO4_4	four mg S/L sulfate isotherm parameter
SO4_8	eight mg S/L sulfate isotherm parameter
SO4_16	sixteen mg S/L sulfate isotherm parameter
SO4_32	thirty-two mg S/L sulfate isotherm parameter
SO4_H2O	water-extractable sulfate
SO4_PO4	phosphate-extractable sulfate
SOW	statement of work
SP_SUR	specific surface
S_TOT	total sulfur
USDA	U.S. Department of Agriculture
VCOS	very coarse sand fraction
VFS	very fine sand fraction

Section 1

Introduction

Overview of the Survey

The Direct/Delayed Response Project (DDRP) is an integral part of the Aquatic Effects Research Program (AERP) of the U.S. Environmental Protection Agency (EPA). The AERP is conducted under the federally mandated National Acid Precipitation Assessment Program (NAPAP) and addresses the concern over potential acidification of surface waters by atmospheric deposition within the United States. The DDRP is administered by the EPA Environmental Research Laboratory in Corvallis, Oregon (ERL-C).

The overall purpose of DDRP is to characterize geographic regions of the United States by predicting the long-term response of watersheds and surface waters to acidic deposition. The DDRP has been designed under the concept of regionalized integrative surveys which initially is approached from a large region of study and leads to the selection and study of regionally characteristic systems. These systems can be assessed through detailed, process-oriented research which will aid in the understanding of the underlying mechanisms responsible for observed effects. The projected responses of watershed systems typical of the regional population can then be extrapolated to a larger regional or national scale.

The Southern Blue Ridge Province (SBRP) of the United States was selected for study because of its suspected sensitivity to acidic deposition. In defining the regions of concern, the intent was to focus on regionally representative watersheds that are potentially sensitive to acidic deposition and that exhibit a wide contrast in soil and watershed characteristics and in levels of deposition. The SBRP Soil Survey focused on the Blue Ridge Mountains geographic area in eastern Tennessee, north-central Georgia, northwestern South Carolina, and western North Carolina. Special interest watersheds in North Carolina and Virginia were also sampled as part of the survey.

The EPA is assessing the role that atmospheric deposition of sulfur plays in controlling long-term acidification of surface waters (EPA, 1985). Recent trend analyses have indicated that the rate of sulfur deposition is slowly declining in the Northeastern United States but is increasing in the Southeastern United States. If a "direct" response exists between increasing sulfur deposition and decreasing surface water alkalinity, then the impact of current effects on surface water probably would increase with increasing levels of deposition, and conditions could improve if the levels of deposition decline. If surface water chemistry changes in a "delayed" manner, e.g., due to chemical changes in the watershed, then future changes in surface water chemistry (even with stable or declining rates of deposition) become difficult to predict. This range of potential effects has clear and significant implications to public policy decisions on sulfur emissions control strategies.

Specific goals of DDRP are to (1) define physical, chemical, and mineralogical characteristics of the soils and define other watershed characteristics across the regions of concern, (2) assess the variability of these characteristics, (3) determine which of these characteristics are most strongly related to surface-water chemistry, (4) estimate the relative importance of key watershed processes in controlling surface water chemistry across the regions of concern, and (5) classify the sample of watersheds with regard to their

response to sulfur deposition and extrapolate the results from the sample of watersheds to the regions of concern.

A variety of data sources and methods of analysis will be used to address the objectives of DDRP. In addition to the data collected during DDRP, other data sources include the following data bases:

- National Surface Water Survey (NSWS) [water chemistry data]
- Acid Deposition Data Network (ADDNET), including GEOECOLOGY [atmospheric precipitation chemistry data]
- Soil Conservation Service (SCS) Soils-5 [soil physical and chemical data]
- Adirondack Watershed [whole watershed chemistry]
- Topographic and Acid Deposition System (ADS) [total sulfur deposition data]
- U.S. Geological Survey [runoff data]

Also, data from EPA long-term monitoring sites, episodic event monitoring sites, and intensively studied watersheds will be used in the data analysis. The data that are collected will be analyzed at three levels:

- Level I -- System description and statistical analysis
- Level II -- Single factor response-time estimates
- Level III -- Dynamic systems modeling

Field and laboratory data collected in DDRP are included in the Level I system description. Next, these data are used in Level II to develop single factor estimates of the response time of watershed properties, e.g., sulfate adsorption capacity, to acidic deposition. The detailed data from special interest watersheds are used in Level III to calibrate three dynamic simulation models, MAGIC (Cosby et al., 1984), ILWAS (Chen et al., 1984), and Trickle-Down (Schnoor et al., 1984), that predict regional responses to acidic deposition.

The soil sampling task leader at ERL-C had overall responsibility for the soil mapping and sampling, including quality assurance/-quality control (QA/QC) for site selection, soil characterization, and collection of bulk samples and clods. Logistical support and analytical QA/QC services were provided by the EPA Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV). There were nine sampling crews, each consisting of three to four soil scientists, involved in the SBRP sampling phase. In addition to collecting 5.5-kilogram routine soil samples, each sampling crew collected one duplicate sample per day for QA purposes. Details of the soil mapping and sampling are contained in a separate QA report (Coffey et al., 1987).

As part of the DDRP, two preparation laboratories were established in the SBRP region to process soil samples collected by the sampling crews and to perform preliminary analyses on these samples. The preparation laboratories were located within the soil science departments at the following land grant universities:

- University of Tennessee, Knoxville, Tennessee
- Clemson University, Clemson, South Carolina

The handling of soil samples at each preparation laboratory is discussed in a separate QA report (Haren and Van Remortel, 1987). Bulk samples were processed, homogenized, and subsampled. Air-dry moisture content, percent rock fragments in the 2- to 20-millimeter fraction, and inorganic carbon were determined. In addition, the bulk density of replicate soil clods was estimated. Approximately 500-gram analytical samples were derived from the homogenized air-dry bulk soil samples. The analytical samples were grouped into batches and were randomized within each batch. Field duplicates, natural audit samples from EMSL-LV, and a preparation duplicate were placed in each batch for QA purposes. The batches were distributed to three analytical laboratories contracted by EPA.

The QA/QC measures were applied in order to maintain consistency in the soil sampling, preparation, and analysis protocols. This ensured that the soil sample analyses would yield results of known quality. The sampling crews and preparation laboratory personnel received training on their respective activities. The QA personnel from EMSL-LV and ERL-C conducted on-site systems audits of the sampling crews, preparation laboratories, and contract analytical laboratories. Weekly communication between QA personnel and laboratory personnel was established to identify, discuss, and resolve issues. Survey participants attended an exit meeting held in Park City, Utah, in July 1986. The purposes of the meeting were to review the mapping, sampling, and preparation activities, resolve any remaining issues, and generate suggestions for future surveys.

The integrity of the QA program affects the ultimate quality of data derived from physical, chemical, and mineralogical analyses of the soil samples. This level of quality enables potential users of the data to determine whether the data meet their specific needs. In addition, the QA program was conceived as a means to ensure that the data are comparable within and across the regions of concern. Soils were described, sampled, and processed according to documented protocols (Bartz et al., 1987) and the contract laboratory analyses were conducted according to documented protocols (Cappo et al., 1987) under three separate EPA solicitations.

Mineralogical analyses are being performed on about 10 percent of the routine samples, including semiquantitative X-ray diffraction and X-ray fluorescence. Data from these analyses will be evaluated in a forthcoming EPA report.

Organization of the Report

This document has been organized into four main sections. The first section provides an overview of DDRP objectives and the SBRP analytical data base. The second section addresses the overall QA program, its relation to data quality assessment, and the use of QA/QC samples during the various stages of data collection. The third section provides results and discussion concerning the QA data analysis and the internal verification checks for eight parameter groups. The fourth section addresses the conclusions and recommendations that have been generated from these findings, particularly in regard to issues of concern, improvement in QA design, and preparation for QA efforts in the DDRP Mid-Appalachian Soil Survey and other future surveys.

Data quality is discussed in terms of detectability, internal consistency, precision, accuracy (interlaboratory differences), representativeness, completeness, and comparability. The relationship of data quality achievements to the data quality objectives (DQOs) established at the beginning of the program is evaluated.

Description of Parameter Groups

The DDRP QA staff organized the 51 analytical parameters into nine groups and subsequently evaluated each group independently according to the DQOs specified for the SBRP survey. The nine parameter groups are briefly summarized below:

(1) Moisture, Specific Surface, and Particle Size -- The air-dry soil moisture content is determined in order to place all subsequent aliquots on an oven-dry weight basis. Specific surface is measured in mineral soils using a gravimetric saturation method and is correlated with data for cation exchange capacity, sulfate adsorption and desorption, and clay mineralogy. Particle size analysis is performed on the less than 2-millimeter size fractions of mineral soils for characterization and classification purposes.

(2) Soil pH -- The pH is an indication of free hydrogen ion activity. The pH measurements are determined in three soil suspensions: deionized water, 0.01M calcium chloride, and 0.002M calcium chloride.

(3) Exchangeable Cations in Ammonium Chloride -- The exchangeable cations (calcium, magnesium, potassium, and sodium) are extracted during the cation exchange capacity (CEC) determinations and can be used to calculate the percent base saturation of the soil and to define selectivity coefficients and cation pools for the DDRP models.

(4) Exchangeable Cations in Ammonium Acetate -- The exchangeable cations (calcium, magnesium, potassium, and sodium) are extracted during the CEC determinations and can be used to calculate the percent base saturation of the soil and to define selectivity coefficients and cation pools for the DDRP models.

(5) Cation Exchange Capacity and Exchangeable Acidity -- The CEC indicates the ability of a soil to adsorb cations, especially the exchangeable basic cations mentioned above. The CEC is highly correlated with the buffering capacity of the soil. Two saturating solutions for the exchangeable cation component are used: buffered ammonium acetate solution to measure "total" CEC and neutral ammonium chloride solution to measure "effective" CEC. Exchangeable acidity is a measure of the exchangeable cations, i.e., hydrogen and aluminum, that are held on a soil particle surface, in contrast to the active acidity of these cations in solution. Two methods of analysis for acidity are used: a buffered barium chloride triethanolamine extraction and a neutral potassium chloride extraction. The first method is a back-titration which indicates "total" exchangeable acidity, including aluminum. The second method is a direct titration which estimates "effective" exchangeable acidity. Exchangeable aluminum was also determined in potassium chloride.

(6) Extractable Cations in Calcium Chloride -- Lime potential [pH - 1/2 pCa] is used in lieu of base saturation as an input for certain predictive models. Aluminum potential [3pH - pAl] is another important characteristic for watershed modeling. The soil is extracted with 0.002M calcium chloride and analyzed for calcium and aluminum concentrations. The magnesium, potassium, sodium, and iron concentrations also are determined and are compared to cation concentrations in other extracts.

(7) Extractable Iron and Aluminum -- The presence of iron and aluminum is highly correlated to sulfate adsorption. Each of three extractions yields an estimate of a specific iron or aluminum fraction: sodium pyrophosphate which estimates organic iron and aluminum; acid oxalate which estimates organic iron and aluminum plus sesquioxides; and citrate-dithionite which estimates nonsilicate iron and aluminum.

(8) Extractable Sulfate and Sulfate Adsorption Isotherms -- Sulfate is determined in two different extracts: deionized water, which estimates interstitial and loosely-bound sulfate; and 500 mg P/L as sodium phosphate, which estimates the readily extractable sulfate on the anion exchange sites. The ability of soil to adsorb sulfate is related to anion adsorption capacity. Isotherms are developed by placing soil samples in six magnesium sulfate solutions of different concentrations: 0, 2, 4, 8, 16, and 32 mg S/L. A determination is made of the amount of sulfate remaining in solution after one- hour contact with the soil and subtraction yields the net sulfate sorption. The isotherms represent the maximum "stable" sulfate adsorption capacity of the soil under laboratory conditions and are used to predict changes in sorbed and dissolved sulfate as a result of altered deposition.

(9) Total Carbon, Nitrogen, and Sulfur -- Total carbon and nitrogen are closely related to the type and amount of soil organic matter. Total sulfur is used as a benchmark to monitor future inputs of anthropogenic sulfur.

Description of Parameters

Throughout this document, parameters are referenced either by a data-variable or descriptive parameter name. A list of data-variable parameters and their corresponding descriptions based on a similar presentation in Turner et al. (1987) is given in Table 1-1. The order of the parameters is consistent with their order of presentation in this report.

Table 1-1. Analytical Parameters Measured in the Southern Blue Ridge Province Soil Survey

Parameter	Description of Parameter
MOIST	Percent air-dry soil moisture measured at the analytical laboratory and expressed as a percentage on an oven-dry weight basis. Mineral soils were dried at 105°C, organic soils at 60°C.
SP_SUR	Specific surface area determined by a gravimetric method of saturation with ethylene glycol monoethyl ether (EGME).
SAND	Total sand is the portion of the sample with particle diameter between 0.05 mm and 2.0 mm. It was calculated as the summation of percentages for individual sand fractions: VCOS + COS + MS + FS + VFS.
VCOS	Very coarse sand is the sand fraction between 1.0 mm and 2.0 mm. It was determined by sieving the sand which had been separated from the silt and clay.
COS	Coarse sand is the sand fraction between 0.5 mm and 1.0 mm. It was determined by sieving the sand which had been separated from the silt and clay.
MS	Medium sand is the sand fraction between 0.25 mm and 0.50 mm. It was determined by sieving the sand which had been separated from the silt and clay.
FS	Fine sand is the sand fraction between 0.10 mm and 0.25 mm. It was determined by sieving the sand which had been separated from the silt and clay.
VFS	Very fine sand is the sand fraction between 0.05 mm and 0.10 mm. It was determined by sieving the sand which had been separated from the silt and clay.
SILT	Total silt is the portion of the sample with particle diameter between 0.002 mm and 0.05 mm. It was calculated by subtracting from 100 percent the sum of the total sand and clay.
COSI	Coarse silt is the silt fraction between 0.02 mm and 0.05 mm. It was calculated by subtracting the fine silt fraction from the total silt.
FSI	Fine silt is the silt fraction between 0.002 mm and 0.02 mm. It was determined by the pipet method (USDA/SCS, 1984) and was calculated by subtracting the clay fraction from the less than 0.02 mm fraction.
CLAY	Total clay is the portion of the sample with particle diameter of less than 0.002 mm and is determined using the pipet method.
PH_H20	pH determined in a deionized water extract using a 1:1 mineral soil to solution ratio and 1:5 organic soil to solution ratio. The pH was measured with a pH meter and combination electrode.
PH_002M	pH determined in a 0.002M calcium chloride extract using a 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio. The pH was measured with a pH meter and combination electrode.
PH_01M	pH determined in a 0.01M calcium chloride extract using a 1:1 mineral soil to solution ratio and 1:5 organic soil to solution ratio. The pH was measured with a pH meter and combination electrode.
CA_CL	Exchangeable calcium determined with an unbuffered 1M ammonium chloride solution. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
MG_CL	Exchangeable magnesium determined with an unbuffered 1M ammonium chloride solution. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
K_CL	Exchangeable potassium determined with an unbuffered 1M ammonium chloride solution. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry was specified.
NA_CL	Exchangeable sodium determined with an unbuffered 1M ammonium chloride solution. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.

(continued)

Table 1-1. Continued.

Parameter	Description of Parameter
CA_OAC	Exchangeable calcium determined with 1M ammonium acetate solution buffered at pH 7.0. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
MG_OAC	Exchangeable magnesium determined with 1M ammonium acetate solution buffered at pH 7.0. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
K_OAC	Exchangeable potassium determined with 1M ammonium acetate solution buffered at pH 7.0. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry was specified.
NA_OAC	Exchangeable sodium determined with 1M ammonium acetate solution buffered at pH 7.0. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
CEC_CL	Cation exchange capacity determined with an unbuffered 1M ammonium chloride solution is the effective CEC which occurs at approximately the field pH, when combined with the acidity component. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Samples were analyzed for ammonium content by one of three methods: automated distillation/titration; manual distillation / automated titration; or ammonium displacement / flow injection analysis.
CEC_OAC	Cation exchange capacity determined with 1M ammonium acetate solution buffered at pH 7.0 is the theoretical estimate of the maximum potential CEC for a specific soil, when combined with the acidity component. A 1:26 mineral soil to solution ratio and 1:52 organic soil to solution ratio were used. Samples were analyzed for ammonium content by one of three methods: automated distillation/titration; manual distillation / automated titration; or ammonium displacement / flow injection analysis.
AC_KCL	Effective exchangeable acidity determined by titration in an unbuffered 1M potassium chloride extraction using a 1:20 soil to solution ratio.
AC_BACL	Total exchangeable acidity determined by titration in a buffered (pH 8.2) barium chloride triethanolamine extraction using a 1:30 soil to solution ratio.
AL_KCL	Extractable aluminum determined by an unbuffered 1M potassium chloride extraction using a 1:20 soil to solution ratio. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
CA_CL2	Extractable calcium determined by a 0.002M calcium chloride extraction. A 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio were used. The calcium is used to calculate lime potential. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
MG_CL2	Extractable magnesium determined by a 0.002M calcium chloride extraction. A 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
K_CL2	Extractable potassium determined by a 0.002M calcium chloride extraction. A 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio were used. Atomic absorption spectrometry was specified.
NA_CL2	Extractable sodium determined by a 0.002M calcium chloride extraction. A 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
FE_CL2	Extractable iron determined by a 0.002M calcium chloride extraction. A 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio were used. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
AL_CL2	Extractable aluminum determined by a 0.002M calcium chloride extraction. A 1:2 mineral soil to solution ratio and 1:10 organic soil to solution ratio were used. The aluminum concentration obtained from this procedure is used to calculate aluminum potential. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.

(continued)

Table 1-1. Continued.

Parameter	Description of Parameter
FE_PYP	Extractable iron determined by a 0.1M sodium pyrophosphate extraction using a 1:100 soil to solution ratio. The pyrophosphate extract estimates organically-bound iron. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
AL_PYP	Extractable aluminum determined by a 0.1M sodium pyrophosphate extraction using a 1:100 soil to solution ratio. The pyrophosphate extract estimates organically-bound aluminum. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
FE_AO	Extractable iron determined by an ammonium oxalate - oxalic acid extraction using a 1:100 soil to solution ratio. The acid oxalate extract estimates organic and amorphous iron oxides. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
AL_AO	Extractable aluminum determined by an ammonium oxalate - oxalic acid extraction using a 1:100 soil to solution ratio. The acid oxalate extract estimates organic and amorphous aluminum oxides. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
FE_CD	Extractable iron determined by a sodium citrate - sodium dithionite extraction using a 1:30 soil to solution ratio. The citrate dithionite extract estimates non-silicate iron. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
AL_CD	Extractable aluminum determined by a sodium citrate - sodium dithionite extraction using a 1:30 soil to solution ratio. The citrate dithionite extract estimates non-silicate aluminum. Atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry was specified.
SO4_H2O	Extractable sulfate determined with a double deionized water extract. This extraction approximates the sulfate which will readily enter the soil solution and uses a 1:20 soil to solution ratio. Ion chromatography was specified.
SO4_PO4	Extractable sulfate determined with a 0.016M sodium phosphate (500 mg P/L) extract. This extraction approximates the total amount of adsorbed sulfate and uses a 1:20 soil to solution ratio. Ion chromatography was specified.
SO4_0	Sulfate remaining in a 0 mg S/L solution following equilibration with a 1:5 mineral soil to solution ratio and 1:20 organic soil to solution ratio. The data are used to develop sulfate isotherms. Ion chromatography was specified.
SO4_2	Sulfate remaining in a 2 mg S/L solution following equilibration with a 1:5 mineral soil to solution ratio and 1:20 organic soil to solution ratio. The data are used to develop sulfate isotherms. Ion chromatography was specified.
SO4_4	Sulfate remaining in a 4 mg S/L solution following equilibration with a 1:5 mineral soil to solution ratio and 1:20 organic soil to solution ratio. The data are used to develop sulfate isotherms. Ion chromatography was specified.
SO4_8	Sulfate remaining in a 8 mg S/L solution following equilibration with a 1:5 mineral soil to solution ratio and 1:20 organic soil to solution ratio. The data are used to develop sulfate isotherms. Ion chromatography was specified.
SO4_16	Sulfate remaining in a 16 mg S/L solution following equilibration with a 1:5 mineral soil to solution ratio and 1:20 organic soil to solution ratio. The data are used to develop sulfate isotherms. Ion chromatography was specified.
SO4_32	Sulfate remaining in a 32 mg S/L solution following equilibration with a 1:5 mineral soil to solution ratio and 1:20 organic soil to solution ratio. The data are used to develop sulfate isotherms. Ion chromatography was specified.
C_TOT	Total carbon determined by rapid oxidation followed by thermal conductivity detection using an automated CHN analyzer. Total carbon can be used to characterize the amount of organic material in the soil.
N_TOT	Total nitrogen determined by rapid oxidation followed by thermal conductivity detection using an automated CHN analyzer. Total nitrogen can be used to characterize the organic material in the soil.
S_TOT	Total sulfur determined by automated sample combustion followed by infrared detection or titration of evolved sulfur dioxide.

Section 2

Quality Assurance Program

Quality assurance has been defined as "those operations and procedures which are undertaken to provide measurement data of stated quality with a stated probability of being right" (Taylor, 1987). The QA/QC procedures for the SBRP survey were designed to ensure that the best possible data were collected and that the quality of the data could be evaluated and documented. These procedures included the preparation of written protocols and manuals describing: (1) soil mapping, sampling, preparation and analysis, (2) application of QA/QC during field and laboratory activities, and (3) verification of the descriptive and analytical data. The protocols were tested and implemented in the survey. Specific aspects of the QA program are described in the following subsections.

Selection of Analytical Laboratories

Specifications for the laboratory analysis were defined during the initial development of the QA program. The estimated number of samples to be analyzed and the schedule of sample collection were defined during logistics planning. No single EPA laboratory had the analytical capabilities or resources to provide the required analytical services, hence, these services were obtained through solicitations with commercial analytical laboratories. The Contract Laboratory Program (CLP) had already been established to support the hazardous waste monitoring activities of EPA. The use of multiple analytical laboratories, however, required that the selection and documentation of analytical methods and QA activities had to be carefully implemented and monitored to ensure consistent and adequate performance in all laboratories. The solicitation process involved the following activities:

- preparation of a detailed statement of work (SOW) which defined the analytical and QA/QC requirements in a contractual format.
- preparation and advertisement of an invitation for bid (IFB) to solicit analytical support.
- an evaluation of all bidders within a competitive range to ensure that qualified laboratories were selected.

Statement of Work

Monitoring of analytical performance at each contractor analytical laboratory was necessary in order to minimize data variability both within and among the laboratories. Although the DDRP Analytical Methods Manual (Cappo et al., 1987) and the DDRP QA Plan (Bartz et al., 1987) were drafted in the early phases of the planning process, the methods and QA/QC requirements had to be restructured in a SOW in order to obtain support services. This involved careful review of the analytical and logistical requirements, i.e., reporting and QC stipulations, to ensure their clarity in the SOW and their ability to be satisfied according to contract specifications. The primary administrative protocols in the SOW were as follows:

- A contractor could bid on the analysis of one or more bid lots (600 samples per bid lot) that would be delivered to the analytical laboratory at a maximum rate of 60 samples per week, grouped in batches of approximately 42 samples per batch.

- Delivery of the completed data package by the contractor was required within 60 days of sample receipt for Solicitation 1 and within 45 days of sample receipt for Solicitations 2 and 3. An incentive for early delivery of data and a consideration for late delivery of data were established.

- Failure of the contractor to provide adequate QA/QC data and deliverables as required by the SOW resulted in a penalty of up to 15 percent of the bid price initially withheld. All analytical laboratories eventually were paid the entire 15 percent withholding after the data were verified and any confirmation/reanalysis requests were serviced.

The analytical laboratories were required to follow the methods exactly as specified in the SOW. The project officer was authorized to provide technical clarifications for the contractor laboratory, but contractual changes were made only with the approval of the EPA contract officer.

Performance Evaluations

The IFB was advertised in *Commerce Business Daily.* All interested laboratories received a set of pre-award performance evaluation (PE) samples as the next step in the qualification process. These laboratories were required to analyze PE samples and to report the results within 25 days after sample receipt. The PE samples were intended to represent soil samples at both the low and high analyte concentrations expected for the survey. Data packages received from each laboratory were evaluated and graded on the accuracy of analytical data as well as the quality and completeness of the data package using the scoring sheet provided in the DDRP QA Plan (Bartz et al., 1987). This procedure identified those laboratories that could not successfully perform the analytical tasks.

All laboratories successfully passing the PE sample evaluation were audited by EPA representatives in order to verify the ability of these laboratories to meet the contractual requirements. The EPA team determined whether or not each analytical laboratory had adequate facilities, equipment, personnel, and technical capabilities to analyze samples in accordance with the SOW. These visits also provided an opportunity to clarify contractual specifications with laboratory personnel and to identify deficiencies that were observed during the PE phase.

Four laboratories successfully passed both the performance and on-site evaluations and were awarded contracts to provide analytical services for the SBRP survey. During the routine analysis of samples, however, it was determined that one of the laboratories could not maintain the specified level of quality in the analyses and this laboratory was eventually disqualified. The remaining samples in archive were retrieved by QA staff and were redistributed to two of the other three laboratories for analysis. Data from the disqualified fourth laboratory have been removed from the SBRP data bases.

Contract Solicitations

The analytical methods and associated QA/QC protocols that were used in the SBRP survey were selected so that the data could be compared with other similar data bases, e.g., the DDRP Northeastern survey data bases. On-site system audits and thorough evaluations of analytical data ensured that the procedures were followed correctly, as certain differences in methodology and reporting units occurred among the three contract solicitations. The distribution of batches among the laboratories, by solicitation, is outlined in Table 2-1.

Table 2-1. Distribution of Batches by Contract Solicitation/Laboratory

Solicitation/ Laboratory	Batch numbers
S1 / L3[a]	20602, 20608, 20609, 20610, 20611, 20612, 20613
S2 / L1	20701, 20702, 20703, 20707, 20708, 20710
S2 / L2	20614, 20704, 20705, 20706, 20709, 20711, 20712
S3[b] / L1	29603, 29605, 29606
S3[b] / L2	29601, 29604, 29607

[a] Laboratory 3 reanalyzed the cations under Solicitation 3.

[b] Reanalysis solicitation for batches retrieved from disqualified analytical laboratory.

Prior to beginning routine sample analysis, the original contract solicitation containing the analytical methodology was employed in the analysis of audit sample data from three referee laboratories. This solicitation was modified to specify the handling of organic samples, clarify the data reporting format, and lower some of the contract-required detection limits (CRDLs), as presented in Table 2-2. This became the basis for Solicitation 1, which required the laboratories to report both raw and blank-corrected data. When the CRDLs were lowered, all samples that were previously analyzed under a higher CRDL were reanalyzed at the lower CRDL.

About half of the SBRP soil samples were analyzed under the requirements of Solicitation 1. The principal changes in specifications for Solicitation 2 were the additional lowering of CRDLs for the cation analyses and the omission of a dilution step for SO4_PO4. Reanalysis of the affected parameters was performed on all previous samples at EPA expense.

Solicitation 3 was initiated to allow two of the other laboratories to provide analysis on the samples that were retrieved from the disqualified laboratory. Certain samples among those retrieved underwent additional processing at EMSL-LV in order to prepare them for analysis, as presented in Table 2-3. This processing consisted of rehomogenization and relabelling of the affected samples.

Table 2-3. Soil Samples which Underwent Secondary Processing Following Retrieval from the Disqualified Analytical Laboratory

Batch	Sample Numbers
29601	All except 22, 27, 29, 33, 34, 35
29603	All except 4, 11, 13, 21, 25, 30, 34, 35, 37, 39
29604	All except 2, 3, 5, 6, 9, 11, 15, 17, 18, 20, 24, 27, 29, 32, 34, 38
29605	All except 3, 30, 34, 36
29606	All except 2, 7, 12, 14, 15, 19, 22, 25, 27, 30, 37, 38
29607	All except 1, 2, 3, 4, 6, 8, 11, 12, 15, 16, 18, 19, 21, 22, 24, 28

The only substantive differences between batches analyzed under the different solicitations are with the CRDLs. Sample reanalyses have corrected all data affected by methods changes which occurred as the survey progressed. An international interlaboratory study is underway using DDRP audit samples to compare analytical data from the SBRP samples to data from other methods currently in use at soil characterization laboratories throughout the United States and Canada. Results of the study will be summarized in a forthcoming report (Palmer et al., in preparation).

Table 2-2. Contract-Required Detection Limits by Contract Solicitation

Parameter	Solicitation 1	Solicitation 2	Solicitation 3
CA_CL, CA_OAC, CA_CL2[a]	0.20 mg/L	0.05 mg/L	0.05 mg/L
MG_CL, MG_OAC, MG_CL2	0.20 mg/L	0.05 mg/L	0.05 mg/L
K_CL, K_OAC, K_CL2	0.20 mg/L	0.05 mg/L	0.05 mg/L
NA_CL, NA_OAC, NA_CL2	0.20 mg/L	0.05 mg/L	0.05 mg/L
CEC_OAC, CEC_CL	0.01 meq/L	0.01 meq/L	0.01 meq/L[b]
AC_KCL	0.25 meq/L	0.25 meq/L	0.25 meq
AC_BACL	0.40 meq/L	0.40 meq/L	0.40 meq
AL_KCL	0.50 mg/L	0.10 mg/L	0.10 mg/L
FE_CL2, AL_CL2	0.50 mg/L	0.05 mg/L	0.05 mg/L
FE_PYP, FE_AO, FE_CD	0.50 mg/L	0.50 mg/L	0.50 mg/L
AL_PYP, AL_AO, AL_CD	0.50 mg/L	0.50 mg/L	0.50 mg/L
SO4_H2O, SO4_PO4, SO4_0-32	0.10 mg SO_4/L	0.10 mg S/L	0.10 mg S/L
C_TOT, N_TOT	0.005 wt %	0.01 wt %	0.01 wt %
S_TOT	0.01 wt %	0.01 wt %	0.01 wt %

[a] CRDL for CA_CL2 reported as standard deviation of ten nonconsecutive blanks.
[b] meq for distillation/titration analysis and meq/L for flow injection analysis.

Analytical Laboratory Operations

Data Reporting

All samples received at the analytical laboratory were checked in by a receiving clerk who: (1) recorded on the shipping form the date the samples were received, (2) checked the samples and sample labels to identify discrepancies on the shipping form, and (3) sent copies of the shipping form to the EPA Sample Management Office in Washington, D.C., and to QA staff at EMSL-LV. If there were any discrepancies or problems, such as sample leakage or insufficient sample volume, the QA manager was notified immediately for instructions. The samples were refrigerated at 4°C as soon as possible and were kept under refrigeration when not in use. After all analyses were completed and the results were checked, the samples were placed in long-term cold storage at 4°C in the event that reanalysis was requested.

Analytical data were reported according to the protocols specified in the DDRP Analytical Methods Manual (Cappo et al., 1987). After each sample was completely analyzed, the results were summarized on summary data forms. Where appropriate, the data were annotated with the data qualifiers, or flags, listed in Appendix A. The laboratory managers signed each completed form to indicate that the data had been reviewed and that the samples were analyzed exactly as described in the SOW. Each manager was responsible for documenting any deviations from the SOW. An index of the data forms used by the analytical laboratories is provided in Appendix B.

Copies of the raw data were submitted upon request of the QA manager when potential discrepancies were found. Otherwise, all original raw data were retained at the analytical laboratories. The raw data include data system printouts, chromatograms, notebooks, individual data sheets, and QC charts.

System Audits

Each analytical laboratory underwent a minimum of two system audits, i.e., on-site evaluations. The first audit was performed after the laboratory had successfully analyzed the set of pre-award PE samples or, occasionally, during the PE sample analyses. The QA manager or authorized representative evaluated each of the laboratory functions that were pertinent to the analyses; a questionnaire was used to assist in this evaluation (see example in Bartz et al., 1987). The auditor summarized all observations in an audit report and brought any discrepancies to the attention of the laboratory manager.

The second on-site audit was conducted after sample analyses had begun. The evaluation questionnaire was completed with an emphasis on all changes occurring since the first audit. Data from the audit sample pairs and QC samples received to date were reviewed. An audit report was written for this and any subsequent on-site evaluations.

Daily communication was maintained between the QA staff and the laboratories during the periods when samples were being analyzed. The objectives of daily communication were to assure that each laboratory was satisfying the QC requirements and to obtain a preliminary evaluation of data quality and laboratory performance. This enabled the QA auditors to become familiar with analytical difficulties and with preliminary data, hence, verification of the data was underway prior to receipt of the data package by QA staff.

General Laboratory Protocols

General laboratory QC protocols included the use of suitable laboratory facilities, appropriate instrumentation with documented performance characteristics, reagents and labware of sufficient quality for the specific purpose, and adequately trained personnel. Documentation of the standard operating procedures of the laboratory, a list of in-house samples, and up-to-date QC charts were required. The laboratories were not required to use specific makes or models of instruments, although recommendations were given.

The analytical instruments for all of the methods required some form of calibration. For most methods, a series of standards was analyzed and a calibration curve was derived. The range of analyte concentrations in the calibration standards was required to bracket the expected analyte concentrations in the

routine samples without exceeding the linear dynamic range of the instrument. This range was determined by a least squares regression analysis (Steel and Torrie, 1960), with a correlation requirement for concentration versus instrument response of 0.99 or greater.

Quality Assurance and Quality Control Samples

The QA samples were used for independently assessing data quality and for monitoring the internal QC procedures. QA samples differ from QC samples in that they are submitted as blind samples to the laboratories, i.e., their identity in the batch and their composition are unknown to the analyst. QA data assessment is undertaken in statistical terms and is accomplished by the inclusion of replicate (usually duplicate) samples with the routine samples for analysis.

The QC samples were used to reduce random errors and systematic errors, or to maintain these errors within specified tolerable limits. These samples are created and used by the laboratories to evaluate the calibration and standardization of instruments and to identify problems such as contamination or analytical interference.

Description of Quality Assurance Samples

Three types of QA samples were used in the SBRP survey: (1) field duplicates, (2) preparation duplicates, and (3) natural audit samples. The number and percentage of QA and routine samples used in data quality assessment was as follows:

- Total QA and routine = 984 samples
- QA field duplicates = 106 samples (11 percent of total)
- QA preparation duplicates = 26 samples (2.5 percent of total)
- QA natural audits = 104 samples (10.5 percent of total)
- Routine = 748 samples (76 percent of total): 704 mineral samples (94 percent of routine) and 44 organic samples (6 percent of routine)

Field Duplicate Samples --

Each sampling crew was required to randomly sample one horizon in duplicate per day, collecting alternate portions of soil for each sample (Bartz et al., 1987). One sample was considered to be the routine sample and the other was designated the field duplicate. Since more than one pedon could be sampled on an average day, not all pedons were sampled for a duplicate. A pedon is a three-dimensional body of soil having a lateral area large enough (1 to 10 square meters) to permit the study of soil horizons. After processing, the field duplicates were placed randomly with their associated pedon samples in batches of approximately 42 soil samples each.

Certain QA data sets utilized only the 106 field duplicates, while other analyses used the 106 field duplicate pairs, i.e., the field duplicates in conjunction with their associated routine samples. The distribution of field duplicate pairs among the preparation laboratories and the analytical laboratories is shown in Table 2-4.

Preparation Duplicate Samples --

Each preparation laboratory selected one routine soil sample per batch and subsampled a duplicate sample with a Jones-type, 3/8-inch riffle splitter. Each preparation duplicate was placed randomly within its associated batch. Certain QA data sets utilized only the 26 preparation duplicates, while other analyses used the 26 preparation duplicate pairs, i.e., the preparation duplicates in conjunction with their associated routine samples. The distribution of preparation duplicate pairs among the analytical laboratories is shown in Table 2-5.

Natural Audit Samples --

Bulk soil samples representing five typical soil horizons of the eastern United States were collected in large storage drums and used as natural audit sample material. The soil horizons represented by these samples were Oa, A, Bs, Bw, and C horizons. Subsamples from each of these bulk samples were prepared by EMSL-LV staff and were forwarded to the preparation laboratories. The

Table 2-4. Distribution of Field Duplicate Sample Pairs Among the Sampling Crews, Preparation Laboratories, and Analytical Laboratories

	Analytical laboratory					
	1		2		3	
	Preparation laboratory					
Sampling crew	Clemson	UTenn	Clemson	UTenn	UTenn	Crew totals
GA01	10	0	5	0	0	15
GA02	0	0	4	2	16	22
NC01	5	0	4	2	0	11
NC02	2	0	0	0	0	2
NC03	4	5	2	4	3	18
NC04	0	0	9	0	0	9
TN01	0	9	0	0	3	12
TN02	0	0	0	7	5	12
VA01	0	0	0	0	5	5
Sampling(total)						106
Preparation	21	14	24	15	32	
Analytical		35		39	32	

Table 2-5. Distribution of Preparation Duplicate Sample Pairs Among the Preparation Laboratories and Analytical Laboratories

Analytical laboratory	Preparation laboratory		Total
	Clemson	UTenn	
1	6	3	9
2	6	4	10
3	0	7	7
Total	12	14	26

samples were randomly placed into batches at a rate of two pairs per batch without further handling or processing by laboratory personnel. One of the two pairs in each batch was always A horizon audit material. The distribution of the natural audit pairs among the analytical laboratories is presented in Table 2-6.

Table 2-6. Distribution of the Natural Audit Sample Pairs Among the Analytical Laboratories

	Audit horizon					
Laboratory	Oa	A	Bs	Bw	C	Total
1	0	9	5	3	1	18
2	0	10	6	2	2	20
3	2	7	0	2	3	14
Total	2	26	11	7	6	52

Since the same audit material (assumed to be homogeneous) was utilized throughout the survey, data from the audit samples were used to evaluate within-batch precision and analytical differences among laboratories. These data were also used for independent QA comparisons to data from the analytical duplicate QC samples. Additional checks on precision were made using the field duplicates and preparation duplicates.

Sample Flow

The routine and field duplicate samples were collected by the nine sampling crews and were delivered to the two preparation laboratories. The laboratories processed and prepared the samples and subsampled the preparation duplicates. Batches of soil samples were assembled, each containing field duplicates (two to six per batch, depending on the number of pedons represented in the batch), one preparation duplicate, and two pairs of audit samples, with the balance of the batch being composed of routine samples from the pedons. The batches were distributed among the contracted analytical laboratories for analysis.

Description of Quality Control Samples

Seven types of QC samples were used in the SBRP survey: calibration blanks, reagent blanks, QC check samples (QCCS), detection limit QC check samples (DL-QCCS), matrix

spikes, analytical duplicates, and ion chromatography (IC) standards, as described below. Control limits were established for measurements of each of the QC samples. The results from each laboratory were examined with reference to these established limits.

One calibration blank per batch was analyzed immediately following the initial instrument calibration in order to detect instrumental drift or to test for evidence of sample contamination. The calibration blank was defined as a "0" concentration standard and contained only the matrix of the calibration standards.

For methods that required sample preparation, e.g., soil extractions, a reagent blank was included in each batch of samples. The reagent blank, sometimes referred to as a process blank, was composed of all the reagents used and in the same quantities used in preparing a soil sample for analysis. This blank underwent the same digestion and extraction procedures as a routine sample and was used to identify contamination of the reagents. If the observed concentration of the calibration blank or the reagent blank was greater than the CRDL, the instrument was rezeroed, the calibration was checked, and the source of contamination or error was investigated and eliminated. A blank exceeding the CRDL for more than 25 percent of the samples in a batch was cause for reanalysis of the affected parameter.

A QCCS containing the analyte of interest at a concentration in the mid-calibration range was analyzed immediately following the standardization of an instrument, after the routine analysis of groups of ten samples, and after the last sample in each batch. The QCCS was prepared from a source which was independent of the calibration standards and was used to determine the accuracy and consistency of instrument calibration. The control limit for the QCCS was 10 percent of the theoretical value (5 percent for sulfate parameters and 1 percent for particle size parameters). When an unacceptable QCCS value was obtained, the instrument was recalibrated and all samples analyzed beyond the last acceptable QCCS were reanalyzed. The QCCS samples were plotted on the QC chart and the 95- and 99-percent confidence intervals were calculated. The 99-percent confidence interval, i.e., the control limit, was required to be within the maximum control limit specified by the QA staff.

The DL-QCCS contained the analyte of interest at a concentration two to three times above the CRDL (Cappo et al., 1987). The purposes of this sample were to eliminate the necessity of formally determining the detection limit on a daily basis and to determine accuracy at the lower end of the linear dynamic range of measurement. The measured value of the DL-QCCS was required to be within 20 percent of the theoretical concentration. If the difference was greater than this limit, the source of error was identified and corrected, and acceptable results were obtained before initiating routine sample analysis. The CRDL often was far below the concentration of the lowest-level analyte, hence, discriminating the DL-QCCS from background or instrument noise was difficult.

One matrix spike, i.e., a known quantity of analyte added to a sample aliquot, was examined in each batch to determine the sample matrix effect on the analytical laboratory measurements for most of the parameters. The spike concentration was approximately equal to the endogenous level or ten times the detection limit, whichever was larger, of the analyte being measured. The volume or weight of the added spike was required to be negligible for the purposes of calculation. Analytes that were extracted prior to analysis were spiked after extraction. If there was insufficient sample volume to spike all of the aliquots from one sample, the matrix spike analysis was performed on a per-aliquot basis.

If the spike recovery was not within 15 percent of the initial spike volume or weight, two additional samples were spiked with each of the analytes in question. The two additional samples were then analyzed and their respective recoveries were calculated. If the spike recovery in one or both of the samples was not within 15 percent, the entire batch of samples was reanalyzed for each of the parameters in question. The samples were diluted or the spike level was adjusted if the concentration of the matrix spike was not within the linear dynamic range for the analytical method.

One soil sample per batch was subsampled and analyzed in duplicate by the analytical laboratories. This QC sample, the analytical duplicate, was used in estimating the within-batch precision for each analytical laboratory and for identifying significant instrumental drift. The percent relative standard deviation (RSD) of each analytical duplicate pair, i.e., the duplicate and its companion routine sample, was calculated by dividing the standard deviation of the pair by the mean of the pair and multiplying this value by 100. If the RSD and the mean concentration of an analytical duplicate pair were greater than 10 percent and ten times the CRDL, respectively, then an explanation for the discrepancy was sought and another duplicate sample was analyzed. Routine sample analyses were stopped until instrumental control was restored, unless permission to proceed was obtained from the QA manager.

An IC resolution test was performed once per analytical run by analyzing a standard that contained approximately equal concentrations (1 mg/L) of sulfate and nitrate ions. If the resolution did not exceed 60 percent, the column was replaced and the resolution test was repeated.

Data Verification

Overview of Data Bases

The field sampling data and the analytical data were entered into the SBRP data base using a compiled *dBase III* entry system at Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee. These data also were sent to the QA staff at EMSL-LV for concurrent data verification. All data were double-entered into data sets and were visually checked, thereby allowing errors in transcription to be identified and removed. The data bases progressed through three stages: (1) raw data base, (2) verified data base, and (3) validated data base.

The raw data base contains the data that were entered directly from the field data forms and analytical data packages through double entry by ORNL and EMSL- LV. The two entries were compared and discrepancies were corrected so that the data sets were identical. One version was discarded and the other was frozen to become the official raw data base. A magnetic tape of this data base was sent to the National Computer Center (NCC) in Research Triangle Park, North Carolina, where the data tape was uploaded and made accessible to the QA staff.

Verification of the raw data base was accomplished by a systematic evaluation of completeness, precision, consistency, and coding accuracy. Discrepancies were flagged unless they could be corrected. After verification was completed, the data base was frozen and became the verified data base. A magnetic tape was generated and was sent to ORNL.

The verified data base underwent additional evaluation through a process called validation. The validation procedures included specific assessment of outlying data points for inclusion or omission in data sets based on assigned levels of confidence. These data warrant special attention or caution by the data user during analysis of the survey results. After the data were evaluated and the suspect values were confirmed or flagged, the data were frozen as the validated data base (Turner et al., in preparation).

Verification of Field Data

After locating specified sampling sites in designated watersheds, the sampling crews excavated, characterized, and sampled soil pedons representing the desired sampling classes (Coffey et al., 1987). The pedons were characterized and sampled by United States Department of Agriculture (USDA) Soil Conservation Service (SCS) soil scientists who consistently utilized SCS computer-coded field data forms (SCS-SOI-232 forms) to record the soil descriptions. Use of the field data forms allowed sampling crews to gather comparable data for each pedon.

The completed field data forms were sent to ORNL where the data were entered into two data base subsets. The 232BA subset includes data from the first page of the field data form concerning general site and pedon characteristics. The 232HO subset includes data from the second, third, and fourth pages of the field data form concerning specific characteristics of the individual soil horizons. As ORNL staff were double-entering

the raw data, QA staff at EMSL-LV were reviewing the data for outliers. An anticipated computer verification system to check field parameters for coding accuracy and completeness was not available, therefore, the data were reviewed manually.

Raw data were evaluated for each sampling crew. Outliers were identified and placed on discrepancy forms (see Appendix B) which were sent to the appropriate SCS state office for confirmation. The individual sampling crews reviewed these forms and entered either the corrected values or a notation indicating that the requested information could not be discerned. In either case, all outliers identified on the form were addressed and initialed by the reviewer. The discrepancy forms were returned to the QA staff, who edited the corrections on the original field data forms. In all, three separate sets of discrepancy forms were sent to the sampling crews during data verification. Because of the hand-checking procedure, various outliers were overlooked during the initial review but surfaced during the second or third reviews.

When the raw data base became accessible to QA personnel, a set of procedures for entering and editing the data base was employed. Editing was accomplished on a working copy of the official raw data base supplied by ORNL. All changes were made on this data base through a special editing program, thereby protecting the official raw data base. A subset of the raw data base was keyed into this analysis program. The subset, sorted by state, was moved into a temporary working file and underwent manual editing. After completion of editing, the manual system was exited and a transaction file of both edited and original data was created automatically. At the end of each editing session, the transaction file was printed and reviewed.

After the edits were verified, the local master data base was updated with the edited information in the transaction file. This information also entered a history file, which recorded all transactions made on the local master data base. The verified master data base was completed in October 1987.

ORNL personnel ran a thorough check by comparing data on the tape with the original field data forms. Occasional entry and editing errors were discovered and, after correction, a second tape was generated. It was decided that QA staff would make no further edits on the official verified data base. Subsequent discoveries of outliers were jointly discussed and documented by EMSL-LV and ORNL. Further changes in the data base were made only upon written confirmation by the QA staff.

Additional tests were performed on the verified data which generated a small set of outliers. Discrepancy forms identifying these outliers were sent to the appropriate SCS state and field offices for confirmation. A new list of edits was compiled by QA staff and was sent to ORNL for entry. EMSL-LV also entered the edits into a working file that was maintained by the QA staff. Comparisons of the ORNL and EMSL- LV files were made to evaluate completeness and consistency of the edits.

Verification of Analytical Data

Analytical data reported on 100-level data reporting forms and 200-level blank-corrected data forms were entered into a data set by ORNL. A magnetic tape of the data was added to the catalogue file at NCC, where it was loaded for remote access by the QA staff. Exceptions programs, used to highlight discrepancies in the data sets, were applied in the data quality assessment.

The steps identified below were established to identify and correct suspected data errors. Information obtained by this process was used to edit data on the magnetic tapes sent by ORNL. New data and flags were entered into the raw data set to correct or flag the original data.

Review of Data Packages --

When data packages were received from the analytical laboratories, the QA staff checked to ensure that the correct sequence and number of forms were submitted and that each form contained data for all samples in the batch. The laboratory manager's signature and the date of analysis was confirmed on each form. Each data package was then subjected to the following QA/QC checks:

- Audit data were evaluated with the data verification template (see Appendix B).
- The RSDs of all duplicate pairs were checked.
- Standard analyte relationships were evaluated.
- Blank concentrations were checked for compliance, i.e., less than the CRDL as outlined previously in Table 2-2.
- Instrument detection limits (IDLs) were checked for compliance, i.e., less than their corresponding CRDLs.
- Matrix spikes were checked for compliance in preparation, i.e., concentrations were ten times the CRDL or twice the endogenous level, whichever was greater; data were checked to ensure a spike recovery within 15 percent of the original spike concentration.
- QCCS data were checked for compliance, i.e., values within the calculated control limits.
- Reported and blank-corrected data were checked for proper calculations.

The QA staff compiled verification reports for each batch data package submitted. A response letter was sent to each laboratory after data package evaluation describing potential discrepancies within the reported data and occasionally suggesting where errors may have occurred, e.g., transposed numbers or erroneous dilution factors. Through use of the Form 500 (see Appendix B), the laboratories were required to respond promptly with confirmation or reanalysis of the parameters in question. Reanalysis was performed on whole batches of samples rather than individual samples.

Compliance for Quality Control Check Samples --

Analysis of QCCS was not required on the titrimetric analytical methods used to determine the CEC and exchangeable acidity parameters. For the other analyses, the QCCS sample concentrations were formulated to represent an approximate mid-range of the routine samples. The QCCS data were used to verify the analytical consistency of the laboratories.

The chemical characteristics and concentrations of the QCCS were known to the analytical laboratories, hence, it was expected that the observed values of the QCCS would be within 10 percent of their respective theoretical values. Due to the importance of the sulfate analysis to DDRP, the observed values were required to be within 5 percent of the theoretical values. The QCCS observations outside of these ranges are tabulated in Table B-1 of Appendix B. The application of a Type I error equation (Aronoff, 1984) generated a list of QCCS values whose compliance was estimated at the 0.05 significance level. The large number of QCCS samples outside of the range for the particle size classes suggests that the control limits were too tight for this parameter group. Other low concentration parameters were also susceptible to falling outside of the range.

Standard Relationships --

The audit pairs and the field, preparation, and analytical duplicates were used in the preliminary QA/QC assessment. The QA acceptance criteria, i.e., audit windows, initially were calculated for each of the parameters as the 95-percent confidence interval of audit sample data from the DDRP Northeastern Soil Survey. The audit sample windows were updated periodically on the basis of incoming data from the analytical laboratories. Audit pairs were first checked for their inclusion within the audit windows. Precision of each pair was estimated by calculating the percent RSD, with less than 10 percent being acceptable if the mean of the pair was greater than ten times the CRDL.

The natural audit pairs were also checked for consistency as set forth in the following standard analyte relationships:

- Particle Size Analysis: SAND + SILT + CLAY = 100

  The summation of total sand, silt, and clay fractions in mineral soil samples should equal 100 percent ±0.1 percent.

Also, samples labeled as organic soils are checked for having 12 percent or more organic carbon.

- Soil pH: PH_H2O > PH_002M > PH_01M

  Calcium ions in the calcium chloride extracts displace hydrogen ions by mass action on the exchange sites, thereby increasing the hydrogen concentration in the soil solution relative to that of the water extract. A higher concentration of calcium will more effectively displace hydrogen ions and will result in a lower pH.

- Cation Exchange Capacity (CEC): CEC_OAC > CEC_CL

  Ammonium in a buffered (pH 7.0) ammonium acetate solution displaces other cations from exchange sites. This method was used in conjunction with AC_BACL to establish a theoretical maximum for CEC in the soil. Ammonium in an unbuffered ammonium chloride solution provides a more accurate estimation of the actual CEC of the soil when included with AC_KCL. Generally, the CEC in ammonium chloride is less than the CEC in ammonium acetate (exceptions include soils with very low CEC or high pH).

- Exchangeable Acidity: AC_BACL > AC_KCL

  A buffered (pH 8.2) barium chloride triethanolamine solution was used to assess the total potential acidity. The unbuffered potassium chloride method estimates the actual exchangeable acidity in soils. Generally, the exchangeable acidity in potassium chloride is less than that in barium chloride triethanolamine (exceptions include some coarse-textured or low CEC soils).

- Extractable Sulfate: SO4_PO4 > SO4_H2O

  The phosphate anion, because of its size and chemical properties, readily exchanges with the sulfate anion. The phosphate extraction gives an indication of the total exchangeable sulfate in the soil. The water extraction measures only those sulfate ions that are easily displaced and is an accepted indicator of available sulfate in the soil. Generally, the sulfate concentration in the water extraction is less than in the phosphate extraction (exceptions include some soils with low sulfate adsorption or high organic matter).

- Sulfate Isotherms: SO4_0 < 2 < 4 < 8 < 16 < 32

  The isotherm relationship is a response to increased concentrations of sulfate and should advance in a linear fashion until the threshold of sulfate adsorption is reached.

Internal Consistency --

Most of the verification checks and evaluations of analytical laboratory measurements were performed on data from QA samples and from analytical QC samples. Although an assessment of data quality could be drawn from these samples, the QA staff decided that an additional evaluation was needed to identify specific errors in the data from the routine soil samples. The purpose of this evaluation was to identify values for each analytical parameter that were not consistent with the majority of values observed. These values were checked for errors in transcription, data entry, or editing. If no discrepancies were encountered, these data values were qualified, or "flagged", as routine data outliers with an "X" flag (see Appendix A). Time did not permit the QA staff to identify the cause of all outliers, nor was it feasible to confirm the accuracy of outliers with the laboratory personnel.

An internal consistency program created at ERL-C was used to identify the routine data outliers (D. L. Cassell, unpublished data). The first step was to correlate analytical data for each parameter with all other analytical parameters measured in the SBRP survey. The strongest correlations, based on the coefficient of determination (r^2), were investigated. When the r^2 value generated by the

correlation of one parameter with another was greater than about 0.80, the correlation having the highest r^2 was selected and the internal consistency computer program was applied to all of the routine data points. If r^2 for the highest correlations was less than 0.80, a separation of data between organic and mineral samples was used in order to ascertain whether or not the groupings had an effect on the correlation. The correlation was used if r^2 increased significantly after the organic and mineral samples were correlated separately.

In some cases, the values for one parameter did not correlate well with values for any other parameter. In these cases, a percentage of the highest and the lowest values for that parameter were checked for errors. Correlations were not performed on parameters within the same extract or from the same measurement, e.g., CA_OAC values were not correlated with MG_OAC values even though the resulting r^2 value had the highest value. The reason for this decision is that it was recognized that certain errors, e.g., incomplete extraction, would not be identified by performing correlations within the same extracting solution. Although correlations were performed for particle size parameters, the highest and lowest values in each particle size class were also checked.

The internal consistency program was designed using a weighted linear regression model (SAS, 1986) because the data exhibited heteroscedasticity, i.e., the variances were not the same for the entire population. The weighting factor (w) which was used in the regression was calculated as the reciprocal of the analyte concentration of the independent variable ($w = 1 / x$). The correlation was run by plotting values for one parameter on the X-axis and values for another parameter on the Y-axis. Outliers were defined as those points having a studentized residual (Belsley et al., 1980) of 3.0 or greater. The X- and Y-axes of each parameter then were reversed and the regression was repeated. The results from both regressions were combined in order to identify the outliers.

For each regression, the studentized residual was calculated by subtracting the regression estimate of the dependent variable from its corresponding observed value and dividing by the estimated standard error of the residual, as follows:

$$(y_i - \hat{y}_i) \div [S(i) \cdot (1 - h_i)^{1/2}]$$

where: y_i = ith value of the dependent variable

$\hat{y}_i$ = ith predicted value of the dependent variable by the regression equation

$S(i)$ = standard error estimated without the ith observation

h_i = ith leverage factor

The studentized residual is an appropriate robust technique used to investigate outlying data points. A possible limitation in the capability of the studentized residual to determine an outlier was that the outlier itself strongly influenced the regression estimates of the slope or intercept, thereby abnormally affecting the value of the residual. Another outlier measurement technique involved the use of a DFFITS statistic (Belsley et al., 1980), which was used to measure the change in the predicted value resulting from the exclusion of a specific observation in the regression analysis. The DFFITS statistic was used to examine the significance of large differences in residuals and was calculated as follows:

$$(\hat{y}_i - \hat{y}(i)) \div (S(i) \cdot h_{(i)}^{1/2})$$

where: $\hat{y}_i$ = ith predicted value with the current observation included

$\hat{y}(i)$ = ith predicted value with the current observation excluded

$S(i)$ = standard error estimated with the ith observation excluded

$h_{(i)}$ = leverage factor with the ith observation excluded

As in the studentized residual, division by the estimated error normalized the statistic to allow comparison among points of varying precision. As a result, controlling data points that might unduly affect the predicted value of the dependent variable tended to have a high

DFFITS value. The critical point which was used to define a high value, i.e., the critical DFFITS, and its corresponding outlier was calculated as follows:

$$2 \cdot [(m + 1) \div n]^{1/2}$$

where: m = number of independent variables

n = number of points or observations regressed

A data outlier was identified as any data point exceeding the critical values which had been defined for the studentized residual or DFFITS statistic. These points were temporarily removed from the set of observations being analyzed. Using the remaining data, a second regression was performed on the same parameters. Utilizing the regression equation, i.e., slope and intercept estimates, from the second regression performed and the mean and corrected sum of squares from data points defined as outliers in the first regression, a residual test was performed to examine and return data outliers to the set of "good" or viable data points. Any outliers that failed to pass this test were considered outliers and underwent additional internal consistency checks. Results were checked for accuracy in transcription against the values in the data package and, where necessary, corrections were made.

After edits were made in the data base, the internal consistency program was repeated and a second set of outliers was generated. Any new outliers which appeared in the second correlation were checked for accuracy. No errors in transcription were found in the second regression.

Table B-2 in Appendix B contains a list of correlations that were performed for each parameter, the parameter groups, and the r^2 values for the first and second correlations. Most of the correlations resulted in r^2 values greater than 0.80. When correlations were performed for the sulfate isotherms and for total sulfur/nitrogen, it was observed that a disproportionate percentage of the outliers were organic samples having high variability. Separating the organic horizons from the mineral horizons aided in identifying mineral soil outliers.

The following types of errors in the SBRP data base were identified by the internal consistency checks and were subsequently confirmed or corrected:

- Data entry errors: values from the analytical laboratory data packages that were entered incorrectly.
- Transcription errors: data that were transposed or transcribed at the analytical laboratories incorrectly, e.g., pH 5.34 instead of 3.54; most of these suspect values had been identified earlier and confirmation requests were sent to the laboratories, where the values were corrected, although the values had missed the editing loop.
- Batch errors: systematic or sporadic calculation errors that were discovered when most or all of the data in specific batches was outlying.
- Laboratory errors: systematic or sporadic calculation errors that were discovered when some or all of the data in batches from a specific laboratory was outlying.

Data Quality Objectives

To address the DDRP objectives, conclusions must be based on scientifically sound interpretations of the data base. To achieve this end, the EPA requires all monitoring and measurement programs to have established objectives for data quality based on the proposed end uses of the data (Blacker et al., 1986). Computer models are being used to predict results and hypotheses have been developed to test the models. The utility of the data, and thus the project itself, is defined by the ability to confirm, reject, or discriminate between hypotheses. The primary purpose of the QA program is to increase the likelihood that the resulting data base meets or exceeds specific DQOs. Through the proper development of DQOs, the quality of data can be quantified, thereby allowing the data user to differentiate hypotheses. In practice, DQOs are statements of the levels of uncertainty that a data user is willing to accept in the results derived from the data.

The DQOs for the SBRP survey were established for detectability, precision, representativeness, completeness, and comparability. Due to the naturally low analyte concentrations in the soils under investigation, contract-required detectability standards were established to further enhance interpretability of the data base. The DQOs for precision are quantitative criteria that were developed for specific components of the data collection activities and measurement system used in the survey. The DQOs for representativeness, completeness, and comparability were somewhat qualitative in nature and were assessed primarily by the research design and selection of methodologies. There were no DQOs established for accuracy, although an attempt has been made in this report to relate accuracy considerations to interlaboratory differences.

Detectability

An important factor to consider in the evaluation of data quality is the detection limit, which is the lowest concentration of an analyte that an analytical process can reliably detect. The primary consideration is whether or not a measured sample value can be considered significantly different than the measured value of a sample blank. The probability that an analytical signal is not simply a random fluctuation of the blank is dependent on how many standard deviations the analytical signal varies from the mean value of blank responses (Long and Winefordner, 1980). The specific application of detectability in the SBRP survey required the investigation of precision in low concentration samples.

A commonly recognized value for the detection limit is three times the standard deviation of the blank samples (American Chemical Society, 1983). A signal measured at this level or greater would have less than a 0.1 percent chance of being the result of a random fluctuation of the blank, assuming the blank samples have a normal distribution. In the absence of blank samples, low concentration replicate samples are often used to estimate the standard deviation expected of blank samples. Although liquid blank samples have been used extensively in the aquatics surveys of the National Surface Water Survey, to date it has been unknown how to develop a soil blank suitable for system-wide use in DDRP. With this in mind, the following three types of detection limits are described in this report.

(1) All analytical laboratories were required to satisfy the contract-required detection limit (CRDL) for specified parameters, as presented in Table 2-7. The CRDLs were established for instrument readings in the analytical phase only.

(2) A calculated instrument detection limit (IDL) was used to estimate the lowest concentration of an analyte that the analytical instruments used by the laboratories could reliably detect. Although IDLs were calculated from analytical blank samples and were reported by the laboratories, these values are not included in this report. Instead, an independent check of the IDLs was possible by examining the variability in the DL-QCCS samples and by assuming that the variability of this low level sample should have been about the same as that of the blank samples. The IDLs reflect variability in the analytical phase only.

(3) It is recognized that laboratory analysis is only one of many steps in the overall process of generating raw data for a soil sample collected from the field. If it were possible to route "soil blank" samples with zero concentration of analyte through the sampling crews and subsequently through all phases of the measurement system, a system detection limit (SDL) could be estimated. Overall variability in the blank sample would encompass variability in sampling, preparation, extraction, and analysis, and would include sample contamination at any of these steps. Calculation of a SDL from such a sample would allow a data user to identify when any given soil sample had a measured concentration that could be considered as statistically different from that of a reagent blank or calibration blank. For this report, reasonable substitutes for blank samples are the field duplicates which are routed through the major components of the measurement system and exhibit many of the features that would be expected in soil blanks. By selecting the field duplicates of least concentration, e.g., the lowest 10 percent of the duplicates, the resulting variability would be expected to parallel that of system-wide blanks.

Table 2-7. Data Quality Objectives for Detectability and Analytical Within-Batch Precision

Parameter	Reporting units	CRDL[a]		Precision[b]		
		units	mg/L	lower (SD)	upper (RSD)	knot
MOIST	wt %	---	---	---	---	---
SP_SUR	m^2/g	---	---	---	---	---
SAND[c]	wt %	---	---	1.0	---	---
SILT[c]	"	---	---	1.0	---	---
CLAY	"	---	---	1.0	---	---
PH_H2O	pH units	---	---	0.15	---	---
PH_002M	"	---	---	0.15	---	---
PH_01M	"	---	---	0.15	---	---
CA_CL	meq/100g	0.003	0.05	0.03	15%	0.20
MG_CL	"	0.011	0.05	0.03	15%	0.20
K_CL	"	0.003	0.05	0.03	15%	0.20
NA_CL	"	0.006	0.05	0.03	15%	0.20
CA_OAC	meq/100g	0.006	0.05	0.03	15%	0.20
MG_OAC	"	0.011	0.05	0.03	15%	0.20
K_OAC	"	0.006	0.05	0.03	15%	0.20
NA_OAC	"	0.006	0.05	0.03	15%	0.20
CEC_CL	meq/100g	0.002	0.01[d,e]	0.25	10%	2.5
CEC_OAC	"	0.002	0.01[d,e]	0.25	10%	2.5
AC_KCL	"	0.11	0.40[e]	0.50	20%	2.5
AC_BACL	"	0.75	0.25[e]	0.50	20%	2.5
AL_KCL	"	0.80	0.10	0.50	20%	2.5
CA_CL2	meq/100g	---	---[f]	---	5%	---
MG_CL2	"	0.0007	0.05	---	10%	---
K_CL2	"	0.0002	0.05	---	10%	---
NA_CL2	"	0.0004	0.05	---	10%	---
FE_CL2	"	0.0005	0.05	---	10%	---
AL_CL2	"	0.0001	0.05	---	10%	---
FE_PYP	wt %	0.005	0.50	0.05	15%	0.33
AL_PYP	"	0.005	0.50	0.05	15%	0.33
FE_AO	"	0.005	0.50	0.05	15%	0.33
AL_AO	"	0.005	0.50	0.05	15%	0.33
FE_CD	"	0.002	0.50	0.05	15%	0.33
AL_CD	"	0.002	0.50	0.05	15%	0.33
SO4_H2O	mg S/kg	2.0	0.10	1.0	10%	10.0
SO4_PO4	"	2.0	0.10	1.0	10%	10.0
SO4_0-32	mg S/L	0.10	0.10	0.05	5%	1.0
C_TOT	wt %	0.01	0.010[g]	0.05	15%	0.33
N_TOT	"	0.01	0.010[g]	0.01	10%	0.10
S_TOT	"	0.01	0.010[g]	0.01	10%	0.10

[a] Contract-required detection limit in reporting units and parts per million, respectively.
[b] Precision objectives below and above the knot separating the lower tier (standard deviation in reporting units) and the upper tier (relative standard deviation in percent); the knot is in reporting units.
[c] DQOs were not established for size fractions of this parameter.
[d] Units are meq/L for this parameter for flow injection analysis.
[e] Units in meq for this parameter for titration.
[f] CRDL reported as standard deviation of ten nonconsecutive blanks.
[g] Units are weight percent (wt %) for this parameter.

The calculated IDL was estimated as three times the pooled standard deviation of a low level DL-QCCS. The SDL was estimated as three times the pooled standard deviations of the lowest ten percent of all field duplicate pairs. These limits, together with the CRDL and the converted IDL (the calculated IDL in comparable reporting units), are given in the results and discussion. The effect of adjusting CRDLs for certain parameters during the course of the survey is also examined.

An important factor to consider in the evaluation of detectability is the implication of the calculated detection limits for data quality of the routine data set. By estimating the percentage of data from the routine samples that were greater than the corresponding SDLs, specific parameters were identified that might not have been measured with sufficient precision to satisfy the requirements of data users. This was not necessarily the result of improper CRDLs, for it is evident that the instrumental error was the source of only a small portion of the variability in the low concentration field duplicates used to estimate the SDLs.

Precision

Development of the Precision Objectives --

The precision DQOs for the SBRP survey were established for analytical within-batch precision of most of the physical and chemical parameters listed in Table 1-1 of Section 1. There were no specific DQOs established for the sampling or preparation phases of the survey. The initial DQOs were based on the requirements of EPA data users, the selection of appropriate methods to obtain the required data, and the results of a pilot study. Modifications were implemented based on review comments from the users and cooperating scientists. In addition, the analytical results from specific methods, procedures, and instrumentation were useful in the adjustment of DQOs for future DDRP surveys.

The primary characteristics of the precision objectives were the development and implementation of a two-tiered system for characterizing the DQOs. Similar parameters were grouped together according to their type of reporting units. Intralaboratory within-batch precision goals were defined, based on a percent RSD for concentrations above a specific level, defined as the "knot", and an absolute standard deviation for concentrations below the knot (see Table 2-7). The upper tier concentration range above the knot defines the region of the data where the analytical results are relative and expressed as a percentage. The lower tier concentration range below the knot defines the region of the data where the analytical results are absolute and expressed as a standard deviation in reporting units. This system avoids setting restrictive precision requirements for low concentration samples which generally are more difficult to analyze with a high degree of precision. The knot was established by dividing the precision objective at the lower tier by the precision objective at the upper tier (see Figure 2-1).

Data from the homogenized natural audit samples were used to assess the DQOs for analytical within-batch precision because they had no sampling error and were assumed to have negligible preparation error. As such, the precision DQOs developed for the SBRP survey were not intended to serve as project level DQOs.

Estimation of the Data Collection Error --

For any large survey, the collection of data is a multi-phase process. In the DDRP, those phases are field sampling, sample preparation, and sample analysis. The QA samples were introduced at these different data collection phases so that analytical data from the samples could be used to control and assess the uncertainty for each phase. For example, data from field duplicates can be used to estimate the confounded error associated with field sampling, sample preparation, and sample analysis. Data from the preparation duplicate samples can be used to estimate the confounded error associated with subsampling in the preparation laboratory and analysis at the analytical laboratory. Data from the audit samples can be used to estimate the error of the sample analysis. The audit samples are assumed to have negligible preparation error for the purposes of the error estimates that are based on the following model:

$$y = \mu + \epsilon$$

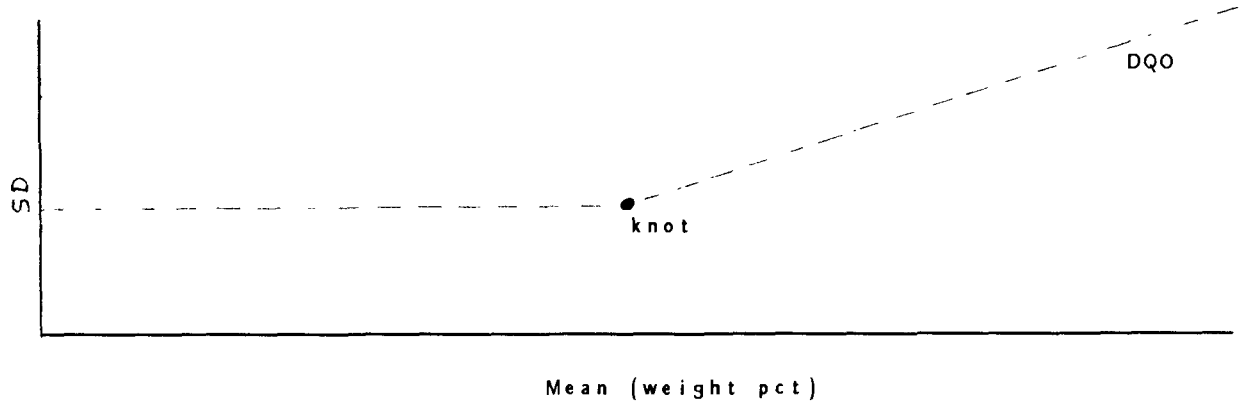


Figure 2-1. Example of a two-tiered precision objective.

where: y is an observed sample characteristic; μ is the true sample characteristic; and ϵ is the data collection error, which is assumed to be the sum of the errors generated by the three independent data collection phases.

Standard operating procedures, or protocols, were followed in each phase of the SBRP survey. Depending on its limitations or assumptions, each operating procedure induces a random error for each physical or chemical characteristic of a soil sample. The sum of the errors induced by each procedure can be defined as data collection error, which is treated as a random variable. It is necessary to characterize this variable in order to identify the effect of the error on the routine soil samples. This involves identifying the distributional form and estimating the moments.

The identification of the error distribution requires a large number of replicate measurements which, from a budgetary and logistical standpoint, imposes a serious limitation; however, a relatively small number of observations can be used to estimate the first two moments, i.e., the mean and variance, of the data collection error. The mean and variance are sufficient to measure the precision and accuracy of the routine data in an additive model, where the observed analyte concentration is assumed to be the sum of the true analyte concentration and the data collection error. For this report, the standard deviation in reporting units and the RSD, i.e., coefficient of variation, in percent are used to measure precision.

The within-batch precision component measures the reproducibility of audit sample data for a given set of soil samples analyzed for one analytical run by one laboratory. The between-batch precision component measures the reproducibility of audit sample data for different batches of soil samples analyzed on different days by different laboratories. It is expected that the within-batch variability is smaller than the between-batch variability.

Two pairs of natural audit samples were placed in each of the 26 batches for a total of 52 audit sample pairs for the SBRP survey. To assess the within- batch precision, the standard deviations for each of the pairs were pooled by averaging the variances and taking the square root to generate a within-batch standard deviation. A standard deviation was calculated for the pooled means of the audit pairs for between-batch precision.

It was found that the variance changes with analyte concentration, and it was not possible to identify a normal relationship between the soil analyte concentration and the error variance. However, the range of the soil analyte concentration was arbitrarily divided into intervals, i.e., windows, by grouping clusters of data in such a way that the error variance was relatively constant within each window. It was then possible to fit a step function across the windows to represent the error variance for the entire concentration range.

For each QA sample type, a step function was used to represent the appropriate standard deviation. Values for the fitted step

function were pooled and used as an estimate of the associated standard deviation, e.g., data from the preparation duplicates were used to estimate the standard deviation of the confounded preparation and analytical error. The standard deviations were pooled (s_p) by using the degrees of freedom (df) as a weighting device according to the formula:

$$s_p = [\Sigma_{\{i=1,k\}} (df_i \cdot s_i^2) \div \Sigma_{\{i=1,k\}} df]^{1/2}$$

where: s_i is the standard deviation for the ith window with corresponding degrees of freedom df_i. The RSD was used to assess data in the upper concentration ranges and was obtained by dividing the pooled standard deviation by the weighted mean.

It was also important to evaluate the effect of measurement precision on the routine sample data. Since the error standard deviation changes with analyte concentration, the expected standard deviation is estimated by considering its variability over the range of routine samples. In order to estimate this effect, the standard deviations for different windows are pooled with weighted proportions of routine samples, grouped by sampling class/horizon criteria, within the respective windows. This pooled value, delta (δ), is used as a measure of system-wide data uncertainty in the routine sample data due to data collection error. Delta is defined as:

$$\delta = \Sigma_{\{i=1,k\}} (P_i \cdot s_i) \div \Sigma_{\{i=1,k\}} P_i$$

where: P_i is the proportion of routine samples in the ith window, and s_i is the estimated standard deviation for the ith window. Occasionally, a lack of QA sample data within the concentration limits of a particular window made it impossible to calculate a standard deviation for that portion of the data set. In those cases, delta is the conditional measure of data uncertainty, the condition being dependent on the availability of QA data. Hence, certain windows are excluded from the calculation of delta.

An assumption is stated that the sampling class/horizon groups define homogeneous sets of soil samples, each having a specific variance. The 12 sampling classes and the 19 primary horizon types associated with these classes are known "effects" that define soil differences in the SBRP survey. By specifying these characteristics in the model, the variation due to these effects was removed. Table 2-8 presents the number and percentage of primary horizon types selected as a basis for the sampling class/horizon criteria used in grouping the 748 routine samples, and the number of sampling classes each horizon spans.

Table 2-8. Primary Horizon Types for Sampling Class/Horizon Groups

Horizon type	Routine samples number	Routine samples percent	Sampling classes represented
A	136	18.2	12
AB	25	3.3	9
AC	1	0.1	1
Ap	12	1.6	4
B	3	0.4	2
BA	21	2.8	8
BC	49	6.6	9
BE	2	0.3	2
Bg	3	0.4	1
Bs	1	0.1	1
Bt	112	15.0	9
Bw	201	26.9	11
Bx	2	0.3	1
C	111	14.8	11
Cg	6	0.8	2
Cr	10	1.3	4
E	10	1.3	6
Oa	2	0.3	2
Oe	41	5.5	9

Accuracy (Interlaboratory Differences)

Accuracy is the ability of a specific component of a measurement system to approximate a true value. The audit samples used in the SBRP survey were natural soil samples, hence, their true chemical composition and physical characteristics are unknown. Natural soil samples were used because a procedure for preparing synthetic samples has not been established. Therefore, accuracy of the analytical data cannot be determined because neither synthetic soil audit samples nor natural soil audit samples of known composition could be used as audit samples. An international interlaboratory comparison study, however, is currently being conducted to provide data on the chemical composition and physical characteristics of the natural audit samples (Palmer et al., in preparation). Data from the analyses of the audit samples by 22

external laboratories can possibly be considered to represent the known composition of these samples. These data will be compared to data in the verified data base to estimate interlaboratory bias. In the interim, data from the natural soil audit samples are used to establish interlaboratory differences for this report.

Absolute Differences --

The absolute difference (d_i) is defined as the variation between the mean of a repeated measurement for a given laboratory and the mean for the measurement among all laboratories, as follows:

$$d_i = | x_i - X |$$

where: d_i = absolute difference for the ith laboratory

x_i = mean for the ith laboratory

X = mean for all laboratories

Significant Differences Among Laboratories --

For each of the parameters, the analysis of variance (ANOVA) was used to determine the significant differences among the audit sample data reported by the analytical laboratories. An initial review of the data showed that the analytical variances across audit sample types were not identical. Because of this lack of homogeneity, a nested ANOVA model (Steel and Torrie, 1960) was used for each audit sample type to test the significance of laboratory differences by comparing laboratory means, based on a similar approach in Schmoyer et al. (1988). The model is as follows:

$$Y_{ijk} = \mu + L_i + \tau_{ij} + \epsilon_{ijk}$$

where: Y_{ijk} = the ith laboratory observation of the kth audit sample in the jth batch

μ = the expected value of the audit samples

L_i = the ith analytical laboratory effect

τ_{ij} = the jth batch effect within the ith laboratory

ϵ_{ijk} = the random error

Where laboratory differences were significant, a pair-wise comparison was performed on the laboratory means by using Scheffe's multiple comparisons test (Arnold, 1981). The results of this test were used to select values of high significance and to describe the ranking order in which the analytical laboratories can be arranged.

Pooled Data for Laboratories and Audit Sample Types --

Data pooled across audit samples to eliminate horizon effects were used to establish each laboratory's performance for individual parameters. This was accomplished by ranking the laboratories according to the magnitude of the difference from the grand mean (smallest to largest) after first comparing the difference to the overall laboratory mean. Three of the five audit sample types, the A, Bw, and C horizons, were analyzed by all three laboratories. Interlaboratory differences were determined, therefore, by pooling only the data for the A, Bw, and C audit samples for each laboratory, as follows:

$$\frac{\Sigma_{\{a = A,Bw,C\}} (d_{ia} \cdot n_{ia}) \div \Sigma_{\{a = A,Bw,C\}} n_{ia}}{\Sigma_{\{a = A,Bw,C\}} (X_a \cdot n_a) \div \Sigma_{\{a = A,Bw,C\}} n_a} \cdot 100$$

where: d_{ia} = absolute difference for the ith laboratory and the ath audit sample

n_{ia} = number of samples from the ith laboratory and the ath audit sample

X_a = mean for all laboratories for the ath audit sample

n_a = total number of samples for the ath audit sample

Pooling audit sample data to eliminate laboratory effects allowed an evaluation to be made of the mean laboratory difference for four of the five audit sample types (the Oa sample was analyzed by only one laboratory and was not used in this evaluation). If the

range of chemical and physical data of the audit samples is comparable to that of the routine samples in the survey, an evaluation can be made of the ability of the laboratories as a group to analyze certain soils using the specified analytical methods. For example, if the differences were very high for all laboratories for a parameter or group of parameters determined by a specific analytical method, the method itself could be in question concerning its selectivity of the parameter. The overall laboratory difference for each audit sample was determined as follows:

$$\frac{\Sigma (d_{ia} \cdot n_{ia}) \div \Sigma n_{ia}}{X_a} \cdot 100$$

where: d_{ia} = difference for the ith laboratory and the ath audit sample

n_{ia} = number of samples for the ith laboratory and the ath audit sample

X_a = mean for all laboratories for the ath audit sample

Representativeness

The evaluation of representativeness includes: (1) determining whether the routine samples collected were representative of the sampling class characteristics, (2) assessing the homogenization procedure by measuring the ability of each preparation laboratory to prepare representative subsamples from the bulk soil samples collected by the sampling crews, and (3) assessing the ability of the QA samples to adequately represent the range and frequency distribution of analyte concentrations in the routine samples. Data from the preparation duplicates were used in the second assessment, while the Kolmogorov-Smirnov two-sample test, i.e., the KS-statistic, was used to estimate the maximum distance between two data sets as a measure of resemblance between the sets (Conover, 1980).

Three data sets encompassing data for the routine samples (RS), the field duplicates (FD), and the preparation duplicates (PD) were used in the latter assessment. The FD and PD sets were tested independently against the RS set by using the p05, p50, and p95 percentiles to assess the range and frequency distribution within the data sets. The significant KS-statistics, i.e., significant at the 0.05 level, were defined according to the critical value (V_c) for each data set comparison. The critical value is based on a sample size n_1 and n_2 for the data sets being compared, where:

$$V_c = 1.36 \cdot [(n_1 + n_2) \div (n_1 \cdot n_2)]^{1/2}$$

This algorithm yielded critical values for the data set pairings, where V_c for FD_RS is 0.141 and V_c for PD_RS is 0.271. If the KS-statistic exceeded the critical value for a particular data set pairing, the QA data set was not representative of the distribution of routine samples.

Completeness

Soil sampling protocols in the SBRP survey specified the sampling of 100 percent of the designated pedons. The soil preparation protocols specified that each batch of samples sent to an analytical laboratory includes a preparation duplicate sample. The soil analysis protocols specified the complete analysis of all samples collected for 90 percent or more of the parameters. These three aspects of completeness were evaluated using the SBRP verified and validated data bases.

Comparability

Data comparability is ensured by the uniform use of documented procedures for soil collection, preparation, and analysis and by the use of equivalent units for reporting the data. The analytical methods and associated QA/QC protocols that were used in the SBRP survey were selected so that the data could be compared with other similar data bases, e.g., the DDRP Northeastern data base. On-site system audits and thorough evaluations of analytical data were employed to ensure that the procedures were being followed correctly.

The DDRP Analytical Methods Manual (Cappo et al., 1987) contains detailed descriptions of each of the analytical techniques, including examples of calculations and appropriate references. The internal QC procedures for each method are described in an introductory section and are summarized in tabular form. The QC protocols also are described within each of the analytical method descriptions. Data quality objectives, data

qualifiers, and decimal reporting requirements are listed in tabular form.

The DDRP Quality Assurance Plan (Bartz et al., 1987) was based on previously developed planning documents for the National Surface Water Survey. The QA Plan includes several introductory sections describing the project organization, sampling strategy, and field operations. The QA objectives and the sampling, preparation, and analytical QC procedures are described in detail and are also summarized in tabular form. Analytical methods are listed with the appropriate references. These methods generally are descriptive of the methods specified in the overall SOW as well as the subsequent EPA special analytical services solicitations.

Before it can be ascertained whether the field sampling or sample preparation activities are comparable between regions, the analytical laboratories must be shown to have provided comparable data. This assessment was made by examining data from the natural audit samples. If the analytical data are comparable across regions, the sample preparation can be compared using data from the preparation duplicates. If the preparation data are comparable across regions, then the field sampling can be compared using data from the field duplicates and from validation activities, e.g., aggregation. For this report, noncomparable field and laboratory methods used in the two surveys were documented and the QA duplicate samples inserted at certain steps during the surveys were used to assess comparability of the soil sampling, preparation, and analysis phases. Comparability of the data bases could not be evaluated because the statistical approach taken for the Northeastern survey data assessment was different from that of the SBRP survey.

Section 3

Results and Discussion

The results described in this section are based on the analysis of data values in the official SBRP verified data base. An assessment of completeness used some data from the official SBRP validated data base.

Detectability

Data relating to detection limits for contract requirements, instrument readings, and system-wide measurement in the SBRP survey are presented in Table 3-1.

The SDLs were always larger than the corresponding IDLs, which indicated the additional sources of variability in system-wide measurement. As anticipated from the experiences of previous surveys, variability in the selected low concentration field duplicates exceeded the variability in the selected DL-QCCS. Only seven parameters did not have over 85 percent of the data from their respective routine samples above the SDL. Only five of the 31 IDLs were higher than their corresponding CRDLs, and all were only slightly higher except for CA_CL2.

Reduction of the CRDL for the exchangeable base cations, from 0.20 to 0.05 mg/L, had little effect on reducing the IDLs. The IDLs were less than the corresponding CRDLs for all cations at the 0.20 mg/L limit, although the IDLs exceeded the CRDLs at the 0.05 mg/L limit for CA_CL. The SDL was high in relation to the routine samples only for NA_CL and CA_OAC.

The IDLs for the CEC and exchangeable acidity parameters were calculated by averaging the IDLs reported by the laboratories because the DL-QCCS data for these parameters were incomplete. The IDLs were slightly higher than the CRDLs for CEC. The reduction of the CRDL for AL_KCL, from 0.50 to 0.10 mg/L, reduced the IDL only slightly. Of this group, the SDL was high in relation to the routine samples only for AL_KCL.

Reduction of the CRDL for the extractable base cations, from 0.20 to 0.05 mg/L, had little effect on reducing the IDLs. Reduction of the CRDL for FE_CL2 and AL_CL2 from 0.50 to 0.05 resulted in a two-fold drop in the IDLs. The IDLs were less than the corresponding CRDLs for all cations at the 0.20 mg/L limit, although the IDLs exceeded the CRDLs at the 0.05 mg/L limit for CA_CL2, NA_CL2 and AL_CL2. The SDL was high in relation to the routine samples only for FE_CL2 and AL_CL2, both of which had very low analyte concentrations.

The IDLs were lower than the CRDL for each of the extractable iron and aluminum parameters. The SDLs were higher than the IDLs by an order of magnitude or more, but were not high in relation to the routine samples.

The IDLs were lower than the CRDLs for the extractable sulfate and sulfate isotherm parameters. The IDLs were converted from a solution concentration to a soil concentration that enabled comparisons to be made with the SDLs. The SDLs for extractable sulfate were three to six times higher than the IDLs, but were not high in relation to the routine samples.

The increase in the CRDL for total carbon and nitrogen, from 0.005 to 0.010 weight percent, resulted in a marked reduction in the IDL for C_TOT but not for N_TOT. The IDL was lower than the CRDL for S_TOT. The SDLs were high in relation to the routine samples for N_TOT and S_TOT.

Table 3-1. Detection Limits for the Contract Requirements, Instrument Readings, and System-wide Measurement

Parameter	CRDL[a]	Calc IDL[b]	Conv IDL[c]	SDL and	%RS>SDL[d]
CA_CL	0.05 mg/L	0.0524 mg/L	0.0068 meq/100g	0.0311 meq/100g	89.8
MG_CL	0.05 mg/L	0.0369 mg/L	0.0079 meq/100g	0.0328 meq/100g	92.4
K_CL	0.05 mg/L	0.0364 mg/L	0.0024 meq/100g	0.0423 meq/100g	90.0
NA_CL	0.05 mg/L	0.0415 mg/L	0.0046 meq/100g	0.0195 meq/100g	69.1
CA_OAC	0.05 mg/L	0.0314 mg/L	0.0041 meq/100g	0.0725 meq/100g	77.5
MG_OAC	0.05 mg/L	0.0121 mg/L	0.0026 meq/100g	0.0220 meq/100g	96.1
K_OAC	0.05 mg/L	0.0330 mg/L	0.0022 meq/100g	0.0363 meq/100g	92.2
NA_OAC	0.05 mg/L	0.0448 mg/L	0.0051 meq/100g	0.0098 meq/100g	92.0
CEC_CL	0.01 meq/L	0.0153 meq/L[e]	0.0306 meq/100g	1.0724 meq/100g	99.9
CEC_OAC	0.01 meq/L	0.0155 meq/L[e]	0.0311 meq/100g	0.5809 meq/100g	100
AC_KCL	0.25 meq/L	0.0060 meq/L[e]	0.0188 meq/100g	0.3870 meq/100g	92.1
AC_BACL	0.40 meq/L	0.1840 meq/L[e]	0.3681 meq/100g	3.7750 meq/100g	89.8
AL_KCL	0.10 mg/L	0.0840 mg/L	0.0186 meq/100g	0.4780 meq/100g	83.1
CA_CL2	— mg/L[f]	0.6071 mg/L	0.0160 meq/100g	0.0565 meq/100g	99.6
MG_CL2	0.05 mg/L	0.0187 mg/L	0.0003 meq/100g	0.0041 meq/100g	99.7
K_CL2	0.05 mg/L	0.0335 mg/L	0.0002 meq/100g	0.0020 meq/100g	99.6
NA_CL2	0.05 mg/L	0.0560 mg/L	0.0005 meq/100g	0.0031 meq/100g	98.9
FE_CL2	0.05 mg/L	0.0402 mg/L	0.0004 meq/100g	0.0021 meq/100g	12.7
AL_CL2	0.05 mg/L	0.0616 mg/L	0.0014 meq/100g	0.0071 meq/100g	51.3
FE_PYP	0.50 mg/L	0.1434 mg/L	0.0015 wt %	0.0273 wt %	93.8
AL_PYP	0.50 mg/L	0.2278 mg/L	0.0023 wt %	0.0220 wt %	99.5
FE_AO	0.50 mg/L	0.1941 mg/L	0.0019 wt %	0.0509 wt %	93.7
AL_AO	0.50 mg/L	0.2282 mg/L	0.0023 wt %	0.0547 wt %	96.3
FE_CD	0.50 mg/L	0.1340 mg/L	0.0004 wt %	0.1449 wt %	98.5
AL_CD	0.50 mg/L	0.1998 mg/L	0.0006 wt %	0.0426 wt %	99.3
SO4_H2O	0.10 mgS/L	0.0141 mgS/L	0.2828 mgS/kg	1.7394 mgS/kg	92.0
SO4_PO4	0.10 mgS/L	0.0367 mgS/L	0.9186 mgS/kg	3.2539 mgS/kg	99.7
SO4_0	0.10 mgS/L	0.0494 mgS/L	—	0.0759 mgS/L	91.4
C_TOT	0.010 wt %	0.0105 wt %	—	0.0821 wt %	96.7
N_TOT	0.010 wt %	0.0114 wt %	—	0.0247 wt %	71.2
S_TOT	0.010 wt %	0.0026 wt %	—	0.0178 wt %	44.6

[a] Contract-required detection limit.
[b] Calculated instrument detection limit, estimated as three times the pooled standard deviation of a low level DL-QCCS.
[c] Converted instrument detection limit, based on the specified reporting units.
[d] System detection limit, estimated as three times the pooled standard deviations of the lowest 10 percent of field duplicates, independent of the CRDL; Percent of routine samples exceeding the system detection limit.
[e] Estimated by averaging laboratory-reported IDLs for incomplete DL-QCCS data.
[f] CRDL reported as standard deviation of ten nonconsecutive blanks.
NOTE: Detection limits not applicable for the physical parameters, soil pH, and the remainder of the sulfate isotherm parameters.

Precision

The following sets of tables, figures, and text are designed to satisfy the requirements of the SBRP data users for summary precision estimates of the routine and QA sample data. The assessment of precision relates directly to the achievement of intralaboratory within-batch DQOs established in the DDRP QA Plan (Bartz et al., 1987). In most cases, the DQOs have knot values which represent the separation point for the data uncertainty expressed as a standard deviation for low concentrations and as a relative standard deviation in percent for higher concentrations.

The precision data are presented in sequential order of the parameters listed in Table 1-1 of Section 1 of this report. For each of the nine parameter groups, a table of statistics presents the QA and routine sample

data below and above the knot. These tables show the relationship of the QA data to the DQOs.

Two figures are presented for each parameter within each parameter group. The first figure is a plot of the mean and standard deviation of data from each of the five audit samples and their relationship to the DQO for each parameter. The second figure is a plot of the mean and standard deviation of data from the routine samples, grouped by sampling class/horizon criteria. The variability seen in the sampling class/horizon data is principally the result of spatial heterogeneity among the population of soils within each group. Also included in this plot are sets of four horizontal lines representing within-batch standard deviations for the field duplicates, preparation duplicates, and natural audit samples, and between-batch standard deviation for the natural audit samples. Each set of lines represents the data uncertainty within the windows that were established by the step function across the total range of concentration. Although the data uncertainty is not always constant within the windows for each type of sample represented, the lines are treated as constants. This latter figure is intended to show the contribution of measurement uncertainty to the overall variability of the routine data.

Additional tables corresponding to the step function statistical procedure for each of the parameters are given in Appendix C as supplemental information for the derivation of the precision data provided in the plots. Appendix D presents tables of data points that were sorted according to the sampling class/horizon group and the batch/sample number. These data correspond to routine or QA samples having inordinately high or low values that exert a disproportionate influence on the assessment of data quality and are of interest to data users when making evaluations of individual data sets represented in the plots or of individual batches of samples from a given analytical laboratory.

Moisture, Specific Surface, and Particle Size Analysis

Table 3-2
Figures 3-1 through 3-6

The analytical within-batch precision DQO for total sand, silt, and clay was not satisfied

Table 3-2. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of Moisture, Specific Surface, and Particle Size Analysis

Data set[a]	Parameter	df	SD[b]	DQO[b]	Pairs>DQO n	Pairs>DQO %
AS	MOIST	50	0.2910			
	SP_SUR	50	2.7636			
	SAND	50	1.9639	1.00	11	22.0
	VCOS	50	0.8262			
	COS	50	1.3240			
	MS	50	0.8099			
	FS	50	1.5903			
	VFS	50	1.0753			
	SILT	50	2.5757	1.00	13	26.0
	COSI	50	3.1846			
	FSI	50	0.9564			
	CLAY	47	1.2016	1.00	7	14.9
PD	MOIST	26	0.1442			
	SP_SUR	26	3.3767			
	SAND	26	1.7419		3	11.5
	VCOS	26	1.0037			
	COS	26	1.5593			
	MS	26	0.5419			
	FS	26	0.6286			
	VFS	26	0.6844			
	SILT	26	1.8274		7	26.9
	COSI	26	1.6481			
	FSI	26	1.2356			
	CLAY	26	0.7125		4	15.4
FD	MOIST	104	0.5149			
	SP_SUR	102	5.2200			
	SAND	102	2.3027		35	34.0
	VCOS	101	1.1022			
	COS	102	0.8049			
	MS	102	0.8018			
	FS	102	0.8429			
	VFS	102	1.0898			
	SILT	102	1.6107		30	29.1
	COSI	102	1.4002			
	FSI	102	1.5825			
	CLAY	102	1.5015		24	23.3
S/H	MOIST	609	1.1321			
	SP_SUR	608	16.1132			
	SAND	608	13.0921			
	VCOS	608	3.5589			
	COS	608	5.2780			
	MS	608	5.5616			
	FS	608	6.5113			
	VFS	608	5.3261			
	SILT	608	10.2580			
	COSI	608	5.0158			
	FSI	608	7.3044			
	CLAY	608	6.8395			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.
[b] Standard deviation data for mineral soil samples, reported in weight percent.

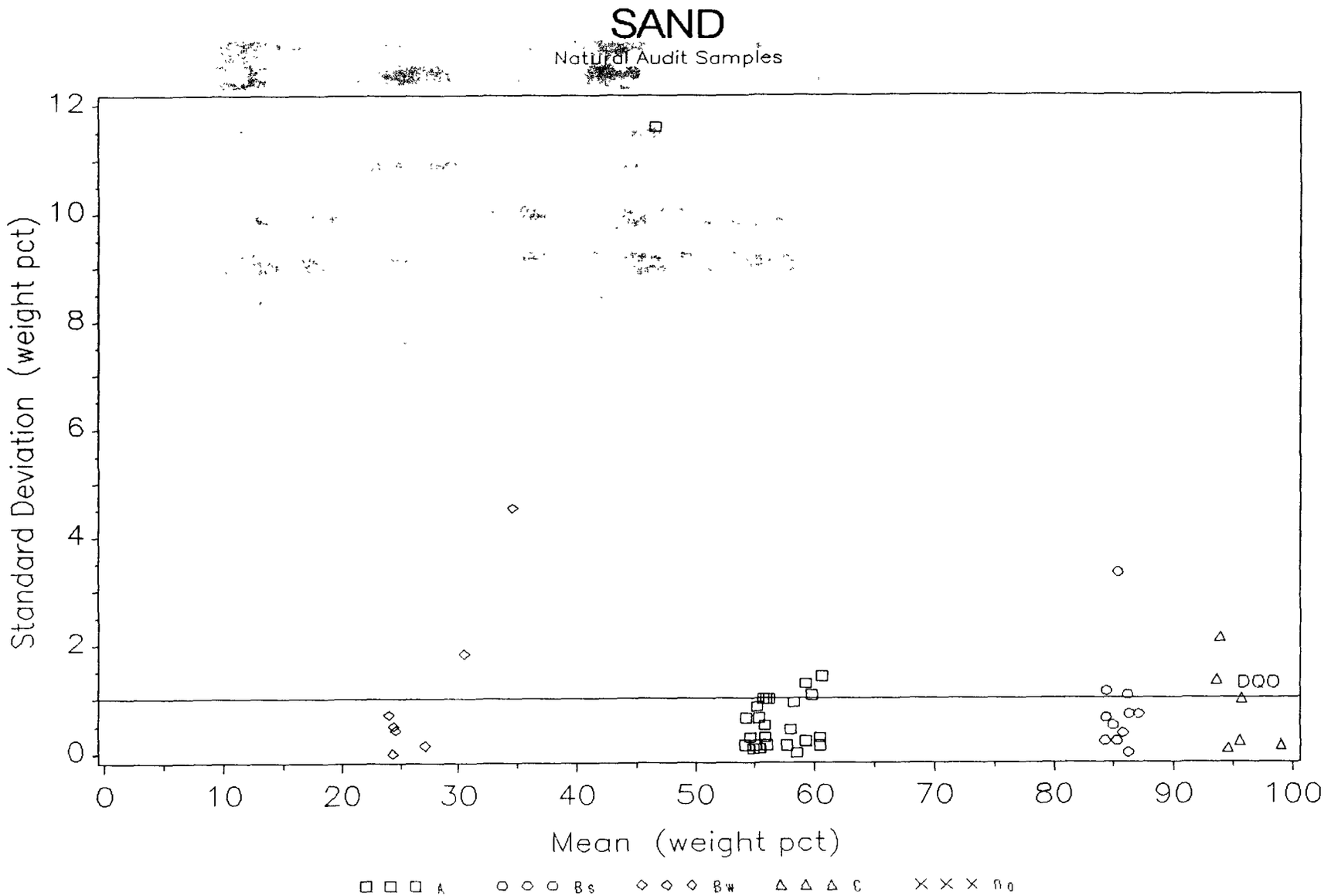


Figure 3-1. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SAND.

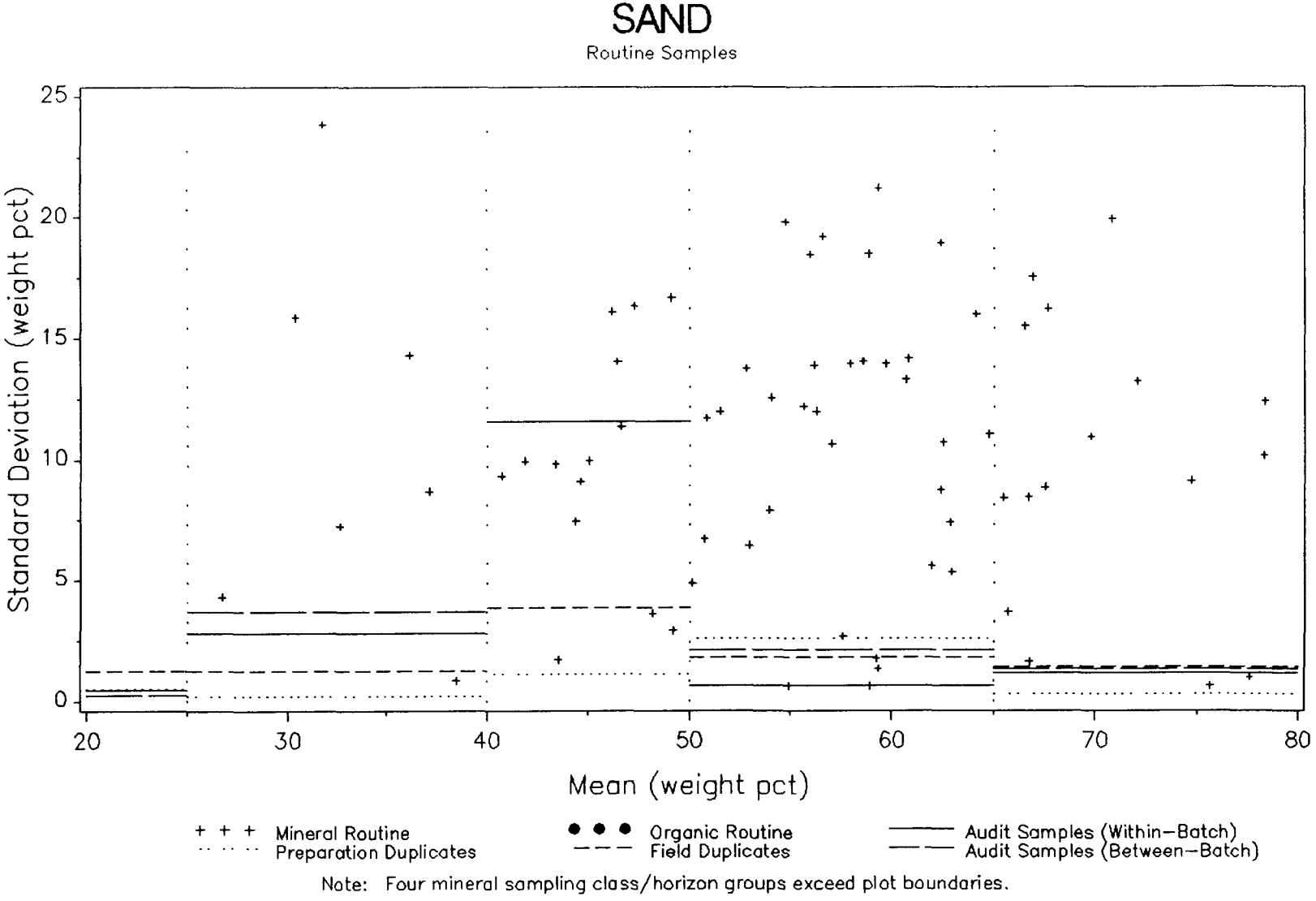


Figure 3-2. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SAND.

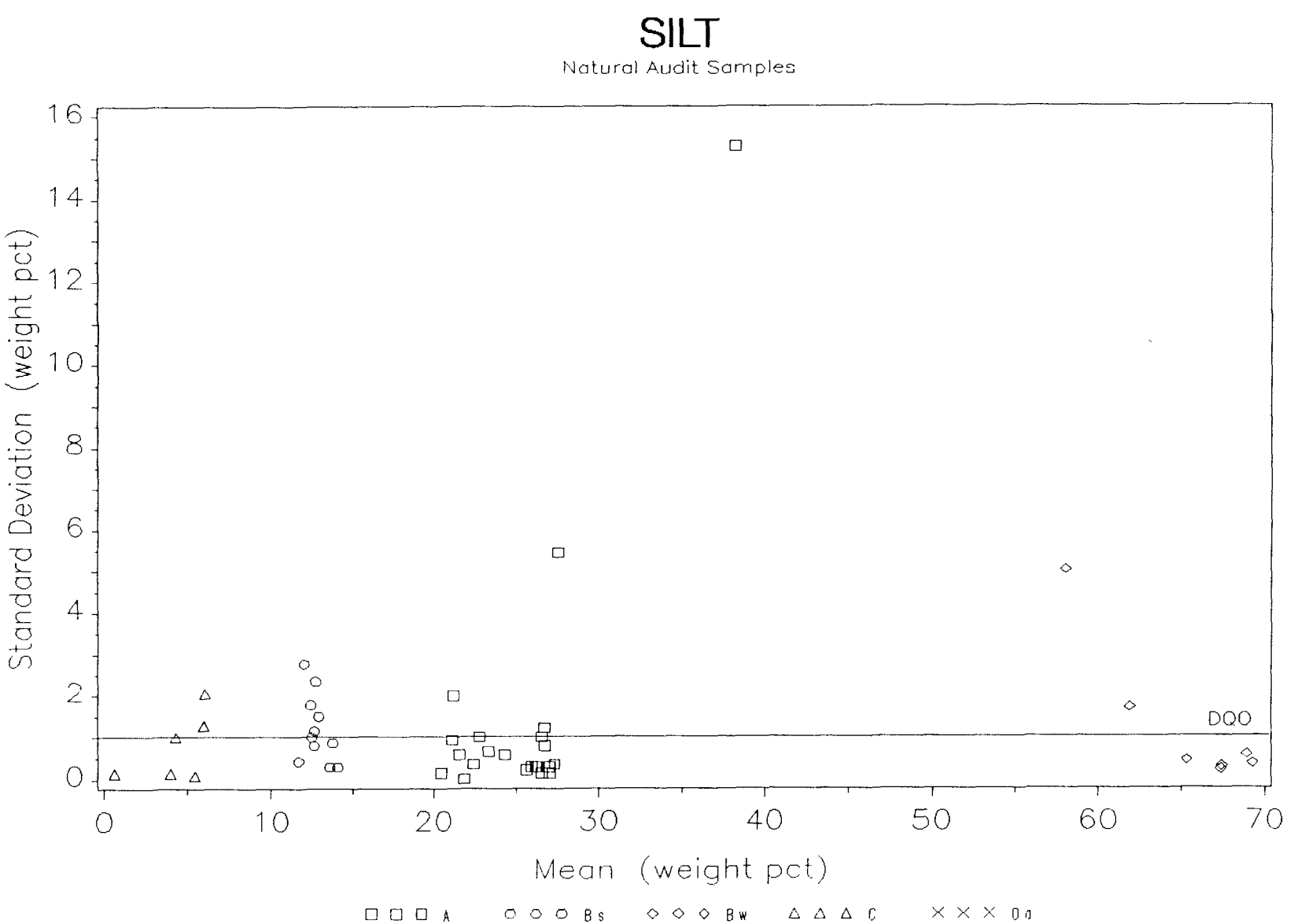


Figure 3-3. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SILT.

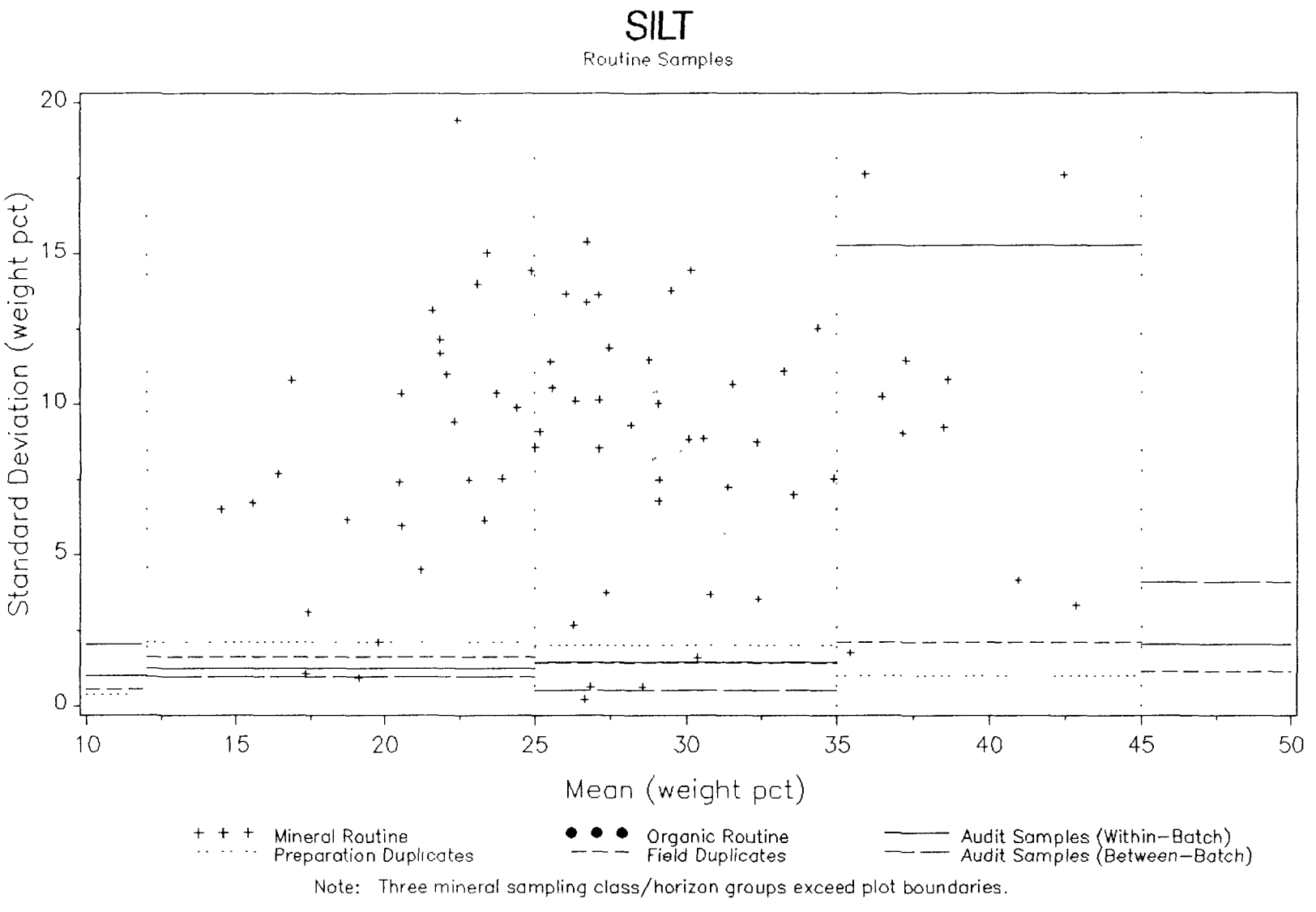


Figure 3-4. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SILT.

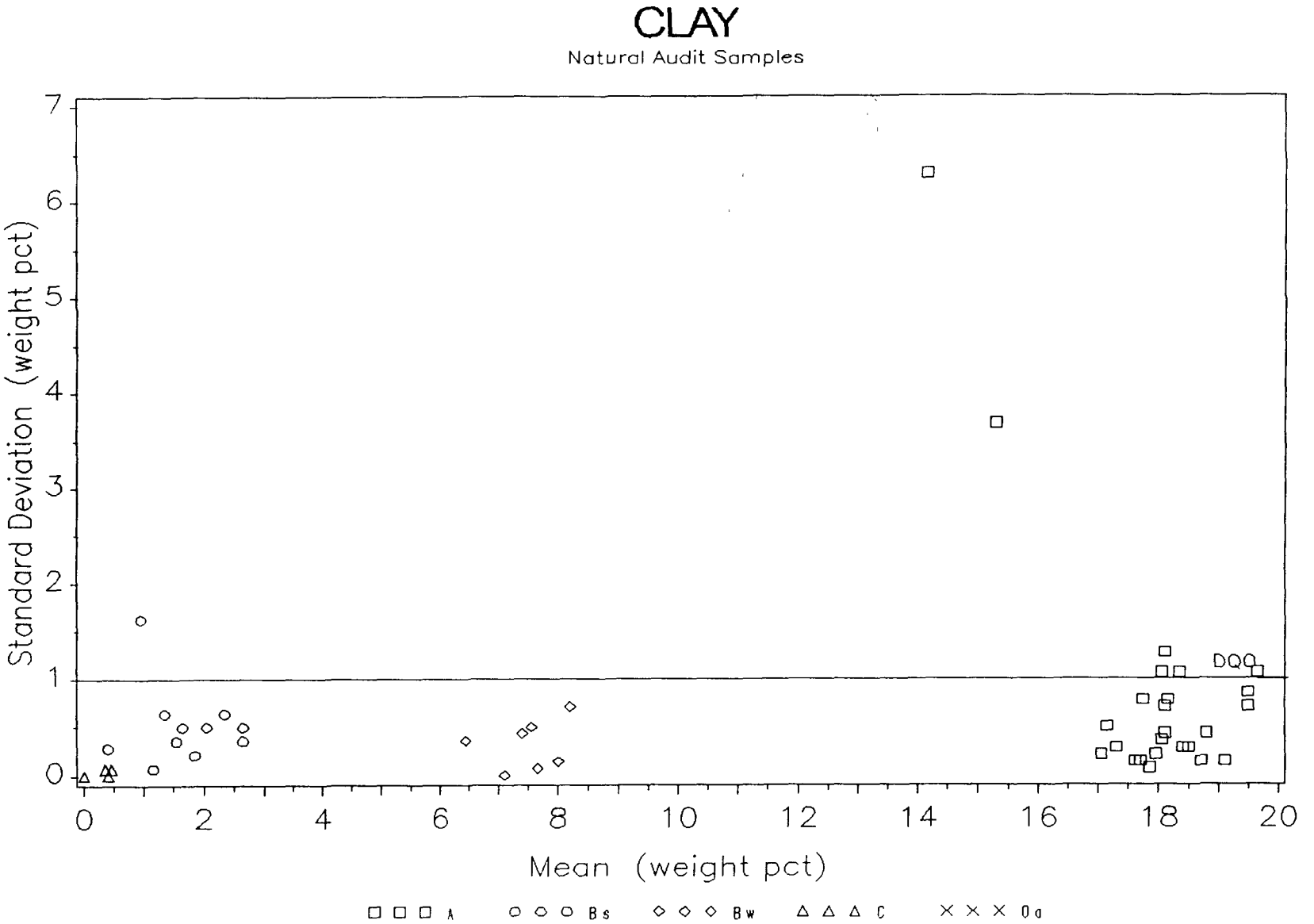


Figure 3-5. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for CLAY.

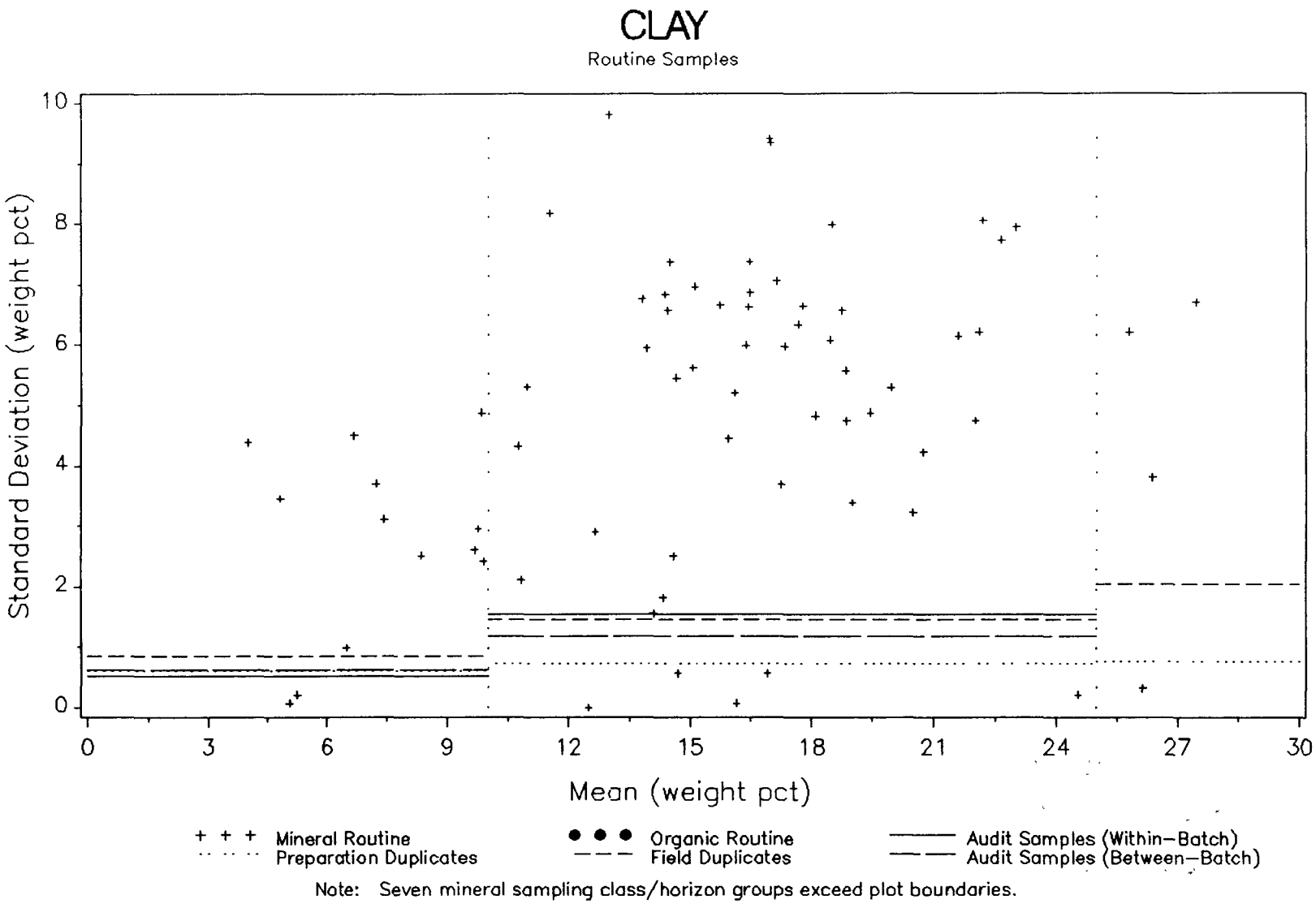


Figure 3-6. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for CLAY.

in the SBRP survey (see Table 3-2). For SAND and SILT, the DQO was exceeded by a factor of two, while the DQO for CLAY was only slightly exceeded. Based on data from the audit sample pairs, however, the DQO was satisfied 75 percent of the time or more for all of the parameters. A general pattern of increasing standard deviation with increased sources of confounded error was found, i.e., the standard deviations for the field duplicates exceeded those of the preparation duplicates and audit samples. Specific DQOs were not defined for moisture, specific surface, or the sand and silt fractions.

For MOIST, the analytical within-batch standard deviation observed in the audit samples was notably higher than the confounded analytical/preparation standard deviation observed in the preparation duplicates. It is thought that the drier climatic conditions under which the QA staff prepared the audit samples may have allowed a greater fluctuation in moisture among the different samples, thereby resulting in greater variability than was observed in the preparation duplicates. This variability had no effect on the calculation of air-dry/oven-dry coefficients for reporting routine sample data on an oven-dry weight basis.

Figures 3-1 through 3-6 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. The plots presented are provided only for those particle size parameters for which precision DQOs were defined, i.e., SAND, SILT, and CLAY. Appendix E contains the routine data plots for the remaining particle size parameters. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Soil pH

Table 3-3
Figures 3-7 through 3-12

The analytical within-batch precision DQO was easily satisfied in all cases for the pH parameters using data from the natural audit samples (see Table 3-3). A comparison of error estimates in the preparation duplicates and the audit samples suggests that the preparation error was negligible. A general pattern of increasing standard deviation with increased sources of confounded error was maintained.

Table 3-3. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of the Soil pH Parameters

Data set[a]	Parameter	df	SD[b]	DQO[b]	Pairs>DQO n	Pairs>DQO %
AS	PH_H2O	50	0.0349	0.15		
	PH_002M	50	0.0361	0.15	1	2.0
	PH_01M	50	0.0354	0.15	1	2.0
PD	PH_H2O	26	0.0350			
	PH_002M	26	0.0253			
	PH_01M	26	0.0307			
FD	PH_H2O	104	0.1009		8	7.7
	PH_002M	104	0.0917		5	4.8
	PH_01M	104	0.0846		4	3.8
S/H	PH_H2O	609	0.3331			
	PH_002M	609	0.3433			
	PH_01M	609	0.3516			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.
[b] Standard deviation data reported in pH units.

The standard deviation did not show any marked pattern of change over the measured pH range, hence, it was not necessary to fit a step function to the data from the three pH parameters. Unlike the other SBRP parameters, the error variance was calculated for the entire concentration range.

Figures 3-7 through 3-12 are plots of the audit sample data in relation to the DQO and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

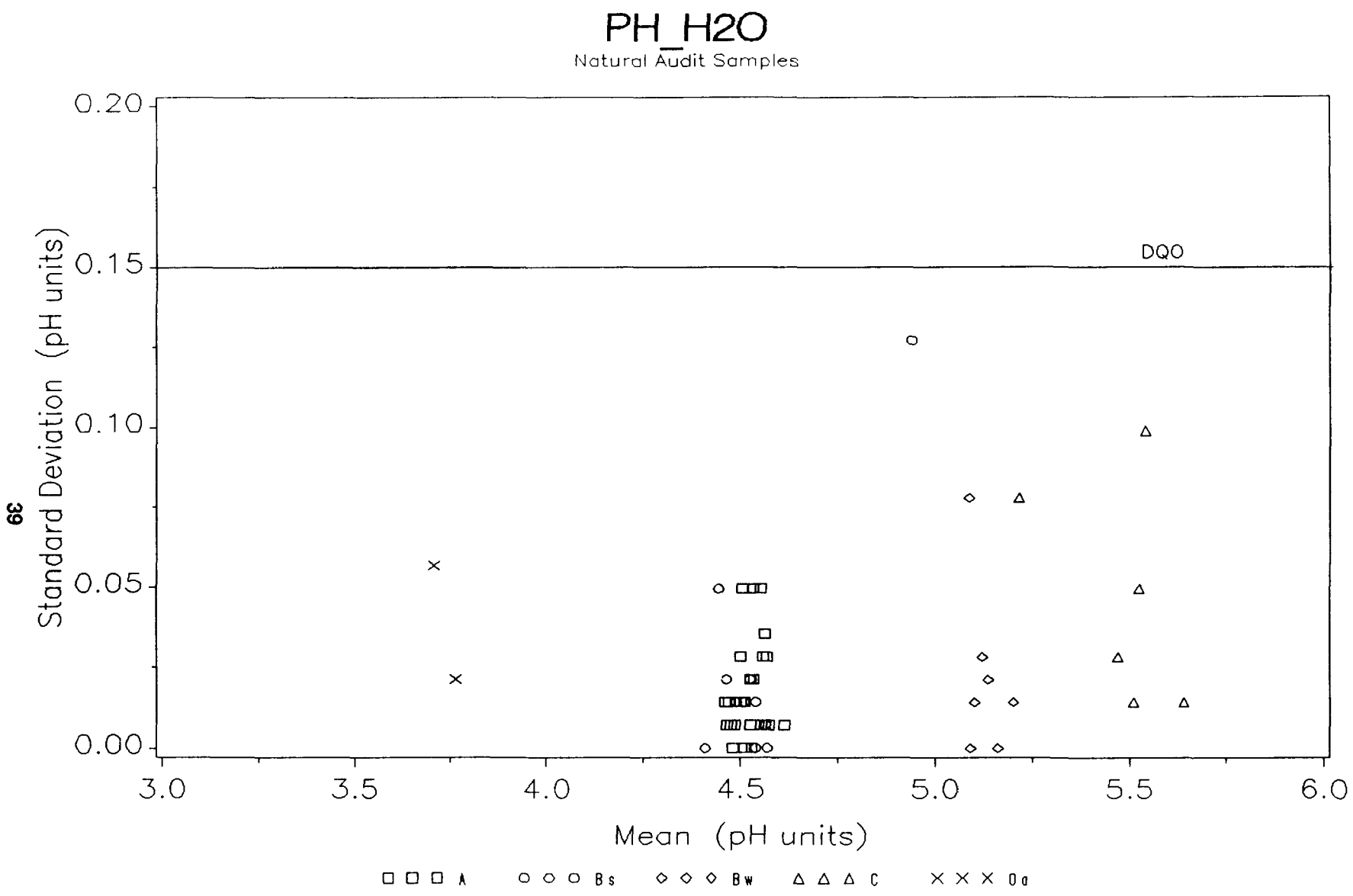


Figure 3-7. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for PH_H20.

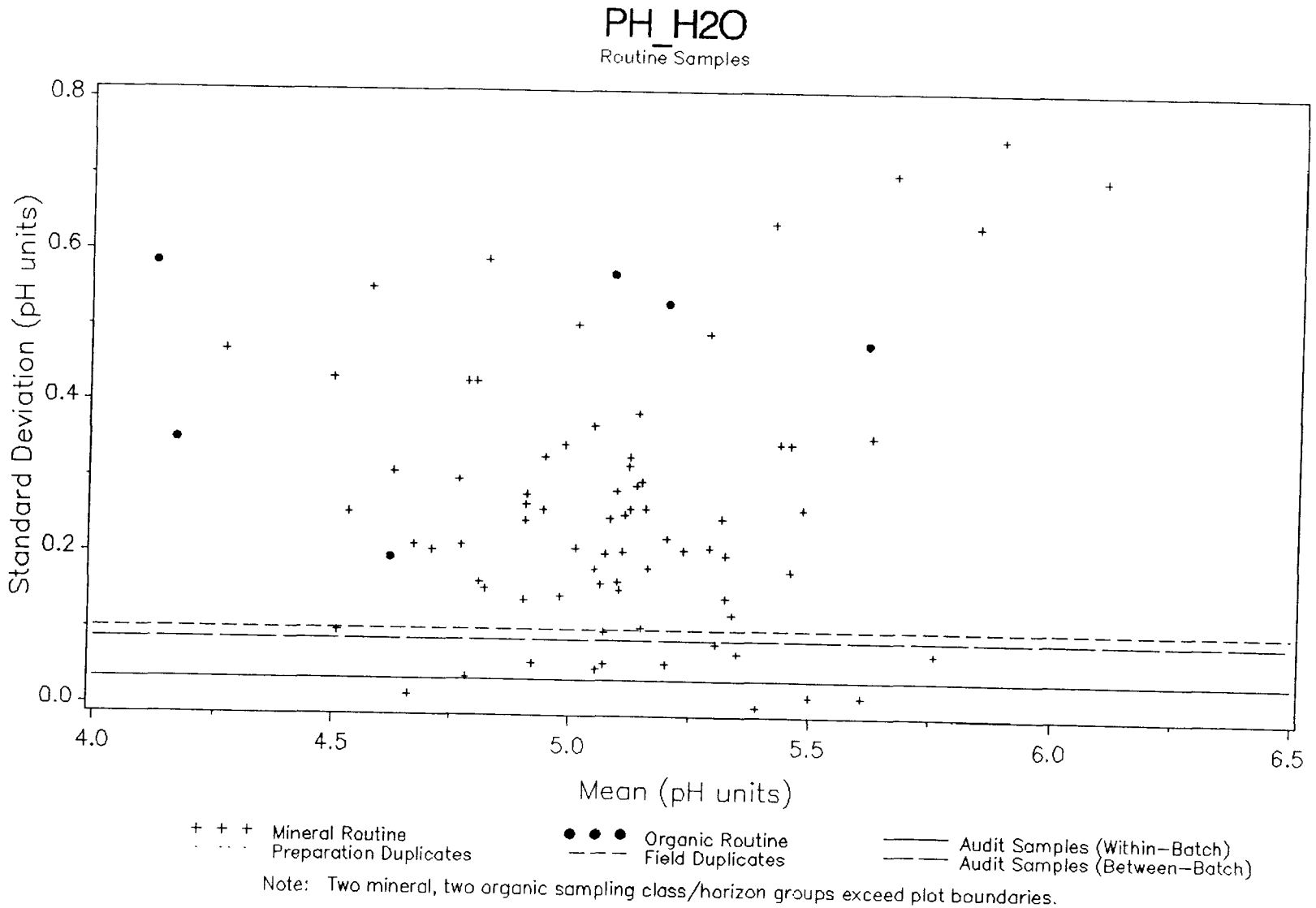


Figure 3-8. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for PH_H20.

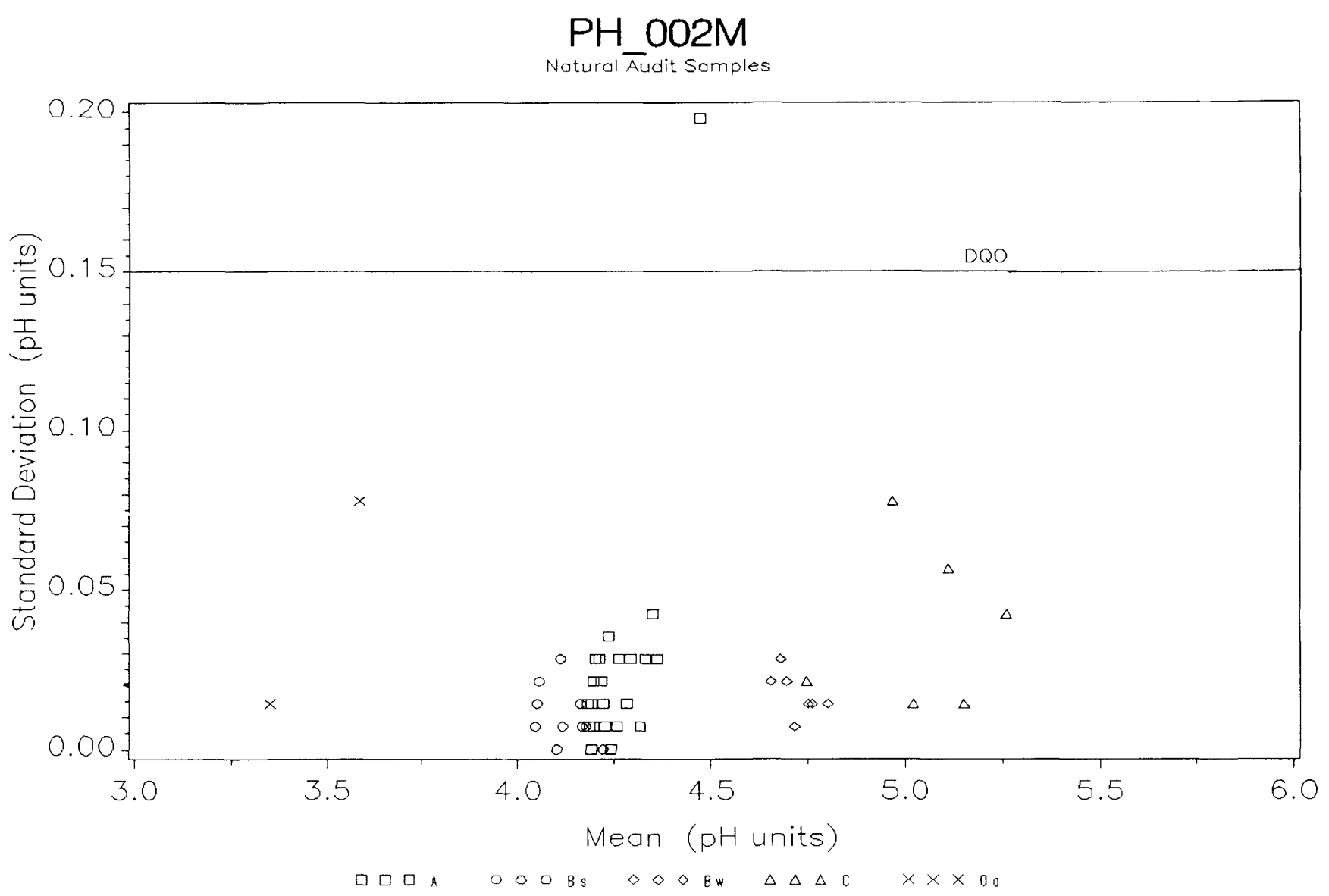


Figure 3-9. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for PH_002M.

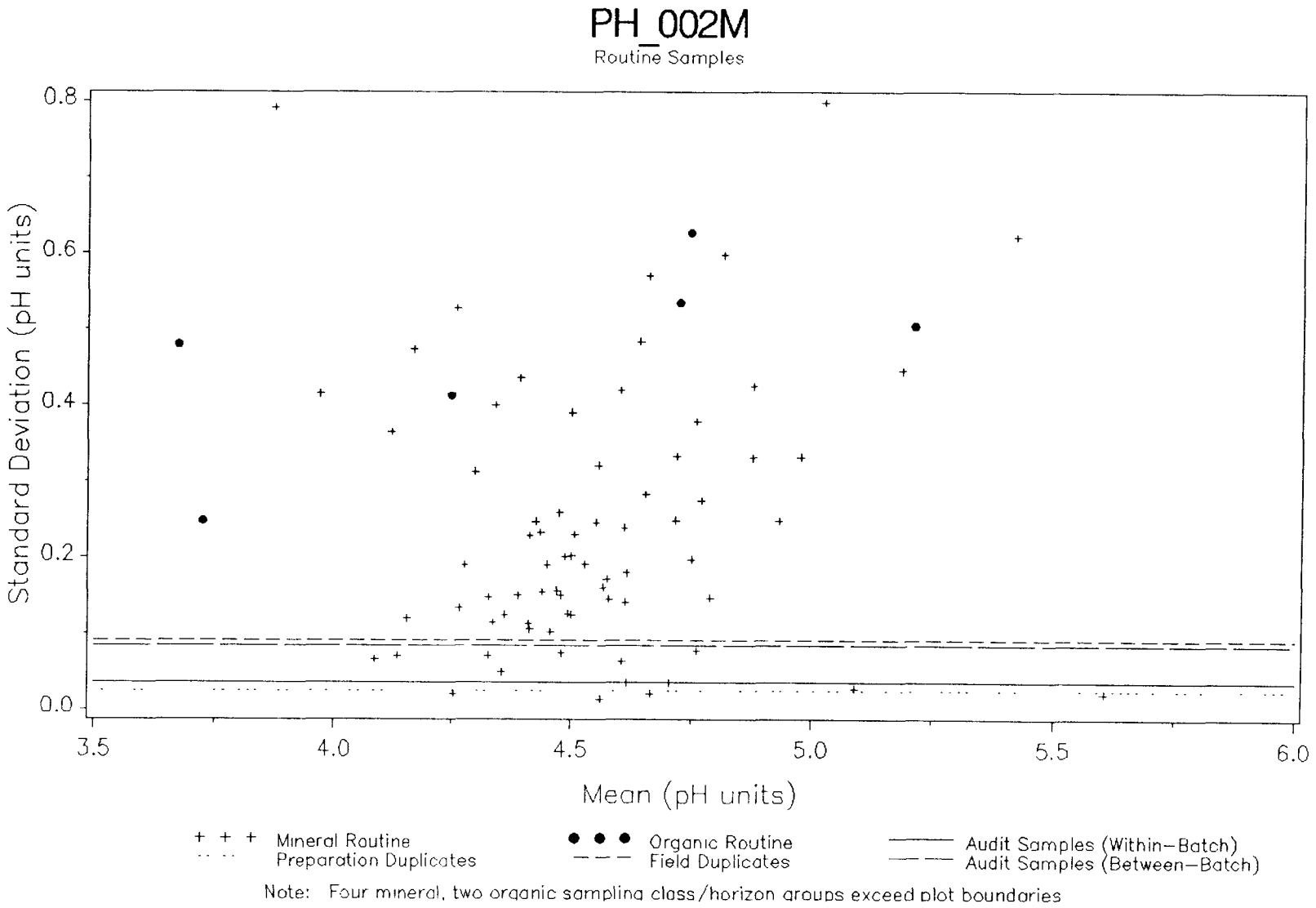


Figure 3-10. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for PH_002M.

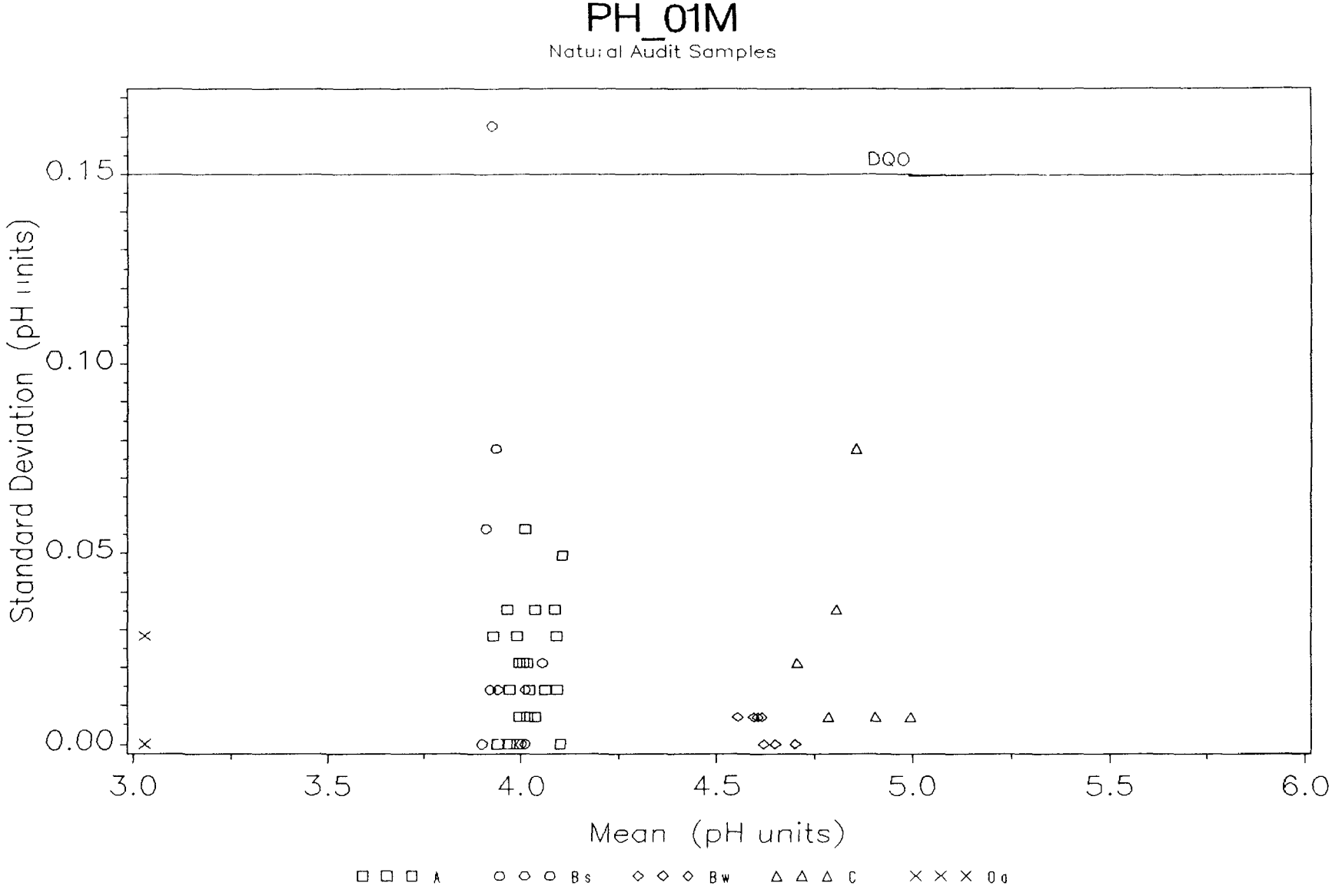


Figure 3-11. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for PH_01M.

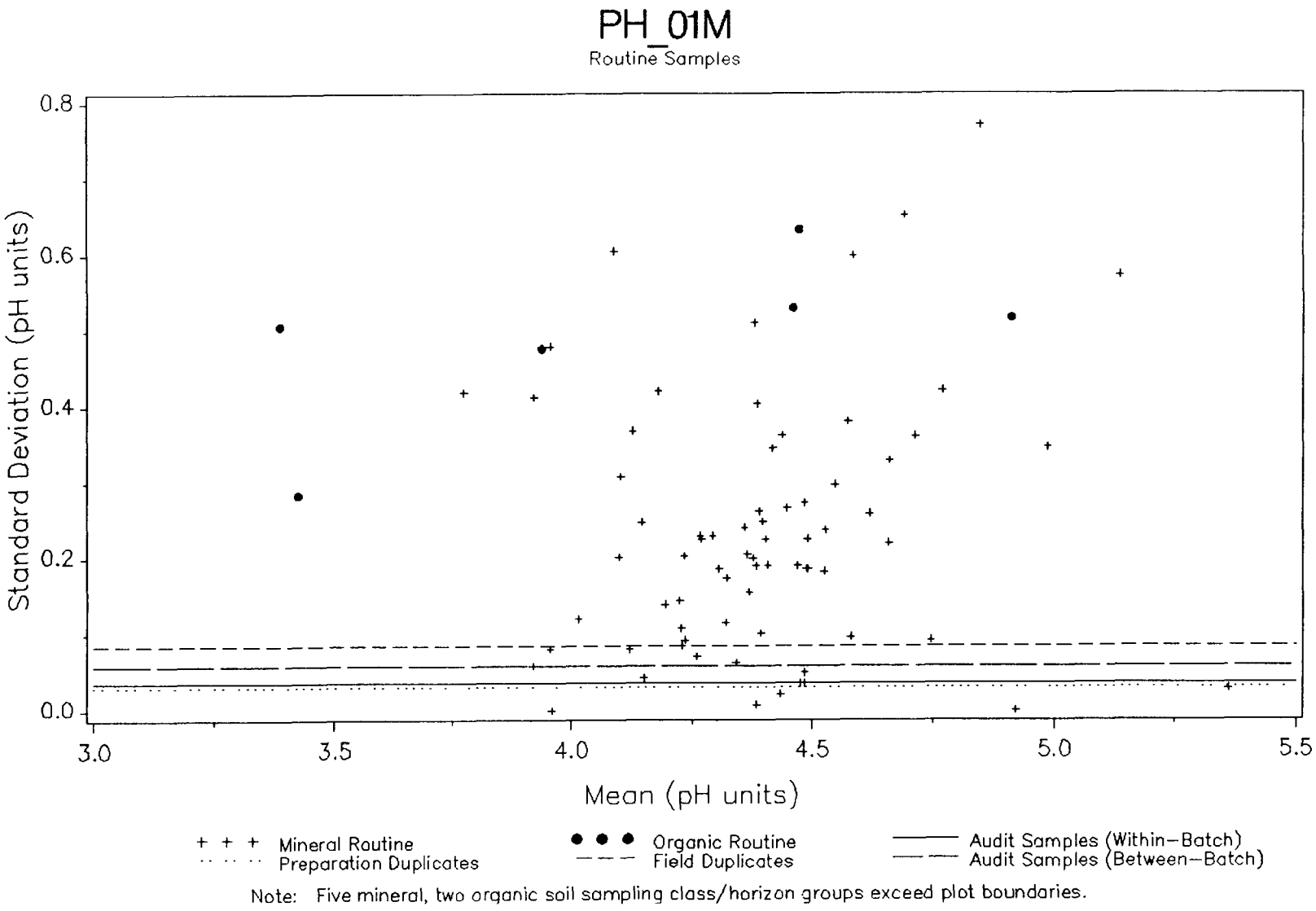


Figure 3-12. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for PH_01M.

Exchangeable Cations in Ammonium Chloride

Table 3-4
Figures 3-13 through 3-20

The analytical within-batch precision DQOs were satisfied for all of the cations except K_CL in the upper tier (see Table 3-4). The inordinate effect of data from one audit sample pair prevented this particular DQO from being met. The preparation duplicates and field duplicates also satisfied the analytical DQO for the lower tier even though these samples were susceptible to additional confounded errors from soil sampling or preparation. The general trend of increasing standard deviation with increased sources of confounded error was maintained. For NA_CL, the lack of data in the upper concentration window renders the precision estimates conditional on the presence of sufficient data within this range.

Figures 3-13 through 3-20 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Table 3-4. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of the Exchangeable Cations in Ammonium Chloride

Data set[a]	Parameter	Below the knot[b] df	SD	DQO	Pairs>DQO n	%	Above the knot[b] df	RSD	DQO	Pairs>DQO n	%
AS	CA_CL	9	0.0250	0.03	1	11.1	41	12.4%	15%	3	7.3
	MG_CL	29	0.0073	0.03			21	4.3%	15%		
	K_CL	23	0.0102	0.03			26	34.7%	15%	1	3.8
	NA_CL	48	0.0187	0.03	4	8.3	.	.	15%	.	.
PD	CA_CL	17	0.0314		4	23.5	9	5.1%			
	MG_CL	13	0.0140		1	7.7	13	16.1%		1	7.7
	K_CL	19	0.0093				7	5.1%			
	NA_CL	24	0.0097				.	.		.	.
FD	CA_CL	56	0.0308		9	15.8	47	42.3%		13	28.3
	MG_CL	59	0.0250		7	11.9	45	47.0%		6	13.3
	K_CL	80	0.0185		10	12.5	24	29.3%		6	25.0
	NA_CL	101	0.0172		8	7.9	1	10.8%			
S/H	CA_CL	224	0.1179				385	170.3%			
	MG_CL	279	0.1147				330	99.4%			
	K_CL	476	0.0817				133	61.4%			
	NA_CL	609	0.0350				.	.			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.
[b] Standard deviation and RSD data in reporting units and percent, respectively, for mineral soil samples below and above the knot point of 0.20 meq/100g; a dot signifies a lack of data occupying that range.

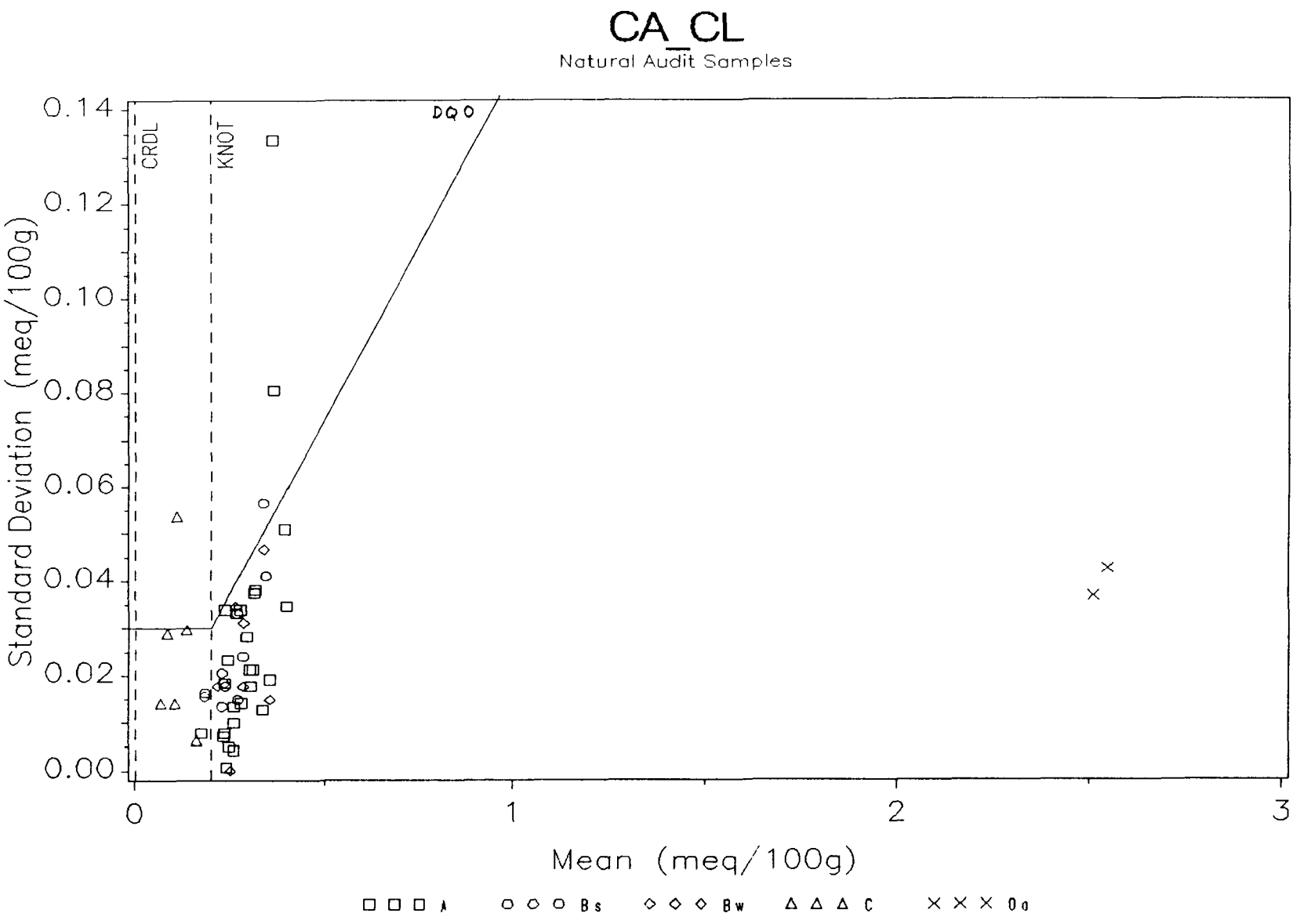


Figure 3-13. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for CA_CL

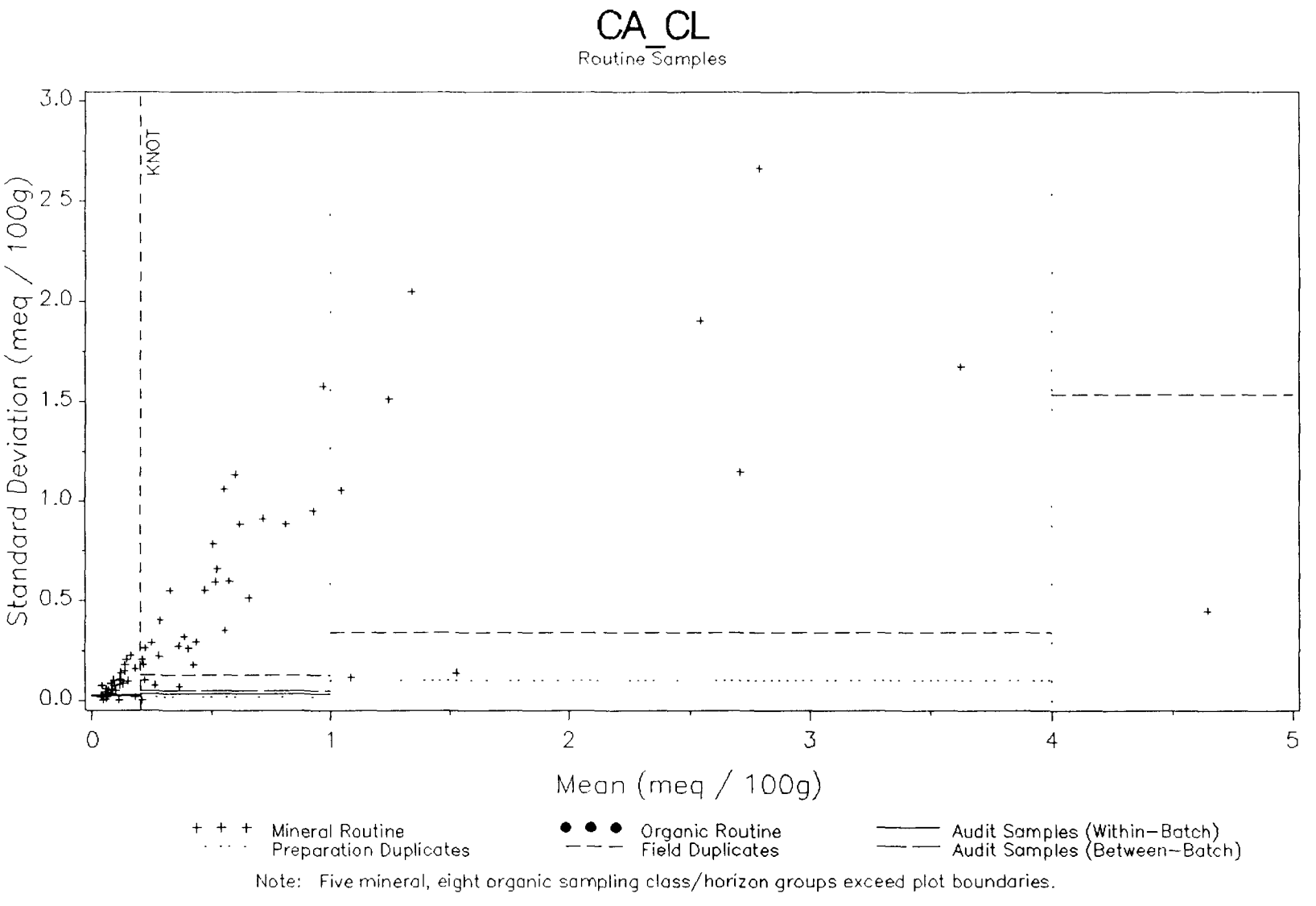


Figure 3-14. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for CA_CL.

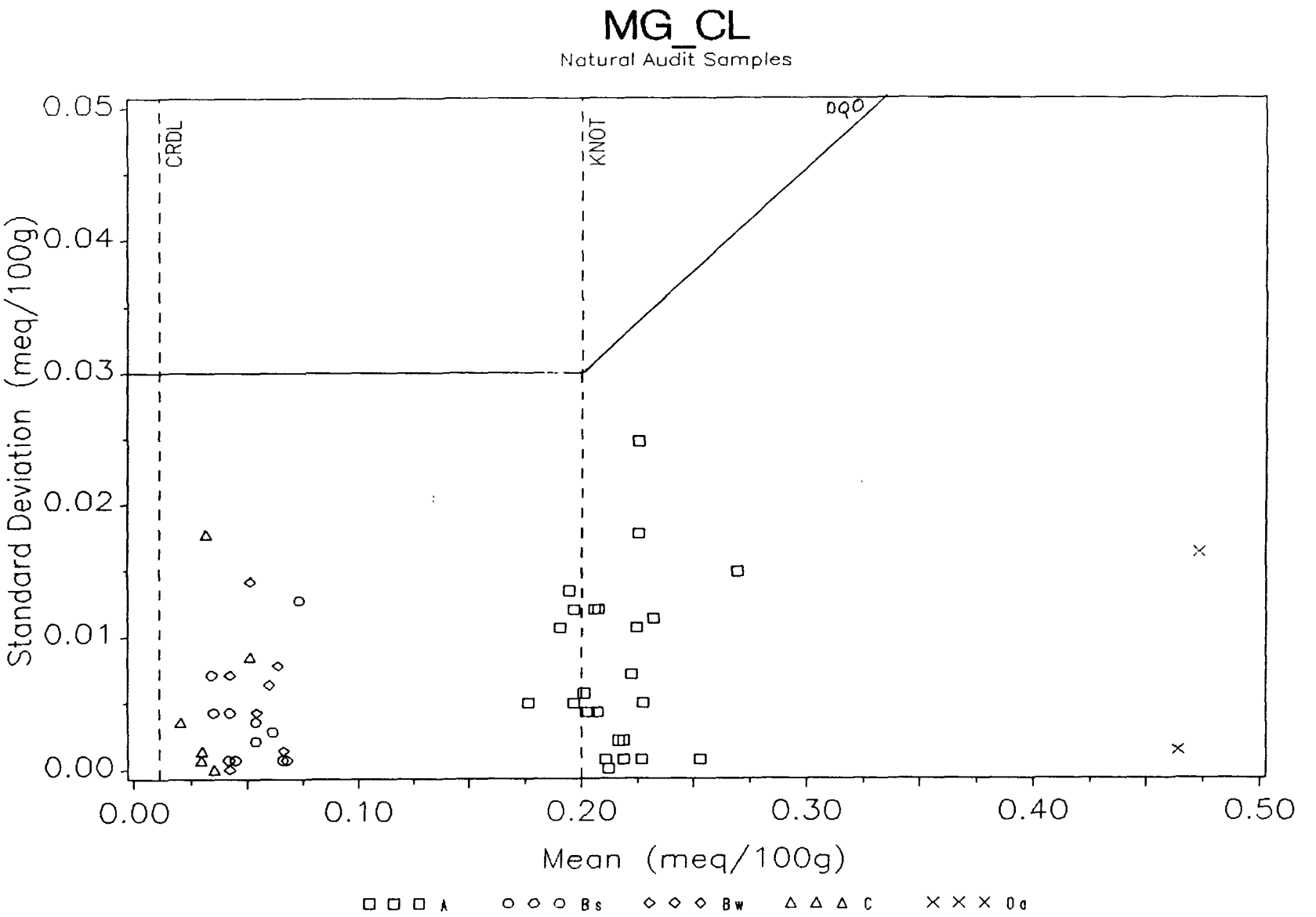


Figure 3-15. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for MG_CL.

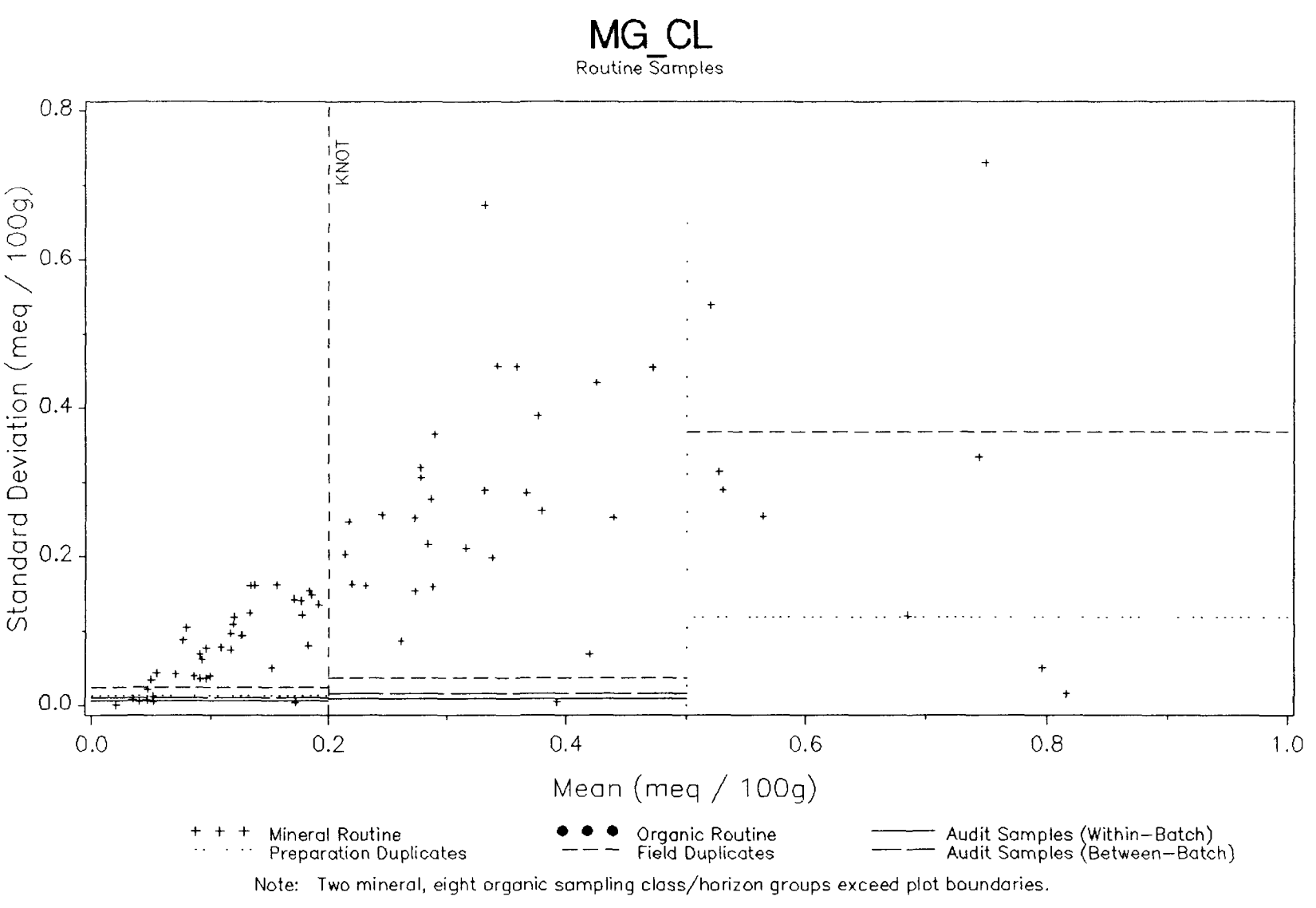


Figure 3-16. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for MG_CL

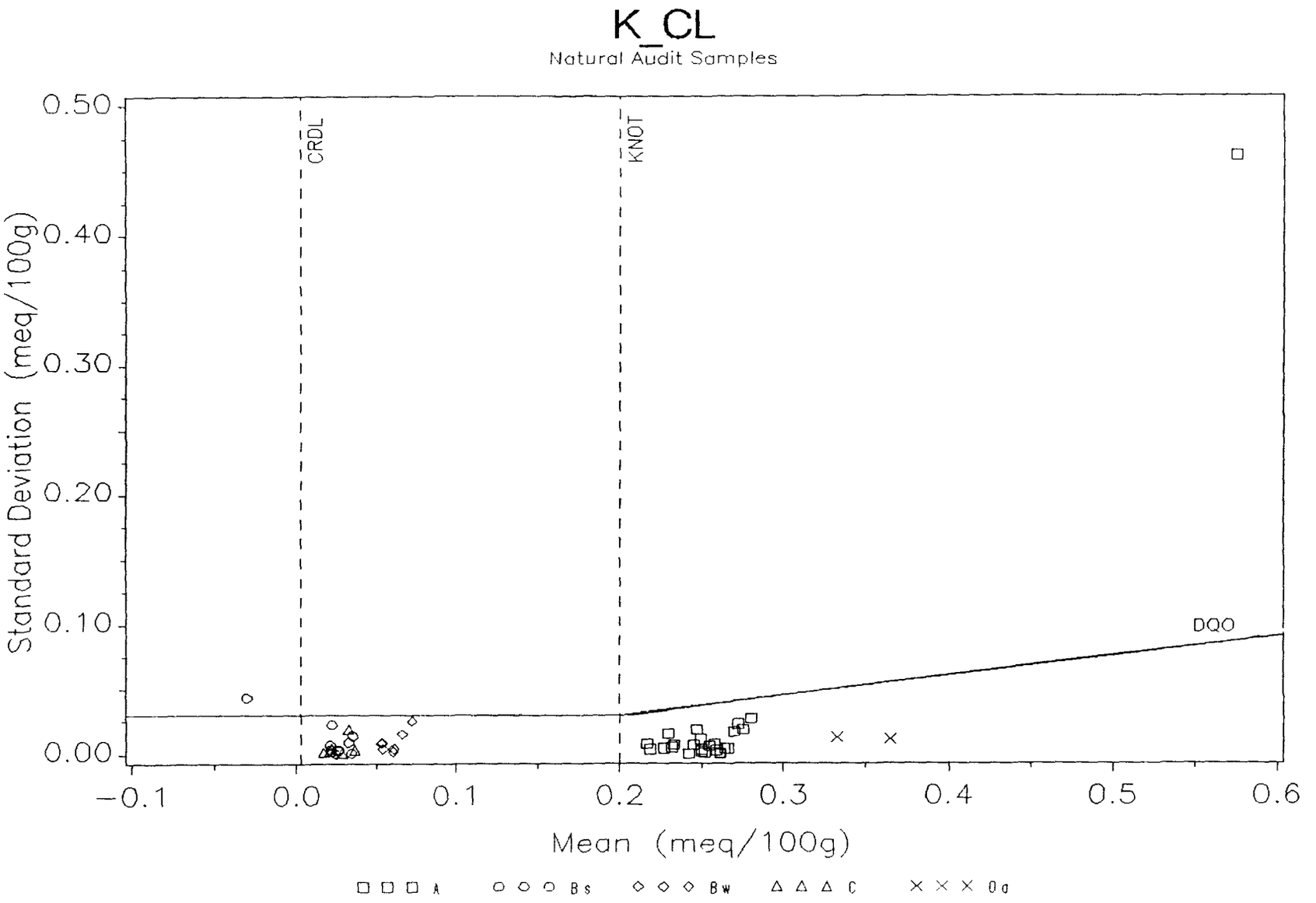


Figure 3-17. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for K_CL

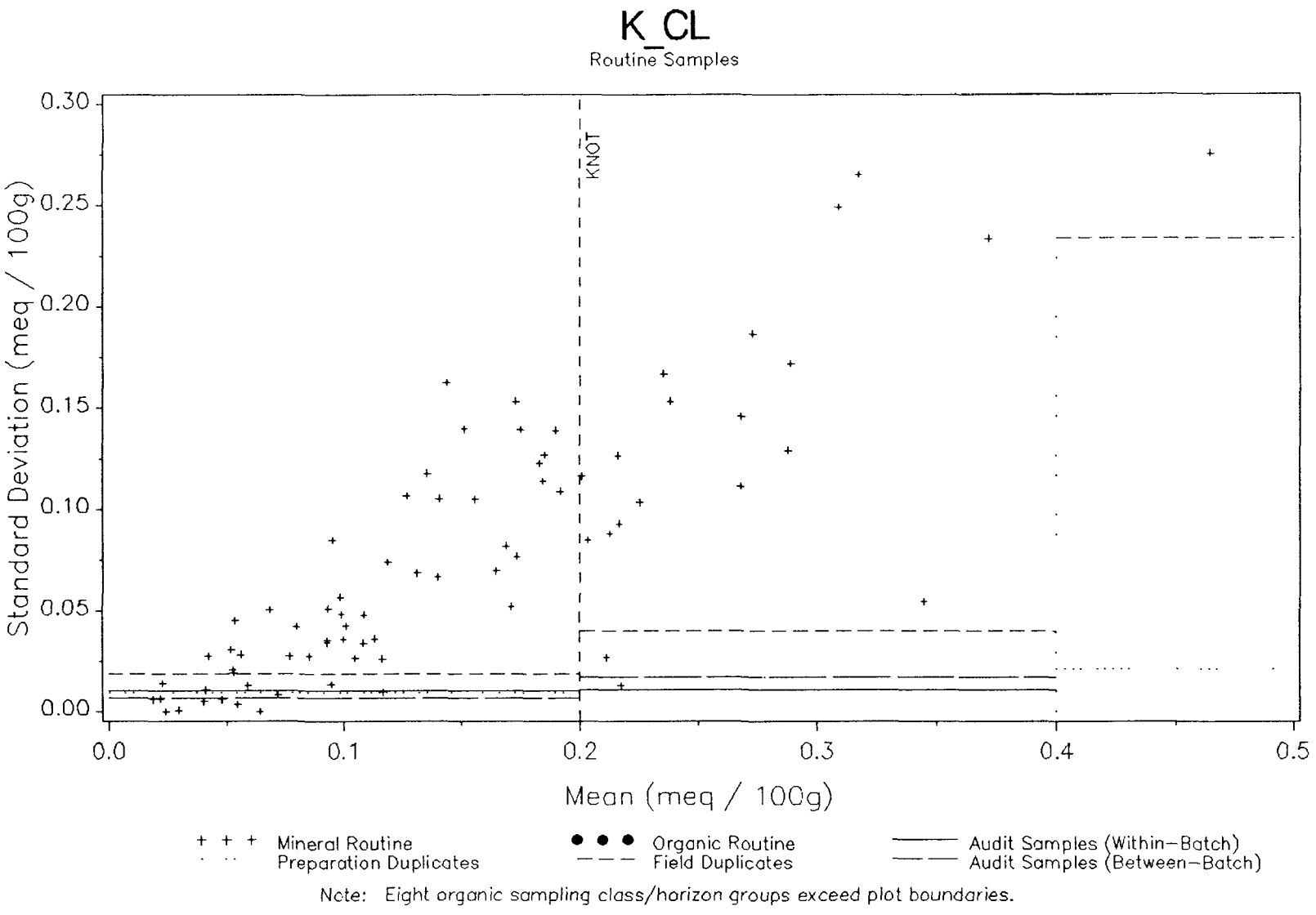


Figure 3-18. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for K_CL

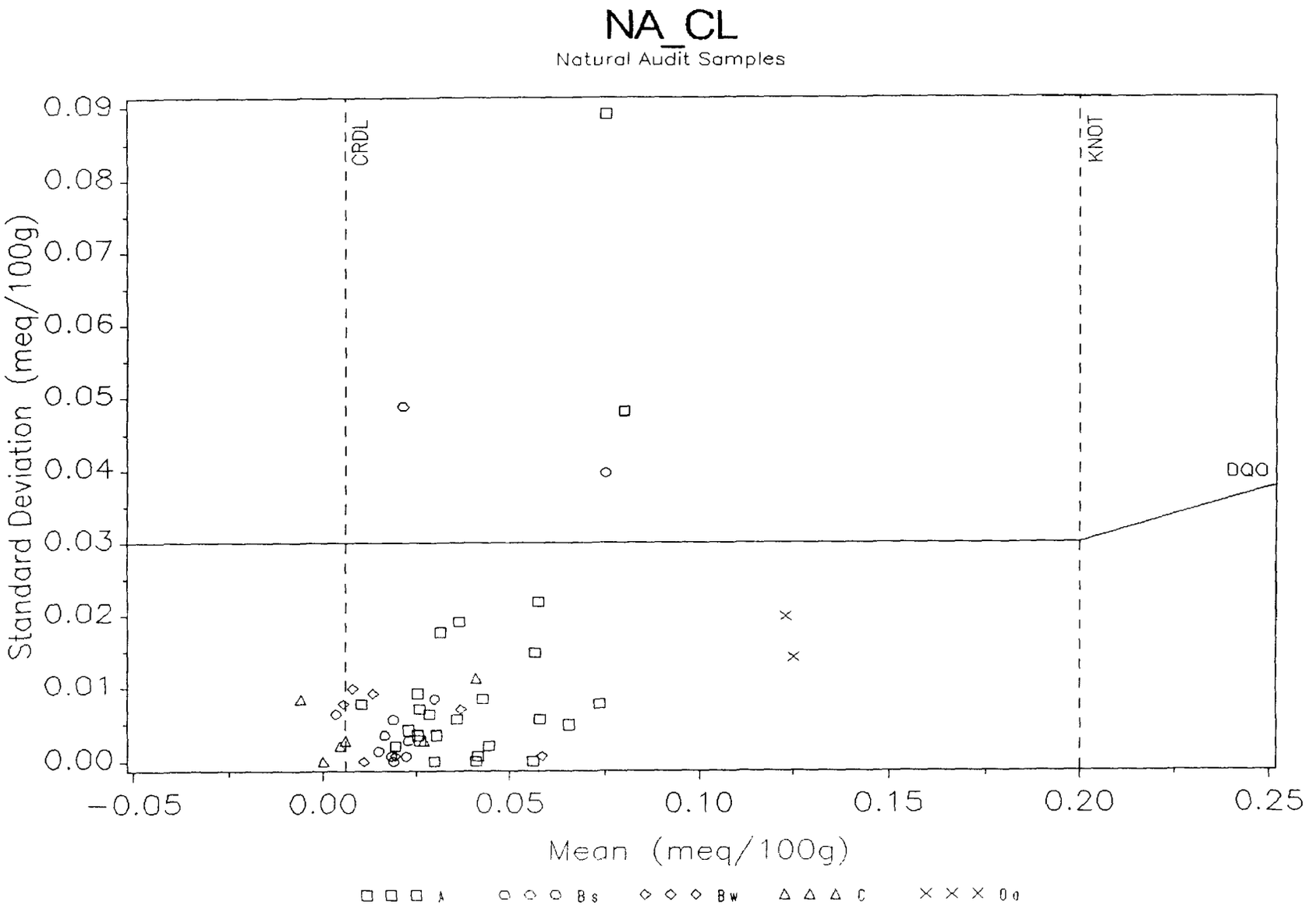


Figure 3-19. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for NA_CL.

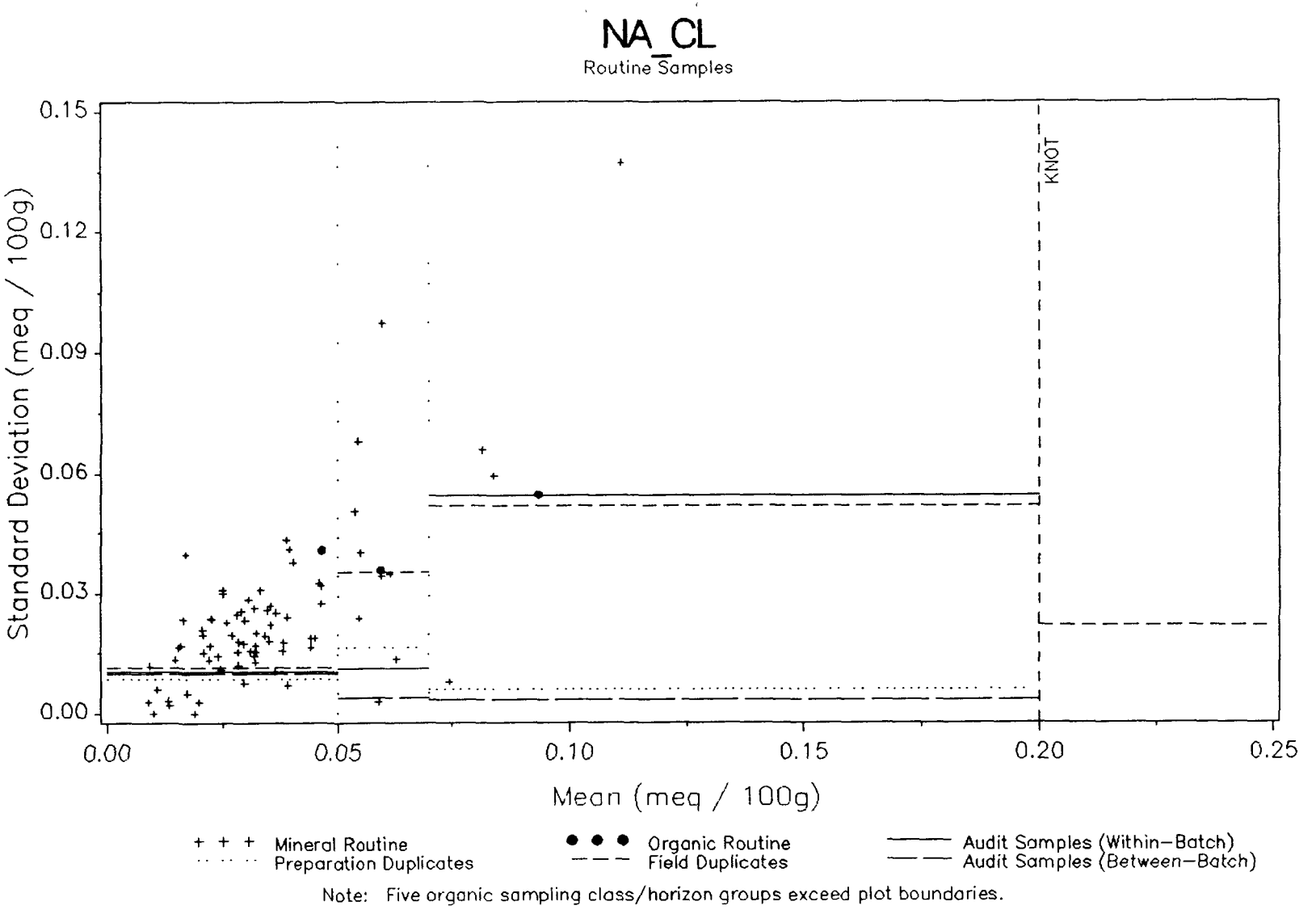


Figure 3-20. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for NA_CL.

Exchangeable Cations in Ammonium Acetate

Table 3-5
Figures 3-21 through 3-28

The analytical within-batch precision DQOs were satisfied for all parameters except for K_OAC data above the knot which slightly exceeded the DQO (see Table 3-5). A comparison of data from the preparation duplicates and the audit samples suggests that the preparation component of the data collection error is very small. A general pattern of increasing standard deviation with increased sources of confounded error was maintained.

Figures 3-21 through 3-28 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Table 3-5. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of the Exchangeable Cations in Ammonium Acetate

Data set[a]	Parameter	Below the knot[b] df	SD	DQO	Pairs>DQO n	%	Above the knot[b] df	RSD	DQO	Pairs>DQO n	%
AS	CA_OAC	17	0.0220	0.03	2	11.8	33	12.1%	15%	8	24.2
	MG_OAC	25	0.0063	0.03			25	6.9%	15%	1	4.0
	K_OAC	24	0.0087	0.03			26	15.8%	15%	3	11.5
	NA_OAC	48	0.0119	0.03	1	2.1	.	.	15%	.	.
PD	CA_OAC	15	0.0214		1	6.7	11	12.4%		2	18.2
	MG_OAC	12	0.0072				14	11.8%		1	7.1
	K_OAC	18	0.0085				7	14.0%		1	14.3
	NA_OAC	25	0.0074				.	.		.	.
FD	CA_OAC	52	0.0270		12	23.1	49	50.8%		16	32.7
	MG_OAC	58	0.0229		6	10.3	46	37.3%		6	13.0
	K_OAC	79	0.0168		7	8.8	25	32.3%		7	29.2
	NA_OAC	101	0.0122		2	2.0	1	9.0%			
S/H	CA_OAC	218	0.1259				391	169.7%			
	MG_OAC	231	0.1195				378	97.0%			
	K_OAC	486	0.0756				123	58.4%			
	NA_OAC	608	0.0420								

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.

[b] Standard deviation and RSD data in reporting units and percent, respectively, for mineral soil samples below and above the knot point of 0.20 meq/100g; a dot signifies a lack of data occupying that range.

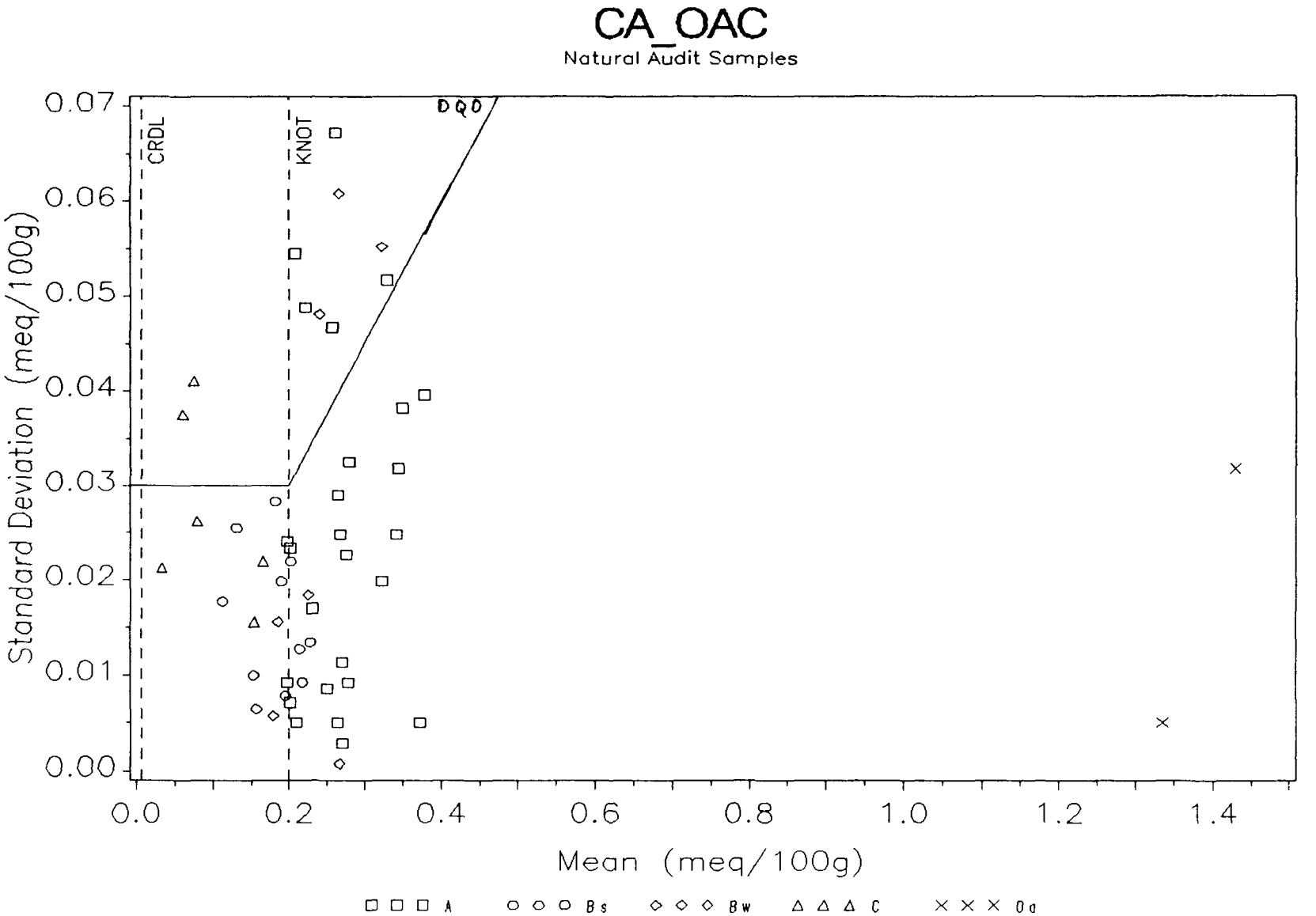


Figure 3-21. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for CA_OAC.

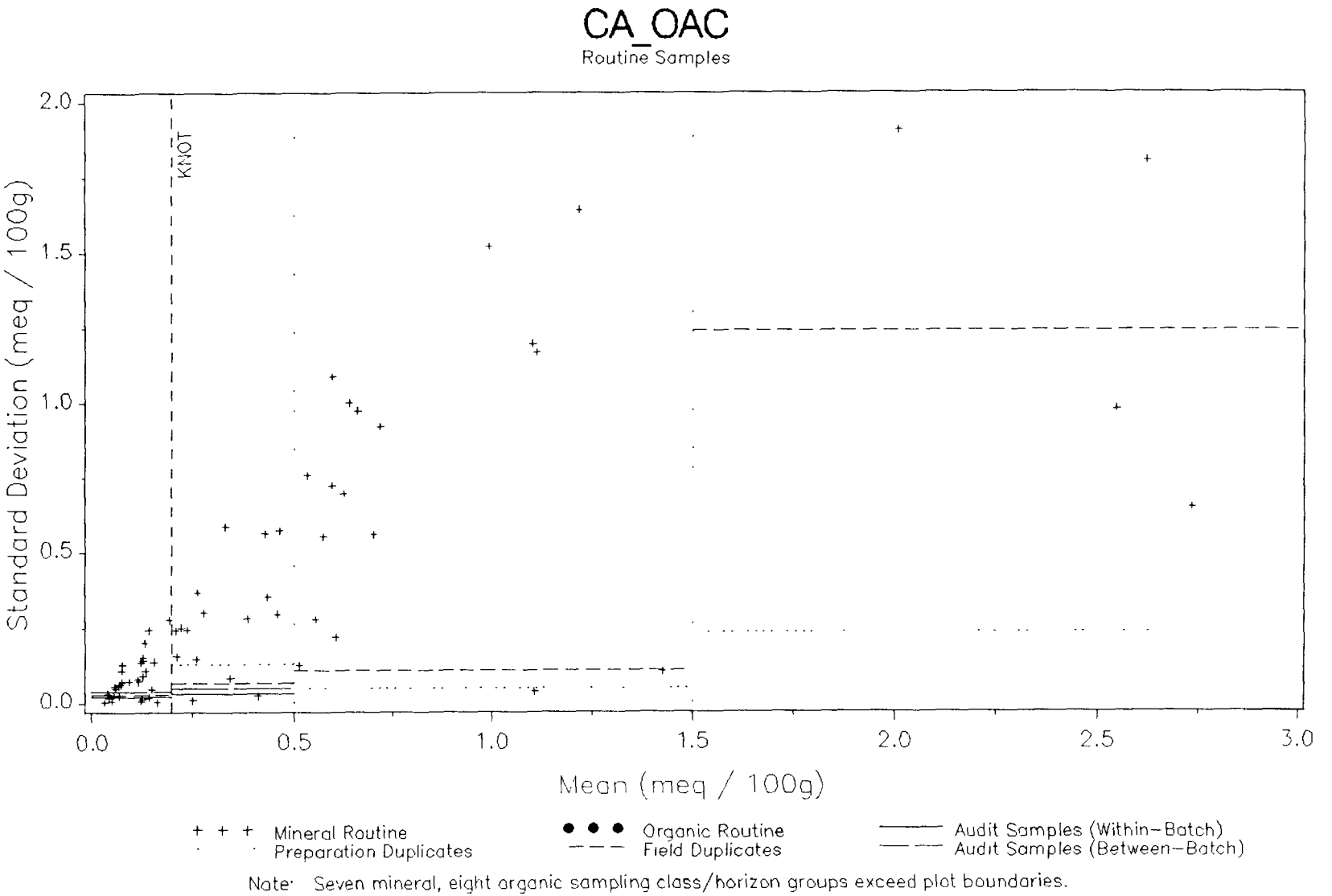


Figure 3-22. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for CA_OAC.

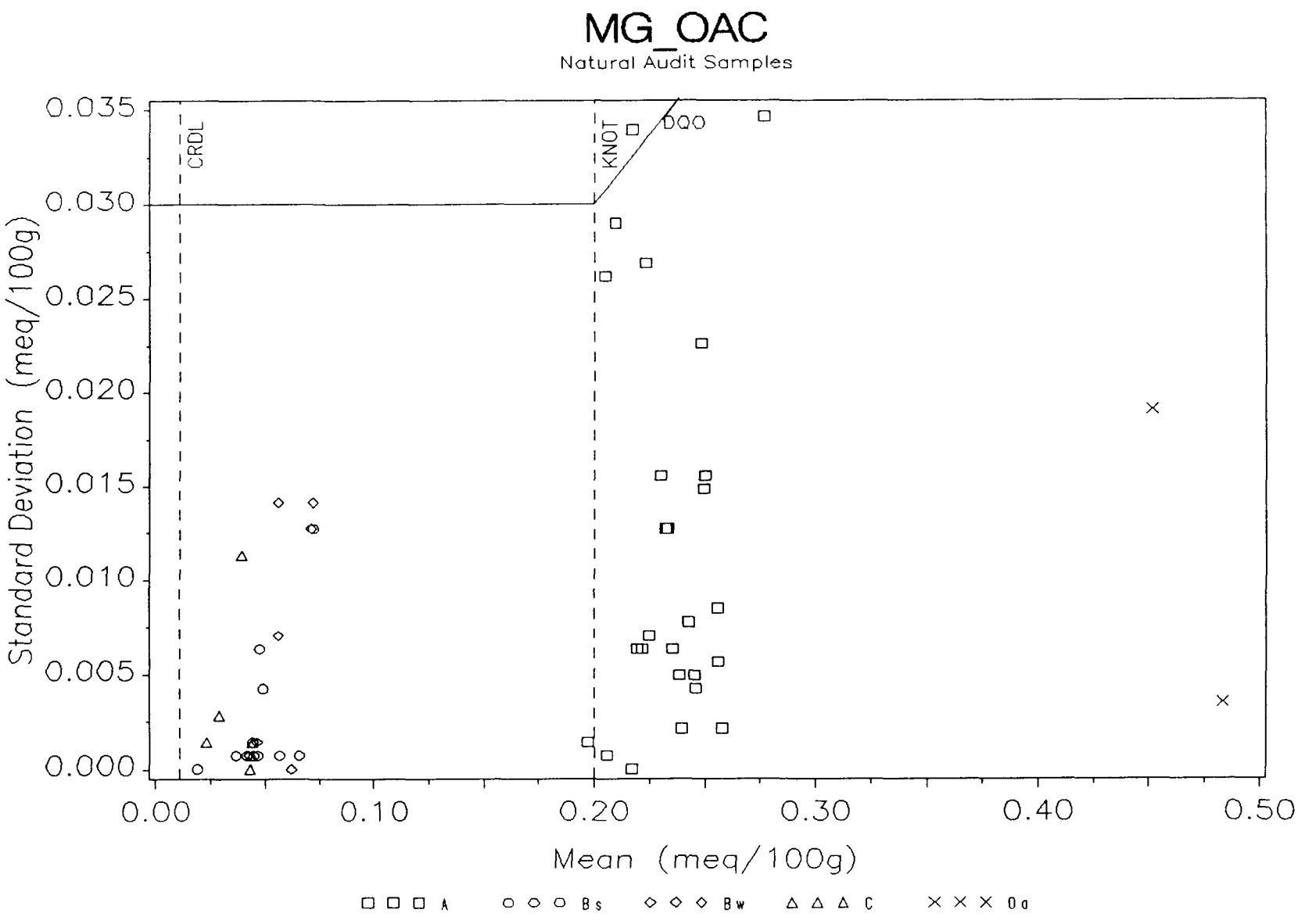


Figure 3-23. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for MG_OAC.

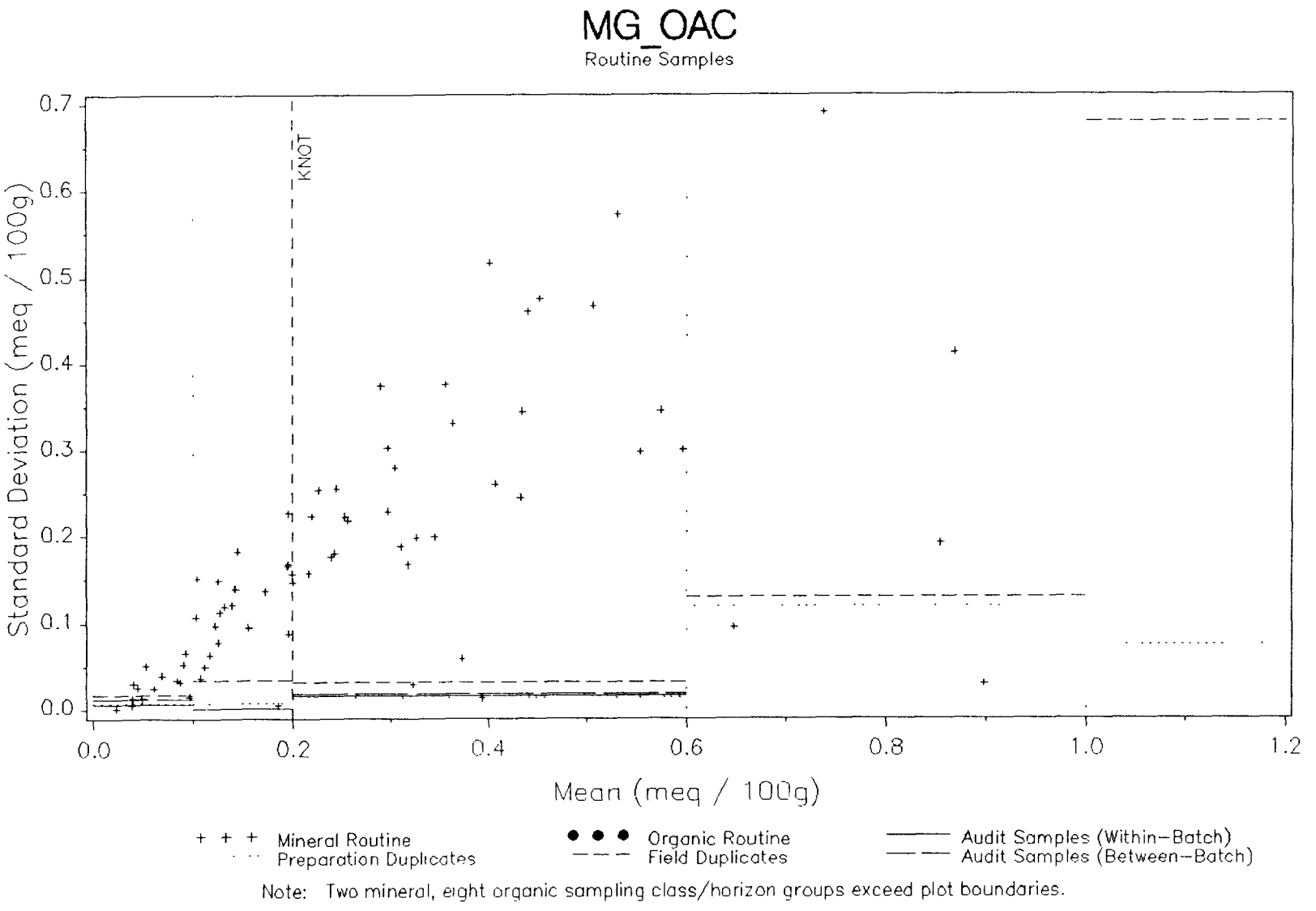


Figure 3-24. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for MG_OAC.

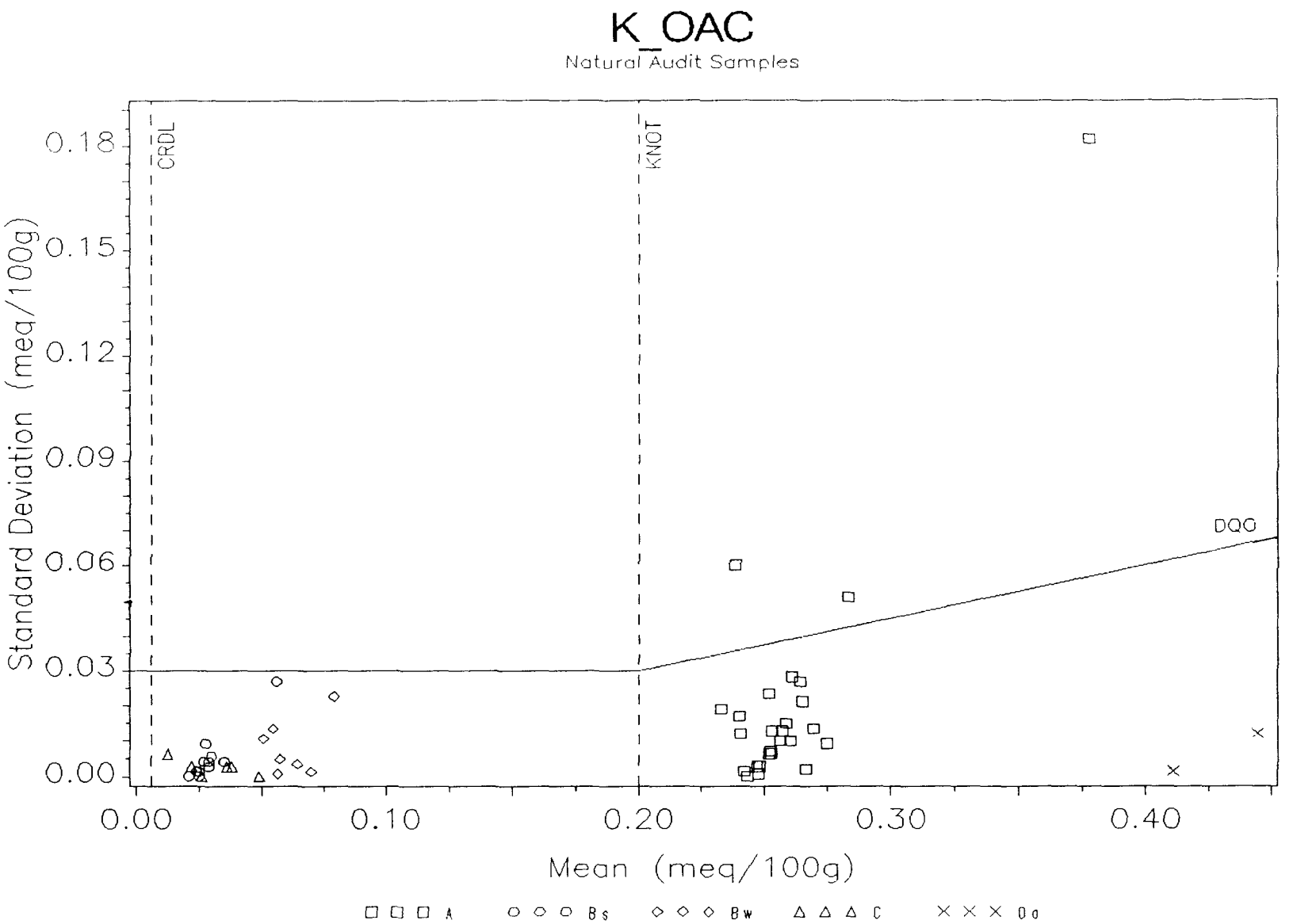


Figure 3-25. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for K_OAC.

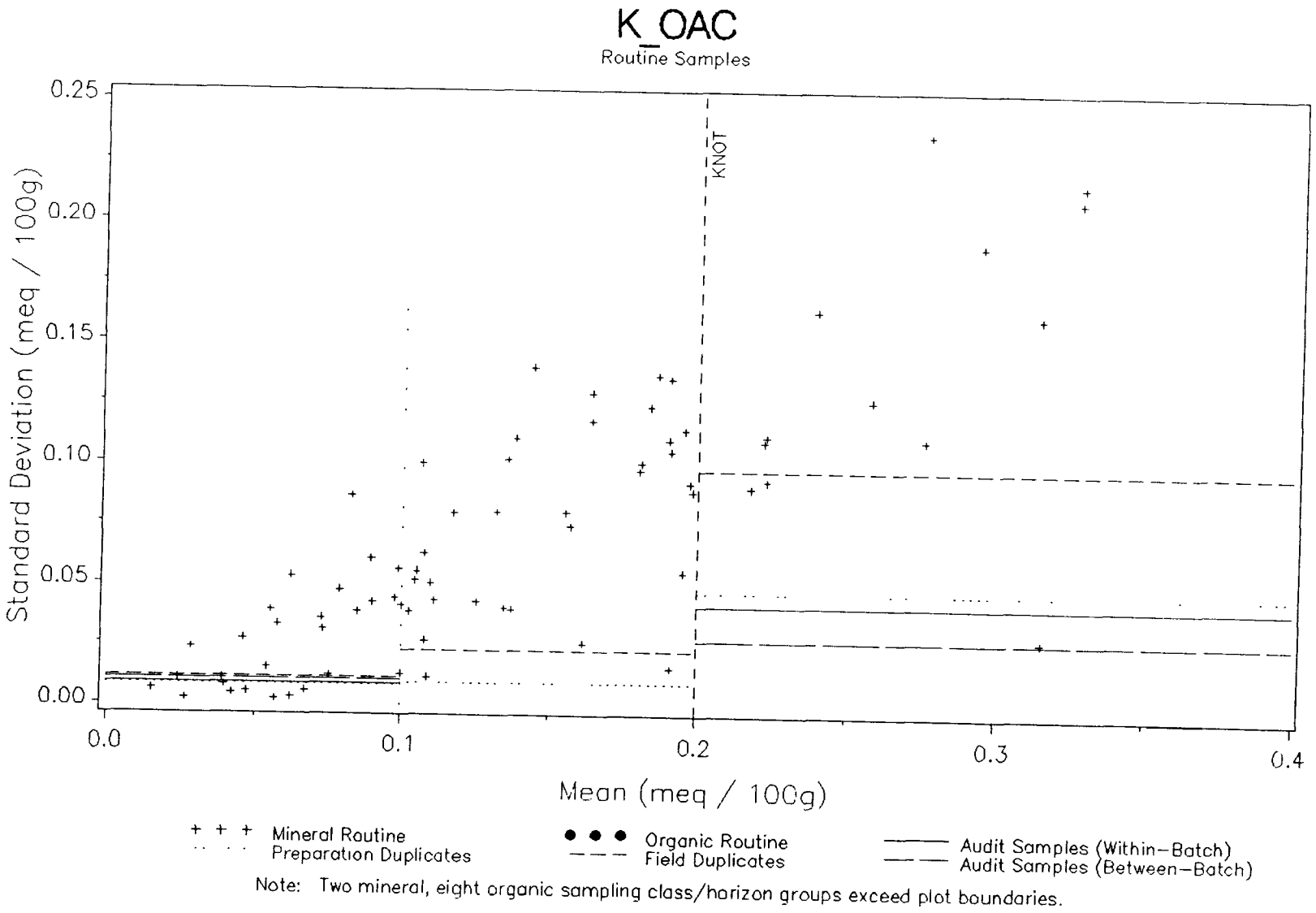


Figure 3-26. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for K_OAC.

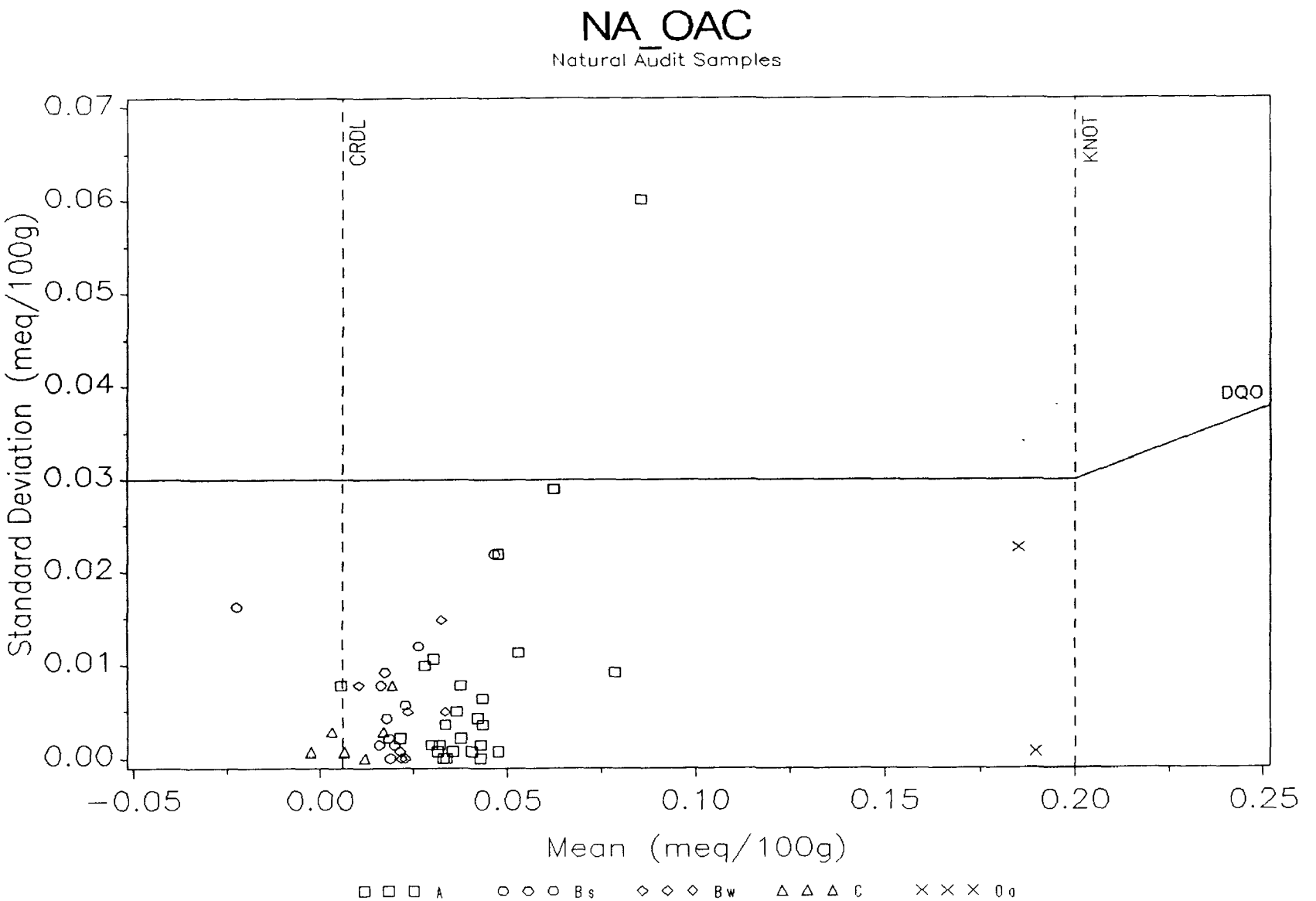


Figure 3-27. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for NA_OAC.

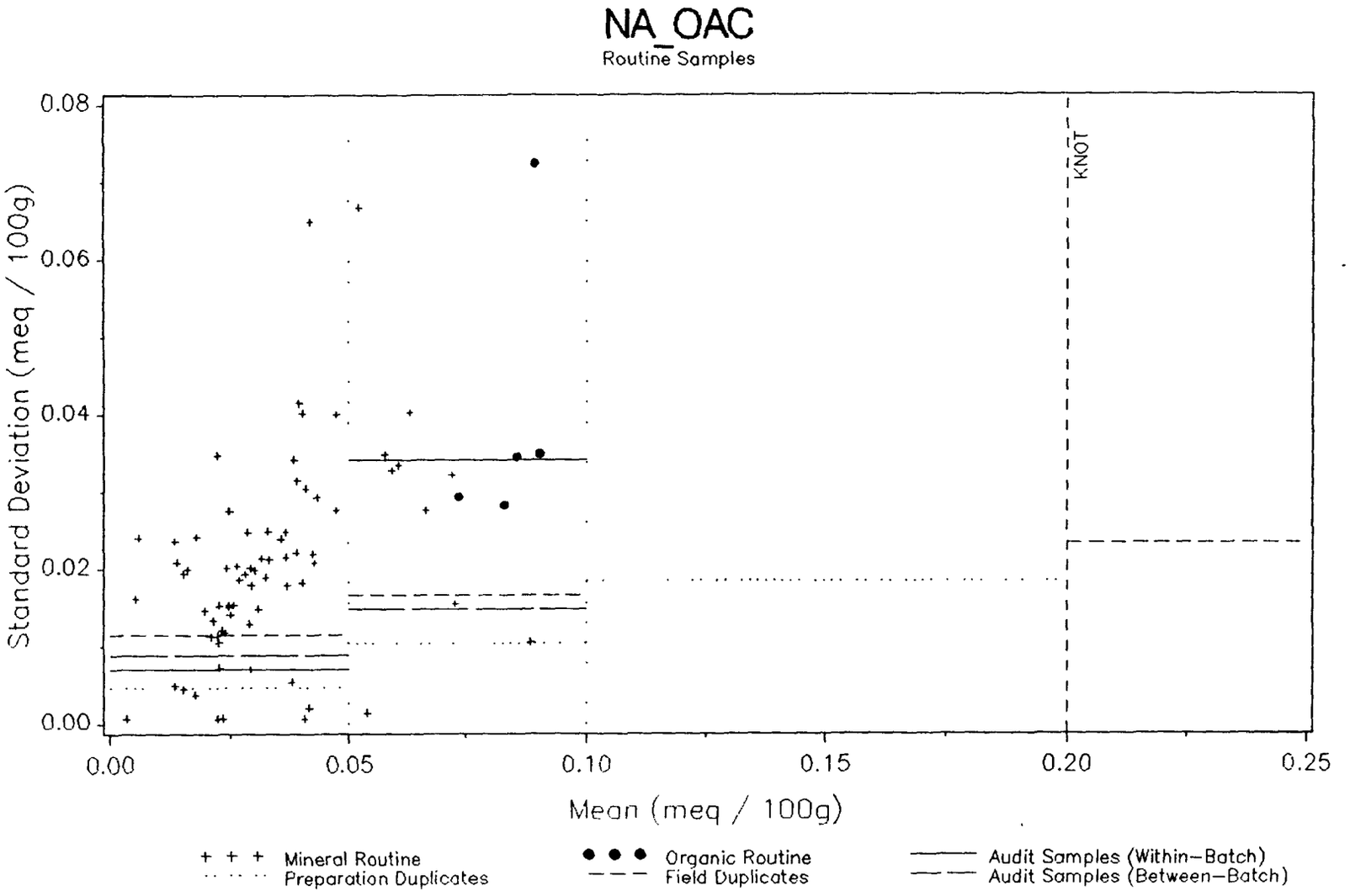


Figure 3-28. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for NA_OAC.

Cation Exchange Capacity and Exchangeable Acidity

Table 3-6
Figures 3-29 through 3-38

The CEC_CL parameter did not meet the DQO for analytical within-batch precision below the knot (see Table 3-6). The AC_BACL parameter was only slightly above the DQO for data below the knot. In all other cases the DQOs for this parameter group were satisfied. In most cases, the preparation duplicates and field duplicates also met the analytical DQOs, even though the samples were susceptible to additional confounded errors from sampling or preparation.

The estimated standard deviations for CEC_CL in the PD and S/H data sets, and CEC_OAC and AC_BACL in the FD and S/H data sets, have insufficient degrees of freedom to place confidence in these portions of the data.

Figures 3-29 through 3-38 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data points is presented in Appendix D.

Table 3-6. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of Cation Exchange Capacity and Exchangeable Acidity

Data set[a]	Parameter	Below the knot[b] df	SD	DQO	Pairs>DQO n	%	Above the knot[b] df	RSD	DQO	Pairs>DQO n	%
AS	CEC_CL	6	0.4028	0.25	2	33.3	44	8.9%	10%	6	13.6
	CEC_OAC	6	0.1333	0.25			44	7.1%	10%	5	11.4
	AC_KCL	12	0.1106	0.50			38	12.8%	20%	1	2.6
	AC_BACL	6	0.5059	0.50	3	50.0	44	10.4%	20%	2	4.5
	AL_KCL	14	0.1231	0.50			36	8.5%	20%	2	5.6
PD	CEC_CL	1	0.1676				25	13.0%		6	24.0
	CEC_OAC	.	.		.	.	26	9.5%		3	11.5
	AC_KCL	17	0.0635				9	10.8%		1	11.1
	AC_BACL	.	.		.	.	26	15.3%		7	26.9
	AL_KCL	17	0.1362				9	12.3%		1	11.1
FD	CEC_CL	6	0.3745		2	33.3	98	14.4%		30	30.6
	CEC_OAC	2	0.1115				102	15.6%		25	24.5
	AC_KCL	63	0.3770		4	6.3	41	15.2%		4	9.8
	AC_BACL	3	0.3227				101	33.1%		22	21.8
	AL_KCL	73	0.2498		3	4.1	30	11.3%		3	10.0
S/H	CEC_CL	1	0.2475				608	51.2%			
	CEC_OAC	1	0.1838				608	52.1%			
	AC_KCL	402	1.0775				207	71.3%			
	AC_BACL	1	0.1768				608	64.9%			
	AL_KCL	412	1.0008				197	77.6%			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.
[b] Standard deviation and RSD data in reporting units and percent, respectively, for mineral soil samples below and above the knot point of 0.20 meq/100g; a dot signifies a lack of data occupying that range.

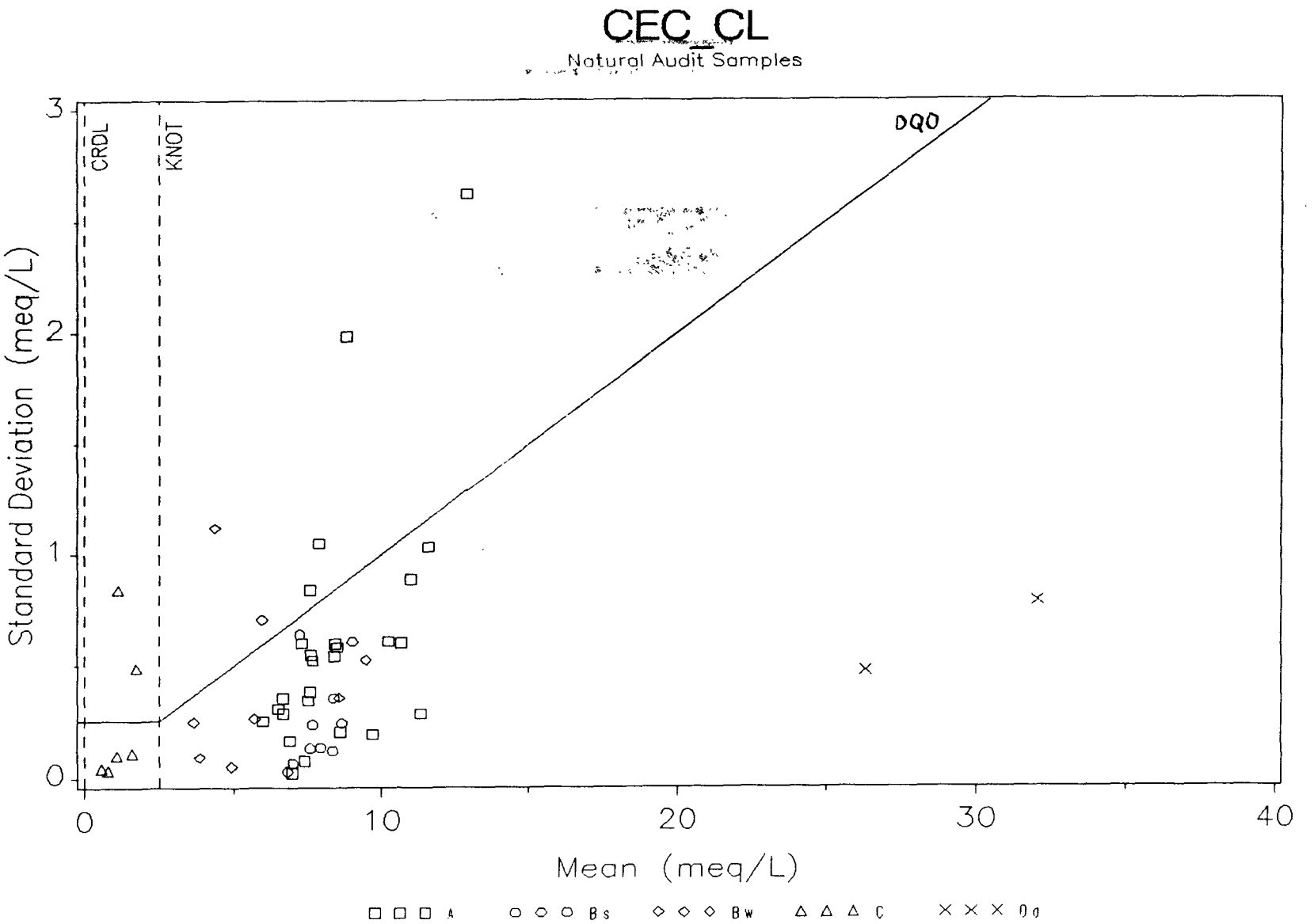
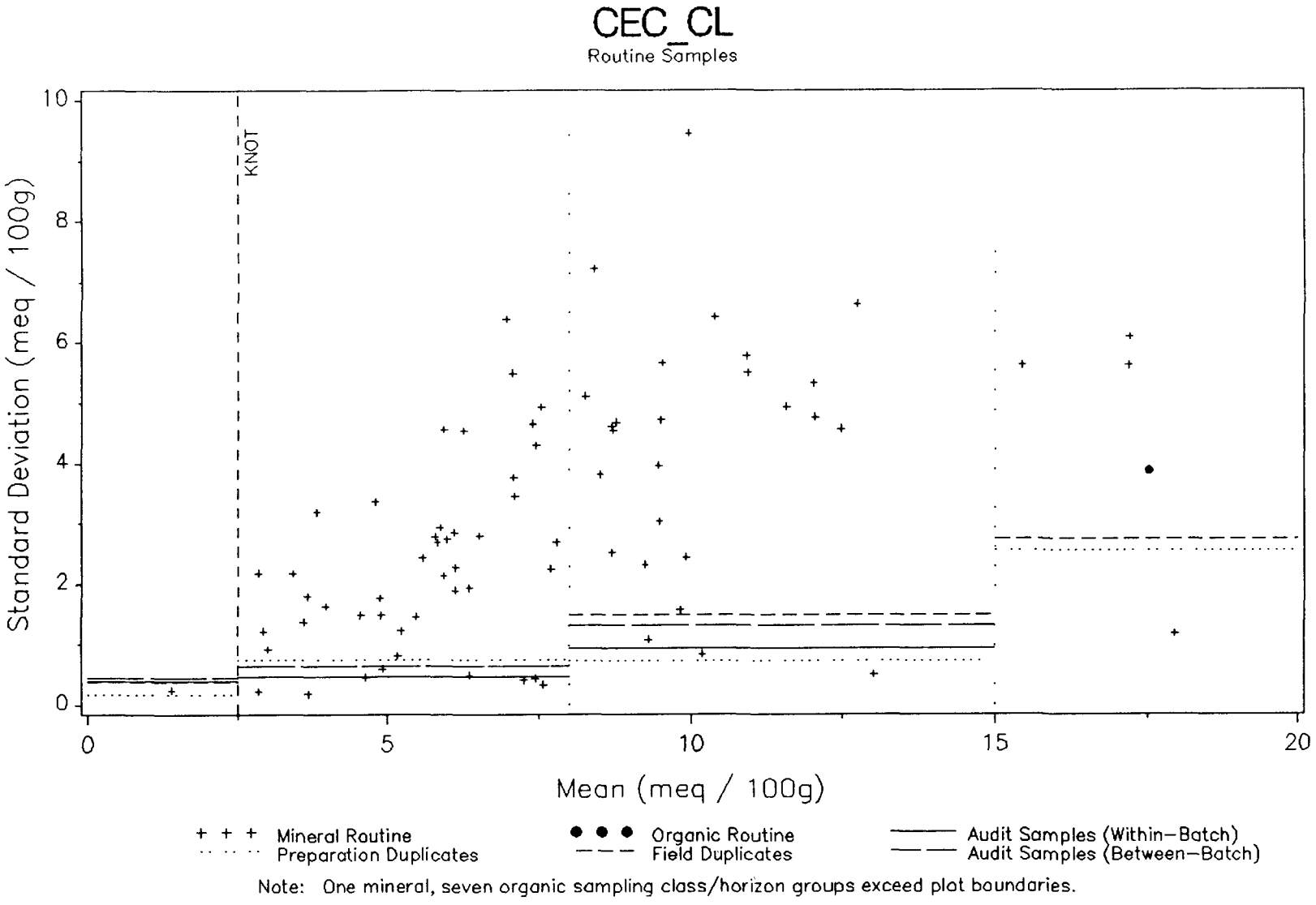


Figure 3-29. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for CEC_CL



Note: One mineral, seven organic sampling class/horizon groups exceed plot boundaries.

Figure 3-30. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for CEC_CL.

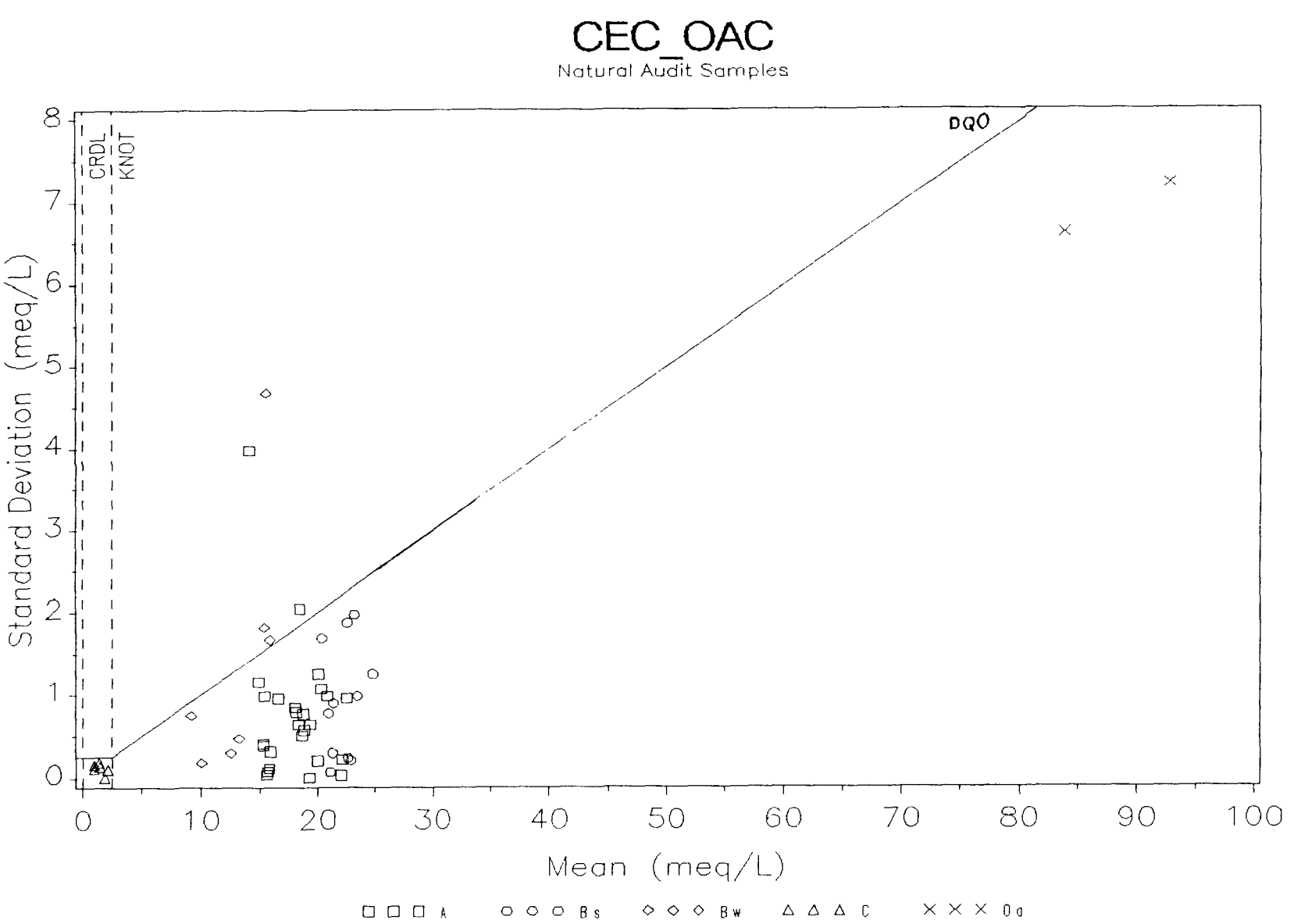


Figure 3-31. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for CEC_OAC.

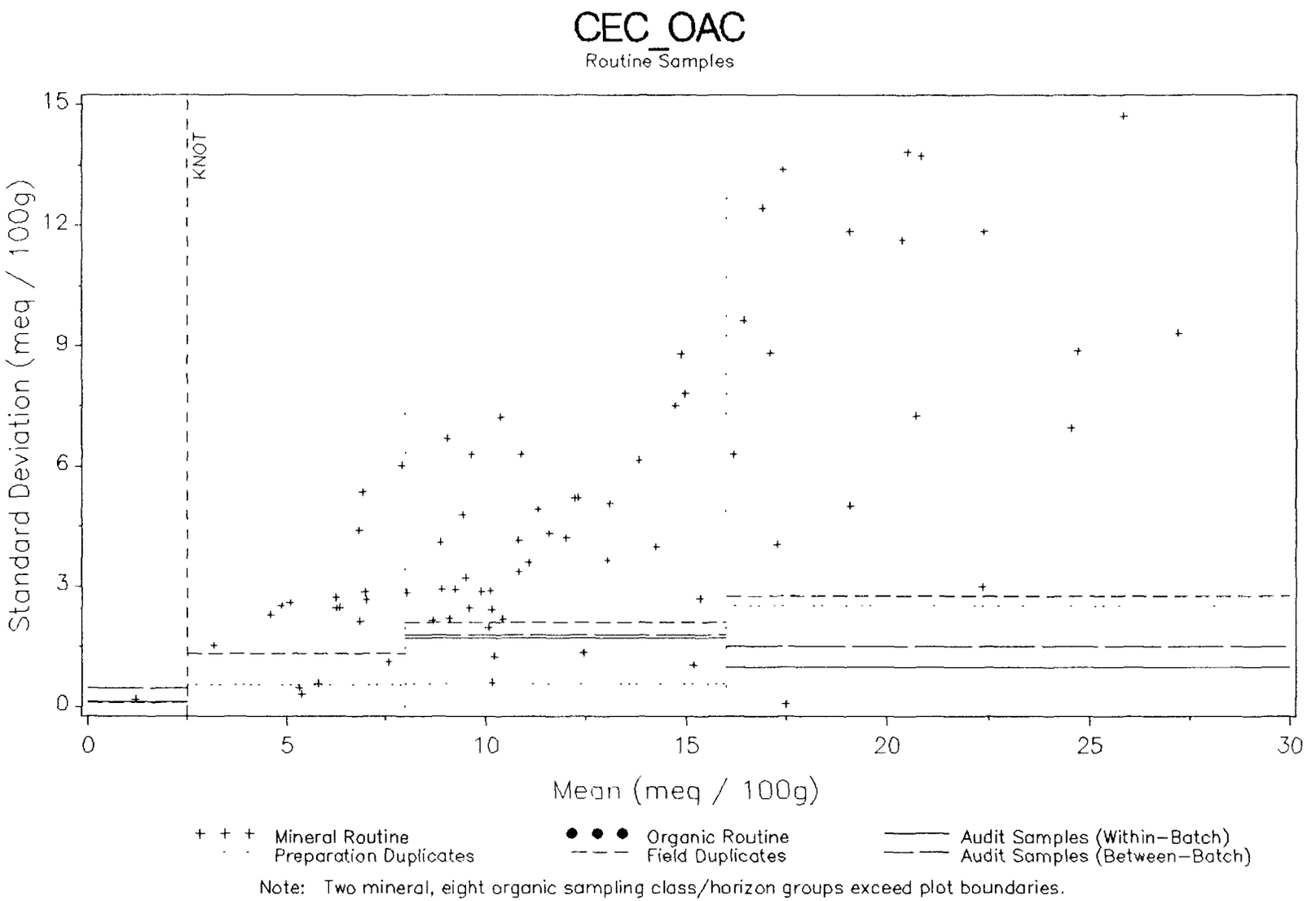


Figure 3-32. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for CEC_OAC.

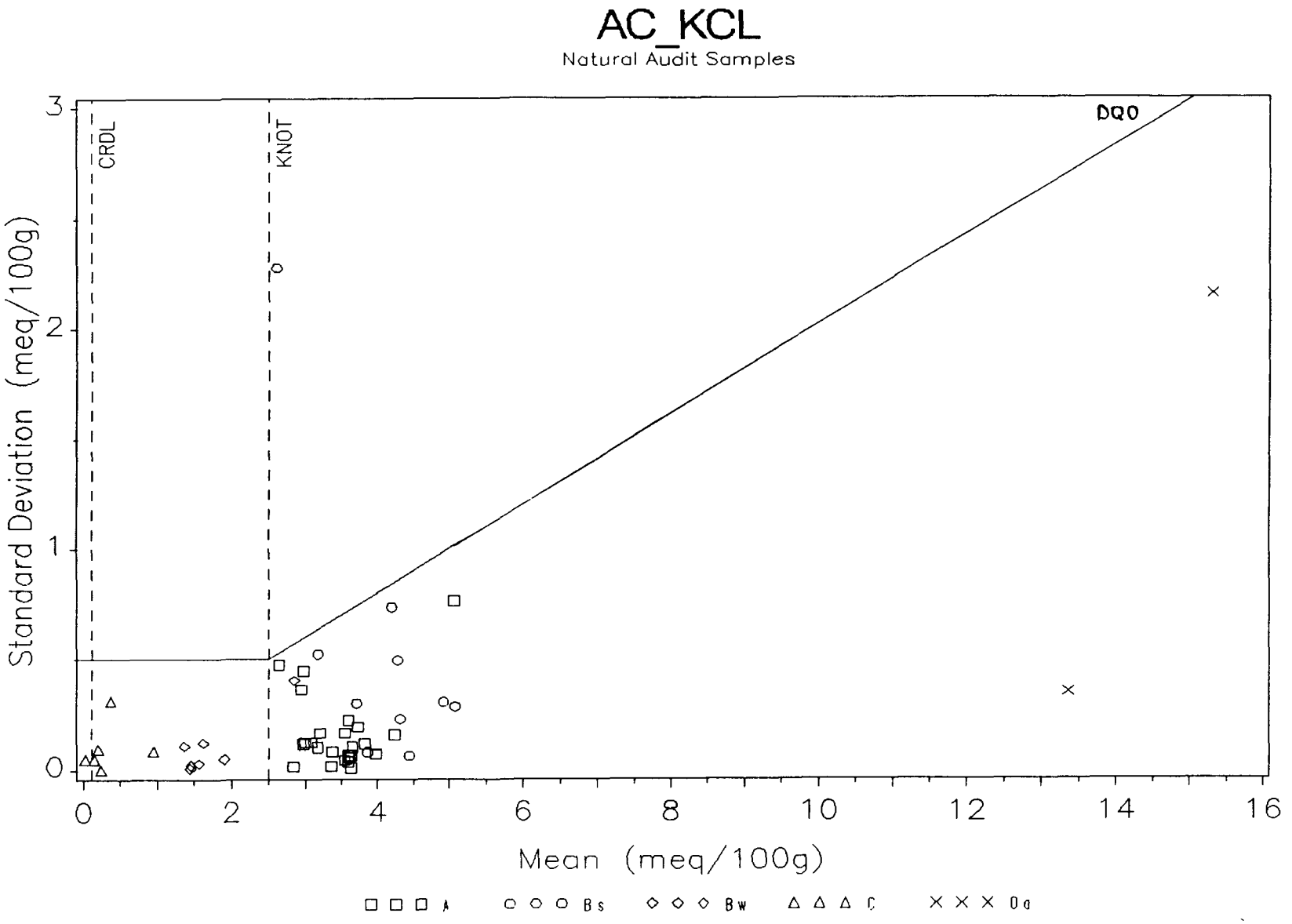
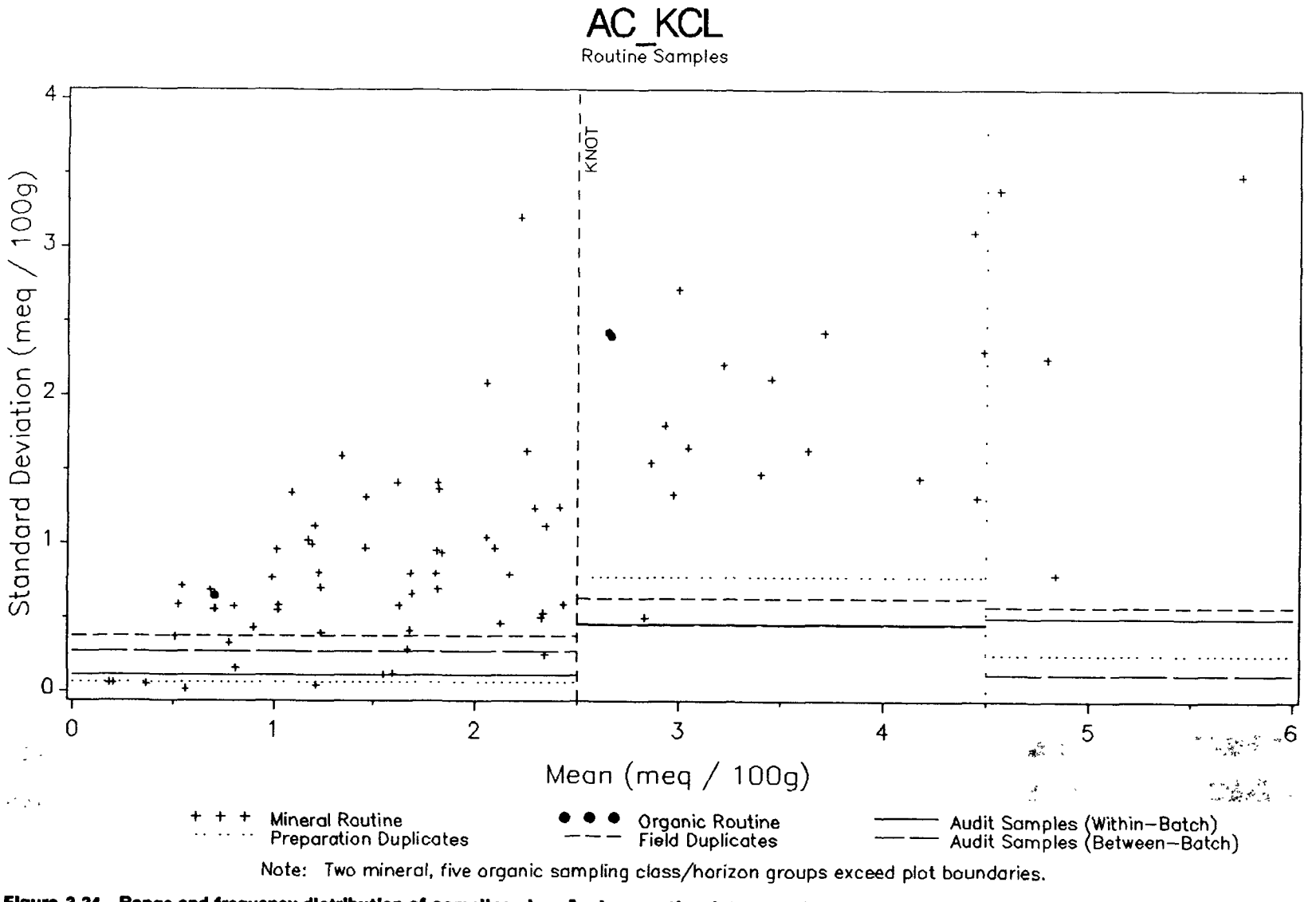


Figure 3-33. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AC_KCL.



Note: Two mineral, five organic sampling class/horizon groups exceed plot boundaries.

Figure 3-34. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AC_KCL.

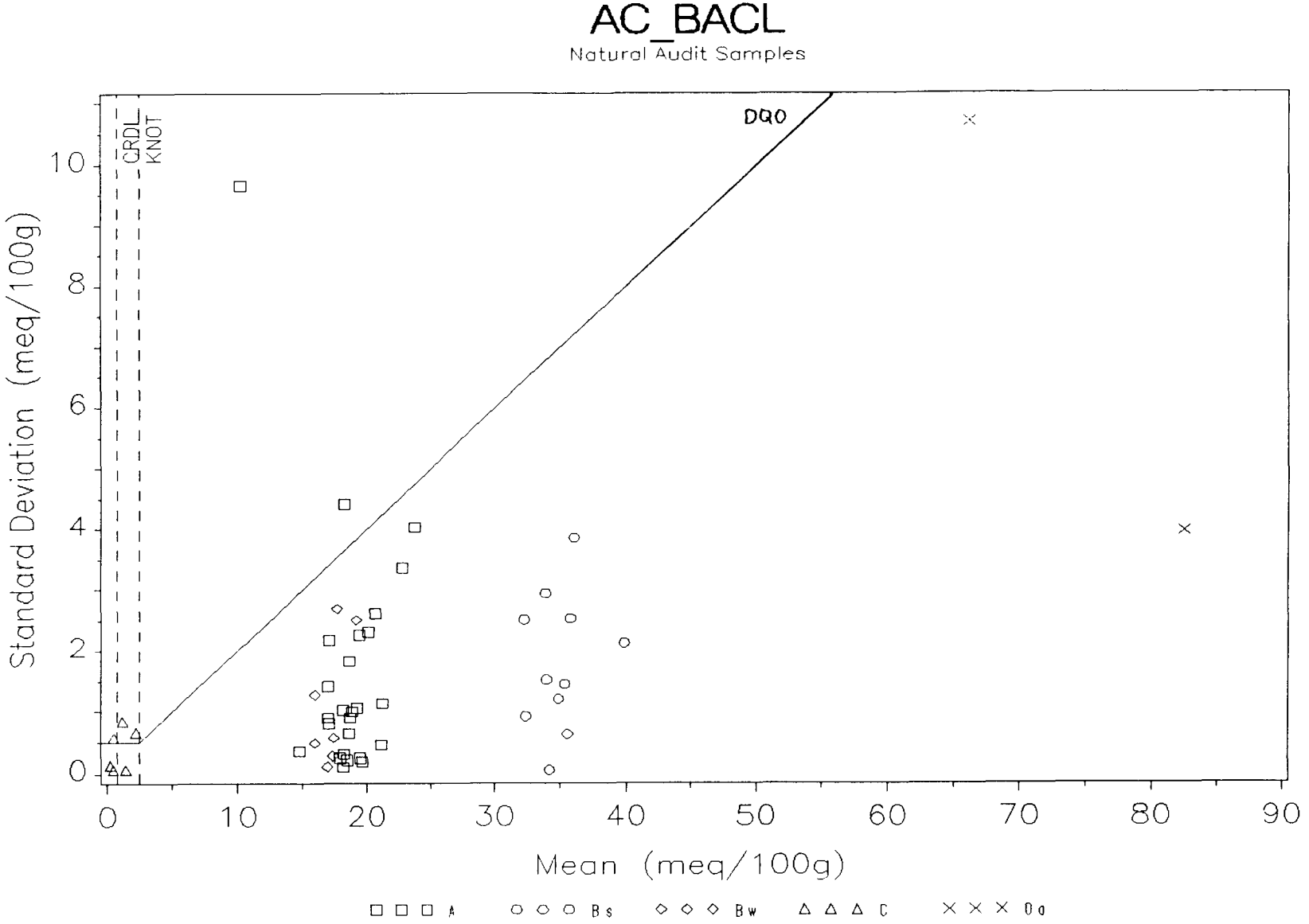


Figure 3-35. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AC_BACL.

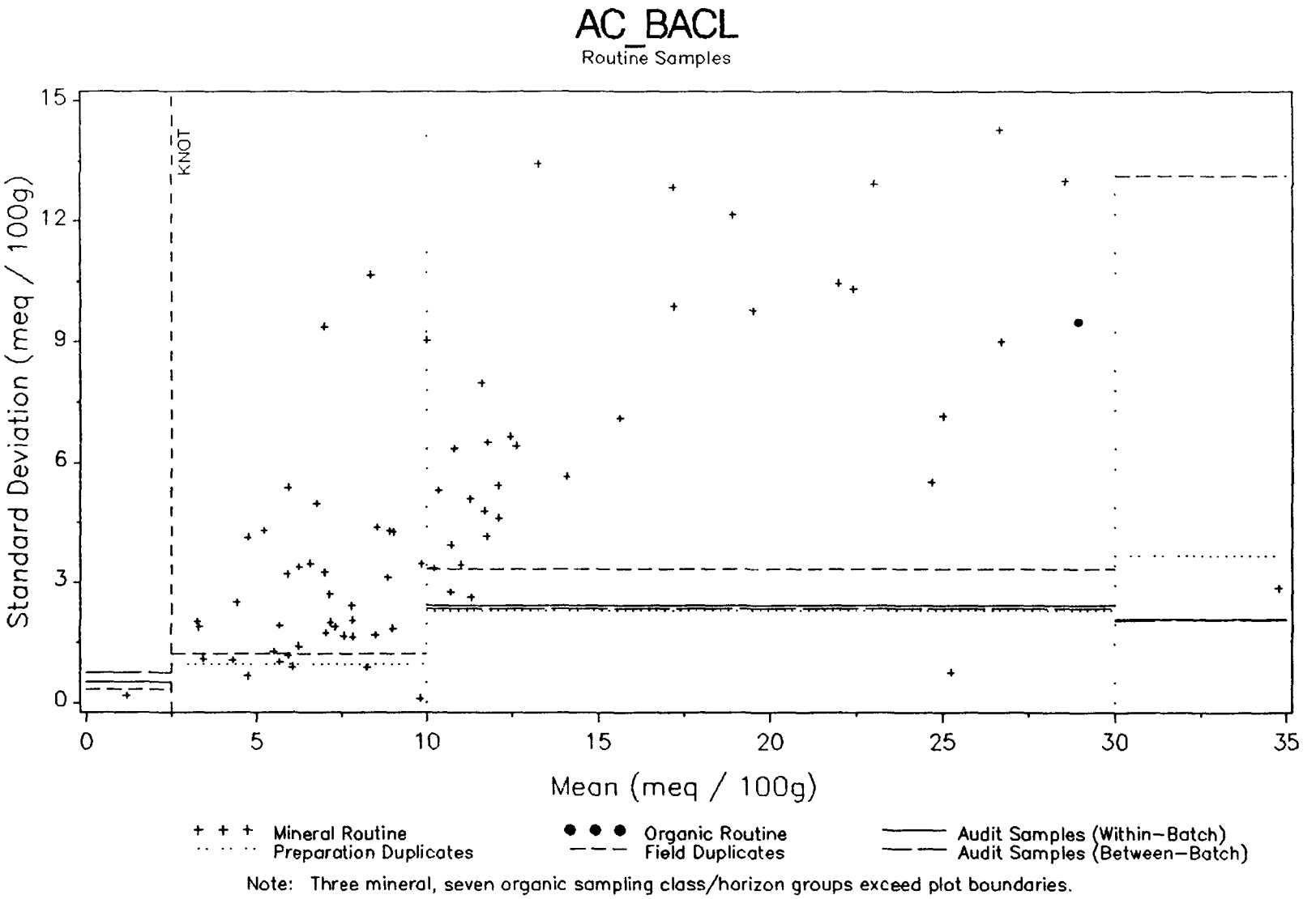


Figure 3-36. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AC_BACL.

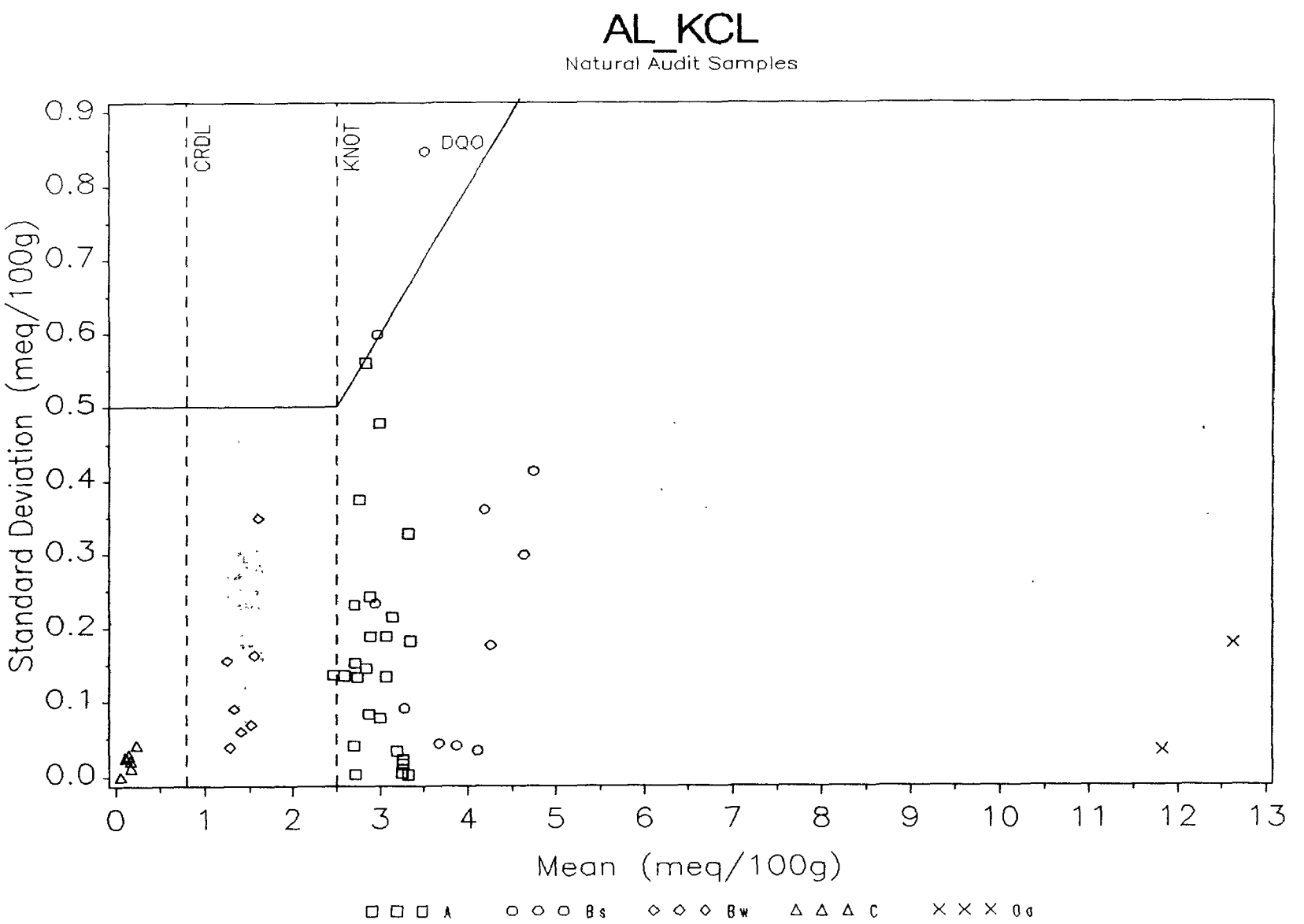


Figure 3-37. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AL_KCL.

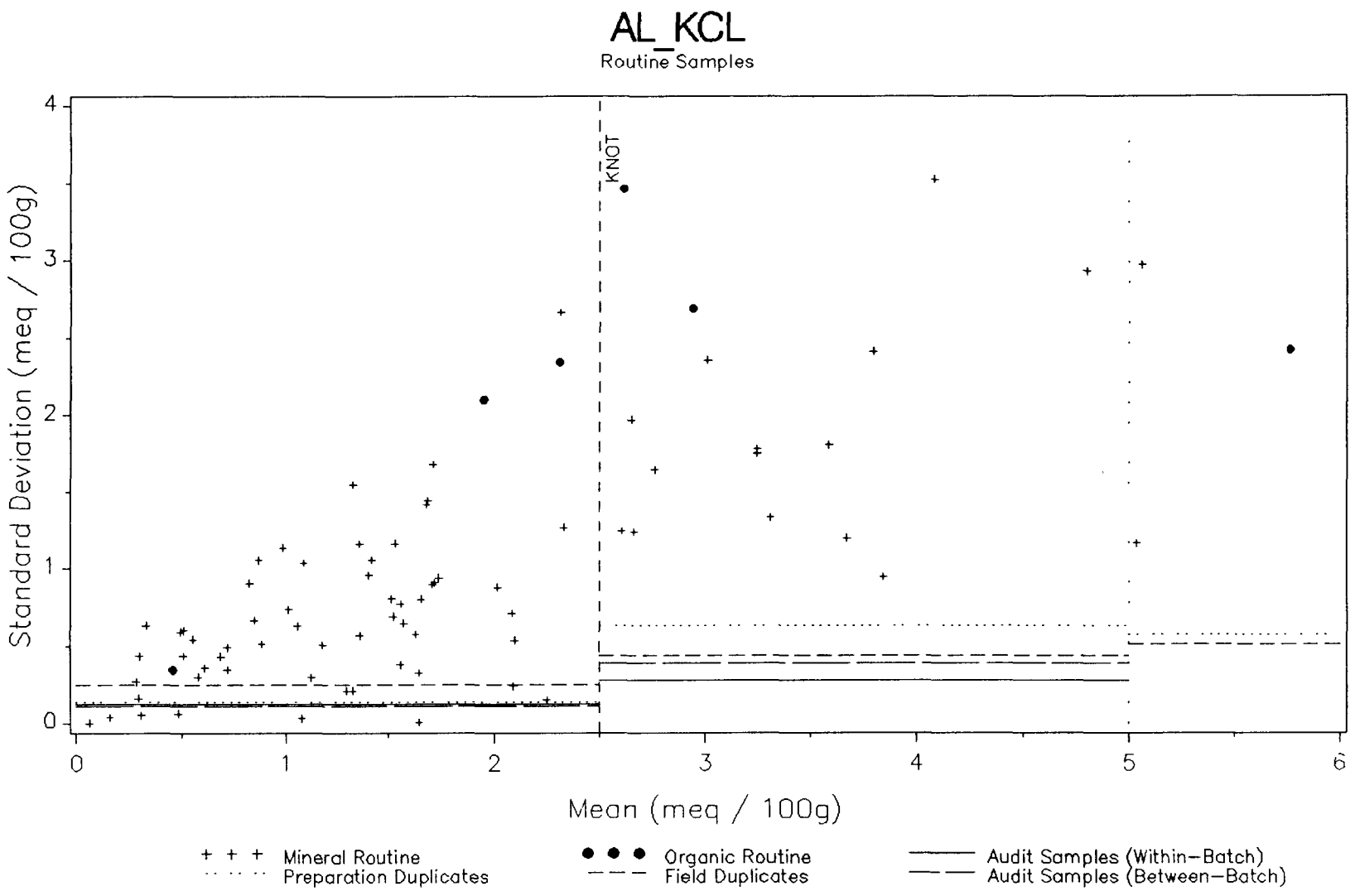


Figure 3-38. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AL_KCL

Extractable Cations in Calcium Chloride

Table 3-7
Figures 3-39 through 3-50

Of the six extractable cations in calcium chloride, the analytical within-batch precision DQO was satisfied only for MG_CL2 (see Table 3-7). The RSD for AL_CL2 only slightly exceeded the DQO, while the RSD for the remaining cations were from 1.2 to 2 times higher than the DQO. It appears that the single-tiered DQO for this parameter group was generally inappropriate and unattainable, as there was no contingency made for a lower-tier DQO to accomodate low analyte concentrations. Indeed, the majority of the routine data for these parameters was distributed in the extremely low zone of concentration near the detection limit. For example, the FE_CL2 concentrations were so low that, after correction for blank analysis, many of the data showed up as negative values. This was the case for 17 of the 26 preparation duplicates and 60 of the 104 field duplicates, as seen in the high RSD value for the FD data set.

Figures 3-39 through 3-50 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Table 3-7. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of the Extractable Cations in Calcium Chloride

Data set[a]	Parameter	df	RSD[b]	DQO[b]	Pairs>DQO n	Pairs>DQO %
AS	CA_CL2	50	18.4%	5%	24	48.0
	MG_CL2	50	9.8%	10%	13	26.0
	K_CL2	50	12.3%	10%	16	32.0
	NA_CL2	50	20.5%	10%	17	34.0
	FE_CL2	42	17.2%	10%	12	28.6
	AL_CL2	49	10.8%	10%	24	49.0
PD	CA_CL2	26	5.4%		8	30.8
	MG_CL2	26	8.7%		8	30.8
	K_CL2	26	12.8%		13	50.0
	NA_CL2	26	12.0%		12	46.2
	FE_CL2	9	36.5%		4	44.4
	AL_CL2	24	67.8%		14	58.3
FD	CA_CL2	104	41.1%		40	38.5
	MG_CL2	104	52.7%		40	38.5
	K_CL2	104	92.8%		61	58.6
	NA_CL2	104	34.5%		72	69.2
	FE_CL2	44	496.7%		24	53.3
	AL_CL2	87	94.6%		63	72.4
S/H	CA_CL2	609	40.7%			
	MG_CL2	609	64.1%			
	K_CL2	609	81.0%			
	NA_CL2	609	274.6%			
	FE_CL2	543	658.9%			
	AL_CL2	602	138.0%			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.
[b] Data reported as %RSD.

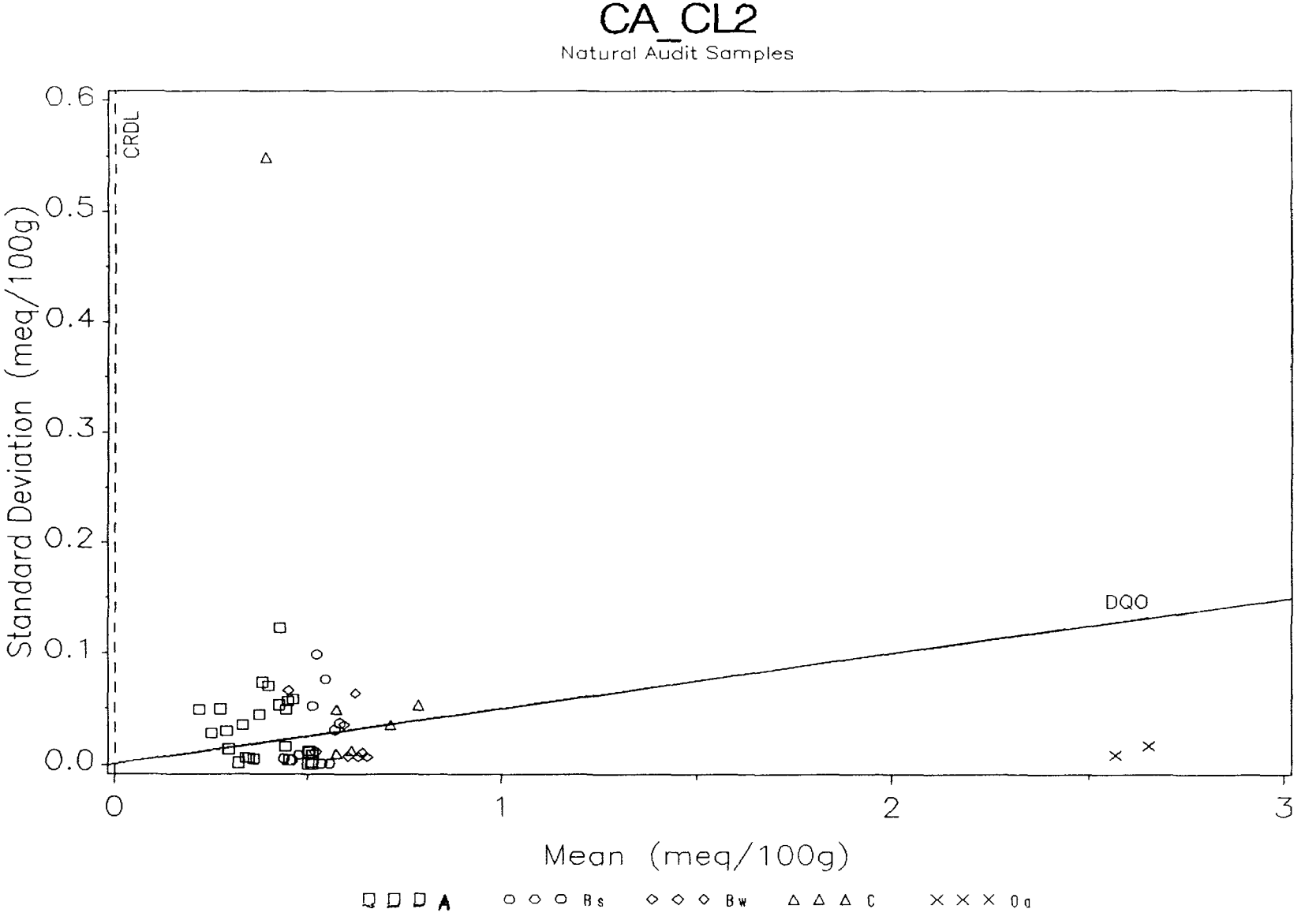


Figure 3-39. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for CA_CL2.

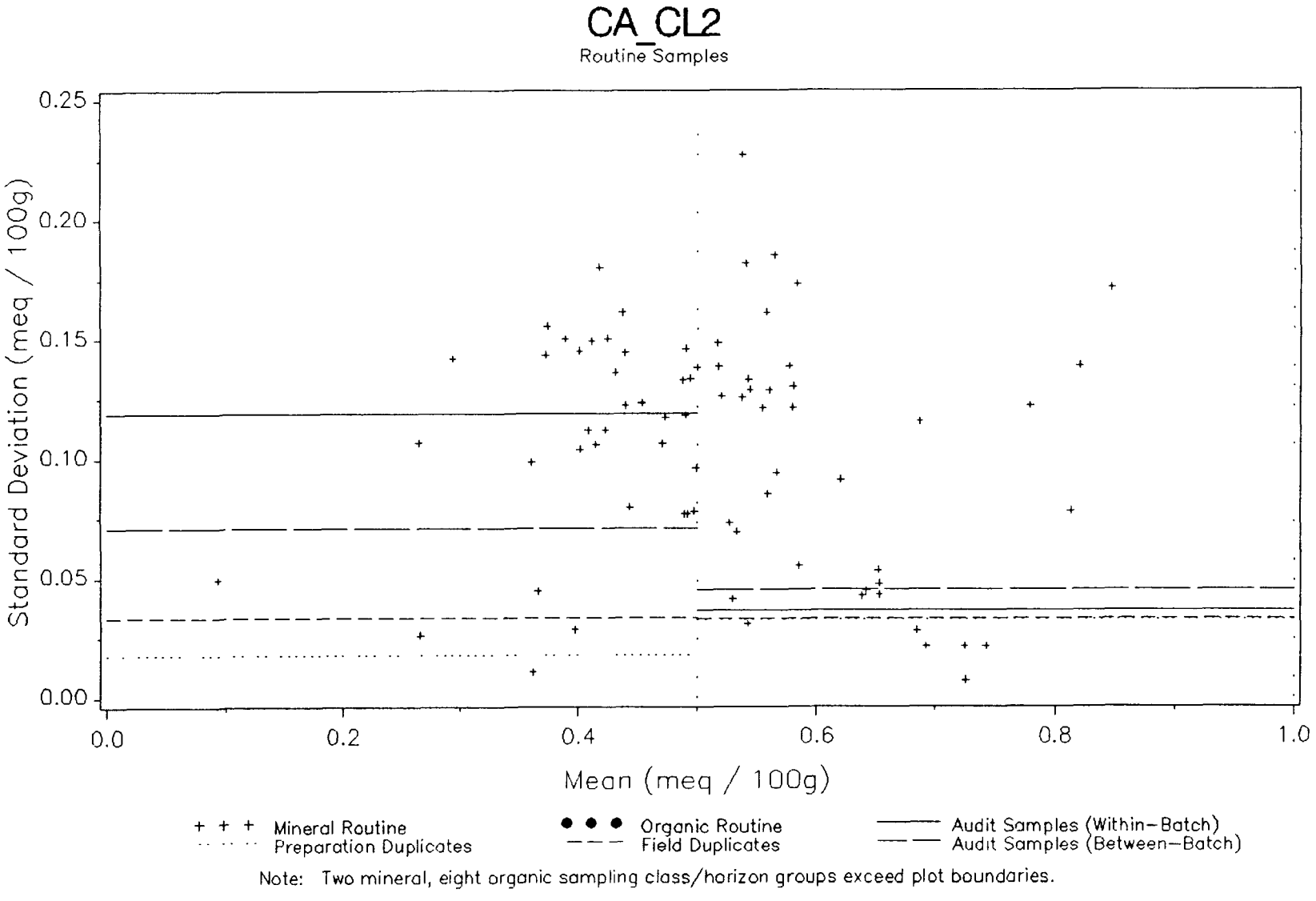


Figure 3-40. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for CA_CL2.

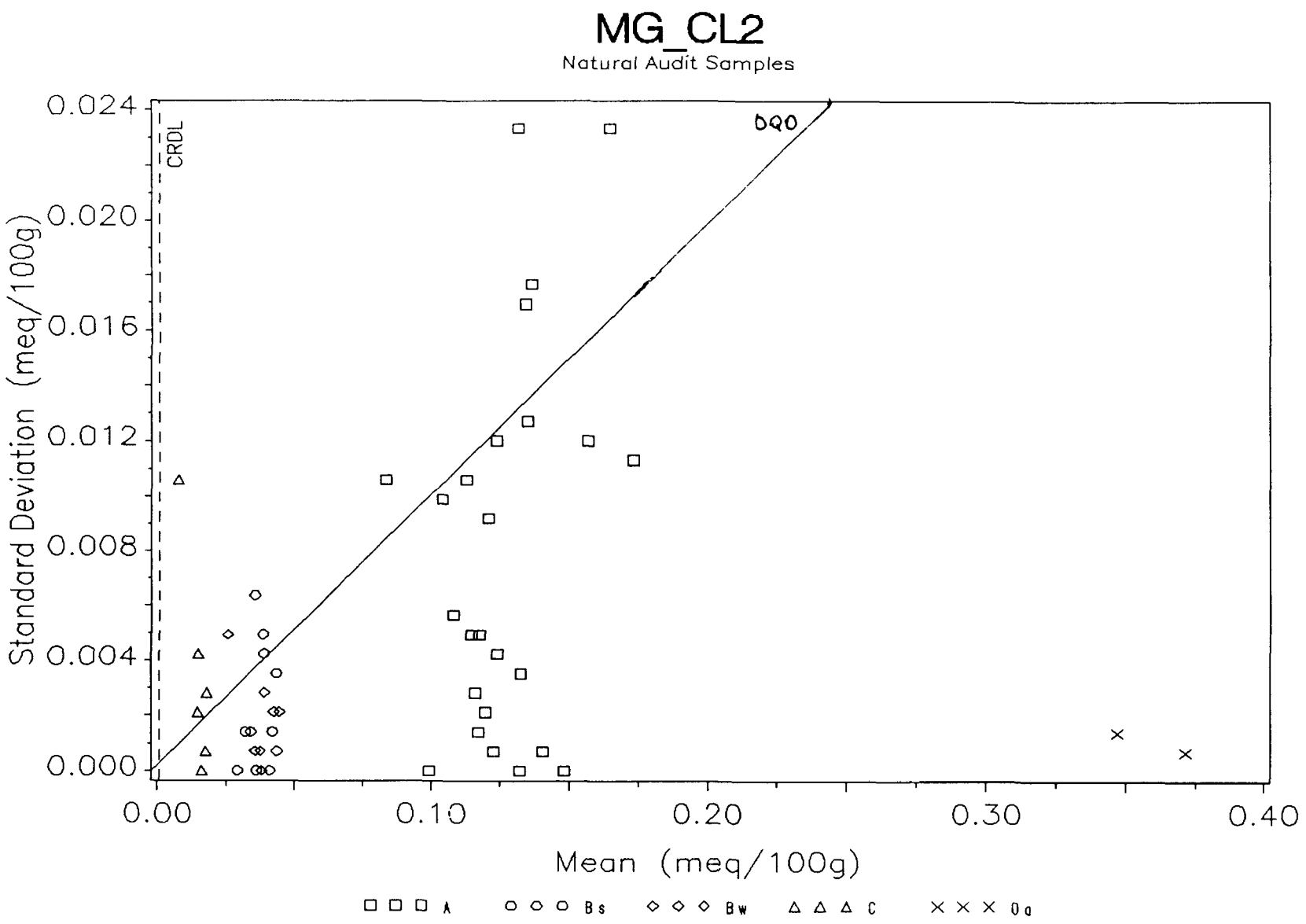


Figure 3-41. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for MG_CL2.

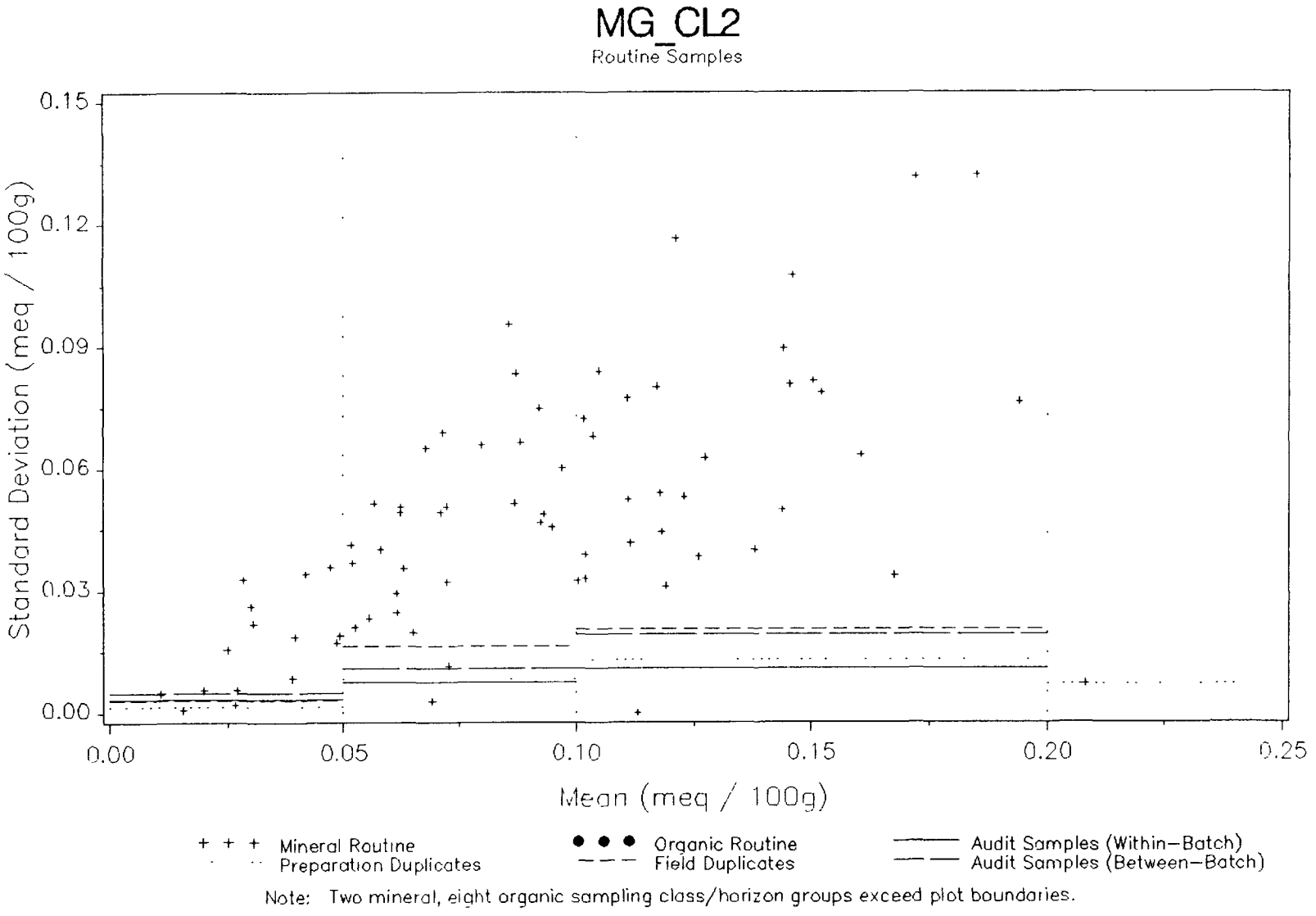


Figure 3-42. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for MG_CL2.

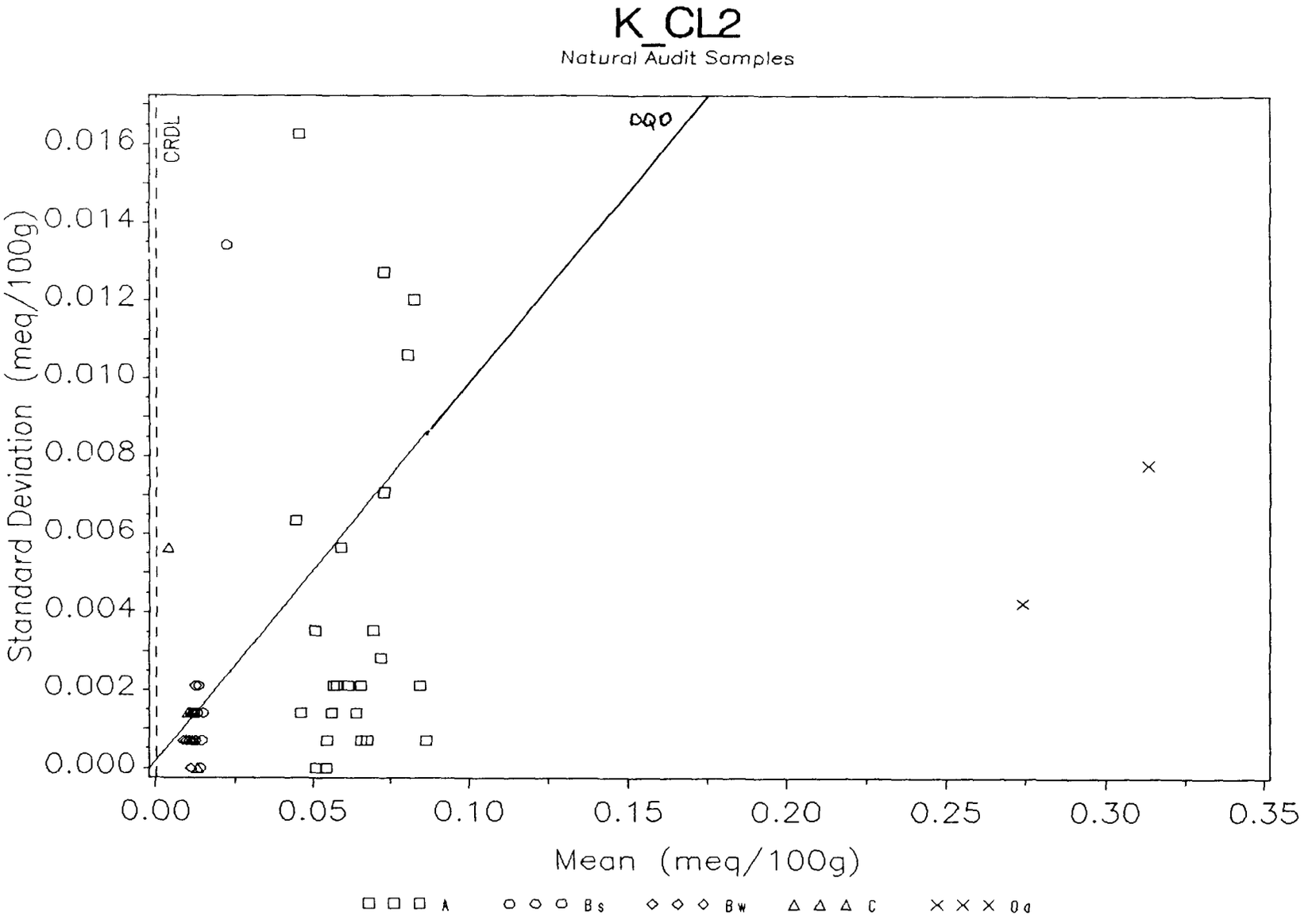


Figure 3-43. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for K_CL2.

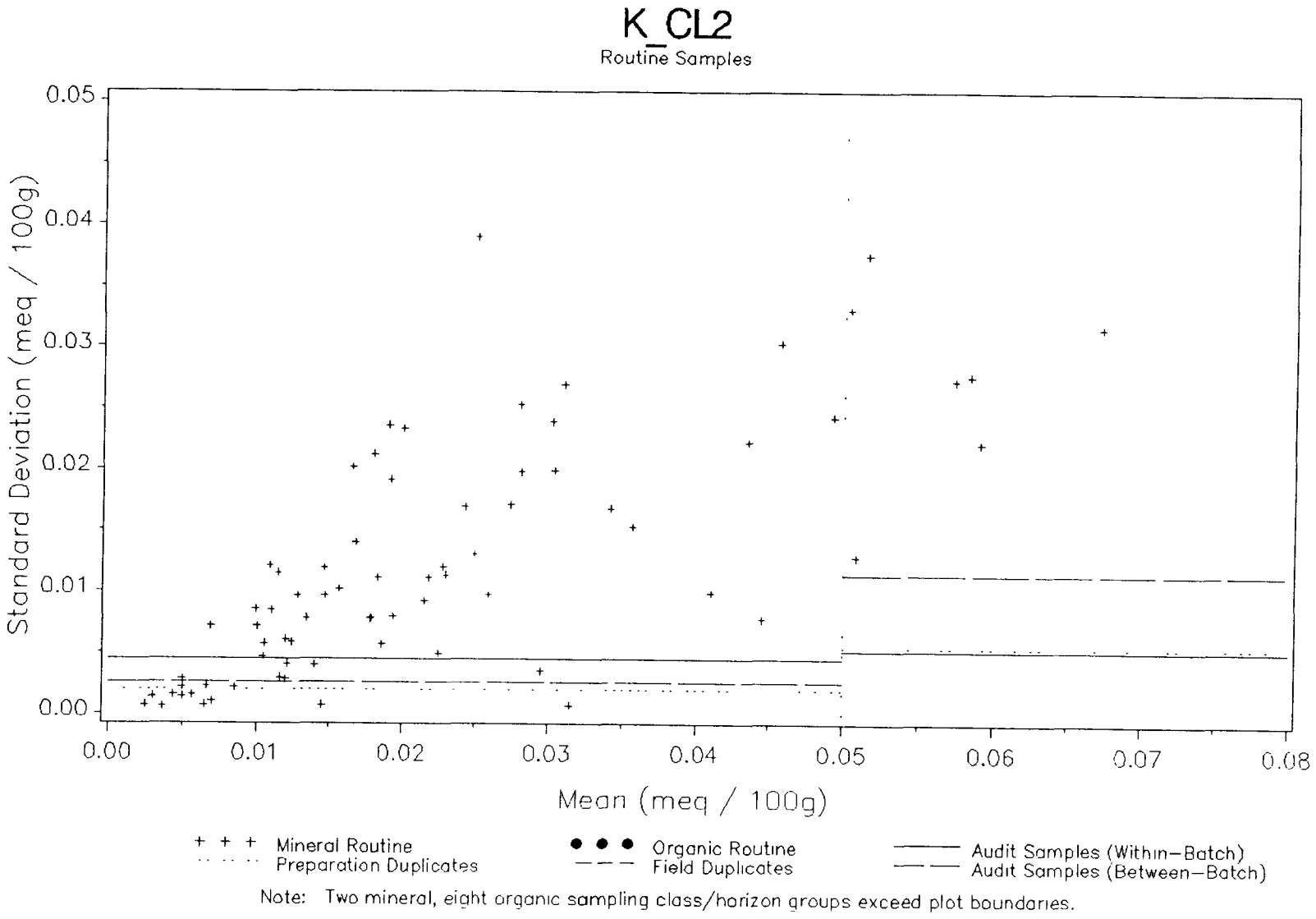


Figure 3-44. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for K_CL2.

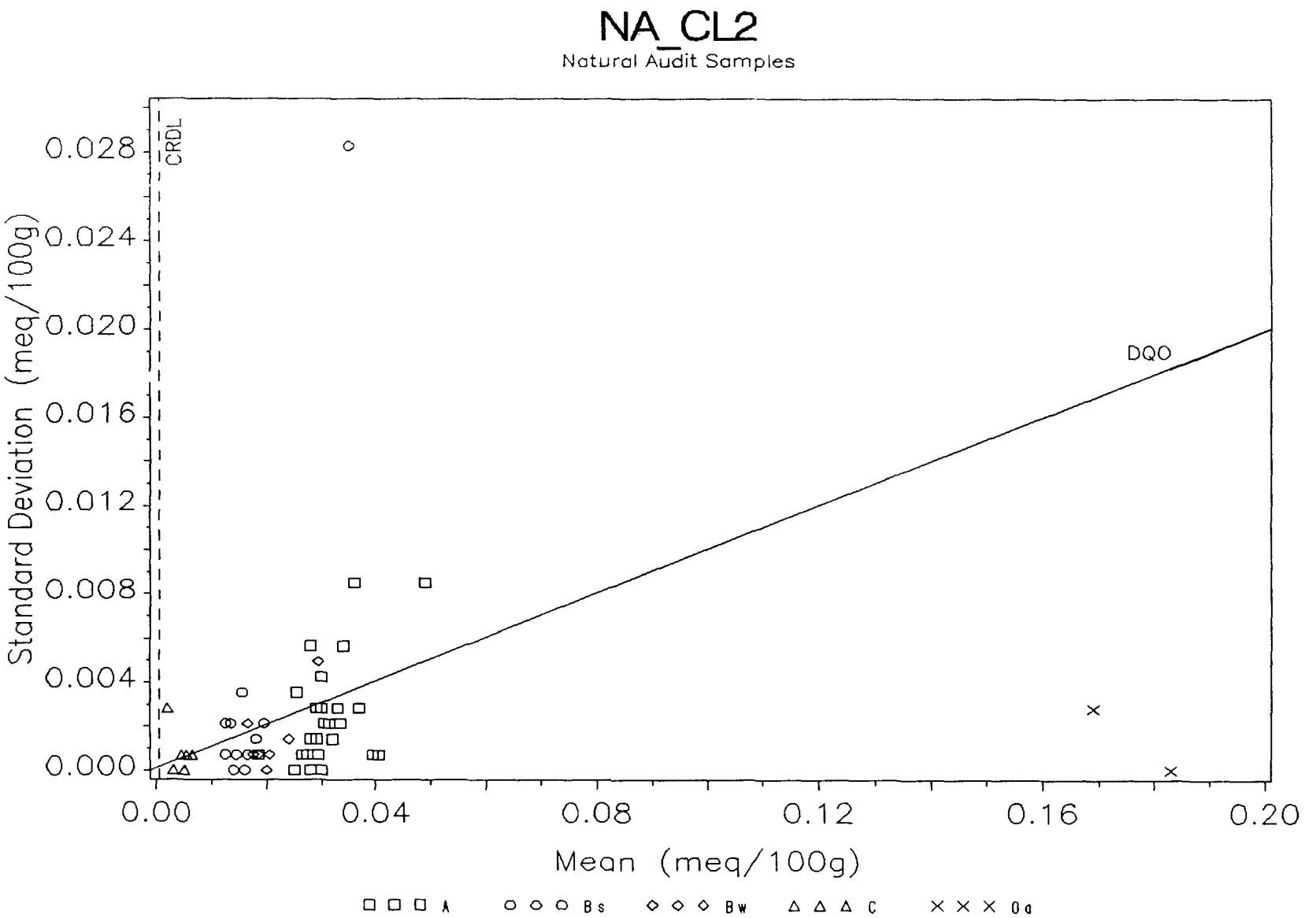


Figure 3-45. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for NA_CL2.

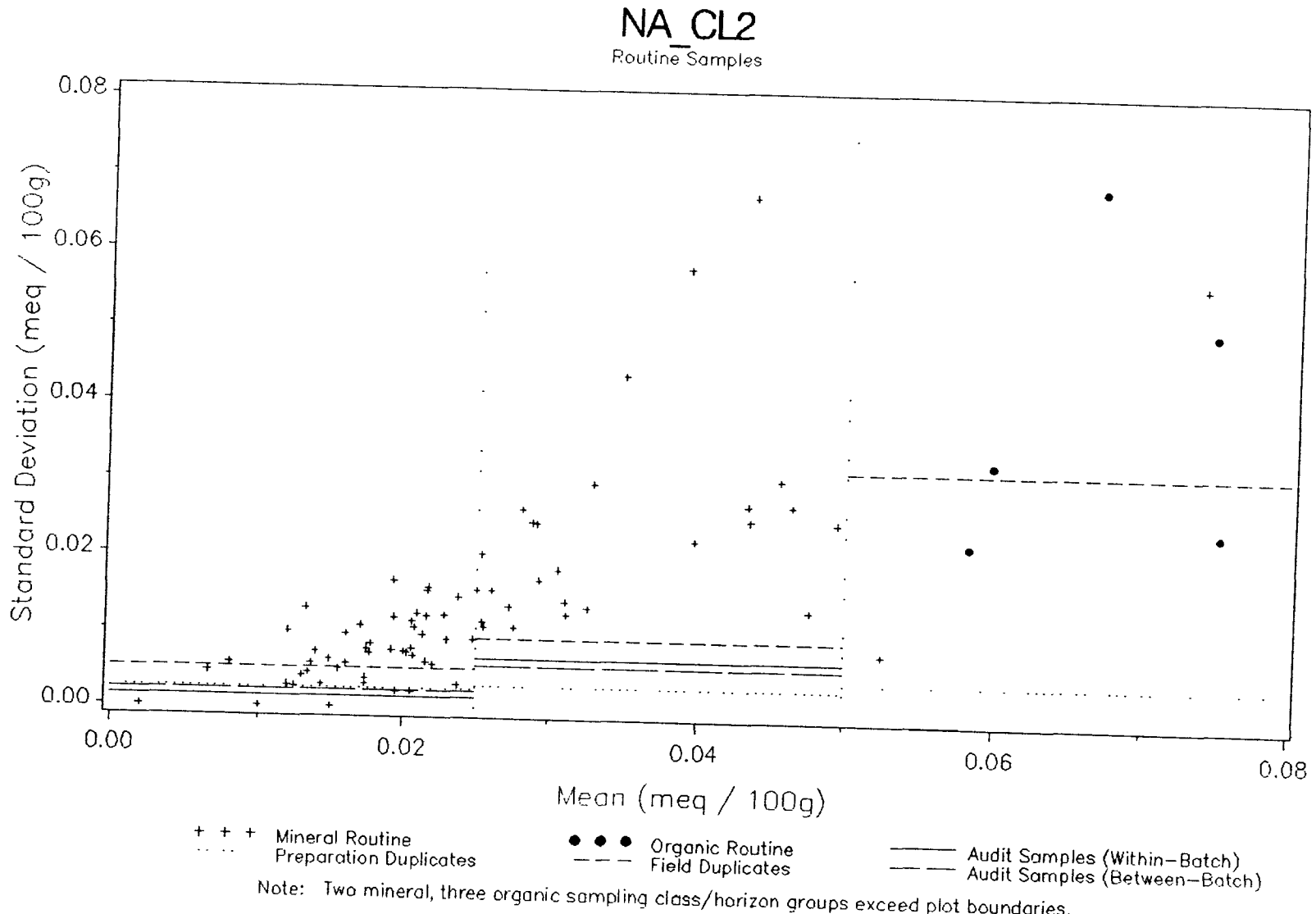


Figure 3-46. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for NA_CL2.

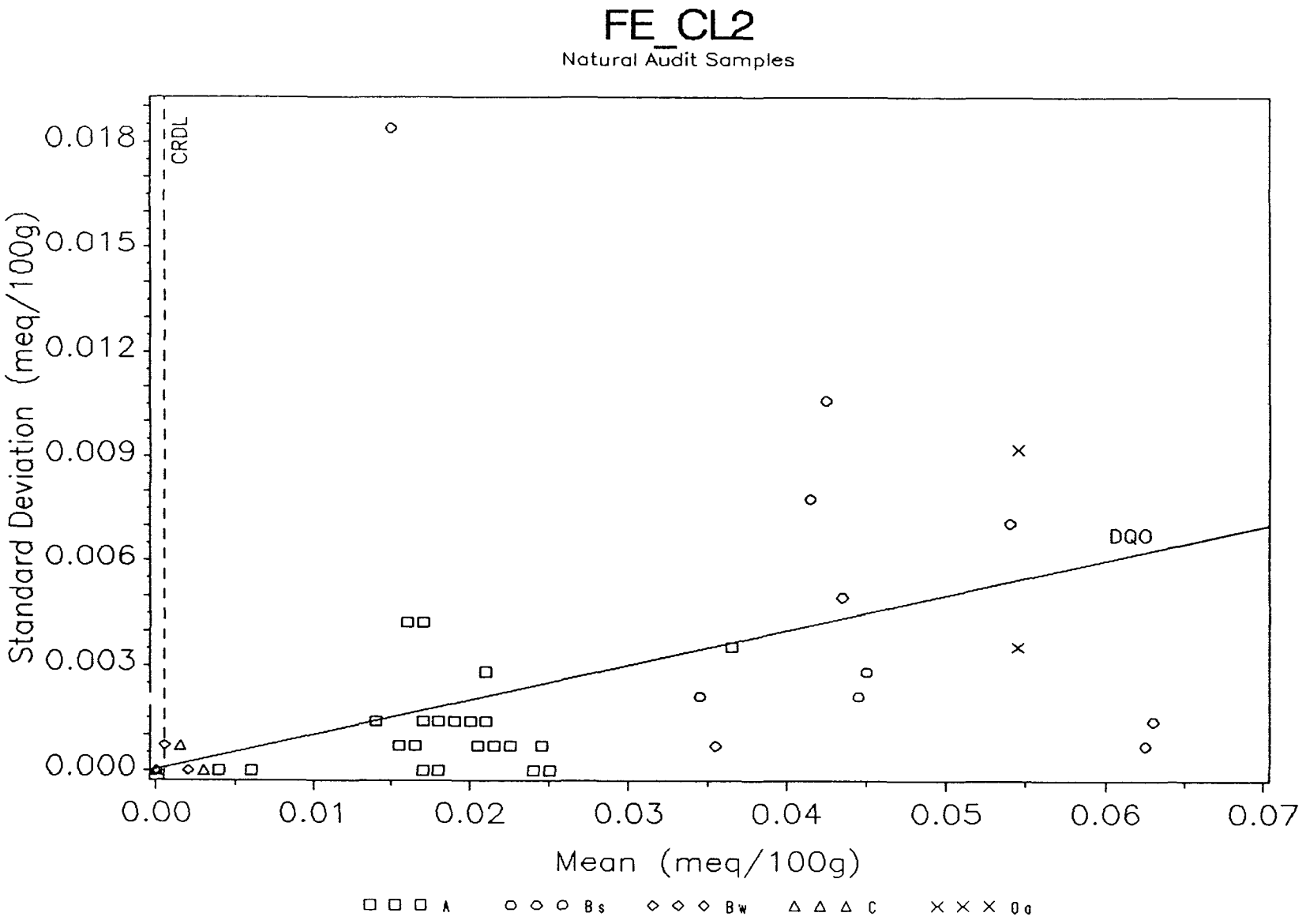


Figure 3-47. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for FE_CL2.

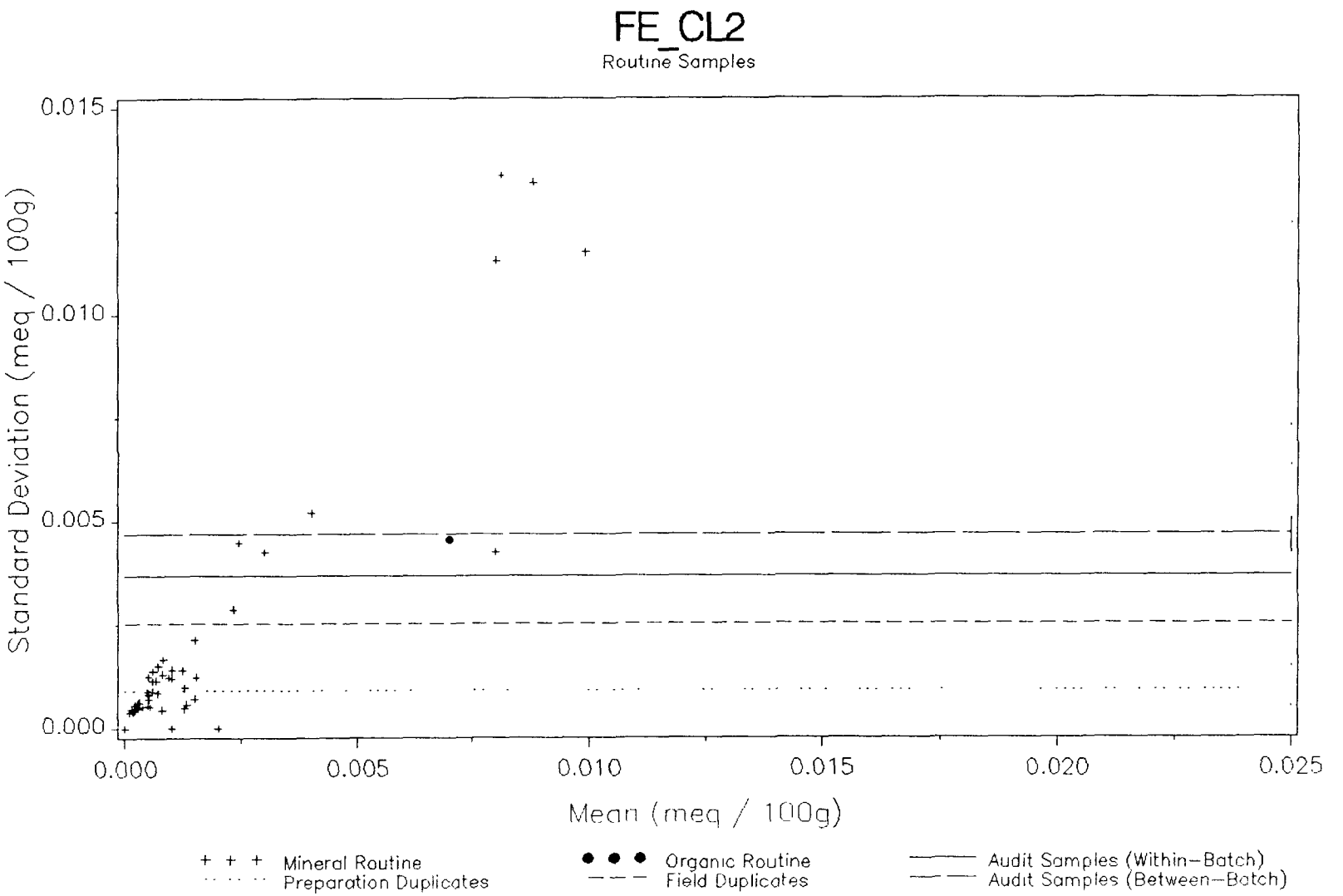


Figure 3-48. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for FE_CL2.

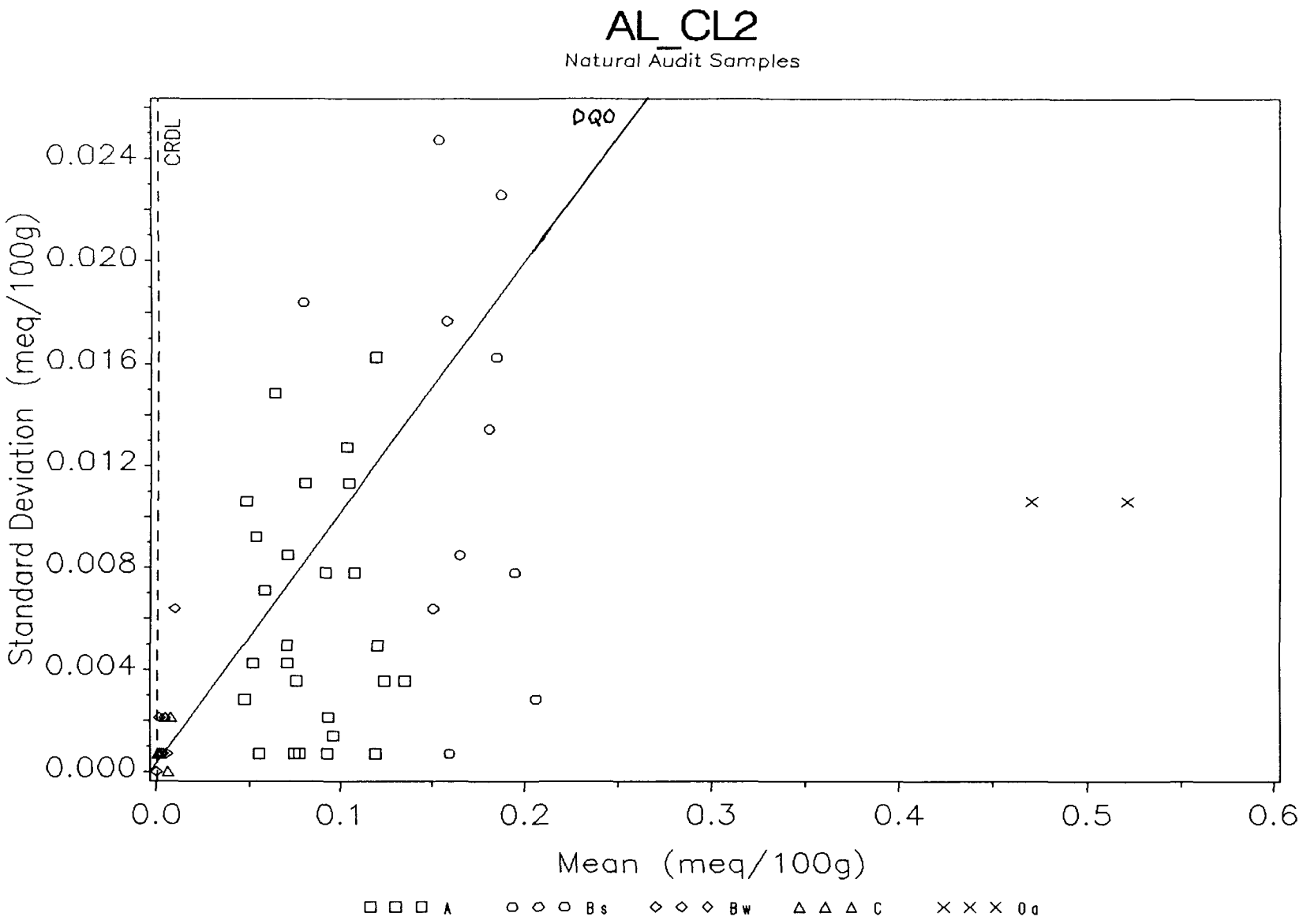


Figure 3-49. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AL_CL2.

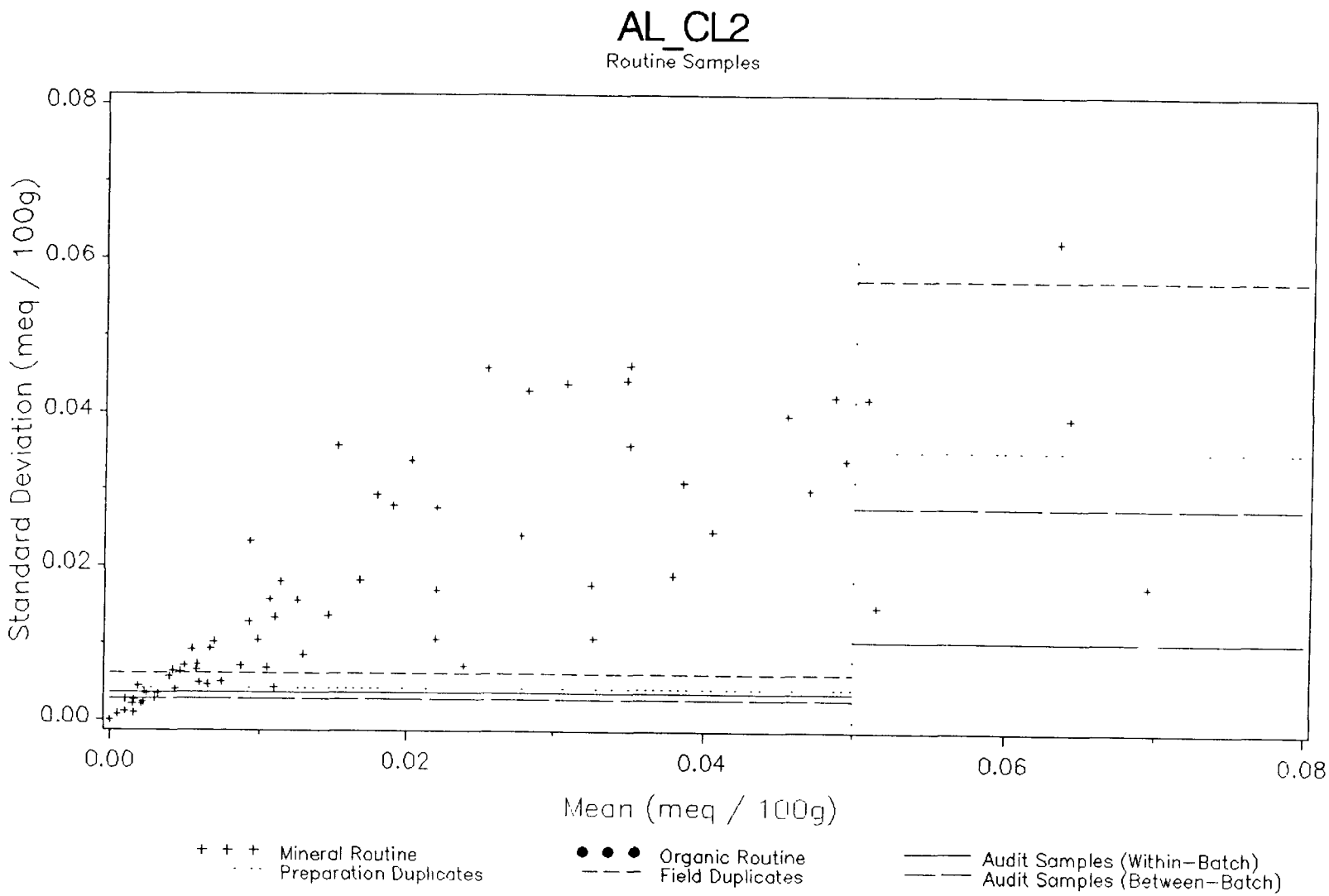


Figure 3-50. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AL_CL2.

Extractable Iron and Aluminum

Table 3-8, Figures 3-51 through 3-62

The analytical within-batch precision DQO for the six extractable iron and aluminum parameters was satisfied except for the FE_AO concentrations below the knot (see Table 3-8). In this case, the achieved precision only slightly exceeded the DQO. In most cases, the preparation duplicates and field duplicates also met the DQO in spite of the additional confounded error due to soil sampling and preparation. The effect of one inordinate preparation pair in the FE_AO data above the knot prevented the data set for this parameter from meeting the DQO as well. Generally, the relationship of increasing standard deviation with increased sources of confounded error was maintained.

Figures 3-51 through 3-62 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Table 3-8. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of Extractable Iron and Aluminum

Data set[a]	Parameter	Below the knot[b] df	SD	DQO	Pairs>DQO n	%	Above the knot[b] df	RSD	DQO	Pairs>DQO n	%
AS	FE_PYP	6	0.0063	0.05			44	6.7%	15%	2	4.5
	AL_PYP	6	0.0063	0.05			44	8.1%	15%	4	9.1
	FE_AO	6	0.0657	0.05	3	50.0	44	10.0%	15%	2	4.5
	AL_AO	7	0.0107	0.05			43	9.3%	15%	3	7.0
	FE_CD	6	0.0319	0.05	1	16.7	44	10.2%	15%	3	6.8
	AL_CD	6	0.0066	0.05			44	10.2%	15%	1	2.3
PD	FE_PYP	17	0.0153				9	5.4%		2	22.2
	AL_PYP	17	0.0227		1	5.9	9	12.0%		1	11.1
	FE_AO	15	0.0353		2	13.3	11	32.4%		2	18.2
	AL_AO	17	0.0177				9	21.3%		1	11.1
	FE_CD	1	0.0127				25	4.9%			
	AL_CD	16	0.0104				10	4.5%			
FD	FE_PYP	51	0.0411		8	15.7	53	14.7%		6	11.3
	AL_PYP	62	0.0356		7	11.5	42	17.6%		6	14.0
	FE_AO	62	0.0324		8	12.9	42	12.0%		10	23.8
	AL_AO	69	0.0357		7	10.1	35	14.4%		7	20.0
	FE_CD	4	0.0237				100	13.4%		9	9.0
	AL_CD	43	0.0184		1	2.3	61	12.7%		8	13.1
S/H	FE_PYP	275	0.1455				334	73.3%			
	AL_PYP	293	0.1116				316	65.1%			
	FE_AO	296	0.1736				313	81.9%			
	AL_AO	330	0.1219				279	76.9%			
	FE_CD	1	0.0283				608	58.0%			
	AL_CD	194	0.1228				415	52.5%			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.

[b] Standard deviation and RSD data in reporting units and percent, respectively, for mineral soil samples below and above the knot point of 0.33 weight percent.

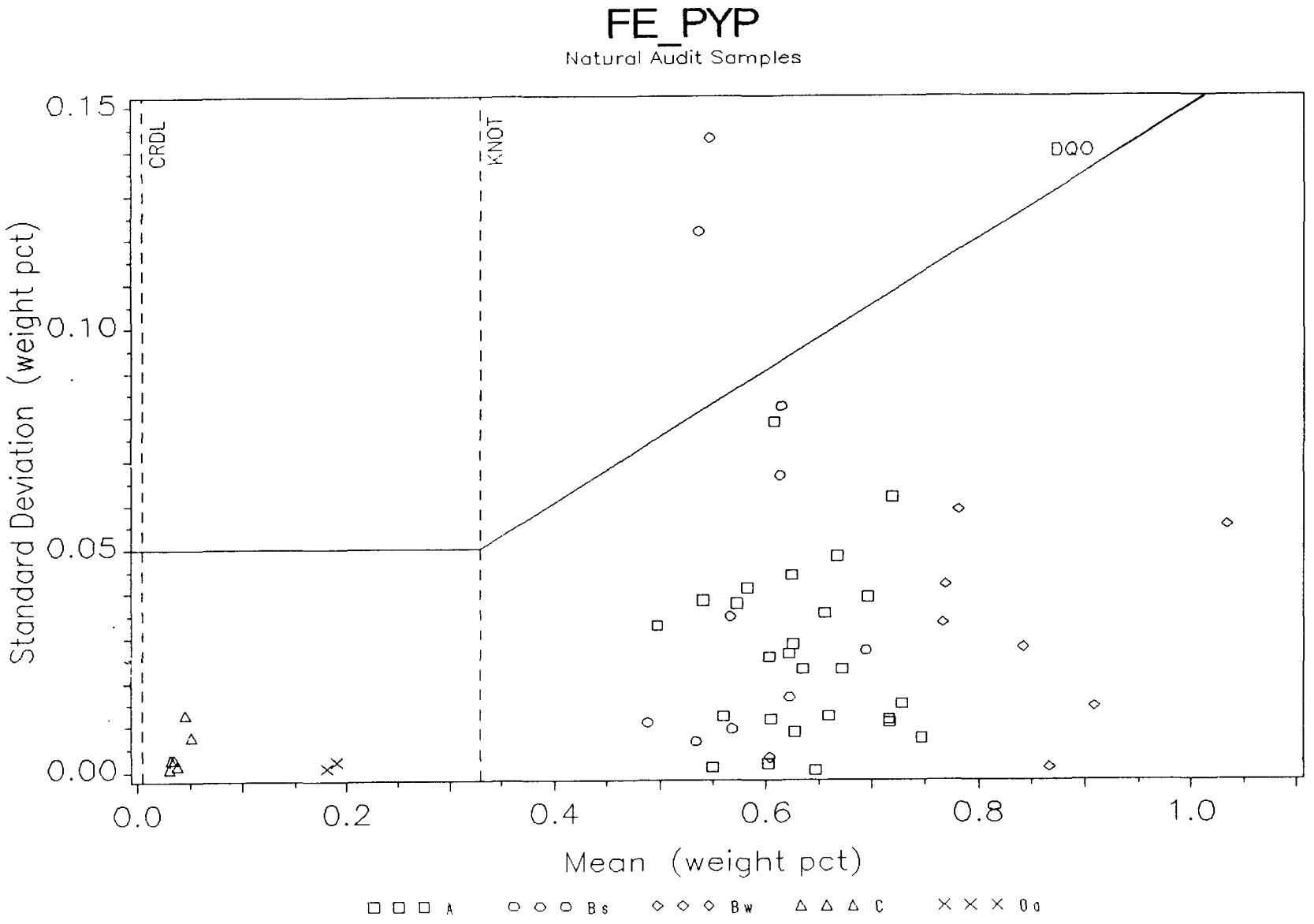
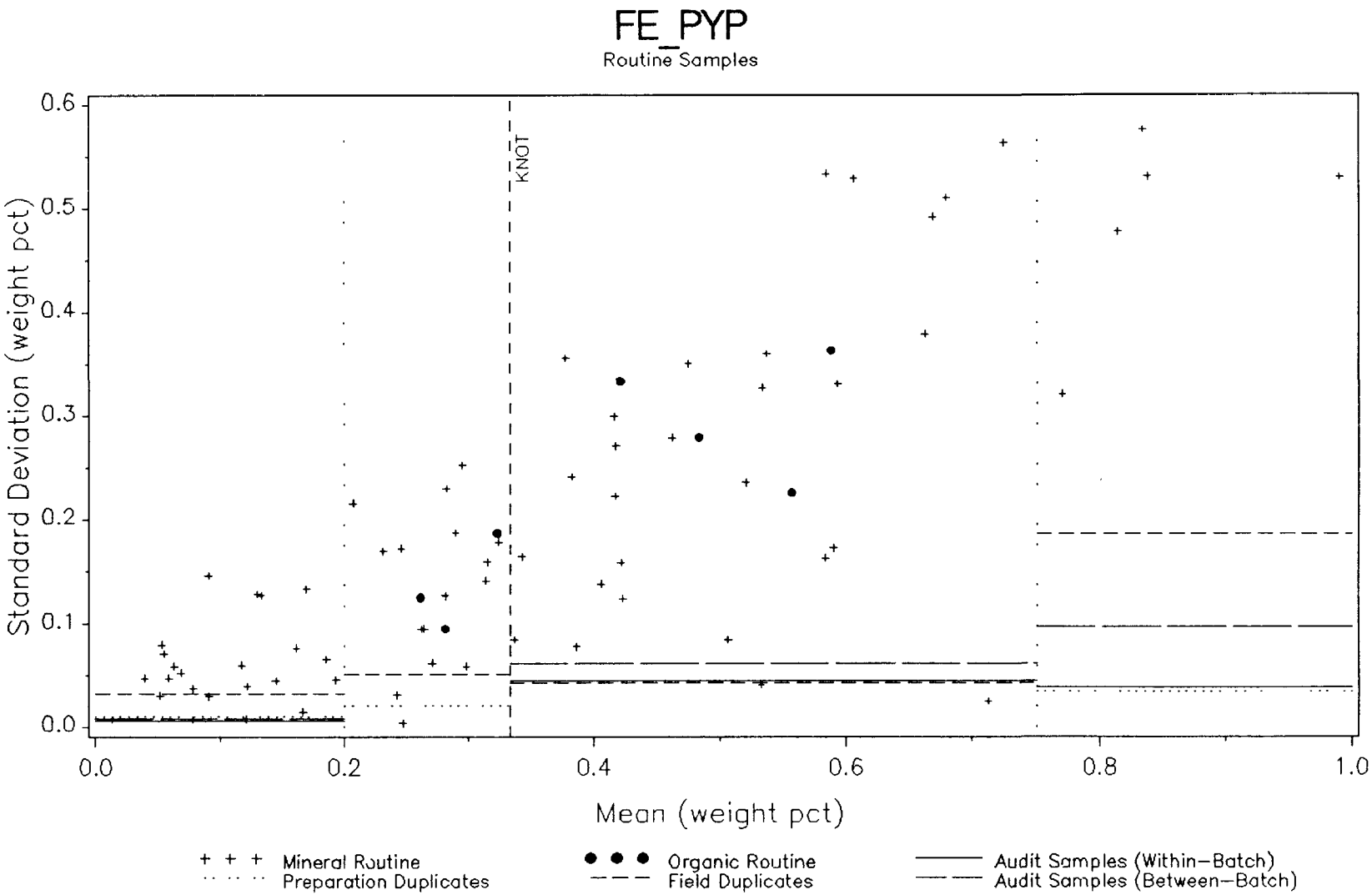


Figure 3-51. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for FE_PYP.



Note: Five mineral, one organic sampling class/horizon groups exceed plot boundaries.

Figure 3-52. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for FE_PYP.

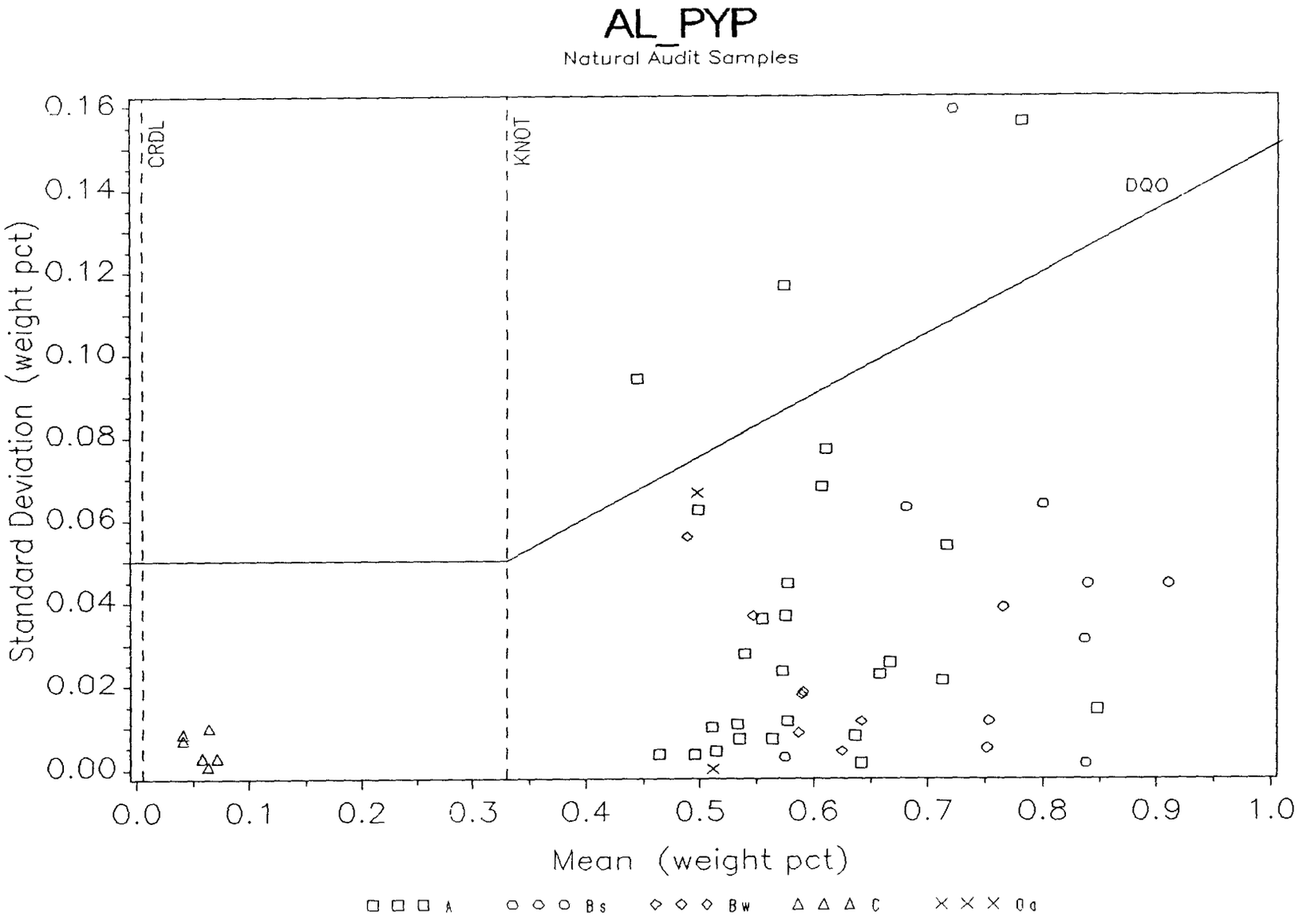


Figure 3-53. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AL_PYP.

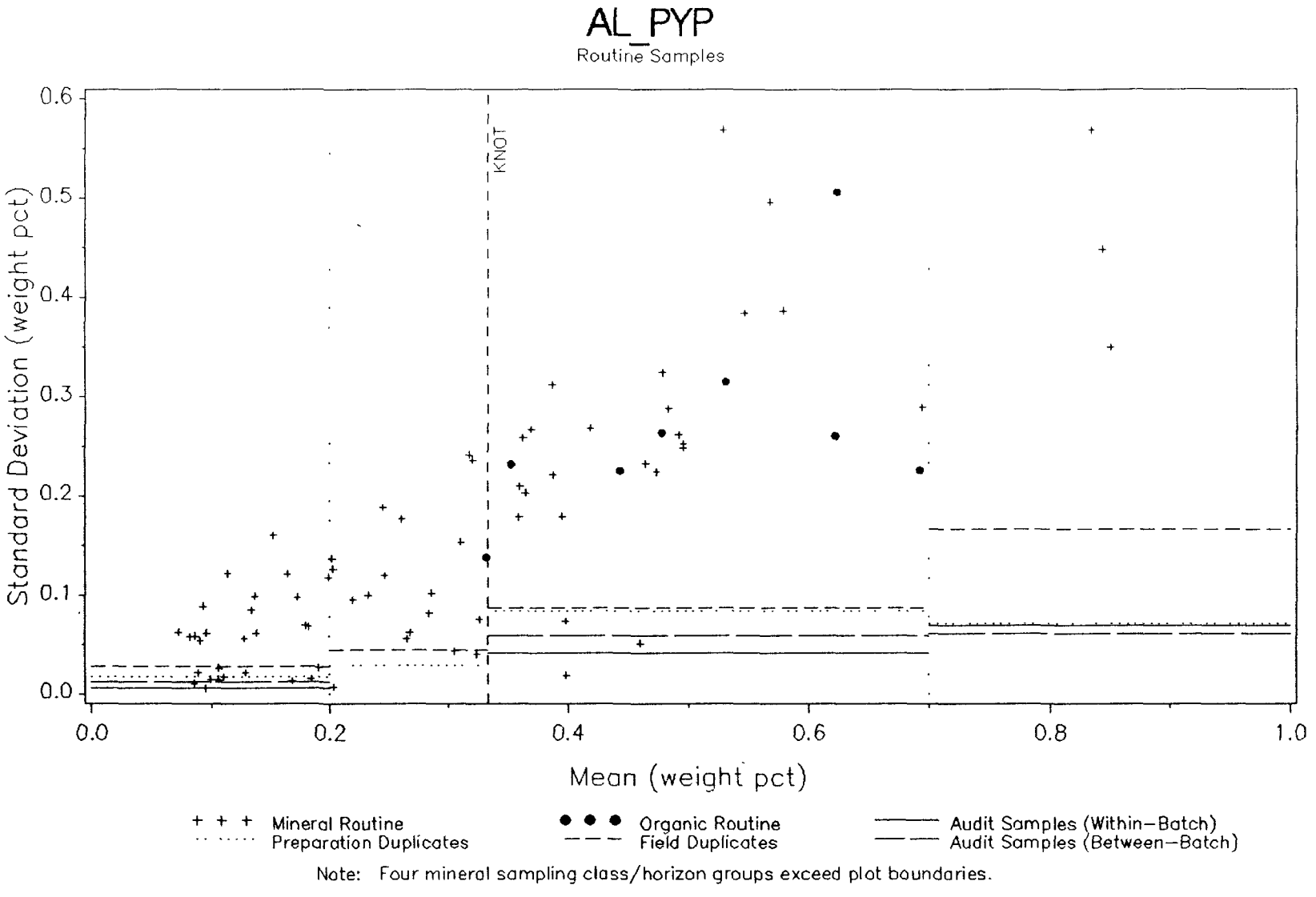


Figure 3-54. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AL_PYP.

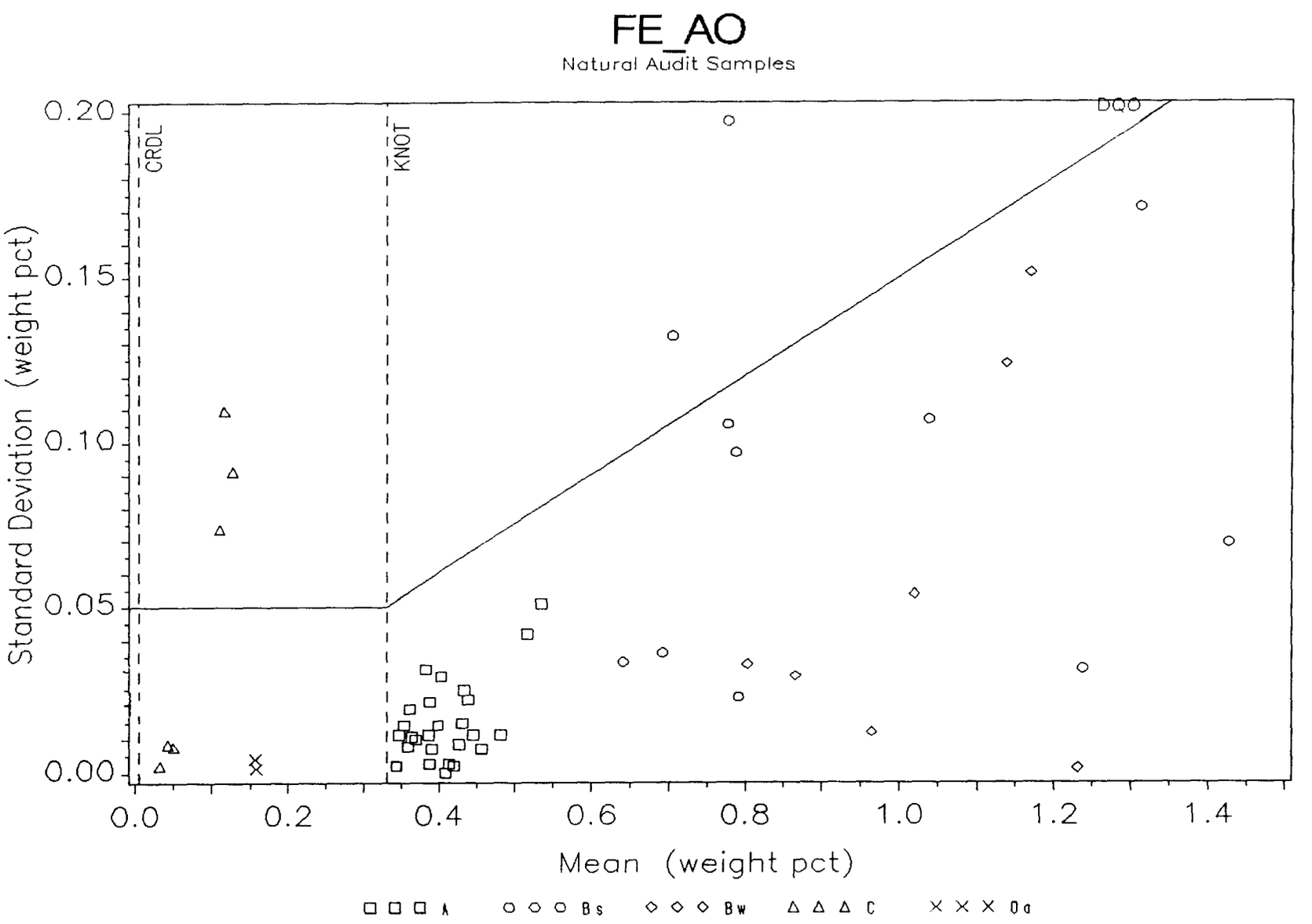


Figure 3-55. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for FE_AO.

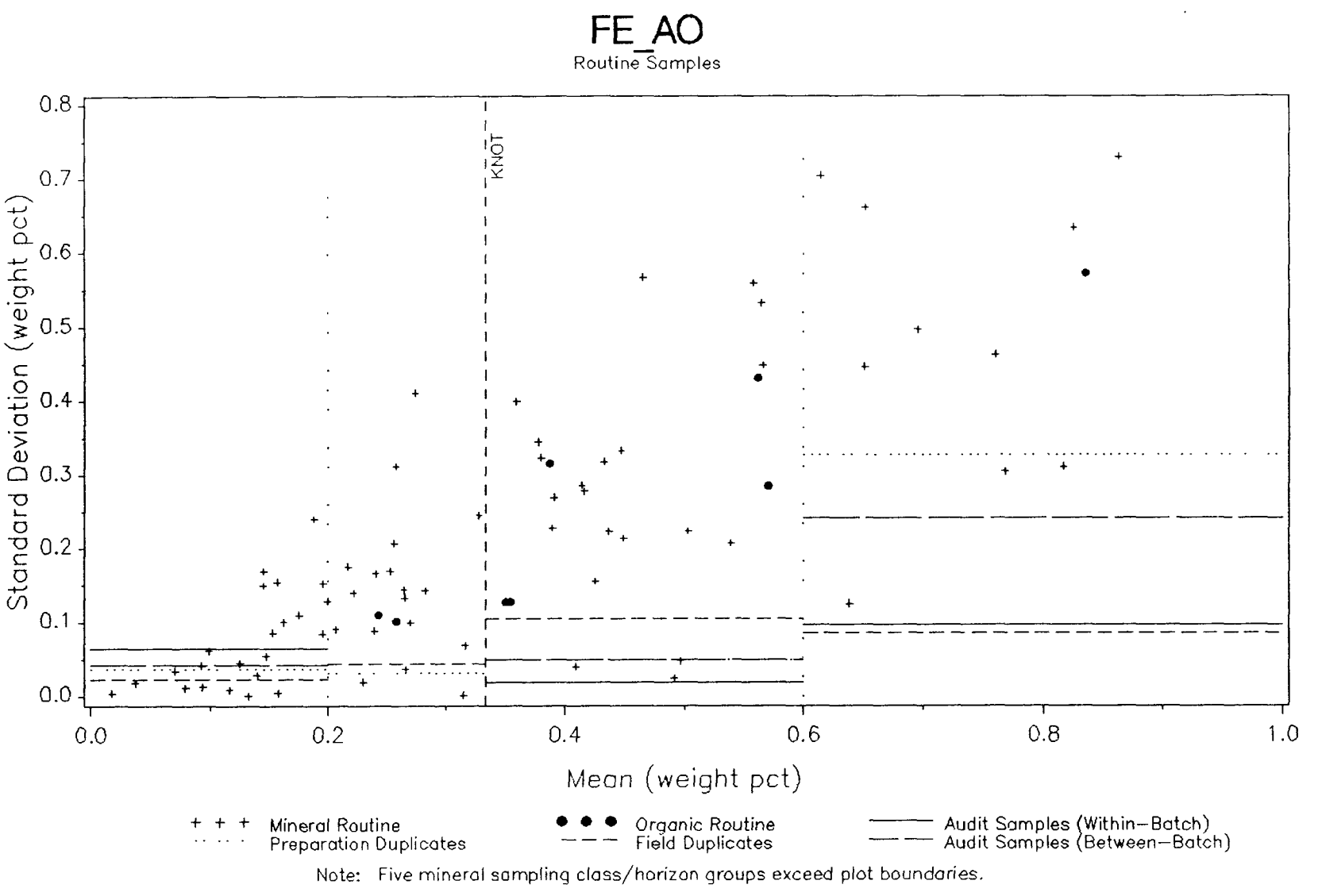


Figure 3-56. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for FE_AO.

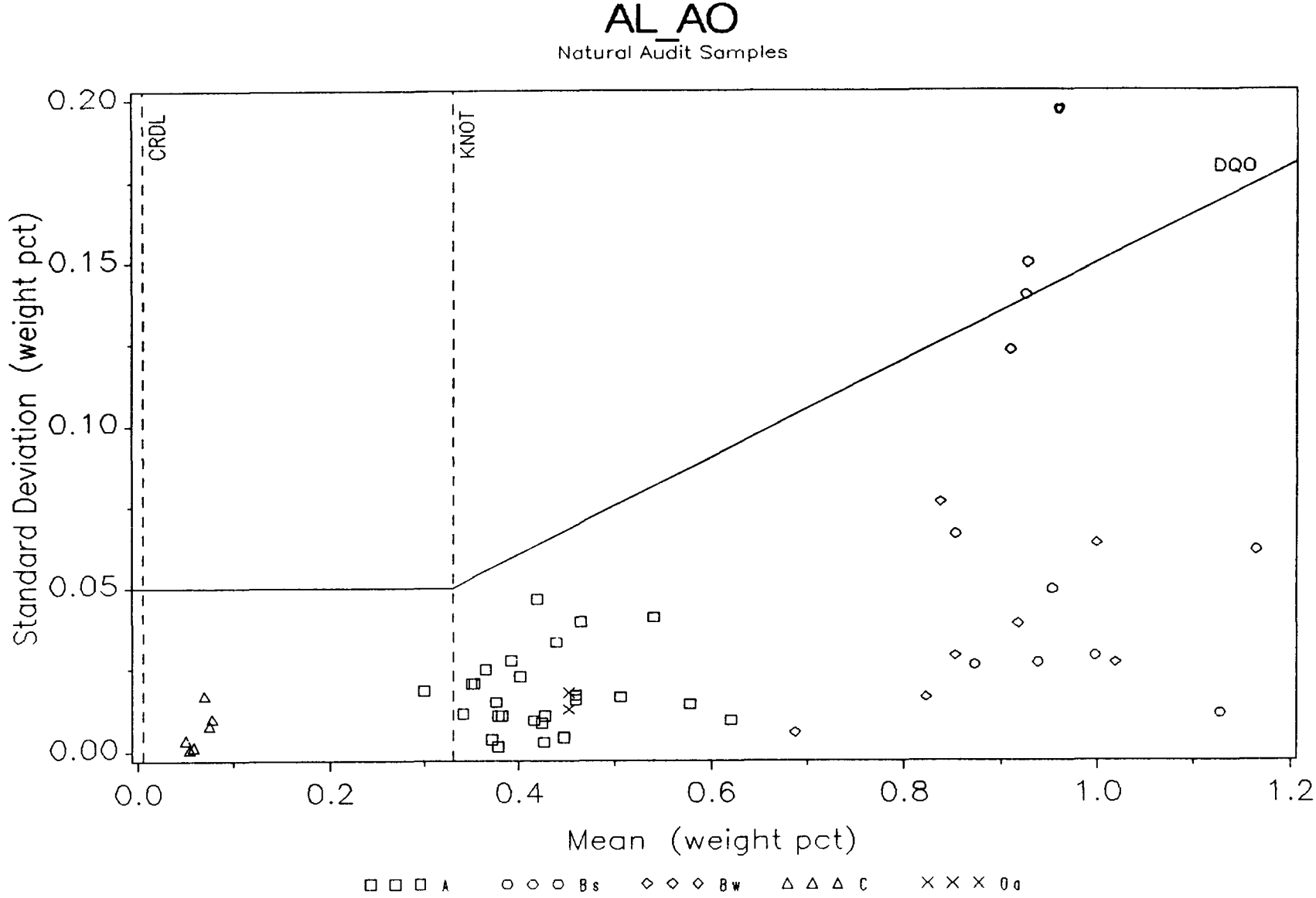


Figure 3-57. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AL_AO.

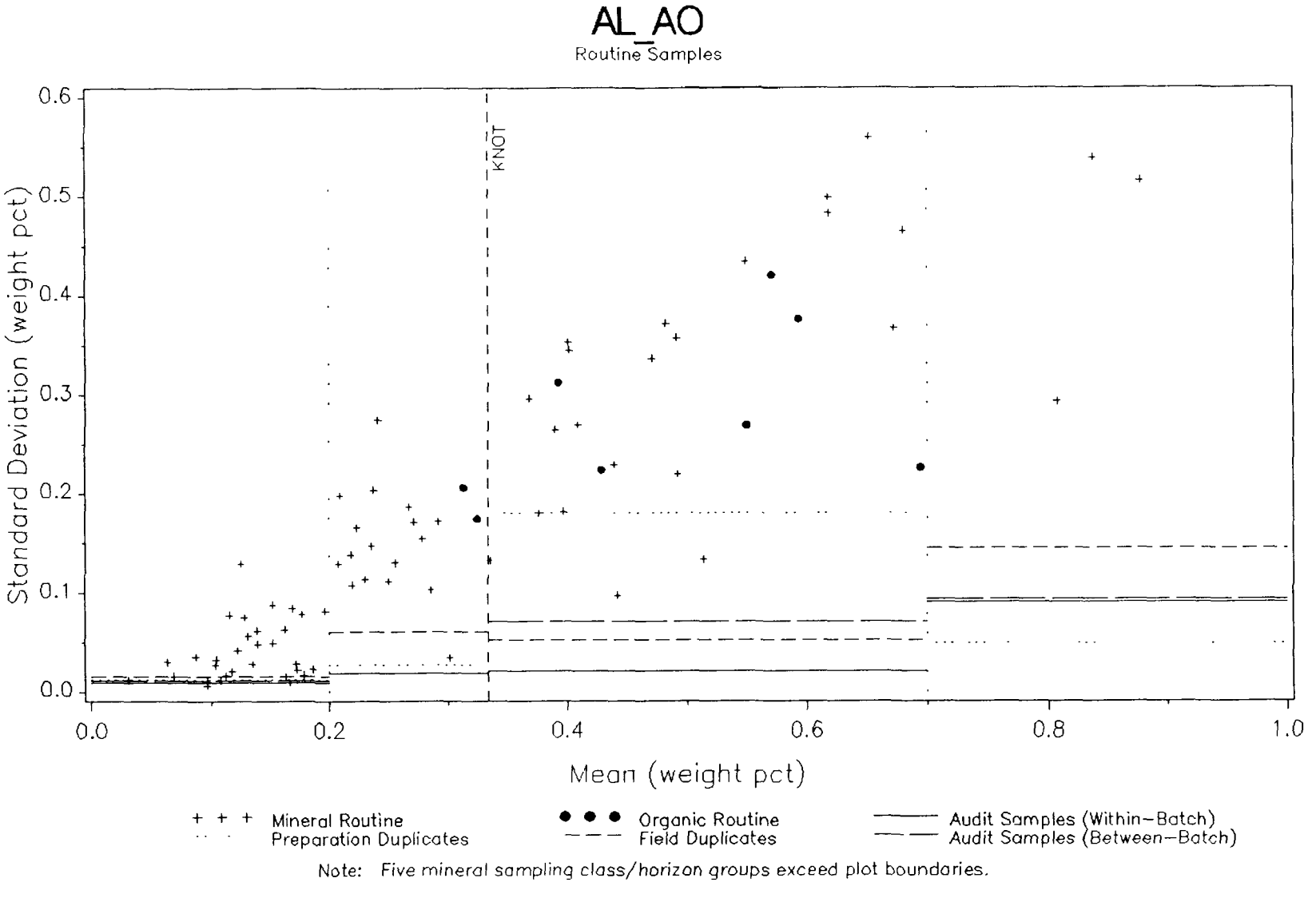


Figure 3-58. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AL_AO.

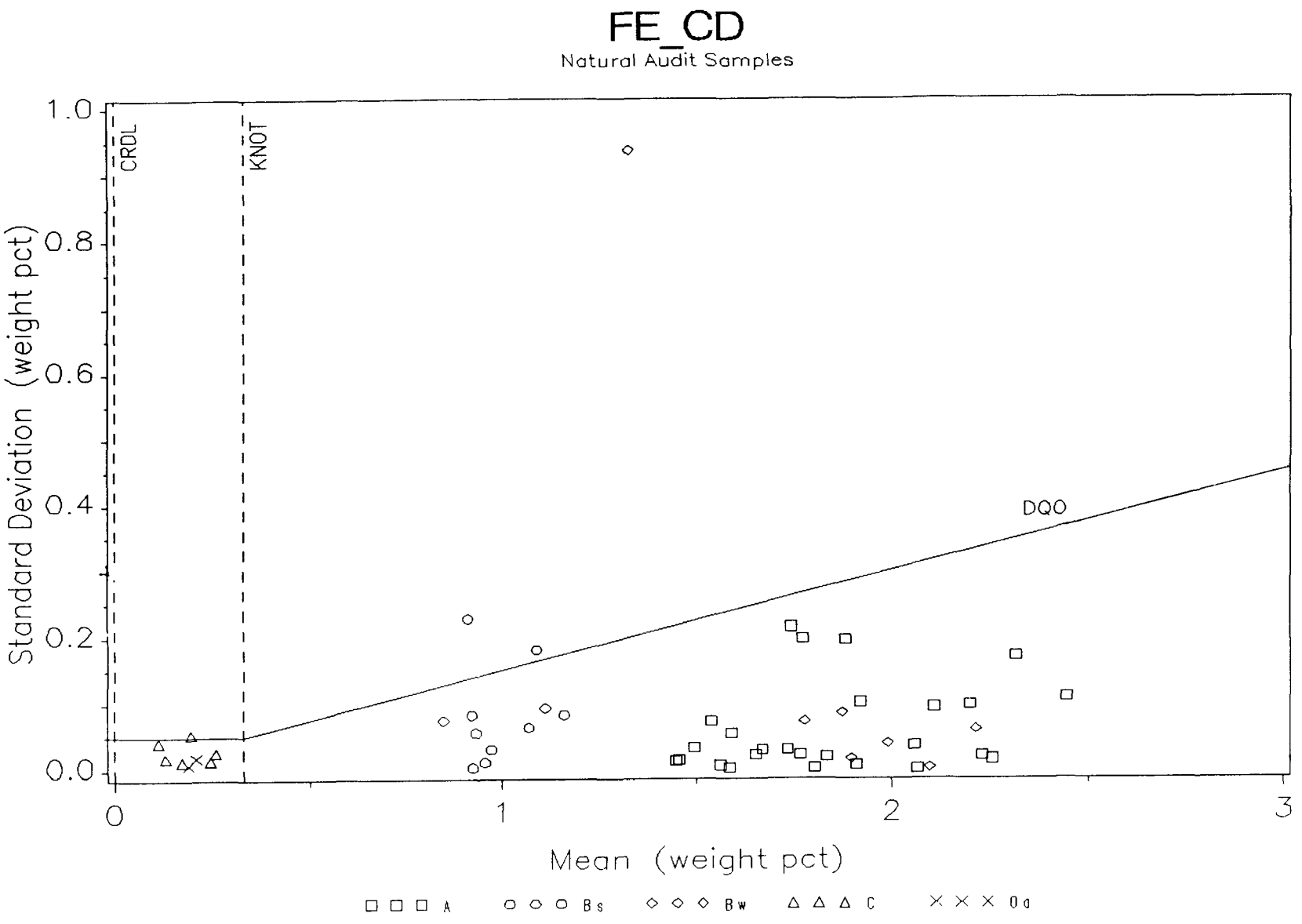


Figure 3-59. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for FE_CD.

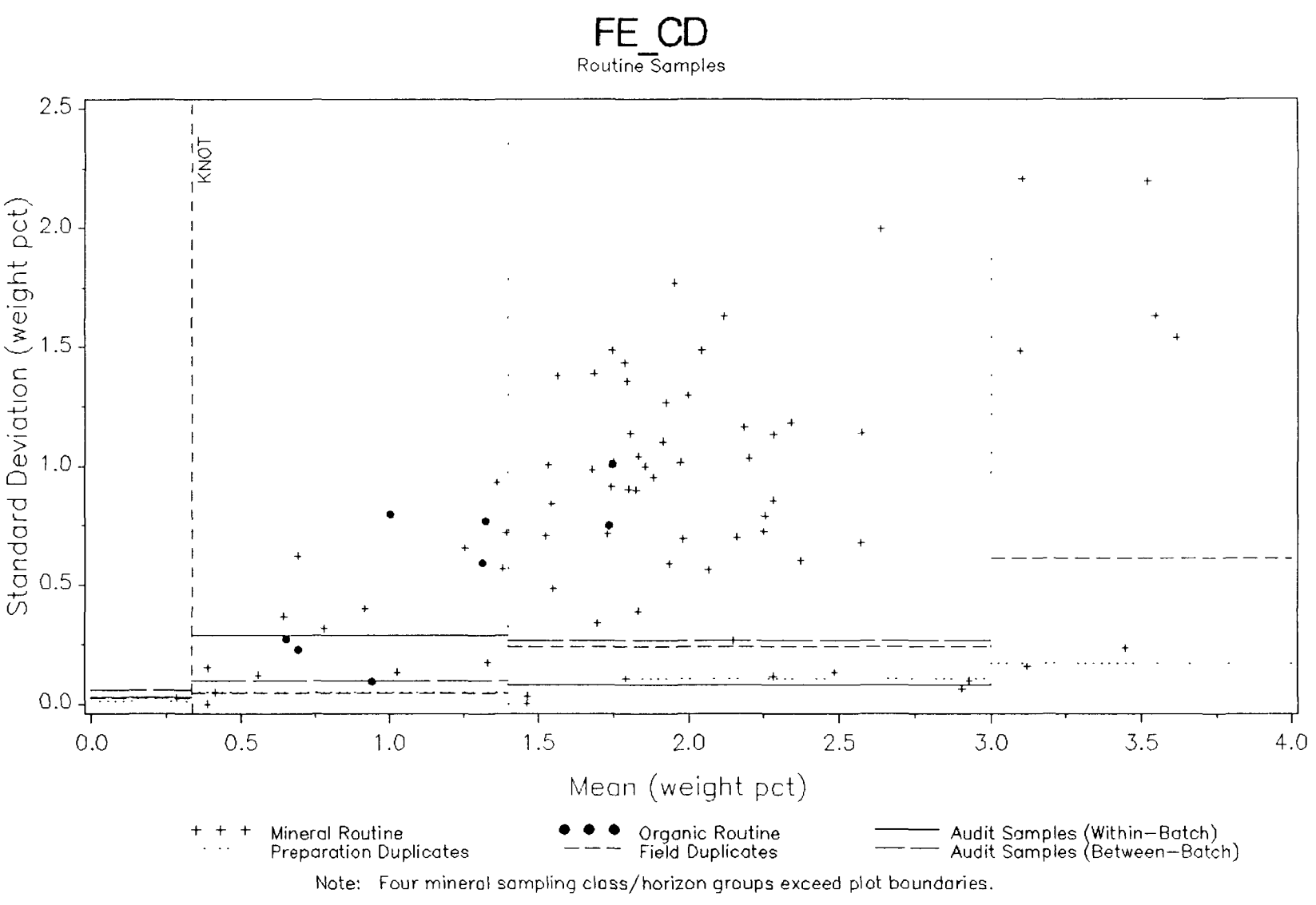


Figure 3-60. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for FE_CD.

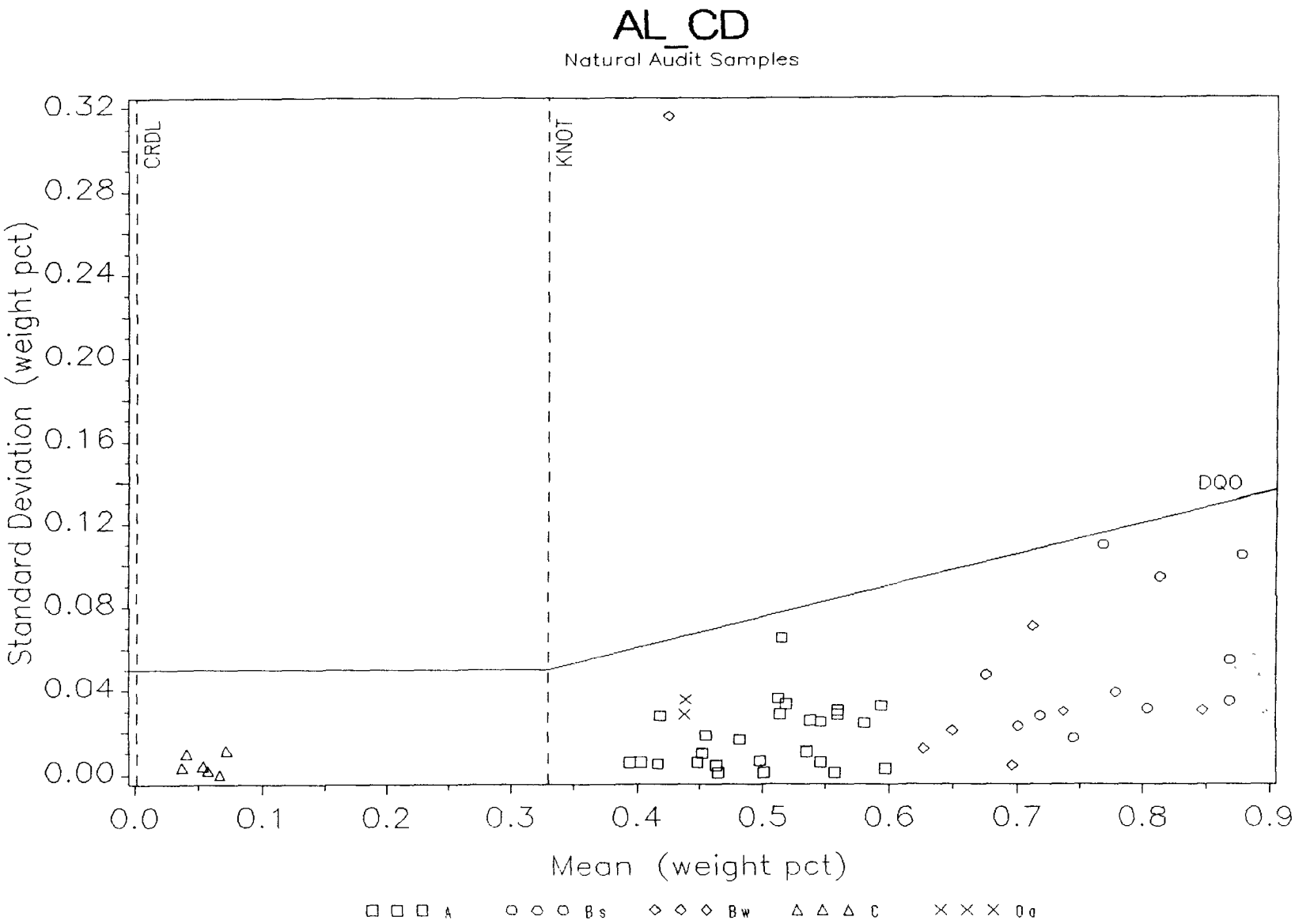


Figure 3-61. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for AL_CD.

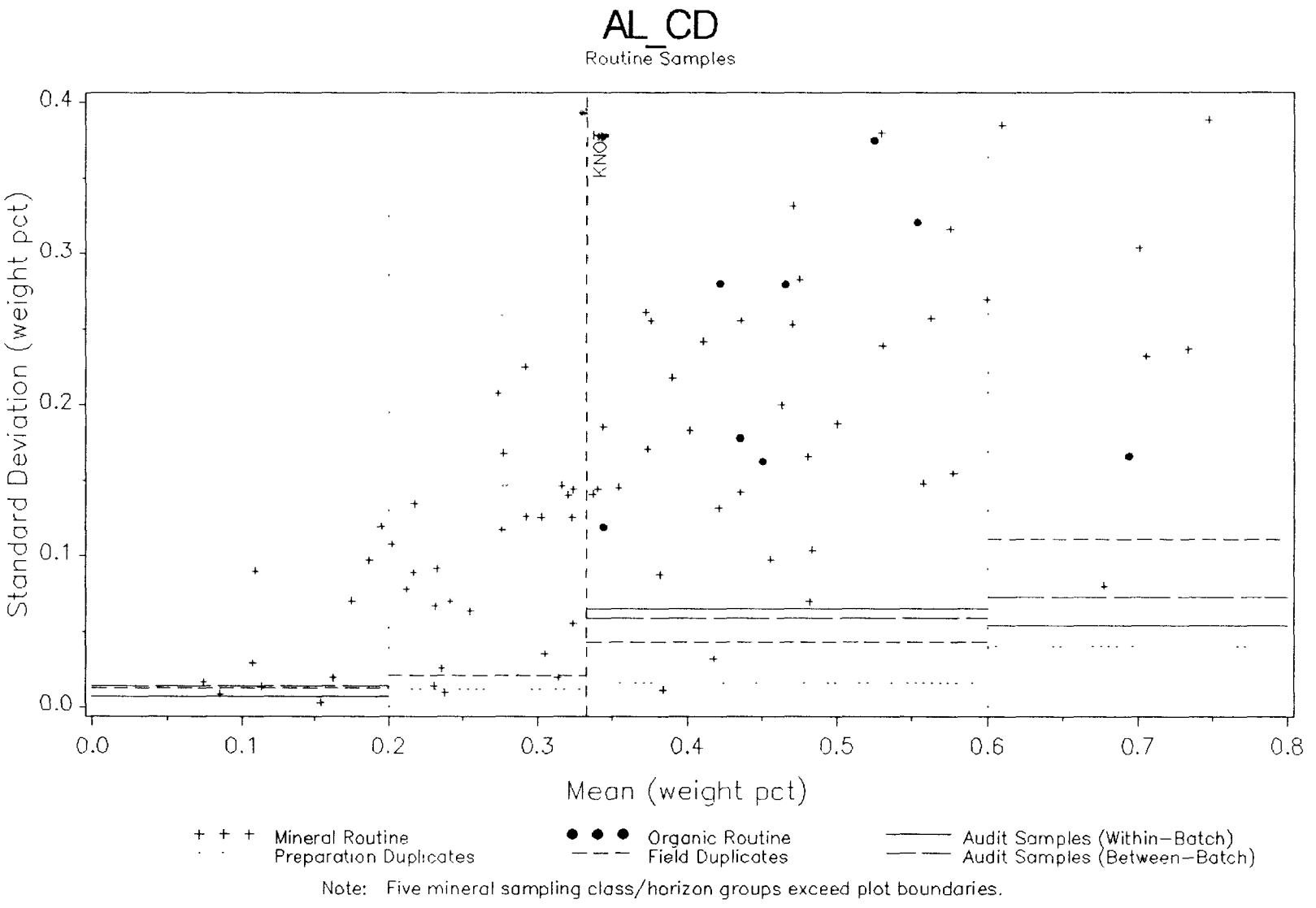


Figure 3-62. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for AL_CD.

Extractable Sulfate and Sulfate Adsorption Isotherms

Table 3-9
Figures 3-63 through 3-78

The analytical within-batch precision DQOs were satisfied for all of the sulfate parameters except SO4_PO4 and SO4_0 (see Table 3-9). In these two cases, a significant amount of scatter in the Bs and C audit samples was responsible for large variability above the knot and below the knot, respectively. In most cases, the preparation duplicates also met the analytical DQOs, which suggests that preparation error is minor for these parameters. The effect of increasing sulfate levels tends to promote decreasing variability in the isotherm parameters. A general pattern of increasing standard deviation with increased sources of confounded error was maintained.

Table 3-9. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of Extractable Sulfate and Sulfate Adsorption

Data set[a]	Parameter	Below the knot[b] df	SD	DQO	Pairs>DQO n	%	Above the knot[b] df	RSD	DQO	Pairs>DQO n	%
AS	SO4_H2O	17	0.8916	1.00	4	23.5	33	4.2%	10%	2	6.1
	SO4_PO4	6	2.2402	1.00	2	33.3	44	15.0%	10%	7	15.9
	SO4_0	8	0.0921	0.05	2	25.0	42	6.2%	5%	13	31.0
	SO4_2	.	.	0.05	.	.	50	4.4%	5%	9	18.0
	SO4_4	.	.	0.05	.	.	50	3.1%	5%	9	18.0
	SO4_8	.	.	0.05	.	.	50	2.7%	5%	2	4.0
	SO4_16	.	.	0.05	.	.	50	5.4%	5%	4	8.0
	SO4_32	.	.	0.05	.	.	50	1.7%	5%		
PD	SO4_H2O	16	0.7341		3	18.8	10	8.8%		3	30.0
	SO4_PO4	3	0.9141		1	33.3	23	6.5%		4	17.4
	SO4_0	20	0.0728		5	25.0	6	3.1%			
	SO4_2	5	0.0177				21	5.0%		4	19.0
	SO4_4	4	0.0773		1	25.0	22	4.3%		3	13.6
	SO4_8	2	0.0015				24	3.2%		3	12.5
	SO4_16	1	0.0028				25	3.7%		4	16.0
	SO4_32	.	.		.	.	26	3.1%		4	15.4
FD	SO4_H2O	53	1.5490		13	25.0	51	18.8%		15	28.8
	SO4_PO4	8	1.2009		4	50.0	96	11.4%		25	26.0
	SO4_0	59	0.0956		21	36.2	45	14.5%		26	56.5
	SO4_2	28	0.1282		16	57.1	76	9.3%		31	40.8
	SO4_4	21	0.1040		11	52.4	83	8.2%		31	37.3
	SO4_8	11	0.1071		6	54.5	93	6.2%		31	33.3
	SO4_16	4	0.3395		3	75.0	100	5.6%		28	8.0
	SO4_32	.	.		.	.	104	4.3%		21	20.2
S/H	SO4_H2O	357	5.3363				252	46.5%			
	SO4_PO4	4	3.8622				605	87.2%			
	SO4_0	397	0.6026				212	53.0%			
	SO4_2	201	0.7242				408	43.7%			
	SO4_4	45	0.9911				564	43.4%			
	SO4_8	1	0.0997				608	41.6%			
	SO4_16	1	0.3769				608	33.9%			
	SO4_32	.	.				609	25.3%			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.

[b] Standard deviation and RSD data in reporting units and percent, respectively, for mineral soil samples below and above the knot point, 10.0 mg S/kg for extractable sulfate and 1.0 mg S/L for the isotherms; a dot signifies a lack of data occupying that range.

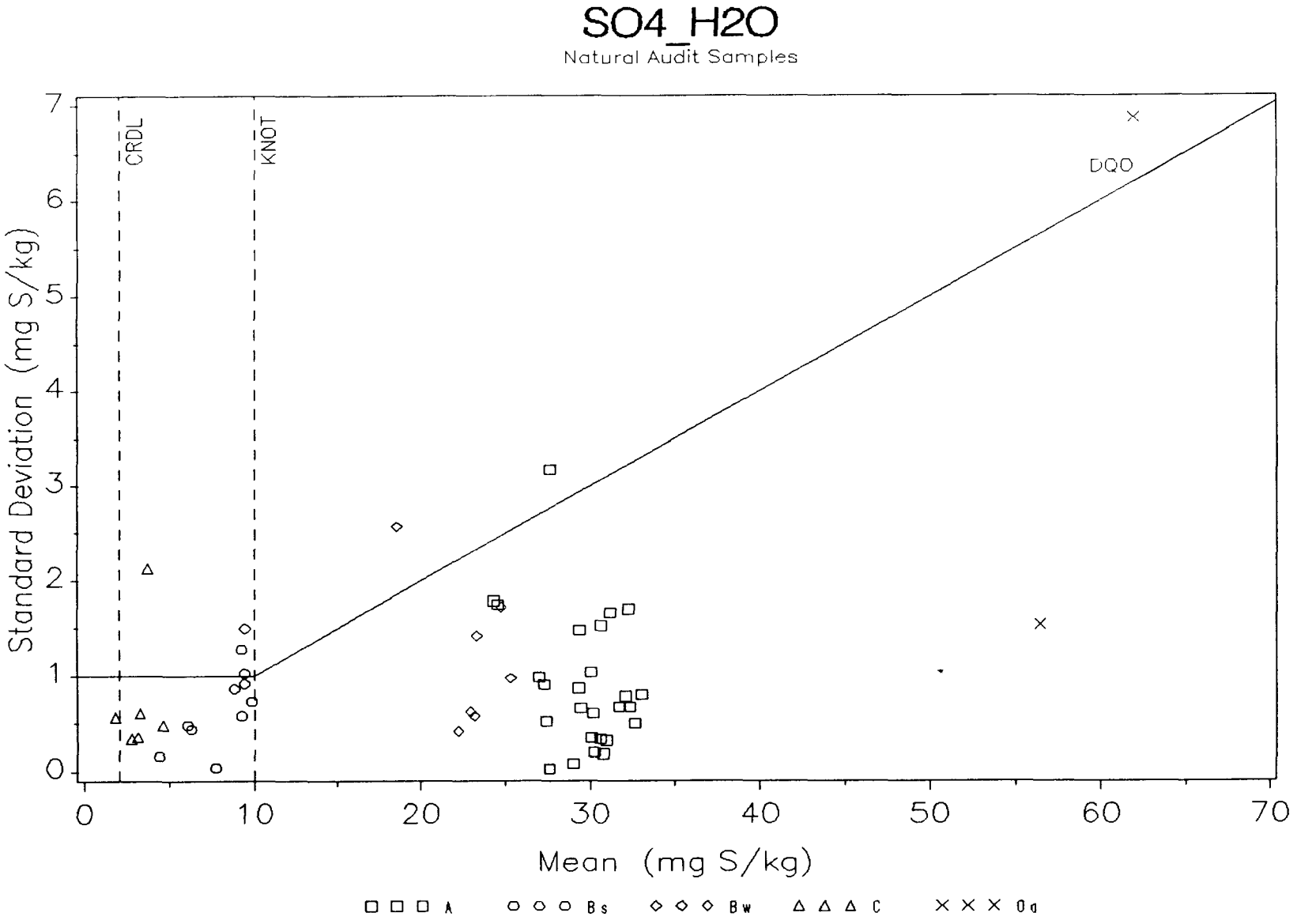


Figure 3-63. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_H20.

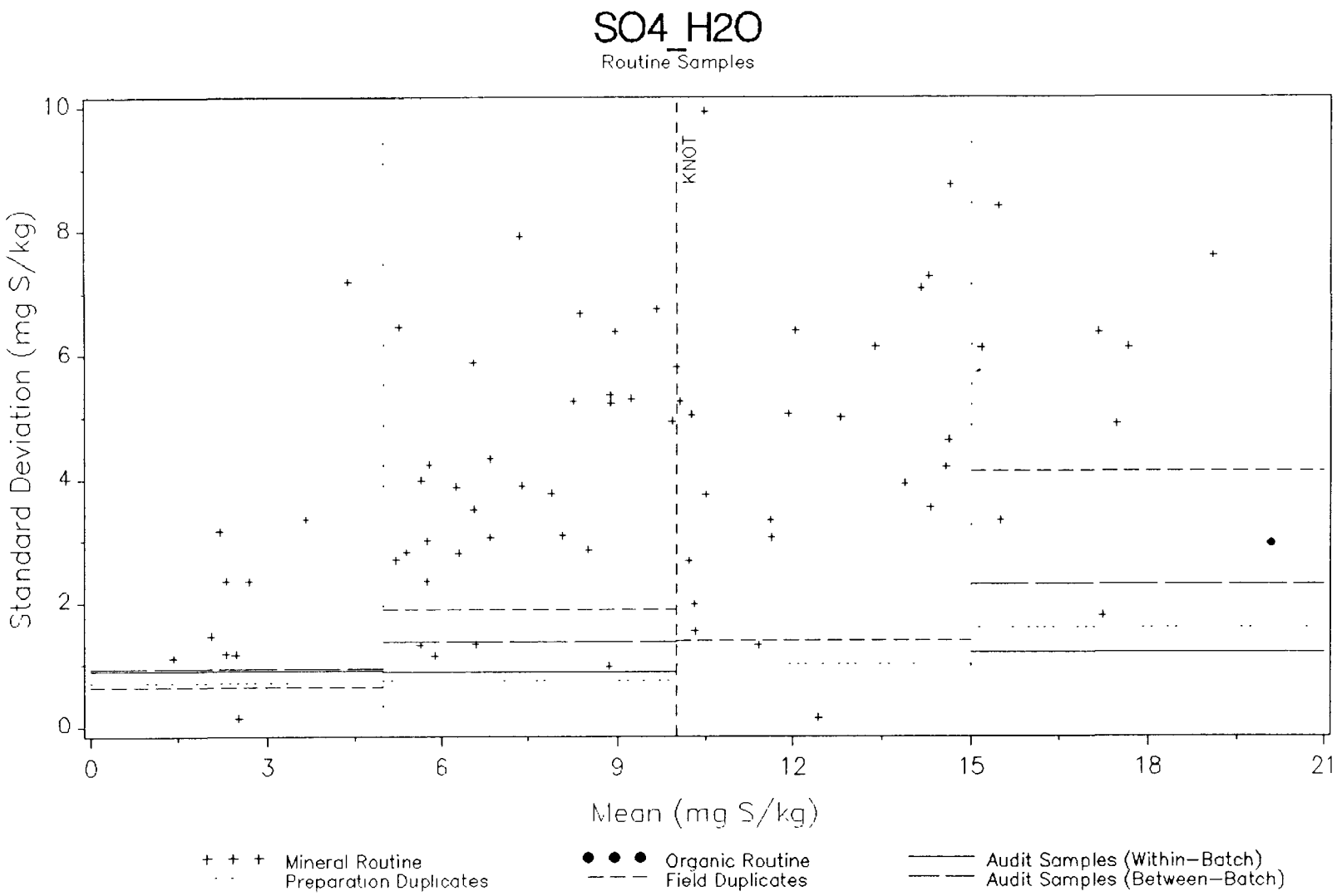


Figure 3-64. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_H2O.

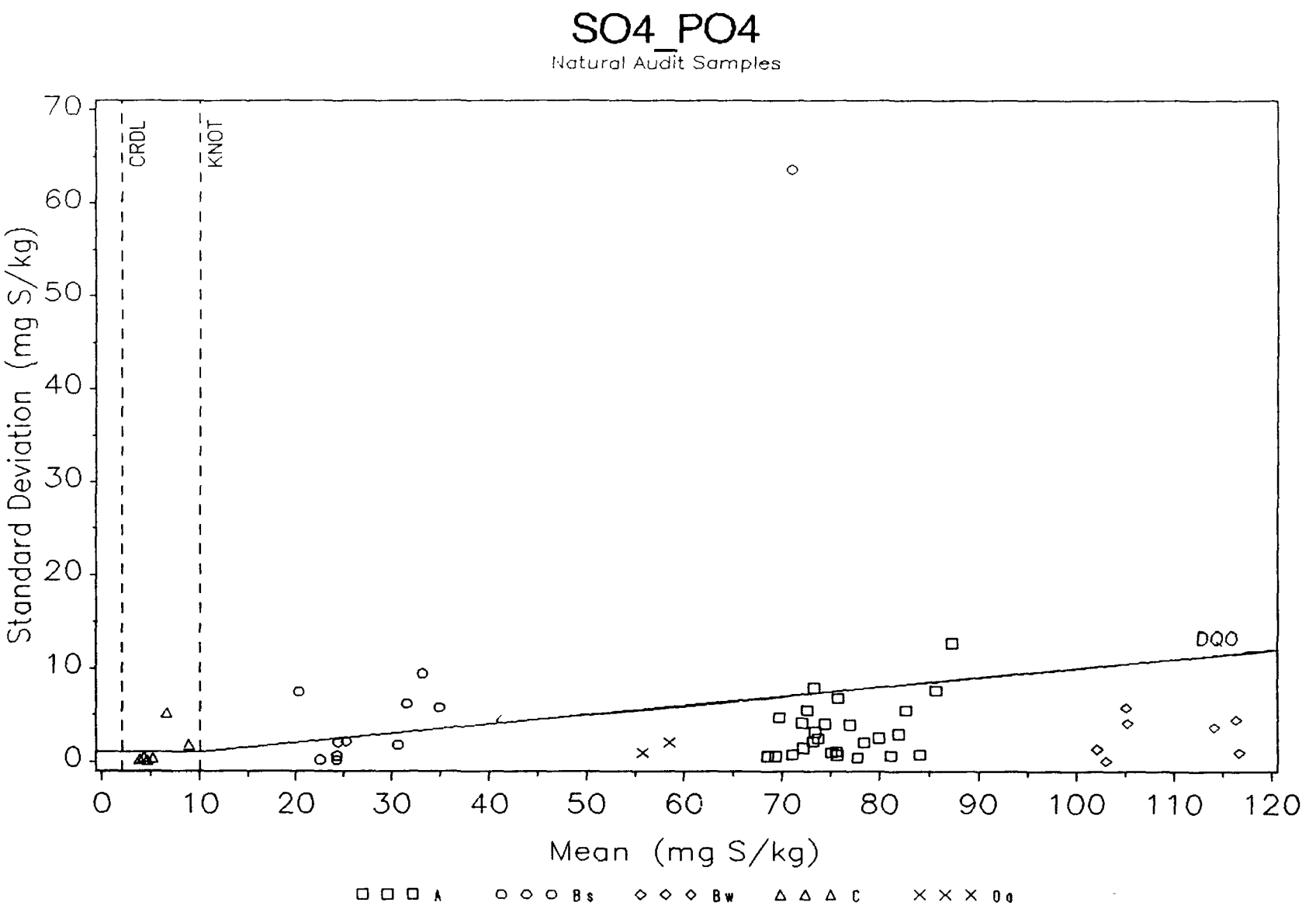


Figure 3-65. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_PO4.

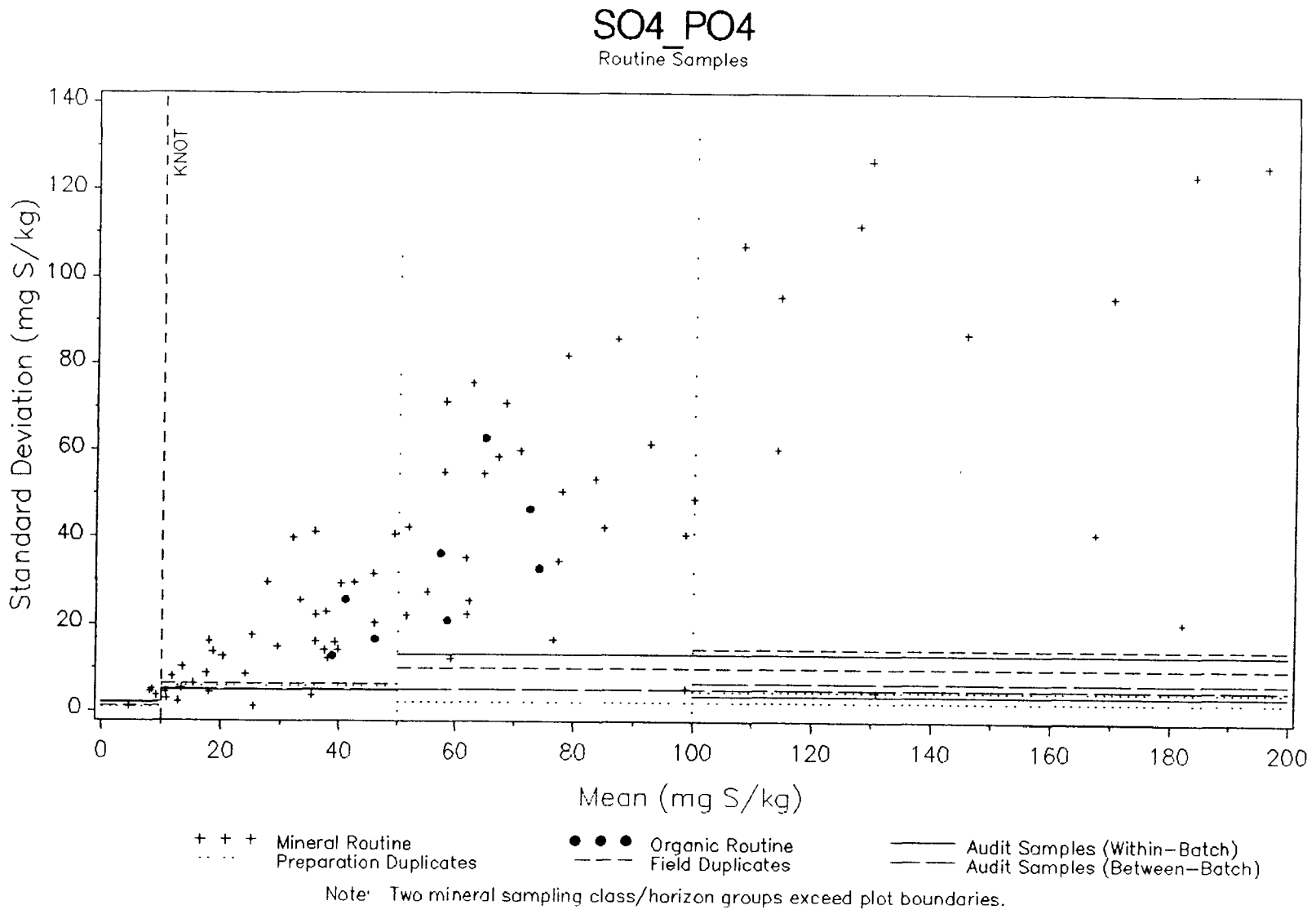


Figure 3-66. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_PO4.

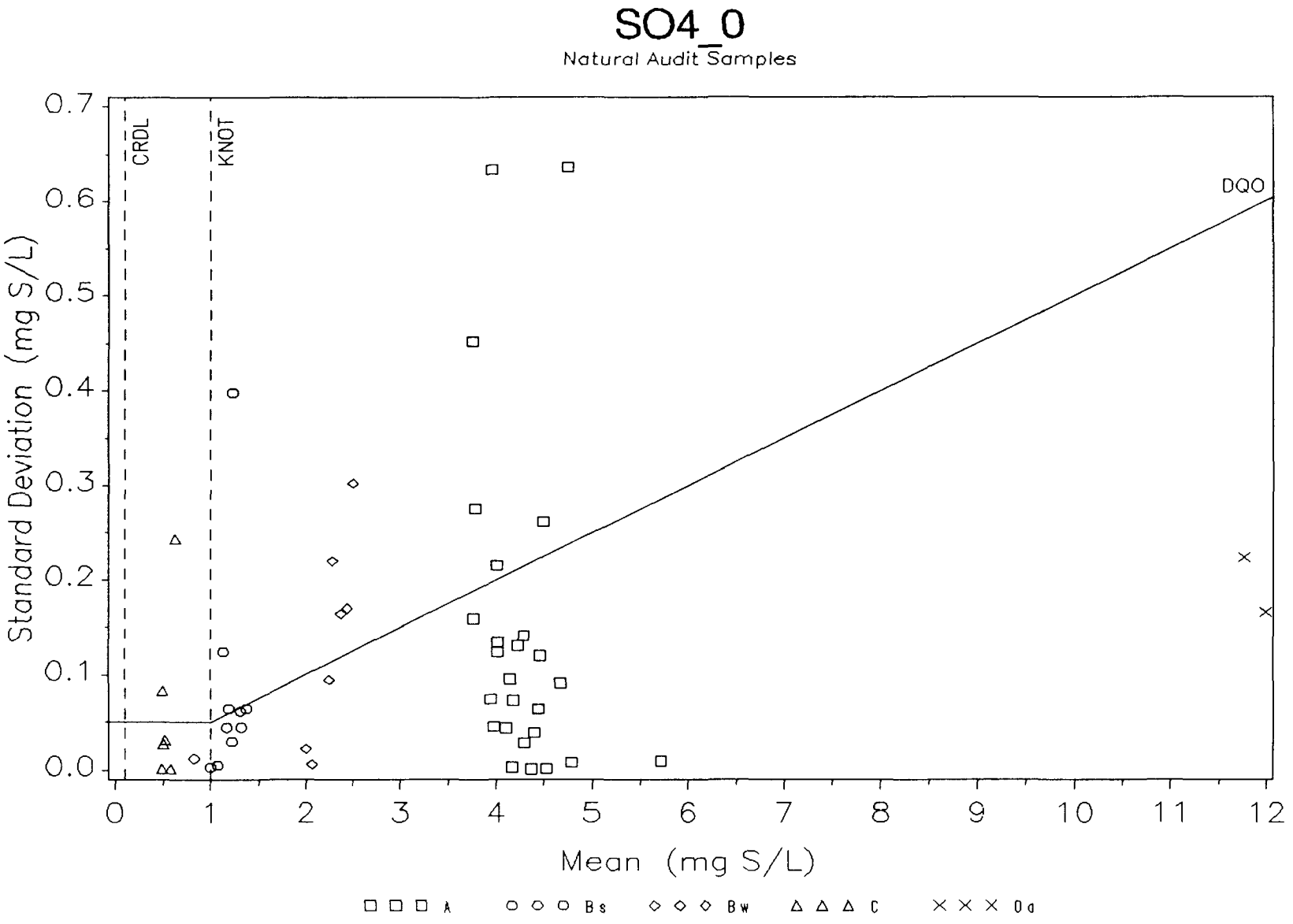


Figure 3-67. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_0.

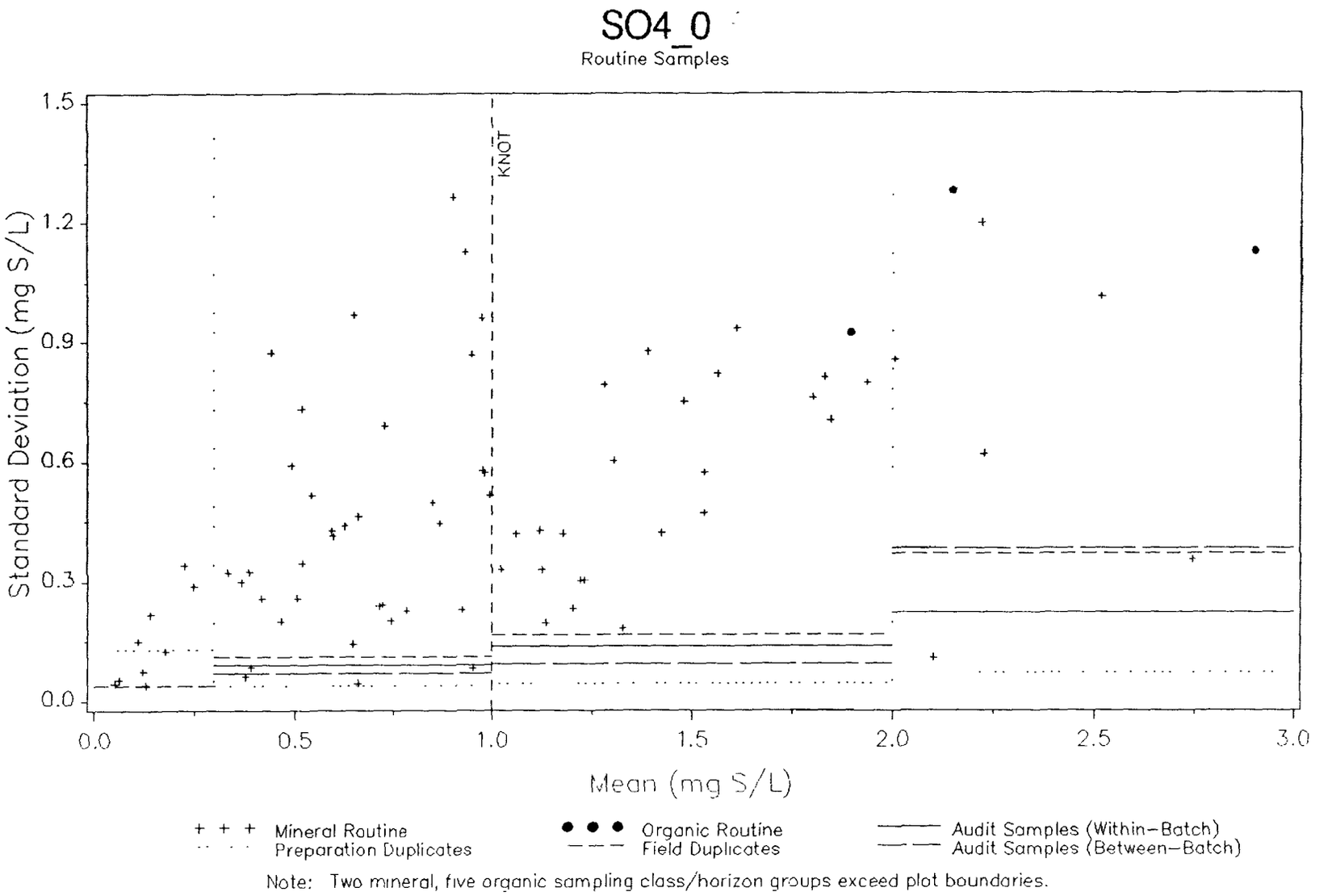


Figure 3-68. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_0.

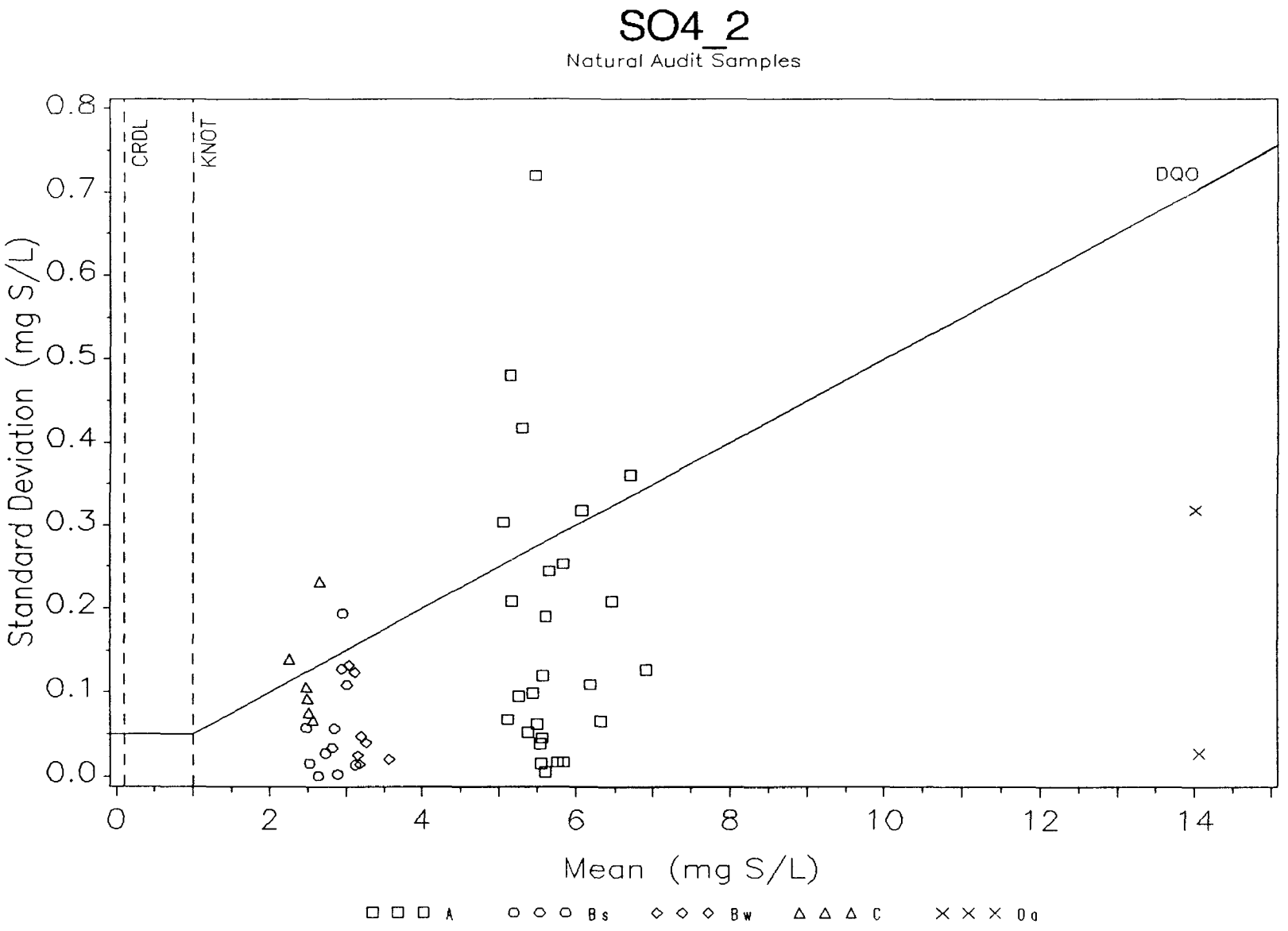


Figure 3-69. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_2.

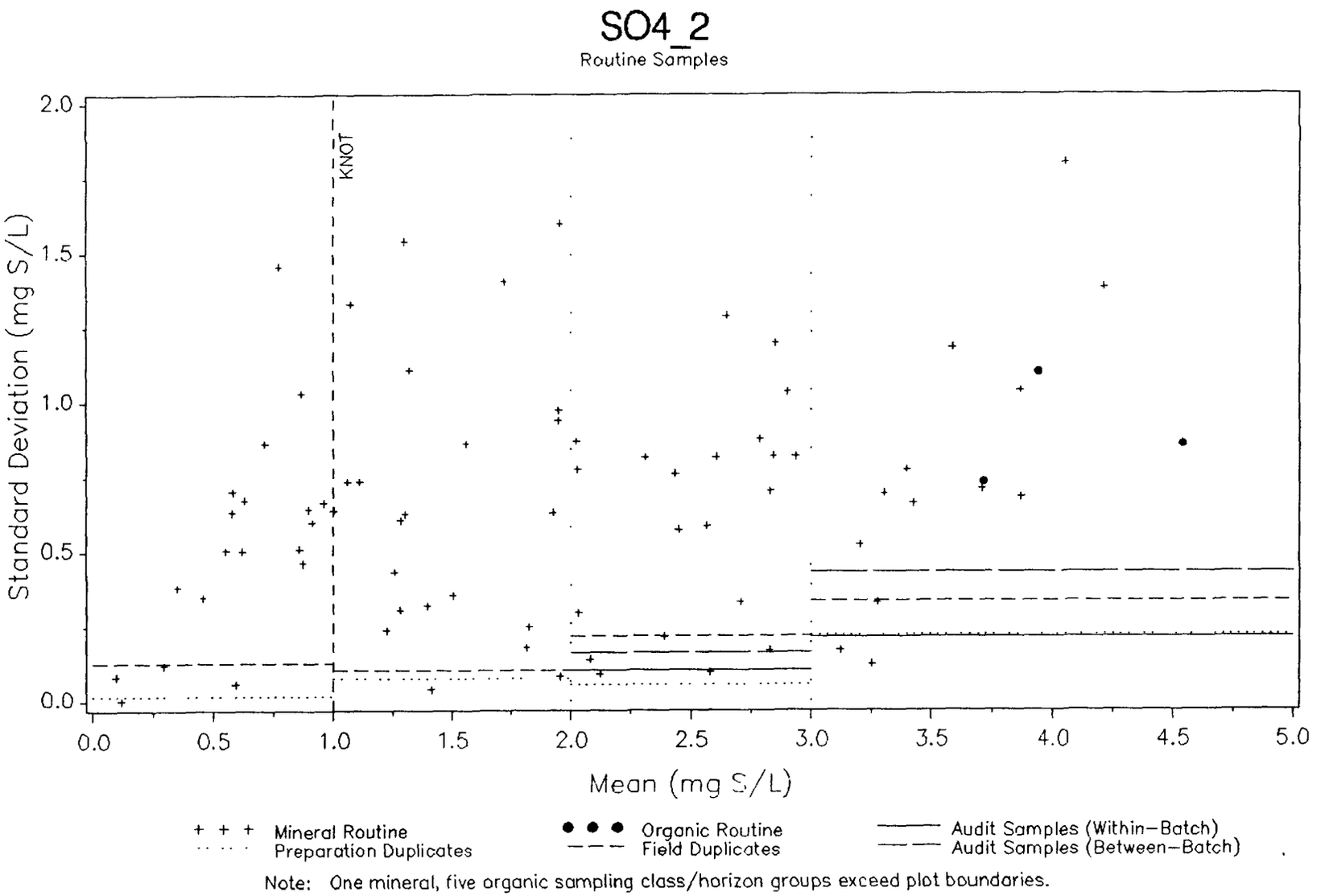


Figure 3-70. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_2.

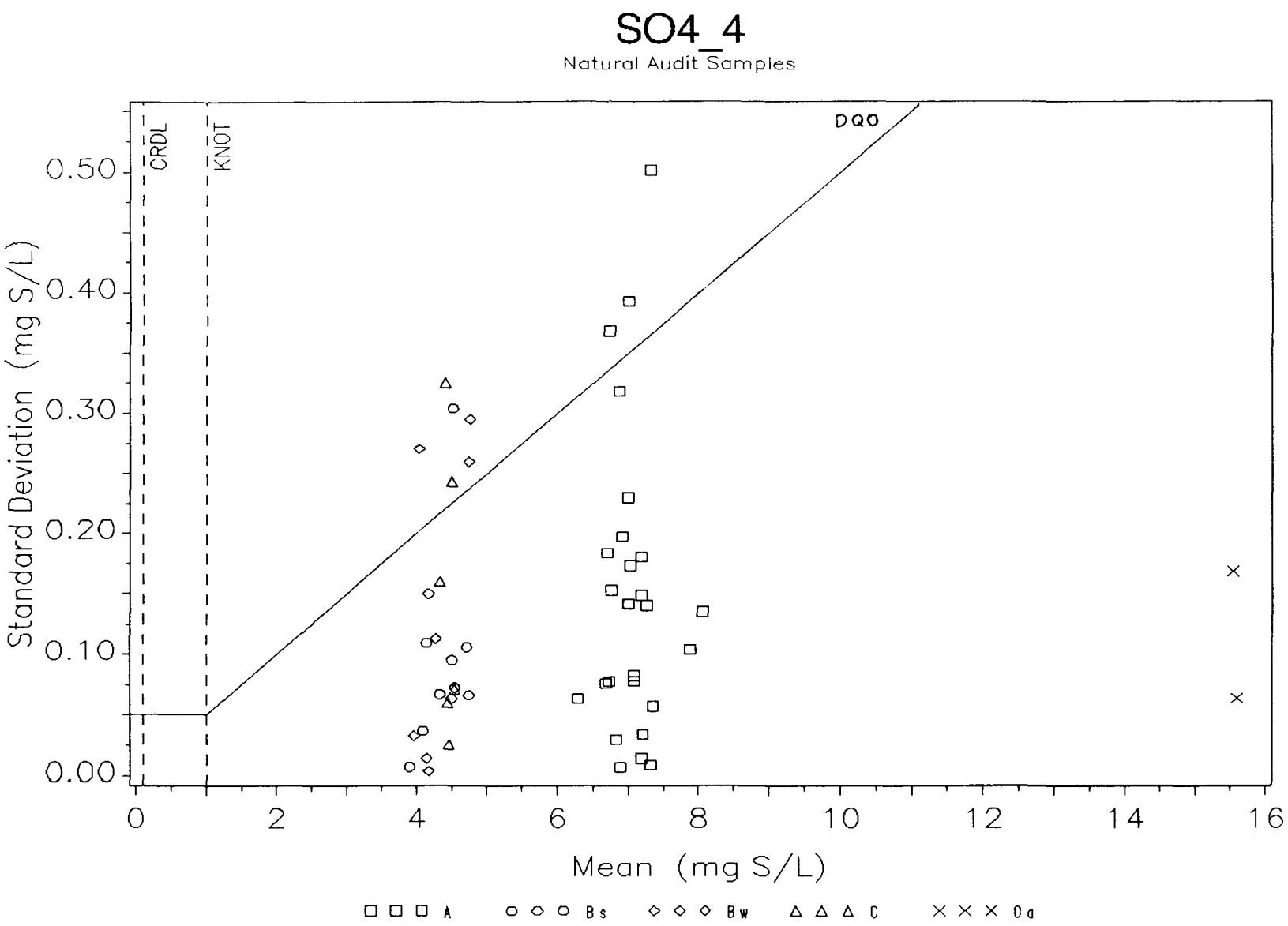


Figure 3-71. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_4.

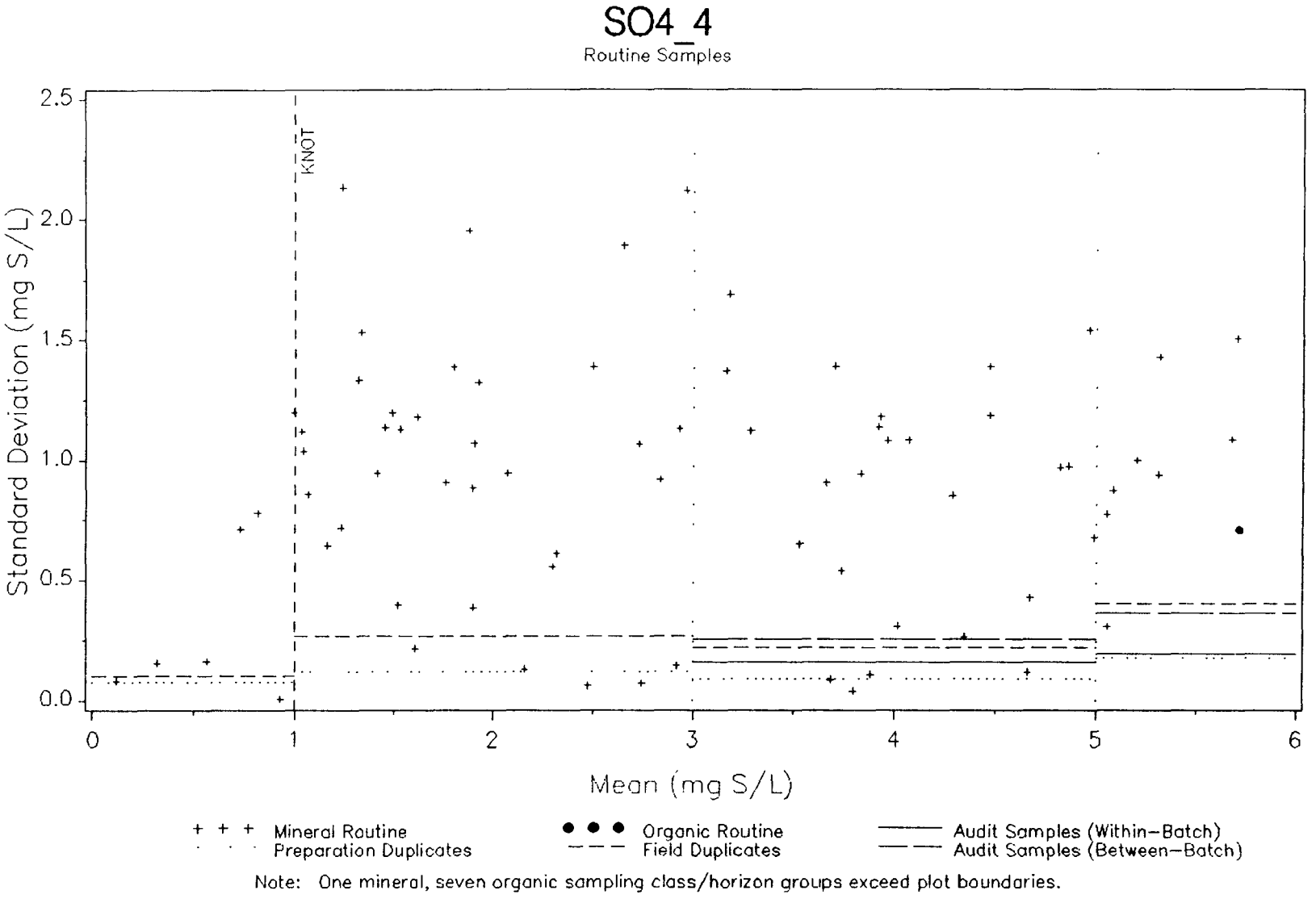


Figure 3-72. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_4.

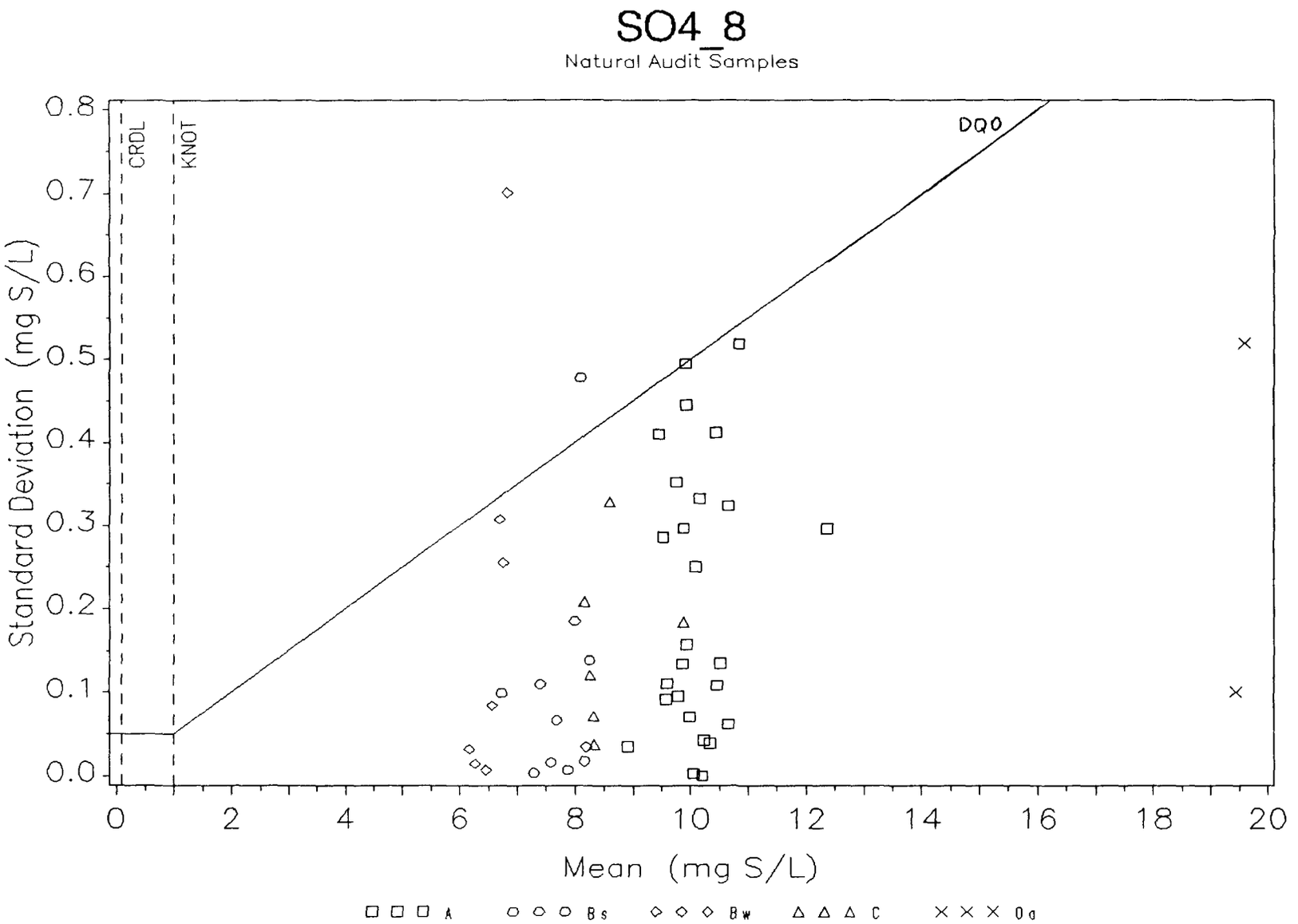


Figure 3-73. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_8.

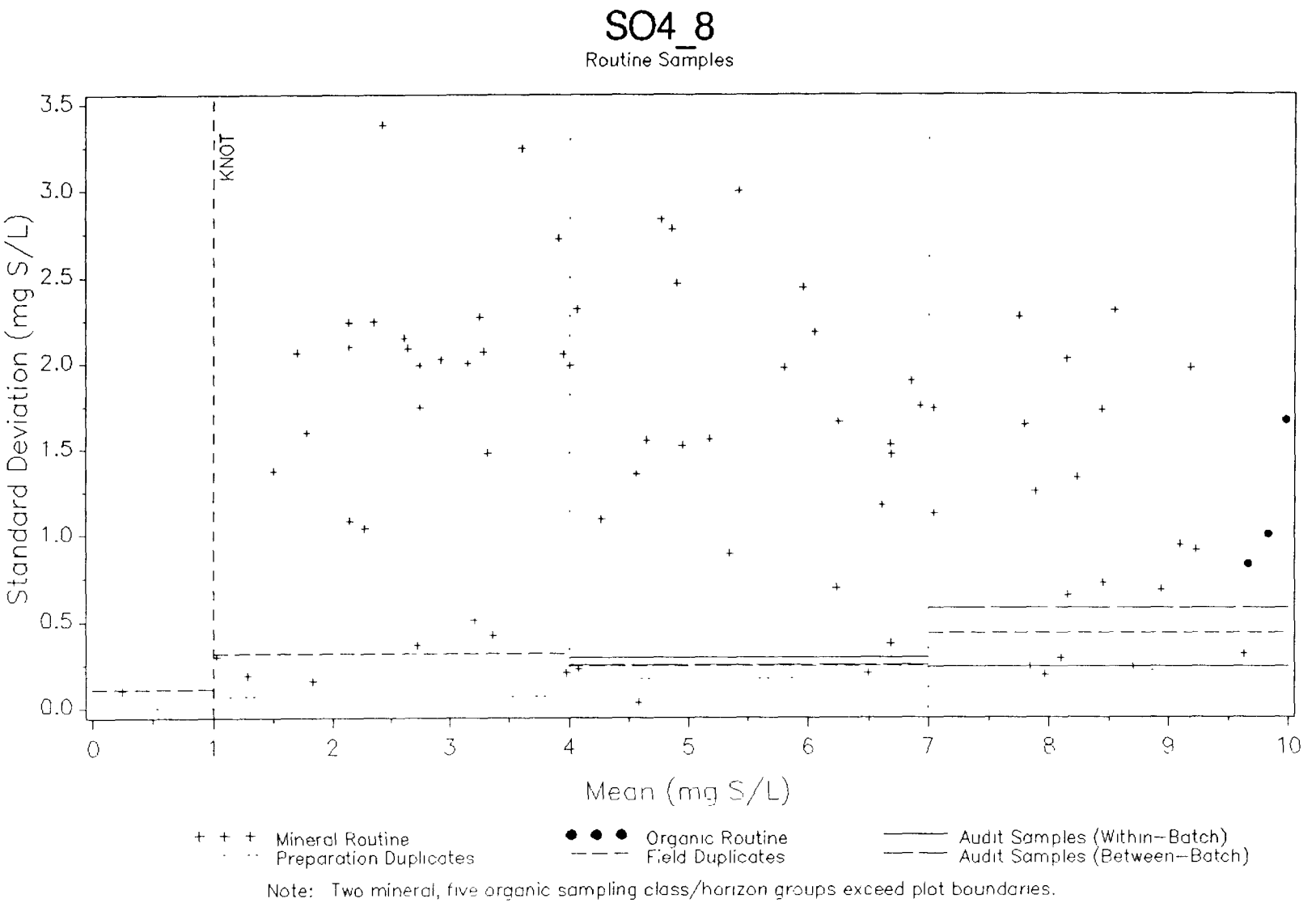


Figure 3-74. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_8.

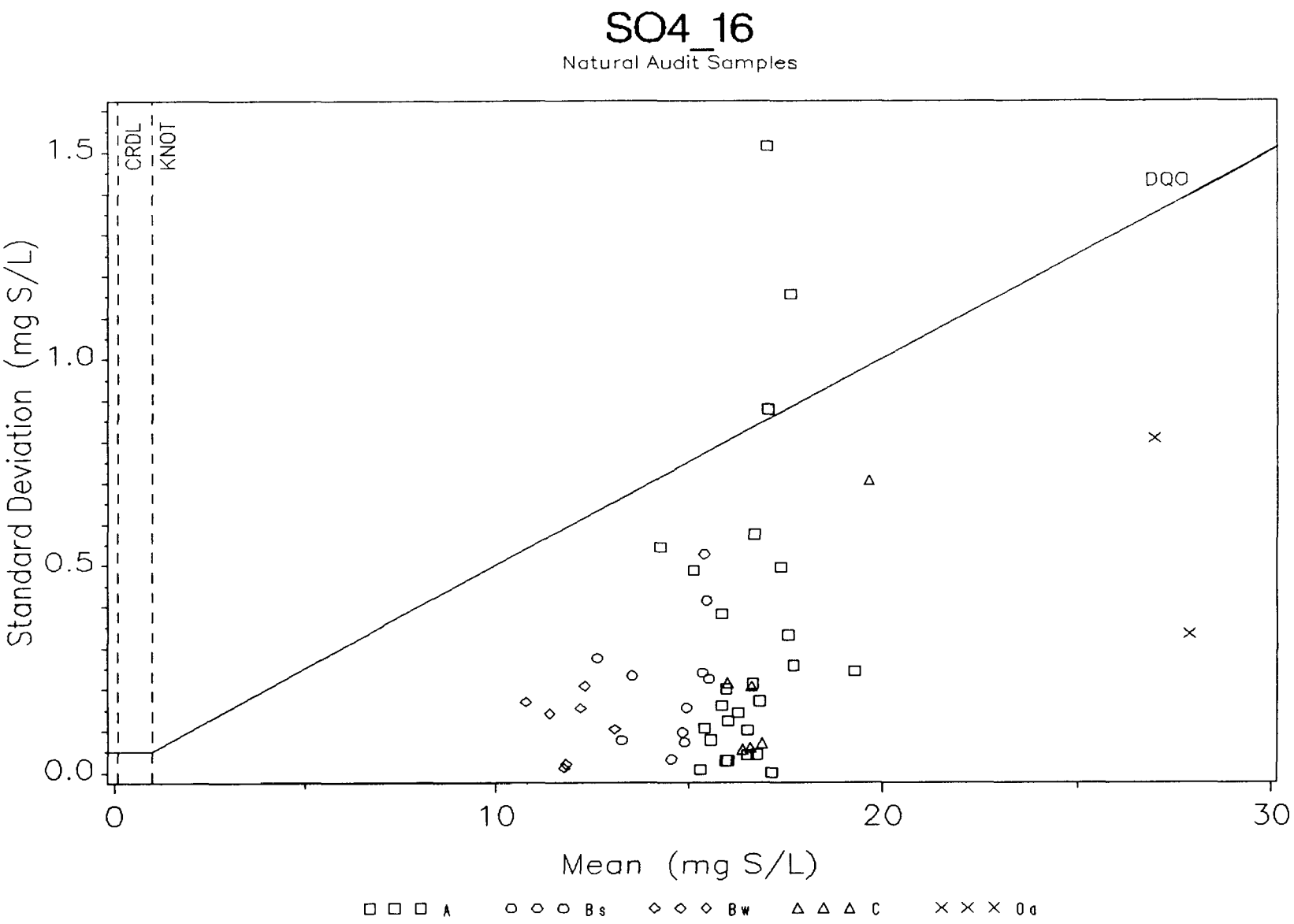


Figure 3-75. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_16.

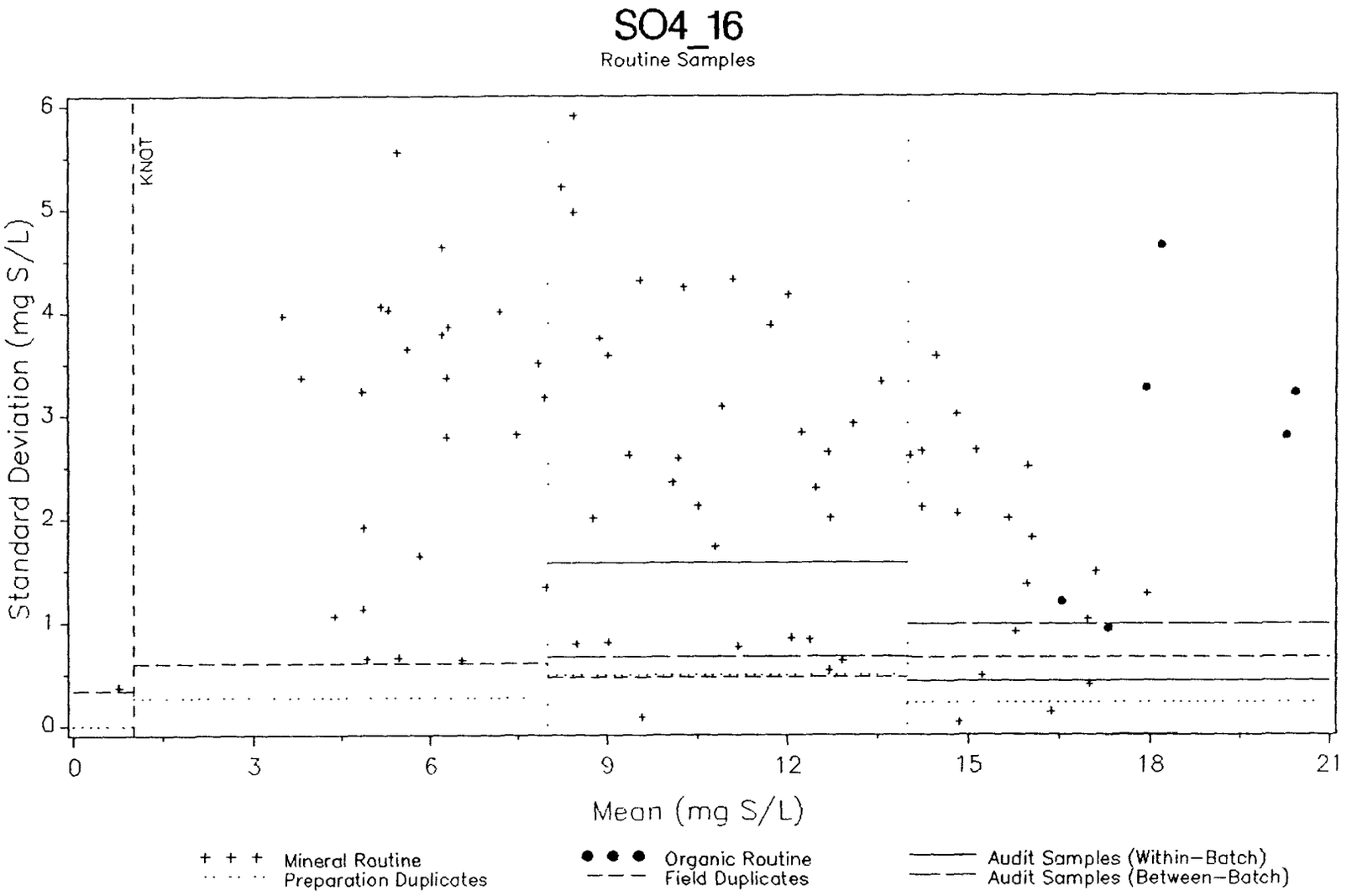


Figure 3-76. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for S04_16.

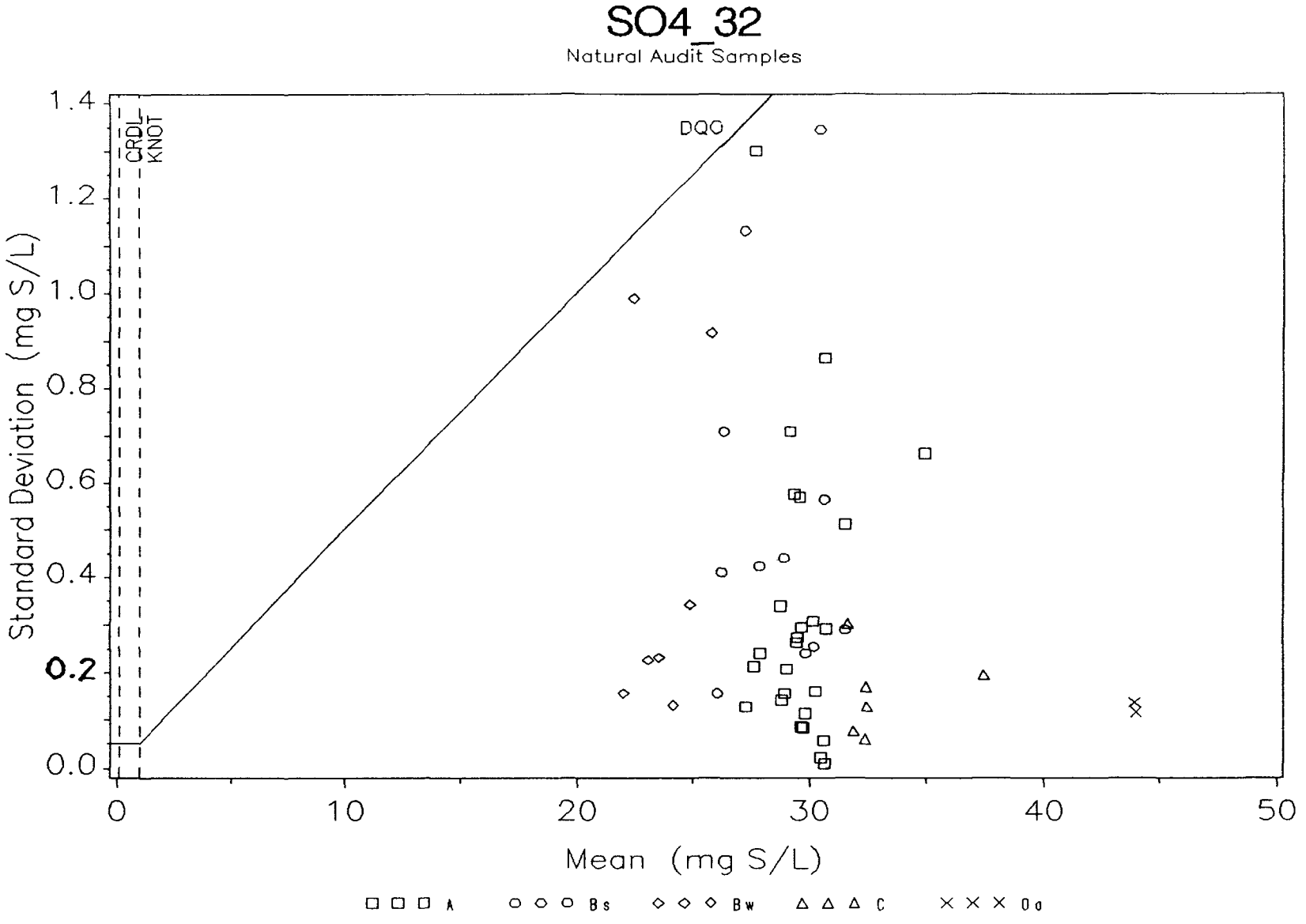


Figure 3-77. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for SO4_32.

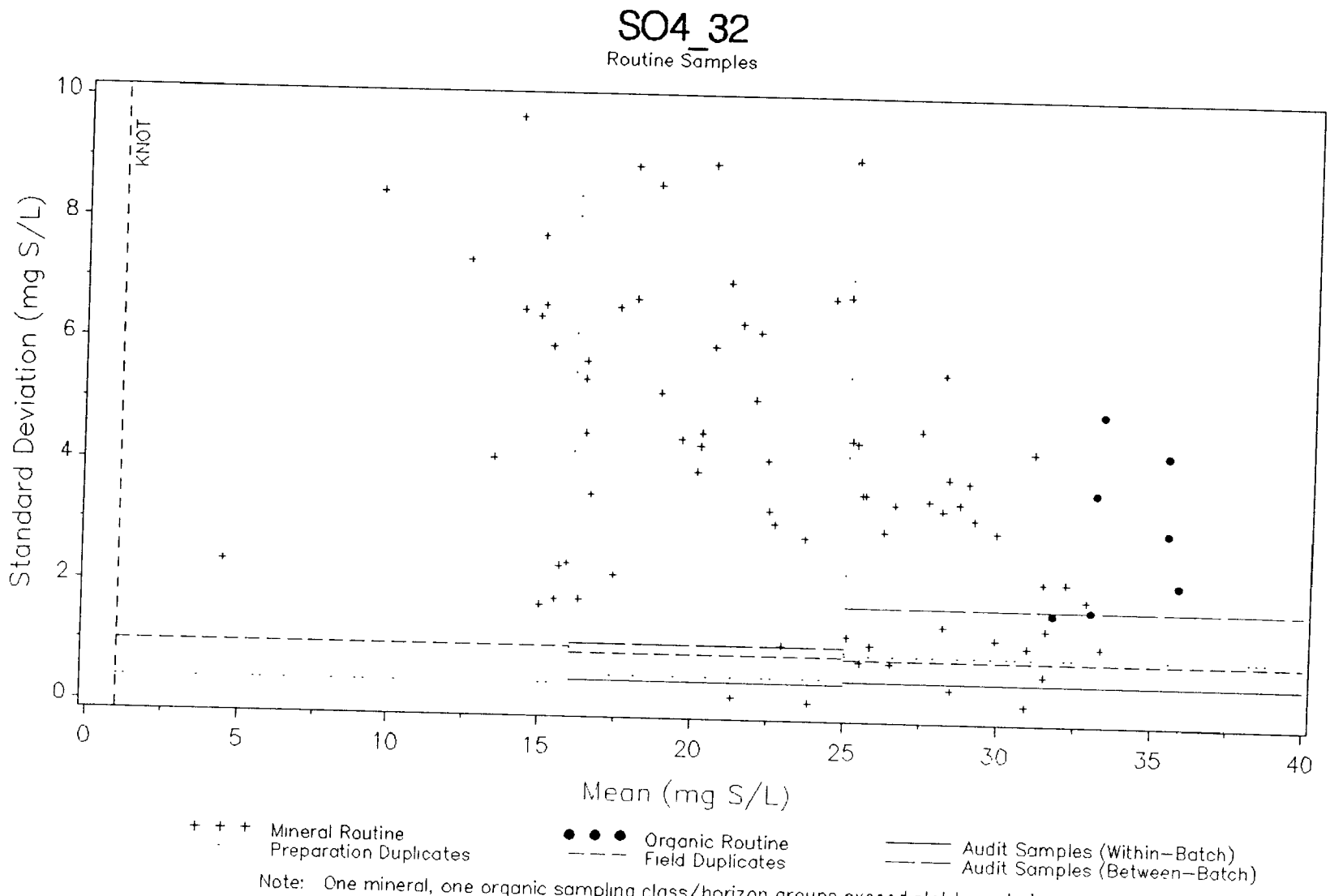


Figure 3-78. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SO4_32.

Figures 3-63 through 3-78 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Total Carbon, Nitrogen, and Sulfur

Table 3-10, Figures 3-79 through 3-84

The analytical within-batch precision DQOs were satisfied for total carbon, nitrogen, and sulfur except for N_TOT data above the knot (see Table 3-10). The preparation duplicates and field duplicates also met the analytical DQOs for the lower tier but not the upper tier. A general pattern of increasing standard deviation with increased sources of confounded error was maintained.

Figures 3-79 through 3-84 are plots of the audit sample data in relation to the DQOs and of the routine sample data in relation to the QA samples. Supplemental information relating to the delta and proportion values is presented in Appendix C, and the identification of inordinate data values is presented in Appendix D.

Table 3-10. Achievement of Data Quality Objectives for Analytical Within-Batch Precision of Total Carbon, Nitrogen, and Sulfur

		Below the knot[b]					Above the knot[b]				
					Pairs>DQO					Pairs>DQO	
Data set[a]	Parameter	df	SD	DQO	n	%	df	RSD	DQO	n	%
AS	C_TOT	6	0.0194	0.05			44	8.5%	15%	2	4.5
	N_TOT	8	0.0023	0.01			42	13.3%	10%	11	26.2
	S_TOT	48	0.0045	0.01	2	4.2	.	.	10%	.	.
PD	C_TOT	7	0.0552		2	28.6	19	20.9%		6	31.6
	N_TOT	20	0.0200		6	30.0	5	12.4%		3	60.0
	S_TOT	22	0.0067		2	9.1	.	.		.	.
FD	C_TOT	22	0.0335		4	18.2	82	40.8%		27	32.9
	N_TOT	72	0.0172		17	23.3	32	23.5%		13	41.9
	S_TOT	99	0.0116		4	4.0	1	79.2%		1	100.
S/H	C_TOT	106	0.1191				503	85.4%			
	N_TOT	434	0.0373				170	69.6%			
	S_TOT	609	0.0376				.	.			

[a] AS = Audit samples; PD = Preparation duplicates; FD = Field duplicates; S/H = Sampling class/horizon routine samples.

[b] Standard deviation and RSD data in reporting units and percent, respectively, for mineral soil samples below and above the knot point, 0.33 weight percent for carbon and 0.10 weight percent for nitrogen and sulfur; a dot signifies a lack of data occupying that range.

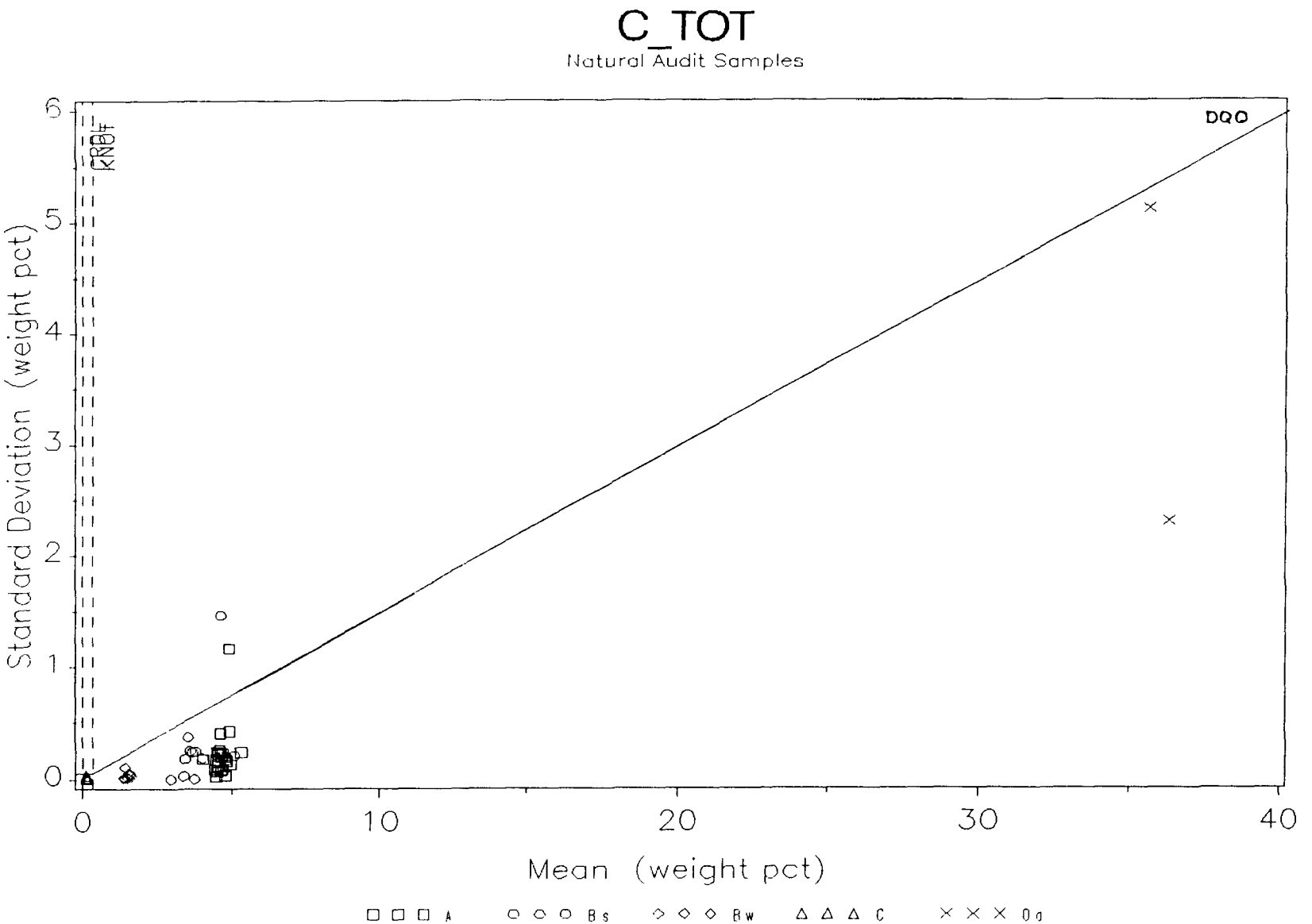


Figure 3-79. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for C_TOT.

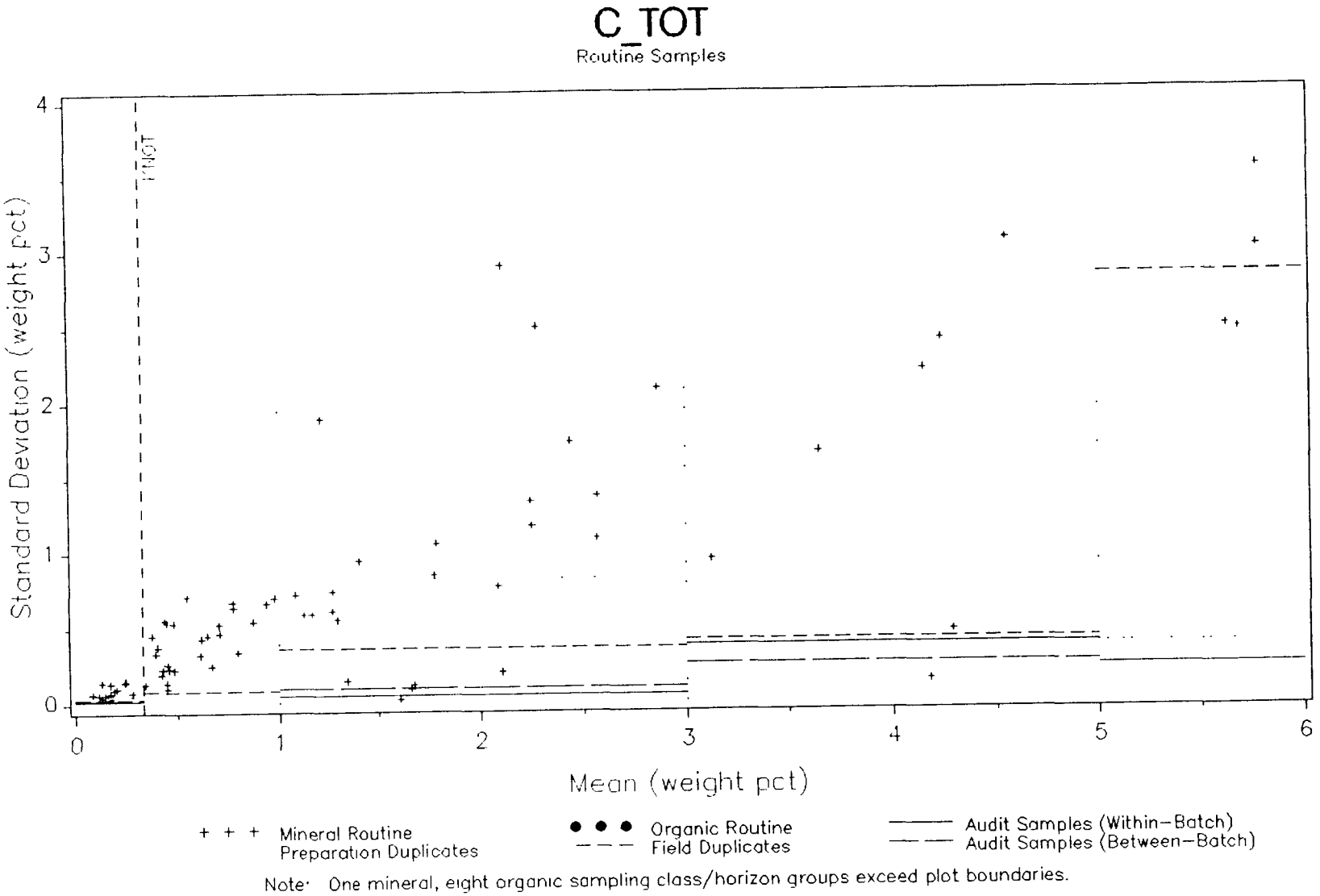


Figure 3-80. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for C_TOT.

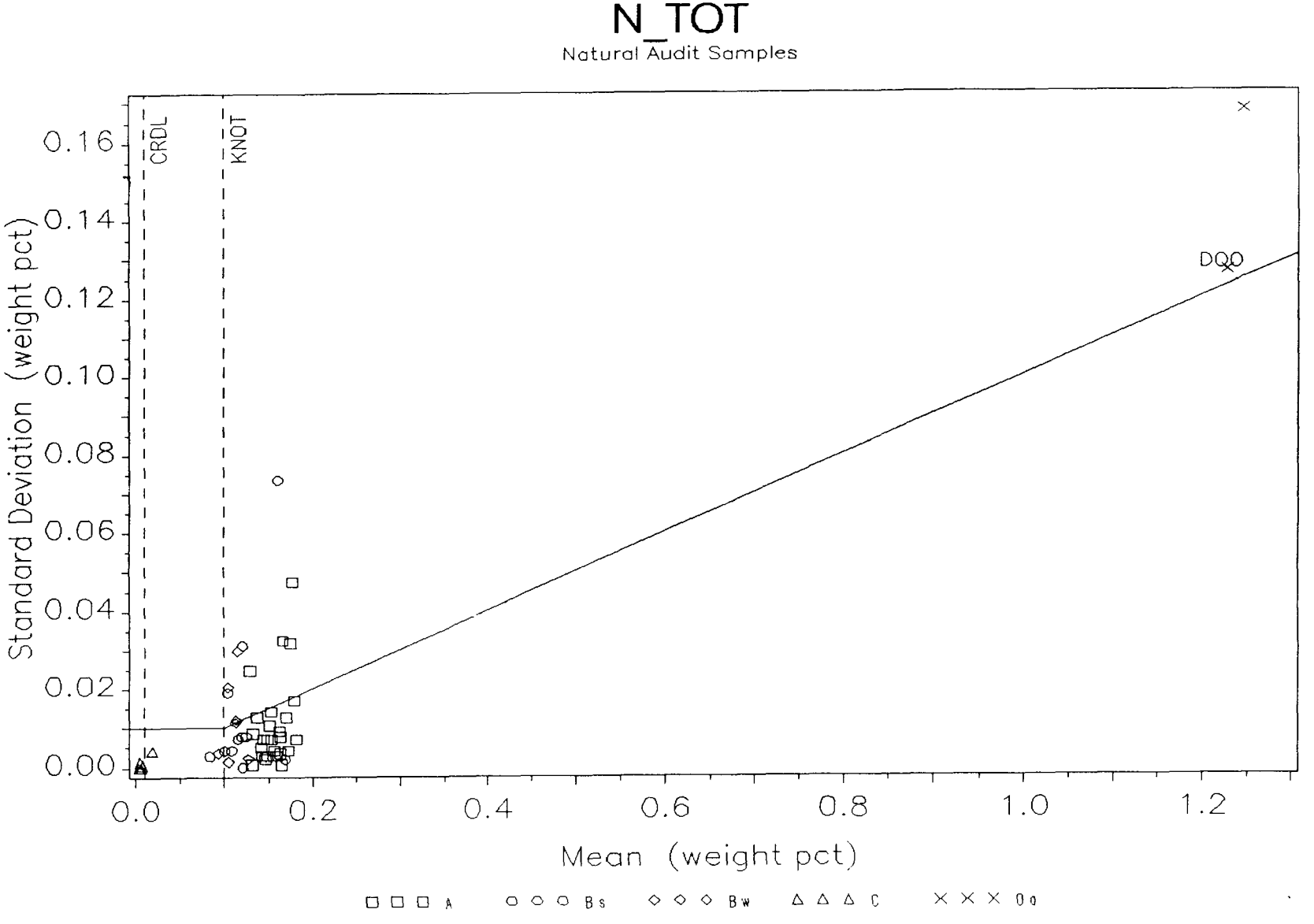


Figure 3-81. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for N_TOT.

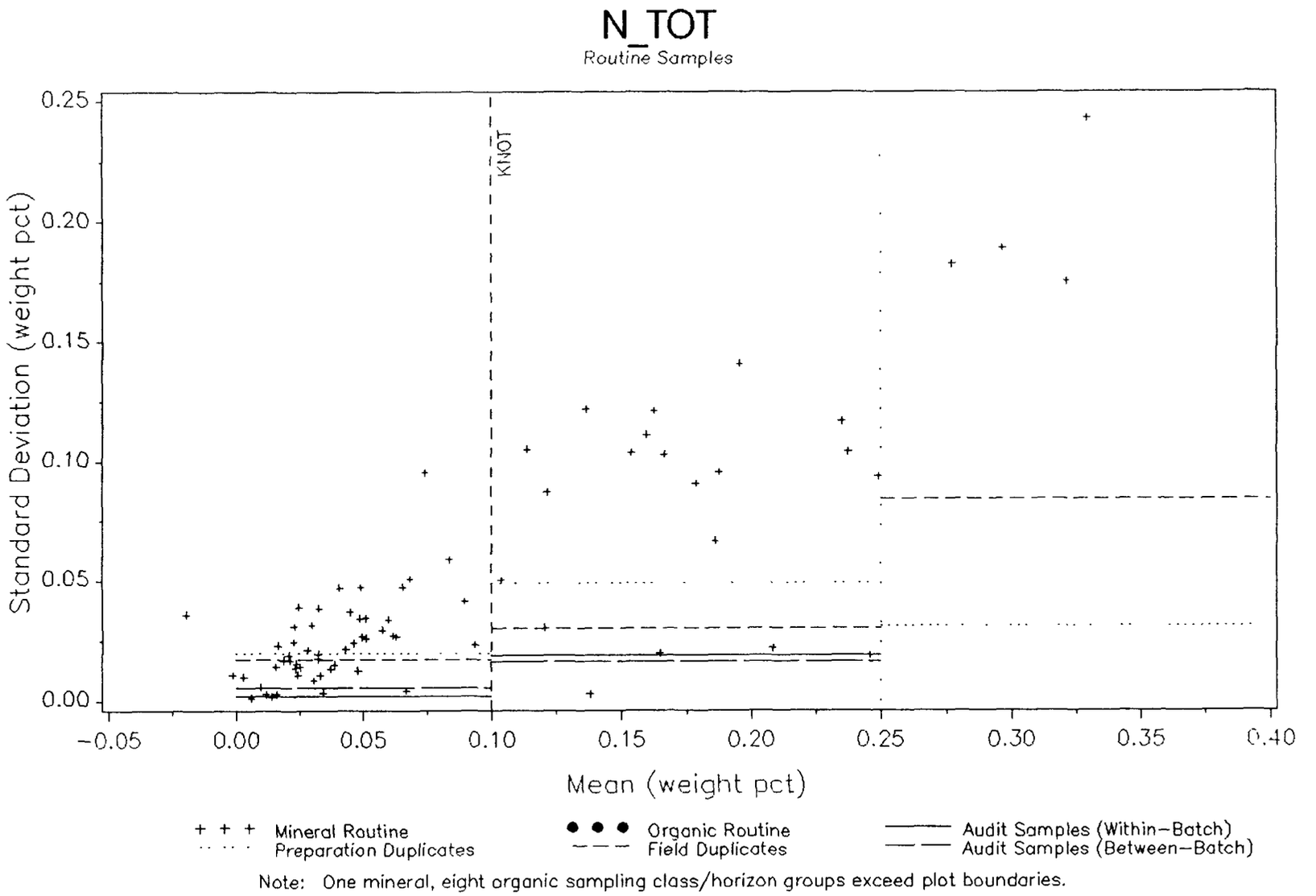


Figure 3-82. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for N_TOT.

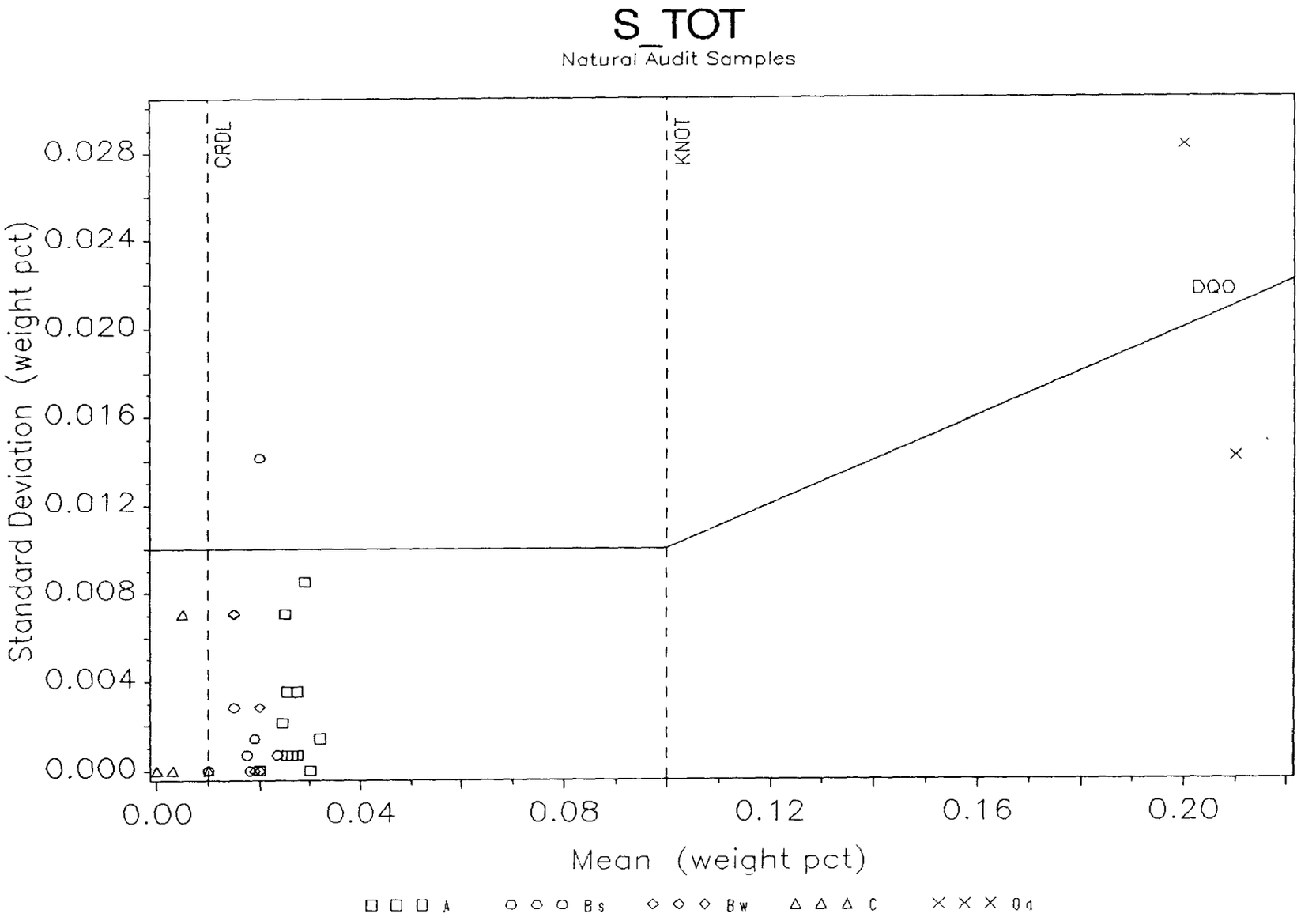


Figure 3-83. Range and frequency distribution of the natural audit samples and their relation to achievement of the analytical within-batch precision objective for S_TOT.

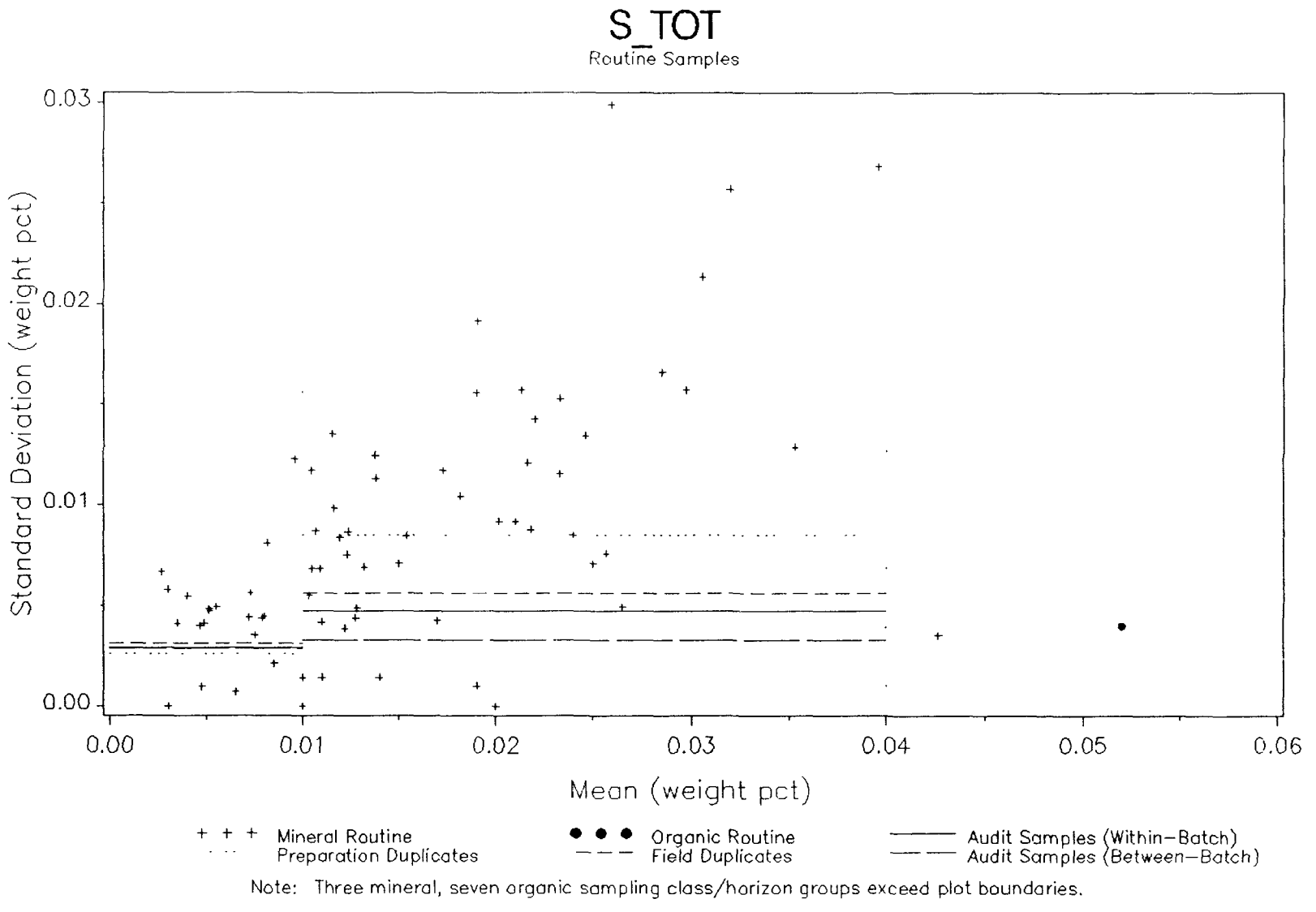


Figure 3-84. *Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for S_TOT.*

Accuracy (Interlaboratory Differences)

The following description of interlaboratory differences focuses on: (1) the significant differences among the analytical laboratories by audit sample horizon type, (2) the relative differences and rank of increasing difference among the laboratories using pooled data for the audit samples, and (3) the relative differences among the audit samples using pooled data from all of the laboratories.

Significant Differences Among Laboratories

Table 3-11 shows the laboratories which were significantly lower (at the 0.05 and 0.01 significance levels) than the other laboratories using Scheffe's pair-wise multiple comparison test for the analytical parameters.

For the A horizon audit sample, laboratory differences were highly significant for 19 parameters and significant for 11 parameters. For the physical parameters, Laboratory 2 showed the greatest number of significant differences. For the sulfate parameters, Laboratory 1 showed the greatest number of significant differences.

For the Bs horizon audit sample, laboratory differences were highly significant for four parameters and significant for eight parameters. All of these cases involved differences between Laboratory 1 and Laboratory 2.

For the Bw horizon audit sample, laboratory differences were highly significant for seven parameters and significant for nine parameters. A majority of the cases involved Laboratory 2.

For the C horizon audit sample, laboratory differences were highly significant for only two parameters and significant for five parameters. An additional five parameters showed significant differences. A majority of the cases involved Laboratory 3.

Overall, the laboratories were less consistent with their analysis of the physical parameters. In terms of sample type, the laboratories were less consistent for the A audit sample, followed by the Bw, Bs, and C samples, respectively.

Relative Differences and Ranking of Laboratories

Table 3-12 shows the relative difference as percent and the rank of increasing relative difference for each of the laboratories pooled for the A, Bw, and C audit samples. The table also shows the mean differences for all laboratories combined for each audit sample type.

For the physical parameters, SP_SUR showed the highest interlaboratory differences followed by VCOS, while CLAY showed the lowest differences. Laboratory 2 showed the highest differences overall for the 12 parameters in this group. For soil pH, the laboratory differences were consistently very low.

For the CEC parameters, Laboratory 1 was consistently lower than the other laboratories. For the exchangeable acidity parameters, Laboratory 2 was consistently lower than the others. For the iron and aluminum in the pyrophosphate and acid oxalate extracts, Laboratory 1 was consistently lower than the other laboratories. For iron and alimimum in citrate dithionite, Laboratory 2 was consistently lower than the others.

For the extractable sulfate parameters, Laboratory 2 showed the lowest differences. For the sulfate isotherm parameters, all laboratories showed low relative differences. For the elemental analysis of carbon, nitrogen, and sulfur, the laboratories were more consistent for C_TOT, followed by N_TOT and S_TOT, respectively.

For the 43 parameters used in determining laboratory differences, the rankings showed that Laboratory 1 had the lowest differences over all parameters, with 19 first-place rankings (43 percent) and 9 third-place rankings (21 percent).

Mean Differences Among the Audit Samples

The laboratories showed the lowest differences overall on the Bs audit sample for the physical parameters, pH, CEC, acidity, and iron and aluminum. The laboratories showed the highest differences overall for the C audit

Table 3-11. Significant Interlaboratory Differences

Parameter[a]	Audit horizon[b] A	Bs	Bw	C
SP_SUR	2 < 1,3 **			
SAND	1,3 < 2 **		1,3 < 2	
COS			2 < 1,3	
MS			2 < 3 **	
FS	1 < 2,3 **	1 < 2	1 < 2,3 **	
VFS	3 < 2 **		3,1 < 2 **	
SILT	2 < 3,1 **		2 < 1,3	
COSI			2 < 1,3	
FSI	2 < 1,3 **		2 < 1 **	
CLAY				3 < 1,2 **
PH_H20	1,2 < 3		1 < 3	1 < 3
PH_002M	1,2 < 3	2 < 1	2 < 3	1 < 3
PH_01M	1,2 < 3 **	2 < 1 **	2 < 3	
CA_CL	2 < 3	2 < 1 **		
MG_CL	3 < 1			
CA_OAC	1,2 < 3 **		2 < 3	
MG_OAC	2 < 1		2 < 1 **	
CEC_CL	1,2 < 3 **		1,2 < 3 **	
CEC_OAC	2 < 1 < 3 **	2 < 1 **	2 < 1,3	
AC_KCL	1 < 3			
AL_KCL	1 < 3,2 **			
CA_CL2	3,1 < 2	1 < 2		
MG_CL2	3 < 1 **	2 < 1		
K_CL2	2,3 < 1	2 < 1		
NA_CL2	2,3 < 1 **			
AL_CL2				2 < 3
AL_PYP				2 < 3
FE_AO	1,3 < 2	1 < 2		
AL_AO	1,3 < 2 **		3 < 2	
FE_CD	1,2 < 3 **			
AL_CD	1 < 2,3 **	1 < 2 **		2 < 3
S04_H20	3 < 1,2 **			
S04_P04	1 < 2			
S04_2	1 < 2 **			
S04_4			1,2 < 3 **	
S04_8	1 < 2 **			
S04_16	1 < 2 **	1 < 2		
S04_32	1 < 2 **			
C_TOT		2 < 1		
N_TOT				2,3 < 1 **
S_TOT	3 < 2			

[a] No significant differences were reported for MOIST, VCOS, K_CL, NA_CL, K_OAC, NA_OAC, AC_BACL, FE_CL2, FE_PYP, and SO4_0.

[b] A double asterisk denotes a highly significant difference at the 0.01 significance level; differences not evaluated for the Oa horizon audit sample.

Table 3-12. Relative Difference and Rank by Laboratory and Mean Laboratory Difference by Audit Sample Type

	Difference (%) Laboratory			Rank Laboratory			Difference (%) Audit Sample			
Parameter[a]	1	2	3	1	2	3	A	Bs	Bw	C
MOIST	3.0	0.6	2.8	3	1	2	2.3	0.4	1.3	2.8
SP_SUR	10.5	25.0	17.9	1	3	2	18.0	2.1	17.2	27.0
SAND	4.3	5.3	1.5	2	3	1	3.7	0.2	11.5	1.3
VCOS	9.5	12.8	17.4	1	2	3	12.6	7.1	5.9	20.1
COS	2.9	6.1	8.7	1	2	3	1.7	4.6	8.6	9.3
MS	2.5	1.4	3.1	2	1	3	1.5	0.3	3.0	3.1
FS	7.3	4.1	3.2	3	2	1	5.7	5.4	7.5	2.5
VFS	4.9	10.8	8.8	1	3	2	5.2	3.0	27.4	6.5
SILT	8.6	10.7	3.1	2	3	1	8.7	0.3	4.8	31.8
COSI	8.2	11.0	3.9	2	3	1	8.5	0.9	4.4	34.3
FSI	9.0	10.4	3.6	2	3	1	8.9	2.9	6.0	23.8
CLAY	0.9	1.1	1.3	1	2	3	0.6	12.3	2.6	100.0
PH_H2O	0.9	0.2	1.1	2	1	3	0.5	0.3	0.6	1.6
PH_002M	0.9	1.0	2.1	1	2	3	1.1	1.3	0.7	2.6
PH_01M	0.5	0.9	1.7	1	2	3	1.0	0.9	0.7	1.6
CA_CL	3.5	13.8	12.4	1	3	2	9.5	18.0	10.0	14.8
MG_CL	4.8	1.8	7.0	2	1	3	3.9	10.1	5.0	17.0
CA_OAC	1.7	18.2	22.4	1	2	3	12.0	5.5	13.8	39.3
MG_OAC	6.5	5.7	1.0	3	2	1	3.6	1.8	15.4	10.7
CEC_CL	7.8	18.5	30.1	1	2	3	15.7	6.8	31.8	28.6
CEC_OAC	4.6	16.7	14.6	1	3	2	11.2	4.2	15.2	25.1
AC_KCL	10.8	2.2	13.0	2	1	3	5.5	2.0	20.7	60.9
AC_BACL	0.5	4.9	5.7	1	2	3	3.0	0.3	4.1	46.8
AL_KCL	9.3	5.4	3.9	3	2	1	5.8	2.9	8.0	25.0
CA_CL2	8.2	12.3	8.5	1	3	2	13.1	5.8	5.1	5.8
MG_CL2	12.7	3.6	10.9	3	1	2	8.7	8.5	8.8	15.8
FE_PYP	6.5	2.3	5.3	3	1	2	4.1	0.1	5.7	13.0
AL_PYP	1.6	6.8	7.4	1	2	3	5.2	2.6	4.3	18.2
FE_AO	5.7	12.0	7.8	1	3	2	6.7	8.1	9.6	35.3
AL_AO	4.8	14.0	11.0	1	3	2	10.7	1.0	8.6	12.7
FE_CD	8.8	6.4	17.0	2	1	3	11.2	5.6	6.7	25.3
AL_CD	11.9	2.1	11.5	3	1	2	7.5	6.4	9.5	19.5
SO4_H2O	6.5	1.0	7.9	2	1	3	5.0	2.2	3.6	14.1
SO4_PO4	4.6	3.6	2.2	3	2	1	3.0	23.2	4.3	17.4
SO4_0	3.5	4.8	2.0	2	3	1	3.4	5.9	3.3	6.3
SO4_2	5.0	5.1	0.7	2	3	1	4.2	2.8	2.2	0.9
SO4_4	1.9	2.8	2.1	1	3	2	2.0	0.6	6.0	0.3
SO4_8	3.5	4.6	1.6	2	3	1	3.6	3.0	1.4	3.4
SO4_16	4.1	4.7	1.3	2	3	1	3.4	3.9	4.1	3.1
SO4_32	4.0	3.5	1.6	3	2	1	2.8	2.4	4.3	3.0
C_TOT	1.3	0.8	1.6	2	1	3	1.0	8.9	3.8	3.5
N_TOT	3.6	4.0	7.4	1	2	3	4.2	8.3	5.7	48.4
S_TOT	6.8	9.2	8.3	1	3	2	4.3	8.1	4.8	42.9

[a] Concentrations were too low to estimate interlaboratory differences for K_CL, NA_CL, K_OAC, NA_OAC, K_CL2, NA_CL2, FE_CL2, and AL_CL2.

sample. The laboratories performed well on all audit samples for the sulfate isotherm parameters.

Over all the audit samples, the laboratories showed the greatest differences for SP_SUR, VCOS, CEC_CL, CEC_OAC, FE_CD, and S04_PO4.

Representativeness

All pedons sampled were within the range of morphological characteristics outlined in their respective sampling classes (Coffey et al., 1987), hence, the DQO for representativeness of the field sampling was satisfied.

The homogenization and subsampling procedures at the preparation laboratories produced representative analytical soil samples of known and accepted quality (Haren and Van Remortel, 1987). More information on this characteristic of the data can be found in the precision discussions of this report, where assessments of the preparation duplicates are made.

Histograms of the range and frequency distribution of the routine samples, field duplicates, preparation duplicates, and natural audit samples for each of the parameters are presented in Appendix F. The field duplicates and preparation duplicates generally were representative of the range and frequency distribution of analyte concentrations for the routine samples. The only exceptions were the SP_SUR, COSI, FE_CL2, AL_CL2, and S_TOT parameters (see Table 3-13). A more rigorous selection method for the preparation duplicates, relative to that of the DDRP Northeastern Soil Survey, was responsible for good representativeness in the PD data set. The audit samples generally were representative of the range of data from the routine samples.

Completeness

Soil sampling protocols specified the sampling of all of the designated pedons. A total of 110 pedons were sampled of the 114 pedons initially selected, resulting in 96.5 percent completeness (Coffey et al., 1987). Although this does not fully satisfy the DQO for sampling completeness, sufficient pedons were sampled to enable estimates and conclusions to be drawn from the data.

As specified in the protocols, each batch of samples sent to a analytical laboratory contained one preparation duplicate pair. The

Table 3-13. Summary of Significant Differences in the Distribution of the Field and Preparation Duplicates Relative to the Routine Samples

Parameter	Data set[a]	n	Mean	p05[b]	p50[b]	p95[b]	KS-stat[c]
SP_SUR	RS	703	34.33	9.48	30.93	74.47	---
	FD	102	35.26	8.70	34.71	67.55	0.151
COSI	RS	703	9.88	3.50	8.70	19.52	---
	FD	102	10.83	3.60	9.85	20.62	0.147
FE_CL2	RS	747	0.01	0.00	0.00	0.02	---
	FD	106	0.01	0.00	0.00	0.02	0.574
	PD	26	0.00	0.00	0.00	0.01	0.584
AL_CL2	RS	747	0.05	0.00	0.01	0.15	---
	FD	106	0.04	0.00	0.01	0.13	0.194
S_TOT	RS	747	0.02	0.00	0.01	0.07	---
	FD	106	0.02	0.00	0.01	0.06	0.270

[a] RS = routine samples, FD = field duplicates, PD = preparation duplicates.
[b] p05, p50, and p95 are the 5th, 50th (median), and 95th percentiles by data set.
[c] Kolmogorov-Smirnov test; statistics are significant at the 0.05 level for the critical value: FD_RS = 0.141, PD_RS = 0.271.

requested soil analyses and sample processing tasks were performed on 100 percent of the bulk samples and clods received by the preparation laboratories (Haren and Van Remortel, 1987).

The number of AO and JJ flags (denoting missing data or insufficient sample for analysis, respectively) assigned to the 748 routine samples was used to assess analytical completeness of the verified data base. There was only one missing sample in the data base and all of the analyses were performed on the remaining 747 samples; hence, the analytical laboratories achieved a 99.9 percent completeness level (see Appendix B).

Five levels of confidence, ranging from 0 to 4, were used to segregate and classify data in the validated data base. A level of confidence of 2 or less, i.e., less than two major flags or less than one major and two minor flags assigned per sample, was used to assess completeness in the validated data base (see Appendix B). The DQO for analytical completeness of 90 percent or higher was satisfied for all of the parameters. The CEC parameters were the only analytes to fall below a completeness level of 95 percent for the validated data base.

Comparability

The entire verified data base was used for the assessment of data quality for both the Northeastern and SBRP reports because the indiscriminate use or non-use of flagged data was felt to be inappropriate for the purposes of quality assessment. The flags were applied in order to caution the data user that certain data points are suspect and may not be suitable for a particular type of data analysis. Data with levels of confidence of 0, 1, and 2 in the validated data base were used only for the assessment of analytical completeness.

Analytical data from an interlaboratory comparison study were recently received by EMSL-LV staff. The study is using data from the DDRP audit samples to compare analytical methods used in the two surveys to methods currently in use at 22 selected soil characterization laboratories throughout the United States and Canada. The results will be summarized in an upcoming report (Palmer et al., in preparation).

Comparison of Analytical and Preparation Methods

Because of significant differences in methods among private laboratories, the preliminary audit sample data provided by three independent referee laboratories prior to the initiation of the DDRP surveys could not be utilized to evaluate the quality of routine data. Sufficient audit sample data were available from the DDRP contract laboratory analyses, however, to provide an estimate of the audit sample composition. These data were used in the assessment of comparability, precision, and interlaboratory differences.

Initial difficulties were encountered in developing and evaluating the analytical methods prior to initiation of the DDRP surveys. As a result, there are certain instances where the methods actually used by the contract laboratories differ from those specified in the DDRP Analytical Methods Manual or in the individual laboratory solicitations. Approval for methods amendments was given only when it was determined by the QA staff that these changes would not significantly affect the analytical results, e.g., changing from a 0.20-micron filter to a 0.45-micron filter. Methods amendments were recorded in an operations log book by QA staff but did not always result in an official EPA contract modification.

During the Northeastern survey, analytical methods for two parameters were changed sufficiently to warrant reanalysis of any previously analyzed samples. The laboratories were contracted to reanalyze all of their samples for AL_KCL by using a method which employed a different acidification procedure. Two of the laboratories also were contracted to adjust the soil:solution ratio for PH_002M and to reanalyze all of the samples; the third laboratory already had been using the amended ratio. Hence, reanalyses have corrected all data significantly affected by methods amendments which occurred as the survey progressed (Byers et al., 1988).

Identical soil preparation methods were used in preparing soil samples for the two surveys. The protocols were revised for clarity in the SBRP survey but the methods remained

comparable. The procedure for selecting a preparation duplicate for each batch was refined for SBRP, resulting in better representativeness of the preparation duplicates.

Comparison of Field Sampling Methods

As a result of information gathered from the Northeastern survey exit meeting, the field sampling protocols were revised to include clarifications of sampling procedures and contamination control for the SBRP survey. It was discovered that the field duplicates in the Northeastern survey were sampled by two different methods, i.e., some crews placed alternate portions of soil from the same horizon into separate bags (the correct method) while other crews collected twice the normal amount of sample, performed a simple homogenization, and split the sample. The former method is meant to determine sampling variability, hence, the data from samples derived by this method are expected to be more variable than the data derived by the latter method. Because of the inconsistent application of the method, the variances of the Northeastern field duplicates tend to fluctuate among pedons. Field duplicates for the SBRP sites were sampled using the correct method. Nevertheless, overall within-batch variability was expected to be greater in the SBRP than in the Northeastern survey because of the additional sampling variability error contained in the field duplicates that were sampled using the correct protocol. This does not mean that the routine data between region is not comparable, as a similar methodology was used for routine soil sampling in each survey. It does suggest, however, that measurement error in the Northeastern survey may have been underestimated. The field sampling audit team did not report any deviations from the sampling protocols that would compromise the integrity of the routine data.

Comparison of Audit Sample Distribution

Although the SBRP was a less extensive survey in terms of the total number of samples collected, two pairs of natural audit samples were placed in each batch in contrast to one pair per batch in the Northeastern survey. This accounts for the similar total number of audit samples (104 versus 112, respectively), even though the number of batches in each survey varied widely. The soil for each audit horizon type in both regions came from the same bulk audit sample, hence, data for each subsample can be compared between regions for any given parameter. Significant differences could then be attributed to differing amounts of measurement error, e.g., differential laboratory bias. Since there were four analytical laboratories in the Northeastern survey and only three of those four in the SBRP survey, Laboratory 4 cannot be regionally compared. Laboratory 3 did not analyze the A or C audit samples in the Northeastern survey or the Bs audit horizon in the SBRP survey, hence, comparisons for this laboratory can be made only with data from the Bw and Oa audit horizons.

Section 4

Conclusions and Recommendations

Data Verification

Verification of Data Packages

A number of improvements can be made in the verification procedure. Principal among them is the development of a computerized data entry and verification system that will calculate all of the final data values and produce a list of flags and data entry errors. This will provide a much faster turnaround time for submission of data packages and completion of the data review phase and confirmation/reanalysis requests. All raw data needed to calculate final values could be entered and a calculation program could be run. This would facilitate the rapid identification of entry errors and ultimately reduce the amount of reanalysis needed. A link between the laboratories and the quality assurance staff should be established that will enable the transfer of preliminary and final data. The verification program should be designed to evaluate the quality control checks and other contractual requirements, thereby inducing the laboratories to assume much of the responsibility for identification and correction of errant data.

Evaluation of the blind audit samples should also be made part of the verification system. However, this portion of the system would be accessible only to the quality assurance staff. This evaluation would be used in conjunction with the quality control and summary checks to determine the acceptance of batches from the laboratories.

Internal Consistency

The internal consistency checks provided a meaningful check of routine data for each analytical parameter. Errors were discovered that might have otherwise gone unnoticed. The checks were performed during the final weeks of data verification for the SBRP survey. Outliers determined by the internal consistency computer program were checked only for transcription errors. The program generated outliers consisting of approximately 1 percent of the total number of data values for each parameter. Of these outliers, approximately 10 percent, i.e., 0.1 percent of the total data values, were found to be in error. A few parameters did contain a relatively large number of outliers which, after correction, improved the quality of data. There were some parameters that did not correlate well with any of the other parameters. Although the highest and lowest one percent of these values for these parameters were reviewed, a better procedure for checking these values should be developed.

The internal consistency checks could provide useful information during the earlier stages of verification. By using these data to influence requests for reanalysis, the checks could become an integral part of the verification process. Difficulties with methodology and data reporting become apparent when significant numbers of outliers are found for specific batches.

If it can be determined that parameter correlations are comparable among regions, then single batches of data from new regions can be incorporated into an overall data file and reviewed to distinguish outliers. However, if the correlations do not compare across regions, a statistically significant population of data must be collected from the region of concern before suspect data points from individual batches can be viewed as outliers.

A method should be developed that will identify outlying, but confirmed, data points which tend to distort the data quality assessment. In addition to the present internal consistency checks, a suitable statistical analysis should be selected to identify which data points are having a disproportionate influence on the overall data. These data points and any associated routine data could be highlighted with a special flag for the benefit of data users.

Data Quality Objectives

Detectability

Considerable effort was expended during the course of this survey to evaluate and improve the detectability of various parameters. In particular, significant improvement was obtained for the exchangeable cations. It is recommended that attention be given to improving detectability in future surveys. Additional methods research is essential to this effort.

Data quality objectives for detectability were not set at the start of the survey with regard to system detection limits. Although instrument detection limits are an integral part of the detectability issue, the actual detection limit that can be applied to the final data set is the system detection limit and not the instrument detection limit. It is recommended that both types of limits be addressed in the data quality objectives for future surveys.

It has been noted that "soil blanks" were not used in this survey, hence, it was difficult to calculate system detection limits or to identify when contamination may have occurred. Although some consideration has been given to the development of a soil blank sample, it is recommended that low concentration audit samples, entered into the system during the sampling phase, be utilized as substitutes for blanks. These samples would serve not only to identify contamination problems and allow for the calculation of detection limits, but could also be used to estimate system precision and accuracy as well as provide additional quality control benefits.

Precision

It was necessary to investigate why the laboratories had difficulty in satisfying certain precision objectives, e.g., the objectives might be unreasonably restrictive, the laboratories had problems with the methods, or there were sample preparation problems. The disproportionate effect of inordinate data points on the estimates were also assessed. The precision results show that the analytical precision objectives for certain parameters were not satisfied, including the particle size parameters, potassium and cation exchange capacity in ammonium chloride, potassium in ammonium acetate, the extractable cations in calcium chloride, iron in acid oxalate, phosphate-extractable sulfate, the sulfate-zero isotherm, and total nitrogen.

Table 4-1 is a summary of the overall achievement of the data quality objectives for analytical within-batch precision. A proportion, or precision index, was determined for each group of parameters by pooling and weighting the standard deviations across the parameters within the group for the lower and upper tiers and dividing by their respective data quality objectives. These values were then summed and divided by the total degrees of freedom for the group. A precision index exceeding 1.0 (denoted by an asterisk in the table) indicates that the precision estimate for this parameter group did not meet the "overall objective" when viewed from the perspective of the entire concentration range. This approach helped to identify which parameter groups should undergo further quality assurance emphasis in order to redefine the objectives for future surveys or to reassess the analytical procedures for the affected parameters.

The lack of a two tiered data quality objective for the particle size parameters might explain the relatively high variability, although the objective could be somewhat restrictive as well. Also, the nature of the hydrometric/sieve/pipet method, such as temperature effects, may have caused variability in particle size percentages. Interlaboratory differences for these parameters were relatively high. It is recommended that additional methods details be provided in order to lower the variability among laboratories.

Table 4-1. Precision Indices Based on Pooled Within-Batch Precision Estimates for Parameter Groups Across Concentration Ranges

Parameter group (parameters included)	Precision index
Particle size analysis (SAND, SILT, and CLAY)	1.93*
Soil pH (PH_: H2O, 002M, and 01M)	0.24
Exchangeable cations (CA_, MG_, K_, and NA_: CL)	0.77
" " (CA_, MG_, K_, and NA_: OAC)	0.55
Cation exchange capacity (CEC_CL and CEC_OAC)	0.83
Exchangeable acidity (AC_KCL, AC_BACL, and AL_KCL)	0.50
Extractable cations (CA_, MG_, K_, MG_, FE_, and AL_: CL2)	1.79*
Extractable iron and aluminum (FE_ and AL_: PYP, AO, and CD)	0.58
Extractable sulfate (SO4_: H20 and PO4)	1.09*
Sulfate isotherms (SO4_: 0, 2, 4, 8, 16, and 32)	0.80
Elemental analysis (C_TOT, N_TOT, and S_TOT)	0.72

In the future, two-tiered precision objectives should be defined for the extractable cations in calcium chloride. For potassium in ammonium chloride, one audit sample pair had high variability which expanded the imprecision for data above the knot. For cation exchange capacity, only a small amount of the data was below the knot and one-third of these data had high variability.

For phosphate-extractable sulfate, the laboratories exceeded the 10 percent objective by a considerable degree both below and above the knot, although this was not the case for water-extractable sulfate. This suggests that there were no problems with biological degradation over time, sample preparation, or sample extraction. However, there may have been a difficulty with the ion chromatography instrumentation, where inadequate separation of the sulfate and phosphate peaks may have occurred for the higher sulfate concentrations in the phosphate extraction. In addition, column loading could occur due to high phosphate concentrations in the extract. For total nitrogen, one laboratory exhibited significant interlaboratory differences which might explain variability in the data.

As expected, increasing sources of confounded data collection error led to increased standard deviations in the precision estimates. Of the 64 cases where the precision estimates below and above the knot for the preparation duplicates were compared to the analytical data quality objectives, only 18 cases exceeded the objective. This indicates that the preparation laboratories performed relatively well in subsampling the bulk soil samples. In many cases, the error estimates for the preparation duplicates were less than that for the audit samples. As a result, error in the preparation of the natural audit samples by QA staff often may exceed error in the preparation of the routine and duplicate samples by the preparation laboratories. For the field duplicates, 39 of the 64 cases exceeded the analytical objectives. Specific data quality objectives should be defined for system-wide measurement in future surveys, using data from the field duplicates. Also, as anticipated, the sampling class/horizon groups showed the highest levels of error due to population variability.

In summary, the preparation duplicates generally had slightly higher precision than the audit samples. This suggests that the quality assurance staff may have had more difficulty in homogenizing the 500-kilogram bulk audit samples as compared to the 5.5-kilogram routine samples homogenized at the preparation laboratories. This has implications for preparation of any future audit samples, but also reflects well on the subsampling procedure followed by the preparation laboratories. In addition, the relatively lower precision for the field duplicates suggests that the component of error from soil sampling is a large portion of the overall data collection error.

Accuracy (Interlaboratory Differences)

The approach taken for this report was to assess interlaboratory differences for the 51

analytical parameters by comparing mean values among the individual laboratories and mean values for the laboratories combined across audit samples. The lack of acceptable or "true" analytical values for the soil parameters prohibited the assessment of accuracy, hence, interlaboratory differences are used to describe the relative systematic error. Three basic comparisons were made: (1) the use of a statistical test to directly compare laboratories, (2) pooling of audit sample data (A, Bw, and C horizons) for each laboratory to compare and rank overall laboratory performance, and (3) pooling data from each laboratory for each audit sample (A, Bs, Bw, and C horizons) to compare laboratory performance by sample type.

For the 624 possible statistical configurations of the laboratories by parameter and by audit horizon type, there were 97 cases (52, 12, 24, and 9 cases for the A, Bs, Bw, and C horizons, respectively) where one laboratory was significantly different at the 0.05 significance level. Considering the number of pairs of audit samples analyzed by the laboratories (36, 40, and 24 pairs for Laboratories 1, 2, and 3, respectively), the number of significant differences attributed to each laboratory were similar (i.e., 36, 45, and 16). In this respect, each of the laboratories performed similarly when compared to the other two laboratories.

Of the 97 significant differences identified, 41 (42 percent) were highly significant at the 99 percent confidence level. A majority of those (25 of 41) were in the A horizon data. The data users should be aware of this when assessing data for specific parameters. Table 4-2 shows the mean laboratory difference for each laboratory and audit sample type for each parameter group or subgroup for 44 of the 51 parameters analyzed.

The lowest interlaboratory differences occurred in the soil pH parameter group. The laboratories showed the highest differences overall in the CEC parameters. The differences were also relatively high for the cations in calcium chloride and the cations in ammonium acetate. The mean interlaboratory differences across all parameters for Laboratories 1, 2, and 3 were 5.2, 6.8, and 8.2 percent, respectively.

The laboratories showed the highest differences on the C audit samples. The mean laboratory differences for the A, Bs, Bw, and C audit samples were 7.1, 5.5, 9.0, and 22.5 percent, respectively. The C audit sample generally had the lowest concentrations for most of the parameters. The lowest difference was in the Bw horizon for pH and the highest difference was in the C audit sample for exchangeable acidity. Overall, the laboratories

Table 4-2. Summary of Interlaboratory Differences by Laboratory and by Audit Sample Type

Parameters	n*	Interlaboratory difference (percent)						
		Laboratory			Sample type			
		L1	L2	L3	A	Bs	Bw	C
Specific surface	1	10.5	25.0	17.9	18.0	2.1	17.2	27.0
Sand & Silt fractions	7	7.0	8.9	6.0	7.0	3.0	7.9	18.7
Sand, Silt, Clay	3	4.6	5.7	2.0	4.3	4.3	6.3	44.4
Soil pH	3	0.8	0.7	1.6	0.9	0.8	0.7	1.9
Cations in NH_4Cl	4	4.2	7.8	9.7	6.7	14.1	7.5	15.9
Cations in NH_4OAc	4	4.1	12.0	11.3	7.8	3.7	14.6	250
CEC	2	6.2	17.6	22.4	13.5	5.5	23.5	279
Exchangeable acidity	3	5.7	3.6	9.4	4.3	1.2	12.4	54.0
Cations in $CaCl_2$	2	10.5	8.0	9.7	10.9	7.2	7.0	108
Extractable Fe & Al	6	6 6	7.3	10.0	7.5	5.5	7.4	16.3
Extractable sulfate	2	5.6	2.3	5.1	4.0	12.7	4.0	158
Sulfate isotherms	6	3 7	4.3	1.6	3.2	3.1	3.6	2.8
Total C, N, S	3	3.9	4.7	5.8	4.3	8.1	4.8	31.6

* Number of parameters included

showed the highest differences for the specific surface and cation exchange capacity parameters.

No single laboratory was consistently superior to the others for all parameters or parameter groups. Each laboratory appears to have individual strengths in specific analytical methods, which is probably a reflection of the combination of experience, instrumentation, and laboratory management and practice within each laboratory. This resulted in a patchwork of differences on a parameter group basis, although a general trend existed for the overall ranking of the laboratories.

In order to limit the interlaboratory differences in future surveys, it is recommended that the DDRP staff consider the possibility of choosing laboratories to perform analyses on a parameter basis for those parameter groups that revealed inherently high differences. One laboratory would analyze all soil samples for a given parameter or parameter group. This approach would require an advanced quality assurance program as well as a program for establishing acceptable data values to monitor laboratory performance.

It is recommended that a more stringent laboratory selection procedure be adopted in the pre-evaluation process for the selection of contract laboratories. Since one of the major goals of any quality assurance program is minimizing random and systematic errors, selection of the best possible laboratories is of primary importance. As shown, laboratories differ substantially in their overall performance for certain parameters.

It is recommended that an additional type of soil audit sample be incorporated into the quality assurance program to better monitor the analytical results of the laboratories. A quality control audit sample would have known and acceptable analyte concentrations, which the laboratory would be required to duplicate within limits designated on a batch-by-batch basis. The sample would not be blind to the laboratory. If the analytical results were not within designated intervals, the batch would be reanalyzed in order to bring the audit sample within tolerance specifications. This would ensure that each laboratory could meet a rigid standard for each batch of samples analyzed. Batch error and laboratory difference would be reduced. Therefore, it is further recommended that the laboratories be required to report the analytical results of the analyses on a batch-by-batch basis to the QA staff immediately after the analysis of each batch.

It was noted that both the precision and the difference estimates were high in some cases. Soil analytical methods, especially those which extract or exchange soil constituents rather than those which determine the total amount present in the soil, are uniquely composed of two main sources of error, extraction error and instrumental error. The former is assumed to be the main cause of differences among the laboratories and is a major reason to maintain interlaboratory analytical survey programs. Without distinguishing between extraction and instrumental error, however, it is not known whether one or both errors are present and where to focus efforts to reduce systematic bias. It is recommended that liquid audit samples be incorporated into the quality assurance program and be used to differentiate between systematic bias resulting from extraction or instrument sources.

It is recommended that the analytical procedures for the specific parameters mentioned above be reviewed, tested, and modified where appropriate. Since quality is a continuum, the need for data quality dictates the objective chosen. This objective may or may not be attainable with the current technology. The analytical procedures within the methods should be examined for their ability to accomplish the analysis at the level of detail specified. If the critical value for relative interlaboratory difference is set at 5 percent, then the results of this survey suggest that only the methods used for the soil pH and sulfate isotherm parameters remain as viable methods. If a level of 10 percent is chosen, several parameter groups still remain a problem (see Table 4-2).

It is recommended that the issue of properly assessing laboratory bias be addressed since the current approach using interlaboratory differences has limited utility. The use of external laboratories to analyze the audit samples according to established DDRP methods would generate accepted "true" values for comparison among laboratories.

It is recommended that data quality objectives for interlaboratory bias be established. If only one laboratory exhibited a high but known difference, correction factors could conceivably be applied to the data set. In this case, however, each of the four laboratories exhibited high differences for certain parameters. Serious consideration should be given to either (1) modifying the method or clarifying a procedure within the method, or (2) replacing the method altogether. A case in point for the former is cation exchange capacity and for the latter is the specific surface determination.

Representativeness

All pedons sampled were representative of their respective sampling classes. The preparation laboratories prepared analytical samples of known and accepted quality.

In evaluating representativeness of the quality assurance samples, some trends can be noted. First, the field duplicates and preparation duplicates are representative of the range and frequency distribution of the routine samples for most parameters. Second, the natural audit samples generally are representative of the concentration range of the routine samples.

It is recommended that the soil sampling and preparation protocols specify a method for representative selection of field duplicates and preparation duplicates. The selection protocol should be reiterated to the field and laboratory personnel during the pre-sampling training sessions. The quality assurance field auditor should ensure that a sufficient amount of soil is collected for each bulk sample during the sampling effort to allow a preparation duplicate to be subsampled.

Completeness

Sampling of the specified pedons had a completeness level of 96.5 percent. Processing was accomplished for 100 percent of the pedon samples received by the preparation laboratories. The analytical completeness level exceeded 99 percent for all parameters. Sufficient data were generated to make conclusions for each parameter in the data bases.

Comparability

Sampling, preparation, and analytical methods and protocols for the DDRP Southern Blue Ridge Province Soil Survey were comparable and nearly identical to those used for the DDRP Northeastern Soil Survey. As described in Section 3, the data user is cautioned that some of the field duplicate data may not be comparable for certain applications. It is recommended that the statistical approach undertaken for this report be applied to the Northeastern survey data bases and a comparable report be generated for the benefit of DDRP data users.

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Appendix A

Verification Flags Used in the Southern Blue Ridge Province Soil Survey

Following is a list of the data qualifiers, i.e., flags, that were applied to specific data in the DDRP Southern Blue Ridge Province Soil Survey data bases. Data users can examine which flags may be relevant to them for the purposes of a specific analysis. Acronyms and descriptions of the flags are presented in alpha-numeric order.

Table A-1. Flags Used in the DDRP Southern Blue Ridge Province Soil Survey

Reagent/Calibration Blanks	
B3	R-blank > 2x CRDL (reagent blank flag)
B4	Blank value is negative
B5	R-blank > CRDL
B6	C-blank > CRDL
B7	C-blank > 1.05 x R-blank
B8[a]	SP_SUR: calibration blank > 1 milligram
W1	R-blank ≥ 0.5 x sample value (sample < 2 x R-blank)
W2[a]	pH measurements: R-blank <6 or >7
Quality Control Check Samples	
Q1	QCCS was above contractual criteria
Q2	QCCS was below contractual criteria
Q3	Insufficient number of QCCS measured
Q4	Theoretical DL-QCCS > 3 x CRDL
Q5[a]	Measured DL-QCCS was not within 20% of theoretical value
Q6[a]	Measured & theoretical DL-QCCS is negative
Duplicates	
F1	Field duplicate precision > 10% RSD and both routine and duplicate values > 10 x CRDL
F2	Field duplicate pH precision > 10% RSD
F3	Field duplicate particle size precision > 10% RSD and both routine and duplicate values > 5.0 wt%
P1	Preparation duplicate precision > 10% RSD and both routine and duplicate sample concentrations > 10 x CRDL
P2	Preparation duplicate pH precision > 10% RSD
P3	Preparation duplicate particle size precision > 10% RSD and both routine and duplicate values > 5.0 wt %
A1	Audit duplicate > 10% RSD and both audit pair concentrations > 10 x CRDL
A2	Audit sample pH precision > 10% RSD
A3	Audit sample particle size precision > 10% RSD and both routine and duplicate values > 5.0 wt%
D1	Analytical duplicate precision > 10% RSD and both the routine and duplicate samples > 10 x CRDL
D2	Analytical duplicate pH precision > 10% RSD
D3	Analytical duplicate particle size precision > 10% RSD and both routine and duplicate values > 5.0 wt%
Matrix Spike	
S1	Percent recovery of matrix spike was above (>115%) contractual criteria
S2	Percent recovery of matrix spike was below (<85%) contractual criteria

(continued)

Table A-1. Continued

Instrument Detection Limit

L1	IDL > CRDL

Sulfur Relation Determination

K0	Elemental parameter out of range; C_TOT > 60% and S_TOT > 0.5%
K1	Organic soil and SO4_H2O > 1.05 x SO4_PO4 (only if both are > 5 mg/kg): both values flagged
K2	Mineral soil and SO4_H2O > 1.05 x SO4_PO4 (only if both are > 5 mg/kg): both values flagged
K5	Organic: SO4_H2O or SO4_PO4 not in range 0- to 100-mg/kg; Mineral: SO4_PO4 or SO4_H2O not in range 0- to 250-mg/kg
K6	Organic sample doesn't meet following criteria: SO4_0 0-20(mg S/L), SO4_2 ≥ 22, SO4_4 ≥ 24, SO4_8 ≥ 28, SO4_16 ≥ 36, SO4_32 ≥ 52 *or* Sample doesn't fall within following relationship: SO4_0 < SO4_2 < SO4_4 < SO4_8 < SO4_16 < SO4_32 (organic and mineral)
K7	Ratio of SO4_H2O:SO4_0 flagged when ratio <5 or >25

Iron/Aluminum Determination

M1	Flag if AL_KCL ≤ AL_CL2

Miscellaneous

A0[b]	Value missing
X0[b]	*Invalid* but confirmed data based on QA/QC data review
X1[a,b]	*Invalid* but confirmed data; potential gross contamination of sample or parameter
X2[b]	*Invalid* but confirmed data; potential sample switch
X3[a]	Possible contamination due to either sampling technique, e.g., bucket augering, or soil ammendments, e.g., herbicides, liming, manure, etc.
X4[b]	Outliers due to internal consistency check; data checked only for transcription errors

Laboratory Tags

CC	Soil retrieved from disqualified analytical laboratory
JJ	Insufficient soil for analysis/reanalysis
RR	Reanalyzed
UU	Unnecessary for parameter
XX	No sample (initiated at preparation laboratory)

[a] New flag -- not on list of flags distributed 7/87.
[b] Sample flag -- parameter flagged only for affected samples.

Appendix B

Data Verification Worksheets and Tables

Data verification was accomplished using the DDRP Quality Assurance Plan (Bartz et al., 1987) as a guideline. Figures B-1 and B-2 from that document serve as examples of some of the prominent quality assurance worksheets used during data verification. Also provided is the Data Verification Template in Figures B-3 through B-13. The template was used to guide the quality assurance staff through the often complex procedures used to verify the data. The template was developed by the Soils Quality Assurance Section of Lockheed Engineering and Sciences Company in Las Vegas, Nevada.

The latter portion of Appendix B includes data from some of the primary verification activities. Tables B-1, B-2, and B-3 provide information on the quality control check sample compliance, the internal consistency checks, and the analytical completeness assessment.

DATE SENT _____

DATE RECEIVED _____

DDRP SOIL SURVEY
FORM 500

Data Confirmation/Reanalysis Request Form

Batch ID _____ Contractor Analytical Laboratory _____ Laboratory Supervisor _____

The following suspect data values require: _____ Confirmation (See I) _____ Reanalysis (See II)

PARAMETER	DDRP FORM NO.	SAMPLE ID	SUSPECT ORIGINAL VALUE	RECONFIRMED NEW VALUE	Explanation: CONTRACT ANALYTICAL LABORATORY	Explanation: LEMSCO

I. Confirmation Request Did ANY values change: __ Yes __ No
If yes, reason (note above in explanation column)
(A) Reporting Error (C) Original reported value did not change
(B) Calculation Error (D) Data Previously Omitted
(E) Other - Explain

Whether values are changed or not, submit supporting RAW DATA.

Additional Comments Regarding Confirmation: _____

_____

_____

II Reanalysis Requested Due to:*
_____ QA (External) Data
_____ QC (Internal) Data Indicated Below.
_____ IDL > CRDL
_____ Matrix Spike Recovery Outside Criteria
_____ Replicate Precision (% RSD) Outside Criteria, Insufficient Number of Replicates
_____ Blank > CRDL (Reagent; Calibration)
_____ QCCS Outside Criteria (DL, Low, High)
_____ IC Resolution Value Below 60%
_____ Air Dry Sample Weight Ouside Criteria
_____ Total Sample Volume, Aliquot Volume, or Dilution Volume Outside Criteria
_____ Standard Relationships Out of Range

Additional Comments Regarding Reanalysis. _____

_____

_____

* All appropriate data forms including QC data forms must be submitted in support of reanalysis.
"Date form completed" must reflect date of reanalysis.

FOR LEMSCO USE ONLY. PRE-VERIFICATION _____ NUMBER OF VALUES SUBMITTED _____
POST-VERIFICATION _____ NUMBER OF VALUES CHANGED _____

0600C

Figure B-1. DDRP form 500 (data confirmation/reanalysis request).

I. OUTSTANDING ISSUES - CONTRACTOR ANALYTICAL LABORATORY

The following items that are identified as missing should be resubmitted and problems should be resolved before verification is completed:

A. General (forms 102-108)
   1. Required forms have been submitted.
   2. Laboratory name, batch ID, preparation laboratory name, laboratory manager's signature, date form completed, and date batch received are included on all forms.
   3. Correct data qualifiers (tags) were used as needed (see Table 1).

B. Data examination (forms 103-108)
   1. Check that audit pairs are within established control criteria.
   2. Estimate %RSD for all paired QA samples for each parameter, and record in Table 3.
   3. Check the internal consistency of the data.
      a. Form 103a: pH, H_2O > 0.002 > 0.01.
      b. Form 103b: sand + silt + clay = 100 $\pm$ 0.2.
      c. Form 104d: CEC NH_4OAc > CEC NH_4Cl.
      d. Form 106: Ext. Sulfate, H_2O < PO_4.
      e. Form 106: Exch. Acidity, $BaCl_2$ > KCl.
      f. Form 107: Sulfate Isotherms are 0 < 2 < 4 < 8 < 16 < 32. Adsorption solution is within 5% of the theoretical value.
      g. Form 104c: Extraction ratio is 1:2 for mineral samples and 1:10 or 1:25 for organic samples.
      h. Forms 103b and 108: For particle size analysis and specific surface, organic samples are reported as a U.

C. General (forms 109-116)
   1. Required forms have been submitted.
   2. Laboratory name, batch ID, and laboratory manager's signature are included on all forms.

D. Data examination (forms 109-116)
   1. Forms 109a-c: Detection Limits
      a. Check that instrumental detection limits (IDL) and associated dates of determination are tabulated. IDL should be updated monthly for each parameter.
      b. IDL should be less than or equal to the contract-required detection limit (CRDL) for each parameter.
   2. Form 110a-c: Matrix Spikes
      a. Identify samples used for spiking.
      b. Check that percent recovery for matrix spikes is reported for each parameter required.

Figure B-2. Data completeness checklist (1 of 3).

c. Check that percent recovery is calculated correctly (recalculate at least three per page).
d. Check that percent recovery is 100 ± 15% for each parameter; if it is not, then spiking must be repeated on two different samples.
e. Verify that the level of spike is 10 times the CRDL or equal to the endogenous level, whichever is greater.
f. Check that the sample used for Total S, N, and C is not an organic sample for each batch.

3. Form 111a-i: Replicates
   a. Replicate precision results are reported for each parameter. For pH and specific surface, triplicates are determined.
   b. Correct equation is used to calculate %RSD (degrees of freedom equal n-1).
   c. %RSDs are 0-10% (except on fractionated sand and silt).

4. Forms 112a-h: Blanks and QCCS
   a. Calibration blanks, reagent blanks, and detection limit (DL) QCCS are reported where required.
   b. Calibration and reagent blanks should be less than or equal to the CRDL.
   c. Form 112g: K-factors are reported correctly.
   d. Form 112h: Three high EGME blanks are reported correctly.
   e. DL QCCS theoretical values are approximately 2 to 3 times the CRDL, and the measured values are within 20% of the theoretical value.
   f. QCCS true values are approximately in the midrange of the reported sample values or of the calibration curve.
   g. Initial, continuing, and final QCCS values are within upper and lower control limits.

5. Form 113: Ion Chromatography
   a. IC resolution test results are reported.
   b. Resolution value exceeds 60%.
   c. Peaks are clean on chromatogram(s).
   d. At least one chromatogram is provided for each day of operation for each instrument.
6. Form 114: Standard Additions
   a. Standard additions are performed and results are reported when matrix spike results do not meet contractual requirements.
7. Forms 115a-e: Air Dry Sample Weights
   a. The air-dried soil weight is reported for each parameter, except for particle-size. analysis (oven dried) and specific surface ($P_2 O_5$ wt. = oven dried).

Figure B-2. Continued (2 of 3).

b. Weights are reported correctly (see Table 2).
c. Form 115a: One sample is determined in triplicate for moisture and specific surface.
d. Duplicates are reported correctly.

8. Forms 116a-h: Dilution Factors
a. Total sample volume, aliquot volume, total dilution volume, dilution concentrations, and dilution blanks are recorded for each sample.

E. Forms 200: Blank-corrected data
1. Required forms 204-208 have been submitted.
2. Laboratory name, batch ID, preparation laboratory name, manager's signature, and date batch received are included on all forms.
3. Correct number of samples were analyzed, and the results for each parameter are tabulated.

F. Forms 300: Raw Data
1. Required forms 303b-308 have been submitted.
2. Laboratory name, batch ID, preparation laboratory name, laboratory manager's signature, and date batch received are included on all forms.
3. Correct number of samples were analyzed, and the results for each parameter are tabulated.

G. Reporting units are correct on the following forms (see Table 4):
1. 103-108
2. 109: Detection Limits
3. 110: Matrix Spikes
4. 111: Replicates
5. 112: Blanks and QCCS
6. 115: Air Dry Sample Weights
7. 116: Dilution Factors/Concentration
8. 200: Blank-Corrected Data
9. 300: Raw Data

Figure B-2. Continued (3 of 3).

	Major	Minor	Non
Audits	Either value out of window %PSD out on graph of %RSD vs. conc.	Either value greater than 1.2 x or less than 0.8 x None	Both within windows. %RSD in on praph of %RSD vs conc.
Prep	%RSD out on graph of %RSD vs. conc.	None	%RSD in on graph of %RSD vs. conc.
Field	None	%RSD out on graph of %RSD vs. conc.	%RSD in on graph of %RSD vs. conc.
Internal Replicates (3 reps)	None	%RSD > 10.0%	%RSD < 10.0%
Blanks	None Effects > 25% of values (Effects=blanks constites 20% of sample conc.)	Not using 3 high EGME blanks (some contracts required low, med, high blanks) EGME blanks did not equil. at < 1 mg	Used 3 high EGME blanks which equil. at < 1 mg.
DL QCCS	NA	NA	NA
QCCS	Initial, final, or continuing QCCS above or below absolute contract limit or lab's 95%, whichever is greater	True QCCS not in mid-calibration range Marked change from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calibration range No significant change w/i QCCS values
Matrix spike	NA	NA	NA
Standard Relationship	None	None	None
IDL > CRDL	NA	NA	NA

Figure B-3. Quality assurance reanalysis template for specific surface.

	Major	Minor	Non
Audits	Either value out of window Sand, silt, or clay: %RSD out on graph of %RSD vs. conc.	Either value greater than 1.2 mean or less than 0.8 mean Sand or silt fractions: %RSD out on graph of %RSD vs. conc.	Both within windows. %RSD in on graph of %RSD vs. conc.
Prep	Sand, silt, or clay: %RSD out on graph of %RSD vs. conc.	Sand or silt fractions: %RSD out on graph of %RSD vs. conc.	%RSD in on graph of %RSD vs. conc.
Field	None	Sand, silt, or clay: %RSD out on graph of %RSD vs. conc.	%RSD in on graph of %RSD vs. conc.
Internal Replicates	%RSD greater than 10.0% and both values greater than 5%	%RSD greater than 10.0% and one value greater than 5%	%RSD greater than 10.0% and no values greater than 5%, or %RSD less than 10.0%
Blanks	None	Clay blank not approx. 0.01	Clay blank approx. 0.01
DL QCCS	NA	NA	NA
QCCS	Initial, final, or continuing QCCS above or below absolute contract limit, or lab's 95%, whichever is greater	True QCCS not in mid-calibration range Marked change from one QCCS to another of 5% or any diff. w/i batch QCCS of 10%	True QCCS within calibration range No significant change w/i QCCS values
Matrix spike	NA	NA	NA
Standard Relationship	None	Sand + silt + clay > 100% ±0.1% Sand fraction not equal to sand Silt fractions not equal to silt	Sand + silt + clay = 100% ±0.1% Sand fractions = sand Silt fractions = silt
IDL > CRDL	NA	NA	NA

Figure B-4. Quality assurance reanalysis template for particle size analysis.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD > to 0%	%RSD in on graph of %RSD vs Conc.
Internal Replicates	Triplicates not within 0.1 pH units	None	Triplicate within 0.1 pH units
Blanks	None	None	Reagent blank is solution added to the soil
DL QCCS	NA	NA	NA
QCCS	Initial, Final, or Cont. QCCS not within 0.1 pH units of true QCCS	Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCSs of 10%	Init. Cont. and Final QCCS w/in 0.1 pH units of true QCCS, and no sign, chg. between QCCS values
Matrix Spikes	NA	NA	NA
Standard Relationships	None	pH_H20 < pH_002 CaC12 < pH_01 CaC12	pH_H20 > pH_002 CaC12>pH_01 CaC12
IDL > CRDL	NA	NA	NA

Figure B-5. Quality assurance reanalysis template for pH.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values > 10 x CRDL; %RSD < 10.0%
Blanks	Blank > Contract Require Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blank > Contract Require Effects 0-25% of values	Blank > Contract Require Effects 0% of values Blank < Contract Require
DL QCCS	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 x CRDL DL QCCS meas. 20% theo.
QCCS	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	Ca < Mg < K < Na	Ca > Mg > K > Na
IDL > CRDL	IDL > CRDL and Effects 25% of values (Effects=CRDL constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-6. Quality assurance reanalysis template for exchangeable cations.

	Major	Minor	None
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values > 10 x CRDL %RSD < 10.0%
Blanks	Blank > Contract Require. Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blank > Contract Require. Effects 0-25% of values	Blank > Contract Require Effects 0% of values Blank < Contract Require
DL QCCS (may be NA if titration)	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 X CRDL DL QCCS, meas. 20% theo.
QCCS (may be NA if titration)	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	NH4OAc < NH4Cl	NH4OAc > NH4Cl
IDL > CRDL	IDL > CRDL and Effects 25% or values (Effects=CRDL constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-7. Quality assurance reanalysis template for cation exchange capacity.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 X CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values >10 x CRDL %RSD < 10.0%
Blanks (No Calibration Blanks)	Blank > Contract Require Effects > 25% of values (Effects = blank constitutes 20% of sample conc.)	Blank > Contract Require Effects 0-25% of values	Blank > Contract Require Effects 0% of values Blank < Contract Require
DL QCCS	NA	NA	NA
QCCS	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	NA	NA	NA
Standard Relationships	None	BaC12 < KCL	BaC12 > KC1
IDL > CRDL	IDL > CRDL and Effects 25% of values (Effects = CRDL constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-8. Quality assurance reanalysis template for exchangeable acidities.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values > 10 x CRDL %RSD < 10.0%
Blanks	Blank > Contract Require. Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blanks > Contract Require Effects 0-25% of values	Blank > Contract Require Effects 0% of values Blank < Contract Require
DL QCCS	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 x CRDL DL QCCS, meas. 20% theo.
QCCS	Initial, FInal, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	None	None
IDL > CRDL	IDL > CRDL and Effects 25% of values (Effects=CRDL constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-9. Quality assurance reanalysis template for KCl-extractable aluminum.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values >10 x CRDL %RSD < 10.0%
Blanks	Blank> Contract Require Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blanks > Contract Require Effects 0-25% of values	Blank > Contract Requires Effects 0% of values Blank < Contract Require
DL QCCS	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 x CRDL DL QCCS, meas. 20% theo.
QCCS	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	None	None
IDL > CRDL	IDL > CRDL and Effects 25% of value (Effects=CRDL Constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-10. Quality assurance reanalysis template for extractable iron and aluminum.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values >10 x CRDL %RSD < 10.0%
Blanks	Blank> Contract Require Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blanks > Contract Require Effects 0-25% of values	Blank > Contract Requires Effects 0% of values Blank < Contract Require
DL QCCS	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 x CRDL DL QCCS, meas. 20% theo.
QCCS	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	H2O ext. SO4 > PO4 ext. SO4	H2O ext. SO4 < PO4 ext. SO4
IDL > CRDL	IDL > CRDL and Effects 25% of value (Effects=CRDL Constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-11. Quality assurance reanalysis template for water-extractable sulfate and phosphate-extractable sulfate.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0% ;no values >10 x CRDL %RSD < 10.0%
Blanks	Blank> Contract Require Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blanks > Contract Require Effects 0-25% of values	Blank > Contract Requires Effects 0% of values Blank < Contract Require
DL QCCS (0 Isotherm only)	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 x CRDL DL QCCS, meas. 20% theo.
QCCS	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	0 < 2 < 4 < 8 < 16 < 32	0 > 2 > 4 > 8 > 16 > 32
IDL > CRDL	IDL > CRDL and Effects 25% of value (Effects=CRDL Constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL
Ion Chromatograph	None	%Resolutions < 60% Chromatographs not clean	%Resolution > 60%; Chromatographs Clean

Figure B-12. Quality assurance reanalysis template for sulfate isotherms.

	Major	Minor	Non
Audits	Either value out of window %RSD out on graph of %RSD vs Conc.	Either value greater than 1.2 mean or less than 0.8 mean None	Both within Windows %RSD in on graph of %RSD vs Conc.
Prep	%RSD out on graph of %RSD vs Conc.	None	%RSD in on graph of %RSD vs Conc.
Field	None	%RSD out on graph of %RSD vs Conc.	%RSD in on graph of %RSD vs Conc.
Internal Replicates	%RSD > 10.0% Both values > 10 x CRDL	%RSD > 10.0% One value > 10 x CRDL	%RSD > 10.0%; no values >10 x CRDL %RSD < 10.0%
Blanks (some instruments R-blk NA for Total N & C)	Blank> Contract Require Effects > 25% of values (Effects=blank constitutes 20% of sample conc.)	Blanks > Contract Require Effects 0-25% of values	Blank > Contract Requires Effects 0% of values Blank < Contract Require
DL QCCS	None	DL QCCS, theo. not 2-3 x CRDL or IDL, where applicable DL QCCS, meas. not 20% theo.	DL QCCS, theo. 2-3 x CRDL DL QCCS, meas. 20% theo.
QCCS (K-Factors for TotN and TotC)	Initial, Final, or Cont. QCCS above or below absolute contract limit or lab's 95%, whichever >	True QCCS not mid calib. range Marked chg. from one QCCS to another of 5% or any diff. w/in batch QCCS of 10%	True QCCS w/in mid calib. range No sign. chg. w/in QCCS values
Matrix Spikes	%Recovery > 100 + or - 15.0%	Spike added not 50-200% of the endogenous level	%Recovery 100 + or - 15.0% Spike added 50-200% of endogenous level
Standard Relationships	None	None	None
IDL > CRDL	IDL > CRDL and Effects 25% of value (Effects=CRDL Constitutes 20% of sample conc.)	IDL > CRDL and Effects 0-25% of values	IDL > CRDL and Effects 0% of values IDL = CRDL IDL < CRDL

Figure B-13. Quality assurance reanalysis template for total sulfur, nitrogen, and carbon.

Table B-1. Occurrences of Less-Than-Complete Compliance for Measurement of Quality Control Check Samples

Parameter	Laboratory	n	Percent compliance
SP_SUR	2	29	72.4
	3	27	77.8
VCOS	1	47	26.1
	2	45	33.4
	3	42	38.1
COS	1	47	29.8
	2	45	73.3
	3	42	83.3
MS	1	47	57.4
	2	45	93.3
	3	42	95.2
FS	1	47	53.2
	3	42	95.3
VFS	2	45	86.7
	3	42	97.6
SILT	2	45	95.6
COSI	2	45	62.2
	3	42	90.5
FSI	2	45	71.1
MG_CL	1	54	98.1
	2	56	98.2
K_CL	1	54	79.6
NA_CL	1	54	94.4
MG_OAC	1	54	94.4
K_OAC	1	54	94.4
CEC_CL	2	55	98.2
AC_KCL	2	54	88.9
AC_BACL	2	52	98.1
AL_KCL	1	52	98.1
MG_CL2	1	54	85.2
NA_CL2	2	58	98.2
AL_CL2	2	58	98.3
AL_PYP	1	54	98.2
	2	57	98.2
FE_CD	2	58	87.9
AL_CD	2	58	96.6
SO4_H20	2	55	96.4
SO4_PO4	2	55	92.7
SO4_0	2	54	96.3
	3	42	97.6
SO4_2	2	52	92.3
SO4_4	3	42	97.6
SO4_8	2	51	98.0
SO4_16	2	53	94.3
SO4_32	2	52	92.3
C_TOT	3	41	65.9
N_TOT	2	55	96.4
	3	41	68.3
S_TOT	3	41	82.9

Table B-2. Internal Consistency Checks Performed for the Southern Blue Ridge Province Analytical Verified Data Base

Parameter	Correlations[a]	Data set[b]	r^2 value[a] 1st check	r^2 value[a] 2nd check
MOIST	+/-	A	x	x
SP_SUR	+/-	A	x	x
SAND	+/-, FSI	A	.95	.95
VCOS	+/-, COS	A	.62	.62
COS	+/-, MS	A	.74	.74
FS	+/-, SAND	A	.67	.67
VFS	+/-, SAND	A	x	x
SILT	+/-, SAND	A	.96	.96
COSI	+/-, SILT	A	.80	.80
CLAY	+/-	A	x	x
PH_H2O	PH_01M	A	.95	.96
PH_002M	PH_H20	A	.98	.98
PH_01M	PH_002M	A	.98	.98
CA_CL	CA_OAC	A	.94	.96
MG_CL	MG_OAC	A	.82	.83
K_CL	K_OAC	A	.92	.92
NA_CL	NA_OAC	A	.70	.90
CEC_CL	CEC_OAC	A	.99	.89
AC_KCL	AL_KCL	A	.81	.83
AC_BACL	C_TOT	A	.92	.92
CA_CL2	+/-	A	x	x
MG_CL2	+/-	A	x	x
K_CL2	+/-	A	x	x
NA_CL2	+/-	A	x	x
FE_CL2	+/-	A	x	x
AL_CL2	+/-	A	x	x
FE_PYP	FE_AO	A	.91	.90
AL_PYP	AL_CD	A	.89	.89
FE_AO	FE_CD	A	.76	.78
AL_AO	AL_CD	A	.86	.76
SO4_H2O	SO4_PO4	A	.50	.50
SO4_0	SO4_H2O	O-M	.93 .88	.94 .80
SO4_2	SO4_H2O	O-M	.97 .86	.97 .68
SO4_4	SO4_H2O	O-M	.96 .82	.96 .51
SO4_8	SO4_H2O	O-M	.96 .76	.96 .29
SO4_16	SO4_H2O	O-M	.89 .62	.90 .07
SO4_32	SO4_H2O	O-M	.89 .07	.89 .08
N_TOT	C_TOT	A	.95	.95
S_TOT	N_TOT	O-M	.34 .76	.36 .66

[a] At times, a variable is used for more than one correlation; x = r^2 not applicable; +/- = outlier check on the highest and lowest 1% of the values.

[b] Routine samples used in correlation: A = all samples; O = organic samples only; M = mineral samples only.

Table B-3. Completeness of Soil Analysis Using Data for Routine Samples from the Verified and Validated Data Bases

Parameter	Verified n*	Verified percent	Validated n*	Validated percent
MOIST	747	99.9	747	99.9
SP_SUR	703	99.9	703	99.9
SAND, SILT, CLAY	703	99.9	703	99.9
VCOS, COS, MS, VFS	703	99.9	703	99.9
FS, FSI	703	99.9	702	99.7
COSI	703	99.9	700	99.5
PH_H2O	747	99.9	747	99.9
PH_002M	747	99.9	747	99.9
PH_01M	747	99.9	747	99.9
CA_CL	747	99.9	731	97.7
MG_CL	747	99.9	747	99.9
K_CL	747	99.9	743	99.3
NA_CL	747	99.9	730	97.6
CA_OAC	747	99.9	733	98.0
MG_OAC	747	99.9	747	99.9
K_OAC	747	99.9	746	99.7
NA_OAC	747	99.9	726	97.1
CEC_CL	747	99.9	705	94.3
CEC_OAC	747	99.9	709	94.8
AC_KCL	747	99.9	746	99.7
AC_BACL	747	99.9	746	99.7
AL_KCL	747	99.9	744	99.5
CA_CL2	747	99.9	726	97.1
MG_CL2	747	99.9	746	99.7
K_CL2	747	99.9	746	99.7
NA_CL2	747	99.9	731	97.7
FE_CL2	747	99.9	745	99.6
AL_CL2	747	99.9	743	99.3
FE_PYP	747	99.9	747	99.9
AL_PYP	747	99.9	744	99.5
FE_AO	747	99.9	746	99.7
AL_AO	747	99.9	747	99.9
FE_CD	747	99.9	744	99.5
AL_CD	747	99.9	747	99.9
SO4_H2O	747	99.9	744	99.5
SO4_PO4	747	99.9	728	97.3
SO4_0	747	99.9	745	99.6
SO4_2	747	99.9	747	99.9
SO4_4	747	99.9	747	99.9
SO4_8	747	99.9	745	99.6
SO4_16	747	99.9	747	99.9
SO4_32	747	99.9	747	99.9
C_TOT	747	99.9	740	98.9
N_TOT	747	99.9	745	99.6
S_TOT	747	99.9	731	97.7

* The total number of routine samples (N) was 748; for the verified data base, n is the number of samples which underwent analysis; for the validated data base, n is the number of samples that are valid at a level of confidence of 2 or less; [Note: 44 of the routine samples were organic soils that were not required to undergo specific surface or particle size analyses; in these cases, N = 704].

Appendix C

Table of Statistics for Step Function Precision Estimates

This table provides statistical information that supplements the precision results and discussion found in Section 3. Included are data relating to the development of delta values, including the standard deviations and proportions. These data can be used to assess the quality of the routine sample data set on the basis of the quality of the QA sample data sets. The table is sorted by parameter and subsorted by data set.

Table C-1. Table of Statistics for Step Function Precision Estimates

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs >DQO n	Pairs >DQO %	Between-batch SD
MOIST	AS	0.2715	0.0-1.0	6	0.15	0.0105			0.0082
			1.0-2.0	17	1.79	0.1153			0.1782
			2.0-3.0	25	2.36	0.1058			0.1287
			3.0-5.0	2	3.42	1.3655			0.4091
			5.0-inf	.	.	.			.
	PD	0.1503	0.0-1.0	4	0.78	0.0871			
			1.0-2.0	8	1.55	0.1515			
			2.0-3.0	12	2.48	0.1566			
			3.0-5.0	.	.	.			
			5.0-inf	2	6.24	0.1276			
	FD	0.2782	0.0-1.0	15	0.70	0.0673			
			1.0-2.0	34	1.59	0.1867			
			2.0-3.0	37	2.46	0.2659			
			3.0-5.0	14	3.53	0.3959			
			5.0-inf	4	5.88	2.3188			P(i)
	S/H	1.0249	0.0-1.0	15	0.74	0.5516			0.036
			1.0-2.0	291	1.59	0.6811			0.482
			2.0-3.0	210	2.40	1.1486			0.334
			3.0-5.0	82	3.73	1.9585			0.128
			5.0-inf	11	6.14	2.1173			0.020

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs >DQO n	Pairs >DQO %	Between-batch SD
SP_SUR	AS	2.8038	0.0-20.0	10	8.54	1.5527			1.1464
			20.0-35.0	15	26.30	2.9952			5.0913
			35.0-50.0	25	41.61	2.9879			3.6237
			50.0-60.0	.	.	.			.
			60.0-inf	.	.	.			.
	PD	2.4631	0.0-20.0	7	12.51	2.6175			
			20.0-35.0	6	26.62	1.8572			
			35.0-50.0	8	41.28	1.8821			
			50.0-60.0	3	55.16	7.9749			
			60.0-inf	2	76.26	2.0807			
	FD	4.3893	0.0-20.0	22	12.55	2.3343			
			20.0-35.0	33	28.02	4.1471			
			35.0-50.0	25	40.56	3.8869			
			50.0-60.0	14	53.89	10.4138			
			60.0-inf	8	75.84	4.9491			P(i)
	S/H	15.3351	0.0-20.0	63	15.70	7.8331			0.118
			20.0-35.0	307	29.07	13.1748			0.500
			35.0-50.0	177	42.74	19.5647			0.283
			50.0-60.0	52	54.30	23.7949			0.083
			60.0-inf	9	86.97	19.7507			0.016
SAND	AS	3.2861	0.0-25.0	4	24.34	0.4809			0.2562
			25.0-40.0	3	30.70	2.8213	2	66.7	3.7041
			40.0-50.0	1	46.50	11.5966	1	100.0	.
			50.0-65.0	25	56.87	0.6598	3	12.0	2.1122
			65.0-inf	17	88.96	1.1548	5	29.4	1.3601
	PD	1.6508	0.0-25.0	1	14.25	0.4950			
			25.0-40.0	1	39.75	0.2121			
			40.0-50.0	8	46.46	1.1150	1	12.5	
			50.0-65.0	10	59.21	2.6087	2	20.0	
			65.0-inf	6	79.15	0.3162			
	FD	2.1400	0.0-25.0	6	17.33	1.2517	3	50.0	
			25.0-40.0	16	31.70	1.2618	4	25.0	
			40.0-50.0	23	44.80	3.8792	11	47.8	
			50.0-65.0	36	57.57	1.8054	11	30.1	
			65.0-inf	21	76.17	1.4229	6	27.9	P(i)
	S/H	13.0018	0.0-25.0	5	9.56	9.2390			0.010
			25.0-40.0	40	34.74	14.5129			0.068
			40.0-50.0	133	44.61	11.0756			0.218
			50.0-65.0	315	57.32	14.0928			0.509
			65.0-inf	115	71.95	11.9727			0.195
VCOS	AS	0.6918	0.0-2.0	26	0.97	0.2033			0.2943
			2.0-5.0	12	2.70	0.6618			0.4318
			5.0-7.0	1	6.65	0.9192			.
			7.0-10.0	5	8.47	0.6025			0.9660
			10.0-inf	6	10.90	2.0469			0.7550
	PD	0.8507	0.0-2.0	6	0.66	0.2291			
			2.0-5.0	10	3.25	0.5599			
			5.0-7.0	3	5.82	1.6376			
			7.0-10.0	5	8.44	1.3784			
			10.0-inf	2	12.35	1.6125			
	FD	0.9102	0.0-2.0	27	1.06	0.2228			
			2.0-5.0	44	3.32	0.7352			
			5.0-7.0	16	5.88	1.0109			
			7.0-10.0	5	8.66	1.6420			
			10.0-inf	9	13.83	2.7438			P(i)
	S/H	3.1052	0.0-2.0	44	1.07	1.0098			0.090
			2.0-5.0	361	3.51	2.1911			0.580
			5.0-7.0	105	6.35	4.1229			0.170
			7.0-10.0	84	8.31	6.1835			0.135
			10.0-inf	14	14.11	8.1345			0.026

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs >DQO n	Pairs >DQO %	Between-batch SD
COS	AS	1.1928	0.0-6.0	33	3.30	0.2926			0.3154
			6.0-10.0	1	9.85	0.2121			.
			10.0-15.0	4	11.66	2.2122			1.4447
			15.0-inf	12	19.53	2.3310			1.6977
	PD	1.0244	0.0-6.0	5	3.46	0.2530			
			6.0-10.0	11	7.62	2.2702			
			10.0-15.0	6	11.91	0.4787			
			15.0-inf	4	21.77	1.0989			
	FD	0.8614	0.0-6.0	42	3.25	0.3901			
			6.0-10.0	29	8.11	1.0590			
			10.0-15.0	21	12.34	0.9607			
			15.0-inf	10	19.59	0.8823			P(i)
	S/H	4.8611	0.0-6.0	126	3.87	2.3924			0.217
			6.0-10.0	185	7.94	4.1560			0.306
			10.0-15.0	250	11.47	5.7904			0.403
			15.0-inf	47	17.87	9.9343			0.074
MS	AS	0.4506	0.0-7.0	7	3.51	0.1000			0.1600
			7.0-15.0	26	8.27	0.4731			0.3725
			15.0-20.0	.	.	.			.
			20.0-inf	17	28.04	1.2580			1.3142
	PD	0.4251	0.0-7.0	4	5.34	0.1369			
			7.0-15.0	12	10.89	0.4026			
			15.0-20.0	7	16.48	0.6745			
			20.0-inf	3	23.50	0.9000			
	FD	0.8863	0.0-7.0	32	3.82	0.1772			
			7.0-15.0	46	10.60	1.0184			
			15.0-20.0	14	17.29	0.6285			
			20.0-inf	10	24.08	1.0644			P(i)
	S/H	5.4795	0.0-7.0	49	5.11	3.2163			0.094
			7.0-15.0	468	11.56	5.4426			0.748
			15.0-20.0	84	16.87	6.9530			0.138
			20.0-inf	7	23.52	7.3224			0.020
FS	AS	1.5968	0.0-5.0	7	3.91	0.1225			0.3297
			5.0-10.0	.	.	.			.
			10.0-15.0	.	.	.			.
			15.0-25.0	32	22.83	1.5760			1.6669
			25.0-inf	11	31.51	2.0641			1.9776
	PD	0.6694	0.0-5.0	.	.	.			
			5.0-10.0	4	6.52	0.3317			
			10.0-15.0	6	13.31	0.6922			
			15.0-25.0	11	18.91	0.6882			
			25.0-inf	5	28.12	0.5916			
	FD	0.8903	0.0-5.0	6	3.17	0.1155			
			5.0-10.0	19	7.51	0.5319			
			10.0-15.0	24	12.37	1.1724			
			15.0-25.0	38	19.89	0.8227			
			25.0-inf	15	28.37	0.7443			P(i)
	S/H	6.4465	0.0-5.0	4	0.94	0.2510			0.007
			5.0-10.0	19	8.49	4.7832			0.041
			10.0-15.0	160	12.34	5.4761			0.254
			15.0-25.0	400	18.70	6.9114			0.647
			25.0-inf	25	27.56	7.5823			0.051

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	>DQO %	Between-batch SD
VFS	AS	0.9815	0.0-8.0	8	6.94	0.8846			0.2696
			8.0-14.0	13	10.28	0.9009			1.0197
			14.0-20.0	11	18.19	1.2454			0.6924
			20.0-inf	18	21.61	1.1533			0.9327
	PD	0.7067	0.0-8.0	5	5.93	0.2588			
			8.0-14.0	14	11.19	0.6170			
			14.0-20.0	4	16.66	1.2171			
			20.0-inf	3	22.37	0.4435			
	FD	0.9105	0.0-8.0	24	5.96	0.4985			
			8.0-14.0	36	10.80	0.7190			
			14.0-20.0	30	16.25	1.5143			
			20.0-inf	12	25.57	1.5215			P(i)
	S/H	5.2680	0.0-8.0	20	5.50	2.7120			0.043
			8.0-14.0	444	11.12	4.9620			0.705
			14.0-20.0	114	16.51	6.1405			0.192
			20.0-inf	30	21.84	7.8953			0.060
SILT	AS	3.0484	0.0-12.0	7	5.44	1.0021	2	28.6	2.0491
			12.0-25.0	19	17.31	1.2367	6	31.6	0.9544
			25.0-35.0	16	26.71	1.4472	2	12.5	0.5231
			35.0-45.0	1	38.20	15.2735	1	100.0	.
			45.0-inf	7	65.45	2.0296	2	28.6	4.1057
	PD	1.9100	0.0-12.0	2	8.95	0.3808			
			12.0-25.0	12	19.03	2.1064	2	16.7	
			25.0-35.0	7	30.14	2.0032	3	42.9	
			35.0-45.0	5	39.43	1.0193	2	40.0	
			45.0-inf	.	.	.	.	.	
	FD	1.5611	0.0-12.0	6	8.58	0.5679	1	15.4	
			12.0-25.0	39	19.56	1.6110	8	20.5	
			25.0-35.0	26	30.24	1.4116	11	42.3	
			35.0-45.0	22	39.62	2.1105	8	35.6	
			45.0-inf	9	52.17	1.1385	2	22.2	P(i)
	S/H	10.2079	0.0-12.0	1	8.32	3.1113			0.007
			12.0-25.0	234	20.97	9.7470			0.379
			25.0-35.0	293	28.39	10.6545			0.476
			35.0-45.0	71	37.57	10.1350			0.121
			45.0-inf	9	55.33	11.4662			0.017
COSI	AS	1.0200	0.0-6.0	6	4.07	1.0042			2.0745
			6.0-15.0	35	10.15	0.8494			1.3269
			15.0-inf	9	31.13	7.2708			2.4468
	PD	0.9736	0.0-6.0	5	4.81	3.3092			
			6.0-15.0	19	9.29	0.8851			
			15.0-inf	2	16.77	0.7018			
	FD	1.1769	0.0-6.0	22	4.46	0.4880			
			6.0-15.0	61	10.35	1.1712			
			15.0-inf	19	19.76	2.4179			P(i)
	S/H	5.0119	0.0-6.0	20	4.83	4.3080			0.038
			6.0-15.0	577	9.89	5.0333			0.936
			15.0-inf	11	17.66	5.2882			0.026

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs >DQO n	Pairs >DQO %	Between-batch SD
FSI	AS	0.8048	0.0-10.0	18	3.09	1.2137			0.5142
			10.0-20.0	25	14.14	0.8212			1.2703
			20.0-30.0	1	27.65	0.6364			.
			30.0-inf	6	32.41	0.5708			1.7588
	PD	1.2626	0.0-10.0	7	7.61	0.2087			
			10.0-20.0	11	15.89	1.2412			
			20.0-30.0	8	24.06	1.6750			
			30.0-inf	.	.	.			
	FD	1.2922	0.0-10.0	12	6.36	0.4770			
			10.0-20.0	55	14.74	1.0247			
			20.0-30.0	27	24.19	2.3842			
			30.0-inf	8	37.25	2.2773			P(i)
	S/H	7.2695	0.0-10.0	37	8.24	5.2270			0.067
			10.0-20.0	439	15.58	7.1082			0.708
			20.0-30.0	124	23.48	8.1474			0.208
			30.0-inf	8	38.42	11.2703			0.017
CLAY	AS	1.3815	0.0-10.0	21	3.44	0.5189	1	4.8	0.6286
			10.0-25.0	26	17.95	1.5467	6	23.1	1.1777
			25.0-inf	.	.	.	.	.	.
	PD	0.7144	0.0-10.0	5	5.67	0.6107			
			10.0-25.0	16	15.98	0.7278	3	18.8	
			25.0-inf	5	33.72	0.7563	1	20.0	
	FD	1.4309	0.0-10.0	17	5.70	0.8438	2	11.8	
			10.0-25.0	66	17.45	1.4499	17	25.4	
			25.0-inf	19	36.20	2.0403	5	26.3	P(i)
	S/H	6.5929	0.0-10.0	83	7.14	3.5364			0.142
			10.0-25.0	455	17.15	6.5828			0.744
			25.0-inf	70	33.71	10.4791			0.114
PH_H2O	AS	0.0349	4.0-inf	50	4.73	0.0349			0.0871
	PD	0.0350	4.0-inf	26	5.17	0.0350			
	FD	0.1009	4.0-inf	104	5.03	0.1009	8	7.7	P(i)
	S/H	0.3331	4.0-inf	609	5.08	0.3331			1.000
PH_002M	AS	0.0361	3.5-inf	50	4.38	0.0361	1	2.0	0.0849
	PD	0.0253	3.5-inf	26	4.59	0.0253			
	FD	0.0917	3.5-inf	104	4.50	0.0917	5	4.8	P(i)
	S/H	0.3433	3.5-inf	609	4.55	0.3433			1.000
PH_01M	AS	0.0354	3.0-inf	50	4.19	0.0354	1	2.0	0.0587
	PD	0.0307	3.0-inf	26	4.43	0.0307			
	FD	0.0846	3.0-inf	104	4.33	0.0846	4	3.8	P(i)
	S/H	0.3516	3.0-inf	609	4.39	0.3516			1.000
CA_CL	AS	0.0309	0.0-0.2	9	0.14	0.0250	1	11.1	0.0311
			0.2-1.0	41	0.29	0.0355	3	7.3	0.0489
			1.0-4.0	.	.	.	.	.	.
			4.0-inf	.	.	.	.	.	.
	PD	0.0329	0.0-0.2	17	0.10	0.0314	4	23.5	
			0.2-1.0	4	0.52	0.0179			
			1.0-4.0	5	2.36	0.1039			
			4.0-inf	.	.	.	.	.	
	FD	0.1608	0.0-0.2	56	0.09	0.0308	9	15.8	
			0.2-1.0	32	0.52	0.1320	11	35.5	
			1.0-4.0	11	1.79	0.3443	1	9.1	
			4.0-inf	4	4.37	1.5367	1	25.0	P(i)
	S/H	0.8124	0.0-0.2	224	0.10	0.1179			0.380
			0.2-1.0	303	0.47	0.6925			0.479
			1.0-4.0	66	1.89	2.0407			0.110
			4.0-inf	16	6.27	6.7793			0.031

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
MG_CL	AS	0.0083	0.0-0.2	29	0.07	0.0073			0.0113
			0.2-0.5	21	0.22	0.0094			0.0166
			0.5-inf	.	.	.	.	.	.
	PD	0.0234	0.0-0.2	13	0.08	0.0140	1	7.7	
			0.2-0.5	7	0.30	0.0099			
			0.5-inf	6	0.74	0.1189	1	16.7	
	FD	0.0668	0.0-0.2	59	0.08	0.0250	7	11.9	
			0.2-0.5	28	0.31	0.0372	5	17.9	
			0.5-inf	17	0.77	0.3683	1	5.9	P(i)
	S/H	0.2547	0.0-0.2	279	0.13	0.1147			0.469
			0.2-0.5	268	0.32	0.3489			0.424
			0.5-inf	62	0.65	0.4968			0.107
K_CL	AS	0.0116	0.0-0.2	23	0.04	0.0102			0.0065
			0.2-0.4	25	0.25	0.0109			0.0173
			0.4-inf	1	0.57	0.4610	1	100.0	.
	PD	0.0110	0.0-0.2	19	0.09	0.0093			
			0.2-0.4	5	0.28	0.0170			
			0.4-inf	2	0.56	0.0212			
	FD	0.0237	0.0-0.2	80	0.09	0.0185	10	12.5	
			0.2-0.4	21	0.28	0.0398	5	23.8	
			0.4-inf	3	0.53	0.2341	1	33.3	P(i)
	S/H	0.1004	0.0-0.2	476	0.12	0.0817			0.780
			0.2-0.4	132	0.27	0.1651			0.218
			0.4-inf	1	0.47	0.2758			0.003
NA_CL	AS	0.0116	0.0-0.05	38	0.02	0.0104	1	2.6	0.0099
			0.05-0.07	6	0.06	0.0112			0.0039
			0.07-0.2	4	0.08	0.0545	3	75.0	0.0034
			0.2-inf	.	.	.	.	.	.
	PD	0.0094	0.0-0.05	19	0.03	0.0085			
			0.05-0.07	3	0.06	0.0165			
			0.07-0.2	2	0.10	0.0060			
			0.2-inf	.	.	.	.	.	
	FD	0.0154	0.0-0.05	90	0.03	0.0115	5	5.6	
			0.05-0.07	8	0.05	0.0351	1	12.5	
			0.07-0.2	3	0.09	0.0519	2	66.7	
			0.2-inf	1	0.20	0.0219			P(i)
	S/H	0.0312	0.0-0.05	523	0.03	0.0248			0.855
			0.05-0.07	74	0.06	0.0580			0.121
			0.07-0.2	12	0.10	0.1214			0.024
			0.2-inf	.	.	.			.
CA_OAC	AS	0.0261	0.0-0.2	17	0.14	0.0220	2	11.8	0.0392
			0.2-0.5	33	0.27	0.0322	8	24.2	0.0490
			0.5-1.5	.	.	.	.	.	.
			1.5-inf	.	.	.	.	.	.
	PD	0.0724	0.0-0.2	15	0.09	0.0214	1	6.7	
			0.2-0.5	3	0.29	0.1298	1	33.3	
			0.5-1.5	4	0.84	0.0472			
			1.5-inf	4	2.45	0.2309	1	25.0	
	FD	0.1543	0.0-0.2	52	0.09	0.0270	12	23.1	
			0.2-0.5	19	0.33	0.0658	7	36.8	
			0.5-1.5	21	0.86	0.1087	6	28.6	
			1.5-inf	9	3.06	1.2389	3	33.3	P(i)
	S/H	0.8353	0.0-0.2	218	0.11	0.1259			0.373
			0.2-0.5	158	0.32	0.3672			0.250
			0.5-1.5	190	0.82	1.2118			0.300
			1.5-inf	43	3.87	4.3309			0.077

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
MG_OAC	AS	0.0106	0.0-0.1	24	0.05	0.0064			0.0123
			0.1-0.2	1	0.20	0.0014			.
			0.2-0.6	25	0.24	0.0162	1	4.0	0.0181
			0.6-1.0	.	.	.	.	.	.
			1.0-inf	.	.	.	.	.	.
	PD	0.0152	0.0-0.1	9	0.05	0.0072			
			0.1-0.2	3	0.16	0.0071			
			0.2-0.6	10	0.35	0.0131			
			0.6-1.0	3	0.80	0.1185	1	33.3	
			1.0-inf	1	1.13	0.0742			
	FD	0.0367	0.0-0.1	43	0.06	0.0172	2	4.7	
			0.1-0.2	15	0.16	0.0343	4	26.7	
			0.2-0.6	32	0.35	0.0317	4	12.5	
			0.6-1.0	11	0.75	0.1296	1	9.1	
			1.0-inf	3	1.22	0.6794	1	33.3	P(i)
	S/H	0.2650	0.0-0.1	54	0.07	0.0463			0.114
			0.1-0.2	177	0.15	0.1341			0.283
			0.2-0.6	355	0.35	0.3481			0.559
			0.6-1.0	22	0.78	0.6105			0.041
			1.0-inf	1	1.67	0.6873			0.003
K_OAC	AS	0.0219	0.0-0.1	24	0.04	0.0087			0.0105
			0.1-0.2	.	.	.	.	.	.
			0.2-inf	26	0.26	0.0410	3	11.5	0.0269
	PD	0.0163	0.0-0.1	10	0.06	0.0083			
			0.1-0.2	8	0.12	0.0089			
			0.2-inf	7	0.33	0.0468	1	14.3	
	FD	0.0342	0.0-0.1	47	0.06	0.0114	2	4.3	
			0.1-0.2	32	0.14	0.0224	5	14.7	
			0.2-inf	25	0.30	0.0973	7	29.2	P(i)
	S/H	0.0880	0.0-0.1	165	0.07	0.0398			0.289
			0.1-0.2	321	0.14	0.0885			0.512
			0.2-inf	123	0.27	0.1567			0.199
NA_OAC	AS	0.0109	0.0-0.0	44	0.03	0.0070			0.0089
			0.0-0.1	4	0.07	0.0342	1	25.0	0.0148
			0.1-inf	.	.	.	.	.	.
	PD	0.0058	0.0-0.0	21	0.02	0.0047			
			0.0-0.1	2	0.06	0.0104			
			0.1-inf	2	0.13	0.0184			
	FD	0.0122	0.0-0.0	89	0.03	0.0115	1	1.1	
			0.0-0.1	12	0.06	0.0167	1	8.3	
			0.1-0.2	.	.	.	.	.	
			0.2-inf	1	0.26	0.0233			P(i)
	S/H	0.0326	0.0-0.0	515	0.03	0.0234			0.838
			0.0-0.1	83	0.06	0.0641			0.139
			0.1-inf	10	0.15	0.2127			0.018

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs >DQO n	Pairs >DQO %	Between-batch SD
CEC_CL	AS	0.6535	0.0-2.5	6	1.14	0.4028	2	33.3	0.4537
			2.5-8.0	26	6.63	0.4715	4	15.4	0.6417
			8.0-15.0	18	9.59	0.9277	2	11.1	1.3158
			15.0-inf	.	.	.	.	.	.
	PD	0.8022	0.0-2.5	1	2.02	0.1676			
			2.5-8.0	16	5.60	0.7372	3	18.8	
			8.0-15.0	6	11.35	0.7286	1	16.7	
			15.0-inf	3	19.11	2.5525	2	66.7	
	FD	1.0434	0.0-2.5	6	2.06	0.3745	2	33.3	
			2.5-8.0	60	5.53	0.6343	20	33.3	
			8.0-15.0	33	11.18	1.4862	7	21.2	
			15.0-inf	5	19.17	2.7369	3	60.0	P(i)
	S/H	3.8552	0.0-2.5	1	1.65	0.2475			0.007
			2.5-8.0	343	5.84	2.9598			0.569
			8.0-15.0	242	10.04	4.9852			0.384
			15.0-inf	23	16.76	6.3937			0.040
CEC_OAC	AS	1.4975	0.0-2.5	6	1.43	0.1333			0.4756
			2.5-8.0	.	.	.	.	.	.
			8.0-16.0	16	14.36	1.7216	4	25.0	1.7984
			16.0-inf	28	20.58	0.9934	1	3.6	1.5125
	PD	1.0692	0.0-2.5	.	.	.	.	.	
			2.5-8.0	7	5.15	0.5376	2	28.6	
			8.0-16.0	13	11.58	0.5799	1	7.7	
			16.0-inf	6	28.38	2.5168			
	FD	2.1533	0.0-2.5	2	1.87	0.1115			
			2.5-8.0	28	6.04	1.3210	7	25.0	
			8.0-16.0	43	12.21	2.1032	10	23.3	
			16.0-inf	31	23.10	2.7704	8	25.8	P(i)
	S/H	6.2257	0.0-2.5	1	1.39	0.1838			0.004
			2.5-8.0	78	5.68	2.7096			0.142
			8.0-16.0	375	11.25	4.8001			0.599
			16.0-inf	155	23.32	11.6441			0.255
AC_KCL	AS	0.2289	0.0-2.5	12	0.94	0.1106			0.2669
			2.5-4.5	35	3.49	0.4592	1	2.9	0.4492
			4.5-inf	3	5.02	0.5019			0.1167
	PD	0.2523	0.0-2.5	17	1.11	0.0635			
			2.5-4.5	4	3.45	0.7769	1	25.0	
			4.5-inf	5	6.40	0.2512			
	FD	0.4554	0.0-2.5	63	1.27	0.3770	4	6.3	
			2.5-4.5	32	3.36	0.6298	3	9.4	
			4.5-inf	9	6.57	0.5756	1	11.1	P(i)
	S/H	1.5754	0.0-2.5	402	1.54	1.0775			0.671
			2.5-4.5	153	3.31	1.8788			0.242
			4.5-inf	54	5.89	4.5821			0.087
AC_BACL	AS	2.3814	0.0-2.5	6	0.99	0.5059	3	50.0	0.7581
			2.5-10.0	.	.	.	.	.	.
			10.0-30.0	33	18.34	2.4070	2	6.1	2.3378
			30.0-inf	11	34.94	2.0840			2.0669
	PD	1.7449	0.0-2.5	.	.	.	.	.	
			2.5-10.0	15	6.44	0.9713	4	26.7	
			10.0-30.0	9	13.56	2.2901	3	33.3	
			30.0-inf	2	47.64	3.6708			
	FD	2.6042	0.0-2.5	3	2.06	0.3227			
			2.5-10.0	46	6.80	1.2158	11	23.9	
			10.0-30.0	47	16.15	3.3417	10	21.3	
			30.0-inf	8	34.52	13.1190	1	12.5	P(i)
	S/H	7.8735	0.0-2.5	1	1.11	0.1768			0.004
			2.5-10.0	256	6.87	4.9756			0.432
			10.0-30.0	341	16.53	10.1933			0.543
			30.0-inf	11	45.36	9.1530			0.020

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
AL_KCL	AS	0.1644	0.0-2.5	14	0.95	0.1231			0.1110
			2.5-5.0	36	3.25	0.2754	2	5.6	0.3911
			5.0-inf	.	.	.	.	.	.
	PD	0.2874	0.0-2.5	17	0.88	0.1362			
			2.5-5.0	6	3.40	0.6320	1	16.7	
			5.0-inf	3	8.16	0.5739			
	FD	0.3124	0.0-2.5	73	1.25	0.2498	3	4.1	
			2.5-5.0	24	3.29	0.4369	3	12.5	
			5.0-inf	6	6.90	0.5138			P(i)
	S/H	1.5319	0.0-2.5	412	1.28	1.0008			0.688
			2.5-5.0	163	3.17	2.2599			0.256
			5.0-inf	34	6.01	4.7630			0.055
CA_CL2	AS	0.0819	0.0-0.5	25	0.38	0.1188	15	60.0	0.0706
			0.5-1.0	25	0.58	0.0365	9	36.0	0.0452
			1.0-inf	.	.	.	.	.	.
	PD	0.0244	0.0-0.5	13	0.36	0.0177	4	30.8	
			0.5-1.0	13	0.62	0.0326	4	30.8	
			1.0-inf	.	.	.	.	.	
	FD	0.0332	0.0-0.5	49	0.37	0.0335	24	50.0	
			0.5-1.0	53	0.60	0.0329	14	25.9	
			1.0-inf	2	1.46	1.4871	2	100.0	P(i)
	S/H	0.1923	0.0-0.5	345	0.43	0.1308			0.552
			0.5-1.0	264	0.58	0.2681			0.448
			1.0-inf	.	.	.			.
MG_CL2	AS	0.0085	0.0-0.05	24	0.03	0.0035	8	33.3	0.0050
			0.05-0.1	2	0.09	0.0075	1	50.0	0.0110
			0.1-0.2	24	0.13	0.0108	4	16.7	0.0194
			0.2-inf	.	.	.	.	.	.
	PD	0.0095	0.0-0.05	8	0.02	0.0017	3	37.5	
			0.05-0.1	5	0.07	0.0083	2	40.0	
			0.1-0.2	10	0.15	0.0129	3	30.0	
			0.2-inf	3	0.24	0.0071			
	FD	0.0179	0.0-0.05	36	0.03	0.0032	14	38.9	
			0.05-0.1	25	0.07	0.0164	12	48.0	
			0.1-0.2	36	0.14	0.0206	11	30.6	
			0.2-inf	7	0.30	0.1882	3	42.9	P(i)
	S/H	0.0586	0.0-0.05	64	0.04	0.0283			0.125
			0.05-0.1	266	0.07	0.0508			0.424
			0.1-0.2	277	0.13	0.0742			0.444
			0.2-inf	2	0.28	0.0827			0.007
K_CL2	AS	0.0046	0.0-0.05	27	0.02	0.0045	13	48.1	0.0026
			0.05-inf	23	0.07	0.0052	3	13.0	0.0114
	PD	0.0024	0.0-0.05	22	0.01	0.0020	12	54.5	
			0.05-inf	4	0.07	0.0054	1	25.0	
	FD	0.0130	0.0-0.05	94	0.02	0.0045	54	57.4	
			0.05-inf	10	0.10	0.0759	7	70.0	P(i)
	S/H	0.0188	0.0-0.05	535	0.02	0.0167			0.881
			0.05-inf	74	0.06	0.0339			0.119

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
NA_CL2	AS	0.0032	0.0-0.02	23	0.01	0.0014	9	39.1	0.0023
			0.02-0.05	27	0.03	0.0065	8	29.6	0.0055
			0.05-inf	.	.	.	.	.	.
	PD	0.0027	0.0-0.02	21	0.02	0.0025	11	52.4	
			0.02-0.05	3	0.03	0.0029	1	33.3	
			0.05-inf	2	0.08	0.0038			
	FD	0.0076	0.0-0.02	73	0.02	0.0051	49	67.1	
			0.02-0.05	29	0.03	0.0092	21	72.4	
			0.05-inf	2	0.15	0.0316	2	100.0	P(i)
	S/H	0.0318	0.0-0.02	382	0.02	0.0104			0.627
			0.02-0.05	202	0.03	0.0294			0.330
			0.05-inf	25	0.11	0.3646			0.043
FE_CL2	AS	0.0037	0.0-0.02	30	0.02	0.0037	8	26.7	0.0047
			0.02-0.05	9	0.04	0.0050	3	33.3	0.0050
			0.05-inf	3	0.06	0.0042	1	33.3	0.0051
	PD	0.0009	0.0-0.02	9	0.00	0.0009	4	44.4	
			0.02-0.05	.	.	.	.	.	
			0.05-inf	.	.	.	.	.	
	FD	0.0025	0.0-0.02	42	0.00	0.0025	23	53.5	
			0.02-0.05	1	0.03	0.0021			
			0.05-inf	1	0.22	0.2524	1	100.0	P(i)
	S/H	0.0052	0.0-0.02	531	0.00	0.0033			0.851
			0.02-0.05	12	0.03	0.0864			0.020
			0.05-inf	.	.	.			.
AL_CL2	AS	0.0043	0.0-0.05	14	0.01	0.0036	12	85.7	0.0028
			0.05-inf	35	0.11	0.0104	12	34.3	0.0278
	PD	0.0073	0.0-0.05	18	0.01	0.0042	11	61.1	
			0.05-inf	6	0.08	0.0351	3	50.0	
	FD	0.0113	0.0-0.05	66	0.01	0.0061	55	84.6	
			0.05-inf	21	0.08	0.0574	8	36.4	P(i)
	S/H	0.0282	0.0-0.05	545	0.02	0.0229			0.882
			0.05-inf	57	0.08	0.0752			0.098
FE_PYP	AS	0.0317	0.0-0.2	6	0.04	0.0063			0.0083
			0.2-0.33	.	.	.	.	.	.
			0.33-0.7	37	0.62	0.0452	2	5.4	0.0613
			0.7-inf	7	0.85	0.0383			0.0964
	PD	0.0295	0.0-0.2	10	0.11	0.0104			
			0.2-0.33	7	0.26	0.0203			
			0.33-0.7	6	0.49	0.0445	2	33.3	
			0.7-inf	3	1.33	0.0343			
	FD	0.0605	0.0-0.2	30	0.10	0.0322	2	6.7	
			0.2-0.33	21	0.27	0.0511	6	28.6	
			0.33-0.7	36	0.52	0.0425	3	8.1	
			0.7-inf	17	1.31	0.1860	3	18.8	P(i)
	S/H	0.2970	0.0-0.2	149	0.10	0.0962			0.252
			0.2-0.33	126	0.27	0.1878			0.209
			0.33-0.7	254	0.51	0.3331			0.408
			0.7-inf	80	1.03	0.7451			0.131

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
AL_PYP	AS	0.0318	0.0-0.2	6	0.06	0.0063			0.0126
			0.2-0.33	.	.	.	.	.	.
			0.33-0.7	31	0.57	0.0417	2	6.5	0.0590
			0.7-inf	13	0.79	0.0701	2	15.4	0.0609
	PD	0.0514	0.0-0.2	10	0.12	0.0171			
			0.2-0.33	7	0.26	0.0290	1	14.3	
			0.33-0.7	6	0.47	0.0842	1	16.7	
			0.7-inf	3	1.06	0.0713			
	FD	0.0701	0.0-0.2	38	0.12	0.0279	3	7.9	
			0.2-0.33	24	0.27	0.0451	4	17.4	
			0.33-0.7	29	0.50	0.0875	2	6.7	
			0.7-inf	13	1.07	0.1668	4	30.8	P(i)
	S/H	0.2233	0.0-0.2	193	0.13	0.0972			0.324
			0.2-0.33	100	0.25	0.1352			0.172
			0.33-0.7	243	0.43	0.2771			0.387
			0.7-inf	73	0.88	0.5254			0.117
FE_AO	AS	0.0607	0.0-0.2	6	0.08	0.0657	3	50.0	0.0425
			0.2-0.33	.	.	.	.	.	.
			0.33-0.6	26	0.41	0.0193			0.0494
			0.6-inf	18	0.97	0.0970	2	11.1	0.2413
	PD	0.1115	0.0-0.2	9	0.13	0.0377	2	22.2	
			0.2-0.33	6	0.28	0.0313			
			0.33-0.6	4	0.46	0.0501			
			0.6-inf	7	1.02	0.3281	2	28.6	
	FD	0.0636	0.0-0.2	40	0.12	0.0230	2	5.0	
			0.2-0.33	22	0.26	0.0446	6	27.3	
			0.33-0.6	21	0.44	0.1053	8	40.0	
			0.6-inf	21	1.13	0.0858	2	9.1	P(i)
	S/H	0.3413	0.0-0.2	181	0.15	0.1301			0.306
			0.2-0.33	115	0.27	0.2257			0.193
			0.33-0.6	156	0.47	0.3491			0.253
			0.6-inf	157	0.86	0.6845			0.248
AL_AO	AS	0.0239	0.0-0.2	6	0.06	0.0088			0.0115
			0.2-0.33	1	0.30	0.0184			.
			0.33-0.7	26	0.44	0.0208			0.0707
			0.7-inf	17	0.95	0.0905	3	17.6	0.0936
	PD	0.0762	0.0-0.2	12	0.14	0.0120			
			0.2-0.33	5	0.25	0.0268			
			0.33-0.7	6	0.54	0.1803	1	16.7	
			0.7-inf	3	1.03	0.0489			
	FD	0.0523	0.0-0.2	48	0.13	0.0153	1	2.1	
			0.2-0.33	21	0.25	0.0604	6	28.6	
			0.33-0.7	21	0.50	0.0524	3	14.3	
			0.7-inf	14	1.00	0.1451	4	28.6	P(i)
	S/H	0.2510	0.0-0.2	187	0.14	0.0722			0.323
			0.2-0.33	143	0.24	0.1658			0.232
			0.33-0.7	211	0.47	0.3497			0.337
			0.7-inf	68	0.93	0.6602			0.108

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
FE_CD	AS	0.1120	0.0-0.33	6	0.19	0.0319	1	16.7	0.0595
			0.33-1.4	12	1.02	0.2884	3	25.0	0.0996
			1.4-3.0	32	1.87	0.0838			0.2683
			3.0-inf	.	.	.	.	.	.
	PD	0.1086	0.0-0.33	1	0.26	0.0127			
			0.33-1.4	12	1.08	0.0500			
			1.4-3.0	10	2.11	0.1059			
			3.0-inf	3	5.02	0.1752			
	FD	0.2724	0.0-0.33	4	0.27	0.0237			
			0.33-1.4	29	0.89	0.0465	1	3.4	
			1.4-3.0	51	2.28	0.2443	6	11.5	
			3.0-inf	20	5.24	0.6112	2	10.5	P(i)
	S/H	1.1583	0.0-0.33	1	0.21	0.0283			0.007
			0.33-1.4	65	1.07	0.5859			0.119
			1.4-3.0	454	1.98	1.0252			0.728
			3.0-inf	89	4.24	2.3534			0.145
AL_CD	AS	0.0551	0.0-0.2	6	0.05	0.0066			0.0137
			0.2-0.33	.	.	.	.	.	.
			0.33-0.6	27	0.50	0.0649	1	3.7	0.0591
			0.6-inf	17	0.76	0.0540			0.0728
	PD	0.0179	0.0-0.2	4	0.16	0.0065			
			0.2-0.33	12	0.27	0.0114			
			0.33-0.6	6	0.46	0.0159			
			0.6-inf	4	0.88	0.0403			
	FD	0.0459	0.0-0.2	15	0.14	0.0124			
			0.2-0.33	28	0.27	0.0209	1	3.6	
			0.33-0.6	39	0.47	0.0431	4	10.3	
			0.6-inf	22	0.81	0.1113	4	18.2	P(i)
	S/H	0.2127	0.0-0.2	52	0.16	0.0997			0.097
			0.2-0.33	142	0.27	0.1302			0.242
			0.33-0.6	311	0.45	0.2099			0.498
			0.6-inf	104	0.76	0.4099			0.164
SO4_H2O	AS	0.9452	0.0-5.0	7	3.37	0.9036	1	14.3	0.9405
			5.0-10.0	10	8.56	0.8830	3	30.0	1.3778
			10.0-15.0	.	.	.	.	.	.
			15.0-inf	33	28.24	1.1965	2	6.1	2.2976
	PD	0.9319	0.0-5.0	6	2.53	0.7120	1	16.7	
			5.0-10.0	10	7.77	0.7470	2	20.0	
			10.0-15.0	4	11.75	1.0011	1	25.0	
			15.0-inf	6	18.32	1.5926	2	33.3	
	FD	1.9537	0.0-5.0	20	2.87	0.6361	3	15.0	
			5.0-10.0	33	7.61	1.8995	10	31.3	
			10.0-15.0	26	13.04	1.3794	10	35.7	
			15.0-inf	25	19.90	4.1298	5	20.8	P(i)
	S/H	5.8528	0.0-5.0	44	2.83	3.2913			0.081
			5.0-10.0	313	7.82	5.5638			0.498
			10.0-15.0	175	12.61	6.1462			0.284
			15.0-inf	77	18.15	7.8160			0.137

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
SO4_PO4	AS	6.8012	0.0-10.0	6	5.52	2.2402	2	33.3	1.8574
			10.0-50.0	10	27.08	4.7974	4	40.0	4.9756
			50.0-100	27	75.99	13.0012	3	11.1	5.1376
			100-inf	7	109.2	3.5376			6.4870
	PD	4.2747	0.0-10.0	3	7.16	0.9141	1	33.3	
			10.0-50.0	12	31.72	5.8440	4	33.3	
			50.0-100	5	74.71	2.0469			
			100-inf	6	164.8	4.5053			
	FD	9.5746	0.0-10.0	8	7.38	1.2009	4	50.0	
			10.0-50.0	45	31.11	6.2803	12	26.7	
			50.0-100	25	73.59	9.9318	11	44.0	
			100-inf	26	198.2	14.2334	2	7.7	P(i)
	S/H	55.803	10.0-10.0	4	7.02	3.8622			0.014
			10.0-50.0	241	31.84	24.7319			0.404
			50.0-100	179	69.27	53.6656			0.293
			100-inf	185	144.9	104.1			0.289
SO4_0	AS	0.1167	0.0-0.3	.	.	.	.	.	.
			0.3-1.0	8	0.62	0.0921	2	25.0	0.0703
			1.0-2.0	10	1.30	0.1383	3	30.0	0.0939
			2.0-inf	32	3.91	0.2206	10	31.3	0.3829
	PD	0.0504	0.0-0.3	5	0.13	0.1290	2	40.0	
			0.3-1.0	15	0.74	0.0391	3	20.0	
			1.0-2.0	4	1.29	0.0446			
			2.0-inf	2	2.61	0.0696			
	FD	0.1395	0.0-0.3	17	0.14	0.0397	3	17.6	
			0.3-1.0	42	0.64	0.1104	18	43.9	
			1.0-2.0	30	1.33	0.1676	16	51.6	
			2.0-inf	15	2.59	0.3689	10	66.7	P(i)
	S/H	0.6741	0.0-0.3	50	0.17	0.2372			0.084
			0.3-1.0	347	0.67	0.6382			0.555
			1.0-2.0	172	1.47	0.6733			0.290
			2.0-inf	40	2.46	1.4729			0.071
SO4_2	AS	0.1377	0.0-1.0	.	.	.	.	.	.
			1.0-2.0	.	.	.	.	.	.
			2.0-3.0	15	2.65	0.1052	3	20.0	0.1612
			3.0-inf	35	5.05	0.2156	6	17.1	0.4348
	PD	0.0685	0.0-1.0	5	0.28	0.0177			
			1.0-2.0	15	1.46	0.0748	3	20.0	
			2.0-3.0	4	2.52	0.0554			
			3.0-inf	2	4.23	0.2203	1	50.0	
	FD	0.1710	0.0-1.0	28	0.46	0.1282	16	57.1	
			1.0-2.0	24	1.44	0.1050	9	36.0	
			2.0-3.0	34	2.37	0.2191	11	33.3	
			3.0-inf	18	3.94	0.3361	11	61.1	P(i)
	S/H	0.9001	0.0-1.0	201	0.75	0.7242			0.313
			1.0-2.0	180	1.47	0.9655			0.292
			2.0-3.0	161	2.55	0.8431			0.279
			3.0-inf	67	3.64	1.3443			0.117

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
SO4_4	AS	0.1648	0.0-1.0	.	.	.	.	.	.
			1.0-3.0	.	.	.	.	.	.
			3.0-5.0	24	4.37	0.1592	6	25.0	0.2572
			5.0-inf	26	7.06	0.1940	3	11.5	0.3647
	PD	0.1129	0.0-1.0	4	0.32	0.0773	1	25.0	
			1.0-3.0	15	2.07	0.1232	3	20.0	
			3.0-5.0	5	3.98	0.0921			
			5.0-inf	2	5.64	0.1764			
	FD	0.2478	0.0-1.0	21	0.50	0.1040	11	52.4	
			1.0-3.0	35	2.10	0.2665	12	34.3	
			3.0-5.0	34	3.75	0.2196	12	35.3	
			5.0-inf	14	5.84	0.4053	7	50.0	P(i)
	S/H	1.2066	0.0-1.0	45	0.81	0.9911			0.074
			1.0-3.0	335	1.80	1.1923			0.529
			3.0-5.0	194	4.03	1.1210			0.333
			5.0-inf	35	5.33	2.0197			0.064
SO4_8	AS	0.2675	0.0-1.0	.	.	.	.	.	.
			1.0-4.0	.	.	.	.	.	.
			4.0-7.0	8	6.56	0.2892	1	12.5	0.2491
			7.0-inf	42	9.36	0.2356	1	2.4	0.5789
	PD	0.1347	0.0-1.0	2	0.23	0.0015			
			1.0-4.0	10	2.57	0.0678	1	10.0	
			4.0-7.0	9	5.10	0.1723	2	22.2	
			7.0-inf	5	8.20	0.2127			
	FD	0.3179	0.0-1.0	11	0.54	0.1071	6	54.5	
			1.0-4.0	30	2.45	0.3169	18	60.0	
			4.0-7.0	35	5.50	0.2439	7	20.0	
			7.0-inf	28	8.44	0.4314	6	21.4	P(i)
	S/H	1.9926	0.0-1.0	1	0.25	0.0997			0.003
			1.0-4.0	282	2.59	1.9205			0.442
			4.0-7.0	198	5.54	1.9462			0.330
			7.0-inf	128	8.04	2.2265			0.225
SO4_16	AS	1.1733	0.0-1.0	.	.	.	.	.	.
			1.0-8.0	.	.	.	.	.	.
			8.0-14.0	11	12.41	1.5700	1	9.1	0.6650
			14.0-inf	39	16.25	0.4291	3	7.7	0.9774
	PD	0.3402	0.0-1.0	1	0.28	0.0028			
			1.0-8.0	10	5.32	0.2701	3	30.0	
			8.0-14.0	9	10.51	0.4836	1	11.1	
			14.0-inf	6	15.10	0.2133			
	FD	0.5620	0.0-1.0	4	0.72	0.3395	3	75.0	
			1.0-8.0	33	4.61	0.6030	17	51.5	
			8.0-14.0	43	11.04	0.4706	4	9.3	
			14.0-inf	24	15.91	0.6561	7	29.2	P(i)
	S/H	3.2410	0.0-1.0	1	0.75	0.3769			0.003
			1.0-8.0	260	5.75	3.3797			0.404
			8.0-14.0	230	10.88	3.4585			0.387
			14.0-inf	118	14.98	2.6010			0.206

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
SO4_32	AS	0.4729	0.0-1.0	.	.	.	.	.	.
			1.0-16.0	.	.	.	.	.	.
			16.0-25.0	6	23.35	0.4551			1.0632
			25.0-inf	44	29.80	0.4953			1.7337
	PD	0.6459	0.0-1.0	.	.	.	.	.	
			1.0-16.0	7	11.41	0.3923	3	42.9	
			16.0-25.0	12	21.44	0.5392			
			25.0-inf	7	28.67	0.9347	1	14.3	
	FD	0.9111	0.0-1.0	.	.	.	.	.	
			1.0-16.0	28	10.45	1.0103	12	42.9	
			16.0-25.0	38	21.25	0.9039	5	13.2	
			25.0-inf	38	29.06	0.8597	4	10.5	P(i)
	S/H	5.2605	0.0-1.0	.	.	.			.
			1.0-16.0	135	13.45	6.5212			0.212
			16.0-25.0	274	19.81	5.4906			0.440
			25.0-inf	200	27.77	4.2035			0.349
C_TOT	AS	0.1132	0.0-0.3	6	0.14	0.0194			0.0263
			0.3-1.0	.	.	.	.	.	.
			1.0-3.0	8	1.69	0.0469			0.0953
			3.0-5.0	34	4.40	0.3757	2	5.9	0.2507
			5.0-inf	2	5.19	0.2221			.
	PD	0.3168	0.0-0.3	7	0.22	0.0552	2	28.6	
			0.3-1.0	12	0.67	0.1090	5	41.7	
			1.0-3.0	5	1.77	0.8215	1	20.0	
			3.0-5.0	.	.	.	.	.	
			5.0-inf	2	11.78	0.3641			
	FD	0.4458	0.0-0.3	22	0.20	0.0335	4	18.2	
			0.3-1.0	32	0.60	0.0809	10	32.3	
			1.0-3.0	32	1.71	0.3602	9	27.3	
			3.0-5.0	11	3.96	0.4092	3	27.3	
			5.0-inf	7	8.02	2.8314	5	71.4	P(i)
	S/H	1.1403	0.0-0.3	106	0.19	0.1191			0.175
			0.3-1.0	247	0.63	0.4774			0.397
			1.0-3.0	141	1.68	1.5394			0.245
			3.0-5.0	50	4.13	2.3361			0.083
			5.0-inf	65	6.56	3.5708			0.101
N_TOT	AS	0.0057	0.0-0.1	8	0.03	0.0023			0.0056
			0.1-0.2	42	0.14	0.0188	11	26.2	0.0165
			0.2-inf	.	.	.	.	.	.
	PD	0.0265	0.0-0.1	20	0.04	0.0200	6	30.0	
			0.1-0.2	3	0.14	0.0492	3	100.0	
			0.2-inf	2	0.65	0.0310			
	FD	0.0264	0.0-0.1	72	0.04	0.0172	17	23.3	
			0.1-0.2	22	0.15	0.0302	9	42.9	
			0.2-inf	10	0.39	0.0840	4	40.0	P(i)
	S/H	0.0702	0.0-0.1	434	0.04	0.0373			0.706
			0.1-0.2	105	0.18	0.1068			0.183
			0.2-inf	65	0.36	0.2333			0.101

(continued)

Table C-1. Continued

Parameter	Data set	Delta	Window	df	Mean	Within-batch SD	Pairs n	Pairs >DQO %	Between-batch SD
S_TOT	AS	0.0042	0.0-0.01	6	0.01	0.0029			0.0029
			0.01-0.04	42	0.02	0.0047	2	4.8	0.0033
			0.04-0.1	.	.	.	.	.	.
			0.1-inf	.	.	.	.	.	.
	PD	0.0065	0.0-0.01	8	0.01	0.0026			
			0.01-0.04	13	0.02	0.0085	2	15.4	
			0.04-0.1	1	0.10	0.0007			
			0.1-inf	.	.	.	.	.	
	FD	0.0070	0.0-0.01	41	0.01	0.0031			
			0.01-0.04	50	0.02	0.0056	2	4.0	
			0.04-0.1	8	0.06	0.0376	2	25.0	
			0.1-inf	1	0.25	0.1980	1	100.0	P(i)
	S/H	0.0203	0.0-0.01	143	0.01	0.0057			0.246
			0.01-0.04	427	0.02	0.0150			0.686
			0.04-0.1	39	0.06	0.1397			0.061
			0.1-inf	.	.	.			.

. A dot indicates a lack of data within the range of this window.
P(i) Proportion of the routine samples within the ith window.

Appendix D

Inordinate Data Points Influencing the Precision Estimates

The Appendix D table provides information on specific data points that have an inordinate effect on the precision estimates presented in the results and discussion of Section 3. Included is information for each datum on the sampling class/horizon group, the batch/sample number, and the reason for its effect on the estimates. Data users interested in the quality of data in specific batches will find this table particularly helpful. The table is sorted by parameter and subsorted by data set.

Table D-1. Inordinate Data Points Having a High Degree of Influence on the Precision Estimates for the Data Sets

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
MOIST	S/H	MSL / Oe		large variability
	FD	FR / A	20612-06,20	high value
		FR / Oe	20614-04,12	high value
	AS	Bs	29601-35	high value
		Bs	29606-14	high value
SP_SUR	S/H	OTC / A		large variability
	FD	ACH / Bw	29601-40,04	high value
		ACL / A	20610-18,34	high value
	PD	MSL / Bt	20609-33,19	large variability
		MSL / Bw	20614-23,02	low value
	AS	Bs	20712-28	low value
SAND	S/H	OTC / A		large variability
	FD	ACL / A	29607-09,26	low value
	PD	MSH / Bw	20711-16,29	high value
		ACH / A	20704-10,14	large variability
		MSL / Bw	20614-23,02	large variability
	AS	A	20703-03	low value
		Bs	20705-30	low value
		Bw	20704-20	high value
VCOS	FD	ACH / C	29607-05,23	high value
		SKV / Bw	20608-02,23	high value
	AS	Bs	20705-21,30	large variability
		Bs	20709-24,37	large variability
		C	20608-27	low value
		C	20614-18,31	high value

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon		Batch/sample	Reason[b]
COS	FD	ACL /	A	29607-09,26	large variability
	PD	MSH /	Bw	20711-16,29	large variability
		ACH /	BA	20710-16,07	high value
	AS		Bs	20705-21,30	large variability
			Bs	20709-24,37	large variability
			C	20614-31	high value
MS	FD	ACL /	A	29607-09,26	low value
	AS		A	20703-03	low value
			Bs	20709-37	low value
			C	29605-36	low value
FS	S/H	MSL /	BA		large variability
		OTC /	A		large variability
			Bt		low value routine
	PD	MSL /	Bw	20614-23,02	large variability
	AS		A	20703-03	low value
			Bs	20705-21,30	large variability
			Bs	20709-24,37	large variability
VFS	S/H	OTC /	A		large variability
		OTC /	Ap		large variability
	FD	SKV /	A	29606-18,26	high value
	AS		A	20703-03	low value
			Bs	20705-21	low value
			Bw	20704-01,20	high value
			C	20614-31	low value
SILT	FD	ACL /	A	29607-09,26	high value
		FL /	A	20711-25,09	low value
	PD	MSH /	Bw	20711-16,29	low value
	AS		A	20703-03	high value
			A	20709-07	high value
			Bw	20704-20	low value
COSI	FD	ACH /	Bt	20705-06,01	high value
		FL /	A	20711-25,09	low value
	PD	MSH /	Bw	20711-16,29	negative value
	AS		A	20703-03	high value
			A	20709-07	high value
			Bw	20704-20	low value
FSI	S/H	MSL /	BA		large variability
		OTC /	Ap		large variability
	FD	ACH /	Bt	20705-06,01	high value
		ACH /	Bw	29601-40,04	high value
		ACL /	A	29607-09,26	large variability
	PD	ACH /	A	20704-10,14	high value
	AS		A	20703-03	low value
			A	20709-07,16	low value
CLAY	S/H	OTC /	A		large variability
		SHL /	Bt		large variability
	AS		A	20703-03	low value
			A	20709-07	low value
			Bs	29601-35	negative value
PH_H2O	S/H	OTC /	A		large variability
		ACL /	Oe		large variability
	FD	MSL /	A	20609-18,24	low value
	AS		Bs	29601-22,35	high value

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
PH_002M	S/H	OTC / A		large variability
		ACL / Oe		large variability
	FD	MSL / A	20609-18,24	low value
	AS	A	20612-07	high value
PH_01M	S/H	OTC / A		large variability
		ACL / Oe		large variability
	FD	MSL / A	20609-18,24	low value
	AS	A	20701-19	low value
CA_CL	S/H	OTC / A		large variability
		OTC / Bt		large variability
		OTC / C		large variability
	FD	MSL / A	20609-18,24	high value
		SKV / A	20709-22,06	high value
		FR / Oe	20614-04,12	high value
	AS	A	20706-31	high value
		A	20703-03,19	large variability
MG_CL	S/H	ACC / Ap		large variability
		SHL / Oe		large variability
	FD	SKV / A	20709-22,06	high value
		FR / Oe	20614-04,12	high value
	PD	ACC / Ap	20701-28,39	large variability
K_CL	S/H	MSH / Oe		large variability
	FD	SKV / A	20709-22,06	high value
		FR / Oe	20614-04,12	high value
	AS	A	20703-19	high value
		Bs	20703-35	low value
NA_CL	S/H	MSH / Oe		large variability
	FD	MSH / Bw	29603-03,40	low value
		FR / Oe	20614-04,12	high value
	PD	SKX / Bw	20608-12,05	high value
	AS	A	29605-30	high value
		A	20707-33	high value
		Bs	20703-18	high value
		Bs	29603-04	low value
CA_OAC	S/H	OTC / A		large variability
		OTC / Bt		large variability
		OTC / C		large variability
		OTC / Bw		large variability
	FD	MSL / A	20609-18,24	high value
		OTC / Ap	20612-24,10	large variability
		SKV / A	20709-22,06	large variability
		FR / Oe	20614-04,12	high value
	PD	ACH / A	20704-10,14	large variability
		MSH / Bw	29605-12,23	high value
MG_OAC	S/H	ACC / Ap		high value
		SHL / Oe		large variability
	FD	OTC / Ap	20612-24,10	large variability
		SKV / A	20709-22,06	large variability
		FR / Oe	20614-04,12	high value
	PD	ACC / Ap	20701-28,39	large variability
		ACH / A	20704-10,14	large variability

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
K_OAC	S/H	OTC / Ap		large variability
	FD	OTC / Ap	20612-24,10	large variability
		SKV / A	20709-22,06	high value
		FR / Oe	20614-04,12	large variability
	PD	ACH / A	20704-10,14	high value
	AS	A	20702-01	high value
		Bs	29603-04	high value
		Bw	20702-12	high value
NA_OAC	S/H	FR / A		large variability
		FR / Bw		large variability
		FR / Oe		large variability
	FD	ACL / Bw	20701-17,10	low value
		FR / Oe	20614-04,12	high value
	PD	ACH / A	20704-10,14	large variability
	AS	A	20707-11	high value
		Bs	20701-16,19	low value
CEC_CL	S/H	OTC / A		large variability
	FD	SKV / A	20709-22,06	large variability
		FR / A	20612-28,08	high value *
	PD	ACH / A	20704-10,14	large variability
		ACC / Bt	20706-40,12	large variability
		FL / A	20613-18,08	high value
	AS	A	20613-31	high value
		A	20610-05,14	large variability
		C	20614-18,31	large variability
CEC_OAC	S/H	FR / Oe		large variability
	FD	SKV / A	20709-22,06	large variability
		FR / A	20612-28,08	high value *
		OTC / C	20708-05,09	high value
	PD	ACH / A	20704-10,14	high value
		FL / A	20613-18,08	high value
	AS	A	20614-06	low value
		Bw	20710-05	high value
AC_KCL	S/H	SKX / A		large variability
		ACL / Oe		large variability
	FD	MSL / A	20609-18,24	low value
		FR / C	29604-26,14	low value
		FR / A	20612-28,08	high value *
	PD	ACH / BC	20611-37,40	high value
	AS	A	20602-14	high value
		Bs	29604-15	low value
		Oa	20613-13	high value
AC_BACL	S/H	ACL / Oe		large variability
		FR / Oe		large variability
		SKX / Oe		large variability
		SKV / C		large variability
	FD	FL / Bg	20614-01,29	large variability
		OTL / Bt	29604-26,16	high value
		FR / A	20612-28,08	large variability *
	AS	A	20612-02	low value
		Oa	20612-12	low value

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
AL_KCL	S/H	MSL / C		large variability
		SKX / A		large variability
	FD	MSL / A	20609-18,24	large variability
		MSH / BC	20711-02,22	high value
		SKX / Bw	20608-18,36	high value
		FR / A	20612-28,08	high value *
	PD	ACH / BC	20611-27,40	high value
CA_CL2	S/H	ACH / Bw		large variability
		OTC / Ap		large variability
		ACL / Oe		large variability
		SHL / Oe		large variability
	FD	FR / A	20612-06,20	high value
		SKV / A	20709-22,06	high value
	PD	ACC / Ap	20701-28,39	large variability
	AS	C	20711-31	low value
MG_CL2	S/H	ACL / E		large variability
		ACC / Ap		large variability
		SHL / Oe		large variability
	FD	SKV / A	20709-22,06	high value
		FR / A	20612-06,20	high value
	AS	C	20711-31	low value
K_CL2	S/H	ACL / Oe		large variability
		FR / A		large variability
		OTC / Ap		large variability
	FD	SKV / A	20709-22,06	high value
		FR / A	20612-06,20	high value
		FR / Oe	20614-04,12	high value
	AS	C	20711-31	low value
		Bs	29606-14	high value
		A	20706-07	low value
NA_CL2	S/H	ACL / Bt		large variability
		FR / A		large variability
		FR / Oe		large variability
	FD	FR / A	20612-06,20	high value
		FR / Oe	20614-04,12	high value
	AS	C	20711-31	low value
		Bs	29606-14	high value
		A	20701-33	high value
		A	20707-11,33	large variability
FE_CL2	S/H	FR / A		large variability
		FR / C		large variability
	FD	FR / A	20612-06,20	high value
		SKV / A	20709-22,06	high value
	PD	FL / A	20613-18,08	high value
	AS	Bs	29601-22,35	low value
AL_CL2	S/H	FR / A		large variability
		ACH / Oe		large variability
	FD	FR / A	20612-06,20	high value
		SKV / A	20709-22,06	high value
	PD	ACH / A	20704-10,14	high value
		ACC / Bt	20706-40,12	large variability
	AS	Bw	20610-15	high value
		Bs	29601-22,35	low value

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
FE_PYP	S/H	FR / A		large variability
		FR / C		large variability
		FR / Bw		large variability
	FD	ACL / A	29607-09,26	high value
		FR / A	20612-06,20	large variability
		MSH / Bw	20602-28,30	high value
		SHL / Bt	20611-24,35	high value
	PD	ACH / A	20704-10,14	large variability
		ACH / Bw	29607-14,25	large variability
		ACH / BA	20710-16,07	low value
	AS	Bs	20705-21	low value
		Bs	20712-28	low value
AL_PYP	FD	FR / C	29604-26,16	high value
	PD	ACH / Bw	29607-14,25	large variability
	AS	Bs	20712-28,31	large variability
		A	29601-33,34	large variability
FE_AO	S/H	FR / C		large variability
		FR / Bw		large variability
	FD	ACL / C	20706-20,19	high value
	PD	ACH / BA	20710-16,07	large variability
AL_AO	S/H	FR / Bw		large variability
	FD	ACL / A	29607-09,26	high value
	PD	ACH / A	20710-16,07	high value
	AS	Bs	20705-21,30	large variability
		Bw	20611-15.25	low value
FE_CD	S/H	ACC / Bt		large variability
		ACC / Cr		large variability
		FR / C		large variability
		SHL / Bt		large variability
	FD	ACL / C	20706-20,19	high value
	PD	SKX / Bw	20608-12,05	high value
		SKX / Bw	20612-14,26	large variability
	AS	Bw	20710-05	low value
AL_CD	S/H	FR / Bw		large variability
	FD	ACH / Bw	20704-07,03	high value
		ACL / A	29607-09,26	high value
		ACL / C	20706-20,19	high value
	PD	ACC / Bt	20706-40,12	high value
	AS	Bw	20710-05	low value
SO4_H2O	S/H	FR / Oe		large variability
	FD	SKV / A	20709-22,06	high value
		FR / Oe	20614-04,12	high value
	PD	ACH / Bw	29607-14,25	large variability
	AS	A	20610-05,14	large variability
		Bw	20610-15	low value
		C	20711-14	high value
		Oa	20612-12	high value
SO4_PO4	FD	ACH / Bw	20703-28,40	large variability
		ACL / C	20706-20,19	high value
		ACH / Bw	20704-07,03	high value
	PD	OTC / C	20705-14,37	high value
		ACH / BA	20710-61,07	low value
		ACC / Bt	20706-40,12	high value
	AS	Bs	20709-24	high value
		A	20706-31	high value

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
SO4_0	S/H	FR / Oe		large variability
		ACH / Oe		large variability
		FR / A		large variability
		MSL / BA		large variability
	FD	ACL / A	20610-35,29	high value
		FR / A	20612-06,20	high value
		FR / A	20612-28,08	large variability*
	PD	MSL / Bt	20609-33,19	low value
	AS	Bs	29601-35	high value
		A	20614-31	high value
SO4_2	S/H	ACH / Oe		large variability
		FR / Oe		large variability
		FR / A		large variability
	FD	FR / A	20612-28,08	large variability*
	PD	ACH / A	20704-10,14	large variability
		ACH / Bw	20709-25,15	large variability
		SKX / Bw	20612-14,26	large variability
	AS	A	20610-14	low value
SO4_4	S/H	FR / Oe		large variability
		FR / A		large variability
	FD	ACH / Bw	20613-28,39	large variability
		FR / A	20612-28,08	large variability*
	PD	OTL / Bw	29601-41,37	large variability
	AS	Bs	20701-19	high value
		C	29605-03	low value
SO4_8	S/H	FR / Oe		large variability
		FR / A		large variability
	AS	Bs	29607-08,19	large variability
		Bw	20704-01	high value
SO4_16	S/H	FR / Oe		large variability
	PD	MSL / Bw	20614-23,02	large variability
	AS	A	20610-14	low value
		C	20614-18,31	large variability
SO4_32	S/H	FR / Oe		large variability
	PD	OTC / C	20705-14,37	low value
	AS	A	29605-30,34	large variability
		C	20614-18,31	high value
C_TOT	S/H	FR / A		large variability
	FD	FR / A	20612-06,20	large variability
		MSL / A	20609-18,24	high value
		SKV / A	20709-22,06	high value
	PD	ACH / A	20704-10,14	high value
		MSL / Bw	20614-23,02	high value
	AS	A	20611-16	high value
		Bs	20707-13	high value
		Oa	20612-12,25	large variability

(continued)

Table D-1. Continued

Parameter	Data set[a]	Sampling class/horizon	Batch/sample	Reason[b]
N_TOT	S/H	FR / A		large variability
		ACH / Cr		negative value
		ACL / Cr		negative value
		FR / Oe		large variability
	FD	MSL / A	20609-18,24	high value
		SKV / A	20709-22,06	large variability
		FR / A	20612-06,20	high value
	PD	MSH / Bw	29605-12,23	high value
		MSH / BC	29606-39,16	negative value
		MSL / Bw	20614-23,02	high value
		ACH / A	20704-10,14	high value
	AS	Bs	20707-13	high value
		A	20611-16	high value
S_TOT	S/H	ACL / A		large variability
		FR / A		large variability
		SKX / Bw		large variability
	FD	ACL / Bt	20706-04,03	high value
		FR / A	20612-06,20	high value
		SHL / A	29606-15,37	high value
		SKV / A	20709-22,06	high value
		FR / Oe	20614-04,12	high value
	PD	ACC / Bt	20706-40,12	large variability
		MSL / Bw	20614-23,02	large variability
		ACH / A	20704-10,14	high value
	AS	Bs	20701-16	high value
		Bs	20707-13	high value
		Bw	20611-15,25	large variability
		C	20608-27,38	large variability

[a] AS = audit samples; PD = preparation duplicates; FD = field duplicates; S/H = sampling class/horizon groups of routine samples.
[b] An asterisk in this column denotes an organic soil type.

NOTE: Sampling class codes are as follows: FR = frigid, OTC = calcareous, SKV = skeletal concave, SKX = skeletal convex, FL = flooded, SHL = low organic shallow, ACH = high organic acid crystalline, MSH = high organic metasedimentary, ACC = low organic clayey acid crystalline, ACL = other low organic acid crystalline, MSL = low organic metasedimentary, OTL = other low organic soils.

Appendix E

Additional Precision Plots for Moisture, Specific Surface, and Particle Size Fractions

Following are precision plots for the routine and QA data sets from the MOIST, SP_SUR, VCOS, COS, MS, FS, VFS, COSI, and FSI parameters. Since these parameters did not have specifically established DQOs, it was decided to place the routine data plots in a place separate from the audit sample / routine sample paired plots found in the results and discussion of the report. Supplemental information relating to these plots can be found in Section 3 under the parameter group heading and in Appendices C and D.

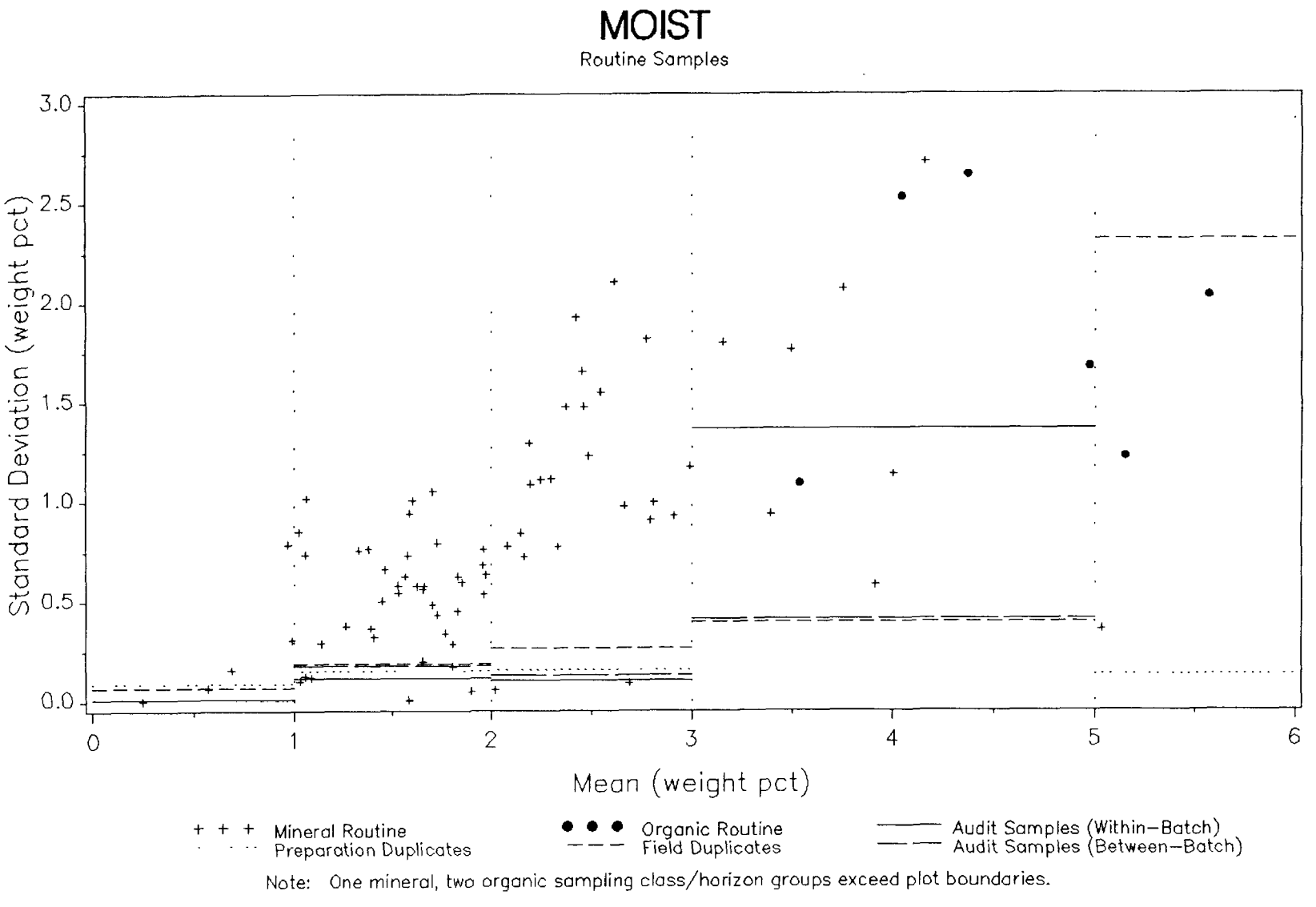

Figure E-1. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled estimates for MOIST.

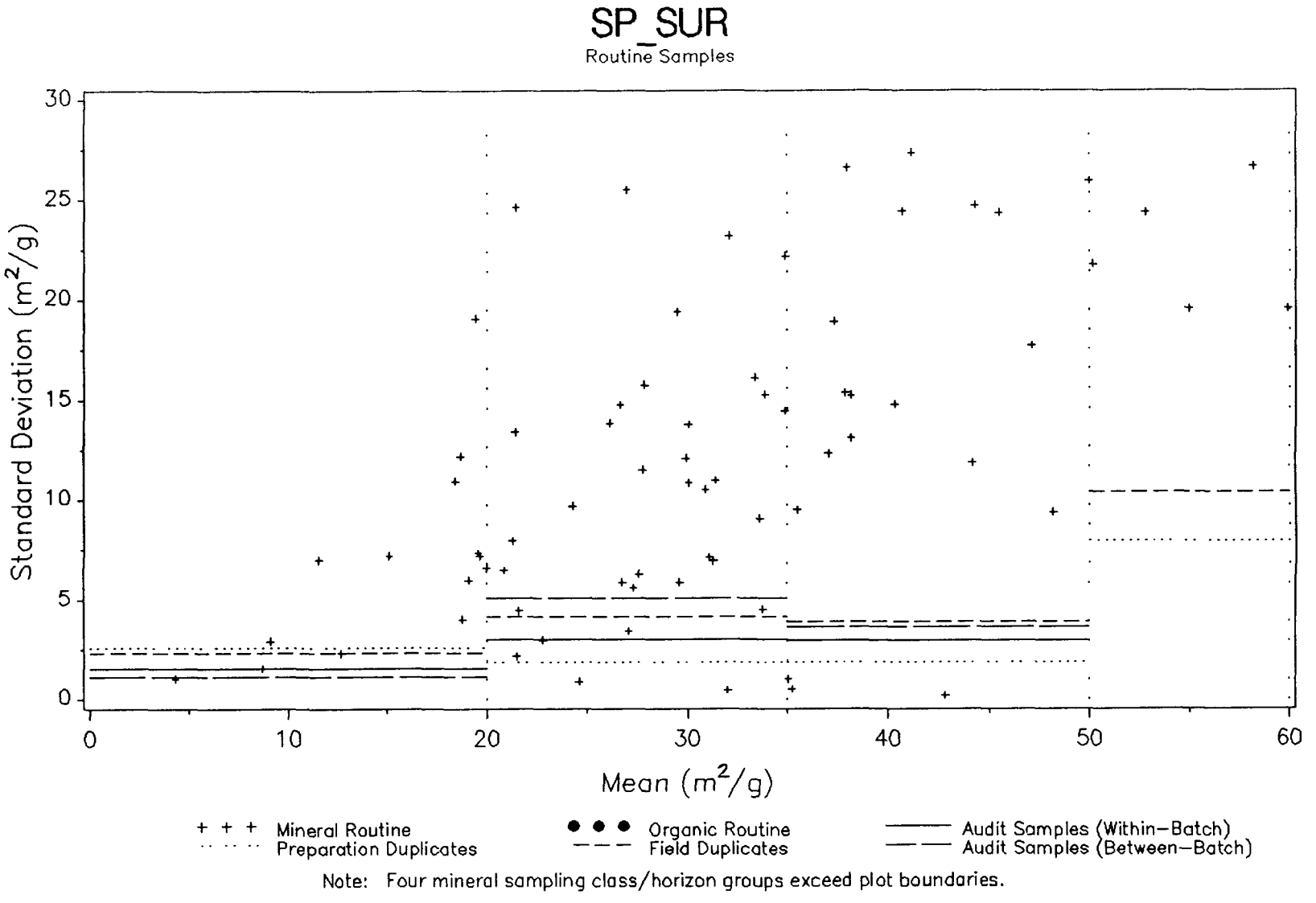

Figure E-2. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for SP_SUR.

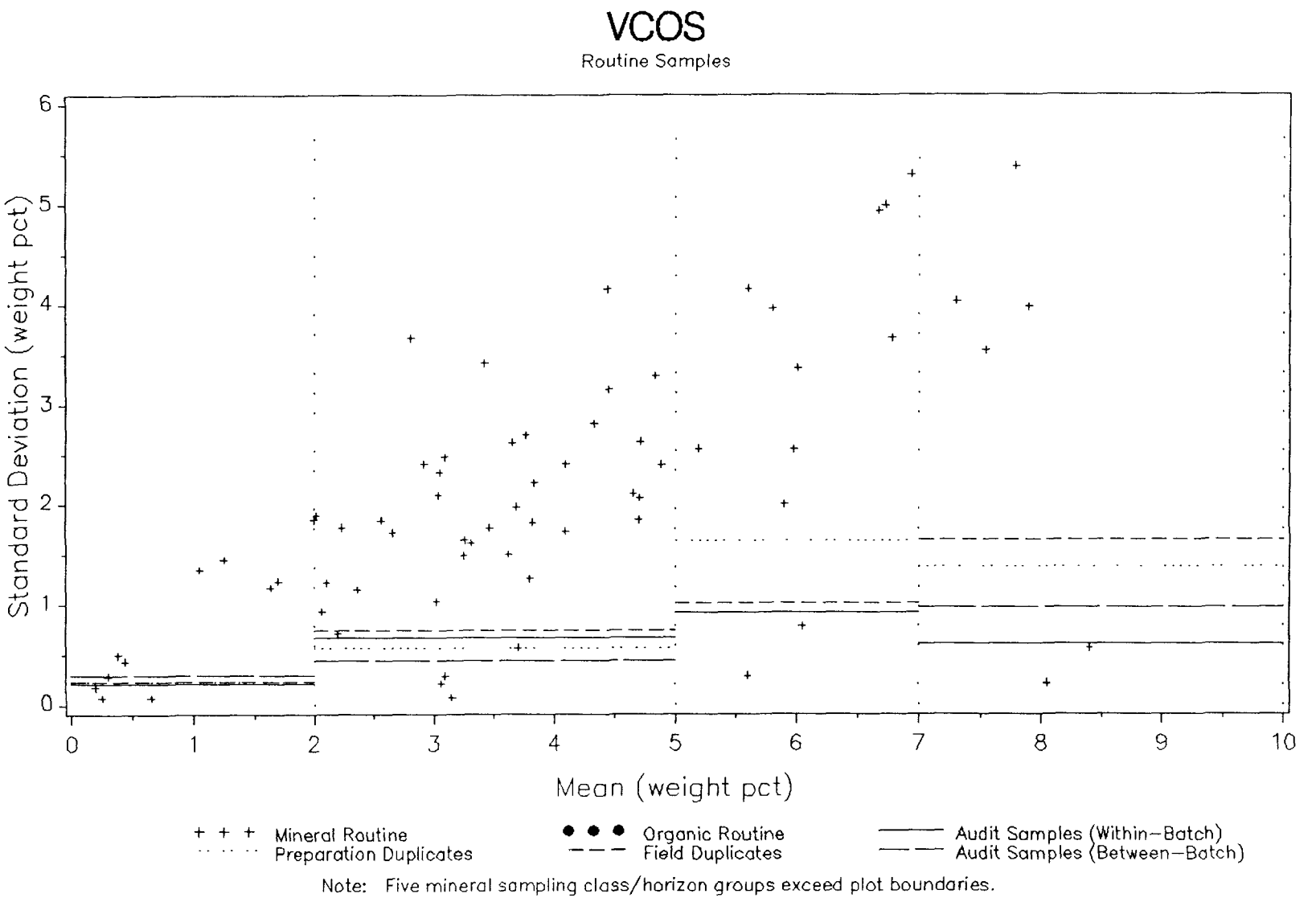

Figure E-3. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for VCOS.

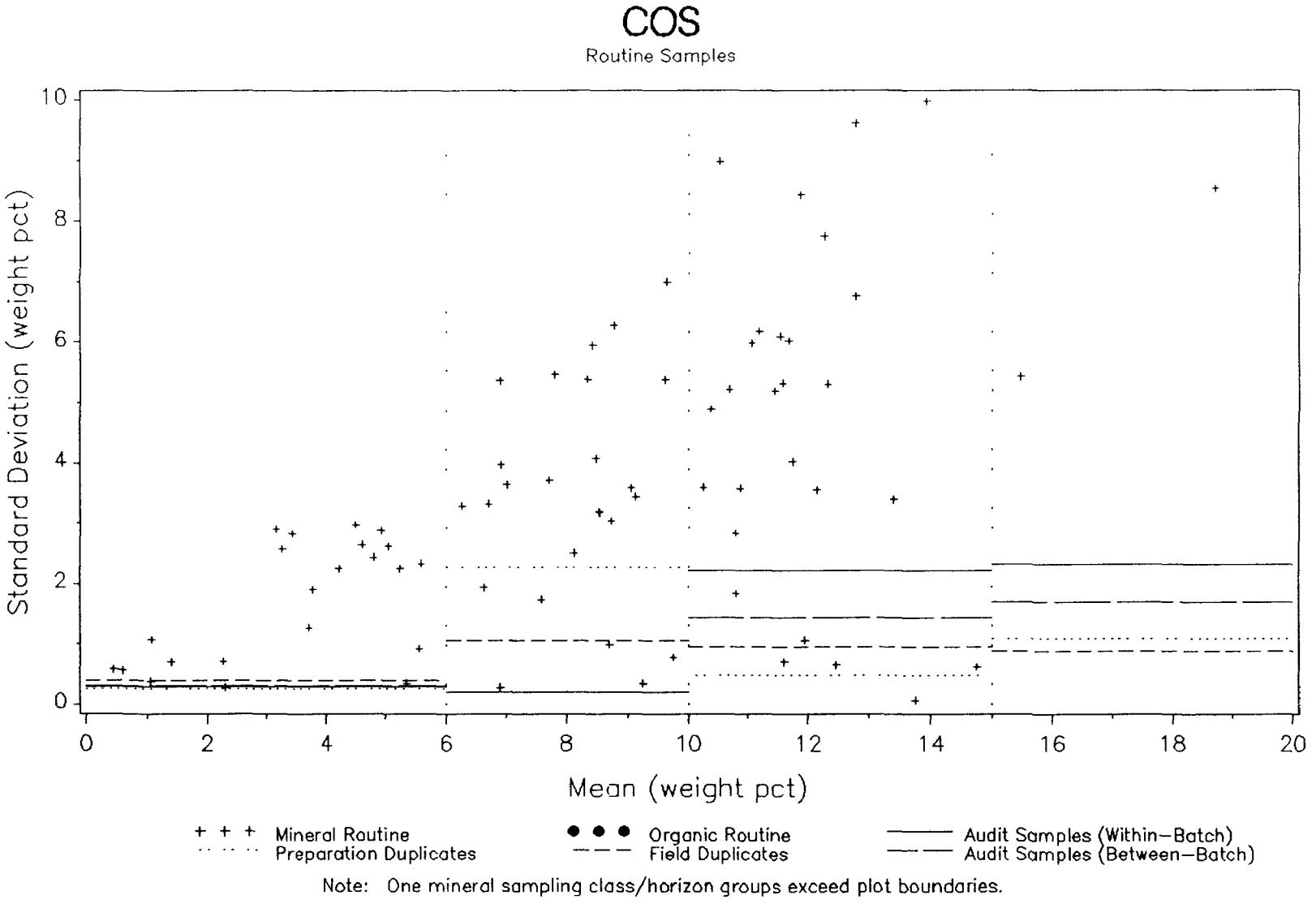

Figure E-4. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for COS.

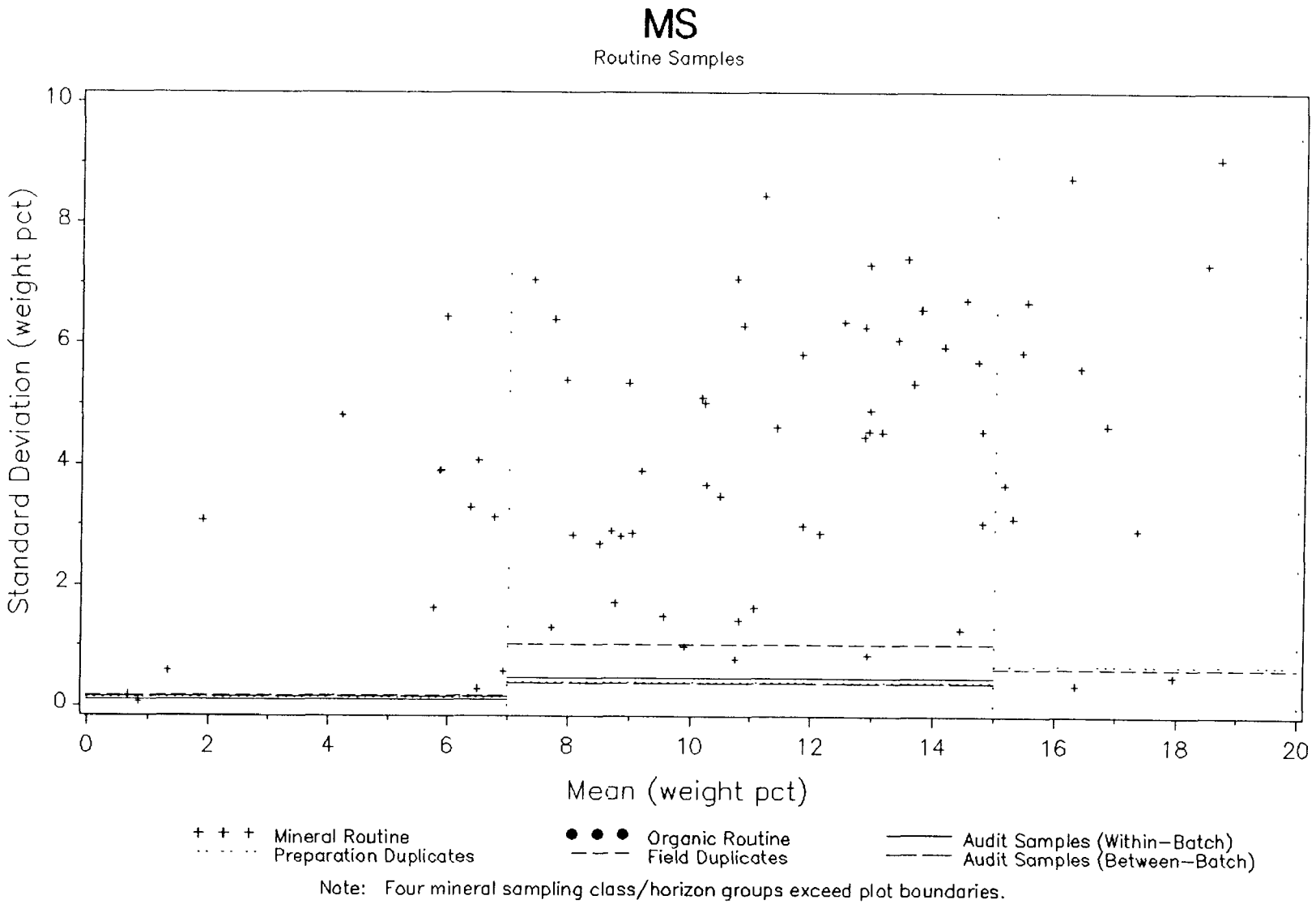

Figure E-5. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for MS.

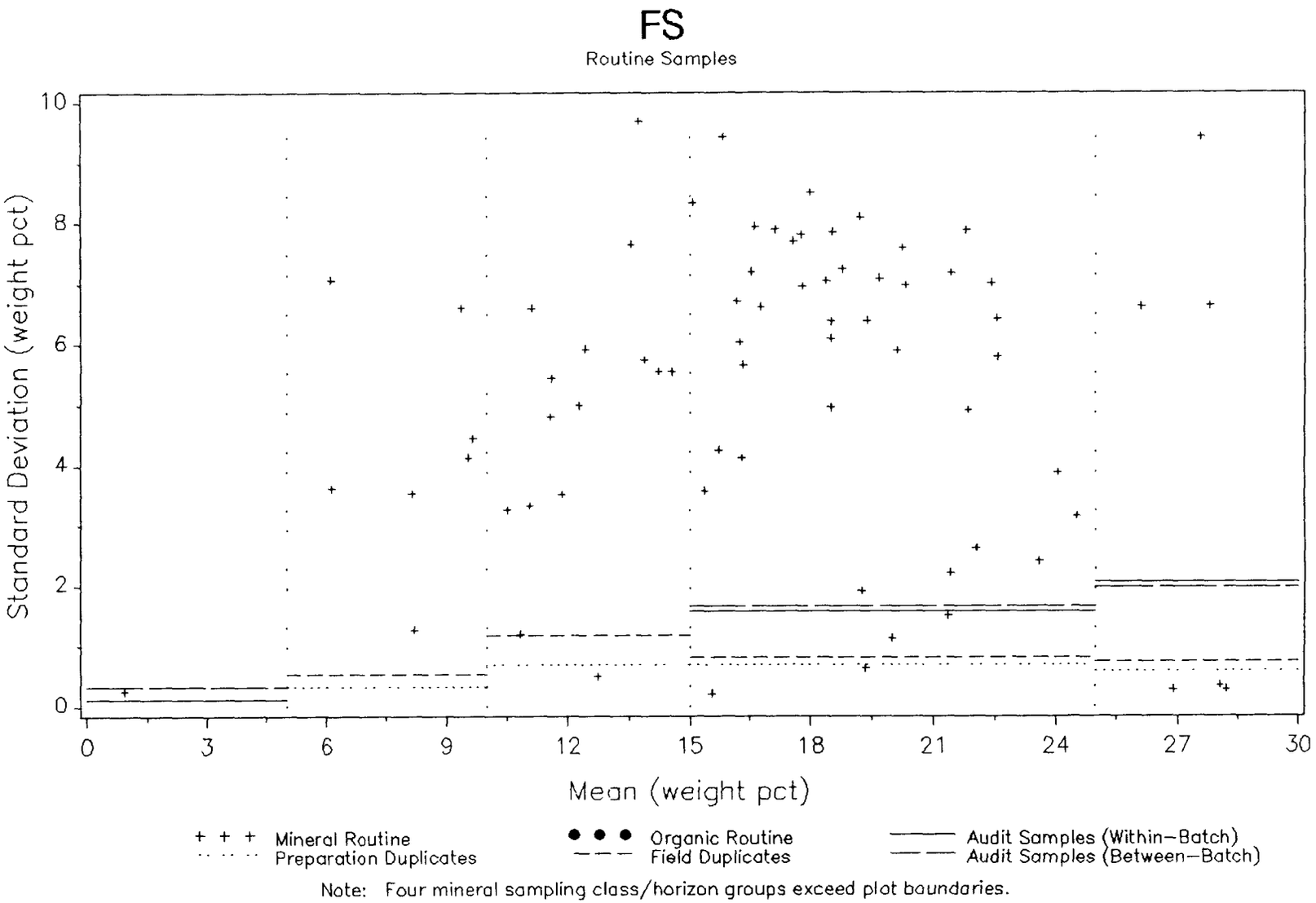

Figure E-6. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for FS.

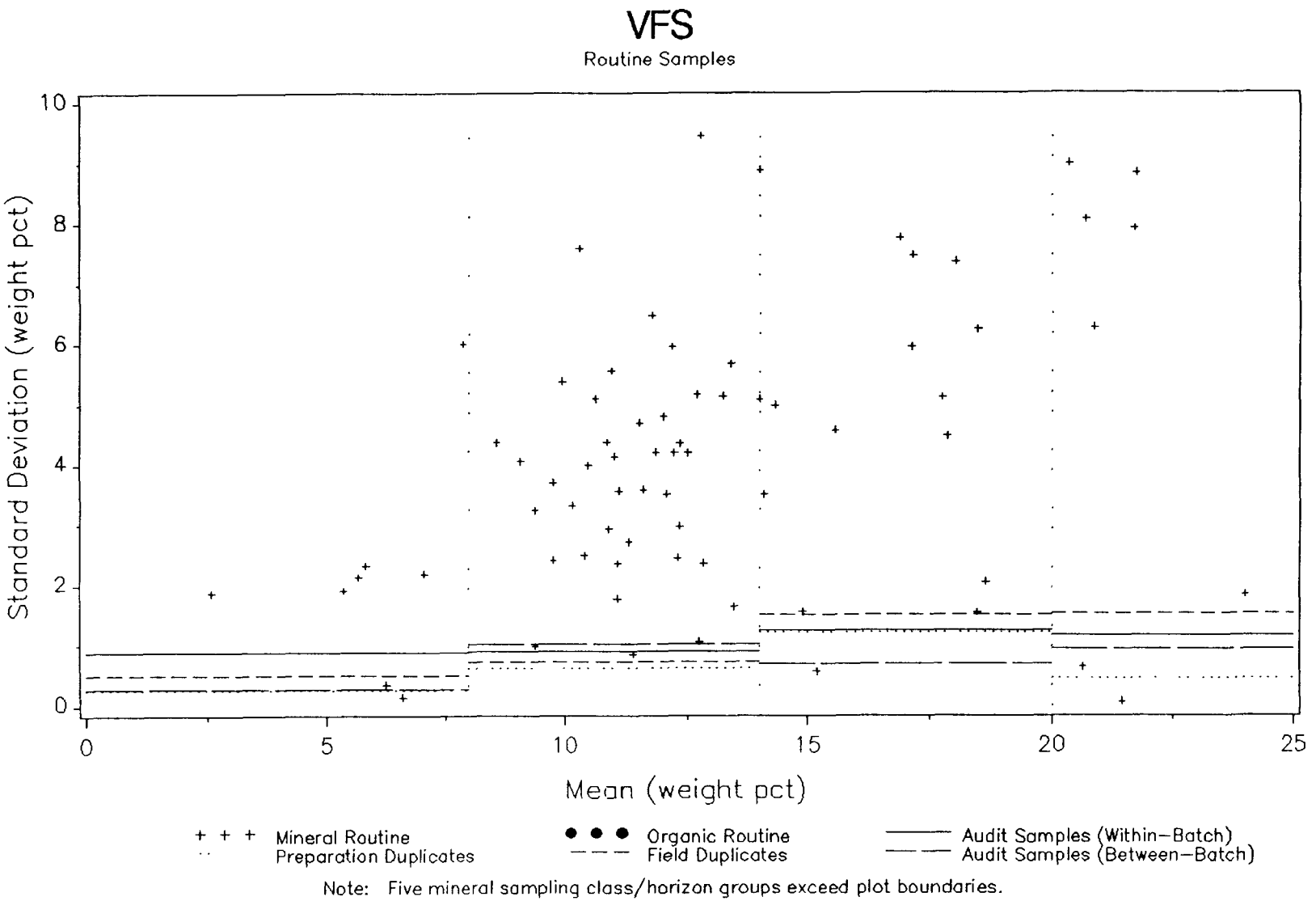

Figure E-7. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for VFS.

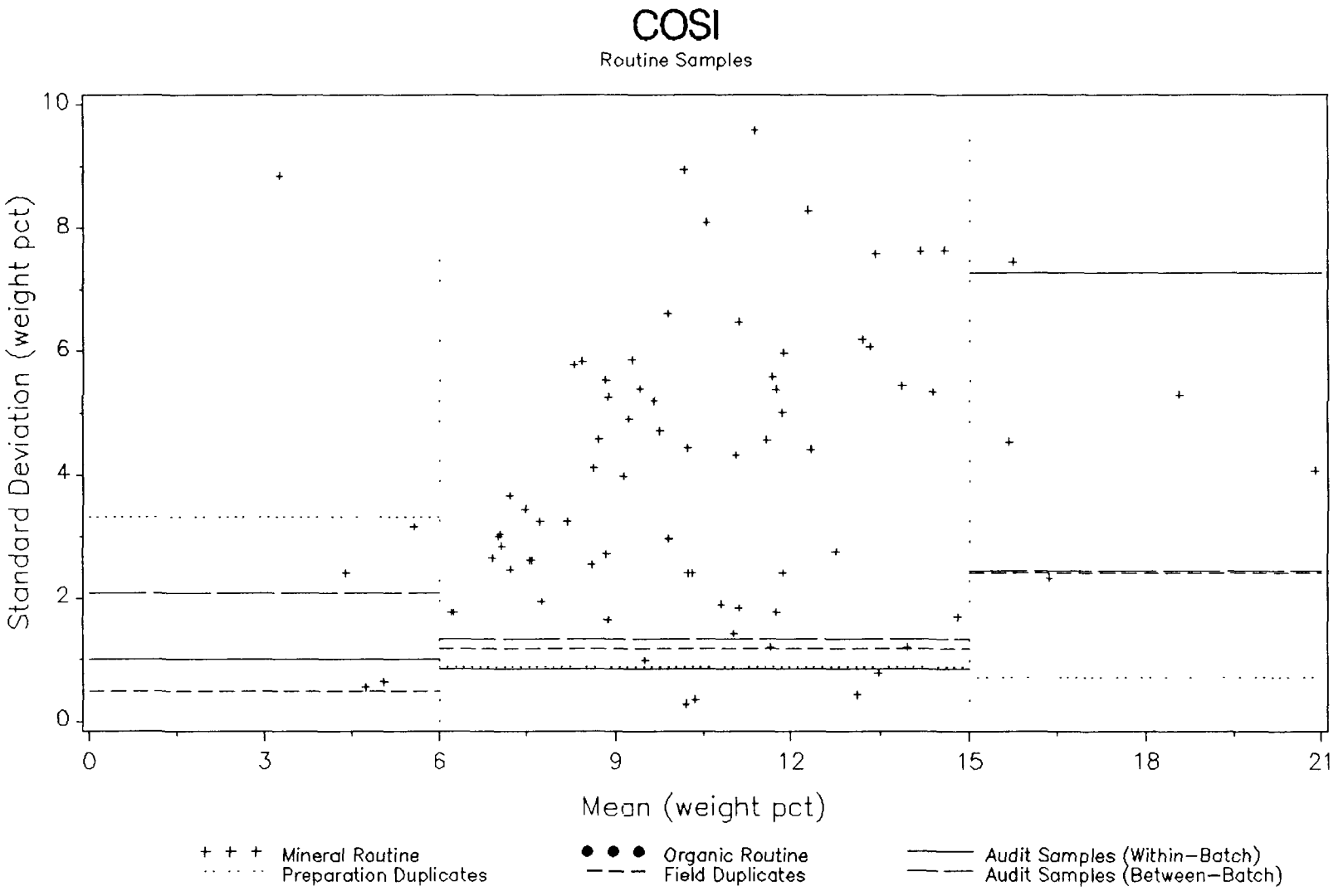

Figure E-8. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for COSI.

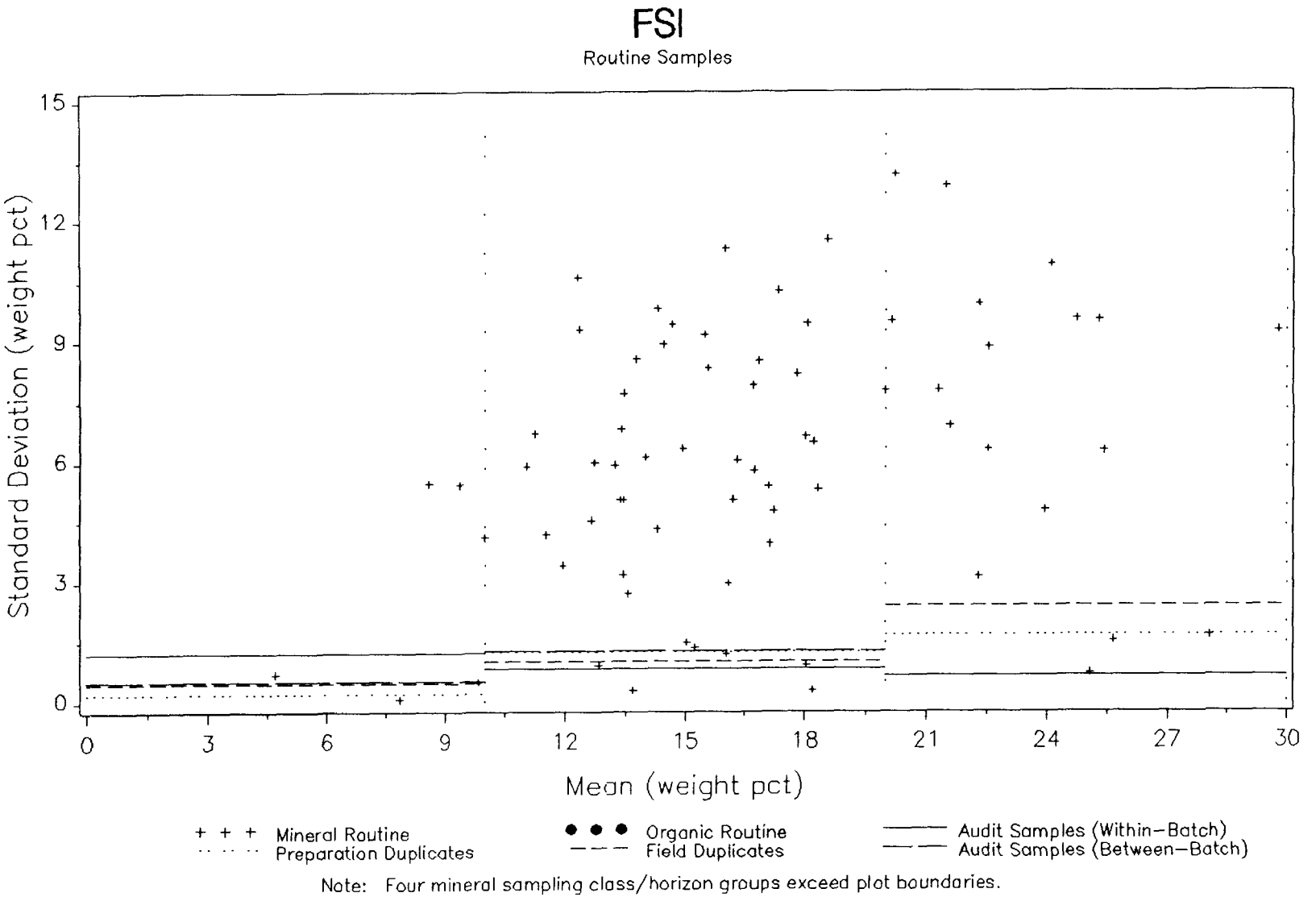

Figure E-9. Range and frequency distribution of sampling class/horizon routine data partitioned into windows and their relation to pooled precision estimates for FSI.

Appendix F

Table of General Statistics for the Analytical Parameters

Appendix F consists of a general summary table for data users. Included are data sorted by laboratory and by audit sample type for mean concentration, standard deviation, and laboratory difference from the interlaboratory mean. Supplemental information relating to this table is contained in the discussion of "interlaboratory differences" in the main body of the report.

Table F-1. Table of General Statistics for the Analytical Parameters

	Audit Sample[a]											
	A			Bs			Bw			C		
Lab	Mean	SD	d	Mean	SD	d	Mean	SD	d	Mean	SD	d
						Moisture %						
1	1.95	0.13	0.06	2.79	0.31	-0.01	2.36	0.12	0.03	0.14	0.01	-0.01
2	1.87	0.28	-0.01	2.81	0.72	0.01	2.34	0.04	0.01	0.15	0.00	0.00
3	1.82	0.20	-0.06	--	--	--	2.28	0.15	-0.05	0.15	0.01	0.00
ALL	1.89	0.22	.	2.80	0.56	.	2.33	0.11	.	0.15	0.01	
						Specific Surface m^2/g						
1	40.3	4.56	3.40	26.0	6.59	-0.63	40.1	1.98	3.9	2.21	0.15	-0.24
2	28.2	9.31	-8.64	27.1	8.23	0.52	25.3	7.28	-10.9	1.58	0.40	-0.87
3	44.8	4.98	7.90	--	--	--	41.4	1.47	5.1	3.11	1.19	0.66
ALL	36.9	9.81	.	26.6	7.38	.	36.3	8.11	.	2.45	1.10	
						Total Sand (2.0-0.05mm) % dry wt						
1	54.1	4.00	-2.40	85.6	1.14	0.18	24.3	0.44	-2.81	93.6	1.34	-1.74
2	59.2	1.18	2.70	85.3	1.36	-0.15	32.5	3.65	5.40	97.3	2.00	1.90
3	55.6	0.61	-0.82	--	--	--	25.8	1.47	-1.21	94.7	1.33	-0.67
ALL	56.5	3.33	.	85.5	1.25	.	27.1	4.10	.	95.4	2.04	
						Very Coarse Sand (2.0-1.0 mm) % dry wt						
1	0.79	0.22	-0.18	10.6	1.03	0.77	2.77	0.36	0.15	3.45	0.78	0.01
2	1.00	0.22	0.03	9.15	2.17	-0.65	2.35	0.47	-0.27	4.47	2.58	1.03
3	1.15	0.45	0.18	--	--	--	2.67	0.64	0.05	2.75	0.88	-0.69
ALL	0.97	0.33	.	9.80	1.86	.	2.62	0.48	.	3.44	1.69	
						Coarse Sand (1.0-0.5mm) % dry wt						
1	3.13	0.34	0.03	20.8	1.44	1.00	4.18	0.28	0.15	13.2	3.04	1.00
2	3.14	0.21	0.04	19.0	2.48	-0.85	3.42	0.43	-0.61	13.3	4.44	1.20
3	3.00	0.44	-0.10	--	--	--	4.42	0.30	0.39	11.0	1.56	-1.13
ALL	3.10	0.33	.	19.8	2.24	.	4.04	0.52	.	12.1	2.95	

(continued)

Table F-1. Continued

Lab	Audit Sample[a] A Mean	A SD	A d	Bs Mean	Bs SD	Bs d	Bw Mean	Bw SD	Bw d	C Mean	C SD	C d
					Medium Sand (0.5-0.25mm) % dry wt							
1	8.09	0.62	-0.18	26.3	0.71	0.10	3.50	0.09	-0.01	29.6	2.90	-1.94
2	8.34	0.29	0.08	26.1	1.22	-0.08	3.35	0.13	-0.16	31.0	2.31	-0.52
3	8.38	0.54	0.11	--	--	--	3.70	0.12	0.19	32.5	1.94	0.99
ALL	8.27	0.50	.	26.2	1.00	.	3.51	0.17	.	31.5	2.29	
					Fine Sand (0.25-0.1mm) % dry wt							
1	21.6	2.09	-1.92	20.8	1.51	-1.48	3.57	0.12	-0.34	34.3	3.89	-2.50
2	24.7	0.51	1.20	23.5	2.05	1.20	4.15	0.13	0.24	36.6	4.33	-0.12
3	24.3	0.67	0.75	--	--	--	4.17	0.15	0.27	37.7	1.94	0.92
ALL	23.5	1.92	.	22.3	2.25	.	3.91	0.33	.	36.7	3.13	
					Very Fine Sand (0.1-0.05mm) % dry wt							
1	20.5	1.78	-0.17	7.15	0.91	-0.24	10.3	0.12	-2.76	13.3	1.48	1.70
2	22.0	1.05	1.40	7.58	1.05	0.20	19.3	2.88	6.30	11.9	3.06	0.32
3	18.8	0.74	-1.77	--	--	--	10.9	0.43	-2.09	10.8	1.20	-0.76
ALL	20.6	1.80	.	7.39	0.99	.	13.0	4.33	.	11.6	2.06	
					Total Silt (0.05-0.002mm) % dry wt							
1	28.0	5.26	2.40	12.9	1.05	0.04	68.5	0.96	3.10	6.00	1.27	1.60
2	22.7	2.43	-2.89	12.8	1.45	-0.03	60.0	3.78	-5.47	2.30	1.97	-2.10
3	26.5	0.67	0.95	--	--	--	66.3	1.25	0.90	5.27	1.29	0.87
ALL	25.6	4.15	.	12.8	1.25	.	65.4	4.22	.	4.40	2.11	
					Coarse Silt (0.05-0.02mm) % dry wt							
1	12.9	6.40	1.30	8.69	0.66	-0.09	34.6	0.81	0.84	5.40	1.13	1.37
2	10.4	4.12	-1.28	8.85	1.24	0.07	31.1	2.95	-2.59	1.97	2.17	-2.09
3	11.8	0.49	0.17	--	--	--	35.1	0.78	1.30	5.02	1.21	0.95
ALL	11.6	4.61	.	8.78	1.00	.	33.7	2.31	.	4.07	2.11	
					Fine Silt (0.02-0.005mm) % dry wt							
1	15.1	1.17	1.20	4.21	0.88	0.13	33.9	0.34	2.20	0.60	0.14	0.25
2	12.3	1.82	-1.61	3.97	0.71	-0.11	28.8	1.46	-2.88	0.35	0.26	0.00
3	14.7	0.36	0.77	--	--	--	31.3	0.83	-0.43	0.27	0.14	-0.08
ALL	13.9	1.84	.	4.08	0.78	.	31.7	2.36	.	0.35	0.21	
					Total Clay (< 0.002mm) % dry wt							
1	17.9	1.42	-0.07	1.46	0.76	-0.23	7.25	0.87	-0.23	0.35	0.07	0.15
2	18.1	2.21	0.15	1.88	0.88	0.19	7.52	0.29	0.05	0.42	0.05	0.23
3	17.8	0.54	-0.12	--	--	--	7.77	0.39	0.30	0.00	0.00	-0.20
ALL	17.9	1.61	.	1.69	0.84	.	7.48	0.63	.	0.20	0.21	
					pH in H_2O							
1	4.50	0.04	-0.02	4.53	0.05	-0.02	5.10	0.04	-0.03	5.21	0.08	-0.27
2	4.51	0.03	-0.01	4.56	0.19	0.01	5.12	0.03	-0.01	5.49	0.03	0.01
3	4.56	0.04	0.04	--	--	--	5.18	0.02	0.05	5.57	0.07	0.08
ALL	4.52	0.04	.	4.55	0.14	.	5.13	0.05	.	5.48	0.14	

(continued)

Table F-1. Continued

	Audit Sample[a]											
		A			Bs			Bw			C	
Lab	Mean	SD	d	Mean	SD	d	Mean	SD	d	Mean	SD	d
						pH in 0.002M $CaCl_2$						
1	4.23	0.03	-0.02	4.19	0.03	0.06	4.72	0.03	-0.00	4.75	0.02	-0.30
2	4.21	0.02	-0.04	4.08	0.03	-0.05	4.67	0.03	-0.05	4.99	0.06	-0.05
3	4.34	0.09	0.09	--	--	--	4.78	0.03	0.06	5.17	0.08	0.13
ALL	4.25	0.07	.	4.13	0.06	.	4.72	0.05	.	5.04	0.17	
						pH in 0.01M $CaCl_2$						
1	4.01	0.04	-0.01	4.00	0.07	0.04	4.61	0.01	-0.01	4.71	0.02	-0.14
2	3.98	0.04	-0.04	3.92	0.03	-0.03	4.57	0.02	-0.04	4.79	0.02	-0.05
3	4.09	0.02	0.07	--	--	--	4.67	0.03	0.06	4.92	0.07	0.08
ALL	4.02	0.06	.	3.96	0.07	.	4.62	0.04	.	4.84	0.10	
						Ca in NH_4Cl meq/100g						
1	0.30	0.04	0.01	0.30	0.04	0.05	0.30	0.05	0.02	0.11	0.05	-0.00
2	0.25	0.06	-0.04	0.22	0.03	-0.04	0.23	0.02	-0.05	0.09	0.02	-0.02
3	0.33	0.06	0.04	--	--	--	0.31	0.05	0.03	0.13	0.04	0.02
ALL	0.29	0.06	.	0.25	0.05	.	0.28	0.05	.	0.11	0.04	
						Mg in NH_4Cl meq/100g						
1	0.23	0.02	0.01	0.06	0.01	0.01	0.05	0.01	-0.00	0.03	0.02	-0.00
2	0.22	0.01	0.00	0.05	0.01	-0.01	0.06	0.01	0.00	0.03	0.01	-0.01
3	0.20	0.01	-0.02	--	--	--	0.05	0.01	-0.00	0.04	0.01	0.01
All	0.21	0.02	.	0.05	0.01	.	0.05	0.01	.	0.03	0.01	
						K in NH_4Cl meq/100g						
1	0.29	0.15	0.02	0.02	0.03	-0.00	0.06	0.01	0.00	0.02	0.00	-0.00
2	0.25	0.02	-0.01	0.02	0.00	0.00	0.06	0.01	0.00	0.02	0.01	-0.00
3	0.24	0.02	-0.02	--	--	--	0.05	0.01	-0.01	0.03	0.01	0.00
ALL	0.26	0.09	.	0.02	0.02	.	0.06	0.01	.	0.03	0.01	
						Na in NH_4Cl meq/100g						
1	0.05	0.03	0.01	0.03	0.03	0.01	0.02	0.03	0.00	0.01	0.01	-0.00
2	0.04	0.01	0.00	0.02	0.01	-0.01	0.02	0.02	0.00	0.00	0.00	-0.01
3	0.03	0.02	-0.01	--	--	--	0.02	0.01	0.00	0.02	0.02	0.01
ALL	0.04	0.02	.	0.02	0.02	.	0.02	0.02	.	0.01	0.02	
						Ca in NH_4OAC meq/100g						
1	0.27	0.03	0.00	0.19	0.03	0.01	0.24	0.04	0.00	0.08	0.03	-0.01
2	0.23	0.05	-0.04	0.17	0.04	-0.01	0.18	0.01	-0.06	0.05	0.03	-0.05
3	0.33	0.05	0.06	--	--	--	0.29	0.05	0.05	0.13	0.05	0.04
ALL	0.27	0.06	.	0.18	0.04	.	0.24	0.06	.	0.09	0.06	
						Mg in NH_4OAC meq/100g						
1	0.25	0.02	0.01	0.05	0.02	0.00	0.07	0.01	0.01	0.04	0.00	0.01
2	0.22	0.02	-0.01	0.05	0.01	0.00	0.04	0.00	-0.01	0.03	0.01	-0.01
3	0.23	0.02	0.00	--	--	--	0.06	0.01	0.00	0.04	0.01	0.00
ALL	0.23	0.02	.	0.05	0.01	.	0.06	0.01	.	0.04	0.01	

(continued)

Table F-1. Continued

	Audit Sample[a]											
	A			Bs			Bw			C		
Lab	Mean	SD	d	Mean	SD	d	Mean	SD	d	Mean	SD	d
						K in NH_4OAC meq/100g						
1	0.27	0.06	0.01	0.03	0.02	0.00	0.06	0.02	0.00	0.05	0.00	0.02
2	0.26	0.01	0.00	0.03	0.00	0.00	0.06	0.01	0.00	0.02	0.00	0.01
3	0.25	0.01	-0.01	--	--	--	0.06	0.01	0.00	0.03	0.01	0.00
ALL	0.26	0.04	.	0.03	0.01	.	0.06	0.01	.	0.03	0.01	
						Na in NH_4OAC meq/100g						
1	0.05	0.03	0.01	0.02	0.03	0.00	0.03	0.01	0.00	0.01	0.00	0.00
2	0.04	0.01	0.00	0.02	0.01	0.00	0.03	0.01	0.00	0.00	0.01	-0.01
3	0.03	0.01	-0.01	--	--	--	0.02	0.01	-0.01	0.01	0.01	0.00
All	0.04	0.02	.	0.02	0.02	.	0.02	0.01	.	0.01	0.01	
						CEC - NH_4Cl meq/100g						
1	8.05	1.08	-0.42	8.24	0.56	0.57	4.82	0.82	-0.97	0.77	0.03	-0.37
2	7.11	0.51	-1.36	7.20	0.92	-0.48	4.01	0.77	-1.77	0.84	0.58	-0.31
3	10.9	1.59	2.43	--	--	--	9.01	0.64	3.22	1.47	0.38	0.33
ALL	8.47	1.90	.	7.67	0.93	.	5.79	2.26	.	1.14	0.52	
						CEC - NH_4OAC meq/100g						
1	18.8	0.76	0.73	23.2	1.21	1.00	13.8	2.51	0.66	1.34	0.18	-0.09
2	15.5	1.19	-2.62	21.3	1.27	0.86	9.64	0.64	3.48	0.94	0.12	-0.49
3	20.9	1.61	2.80	--	--	--	15.6	1.45	-2.50	1.79	0.33	0.36
ALL	18.1	2.53	.	22.2	1.55	.	13.1	2.97	.	1.43	0.46	
						KCl Acidity meq/100g						
1	3.19	0.26	-0.28	4.10	0.40	0.09	1.42	0.07	-0.32	0.02	0.05	-0.29
2	3.50	0.33	0.02	3.94	1.19	-0.07	1.59	0.08	-0.15	0.17	0.07	-0.15
3	3.80	0.76	0.33	--	--	--	2.38	0.59	0.63	0.52	0.36	0.20
ALL	3.47	0.52	.	4.01	0.90	.	1.74	0.51	.	0.32	0.33	
						$BaCl_2$ Acidity meq/100g						
1	18.6	1.45	0.00	34.8	2.14	-0.13	17.4	1.29	0.13	0.44	0.07	-0.55
2	19.4	2.45	0.73	35.0	2.89	0.11	18.3	1.80	1.10	1.69	0.88	0.70
3	17.6	4.99	-1.05	--	--	--	16.0	0.79	-1.24	0.71	0.62	-0.28
ALL	18.6	3.13	.	34.9	2.52	.	17.2	1.53	.	0.99	0.81	
						KCl Extractable Al meq/100g						
1	2.72	0.21	-0.25	3.72	0.62	-0.12	1.29	0.09	-0.13	0.06	0.00	-0.09
2	3.12	0.29	0.15	3.93	0.70	0.10	1.50	0.23	0.08	0.14	0.03	-0.01
3	3.07	0.18	0.10	--	--	--	1.54	0.10	0.12	0.18	0.05	0.04
ALL	2.97	0.30	.	3.84	0.66	.	1.42	0.18	.	0.15	0.06	
						Ca in 0.002M $CaCl_2$ meq/100g						
1	0.35	0.05	-0.04	0.49	0.05	-0.03	0.61	0.04	0.01	0.71	0.04	0.11
2	0.46	0.06	0.07	0.55	0.04	0.03	0.64	0.01	0.04	0.59	0.39	-0.02
3	0.35	0.12	-0.04	--	--	--	0.55	0.12	-0.05	0.59	0.03	-0.02
ALL	0.40	0.09	.	0.52	0.06	.	0.60	0.07	.	0.61	0.21	

(continued)

Table F-1. Continued

Lab	A Mean	A SD	A d	Bs Mean	Bs SD	Bs d	Bw Mean	Bw SD	Bw d	C Mean	C SD	C d
						Audit Sample[a]						
						Mg in 0.002M $CaCl_2$ meq/100g						
1	0.14	0.03	0.02	0.04	0.00	0.00	0.04	0.00	0.00	0.02	0.00	0.00
2	0.12	0.01	0.00	0.03	0.00	0.00	0.04	0.00	0.00	0.01	0.01	0.00
3	0.11	0.02	-0.02	--	--	--	0.03	0.01	-0.01	0.02	0.00	0.00
All	0.13	0.02	.	0.04	0.01	.	0.04	0.01	.	0.01	0.01	
						K in 0.002M $CaCl_2$ meq/100g						
1	0.07	0.01	0.01	0.02	0.01	0.00	0.01	0.00	0.00	0.01	0.00	0.00
2	0.06	0.01	0.00	0.01	0.00	0.00	0.01	0.00	0.00	0.01	0.01	0.00
3	0.06	0.01	-0.01	--	--	--	0.01	0.00	0.00	0.01	0.00	0.00
All	0.06	0.01	.	0.01	0.00	.	0.01	0.00	.	0.01	0.00	
						Na in 0.002M $CaCl_2$ meq/100g						
1	0.04	0.01	0.00	0.02	0.01	0.00	0.02	0.01	0.00	0.00	0.00	0.00
2	0.03	0.00	0.00	0.02	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00
3	0.03	0.00	0.00	--	--	--	0.02	0.00	0.00	0.01	0.00	0.00
All	0.03	0.01	.	0.02	0.01	.	0.02	0.00	.	0.00	0.00	
						Fe in 0.002M $CaCl_2$ meq/100g						
1	0.02	0.01	0.00	0.05	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00
2	0.02	0.01	0.00	0.04	0.01	-0.01	0.00	0.00	0.00	0.00	0.00	0.00
3	0.02	0.00	0.00	--	--	--	0.00	0.00	0.00	0.00	0.00	0.00
ALL	0.02	0.01	.	0.04	0.01	.	0.00	0.00	.	0.00	0.00	
						Al in 0.002M $CaCl_2$ meq/100g						
1	0.10	0.03	0.02	0.19	0.02	0.02	0.00	0.00	0.00	0.00	0.00	0.00
2	0.08	0.02	0.01	0.15	0.04	-0.02	0.00	0.00	0.00	0.00	0.00	0.00
3	0.07	0.02	-0.01	--	--	--	0.01	0.00	-.00	0.01	0.00	-.00
ALL	0.08	0.03	.	0.17	0.03	.	0.00	0.00	.	0.00	0.00	
						Fe in Pyrophosphate % dry wt						
1	0.60	0.05	-0.04	0.58	0.04	0.00	0.80	0.06	-0.05	0.05	0.01	0.01
2	0.65	0.06	0.01	0.58	0.10	0.00	0.84	0.08	-0.01	0.03	0.00	-0.01
3	0.66	0.08	0.03	--	--	--	0.94	0.12	0.09	0.04	0.01	0.00
ALL	0.63	0.07	.	0.58	0.07	.	0.85	0.10	.	0.04	0.01	
						Al in Pyrophosphate % dry wt						
1	0.60	0.09	0.00	0.79	0.05	0.02	0.60	0.02	0.02	0.06	0.00	0.01
2	0.63	0.12	0.04	0.75	0.13	-0.02	0.54	0.07	-0.04	0.04	0.01	-0.02
3	0.54	0.06	-0.06	--	--	--	0.59	0.06	0.01	0.06	0.01	0.01
ALL	0.59	0.10	.	0.77	0.10	.	0.58	0.05	.	0.06	0.01	
						Fe in Acid Oxalate % dry wt						
1	0.39	0.05	-0.02	0.84	0.23	-0.08	0.99	0.16	-0.04	0.11	0.07	0.03
2	0.45	0.04	0.04	0.99	0.31	0.07	1.20	0.09	0.17	0.04	0.01	-0.04
3	0.39	0.02	-0.02	--	--	--	0.91	0.06	-0.11	0.10	0.07	0.02
ALL	0.41	0.05	.	0.93	0.28	.	1.03	0.16	.	0.08	0.06	

(continued)

Table F-1. Continued

Lab	Audit Sample[a] A Mean	A SD	A d	Bs Mean	Bs SD	Bs d	Bw Mean	Bw SD	Bw d	C Mean	C SD	C d
					Al in Acid Oxalate % dry wt							
1	0.40	0.06	-0.03	0.95	0.12	-0.01	0.87	0.06	-0.01	0.07	0.01	0.01
2	0.48	0.07	0.06	0.98	0.13	0.01	1.01	0.04	0.13	0.05	0.00	-0.01
3	0.38	0.04	-0.05	--	--	--	0.75	0.08	-0.12	0.07	0.01	0.01
ALL	0.42	0.07	.	0.97	0.12	.	0.88	0.11	.	0.06	0.01	
					Fe in Citrate Dithionite % dry wt							
1	1.68	0.21	-0.17	0.93	0.06	-0.06	1.77	0.55	-0.11	0.17	0.01	-0.01
2	1.73	0.15	-0.12	1.05	0.14	0.05	1.83	0.08	-0.06	0.12	0.03	-0.06
3	2.23	0.14	0.38	--	--	--	2.10	0.13	0.22	0.23	0.04	0.05
ALL	1.85	0.29	.	0.99	0.12	.	1.88	0.38	.	0.19	0.06	
					Al in Citrate Dithionite % dry wt							
1	0.45	0.03	-0.05	0.73	0.05	-0.05	0.60	0.20	-0.07	0.05	0.00	0.00
2	0.51	0.05	0.01	0.83	0.07	0.05	0.67	0.03	0.00	0.04	0.01	-0.02
3	0.56	0.04	0.06	--	--	--	0.78	0.09	0.11	0.07	0.01	0.01
ALL	0.50	0.06	.	0.78	0.08	.	0.67	0.15	.	0.05	0.01	
					Sulfate in H_2O mg S/kg							
1	31.6	1.20	1.90	8.38	1.53	0.20	23.8	1.40	0.96	2.74	0.34	-0.46
2	29.9	1.45	0.21	8.01	2.16	-0.17	22.7	0.68	-0.21	2.75	1.68	-0.45
3	26.9	2.25	-2.76	--	--	--	21.7	3.98	-1.23	3.65	0.84	0.45
ALL	29.7	2.44	.	8.18	1.87	.	22.9	2.33	.	3.20	1.15	
					Sulfate in PO_4 mg S/kg							
1	73.4	5.30	-2.77	23.1	3.09	-7.95	103	2.42	-5.50	4.59	0.12	-0.92
2	79.1	6.72	2.90	37.7	25.2	6.60	111	7.50	1.95	6.96	2.32	1.44
3	75.6	3.74	-0.63	--	--	--	115	3.62	6.30	4.86	2.68	-0.65
ALL	76.2	5.99	.	31.1	19.8	.	109	6.75	.	5.52	2.42	
					Sulfate 0 mg S/L							
1	4.12	0.27	-0.16	1.24	0.14	0.08	2.29	0.21	0.02	0.57	0.00	0.04
2	4.47	0.56	0.19	1.10	0.19	-0.06	2.14	0.21	-0.13	0.56	0.16	0.03
3	4.20	0.34	-0.07	--	--	--	2.37	0.23	0.10	0.50	0.04	-0.03
ALL	4.27	0.44	.	1.17	0.18	.	2.27	0.22	.	0.53	0.09	
					Sulfate 2 mg S/L							
1	5.36	0.29	-0.33	2.90	0.10	0.09	3.20	0.06	-0.02	2.56	0.07	0.07
2	6.00	0.58	0.31	2.74	0.25	-0.07	3.12	0.12	-0.10	2.46	0.27	-0.04
3	5.66	0.35	-0.02	--	--	--	3.34	0.27	0.13	2.49	0.07	0.00
ALL	5.69	0.51	.	2.81	0.21	.	3.22	0.17	.	2.49	0.16	
					Sulfate 4 mg S/L							
1	6.93	0.30	-0.13	4.35	0.27	-0.03	4.20	0.08	-0.10	4.41	0.33	-0.04
2	7.24	0.47	0.18	4.40	0.30	0.02	4.00	0.16	-0.30	4.45	0.04	0.01
3	6.96	0.26	-0.09	--	--	--	4.75	0.23	0.45	4.45	0.17	0.01
ALL	7.06	0.39	.	4.38	0.28	.	4.30	0.34	.	4.45	0.15	

(continued)

Table F-1. Continued

Lab	Audit Sample[a] A Mean	A SD	A d	Bs Mean	Bs SD	Bs d	Bw Mean	Bw SD	Bw d	C Mean	C SD	C d
						Sulfate 8 mg S/L						
1	9.69	0.41	-0.44	7.49	0.52	-0.26	6.44	0.27	-0.09	8.60	0.33	0.01
2	10.6	0.71	0.48	7.97	0.31	0.21	6.70	0.43	0.16	9.02	1.01	0.43
3	10.0	0.28	-0.13	--	--	--	6.51	0.32	-0.02	8.30	0.07	-0.29
ALL	10.1	0.65	.	7.75	0.47	.	6.53	0.33	.	8.59	0.64	
						Sulfate 16 mg S/L						
1	15.6	0.84	-0.72	13.9	1.10	-0.63	11.3	0.48	-0.57	16.9	0.07	-0.13
2	17.0	0.98	0.72	15.1	0.44	0.53	12.6	0.51	0.72	17.8	2.14	0.80
3	16.2	1.90	-0.10	--	--	--	12.0	0.33	0.14	16.5	0.15	-0.49
ALL	16.3	1.38	.	14.6	0.99	.	11.9	0.70	.	17.0	1.28	
						Sulfate 32 mg S/L						
1	28.5	0.95	-1.21	27.9	1.92	-0.75	22.5	0.66	-1.19	32.4	0.13	-0.58
2	30.6	1.73	0.88	29.3	1.95	0.63	25.0	1.10	1.30	34.5	3.33	1.50
3	30.0	0.49	0.30	--	--	--	24.2	0.80	0.50	32.2	0.29	-0.81
ALL	29.7	1.52	.	28.7	2.01	.	23.7	1.35	.	33.0	2.07	
						Total Carbon % dry wt						
1	4.62	0.20	-0.04	4.19	0.80	0.38	1.55	0.04	0.05	0.13	0.01	-0.01
2	4.64	0.17	-0.02	3.49	0.31	-0.32	1.41	0.02	-0.10	0.14	0.02	0.00
3	4.75	0.52	0.09	--	--	--	1.54	0.14	0.03	0.15	0.04	0.00
ALL	4.66	0.31	.	3.81	0.67	.	1.51	0.10	.	0.14	0.03	
						Total Nitrogen % dry wt						
1	0.15	0.02	0.00	0.14	0.04	0.01	0.11	0.02	0.00	0.02	0.00	0.01
2	0.15	0.01	0.00	0.11	0.01	-0.01	0.10	0.01	-0.01	0.00	0.00	0.00
3	0.17	0.02	0.01	--	--	--	0.12	0.01	0.01	0.01	0.00	0.00
ALL	0.16	0.02	.	0.12	0.03	.	0.11	0.02	.	0.01	0.01	
						Total Sulfur % dry wt						
1	.023	.001	-0.002	.015	.002	-0.002	.020	.001	0.001	.000	.004	-0.004
2	.027	.002	0.002	.018	.001	0.001	.020	.000	0.001	.003	.001	-0.001
3	.023	.002	-0.002	--	--	--	.018	.002	-0.001	.005	.002	0.001
ALL		.025	.002	.	.017	.001	.	.019	.001	.	.004	.002

[a] Mean, standard deviation (SD), and laboratory difference (d) for audit samples; differences were not estimated for the Oa horizon audit samples.

Appendix G

Histograms of Range and Frequency Distributions

Appendix G consists of figures displaying the histograms of four data sets which show the range and frequency distribution of the routine samples (RS), the field duplicates (FD), the preparation duplicates (PD), and the natural audit samples (AS). Additional information relating to these plots can be found under the heading "Representativeness" in Sections 2 and 3 of the report. Histograms are presented for each of the 51 analytical parameters in the order described in Table 1-1 of Section 1 of the report.

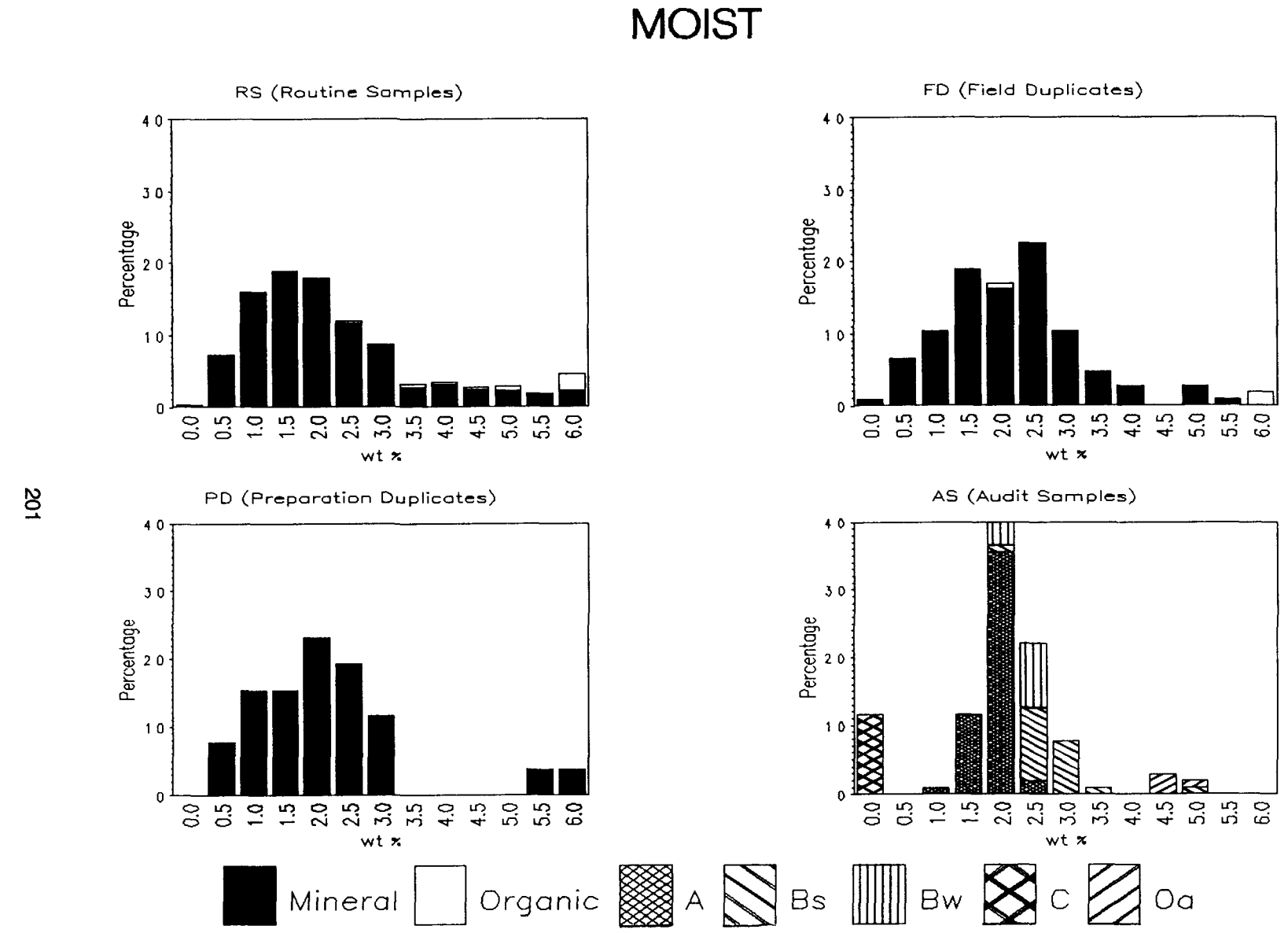

Figure G-1. Histogram of range and frequency distribution for air-dry moisture.

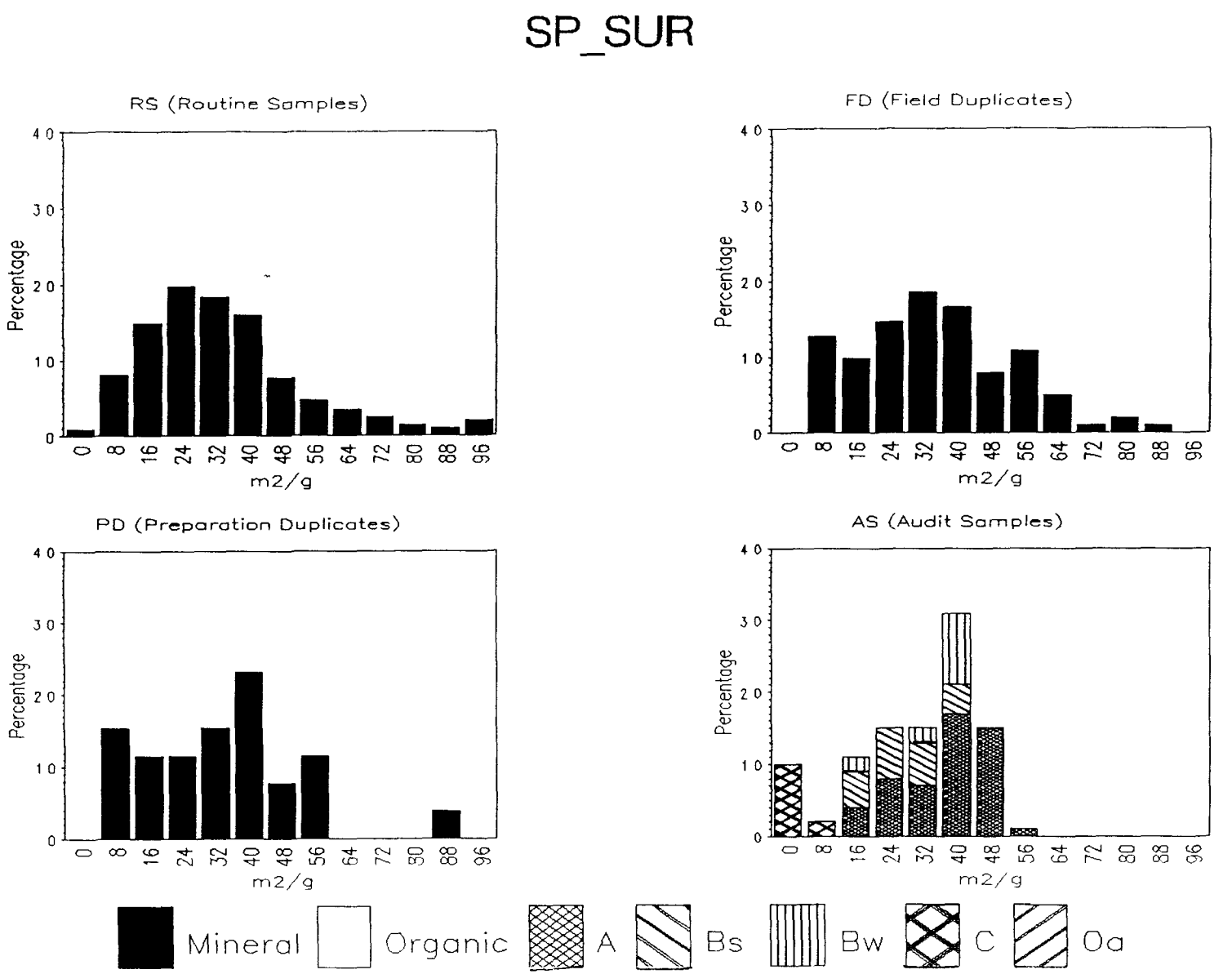

Figure G-2. Histogram of range and frequency distribution for specific surface.

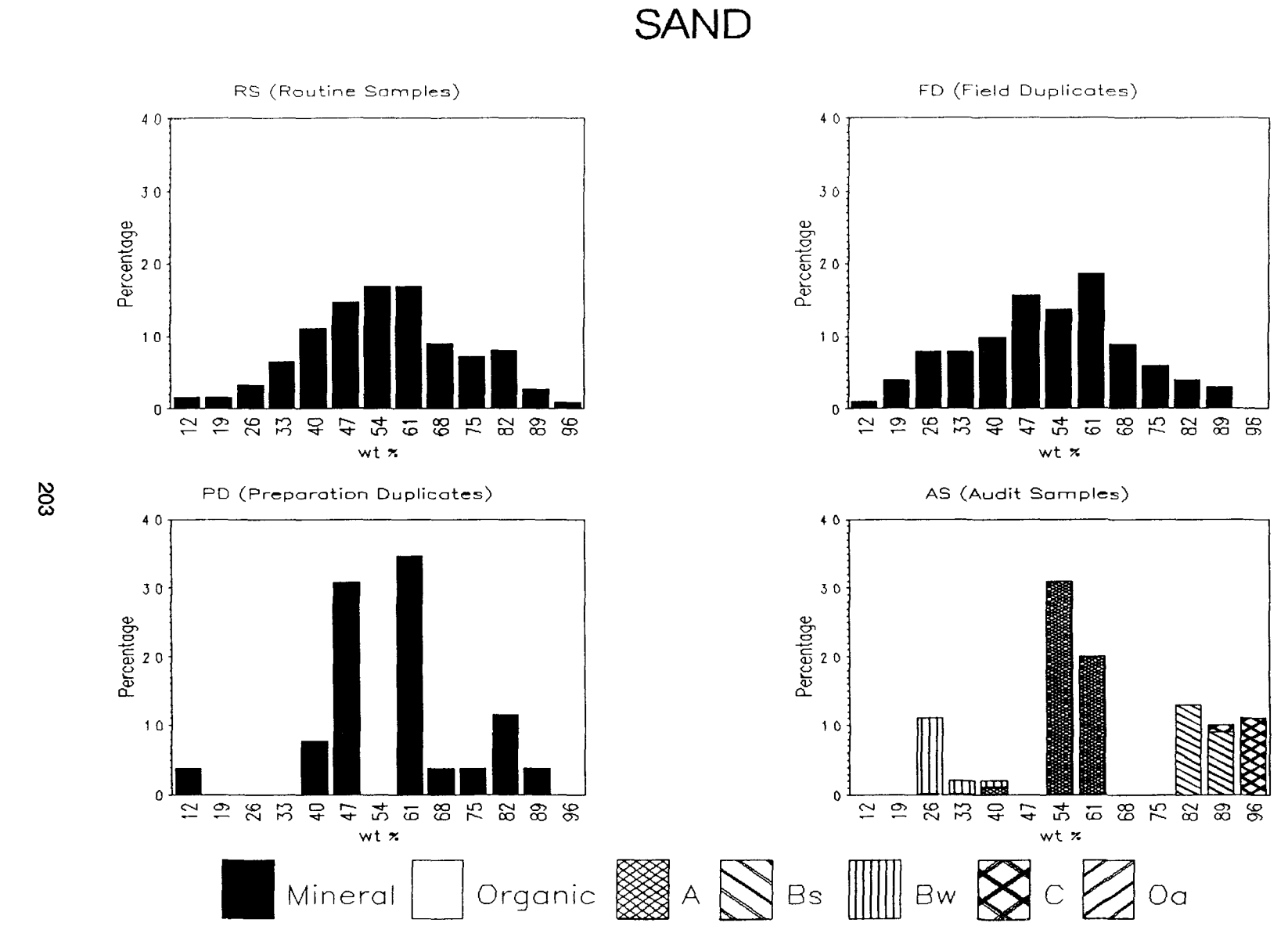

Figure G-3. Histogram of range and frequency distribution for total sand.

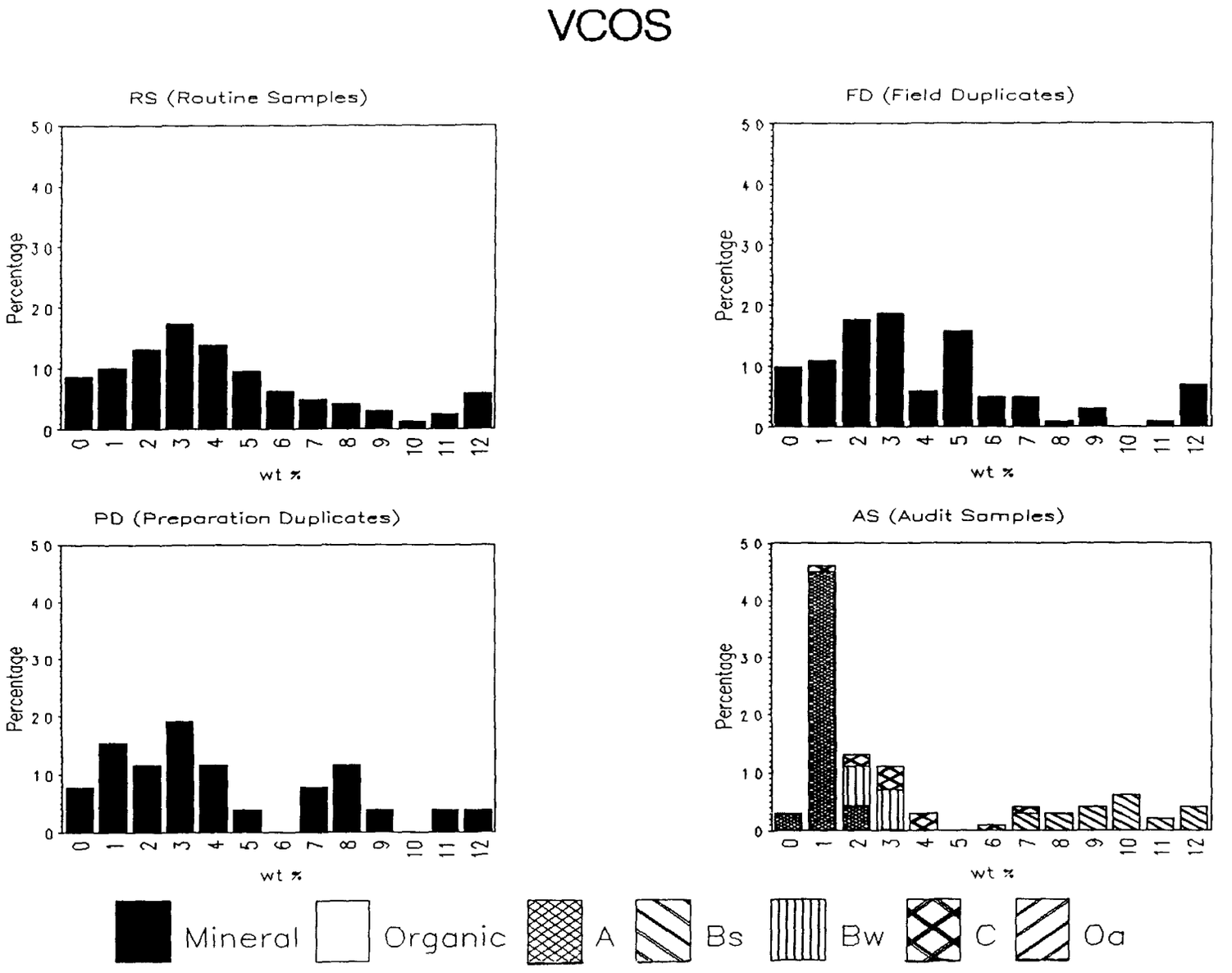

Figure G-4. Histogram of range and frequency distribution for very coarse sand.

COS

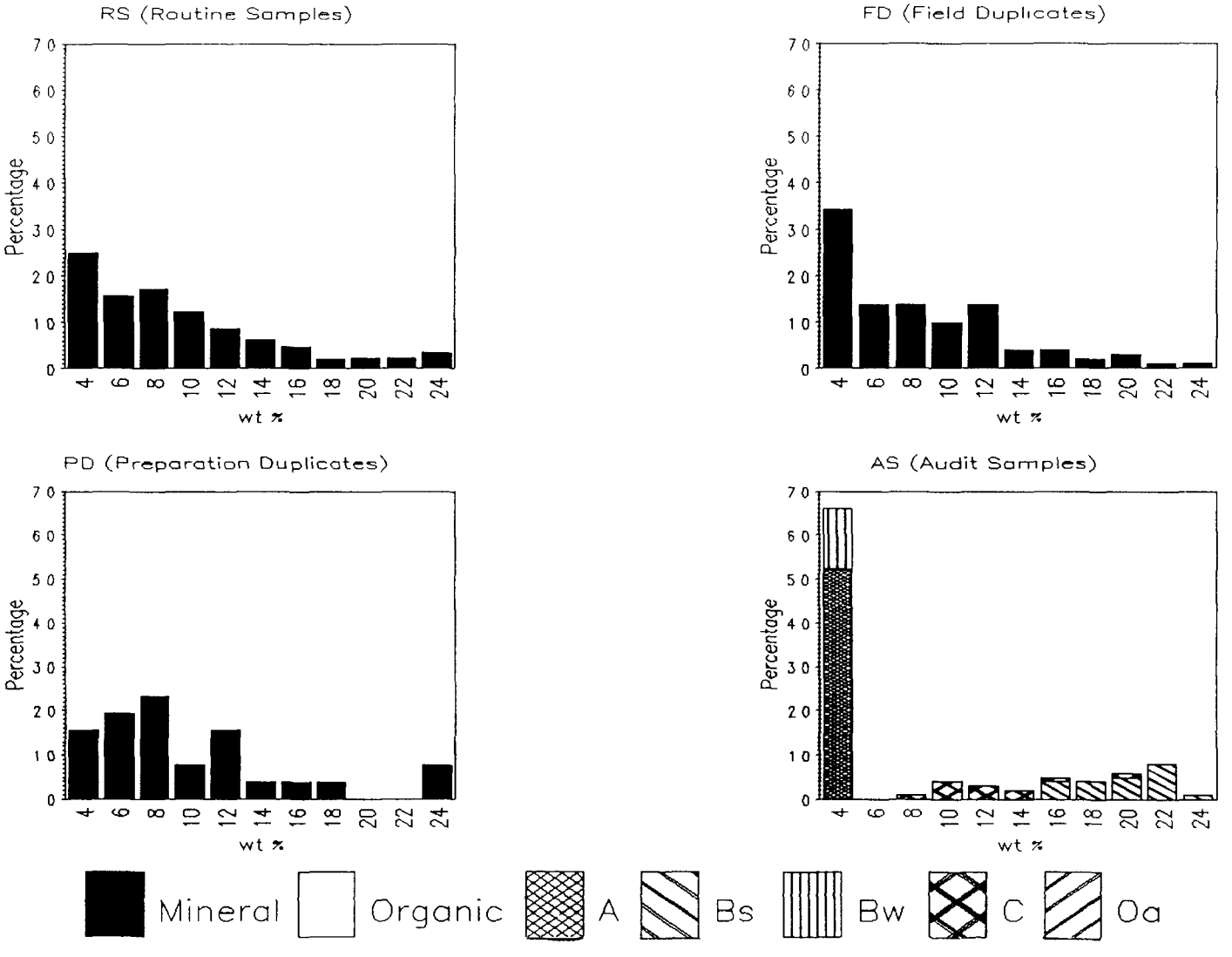

Figure G-5. Histogram of range and frequency distribution for coarse sand.

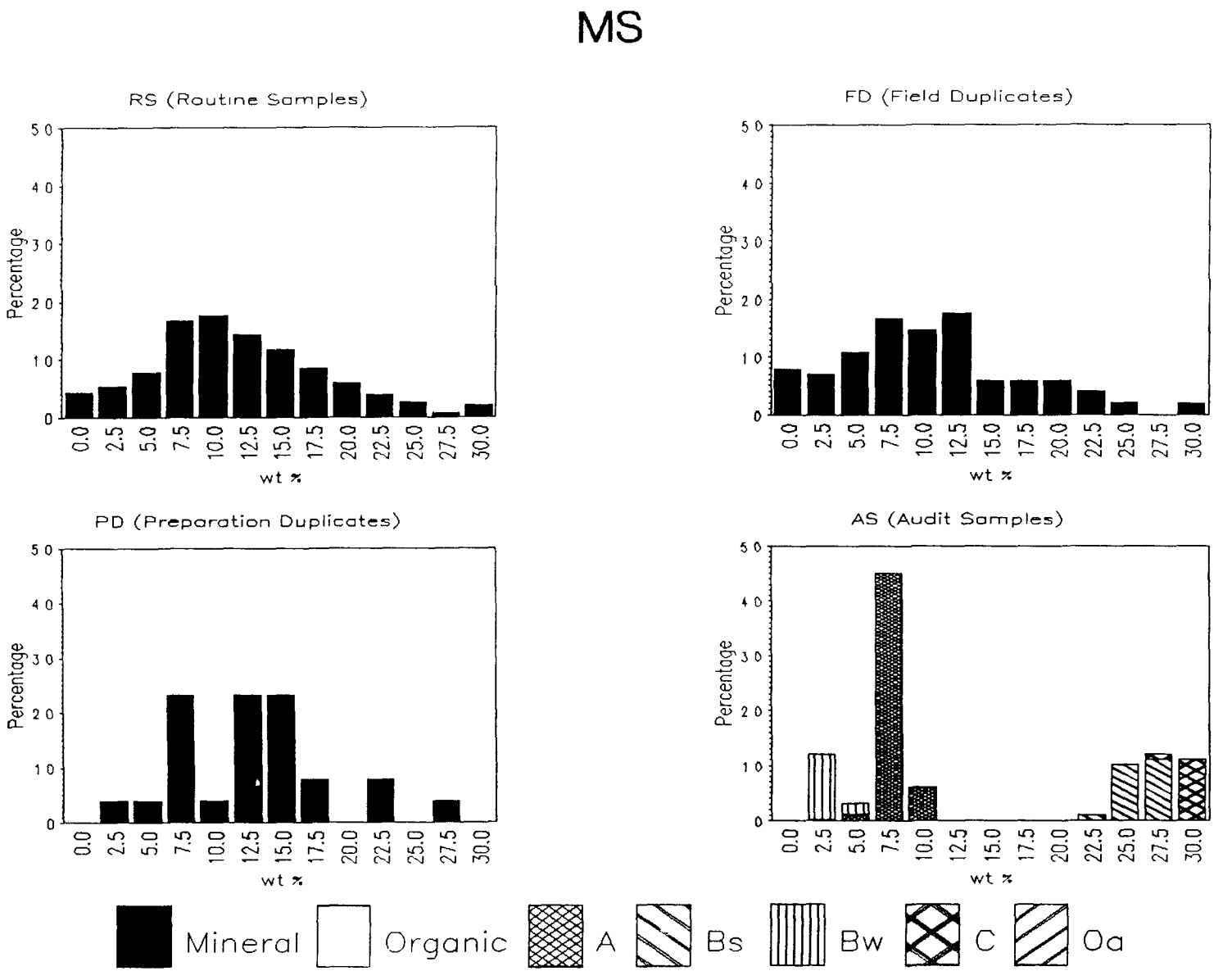

Figure G-6. Histogram of range and frequency distribution for medium sand.

FS

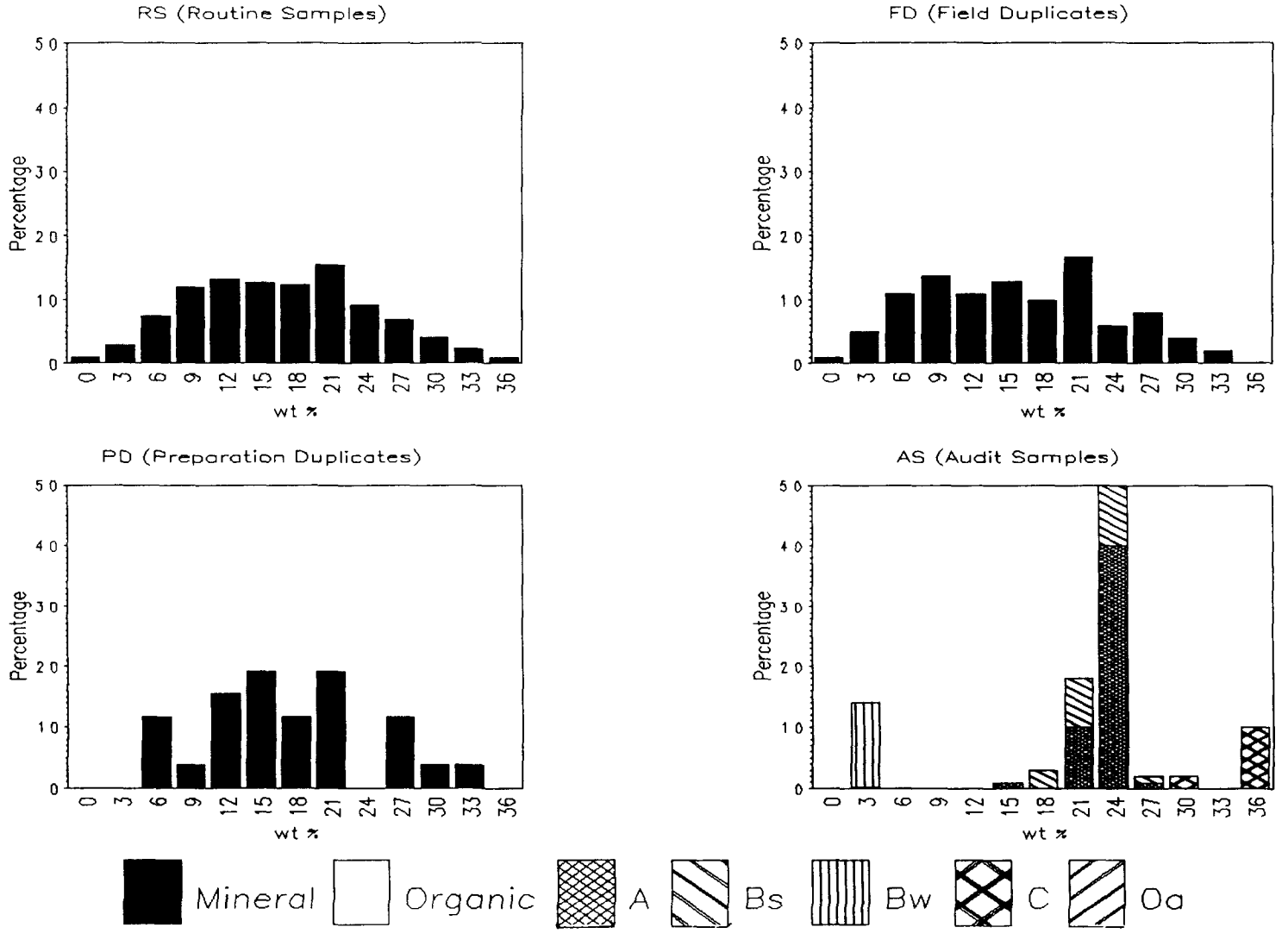

Figure G-7. Histogram of range and frequency distribution for fine sand.

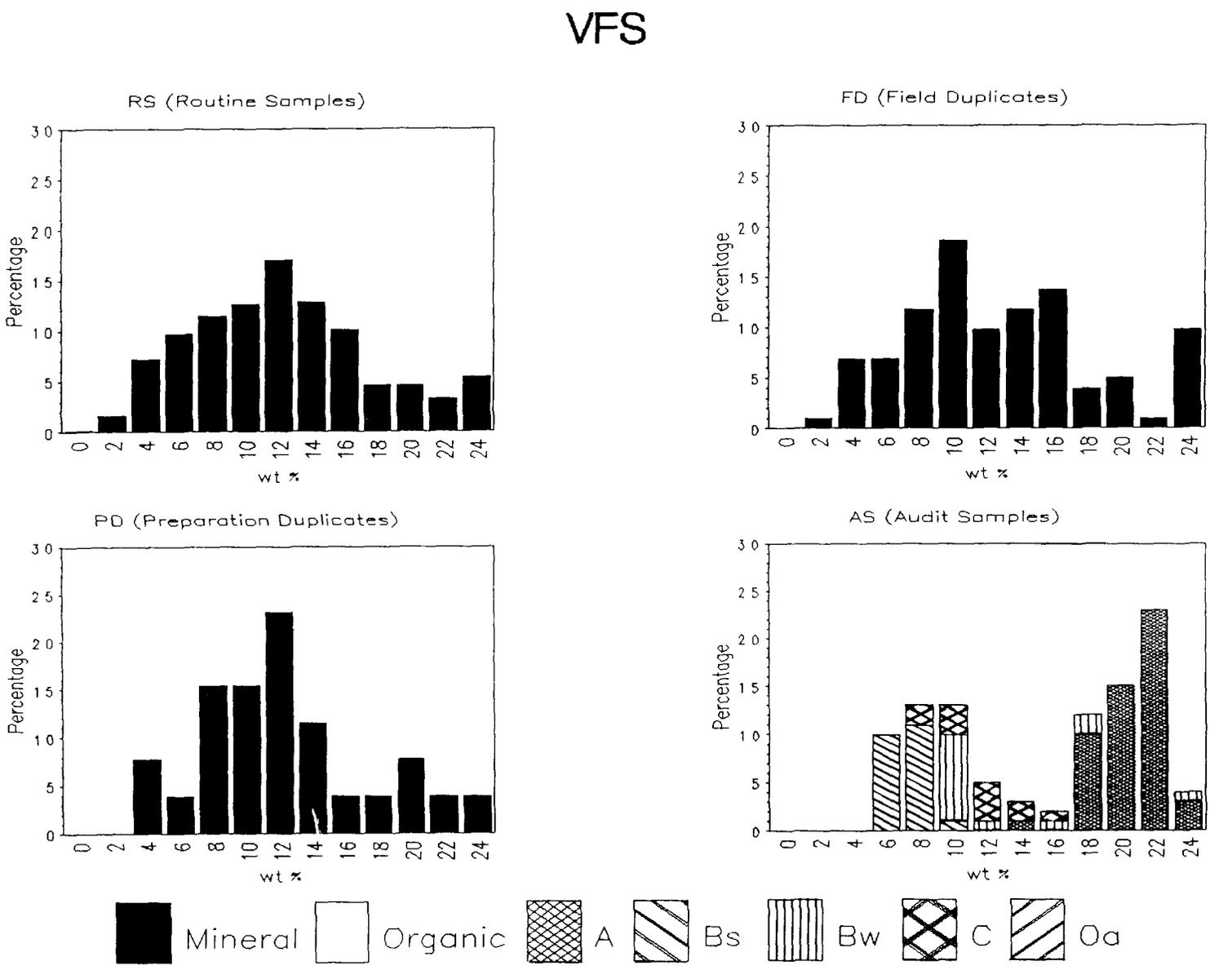

Figure G-8. Histogram of range and frequency distribution for very fine sand.

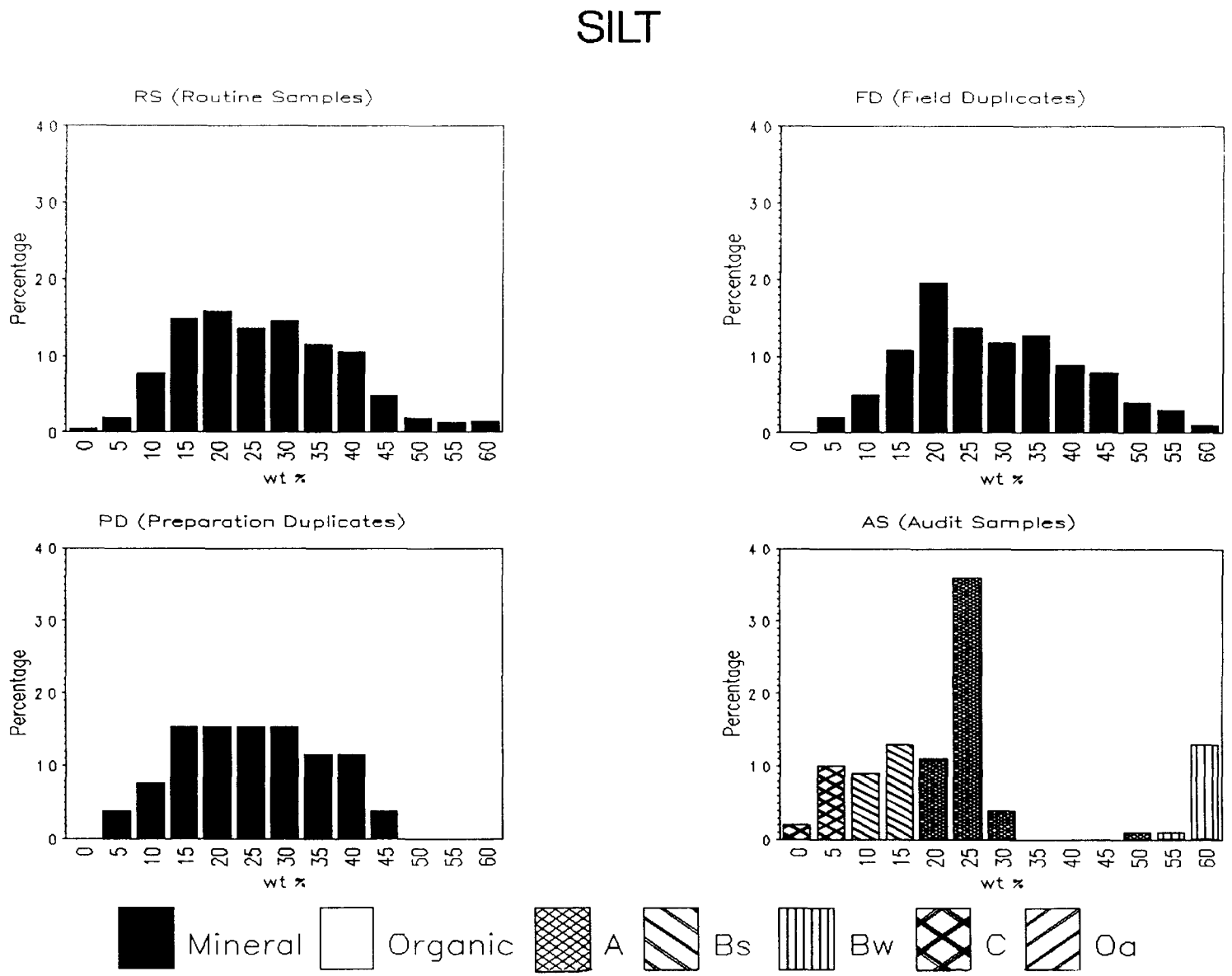

Figure G-9. Histogram of range and frequency distribution for total silt.

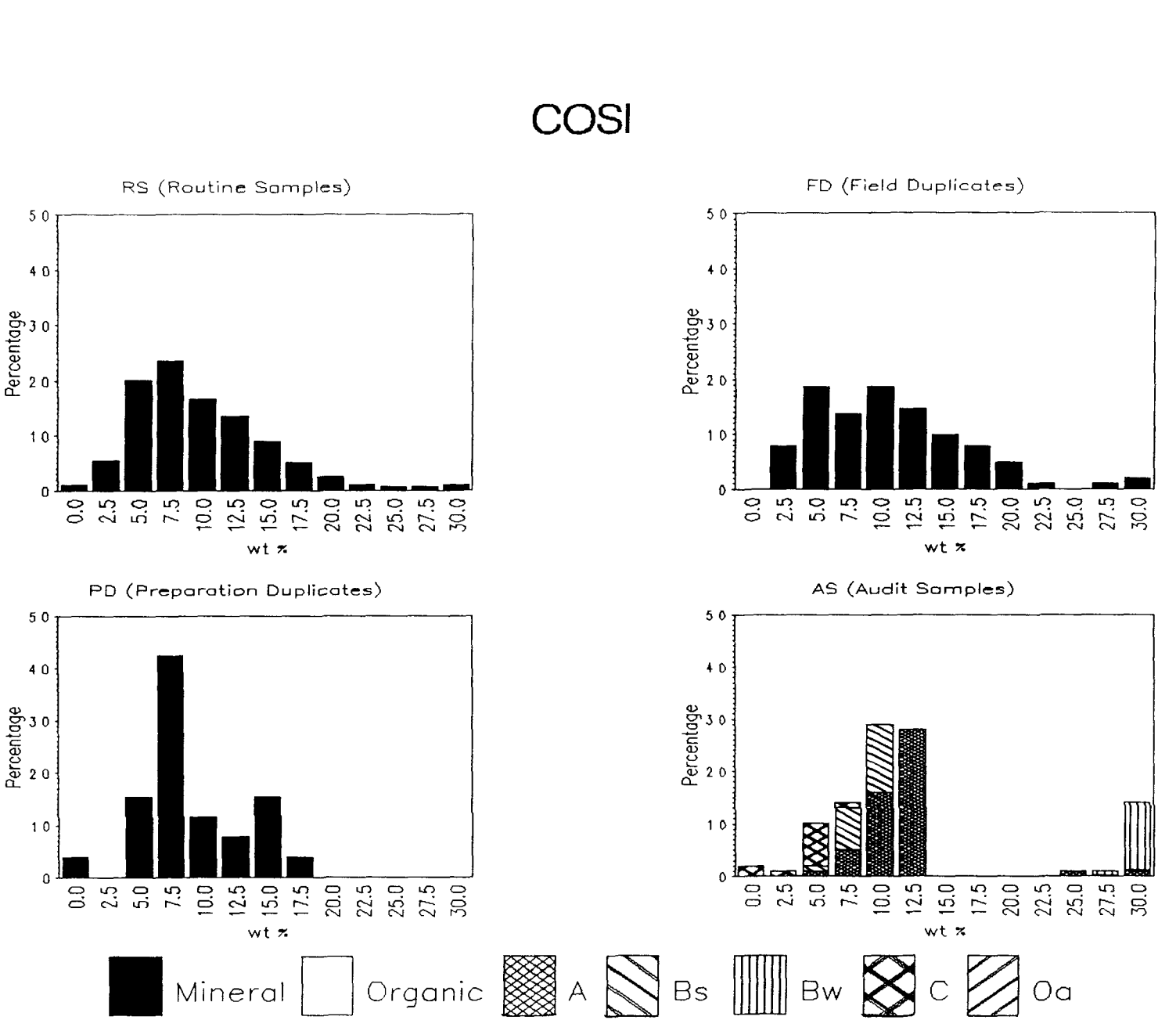

Figure G-10. Histogram of range and frequency distribution for coarse silt.

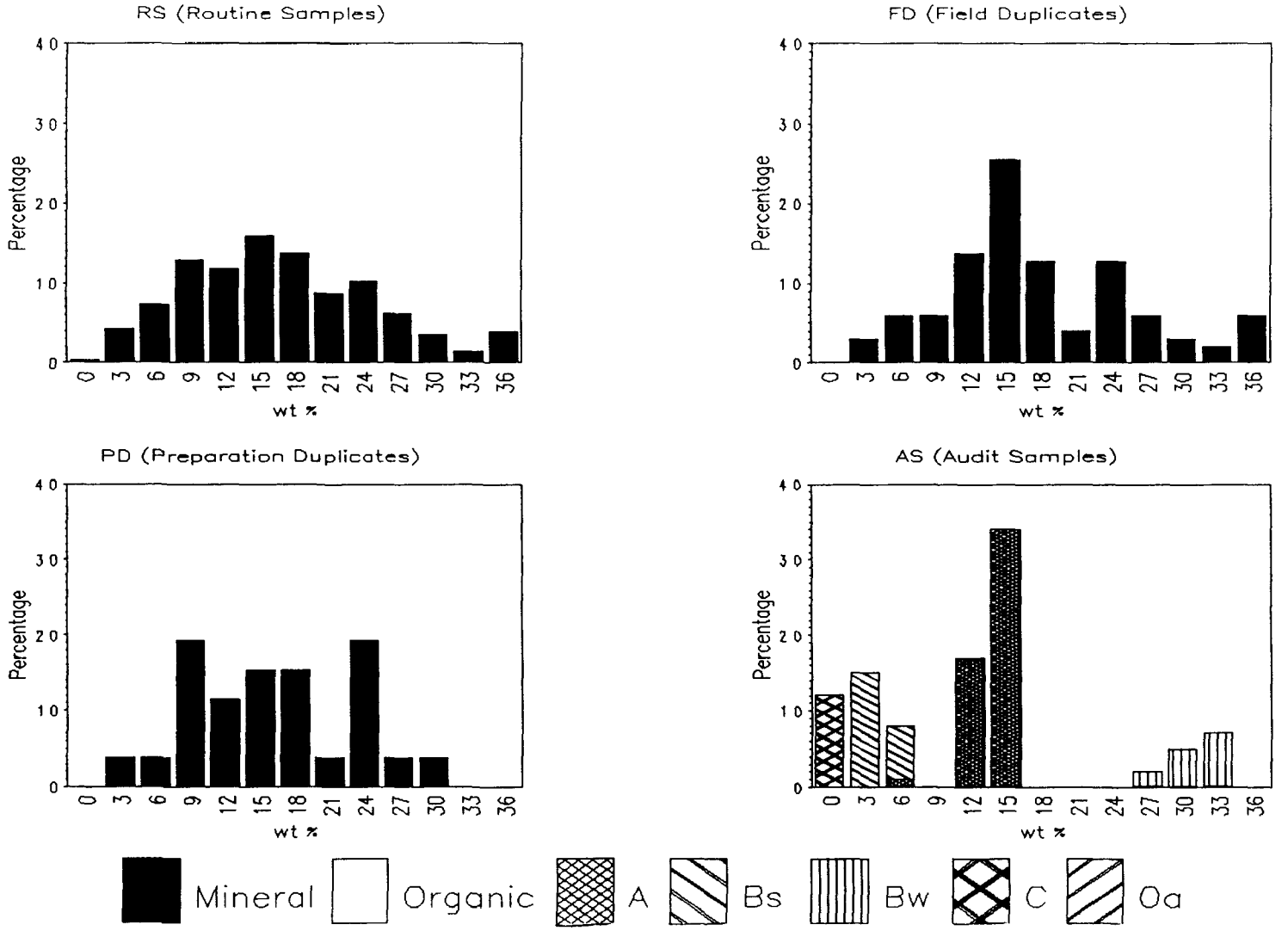

Figure G-11. Histogram of range and frequency distribution for fine silt.

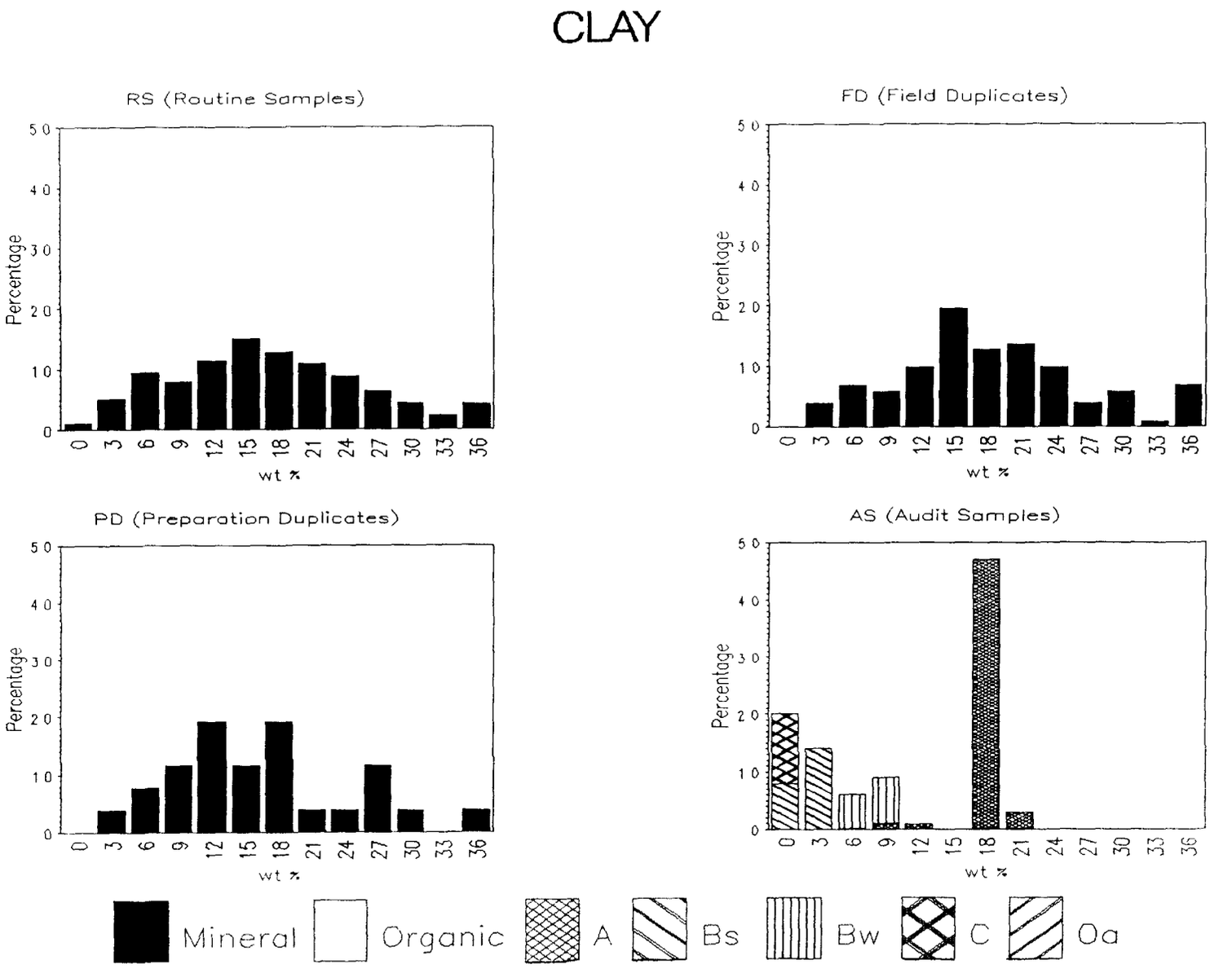

Figure G-12. Histogram of range and frequency distribution for total clay.

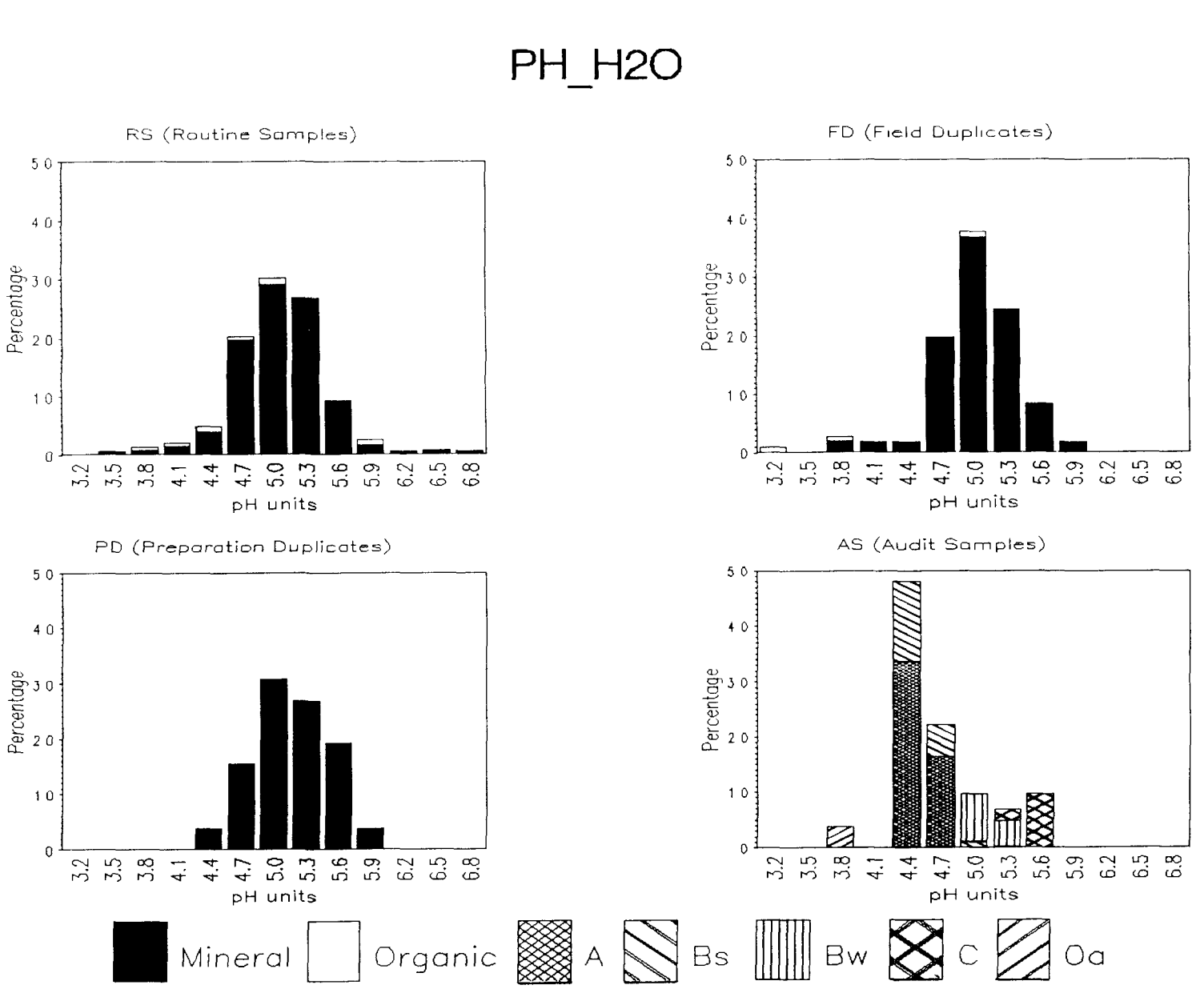

Figure G-13. Histogram of range and frequency distribution for pH in water.

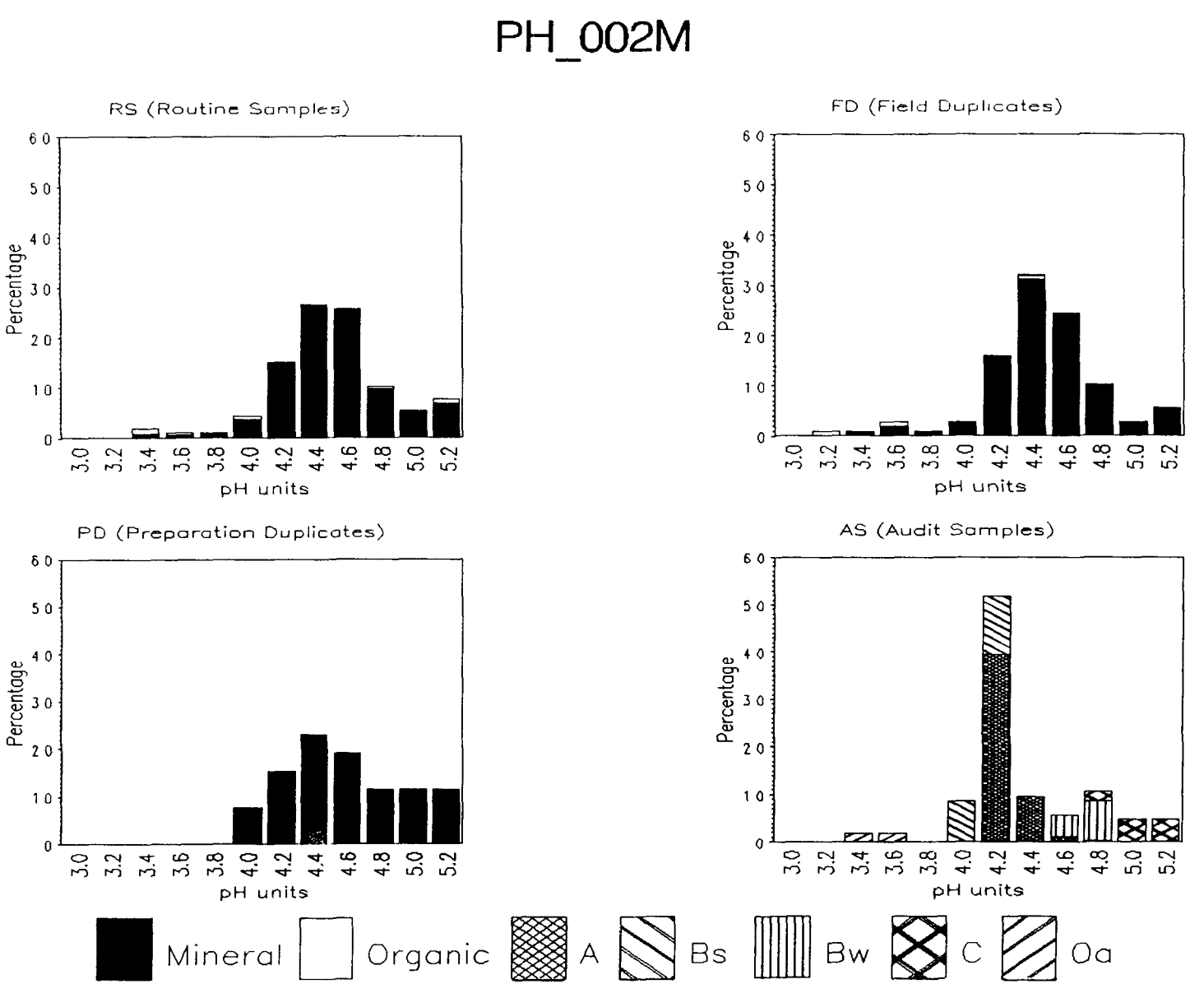

Figure G-14. Histogram of range and frequency distribution for pH in 0.002M calcium chloride.

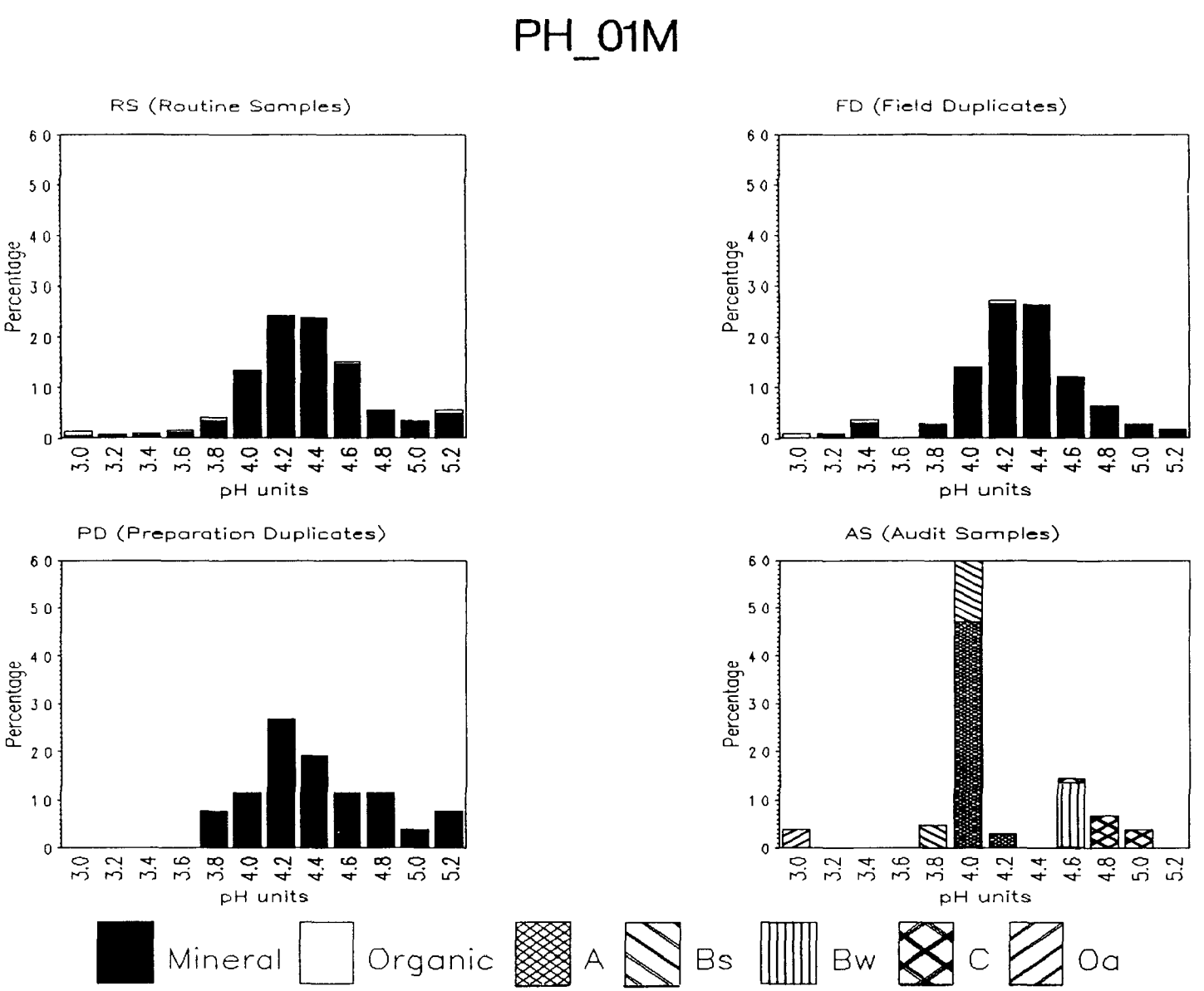

Figure G-15. Histogram of range and frequency distribution for pH in 0.01M calcium chloride.

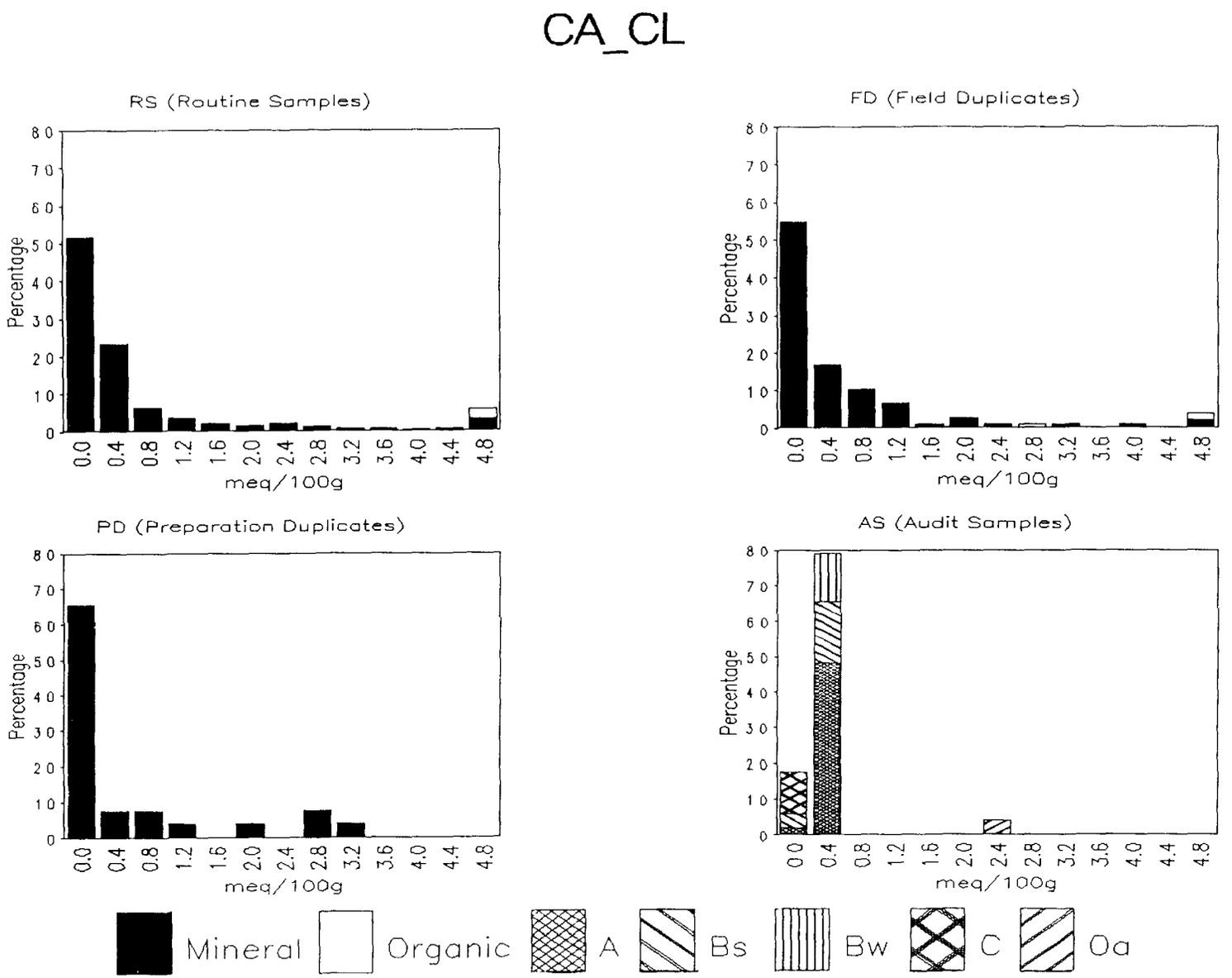

Figure G-16. Histogram of range and frequency distribution for calcium in ammonium chloride.

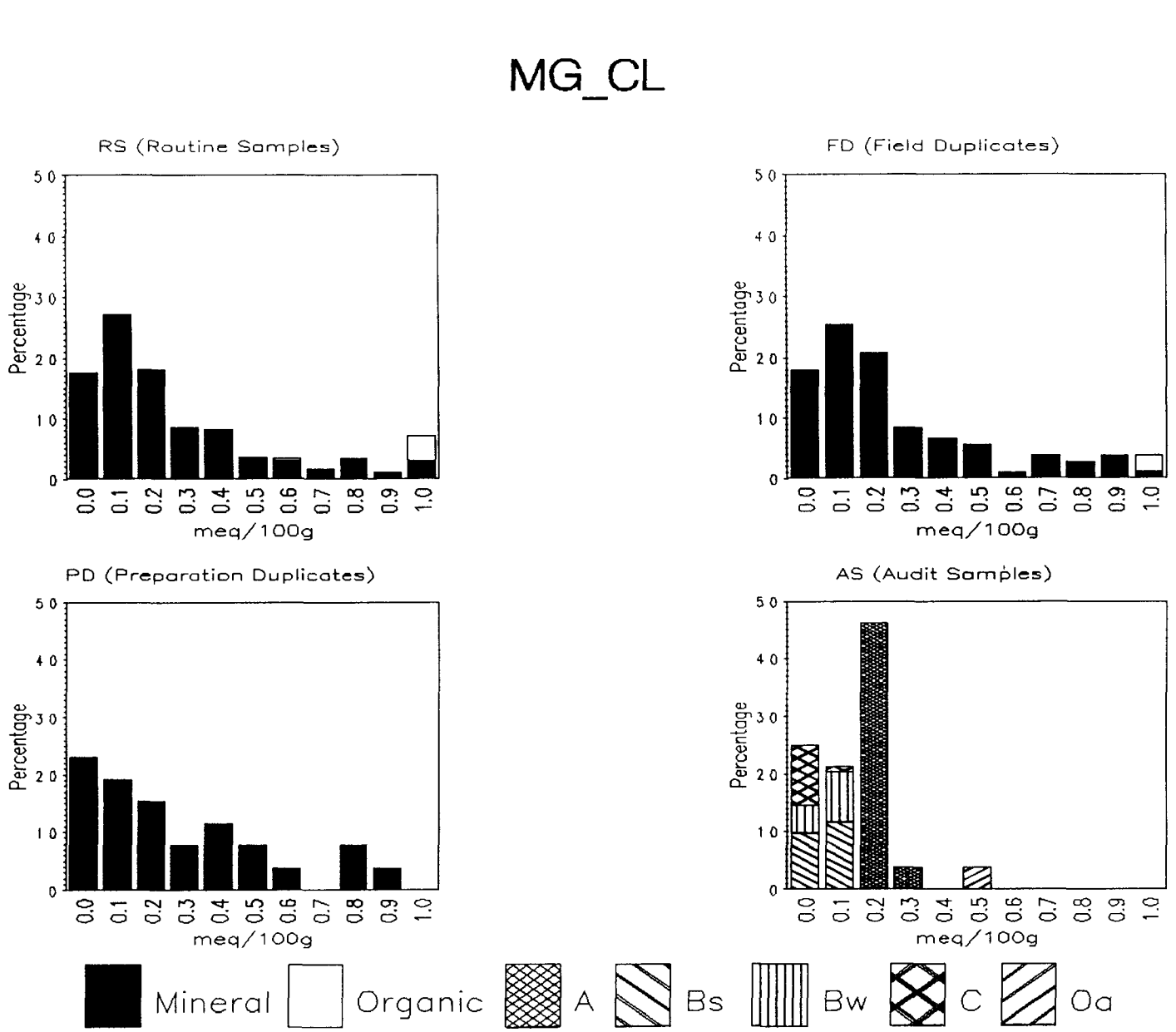

Figure G-17. Histogram of range and frequency distribution for magnesium in ammonium chloride.

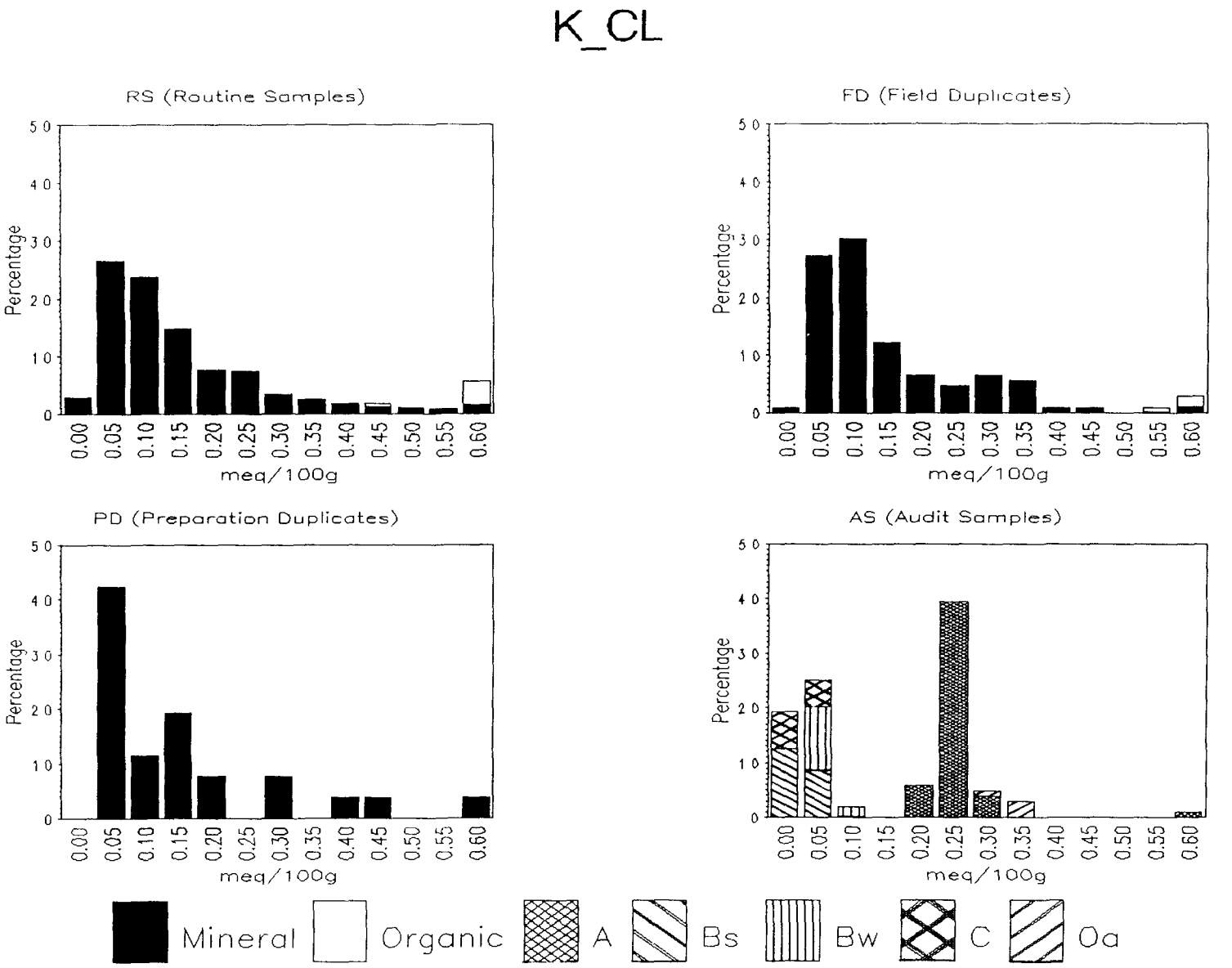

Figure G-18. Histogram of range and frequency distribution for potassium in ammonium chloride.

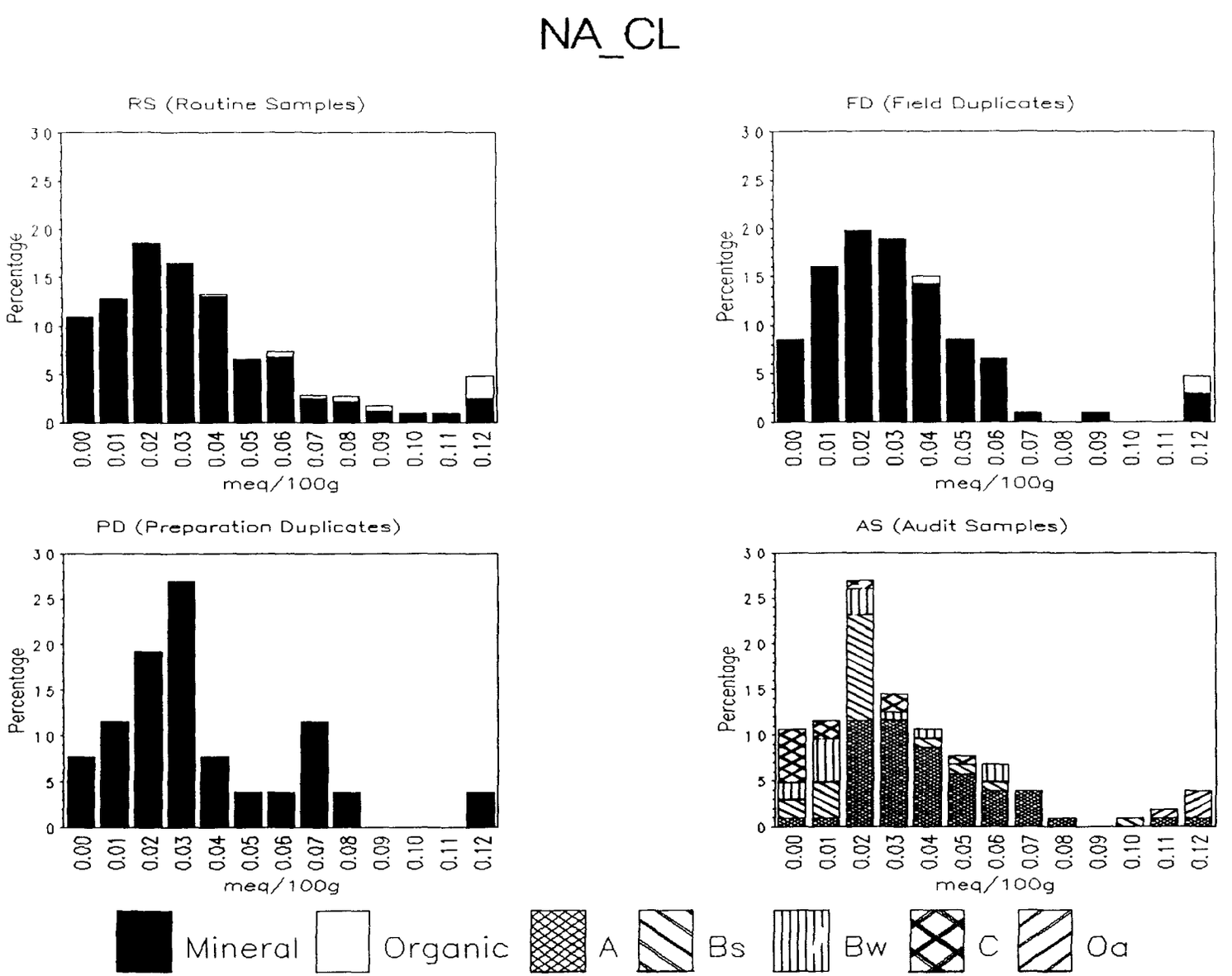

Figure G-19. Histogram of range and frequency distribution for sodium in ammonium chloride.

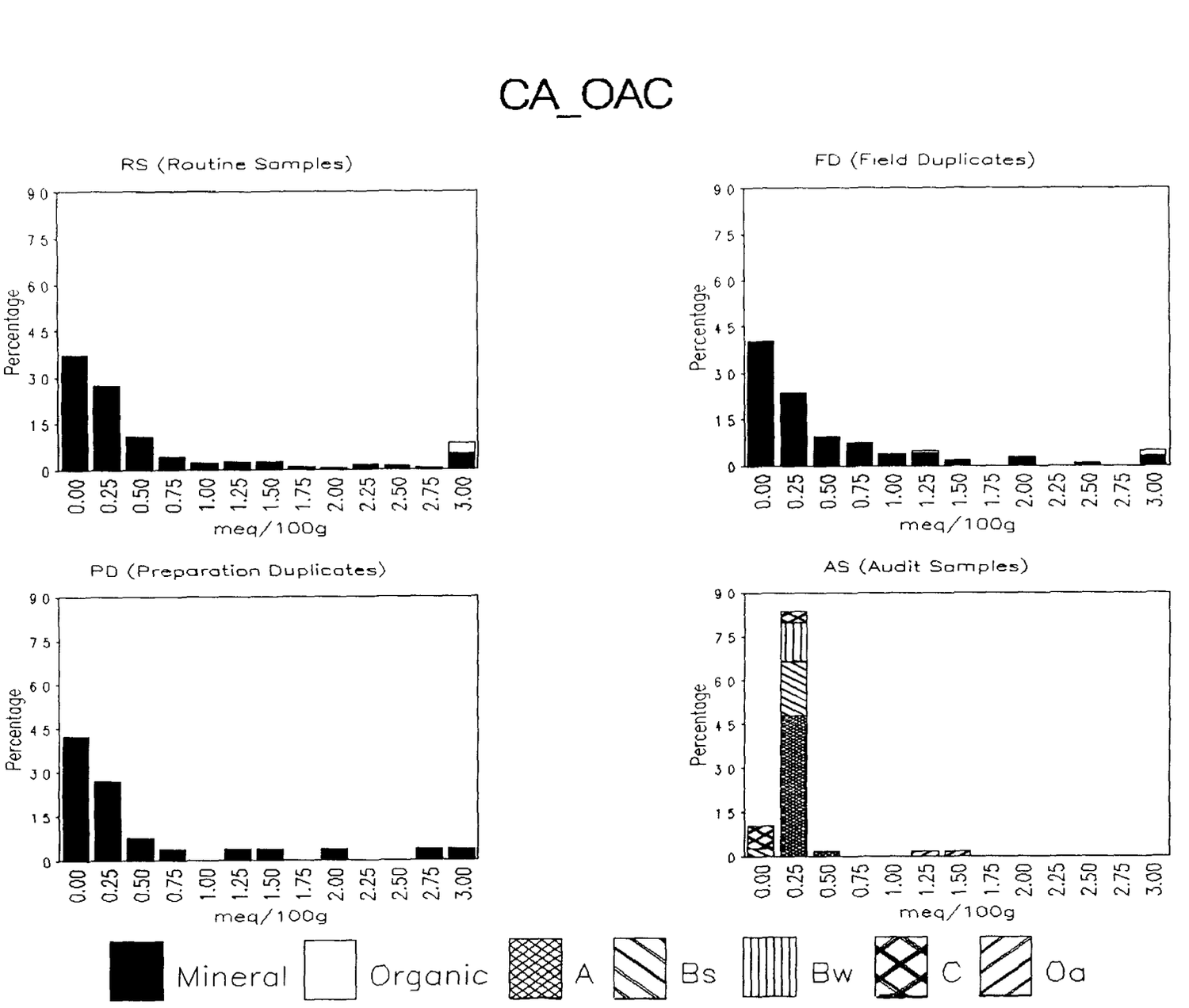

Figure G-20. Histogram of range and frequency distribution for calcium in ammonium acetate.

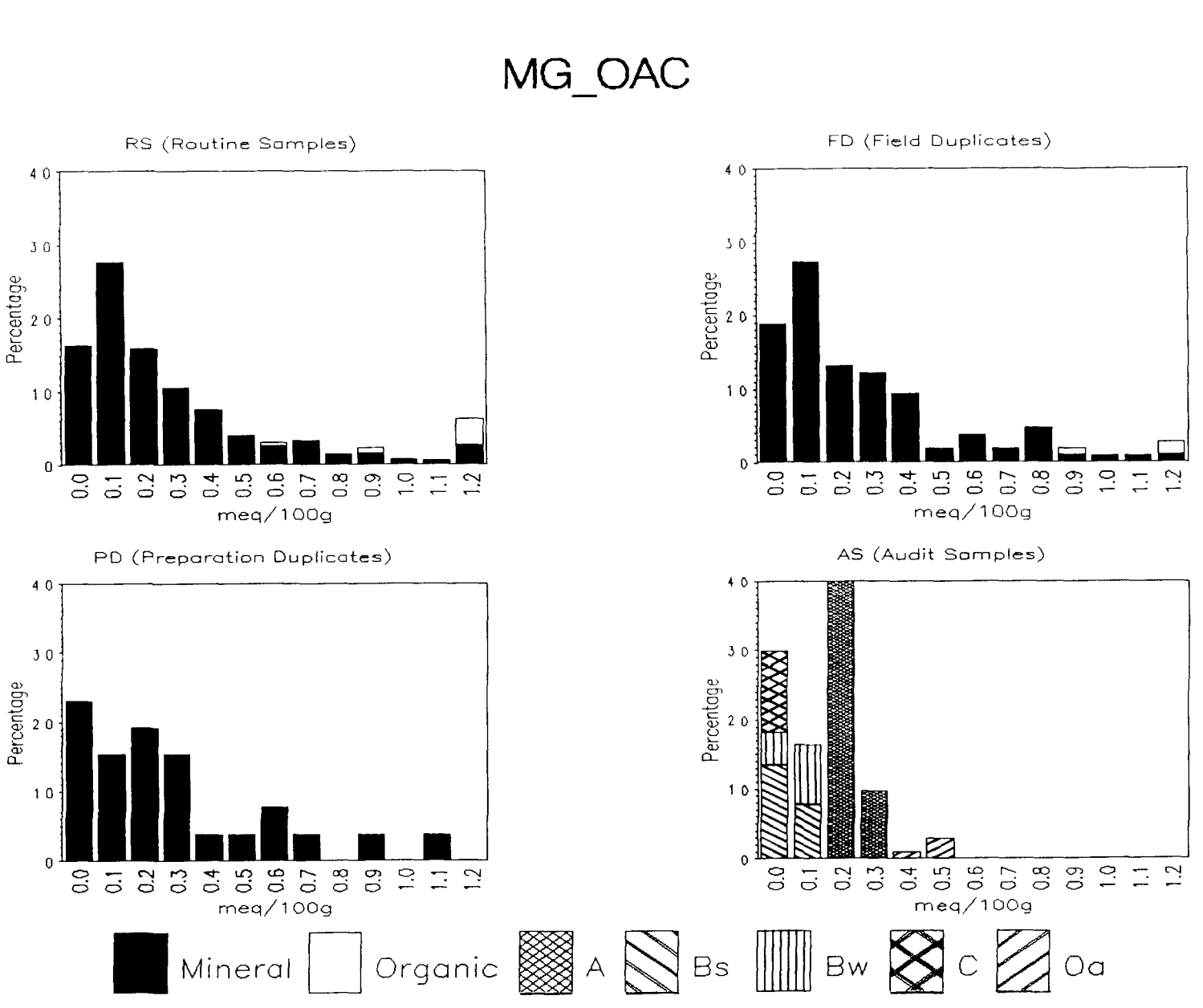

Figure G-21. Histogram of range and frequency distribution for magnesium in ammonium acetate.

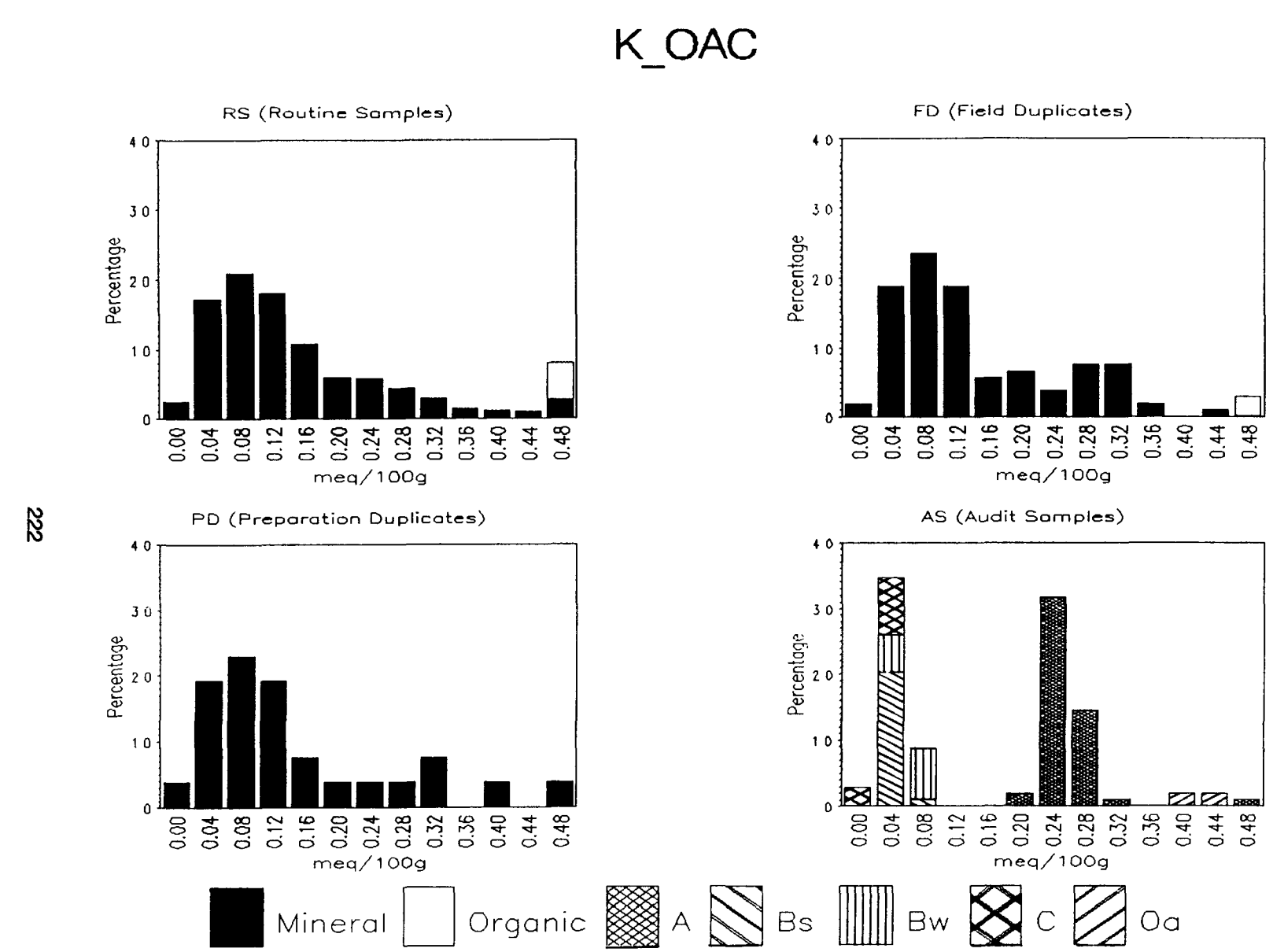

Figure G-22. Histogram of range and frequency distribution for potassium in ammonium acetate.

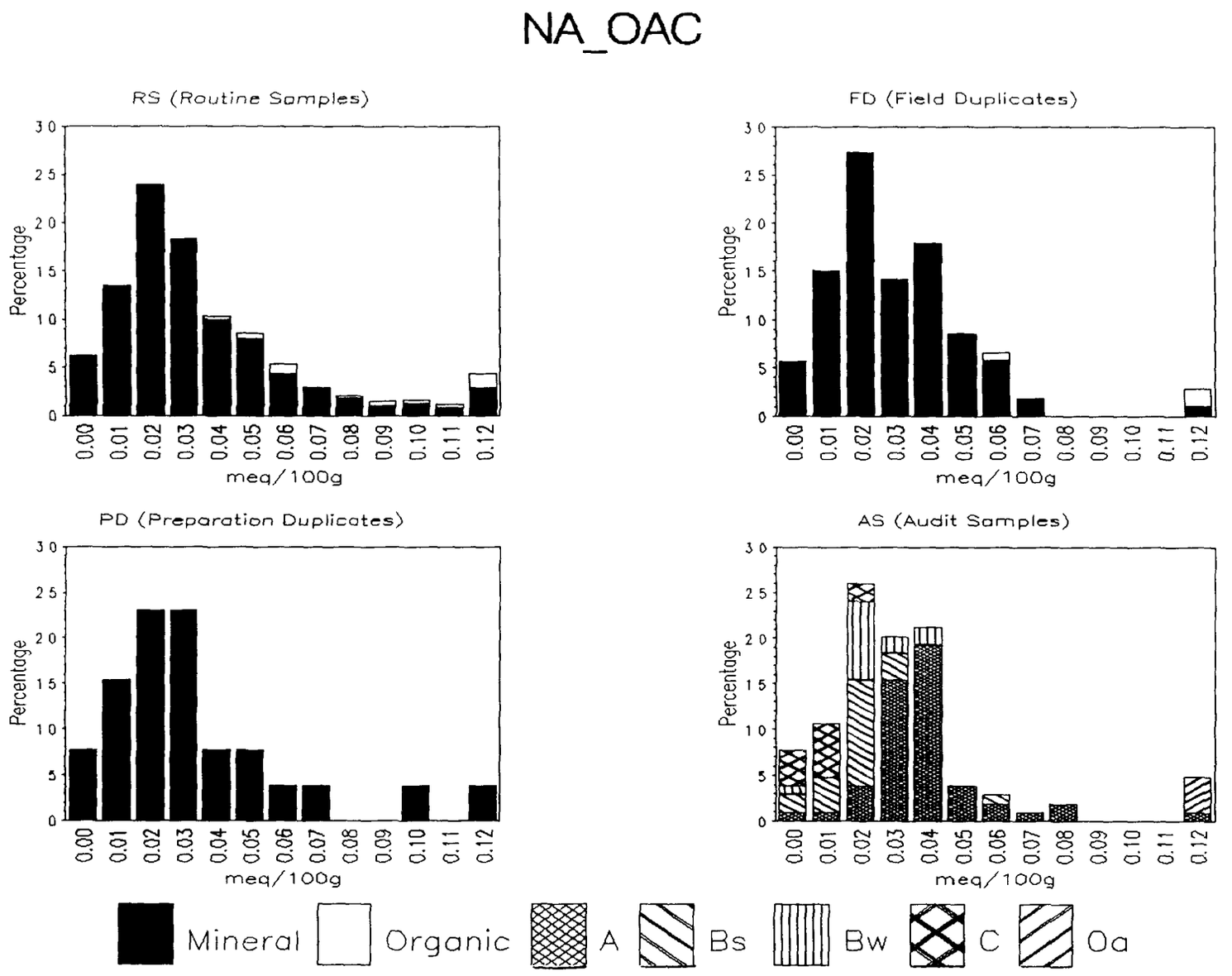

Figure G-23. Histogram of range and frequency distribution for sodium in ammonium acetate.

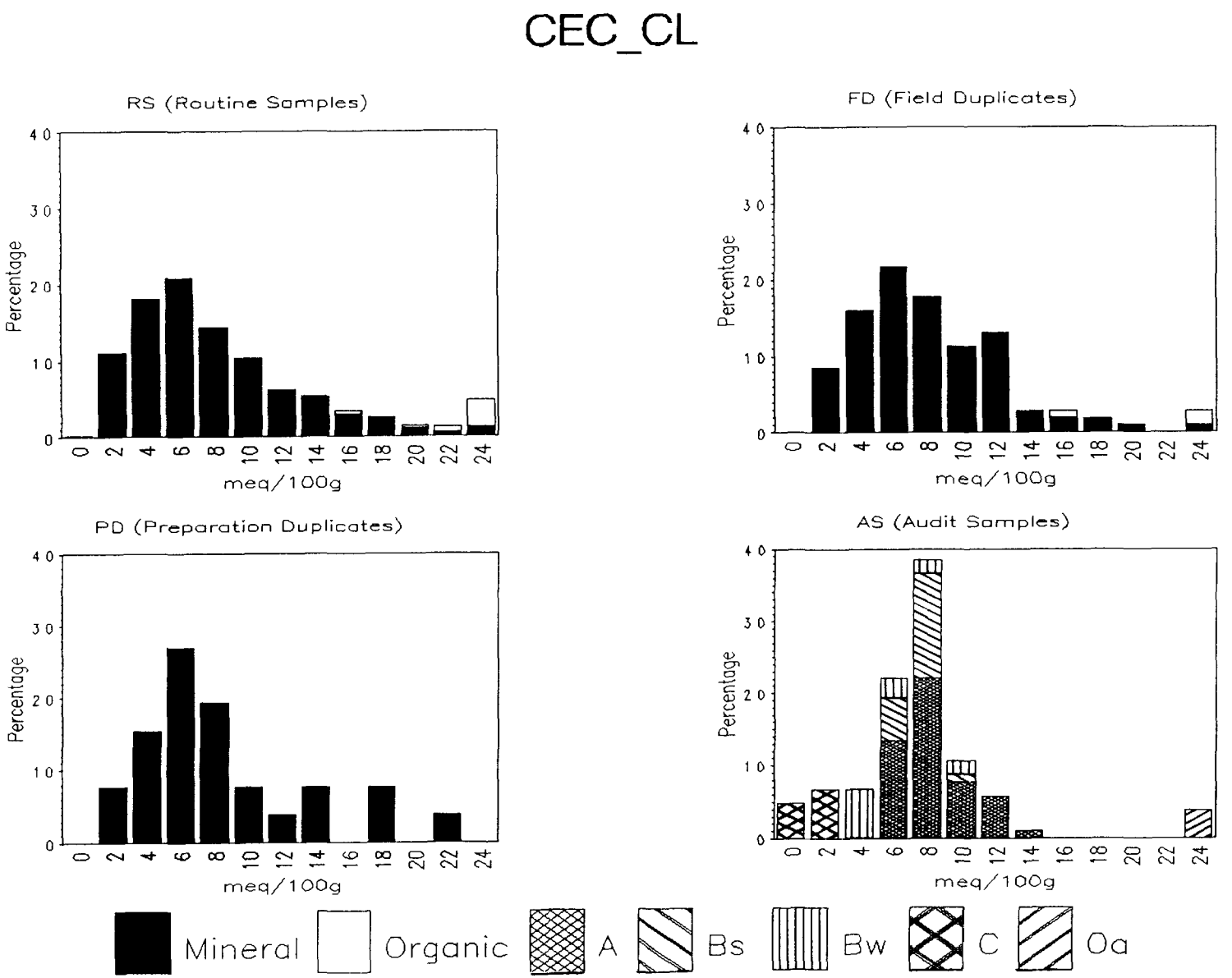

Figure G-24. Histogram of range and frequency distribution for cation exchange capacity in ammonium chloride.

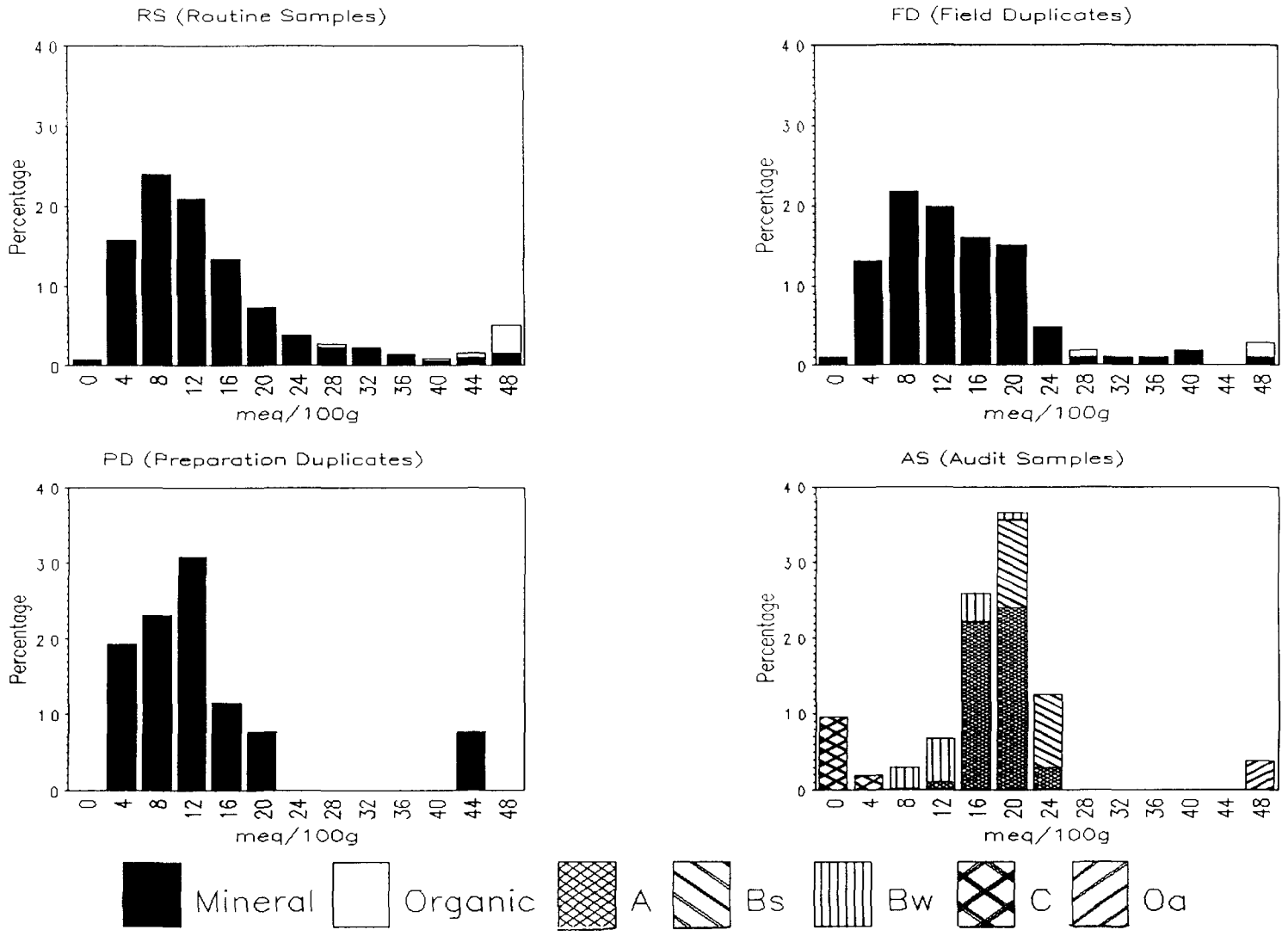

Figure G-25. Histogram of range and frequency distribution for cation exchange capacity in ammonium acetate.

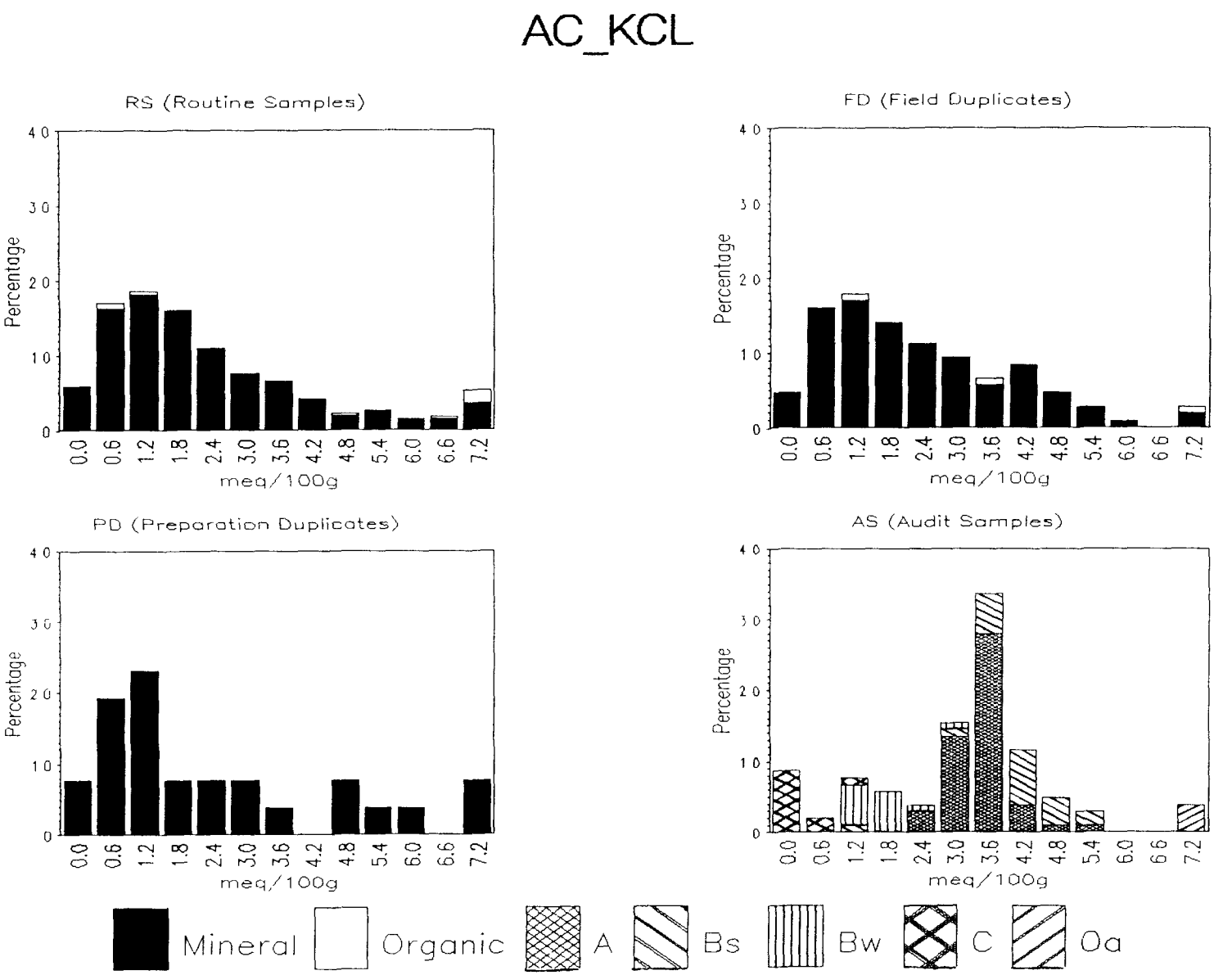

Figure G-26. Histogram of range and frequency distribution for exchangeable acidity in potassium chloride.

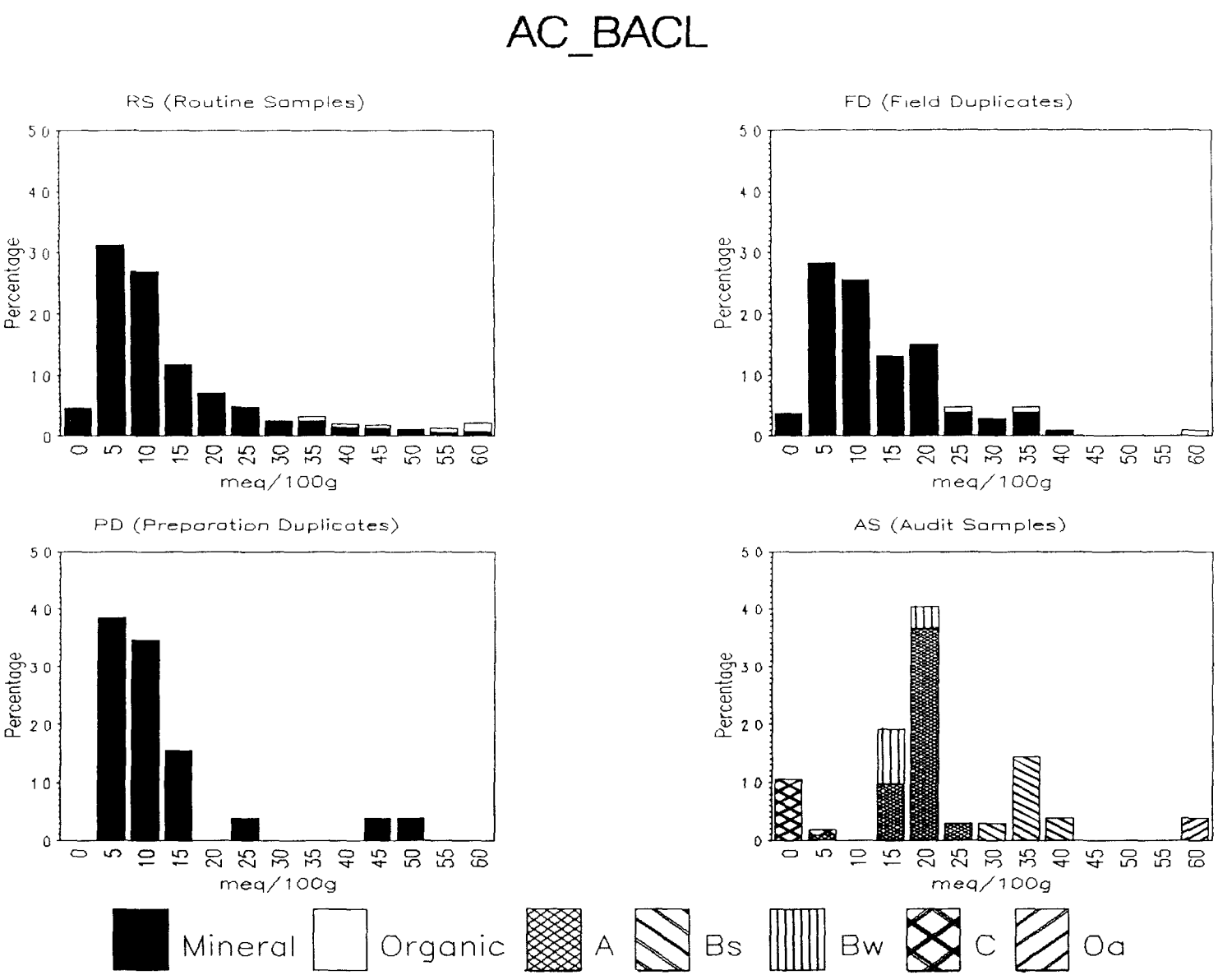

Figure G-27. Histogram of range and frequency distribution for exchangeable acidity in barium chloride triethanolamine.

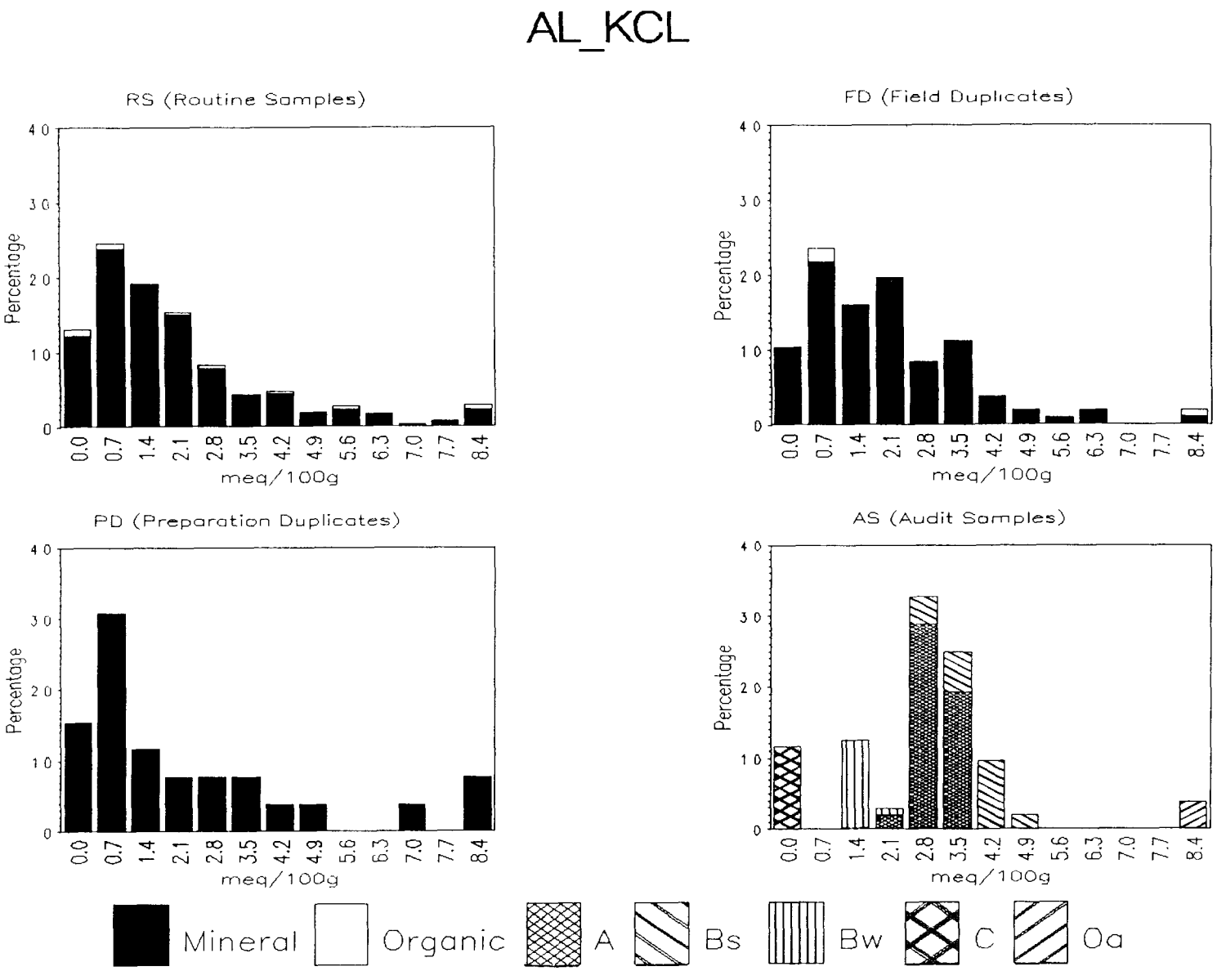

Figure G-28. Histogram of range and frequency distribution for exchangeable aluminum in potassium chloride.

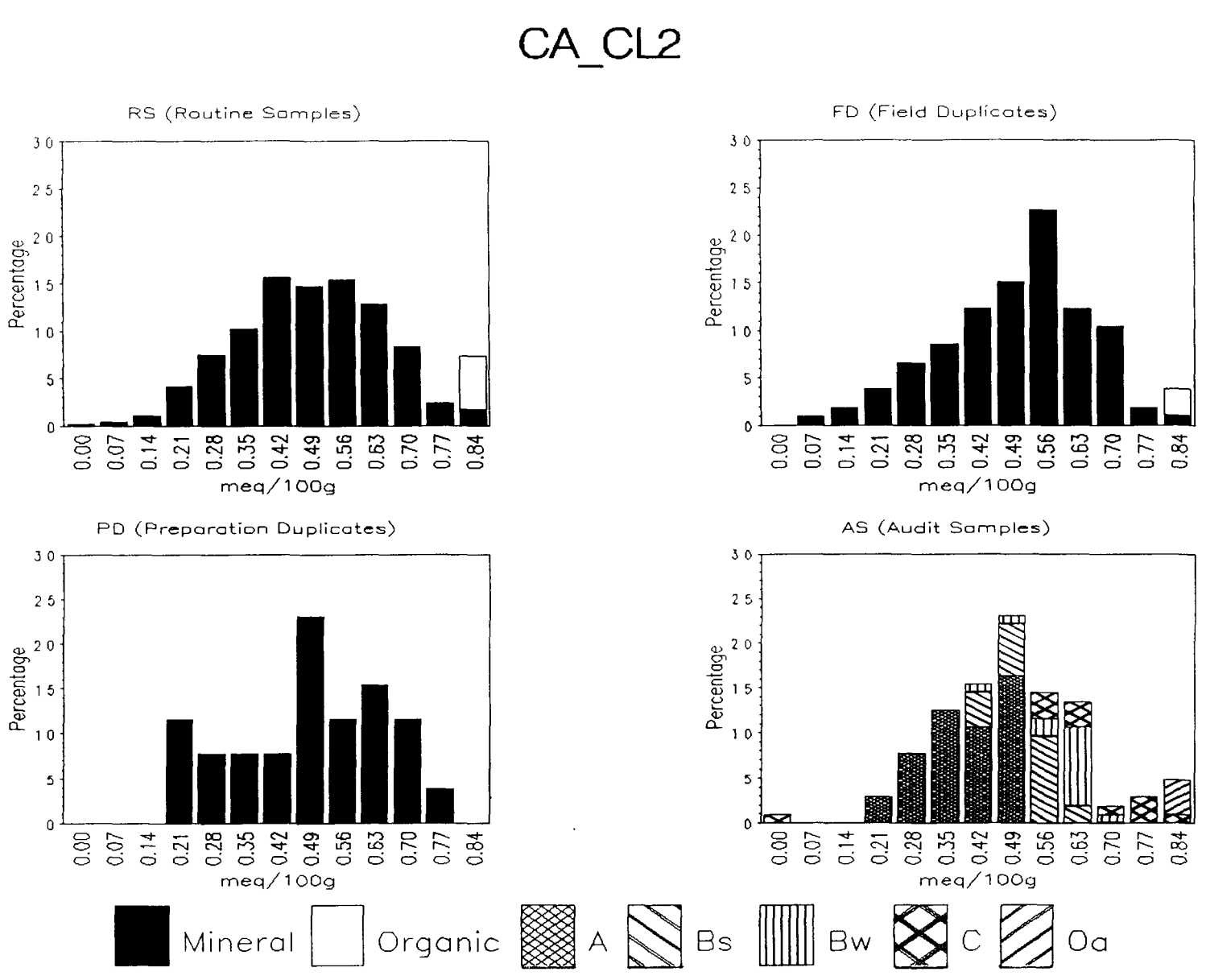

Figure G-29. Histogram of range and frequency distribution for calcium in calcium chloride.

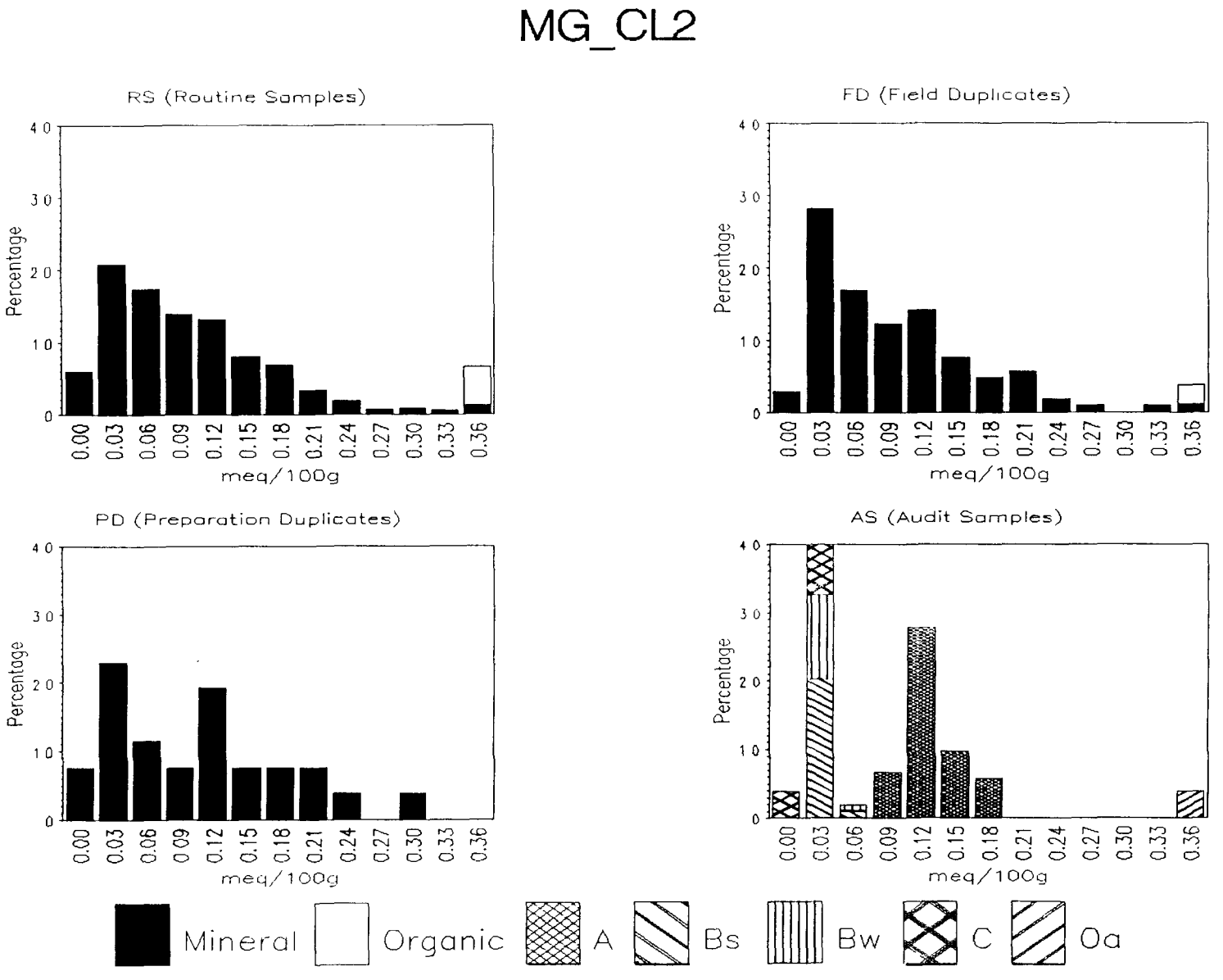

Figure G-30. Histogram of range and frequency distribution for magnesium in calcium chloride.

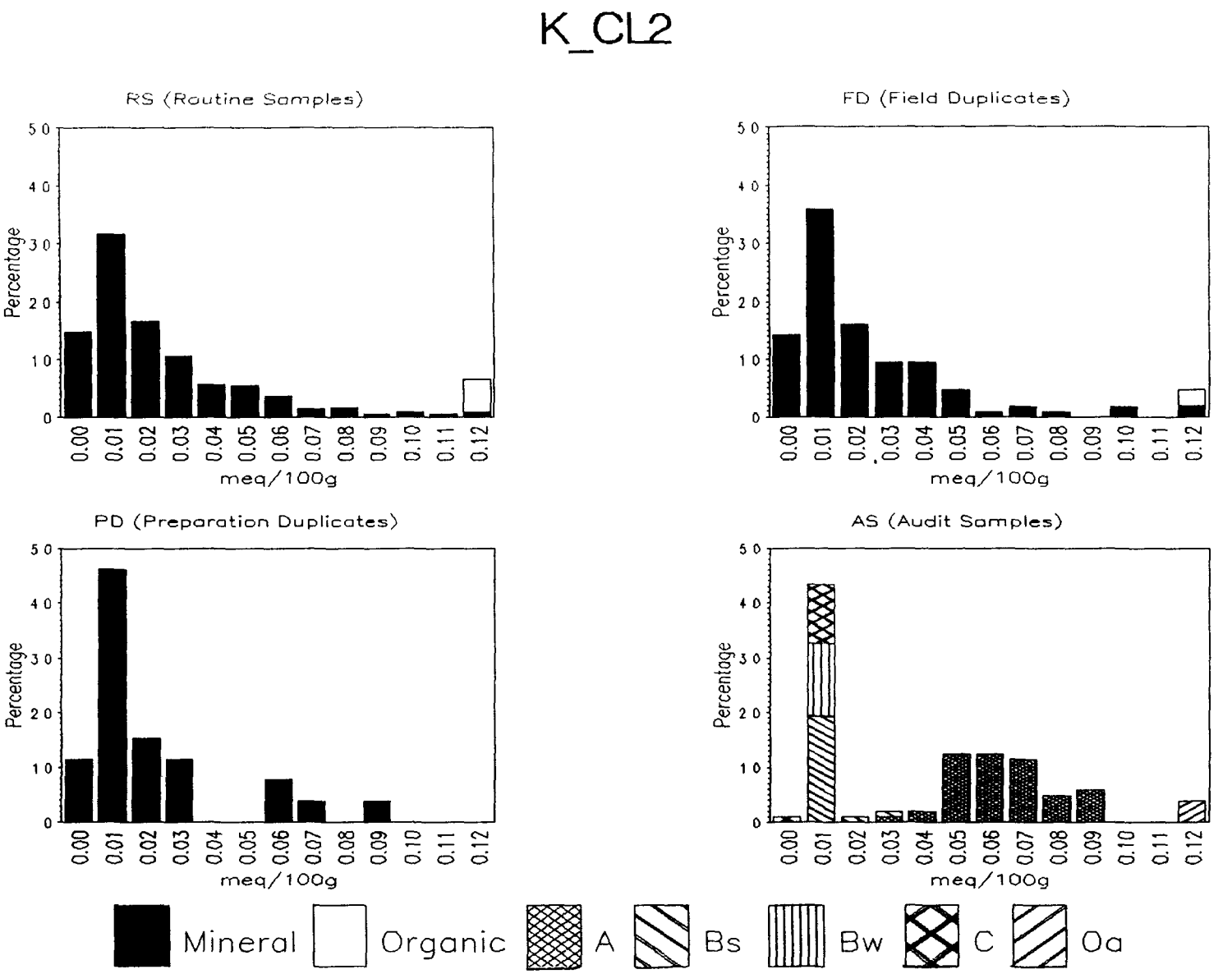

Figure G-31. Histogram of range and frequency distribution for potassium in calcium chloride.

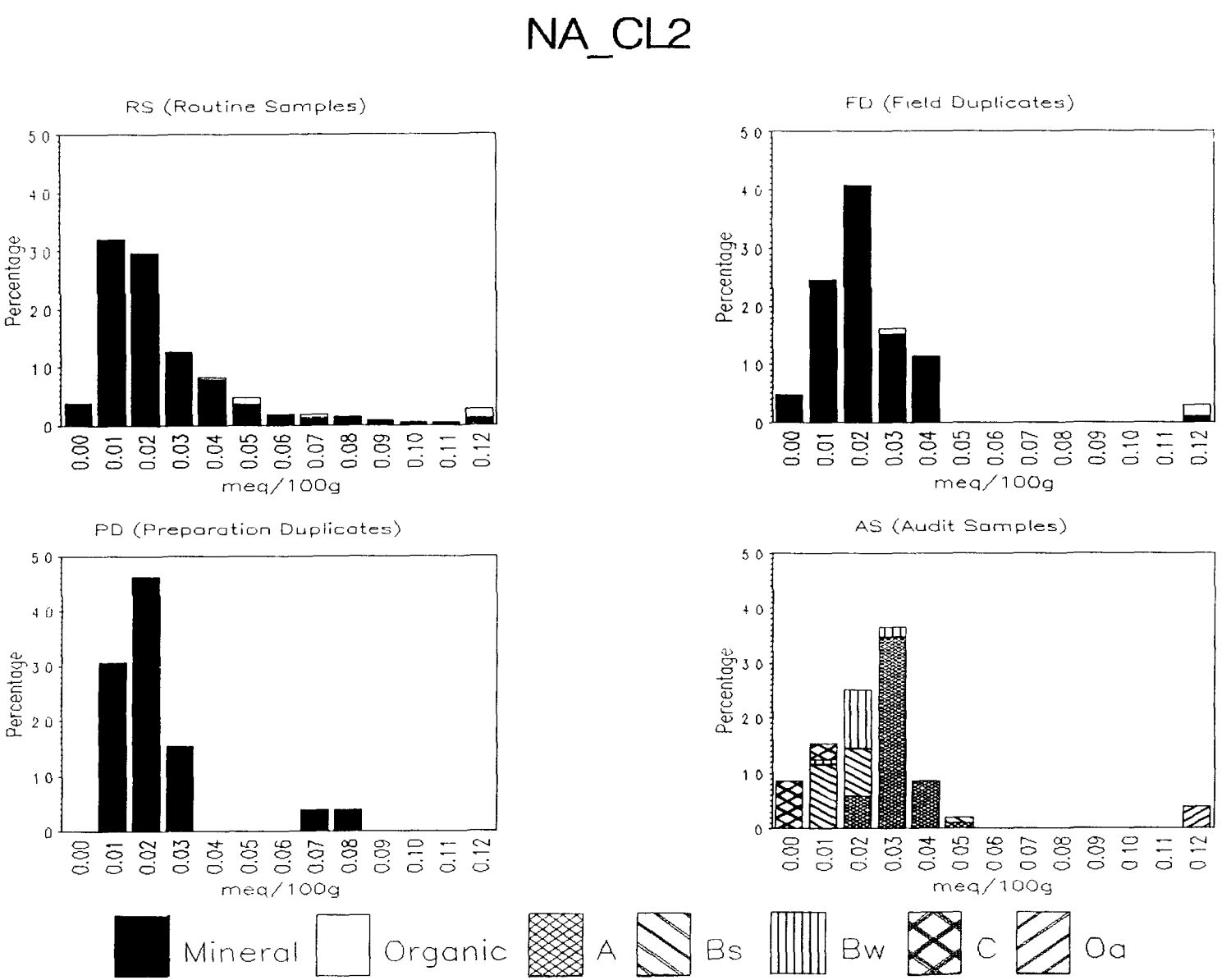

Figure G-32. Histogram of range and frequency distribution for sodium in calcium chloride.

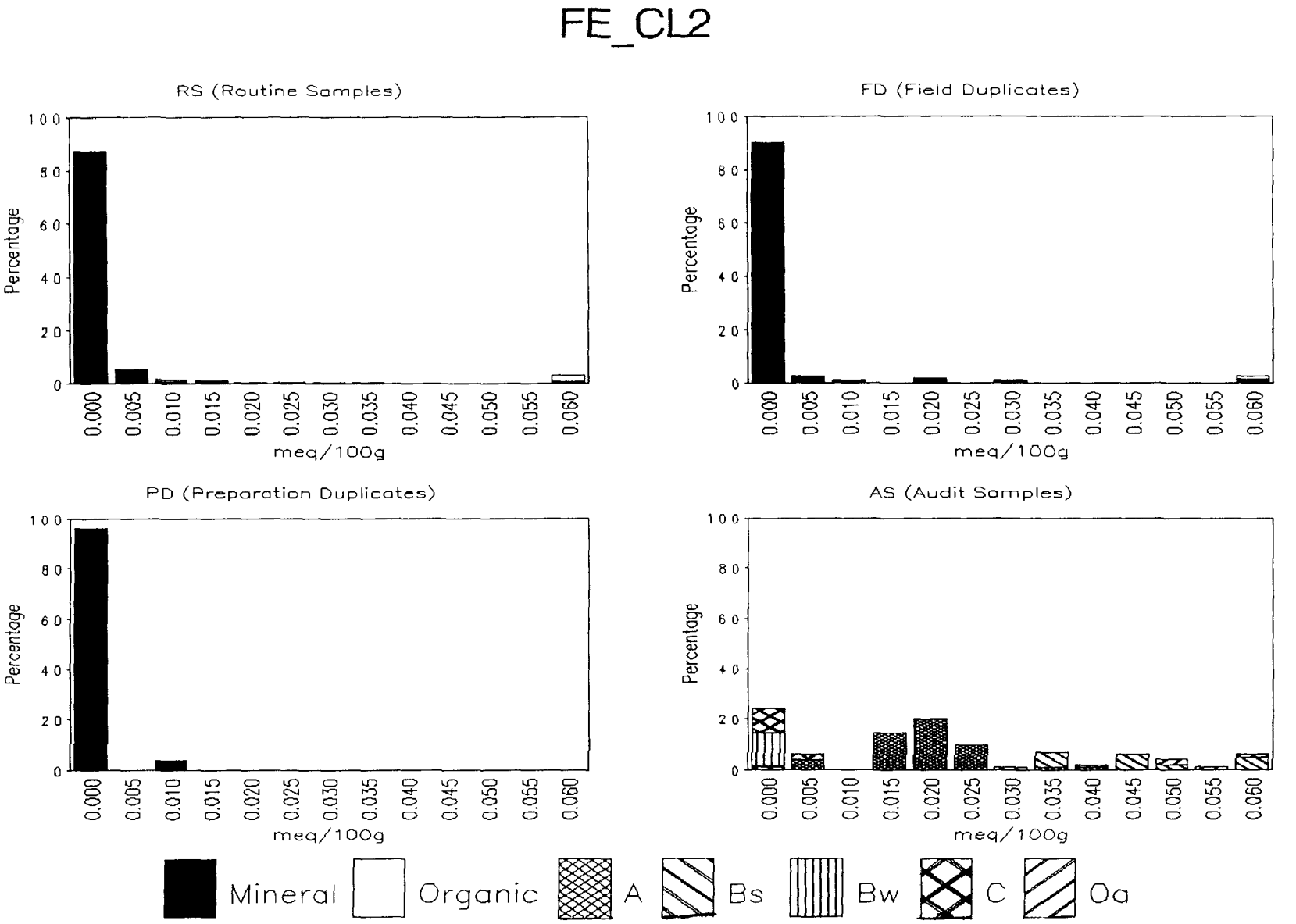

Figure G-33. Histogram of range and frequency distribution for iron in calcium chloride.

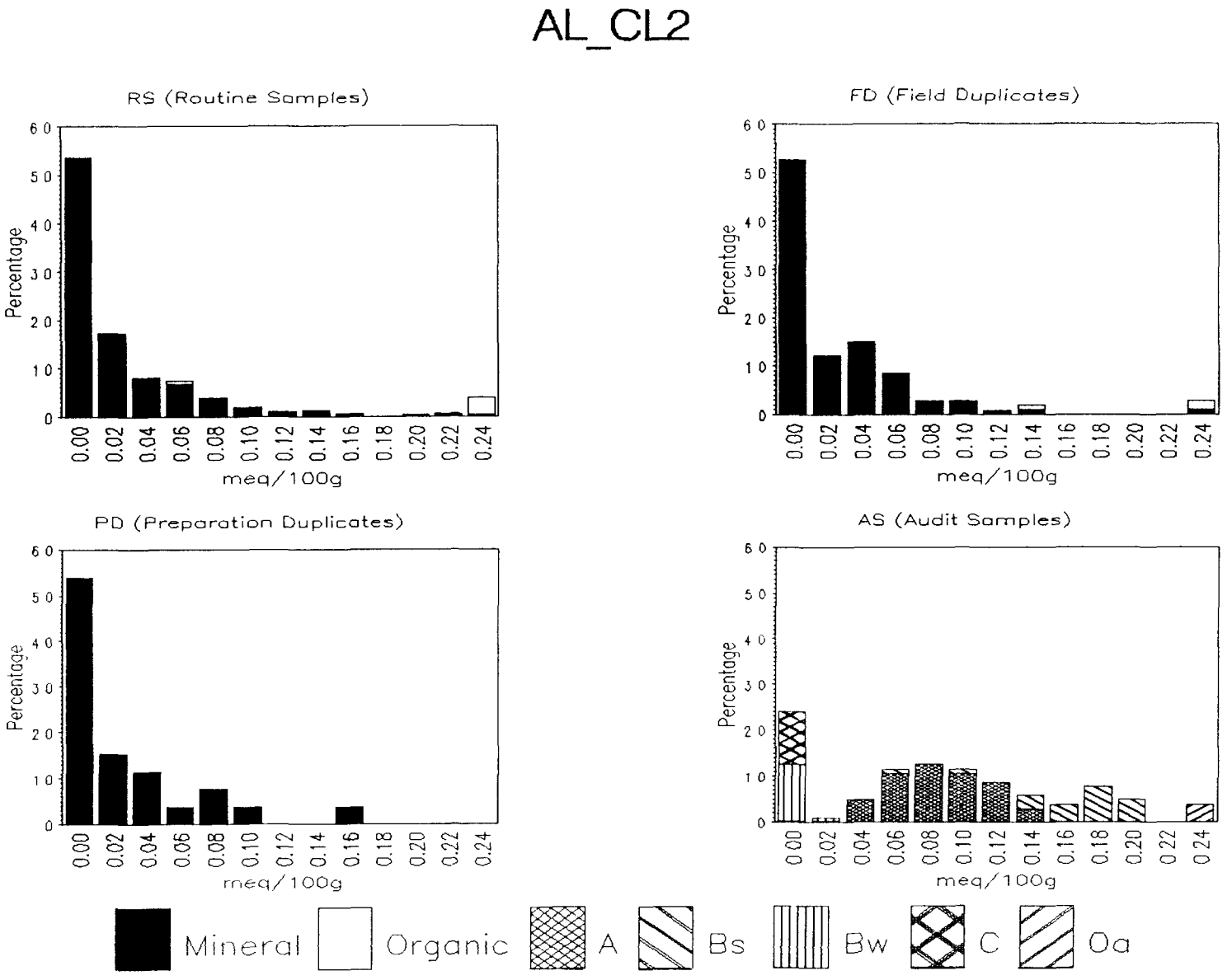

Figure G-34. Histogram of range and frequency distribution for aluminum in calcium chloride.

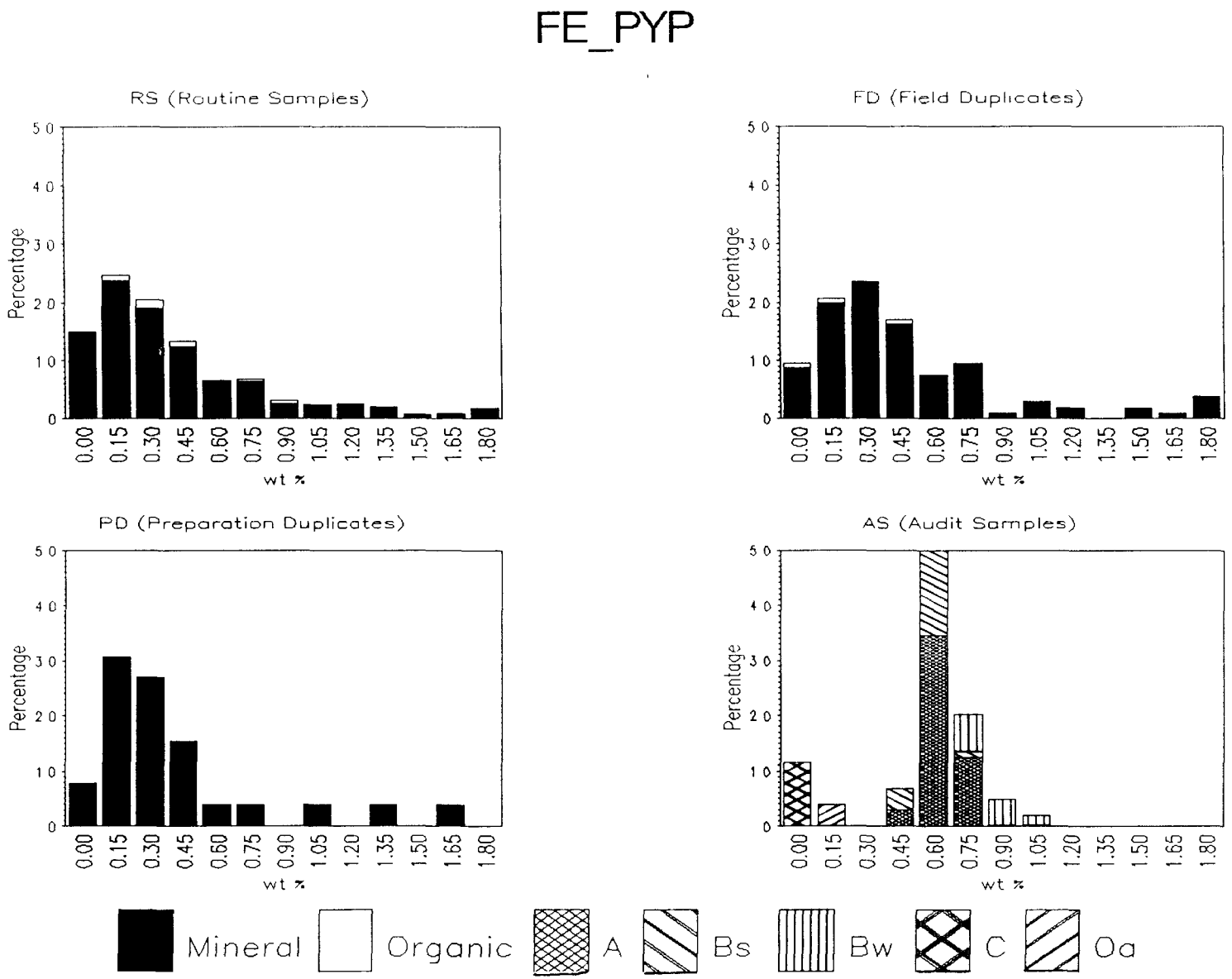

Figure G-35. Histogram of range and frequency distribution for iron in pyrophosphate.

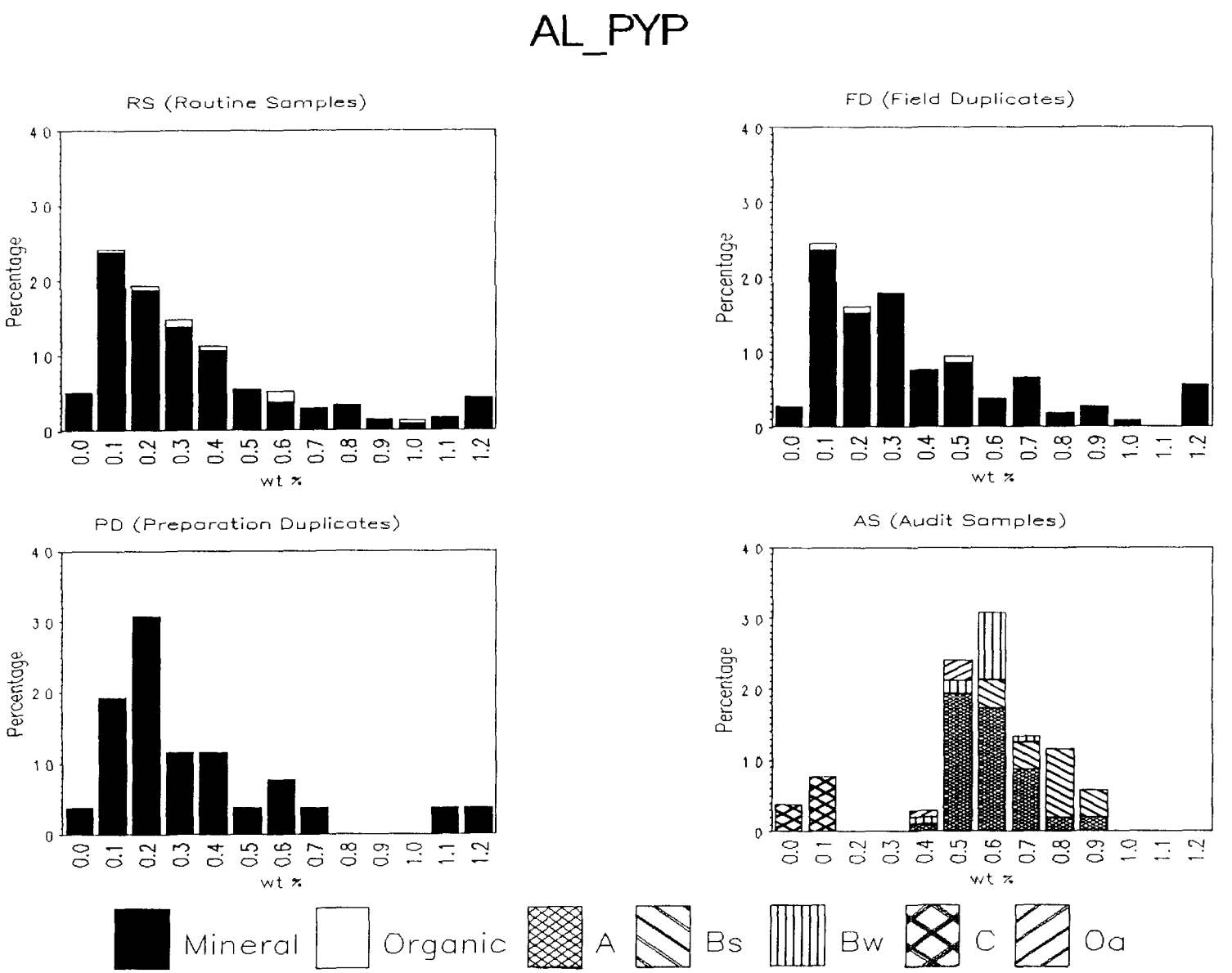

Figure G-36. Histogram of range and frequency distribution for aluminum in pyrophosphate.

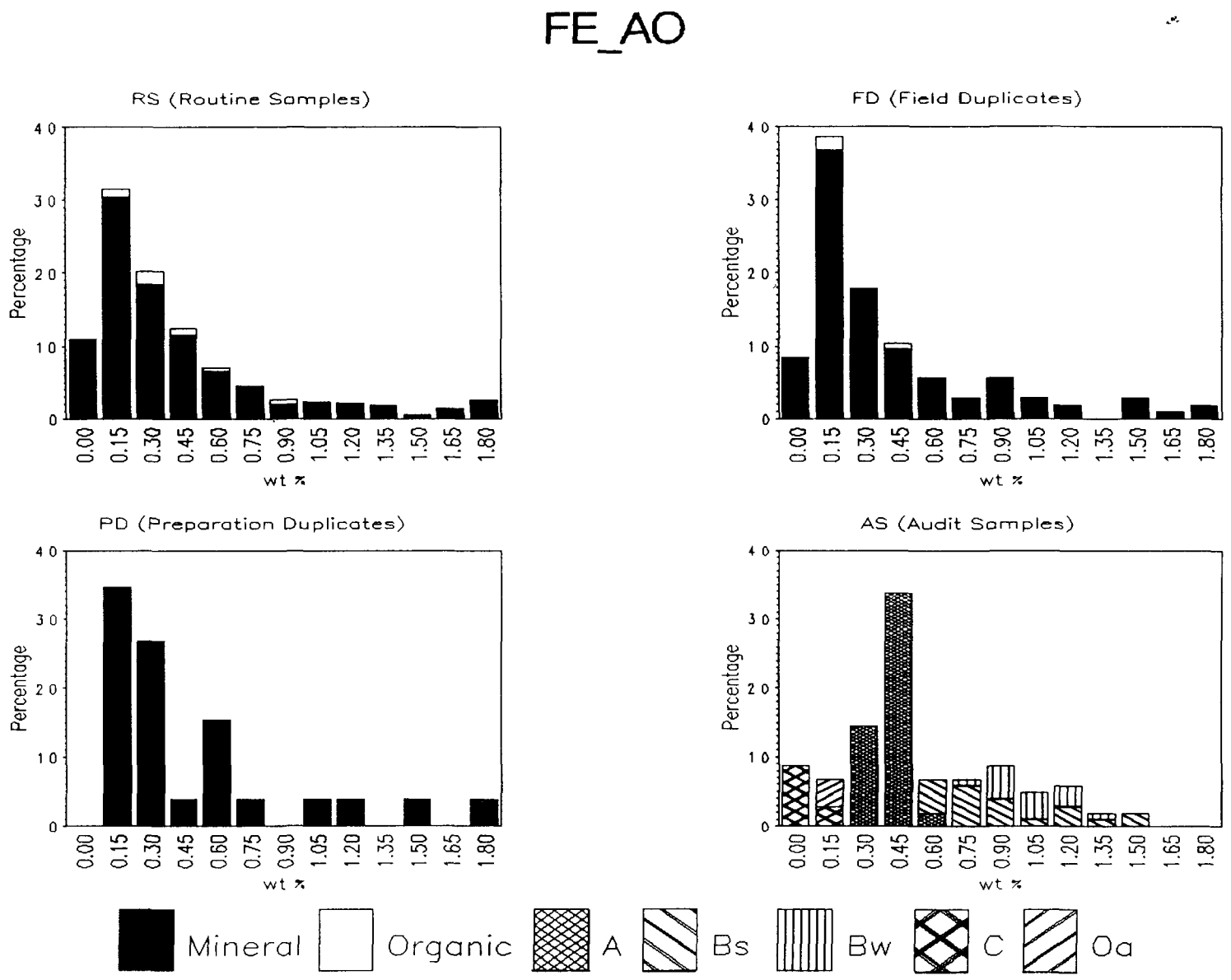

Figure G-37. Histogram of range and frequency distribution for iron in acid oxalate.

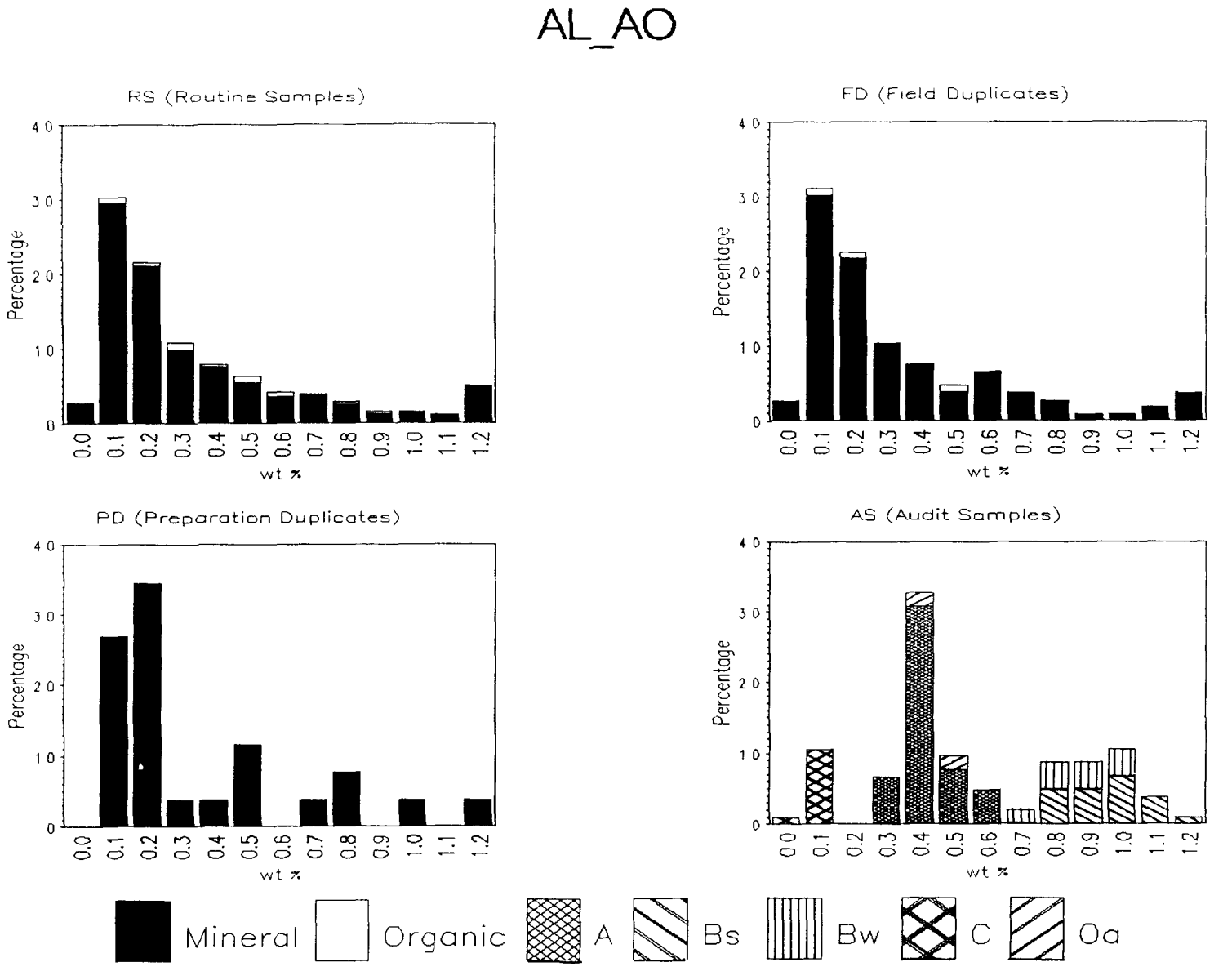

Figure G-38. Histogram of range and frequency distribution for aluminum in acid oxalate.

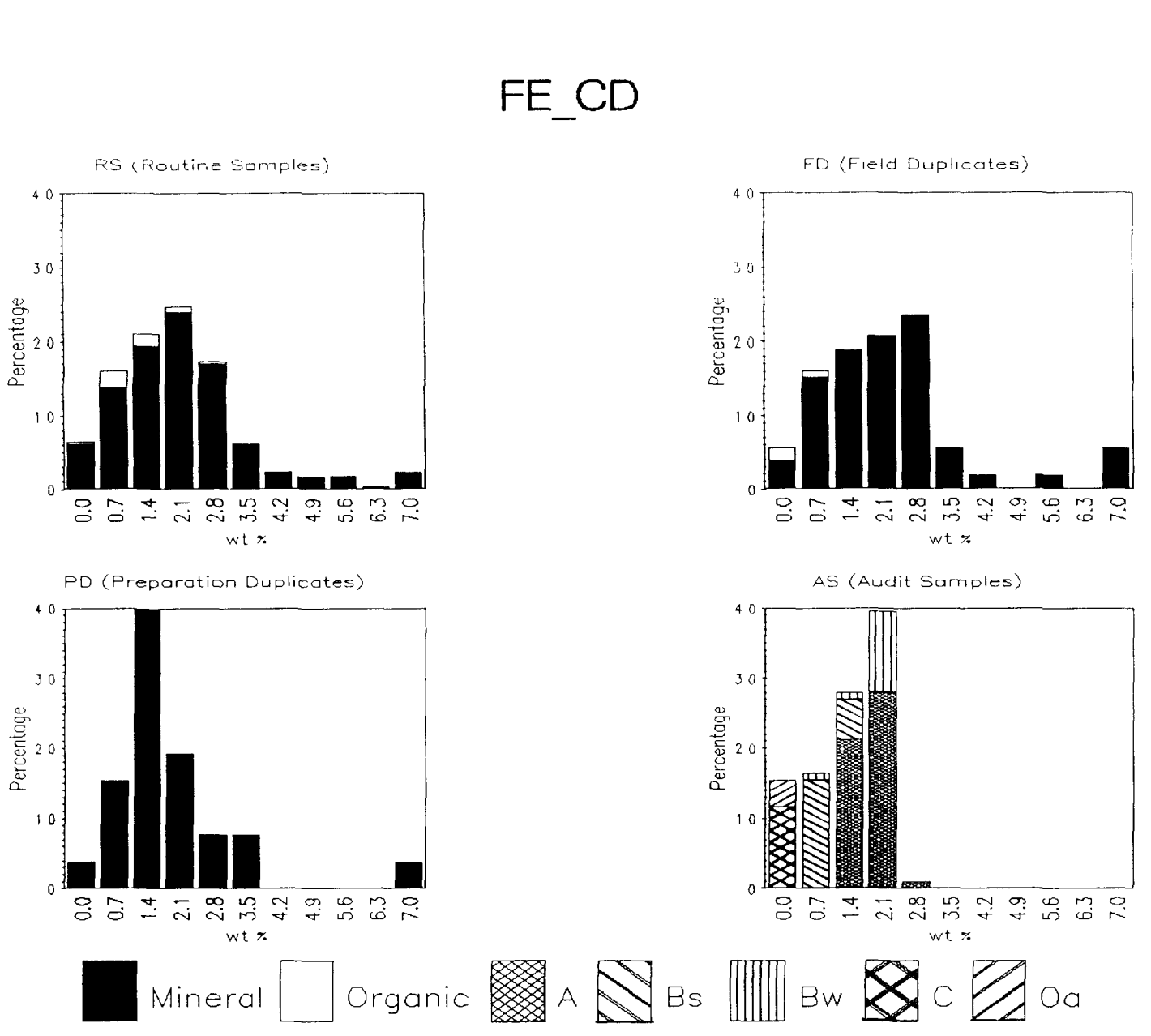

Figure G-39. Histogram of range and frequency distribution for iron in citrate dithionite.

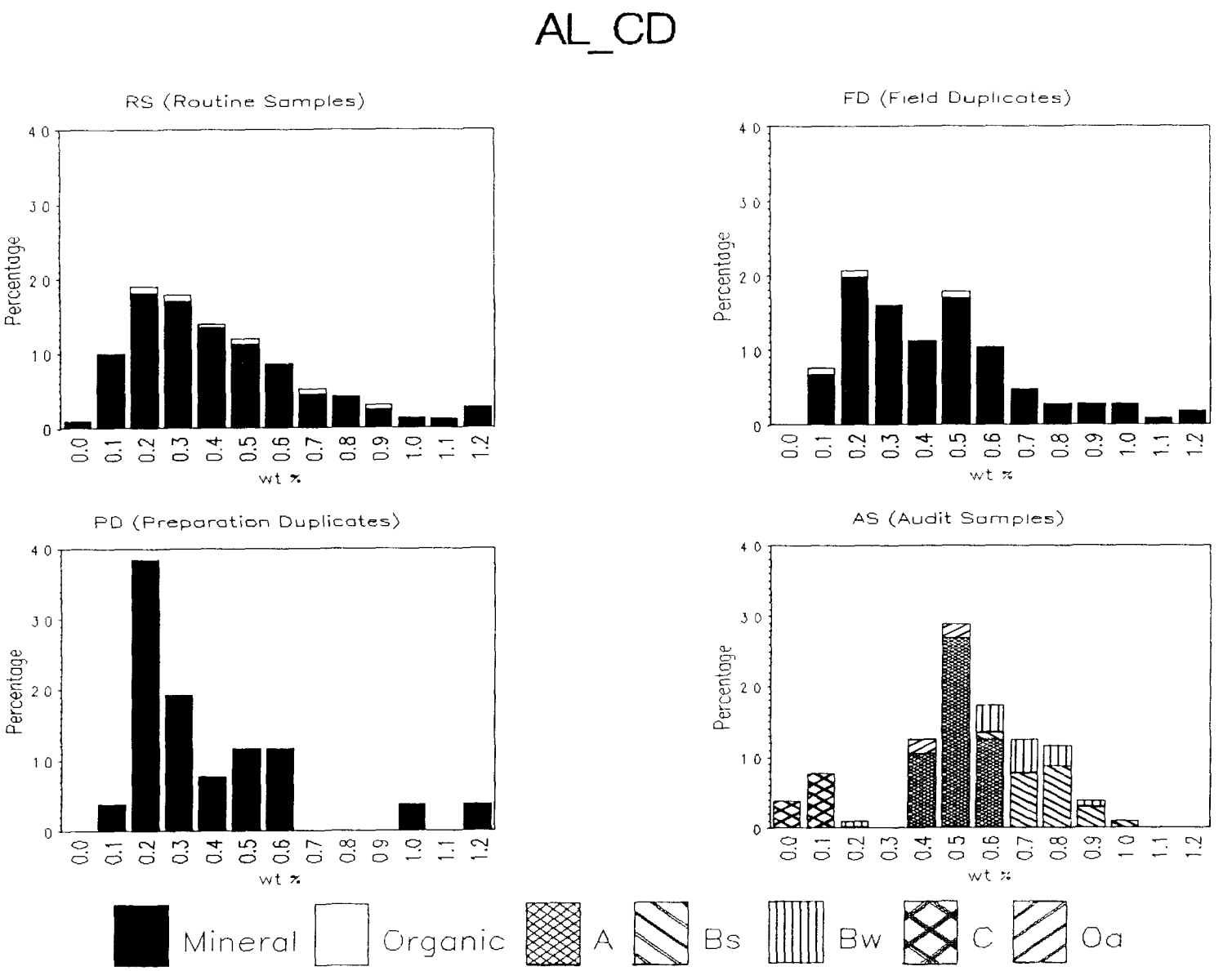

Figure G-40. Histogram of range and frequency distribution for aluminum in citrate dithionite.

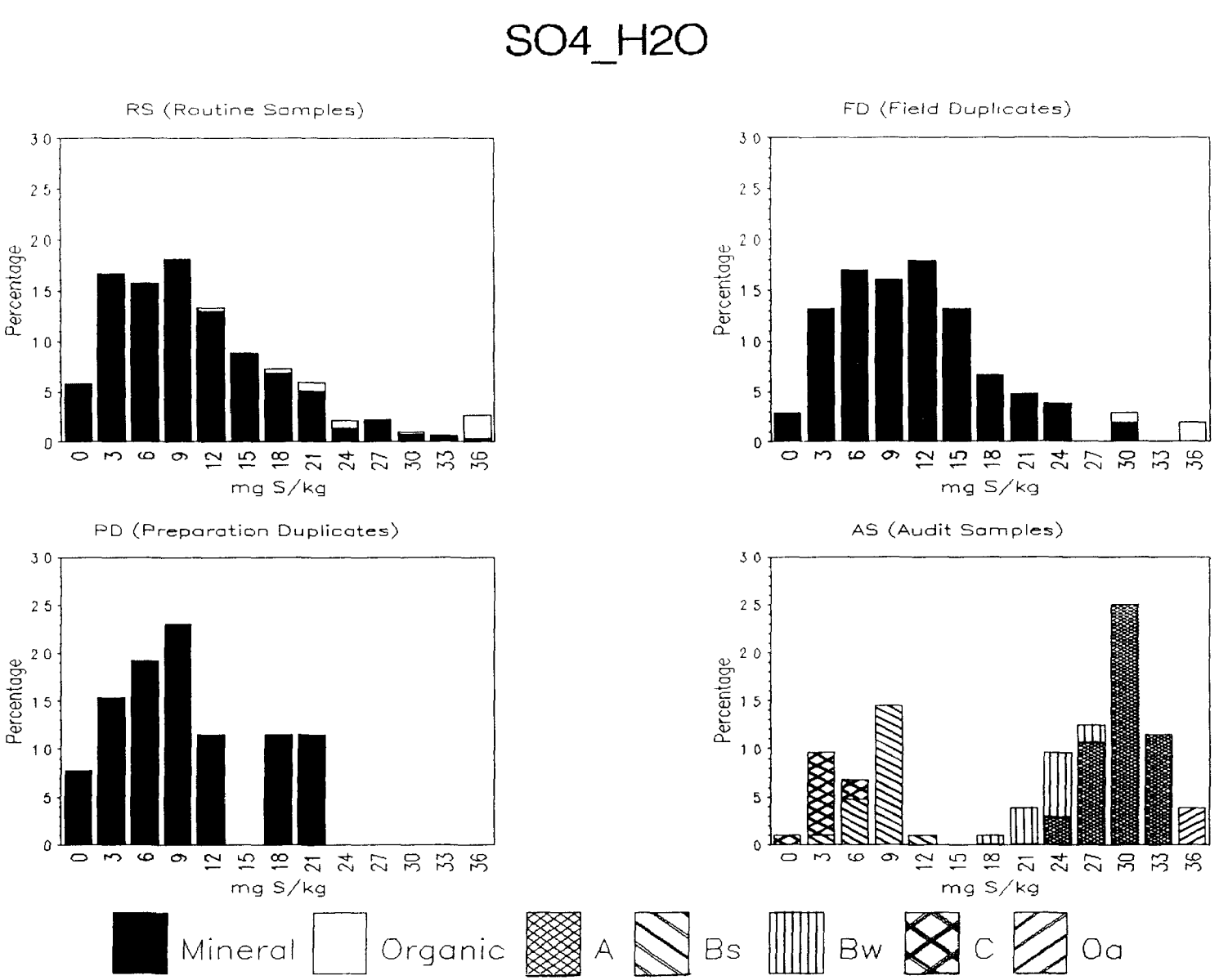

Figure G-41. Histogram of range and frequency distribution for water-extractable sulfate.

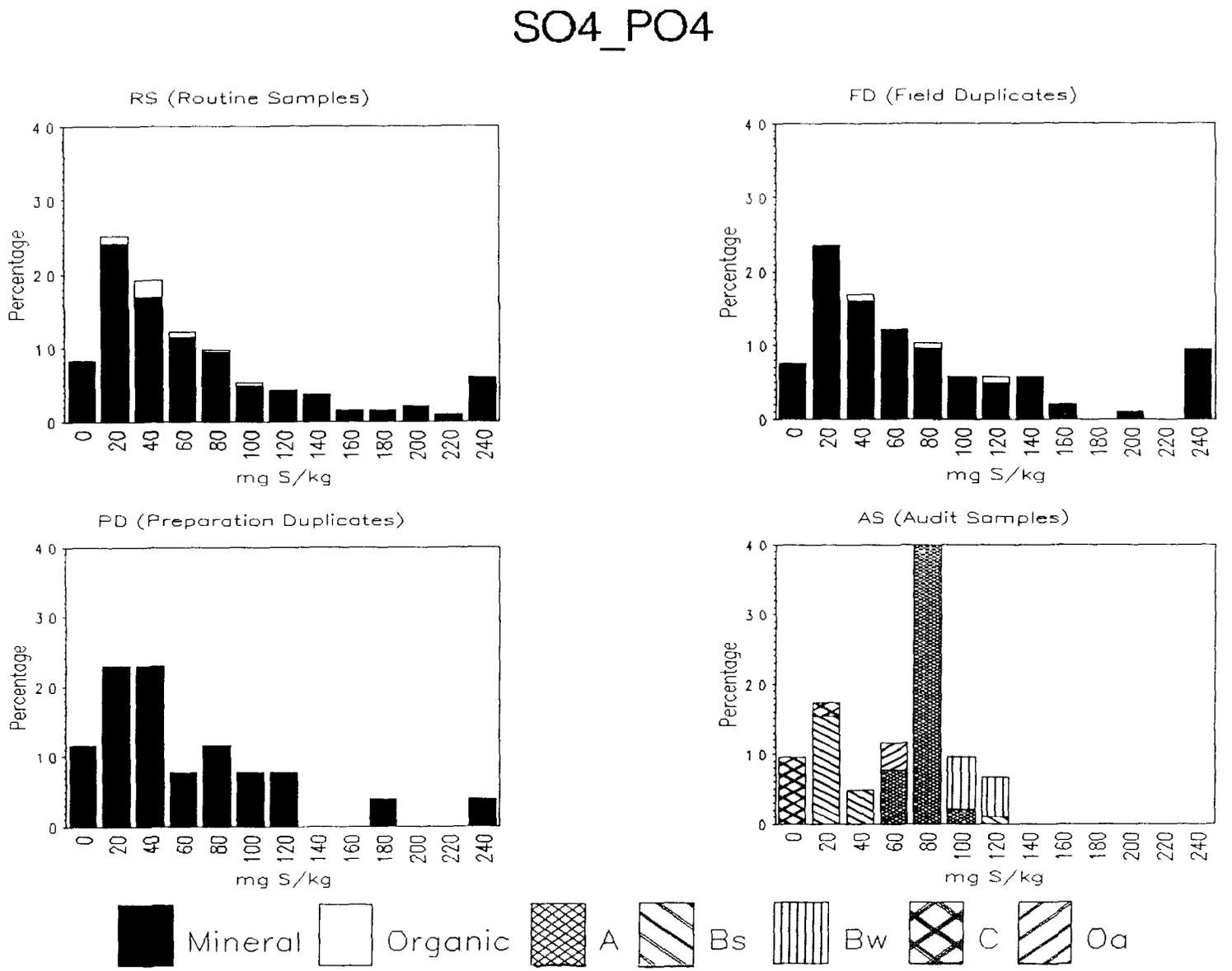

Figure G-42. Histogram of range and frequency distribution for phosphate-extractable sulfate.

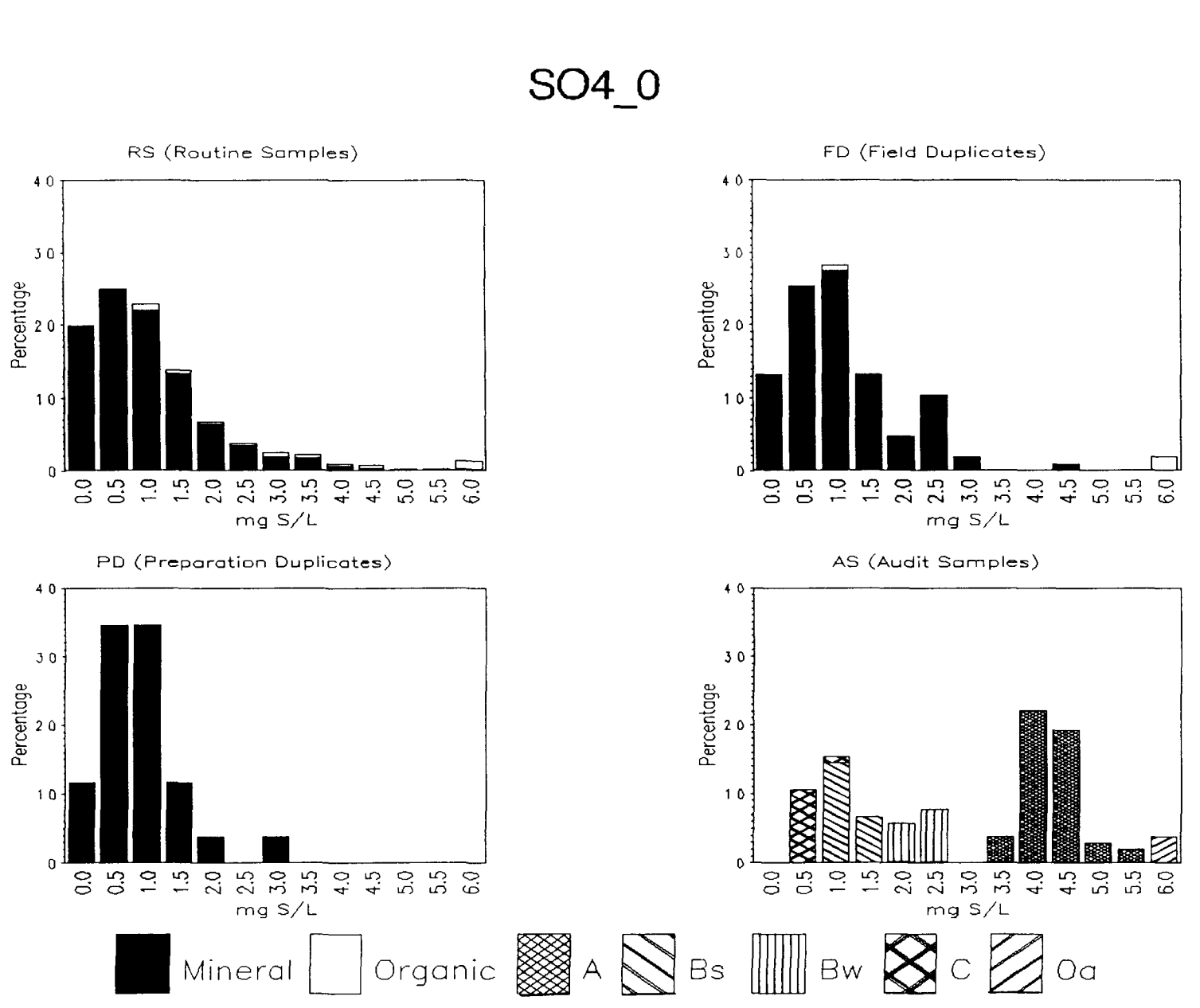

Figure G-43. Histogram of range and frequency distribution for the sulfate-zero isotherm.

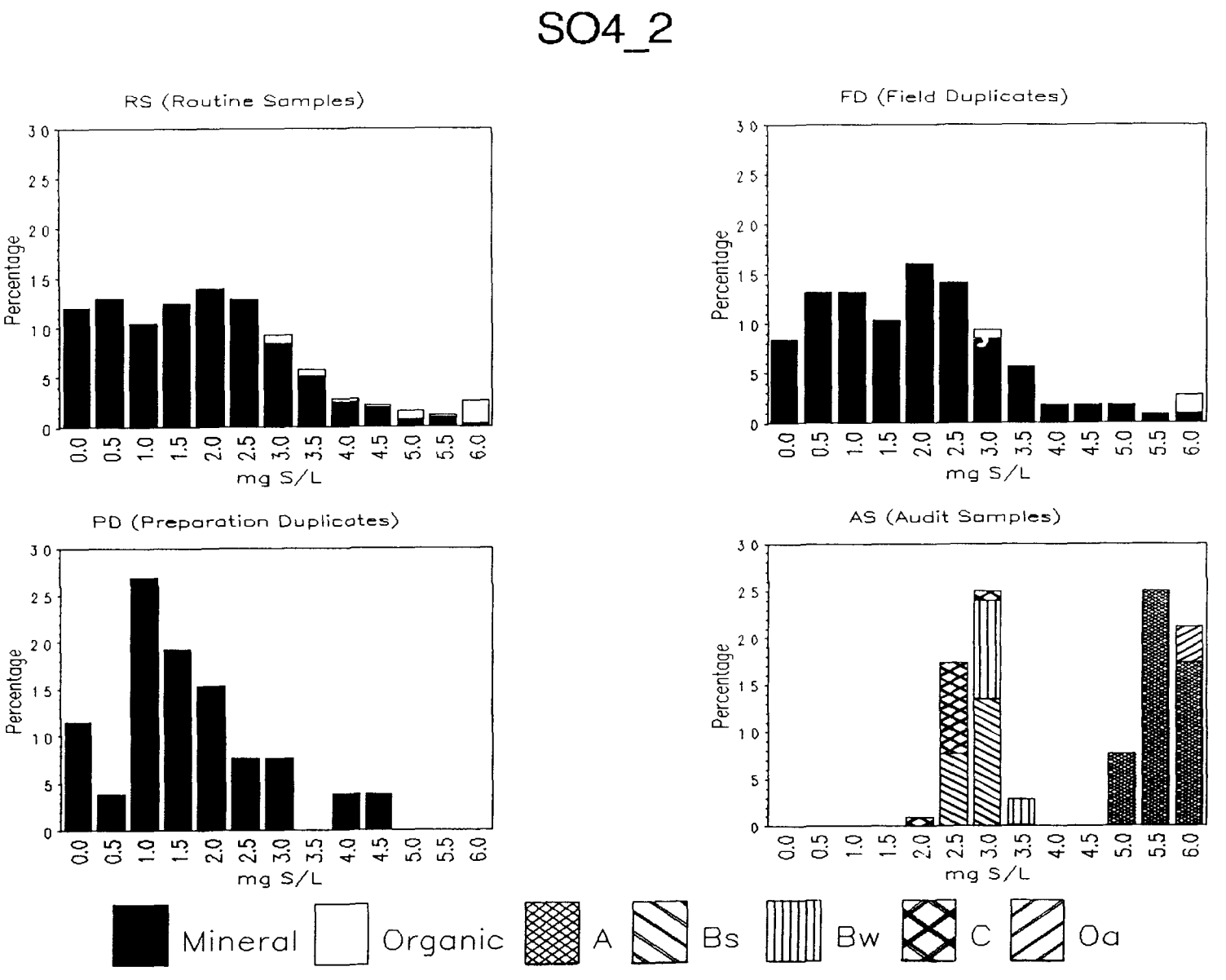

Figure G-44. Histogram of range and frequency distribution for the sulfate-two isotherm.

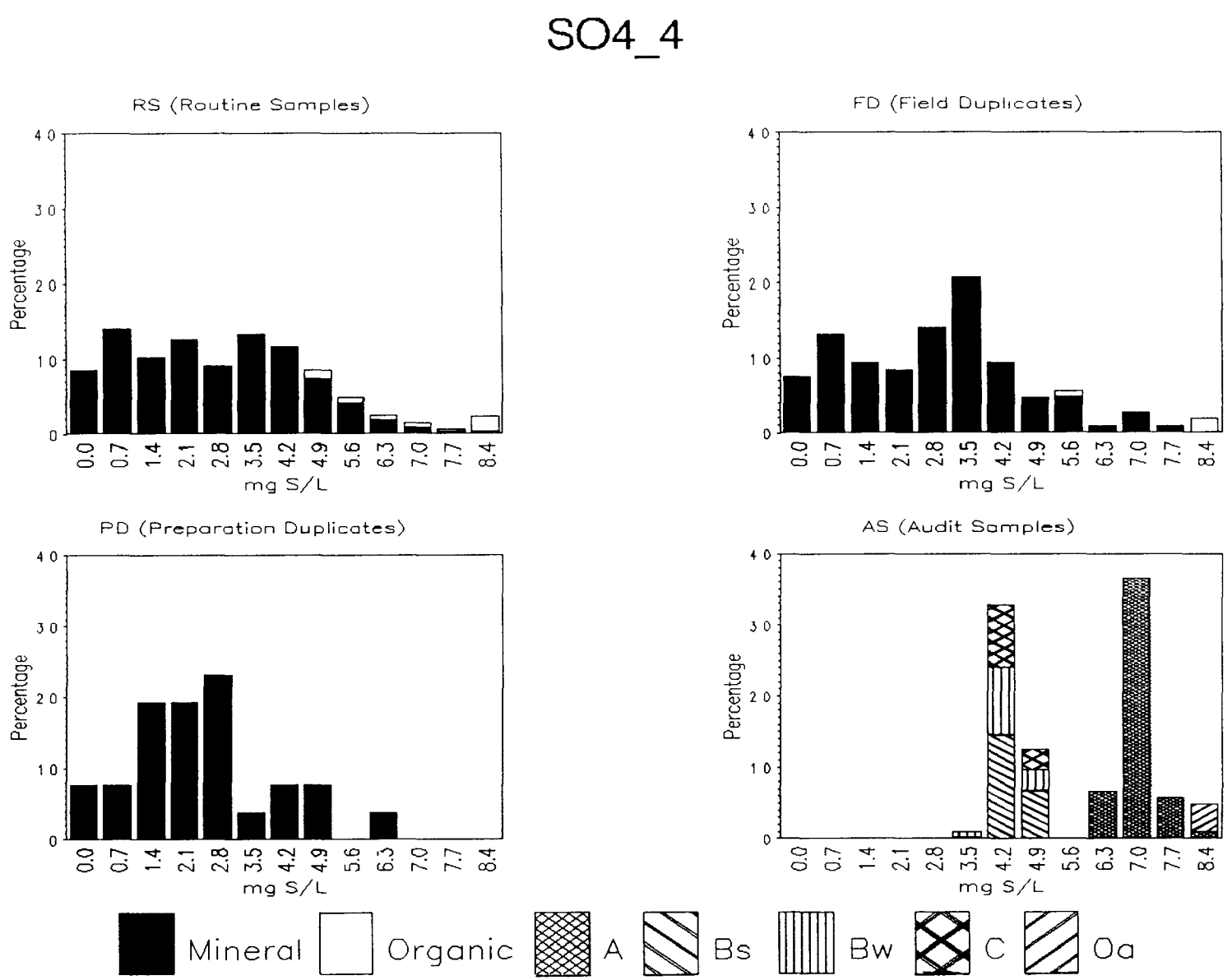

Figure G-45. Histogram of range and frequency distribution for the sulfate-four isotherm.

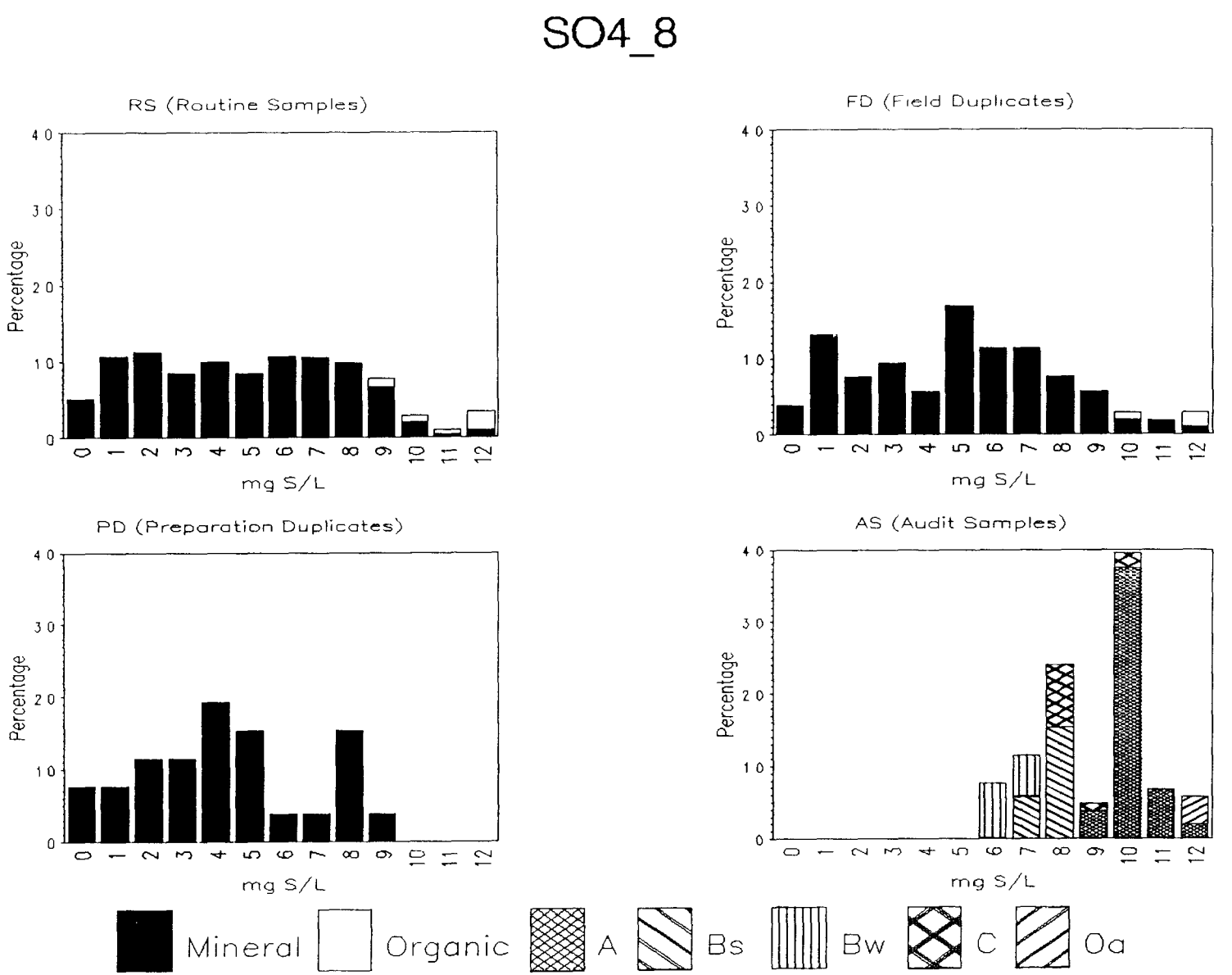

Figure G-46. Histogram of range and frequency distribution for the sulfate-eight isotherm.

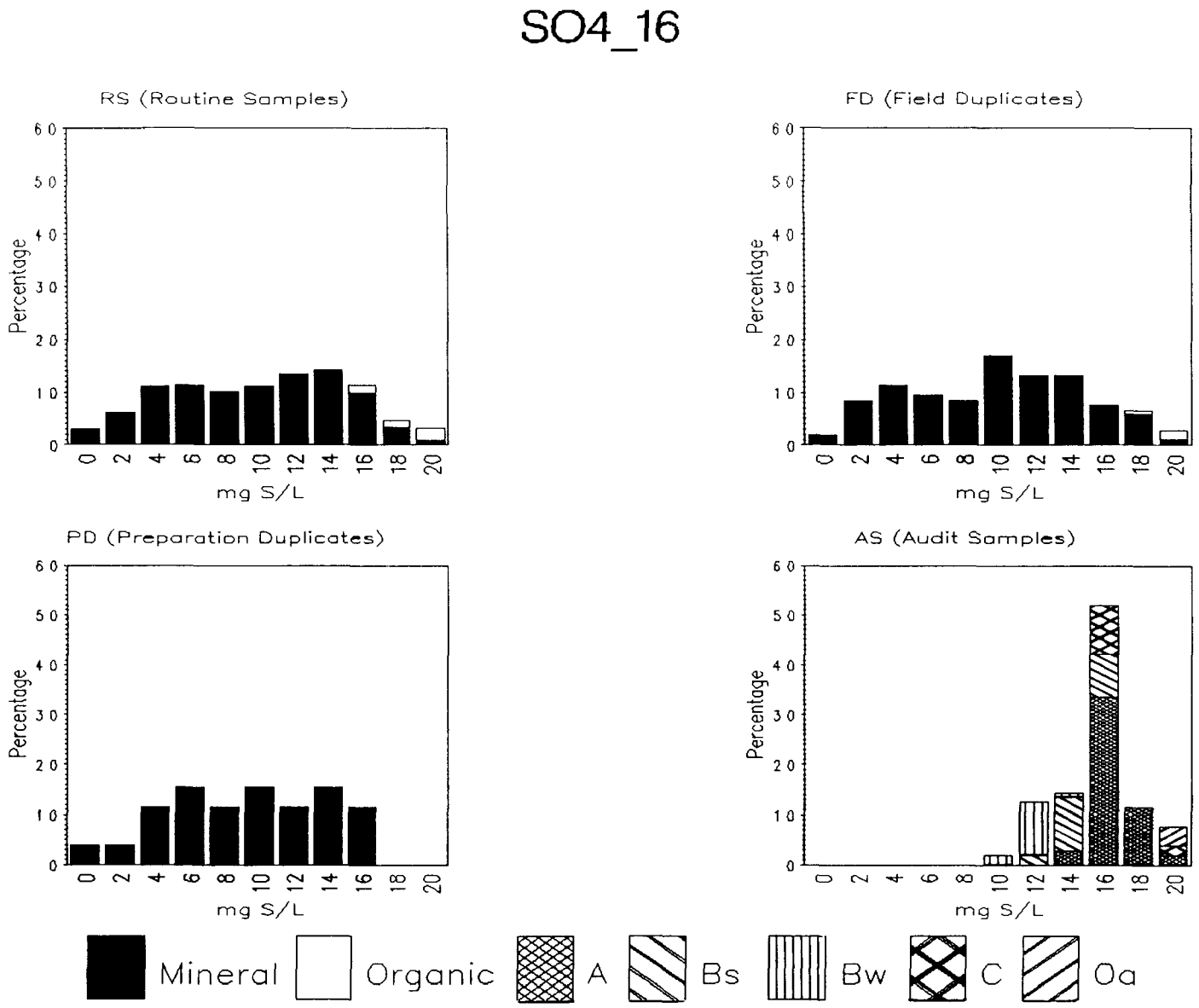

Figure G-47. Histogram of range and frequency distribution for the sulfate-16 isotherm.

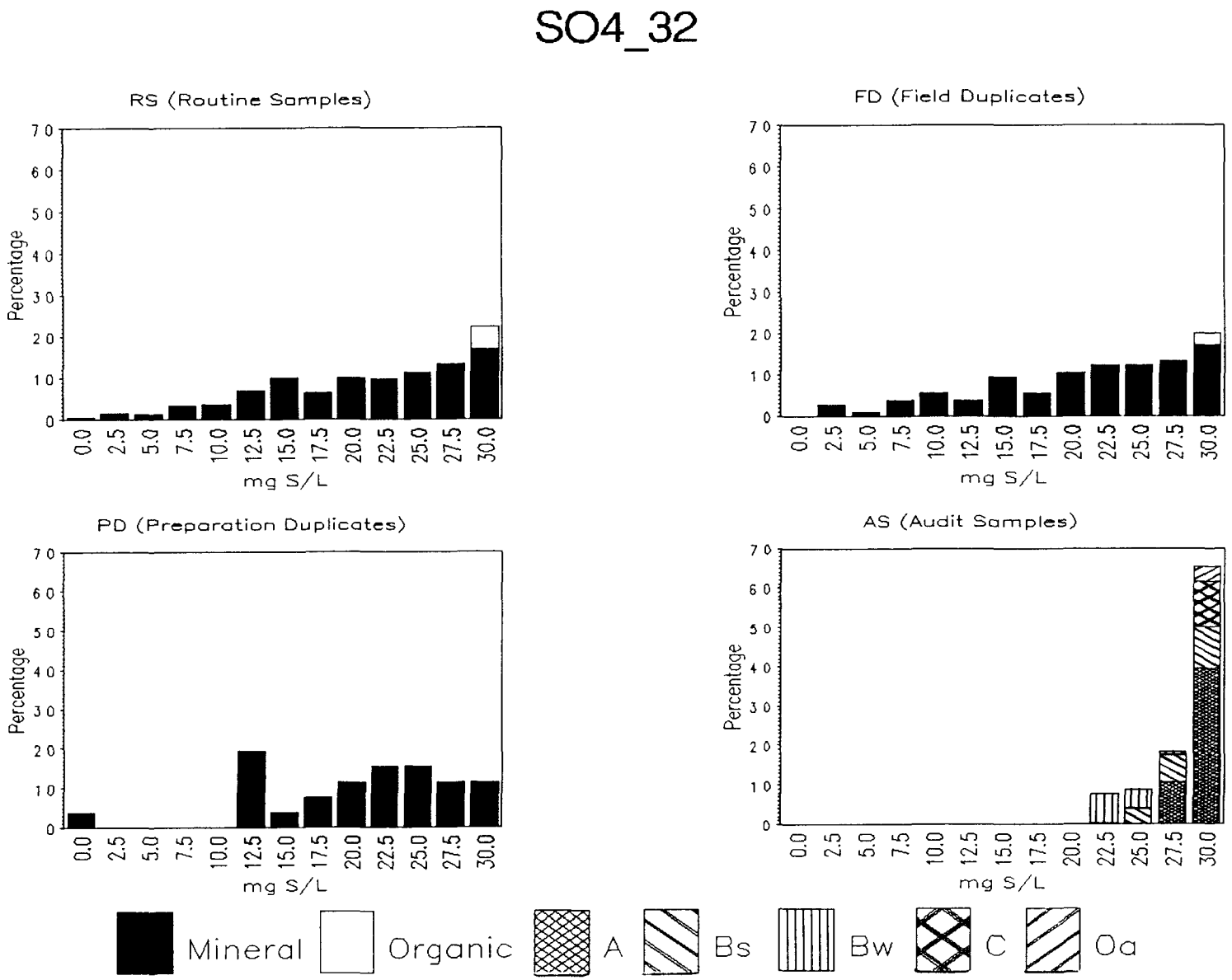

Figure G-48. Histogram of range and frequency distribution for the sulfate-32 isotherm.

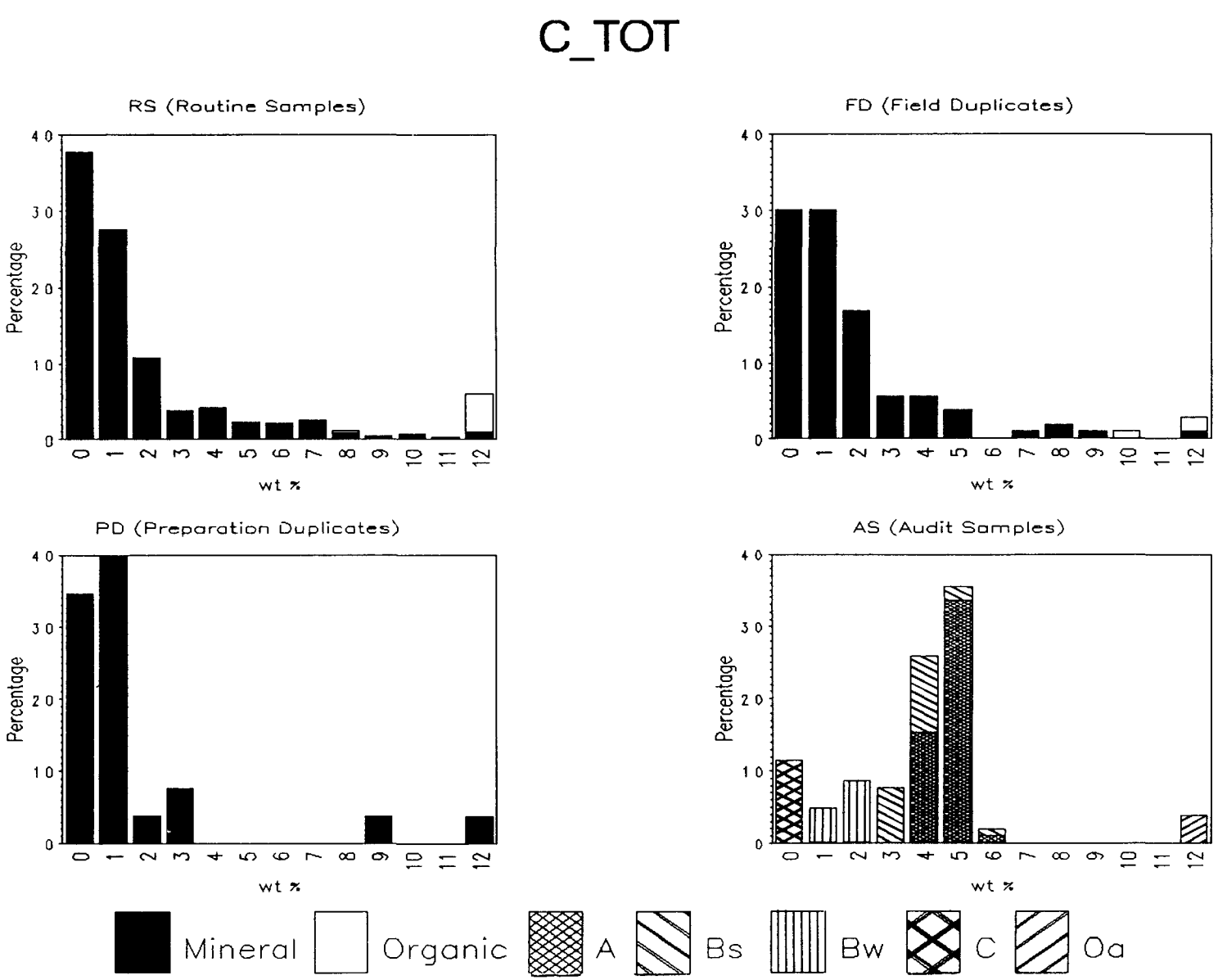

Figure G-49. Histogram of range and frequency distribution for total carbon.

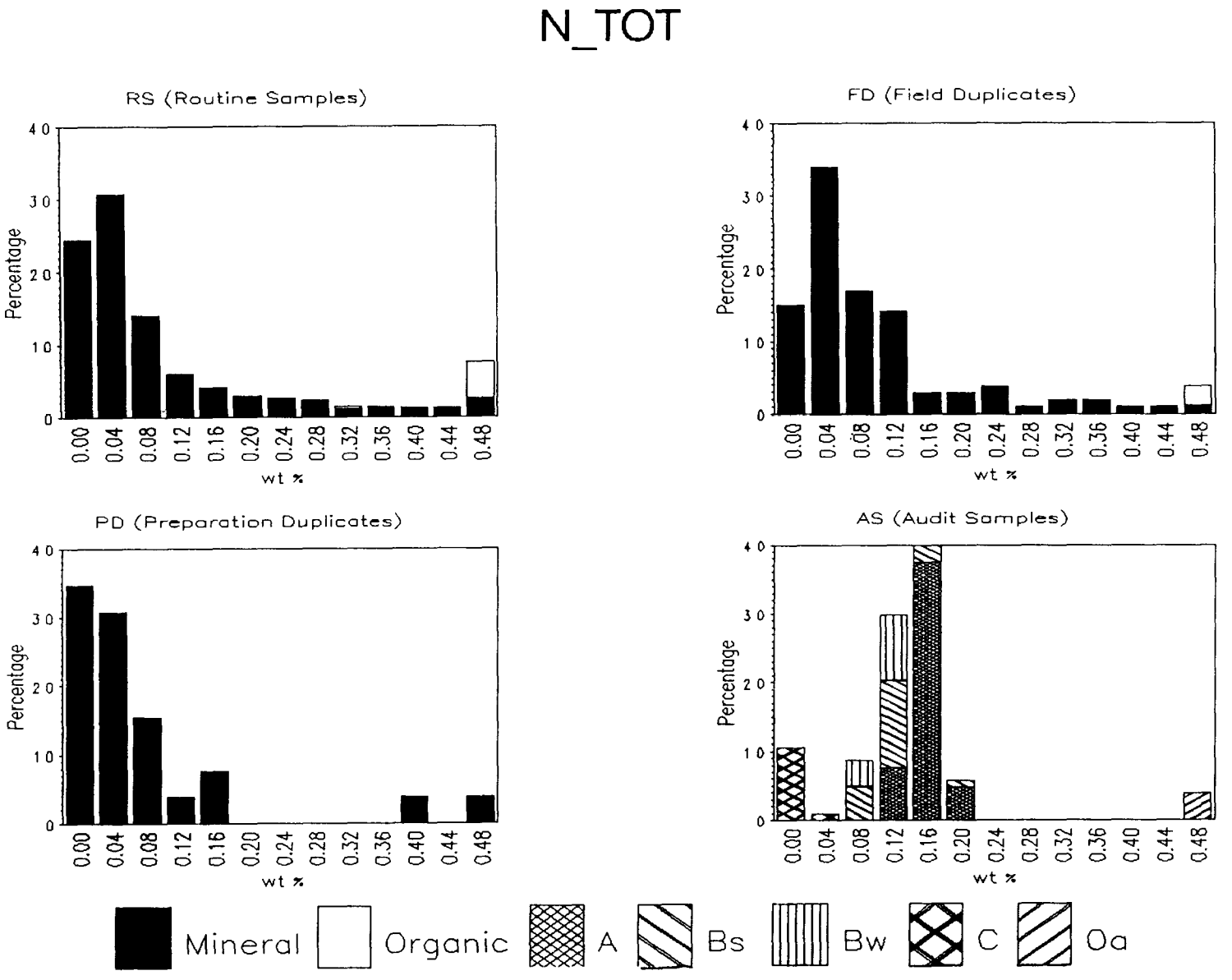

Figure G-50. Histogram of range and frequency distribution for total nitrogen.

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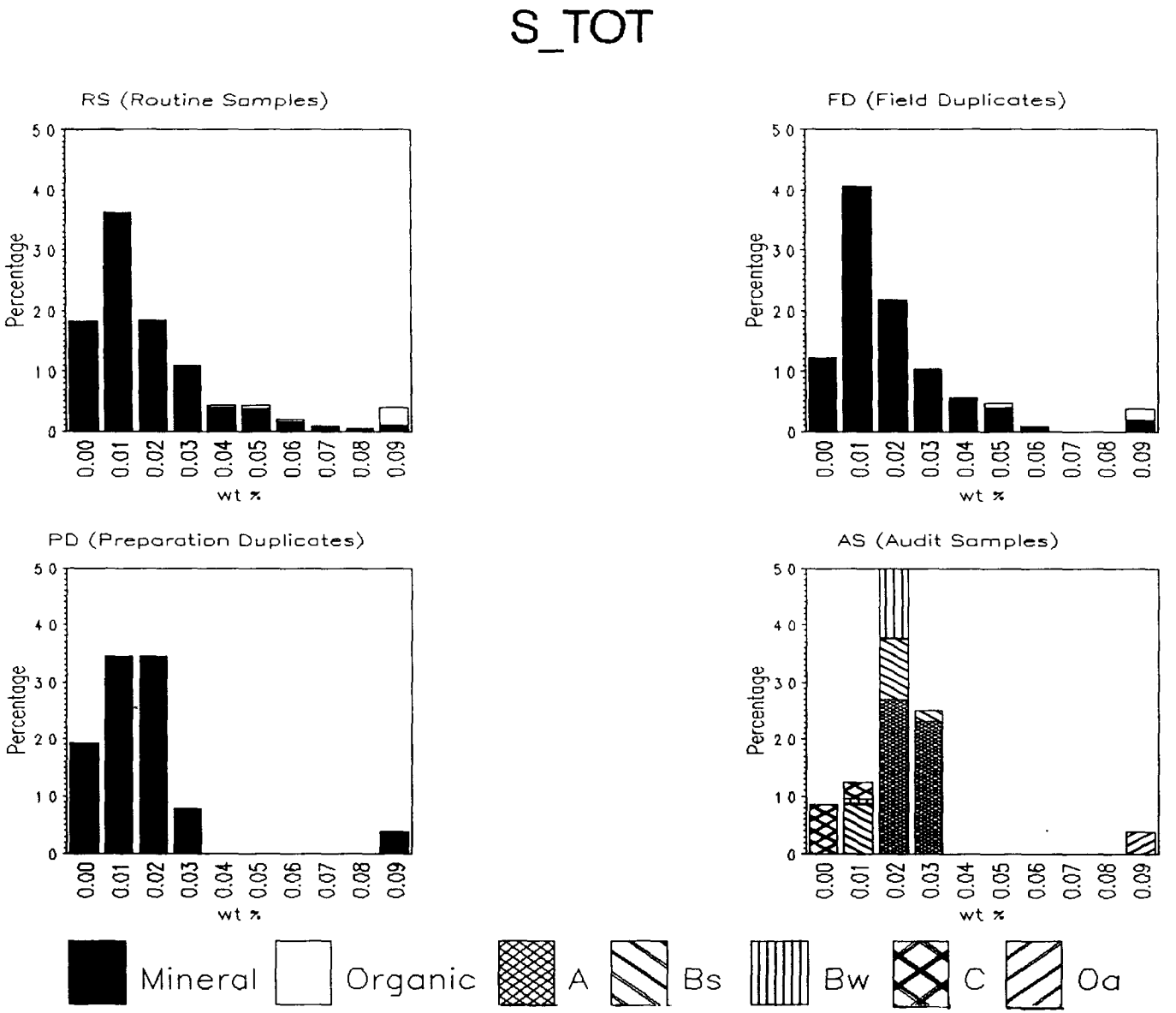

Figure G-51. Histogram of range and frequency distribution for total sulfur.