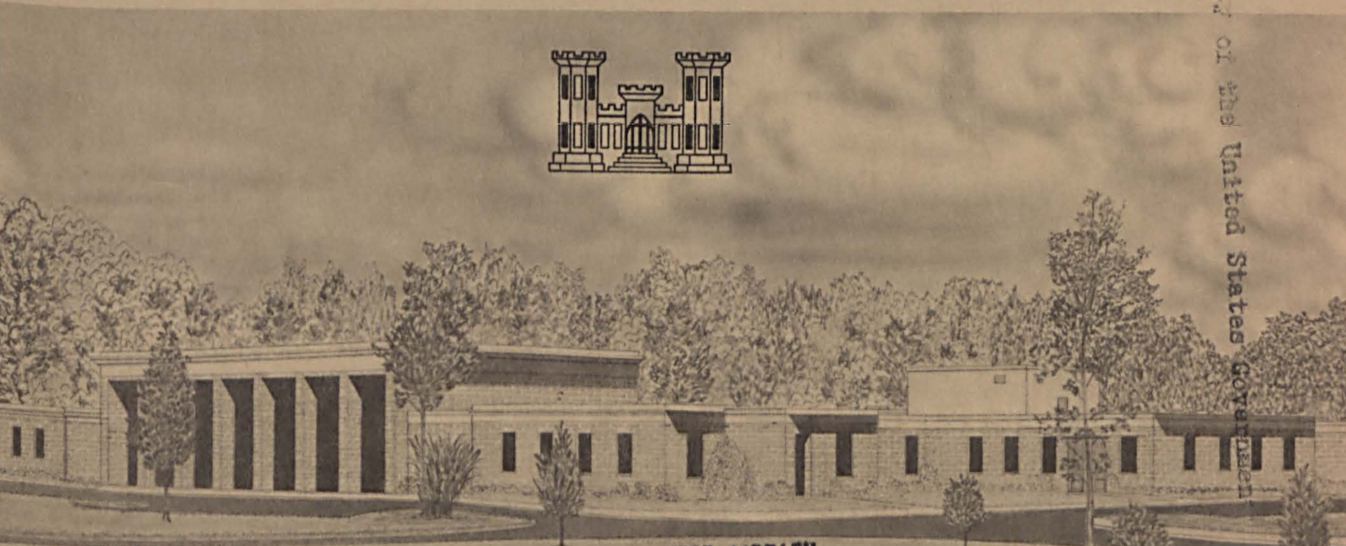
MISCELLANEOUS PAPER C-70-24

AN EVALUATION OF THE MSA METHOD FOR DETERMINING PARTICLE SIZE DISTRIBUTION

by

R. L. Curry



November 1970

Sponsored by Office, Chief of Engineers, U. S. Army

Conducted by U. S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi

MISCELLANEOUS PAPER C-70-24

AN EVALUATION OF THE MSA METHOD FOR DETERMINING PARTICLE SIZE DISTRIBUTION

by

R. L. Curry

November 1970

Sponsored by Office, Chief of Engineers, U. S. Army

Conducted by U. S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi

ARMY-MRC VICKSBURG, MISS.

THE CONTENTS OF THIS REPORT ARE NOT TO BE USED FOR ADVERTISING, PUBLICATION, OR PROMOTIONAL PURPOSES. CITATION OF TRADE NAMES DOES NOT CONSTITUTE AN OFFICIAL ENDORSEMENT OR APPROVAL OF THE USE OF SUCH COMMERCIAL PRODUCTS.

FOREWORD

This investigation was conducted under the sponsorship of the Engineer Studies Program of the Office, Chief of Engineers (ES Item No. 622.6, "Investigation of Testing Methods and Apparatus").

The work was conducted at the Concrete Division of the U. S. Army Engineer Waterways Experiment Station under the direction of Messrs. Thomas B. Kennedy, Bryant Mather, and James M. Polatty. The testing was supervised by Mr. Rembert L. Curry, who prepared this report.

CONTENTS

SUMMARY

This investigation compares fineness test results for 17 fly ashes as obtained by testing in accordance with a number of test methods. The test methods were the Blaine and Fisher air permeability methods, the Klein hydrometer and MSA centrifuging sedimentation methods, and the No. 325 sieving method.

The objectives of this investigation were to compare particle size data on the various materials as tested by the different methods for evaluating the MSA method, which requires a short time for its performance. The MSA results as compared with those of the other tests show that average particle diameters by that method generally exceed the average particle diameters by the Blaine, Fisher, and Klein hydrometer methods. Determined amounts finer than selected particle sizes, expressed as percentages, are, in general, smaller for the MSA than for the Klein hydrometer or No. 325 sieve tests.

The comparisons of test values for all of the materials were based on three rounds of tests.

Based on the comparisons and student t values, it would appear that the MSA equipment can be used to obtain particle size distribution percentages for samples of fly ash.

AN EVALUATION OF THE MSA METHOD FOR DETERMINING PARTICLE SIZE DISTRIBUTION

PART I - BACKGROUND AND PURPOSE

1. One of the problems in the study of pulverized materials has been to determine the size distribution of particles too small for analysis by conventional sieving methods. Hydrometer sedimentation by gravity, studied by Klein[1] and others, has afforded one means of determining percentages of particles on subsieve size ranges, however, there have been two main objections to this method: (a) the testing time is long when particle sizes are very small, and (b) flocculation is not easily controlled. Comparative mean particle sizes of materials determined by air permeability methods such as CRD-C 263[2] Blaine fineness, and ASTM Designation B 330 [3] Fisher subsieve fineness, and a comparative 44 micron separation as percent retained on a No. 325 sieve by method CRD-C 214,[2] have been useful. However, these tests do not measure size distribution. A method has been proposed by the Mine Safety Appliance Company[4] (MSA) whereby centrifugal force is used as a means of decreasing the time required for small particles to settle out of suspension. Centrifuges of varying speeds are utilized to accomplish settling within a matter of an hour or so that could only be accomplished in 3 or 4 days of gravity settling by the Klein hydrometer test. This investigation compares results of tests by the various methods in an effort to find the advantages and disadvantages of the MSA method. Development of techniques was sought to make the method effective for the testing encountered in concrete research.

PART II: MATERIALS

2. The materials for the investigation were taken from stocks left from research investigations carried on previously, and included 17 comparison study fly ashes, AD-273, AA through NN, Q, R, and S.

3. Each material was well blended before use in this investigation, and quantities not being actually subjected to testing were stored in closed containers.

PART III: TESTS, PROCEDURES, AND RESULTS

Tests

4. Each of the fly ashes was tested for specific gravity by method CRD-C 263.

5. Three rounds of each of the 17 fly ashes were tested using the MSA method given in Appendix A, the Klein hydrometer in Appendix B, the 325 sieve method (CRD-C 115), the Blaine fineness method (CRD-C 263), and the Fisher fineness method (ASTM 330).

Procedures

6. The MSA test method (Appendix A) recommends that the dispersion system consists of a sedimentation liquid, a dispersing agent, and a feeding liquid.* Those tests indicated that a satisfactory sedimentation liquid was distilled water, a dispersing agent of 3 drops of 25 percent aerosol and a feeding liquid of 30 percent acetone and 70 percent water. These materials were used in this investigation. It was found extremely difficult to clean the small 0.75 mm diameter capillary portions of the centrifuge tube; therefore, a tube with a 1 mm diameter capillary portion was used in this program. Tests indicated that there was no measurable difference in results using the two diameter tubes.

7. The particle size percentage values, reported for the MSA and Klein methods, were taken from gradation curves prepared from the test data. The average particle diameters were calculated by dividing 60,000 by the surface area in square centimeters per cubic centimeters.

---

* A preliminary investigation to determine what materials to use as liquids and dispersants was made; one of the problem areas being a dispersing agent that would prevent the fly ash from floculating out of suspension.

Results

8. Table 1 contains the results of the specific gravity tests of all of the materials.

9. Table 2 contains the results of the tests for average particle diameter by the MSA, Blaine, Fisher, and Klein methods.

10. Table 3 contains the results of percents finer than 44 micron diameter size by the MSA, 325 sieve, and Klein methods.

11. Table 4 contains the results of tests of percents finer by weight for 33.5, 19, 12.5, 10, 7.5, 5, 4, 3, 2, and 1 micron size particles by the MSA and Klein methods.

12. Table 5 contains student t evaluations of: (a) average particle diameter comparisons for three tests each of the 17 fly ashes; MSA versus Klein, Blaine, and Fisher methods; Klein versus Blaine and Fisher methods; and Blaine versus Fisher methods; (b) percent finer than 44 micron size comparisons for three tests of each of the 17 fly ashes; MSA versus Klein and No. 325 sieve methods, and Klein versus No. 325 sieve methods; (c) percentages finer than 33.5, 19, 12.5, 10, 7.5, 5, 4, 3, 2, and 1 micron size comparisons for three tests of each of the 17 fly ashes; MSA versus Klein methods.

13. Figures 1-15 are graphic representations of comparisons of the data in tables 2-4.

PART IV: DATA ANALYSES

14. Comparisons of average particle diameter for three tests of each of the 17 fly ashes tested in table 2 and plotted in figures 1, 2, and 3 show the following:

a. In the 51 MSA versus Blaine test comparisons, the MSA average particle diameter exceeded the Blaine value in 43 cases, whereas the Blaine value exceeded the MSA value in the other eight cases. Observing the student t values for the MSA versus Blaine comparisons in table 5, it will be noted that only for sample BB did the t value exceed the 0.01 table significance value of 4.604.

b. In the 51 MSA versus Fisher test comparisons, the MSA average particle diameter exceeded the Fisher value in 44 cases, and the Fisher value exceeded the MSA value in seven cases. Observing the student t values for the MSA versus Fisher comparisons in table 5, it will be noted that none of the values exceeded the 0.01 table significance value of 4.604.

c. In the 51 MSA versus Klein test comparisons, the MSA average particle diameter exceeds the Klein value in 46 cases and the Klein value exceeds the MSA value in five cases. Observing the student t values for the MSA versus Klein comparisons in table 5, it will be noted that the only t values exceeding 4.604 were for samples EE and S.

d. A further study of the table 5 average particle diameter data shows that, for the Klein versus Blaine comparisons, the only t value exceeding 4.604 were for samples EE and JJ, for the Klein versus Fisher comparisons the only t values exceeding 4.604 were for samples EE and HH, and for the Blaine versus Fisher comparisons of samples BB, EE, GG, HH, KK, NN, and S the t values exceeded 4.604.

15. In comparisons of percentages finer than 44 micron size for three tests each of the 17 fly ashes, listed in table 3 and plotted in figure 4, the following information is obtained:

a. MSA versus No. 325 test values compared show that in 51 comparisons 41 times the No. 325 sieve test percent finer than 44 micron size was greater than the corresponding MSA test percent and 10 times the percent finer than 44 micron size was greater by the MSA test. The table 5 MSA versus No. 325 sieve test student t values for percent finer than 44 micron size comparisons exceed 4.604 for samples AA, CC, EE, FF, II, JJ, KK, Q, and S.

b. MSA versus Klein test values compared in table 3 and figure 5 show that in 51 comparisons 26 times the percent finer than 44 micron size was greater by the Klein method, 24 times the percent was greater by the MSA method, and one time the percent by the two methods was the same. The table 5 MSA versus Klein test student t values for percent finer than 44 micron size comparisons exceed 4.604 for samples BB and EE only.

c. The table 5 Klein versus No. 325 sieve test student t values for percent finer than 44 micron size comparisons exceed 4.604 for samples AA, BB, CC, DD, GG, Q, R, and S.

16. MSA versus Klein comparisons of percentages finer than 33.5, 19, 12.5, 10, 7.5, 5, 4, 3, 2, and 1 micron sizes for three tests each of the 17 fly ashes shown in table 4 and figures 6-15 show the following:

a. For the 51 percentages finer than 33.5 micron size, comparisons the Klein values were higher 38 times and the MSA values were higher 13 times.

b. For the 51 comparisons at 19 micron size, the Klein values were higher 43 times, the MSA values were higher seven times, and the values were the same one time.

c. For the 51 comparisons at 12.5 micron size, the Klein values were higher 38 times, the MSA values were higher 10 times, and three times the values were the same.

d. For the 51 comparisons at 10 micron size, the Klein values were higher 34 times, the MSA values were higher 15 times, and two times the values were the same.

e. For the 51 comparisons at 7.5 micron size, the Klein values were higher 24 times, the MSA values were higher 24 times, and three times the values were the same.

f. For the 51 comparisons at 5 micron size, the Klein values were higher 23 times, the MSA values higher 26 times, and two times the values were the same.

g. For the 51 comparisons at 4 micron size, the Klein values were higher 22 times, the MSA values higher 26 times, and three times the values were the same.

h. For the 51 comparisons at 3 micron size, the Klein values were higher 26 times, the MSA values higher 25 times.

i. For the 51 comparisons at 2 micron size, the Klein values were higher 31 times, the MSA values higher 12 times, and eight times the values were the same.

j. For the 51 comparisons at 1 micron size, the Klein values were higher 47 times, the MSA values higher two times, and two times the values were the same.

17. The table 5 values for MSA versus Klein tests of percentages finer than the micron sizes tested show the following:

a. For 33.5 micron size comparisons, samples BB and EE had student t values exceeding 4.604.

b. For 19 micron size comparisons, samples AA, BB, DD, EE, HH, and JJ had student t values exceeding 4.604.

c. For 12.5 micron size comparisons, samples BB and EE had student t values exceeding 4.604.

d. For 10 micron size comparisons, samples BB and EE had student t values exceeding 4.604.

e. For 7.5 micron size comparisons, samples BB, EE, and JJ had student t values exceeding 4.604.

f. For 5 micron size comparisons, samples BB, EE, and JJ had student t values exceeding 4.604.

g. For 4 micron size comparisons, samples BB, EE, HH, and JJ had student t values exceeding 4.604.

h. For 3 micron size comparisons, samples EE and JJ had student t values exceeding 4.604.

i. For 2 micron size comparisons, samples CC, EE, and JJ had student t values exceeding 4.604.

j. For 1 micron size comparisons, samples CC, EE, GG, and R had student t values exceeding 4.604.

PART V: CONCLUSIONS

18. This investigation used two methods (Klein and MSA) of determining a complete particle distribution for materials of predominantly -100 sieve size. In addition, percentages passing the 325 sieve, using a mechanical sieving test sieve and the average particle diameter as calculated from the Blaine and Fisher procedure, were used to evaluate the two basic methods.

19. The results indicate for the two basic methods:

a. For the three tests of each of the 17 fly ash specimens, in the majority of cases, the micron sizes indicate that the materials were finer by the Klein method than by the MSA method. The student t test indicated that the variation between test results for the 10 micron sizes for the 51 tests did not, in the great majority of results, indicate a probability of significant difference except for three of the 17 fly ashes; however, the variation between the Klein and MSA results were greater in the 19, 4, and 1 micron sizes.

b. Using the mean particle diameters as calculated from the Blaine, Klein, Fisher, and the MSA methods, for the three tests of each of 17 fly ashes, the MSA results were higher, except for two of the materials, than those obtained by the other three methods. In addition, the variation in individual results for the Klein and MSA methods were greater than for the other two methods.

c. A student t test analysis of the mean particle diameter for the 51 fly ash test indicated that the probability of variation was usually not significant.

d. A study of the results of a mechanical sieving test, using a No. 325 sieve (44 microns) for three tests of each of the 17 fly ashes, indicated that the student t probability for variation was significant when compared with either the Klein or MSA values; however, the difference between the Klein and MSA values were not significant.

20. Based on the results of this study, it would appear that the MSA equipment can be used to obtain particle size distribution percentages for samples of fly ash.

Literature Cited

1. Klein, Alexander, Surface - Increment Scale Hydrometer Instructions For Use, Berkeley, California, January 1946.
2. U. S. Army Engineer Waterways Experiment Station, CE, Handbook For Concrete and Cement, with quarterly supplements, Vicksburg, Miss., August 1949.
3. American Society for Testing and Materials, 1958 Book of ASTM Standards; Part 3, Methods of Testing Metals (Except Chemical Analysis), Philadelphia, Pa., December 1958.
4. Mine Safety Appliances Company, MSA Particle-Size Analyses Operating Procedures and Applications, Pittsburgh,Pa., January 1959.

TABLE 1 - SPECIFIC GRAVITY OF TEST FLY ASHES

Designation	Specific Gravity
AD-273 AA	2.60
AD-273 BB	2.39
AD-273 CC	2.43
AD-273 DD	2.55
AD-273 EE	2.50
AD-273 FF	2.40
AD-273 GG	2.32
AD-273 HH	2.29
AD-273 II	2.55
AD-273 JJ	2.42
AD-273 KK	2.42
AD-273 LL	2.20
AD-273 MM	2.27
AD-273 NN	2.18
AD-273 Q	2.46
AD-273 R	2.50
AD-273 S	2.47

TABLE 2 - AVERAGE PARTICLE DIAMETER, MICRONS
MSA, BLAINE, FISHER, KLEIN TESTS, 17 FLY ASHES

	Average Particle Diameter in Microns															
	MSA Fineness				Blaine Fineness				Fisher Fineness				Klein Fineness			
Sample	Run 1	Run 2	Run 3	Avg	Run 1	Run 2	Run 3	Avg	Run 1	Run 2	Run 3	Avg	Run 1	Run 2	Run 3	Avg
AD-273 AA	8.2	9.0	10.1*	9.1	6.7	6.7	6.8	6.7	6.7	6.8	6.7	6.7	5.1	5.1	8.9*	5.1
AD-273 BB	11.5	9.9	8.5	9.2	16.6	16.3	16.2	16.4	19.0	19.0	17.7	18.6	8.5	9.5	14.8*	9.0
AD-273 CC	9.4	15.6*	11.1	10.2	7.9	7.8	8.0	7.9	7.4	7.8	7.4	7.5	7.3	8.9	4.4*	8.1
AD-273 DD	9.3	12.9*	10.1	9.7	8.3	8.5	8.4	8.4	8.3	8.4	8.2	8.3	13.7*	5.7	9.5	7.6
AD-273 EE	12.3	8.9*	14.6	13.4	5.9	5.9	5.9	5.9	5.8	5.8	5.8	5.8	2.7	2.7	2.7	2.7
AD-273 FF	10.5	10.3	10.5	10.4	9.0	9.4	9.9	9.4	9.4	9.8	9.0	9.4	7.4	5.6	10.4*	6.5
AD-273 GG	9.0	7.9	15.0*	8.4	6.7	6.9	7.0	6.9	4.7	4.8	4.5	4.7	6.4	5.8	8.7*	6.1
AD-273 HH	9.2*	14.0	15.6	14.8	5.5	5.4	5.5	5.5	4.8	4.9	4.9	4.9	11.6	10.5	13.1*	11.0
AD-273 II	8.8	11.2*	7.3	8.0	7.0	7.0	7.4	7.1	7.0	7.0	6.8	6.9	4.3	5.3	6.8*	4.8
AD-273 JJ	10.0	14.2	21.9*	12.1	6.5	6.3	6.3	6.4	6.2	6.3	6.1	6.3	2.8*	3.2	3.2	3.2
AD-273 KK	7.9*	12.3	12.1	12.2	7.2	7.0	7.4	7.2	5.6	5.7	5.5	5.6	4.6	6.6	8.8*	5.6
AD-273 LL	5.1*	7.7	7.0	7.4	6.9	6.7	6.6	6.7	6.6	6.6	6.6	6.6	4.3	6.0	11.1*	5.2
AD-273 MM	4.6*	6.1	6.5	6.3	6.6	7.8	7.7	7.4	7.4	7.4	7.2	7.3	11.0*	4.2	5.7	5.0
AD-273 NN	12.1	20.4*	13.8	13.0	10.1	10.0	10.4	10.2	5.8	6.0	5.8	5.9	5.9	7.2	11.4*	6.6
AD-273 Q	6.4	7.7	9.5*	7.0	5.0	5.3	5.0	5.1	5.1	5.2	4.9	5.1	5.1	5.3	6.2*	5.2
AD-273 R	5.0	6.6	22.6*	5.8	4.5	4.6	4.6	4.6	4.5	4.5	4.4	4.5	2.9	3.1	3.9*	3.0
AD-273 S	6.3	7.3	5.5*	6.8	4.0	4.2	4.0	4.1	3.6	3.6	3.6	3.6	3.0	3.6*	2.5	2.8

* Values varied more than 10 percent from average of 3 and were not used in final averaging for the test methods.

TABLE 3 - PERCENT FINER THAN 44 MICRON DIAMETER
MSA, 325 SIEVE, AND KLEIN TESTS, 17 FLY ASHES

	Percent by Weight Finer Than 44 Micron Diameter Size											
	MSA Test				325 Sieve Test				Klein Test			
Sample	Run 1	Run 2	Run 3	Avg	Run 1	Run 2	Run 3	Avg	Run 1	Run 2	Run 3	Avg
AD-273 AA	83.0	84.2	83.0	83.4	94.2	94.2	94.7	94.4	91.8	85.0	80.9	85.9
AD-273 BB	80.1	74.5	79.0	77.9	70.8	72.8	70.8	71.5	65.6*	54.5	52.6	53.6
AD-273 CC	81.8	80.0	84.2	82.0	88.6	88.0	88.3	88.3	72.8*	82.0	82.0	82.0
AD-273 DD	73.0	70.2	80.2*	71.6	87.6	87.8	87.7	87.7	70.0*	78.3	82.5	80.4
AD-273 EE	86.5	82.7	84.2	84.5	97.5	97.2	97.2	97.3	92.0	92.0	98.4	94.1
AD-273 FF	76.0	78.3	72.0	75.4	88.3	87.6	87.5	87.8	82.0	82.0	80.0	81.3
AD-273 GG	88.3	85.5	83.8	85.9	84.2	85.3	85.8	85.1	83.0	81.0	81.0	81.7
AD-273 HH	81.5	74.9	74.0	76.8	86.9	86.5	85.8	86.4	81.5	82.0	73.0*	81.8
AD-273 II	83.0	83.0	84.0	83.3	90.1	90.0	89.5	89.9	78.8	82.0	74.5	78.4
AD-273 JJ	75.6	73.7	73.0	74.1	87.7	87.4	88.2	87.8	82.0	70.0*	78.0	80.0
AD-273 KK	80.2	76.7	75.5	77.5	88.5	88.2	87.5	88.1	84.0	80.0	80.0	81.3
AD-273 LL	89.7	82.5	89.7	87.3	89.1	89.7	89.4	89.4	84.8	86.0	84.0	84.9
AD-273 MM	93.0	88.5	89.5	90.3	91.8	91.2	90.8	91.3	86.2	86.0	93.5	88.6
AD-273 NN	84.2	79.8	84.2	82.7	82.9	83.4	81.1	82.5	81.5	84.5	81.5	82.5
AD-273 Q	91.8	90.0	89.0	90.3	94.3	94.1	94.2	94.2	88.2	92.0	92.0	90.7
AD-273 R	94.0	87.5	93.0	91.5	95.7	95.6	95.4	95.6	95.8	90.0	91.8	92.5
AD-273 S	91.0	87.5	90.5	89.7	95.6	95.6	95.6	95.6	90.5	90.2	91.2	90.6

* Values varied more than 10 percent from the average of the three test values and were not used in the final averaging for the test methods.

TABLE 4 - PERCENTS FINER THAN MICRON SIZES MSA VERSUS KLEIN TESTS, 17 FLY ASHES

		Percent by Weight Finer Than Indicated Micron Sizes							
	Diam	MSA Test				Klein Test			
Sample	Microns	Test 1	Test 2	Test 3	Avg	Test 1	Test 2	Test 3	Avg
AD-273 AA	33.5	75	76	75	75	84	78	76	79
	19	59	51	43*	55	64	60	59	61
	12.5	43*	35	28	32	49*	41	39	40
	10	36*	28	23	26	38*	30	29	30
	7.5	29*	21	18	20	25*	20	18	20
	5	19*	13	12	12	16	12	8	12
	4	14*	10	10	10	13	10	5*	12
	3	9*	7	6	6	11	8	3*	10
	2	4	3	4	4	8	7	3*	8
	1	0	0	0	0	5	6	2*	6
AD-273 BB	33.5	67	73	67	69	52*	38	40	39
	19	49	56*	46	48	28*	17	18	18
	12.5	35	40*	36	36	16*	8	8	8
	10	29	33	31	31	11*	5	5	5
	7.5	23	25	26	25	7*	4	3	4
	5	15	17	19	17	6	4	2	4
	4	11	13	15	13	5	4	2*	4
	3	6	8	12*	7	4	4	2*	4
	2	0*	4	7	6	4	4	2*	4
	1	0	0	0	0	4	4	2*	4
AD-273 CC	33.5	65	60*	67	66	72	67	72	70
	19	44*	35	39	37	51	47	51	50
	12.5	32	22*	30	31	36	34	38	36
	10	27	16*	26	26	28	28	31*	28
	7.5	21	11*	21	21	21	21	24	22
	5	14	5*	14	14	14	14	16*	14
	4	11	4*	11	11	12	11	13	12
	3	8	3*	8	8	10	8*	11	10
	2	4	3*	4	4	8	5*	9	8
	1	0	3*	0	0	8	2*	8	8
AD-273 DD	33.5	60	64	64	63	67	76*	70	68
	19	37	34	37	36	49	54	51	51
	12.5	23	20*	23	23	27*	34	34	34
	10	19	17*	19	19	19*	26	26	26
	7.5	15	13*	16	16	12*	18	17	18
	5	12	9*	13	12	6*	12	10	11
	4	10	8*	11	10	4*	10	8	9
	3	6*	7	9	8	2*	8	7	8
	2	5	4	6	5	1*	6	5	6
	1	0	0	0	0	0*	5	5	5

*Value varies more than 10 percent from average of closest two and was not used to compute average percent finer than indicated micron size.

(Continued)

Table 4
(Continued)

Sample	Diam Microns	MSA Test: Test 1	MSA Test: Test 2	MSA Test: Test 3	MSA Test: Avg	Klein Test: Test 1	Klein Test: Test 2	Klein Test: Test 3	Klein Test: Avg
		Percent by Weight Finer Than Indicated Micron Sizes							
AD-273 EE	33.5	73	72	76	74	94	88	87	89
	19	46*	28	34	31	76	74	72	74
	12.5	32*	14	19	16	55	59	56	57
	10	27*	10	14	12	47	48	47	47
	7.5	20*	6	10	8	37	37	37	37
	5	13*	2	5	4	26	27	26	26
	4	9*	1	4	2	22	22	22	22
	3	6*	0	2	1	18	18	18	18
	2	3*	0	1	0	15	15	15	15
	1	0	0	0	0	14	13	13	13
AD-273 FF	33.5	55	69*	53	54	75	74	72	74
	19	37	42	29*	40	51	51	51	51
	12.5	27	29	20*	28	27	29	30	29
	10	23	25	17*	24	20	21	20	20
	7.5	19	19	13*	19	14	13	11*	14
	5	14	14	9*	14	9	9	6*	9
	4	11	11	7*	11	8	8	5*	8
	3	9	9	5*	9	6	7	4*	6
	2	5	5	3*	5	5	6	3*	6
	1*	0	0	0	0	3	3	2*	3
AD-273 GG	33.5	82	78	75	78	78	76	76	77
	19	57	54	36*	56	62	59	58	60
	12.5	35	36	19*	36	43	41	38	41
	10	27	30	15*	28	30	30	24*	30
	7.5	21	24	10*	22	18	20	14*	19
	5	15	17	5*	16	10	12	6*	11
	4	12	14	4*	13	8	9	4*	8
	3	8	10	2*	9	6	7	3*	6
	2	4	5	1*	4	5	5	3*	5
	1	0	0	0	0	2	4	2	3

*Value varies more than 10 percent from average of closest two and was not used to compute average percent finer than indicated micron size.

(Continued)

Table 4
(Continued)

Sample	Diam Microns	MSA Test: Test 1	MSA Test: Test 2	MSA Test: Test 3	MSA Test: Avg	Klein Test: Test 1	Klein Test: Test 2	Klein Test: Test 3	Klein Test: Avg
		Percent by Weight Finer Than Indicated Micron Sizes							
AD-273 HH	33.5	73*	49	45	47	77	68*	80	78
	19	44*	29	27	28	58	52*	64	61
	12.5	29*	21	19	20	31	33	36*	32
	10	23*	18	16	17	20	22	22	21
	7.5	18*	14	13	14	12	13	13	13
	5	12*	9	8	8	6	6	4*	6
	4	10*	8	7	8	4	4	2*	4
	3	7*	5	5	5	3	3	1*	3
	2	4	3	2	3	2	3	1	2
	1	0	0	0	0	1	2	0	1
AD-273 II	33.5	75	73	77	75	73	75	68	72
	19	52	40*	56	54	56	56	52	55
	12.5	37	27*	44	40	41	39	37	39
	10	31	23*	38	34	32	32	30	31
	7.5	24	18	32*	21	23	24	22	23
	5	17	12	23*	15	15	16	13*	16
	4	13	10	19*	12	13	13	10*	13
	3	10	7*	14	12	11	10	8*	10
	2	6	4	8	6	9	7	5	7
	1	0	0	0	0	8	6	4	6
AD-273 JJ	33.5	64	61	55*	62	81	70*	77	79
	19	43	35	19*	39	71	65	66	67
	12.5	30	21	12	21	41*	52	50	51
	10	25	17	9	17	35*	41	41	41
	7.5	20*	13	7	10	30	32	32	31
	5	14*	8	4	6	23	23	24	23
	4	11*	6	3	4	20	20	21	20
	3	8*	4	1	2	17	16	17	17
	2	5*	2	1	2	14	12*	14	14
	1	0	0	0	0	2	4*	2	2
AD-273 KK	33.5	69	63	57	63	73	74	76	74
	19	47*	38	35	36	48	52	55	52
	12.5	33	31	22*	32	28	30	32	30
	10	28*	20	18	19	21	22	25*	22

*Value varies more than 10 percent from average of closest two and was not used to compute average percent finer than indicated micron size.

(Continued)

Table 4
(Continued)

Sample	Diam Microns	MSA Test: Test 1	MSA Test: Test 2	MSA Test: Test 3	MSA Test: Avg	Klein Test: Test 1	Klein Test: Test 2	Klein Test: Test 3	Klein Test: Avg
AD-273 KK	7.5	23*	16	14	15	16	17	17	17
	5	17*	11	9	10	11	11	10	11
	4	14*	8	7	8	10	10	8*	10
	3	11*	6	5	6	8	8	6*	8
	2	7*	4	3	4	8	7	4*	8
	1	3*	1	0	1	8	6	3*	7
AD-273 LL	33.5	85	73*	83	84	79	79	77	78
	19	74*	50	58	54	56	59	58	58
	12.5	61*	35	39	37	34*	38	38	38
	10	53*	30	33	32	25	27	28	27
	7.5	41*	24	26	25	17*	20	19	20
	5	29*	18	20	19	12	13	11	12
	4	23*	15	16	16	11	12	8*	12
	3	17*	11	13	12	9	9	6*	9
	2	11*	7	8	8	7	7	4*	7
	1	3	3	3	3	6	5	4	5
AD-273 MM	33.5	89	82	84	85	85	83	89	86
	19	77*	59	67	63	71	70	70	70
	12.5	62*	39	48	44	43*	53	48	50
	10	53*	33	39	36	31*	42	37	40
	7.5	41*	27	30	28	21*	24	26	25
	5	30*	20	21	20	13	14	16*	14
	4	26*	16	16	16	10	11	13*	10
	3	20*	13	12	12	7	8	10*	8
	2	14*	9	7	8	4*	7	7	7
	1	5	3	0*	4	2	7	4	3
AD-273 NN	33.5	75*	58	66	62	73	76	65*	74
	19	43	21*	36	40	47	50	31*	48
	12.5	25	13*	20	22	27	27	17*	27
	10	20	10	15	15	19	19	11*	19
	7.5	15	7	11	11	13	13	7*	13

*Value varies more than 10 percent from average of closest two and was not used to compute average percent finer than indicated micron size.

(Continued)

Table 4
(Continued)

Sample	Diam Microns	Percent by Weight Finer Than Indicated Micron Sizes — MSA Test: Test 1	Test 2	Test 3	Avg	Klein Test: Test 1	Test 2	Test 3	Avg
AD-273 NN	5	9*	4	6	5	10	10	3*	10
	4	7*	3	4	4	9	9	2*	9
	3	5	1	3	3	8	8	2*	8
	2	2	1	1	1	7	6	2*	6
	1	0	0	0	0	6	4	2	4
AD-273 Q	33.5	87	84	83	85	86	88	88	87
	19	70*	60	60	60	76	75	75	75
	12.5	57*	38	44	41	65	63	62	63
	10	50*	30	37	34	58	55	54	56
	7.5	39*	22	28	25	48	44	41	44
	5	25*	13	17	15	34	30	23*	32
	4	19*	9	13	11	28	23	17*	26
	3	13*	6	8	7	20	16	11*	18
	2	7*	2	3	2	12	9	5*	10
	1	0	0	0	0	3	2	1	2
AD-273 R	33.5	91	85	87	88	94	89	89	91
	19	79	15*	68	74	84	81	79	81
	12.5	67	10*	50	58	73	68	68	70
	10	60	8*	42	51	64	60	61	62
	7.5	48	5*	33	40	52*	45	49	47
	5	33	3*	23	28	38*	29	25	27
	4	27	2*	19	23	31	24	16*	27
	3	19	1*	13	16	23	18	10*	20
	2	11	0*	7	9	15	13	7*	14
	1	0	0	0	0	8	10	6*	9
AD-273 S	33.5	86	81	89	85	90	90	92	91
	19	70	57*	78	74	85	84	86	85
	12.5	56	40*	64	60	78	76	78	77
	10	44	33	56*	38	73	71	71	72
	7.5	35	26	47*	30	66	64	61	64
	5	24	18	32*	21	52	48	39*	50
	4	19	14	26*	16	40	36	23*	38
	3	14	10	18	14	25	25	12*	25
	2	7	5	10*	6	14	14	6*	14
	1	0	0	0	0	12*	5	6	6

*Value varies more than 10 percent from average of closest two and was not used to compute average percent finer than indicated micron size.

TABLE 5 - STUDENT t EVALUATIONS OF RESULTS OF COMPARATIVE TESTS OF 17 FLY ASHES

Student t Evaluations	AD-273 Fly Ashes																
	AA	BB	CC	DD	EE	FF	GG	HH	II	JJ	KK	LL	MM	NN	Q	R	S
Avg Part. Diam																	
MSA vs Klein	1.95	0.42	2.24	0.91	5.53	1.77	1.49	1.17	2.66	3.51	2.18	0.28	0.62	2.37	2.48	1.43	5.54
MSA vs Blaine	4.31	7.29	2.20	3.29	3.61	3.70	1.67	3.83	1.73	2.57	2.49	0.09	2.44	2.05	3.08	1.21	4.38
MSA vs Fisher	4.37	2.70	2.42	2.28	3.67	4.06	2.40	4.14	1.93	2.63	3.61	0.15	2.74	3.74	3.10	1.22	4.38
Klein vs Blaine	0.23	2.79	0.76	0.52	[illegible]	1.12	0.00	2.59	1.78	21.2	0.41	0.20	0.95	0.22	1.19	4.28	3.35
Klein vs Fisher	2.36	3.84	0.45	0.56	[illegible]	1.12	2.46	9.00	1.92	2.10	0.90	0.24	1.93	1.38	1.13	3.84	1.88
Blaine vs Fisher	0.00	5.36	3.05	0.61	[illegible]	0.00	8.38	10.6	4.14	2.22	11.5	1.12	0.25	30.9	0.00	1.71	7.02
% Finer Than 44 Micron Size																	
MSA vs Klein	0.31	13.4	0.00	1.83	4.66	2.93	3.52	0.55	2.26	0.60	1.86	0.93	0.35	0.06	1.40	0.05	0.88
MSA vs 325 S. Test	25.3	3.47	5.12	4.42	11.5	6.64	0.57	4.01	14.7	16.9	7.34	0.87	0.71	0.12	4.72	2.04	5.37
Klein vs 325 S. Test	6.74	20.5	14.0	5.97	1.38	1.08	11.6	2.50	3.03	3.83	1.77	0.72	0.85	0.27	5.88	5.56	16.6
% Finer Than Micron Sizes																	
MSA vs Klein																	
33.5 Micron Size	1.26	5.56	2.83	3.77	5.91	2.72	0.50	2.63	1.25	3.83	3.15	0.53	0.25	0.84	1.89	1.23	2.22
19 Micron Size	5.59	10.8	4.46	11.4	6.79	3.94	1.80	4.99	1.08	4.78	2.81	0.42	0.50	1.07	3.57	1.37	2.71
12.5 Micron Size	1.27	9.60	2.96	3.80	6.31	1.27	1.68	3.23	0.59	4.30	0.36	0.90	0.23	0.94	2.98	1.61	3.37
10 Micron Size	0.62	10.4	1.91	2.61	5.83	0.71	0.40	1.05	0.14	4.36	0.21	1.64	0.74	0.32	1.52	1.64	4.08
7.5 Micron Size	0.59	12.4	1.53	0.32	5.98	2.23	0.67	1.46	0.41	4.69	0.36	2.12	1.99	0.00	2.73	1.56	4.42
5 Micron Size	1.16	7.73	1.39	1.44	5.85	2.72	1.24	3.63	0.79	4.98	0.66	2.99	2.84	0.51	2.23	1.14	3.86
4 Micron Size	1.42	7.78	1.69	0.51	7.38	2.24	1.38	5.06	0.35	5.73	0.17	2.02	2.32	0.76	2.08	0.90	2.14
3 Micron Size	0.57	3.17	2.58	1.58	7.68	2.00	1.03	4.21	0.27	6.01	0.00	2.37	2.88	1.29	2.00	0.92	1.36
2 Micron Size	0.32	0.48	10.5	0.89	15.5	0.64	0.29	2.53	0.61	7.69	0.94	1.71	1.73	2.35	1.85	1.41	1.31
1 Micron Size	3.00	4.02	7.00	2.31	39.6	4.60	∞	0.89	2.59	4.02	2.58	3.44	0.77	3.46	3.44	6.91	3.50

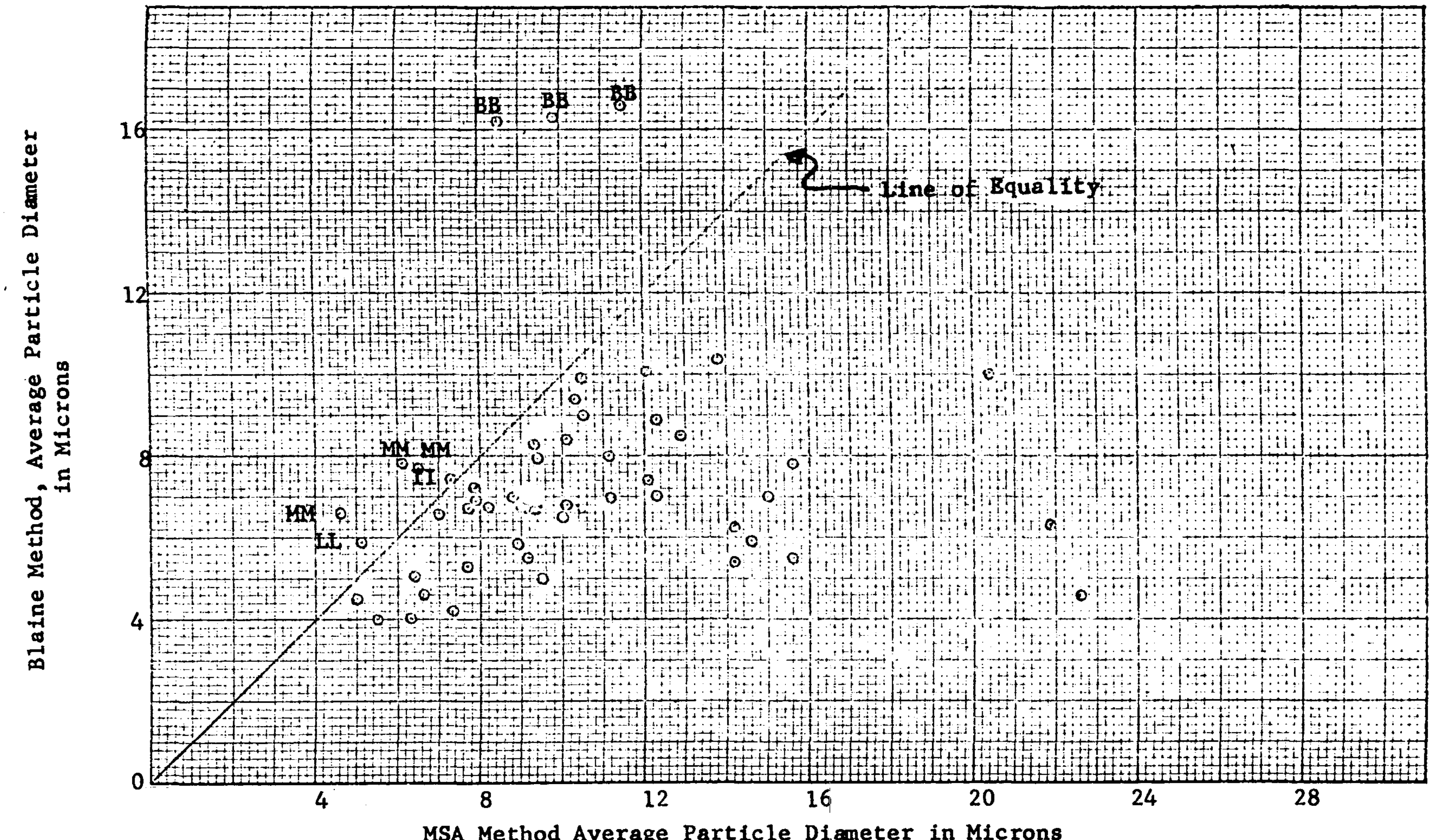


Figure 1. Average particle diameter comparisons, MSA vs Blaine methods, 17 fly ashes, three tests each by each method (table 2).

WES FORM NO. 1780
JULY 1968

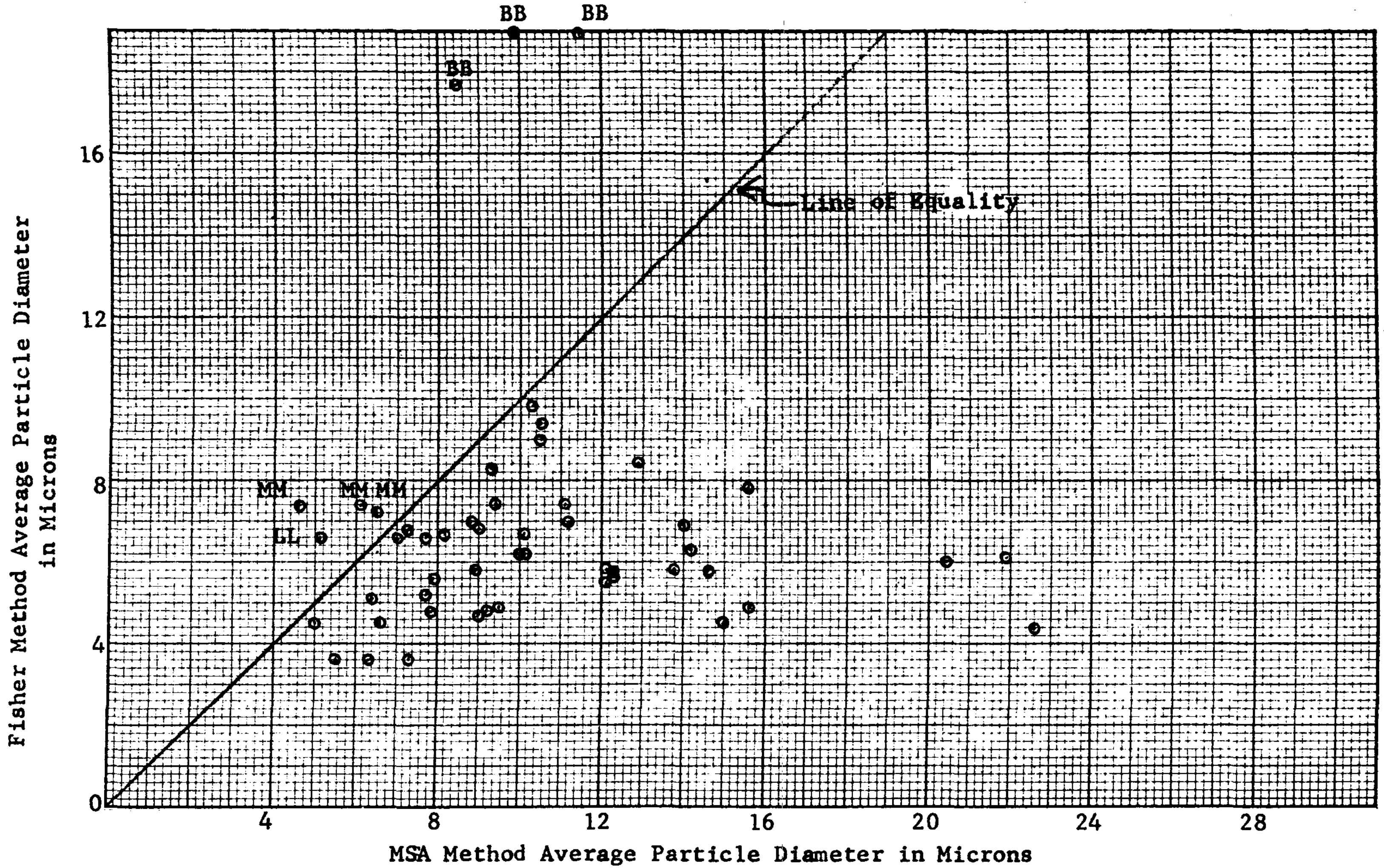


Figure 2. Average particle diameter comparisons, MSA vs Fisher methods, 17 fly ashes, three tests each by each method (table 2).

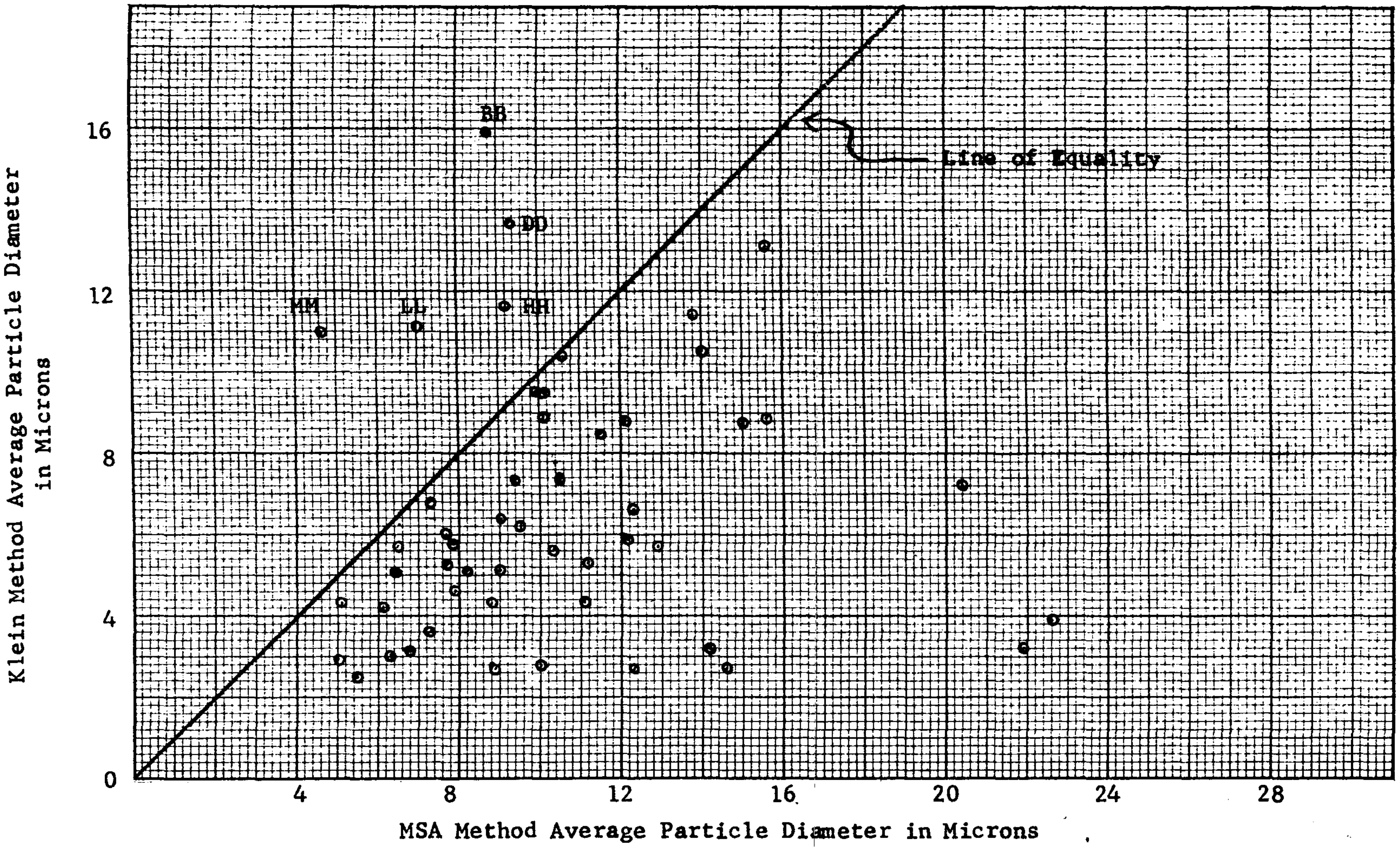


Figure 3. Average particle diameter comparisons, MSA vs Klein methods, 17 fly ashes, three tests each by each method (table 2).

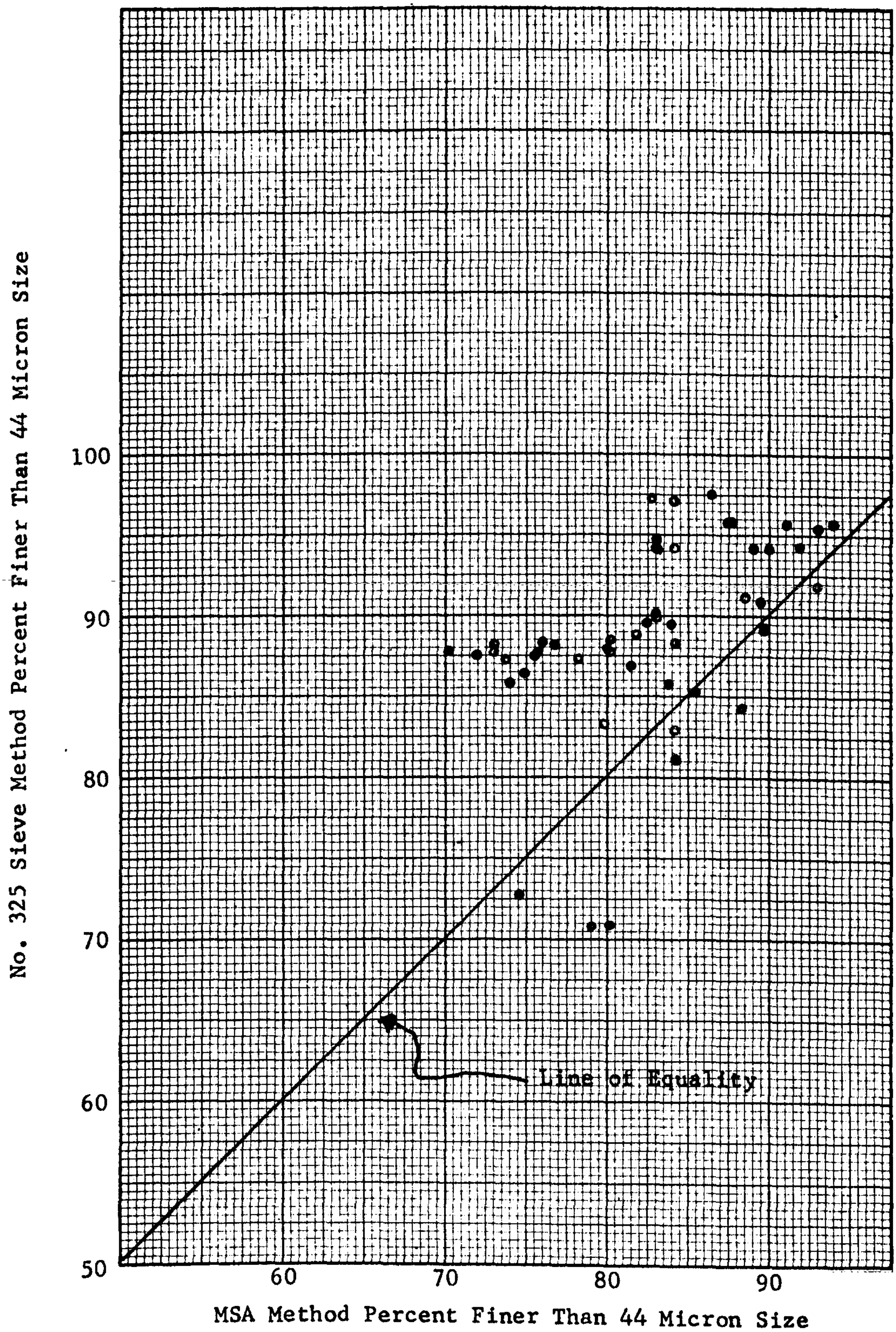


Figure 4. Percent finer than 44 micron size comparisons, MSA vs 325 sieve methods, 17 fly ashes, three tests each by each method (table 3).

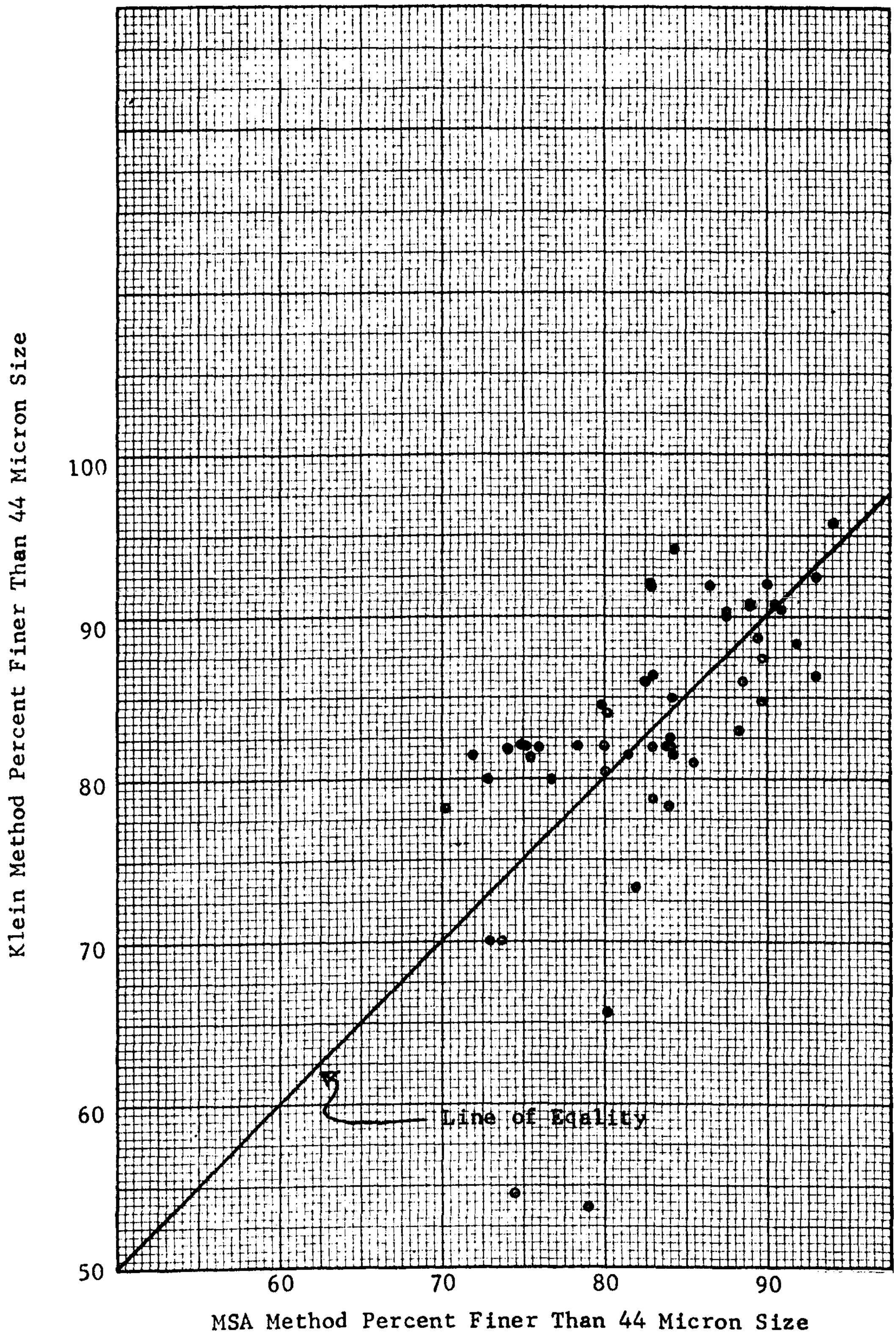


Figure 5. Percent finer than 44 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests each by each method (table 3)

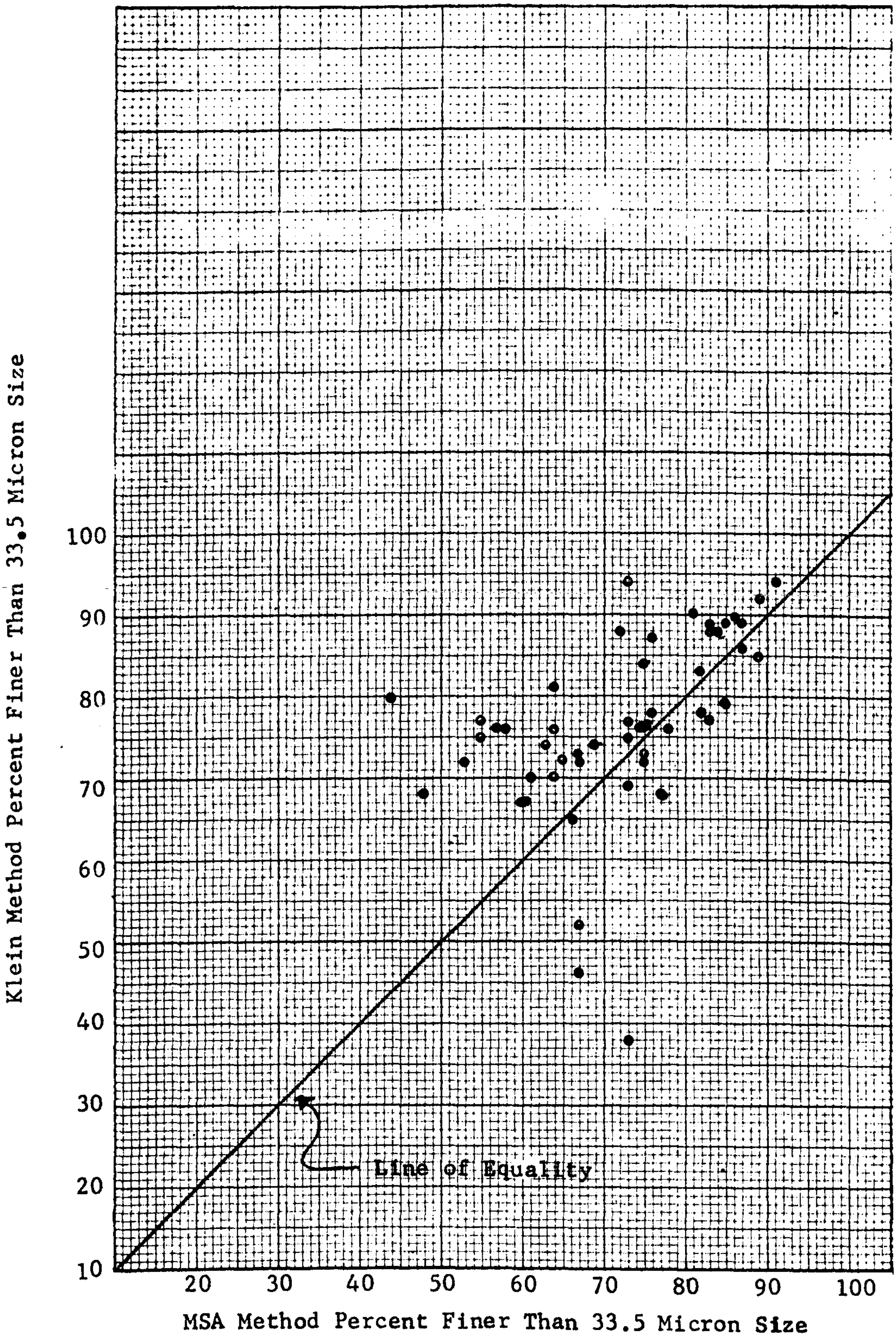


Figure 6. Percent finer than 33.5 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests each by each method (table 4).

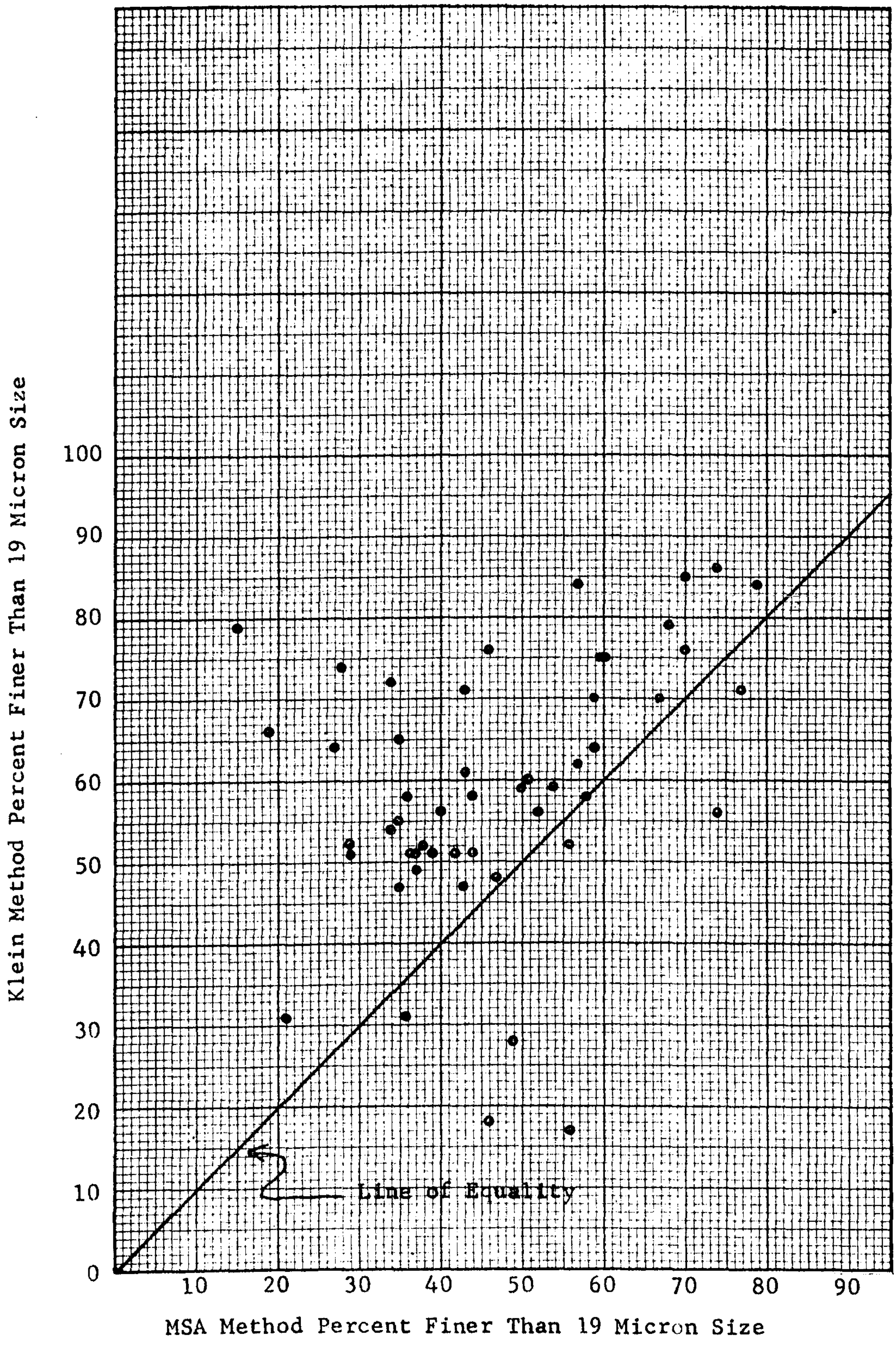


Figure 7. Percent finer than 19 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests each by each method (table 4).

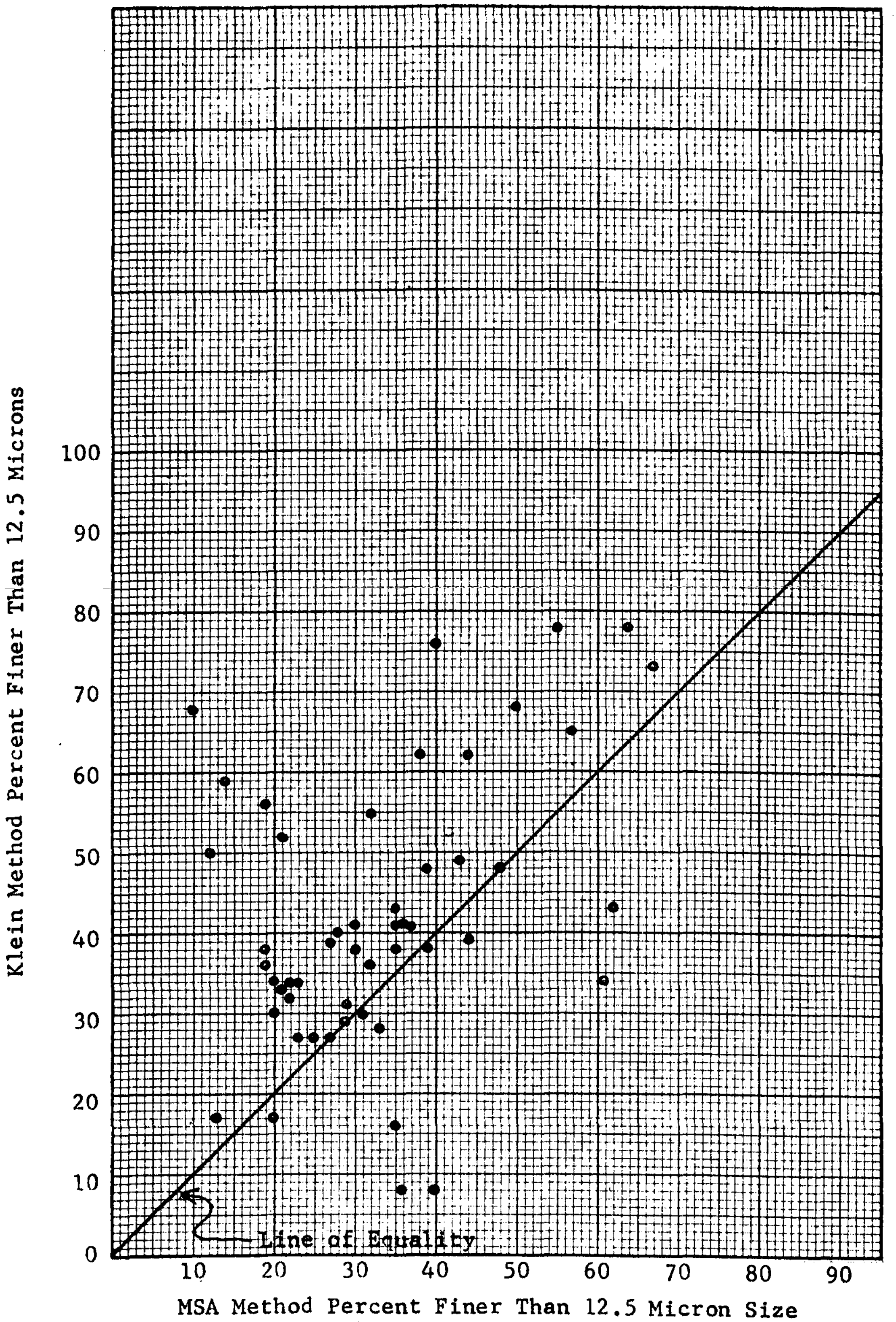


Figure 8. Percent finer than 12.5 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4).

WES FORM NO. 1779
JULY 1968

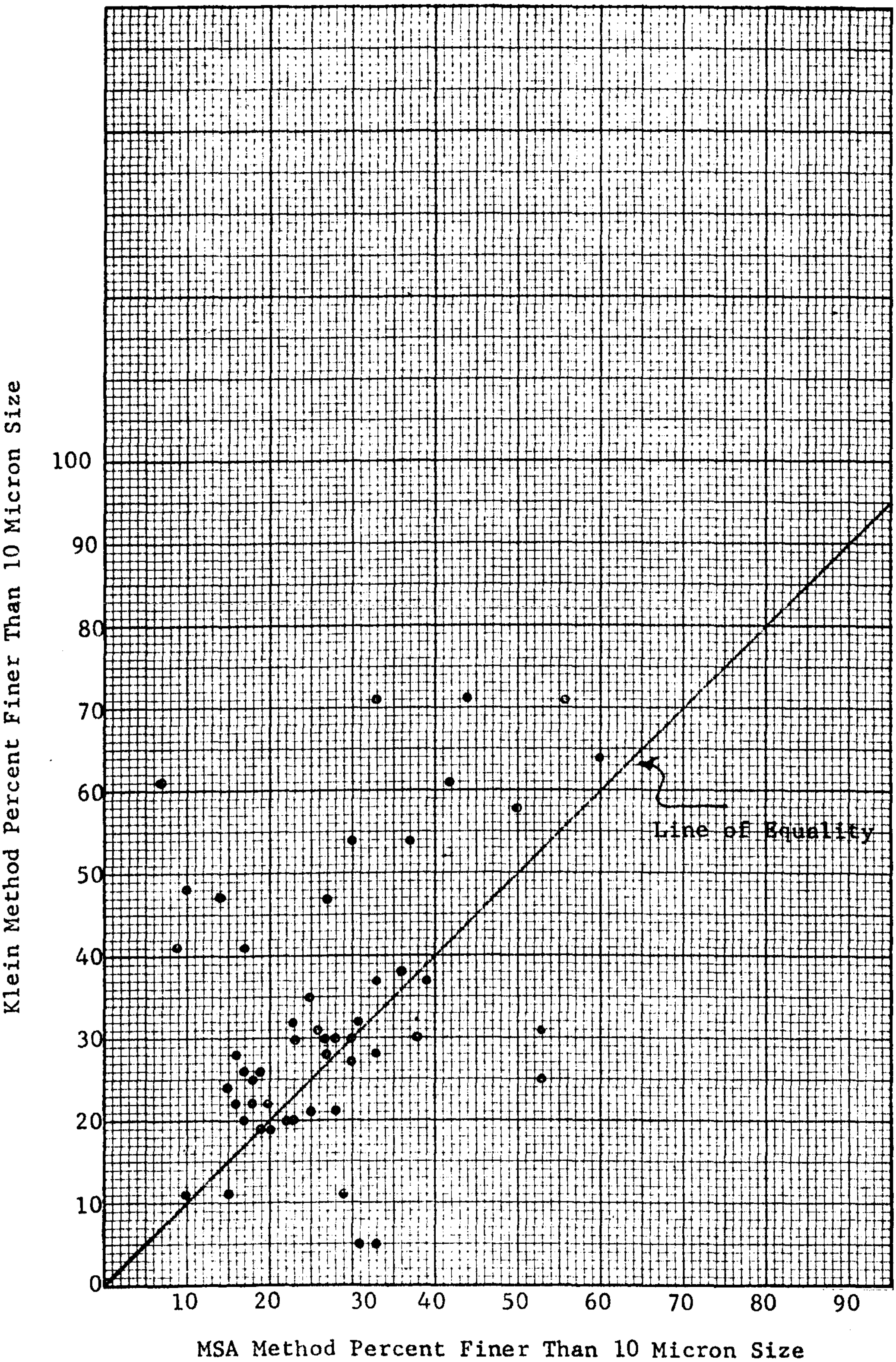


Figure 9. Percent finer than 10 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4).

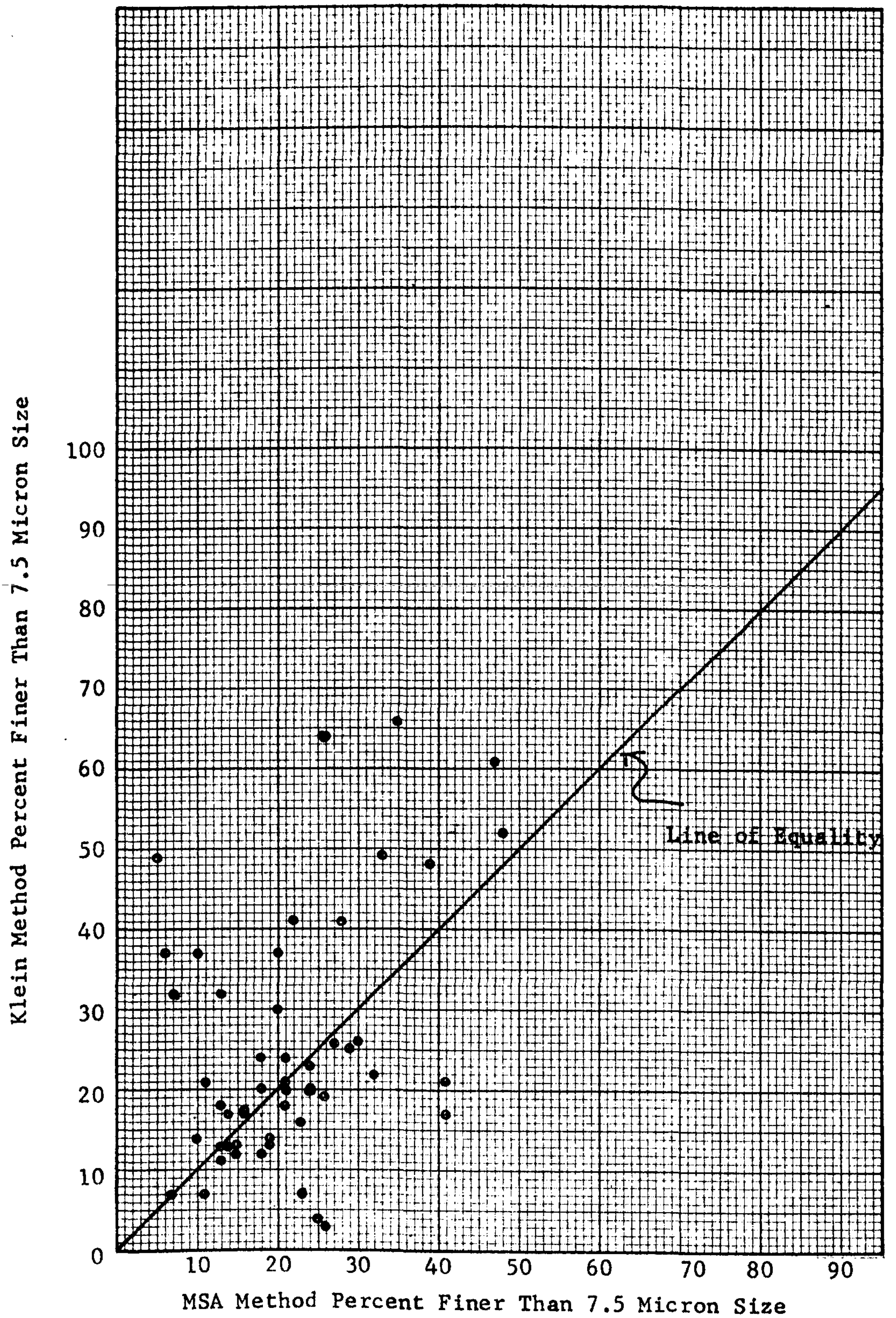


Figure 10. Percent finer than 7.5 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4).

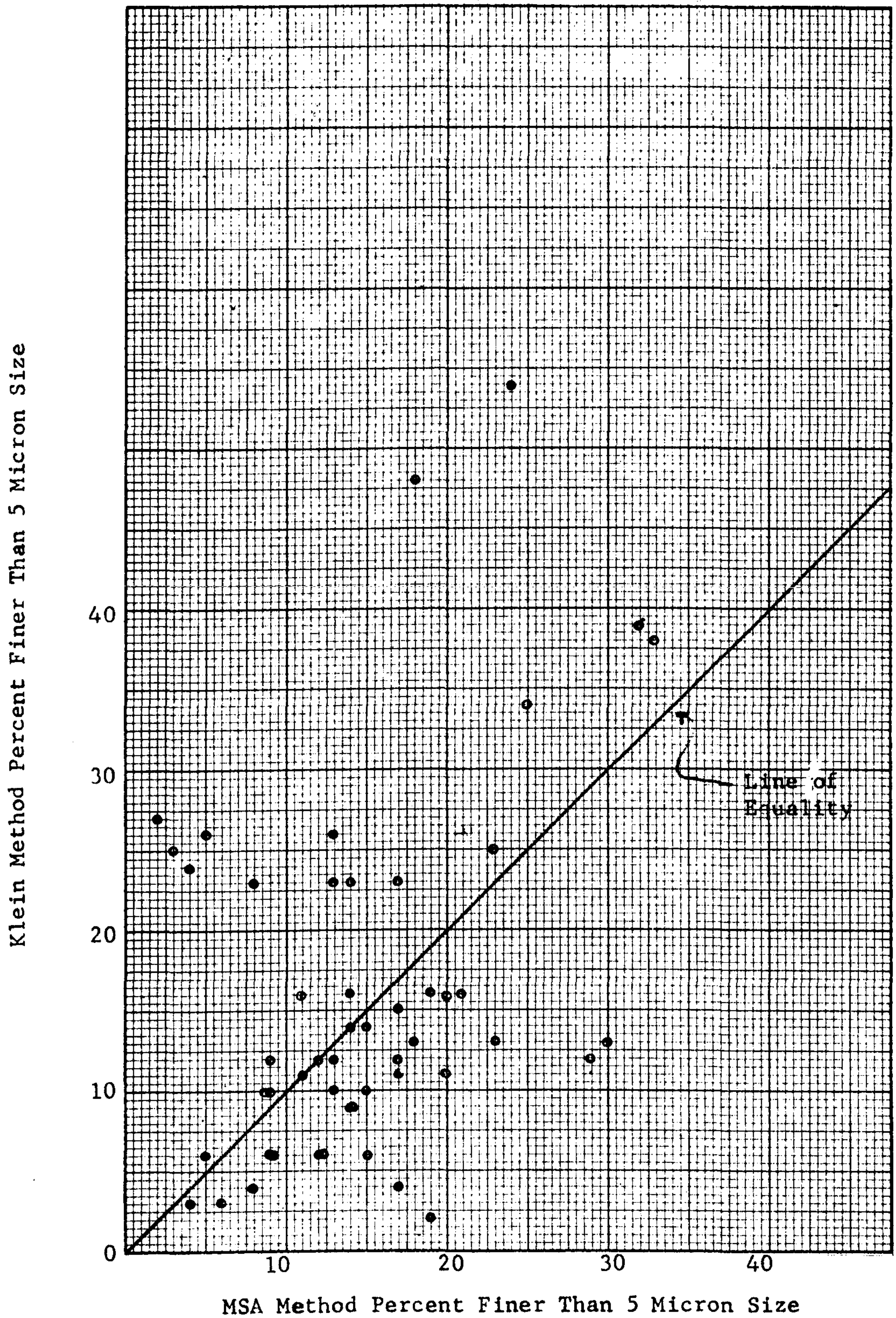


Figure 11. Percent finer than 5 micron size comparisons, MSA vs Klein Methods, 17 fly ashes, three tests by each method (table 4).

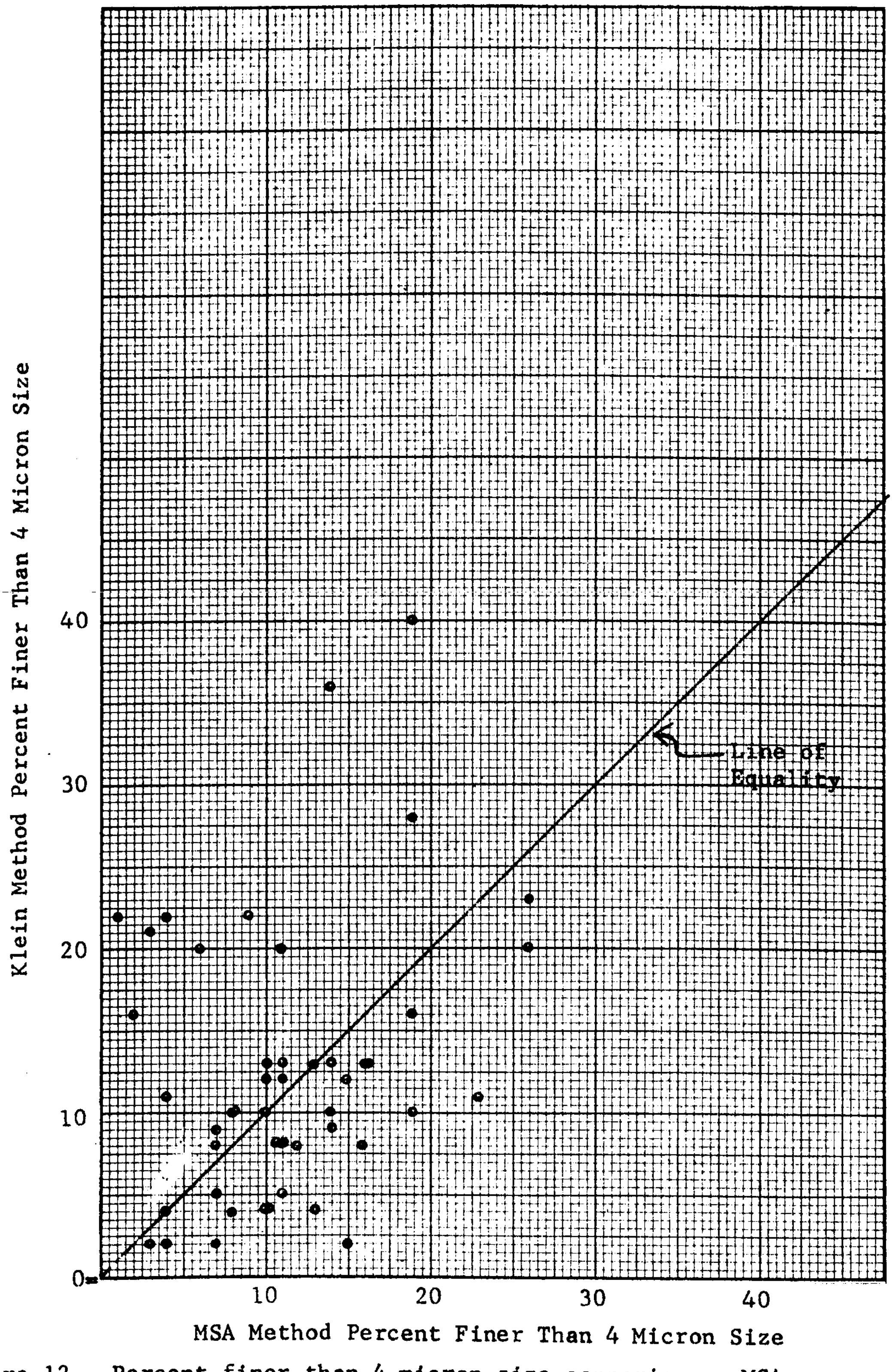


Figure 12. Percent finer than 4 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4)

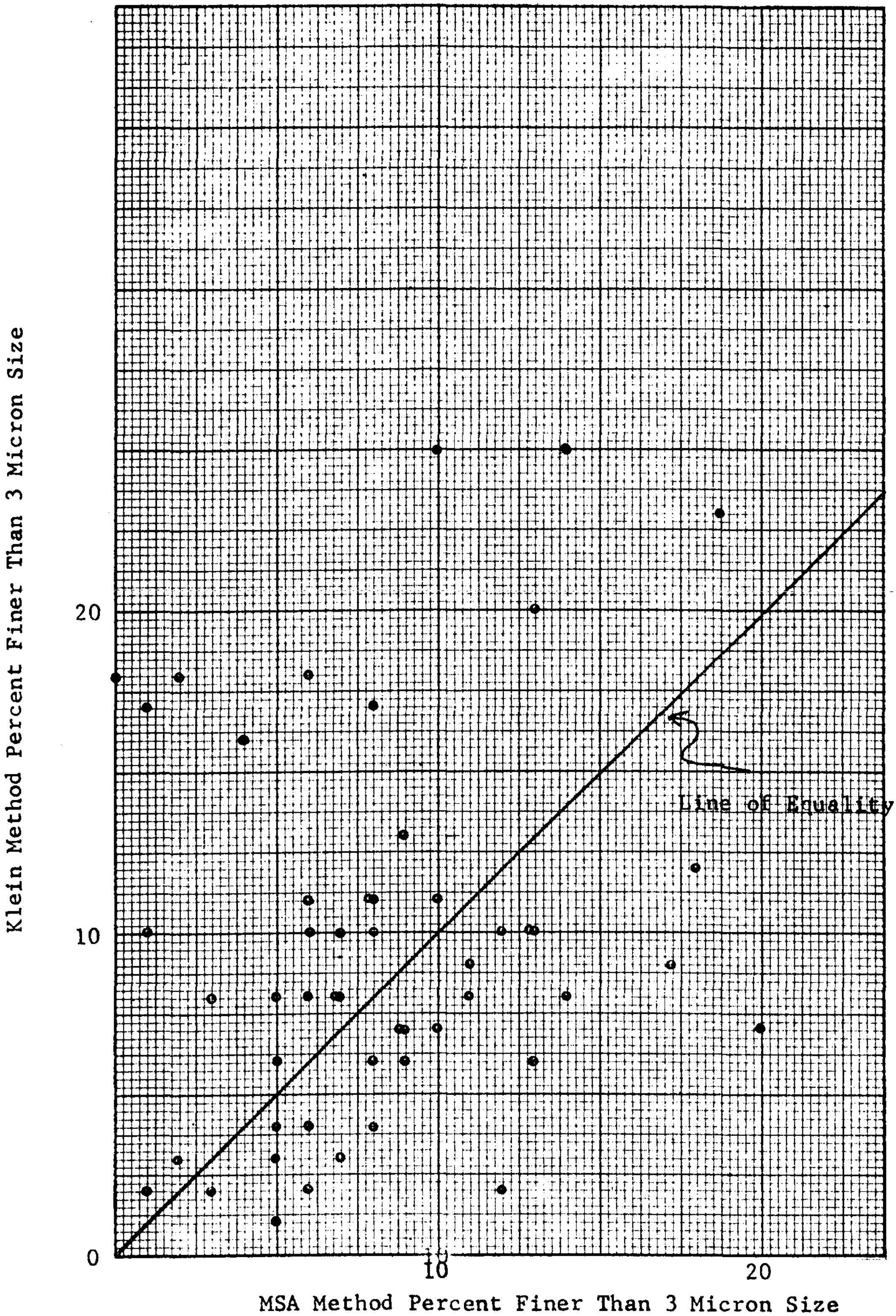


Figure 13. Percent finer than 3 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4).

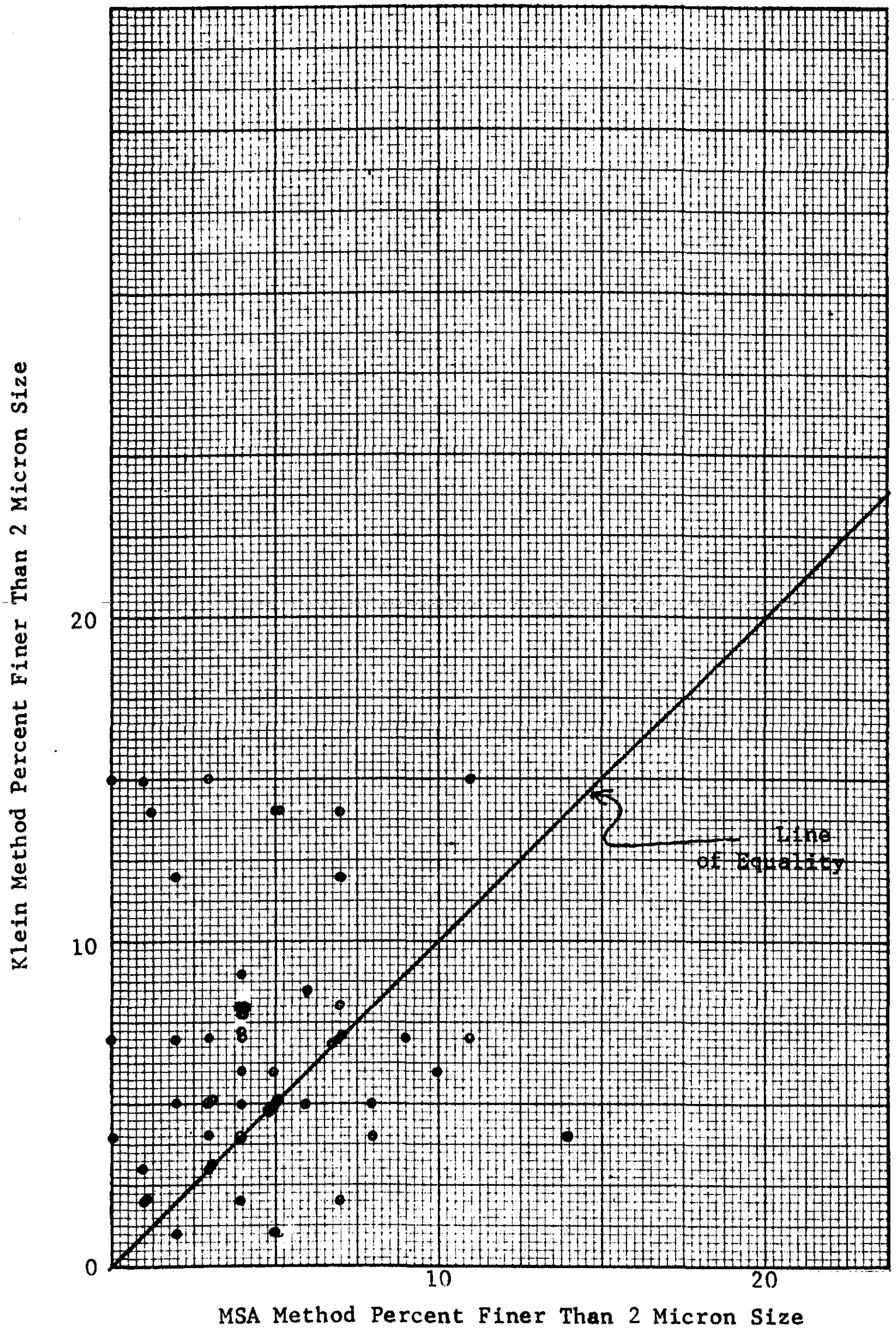


Figure 14. Percent finer than 2 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4).

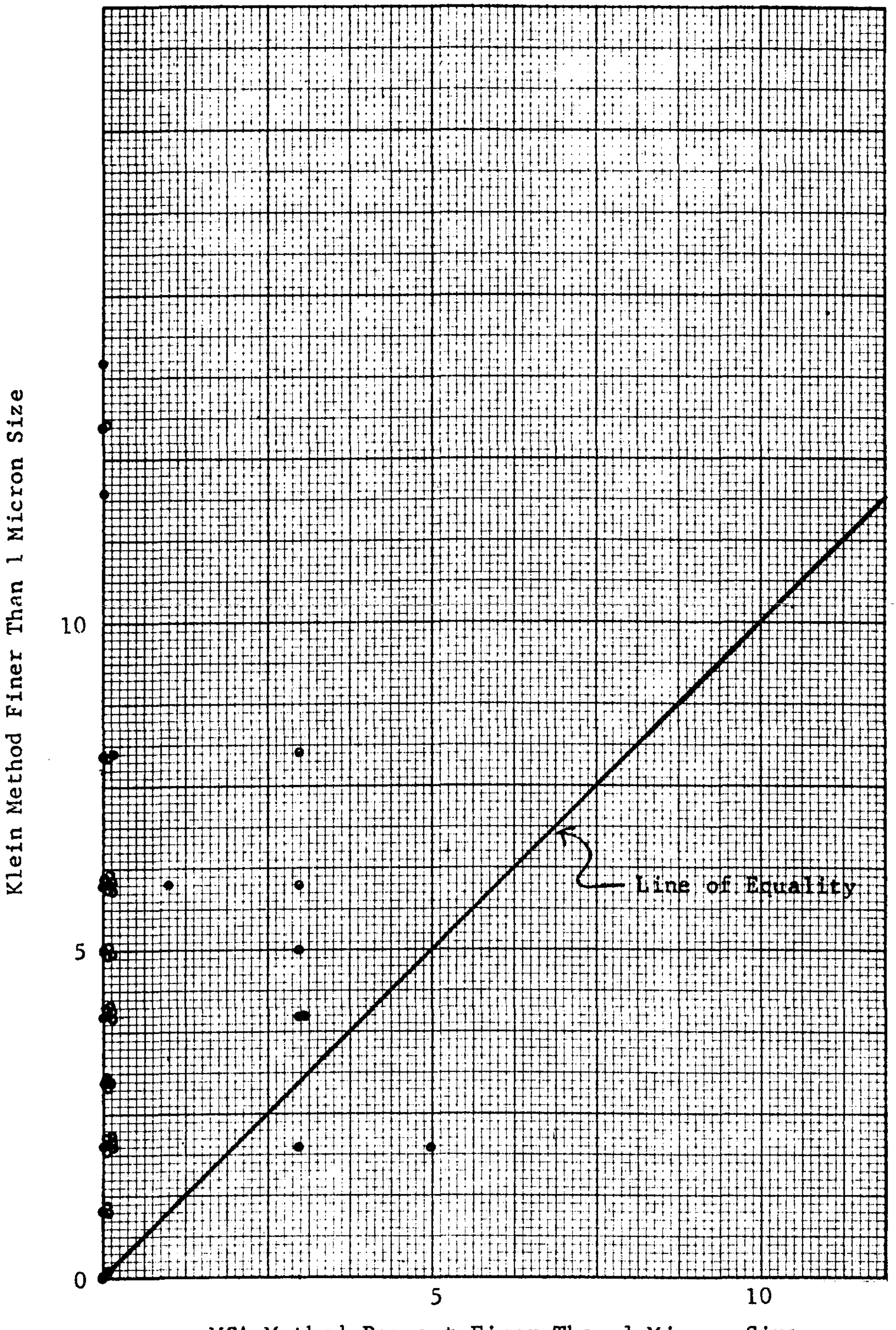


Figure 15. Percent finer than 1 micron size comparisons, MSA vs Klein methods, 17 fly ashes, three tests by each method (table 4).

APPENDIX A

MSA FINENESS METHOD

METHOD OF TEST FOR
FINENESS OF POWDERED MATERIALS
BY THE MSA ANALYZER

Scope

1. This method of test covers the procedure for using the MSA analyzer to determine the fineness and particle size distribution of powdered materials. The possible test particle size range varies in diameter from 0.5 to 150 microns.

Apparatus

2. The apparatus shall consist of the following:

a. Centrifuges - Three centrifuges with rated speeds as follows: (1) 300 rpm, (2) 600 and 1200 rpm, and (3) 1800 rpm. The speeds shall be constant within one percent of the rated values. Maximum acceleration or deceleration during starting or stopping shall be five rad per sec^2. The starting or stopping time shall not vary more than 0.5 sec.

b. Centrifuge tubes - The centrifuge tubes shall be specially designed with capillary portions at the bottom measuring approximately 30 mm in length and 0.75 $\pm$ 0.25 mm in inside diameter. The overall outside length of the tube shall be approximately 12.5 cm.with a line completely around the tube approximately 1 cm from the top. The inside diameter of the top 6 cm of the tube shall be approximately 1-1/2 cm, and the inside of the tube shall be tapered from the larger upper portion to the lower capillary portion.

c. Reading apparatus - The reading apparatus shall consist of a light box equipped to shine a light through the centrifuge tube containing the suspension so the height of the settling particles will show on an adjustable screen having a scale graduated 0 to 100 at increments of 1, the distance between graduations being 1.5 mm. In the reading apparatus a device shall be incorporated to gently jig the centrifuge tube along its long axis during settling.

d. Feeding chamber - The feeding chamber shall consist of a metal tube approximately 1 cm in inside diameter and 2 cm in length, with a screen of approximately No. 40 sieve cloth across the lower open end.

e. Sampler - The sampler shall have a compartment with a volume approximately equal to that of the capillary portion of the centrifuge tube.

Dispersion System

3. Prior to the start of the test,a suitable dispersion system shall be determined for the material to be tested. This shall consist of a sedimentation liquid, a dispersing agent, and a feeding liquid. Some suitable sedimentation liquids, dispensing agents, and feeding liquids are listed in the manufacturer's operating procedures and applications.

Determining test schedule

4. The particles of the test material having diameters of 40 microns or more shall be tested by gravity settling for which the testing times shall be determined by using the following formula: $tg = Kg/d^2$

where:

tg = time of settling of particles in sec,

d = diameter of particles in microns, and

Kg = a value calculated from the formula:

$$Kg = 18.37 \times 10^6 \frac{\mu^o}{p - p_o},$$

where:

μ_o = viscosity of the sedimentation liquid in poises,

p = density of test material in g per ml, and

p_o = density of the sedimentation liquid in g per ml.

5. The test sample particles smaller than 40 microns in diameter shall be settled out of suspension by centrifuging, and the time of settling shall be determined using the following formula: $t_c = K_c\, Q + C$,

where:

t_c = corrected time interval for centrifuging in sec.

K_c = the constant for the centrifuge settling obtained by multiplying Kg by 992×10^{-4} for the 300 rpm centrifuge, by 248×10^{-4} for the 600 rpm centrifuge, by 62×10^{-4} for the 1200 rpm centrifuge, and by 27.6×10^{-4} for the 1800 rpm centrifuge.

Q = the value taken from table 3* or calculated by the formula accompanying the table, and

C = a standard correction (0 for 300 rpm values, 9 for 600 rpm, 10 for 1200 prm, and 30 for 1800 rpm)

Test Procedure

6. Prepare the dispersing liquid by adding dispersing agent to the sedimentation liquid. Prepare the feeding liquid. Add the sample to the feeding liquid, and agitate vigorously, (at least 25 inversions). Place the mixture of sample and feeding liquid above the sedimentation

* MSA Operating Procedures and Manual Table Number.

and dispersing mixture in the test tube. Allow them to come into contact, immediately after starting the timer. Take readings on the reading apparatus chart at the settling times for the desired particle sizes falling out of suspension. (150, 100, 70, 40, 20, 10, 5, 3, 2, 1, 0.6 and 0.5 microns are satisfactory for most fly ashes). Enter the data on the analysis sheet furnished by the manufacturer, and make the calculations needed to determine the percentages finer than each micron size. To determine the surface area in sq cm per cu cm from the percentages passing the sizes, compute the individual percent that was represented by each micron size interval and make weighted average surface area calculations as shown in the example in table 1. Percentages finer than other sizes than tested may be determined from a plotting obtained as follows: Plot the particle diameters versus percentages on semi-log paper, the diameters on the log scale and the percentages on the linear scale. Connect the points to form a line.

Report

7. The report shall include the source and type of the test material; descriptions of the sedimentation liquid, the dispersing agent, and the feeding liquid, the cumulative percents finer than the micron test sizes of the test material, and the surface area of the test material in sq cm per cu cm.

Table A1 - Typical Surface Area Calculation

Micron size d(1)	Percent finer < d (2)	Average Micron size Ret (3)	Ind % Ret (4)	Factor (5)	Cum. Fineness sq cm/cu cm(6)
200	100				
150	100	175	0.0	342.6	0
100	98.9	125	1.1	480.0	5.3
70	95.5	85	3.4	705.88	24.0
40	89.8	55	5.7	1090.91	62.2
20	57.9	30	31.9	2000	638.0
10	23.9	15	34.0	4000	1360.0
5	18.8	7.5	5.1	8000	408.0
3	14.8	4	4.0	15000	600.0
2	4.0	2.5	10.8	24000	2592.0
1	0.0	1.5	4.0	40000	1600.0
0.6	0.0	0.8	0.0	75000	0.0
0.5	0.0	0.55	0.0		
					7297.5

(1) From work sheet except that the 200 micron size was added because that was the apparent size of the largest particles of materials after size tests.

(2) From the work sheet.

(3) 200-150, 150-100, etc from (1) averaged.

(4) 100-98.9, 98.9-95.5, etc from (2).

(5) $\frac{60,000}{175}$, $\frac{60,000}{125}$, etc (see 3).

(6) (4) percent of (5).

APPENDIX B

HYDROMETER FINENESS METHOD

Method of Test for Fineness of Powdered Materials Using a Klein Hydrometer

Scope.

1. This method of test covers the procedure for using a Klein Hydrometer to determine fineness and particle size distribution of powdered materials.

Apparatus

2. The apparatus shall consist of the following:

a. Graduate. A 500 ml Kollegiate graduate cut off at the 375 ml mark.

b. Hydrometer. A special Klein Hydrometer provided with a calibration data sheet for use in the testing for which it was designed. Two hydrometers are available, one for use with nonhydrous, and one with hydrous materials.

Test Sample

3. The sample size shall be determined by one of the following formulae:

a. For nonhydrous materials, using water as the suspending liquid:

$$M = 0.35 \frac{24.9G}{G - P}$$

where: M = weight of sample,

G = specific gravity of test material, and

P = density of the suspending medium in g per cc.

b. For hydrous materials using Iso-propyl alcohol as the suspending liquid: $M = 0.35 \frac{30G}{G - P}$

The suspension:

4. The suspension will be made in one of the following ways:

a. Nonhydrous materials - Mix 3.5 ml 10 percent TDA and test sample with water, bringing the volume to 150 ml. Stir 1 min with turbidimeter stirrer. Bring volume to 350 ml with water. Put hand over top and invert 30 times in 1 min.

b. Hydrous materials - Mix 15 drops, 25 percent areosol, and test sample with isopropyl alcohol, bring the volume to 150 ml. Stir 1 min with turbidimeter stirrer. Bring volume to 350 ml with isopropyl alcohol. Put hand over top and invert 30 times in 1 minute.

The Blank

5. The blank shall be 3.5 ml, 10 percent TDA, brought up to 350 ml with water, or 15 drops, 25 percent aerosol brought up to 350 ml with isopropyl alcohol.

Procedure

6. Start timing on completion of the 30 inversions. Take at least 5 sec to place the hydrometer in the suspension, and to remove it from the suspension. Do not allow the small bulb at the top of the hydrometer to become immersed. Allow 30 seconds immersion before each reading. Take the blank reading as soon as possible after the suspension reading. Wipe the hydrometer thoroughly dry after each immersion. Take a temperature reading in the suspension at each test interval.

Test Intervals

7. The test intervals shall be as follows:

a. With water - 1 min, 3.5 min, 9 min, 15 min, 27 min, 1 hr, 1-1/2 hr, 2 hr 40 min, 6 hr, 24 hr, 96 hr.

b. With isopropyl alcohol - 2 min, 6.5 min, 16 min, 26 min, 48 min, 1-1/2 hr, 2-1/2 hr, 4-1/2 hr, 6 hr, 24 hr, 48 hr.

Calculation of weight percent of suspended particles,

8. The percent of the weight of the particles suspended will be determined using the calibration formula for the hydrometer determined at calibration.

Calculation of the Diameter of Suspended Particles

9. The diameter of the suspended particles shall be determined by the formula:

$$D = \sqrt{\frac{KH}{t}}$$

where:

D = diameter in microns,

K = the hydrometer constant,

H = the distance of fall determined from the calibration formula for the hydrometer, in centimeters, and

t = sedimentation interval in seconds.

$$K = \frac{1837\ U(1000)}{G - P}$$

where:

U = viscosity in poises of liquid medium.

G = average specific gravity of solids in suspension, and

P = density of liquid medium in g per cc.

Plotting Results

10. Plot the particle diameters versus weight percentages on semi-log paper, the diameters on the log scale, and the percentages on the linear scale. From this plotted curve read the weights suspended as percents for the following diameters: 33.5, 19, 12.5, 10, 7.5, 5, 4, 3, 2, and 1 microns.

Calculation of Surface Area

11. Determine the surface area from the following formula:

$$S = 15.25 \quad (W_1 + W_2 + W_3 + W_4 + 2W_5 + 2W_6 + 2W_7 + 6W_8 + 10W_9 + 52W_{10} + 50)$$

where:

S = surface area of test material in sq cm per cu cm,

W_1 to W_{10} = the percentages of weight suspended for the size particles in paragraph 10, respectively.

Report.

12. The report shall contain identification data for the test material, the suspending medium, the weight percent of particles finer than the ten micron sizes listed in paragraph 10, and the surface area in sq cm per cu cm.