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REPORT OF TEST

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ROCKWELL HANFORD OPERATIONS
NORTH AMERICAN SPACE OPERATIONS
2401 Stevens Dr. P.O. Box 800
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CERAMIC SUBSURFACE MARKER PROTOTYPES -
SUBCONTRACT SA-5028

May 2, 1985

APPROVED FOR
PUBLIC RELEASE

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TEST REPORT NO. LA 50205

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REFERENCES

1. Tests and charges were authorized by Mr. J.R. Olson, Subcontracts Group Administrator, MO-404/Room 6/1100 Area in Subcontract SA-5028 dated February 8, 1985 and in alteration No. 2 dated March 19, 1985.
2. Tests performed under the direction of Mr. Mel Adams, Technical Representative (2750 East Bldg/Room 200).
3. "Annual Book of ASTM Standards (1983), American Society for Testing and Materials, Philadelphia, PA." (also designated as ASTM).

REQUEST

Determine the following properties per ASTM Methods:

1. Compressive Strength (ASTM-C648-78).
2. Thermal Shock ((ASTM-C484).
3. Thermal Crazing (ASTM-C554).
4. Alkali Resistance (ASTM-C614).
5. Color Resistance (ASTM-C538).
6. Chemical Resistance (ASTM-C650-83).

SAMPLE DESCRIPTION

The Client submitted five (5) sets of porcelain and stone-ware subsurface (Radioactive site) marker prototypes (31 markers each set) and identified the sets as A, B, C, D, and E.

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TEST PROCEDURES

1. Compressive Shock

a) Method (ASTM-C648: Breaking Strength of Ceramic Tile)

Ten specimens from each set of samples were tested for breaking strength employing the support and force apparatus described in para. 6.1 and 6.2 of ASTM-C648 with the following equipment and conditions:

Load Instrument: Instron Universal Testing Machine;
Model TTC

Load Cell: 10K (10,000 lbs.)

Load Rate: 800 lbs./min.

Full Scale Load: 2,000 lbs. (unless otherwise indicated)

RESULTSCOMPRESSIVE STRENGTH, MAX. LOAD (LBS.)Marker Samples

<u>Specimen No.</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>
1	640	1340	1525	1380	1650
2	700	1380	1530	730	2800
3	1300	1400	1230	500	1180
4	940	1280	1690	960	2550
5	780	1740	1540	2230	1460
6	700	1700	1620	2600	2950
7	1320	1340	1780	1200	2750
8	830	1710	1500	860	2600
9	1000	1560	1920	2060	2000
10	<u>1230</u>	<u>1660</u>	<u>1400</u>	<u>1800</u>	<u>3050</u>
Average	940	1510	1575	1600	2300

The following observations were noted on the exposed interiors of the specimens:

Set A - This stoneware sample possesses a white interior with a consistent finely-sintered appearance.

Set B - This stoneware possesses a yellow interior which, for the most part, has a consistent finely-sintered appearance. However, the interior also contains a few singular cracks or voids.

RESULTS

(Cont)

Set C - This stoneware contains a yellow interior with many small fractures or cracks.

Set D - This stoneware has a yellow interior with many small cracks or fractures. The interior yellow is not homogeneous in consistency or color: some areas are a dull baked yellow while other areas are a raw porous yellow.

Set E - The stoneware possess a yellow interior with many small cracks or fractures.

It should also be noted that during the compression test, Sets D and E (the rounded top specimens) occasionally broke into two pieces rather than three pieces. Sometimes the breakage was in the form of large chips from the surface, rather than actual splitting of the specimen.

TEST PROCEDURES

2. Thermal Shock Resistance

a) Method (ASTM-C484; Thermal Shock Resistance of Glazed Ceramic Tile)

Three specimens from each set were heated for 30 minutes in an oven at $293 \pm 9^{\circ}\text{F}$ ($145 \pm 5^{\circ}\text{C}$). The specimens were then removed from the oven and placed glazed side up on an aluminum sheet which was maintained at a room temperature of $75 \pm 5^{\circ}\text{F}$ ($24 \pm 3^{\circ}\text{C}$). After 15 minutes each specimen was visually evaluated for any shivering (i.e. breaking into small pieces), using a dye solution (methylene blue solution) to demarcate any physical changes to the specimen. Unchanged specimens were retested for an additional four cycles of heating (293°F) and cooling (75°F).

b) RESULTS

(Thermal Shock Resistance)

Set Identification

Evaluation

A	No failure for any of the specimens through 5 cycles.
B	No failure for any of the specimens through 5 cycles.
C	No failure for any of the specimens through 5 cycles.
D	No failure for any of the specimens through 5 cycles.
E	No failure for any of the specimens through 5 cycles.

TEST PROCEDURES

3. Thermal Crazing Resistance

a) Method (per ASTM-C554; Crazing Resistance of Fire Glazed Ceramic Whitewares by a Thermal Shock Method)

Five specimens from each set were heated in an oven maintained at a temperature of $300 \pm 5^{\circ}\text{F}$ ($149 \pm 3^{\circ}\text{C}$) for not less than 45 minutes and not more than 60 minutes. The specimens were then removed from the oven and immediately quenched in water at $68 \pm 5^{\circ}\text{F}$ ($20 \pm 3^{\circ}\text{C}$). After the quenched specimens had cooled they were dried and examined for body cracks or craze marks. Failed specimens were eliminated from further testing. Undamaged specimens were retested for an additional four (4) cycles of heating (300°F), quenching (68°F), and examination (for cracking or crazing).

Any crack-or craze-resistant specimens were retested at higher oven temperatures in increments of 30°F up to a temperature of 510°F (266°C). At each temperature step, the specimens were tested for a total of five (5) cycles. Again, any specimens which exhibited cracking or crazing were eliminated from further testing.

b) RESULTS(Thermal Crazing Resistance)Evaluations of Individual Sets

<u>Set, ID</u>	<u>Temperature Level (°F)</u>	<u>Cycle Completed</u>	<u>Number of Specimens</u>	<u>Defect</u>
A	300	5	1	Crazing
	390	1	3	Crazing
	420	5	1	Crazing
B	390	1	1	Crazing
	420	2	1	Crazing
	480	2	2	Cracking
	510	1	1	Cracking
C	330	3	1	Crazing
	480	4	1	Crazing
	510	2	3	Crazing
D	390	4	1	Cracking
	420	3	3	Cracking/ Crazing
	420	4	1	Crazing
E	390	1	3	Cracking
	390	4	1	Cracking
	420	4	1	Cracking

SUMMARY OF RESULTS

SET IDENTIFICATION

<u>Temperature (°F)</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>
	<u>Total Specimen Failures</u>				
300	1	0	0	0	0
330	1	0	1	0	0
360	1	0	1	0	0
390	4	1	1	1	4
420	5	2	1	5	5
450	-	2	1	-	-
480	-	4	2	-	-
510	-	5	5	-	-

TEST PROCEDURES

4. Alkali Resistance

a) Method (per Modified ASTM-C614; Alkali Resistance of Porcelain Enamels)

Six specimens from each set were rinsed with tap water, washed with 1% trisodium phosphate solution, rinsed with distilled water, dried at 110°C for 15 minutes, cooled and then weighed.

Because of the ridges running through the middle of the pictograph, the opposite face with the lettered warning was tested for alkali resistance.

Six weighed specimens at a time were fastened to the ports of the extraction assembly adapted from both ASTM C614 and Mil Spec. T-12295 (see notes). The assembled apparatus was filled with fresh preheated (to 96°C) extraction solution (composed of 260 grams of hydrated tetrasodium phosphate ($\text{Na}_4 \text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$) dissolved in 4.90 liters of distilled water). The entire assembly was then placed on a hot plate, and the solution stirred and refluxed for 6 hours at 96°C.

TEST PROCEDURES

(Cont)

At the end of the exposure time, the specimens were removed from the extraction assembly and washed, dried and reweighed.

The dimensions of the exposed areas were measured and the alkali resistance determined by dividing the loss (or gain) in weight of the specimens by the area of exposure.

NOTE:

The extraction assembly consisted of a stainless steel chamber with six 3" diameter ports along the four sides of the chamber. The specimens were sealed with gaskets and fastened with steel backing plates over the ports.

Photographs (1-4) of the assembly are included with this report.

b) RESULTS

(Alkali Resistance)

Average Area of Exposure 6 Sq.Inches

<u>Set Identification</u>	<u>Weight Change, mg./sq. in.</u>
A	< 0.5
B	(+) 0.7
C	< 0.5
D	< 0.5
E	(-) 0.9

NOTE: (+) - weight gain
(-) - weight loss

Set B showed a consistent weight gain while Set E showed a consistent weight loss. Only Set E exhibited any visible changes. The glazed surface developed a white glassy appearance.

TEST PROCEDURES

5. Color Retention

a) Method (per ASTM-C538; Color Retention of Red, Orange, and Yellow Porcelain Enamels)

One specimen from each set was cleaned with soap and water and rinsed with distilled water. The surface was dried with clean dry towels.

Several drops of 50% cupric sulfate solution were placed on a test area of the specimen and immediately covered with a one inch (25mm) diameter watch glass. The quantity of cupric sulfate solution was sufficient to fill the inverted watch glass. To reduce leakage of solution, the area of contact between the watch glass and the surface of the specimen was sealed with a Dow Corning silicone sealant.

Each specimen was placed under the light chamber apparatus described in para. 5.1 of C 538 for 20 hours at 80°F.

After exposure, the watch glass was removed. The treated area was rinsed with water and dried with a clean cloth. Immediately after drying, the treated area was visually examined for any darkening or discoloration.

b) RESULTS
(Color Retention)

<u>Set Identification</u>	<u>Result of Test/Observation</u>
A	No darkening
B	No darkening
C	No darkening
D	No darkening
E	No darkening

NOTE:

The color retention requirement is that no darkening of the treated area shall be visible.

TEST PROCEDURES

6. Chemical Resistance

a) Method (ASTM-C650; Resistance of Ceramic Tile to Chemical Substances)

Twelve specimens from each set were cleaned with ethyl alcohol and then dried. After cleaning, six specimens were immersed in a 10% hydrochloric acid (HCl) solution and the other six specimens were immersed in a 10% potassium hydroxide (KOH) solution. Each specimen was immersed to about one half of its diameter, for 24 hours at $75 \pm 5^{\circ}\text{F}$ ($24 \pm 3^{\circ}\text{C}$). After immersion the specimens were removed from the solution, washed with water, dried at 100°C for 4 hours and cleaned again with ethyl alcohol.

A visual evaluation of the exposed areas of the tiles was made by comparing at various angles of illumination the exposed surfaces with the unexposed surfaces of the tiles.

b) RESULTS(Chemical Resistance)

<u>Set Identification</u>	<u>10% Hydrochloric Acid</u>	<u>10% Potassium Hydroxide</u>
A	Not affected.	Not affected.
B	Not affected.	Not affected.
C	Not affected.	Not affected.
D	Not affected.	Not affected.
E	Not affected.	Slight loss of gloss.

NOTES:

Not affected=No change in color or tint of surface of specimen.

Three of the acid exposed and three of the alkali exposed specimens for each set were employed also in the compression (breaking strength) test. The type of exposure (acid or alkali) had no apparent effect on the breaking strength. As described in para. 8.1 of C650, after breakage, the outer layer (skin) of the specimens was examined for any changes. No observable differences between the immersed outer layer and the unimmersed outer layer was observed in any of the specimens.

Photo 1 - Alkali Resistance (ASTM-C614). Test chamber with six mounted specimens.

Photo 2 - Interior of alkali resistance chamber

Photo 3 - Alkali resistance test chamber with back plates and orientation of specimens.

Photo 4 - Alkali resistance test chamber with unmounted specimens and gaskets over ports.

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