AD-751 103

PORTLAND-POZZOLAN CEMENT MADE WITH TENNESSEE VALLEY AUTHORITY FLY ASH

Bryant Mather

Army Engineer Waterways Experiment Station
Vicksburg, Mississippi

May 1960

PORTLAND-POZZOLAN CEMENT MADE WITH TENNESSEE VALLEY AUTHORITY FLY ASH

TECHNICAL REPORT NO. 6-541

May 1960

U. S. Army Engineer Waterways Experiment Station
CORPS OF ENGINEERS
Vicksburg, Mississippi

ARMY-MRC VICKSBURG, MISS

THE CONTENTS OF THIS REPORT ARE NOT TO BE USED FOR ADVERTISING, PUBLICATION, OR PROMOTIONAL PURPOSES

PREFACE

The work reported herein was conducted in accordance with a letter of agreement, dated 22 May 1959, from Mr. G. O. Wessenauer, Manager of Power, Tennessee Valley Authority, accepted by the Director, U. S. Army Engineer Waterways Experiment Station, on 26 May 1959. The portland cement, portland-cement clinker, and gypsum were supplied, without charge, by the Brandon, Mississippi, plant of the Marquette Cement Manufacturing Co. through the courtesy of Mr. C. E. Wuerpel, Vice President.

The investigation was planned cooperatively by Messrs. William E. Dean, Jr., and Harry L. Balzan of the Power Research Staff, Tennessee Valley Authority, and Messrs. Thomas B. Kennedy, Bryant Mather, and James M. Polatty of the Concrete Division, Waterways Experiment Station. The work was done at the WES Concrete Division by or under the immediate supervision of Mr. Leonard Pepper, Mrs. Katharine Mather, Mr. W. O. Tynes, and Mr. R. L. Curry. Mr. Mather was project leader and prepared this report. Col. Edmund H. Lang, CE, was Director, and Mr. J. B. Tiffany was Technical Director of the Waterways Experiment Station during the course of this work.

CONTENTS

SUMMARY

Fly ash, mechanically collected at the Colbert, Alabama, steam generating plant of the Tennessee Valley Authority, has a chemical composition such that it would be satisfactory for use as a pozzolanic admixture in portland-cement concrete. However, it does not meet current specifications for such use because it is too coarse. The investigation described herein revealed that this fly ash can be used to make a satisfactory portland-pozzolan cement either by grinding the fly ash separately and blending it with commercially manufactured portland cement or by intergrinding fly ash, portland-cement clinker, and gypsum. The rate of chemical activity, as indicated by heat of hydration and the rate of development of compressive strength, decreases with increasing fly ash content of the cement-fly ash blend or the fly ash-portland-pozzolan cement when the fineness is held constant, and increases with increasing fineness when the fly ash content is held constant. Federal Specifications for portland-pozzolan cement are included as Appendix A.

PORTLAND-POZZOLAN CEMENT MADE WITH TENNESSEE VALLEY AUTHORITY FLY ASH

PART I: INTRODUCTION

Fly Ash

1. The portion of the residue that is carried up the stack by flue gases when powdered coal is burned is referred to as fly ash. The chief sources of fly ash are the steam plants used in the generation of electric power. Two principal kinds of precipitators employed in collecting fly ash are: (a) mechanical precipitators, which generally collect a coarser product, and (b) electrostatic precipitators, which generally collect a finer product. The nature and properties of fly ashes differ not only as a function of the type and efficiency of the precipitation equipment used in their collection, but also as a function of the composition and fineness of the coal from which they are produced and the efficiency and type of combustion procedure by which the coal is burned.

Use of Fly Ash in Concrete

2. Fly ash has been extensively investigated and widely used in the United States and other countries as an ingredient in portland-cement concrete mixtures. In some cases the fly ash has been incorporated in the concrete as an interground or interblended ingredient of portland-pozzolan cement, but in most cases the fly ash has been employed as an admixture added as a separate ingredient to the concrete mixture.

3. The inclusion of fly ash in concrete mixtures may have different effects, depending on the characteristics of the cement, aggregates, and fly ash and the proportions of the mixture. Generally, however, when fly ash is used in a concrete, the compressive strength of the concrete is increased, at least at later ages; volume change is affected little;[4,6,7]*

* Raised numbers refer to similarly numbered items in the List of References.

resistance to chemical attack is improved;[4,6,11] permeability is reduced;[3,4,5] and heat evolution is also reduced.[4,6]

TVA Fly Ash Production and Use

4. The steam generating stations of the Tennessee Valley Authority currently burn over 18 million tons of pulverized coal and produce over a million tons of mechanically collected fly ash a year. This mechanically collected ash is too coarse to comply with current specifications for fly ash used as an admixture in portland-cement concrete.[2,12] However, experience with the use of this ash (from the Johnsonville, Tennessee, steam plant and Widows Creek and Colbert steam plants, Alabama) as an admixture in concrete has been satisfactory in connection with construction at the Johnsonville and Widows Creek steam plants (50,000 cu yd of concrete, each) and Wilson Dam lock (425,000 cu yd of concrete) and also in connection with construction of a Du Pont plant near Waverly, Tennessee, with regular concrete-block production at Sheffield, Alabama, and with experimental concrete masonry units made at Tupelo, Mississippi.[10]

Previous Studies of Fly Ash-Portland-Pozzolan Cement

5. A brief review of the limited literature on the use of fly ash as an ingredient of portland-pozzolan cement is given in the following paragraphs.

Studies reported by Davis and others in 1937

6. Davis and others[6] in 1937 described studies in which samples of fly ash from 15 different sources were used in blends with portland cements of seven different compositions, the proportion of fly ash to cement ranging from 1:9 to 1:1. In most of this work fly ash was blended with portland cement at the concrete mixer, but for 12 cements, the materials were interground. Eight of these cements were made by intergrinding two portland cements with two fly ashes as shown in the tabulation on the following page. Each fly ash was used in the ratios (fly ash to cement) of 1:9, 1:4, 1:2.3, and 1:1.5. The cements were first ground to attain a specific

Test Result	Cement 1*					Cement 2**				
	Amount of Fly Ash Interground					Amount of Fly Ash Interground				
	0%	10%	20%	30%	40%	0%	10%	20%	30%	40%
Passing No. 325 sieve, %	97.5	97.9	97.7	99.0	99.3	98.0	98.6	99.1	99.4	99.7
Surface area, sq cm/g (Wagner)	2350	2570	2430	2350	2400	2420	2570	2620	2740	2760
Water required for normal construction, %	24.5	25.0	24.8	25.8	26.5	25.5	25.7	26.5	26.6	28.0
Setting time, Gillmore:										
Initial, hr:min	1:00	1:15	1:20	1:40	2:05	1:15	1:35	2:30	3:45	4:[illegible]
Final, hr:min	1:45	2:05	2:20	2:35	3:20	2:15	3:15	4:15	[illegible]	6:00
Mortar, W/C† by wt	0.42	0.43	0.43	0.43	0.44	0.43	0.43	0.44	0.44	0.45
Tensile strength, psi										
1 day	300	320	305	315	260	300	260	250	200	170
3 days	380	385	390	390	340	380	345	340	310	270
7 days	435	440	420	425	425	400	370	375	315	290
28 days	460	475	500	515	510	440	450	470	415	330
Concrete, W/C by wt	0.43	0.43	9.43	9.43	0.43	0.46	0.46	0.45	0.45	0.45
Compressive strength, psi										
1 day	2570	2330	2490	2130	1680	2680	2720	2380	2040	1600
3 days	3540	3560	3560	2990	2850	4170	4030	4170	3580	3000
7 days	4740	4720	4590	4040	3560	4470	4410	4190	3800	3300
28 days	5720	5870	5950	5600	5260	5890	6470	6200	5780	5230

* Clinker: 50% C_3S, 8% C_3A; fly ash used with cement 1 had 1.1% carbon and a surface area before blending of 3220 sq cm/g.

** Clinker: 56% C_3S, 8% C_3A; fly ash used with cement 2 had 3.3% carbon and a surface area before blending of 2580 sq cm/g.

† W/C is water-cement ratio.

surface of 1600 sq cm/g (Wagner); then the fly ash was added, and the mixture ground for the same length of time required to increase the specific surface of the portland cement from 1600 to approximately 2400 sq cm/g (Wagner). This gave portland-fly ash cement of a fineness equal to or greater than that of the corresponding portland cement.

7. The four other fly ash cements were made in which the clinker-fly ash mixture was ground for the same length of time as that required to grind portland cement to a surface area of 1600 sq cm/g. In the tabulation below the results of tests on the interground cements are compared with those obtained in tests on corresponding mixed cements.

	Fly Ash			Surface Area of Fly Ash Cement sq cm/g		Compressive Strength of Concrete, psi				Ratio of Interground Cement Compressive Str to Mixed Cement Compressive Str %	
						7 Days		1 Year			
No.	Carbon %	Surface Area sq cm/g	%	Mixed	Interground	Mixed	Interground	Mixed	Interground	7 Days	1 Year
1	1	3220	20	1920	2110	3760	4090	7610	7940	109	104
2	10	3800	20	2040	2790	3220	3390	7080	6480	105	92
3	12	2390	20	1760	2320	3270	3600	6840	6970	110	102
1	1	3220	30	2090	2570	3210	3150	7770	6930	98	89

8. The effect of intergrinding was to increase the fineness of the fly ash cement. The interground cements containing 20% fly ash had 7-day compressive strengths that averaged 8% higher than those of the corresponding mixed cements; their 1-year compressive strengths were little or no higher. Therefore, it was concluded that intergrinding was of benefit only at early ages.

9. In the general conclusions reported by Davis et al.,[6] it was contemplated that only fly ashes having surface areas of 2500 sq cm/g would be used and, therefore, intergrinding would not be of significant benefit.

Studies reported by G. and W. H. Corson, Inc., in 1948

10. G. and W. H. Corson, Inc.,[8] described studies made in 1946 and 1947 in which comparisons were made among grinds containing from 0 to 30% fly ash. The results were described as indicating improvement in strength at all ages as a result of intergrinding, and also as indicating that 20% fly ash seemed to be a "satisfactory optimum." Test results included the following:

Fly Ash in Cement, %	Compressive Strength of 2-in. Cubes Composed of 1 Part Cement:2.90 Parts Ottawa Sand, psi 7 Days	28 Days	6 Months	1 Year
0	3540	4760	4880	5780
10	3930	5570	6080	7010
20	4220	4860	5750	6880

Material	Percentage Passing No. 325 Sieve After Grinding for Indicated Time, hr 0	1	2	3	4	5
Clinker	0.4	17.3	33.2	43.5	52.0	57.2
80% clinker, 20% fly ash	18.8	36.5	51.3	59.9	68.8	76.4

11. Several carloads of cement were made by introducing 20% of fly ash by weight to the ground clinker stream before it entered the ball mill for final grinding. The results of tests on this cement included:

Test Result	Cement with No Fly Ash	Cement with 20% Fly Ash Interground
Passing No. 325 sieve, %	90.7	96.9
Surface area, turbidimeter, sq cm/g	1735	2230
Specific gravity	3.140	3.045
Autoclave expansion, %	0.19	0.11
Normal consistency, %	24.5	24.0
Compressive strength, psi		
1 day	950	1330
3 days	2230	2480
7 days	3480	3820
28 days	5470	5870

Letter comments on use of fly ash cements

12. In a letter to the Waterways Experiment Station dated 5 February 1959, Dr. L. John Minnick of G. and W. H. Corson, Inc., commented that after extensive studies of fly ash cements, he concluded that "it is very difficult to grind down to a finer state the small glassy spheres of fly ash." He also stated that when grinding was employed on fly ash cements for extended periods, a substantial improvement in the strength resulted, probably due to grinding of the cement rather than to grinding of the fly ash. On the other hand, Dr. Minnick also reported that the nonspherical particles of fly ash are readily ground, and that the effect is beneficial in reducing water requirement.

13. In another letter to the WES, dated 16 March 1959, Mr. E. Carl Kreager of Columbia Cement Division, Columbia Southern Chemical

Corporation, described cement blended in their plant in 1952-1953 for the Ohio Power Company for use in construction of a generating plant near Beverly, Ohio. According to Mr. Kreager, fly ash was fed into the discharge of the finish grinding mill at the rate of 20%:80% portland cement, and mixing was accomplished by passage through 100 ft of 16-in. screw feeder, after which the mixture was transported through a Fuller-Kinyon pump to storage. The fly ash came from the Ohio Power Company plant at Philo, Ohio. Typical data on the cement in question include:

Chemical and Physical Properties	Fly Ash	Clinker	Blend
SiO_2, %	48.78	22.37	27.45
Al_2O_3, %	24.70	5.78	9.39
Fe_2O_3, %	17.42	3.40	5.78
CaO, %	1.90	64.86	51.28
MgO, %	1.35	2.86	2.59
Passing No. 325 sieve, %	92.8	--	--
Surface area, air permeability, sq cm/g	2165	--	--

	Compressive Strength, psi			
	2-in. Cubes		4-1/2 bag/cu yd Concrete	
Age of Sample	No Fly Ash	Fly Ash Blend	No Fly Ash	Fly Ash Blend
3 days	1780	1620	--	--
7 days	2710	2570	2250	1880
28 days	4300	4270	3180	2650
90 days	6640	5850	3350	3750
1 year	6950	7630	--	--

14. Concrete made with the blended fly ash-portland cement was used in all concrete construction on the Beverly, Ohio, generating plant.

French fly ash cements

15. The Société des Matériaux de Construction de la Loisne, Paris, has, since 1951, manufactured pozzolan-slag cements with fly ash in two strength grades. Production rose from 24,000 tons in 1952 to 83,000 tons in 1956. The company began making portland-fly ash cement in 1956 and now makes three strength grades of this cement. Between 1957 and 1958, the proportion of their total cement production that contained fly ash increased from 81 to 91%. The three grades of fly ash-portland cement have approximately the following contents and surface areas:

Grade Designation	Clinker %	Fly Ash %	Gypsum %	Surface Area, sq cm/g (Air Permeability)
CPA+C 250-315	75	20	5	3700
HRI+C 315-400	84	12	4	4300
Super+C 355-500	85	10	5	4700

16. The fly ash used contains about 2% carbon, 2% lime, and 90% acid-insoluble material, and has a surface area (air permeability) of 2000 to 3800 sq cm/g. These cements are made under a patent issued on 22 April 1953 to Mr. Fouilloux. Comments on the use of these cements are given in a paper by Jarrige.[9]

New England

17. Rock Products magazine for December 1959 (vol 62, No. 12, p 52) stated that the Lee Lime Corporation, Lee, Massachusetts, is a copartner in Pozament Corporation, Bridgeport, Connecticut, to produce and market a lime-fly ash and pozzolanic cement for concrete block manufacture.

PART II: PRESENT INVESTIGATION

Purpose and Scope

Purpose

18. The purpose of this investigation was to determine the potentialities of using TVA fly ash, ground to increase its surface area, and blended with portland cement; and also of using the same fly ash ground with portland-cement clinker to produce portland-pozzolan cement meeting or exceeding ASTM and Federal Specification[1,13] requirements for such material.

Scope

19. Fly ash from the TVA Colbert, Alabama, steam plant, Type I portland cement and portland-cement clinker, and gypsum were used. All combinations of cement and fly ash or clinker and fly ash were made on a weight basis. The test program included the following:

- a. The cement without fly ash was tested for control purposes. The work consisted of chemical analyses, petrographic examination, and physical property determinations.
- b. The cement and unground fly ash were combined in one proportion and subjected to the types of tests mentioned in a.
- c. Samples of the fly ash were ground alone to two degrees of fineness and combined with commercially ground cement for chemical analyses, petrographic examinations, and determination of physical properties.
- d. Portland-cement clinker and fly ash were ground together in several proportions and to two degrees of fineness for chemical analyses, petrographic examination, and determination of physical properties.

Materials and Processing

Materials

20. The following materials were used:

- a. Cement. Twenty bags of Type I portland cement, serial No. RC-454, from Marquette Cement Manufacturing Company, Brandon, Mississippi, received 25 May 1959.
- b. Clinker. 2000 lb of portland-cement clinker from the same

burn as that from which the cement was made, serial No. RC-454-C, received 25 May 1959.

c. Gypsum. 50 lb of gypsum from the same suppl; as that used in making the cement, serial No. RC-454-G, received 25 May 1959.

d. Fly ash. 2000 lb of fly ash from the Colbert, Alabama, steam power-generating plant of the Tennessee Valley Authority, serial No. AD-94(3), shipped from Wilson Dam on 2 June 1959 and received 4 June 1959.

Processing

21. Laboratory ball milling equipment (figs. 1 and 2), modified to

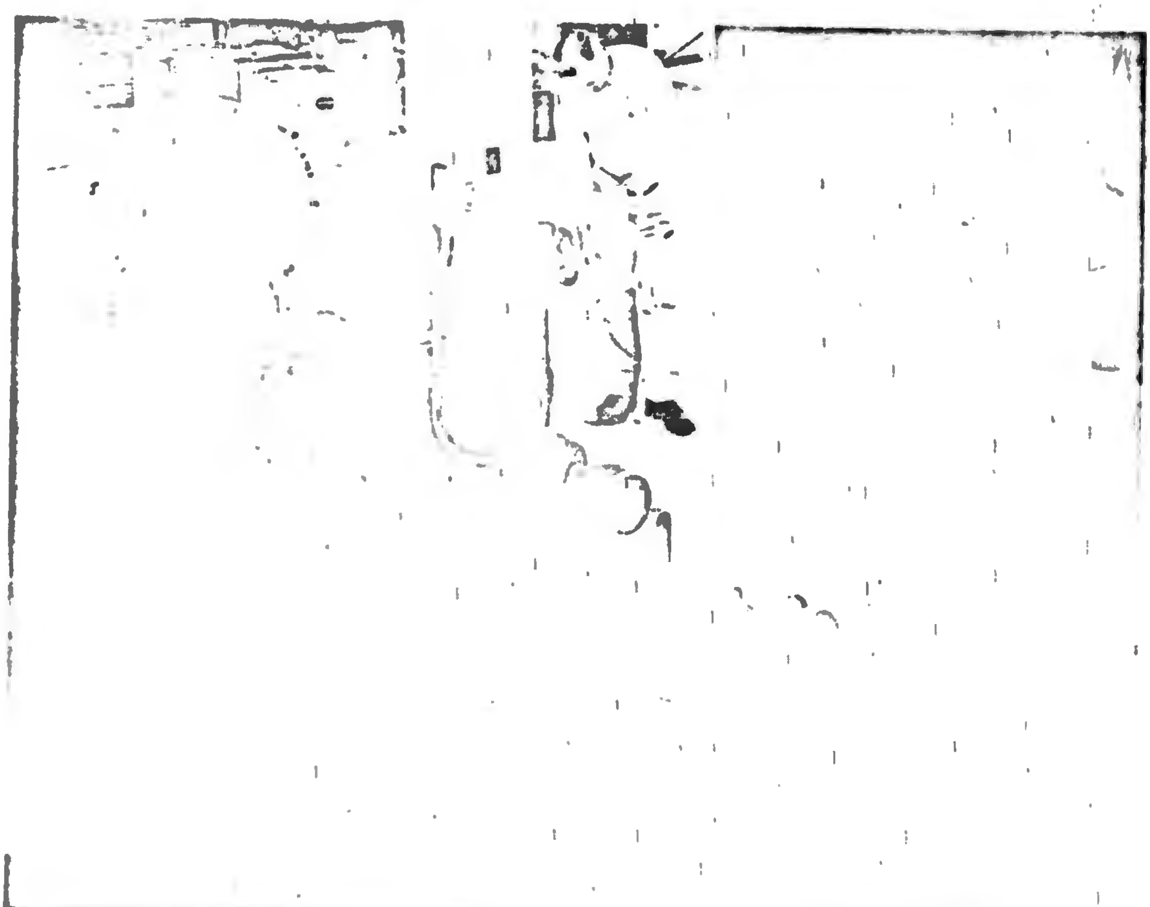

Fig. 1. Roller mill

provide a removable plate opening in one end to permit sampling without removing the entire end plate, was used to prepare the following materials, in the quantities indicated, for use in the tests. All finenesses were determined by the air-permeability method (CRD-C 218; Federal Test Method Standard No. 158, Method 2101).

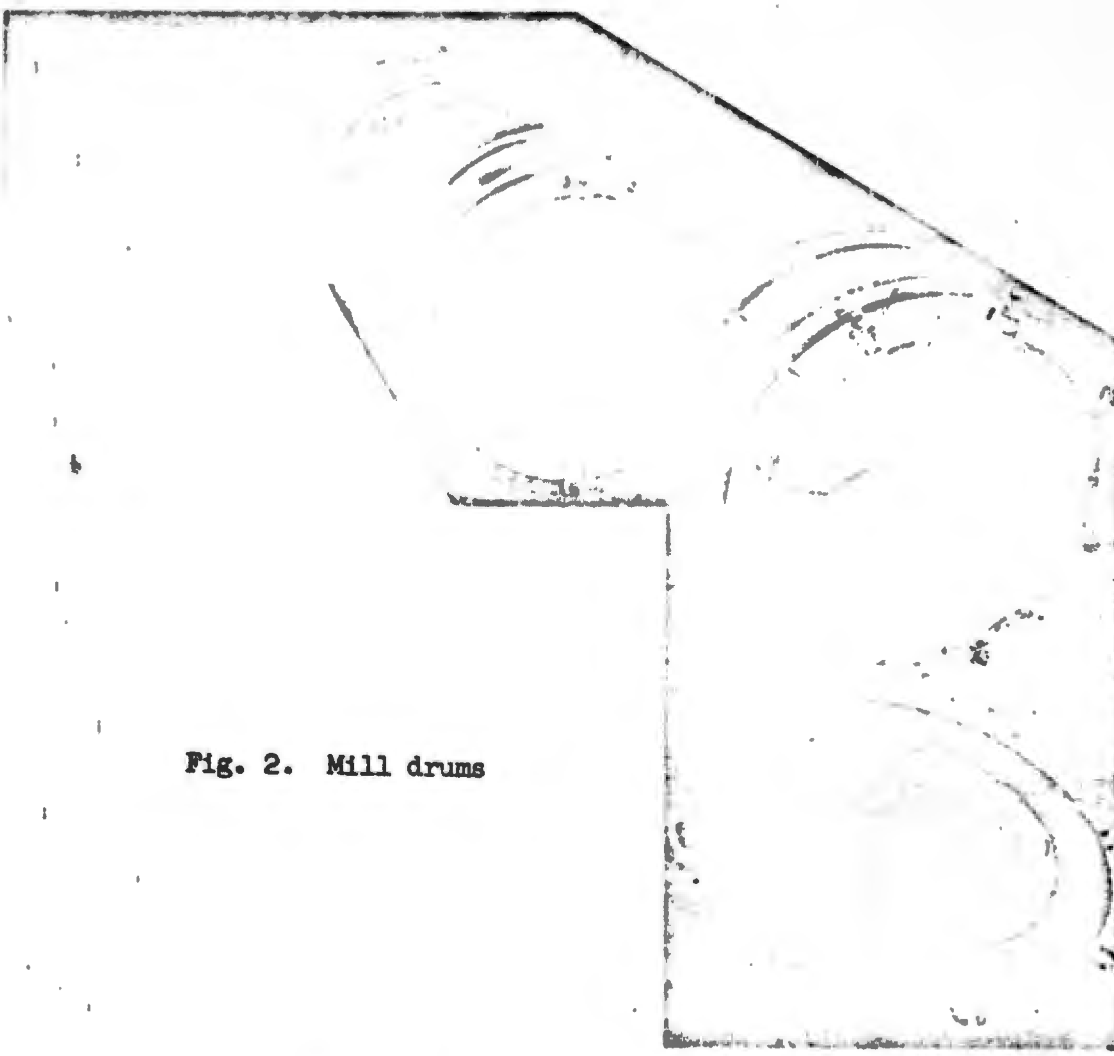

Fig. 2. Mill drums

a. Fly ash ground to n* ± 500 sq cm/g, plus 100 lb sent to TVA.**

b. Fly ash ground to n + 2000 ± 500 sq cm/g, plus 100 lb sent to TVA.

c. Blend 1: 85% clinker and 15% fly ash by weight ground to n ± 500 sq cm/g.†

d. Blend 2: 80% clinker and 20% fly ash by weight ground to n ± 500 sq cm/g.

---

* n = the fineness, sq cm/g, of cement (RC-454), obtained according to CRD-C 218.

** The 100-lb lots of ground fly ash were shipped to TVA on 12 August 1959 for them to check.

† In each blend in which clinker was used, gypsum was added in an amount sufficient to produce an SO_3 content, expressed as percentage by weight of clinker in the blend, equal to the SO_3 content of the cement.

e. Blend 3: 75% clinker and 25% fly ash by weight ground to n ± 500 sq cm/g.

f. Blend 4: 65% clinker and 35% fly ash by weight ground to n ± 500 sq cm/g.

g. Blend 5: 80% clinker and 20% fly ash by weight ground to n + 1000 ± 500 sq cm/g.

h. Blend 6: 80% clinker and 20% fly ash by weight ground to n + 2000 ± 500 sq cm/g.

Tests

Petrography and X-ray diffraction

22. X-ray diffraction studies of all four materials and petrographic examinations of the clinker and gypsum were made. Petrographic examination and X-ray diffraction study of fly ash, crushed to approximately n and n + 2000 sq cm/g, were made.

Chemical analysis and heat of hydration

23. The cement and clinker were analyzed for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, loss on ignition, Na_2O flame, K_2O flame, and insoluble residue (C_3S, C_2S, C_3A, C_4AF, $CaSO_4$). The fly ash was analyzed for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, loss on ignition, Na_2O flame, K_2O flame, insoluble residue, moisture content, sulfide sulfur, and total carbon. The gypsum was analyzed for CaO, SO_3, and H_2O. Heat of hydration (by the heat-of-solution method) at 7 and 28 days was determined on the cement and the six blends. Fly ash ground to n ± 500 sq cm/g, and to n + 2000 ± 500 sq cm/g was also analyzed for Fe_2O_3; based on the resulting values, together with the Fe_2O_3 value determined on the fly ash, the amount of iron picked up during grinding was calculated. Specific gravities were determined, also, of fly ash unground, 2.44; fly ash ground to 4250 sq cm/g, 2.74; fly ash ground to 5860 sq cm/g, 2.78.

Physical tests

24. The following physical tests were conducted:

a. Fineness (CRD-C 218) of the cement, fly ash, and the eight processed materials and blends was determined.

b. Autoclave expansion. Autoclave expansion of the cement;

cement + 20% fly ash; cement + 20% fly ash ground to n ; cement + 20% fly ash ground to n + 2000; and the six blends was determined.

c. Time of setting (Gillmore) was determined on the materials listed in b above.

d. Compressive strength at 3, 7, and 28 days was determined on the materials listed in b above.

e. Air content of mortar was determined on cement and blend 4 (65% clinker and 35% fly ash). Since neither of these air contents was more than 12.0% by weight, the other materials listed in b above were not tested.

f. False set of the cement and blend 4 was tested according to Federal Test Method Standard 158, Method 2501, and CRD-C 259. Materials so tested are judged to be false setting if the difference between initial and final penetration is more than 17 mm. Since neither material showed false set, the other five blends were not tested.

g. Residue after wet-sieving on No. 100 and No. 325 sieves of the cement, fly ash, and six blends was tested.

h. Water requirements of the six blends and cement, cement + 20% fly ash, cement + 20% fly ash ground to n , and cement + 20% fly ash ground to n + 2000 were tested in accordance with CRD-C 206 and ASTM C 340.

i. Drying shrinkage of the cement and the six blends was tested in accordance with CRD-C 206 and ASTM C 340.

j. Mortar expansion of the cement and blend 4 (65:35) was tested in accordance with CRD-C 206 and ASTM C 340.

k. Bleeding of pastes of the materials listed in b above was tested in accordance with CRD-C 245.

l. Sulfate resistance with added SO_4 of the cement, cement + 20% fly ash, and blends 1, 2, 4, and 6 was tested.

m. Sulfate resistances of lean mortar bars of the same materials listed in l were tested.

Results of tests of the cement were compared with the requirements of Federal Specification SS-C-192b (CRD-C 200) and ASTM C 150 for Type I portland cement. Results of tests of all blends of cement (or cement clinker) and fly ash were compared with the requirements of CRD-C 206 and ASTM C 340.

PART III: RESULTS

Processing

Roller-mill charges used

25. The fly ash used in the portland cement-fly ash blends, and the six, interground portland-pozzolan blends were ground in a drum roller mill, 30 in. in diameter and 24 in. wide, turning at a speed of 40 rpm. Based on information on grinding operations in cement manufacturing, it was decided that a graded steel ball charge of approximately 850 lb would produce the best results; however, the first grinding of the fly ash for each fineness resulted in failure of the support rollers of the mixing drum, and the ball charge was reduced to approximately 500 lb for the rest of the grinding operations.

26. The approximate gradings for the two weights of ball charges were as follows:

- a. The 853-lb charge was composed of:
  - (1) 78 lb of 8/16-in. diameter
  - (2) 117 lb of 11/16-in. diameter
  - (3) 404 lb of 13/16-in. diameter
  - (4) 76 lb of 15/16-in. diameter
  - (5) 178 lb of 1-1/16-in. diameter
- b. The 496-lb charge was composed of:
  - (1) 78 lb of 8/16-in. diameter
  - (2) 117 lb of 11/16-in. diameter
  - (3) 301 lb of 13/16-in. diameter

Fly ash

27. The air-permeability fineness of the fly ash before grinding was 1855 sq cm/g, and the fineness of the portland cement used in the processing* was 4010 sq cm/g. Two portions of fly ash, one weighing 194 lb and the other 180 lb, were ground. The first was ground to a fineness of 4250 sq cm/g based on desired fineness of 4010 ± 500; the other was ground

* Later tests revealed a value of 3940 sq cm/g; this value is given in table 1.

to a fineness of 5860 sq cm/g based on desired fineness of 4010 + 2000 ± 500.

28. The data on grinding were as follows:

a. For the 194 lb of 4250 sq cm/g material:

Total Grinding Time, min	Wt of Grinding Media lb	Air-Permeability Fineness at End of Grinding sq cm/g	Specific Gravity
60	853	3565	2.70
120	496	3845	2.72
180	496	4250	2.74

b. For the 180 lb of 5860 sq cm/g material:

Total Grinding Time, min	Wt of Grinding Media lb	Air-Permeability Fineness at End of Grinding sq cm/g	Specific Gravity
90	853	4660	2.75
150	496	4960	--
240	496	5615	--
270	496	5860	2.78

29. The specific gravity of the fly ash before grinding was 2.44. The specific gravity of the ground material was considerably higher than that of the unground. The 4250 fineness value shown is based on two tests, the results of which were 4249 and 4254; the 5860 value is also based on two tests, the results of which were 5856 and 5864. Of the 194 and 180 lb of material, respectively, there were 190 and 173 lb of usable product available after grinding.

30. The relations of grinding time, surface area, and specific gravity for the fly ash tested are shown in fig. 3. It appears that the relation of increase in surface area to grinding time is essentially linear for each charge of grinding media used. Based on the slope of the surface area increase-grinding time curves for the 500-lb grinding-media charge, it would be predicted that a grinding time of approximately 10 hr and 20 min would have been required to increase the surface area of the fly ash from 1855 to 6000 sq cm/g. If the mill had been capable of handling the desired 850-lb ball charge, a grinding time of about 2 hr and 20 min is indicated.

31. The increase in specific gravity of the fly ash from 2.44 to

2.78 as a result of grinding from 1855 to 5860 sq cm/g was assumed to be due to (a) the effects of crushing porous particles (such as hollow glass spheres) having impermeable pore space and thus reducing or eliminating such pore space, and (b) the addition to the ground fly ash of metallic iron produced by wear of the grinding media and the mill liner during grinding. Assuming a specific gravity of 7.5 for iron, it would be necessary to add approximately 6% of metallic iron by weight to the charge of 190 lb of fly ash in order for the specific gravity of the fly ash-iron mixture to be increased from 2.44 to 2.78 by this factor alone. Chemical analysis of the fly ash for iron, calculated as Fe_2O_3, yielded the following results.

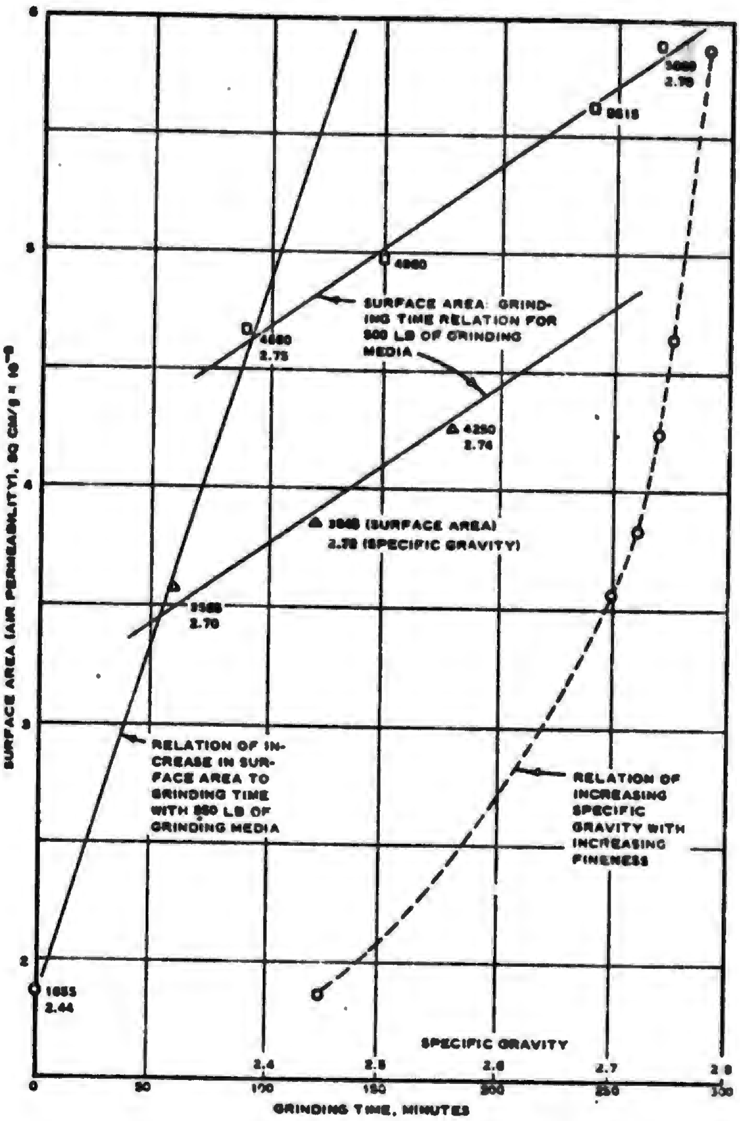

Fig. 3. Surface area-grinding time-specific gravity relations for Colbert fly ash (190-lb batch)

Fineness sq cm/g	Specific Gravity	Fe_2O_3, %
1855	2.44	25.41
4250	2.74	25.73
5860	2.78	26.04

32. The increase in iron content, Fe_2O_3, accompanying the indicated increase in surface area and specific gravity is only 0.63%. It is therefore concluded that the predominant cause of the increase in specific

gravity with increasing fineness produced by grinding is the loss of impermeable pore space.

Fly ash-portland-pozzolan cement

33. The results of grinding 75-lb portions of the clinker-fly ash mixtures with the 500-lb charge were as follows:

Blend No.	Composition	Desired Fineness ±500 sq cm/g	Grinding Time hr	Final Fineness sq cm/g*
1	85% clinker 15% fly ash	4010	1(+)	4485
2	80% clinker 20% fly ash	4010	1	3922
3	75% clinker 25% fly ash	4010	1	4140
4	65% clinker 35% fly ash	4010	1	3659
5	80% clinker 20% fly ash	5010	1-1/2	4743
6	80% clinker 20% fly ash	6010	2	5680

* These values were obtained in tests of grab samples taken to determine the extent of processing, and do not agree with final test results on carefully mixed samples.

34. For the first grind of clinker, fly ash, and gypsum (used in the proportion given in paragraph 21), the desired quantities were weighed out and then interground for successive 60-, 90-, or 120-min periods. After eight such periods, aggregating 12 hr of grinding, the product remained unsatisfactory due to the presence of lumps. Consequently, the material was discarded. It was suspected that an important factor in the continued presence of lumps was the moisture introduced into the mixture with the gypsum. Accordingly, when a new set of the desired quantities of materials was weighed out, the gypsum was first hand-ground to pass a No. 8 sieve. The clinker was weighed and then sieved over a No. 4 sieve; the particles coarser than No. 4 were passed through a hammer mill and returned to the batch. The gypsum was dried in the sun after having been weighed. The ball charge, which had become coated in the initial grinding, was cleaned by using it to grind some dry sand. After a single intergrinding

operation of 60 min, a fineness of 4200 sq cm/g was attained for the clinker-fly ash-gypsum mixture, but approximately 1-1/2 lb of clinker remained in the form of particles coarser than a No. 20 sieve. These particles were returned to the mill alone and ground for an additional 60 min. This additional grinding pulverized only about half of the clinker lumps. The lumps were then run through a disk pulverizer and the product was returned to the batch. The fineness of the final product was 4485 sq cm/g. The weight of the ball charge before and after grinding was 493 lb.

35. Subsequent grinds of clinker, gypsum, and fly ash were made on weighed quantities with the clinker particles coarser than No. 4 being hammer-milled before grinding, the gypsum being hand-ground to pass the No. 8 sieve before grinding and then sun-dried, and the clinker particles coarser than the No. 20 sieve after grinding being reduced by the disk pulverizer.

Petrographic Examinations and X-ray Diffraction Studies

Petrographic procedures

36. Gypsum. A representative sample of the gypsum was examined with the stereoscopic microscope and as immersion mounts with the petrographic microscope.

37. Portland-cement clinker. Thin sections were made from samples of the clinker and examined with the petrographic microscope.

38. Fly ash. Representative samples of the fly ash as received, after it was ground to the same fineness as the cement, and after it was ground to finenesses of 4250 and 5860 sq cm/g were examined as immersion mounts with the petrographic microscope. The particle shape and size distribution of the fly ash as received were noted. Estimates of amounts of glass, magnetite, carbon, and crystalline material were made on about 25 immersion mounts. The range in indices of refraction and predominant indices of refraction of the glass were determined. In the examination of the two samples of ground fly ash, particular attention was paid to the effects of grinding on the particle shape. Known weights of the three size groups of fly ash were separated with a Sepor laboratory magnet. The magnetic portions were weighed and the percentage of magnetic material was

calculated. Both the nonmagnetic and magnetic portions were examined as immersion mounts.

X-ray diffraction procedure

39. Clinker, portland cement, gypsum, and fly ash were examined using a General Electric XRD-3 X-ray diffractometer with No. 2 detector. Nickel-filtered copper radiation was used on all samples except the fly ash, which because of its high iron content required iron radiation to obtain a pattern that could be satisfactorily interpreted. All samples were run as powders packed in aluminum sample holders; samples that were not sufficiently fine when they were received were ground until they could pass the No. 325 sieve. The cement, clinker, and gypsum samples were run at 0.2 degree per min using a 1-degree slit, a 3-degree beam slit as a soller slit, and a 0.2-degree detector slit in the range from 2 to 20 degrees 2-theta. Beyond 20 degrees 2-theta, the 3-degree beam slit, MR soller slit, and 0.2-degree detector slit were used. The 1-3-0.2-degree collimation was used over the entire angular range studied for the fly ash samples. The target angle was 4 degrees; log scale, 4000; time constants, 30-7.5-2; I.S., 3.5 volts; reverter acceptance, 96%.

Results of examinations

40. Gypsum. The sample consisted of slightly damp, very light gray, sand-sized, broken crystals and crushed, medium-light-gray lumps as large as about 3/4 in. in maximum dimension. In immersion mounts, gypsum made up about 97% of the whole, with traces of anhydrite and goethite. The X-ray diffractometer trace showed that the sample examined consisted of about 95% gypsum with about 5% anhydrite.

41. Unground fly ash. This sample resembled other fly ashes examined by WES, but was much coarser. It was composed of glass, opaque carbon and magnetite, hematite, and a small amount of crystals that could not be identified by optical methods. Most of the unground fly ash consisted of spherical and ellipsoidal beads of glass and magnetite, with smaller amounts of irregularly rounded composite grains of similar composition, and some very irregular carbon particles. The grains ranged from larger than [illegible] in diameter to less than 1μ, the predominant particle size being between 40 and 15μ. About 95% of the particles were larger than 5μ. The index of refraction of the glass ranged from 1.500 to 1.630, with most of

the glass having indices in the range from 1.530 to 1.560. The particles of lower index tended to be clear, in part vesicular, and somewhat larger in size than the rest of the glass particles. The glass of higher index was generally green or amber. A few bright red particles with an index much higher than 1.630 were presumed to be hematite, or hematite enclosed in glass. Black spheres with a dull metallic luster in reflected light, assumed to be magnetite, and a much smaller number of red spheres with high refractive index made up from 15 to 25% of the sample. Irregularly shaped, large carbon particles made up 5 to 10% by volume of the sample (the carbon content determined by chemical analysis, paragraph 49, was 3.56%). Many of the carbon particles were vesicular, others looked like splinters; a few were somewhat ellipsoidal. The original woody structure was seen on some of these particles. Transparent or translucent glass, glass with inclusions, and crystals made up 60 to 75% of the sample. Less than 25% of the glass was free from inclusions. The glass containing inclusions showed low over-all birefringence or birefringent spots between crossed nicols. Some of the inclusions were crystals, but many were bubbles. The particles containing inclusions were white in reflected light, but the glass spheres without inclusions were clear in reflected light. Less than 5% of the sample was crystalline material other than magnetite and hematite.

42. Results of the magnetic separations were:

Condition of Material	Magnetic Portion % by Weight
As received	42.0
Ground to 4250 sq cm/g	40.1
Ground to 5860 sq cm/g	45.4

The results are not entirely accurate, since examination of the magnetic and nonmagnetic separates revealed that neither was free of foreign particles. The magnetic portion contained glass spheres that were obviously nonmagnetic, and the nonmagnetic portion contained weakly magnetic particles, particularly in the ground samples. Magnetic and nonmagnetic particles were found to be feebly attracted to each other by some force, probably static electricity, so as to form mixed aggregates. The aggregation was partially overcome by placing the fly ash in distilled water and stirring with a glass rod before magnetic separation. The magnetic portion was then separated by putting the magnet in the container and removing the magnetic material. Had this procedure not been used, all of the material ground to 5860 sq cm/g would have been picked up by the magnet.

43. Ground fly ash. The material ground to finenesses of 4250 and 5860 sq cm/g in the laboratory ball mill was examined to observe the effect of grinding on particle shape and to detect any change in composition, particularly an increase in iron produced by abrasion of the grinding balls. The 5860 sq cm/g sample consisted of from 30 to 50% glass and magnetite spheres, with the rest consisting of dustlike particles of very irregular shape in aggregates and broken spheres. A few particles larger than 40μ were observed, but the great majority were less than 10μ in diameter. The ground sample appeared to contain more opaque material than the unground sample. The aggregation of the particles and their extremely small size made it difficult to estimate the amount of opaque material present, but a conservative estimate would be more than 25%. The material ground to 4250 sq cm/g was intermediate between the unground and the sample ground to 5860 sq cm/g. The grains were larger, less broken, and less dusty than in the finer sample. If pieces of metallic iron from the grinding balls were present, they were not detected because of their close resemblance to crushed magnetite.

44. The X-ray examination of the three samples of fly ash agreed with the petrographic results, and established that quartz and mullite were present. The crystalline phases in the fly ash samples are listed below in order of decreasing abundance:

Crystalline Constituent	Order of Decreasing Abundance
Magnetite	1
Quartz	2
Hematite	3
Mullite	4

45. Carbon was not identified by X-ray because the carbon in fly ashes is very poorly crystalline, has a low absorption coefficient, and is hard to detect in the presence of quartz and mullite. The diffraction patterns of the three sizes of fly ash were practically identical. Corresponding lines on each pattern were almost equal in intensity. No elemental iron was detected in either of the two ground samples.

46. Clinker and cement. The thin sections of the portland-cement

clinker showed that it was fine-grained even as compared with modern cements produced in the eastern United States, and that the clinker pebbles were very porous. The most conspicuous constituents were phenocrysts of colorless alite in subhedral crystals with many inclusions, quite closely packed, with the interstices filled with ragged to prismatic, dark greenish brown pleiochroic aluminoferrite. Groups of rounded belite crystals were spottily distributed and had a yellowish brown tint in plane light and higher birefringence between crossed nicols than the alite. No tricalcium aluminate was definitely recognized. Parts of the large open pores in the clinker were filled with calcium hydroxide. The X-ray diffraction patterns of the clinker and cement indicate that the proportion of alite to belite was 9 to 1 or greater; aluminoferrite was quite abundant and tricalcium aluminate quite minor. Some of the clinker patterns contained free CaO, less than 1% in all probability, and some of the cement and clinker patterns contained calcium hydroxide. The recognizable calcium sulfate in the cement was anhydrite and hemihydrate; no gypsum was found.

Summary of results

47. Examination of the materials used in the experimental cement yielded the results listed below.

a. The gypsum sample consisted of at least 95% gypsum, with a very small amount of anhydrite, and a trace of goethite.

b. The unground fly ash was coarse and unusually rich in iron oxides as compared to fly ashes examined previously. About 42% of the fly ash as received was magnetic. The major constituents were glass and magnetite, with minor quartz, hematite, mullite, and some carbon. Ball-milling tended to concentrate the glass in the broken, and the opaque material in the unbroken portions. The indices of refraction of the glass ranged from 1.500 to 1.630, with most lying in the range from 1.530 to 1.560. These values are within the ranges of those determined on fly ashes previously examined at the WES.

c. The cement clinker and cement are fine-grained as compared with many modern cements produced in the eastern United States. The X-ray patterns indicated that the proportion of alite (C_3S with substituted ions) to belite (C_2S with substituted ions) was greater than 9 to 1; aluminoferrite was quite abundant; tricalcium aluminate was very minor; there were small amounts of periclase (MgO) and free CaO. The calcium sulfate in the cement was in the forms of hemihydrate and anhydrite. The relative abundance and ratio of the silicates suggest a Type III cement.

Discussion of results

48. It appears reasonable to expect that the fly ash, clinker, and calcium sulfate examined will make a satisfactory portland-pozzolan cement. The fly ash would be improved as a pozzolan if the iron oxide were removed by magnetic separation. The iron oxide concentrate might be useful as fine sand in concrete to be used for radiation shielding, or it might be sold to the steel mills in the Birmingham, Alabama, area, or elsewhere within economic shipping range.

Chemical Analysis

49. Results of the chemical analyses were:

Test Result	Cement RC-454	Clinker RC-454-C	Gypsum RC-454-G	Fly Ash AD-94(3)
Moisture, %	---	---	19.44*	0.26
SiO_2, %	19.71	20.53	---	43.29
Al_2O_3, %	5.34	5.62	---	18.19
Fe_2O_3, %	4.32	4.32	---	25.41
CaO, %	64.94	65.24	33.52	3.57
MgO, %	1.64	1.69	---	0.78
SO_3, %	2.30	0.36	45.92	0.50
S, %	---	---	---	0.00
Total carbon, %	---	---	---	3.56
Ignition loss, %	1.59	1.60	---	4.15
Insoluble residue, %	0.14	0.12	---	86.95
Na_2O, %	0.10	0.11	---	0.23
K_2O, %	0.30	0.31	---	1.84
Total alkalies as Na_2O, %	0.30	0.31	---	1.44
C_3S, %	65.9	64.5	---	---
C_2S, %	6.9	10.3	---	---
C_3A, %	6.8	7.6	---	---
C_4AF, %	13.1	13.1	---	---
$CaSO_4$, %	3.9	0.6	---	---

* Moisture loss at 890 C.

50. The petrographic examination of the gypsum revealed that of the material examined in immersion mounts with the petrographic microscope about 97% consisted of gypsum with traces of anhydrite and goethite, and that of the sample examined by X-ray diffraction about 95% was gypsum and about 5% anhydrite. From the results of the chemical analysis, the following computations were made:

$$\text{Moles } SO_3 \text{ in 100 g} = \frac{45.92}{80.06} = 0.5736$$

$$\text{Moles CaO in 100 g} = \frac{33.52}{56.08} = 0.5977$$

$$\text{Moles CaO excess} = 0.0241$$

$$\text{Moles } CaSO_4 \text{ in 100 g} = 0.5736$$

$$\text{Moles } H_2O \text{ for gypsum} = 0.5736 \times 2 = 1.1472$$

$$\text{Moles } H_2O \text{ in 100 g} = \frac{19.44}{18.016} = 1.0790$$

$$\text{Moles } H_2O \text{ deficient} = 0.0682$$

$$\text{Moles anhydrite} = 0.0341$$

$$\text{Moles gypsum} = 0.5395$$

$$\% \text{ anhydrite} = 0.0341 \times 136.14 = 4.64$$

$$\% \text{ gypsum} = 0.5395 \times 172.17 = 92.89$$

Heat of Hydration

51. Results of tests for heat of hydration by the heat-of-solution method were:

Material	Surface Area sq cm/g	Heat of Hydration, cal/g at 7 Days	28 Days
Cement	(n) 3940	75.0	87.6
Blend 1 (85:15)	(n) 4170	72.9	84.3
Blend 2 (80:20)	(n) 3865	66.4	79.5
Blend 3 (75:25)	(n) 4090	66.7	77.4
Blend 4 (65:35)	(n) 4035	61.6	71.7
Blend 5 (80:20)	(n+1000) 4610	73.6	84.9
Blend 6 (80:20)	(n+2000) 5705	75.4	86.7

52. These results indicate that the three blends containing 20, 25, and 35% fly ash (blends 2, 3, 4) and ground to the same approximate fineness as the Type I cement had values for heat of hydration at both 7 and 28

days that would comply with the optional maximum limits of 70 and 80 cal per g, respectively, that are applicable to Type II portland cement.

53. The effects of fly ash content and fineness on heats of hydration are shown in fig. 4. There is an indicated reduction of 10 cal per g

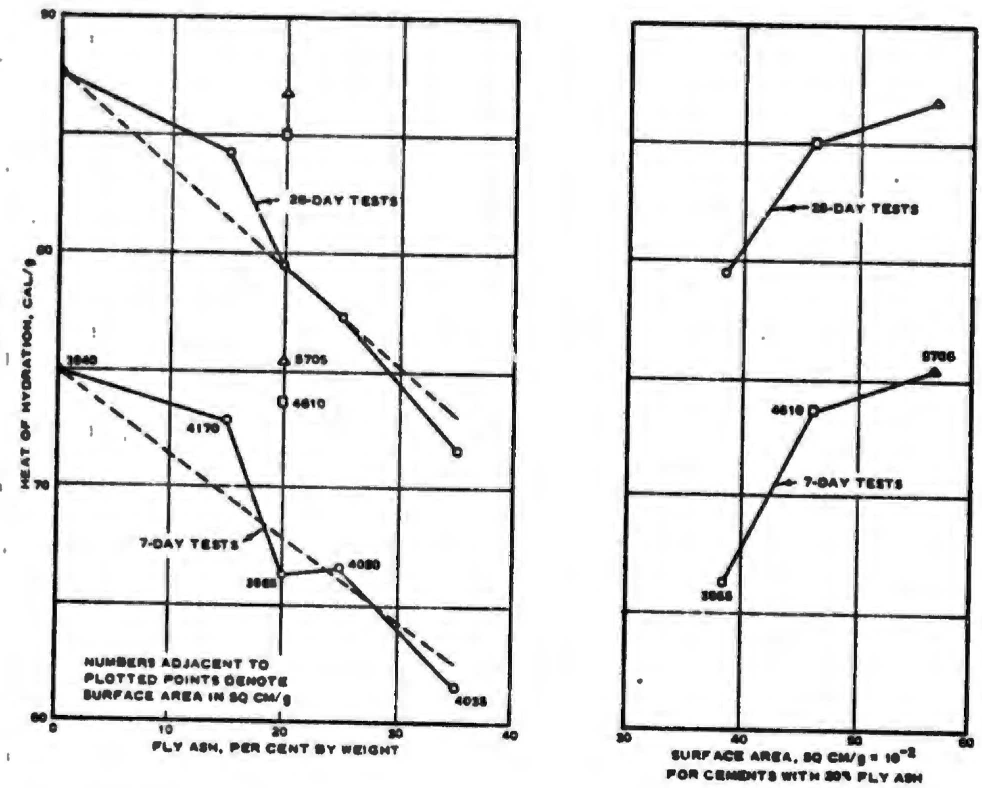

Fig. 4. Effects of fly ash content and fineness on 7- and 28-day heats of hydration of fly ash-portland-pozzolan cements

in both 7- and 28-day heats of hydration for each 25% of fly ash replacement of cement for equal surface area of about 4000 sq cm/g. It is also indicated that for a 20% replacement by fly ash, an increase in fineness of 2000 sq cm/g will increase the heat of hydration to a level approximately equal to that of the cement without fly ash.

Physical Tests

54. Results of tests for fineness, specific gravity, soundness, time of setting, compressive strength of mortar, air content of mortar, early stiffening, water requirement, bleeding, mortar expansion, drying shrinkage, and sulfate resistance are given in table 1 for materials and blends tested.

55. Relations of compressive strength to fly ash content and fineness, similar to those shown for heat of hydration in fig. 4, are shown in fig. 5. For fly ash-portland cement having a fineness of about 4000 sq cm/g, these data tend to suggest an optimum fly ash content of about 25% by weight.

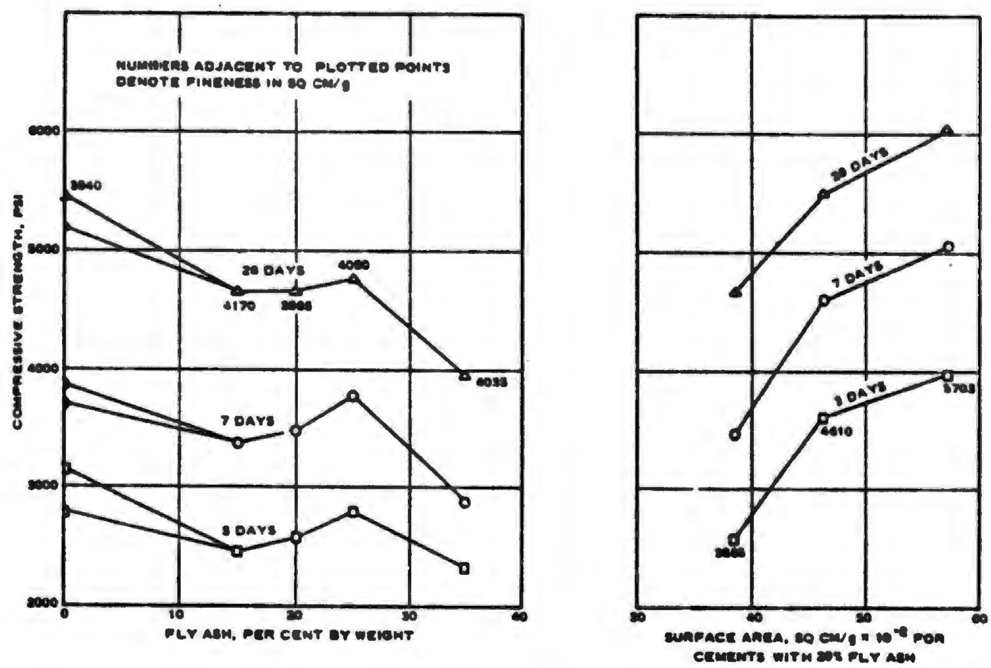

Fig. 5. Effect of fly ash content and fineness on 3-, 7-, and 28-day compressive strengths of fly ash-portland-pozzolan mortars

56. The relation between the specific gravity of the components of a portland-pozzolan cement, the proportions in which they are used, and the specific gravity of the product are illustrated in fig. 6. From such relations it should be possible to compute the actual fineness of the fly ash constituent, assuming that the variation in specific gravity of fly ash with fineness is as indicated in fig. 3 (page 15). The relation of actually determined specific gravities to those calculated graphically from the relation shown in fig. 6 is shown in fig. 7. The theoretical specific gravities are based on the assumption that in a portland-pozzolan cement of 4000 sq cm/g surface area, the fly ash and the portland cement will each have surface areas of 4000 sq cm/g and specific gravities equal to those possessed by the two ingredients when each is separately ground to that surface area; plus the further assumption that the specific gravity of the portland-cement clinker constituent does not change with change in surface

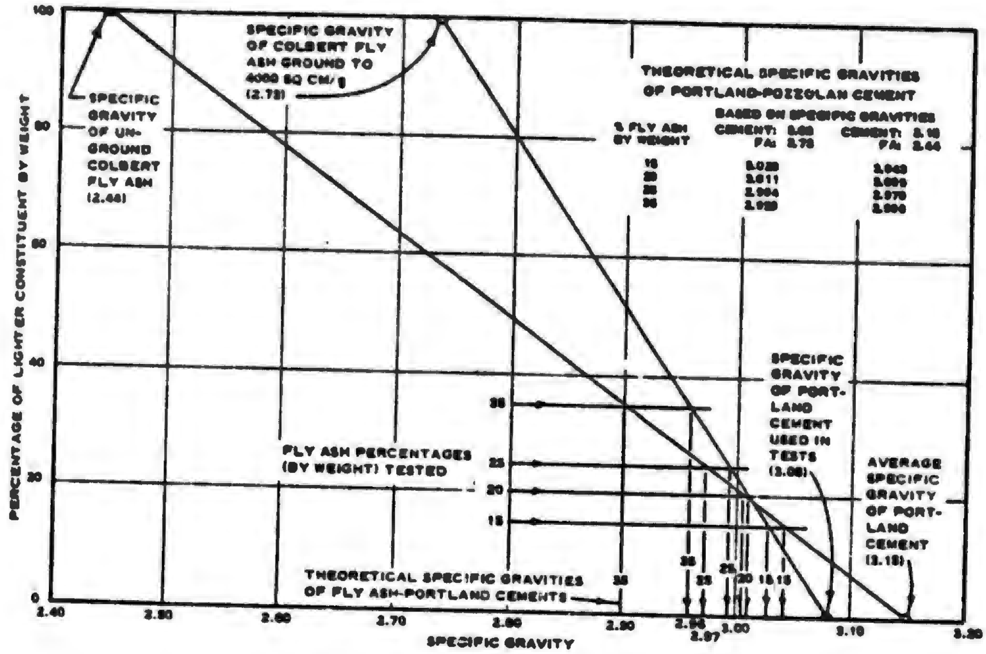

Fig. 6. Relation of fly ash content to specific gravity of fly ash-portland-pozzolan cement

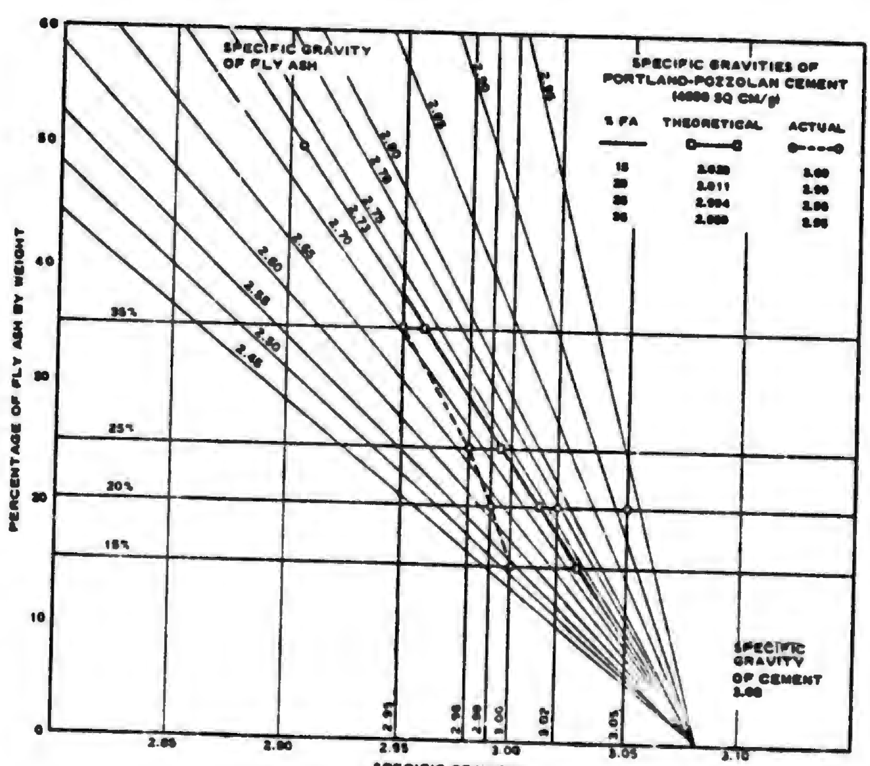

Fig. 7. Relation of theoretical and actual specific gravities of fly ash-portland-pozzolan cement

area within the limits of interest. Fig. 3 shows that the fly ash had a specific gravity of 2.72 at a surface area of 3845, and 2.74 at 4250; consequently, a specific gravity of 2.73 was assumed for a surface area of 4000 sq cm/g. The theoretical and actual specific gravities are:

	Assumed for Fly Ash		Sp Gr, Portland-Pozzolan Cement		Indicated		
					Fly Ash		Portland-Cement Clinker
Fly Ash %	Fineness sq cm/g	Sp Gr	Theoretical	Actual	Sp Gr	Surface Area sq cm/g	Surface Area sq cm/g
15	4000	2.73	3.028	3.00	2.54	2200	4330
20	4000	2.73	3.011	2.99	2.62	2900	4280
25	4000	2.73	2.994	2.98	2.68	3400	4200
35	4000	2.73	2.959	2.95	2.71	3700	4160

The values for indicated surface area of the portland-cement clinker constituent given in the last column above were obtained from the relations shown in fig. 8. The relations indicated in fig. 8 suggest that for the particular fly ash and portland-cement clinker studied and the proportions and grinding facilities employed, the fly ash was harder to grind than was the clinker (see paragraph 12), since for all proportions it is indicated that the 4000 sq cm/g product included fly ash having a surface area less than 4000 sq cm/g and clinker having a surface area greater than 4000 sq cm/g. The additional indicated relation, that the difference between the finenesses of the component constituents decreases as the percentage of the more resistant component increases, suggests that had the ingredients been used in approximately equal proportions their

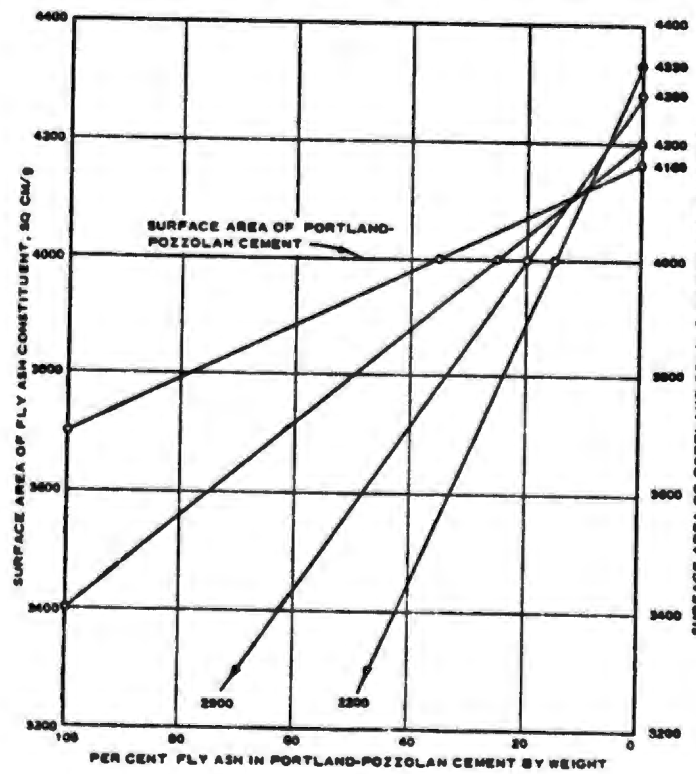

Fig. 8. Relation of surface areas of constituents of portland-pozzolan cements

surface areas might have been more nearly equal. The relation of the theoretical and actual curves in fig. 7 does not, however, suggest that the curves will cross. To the extent that these indications may apply to commercial grinding equipment, they suggest that economical production of fly ash-portland-pozzolan cement intended to have given properties may be achieved best by either pregrinding the components separately or by pregrinding a mixture of the components in other than the final proportions, followed by a final intergrinding of the desired product in selected proportions.

PART IV: DISCUSSION OF RESULTS AND CONCLUSIONS

Comparison of Test Results with Applicable Specifications

Specifications for portland cement

57. The Type I cement (RC-454) complied with the requirements of Federal Specifications for portland cement, SS-C-192b, in all respects. It may be noted that the strength and fineness limits were greatly exceeded, as indicated below.

		Specification Minimum	Cement RC-454 1st Specimen*	Cement RC-454 2nd Specimen**
Fineness, sq cm/g		2800	4010	
Compressive strength, psi	3 days	1200	3125	2760
	7 days	2100	3695	3865
	28 days	3500	5440	5190

* Specimens made 28 July.
** Specimens made 22 August.

58. Since the cement used greatly exceeded the specified minima, it follows that it could be blended or interground with a considerable amount of a less cementitious material and still comply with the specification limits.

Specifications for portland-pozzolan cement

59. Federal Specifications for portland-pozzolan cement, SS-C-208b, included herein as Appendix A, and ASTM Tentative Specifications for Portland-Pozzolan Cement, Designation: C 340-58T, give limits which may be compared with performance as developed in the experimental cements in the tabulation on the following page.

60. Except for two of the 80:20 blends (2 and 5) in which slightly more than the 1.0% allowable amount retained on the No. 100 sieve was found, all interground cements tested met all ASTM requirements and all Federal requirements.

Test Result	Federal Specification	ASTM Specification	Experimental Blends 1 85:15	2 80:20	3 75:25	4 65:35	5 80:20	6 80:20
MgO, %, max	---	5.0	Note 2					
SO_3, %, max	2.5	2.5	Note 3					
Moisture content, %, max	3.5	---	Note 4					
Ignition loss, %, max	3.0	3.0	Note 5					
Retained on: No. 100 sieve, %, max (Note 1)	---	1.0	0.6	1.4	0.6	0.4	1.3	0.7
No. 325 sieve, %, max	12	12.0	3.5	5.1	3.3	2.5	2.8	1.3
Surface area, air permeability, sq cm/g, min	2900	---	4170	3865	4090	4035	4610	5705
Soundness, exp, %, max	0.5	0.5	-0.03	-0.02	-0.03	-0.03	-0.03	-0.03
Setting: Initial, min, min	60	60	3:00	2:40	2:05	1:40	2:42	3:52
Final, hr, max	10	10	5:18	4:45	6:35	7:05	6:42	6:30
Compressive strength, psi, min:								
7 days	1500	1500	3385	3460	3760	2870	4595	5030
28 days	3000	3000	4640	4660	4735	3920	5510	6010
Air content, %, max	12	12	Note 6		---	2.9	---	---
Water requirement: Max, ml	320	---	244	240	238	234	238	244
Max, %	---	64						
Shrinkage. %, max	0.12	0.15	0.09	0.08	0.08	0.08	0.09	0.10
Expansion, %: 14 days, max	0.020	0.020	Note 6		---	-0.009	---	---
8 weeks	0.060	0.060	Note 6		---	-0.013	---	---
Early stiffening, differential max*	17	---	Note 6		---	7	---	---

* SS-C-208b will call for this when revision now in progress is completed.

Note 1. Both specifications provide that a pozzolan that is not to be interground must have not more than 12.0% by weight retained on a No. 325 sieve, and the Federal also requires that it have an air-permeability surface area of at least 3000 sq cm/g. The Colbert fly ash having 18.2% by weight retained on the No. 325 sieve and a surface area of 1855 sq cm/g does not comply with these requirements. The foregoing comparisons are thus made on the interground cements.

Note 2. MgO not determined on blends since percentage of MgO was found to be 1.69 in the clinker and 0.78 in the fly ash; thus, percentage of MgO in the blends would necessarily be less than 5.0.

Note 3. SO_3 not determined on blends since gypsum was added to provide 2.30 as in the reference Type I portland cement.

Note 4. Moisture content not determined since these experimental cements were made dry.

Note 5. Ignition loss not determined on blends since ignition loss of clinker was 1.60% and that of fly ash 4.15.

Note 6. Air in mortar, expansion of mortar, and early stiffening tests made only on the blend of maximum fly ash content and on the reference Type I cement; since both of these gave results within the applicable limits the other blended cements were not tested.

Conclusions

61. The data developed in this investigation suggest the following conclusions.

- a. Fly ash produced at the TVA Colbert, Alabama, steam generating plant is a satisfactory pozzolanic material for use as an ingredient in portland-cement concrete.
- b. Colbert fly ash, as now collected, will not meet current specifications for pozzolanic admixtures for use in concrete because it is too coarse.
- c. Colbert fly ash when ground in a ball mill and blended with commercial portland cement can yield a satisfactory portland-pozzolan cement.
- d. Satisfactory portland-pozzolan cement can also be produced by intergrinding Colbert fly ash, portland-cement clinker, and gypsum.
- e. The rate of chemical activity of portland-pozzolan cements containing ground Colbert fly ash decreases with increasing fly ash content for a given fineness, and increases with increasing fineness for a given fly ash content.
- f. The optimum fly ash content and fineness of a portland-pozzolan cement made with Colbert fly ash will depend on the use for which it is intended, the properties of the portland-cement clinker employed in its manufacture, and the properties of the portland cement or cements with which it is intended to compete.
- g. Portland-pozzolan cements having performance characteristics such as to comply with the requirements for Type I or Type II portland cement could be produced using Colbert fly ash. A product complying with the optional requirements on heat of hydration given in the Federal Specifications for Type II portland cement could be made from fly ash and Type I portland-cement clinker such as those used in these studies if 20% or more by weight of the cement were fly ash and the product ground to a specific surface (air permeability) of approximately 4000 sq cm/g.
- h. The desired rate of chemical activity for a combination of Colbert fly ash and a given portland-cement clinker can probably be obtained at more than one combination of fly ash content and fineness; the optimum fly ash content-fineness combination will depend on relative cost of the cement and fly ash, and the cost of grinding to produce additional surface area.
- i. Since the fly ash appears to be harder to grind than the portland-cement clinker, and since the difference in relative grindability appears to increase as the percentage of

fly ash decreases, it may be more economical to grind fly ash-portland-pozzolan cements in stages rather than in a single operation.

LIST OF REFERENCES

1. American Society for Testing Materials, "Tentative specifications for portland-pozzolan cement." 1958 Book of ASTM Standards, Part 4, Designation: C 340-58T, pp 15-21.

2. _________, "Tentative specifications for fly ash for use as an admixture in portland cement concrete." 1958 Book of ASTM Standards, Part 4, Designation: C 350-57T, pp 616-618.

3. Avery, W. M., "Fly ash teams up with portland cement to make better concrete." Pit and Quarry, vol 39 (May 1947), pp 157-159.

4. Blanks, R. F., "Fly ash as a pozzolan." Journal American Concrete Institute, Proceedings, vol 46 (1950), p 701.

5. Davis, Raymond E., "Use of pozzolans in concrete." Journal American Concrete Institute, Proceedings, vol 46 (1950), pp 377-384.

6. Davis, Raymond E., Carlson, Roy W., Kelly, J. W., and Davis, Homer E., "Properties of cements and concretes containing fly ash." Journal American Concrete Institute, Proceedings, vol 33 (1937), pp 577-612.

7. Davis, Raymond E., Davis, Homer E., and Kelly, J. W., "Weathering resistance of concretes containing fly ash cements." Journal American Concrete Institute, Proceedings, vol 37 (1941), pp 281-296.

8. G. and W. H. Corson, Inc., Fly Ash and Slag, Their Use in Portland Cement Products. Plymouth Meeting, Pa., June 1948 (mimeographed).

9. Jarrige, M. A., "Les utilisations des cendres volantes dans la construction." Annales de l'Institut Technique du Bâtiment et des Travaux Publics, No. 138 (June 1959), pp 521-544.

10. Leonard, George K., and Schwab, Philip A., "TVA uses non-specification fly ash." Civil Engineering (New York), vol 28 (March 1958), pp 188-192.

11. Nelles, J. S., Pilcher, J. M., and Vilbrandt, F. C., "Concrete exposed to sulfur water." Journal American Concrete Institute, Proceedings, vol 37 (1941), p 441.

12. U. S. Army Engineer Waterways Experiment Station, CE, "Corps of Engineers specifications for pozzolan as an admixture for use in portland-cement concrete." Handbook for Concrete and Cement, CRD-C 262-57.

13. U. S. General Services Administration, Federal Specification, Cement; Portland-Pozzolan, SS-C-208b. 9 April 1954, with Amendment 1, 17 May 1955 (included in Handbook for Concrete and Cement as CRD-C 206-55).

Table 1

Results of Physical Tests

	Type I Port-land Cement	Colbert Fly Ash	Mixed Blends of 80% 4010 sq cm/g Cement to 20% Fly Ash of Fineness of 1855 sq cm/g	4250 sq cm/g	5860 sq cm/g	Interground Portland-Cement Clinker and Fly Ash in Percentages of 85 PC 15 FA	80 PC 20 FA	75 PC 25 FA	65 PC 35 FA	80 PC 20 FA	80 PC 20 FA
Fineness, air permeability, sq cm/g	3940	1855	----	----	----	4170	3865	4090	4035	4610	5705
Fineness, per cent retained											
No. 100 sieve	0.04	2.2	----	----	----	0.6	1.4	0.6	0.4	1.3	0.7
No. 325 sieve	5.6	18.2	----	----	----	3.5	5.1	3.3	2.5	2.8	1.3
Specific gravity	3.08*	2.44	----	----	----	3.00	2.99	2.98	2.95	3.02	3.05
Soundness, autoclave expansion, %	0.00	----	-0.02	-0.03	-0.03	-0.03	-0.02	-0.03	-0.03	-0.03	-0.03
Time of setting (Gillmore)											
Initial, hr:min	1:25	----	1:20	3:09	3:07	3:00	2:40	2:05	1:40	2:42	3:52
Final, hr:min	3:00	----	2:50	5:34	5:30	5:18	4:45	6:35	7:05	6:42	6:30
Compressive strength, psi											
3 days**	3185	----	2075	2370	2715	2435					
3 days†	2760	----	----	----	----	----	2550	2705	2300	3995	3950
7 days**	3695	----	2905	3475	3395	3305	----	----	----	----	----
7 days†	3865	----	----	----	----	----	3460	3760	2070	4995	5030
28 days**	5440	----	3900	4510	4790	4640	----	----	----	----	----
28 days†	5190	----	----	----	----	----	4660	4735	3980	5510	6010
Air content of mortar, %	7.1	----	----	----	----	----	----	----	2.9	----	----
Early stiffening, penetration, mm											
Initial	37	----	----	----	----	----	----	----	33	----	----
Final	28	----	----	----	----	----	----	----	26	----	----
Difference	9	----	----	----	----	----	----	----	7	----	----
Water requirement for normal consistency, ml	250	----	244	246	246	244	240	238	234	238	244
Bleeding											
Rate, ml/sq cm/sec x 10^{-6}	65	----	89	61	53	73	50	49	64	55	9
Capacity, ml/ml x 10^{-3}	15	----	24	19	17	6	7	7	10	9	3
Mortar expansion, %											
14 days	-0.002	----	----	----	----	----	----	----	-0.009	----	----
8 weeks	+0.005	----	----	----	----	----	----	----	-0.013	----	----
Drying shrinkage,†† %											
28 days	0.08	----	----	----	----	0.09	0.08	0.08	0.08	0.09	0.10
Sulfate resistance (lean mortar bars)‡											
Expansion, %											
28 days	0.016	----	0.019	----	----	0.016	0.018	----	0.023	----	0.021
90 days	0.033	----	0.036	----	----	0.027	0.029	----	0.037	----	0.046
1 year	----	----	----	----	----	----	----	----	----	----	----
Sulfate resistance (added sulfate)‡‡											
Expansion, %											
28 days	0.039	----	0.040	----	----	0.034	0.037	----	0.045	----	0.038
90 days	0.070	----	0.080	----	----	0.055	0.066	----	0.086	----	0.056
1 year	----	----	----	----	----	----	----	----	----	----	----

* The specific gravity value of 3.08 for RC-454 was confirmed by check tests. A later sample of commercial Type I cement produced by the same mill in November 1959 had a specific gravity of 3.16 as determined both by the producer and the WES.
** Data in this line from specimens made 28 July 1959.
† Data in this line from specimens made 22 August 1959.
†† Average of 3 specimens.
‡ Average of 4 specimens.
‡‡ Average of 6 specimens.

SS–C–208b

APRIL 9, 1964
SUPERSEDING
Fed. Spec. SS–C–208a
18 November 1961

with

AMENDMENT - 1
MAY 17, 1965

APPENDIX A

FEDERAL SPECIFICATION

CEMENT; PORTLAND-POZZOLAN

This specification was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

1. CLASSIFICATION

1.1 Types.—The portland-pozzolan cement covered by this specification shall be of the following types, as specified.

Type I.—Portland-pozzolan cement non-air-entraining.

Type IA.—Portland-pozzolan cement, air-entraining.

2. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

2.1 The following Federal Specifications, of the issues in effect on the date of the invitation for bids, shall form a part of this specification:

SS–C–192—Cements; Portland.

SS–C–158—Cements, Hydraulic Methods for Sampling, Inspection and Testing.

(Non-Government activities may obtain copies of Federal Specifications and Standards upon application accompanied by check, money order, cash, or Government Printing Office coupons, to the General Services Administration, Business Service Center, Region 3, Seventh and D Streets SW., Washington 25, D. C. This office will also honor deposit account numbers issued by the Government Printing Office. Copies of the Index of Federal Specifications and Standards may be obtained from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D. C. Prices may be obtained from the Index of Federal Specifications and Standards or from the GSA Regional Offices.

(Single copies of this specification and other product specifications required by non-Government activities for bidding purposes are available without charge at the GSA Regional Offices in Boston, New York, Atlanta, Chicago, Kansas City, Mo., Dallas, Denver, San Francisco, Los Angeles, Seattle, and Washington, D. C.

(Government activities may obtain copies of Federal Specifications and Standards and the Index of Federal Specifications and Standards through departmental channels from the established sources of supply.)

2.2 Other publications.—The following publication of the issue in effect on date of invitation for bids forms a part of this specification.

American Society for Testing Materials Publication:

ASTM Standard C 188—Method of Test for Specific Gravity of Hydraulic Cement.

(ASTM Standard are published by the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.)

2.3 Specifications and other publications applicable only to individual departments are listed in section 7.

3. REQUIREMENTS

3.1 Portland cement or portland-cement clinker used in the portland-pozzolan cement shall conform to the requirements for chemical composition for type I, Federal Specification SS–C–192. Pozzolans used in portland-pozzolan cement shall conform to the requirements specified in 3.4.

3.2 The percentage of pozzolan in the portland-pozzolan cement shall not be less than 15 percent nor more than 35 percent, by weight. The manufacturer shall state the source, amount, and composition of the pozzolan used in the finished portland-pozzolan cement. The amount of pozzolan in the finished cement shall not vary more than ±5.0 percentage points by weight from the amount stated by the manufacturer.

3.3 Detail requirements.—The portland-pozzolan cement shall conform to the requirements for chemical properties prescribed in table I, and to physical properties prescribed in table II.

TABLE I.—*Chemical requirements*

Test	Type I	Type IA
	Percent	*Percent*
Sulfur trioxide (SO_3), maximum	2.5	2.5
Moisture content, maximum	3.0	3.5
Ignition loss, maximum	3.0	3.0

TABLE II.—*Physical requirements*

Test	Type I	Type IA
Fineness:		
Residue No. 325 sieve, maximum, percent	12	12
Blaine fineness meter:		
Average value, minimum, sq. cm./g	2900	2900
Minimum value, any one sample, sq. cm./g	2700	2700
Soundness, autoclave expansion, maximum, percent	0.5	0.5
Time of setting, Gillmore test:		
Initial, minimum, minutes	60	60
Final, maximum, hours	10	10
Compressive strength: (4.4.6) minimum:		
1 day in moist air, 6 days in water, lb./sq. in	1500	1250
1 day in moist air, 27 days in water, lb./sq. in	3000	2500
(The strength at 28 days shall be greater than at 7 days.)		
Air entrainment: percent by volume	0–12	15–21
Water requirement, maximum, ml	320	280
Drying shrinkage (4.4.11), maximum, percent	0.12	0.11
Mortar expansion:		
At age of 14 days, maximum, percent	0.020	0.020
At age of 8 weeks, maximum, percent	0.060	0.060
False set, minimum, penetration mm	10	10
(This requirement applies only when specifically requested in the invitation for bids.)		

3.4 **Pozzolan.**—Pozzolan which is blended with finished portland cement to produce portland-pozzolan cement shall meet the requirements prescribed in table III. The fineness requirements need not apply to pozzolan which is interground with portland-cement clinker to produce the portland-pozzolan cement. For evaluating the contribution to compressive strength, coarse pozzolans shall be ground only to the extent of having a residue on the No. 325 sieve of 12±2 percent. For materials such as diatomaceous earth with essentially no residue on a No. 325 sieve, the minimum residue need not apply.

TABLE III.—*Physical requirements—Pozzolan*

Test	Type P [1]	Type F [2]
Fineness:		
Residue No. 325 sieve, maximum, percent	12.0	12.0
Blaine fineness meter, minimum, sq. cm./g	-------	3000
Contribution to compressive strength (4.5.3):		
Percent of control 28 days, minimum	75	85
(The compressive strength of the portland-pozzolan cubes at 90 days shall not be less than the strength at 28 days.)		

[1] Type P refers to natural materials such as clays, shales, diatomaceous earths, tuffs, volcanic ash and pumicite, either calcined or uncalcined. Many natural pozzolans are improved by calcining between 1,400° F. and 1,800° F.

[2] Type F refers to fly ash, an artificial pozzolan which is a fine ash collected from the flue gases at the stacks of power plants burning pulverized coal.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 **Inspection.**—The manufacturer shall provide suitable facilities to enable the inspector to check the relative weights of portland cement and pozzolan used to produce the portland-pozzolan cement. The plant facilities for blending and inspection shall be adequate to insure compliance with the requirements in 3.2.

4.2 **Sampling.**—The Government will sample the portland cement and pozzolan prior to blending or the portland-cement clinker and pozzolan prior to intergrinding, and will sample the portland-pozzolan cement. One 5-pound sample of portland-cement or portland-cement clinker shall be taken from approximately each 400 tons. One 5-pound sample shall be taken from approximately each 100 tons of portland-pozzolan cement. Samples of portland cement and samples of portland-pozzolan cement, except samples for false set tests, shall be taken from the conveyor delivering the materials to the bins. A 5-pound sample of portland-pozzolan cement for use in false set tests shall be taken from each car prior to shipment when the purchaser has requested that this test be applied. Samples of portland-cement clinker and of pozzolan shall be taken from bins, or from lots of these materials that have been so stored as to prevent mixing with other lots and prevent contamination.

4.3 The Government will test the portland-cement or portland-cement clinker,[1] the pozzolan and the portland-pozzolan cement. Tests of portland-cement or portland-cement clinker for chemical composition shall be made on composite samples in accordance with Federal Specification SS-C-158. Tests of pozzolan for physical properties shall be made on composite samples in accordance with the provision of 4.5. Tests of portland-pozzolan cement for chemical composition and physical properties, except fineness and false set, shall be made on composite samples in accordance with provisions of 4.4. Fineness tests of portland-pozzolan cement shall be made on each 5-pound sample. The test for false set of portland-pozzolan cement (when required) shall be made on each car sample.

4.3.1 The specific gravity of the pozzolan and the portland-pozzolan cement shall be determined in accordance with the ASTM Standard C188, using kerosene as the liquid.

4.3.2 *Mixing mortars.*—Mortars for compressive strength, air-entrainment, drying shrinkage, mortar expansion of portland-pozzolan cement and for contribution to compressive strength of pozzolan, shall be mixed using the apparatus and mixing procedure specified in Federal Specification SS-C-158.

4.3.3 *Determination of flow and entrained air.*—Immediately after completion of the mixing, determine the flow of the mortar. If the mortar has the correct flow, determination of the entrained air should be made on the portion of the mortar not used for the flow determination. For other mortars, the part used for determination of the flow shall be returned to the bowl and the mortar remixed 15 seconds at the medium speed. Specimens shall then be made in the prescribed manner.

[1] If the portland-cement clinker has been ground in a steel laboratory mill, a magnetic separation of iron particles shall be made prior to chemical analysis.

4.4 **Methods of test, portland-pozzolan cement.**—Tests for chemical and physical properties of portland-pozzolan cement shall be made in accordance with the following methods:

4.4.1 *Sulfur trioxide.*—The sulfur trioxide content of the portland-pozzolan cement shall be determined in accordance with method 2 of Federal Specification SS-C-158, except that the pozzolan need not be completely decomposed by the acid.

4.4.2 *Moisture content.*—Heat a 1-gram sample in a heat-resistant porcelain crucible to constant weight in an oven at 105° C. The percent moisture content equals loss in weight times 100.

4.4.3 *Loss on ignition.*—The loss on ignition shall be determined in accordance with the method given in Federal Specification SS-C-158, except that the material remaining from the determination of moisture content shall be ignited to constant weight in the porcelain (not platinum) crucible at a temperature between 700° and 800° C. The weight after heating at 105° C. minus the weight after heating at 700° C. times 100 equals the percent loss in ignition.

4.4.4 *Fineness.*—The fineness of the portland-pozzolan cement shall be determined by the Blaine apparatus in accordance with the procedure described in Federal Specification SS-C-158, except that the determined specific gravity of the portland-pozzolan cement shall be used in computing the weight of the sample and the fineness. (In calculation of the constants for the apparatus, the K_4 formula shall be used. All the terms used in this formula refer to the standard sample. When fineness of a portland-pozzolan cement is determined it is desired to have a sample weight that will give a porosity as near as possible to the standard sample and yet insure proper compaction.)

4.4.5 *Residue No. 325 sieve.*—The test for material retained on the No. 325 sieve shall be made in accordance with the procedure for sieve determination described under Turbidimeter Fineness in Federal Specification SS-C-158.

4.4.6 *Compressive strength.*—Compressive strength shall be determined in accordance with Federal Specification SS-C-158.

4.4.7 *Time of set.*—Time of set shall be determined by the Gillmore test in accordance with Federal Specification SS-C-158.

4.4.8. *Soundness.*—Autoclave expansion of the portland-pozzolan cement shall be determined in accordance with Federal Specification SS-C-158.

4.4.9 *Air-entrainment.*—Air-entrainment of portland-pozzolan cement shall be determined in accordance with Federal Specification SS-C-158. The air content of the mortar shall be calculated using the general formula and the specific gravity of the cement being tested.

4.4.10 *Water requirement.*—The water requirement shall be determined as the milliliters of water required to produce a flow of between 100 and 115 on the flow table.

4.4.11 *Drying shrinkage.*—The mortar for the three shrinkage specimens shall be the same as the mortar used for compressive strength. The molds shall be the same as specified for autoclave test specimens in Federal Specification SS-C-158. The specimens shall be molded by placing a small amount of mortar around each reference insert and thoroughly tamping with a 1- by ½- by 6-inch tamper. Fill the molds and tamp 25 times with the above specified tamper held in a vertical position with the 1-inch side of the tamper held parallel to the sides of the mold. This tamping shall be with sufficient force and evenly distributed over the total surface of the bar so that uniform filling of the mold is obtained. After compaction the excess material shall be cut off flush with the top of the mold with a thin-edged trowel and the surface smoothed with a few strokes of the trowel. The shrinkage bars shall be cured 24 hours in the damp closet and then removed from the molds. They shall then be further cured for 6 days in water at 23° C. (73° F.) ± 1.7° C. (3° F.). The specimens shall then be read for length and stored at 23° C. (73° F.) ± 1.7° C. (3° F.) and 50 ± 2 percent relative humidity. The bars shall again be read for length change at 28 days after their initial reading. The average difference in length of the three specimens between initial and final reading shall be expressed as a percent of the effective gage length and shall be reported to the nearest 0.01 percent.

4.4.12 *Mortar expansion.*—The test for mortar expansion shall be made using molds and comparator as described in Federal Specification SS-C-158 for autoclave expansion test.

4.4.12.1 *Storage containers.*—Specimens shall be stored in containers made of noncorrodible material or in containers treated to prevent rusting. Water to a depth of 1 inch shall be provided in the containers at all times and the specimens shall be supported so that they cannot contact the water or the other specimens. Containers shall be sealed. Each container shall have an internal volume no greater than 1 cubic foot.

4.4.12.2 *Pyrex glass.*—Uncontaminated, No. 774, Pyrex glass (obtainable as "lump cullet" from the Corning Glass Works, Corning, N. Y.), crushed, graded, and washed shall be used as aggregate in the fabrication of expansive mortar bars. The grading shall be as follows:

	Percent
Retained on No. 100 sieve	100
Retained on No. 50 sieve	80
Retained on No. 30 sieve	60
Retained on No. 16 sieve	40
Retained on No. 8 sieve	20
Retained on No. 4 sieve	0

4.4.12.3 *Proportioning and mixing.*—The mortar for the mortar expansion test specimens shall consist of 900 grams of crushed and graded pyrex glass, 400 grams of cement, and enough water to give a flow of 100 to 115 on the flow table. The mortar shall be mixed as directed in 4.3.2.

4.4.12.4 *Molding of specimens.*—Molds shall be thinly covered with mineral oil; after this operation the reference gage points shall be set in the molds, care being taken to keep them free of oil. Three 1- by 1- by 11¼-inch specimens, having an effective gage length of 10 inches, shall be fabricated from each mortar batch. The specimens shall be molded as described in 4.4.11 for shrinkage bars.

4.4.12.5 *Treatment of specimens.*—After each mold has been filled, it shall be immediately placed in the moist closet and kept there until the specimen has developed sufficient strength for the mold to be removed without injuring the specimen, but, in any event, no longer than 24 hours. After 24 hours' curing in the moist closet, the specimens shall be placed in the storage container with a small amount of water. The container shall be sealed and placed in a cabinet or room kept at a constant temperature of 38° C. (100° F.) ± 1.7° C. (3° F.) for a period of 13 days. After the 24-hour curing period and before placing in storage, each specimen shall be measured in the comparator for length. When the specimens are 14 days old, they shall be measured again in the comparator for length. The container and specimens shall be cooled at 23° C. (73° F.) ± 1.7° C. (3° F.) for 5 hours before the container is opened and the final reading made. All readings shall be to the nearest 0.0001 inch. The average difference in length of the three specimens between initial and final readings shall be expressed as a percent of the effective gage length and shall be reported to the nearest 0.001 percent. After the readings at 14 days' age have been completed, the specimens shall be returned to the containers and these shall again be sealed and placed in the 38° C. (100° F.) cabinet or room. The specimens shall remain in this storage until they reach an age of 8 weeks when they shall again be measured in the comparator for length. The average difference in their length, between initial and 8 weeks' reading shall again be reported as a percent of the effective gage length.

4.4.13 *False set.*—The test for false set shall be made in accordance with the provisions of Federal Specification SS-C-158.

4.5 Methods of test, pozzolan.—Tests for physical properties of pozzolan shall be made in accordance with the following methods.

4.5.1 *Fineness.*—The fineness of the pozzolan shall be determined by the Blaine apparatus in accordance with the procedure described in Federal Specification SS-C-158, except that the determined specific gravity of the pozzolan shall be used in computing the weight of the sample and the fineness. (In calculation of the constants for the apparatus the K_4 formula shall be used. All the terms used in this formula refer to the standard sample. When the fineness of a pozzolan is determined, it is desired to have a sample weight that will give a porosity as near as possible to the porosity of the standard sample and yet insure proper compaction.)

4.5.2 *Residue retained on No. 325 sieve.*—The test for material retained on the No. 325 sieve shall be made in accordance with the procedure for sieve determination described under Turbidimeter Fineness in Federal Specification SS-C-158.

4.5.3 *Contribution to compressive strength.*—Compressive strengths of 2-inch plastic mortar cubes shall be made for a control mix containing portland cement, and a test mix of the same portland cement and pozzolan for comparison. These cubes shall be fabricated in accordance with Federal Specification SS-C-158, except for the following:

4.5.3.1 *Mix parts.*—The mix parts for the control mix shall be one part of any type I cement meeting Federal Specification SS-C-192, 2.75 parts of graded standard Ottawa sand, and sufficient water to obtain a flow of 100 to 115. The test-mix parts shall be identical to the control-mix parts, except that 35 percent by absolute volume of the control cement shall be replaced with pozzolan, and the water used shall be such as to produce a flow of 100 to 115. Mortars shall be mixed as directed in 4.3.2.

Example:	6-cube batch
Control mix	500 grams cement 1,375 grams Ottawa sand X = ml. of water for flow of 100 to 115
Test mix	325 grams of cement $175 \times \frac{\text{specific gravity of pozzolan}}{\text{specific gravity of cement}} =$ grams of pozzolan 1,375 grams of Ottawa sand Y = ml. of water for flow of 100 to 115

4.5.3.2 *Storage of specimens.*—When cube specimens are taken from the molds after 24 hours, they shall be placed in close-fitting containers, sealed airtight, and stored at 38° C. (100° F.) for 27 days, then removed from 38° C. (100° F.) room and stored at 23° C. (73° F.) ± 1.7° C. (3° F.) until the date of test. Cubes broken at 28 days shall be allowed to cool in the containers to 23° C. (73° F.) ± 1.7° C. (3° F.) for 5 hours before being broken. (Any container having a capacity for 3 cubes may be used if it can be made airtight. The use of containers made of light sheet metal with inside dimensions of approximately 2⅟₃₂ by 2⅟₃₂ by 6¼ inches is suggested.)

4.5.3.3 *Test ages.*—Compressive strength shall be determined at 28 and 90 days only. Three specimens shall be made for each age. (Ninety-day specimens of the control batch need not be made, but a 6-cube batch shall be mixed.)

4.5.3.4 *Percent of control strength.*—The percent strength is calculated by the following formula:

$$\text{Percent strength} = \frac{\text{average compressive strength of the test mix cubes, in p. s. i.} \times 100}{\text{average compressive strength of the control mix cubes, in p. s. i.}}$$

5. PREPARATION FOR DELIVERY

5.1 **Packaging.**—The portland-pozzolan cement shall be delivered in bags, barrels, or in bulk, as specified. A bag shall contain 0.48 cubic foot of material by absolute volume. All packages shall be in good condition at the time of inspection. Packages varying more than 5 percent from the weight corresponding to 0.48 cubic foot may be rejected and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected. The portland-pozzolan cement shall be packaged in commercial containers, unless otherwise specified by the purchaser in the invitation for bids, contract or order.

5.2 **Marking.**—When the cement is delivered in packages, the words "Portland-Pozzolan", the name of the manufacturer, and the type of the portland-pozzolan cement, whether it is air-entraining or non-air-entraining, the net weight and absolute volume shall be plainly indicated thereon. Similar information shall be provided in the shipping advices accompanying the shipment of packaged or bulk cement. The manufacturer shall also advise the purchaser of the source of the pozzolan and the amount used in manufacturing the portland-pozzolan cement and the composition of the pozzolan.

6. NOTES

6.1 **Intended use.**—The term "pozzolan" is used to designate siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties. The term "portland-pozzolan" cement is used to designate an interground mixture of portland-cement clinker, pozzolan and gypsum or an intimate and uniform blend of portland cement and fine pozzolan.

6.2 **Ordering data.**—Purchasers should specify type required and should exercise any desired options offered herein. (See 1.1, 3.4.5.1, 5.1 and section 7).

6.2.1 If purchaser does not specify the type of cement to be furnished, type I will be furnished.

6.2.2 If the purchaser does not specify that the portland-pozzolan cement be made with either type P or type F pozzolan, the manufacturer may choose for himself the type of pozzolan used. The manufacturer shall state in his proposal what type of pozzolan he will use and be governed by the requirements for that type.

6.2.3 If the purchaser desires that the portland-pozzolan cement be free of false set, he should specify that the test for false set apply in his invitation for bids.

6.3 It is believed that this specification adequately describes the characteristics necessary to secure the desired material, and that normally no samples will be necessary prior to award to determine compliance with this specification. If, for any particular purpose, samples with bids are necessary, they should be specifically asked for in the invitation for bids, and the particular purpose to be served by the bid sample should be definitely stated, the specification to apply in all other respects.

6.4 Federal specifications do not include all types, classes, grades, sizes, etc., of the commodities indicated by the titles of the specifications, or which are commercially available, but are intended to cover the types, etc. which are suitable for Federal Government requirements.

6.5 Transportation description.—The proper transportation description applicable is:

Cement, natural or Portland:
Carload weight 50,000 pounds.
Truckload weight 50,000 pounds.

Patent Notice.—When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

7. DEPARTMENTAL REQUIREMENTS

7.1 The following specifications, standard, and publication, of the issues in effect on date of invitation for bids, and special requirements, form a part of this specification for purchases made under this specification by the respective departments:

7.2 Army, Navy, and Air Force.

7.2.1 *Applicable specifications and standard.*

Military Specifications:

JAN-P-115—Packaging and Packing for Overseas Shipment—Compound, Sealing, Dipcoating.

MIL-T-704—Treatment and Painting of Materiel.

MIL-B-1190—Bags, Paper, Shipping, Multiwall (for Portland Cement).

Military Standard:

MIL-STD-129—Marking for Shipment and Storage.

(Copies of specifications and standards required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

7.2.2 *Other publications.*—The following document forms a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

Interstate Commerce Commission Regulation:

49 CFR 78.125—Single Trip Container Steel Drums (37D).

(The Interstate Commerce Commission regulations are now a part of the Code of Federal Regulations (1949 Edition—Revised 1950) available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for the above publication should cite "49 CFR 78-125 (Rev. 1950).")

7.2.3 *Packaging.*

7.2.3.1 *Multiwall paper bags.*—Unless otherwise specified, the cement shall be packaged in quantities of 0.48 cubic feet of material by absolute volume, in multiwall paper bags conforming to Military Specification MIL-B-1190, type E, with waterproofed ends. The bags shall be closed in conformance with the specification and the closure waterproofed with dipcoating sealing compound conforming to Military Specification JAN-P-115.

7.2.4 *Packing.*—Normally, no further packing will be required. When specified, cement in paper bags shall be palletized.

7.2.5 *Marking for shipment and storage.*—In addition to any special marking required by the contract or order, marking for identification, shipment, and storage shall be in accordance with Military Standard MIL-STD-129. The nomenclature shall be as follows:

CEMENT, PORTLAND-POZZOLAN
Type ________
Fed. Spec. SS-C-208

MILITARY INTEREST:

Army—E

Navy—MC Y

Air Force.