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RESEARCH SECTION  
Office of Materials  
Iowa Dept. of Transportation

**REPORT**

**ON**

**FLYASH VARIABILITY**



**HIGHWAY DIVISION**

**OFFICE OF MATERIALS**

**MARCH 1981**

REPORT  
ON  
FLY ASH VARIABILITY

by  
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## SUMMARY

The physical-chemical testing of fly ashes indicates that, under normal operating conditions, a low variability of results can be expected from a particular generating plant unit. However, unannounced changes in coal source and/or plant operations do occur and they may result in an ash with undesirable properties. Since these properties can be detected by physical-chemical testing, it is recommended that this testing be performed on a lot-by-lot basis when a plant is supplying fly ash to a construction project.

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## PURPOSE AND SCOPE

The purpose of this study of fly ashes was to characterize their variability so as to determine if future testing should be on a certified source basis or on a lot-by-lot basis.

The study concentrated on three of the fly ashes being marketed in the state.

## MATERIALS

The fly ashes used were obtained from Council Bluffs No. 3, Sioux City Port Neal No. 3 and Sioux City Port Neal No. 4 generating plants.

Several other ashes were also included on a limited basis. These were Dubuque, Nebraska City, Lansing, Clinton and North Omaha.

## LABORATORY PROCEDURES

The variability of the ashes was measured by performing the following physical-chemical tests:

## Chemical

<u>Test</u>	<u>Method</u>
Loss on Ignition	ASTM C311 & 114
Sulfur Trioxide	ASTM C311
Silicon Dioxide	ASTM C311 & 114
Aluminum Oxide	ASTM C311 & 114
Iron Oxide	ASTM C311 & 114
Total Oxide	ASTM C311 & 114
Moisture Content	ASTM C311
Alkali Equivalent	ASTM C311

## Physical

<u>Test</u>	<u>Method</u>
Autoclave Soundness	ASTM C141
Percent passing #325 Mesh	ASTM C430
Mortar Cube Strength	Adapted from ASTM C109
*Specific Gravity	ASTM C188
*Pozzanlic Activity	ASTM C311

\*Performed on every third sample

## TEST RESULTS AND INTERPRETATION

Variability

The mortar strength test was one of the least labor and time intensive tests run; therefore, information from it becomes available soon after samples are received. It was obvious from the first test results that Council Bluffs and Neal No. 4 were quite dissimilar. Council Bluffs set up much quicker when mixed with water and considerable more heat was evolved. Results from the other physical-chemical tests also confirmed the difference as being something other than natural or laboratory variability. The generating plant characteristics as to degree of coal pulverization, boiler design and efficiency, precipitator design, etc., are similar at both plants. Company personnel at both plants had indicated that the coal source was the Belle Ayr Mine at Gillette, Wyoming. In attempting to confirm this through the coal suppliers, it was determined that the coal source for Port Neal No. 4 is Carter Mining's Rawhide Ranch mine and the source for Council Bluffs is Amax Coal Co.'s Belle Ayr (50%)

and Eagle Butte (50% mines. Although these mines operate in the same coal unit, the Belle Ayr Mine lies eighteen miles south of the Eagle Butte Mine and the Rawhide operation lies four miles east of the Eagle Butte Mine.

The following list of coal sources for Iowa area generating plants includes ashes not studied in detail but included in a very limited way in the physical-chemical determinations.

<u>Plant Name</u>	<u>Coal Source</u>
Port Neal No. 3	Hannah South, Rosebud and Medicine Bow
Port Neal No. 4*	Rawhide Ranch Mine
Council Bluffs No. 3*	Belle Ayr and Eagle Butte Mines
Nebraska City*	Caballo Mine
North Omaha	Rosebud Mine
Lansing*	Belle Ayr and Eagle Butte Mines
Clinton	Illinois-Montana Blend
Dubuque	Illinois-Montana Blend

Although other coals may be burned in other units at these plants, the ash being collected, stored and marketed is derived from the coals listed.

The denoted (\*) plants call their coal source "Gillette, Wyoming coal"; however, the specific mines are separated by significant distances as the following map indicates (Figure 1).

At the same time that differences between Council Bluffs

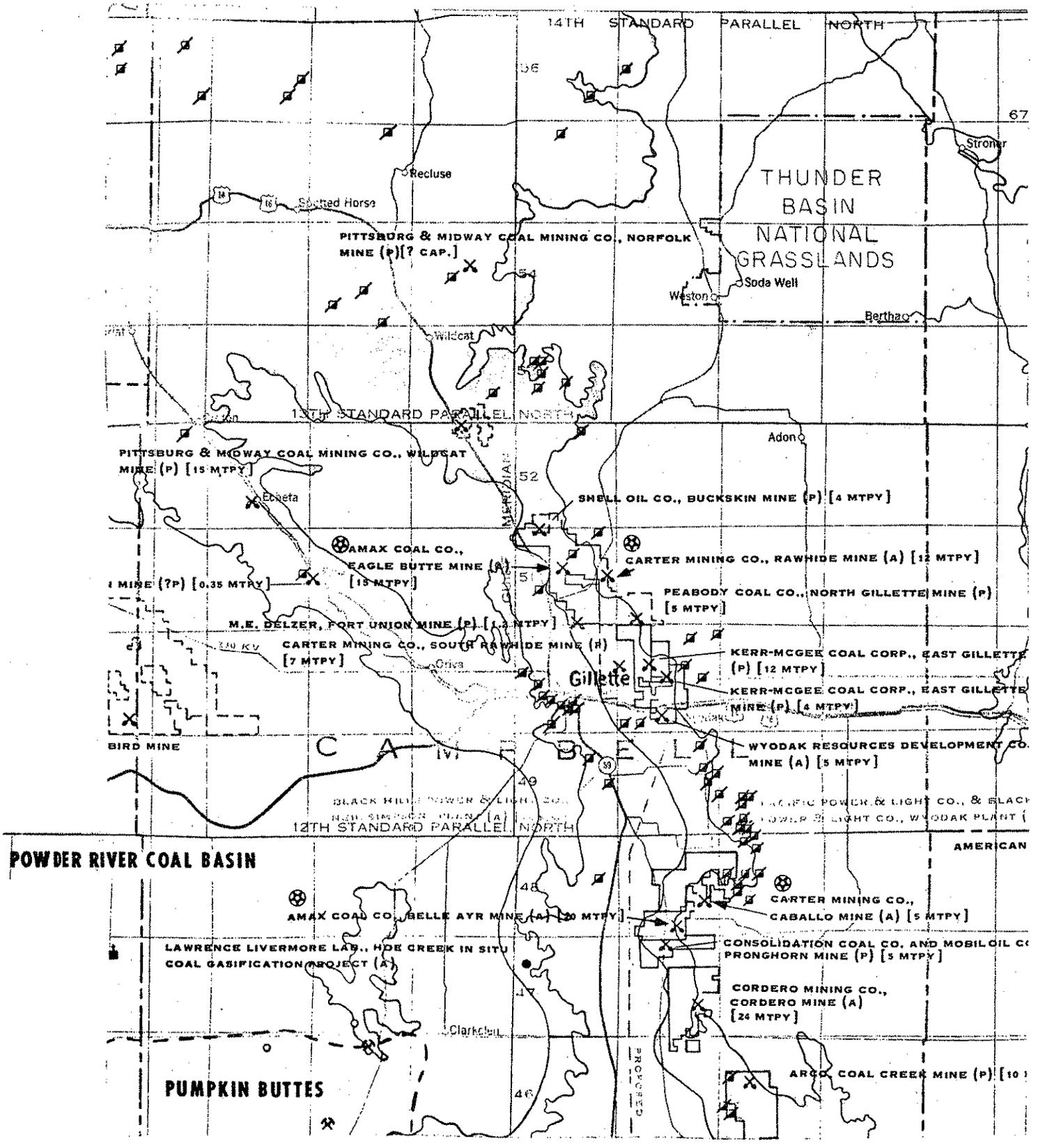


FIGURE 1

⊗ Locations supplying coal to Iowa and nearby plants

and Neal No. 4 were becoming evident, the characteristics of Council Bluffs ash also changed. The ash was noticeably faster setting after the plant came back on line after a six-week maintenance shutdown. Company personnel could offer no explanation for this, but a trip to the plant and subsequent conversations revealed that, prior to the shutdown, a boiler leak was releasing moisture into the exhaust gases. The hardening fly ash was then collected on the precipitator grids where it could not be removed during the normal "rapping" cycle. It was estimated that the precipitators were 50% more efficient after the boiler leak was repaired and the grids cleaned. Since the finer size fractions of fly ash are the most reactive, it is understandable that the ash would be more reactive when the precipitators are operating more efficiently.

This occurrence further supports the need for testing fly ash on a lot-by-lot basis and the need for a product history file on each source of fly ash.

The results of the physical and chemical tests to date are shown in Figures 2 thru Figure 12. The ASTM C618 or the Iowa Special Provision limits are shown on each figure.

#### Alkali Equivalent

Alkali equivalent values are of importance when fly ash is used in concrete containing reactive aggregates. Since Iowa has little trouble with alkali-aggregate reactions, the indicated ASTM limit should not be considered applicable. This test was run for characterization purposes only.



FIGURE 3  
LOSS ON IGNITION VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DURBUQUE 7 = CLINTON 9 = LANSTING

LOT	79960	79980	80000	80020	80040	80060	80080	80100	80120	80140
9										
8										
7										
6										
5										
4										
3										
2										
1										
0	4444 4 4 444 4		2 22 4 44 44	7 22 441 2	2 22 2 4444 4 444 444 444	2 22 2 44 4444 44	2 22 2 444 4434 44	2 22 33 3 3333	2 22 33 3 3333	1 4 35333343 3

6



FIGURE 5  
 MOISTURE CONTENT VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL#3 4 = NEAL#4  
 5 = NEB. CITY 6 = DUBOQUE 7 = CLINTON 9 = LANING

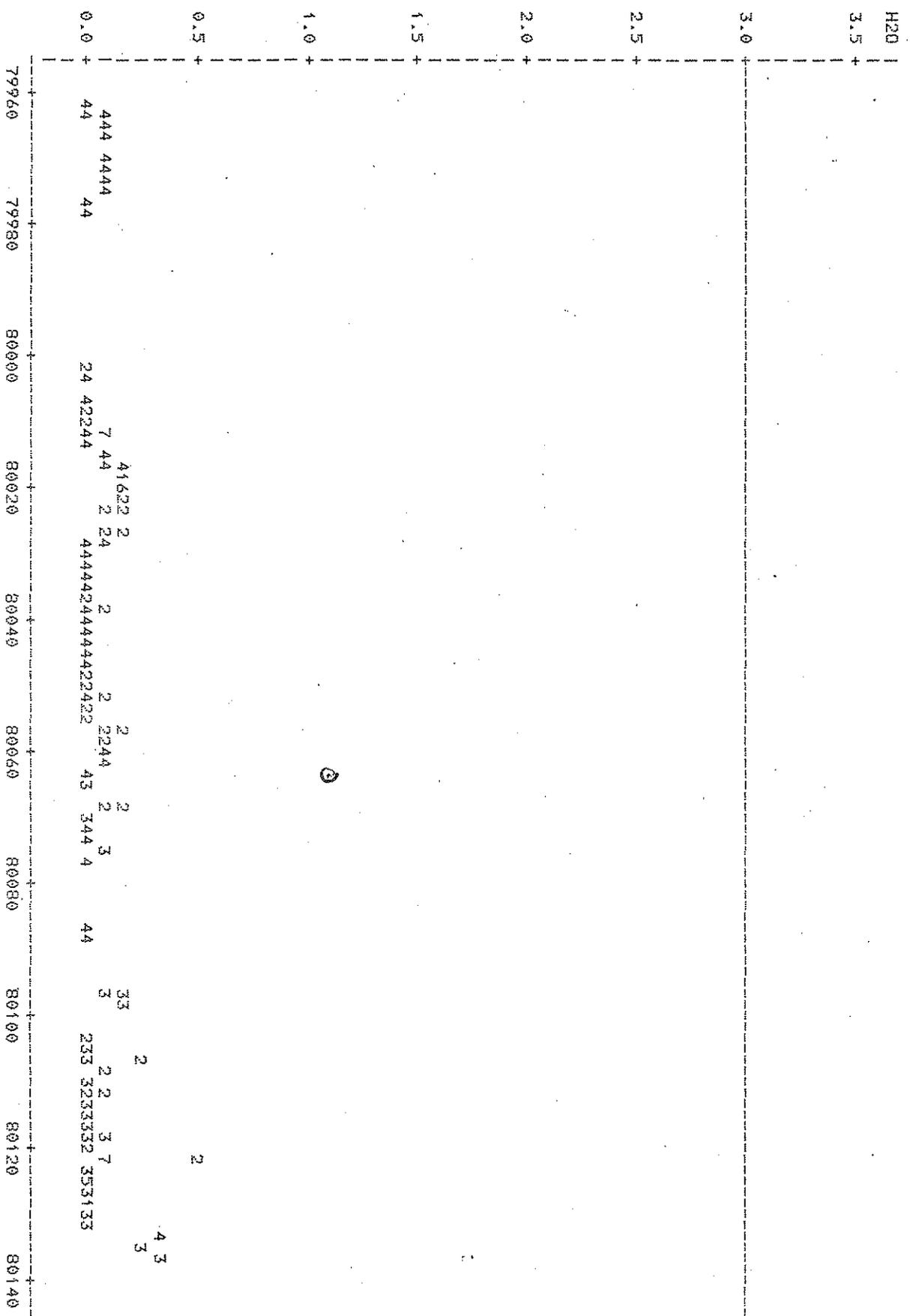


FIGURE 6.  
TOTAL OXIDES VS SEQUENCE NUMBER.

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DURBUQUE 7 = CLINTON 9 = LANSING

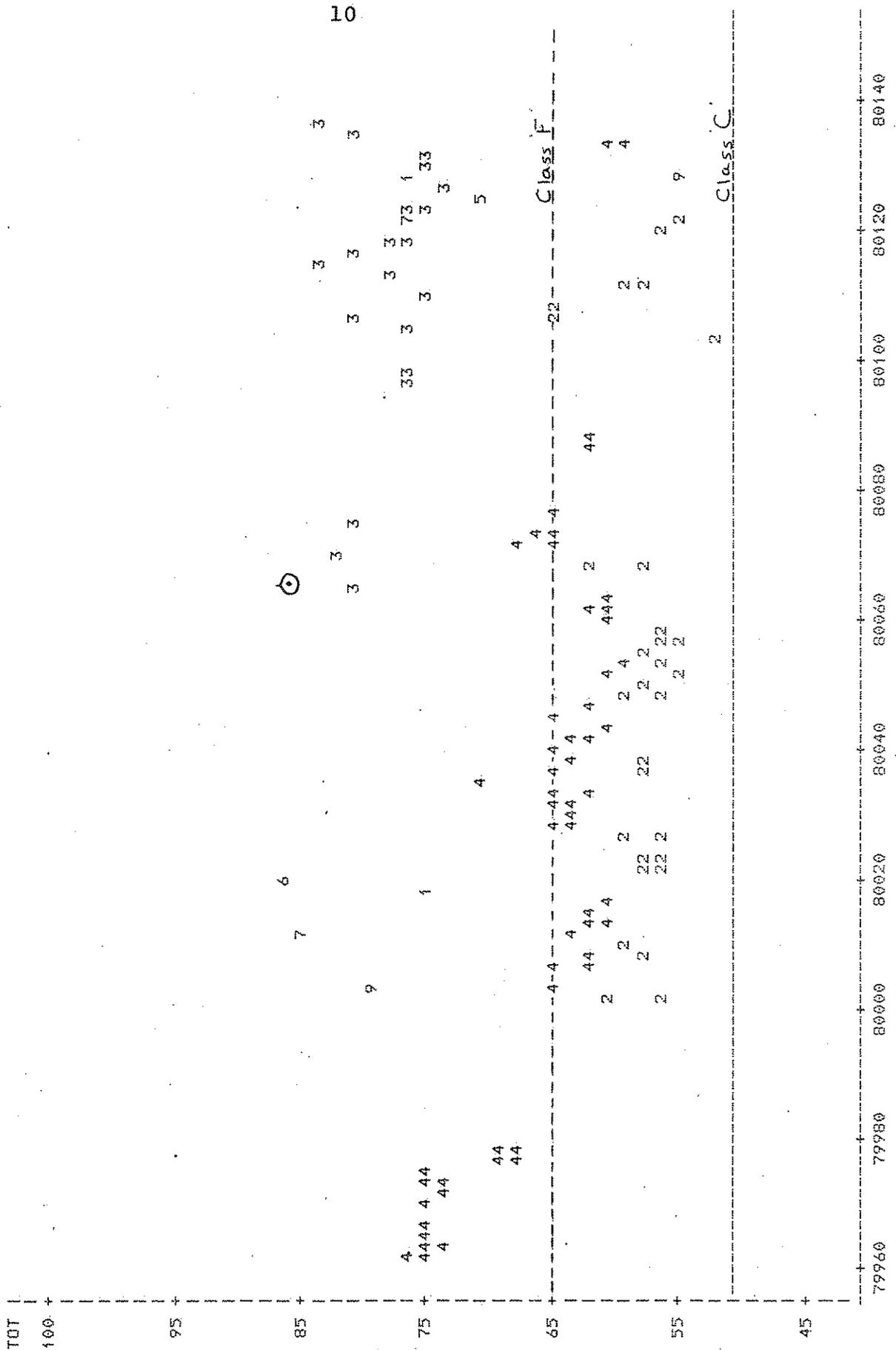


FIGURE 7  
 AUTOCLAVE EXPANSION VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DURBUQUE 7 = CLINTON 9 = LANSING

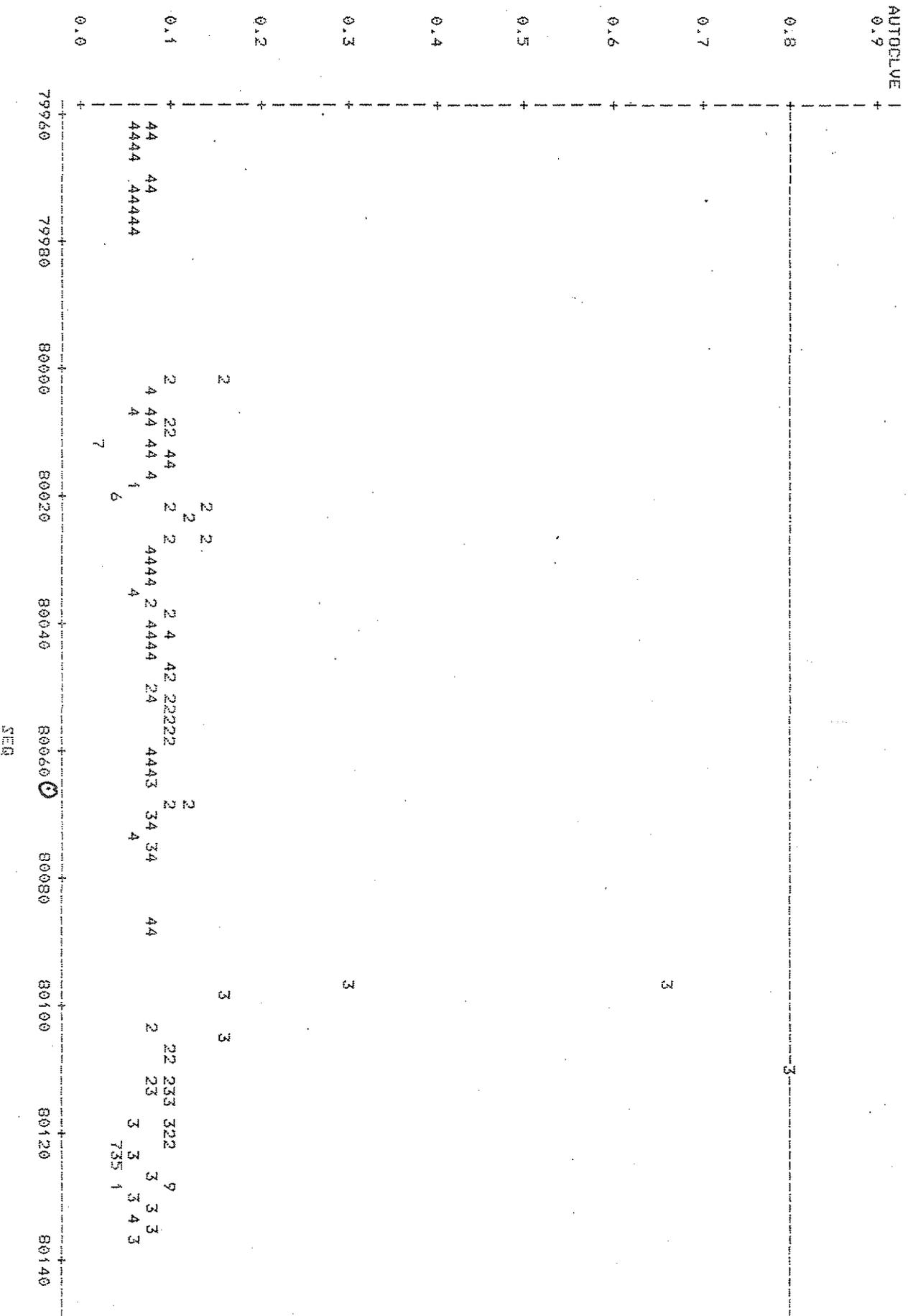


FIGURE 8  
 PERCENT PASSING #325 MESH VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DURORQUE 7 = CLINTON 9 = LANSING

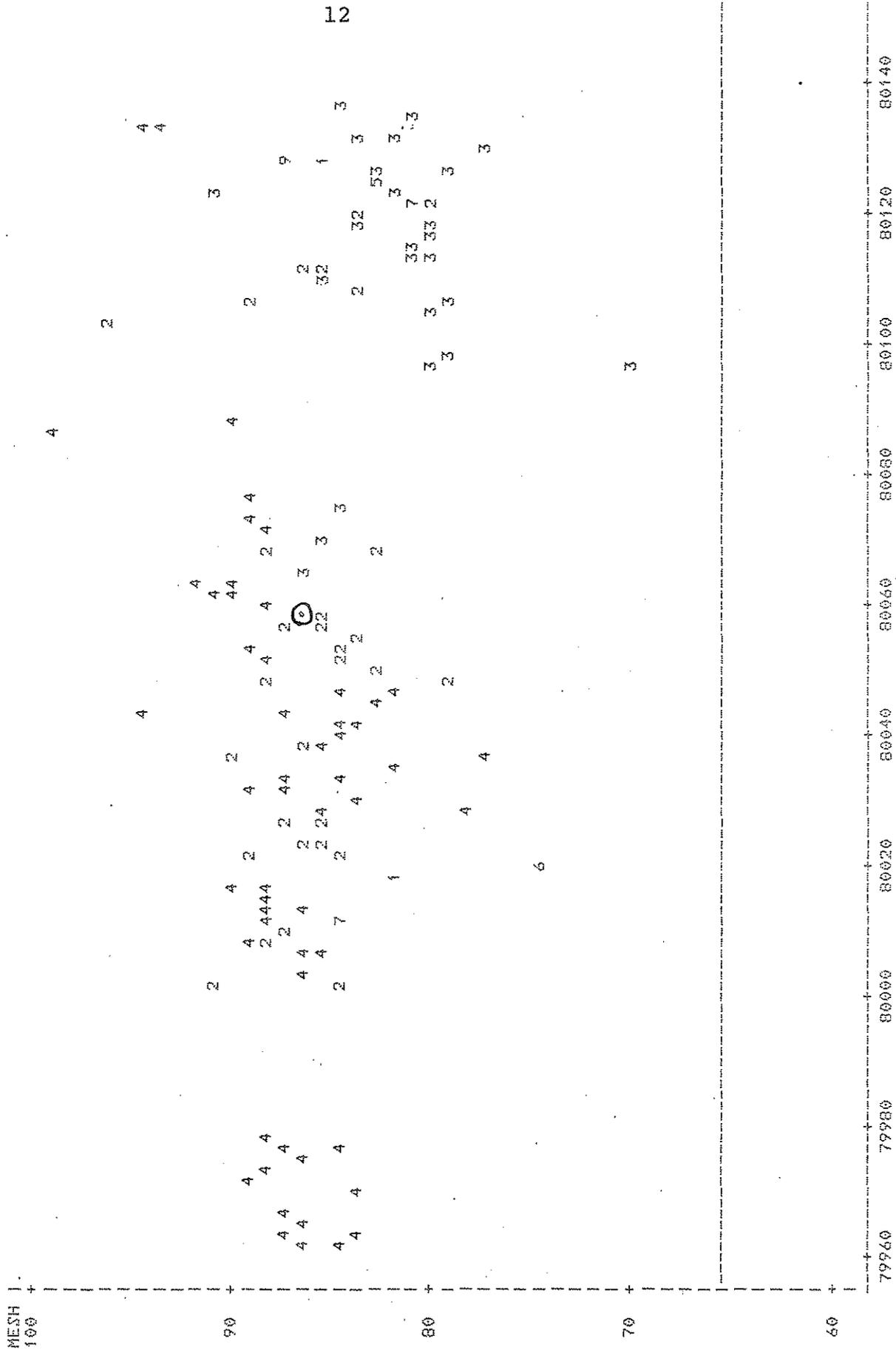


FIGURE 9  
3 DAY MORTAR CURE STRENGTH (PSI) VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DURBUQUE 7 = CLINTON 9 = LANING

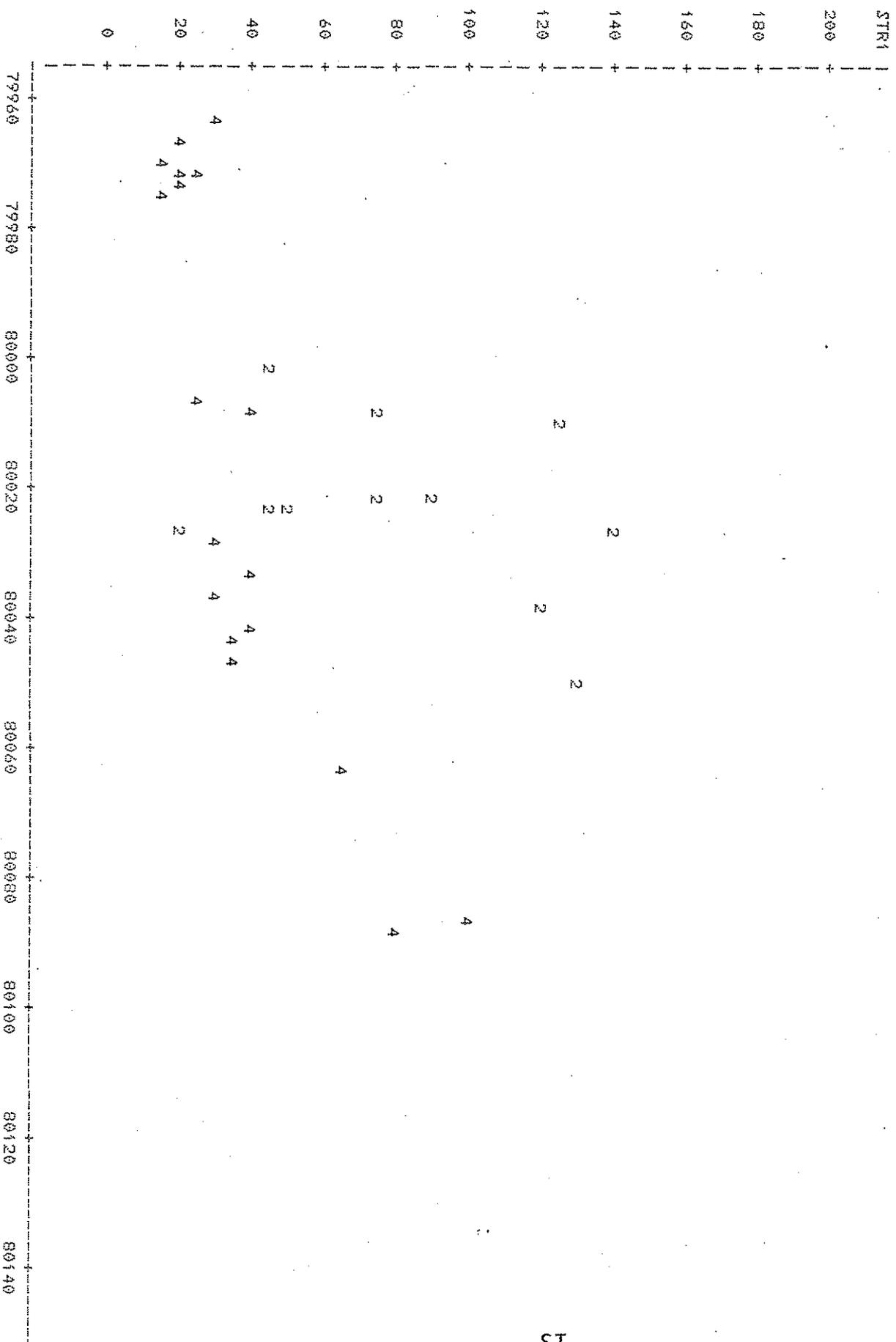


FIGURE 10  
7 DAY MORTAR CUBE STRENGTH (PSI) VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
5 = NEB. CITY 6 = DUBUQUE 7 = CLINTON 8 = LANSTING

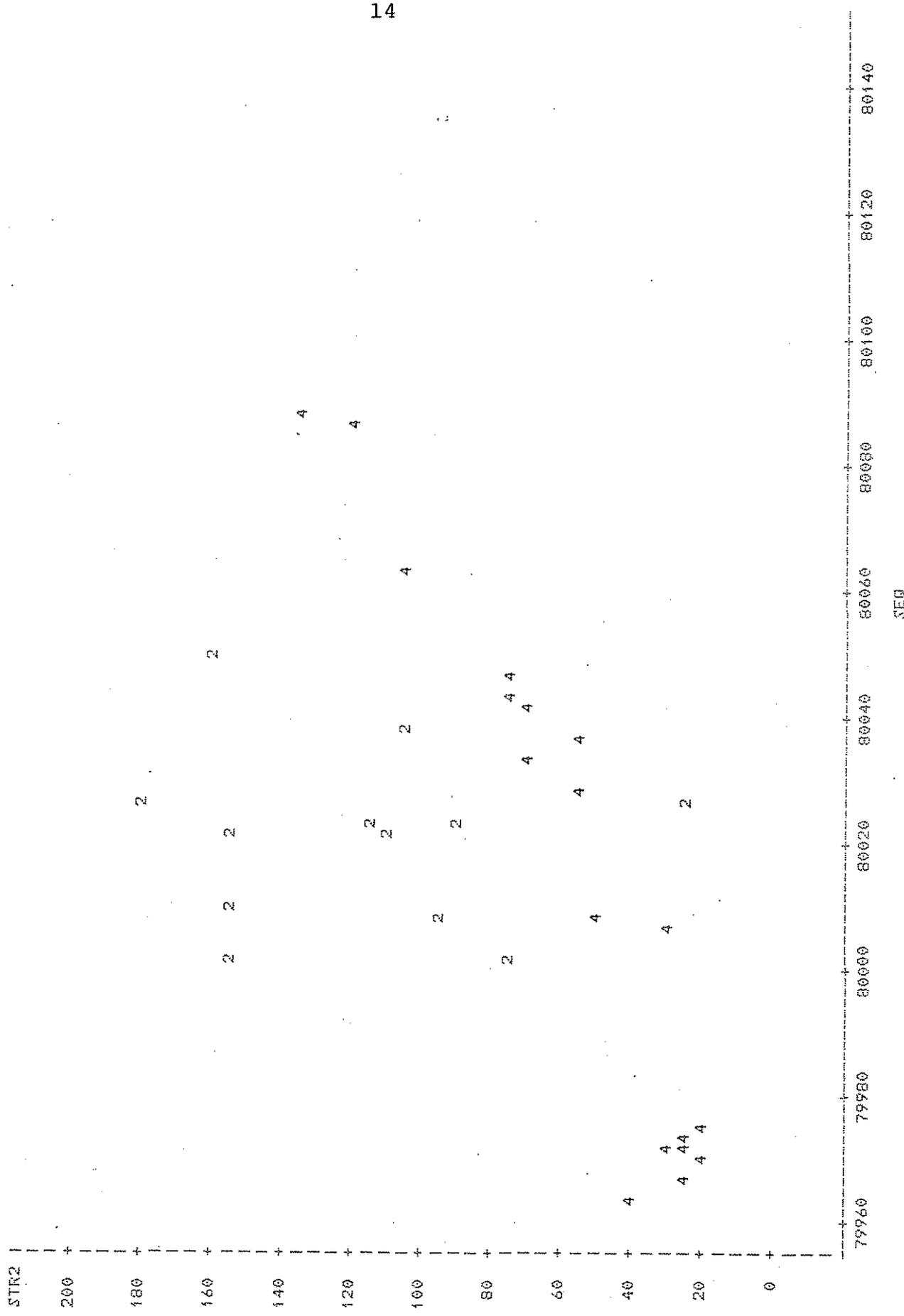


FIGURE 11  
 SPECIFIC GRAVITY VS SEQUENCE NUMBER  
 SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DUBURDE 7 = CLINTON 9 = LANSTING

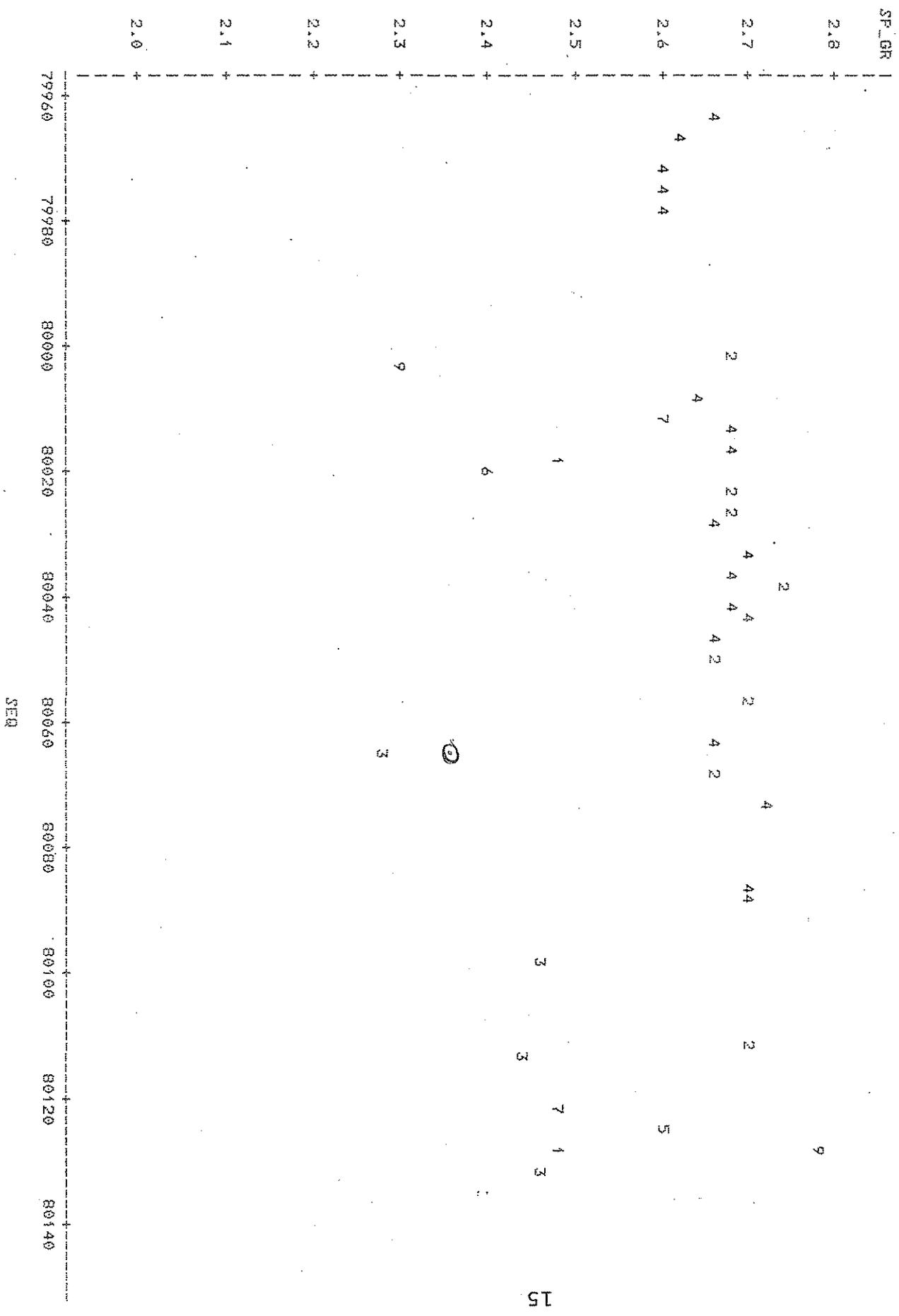
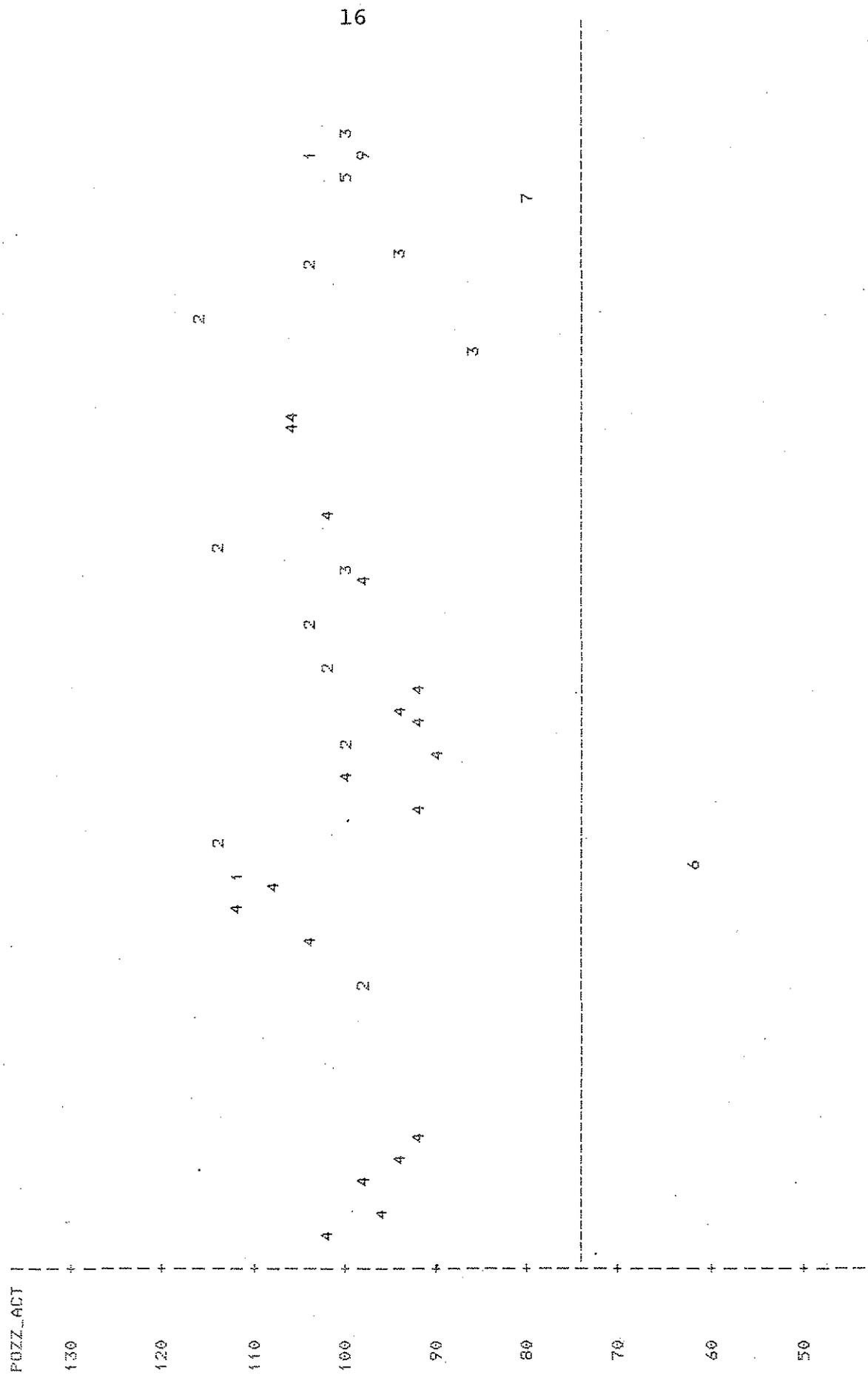


FIGURE 12  
POZZOLANIC ACTIVITY VS SEQUENCE NUMBER

SYMBOL IS PLANT NUMBER  
 1 = NO. OMAHA 2 = CO. BLUFFS 3 = NEAL #3 4 = NEAL #4  
 5 = NEB. CITY 6 = DUBUQUE 7 = CLINTON 8 = LANSING



79960 79980 80000 80020 80040 80060 80080 80100 80120 80140

### Loss on Ignition

Loss on ignition values are primarily an indicator of the carbon content of an ash, although minor amounts of other volatiles may be driven off during heating giving an exaggerated figure. The presence of excess carbon can affect the entrained-air content and subsequently the freeze-thaw durability of concrete made with fly ash. The ASTM limit was exceeded by one sample - that from the Interstate Power Co. plant at Dubuque.

Data from the other sources fall well below the specification limit and exhibited low variability with the exception of Council Bluffs. A partial explanation of its variability lies with periodic problems and shutdowns of the generating plant.

### Sulfur Trioxide

The ASTM C618 limit is low in view of various reports that higher than normal  $\text{SO}_3$  fly ashes have shown no detrimental effects when used in concrete.

All fly ashes tested so far fall below the 5% maximum  $\text{SO}_3$  limit and all exhibited low variability.

### Moisture Content

The ASTM C618 limit is 3.0% maximum moisture content. All the fly ashes tested fall below this limit and exhibited low variability.

### Total Oxides

Iowa specifications require a minimum total oxide

( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) percentage of 65% for Class F (non-cementing) ash and 50% for Class C (self-cementing) ash. Although both Council Bluffs and Neal No. 4 ash have self-cementing properties, other work shows Council Bluffs to be more reactive. Results of this study show that Neal 4 meets both Class F and Class C requirements for total oxides while Council Bluffs ash meets only the Class C requirements.

It was expected, based on the indicated coal source, that Lansing ash would be Class C. However, the total oxides did not bear this out. Subsequently, it was learned that when sampled, the plant was burning a blend of Montana and Wyoming coals. A second sample taken when only Wyoming coal was being burned, gave results that categorized the ash as Class C.

It appears that there is a degree of variability in the total oxide content within a source and that an abnormal deviation from the expected results can indicate changed conditions in coal source, plant operations, etc.

#### Autoclave Soundness

The autoclave soundness test is designed to provide a measure of potential delayed expansion. In portland cement this can be caused by the hydration of calcium or magnesium oxides after the calcium silicates have essentially completed their hydration. Since the Class C ashes have high amounts of calcium oxides, this test has been made a requirement of ASTM C618.

Results fall below the maximum limit of 0.8% and exhibit low variability within a source - except Council Bluffs.

### Percent Passing #325 Mesh

The percent passing the #325 mesh is a measure of ash quality in that the finer ashes react quicker and more completely.

The ashes tested gave values well above the minimum 66% passing the #325 mesh and no correlation was found with loss on ignition or specific gravity.

### Mortar Strength

Water-sand-fly ash mortar cubes were cast according to Iowa Test Method No. 212 (modified ASTM C109) with the fly ash totally replacing the portland cement. If the resultant strengths were consistent and predictable indicators of other more difficult to determine variables, then the strengths could be used as substitute tests.

Difficulty was experienced with the reactive ashes since they started to set up during the mixing procedure and could not be put into the cube molds properly. As a possible solution, the water-ash-sand mixture was only mixed until visibly blended and then put into the molds. This procedure left room for a significant amount of operator variability as shown by the results. Further work on this test method, possibly involving the addition of retarders, is suggested.

### Specific Gravity

Specific gravity of the various ashes ranged from 2.27 to 2.72, with the ash from a particular source exhibiting low variability. No significant correlation of specific gravity

with loss on ignition or percent passing #325 mesh was found.

#### Pozzolanic Activity Index

As a measure of the pozzolanic capabilities of the ashes, mortar cubes were cast according to ASTM C109 and 311 using the ash material to replace part of the normal portland cement content. The 28-day strength of these cubes must be at least 75% of the strength of the control specimens (made with all portland cement).

All ashes studied met the pozzolanic activity requirement, except the ash from Dubuque which also failed the loss on ignition requirement.

#### FURTHER STUDIES

The accumulation of physical-chemical data from the three plants will continue. When the quantity of data is sufficient, an investigation of the interrelationships between the various parameters will be made. It is hoped that parameters can be found that will be indicators or predictors of other, more difficult to test for, parameters or requirements.