United States
Environmental Protection
Agency

National Exposure Research
Laboratory
Research Triangle Park, NC 27711

September 1997

Research and Development

EPA/600/R-95/112

EPA

An Intercomparison of Grinding Techniques Used for the Preparation of Lead-in-Paint Samples

An Intercomparison of Grinding Techniques Used for the Preparation of Lead-in-Paint Samples

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EPA Contract Nos. 68-D1-0009 and 68-D5-0040
RTI Project Nos. 91U-6660-014/91U-6970-255

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The information in this document has been funded wholly or in part by the United States Environmental Protection Agency (U.S. EPA) under EPA Contract Nos. 68-D1-0009 and 68-D5-0040 to Research Triangle Institute (RTI). It has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ACKNOWLEDGMENT

This document was prepared under the direction of Ms. Sharon L. Harper, National Exposure Research Laboratory (NERL), U.S. Environmental Protection Agency, Research Triangle Park, NC.

EXECUTIVE SUMMARY

The method used to grind a dried paint sample prior to acid digestion and measurement (by inductively coupled plasma (ICP) emission spectrometry or some other means), may affect the recovery of lead from the paint. In this study, five grinding techniques were compared. These were: (1) grinding with a glass rod at room temperature in a 50-mL centrifuge tube, (2) grinding with a glass rod at dry ice temperature in a 50-mL centrifuge tube, (3) grinding with a mortar and pestle, (4) grinding with a motorized blender, and (5) grinding with a cryogenic mill that operated at liquid nitrogen temperature. Preliminary testing of these five different procedures using 2" x 2" paint samples removed from real-world fiberboard walls and wooden cabinet boards showed no statistical differences in precision or bias of the measured lead content when a minimum of 5 minutes of grinding was performed with the manual methods. The mortar and pestle and the glass rod/room temperature centrifuge tube techniques were selected for more rigorous examination because of their relative ease of use. This second phase of the study involved optimizing the grinding time using 1" x 1" paint samples. Ten out of twelve tests demonstrated no statistical differences in the lead analysis results between the two methods or between grinding for 30 seconds versus 5 minutes. In the two statistically different tests, more lead was recovered from a sample ground with the mortar and pestle than from a sample ground with the glass rod in a centrifuge tube. When particle size analyses were conducted on the ground paint samples, it was observed that manual grinding times beyond 1½ minutes did not affect the particle size distribution. Since ten out of twelve tests had already shown that the lead recovered after 30 seconds of grinding was equivalent to 5 minutes grinding, no lead measurements were conducted on the paints ground for 1½ minutes. Based on the particle size results, it is recommended that a minimum of 1 to 1½ minutes of grinding be performed. The ground paint should have the consistency of coarsely ground coffee or cornmeal.

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SECTION 1.0
INTRODUCTION

1.1 PURPOSE OF THE STUDY

An early study to evaluate laboratory methods for digestion of paint, soil, and dust samples showed that the recovery of lead from paint chips was dependent, in part, upon the extent of the grinding of the paint.[1] Finely ground paint chips tended to yield higher lead levels than crushed paint chips. To test the hypothesis that the method of grinding may affect the ability to extract the lead from ground paint, five different procedures for grinding of paint samples were applied to a series of samples; processing was completed using acid/microwave digestion and analysis by inductively coupled plasma (ICP) emission spectrometry.[1] The grinding procedures tested included: (1) using a solid glass rod to crush and grind the paint in a centrifuge tube, (2) using a solid glass rod to crush and grind the paint in a plastic centrifuge tube while the centrifuge tube was immersed in dry ice, (3) grinding the paint with a glass mortar and pestle, (4) grinding the paint in a Bel Art Products Micro Mill,[2] and (5) grinding the paint in a Spex freezer mill.[3]

1.2 LIMITATIONS TO THE STUDY

Paint samples vary greatly in both physical and chemical composition. Paint sample thickness may vary from a fraction of a millimeter to several millimeters. Paint samples may be brittle or rubbery; outer layers of multilayered paint are often found to be latex, which is rubbery and difficult to grind. Due to resource limitations, only a few different samples of paint were included in this evaluation of grinding techniques. The conclusions reached with these samples may not apply to all paint samples.

1.3 STUDY APPROACH

Using a hot-air gun, twenty-five 4-in^2 samples of paint were removed from sections of both wooden cabinet doors and fiberboard walls. A group of five paint

samples of each type (doors and walls) was randomly assigned to each of the five grinding procedures (Section 1.1). After each sample was ground, determination of the concentration of lead was performed. The concentrations of lead for samples prepared using each of the grinding techniques were statistically evaluated to determine any differences in bias and precision that could be attributed to the grinding techniques. Particle analysis was performed on a limited number of the ground samples.

Preliminary testing on all five methods with 4-in^2 paint samples showed no statistical differences in precision or bias of the measured lead content when a minimum of 5 minutes of grinding was performed with the manual methods. Two techniques (the glass mortar and pestle method and the glass rod/plastic centrifuge tube at room temperature method) were selected for a second phase of testing to optimize grinding times because of their relative ease of use. This time 1-in^2 samples were collected to match the HUD guidelines.[4] The lead concentrations were statistically evaluated for differences in bias and precision for 30-second versus 5-minute grinding times. Particle size analyses were also conducted on the 30-second verus 5-minute ground samples. Since the histograms of the particle size distributions were different for the 30-seconds versus 5-minute samples, several more 4-in^2 and 1-in^2 samples were selected for grinding 1 - 1½ minutes for particle size analyses.

SECTION 2.0
EXPERIMENTAL

2.1 PHASE I: DESCRIPTION OF GRINDING TECHNIQUES AND ANALYSIS

2.1.1 Removal of Paint Samples

Two wooden cabinet doors and two sections of fiberboard wall taken from inside two different single family houses were selected as sources of paint samples for this test. The dwellings, which were built in the 1940's, were also used for an earlier pilot study for in-situ testing of lead in paint.[5] Originally ten inch (10" by 10") squares were to be drawn on each door and wall section, and each square divided into twenty-five 2" x 2" squares. However, it was necessary to modify the 10" x 10" square approach to accommodate the dimensions of the wall and board sections (See Appendix A) to obtain the 4-in^2 samples. The 4-in^2 sections were numbered and circumscribed with a scalpel. The paint was then removed using a spatula and a hot-air gun. The hot air gun [Ungar 6970HD Heavy Duty Gun] was set to the lower temperature (700°F) and an edge of the section heated just enough to soften the paint. The softened edge was lifted with a spatula and then more of the paint was heated. This process was continued until the entire section of paint was removed. Each sample was placed in a labeled plastic cup. The paint samples were then weighed. Five groups of five samples from each door and each wall section were then randomly assigned to the five preparation methods described in the following sections. Thus each grinding method was used for five paint samples from each of two cabinet doors and five paint samples from each of two wall sections.

2.1.2 Glass Rod/Room Temperature Grinding

The paint sample was placed in a labeled 50-mL plastic centrifuge tube. Using a tapered glass rod, the paint was broken into small pieces and then ground for approximately 5 minutes, using the glass rod like a pestle, until a fine powder (similar to coarsely ground coffee or corn meal) was obtained. After grinding, the tube containing the ground sample was capped for storage.

2.1.3 Glass Rod/Cryogenic Grinding

The paint sample was placed in a labeled 50-mL centrifuge tube and capped. The tube was immersed 1/3 to ½ way in a styrofoam cooler containing crushed dry ice (solid CO_2). The paint was chilled for 5-10 minutes, and then a tapered glass rod was used to grind the paint in the tube while it remained immersed in the dry ice. The paint was ground for 5 minutes until a fine powder (similar to coarsely ground coffee or corn meal) was obtained. After grinding, the tube was capped and removed from the dry ice. The paint remained in the tube for storage.

2.1.4 Mortar and Pestle

The paint sample was placed in a clean, glass, 2-ounce mortar and ground with a glass pestle for 5 minutes until only fine powder (similar to coarsely ground coffee or corn meal) was obtained. The ground paint was transferred back into a labeled sample cup.

2.1.5 Motorized Mechanical Grinder

The paint sample was placed in a Bel Art Products Micro-Mill and ground for 30 seconds; this small electric grinder uses a rotating blade to perform the grinding.[2] The ground paint was transferred back into a labeled sample cup. The mill was thoroughly cleaned between samples using a brush and laboratory wipes.

2.1.6 Cryogenic Motorized Mechanical Grinder

The paint sample was placed in a sample tube with a steel impactor rod, the tube was capped, and the sample was placed in a cryogenic mechanical mill, the Spex Industries Model 6700 freezer mill.[3] The sample was chilled in liquid nitrogen for 5 minutes and ground for 15 seconds. The cold, brittle paint chips were ground as the steel impactor rod rapidly moved electromagnetically back and forth along the length of the plastic sample tube. After allowing a 20-minute warm-up time, the sample was transferred back into a labeled sample cup.

2.1.7 Digestion and Analysis

A 0.1 g aliquot of each ground paint sample was weighed into a 50-mL, plastic centrifuge tube and digested by the Environmental Protection Agency (EPA) National Exposure Research Laboratory's (NERL's) microwave method prior to analysis by ICP emission spectrometry.[6]

2.1.8 Particle Size Analysis

One sample representing each material and each method of grinding described in Sections 2.1.2 through 2.1.6 was subjected to analysis by polarized light microscopy (PLM) to determine the average, mode, range and frequency of particle sizes. In preparation for this analysis, a random pinch of each paint was deposited in deionized water on a slide, and a cover slip was placed on top. The Olympus BHA microscope used for the particle analysis was calibrated using a Walton-Beckett graticule with a stage micrometer. The particle size statistics were performed using a Microsoft Excel spreadsheet.

2.1.9 Residue Analysis

Wall and board paint samples were selected for an analysis of the residue remaining following the microwave digestion. Three aliquots were selected from samples which had been ground using the glass rod/room temperature technique. The samples were digested, decanted and the remaining residue redigested prior to analysis. The samples which had been ground using the glass rod/room temperature technique were then reground using a mortar and pestle, and three aliquots were removed for normal analysis followed by residue analysis.

A particle size analysis was conducted on the original samples ground with the glass rod/room temperature technique and those that were reground using a mortar and pestle.

2.2 PHASE II: OPTIMIZATION OF MORTAR AND PESTLE AND GLASS ROD/ROOM TEMPERATURE PROCEDURES

2.2.1 Sample Selection

The Phase II test samples consisted of 1" x 1" pieces of paint removed from one cabinet door and one wall board previously used in Phase I. Additionally, to provide information on behavior of an exterior paint with grinding, an exterior board from a different site dating from the 1950's was used as a third substrate. The 1" x 1" sections were selected for Phase II to more closely match the HUD guidelines for removal of lead paint verification samples.[4] Ten 1" x 1" contiguous areas were marked on each of the three substrates, numbered, and circumscribed with a scalpel, and the paint was removed using a spatula and the heat-gun. The paint samples were weighed and placed in labeled 50-mL plastic centrifuge tubes.

2.2.2 Preparation and Analysis

Five samples from each substrate were assigned to either the mortar and pestle method or the glass rod/room temperature method. Each 1" x 1" sample was ground for 30 seconds, a 0.1 g aliquot was removed, and the remaining sample was ground for 4 ½ more minutes. A second 0.1 g aliquot was removed from the sample that had been ground for a total of 5 minutes. The 0.1 g aliquots were digested by the microwave method and analyzed by ICP emission spectrometry.[6]

2.2.3 Particle Size Analysis

In Phase II, particle size analyses were conducted on the 30-second and 5-minute ground samples. Since the average particle size was less for a sample which was ground for 5-minutes versus 30-seconds, it was decided to determine the optimum time for obtaining a fine powder of paint similar to the 5-minute samples. Therefore additional 4-in^2 and 1-in^2 samples were collected from the three substrates and ground for 1½ minutes. These samples were analyzed only for particle size distributions, since earlier results demonstrated for 10 out of 12 samples that there was no statistical differences in the amount of lead observed in samples ground for 30-seconds versus 5-minutes.

SECTION 3.0
RESULTS

3.1 PAINT DESCRIPTION

The paint from the cabinet doors consisted of three layers: white over bright green over beige. The top layer of white paint was somewhat rubbery compared to the brittleness of the green and beige paints, and it is presumed that the white paint was a latex paint while the other two layers were oil-based paints.

The paint from the fiberboard consisted of three layers: white over green over beige. Again, the white paint was somewhat rubbery compared to the brittleness of the green and beige paints. It was difficult to remove the paint from the fiberboard and a thin layer of fibrous fiberboard material remained on the back of the paint sections. A spatula was used to scrape and remove this material prior to grinding.

The paint on the exterior board consisted of two layers: beige over green. The beige top layer was somewhat rubbery compared to the brittleness of the green.

3.2 SAMPLE WEIGHT

The paint samples were weighed prior to grinding and removing the 0.1 g aliquots for digestion. The 4-in^2 samples of Phase I weighed an average of 2.3 g for the wall sections and 1.2 g for the cabinet door sections (see Table 1). The relative standard deviations (RSD) for $n = 25$ per substrate were 7.6% and 13.6% for the two wall sections and 9.7% and 14.3% for the two door sections.

The 1-in^2 samples of Phase II (see Table 2) weighed an average of 0.6 g for the wall sections, 0.3 g for the cabinet door sections, and 0.5 g for the exterior board sections. The RSD for $n = 10$ per set ranged from 3.9% to 8.0%.

TABLE 1.

SUMMARY OF 4-in^2 PAINT SAMPLE WEIGHTS - PHASE I

	Wall Section 6	Wall Section 7	Cabinet Door 5	Cabinet Door 8
Avg. wt (g)	2.46	2.23	1.31	0.99
RSD (%)	7.57	13.6	9.67	14.3
n =	25	25	25	25

TABLE 2.

SUMMARY OF 1-in^2 PAINT SAMPLE WEIGHTS - PHASE II

	Wall Section 7	Cabinet Door 5	Board X
Avg. wt (g)	0.59	0.31	0.52
RSD (%)	3.89	7.99	4.33
n =	10	10	10

3.3 QUALITY ASSURANCE

The data quality was assessed three ways, including the analysis of National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs), the analysis of duplicate samples, and the analysis of blanks. The average recovery for the NIST 1579 SRM (certified value 11.87% Pb) was 98% and ranged from 92.7% to 101.9% for the six analyses.

One sample for each method on each substrate was selected for duplicate extraction and analysis. For twenty duplicate pairs, the mean difference in lead concentration was 0.175% with a range of 0.0% to 0.56% (Table 3). The mean concentration measured in these duplicates was 2.56% lead.

Six blank extractions were conducted. No lead concentration above the limit of detection (LOD) of 0.005% lead was observed in the blanks.

TABLE 3.

DUPLICATE ANALYSES

Grinding Procedure	Sample ID	% Pb Initial	% Pb Duplicate	Difference in % Pb	Relative % Difference
Glass Rod,	Wall 6-4	1.59	1.62	0.03	1.89
Room	Wall 7-5	1.39	1.02	0.37	36.3
Temperature	Board 5-5	3.31	3.45	0.14	4.23
------	Board 8-1	4.41	4.97	0.56	12.7
$\bar{x}$ ± Std. Dev.				0.28 ± 0.24	13.8 ± 15.7
Glass Rod,	Wall 6-24	1.48	1.25	0.23	18.4
Dry Ice	Wall 7-25	1.49	1.49	0.00	0.0
-------	Board 5-25	3.02	2.83	0.19	6.71
-------	Board 8-24	4.33	4.21	0.12	2.85
$\bar{x}$ ± Std. Dev.				0.14 ± 0.10	6.99 ± 8.09
Mortar & Pestle	Wall 6-7	1.87	1.81	0.06	3.31
-------	Wall 7-8	0.708	0.653	0.055	8.42
-------	Board 5-7	2.98	2.57	0.41	15.9
-------	Board 8-8	4.49	4.36	0.13	2.98
$\bar{x}$ ± Std. Dev.				0.16 ± 0.17	7.67 ± 6.06
Bell Art Mill	Wall 6-11	1.66	1.76	0.10	6.02
-------	Wall 7-12	1.06	1.34	0.28	26.4
-------	Board 5-14	3.39	3.63	0.24	7.08
-------	Board 8-12	3.46	3.52	0.06	1.73
$\bar{x}$ ± Std. Dev.				0.17 ± 0.11	10.3 ± 10.9
Spex Freezer Mill	Wall 6-18	1.64	1.74	0.10	6.10
-------	Wall 7-16	1.24	1.30	0.06	4.84
-------	Board 5-13	3.63	3.81	0.18	4.96
-------	Board 8-20	4.59	4.41	0.18	4.08
$\bar{x}$ ± Std. Dev.				0.13 ± 0.06	4.99 ± 0.83

3.4 LEAD RECOVERIES

The lead concentrations determined from paint samples ground using the five techniques in Phase I are summarized in Table 4. Statistical interpretations included: (1) an examination of the structure of error, which concluded that using the RSD rather than the standard deviation (SD) was valid: (2) an F-test on ln-transformed computed variances: and (3) a Youden test for ranking.[7] These statistical interpretations (Appendix D) showed no differences for the Phase I study in terms of bias and precision between the five grinding techniques.

An analysis of the residue remaining following the microwave digestion was conducted for 12 samples. In all cases, the lead content of the residue was less than 2% of the lead content of the initial extract (Table 5).

Table 6 is a summary of Phase II results, comparing the two grinding techniques and optimizing grinding times. Student's t-test was utilized to determine if there was a difference in lead concentration dependent upon (1) grinding time (30 seconds versus 5 minutes) or (2) grinding technique (mortar and pestle versus glass rod/room temperature). Statistically significant differences at the 95% confidence limit were observed for two out of 12 tests. A statistically significant difference was observed for the 30-second grinding of Board X, with 4.9% lead in the paint ground using the glass rod/room temperature technique and 7.1% lead in the paint ground using the mortar and pestle technique. A statistically significant difference was also observed for the 5-minute grinding of Cabinet Door 5, with 3.5% lead in the paint ground using the glass rod/room temperature technique and 4.6% lead in the paint ground using the mortar and pestle technique. No other significant differences were noted; i.e., a grinding time of 5 minutes versus 30 seconds did not statistically alter the concentration of lead in the paint for any of the three substrates tested.

3.5 PARTICLE SIZE

Paint samples representing each substrate type and grinding method and grinding method combination were analyzed by PLM at 400x. The particle sizes, shown in Table 7 and presented as histograms in Appendix E, ranged from 0.9 to 634 microns (μm). The

mode (most frequent value) ranged from < 1.2 μm to 24 μm. In general, the glass rod/centrifuge tube at room temperature and the 30-second mortar and pestle techniques yielded larger average particle sizes than the methods using either longer grinding times or mechanical grinders (see histograms in Appendix E). Comparison of the particle size distributions resulting from both the 2" x 2" and 1" x 1" starting materials from Wall Section No. 7 and Cabinet Door No. 5 showed similar mean sized particles.

TABLE 4.

SUMMARY OF LEAD CONCENTRATIONS DETERMINED USING VARIOUS PAINT GRINDING TECHNIQUES - PHASE I

Grinding Technique	Wall Section 6 (n = 5)		Wall Section 7 (n = 5)		Cabinet Door 5 (n = 5)		Cabinet Door 8 (n = 5)	
	% Pb	mg Pb/cm^2	% Pb	mg Pb/cm^2	% Pb	mg Pb/cm^2	% Pb	mg Pb/cm^2
A) Glass Rod								
$\bar{x}$	1.87	1.82	1.30	1.12	3.83	2.03	4.1	1.66
RSD (%)	16.1	21.9	17.8	21.9	18.9	20.5	11.4	15.2
B) Dry Ice/Glass Rod								
$\bar{x}$	1.72	1.65	1.28	1.13	3.58	1.81	4.02	1.54
RSD(%)	14.9	23.5	16.9	31.6	11.6	14.9	24.5	21.8
C) Mortar & Pestle								
$\bar{x}$	1.58	1.46	1.14	0.985	3.51	1.77	4.40	1.67
RSD(%)	17.5	20.3	30.4	42.5	23.7	34.0	11.1	31.2
D) Bel Art Mill								
$\bar{x}$	1.56	1.54	1.14	1.00	3.52	1.76	3.30	1.19
RSD(%)	13.2	19.8	29.3	42.0	10.0	11.5	11.4	16.0
E) Spex Freezer								
$\bar{x}$	1.50	1.42	1.18	1.07	3.74	1.92	4.94	1.87
RSD(%)	5.8	10.7	18.3	18.8	15.8	19.4	6.9	12.3

TABLE 5.
RESULTS OF ANALYSIS OF RESIDUE REMAINING FOLLOWING MICROWAVE DIGESTION

Substrate	Sample Aliquot No.	Grinding Technique	μg Pb Sample	μg Pb Residue	Residue as % of Total Pb
Wall Section 7	5a	Glass Rod/Centrifuge Tube[a]	829	14.3	1.8
Wall Section 7	5b	Glass Rod/Centrifuge Tube[a]	860	14.5	1.7
Wall Section 7	5c	Glass Rod/Centrifuge Tube[a]	854	10.8	1.3
Wall Section 7	5d	Mortar & Pestle[b]	917	11.1	1.2
Wall Section 7	5e	Mortar & Pestle[b]	1,020	18.6	1.8
Wall Section 7	5f	Mortar & Pestle[b]	935	10.8	1.2
Cabinet Doors	8a	Glass Rod/Centrifuge Tube[a]	6,040	10.8	1.8
Cabinet Doors	8b	Glass Rod/Centrifuge Tube[a]	4,390	82.3	1.9
Cabinet Doors	8c	Glass Rod/Centrifuge Tube[a]	3,890	70.0	1.8
Cabinet Doors	8d	Mortar & Pestle[b]	3,630	41.8	1.2
Cabinet Doors	8e	Mortar & Pestle[b]	4,110	46.0	1.1
Cabinet Doors	8f	Mortar & Pestle[b]	3,920	59.2	1.5

a. Samples were initially ground for 5 minutes using a glass rod/centrifuge tube method and three aliquots removed for analysis.
b. The samples that were initially ground for 5 minutes using a glass rod/centrifuge tube method were reground using a mortar and pestle for 3 minutes and three aliquots removed for analysis.

TABLE 6.

SUMMARY OF LEAD CONCENTRATIONS DETERMINED USING VARIOUS PAINT GRINDING TECHNIQUES - PHASE II
(n = 5)

	Wall Section 7	Cabinet Door 5	Board X
A) Glass Rod/Centrifuge Tube t = 30 seconds			
$\bar{x}$	1.12	3.68	4.86
RSD(%)	8.8	18.4	13.9
B) Glass Rod/Centrifuge Tube t = 5 minutes			
$\bar{x}$	1.01	3.53	5.18
RSD(%)	9.0	26.9	6.4
C) Mortar & Pestle t = 30 seconds			
$\bar{x}$	1.03	4.44	7.07
RSD(%)	12.2	16.5	21.1
D) Mortar & Pestle t = 5 minutes			
$\bar{x}$	1.06	4.61	6.13
RSD(%)	10.8	14.7	15.6

TABLE 7.

PARTICLE SIZE OF GROUND PAINT

Procedure	Wall Section 7				
	Particle Diameter		Mode	Range (μm)	Sample Size (in^2)
	$\overline{X}$, μm	s			
Glass Rod, Room Temperature, 5 min	40.9	32.4	24	2.4 - 204.0	4
Glass Rod reground by Mortar & Pestle, 5 min + 3 min	17.5	25.1	6	1.2 - 150.0	4
Glass Rod/Dry Ice, 5 min	23.9	22.6	12	1.2 - 120.0	4
Mortar & Pestle, 30 s	50.7	54.5	9.2	6.1 - 314.2	1
Mortar & Pestle, 1.5 min	15.8	36.0	<1.2	<1.2 - 186.0	1
Mortar & Pestle, 1.5 min	11.4	35.0	2.4	<1.2 - 324.0	4
Mortar & Pestle, 5 min	9.7	8.4	6	1.2 - 44.4	4
Bel Art Mill, 30 sec	13.5	8.8	6	1.2 - 42.0	4
Spex Freezer Mill, 15 sec	10.2	14.7	2.4	1.2 - 126.0	4

Note: Mode is the most frequent value in a data set.

TABLE 7. CONTINUED

PARTICLE SIZE OF GROUND PAINT

Procedure	Wooden Cabinet Door 5				
	Particle Diameter				
	$\overline{X}$ (μm)	s	Mode	Range (μm)	Sample Size (in^2)
Glass Rod, Room Temperature, 5 min	34.5	107.0	3.1	0.9 - 634.4	4
Glass Rod reground by Mortar & Pestle, 5 min + 3 min	21.5	34.1	1.5	1.5 - 201.3	4
Glass Rod/Dry Ice	17.4	29.0	3.1	1.5 - 152.5	4
Mortar & Pestle, 30 s	61.0	56.0	12.1	3.1 - 292	1
Mortar & Pestle, 1.5 min	11.7	32.3	1.2	1.2 - 198.0	1
Mortar & Pestle, 1.5 min	10.0	19.0	<1.2	<1.2 -120.0	4
Mortar & Pestle, 5 min	15.4	23.7	3.1	1.5 - 167.8	4
Bel Art Mill, 30 sec	14.8	26.8	3.1	1.5 - 183.0	4
Spex Freezer Mill, 15 sec	13.7	23.7	6.1	0.9 - 152.5	4

Note: Mode is the most frequent value in a data set.

TABLE 7. CONTINUED

PARTICLE SIZE OF GROUND PAINT

Procedure	Board X				
	Particle Diameter		Mode	Range (μm)	Sample Size (in^2)
	$\bar{X}$ (μm)	s			
Mortar & Pestle, 30 sec	51.9	50.4	24.4	6.1 - 375.2	1
Mortar & Pestle, 1.5 min	6.3	16.2	1.2	1.2 - 156.0	1
Mortar & Pestle, 5 min (Trial 1)	51.9	44.5	24.4	3.0 - 244	1
Mortar & Pestle, 5 min (Trial 2, new sample)	8.4	11.6	<1.2	<1.2 - 90.0	1

Note: Mode is the most frequent value in a set of data.

SECTION 4.0
DISCUSSION

4.1 METHOD OVERVIEW

The five paint grinding techniques vary in performance parameters. An overview of the grinding techniques is presented in Table 8.

4.2 GLASS ROD/ROOM TEMPERATURE GRINDING

This method is relatively easy to perform. No electrical power is required. There is minimal opportunity for contamination because the sample remains in the collection/storage tube and a clean glass rod is used for each sample. To obtain a fine, uniform powder from the paint, approximately 5 minutes of grinding time was necessary. The consistency of the final product depends on the characteristics of the dried paint, the grinding time, and the individual pressure applied. The latex paint in these samples could not be ground into a fine powder but ended up as small chips 1-2 mm in diameter with a rubber-like consistency. The fact that the latex paint material was not ground as finely as the oil-based paint material did not present a lead measurement problem because the latex usually contains little lead; that is, reduced extraction efficiency with the larger latex particles was not expected to significantly affect the overall lead analysis result. However, the larger latex particles did affect attempts at subsampling. It was difficult to remove a subsample that had a mixture of small oil-based particles and larger latex-based particles that was representative of the original paint sample. That is, representative subsampling was difficult to achieve. The wallboard paint chips (which had a backing similar to cardboard) also were difficult to grind. Also, the paint particles became electrostatically charged and accumulated at the top of the plastic centrifuge tube.

TABLE 8.

OVERVIEW OF FIVE PAINT GRINDING TECHNIQUES

Equipment/Material	Glass Rod/ Centrifuge Tube at Room Temp.	Glass Rod/ Centrifuge Tube in Dry Ice	Mortar and Pestle	Bel Art Products Mill	Spex Freezer Mill
Approximate Costs in 1997	~$1.00	~$1.00 (plus cost of dry ice)	$14.00 (glass)	$800 - $1000	~ $3,000 (plus cost of liquid nitrogen)
Time to process one sample (condition, grind, cleanup)	<5 min.	14 min.	<5 min.	15 min.	20 min.
Electricity requirements	No	No	No	Yes	Yes
Safety considerations	No	Dry Ice	No	No	Liquid Nitrogen
Skill level * least *** most	*	**	*	*	***
Opportunity for contamination	No	No	Minimal	Yes	Yes
Minimum sample size	~0.2 g	~0.2 g	~0.2 g	~0.5 g	~0.2 g
Maximum sample size	~8 g	~8 g	~8 g	~8 g	~4 g
Sample transfer from collection container?	No	No	Yes	Yes	Yes

4.3 GLASS ROD/CRYOGENIC GRINDING

This method was very similar to the room temperature grinding. One disadvantage to this method is the requirement of an ice bucket and dry ice. The centrifuge tube was immersed in the dry ice for up to 10 minutes prior to grinding. This increases the preparation time, unless an assembly line approach is taken; i.e. Samples No. 2 and No. 3 can be chilled while Sample No. 1 is being ground. Handling dry ice also requires the use of gloves and safety glasses. These samples were not as electrostatically charged as those ground at room temperature, but daily relative humidities were not recorded, and humidity could contribute to the electrostatic charges observed. Also, there was potential problem of condensation of water vapor into the cold samples.

4.4 MORTAR AND PESTLE

This classic method of grinding a sample is very effective. A freshly cleaned, acid-rinsed glass mortar and pestle were used for each sample. Because cleaning facilities may be limited in the field, several pre-cleaned mortars and pestles are recommended, increasing the equipment burden. The samples can be ground to a fine consistency in 1 - 1½ minutes.

The problems encountered in grinding dried latex paint and paint chips with wallboard backing were the same as those encountered with the glass rod methods. The biggest disadvantage of this method is that it can be very tiring when multiple samples are ground.

4.5 MOTORIZED MECHANICAL GRINDER

The Bel Art Products Mill is similar in design to a "coffee grinder." This mill was fast, requiring only 30 seconds of grinding time. An external power source (electricity) is required. The time required to properly clean the mill between samples can be as much as 5-10 minutes. The possibility for cross-contamination exists because all paint chips come into contact with the mill. The ground paint chips were similar in appearance to those from the previously discussed methods in that the latex

paint yielded visible small chips in conjunction with the fine powder of the other paint components.

4.6 CRYOGENIC MOTORIZED MECHANICAL GRINDER

The Spex freezer mill requires electricity and liquid nitrogen. The liquid nitrogen could be a problem for field use because the reservoir is fairly large (2L) and requires frequent replenishing. It would be necessary to train the operator in the proper handling of liquid nitrogen and the use of personal protective equipment.

The paint was placed in a plastic cylinder with a steel impactor rod and then chilled for 5 minutes. The actual milling time was only 15 seconds, but the sample cylinder had to be removed and allowed to warm to a reasonable temperature before handling. This warm up can take up to 20 minutes. If several cylinders and steel impactor rods were available, this technique could be adapted to an assembly line approach. The opportunity for cross-contamination occurs if the sample cylinders and steel rods are not properly cleaned. The paint was ground to a fine powder but, consistent with the other methods, the latex chips appeared larger in size than the other paint constituents.

4.7 LEAD RECOVERIES

As stated, statistical analyses in Phase I of the study showed no differences in the methods in terms of bias and precision. In Phase II, statistically significant differences were observed for only two out of twelve tests. It is noted in these evaluations that the RSDs are large, which would impact the statistical analyses. These large RSDs reflect, at least in part, the variability in lead in paint from location to location as has been observed in another study.[5] Differences in the samples are reflected in the variations in the weights of the samples of the same area as presented in Tables 1 and 2. The conclusion about the equivalency of the five methods is further supported, however, by two other pieces of evidence. First, the average difference between the pairs of duplicate samples for the five methods (Table 3), with the exception of the glass rod/room temperature technique, are very similar,

indicating equivalency of sample homogeneity; they are statistically equivalent at the 95% confidence level. Second, the residue values for the two methods expected to yield the lowest recoveries (glass rod/room temperature and mortar and pestle) are less than 2%, indicating greater than 95% recovery.

4.8 PARTICLE SIZE HISTOGRAMS

The physical characteristics of the scraped paint sample appear to affect the particle size distribution of the ground sample. The paint removed from the fiberboard had an outer latex layer that made the sample more "rubbery" and difficult to grind; the paint from the boards was more brittle and easier to grind.

Histograms showing the particle size distributions for the different grinding techniques are presented in Appendix E. Immersing the centrifuge tube in dry ice before grinding with a glass rod reduced the mean particle diameter to approximately half that obtained by grinding with a glass rod at room temperature for both the wall and board paint samples. However, there was a more notable effect on the shape of the histogram for the wall sample than the board sample, with a shift toward smaller particle sizes. Regrinding the samples by mortar and pestle had a more dramatic effect on the histogram of the wall sample, resulting in a histogram similar to that for the board sample that was reground by mortar and pestle.

The histograms from the experiments to determine the effect of grinding time showed significant differences between 30 seconds and 1.5 minutes of grinding with the mortar and pestle for both wall and board paint samples. The longer grinding time reduced the mean particle diameter from 61.6 μm (for 30 seconds) to 11.7 μm for Cabinet Door 5 and from 51.9 μm (for 30 seconds) to 6.3 μm for Board X. However, increasing the grinding time from 1.5 minutes to 5 minutes did not significantly affect the histograms for either sample.

The Bel Art Mill and the Spex freezer mill ground both samples (Cabinet Door 5 and Wall Section 7) to approximately the same mean particle diameter (10 - 15 μm). However, the particle size range was larger for the board samples than for the wall samples ground by each mill.

SECTION 5.0
CONCLUSIONS

For Phase I of this study, statistical interpretations, including an F-test on ln-transformed computed variances and a Youden test for ranking, showed no differences in bias and precision for lead measurements made on samples prepared by five different grinding techniques when a minimum of five minutes of manual grinding was used.

For Phase II, two techniques (the mortar and pestle method and the glass rod/centrifuge tube method at room temperature), were selected to determine optimum grinding times for three different substrates. Tests demonstrated at the 95% confidence level using Student's t-test, that a minimum of 30 seconds of grinding yielded lead concentrations equivalent to that obtained after 5 minutes of grinding for 10 out of 12 comparisons. There was a difference in grinding techniques as observed in the amount of lead determined after a 30-second grinding, with 7.1% lead measured in the sample ground using a mortar and pestle and 4.9% lead measured in the sample ground by glass rod/centrifuge tube. A similar disparity was noted after a 5-minute grinding, with 4.6% lead measured in the sample ground by mortar and pestle and 3.5% lead measured in the sample ground by glass rod/centrifuge tube. This second finding is in conflict with the statistical interpretations of the Phase I study.

The mortar and pestle and the glass rod/centrifuge tube (at room temperature) techniques can be easily adapted to field use. Both techniques require no electricity and minimal support materials. It is essential that the mortar and pestle and the glass rod be thoroughly cleaned between paint samples to prevent cross-contamination. The "optimum" grinding time will depend upon the paint and the individual performing the grinding (i.e., how much force is exerted on the paint). Histograms showed that the particle size distributions did not change significantly after 1½ minutes of

grinding. Therefore, it is recommended that the paint be manually ground for a minimum of 1 to 1½ minutes. The ground paint should have the consistency of coarsely ground coffee or cornmeal.

SECTION 6.0

REFERENCES

1. Williams, E. E., D. A. Binstock, J. A. O'Rourke, P. M. Grohse, and W. F. Gutknecht. Evaluation of Hotplate- and Microwave-Based Methods for Extracting Lead in Paint, Dust, and Soil with Measurement by Atomic Absorption Spectrometry and Inductively Coupled Plasma Emission Spectrometry. EPA 600/R-94/147, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1995, 98 pp.

2. Bel Art Micro-Mill. Bel-Art Products, 6 Industrial Rd., Pequannock, NJ, 07440, (201) 694-5000.

3. Spex Freezer Mill. Spex Industries, Inc., 3880 Park Avenue, Edison, NJ, 08820, (908) 549-7144.

4. HUD Guidelines for Testing, Abatement, Clean-Up and Disposal of Lead Based Paint in Housing. The National Institute of Building Sciences, The Department of Housing and Urban Development, 1989.

5. Gutknecht, W.F., L.L. Hodson, K.K. Luk, D.A. Binstock, C.C. Van Hise, and A. R. Turner. Pilot Field Study for the Assessment of Techniques Used for Field Measurement of Lead in Paint. EPA 600/R-97/057, U.S. Environmental Protection Agency, Research Triangle Park, NC., September, 1997.

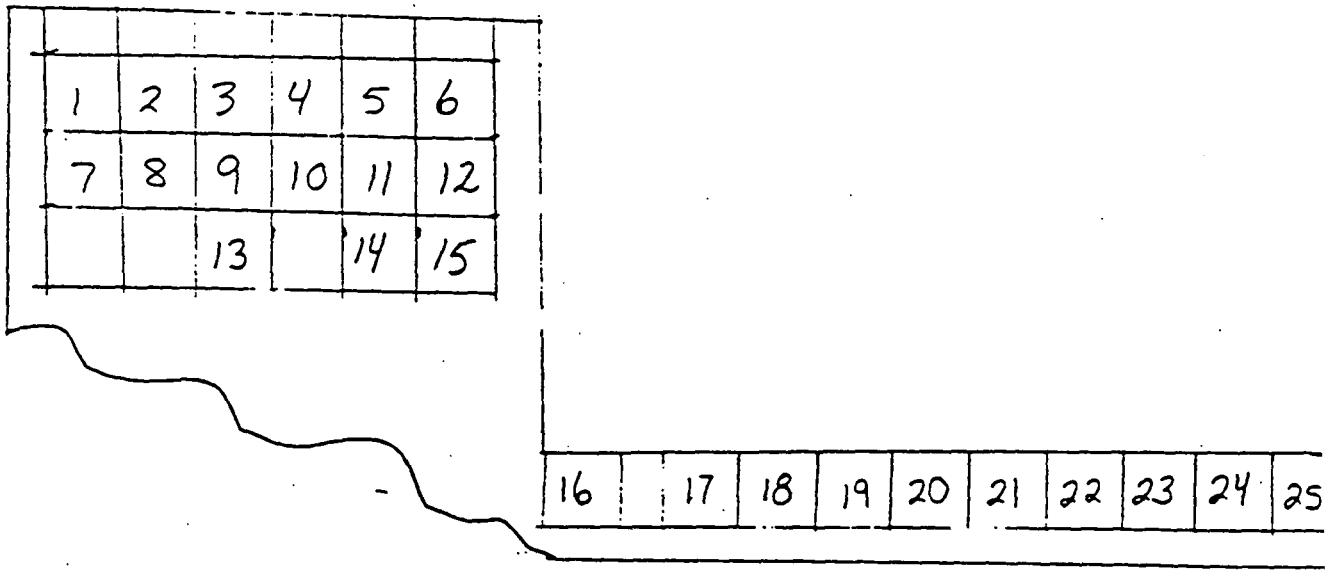
6. Binstock, D. A., D. L. Hardison, P. M. Grohse, and W. F. Gutknecht. Standard Operating Procedures for Lead in Paint by Hotplate- or Microwave-based Acid Digestions and Atomic Absorption or Inductively Coupled Plasma Emission Spectrometry. EPA 600/R-91/213. U.S. Environmental Protection Agency, Research Triangle Park, NC, 1991, 19 pp. *Available from NTIS, Springfield, VA; NTIS PB92-114172.

7. Youden Test for Ranking. Taylor, J.K., Statistical Techniques for Data Analysis, pp. 92-94, Lewis Publishers, 1990.

APPENDIX A

Diagrams of Cabinet Boards and Wall Sections

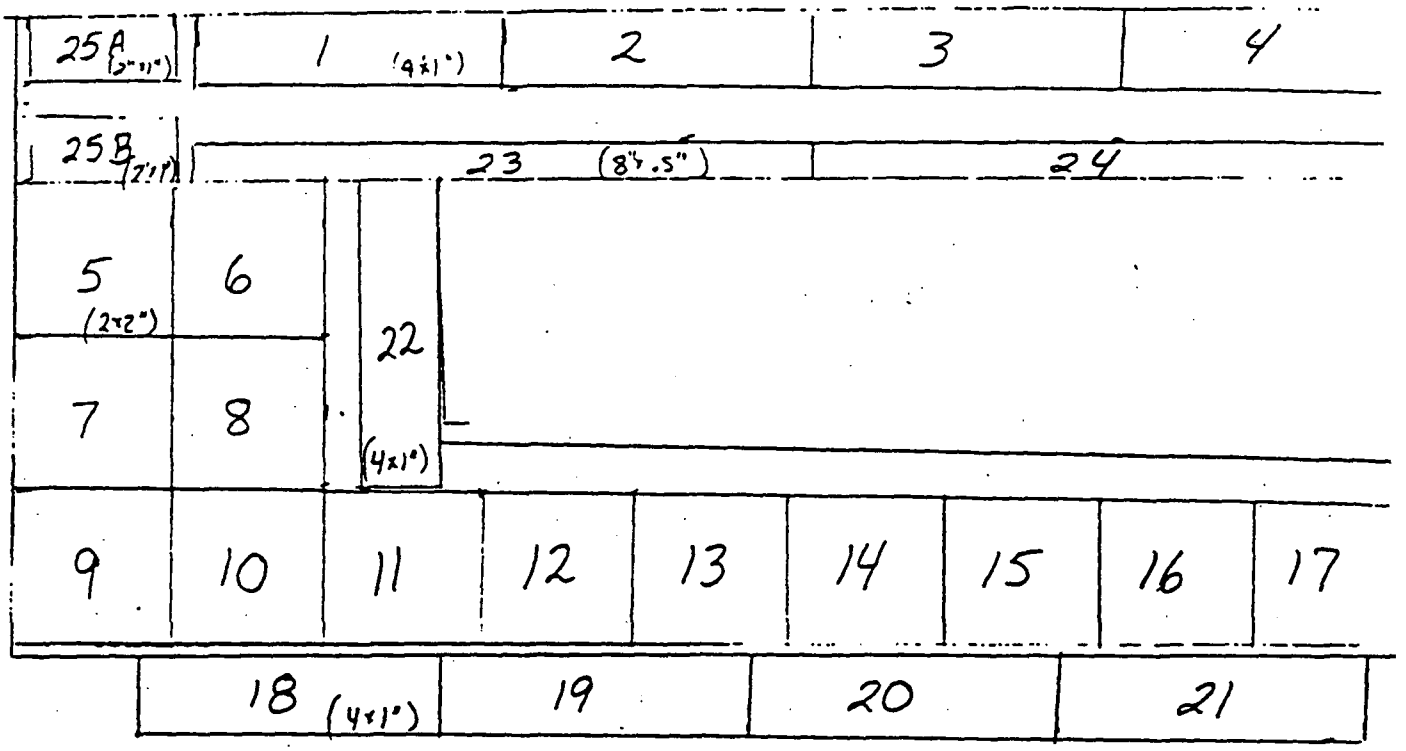
WALL #7



WALL #7

#	SET A	B	C	D	E
	5	3	1	4	2
	9	6	8	7	10
	11	15	13	12	14
	18	20	17	19	16
	22	25	23	21	24

WALL #6



WALL #6

#	SET A	B	C	D	E
	4	5	2	3	1
	6	10	7	9	8
	13	14	12	11	15
	20	16	19	17	18
	22	24	23	25	21

BOARD #5

1	2	3
4	5	6
7	8	chips X
10	11	12
13	14	15
16	chips X	17
18	19	20
21	22	23
24	25	chips X
		9

#	SET A	B	C	D	E
	5	2	3	1	4
	8	9	7	10	6
	15	12	11	14	13
	19	16	20	17	18
	21	25	24	22	23

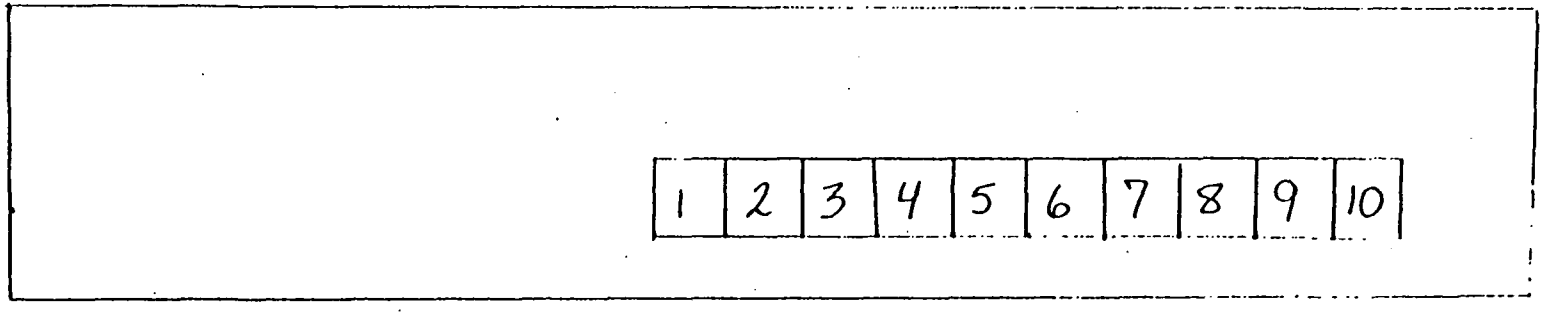
BOARD #8

1	2	3
4	5	6
7	8	9
10	11	12
13	14	15

	SET				
#	A	B	C	D	E
	1	2	3	4	5
	6	7	8	9	10
	15	14	13	12	11
	16	17	18	19	20
	25	24	23	22	21

16	17	18
19	20	21
22	23	24
	25	

BOARD X



APPENDIX B

Phase I Data

Resolution of Paint Grinding Techniques: Procedure A (glass rod)

Sample ID	Paint Sample Weight (g)	Analysis Aliquot Weight (g)	Pb Conc. Measured by ICP (%)	Pb Conc. Measured by ICP (ug/g)	Total Pb in Paint Sample (ug)	Paint Sample Weight (g/cm^2)	Pb Conc. Measured by ICP (ug/cm^2)
Wall 6-4	2.3138	0.1032	1.59	15900	36789	0.0897	1426
Wall 6-6	2.6393	0.1068	2.33	23300	61496	0.1023	2383
Wall 6-13	2.4009	0.1013	1.8	18000	43216	0.0930	1675
Wall 6-20	2.4144	0.1076	1.64	16400	39596	0.0936	1534
Wall 6-22	2.6692	0.103	2	20000	53384	0.1034	2069
Avg	2.4875		1.8720	18720	46896	0.0964	1817
sd	0.1574		0.3019	3019	10298	0.0061	399
RSD %	6.33		16.13	16.13	21.96	6.33	21.96
Wall 7-5	1.7879	0.1027	1.39	13900	24852	0.0693	963
Wall 7-9	1.9999	0.1024	1.16	11600	23199	0.0775	899
Wall 7-11	2.5956	0.1023	0.968	9680	25125	0.1006	974
Wall 7-18	2.3422	0.1068	1.54	15400	36070	0.0908	1398
Wall 7-22	2.4787	0.1006	1.44	14400	35693	0.0960	1383
Avg	2.2409		1.2996	12996	28988	0.0868	1123
sd	0.3376		0.2319	2319	6338	0.0131	246
RSD %	15.07		17.84	17.84	21.86	15.07	21.86
Board 5-5	1.2568	0.1009	3.31	33100	41600	0.0487	1612
Board 5-8	1.4622	0.1038	4.55	45500	66530	0.0567	2578
Board 5-15	1.254	0.105	4.65	46500	58311	0.0486	2260
Board 5-19	1.4841	0.1023	3.62	36200	53724	0.0575	2082
Board 5-21	1.3778	0.1064	3.05	30500	42023	0.0534	1628
Avg	1.3670		3.8360	38360	52438	0.0530	2032
sd	0.1093		0.7269	7269	10732	0.0042	416
RSD %	8.00		18.95	18.95	20.47	8.00	20.47
Board 8-1	1.1495	0.1037	4.41	44100	50693	0.0445	1964
Board 8-6	1.018	0.1047	4.36	43600	44385	0.0394	1720
Board 8-15	1	0.1006	4.44	44400	44400	0.0388	1721
Board 8-16	1.257	0.1019	3.36	33600	42235	0.0487	1637
Board 8-25	0.8419	0.1078	3.88	38800	32666	0.0326	1266
Avg	1.0533		4.0900	40900	42876	0.0408	1661
sd	0.1577		0.4677	4677	6526	0.0061	253
RSD %	14.98		11.43	11.43	15.22	14.98	15.22

Resolution of Paint Grinding Techniques: Procedure B (dry ice and glass rod)

Sample ID	Paint Sample Weight (g)	Analysis Aliquot Weight (g)	Pb Conc. Measured by ICP (%)	Pb Conc. Measured by ICP (ug/g)	Total Pb in Paint Sample (ug)	Paint Sample Weight (g/cm^2)	Pb Conc. Measured by ICP (ug/cm^2)
Wall 6-5	2.7417	0.1062	1.67	16700	45786	0.1062	1774
Wall 6-10	2.6778	0.1058	2.16	21600	57840	0.1038	2241
Wall 6-14	2.231	0.1078	1.62	16200	36142	0.0865	1401
Wall 6-16	2.4076	0.1056	1.68	16800	40448	0.0933	1567
Wall 6-24	2.1735	0.1044	1.48	14800	32168	0.0842	1247
Avg	2.4463		1.7220	17220	42477	0.0948	1646
sd	0.2565		0.2575	2575	9969	0.0099	386
RSD %	10.48		14.96	14.96	23.47	10.48	23.47
Wall 7-3	1.7809	0.1006	1.04	10400	18521	0.0690	718
Wall 7-6	1.9188	0.1063	1.08	10800	20723	0.0744	803
Wall 7-15	2.3969	0.1009	1.28	12800	30680	0.0929	1189
Wall 7-20	2.5352	0.1072	1.49	14900	37774	0.0982	1464
Wall 7-25	2.5463	0.108	1.49	14900	37940	0.0987	1470
Avg	2.2356		1.2760	12760	29128	0.0866	1129
sd	0.3604		0.2155	2155	9192	0.0140	356
RSD %	16.12		16.89	16.89	31.56	16.12	31.56
Board 5-2	1.2193	0.1042	3.74	37400	45602	0.0472	1767
Board 5-9	1.2007	0.1061	3.43	34300	41184	0.0465	1596
Board 5-12	1.3845	0.1022	4.15	41500	57457	0.0536	2226
Board 5-16	1.3845	0.1003	3.57	35700	49427	0.0536	1915
Board 5-25	1.3388	0.1023	3.02	30200	40432	0.0519	1567
Avg	1.3056		3.5820	35820	46820	0.0506	1814
sd	0.0894		0.4143	4143	6964	0.0035	270
RSD %	6.85		11.57	11.57	14.87	6.85	14.87
Board 8-2	1.2671	0.1018	3.53	35300	44729	0.0491	1733
Board 8-7	1.0275	0.1021	3.17	31700	32572	0.0398	1262
Board 8-14	0.9091	0.1015	3.48	34800	31637	0.0352	1226
Board 8-17	0.9309	0.1029	5.61	56100	52223	0.0361	2024
Board 8-24	0.8741	0.1056	4.33	43300	37849	0.0339	1467
Avg	1.0017		4.0240	40240	39802	0.0388	1542
sd	0.1589		0.9847	9847	8679	0.0062	336
RSD %	15.86		24.47	24.47	21.81	15.86	21.81

Resolution of Paint Grinding Techniques: Procedure C (mortar and pestle)

Sample ID	Paint Sample Weight (g)	Analysis Aliquot Weight (g)	Pb Conc. Measured by ICP (%)	Pb Conc. Measured by ICP (ug/g)	Total Pb in Paint Sample (ug)	Paint Sample Weight (g/cm^2)	Pb Conc. Measured by ICP (ug/cm^2)
Wall 6-2	2.423	0.1043	1.25	12500	30288	0.0939	1174
Wall 6-7	2.631	0.1077	1.87	18700	49200	0.1020	1906
Wall 6-12	2.2869	0.101	1.61	16100	36819	0.0886	1427
Wall 6-19	2.2178	0.1022	1.81	18100	40142	0.0859	1556
Wall 6-23	2.3491	0.105	1.34	13400	31478	0.0910	1220
Avg	2.3816		1.5760	15760	37585	0.0923	1456
sd	0.1587		0.2758	2758	7625	0.0062	295
RSD %	6.66		17.50	17.50	20.29	6.66	20.29
Wall 7-1	2.0692	0.1055	0.891	8910	18437	0.0802	714
Wall 7-8	1.6668	0.1094	0.708	7080	11801	0.0646	457
Wall 7-13	2.3255	0.1021	1.44	14400	33487	0.0901	1298
Wall 7-17	2.1541	0.1029	1.16	11600	24988	0.0835	968
Wall 7-23	2.5248	0.1029	1.52	15200	38377	0.0978	1487
Avg	2.1481		1.1438	11438	25418	0.0832	985
sd	0.3207		0.3476	3476	10810	0.0124	419
RSD %	14.93		30.39	30.39	42.53	14.93	42.53
Board 5-3	1.0072	0.1032	2.87	28700	28907	0.0390	1120
Board 5-7	1.296	0.1	2.98	29800	38621	0.0502	1497
Board 5-11	1.3482	0.1022	4.73	47300	63770	0.0522	2471
Board 5-20	1.4967	0.1084	4.02	40200	60167	0.0580	2331
Board 5-24	1.2327	0.1078	2.94	29400	36241	0.0478	1404
Avg	1.2762		3.5080	35080	45541	0.0495	1765
sd	0.1792		0.8312	8312	15470	0.0069	599
RSD %	14.04		23.70	23.70	33.97	14.04	33.97
Board 8-3	1.2593	0.1003	5.06	50600	63721	0.0488	2469
Board 8-8	0.9333	0.103	4.49	44900	41905	0.0362	1624
Board 8-13	1.0239	0.1027	4.55	45500	46587	0.0397	1805
Board 8-18	0.8517	0.1036	4.18	41800	35601	0.0330	1380
Board 8-23	0.7486	0.1082	3.74	37400	27998	0.0290	1085
Avg	0.9634		4.4040	44040	43162	0.0373	1673
sd	0.1941		0.4874	4874	13445	0.0075	521
RSD %	20.15		11.07	11.07	31.15	20.15	31.15

Resolution of Paint Grinding Techniques: Procedure D (Bel Arts Products Micro Mill)

Sample ID	Paint Sample Weight (g)	Analysis Aliquot Weight (g)	Pb Conc. Measured by ICP (%)	Pb Conc. Measured by ICP (ug/g)	Total Pb in Paint Sample (ug)	Paint Sample Weight (g/cm^2)	Pb Conc. Measured by ICP (ug/cm^2)
Wall 6-3	2.3094	0.1039	1.35	13500	31177	0.0895	1208
Wall 6-9	2.7504	0.1082	1.84	18400	50607	0.1066	1961
Wall 6-11	2.4115	0.1044	1.66	16600	40031	0.0934	1551
Wall 6-17	2.4201	0.1065	1.37	13700	33155	0.0938	1285
Wall 6-25	2.7049	0.1019	1.6	16000	43278	0.1048	1677
Avg	2.5193		1.5640	15640	39650	0.0976	1536
sd	0.1958		0.2062	2062	7864	0.0076	305
RSD %	7.77		13.19	13.19	19.83	7.77	19.83
Wall 7-4	1.7809	0.1006	0.816	8160	14532	0.0690	563
Wall 7-7	1.9703	0.1023	0.912	9120	17969	0.0763	696
Wall 7-12	2.4146	0.1038	1.06	10600	25595	0.0936	992
Wall 7-19	2.2965	0.1051	1.25	12500	28706	0.0890	1112
Wall 7-21	2.5511	0.1034	1.66	16600	42348	0.0989	1641
Avg	2.2027		1.1396	11396	25830	0.0854	1001
sd	0.3190		0.3339	3339	10847	0.0124	420
RSD %	14.48		29.30	29.30	41.99	14.48	41.99
Board 5-1	1.2312	0.1001	3.51	35100	43215	0.0477	1675
Board 5-10	1.2478	0.1043	3.93	39300	49039	0.0484	1900
Board 5-14	1.2681	0.1017	3.39	33900	42989	0.0491	1666
Board 5-17	1.4023	0.105	3.75	37500	52586	0.0543	2038
Board 5-22	1.3137	0.1005	3.01	30100	39542	0.0509	1532
Avg	1.2926		3.5180	35180	45474	0.0501	1762
sd	0.0687		0.3529	3529	5238	0.0027	203
RSD %	5.31		10.03	10.03	11.52	5.31	11.52
Board 8-4	n/a	0.1044	3.18	31800			
Board 8-9	0.9141	0.1007	2.99	29900	27332	0.0354	1059
Board 8-12	0.9747	0.1029	3.46	34600	33725	0.0378	1307
Board 8-19	0.8654	0.1065	3	30000	25962	0.0335	1006
Board 8-22	0.9331	0.1036	3.88	38800	36204	0.0362	1403
Avg	0.9218		3.3020	33020	30806	0.0357	1194
sd	0.0453		0.3750	3750	4939	0.0018	191
RSD %	4.92		11.36	11.36	16.03	4.92	16.03

Resolution of Paint Grinding Techniques: Procedure E (Spex Freezer Mill)

Sample ID	Paint Sample Weight (g)	Analysis Aliquot Weight (g)	Pb Conc. Measured by ICP (%)	Pb Conc. Measured by ICP (ug/g)	Total Pb in Paint Sample (ug)	Paint Sample Weight (g/cm^2)	Pb Conc. Measured by ICP (ug/cm^2)
Wall 6-1	2.6394	0.1069	1.45	14500	38271	0.1023	1483
Wall 6-8	2.3447	0.1072	1.42	14200	33295	0.0909	1290
Wall 6-15	2.2122	0.1089	1.51	15100	33404	0.0857	1294
Wall 6-18	2.607	0.1047	1.64	16400	42755	0.1010	1657
Wall 6-21	2.4714	0.1047	1.46	14600	36082	0.0958	1398
Avg	2.4549		1.4960	14960	36762	0.0951	1425
sd	0.1792		0.0868	868	3935	0.0069	152
RSD %	7.30		5.80	5.80	10.71	7.30	10.71
Wall 7-2	2.2094	0.1008	1.05	10500	23199	0.0856	899
Wall 7-10	2.2653	0.1059	1.1	11000	24918	0.0878	966
Wall 7-14	2.6764	0.1011	0.986	9860	26389	0.1037	1023
Wall 7-16	2.2096	0.1016	1.24	12400	27399	0.0856	1062
Wall 7-24	2.3912	0.1006	1.53	15300	36585	0.0927	1418
Avg	2.3504		1.1812	11812	27698	0.0911	1073
sd	0.1968		0.2162	2162	5214	0.0076	202
RSD %	8.37		18.31	18.31	18.83	8.37	18.83
Board 5-4	1.2682	0.1019	3.03	30300	38426	0.0491	1489
Board 5-6	1.1333	0.1002	4.56	45600	51678	0.0439	2003
Board 5-13	1.2387	0.1031	3.63	36300	44965	0.0480	1742
Board 5-18	1.4199	0.1051	3.42	34200	48561	0.0550	1882
Board 5-23	1.5795	0.1029	4.08	40800	64444	0.0612	2497
Avg	1.3279		3.7440	37440	49615	0.0515	1923
sd	0.1740		0.5930	5930	9644	0.0067	374
RSD %	13.11		15.84	15.84	19.44	13.11	19.44
Board 8-5	0.98	0.1007	5.14	51400	50372	0.0380	1952
Board 8-10	1.0966	0.1013	4.56	45600	50005	0.0425	1938
Board 8-11	1.0085	0.1037	5.13	51300	51736	0.0391	2005
Board 8-20	0.8232	0.1013	4.59	45900	37785	0.0319	1464
Board 8-21	0.9769	0.1057	5.3	53000	51776	0.0379	2006
Avg	0.9770		4.9440	49440	48335	0.0379	1873
sd	0.0987		0.3437	3437	5951	0.0038	231
RSD %	10.10		6.95	6.95	12.31	10.10	12.31

Weight of a 1 square inch sample (g)

Door #5		Wall #7		Board X	
# 26	0.28	# 26	0.58	# 1	0.51
# 27	0.31	# 27	0.60	# 2	0.51
# 28	0.31	# 28	0.54	# 3	0.5
# 29	0.28	# 29	0.58	# 4	0.53
# 30	0.32	# 30	0.57	# 5	0.55
# 31	0.30	# 31	0.57	# 6	0.56
# 32	0.28	# 32	0.59	# 7	0.49
# 33	0.32	# 33	0.62	# 8	0.54
# 34	0.36	# 34	0.61	# 9	0.51
# 35	0.31	# 35	0.59	# 10	0.53
Avg	0.31		0.59		0.52
s.d.	0.02		0.02		0.02
RSD	7.99		3.89		4.33

APPENDIX C

Phase II Data

Lead Concentration in Paint (%)

Grinding time		30 sec	5 min	diff
Door #5				
glass rod	# 26	2.74	2.20	0.54
centrifuge	# 27	4.17	3.44	0.73
	# 28	3.81	4.03	-0.22
	# 32	3.28	3.22	0.06
	# 34	4.41	4.74	-0.33
Avg		3.68	3.53	0.16
s.d.		0.68	0.95	0.46
RSD		18.4	26.9	---
mortar &	# 29	3.29	4.23	-0.94
pestle	# 30	4.99	5.46	-0.47
	# 31	5.06	4.83	0.23
	# 33	4.15	3.68	0.47
	# 35	4.69	4.84	-0.15
Avg		4.44	4.61	-0.17
s.d.		0.73	0.68	0.56
RSD		16.5	14.7	-
Wall #7				
glass rod	# 26	1.10	0.958	0.142
centrifuge	# 27	1.04	0.965	0.075
	# 28	1.14	0.986	0.154
	# 32	1.04	0.962	0.078
	# 34	1.28	1.17	0.11
Avg		1.12	1.01	0.11
s.d.		0.10	0.09	0.04
RSD		8.8	9.0	-
mortar &	# 29	0.833	1.02	-0.18
pestle	# 30	1.04	1.02	0.02
	# 31	1.07	0.988	0.082
	# 33	1.03	1.00	0.03
	# 35	1.18	1.26	-0.08
Avg		1.03	1.06	-0.03
s.d.		0.13	0.11	0.11
RSD		12.2	10.8	-
Board X				
glass rod	# 1	3.7	5.53	-1.83
centrifuge	# 3	5.44	5.25	0.19
	# 5	5.08	5.34	-0.26
	# 7	4.92	5.14	-0.22
	# 9	5.14	4.65	0.49
Avg		4.86	5.18	-0.33
s.d.		0.67	0.33	0.90
RSD		13.9	6.4	---
mortar &	# 2	5.36	5.43	-0.07
pestle	# 4	8.48	6.5	1.98
	# 6	5.8	4.99	0.81
	# 8	8.62	7.43	1.19
	# 10	7.08	6.29	0.79
Avg		7.07	6.13	0.94
s.d.		1.49	0.95	0.74
RSD		21.1	15.6	---

APPENDIX D

Statistical Data Interpretations

Following a log-transformation of measurement data, variances were estimated for each grinding technique. These were then compared using an F test. No significant differences were found at the 1% level (critical value = 3.4). The table below shows the estimated standard deviation (ln-scale) and F ratios, where F = max (s^2_x/s^2_y, s^2_y/s^2_x). The greatest ratio found, 3.17, is less than the critical value.

		A 0.165961	B 0.169416	C 0.223662	D 0.17162	E 0.125582
Procedure A (glass rod)	0.165961	1	1.04207	1.816241	1.069365	1.746449
Procedure B (dry ice glass rod)	0.169416	1.04207	1	1.742916	1.026193	1.819923
Procedure C (Mortar)	0.223662	1.816241	1.742916	1	1.69843	3.171973
Procedure D (grinder)	0.17162	1.069365	1.026193	1.69843	1	1.867591
Procedure E (Spex)	0.125582	1.746449	1.819923	3.171973	1.867591	1

F crit = 3.4
MAX = 3.171973

Lead Concentration in Paint (%)

Grinding	time / Board X		30 sec	5 min	diff	Statistics on 30 s vs. 5 min grinding
glass rod centrifuge		# 1	3.7	5.53	-1.83	
		# 3	5.44	5.25	0.19	
		# 5	5.08	5.34	-0.26	
		# 7	4.92	5.14	-0.22	
		# 9	5.14	4.65	0.49	
	Avg		4.86	5.18	-0.33	F = 4.12
	s.d.		0.67	0.33	0.90	s pooled 0.5281
	RSD		13.9	6.4	-275	t test 0.32 < 0.77
						no difference
mortar & pestle		# 2	5.36	5.43	-0.07	
		# 4	8.48	6.5	1.98	
		# 6	5.8	4.99	0.81	
		# 8	8.62	7.43	1.19	
		# 10	7.08	6.29	0.79	
	Avg		7.07	6.13	0.94	F = 2.46
	s.d.		1.49	0.95	0.74	s pooled 1.2495
	RSD		21.1	15.6	79.0	t test 0.94 < 1.82
						no difference
Stats grind methods	F =		4.76	8.33 (Failed)		
	s pooled		1.1552	Alt method		
	t test		2.21 < 1.6866	0.95 < 1.1564		
			DIFFERENCE	no difference		

Lead Concentration in Paint (%)

Grinding	time		30 sec	5 min	diff	Statistics on 30 s vs. 5 min grinding
	Door #5					
glass rod		# 26	2.74	2.20	0.54	
centrifuge		# 27	4.17	3.44	0.73	
		# 28	3.81	4.03	-0.22	
		# 32	3.28	3.22	0.06	
		# 34	4.41	4.74	-0.33	
	Avg		3.68	3.53	0.16	F = 1.96
	s.d.		0.68	0.95	0.46	s pooled = 0.8261
	RSD		18.4	26.9	297.9	t test 0.15 < 1.20
						no difference
mortar &		# 29	3.29	4.23	-0.94	
pestle		# 30	4.99	5.46	-0.47	
		# 31	5.06	4.83	0.23	
		# 33	4.15	3.68	0.47	
		# 35	4.69	4.84	-0.15	
	Avg		4.44	4.61	-0.17	F = 1.15
	s.d.		0.73	0.68	0.56	s pooled = .7054
	RSD		16.5	14.7	-325	t test 0.17 < 1.02
						no difference
Stats	F =		1.15	1.95		
grind.	s pooled		0.7054	0.4808		
methods	t test		0.76 < 1.0299	1.08 < 0.7020		
			no difference	DIFFERENCE		

Procedure*	Example
(1) Choose α, the significance level of the test. (Actually, the procedure outlined will give a significance level of only approximately α).	(1) Let $\alpha = .05$
(2) Compute: $\bar{X}_A$ and s_A^2, $\bar{X}_B$ and s_B^2, for the n_A and n_B measurements from A and B.	(2) $\bar{X}_A = 3166.0$ $s_A^2 = 6328.67$ $n_A = 4$ $\bar{X}_B = 2240.4$ $s_B^2 = 221,661.3$ $n_B = 9$
(3) Compute: $V_A = \frac{s_A^2}{n_A}$ and $V_B = \frac{s_B^2}{n_B}$, the estimated variances of $\bar{X}_A$ and $\bar{X}_B$, respectively.	(3) $V_A = \frac{6328.67}{4}$ $= 1582.17$ $V_B = \frac{221,661.3}{9}$ $= 24629.03$
(4) Compute the "effective number of degrees of freedom" $f = \frac{(V_A + V_B)^2}{\frac{V_A^2}{n_A + 1} + \frac{V_B^2}{n_B + 1}} - 2$	(4) $f = \frac{(26211.20)^2}{500652.4 + 60658911.9} - 2$ $= \frac{687027005}{61159564} - 2$ $= 11.233 - 2$ $= 9.233$
(5) Look up $t_{1-\alpha/2}$ for f' degrees of freedom in Table A-4, where f' is the integer nearest to f; denote this value by $t'_{1-\alpha/2}$.	(5) $f' = 9$ $t'_{.975} = 2.262$
(6) Compute $u = t_{1-\alpha/2}\sqrt{V_A + V_B}$	(6) $u = 2.262\sqrt{26211.20}$ $= 2.262\ (161.9)$ $= 366.2$
(7) If $\lvert\bar{X}_A - \bar{X}_B\rvert > u$, decide that A and B differ with regard to their average performance; otherwise, decide that there is no reason to believe A and B differ in average performance.	(7) $\lvert\bar{X}_A - \bar{X}_B\rvert = 925.6$, which is larger than u. Conclude that A and B differ with regard to average performance.

Experimental Statistics, National Bureau of Statistics Handbook 91, U.S. Department of Commerce, M.G. Natrella, ed., Aug. 1963

COMPARING AVERAGE PERFORMANCE (WHEN F TEST FAILED)

1. $\alpha = 0.05$

2. $\bar{x}_A = 5.18 \quad S^2_A = 0.1089 \; n = 5$

   $\bar{x}_B = 6.13 \quad S^2_B = 0.9025 \; n = 5$

3. $V_A = \underline{0.1089} = 0.0218$

   $V_B = \underline{0.9025} = 0.1805$
   $\quad\quad 5$

4. $$f = \frac{(.0218 + .1805)^2}{\frac{0218^2}{5+1} + \frac{.1805^2}{5+1}} (-2)$$

   $f = 5.43$
   $f^1 = 5$

5. $t_1 - \alpha/_2$ for 5 df = 2.571

6. $\mu = 2.571 \quad (0.0218 + 0.1805)^{1/2}$

   $\mu = 1.1564$

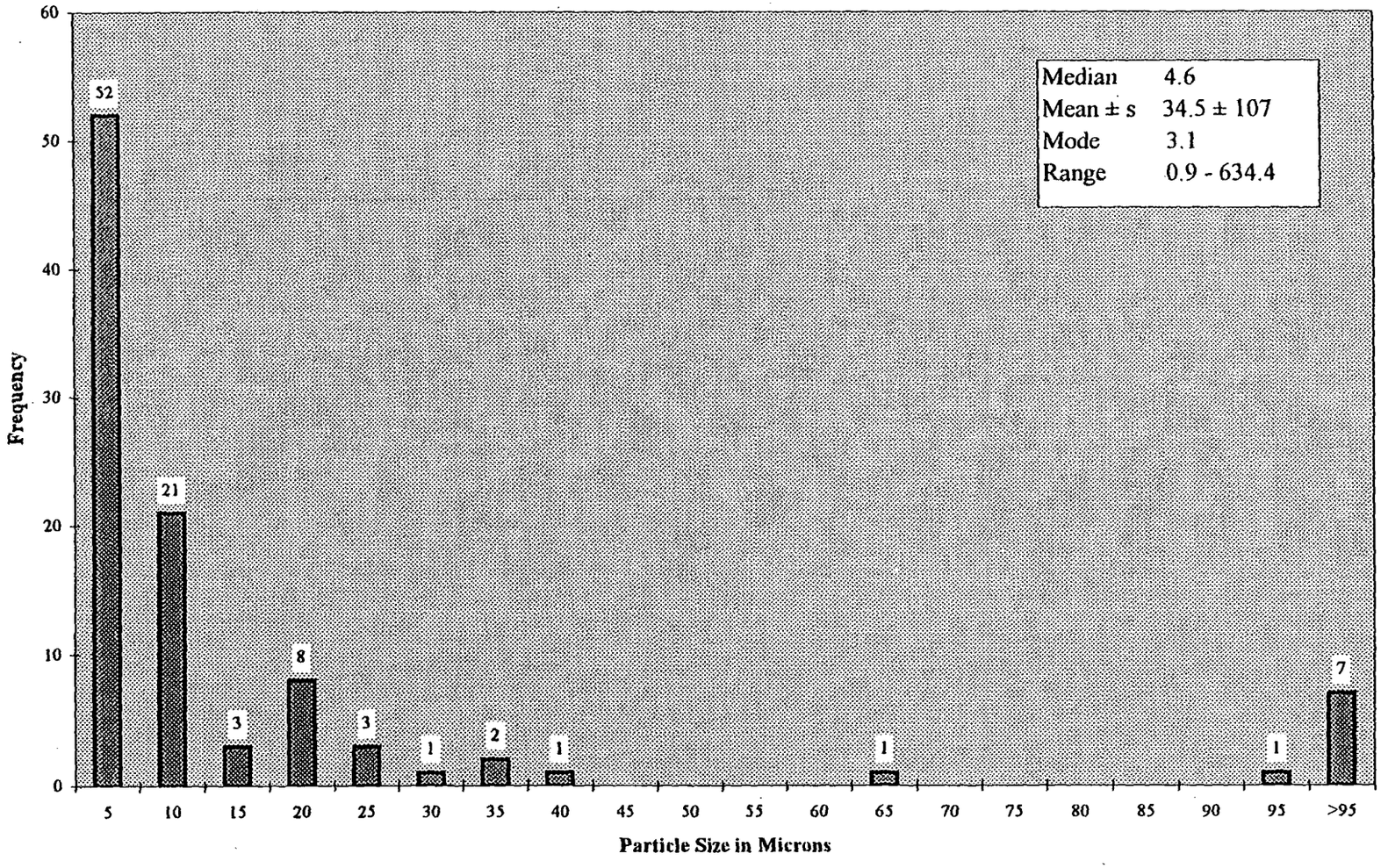
7. Is $(X_A - X_B) \geq 1.1564$?
   5.18 - 6.13 > 1.1564 ?
   .9500 > 1.1564 No

   Therefore, no reason to believe that they are different

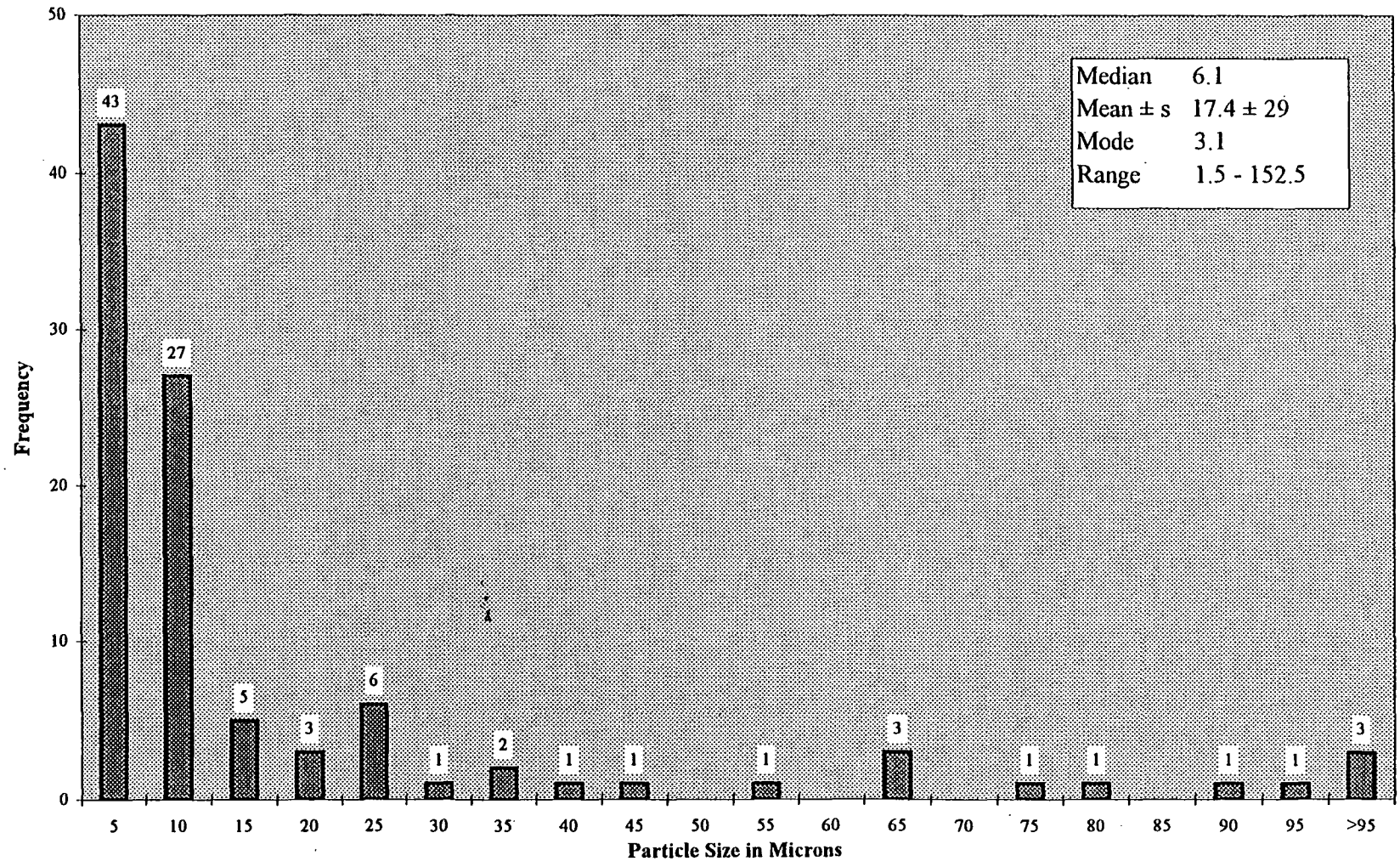
APPENDIX E

Histograms of Particle Size Distribution

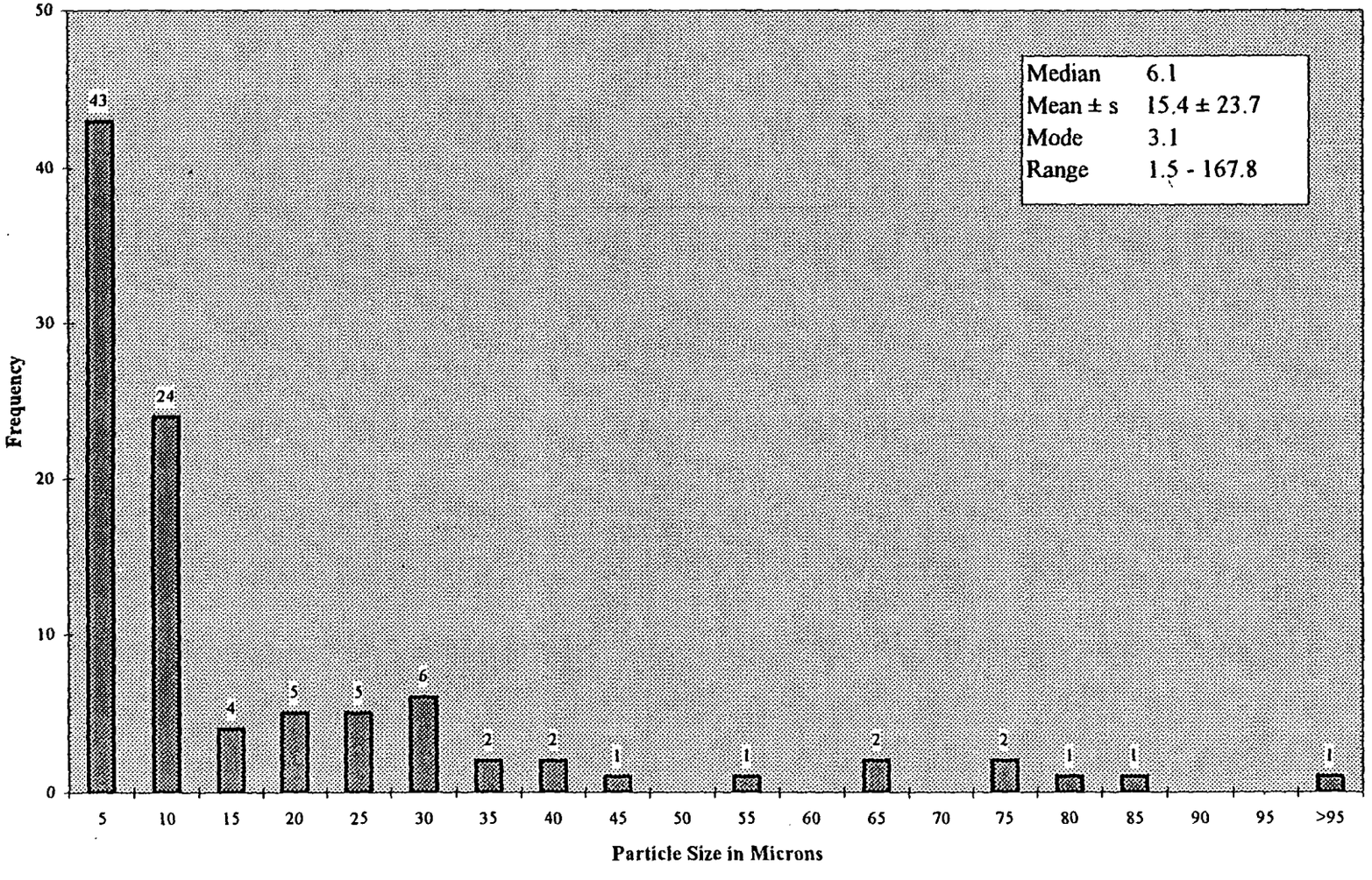
TE-Sample No. 8, Board 5 ($4in^2$)
Glass Rod/ Room Temperature
Ground 5 Minutes



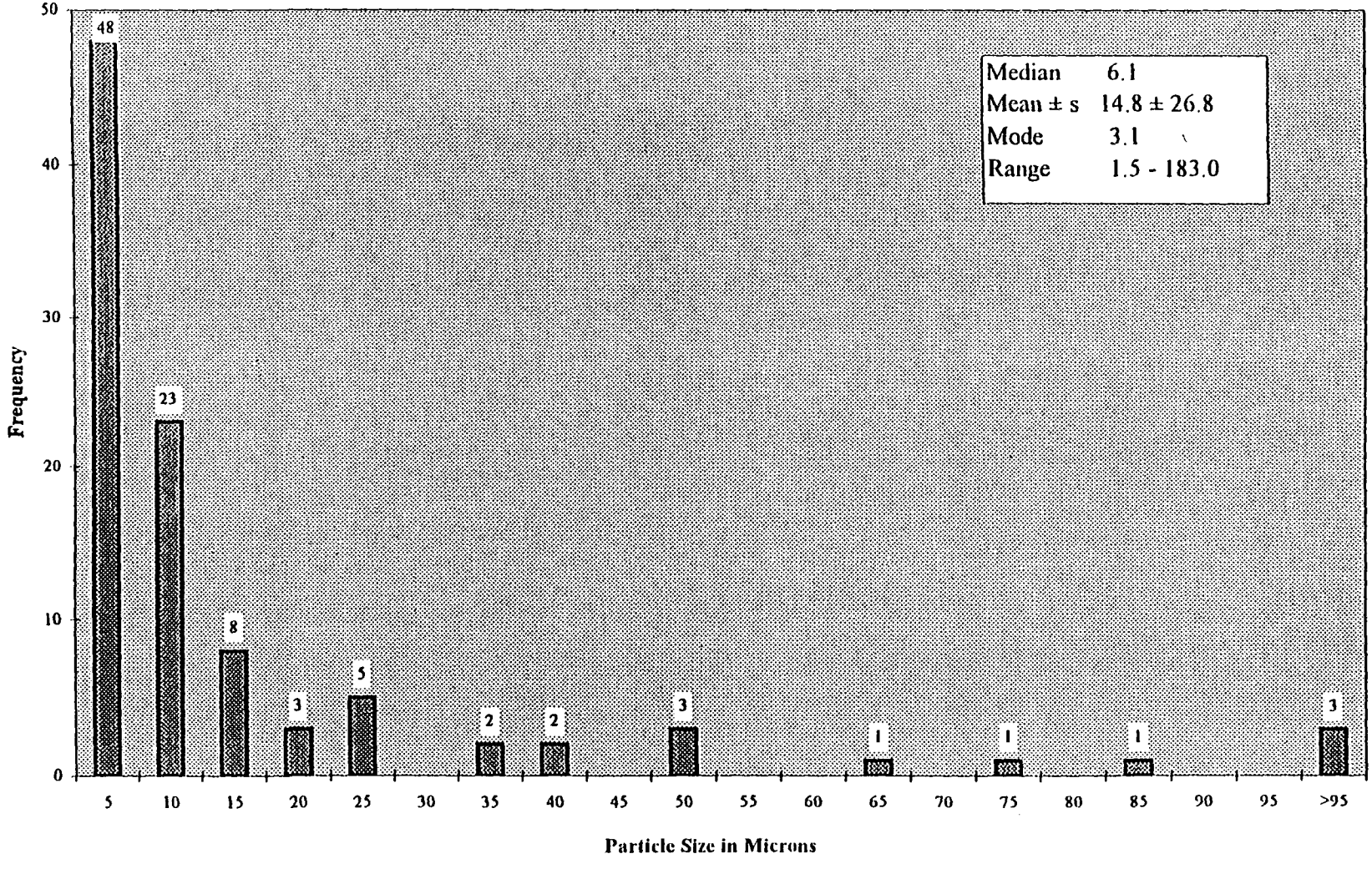
TE-Sample No. 9, Board 5 ($4in^2$)
Glass Rod/ Dry Ice
Ground 5 Minutes



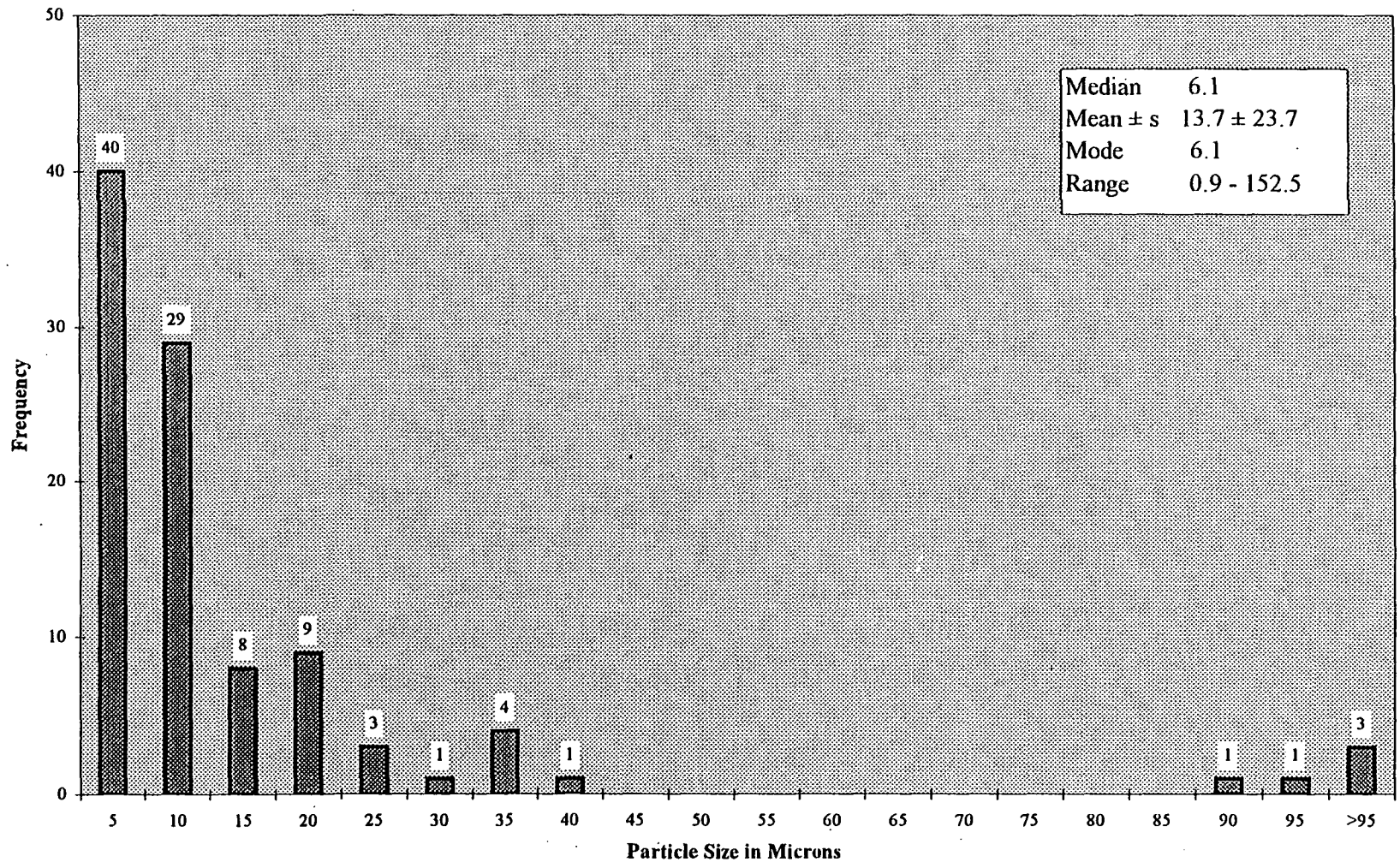
TE-Sample No. 7, Board 5 ($4in^2$)
Mortar & Pestle
Ground 5 Minutes



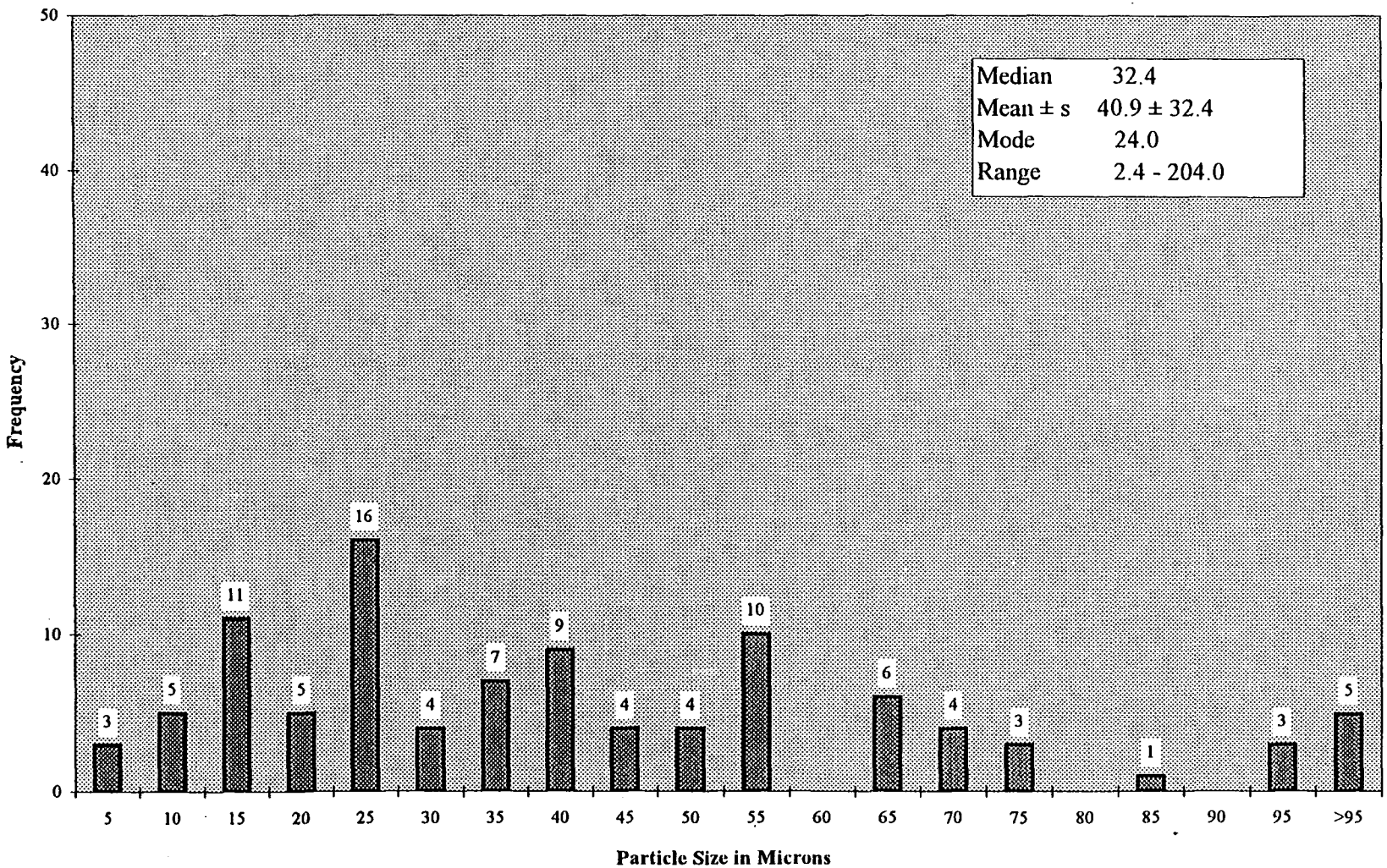
TE-Sample No. 10, Board 5 ($4in^2$)
Bell Art Mill
Ground 30 Seconds



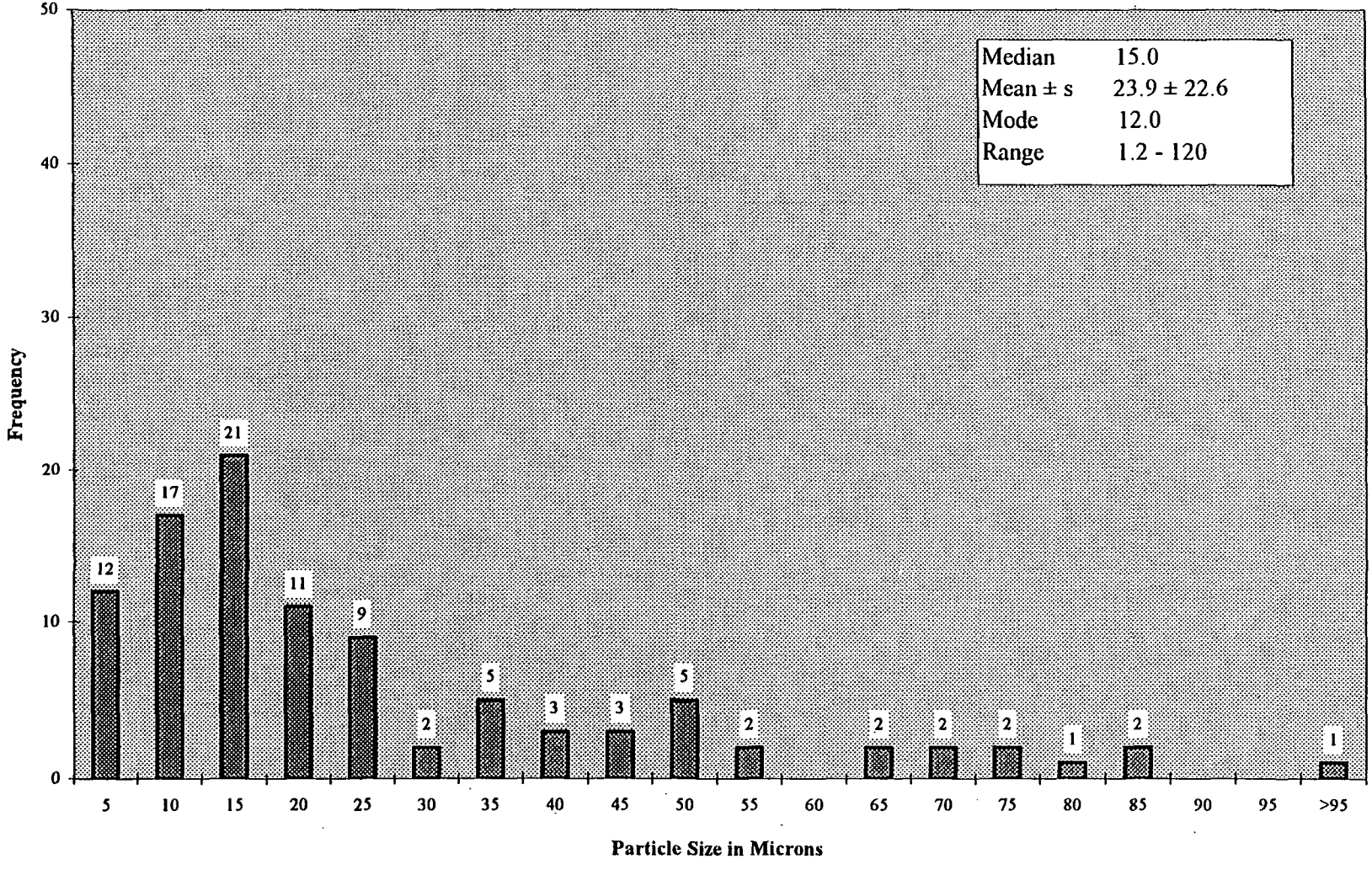
TE-Sample No. 6, Board 5 ($4in^2$)
Spex Freezer Mill
Ground 15 Seconds



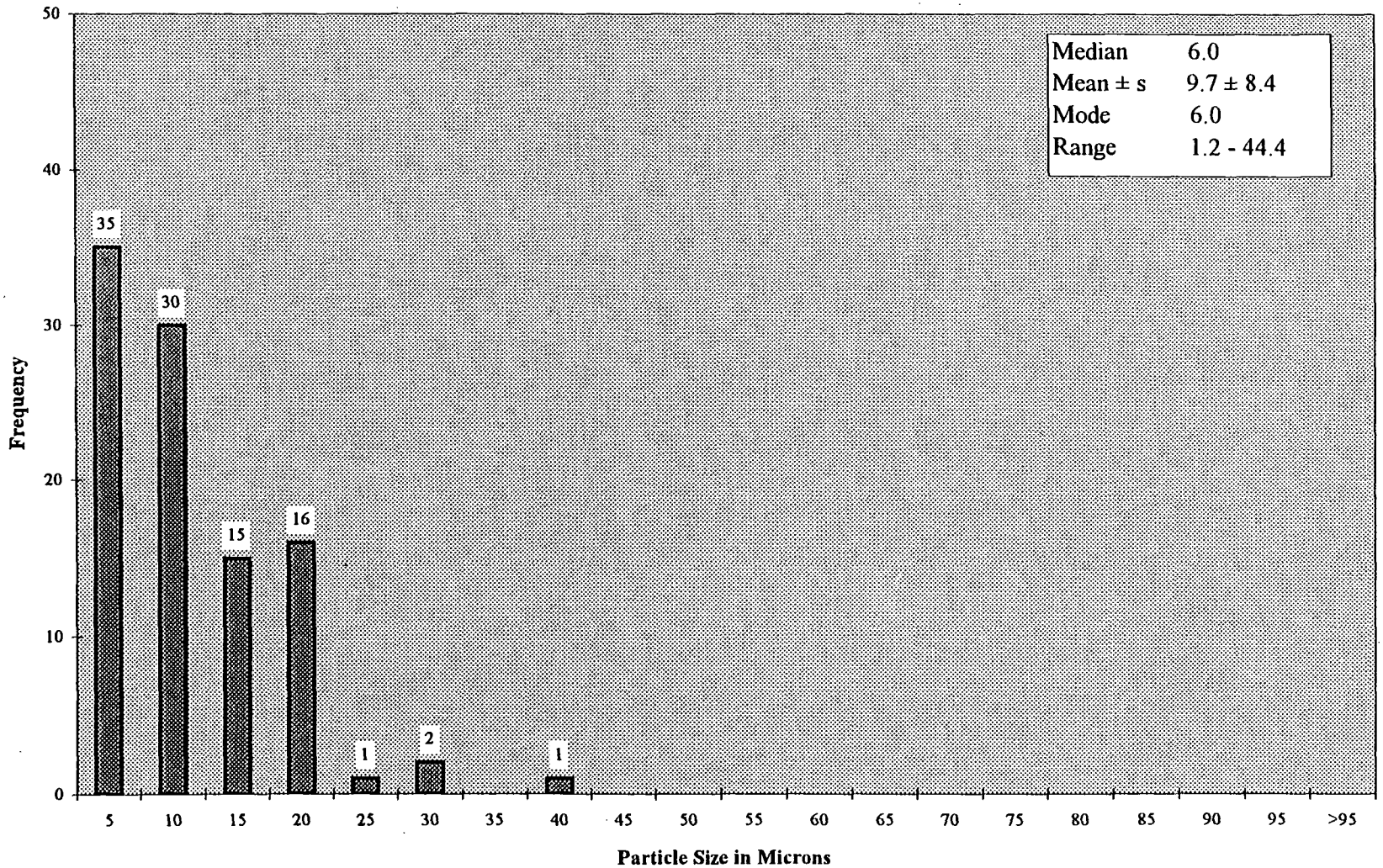
LG-Sample No. 5, Wall 7 ($4in^2$)
Glass Rod/Room Temperature
Ground 5 Minutes



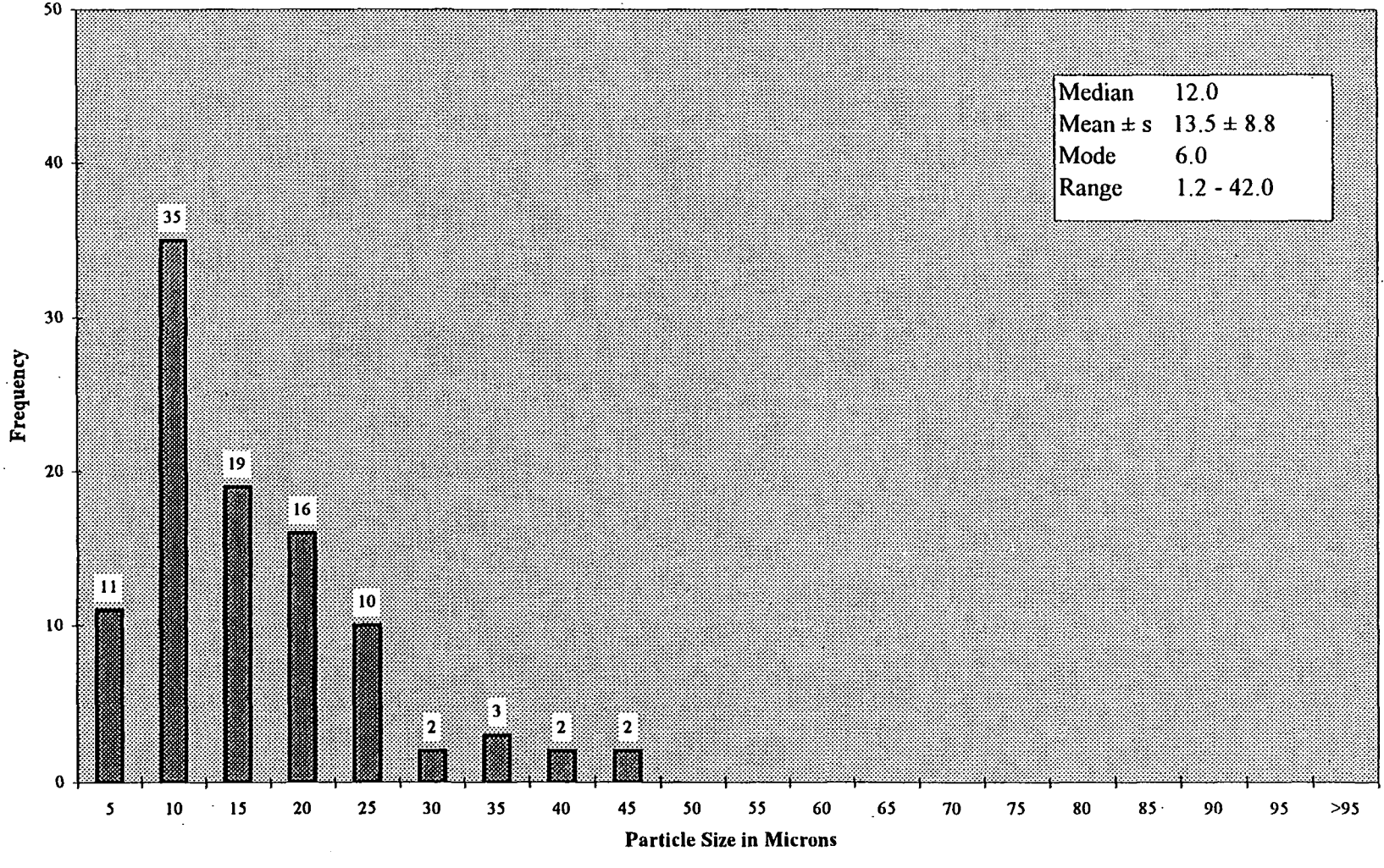
LG-Sample No. 3, Wall 7 ($4in^2$)
Glass Rod/Dry Ice
Ground 5 Minutes



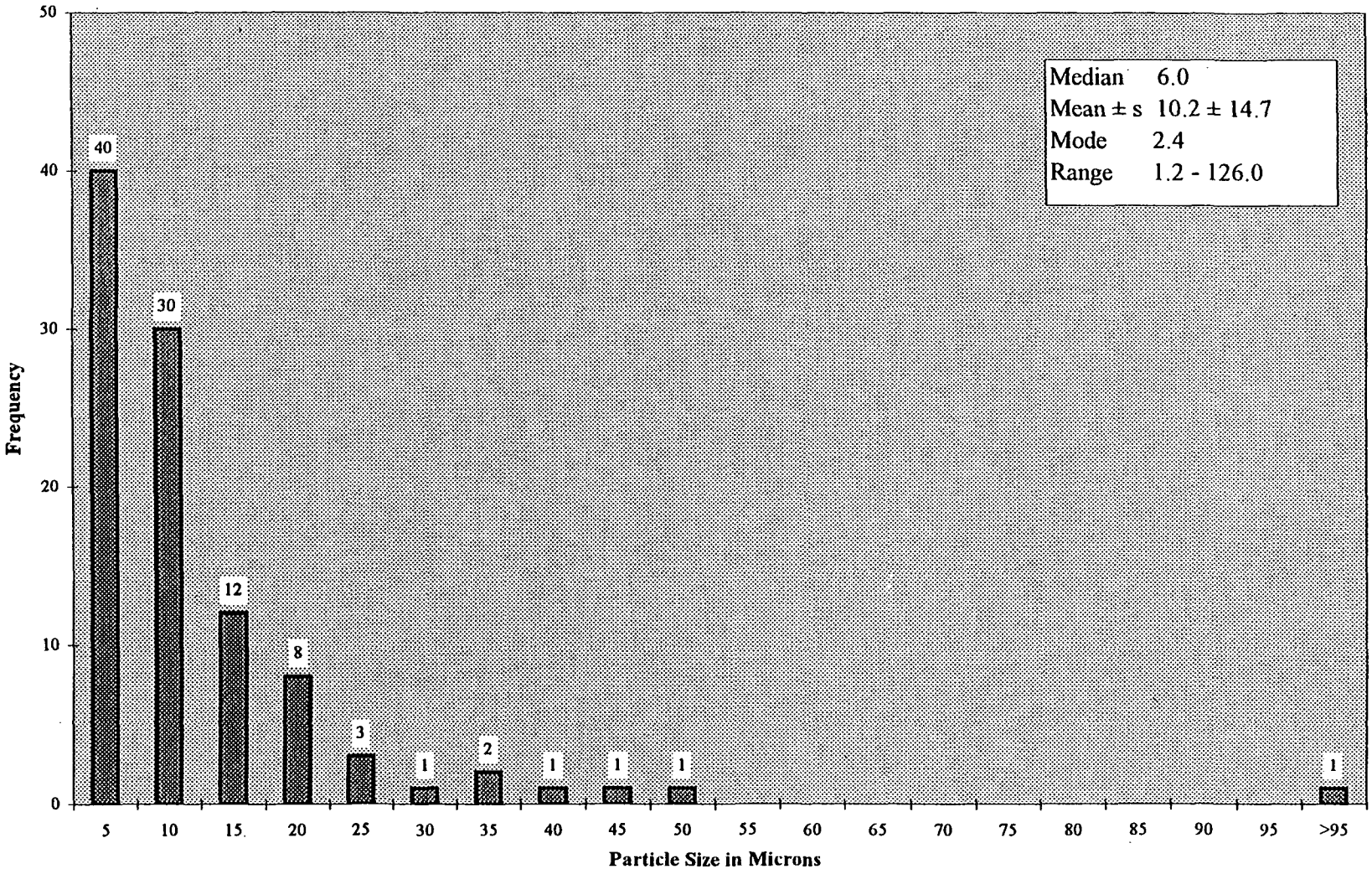
LG-Sample No. 1, Wall 7 ($4in^2$)
Mortar & Pestle
Ground 5 Minutes



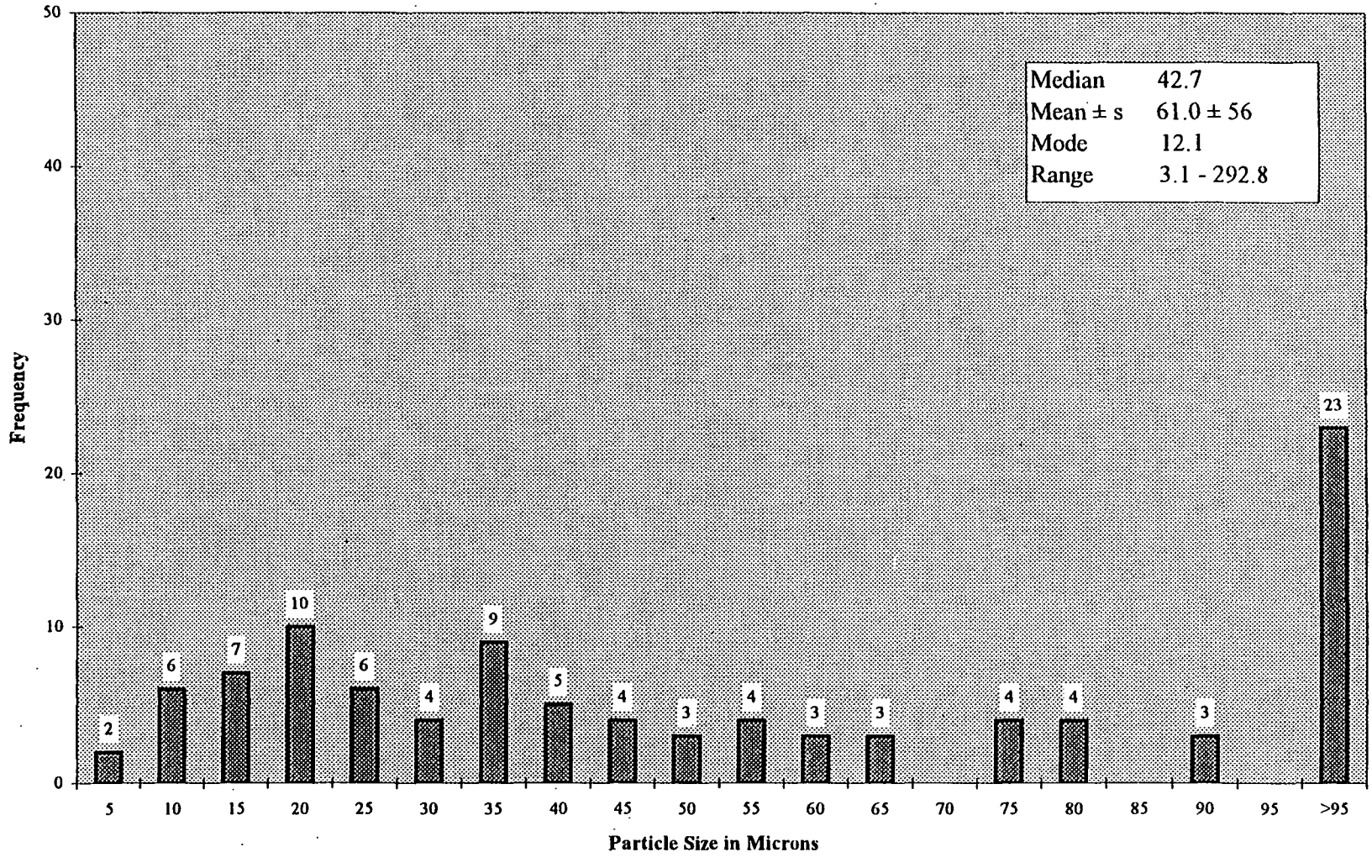
LG-Sample No. 4, Wall 7 ($4in^2$)
Bell Art Mill
Ground 30 Seconds



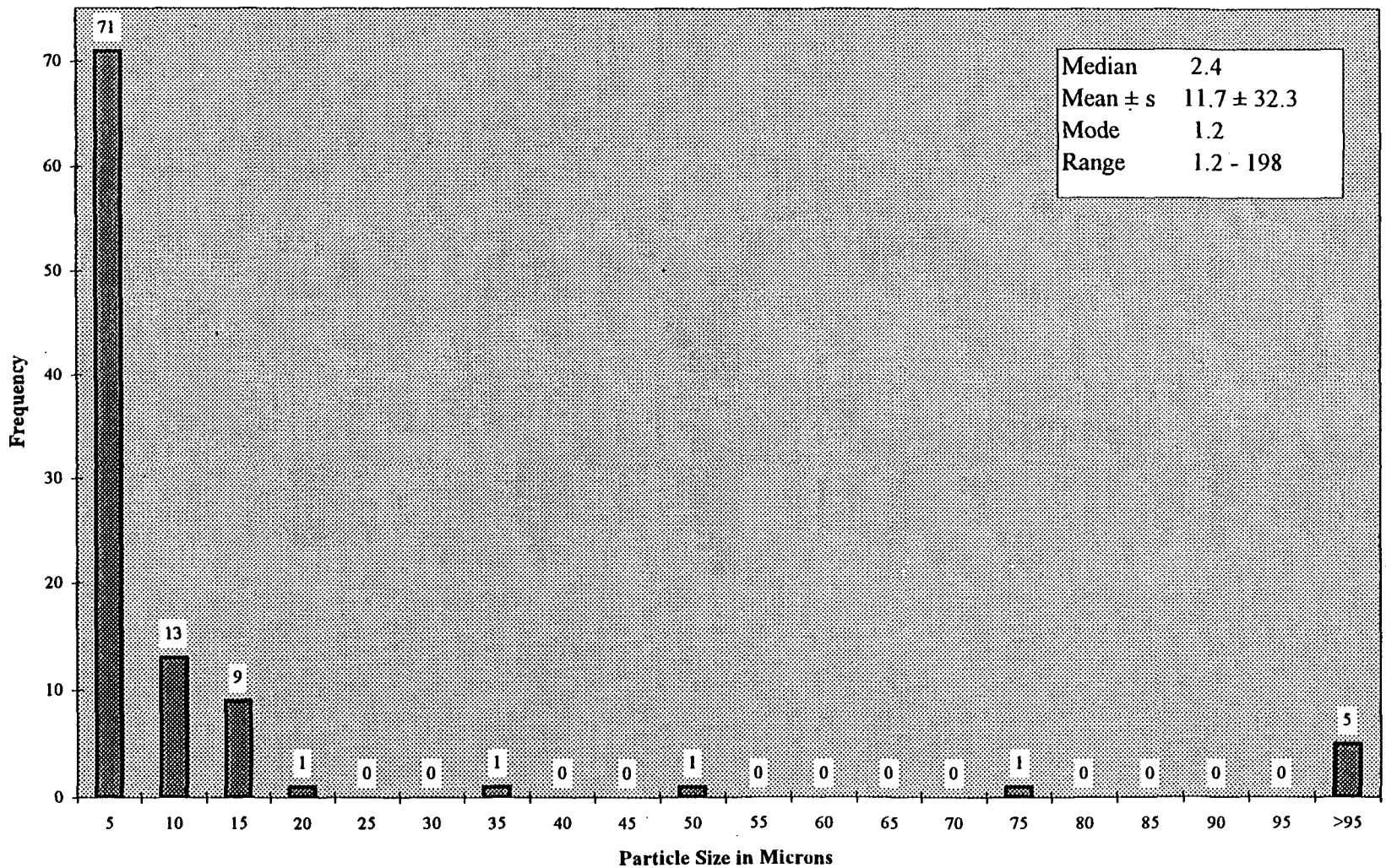
LG-Sample No. 2, Wall 7 ($4in^2$)
Spex Freezer Mill
Ground 15 Seconds



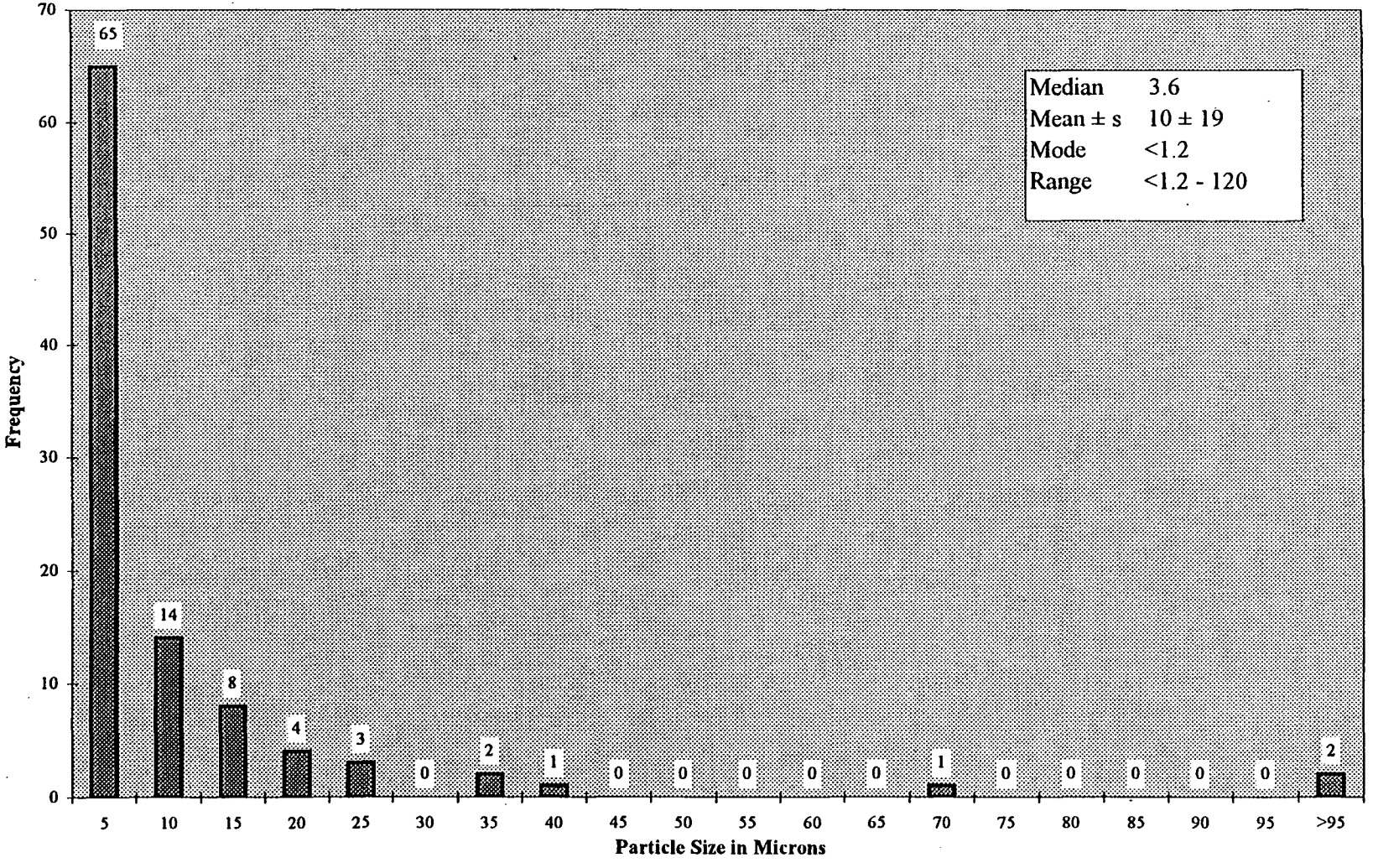
TE-Sample No. 36, Board 5 (1in^2)
Mortar & Pestle
Ground 30 Seconds



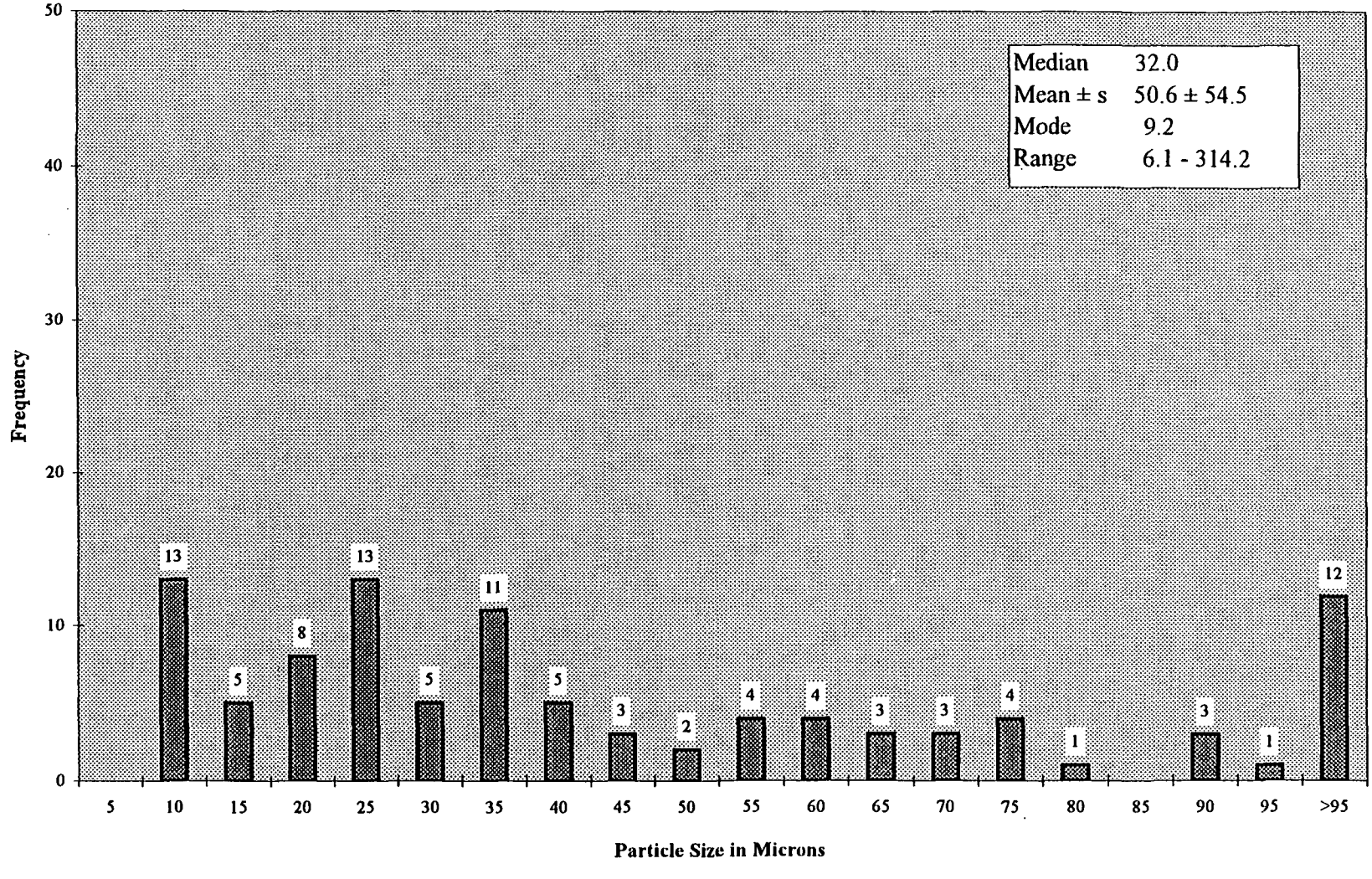
LG-Sample No. 37, Board 5 ($1in^2$)
Mortar & Pestle
Ground 1 1/2 Minutes



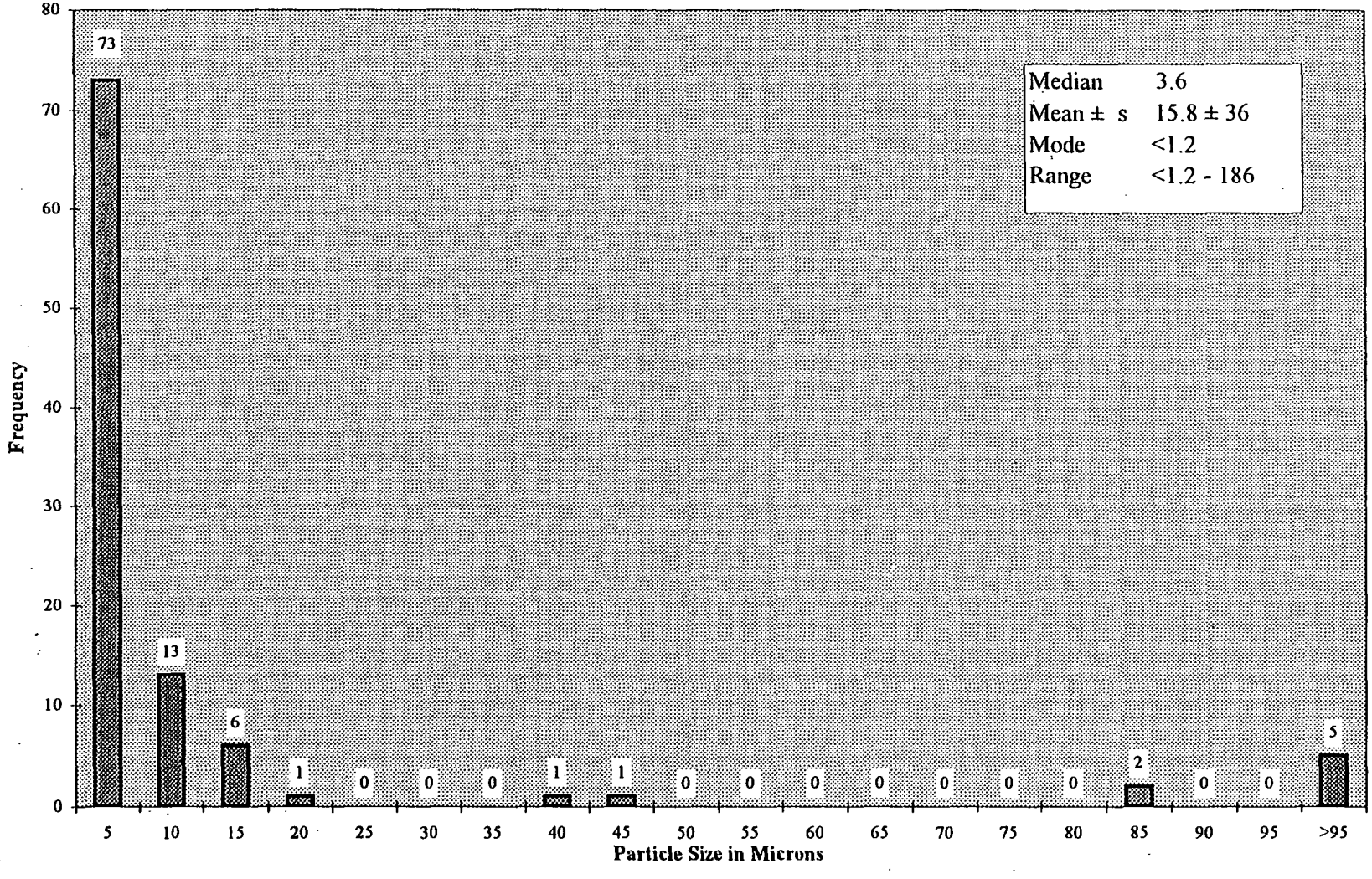
LG-Sample No. 38, Board 5 ($4in^2$)
Mortar & Pestle
Ground 1 1/2 Minutes



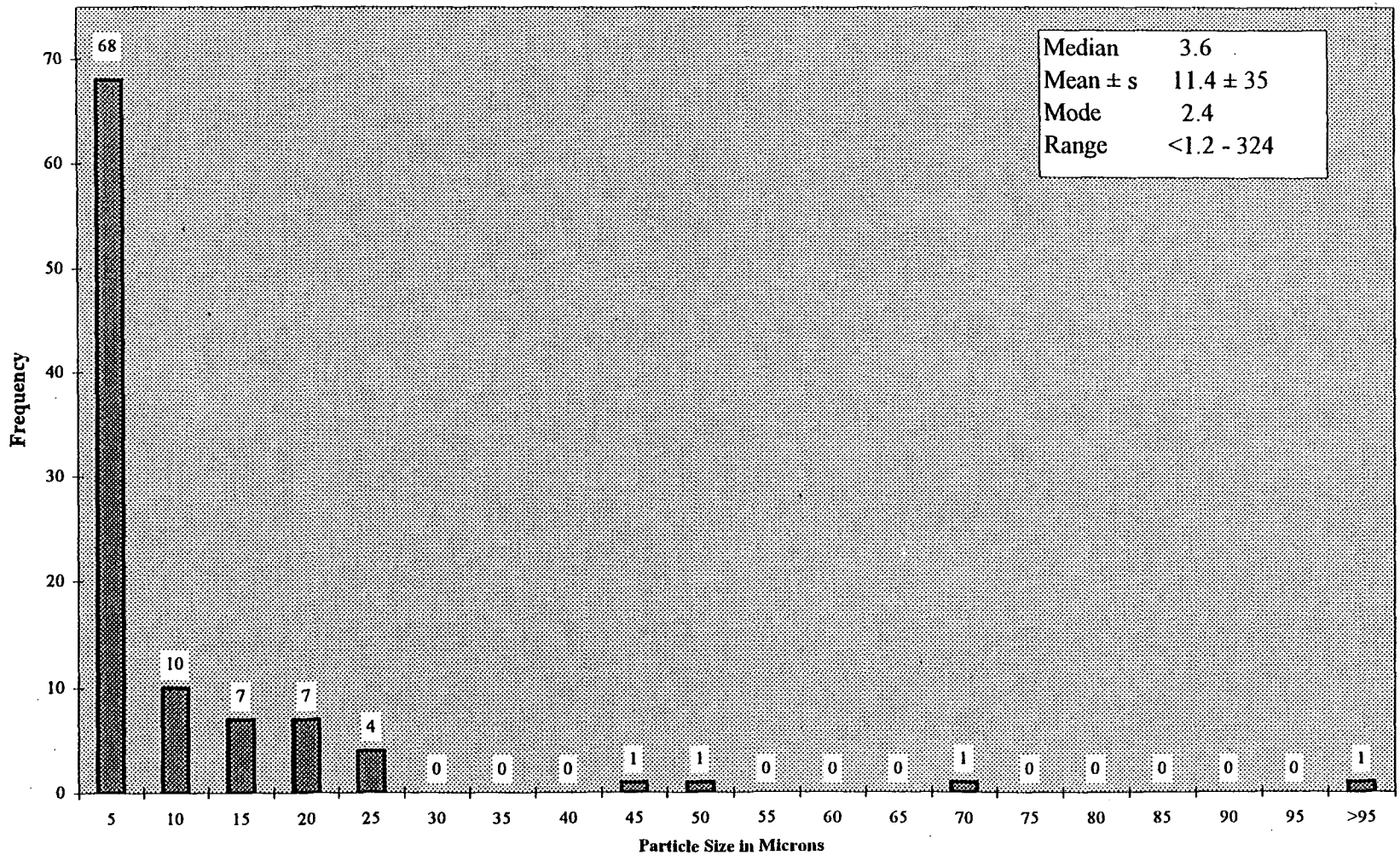
TE-Sample No. 36, Wall 7 (1in^2)
Mortar & Pestle
Ground 30 Seconds



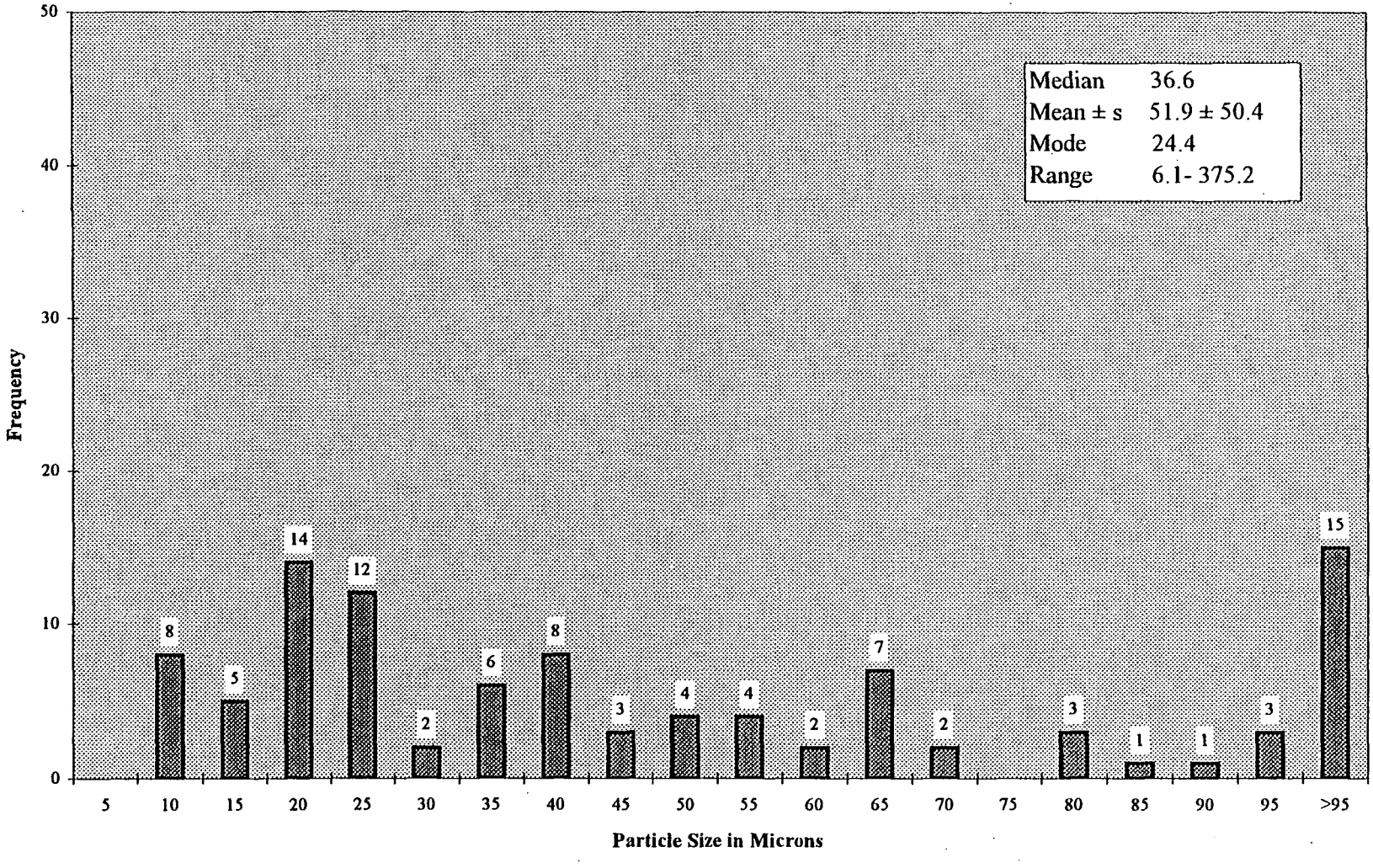
LG-Sample No. 37, Wall 7 (1in^2)
Mortar & Pestle
Ground 1 1/2 Minutes



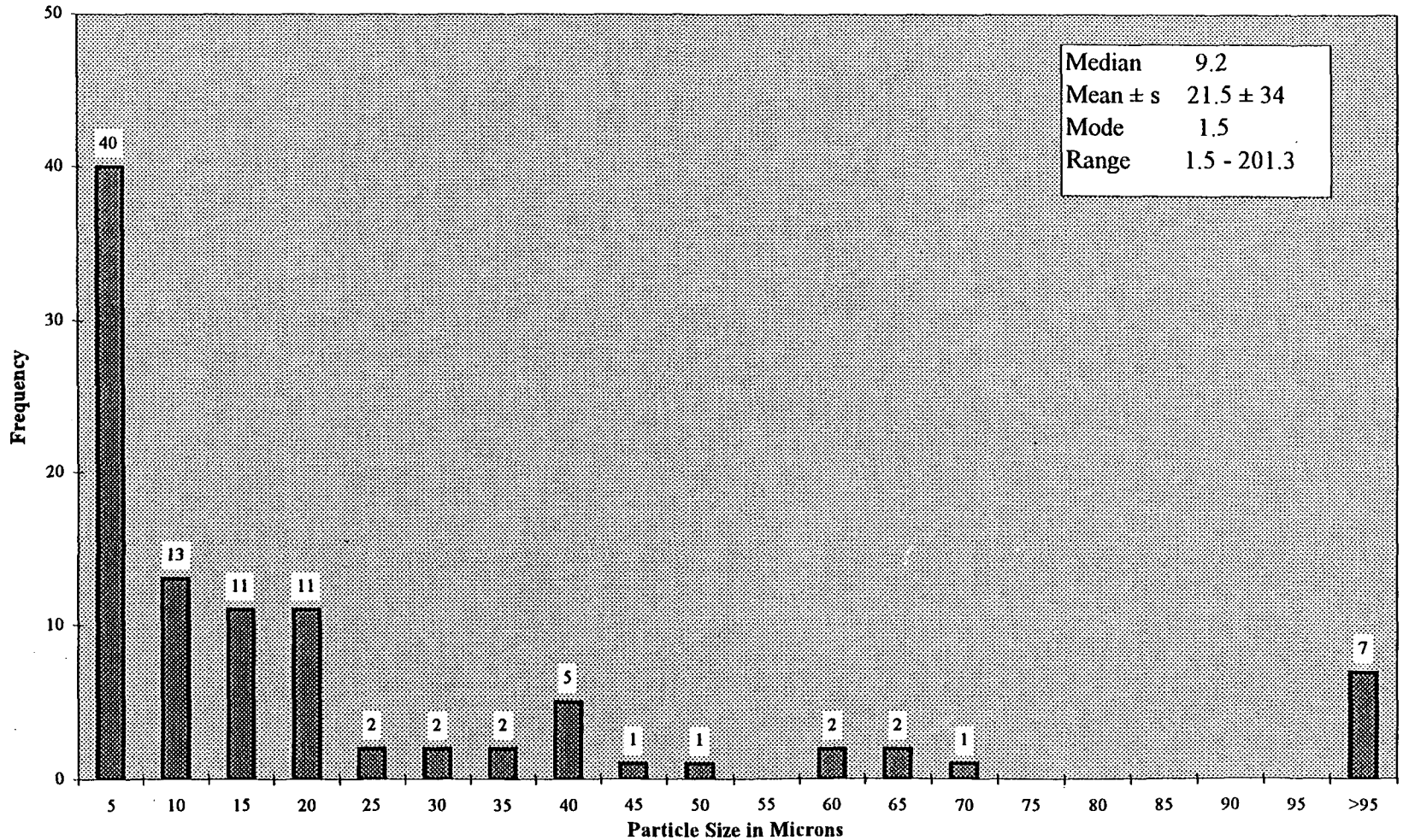
LG-Sample No. 38, Wall 7 ($4in^2$)
Mortar & Pestle
Ground 1 1/2 Minutes



TE-Sample No. 11, Board X (1in^2)
Mortar & Pestle
Ground 30 Seconds



TE-Sample No. 8, Board 5 ($4in^2$)
Glass Rod/ Room Temperature
Ground 5 Minutes, Followed by
3 Minutes with a Mortar & Pestle



LG-Sample No. 5, Wall 7 ($4in^2$)
Glass Rod/ Room Temperature
Ground 5 Minutes, Followed by
3 Minutes with a Mortar & Pestle