

A DIFFERENT PERSPECTIVE FOR INVESTIGATION OF PCC PAVEMENT DETERIORATION

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Interim Report
for
Iowa DOT
Research Project HR-2074

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**Iowa Department
of Transportation**

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ABSTRACT

Portland Cement Concrete (PCC) pavement has served the State of Iowa well for many years. The oldest Iowa pavement was placed in LeMars in 1904. Beginning in 1931, many miles of PCC pavement were built to "get out of the mud." Many of these early pavements provided good performance without deterioration for more than 50 years.

In the late 1950's, Iowa was faced with severe PCC pavement deterioration referred to as D cracking. Research identified the cause of this deterioration as crushed limestone containing a bad pore system. Selective quarrying and ledge control has alleviated this problem.

In 1990, cracking deterioration was identified on a three year old pavement on US 20 in central Iowa. The coarse aggregate was a crushed limestone with an excellent history of performance in PCC pavement. Examination of cores showed very few cracks through the coarse aggregate particles. The cracks were predominately confined to the matrix. The deterioration was identified as alkali-silica reactivity (ASR) by a consultant (1).

To investigate the cause of the deterioration, the Iowa DOT and Iowa State University jointly purchased a high-resolution, low-vacuum Hitachi Scanning Electron Microscope (SEM) with an energy

dispersion detector. Subsequent evaluation identified no concentration of silica gel (sodium-Na, potassium-K or silicon-Si), but did identify substantial amounts of sulfur-S, aluminum-Al and calcium-Ca (assumed to be ettringite) in the air voids. Some of these voids have cracks radiating from them leading us to conclude that the ettringite filled voids were a center of pressure causing the crack. The ettringite in the voids, after being subjected to sodium chloride (NaCl), initially swells and then dissolves.

This low vacuum SEM research of PCC pavement deterioration supports the following conclusions:

1. A low vacuum SEM and an energy dispersion detector are very important for proper evaluation of PCC pavement deterioration.
2. There are instances today where PCC pavement deterioration is mistakenly identified as ASR.
3. Ettringite initially expands when subjected to NaCl. The ettringite filled voids are a center-of-pressure that cracks the PCC.
4. The deterioration of some current premature PCC pavement distress locations is caused by factors related to the formation of excessive ettringite.

INTRODUCTION

The first PCC pavement placed in Iowa was at LeMars in 1904. PCC pavement has served the State of Iowa well for many years. Beginning in 1931, many miles of pavement were built on primary roads across Iowa "to get out of the mud." Many of these early pavements provided good performance without deterioration for more than 50 years. Based on these successes, Iowa over the years has constructed substantial miles of PCC pavement that have been the backbone of our highway system. Each time deterioration mechanisms have threatened the success of Iowa PCC pavement, researchers have identified the problem and appropriate changes have been made to ensure longevity and performance.

In the late 1950's, Iowa was faced with severe PCC pavement deterioration. Some pavements that were only 15 years old were exhibiting a substantial crack pattern. This deterioration was referred to as D cracking (Figure 1). After substantial research, the cause of this deterioration was related to a certain type of carbonate coarse aggregate. The initiating cause was identified as crushed limestone aggregate containing a predominance and substantial volume of pores in the 0.04 to 0.2 μm diameter pore size. Since the 1930's, Iowa has maintained an excellent record of the ledges and quarry locations of crushed stone used in Iowa PCC pavement and their performance by time to visible deterioration. The D cracking problem was solved by

identifying the ledge or ledges in each quarry that would cause D cracking and prohibiting their use in Iowa Primary pavement. The Iowa limestone producers use selective quarrying and bench their operation to remove the undesirable ledges. The D cracking susceptible crushed stone is used in different applications.

A NEW TYPE OF PCC PAVEMENT DETERIORATION

In 1990, the Iowa DOT was presented with a very challenging problem. A ten mile section of new four-lane divided highway in central Iowa (US 20 from Fort Dodge to Webster City) was exhibiting distress cracking (Figure 2) at an age of only three years. In other premature PCC pavement failures, the primary factor relating to the deterioration was the coarse aggregate. In this case, the coarse aggregate was a crushed limestone (Gilmore City Ledge) from the Fort Dodge Mine that had an excellent performance record when used in PCC pavement. An examination of cores from the pavement and particularly one adjacent to the joint (Figure 3) revealed very few cracks passing through the coarse aggregate particles. The cracks were predominately confined to the matrix.

During construction of this pavement the contractor had made a number of changes in materials sources. This provided five different PCC material combinations. These included two cement sources, three fly ash sources and two fine aggregate sources

with the Fort Dodge coarse aggregate being used for the entire project. Four combinations containing fly ash exhibited varying degrees of distress while one section without fly ash exhibited no distress cracking.

There were a number of Iowa DOT personnel with substantial experience in evaluating PCC pavement performance and in identifying distress mechanisms that participated in the investigation (beginning in 1991) of the new type of pavement deterioration. No consensus as to the cause of deterioration was reached. In 1992, a noted concrete consultant was retained to identify the cause of the US 20 deterioration. After an investigation, the consultant concluded in September 1992 that the cause of deterioration was ASR (1).

EVALUATION OF PCC PAVEMENT DETERIORATION USING NEW TECHNOLOGY

Wendell Dubberke of the Iowa DOT and Scott Schlorholtz of Iowa State University (ISU) conducted an evaluation of the US 20 concrete using an ISU high-vacuum scanning electron microscope (SEM). Unfortunately, the high-vacuum would cause cracks in the specimen and there was concern that the removal of water was distorting the findings.

March 1, 1993 the Iowa DOT and ISU entered into a contract to conduct research project HR-358, "Evaluation of Microcracking and

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Chemical Deterioration in Concrete Pavement." The principal investigator of HR-358 is Scott Schlorholtz. Under HR-358 the Iowa DOT and ISU jointly purchased a low-vacuum, high-resolution Hitachi SEM with a germanium energy dispersion detector. It took substantial time for ordering and delivery of the SEM. Delivery was late and there have since been many issues requiring time to be resolved. The project finally began to progress about July 1994.

The current evaluation procedure is to obtain backscattered electron (BSE) SEM, photomicrographs at 30X and 100X. Energy dispersion mapping of sodium, potassium, silicon, calcium, sulfur and aluminum is obtained on the same area at the same time for both the 30X and 100X micrographs. The germanium detector is top-of-the line and does an excellent job of identifying the elements present on the sawed and polished surfaces.

WHITISH MATERIAL IN THE PCC AIR VOIDS

Iowa US 20 Webster County Project

Examination of a sawed and polished face of PCC from the US 20 pavement (with a light microscope) identified a whitish material in many of the air voids. This whitish material may have been mistakenly identified as silica gel by the consultant, leading to the conclusion that the US 20 deterioration was ASR.

This same sawed and polished piece of PCC was then observed using the Hitachi low-vacuum SEM. A BSE photomicrograph of nice round air voids was obtained (Figure 4). This identified smaller voids nearly filled and larger voids partially filled with the whitish material.

The alkali in silica gel has been determined to be either sodium or potassium or a combination of sodium and potassium. This alkali combines with silicon to produce silica gel (2).

Elemental maps were obtained on the US 20 polished sample for sodium, potassium and silicon. In the elemental mapping a dark area would indicate very little of an element was present while a very white area would identify a concentration of the element.

The particular interest in this case was "What elements are present in the whitish material filling the air voids?" An energy dispersion spectrum of the whitish material in the air voids was obtained (Figure 5). This spectrum showed major peaks for oxygen, aluminum, sulfur and calcium, supporting the conclusion that the whitish material in the voids is ettringite. There is a minor silicon peak, but no sodium or potassium peak, thereby, supporting the finding that the material in the void is not silica gel.

The SEM also identified cracks radiating from some of these air voids (Figure 6). This would support a conclusion that this air

void was a center-of-pressure in the development of these cracks. There was another interesting feature of the ettringite in these voids with the radiating cracks. The ettringite was filling these voids, but the ettringite did not extend outside of the round voids into the radiating cracks. This led to the conclusion that the void was filled with ettringite before the cracks developed.

The elemental maps for sodium and potassium (Figure 7) showed a general distribution of the elements and the round air voids could not be identified. The elemental map for silicon was a different story. The round air voids could easily be identified because they were black, showing that there was almost no silicon in the whitish material in the air voids and substantially more silicon in the PCC surrounding the air voids. This led to the conclusion that the whitish material in the air voids is not silica gel.

Elemental maps were obtained for 11 different elements. The map that best outlined the round air voids was sulfur-S. In the map for sulfur, the air voids were very white showing a high concentration of sulfur in the whitish material in the air voids. The aluminum map also showed whiter than the surrounding PCC. The round air voids could also be identified in the calcium map. There was substantial calcium present in both the air voids and

the surrounding matrix, but the whitish material in the air voids had less calcium than the surrounding matrix. These maps led to the conclusion that the whitish material in the air voids was very likely ettringite.

Nebraska I-80 Aurora Project

Core samples were obtained from two Nebraska locations where premature deterioration had been identified. The deterioration at both of these locations (I-80 at Aurora (3) and the Omaha Airport) had also been identified by others as ASR. The aggregates at both of these locations contained sodium-potassium feldspars that others identified as moderately reactive in regard to ASR. Samples from both of these sites contained the whitish material in the air voids.

An investigation of the I-80 Aurora PCC (Figures 8 & 9) show a high concentration of sulfur and very little, if any, silicon again leading to the conclusion that the material is ettringite and not silica gel. An interesting note to make on the I-80 Aurora SEM data is that some of the air voids are in close proximity to these so called ASR reactive aggregates. If these reactive aggregate particles are producing silica gel, it would be a reasonable assumption that some of this expansive gel would be forced into the voids in close proximity. No concentration of silicon has been identified in these air voids.

Nebraska Omaha Airport

The data from the Omaha Airport is very interesting for a couple of other reasons. First, for those who may question the capability of the energy dispersion detector to properly identify elements, a sodium-potassium feldspar aggregate will demonstrate the accuracy of the data. The photomicrograph using the backscattered detector (Figure 10) shows darker and lighter portions of the large aggregate in the left side of the picture. This is due to differences in atomic weight. In the elemental maps (Figure 11), the dark portions are shown to be sodium feldspar while the light portions are shown to be potassium feldspar.

There was also a rim deposit around an aggregate particle (Figure 10). Many petrographic researchers would probably identify this rim as silica gel. The elemental maps of Figure 11 show that this rim does not contain silicon, so it is not silica gel. The rim material contains substantial sulfur and is, therefore, probably ettringite.

The material in the Omaha Airport voids contains substantial sulfur, but almost no silicon, so it again is not silica gel, but is very likely ettringite.

GROWTH OF ETTRINGITE IN THE LABORATORY

Additional research was conducted in the Iowa DOT Materials Laboratory to investigate the formation of ettringite in air voids. Concrete beams were made and left in the molds for approximately 24 hours. After removal from the molds, they were stored in a 100% humidity moist room at approximately 21°C (70°F). They are still being stored in the moist room. Slices are periodically taken from the end of the beam. These slices were examined using the low-vacuum SEM. At an age of two months, Mix #1 showed ettringite nodules either growing in the air voids or being extruded into the air voids from the capillary pore system. At an age of seven months, Mixes #2 & 3 exhibited ettringite nodules in the air voids. These ettringite nodules in the air voids developed in an environment where it would be impossible to have evaporation or an external source of sulfur.

INITIAL EXPANSION OF ETTRINGITE WHEN SUBJECTED TO NaCl BRINE

Canadian research at the University of Manitoba had shown that ettringite would dissolve in the presence of chloride ions (4). In an effort to substantiate this in regard to the ettringite in the voids, a polished PCC surface with a few large, tightly filled voids was selected. A photomicrograph and elemental maps were recorded prior to treatment with NaCl brine (Figure 12). The elemental maps (Figure 13) exhibited no silicon, but substantial sulfur and aluminum indicating that the material in

the air voids was very likely ettringite. The NaCl treated, polished sample was put into the low vacuum SEM. The ettringite in the large voids had either been dissolved or dislodged (Figure 14). Another very interesting finding was that the ettringite in the small voids had swelled and expanded. After 48 hours, the ettringite had dissolved from all of the air voids, but the expansion of the ettringite had been sufficient to lift the boundaries of the air voids (Figure 15). Another significant feature that became visible was the substantial cracking in the large air voids and the cracking through a series of air voids.

The SEM data had shown relatively conclusively that when subjected to NaCl, ettringite initially expands and then dissolves. Duggan cores (5) 25 mm (1 in.) in diameter and 51 mm (2 in.) long were drilled from the laboratory prepared PCC that had been stored in the moist room. This PCC had exhibited ettringite either growing in or being extruded into the air voids as described earlier in this paper. These four cores had exhibited a relatively constant rate of expansion through 50 days during a distilled water soak (Figure 16). At 50 days after pretreatment, the four cores were changed to a NaCl brine soak. From 50 to 60 days all four cores exhibited an increased rate of expansion. After 60 days there was very little expansion indicating that the expansive mechanism had been destroyed (or dissolved). There is speculation that the expansive pressure was

due to ettringite through 50 days. When subjected to NaCl at 50 days, there was increased expansion of the ettringite. The ettringite then dissolved and there was very little additional expansion.

FUTURE RESEARCH

The data contained in this paper was presented to representatives of the Portland Cement Association (PCA) on January 5, 1995. They were very interested and believed the data demonstrated that ettringite in the air voids may well be the distress mechanism in some PCC pavement deterioration. Field reviews and laboratory investigations have identified excessive consolidation during PCC placement, the presence of deicing chemical and water freeze-thaw damage as contributing factors. The PCA has agreed to cosponsor research on this subject at Iowa State University. The co-principal investigators will be Dr. Scott M. Schlorholtz and Dr. James K. Cable of Iowa State University. At this time, the intent is to include the analysis of core samples from two PCC pavement sites exhibiting substantial deterioration, two exhibiting moderate deterioration and two demonstrating good performance. These samples will be examined to determine the chemical and/or physical differences. The research will be aimed at determining what factors enhance or curtail the growth of ettringite in the air voids. Freedom to modify or redirect the direction of the research will be retained to address areas of

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interest by others who participate in the research. The final proposal has yet to be agreed upon. Other researchers have identified ettringite filled or partially filled voids in deteriorated PCC pavement (6).

The Iowa DOT would welcome other states to join in this very important research. This research is essential in improving the performance and longevity of PCC pavement. If any other state DOTs are interested in participating, please contact Vernon J. Marks of the Iowa Department of Transportation (phone 515-239-1447).

CONCLUSIONS

This low-vacuum SEM research of PCC pavement deterioration supports the following conclusions.

1. A low-vacuum SEM and an energy dispersion detector are very important for proper evaluation of PCC pavement deterioration.
2. There are instances today where PCC pavement deterioration is mistakenly identified as ASR.
3. Ettringite initially expands when subjected to NaCl. The ettringite filled voids are a center-of-pressure that crack the PCC.
4. The deterioration of some current premature PCC pavement distress locations is caused by factors related to the formation of excessive ettringite.

ACKNOWLEDGEMENTS

The authors wish to express their appreciation to a number of people who have made substantial contribution to this paper. Much of the SEM investigation has been conducted by Scott Schlorholtz and Jerry Amenson of ISU. In addition to the authors, the Iowa DOT PCC pavement deterioration team included Bernard C. Brown, Jerry Bergren, John Lane, Jim Grove, Jim Myers and Chen Ouyang. Kathy Davis, Bob Steffes and Shane Tymkowicz made substantial contribution in the preparation of the paper. Gordon Smith of the Iowa Concrete Paving Association has been very supportive of this research.

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FIGURE CAPTIONS

1. Typical D Cracking of PCC Pavement
2. The Cracking Pattern of US 20 in Central Iowa
3. A US 20 Core Showing Few Cracks in the Coarse Aggregate
4. A 200X BSE Photomicrograph of the PCC air voids on Iowa US 20
5. An Energy Dispersion Spectrum of the Whitish Material in the Air Voids
6. A 100X BSE Photomicrograph of Iowa US 20 Showing Cracks Radiating from the Ettringite Filled Air Voids
7. The Elemental Maps for the Figure 6 Photomicrograph
8. A 100X BSE Photomicrograph of a Nebraska I-80 Aurora PCC Sample
9. The Elemental Maps for the Figure 8 Photomicrograph
10. A 100X BSE Photomicrograph of a Nebraska Omaha Airport PCC Sample
11. The Elemental Maps for the Figure 10 Photomicrograph
12. A 100X BSE Photomicrograph of Air Voids Tightly Filled with Ettringite
13. The Elemental Maps for the Figure 12 Photomicrograph
14. A 100X BSE Photomicrograph of the Figure 12 Air Voids 45 Minutes After Treatment with NaCl Brine.
15. A 100X BSE Photomicrograph of the Figure 12 Air Voids 48 Hours After Treatment with NaCl Brine.
16. Duggan Test Results with Sodium Chloride Brine Modification

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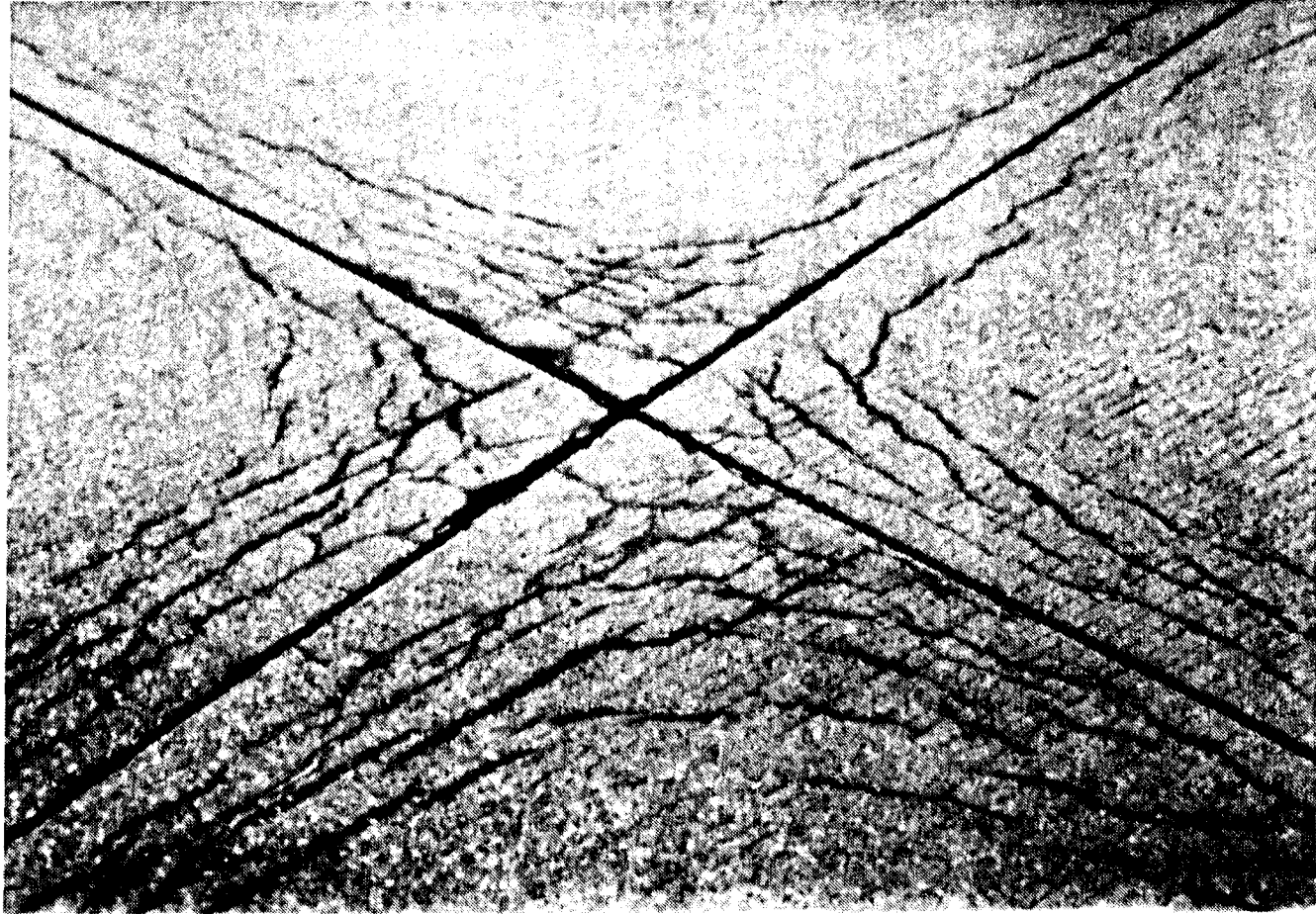


FIGURE 1 TYPICAL D CRACKING OF PCC PAVEMENT

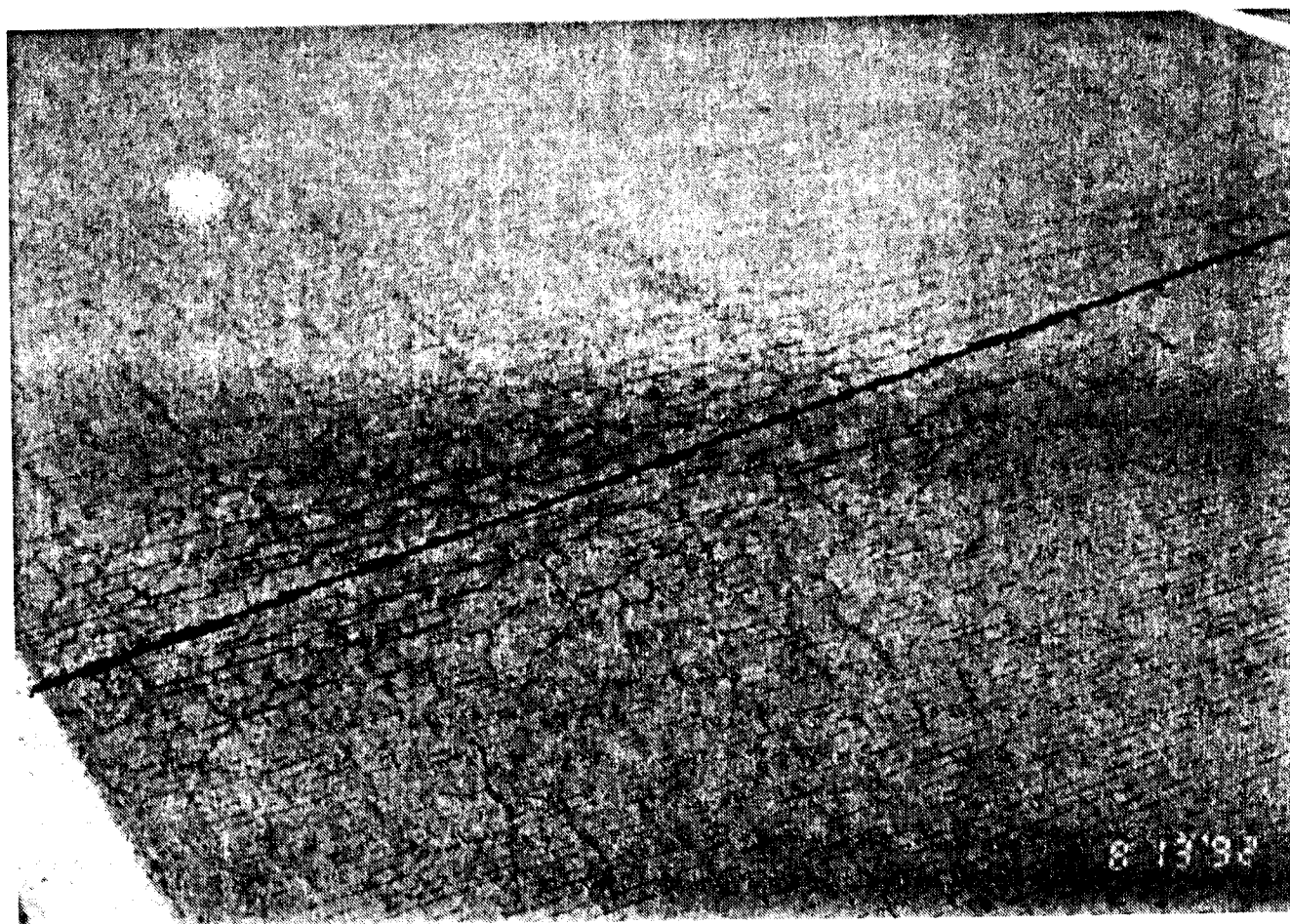


FIGURE 2 THE CRACKING PATTERN OF US 20 IN CENTRAL IOWA



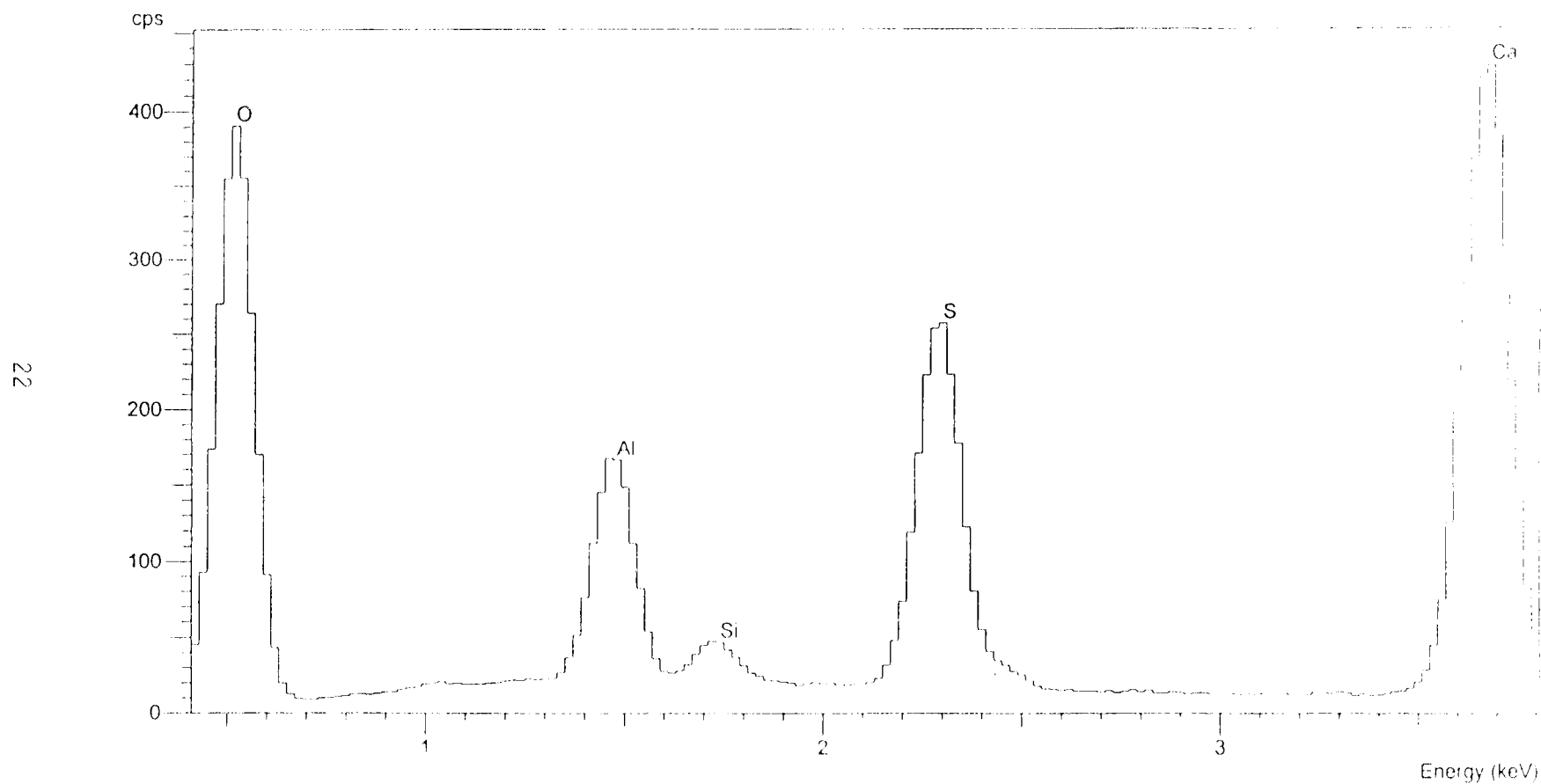
**FIGURE 3 A US 20 CORE SHOWING FEW CRACKS
IN THE COARSE AGGREGATE**

Operator: Scott S
Client: Iowa Dept. of Trans.
Job: HR-358 Task 1
Res: High
Label: 3T-2, US 520, EB lane (2 Nov 94 11:49:55)



**FIGURE 4 A 200X BSE PHOTOMICROGRAPH
OF THE PCC AIR VOIDS ON IOWA US 20**

**FIGURE 5 AN ENERGY DISPERSION SPECTRUM
OF THE WHITISH MATERIAL IN THE AIR
VOIDS**

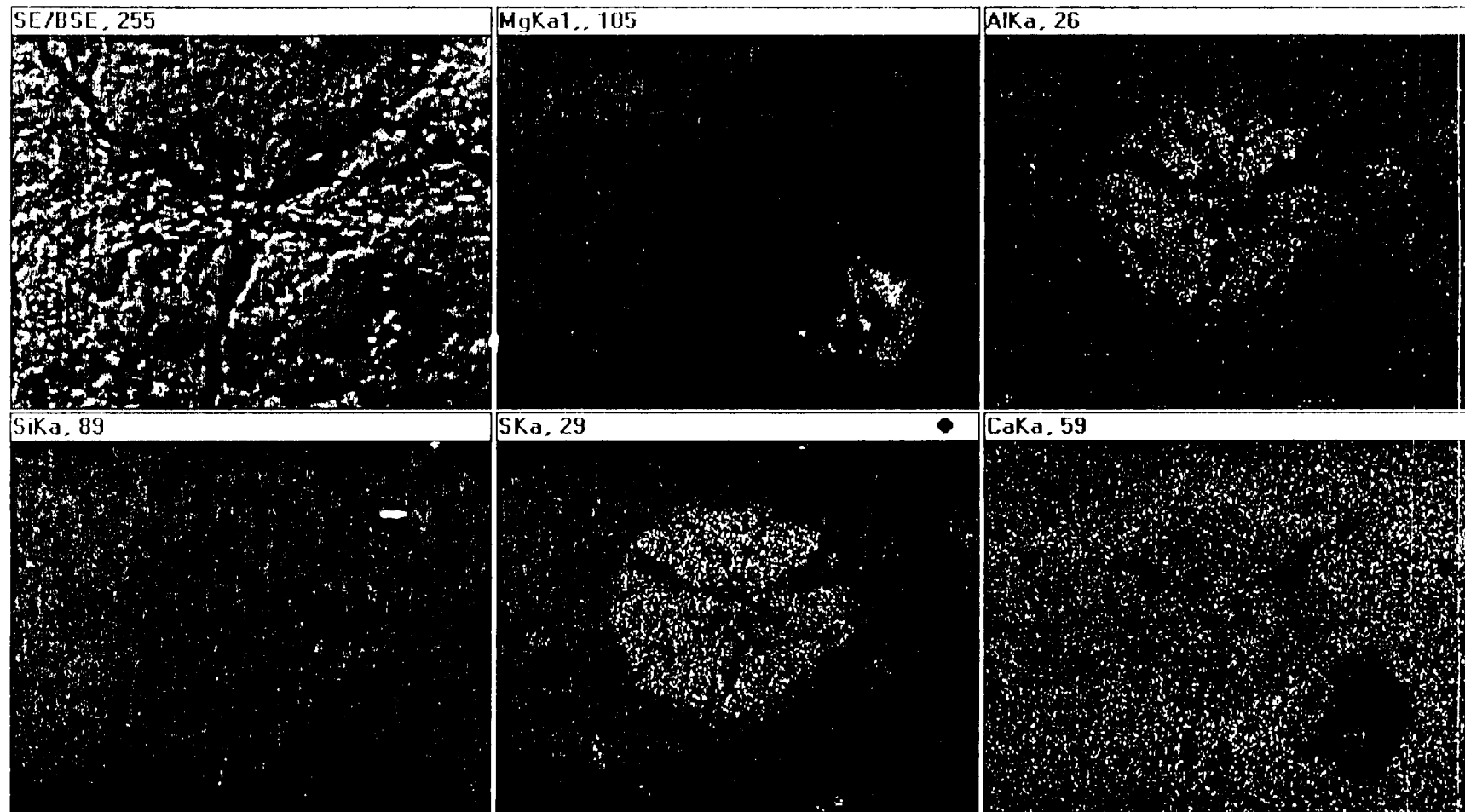


Operator: Scott S
Client: Iowa Dept. of Trans.
Job: HR-358 Task 1
Res: High
Label: 3T-2, US 520, EB lane (2 Nov 94 12:17:29)



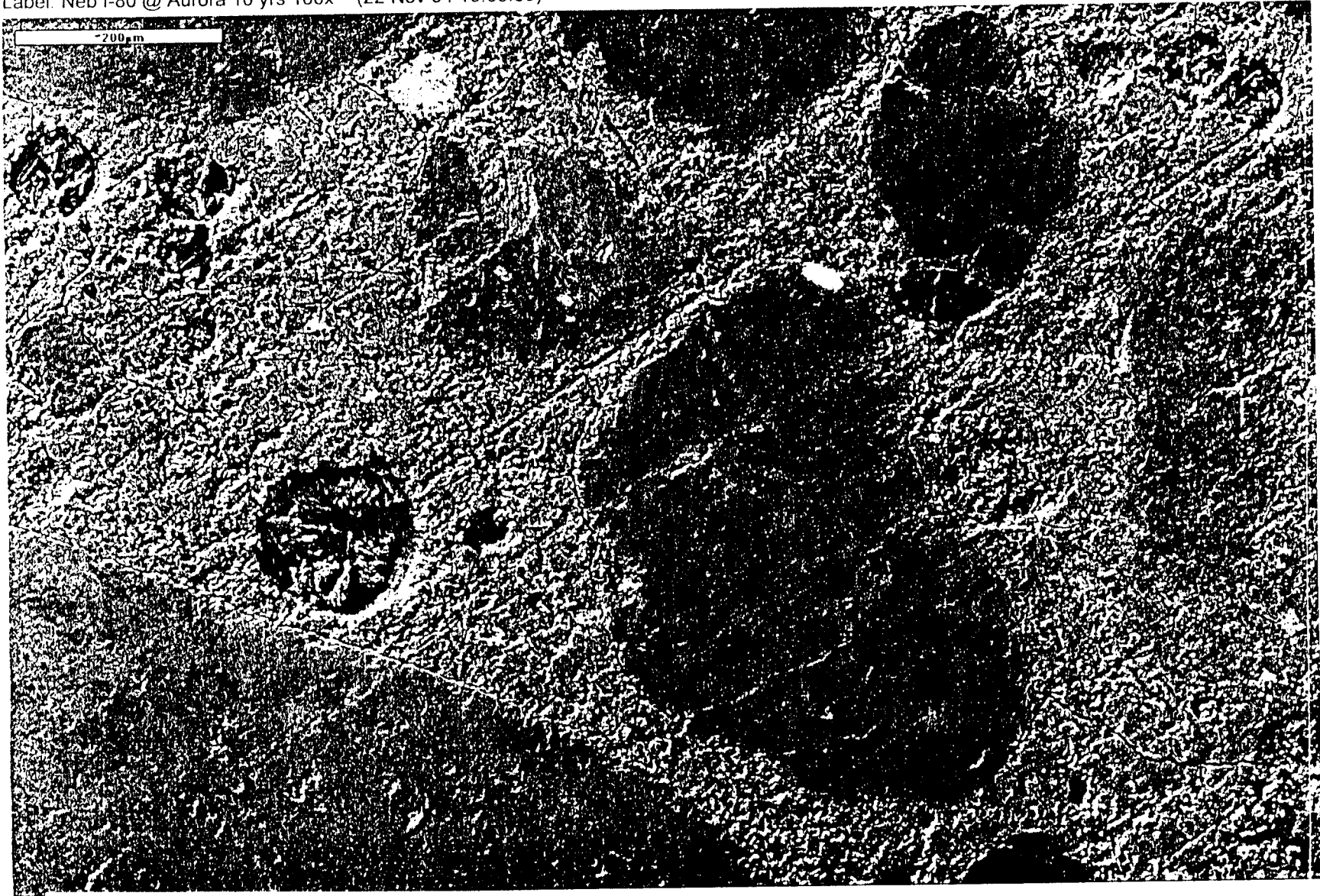
**FIGURE 6 A 100X BSE PHOTOMICROGRAPH OF IOWA 20 SHOWING CRACKS
RADIATING FROM THE ETTRINGITE FILLED AIR VOIDS**

Operator: Scott S
Client: Iowa Dept. of Trans.
Job: HR-358 Task 1
Label: 3T-2, 1500X, US 520, EB LANE (2 Nov 94 12:39:57)



**FIGURE 7 THE ELEMENTAL MAP FOR THE
FIGURE 6 PHOTOMICROGRAPH**

Operator: Wendell D
Client: Iowa DOT
Job: Iowa DOT
Res: High
Label: Neb I-80 @ Aurora 10 yrs 100x (22 Nov 94 10:05:39)



**FIGURE 8 A 100X BSE PHOTOMICROGRAPH OF
A NEBRASKA I-80 AURORA PCC SAMPLE**

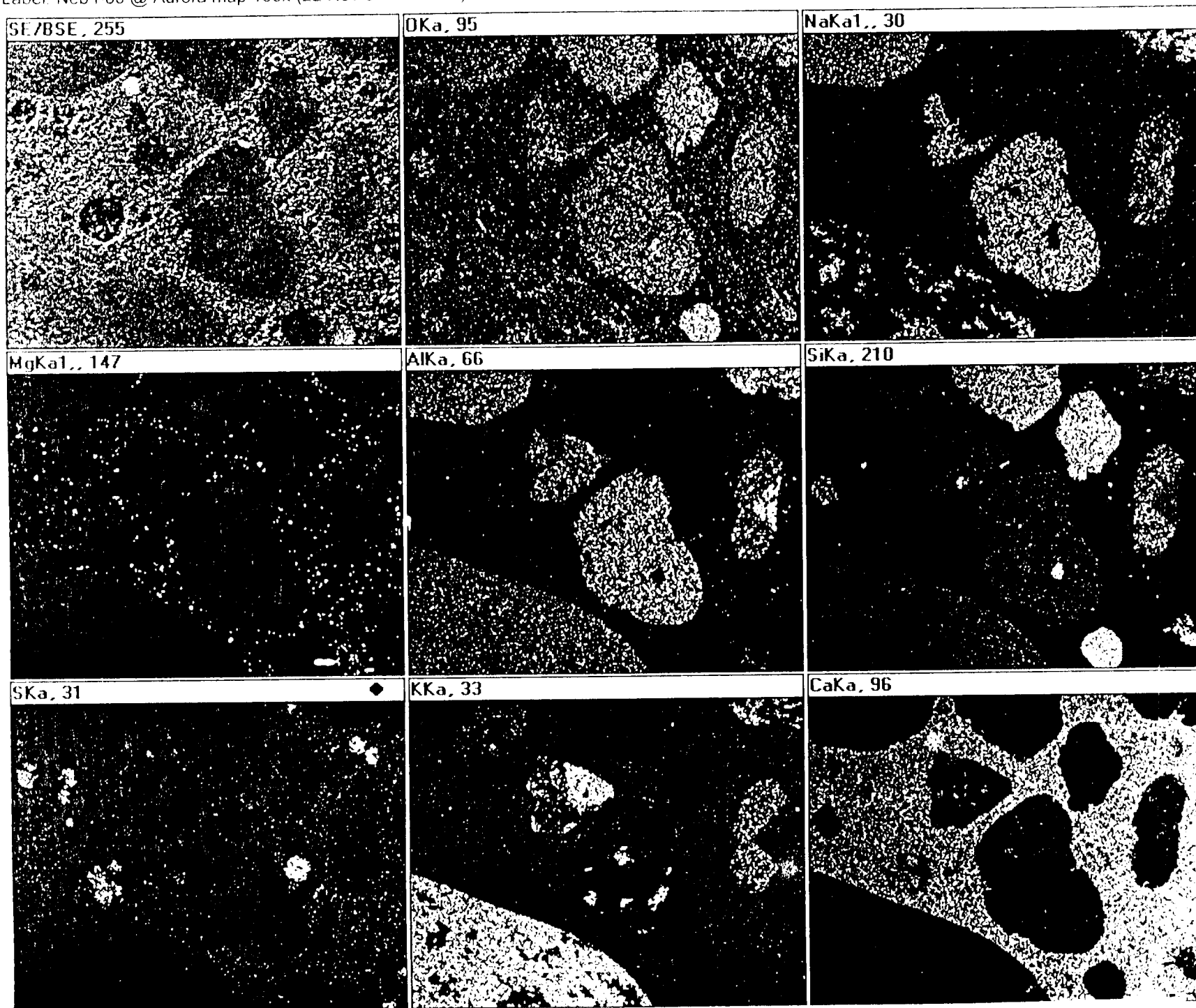
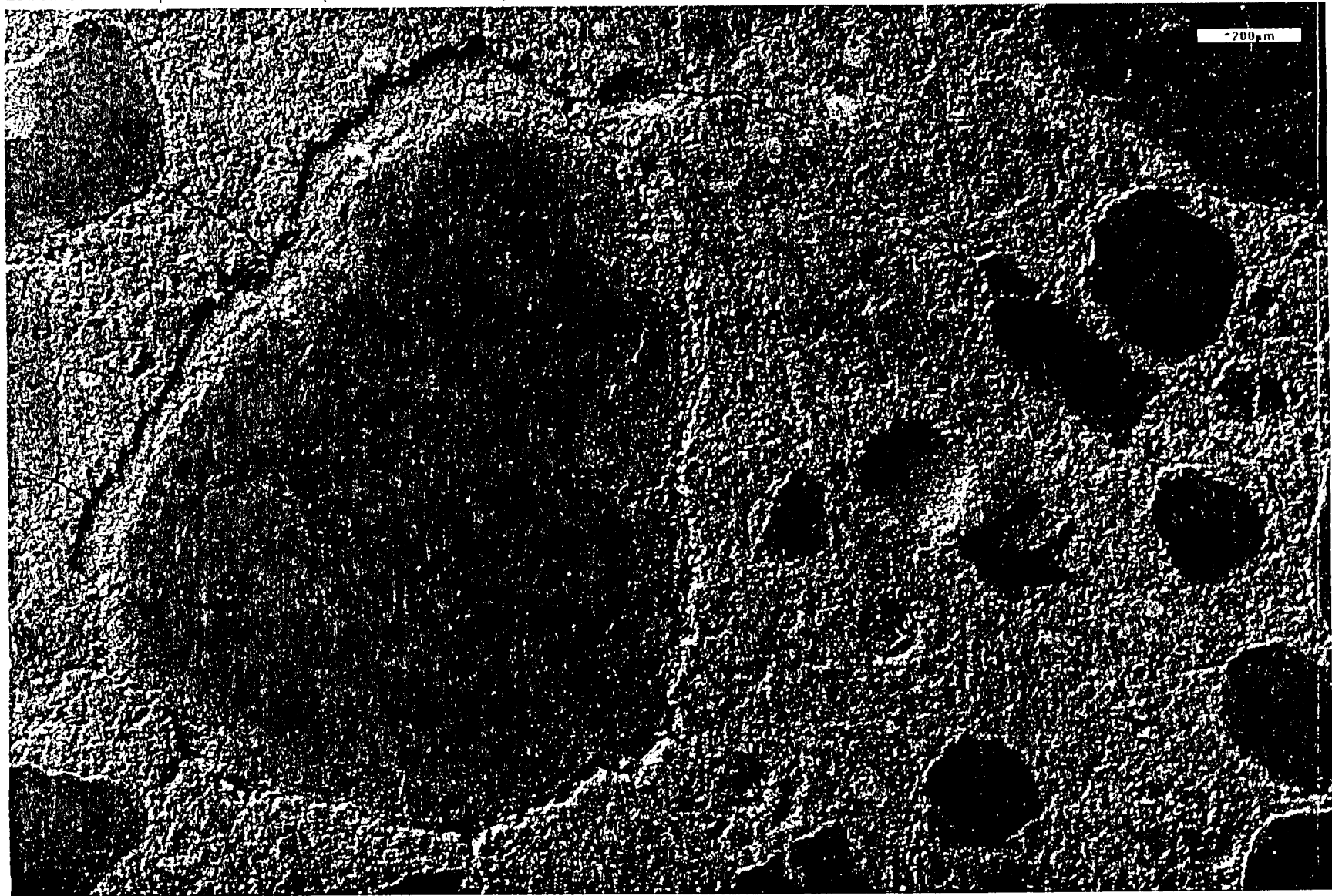


FIGURE 9 THE ELEMENTAL MAPS FOR THE FIGURE 8 PHOTOMICROGRAPH

Operator: Wendell D
Client: Iowa DOT
Job: Iowa DOT
Res: High
Label: Omaha Airport 50x

(22 Nov 94 15:10:12)



**FIGURE 10 A 100X BSE PHOTOMICROGRAPH OF A
NEBRASKA OHMAHA AIRPORT PCC SAMPLE**

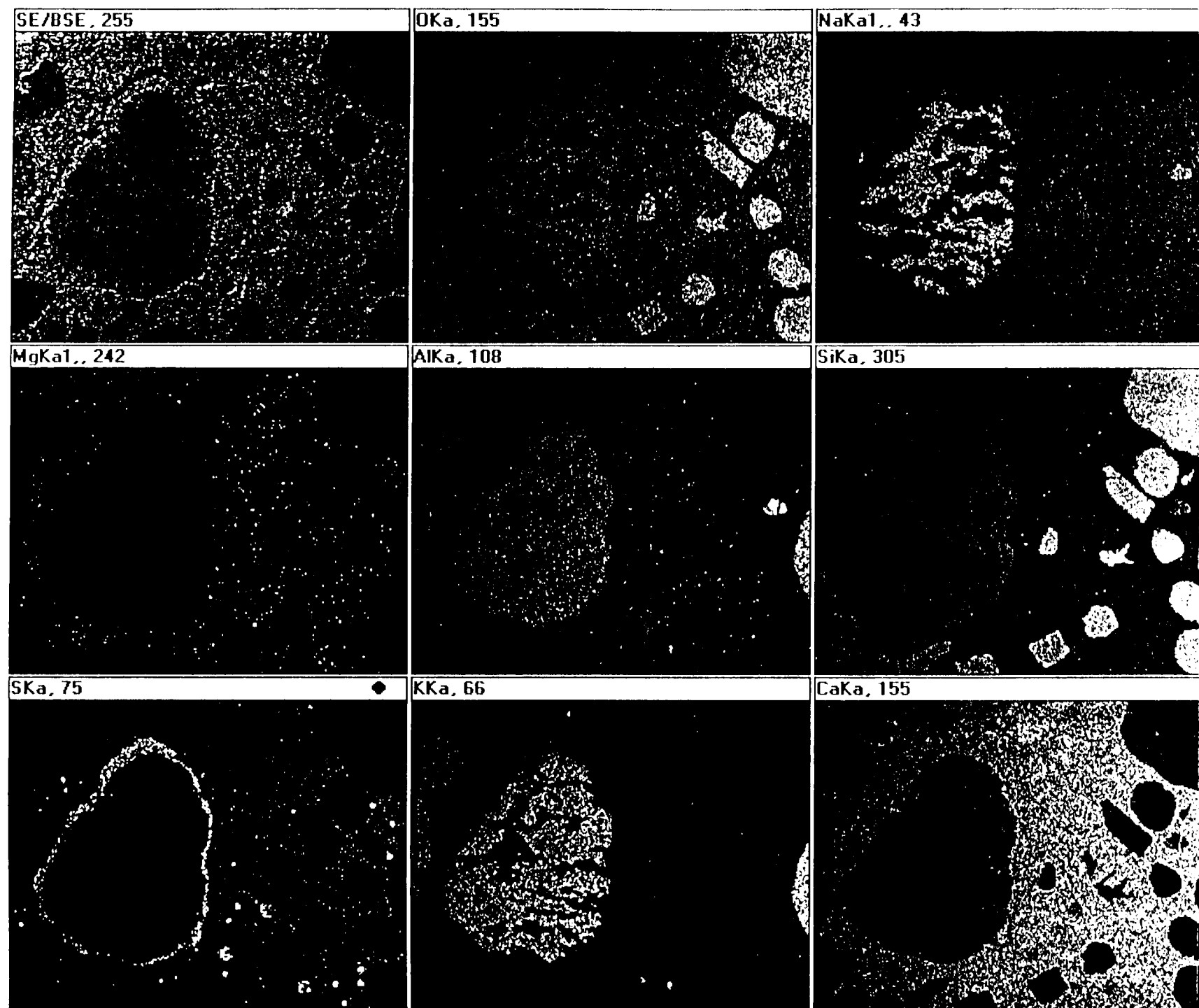


FIGURE 11 THE ELEMENTAL MAPS FOR THE FIGURE 10 PHOTOMICROGRAPHS

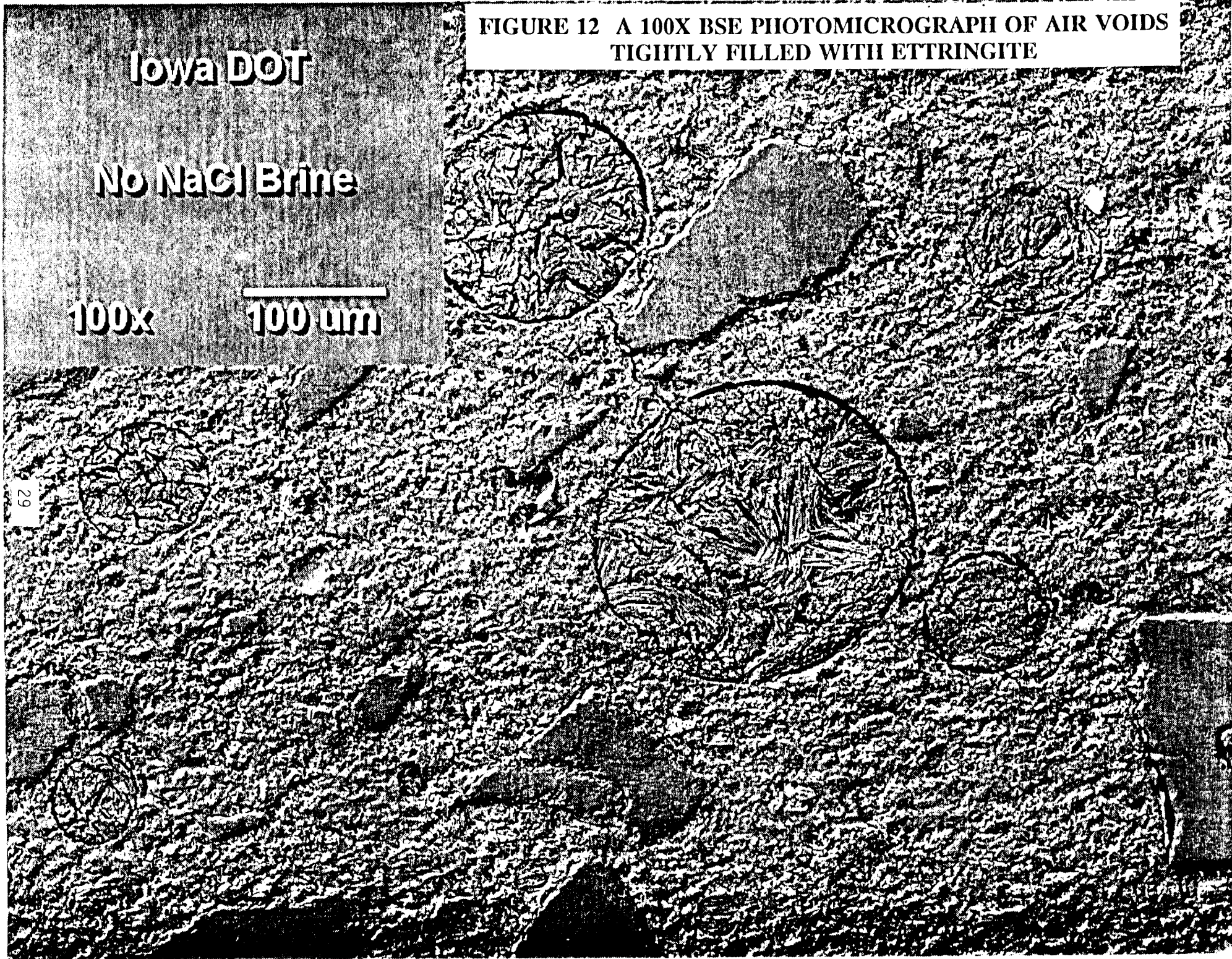
**FIGURE 12 A 100X BSE PHOTOMICROGRAPH OF AIR VOIDS
TIGHTLY FILLED WITH ETTRINGITE**

Iowa DOT

No NaCl Brine

100x

100 μ m



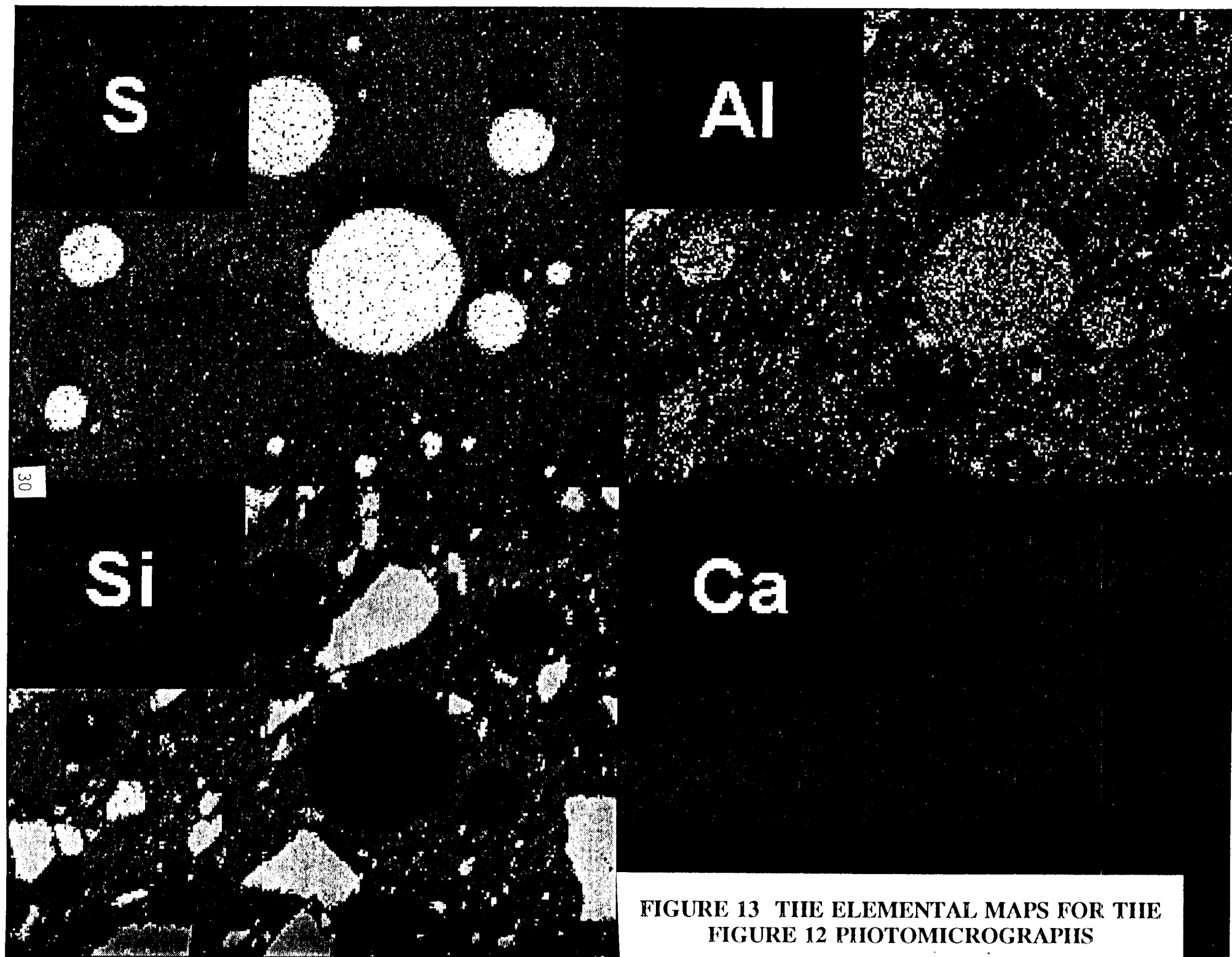


FIGURE 13 THE ELEMENTAL MAPS FOR THE
FIGURE 12 PHOTOMICROGRAPHS

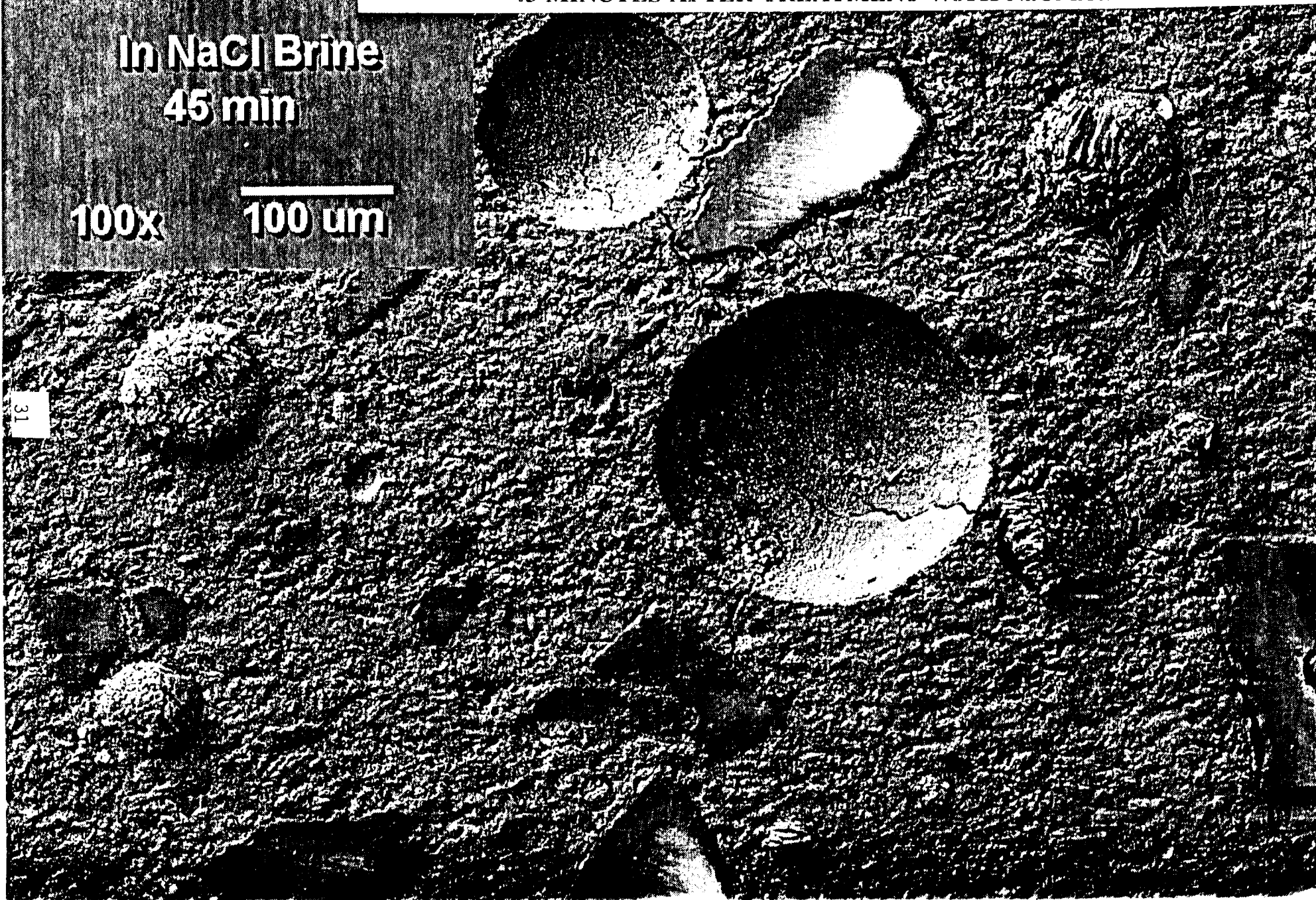
Iowa DOT

**FIGURE 14 A 100X BSE PHOTOMICROGRAPH OF THE FIGURE 12 AIR VOIDS
45 MINUTES AFTER TREATMENT WITH NaCl BRINE**

**In NaCl Brine
45 min**

100x

100 μ m



**FIGURE 15 A 100X BSE PHOTOMICROGRAPH OF THE FIGURE 12 AIR VOIDS
48 HOURS AFTER TREATMENT WITH NaCl BRINE**

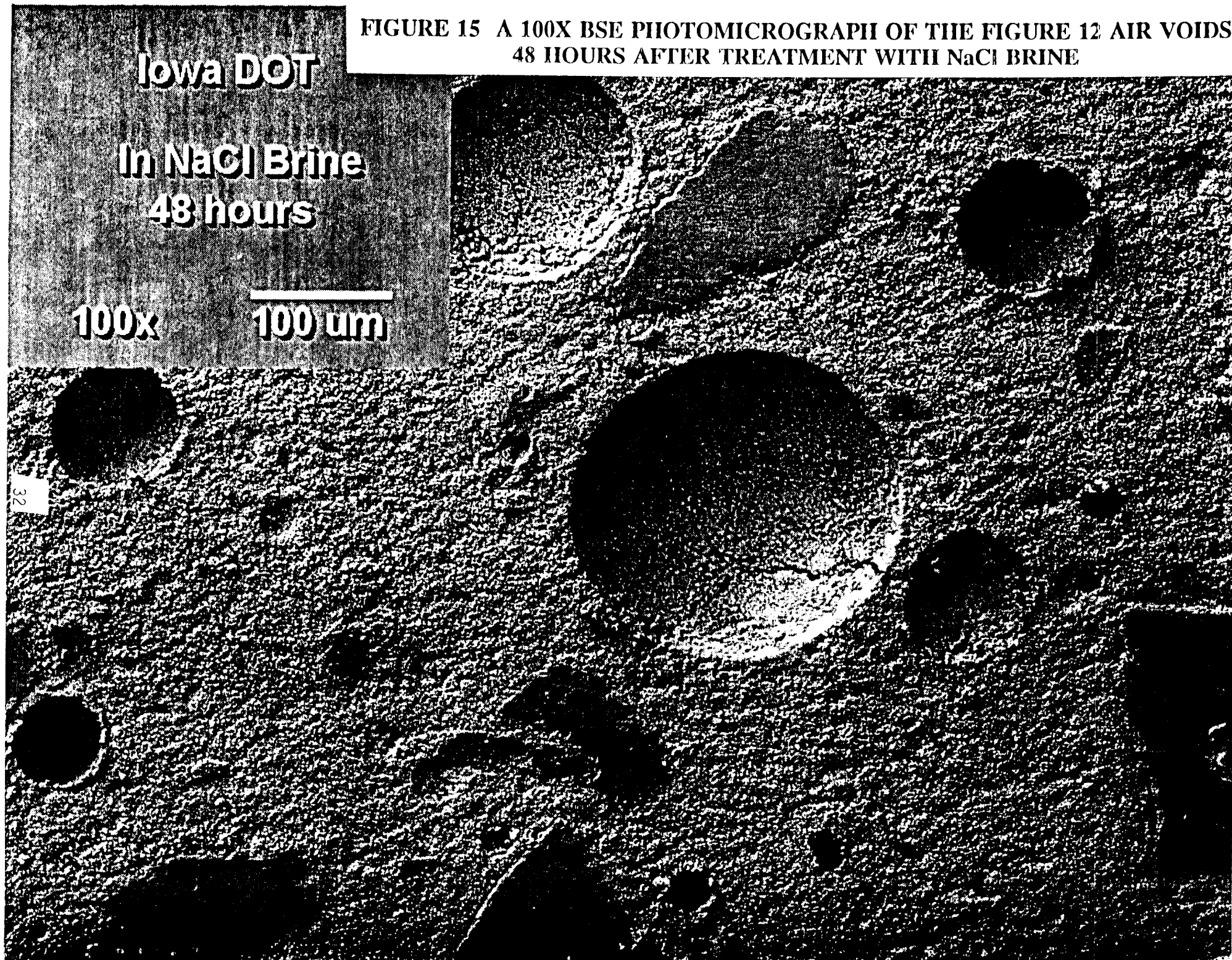


Figure 16 DUGGAN TEST RESULTS
WITH SODIUM CHLORIDE BRINE MODIFICATION

