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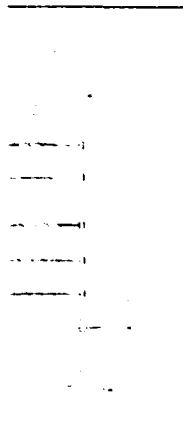
# POSTTEST ANALYSIS OF A LABORATORY-CAST MONOLITH OF SALT-SATURATED CONCRETE

by

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19 ABSTRACT (Continue on reverse if necessary and identify by block number)

A salt-saturated concrete was formulated for laboratory testing of cementitious mixtures with potential for use in disposal of radioactive wastes in a geologic repository in halite rock. Cores were taken from a laboratory-cast concrete monolith on completion of tests of permeability, strain and stress. The cores were analyzed for physical and chemical evidence of brine migration through the concrete, and other features with potential impact on installation of concrete plugs at the Waste Isolation Pilot Plant (WIPP) in New Mexico.

The posttest analyses of the cores provided evidence of brine movement along the interface between concrete and pipe, and little indication of permeability through the monolith itself. There may also have been diffusion of chloride into the monolith without actual brine flow.

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## Preface

This report was prepared for Sandia National Laboratories (SNL) under
Federal Agency Order 104-KF3, specified in SNL Document No. 32-8741.
Mr. C. W. Gulick coordinated the project for SNL, and Mr. John C. Stormont
was SNL Project Manager.

The work was accomplished in the Concrete Technology Division (CTD) of
the Structures Laboratory (SL), US Army Engineer Waterways Experiment Sta-
tion (WES), under the direction of Mr. J. M. Scanlon, Chief, CTD, and
Mr. B. Mather, Chief, SL. Dr. L. D. Wakeley and Mr. T. S. Poole prepared
this report, with the assistance of: Messrs. D. M. Walley, A. D. Buck,
B. R. Sullivan, and J. P. Burkes; Dr. C. E. Pace; and Ms. J. C. Ahlvin.
Dr. Wakeley was project leader.

COL Allen F. Grum, USA, was the previous Director of WES. COL Dwayne G.
Lee, CE, is the present Commander and Director. Dr. Robert W. Whalin is
Technical Director.

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## List of Abbreviations

| | |
|---|---|
| SNL | Sandia National Laboratories |
| SL | Structures Laboratory |
| CTD | Concrete Technology Division |
| WES | US Army Engineer Waterways Experiment Station |
| WIPP | Waste Isolation Pilot Plant |
| R&D | Research and Development |
| LOI | Loss on Ignition |
| XRD | X-ray Diffraction |

Conversion Factors, Non-SI to SI (Metric)
Units of Measurement

Non-SI units of measurements used in this report can be converted to SI
(metric) units as follows:

| Multiply | By | To Obtain |
|---|---|---|
| Fahrenheit degrees | 5/9 | Celsius degrees or Kelvins* |
| feet | 0.3048 | metres |
| inches | 25.4 | millimetres |
| pounds (force) per square inch | 6.894757 | kilopascals |
| pounds (mass) per cubic foot | 16.018463 | kilograms per cubic metre |
| pounds (mass) per gallon (US liquid) | 80.51963 | kilograms per cubic metre |
| fluid ounces | 29.574 | cubic centimetres |
| inches | 2.54 | centimetres |
| darcy | $9.87 \times 10^{-9}$ | centimetres squared |

---

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings,
use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K)
readings, use: $K = (5/9)(F - 32) + 273.15$.

## POSTTEST ANALYSIS OF A LABORATORY-CAST
## MONOLITH OF SALT-SATURATED CONCRETE

### Introduction

1. This report describes a study of a concrete monolith cast as a laboratory simulation of field tests of concrete for use in a repository for radioactive wastes in bedded salt. It includes compositional and chemical analyses of this concrete after termination of simulated in situ tests of stress and permeability, and consideration of the implications for field placement indicated by post-test observations.

### Background

2. A geologic repository for radioactive wastes requires a series of barriers designed to minimize release of radionuclides. The Waste Isolation Pilot Plant (WIPP) in southeast New Mexico is a research and development (R&D) facility the purpose of which is to demonstrate safe disposal in a geologic repository of low-level and transuranic wastes derived from United States defense activities. Sandia National Laboratories (SNL) conducts a major part of the current R&D program at the WIPP. This program involves the Concrete Technology Division (CTD) of the US Army Engineer Waterways Experiment Station (WES) for research on cement-based materials for plugging and sealing man-made openings in the rock.

3. In July 1985, several short concrete plugs were placed in vertical holes in salt beds at the WIPP repository horizon. These plugs provided opportunities to measure stresses, strains, and temperature at the interface between plug and rock and in rock immediately adjacent to the seal (Pace, in prep.), and to measure the permeability of concrete and interfaces. The concrete used for these plugs was developed and tested at WES, (Wakeley & Walley, in prep.) as was the arrangement of instrumentation for measuring stresses (Ainsworth, in prep.) Plans for these small-scale tests of seal performance are described in an SNL report (Stormont, in prep.).

4. During development of the concrete mixture and of instrumentation, procedures for concrete placement and data acquisition were tested in a laboratory simulation. Stress and strain gages were installed in a steel pipe 36 in. (914 mm) in diameter over a bed of crushed rock salt from the WIPP site. The pipe was then filled with a candidate concrete mixture, encasing the gages in concrete in a preplanned array. The gages were wired to a

computer-operated data acquisition system, and data were collected from April to late June, 1985. These data are reported elsewhere (Pace, in prep.). Components of the salt (NaCl)-saturated concrete mixture used in the laboratory simulation are given in Table 1. This was a developmental concrete mixture, with a different water reducing admixture and somewhat different properties than the concrete used underground at the WIPP in 1985 and 1986 (Wakeley and Walley, in prep.).

5. The bed of crushed salt beneath the plug, placed in the pipe below the instrumentation array, was intended as a porous medium to permit fluid flow upward from the bottom of the concrete monolith, when pressure was applied for permeability tests, and to mitigate end effects. It also provided an additional source of salt, to keep the permeating brine saturated with salt even at the elevated pressures of permeability tests. The fluid inlet was slightly below the salt-concrete interface at the bottom of the monolith (Figure 1).

6. During the late stages of permeability testing, dyes were added to two successive batches of test brine. A commercial fluorescein dye was used first, followed by brine containing potassium permanganate ($KMnO_4$). Tests involving dyes cover the period 13 Jun 85 to termination of permeability testing on 1 Jul 85 (Table 2).

## Purposes of this Report

7. At the completion of in situ tests, a scheme for sampling and analysis was designed to answer the following questions:

   a. Had the salt bed been disturbed by concrete placement?

   b. Did the salt compact or recrystallize to a solid mass during permeability testing, from pressure or fluid effects?

   c. Had the salt bed been dissolved during permeability testing?

   d. Did concrete placing techniques result in a uniform, well-compacted monolith?

   e. Was there physical, chemical, or compositional evidence of brine movement, either through the concrete or at the perimeter of the monolith (between concrete and steel pipe)?

## Removing the Pipe from the Monolith

8. The steel pipe that had provided restraint for simulated in situ tests was removed to permit assessment of the condition of the salt bed, and to reveal any evidence of dye along the perimeter. Yellowish brine, having either permeated the concrete or flowed upward along the perimeter during the final two days of testing, was 2.75 in. (70 mm) deep on the upper surface of the specimen. After this fluid was removed, the pipe was cut most of the way through with a torch cutter, down the length of the pipe on both sides and around the previously welded seam connecting the pipe to its base plate. Brine leaked out through the base cut and flow continued slowly thereafter, apparently draining the salt bed. This slow flow suggested that there was not extensive channeling through the salt, which probably would have caused faster drainage.

9. The specimen was then lowered to a horizontal position, with the side cuts defining a plane parallel to the floor through the diameter of the monolith, and the inlet pipe from permeability tests on the upper half near one cut (Figure 2). Cutting was completed around the base, and the base plate removed. This exposed the bottom surface of the crushed salt bed. The salt remained in place, exposed on a vertical surface (Figure 3) at the newly opened bottom end of the pipe, indicating that it had at least been compacted by the column of concrete plus brine (although this pressure was less than 4 psi (0.03 MPa)).

10. The upper half of the pipe, as defined by the side cuts, was then removed from the concrete monolith, by raising it with a fork lift. The ease of this procedure indicated minimal bonding between concrete and pipe, although there was resistance enough to show some bonding. Removing this portion of the pipe exposed half of the perimeter of the interface between concrete and crushed salt (Figure 4), as well as the region of salt bed around the permeability-test inlet.

## Observations of the Salt Bed and Surfaces of Monolith

11. The location of the permeability inlet was clearly marked in the salt bed by a large void, from which salt had been removed. The resultant

irregular gap between salt and concrete was more than 2 in. (50 mm) wide at its widest point, and extended several inches inward along the interface (Figure 5). Its width and extension decreased away from the fluid inlet, but it was readily visible along the entire exposed half of the circumference of the monolith.

12. The bottom surface of the concrete was pitted, showing casts of particles of crushed salt that had been in contact with the concrete when it was placed, and at least partly incorporated into the lower surface of the concrete (Figure 6). This rough surface was evidence of some salt dissolution, although much of the gap could be attributable to compaction. After this initial examination, a larger portion of the crushed salt was scraped away from the bottom surface of the concrete, exposing more of the interface. This revealed that the salt, although damp with remaining brine, was still granular; and that there was only about an inch of relief on the bottom surface of the monolith. This showed minimal disturbance of the salt bed at the time of concrete placement.

13. Brown staining, apparently from the $KMnO_4$ used in the final test of brine permeability, was visible along the perimeter of the specimen, but was not obvious on the exposed lower surface after removal of the salt. The cylindrical surface of the concrete showed no evidence of wash-out or other detrimental effects from flow. Other than the obvious staining, some of which may have been iron oxides from the pipe, the concrete monolith surface appeared fairly uniform, with no evidence of either aggregate segregation or locally stronger bonding to the pipe.

## Sampling the Monolith for Analyses

14. In addition to visual inspection, analyses by both chemical and x-ray diffraction techniques were planned, to search for evidence of brine movement through the sample. These analyses were performed on five cores taken from the monolith. Alteration of the monolith seemed most likely near the permeability inlet. Therefore, the distribution of coring was intended to sample the monolith at several locations relative to the permeability-test inlet, both vertically and circumferentially (Figure 7).

15. The first core was located 5 in. above the permeability-fluid inlet (5P). Two others were cut further up the specimen on the same side

(14P, 31P). The two remaining cores were on the opposite side of the exposed portion of the specimen, roughly 140 degrees from 5P (7A and 32A). Core designations give distance up from the base of the monolith, in inches, and a one-letter suffix: P for samples taken from the side of the permeability inlet, A for samples from the side away from the inlet. All cores were at least 7 in. (178 mm) long, cut approximately along the diameter of the mono-lith with a 4-in. (102-mm) coring bit. They were cut dry to avoid washing soluble salts out of the concrete. Cores were stored in double polyethylene bags at room temperature, to prevent moisture loss.

## Observations of the Five Cores

16. Visual observation of the 4-in. cores showed no evidence that either of the dyes -- fluorescein and $KMnO_4$ -- had penetrated the monolith. In fact, the local concentration of fluorescein indicated that this dye had collected in a reservoir immediately around the permeability inlet, in an open space left by local dissolution of the salt bed on which the monolith had been placed. Visual evidence of the $KMnO_4$ consisted of brown staining of the peripheral surface of the monolith, which had been in contact with the steel pipe and to which the concrete was not strongly bonded.

17. The cores also were briefly inspected for obvious evidence of aggregate segregation. The concrete had been placed by hand to avoid disturb-ing the salt bed, and to assure continuity around the gage array (Ainsworth, in prep.). No gaps in aggregate distribution were noted in this initial inspection.

18. The first subsamples were taken from the cores on 24 Jul 85. One to two millimetres of the outer layer of the perimeter surface was ground off to remove the apparent surface coatings of iron oxide or $KMnO_4$. A masonry bit was then used to drill five holes, 10 to 20 mm deep and 3/8 in. (9.5 mm) in diameter into each end of each core. These drillings were spaced approxi-mately at random over the end surfaces of each core. The powered material from each five- hole set was combined to provide two composite samples of 10-g each, from each of the five cores: one representing the outer edge; and one representing the interior of the monolith at a depth of about 7 in. (178 mm). Each of the ten composite samples was ground in a porcelain mortar to pass a 300-$\mu$m (No. 50) sieve. The samples were then stored in plastic

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vials (Nalgene) with tight-fitting lids. Replicate subsamples were obtained on 13 Sep 85 and 11 Nov 85.

19. Four analyses were performed: moisture loss at 105°C, loss on ignition at 900°C, chloride content, and manganese content. The purpose of these analyses was to define the pattern of movement of the brine from the point at which it entered the system to the reservoir on top of the monolith. Measurements of moisture loss were not originally planned, but observed differences in the texture of the samples taken from the edge of the monolith, when compared to those taken from interior locations, suggested that the free moisture contents might be different. Moisture loss at 105°C and ignition loss at 900°C are not valid techniques for the absolute determination of free water and water of hydration, respectively (Lea, 1971, p. 270.), but they are useful for making relative comparisons of these variables within a group of related samples.

20. Moisture loss was determined on the entire 10-g sample. Each sample was transferred to a 30-mL porcelain crucible and dried to a constant mass at 105°C. Some moisture was undoubtedly lost in the drilling and grinding process, but this was assumed to be constant among samples, and was not considered to be a source of bias in making comparative measurements. Loss on ignition was determined on a 1-g sample of the dried material. This sample was weighed into a 10-mL platinum crucible, covered, and ignited to a constant weight at 900°C. The result is reported as a percentage of the dried sample. Chloride content was determined according to "Sampling and Testing for Chloride Ion in Concrete," Federal Highway Administration Report No. FHWA-RD-77-85, except that a 0.5-g sample was used. Manganese levels were determined by atomic absorption spectroscopy. Samples were decomposed by fusion with lithium metaborate (0.5-g of sample to 1-g $LiBO_2$) at 1100°C, then dissolved in 20% HCl solution and diluted to 200 mL (to 500 mL in the second replicate). Manganese levels were reported in absorption units; no efforts was made to calculate actual contents.

21. Data from each of these four types of measurements were analyzed for spatial patterns using a nested designed analysis of variance. This procedure first compares mean levels among the 5 cores, then compares mean levels within each core, between the end of the core representing the edge of the monolith and the end representing the interior of the monolith. Data are

presented in Appendix A and details of the statistical analyses are described in Appendix B.

22. Statistical analysis indicated that there was a pattern in moisture contents among sampling locations. These are illustrated in Figure 8a. Samples taken from the edge of the monolith were consistently higher in moisture content than samples taken from the interior of the monolith. Differences in moisture levels among cores were not so distinct. The moisture level in core 5P (closet to the brine inlet) was the lowest among the 5 cores, followed by cores 32A, 7A, 31P, and 14P respectively. Moisture contents at the extreme levels, i.e. cores 5P and 14P, were statistically significant, but comparisons among other cores were not significant.

23. There were no differences in loss on ignition in comparisons of samples taken from the edge of the monolith with samples taken from the interior, but there were differences in mean loss on ignition among core locations. These are illustrated in Figure 8b. Loss on ignition was highest in core 5P, followed by cores 31P, 32A, 14P, and 7A, respectively. Ignition loss in core 5P was statistically higher than in cores 32A, 14P, and 7A. Ignition loss in core 31P, which was of intermediate value between these groups, could not be statistically distinguished from either of them.

24. Analysis of chloride contents showed a pattern similar to the moisture distribution pattern. This is illustrated in Figure 8c. Chloride levels were significantly higher in samples taken from the edge of the monolith than in samples taken from the interior of the monolith. Even though the chloride concentration on the interior of the monolith was lower than on the edge, it was higher than expected from the concentration of chloride ion in the original mixture ($t=5.28$, 4 degrees of freedom, $P<0.01$), indicating some penetration of brine. The chloride content of core 5P was significantly lower than levels in the other cores, among which there were no significant differences.

25. There was no significant pattern in manganese contents either between the edge and interior material or among cores. These data are illustrated in Figure 8d.

## Analyses Using X-ray Diffraction

26. A portion of each of the samples drilled from the ends of the five cores was frozen at 70-days age, to be maintained without further hydration

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pending analysis by X-ray diffraction (XRD). Sub-samples were scanned later in packed powder mounts at 2° 2-theta per minute, with nickel-filtered copper radiation. Patterns were studied to identify phases present, and to compare relative peak heights of the most intense peaks of phases considered to be important in determining the effects of hydration and of the NaCl brine. Although these peak heights do not indicate directly the amounts of each phase present, they can indicate that more or less of a phase is present when studied systematically through a series of related patterns with uniform scan parameters.

27. Comparisons among the XRD results from these 10 samples involved three hydration products: ettringite, chloroaluminate, and portlandite ($Ca(OH)_2$); and two residual phases: gypsum and halite. Patterns also were studied for presence or absence of tetracalcium trialuminate sulfate ($C_4A_3\bar{S}$). Chemical compositions and standard powder diffraction data for these phases are listed in Table 3.

28. Ettringite and chloroaluminate are present in approximately equal amounts in each sample. Calcium hydroxide and halite are minor constituents of cores taken from the upper portion of the monolith, and are minor to absent in samples from the lower cores. Gypsum range from minor to absent, and for all cores except 5P it appeared to be more abundant in interior samples than at the perimeter.

## Discussion

29. Because neither dye was visible in any of the cores, there was no direct evidence of brine movement through the monolith. The $KMnO_4$ was found to oxidize in contact with the iron pipe, so in retrospect its failure to appear in cores was predictable. Molecules of fluorescein may be too large to be carried freely into the concrete matrix. Brine movement along the interface was indicated by each individual core sample having more moisture and chloride in subsamples taken from the perimeter than in those from the interior (Figure 8). From this evidence, it appears that most of the measured fluid flow occurred along the interface between concrete and steel, possibly with secondary penetration into the concrete mass. Permeabilities measured in tests of the monolith were far more rapid (in the millidarcy rather than microdarcy range) than were those measured for smaller specimens of this and

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other similar concretes (Buck 1985). The calculations for permeability assume
uniform flow over the entire cross section, which probably was not the case.
In fact, flow apparently occurred at the relatively open interface, rather
than through the monolith. There was poor bonding between concrete and steel,
as indicated by ease of removing the pipe segment.

30. Phase distribution from XRD provides another line of evidence for
flow along the interface. The interior of the monolith was warmer than the
edge, at least for the first ten days of curing (Figure 9), and warmer tem-
peratures usually favor more rapid hydration. However, the hydration products
ettringite and chloroaluminate were more prevalent in samples from the edge,
and there was more unreacted gypsum in the interior, for any given core. More
hydration at the edge is compatible with a greater supply of water there,
especially since the temperature difference should have favored more rapid
hydration in the interior.

31. The surprisingly low chloride and moisture contents of 5P, which
had been closest to the permeability inlet, led to additional study of aggre-
gate distribution after the chemical and XRD studies were complete. When
observed systematically, the five cores revealed notable differences in aggre-
gate content, grading from most to least aggregate as:

> most aggregate -- 5P and 32A
> intermediate -- 7A
> least aggregate -- 31P and 14P.

Thus, the amount of aggregate in any given core is random relative to the
position of that core in the monolith, and indicates localized differences
resulting from hand placement of the concrete, rather than settling of aggre-
gate in the concrete after placement. Subsamples from the ends of the cores
had been taken without consideration of aggregate. This introduces another
variable to complicate interpretations of moisture and chloride contents, and
phase distribution, relative to position in the monolith and distance from the
permeability inlet; and makes discussion more speculative.

32. In general, the chloride levels in interior samples increase upward
through the monolith, although with only five cores taken this is not a
convincing gradation in chloride content. However, if there was significant
brine movement through the monolith, especially if it involved a wetting front
rather than steady-state flow, one would expect both moisture and chloride

contents to be high near the bottom, and at least remain constant if not diminish upward, away from the primary brine source.

33. An alternative explanation for the relatively high chloride content in interior samples taken from the upper cores is movement of brine, and thus of chloride, downward into the concrete from the brine pool on top of the monolith, the pool being fed by flow at the perimeter. Consistent with this possibility is the fact that the two uppermost cores had the greatest amounts of unbound NaCl, as determined by XRD.

34. Additional evidence for possible brine movement downward from the top of the monolith is that the two uppermost cores have the least difference in moisture content from interior and edge samples (Figure 8a). The highest interior moisture content is from core 32A, which was taken furthest from the permeability inlet. In fact, the lowest interior moisture content, and the greatest difference in moisture from edge to interior, is shown by core 5P, which was closest to the inlet. Aggregate content, then, does not account for all trends in the data, because 32A and 5P both had high aggregate contents.

35. Some of the data do appear to be attributable to aggregate distribution, however. Edge samples from cores 31P and 14P, which had the least aggregate, had the highest levels of both chloride and moisture. This is consistent with the assumption that brine moving along the interface would penetrate the outermost portion of the concrete preferentially where the cement paste fraction was more abundant. Core 5P has much lower levels of moisture and chloride, and the largest difference in moisture between its ends, with its large aggregate content; and that its location in the monolith precluded brine penetration downward from the pool on the upper surface.

36. There is some evidence of chloride diffusion independent of brine flow. All specimens, from both interior and edge, have more chloride than would be expected from the original concrete formulation. The only source of additional chloride is the brine. But the distribution of hydrated vs. remnants phases, moisture content, location of staining from dyes, and measured flow rates all indicate brine movement mostly along the concrete-pipe interface, and secondarily inward from the edges. One possible explanation for the increase chloride is diffusion of this ion occurring to some extent without actual brine flow.

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## Conclusions

a. There was little disturbance of the crushed salt bed at the time of concrete placement.

b. Although the salt was compacted by the pressure of the overlying concrete, and was saturated with brine during permeability tests, it did not form a solid mass during the 10 weeks between placement and post-test examination.

c. Crushed salt was dissolved away from the lower surface of concrete immediately around the permeability inlet, and also compacted as it became saturated, leaving a gap between salt and the base of the plug.

d. Aggregate was unevenly distributed in the monolith, but not in a way that suggested aggregate segregation. It was not concentrated in lower cores, nor in fact at any one level in the monolith. Its distribution suggested some unevenness from hand placement, in spite of vibration.

e. There was strong evidence of brine movement along the interface between concrete and pipe, and little indication of permeability through the monolith itself. There was secondary evidence of brine soaking into the concrete from the pool that formed on the upper surface. It appears that under driving pressure, the brine moved fairly rapidly along the path of least resistance, where the concrete was poorly bonded to its steel jacket. There may also have been diffusion of chloride into the monolith without actual brine flow.

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Pace, C. (in prep.). The Behavior of Expansive Cementitious Seals Sealing Hazardous Waste Underground.

Stormont, V. C. 1985. "In Situ Seal Tests at the Waste Isolation Pilot Plant," SAND85-1989, Sandia National Laboratories, Albuquerque, NM.

Table 1
Ingredients and Properties of Salt-Saturated Concrete
Used in Laboratory Studies and Properties of Fresh Concrete

| Ingredients | Mass per Cubic Foot of Concrete lb. (or fl. oz) |
|---|---|
| Class H cement | 13.5 |
| Chem Comp III® (expansive additive) | 9.0 |
| Cal Seal® (expansive additive) | 2.7 |
| Class C fly ash | 7.6 |
| Coarse salt | 3.7 |
| Defoamer | 0.16 |
| Water-reducing admixture | (8.32) |
| Coarse aggregate (19.0-mm (3/4-in)) | 51.7 |
| Sand | 51.0 |
| Water | 10.5 |

Property[a,b]

| | |
|---|---|
| Slump, in | 10.25 |
| Unit weight, lb/ft$^3$ | 131.3 |
| Air content, % | 13.5 |
| Mixture temperature, °F | 70 |

[a] Average of 3 batches.
[b] Determined according to standard procedure in the Handbook for Concrete and Cement, USA Engineers Waterways Experiment Station, Vicksburg, Mississippi, 1947 with quarterly supplements.

Table 2

Data from Laboratory Tests of Permeability

of Salt-Saturated Concrete

| Permeant | Date Start | End | Test Length | Pressure, psi Start | End | Δ | Permeability Millidarcy |
|---|---|---|---|---|---|---|---|
| NITROGEN GAS | 5/22 | 5/26 | 100 hr | 50 | 15 | 35 | 0.60 |
| " | 5/26 | 5/28 | 50 hrs | 50 | 19 | 31 | 1.35 |
| " | 5/29 | - | Same day | 100 | 65 | 34 | 21.00 |
| " | 5/29 | - | Same day | 50 | 13 | 37 | 0.40 |
| " | 5/31 | - | Same day | 50 | 21 | 29 | 0.80 |
| BRINE | 6/05 | 6/06 | 24 hrs | 50 | 40 | 10 | 404.00 |
| " | 6/07 | 6/13 | 6 days | 50 | 48 | 2 | 5.40 |
| " | 6/13 | 6/14 | 20.5 hrs | 100 | 66 | 4 | 13.40 |
| " | 6/26 | 6/27 | 28.5 hrs | 100 | 92 | 8 | ~3.00 |
| " | 6/27 | 7/01 | 90 hrs | 150 | 96 | 54 | ~2.00 |

Table 3

Phases Identified by X-Ray Diffraction, and Chemical Composition,

and Power Diffraction Data for Six Phases Studied

| Phase | Chemical Composition | JCPDS File No[a] | Location of Most Intense Peak | Relative Abundance in this Study |
|---|---|---|---|---|
| **HYDRATED:** | | | | |
| Ettringite | $6CaO \cdot Al_2O_3 \cdot 3SO_3 \cdot 32H_2O$ | 9-414 | 9.73 | 1 |
| Chloroaluminate (Friedel's salt) | $3CaO \cdot Al_2O_3 \cdot CaCl_2 \cdot 10H_2O$ | 19-202 | 7.89 | 1 |
| Portlandite | $Ca(OH)_2$ | 4-733 | 4.90 | 0.5 |
| **UNHYDRATED:** | | | | |
| Halite | $NaCl$ | 5-628 | 2.82 | 0.5 |
| Gypsum | $CaSO_4 \cdot 2H_2O$ | 6-46 | 7.56 | 0.3 |
| Tetracalcium Trialuminatesulfate ($C_4A_3\bar{S}$) | $Ca_4(Al_6O_{12})SO_4$ | 16-335 (discontinued) | 3.75 | Not Detected |

a) Joint Committee on Power Diffraction Standards, 1601 Park Lane, Swarthmore, PA, 19081.
b) Based on the arbitrary assignment of 1.0 to peak height for ettringite, and others as a fraction thereof.

21
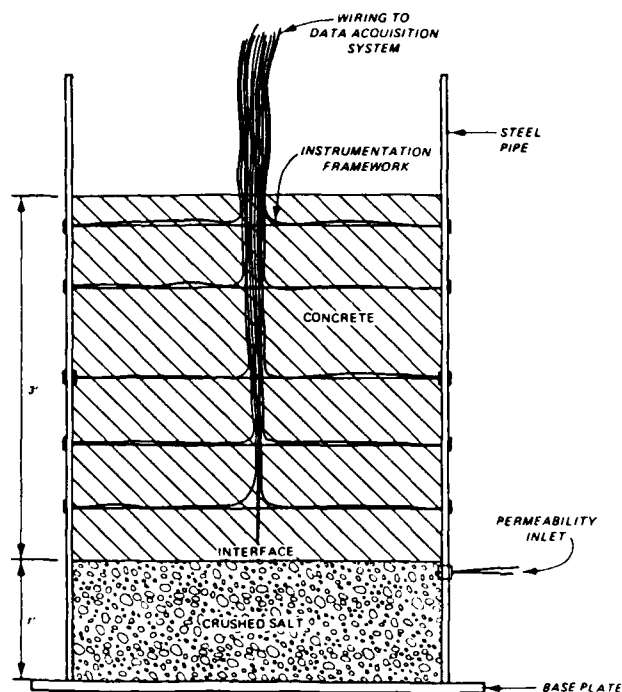
Figure 1. Concrete monolith in configuration for studies of stress, temperature, and permeability



Figure 2. Bottom of 36 in. diameter salt bed at base of concrete monolith, with permeability inlet shown by small tube at right center

Figure 3.   Exposed salt bed during removal of pipe.   Note compacted salt remaining attached to pipe
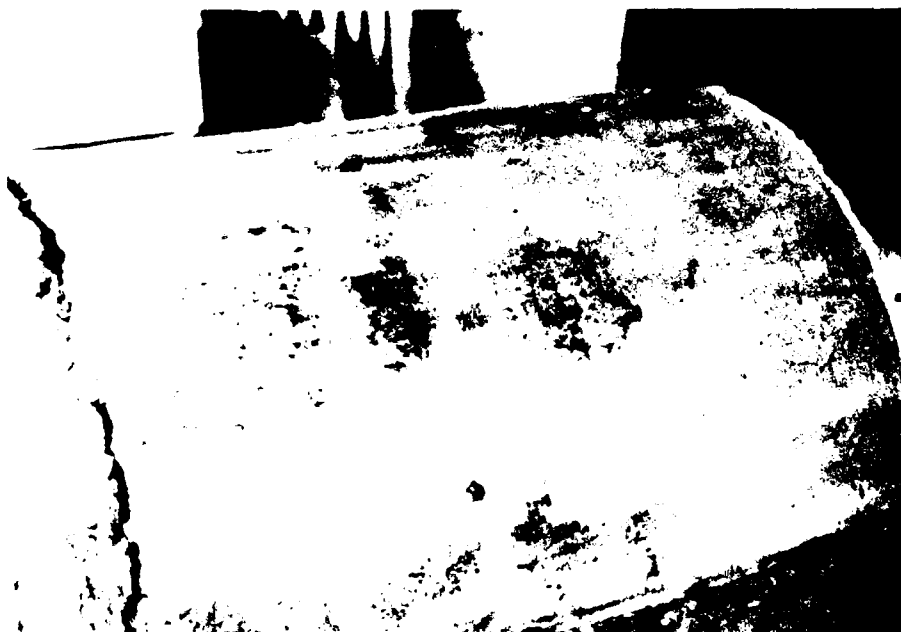


Figure 4.   Exposed surface of concrete after removal of half of the pipe, showing one gage attachment (lower center) and staining at interface
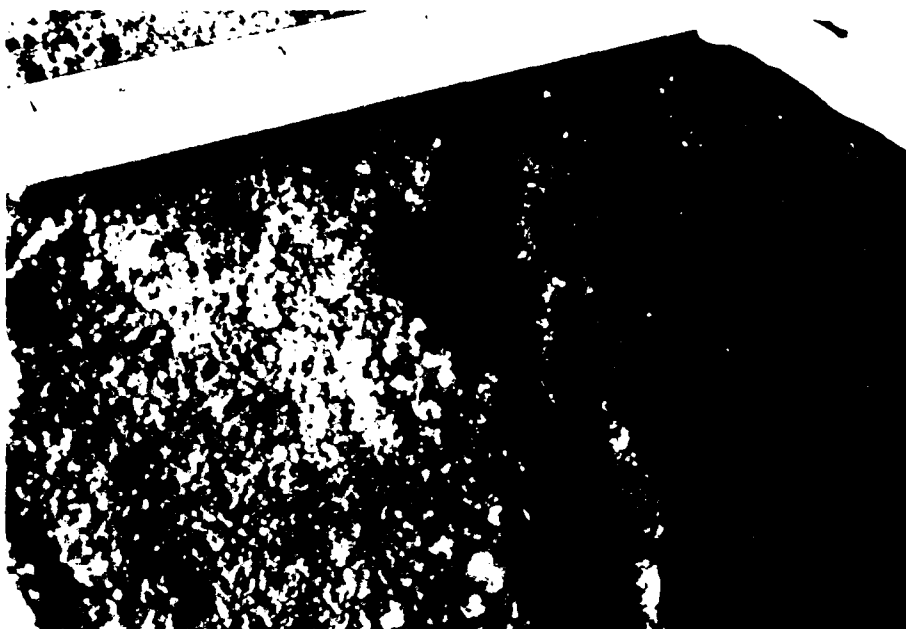
(a)

(b)

Figure 5.  Gap along plane of contact between concrete and salt bed, (a) near permeability inlet, (b) on opposite side from inlet

Figure 6. Pitted lower surface of concrete, showing casts of salt crystals where salt bed removed
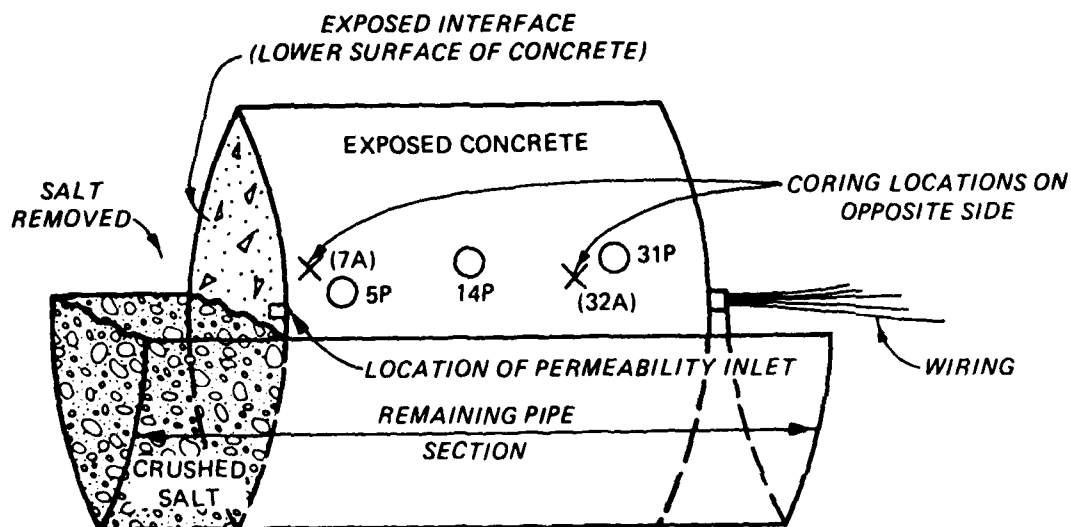


Figure 7. Concrete monolith after removal of base plate, half of pipe, and half of salt bed, showing locations of five cores taken for posttest analyses

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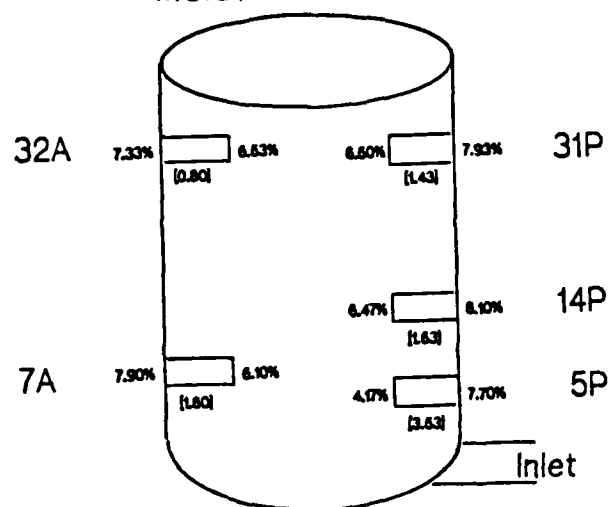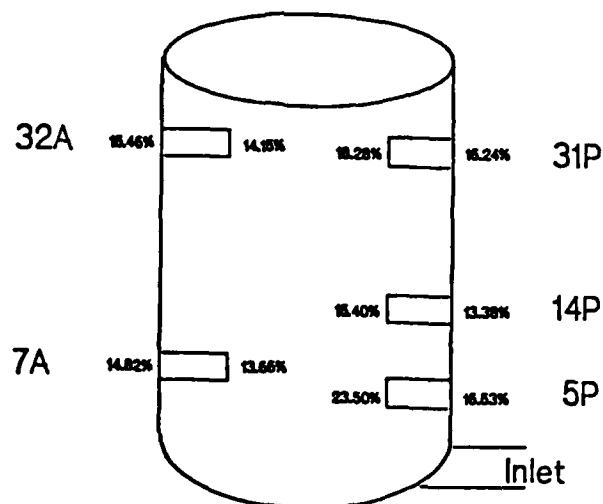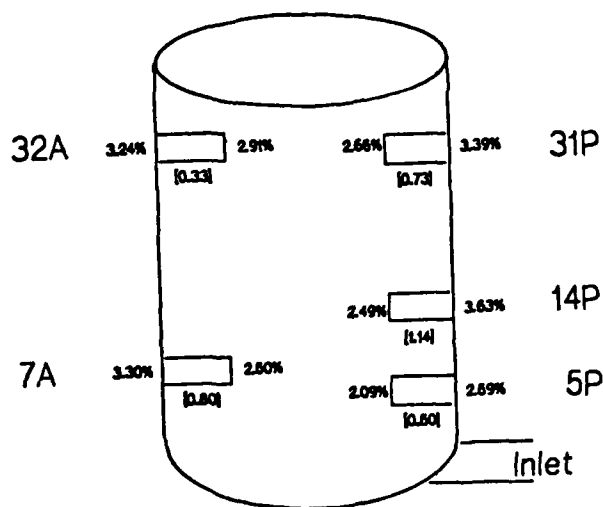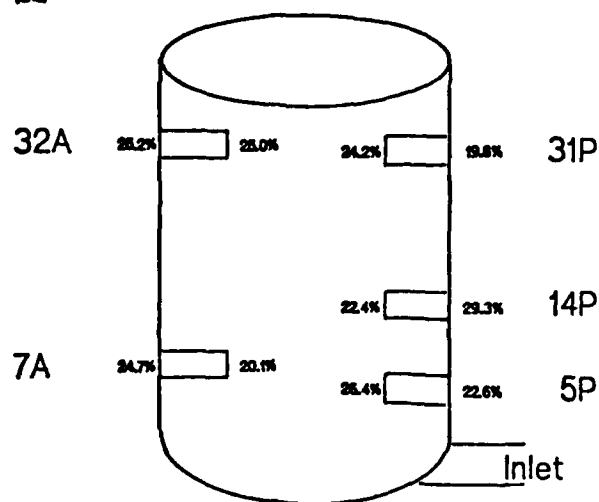
Figure 8. Core locations showing distributions of: (a) moisture loss at 105°C, (b) chloride content, (c) manganese, and (d) loss on ignition (LOI); and differences between interior and edge samples of moisture loss and chloride [in brackets]

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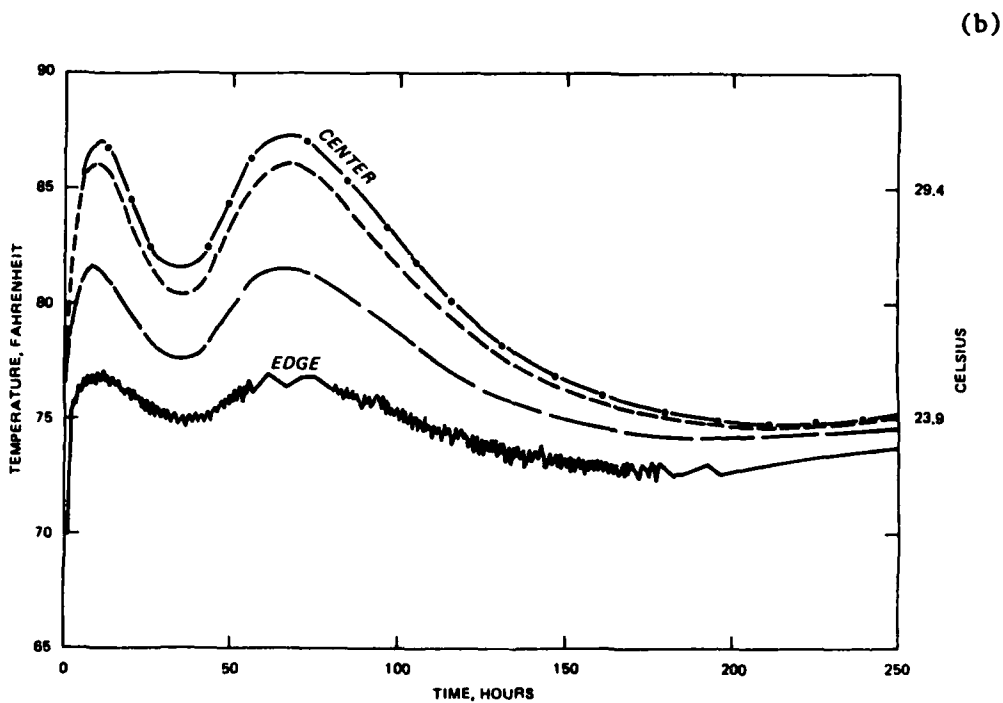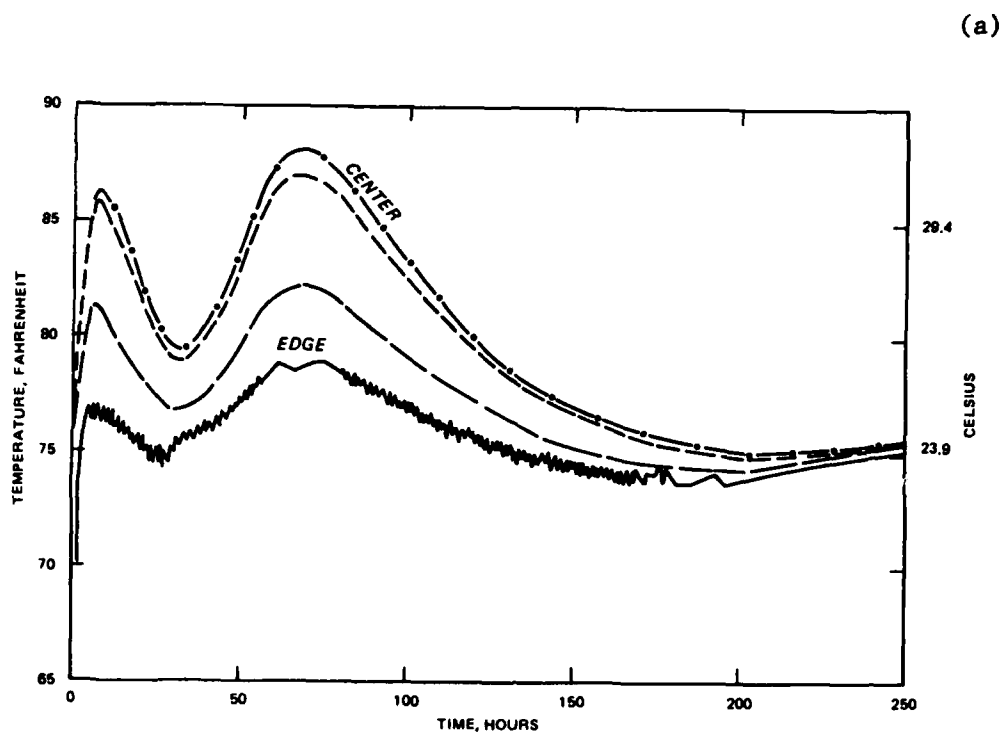
Figure 9.   Temperature profiles from top(a) and bottom(b) gage levels, at
            four positions from center to edge of the monolith

27
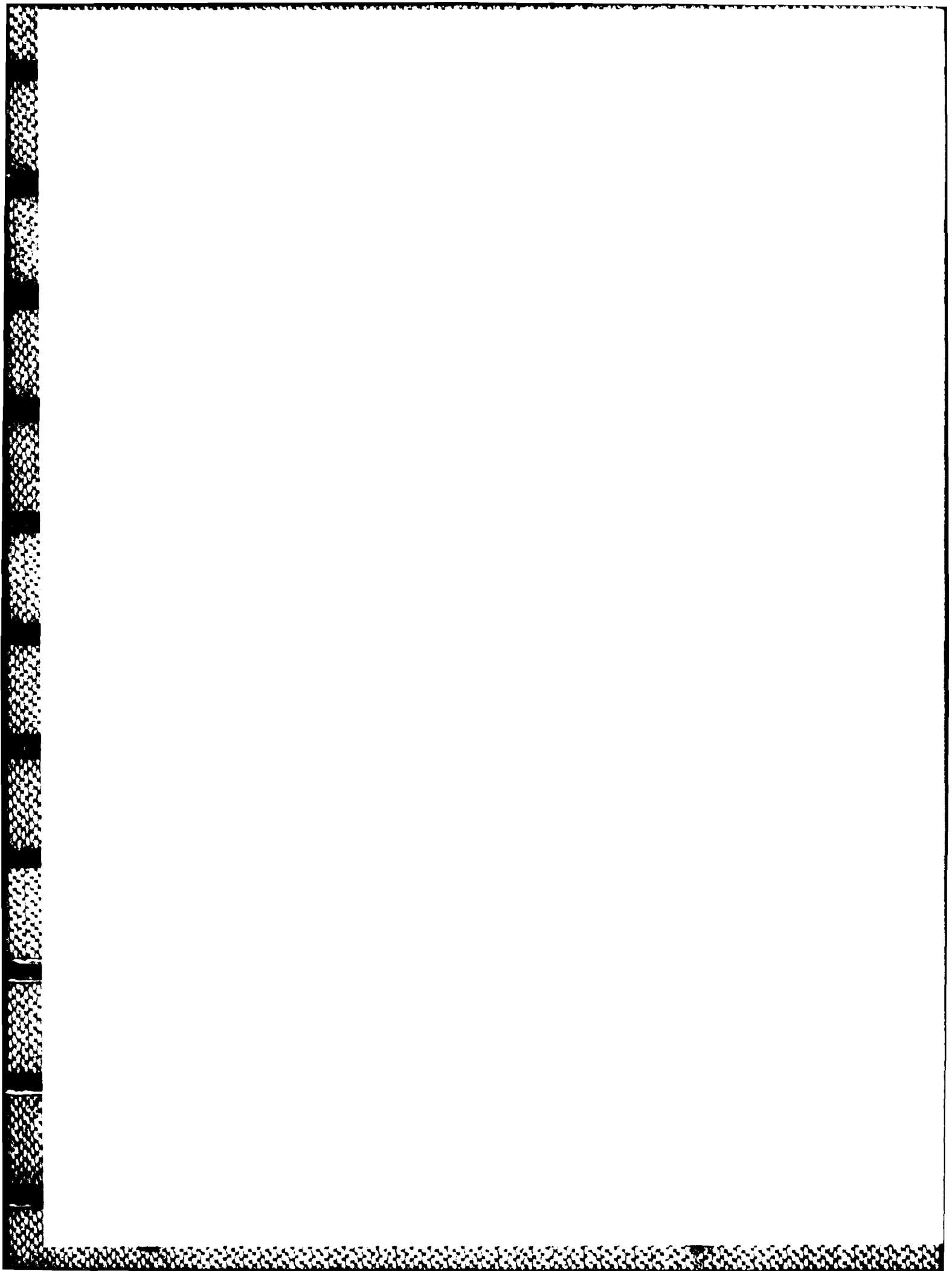
## APPENDIX A: DATA FROM ANALYSIS OF MOISTURE, LOSS ON IGNITION, CHLORIDE, AND MANGANESE LEVELS ON CORES OF THE MONOLITH

| Core | Location | Repl. | Moisture (%) | Loss on Ignition (%) | Cl (%) | Mn* |
|------|----------|-------|--------------|----------------------|--------|------|
| 5P | CENTER | 1 | 4.7 | 24.0 | 2.05 | 35.8 |
| | | 2 | 4.1 | 23.0 | 2.12 | 17.0 |
| | | 3 | 3.7 | | | |
| | EDGE | 1 | 8.8 | 19.4 | 2.82 | 30.2 |
| | | 2 | 6.8 | 13.7 | 2.36 | 15.0 |
| | | 3 | 7.5 | | | |
| 14P | CENTER | 1 | 6.6 | 13.8 | 2.43 | 29.8 |
| | | 2 | 6.1 | 17.0 | 2.57 | 15.0 |
| | | 3 | 6.7 | | | |
| | EDGE | 1 | 10.0 | 14.0 | 3.43 | 45.6 |
| | | 2 | 9.0 | 12.8 | 3.82 | 13.0 |
| | | 3 | 5.3 | | | |
| 31P | CENTER | 1 | 6.6 | 19.7 | 2.50 | 29.3 |
| | | 2 | 6.9 | 16.9 | 2.81 | 19.0 |
| | | 3 | 6.0 | | | |
| | EDGE | 1 | 8.1 | 16.0 | 3.45 | 26.5 |
| | | 2 | 7.5 | 16.5 | 3.32 | 13.0 |
| | | 3 | 8.2 | | | |
| 7A | CENTER | 1 | 6.5 | 14.5 | 2.48 | 27.2 |
| | | 2 | 6.0 | 12.8 | 2.52 | 13.0 |
| | | 3 | 5.8 | | | |
| | EDGE | 1 | 9.6 | 12.0 | 3.69 | 30.3 |
| | | 2 | 8.2 | 17.7 | 2.92 | 19.0 |
| | | 3 | 5.9 | | | |
| 32A | CENTER | 1 | 5.8 | 13.9 | 2.90 | 34.9 |
| | | 2 | 7.6 | 14.4 | 2.92 | 15.0 |
| | | 3 | 6.2 | | | |
| | EDGE | 1 | 7.6 | 16.5 | 3.24 | 31.3 |
| | | 2 | 7.8 | 14.4 | 3.24 | 19.0 |
| | | 3 | 6.6 | | | |

* Units of absorbance, as measured by atomic absorption spectroscopy.

APPENDIX B:   ANALYSIS OF VARIANCE ON MOISTURE CONTENT, LOSS ON IGNITION,
CHLORIDE CONTENT AND MANGANESE CONTENT

## 1.  General Notes on Statistical Analyses

Moisture contents, loss on ignitions, chloride contents, and manganese
contents of the cores were each analyzed in a nested designed analysis of
variance model.  Core location was treated as the main effect, with location
within each core (edge, interior) treated as the nested effect.  Replicates
were treated as blocking variables rather than as pure replicates because they
were separated by considerable time and, in one instance (manganese), by a
change in methods.  Time is a critical factor in analysis of hardened con-
cretes because of continuing hydration.  Moisture content and loss on ignition
would be sensitive to this factor.  Treatment of repeated measures as true
replicates would result in an inflated experimental error because of these
time dependent changes.  Because there is no true replication, interaction
effects could not be examined.

The analysis of variance procedure indicates whether there are, in gen-
eral, significant differences in the main effects (cores) or in the nested
effects (locations within each core), but it does not indicate specifically
which cores or locations within cores are responsible for the significant
results.  Individual means are compared by Duncan's Multiple Range Test
(Steele and Torrie, 1960).  The result of this test is a grouping of means
into homogeneous subsets.  Some means may be included in more than one subset.

Statistical analyses were performed using the Statistical Analysis Sys-
tem (ASA) software (SAS Institute, Box 8000, Cary, NC 27511).  The format of
the following tables is largely that of the SAS software output.

## 2. Analysis of Moisture Distribution

| CLASS | LEVELS | VALUES |
|-------|--------|--------|
| CORE  | 5      | 14P 31P 32A 5P 7A |
| LOC   | 2      | CENTER EDGE |
| REP   | 3      | 1 2 3 |

NUMBER OF OBSERVATIONS IN DATA SET = 30

Model:  MOISTURE = REPLICATE CORE LOC(CORE)

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE |
|--------|----|----------------|-------------|
| MODEL | 11 | 46.69400000 | 4.24490909 |
| ERROR | 18 | 17.66466667 | 0.98137037 |
| CORRECTED TOTAL | 29 | 64.35866667 | |

| MODEL F = | 4.33 | | PR > F = 0.0030 |
|-----------|------|--|-----------------|

| R-SQUARE | C.V. | ROOT MSE | MOIS MEAN |
|----------|------|----------|-----------|
| 0.725528 | 14.4128 | 0.99064139 | 6.87333333 |

| SOURCE | DF | ANOVA SS | F VALUE | PR > F |
|--------|----|----------|---------|--------|
| REP | 2 | 7.92866667 | 4.04 | 0.0355 |
| CORE | 4 | 7.13533333 | 1.82 | 0.1694 |
| LOC(CORE) | 5 | 31.63000000 | 6.45 | 0.0013 |

A.  DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:  MOISTURE CONTENT
NOTE:  THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
       NOT THE EXPERIMENTWISE ERROR RATE


ALPHA=0.05  DF=18  MSE=0.98137


| NUMBER OF MEANS | 2 | 3 | 4 | 5 |
|---|---|---|---|---|
| CRITICAL RANGE | 1.19979 | 1.25955 | 1.30084 | 1.32443 |


MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.


| CORE | 14P | 31P | 7A | 32A | 5P |
|---|---|---|---|---|---|
| N | 6 | 6 | 6 | 6 | 6 |
| MEAN | 7.28 | 7.22 | 7.00 | 6.93 | 5.93 |

B.  DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:  MOISTURE CONTENT
NOTE:  THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
       NOTE THE EXPERIMENTWISE ERROR RATE


ALPHA=0.05    DF=18  MSE=0.98137
NUMBER OF MEANS        2
CRITICAL RANGE       0.758812


MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.


| LOCATION | EDGE | CENTER |
|---|---|---|
| N | 15 | 15 |
| MEAN | 7.79 | 5.95 |

## 3. Analysis of Loss on Ignition (LOI) Distribution

| CLASS | LEVELS | VALUES |
|-------|--------|--------|
| CORE | 5 | 14P 31P 32A 5P 7A |
| LOC | 2 | CENTER EDGE |
| REP | 3 | 1 2 3 |

NUMBER OF OBSERVATIONS IN DATA SET = 30

NOTE: ALL DEPENDENT VARIABLES ARE CONSISTENT WITH RESPECT TO THE
PRESENCE OR ABSENCE OF MISSING VALUES. HOWEVER,
ONLY     20 OBSERVATIONS CAN BE USED IN THIS ANALYSIS.

Model: LOI = REPLICATE CORE LOCATION(CORE)

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE |
|--------|-----|----------------|-------------|
| MODEL | 10 | 159.89335000 | 15.98933500 |
| ERROR | 9 | 45.11490500 | 5.01276722 |
| CORRECTED TOTAL | 19 | 205.00825500 | |

| MODEL F = | 3.19 | | PR > F = 0.0477 |
|-----------|------|---|-----------------|

| R-SQUARE | C.V. | ROOT MSE | LOI MEAN |
|----------|------|----------|----------|
| 0.779936 | 13.8732 | 2.23892100 | 16.13850000 |

| SOURCE | DF | ANOVA SS | F VALUE | PR > F |
|--------|-----|----------|---------|--------|
| REP | 1 | 0.98124500 | 0.20 | 0.6686 |
| CORE | 4 | 99.01588000 | 4.94 | 0.0220 |
| LOC(CORE) | 5 | 59.89622500 | 2.39 | 0.1211 |

A.  DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:  LOI
NOTE:  THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
       NOT THE EXPERIMENTWISE ERROR RATE


ALPHA=0.05  DF=9   MSE=5.01277


| NUMBER OF MEANS | 2 | 3 | 4 | 5 |
|---|---|---|---|---|
| CRITICAL RANGE | 3.57556 | 3.73459 | 3.83298 | 3.88227 |


MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.


| CORE | 5P | 31P | 32A | 14P | 7A |
|---|---|---|---|---|---|
| N | 4 | 4 | 4 | 4 | 4 |
| MEAN | 20.02 | 17.26 | 14.80 | 14.39 | 14.24 |

B.  DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:  LOI
NOTE:  THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
       NOTE THE EXPERIMENTWISE ERROR RATE


ALPHA=0.05    DF=9   MSE=5.01277


| NUMBER OF MEANS | 2 |
|---|---|
| CRITICAL RANGE | 2.26138 |


MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.


| LOCATION | EDGE | CENTER |
|---|---|---|
| N | 10 | 10 |
| MEAN | 16.99 | 15.28 |

## 4. Analysis of Chloride Distribution

| CLASS | LEVELS | VALUES |
|-------|--------|--------|
| CORE | 5 | 14P 31P 32A 5P 7A |
| LOC | 2 | CENTER EDGE |
| REP | 3 | 1 2 3 |

NUMBER OF OBSERVATIONS IN DATA SET = 30

NOTE:   ALL DEPENDENT VARIABLES ARE CONSISTENT WITH RESPECT TO THE
       PRESENCE OR ABSENCE OF MISSING VALUES.  HOWEVER,
       ONLY    20 OBSERVATIONS CAN BE USED IN THIS ANALYSIS.

Model:  CHLORIDE = REPLICATE CORE LOCATION(CORE)

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE |
|--------|-----|----------------|-------------|
| MODEL | 10 | 4.35791235 | 0.43579123 |
| ERROR | 9 | 0.54310364 | 0.06034485 |
| CORRECTED TOTAL | 19 | 4.90101599 | |

MODEL F =        7.22                      PR > F = 0.0033

| R-SQUARE | C.V. | ROOT MSE | CL-IG MEAN |
|----------|------|----------|------------|
| 0.889185 | 8.5315 | 0.24565189 | 2.87936501 |

| SOURCE | DF | ANOVA SS | F VALUE | PR > F |
|--------|-----|----------|---------|--------|
| REP | 1 | 0.00800176 | 0.13 | 0.7242 |
| CORE | 4 | 1.54404439 | 6.40 | 0.0101 |
| LOC(CORE) | 5 | 2.80586620 | 9.30 | 0.0023 |

A.  DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:  CHLORIDE

NOTE:  THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
       NOT THE EXPERIMENTWISE ERROR RATE

ALPHA=0.05  DF=9  MSE=.0603448

| NUMBER OF MEANS | 2 | 3 | 4 | 5 |
|---|---|---|---|---|
| CRITICAL RANGE | 0.392306 | 0.409755 | 0.42055 | 0.425958 |

MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.

| CORE | 32A | 14P | 31P | 7A | 5P |
|---|---|---|---|---|---|
| N | 4 | 4 | 4 | 4 | 4 |
| MEAN | 3.08 | 3.06 | 3.02 | 2.90 | 2.34 |

B.  DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:  CHLORIDE

NOTE:  THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
       NOTE THE EXPERIMENTWISE ERROR RATE

ALPHA=0.05    DF=9   MSE=.0603448

| NUMBER OF MEANS | 2 |
|---|---|
| CRITICAL RANGE | 0.248116 |

MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.

| LOCATION | EDGE | CENTER |
|---|---|---|
| N | 10 | 10 |
| MEAN | 3.23 | 2.53 |

## 5. Analysis of Manganese Distribution

| CLASS | LEVELS | VALUES |
|-------|--------|--------|
| CORE | 5 | 14P 31P 32A 5P 7A |
| LOC | 2 | CENTER EDGE |
| REP | 3 | 1 2 3 |

NUMBER OF OBSERVATIONS IN DATA SET = 30

NOTE: ALL DEPENDENT VARIABLES ARE CONSISTENT WITH RESPECT TO THE
PRESENCE OR ABSENCE OF MISSING VALUES. HOWEVER,
ONLY    20 OBSERVATIONS CAN BE USED IN THIS ANALYSIS.

Model: MANGANESE = REPLICATE CORE LOCATION(CORE)

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE |
|--------|----|----------------|-------------|
| MODEL | 10 | 1475.38500000 | 147.53850000 |
| ERROR | 9 | 188.80450000 | 20.97827778 |
| CORRECTED TOTAL | 19 | 1664.18950000 | |

| MODEL F = | 7.03 | PR > F = 0.0036 |
|-----------|------|-----------------|

| R-SQUARE | C.V. | ROOT MSE | MN MEAN |
|----------|------|----------|---------|
| 0.886549 | 19.1280 | 4.58020499 | 23.94500000 |

| SOURCE | DF | ANOVA SS | F VALUE | PR > F |
|--------|----|----------|---------|--------|
| REP | 1 | 1326.82050000 | 63.25 | 0.0001 |
| CORE | 4 | 46.41200000 | 0.55 | 0.7022 |
| LOC(CORE) | 5 | 102.15250000 | 0.97 | 0.4824 |

A.   DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:   MANGANESE

NOTE:   THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
        NOT THE EXPERIMENTWISE ERROR RATE

ALPHA=0.05   DF=9    MSE=20.9783

| NUMBER OF MEANS | 2 | 3 | 4 | 5 |
|---|---|---|---|---|
| CRITICAL RANGE | 7.31459 | 7.63992 | 7.8412 | 7.94204 |

MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.

| CORE | 14P | 32A | 5P | 7A | 31P |
|---|---|---|---|---|---|
| N | 4 | 4 | 4 | 4 | 4 |
| MEAN | 25.85 | 25.04 | 24.50 | 22.38 | 21.95 |

B.   DUNCAN'S MULTIPLE RANGE TEST FOR VARIABLE:   MANGANESE

NOTE:   THIS TEST CONTROLS THE TYPE I COMPARISONWISE ERROR RATE,
        NOT THE EXPERIMENTWISE ERROR RATE

ALPHA=0.05    DF=9   MSE=20.9783

| NUMBER OF MEANS | 2 |
|---|---|
| CRITICAL RANGE | 4.62615 |

MEANS WITH THE SAME UNDERLINE ARE NOT SIGNIFICANTLY DIFFERENT.

| LOCATION | EDGE | CENTER |
|---|---|---|
| N | 10 | 10 |
| MEAN | 24.29 | 23.60 |

# END

# 11-86

# DTIC