GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[x] ORIGINAL [ ] REVISION NO. ______

Project No. A-3073 DATE 10/13/81

Project Director: Mr. J. N. Harris School/Lab EMSL/MRB

Sponsor: University of Houston Central Campus; Houston, Texas 77004

Type Agreement: Subcontract under DOE Prime Contract No. DE-AC03-81SF11492

Award Period: From 8/1/81 To ~~11/30/81~~ 12/31/81 (Performance) ----- (Reports)

Sponsor Amount: $39,243 Contracted through:

Cost Sharing: None GTRI/GIT

Title: Solar Energy Treatment of Ceramic Tile

ADMINISTRATIVE DATA

OCA Contact Duane Hutchison x4820

1) Sponsor Technical Contact:

Mr. Frank B. Smith
Solar T hermal Test Facilities Users Association
Suite 1204, Central & San Mateo N.E.
First National Bank Building East
Albuquerque, N.M. 87108
505/268-3994

2) Sponsor Admin/Contractual Matters:

Ms. Rosemary Grimmet
Deputy Director
Office of Sponsored Programs
University of Houston
Central Campus
Houston, Texas 77004
713/749-3412

Defense Priority Rating: None

Security Classification: None

RESTRICTIONS

See Attached Government Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval – Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with Government; except that items costing less than $1,000 vests with GIT if prior approval to purchase is obtained from the Contracting Officer.

COMMENTS:

OCT 1981 RECEIVED Research Reports Office

COPIES TO:

Administrative Coordinator	Research Security Services	EES Public Relations (2)
Research Property Management	Reports Coordinator (OCA) ✓	Computer Input
Accounting	Legal Services (OCA)	Project File
Procurement/EES Supply Services	Library	Other ______

FORM OCA 4:781

GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION SHEET

Date 3/12/82

Project Title: Solar Energy Treatment of Ceramic Tile

Project No: A-3073

Project Director: Harris

Sponsor: University of Houston

Effective Termination Date: 12/31/82

Clearance of Accounting Charges: 1/31/82 (Rpts)

Grant/Contract Closeout Actions Remaining:

- [x] Final Invoice and Closing Documents
- [ ] Final Fiscal Report
- [x] Final Report of Inventions (DOE Form)
- [ ] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other ____________

Assigned to: EMSL (School/Laboratory)

COPIES TO:

Administrative Coordinator
Research Property Management
Accounting
Procurement/EES Supply Services
Research Security Services
Reports Coordinator (OCA) ✓
Legal Services (OCA)
Library
EES Public Relations (2)
Computer Input
Project File
Other ____________

FORM OCA 10:781

A-3073

FINAL REPORT

SOLAR ENERGY TREATMENT OF CERAMIC TILE

By

J. N. Harris
M. E. Clayton

Prepared for

THE UNIVERSITY OF HOUSTON

Subcontract Under

Contract No. DE–AC03–81SF11492

December 1981

GEORGIA INSTITUTE OF TECHNOLOGY

A Unit of the University System of Georgia
Engineering Experiment Station
Atlanta, Georgia 30332

1981

FINAL REPORT

SOLAR ENERGY TREATMENT OF CERAMIC TILE

By

J. N. Harris
M. E. Clayton

For

The University of Houston

Subcontract Under Contract No. DE-AC03-81SF11492

GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station
Atlanta, Georgia 30332

December 1981

ABSTRACT

The Department of Energy 400 kW Advanced Components Test Facility, located on the Georgia Institute of Technology campus, was used to provide a concentrated source of solar energy for firing ceramic wall tile. A domed top cylindrical cavity with a white refractory fiber lining provided diffuse reflection of the concentrated solar beam directly onto the upper surface of the unfired wall tile. The tile were placed directly on the cavity floor in a circular pattern, centered at 45 degree intervals so that eight tile could be fired at one time. The tile and cavity walls were instrumented with thermocouples, and pyrometric cones were used to determine temperature distribution within the cavity.

The glazed and unglazed solar fired tiles were subjected to standard ceramic testing procedures to determine: flatness, modulus of rupture, water absorption, porosity, bulk density, apparent specific gravity, percent linear thermal expansion and crystalline phases present in the fired bodies. These data were compared with the same data for commercial fired tiles from the same batch of raw materials. The glazed tile surfaces were compared with commercially fired tile for reflectance and color match.

The major problems encountered were: cracking by thermal shock, and uneven shrinkage and glaze maturity across individual tile. The cavity also failed to provide even heating at all eight tile positions. The hottest position in each tile firing depended on the sun's position in the sky and on the velocity and direction of the wind. Despite these problems some acceptable tile were fired that met the flatness standard of the comparable commercially fired tile and some came very close to matching the glaze color of the commercial tile.

An alternate air heat exchanger system is recommended to fire the tile by convection rather than direct radiation.

TABLE OF CONTENTS

(Continued)

TABLE OF CONTENTS (Continued)

LIST OF ILLUSTRATIONS

(Continued)

(Continued)

LIST OF ILLUSTRATIONS (Concluded)

LIST OF TABLES

(Continued)

LIST OF TABLES (Continued)

I. OBJECTIVE

The objective of this program was to show that solar energy could be used to translate ceramic wall tile from the unreacted raw state to the completely reacted fired state.

II. INTRODUCTION

Manufacture of ceramic tile is an energy intensive process requiring temperatures of about 1100^{o} C through part of the firing cycle. The possibility for utilizing radiant solar energy in this process should be evaluated with electrical energy considered particularly as an auxiliary or standby energy source.

Presently, gas and oil fossil fuels provide energy, including the high temperature radiant heat, required for ceramic tile manufacture. A great excess of combustion air is necessarily heated when fossil fuel is used. Also, a great mass of refractory including refractory product support and kiln walls are raised to the incandescent radiant temperature in typical factories. About six trillion Btu's, or 1.8 billion kwhr, were required to produce 300 million square feet of ceramic tile in the United States during 1979.* Energy consumption by the world ceramic tile industry is estimated to be 300 trillion Btu's. Thus, a typical tile factory consuming about 240 billion Btu's to produce 12,000,000 square feet paid $864,000 for fuel in 1980. The modern factory of 12,000,000 square foot capacity requires about $11,000,000 capital and is predicted as being reliably profitable in today's market. Small solar energy collectors of 2 to 20 acres in area conceivably could: (1) reduce consumption of fossil fuels, (2) improve

---

*From reports to the Department of Energy by Tile Council of America, Inc. (Please see enclosed "PROFILE OF THE CERAMIC TILE INDUSTRY.")

overall energy efficiency, and (3) improve the quality of the product. Clean solar radiation is not likely to result in the contamination of glazes.

This program examined the feasibility of using direct solar radiation to fire a commercial ceramic wall tile body. The Department of Energy Advanced Components Test Facility located on the Georgia Institute of Technology campus and operated by the Engineering Experiment Station, Energy and Materials Sciences Laboratory was used to provide the source of concentrated solar energy required to process the raw tile bodies to maturity.

III. EXPERIMENTAL PROCEDURE

A. Test Facility

1. DOE 400 kW Advanced Components Test Facility (ACTF)

The Advanced Components Test Facility (ACTF) is a tracking mirror solar concentrator system operated by the Georgia Institute of Technology's Engineering Experiment Station (Georgia Tech EES) for the U. S. Department of Energy (DOE). This facility, shown in Figure 1, is located on the campus of Georgia Tech in Atlanta, Georgia.

Major elements of the facility include a solar concentrating mirror field, a rigid structural steel test tower located at the geometric center of the mirror field, an experiment support platform (tower deck) mounted on top of the tower, an instrument and control building, a computerized data collection system, and a heat rejection system. The mirror field consists of 550 heliostats (tracking mirrors) that reflect and concentrate direct solar radiation to a stationary focal zone centered in the aperture of the elevated

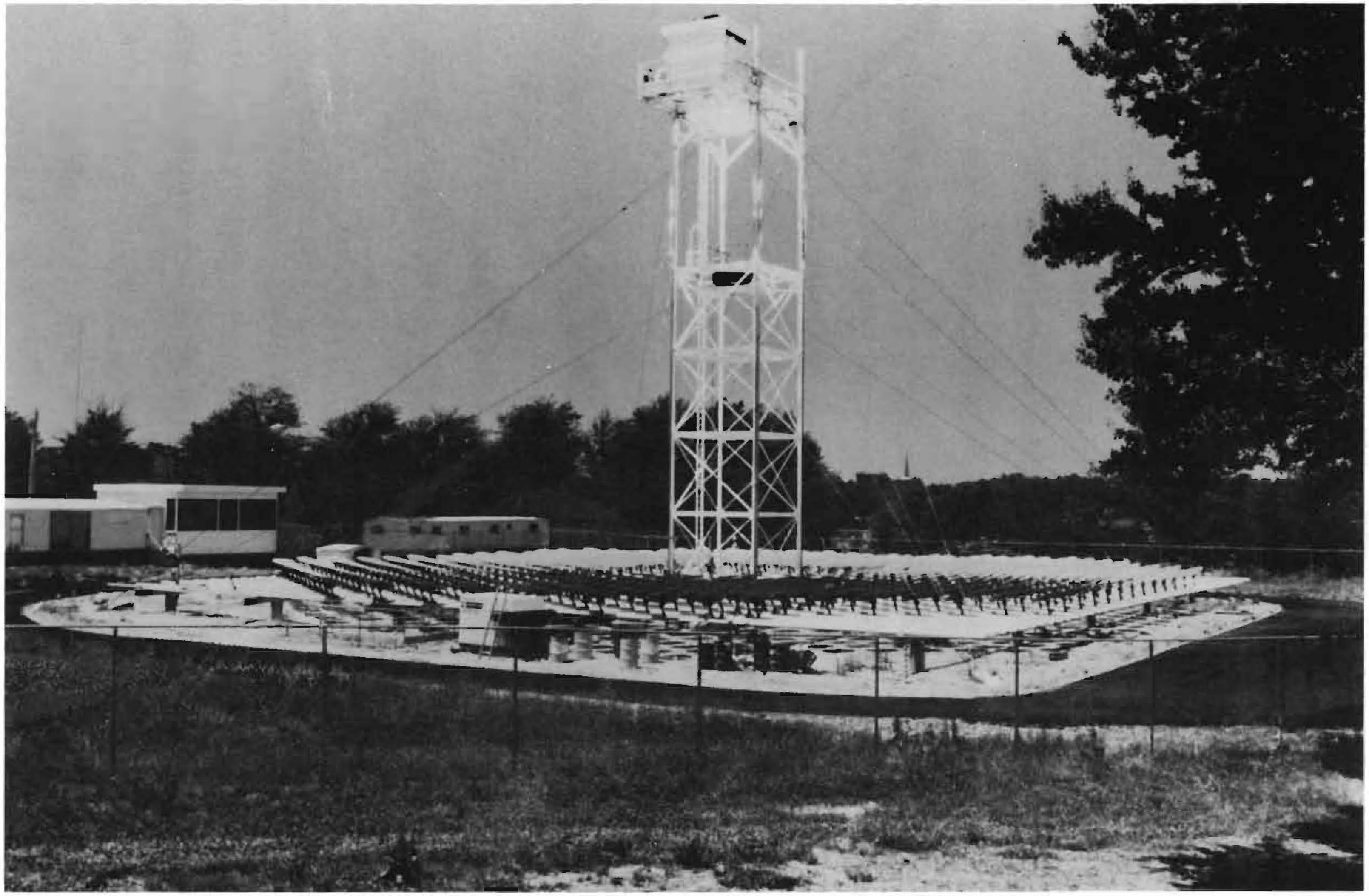

Figure 1. U. S. Department of Energy Advanced Components Test Facility (ACTF), Located on the Georgia Tech Campus, Atlanta, Georgia.

tower deck. The maximum solar radiative flux available at the focus is approximately 125 W/cm^2 (3.96×10^5 Btu/ft^2-hr) representing a total power input of 325 kW (1.11×10^6 Btu/hr) based on a nominal local insolation of 900 W/m^2 (285 Btu/ft^2-hr).

A computerized data collection system is available at the facility to record, condition, display and reduce user data. This system, shown schematically in Figure 2, consists of two PDP-8/a minicomputers,* a twelve-bit multiplexed A-to-D converter, a graphics terminal to allow real-time display of data, two disk-type mass storage devices, and a hard-copy graphics terminal. The first of the two computers is located in the control building and serves as the master control for the system. The second computer, located in the tower deck building, serves as an interface to a Digital Equipment Corporation Industrial Control Subsystem (ICS) and is configured to allow unattended operation. The ICS subsystem is an analog to digital and digital to analog converter/multiplexer system currently capable of accepting up to 120 channels of analog input. These channels can be scanned at rates up to 200 channels/second, with a single channel capable of being scanned 20 times/second. All scanning routines are under computer software control. The digitized raw data is stored on high speed magnetic disk for later retrieval.

2. Firing Chamber

The test set up for firing the ceramic wall tile on the ACTF test tower consisted of a water cooled shield with an 18 inch diameter aperture. This shield was located just below the center of focus of the solar beam.

---

*Digital Equipment Corporation, Maynard, Massachusetts.

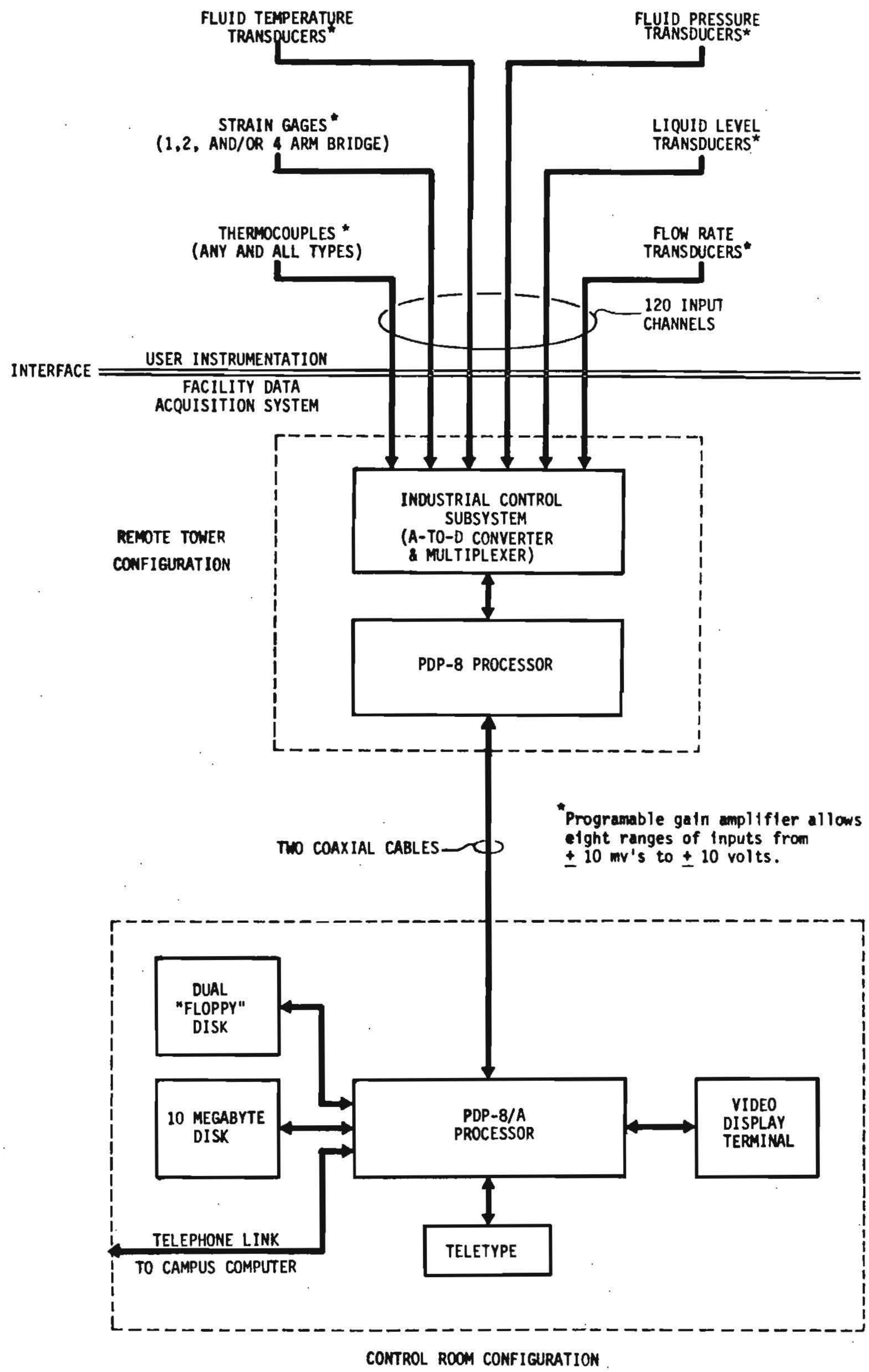

Figure 2. ACTF Data Collection Schematic

A 45 degree tapered opening was cut in the center of a 1 inch thick Babcock and Wilcox (B&W) 3000 board (Saffil Fiberboard) so that the maximum diameter of the opening on the under side of the board was 11 inches. This board served as the base of the firing chamber and tile were placed directly on the board for firing. The rest of the chamber consisted of a DOE owned cavity, Figure 3. The stainless steel domed cavity was lined with a molded in place white refractory ceramic fiber blanket. This white layer served as a diffuse reflector to redirect and disperse the solar energy entering the base of the cavity through the 11 inch diameter aperture. The inside diameter of the cavity was 22 inches, thus, giving a 5½ inch wide circular shelf on which eight tiles could be fired at the same time. The tile were placed in a circular pattern with each tile centered on a 45 degree radial line.

The cavity was suspended on a tripod arrangement by means of a cable attached to a winch so that it could be easily raised or lowered onto the refractory fiberboard base, Figure 4. This ability to raise the cavity off the base was designed as a means of temperature control, but this also facilitated loading and unloading of tile through the aperture plate.

The cavity walls and top were instrumented with ANSI Type K thermocouples at the positions indicated in Figure 5. In addition, the 3000 degree board base had eight thermocouples located on 45 degree lines on a circle 16-3/4 inch in diameter, so that a thermocouple was centered beneath each tile being fired.

B. Ceramic Wall Tile

Glazed and unglazed as pressed commercial wall tile were obtained from a U. S. manufacturer who requested to remain anonymous. In addition, the

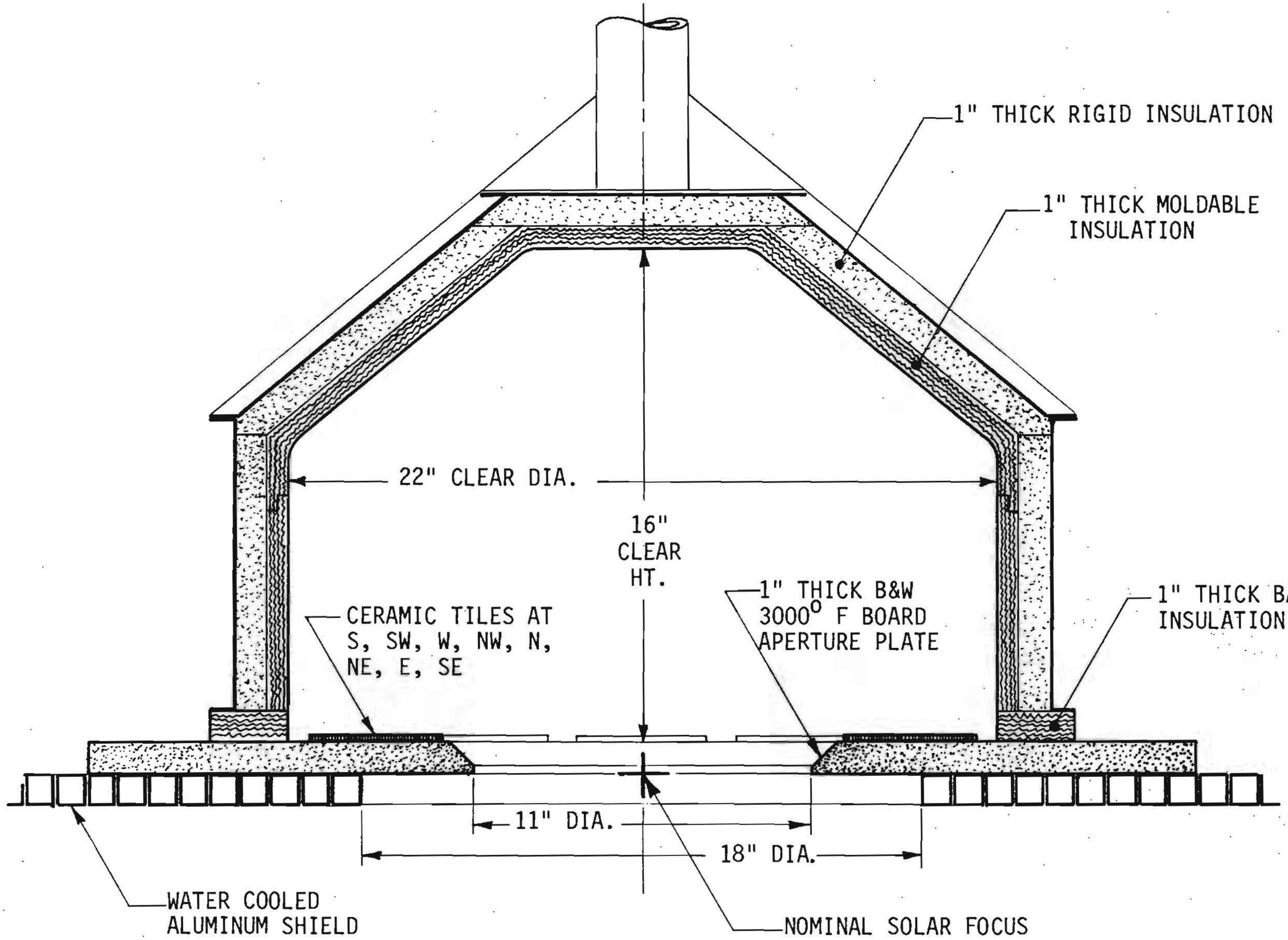

Figure 3. Cross-Sectional View of Cavity Used for Solar Firing Ceramic Wall Tile.

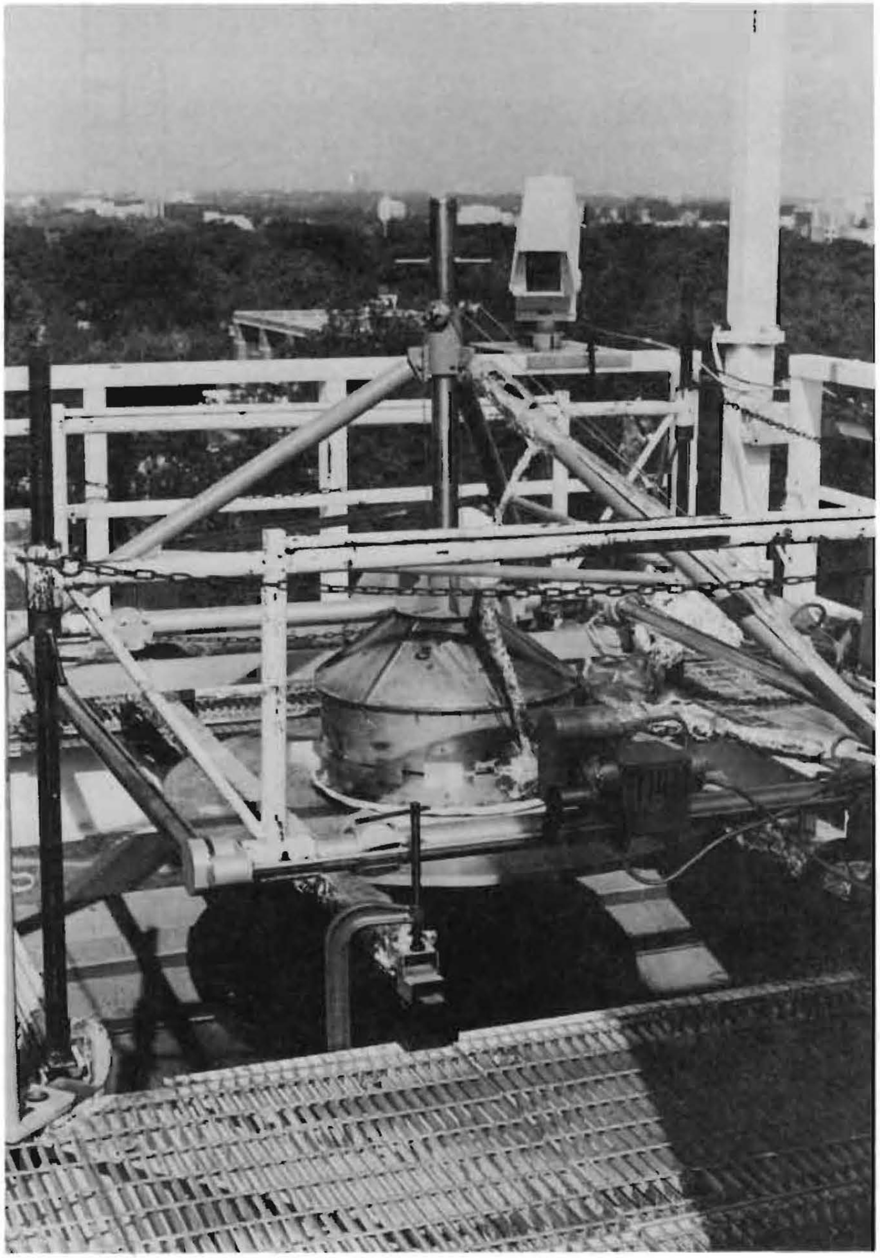

Figure 4. External View of Cavity Showing Tripod and Cable Arrangement for Raising Cavity.

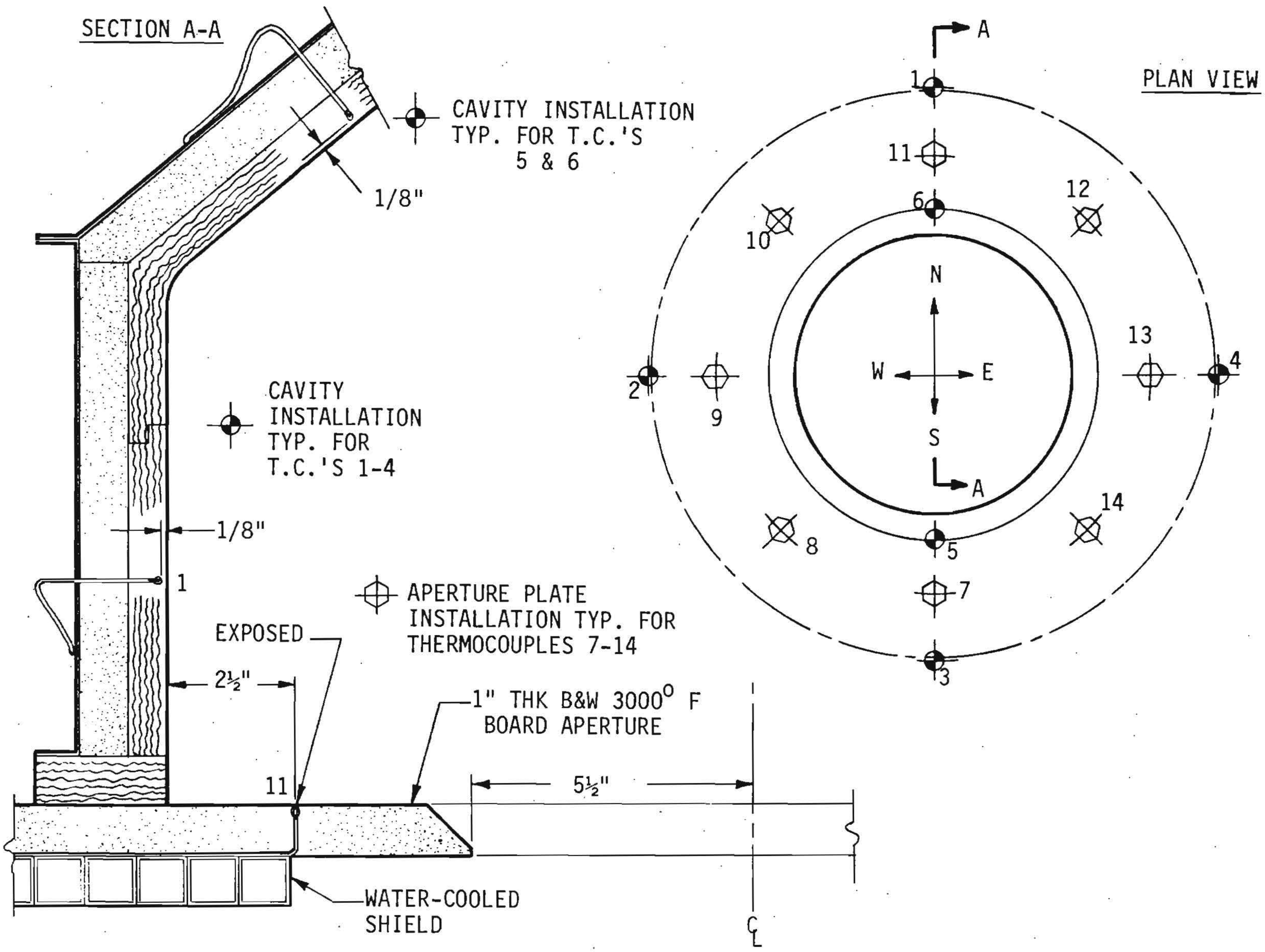

Figure 5. Thermocouple Locations in Cavity Wall.

manufacturer provided five glazed and five bisque fired tiles to be used for comparison of properties with the solar fired tiles. Approximately one-half of the unfired tiles were broken in transit, therefore, during the early solar tests broken tile sections were fired to conserve the whole tiles for the final tests. Twelve test firings were made. Instrumented tiles had one to three type K thermocouples attached. Thermocouples were secured to the tiles by drilling a 1/16 inch diameter hole 1/8 inch deep in the back of the tile. A 26 gauge type K thermocouple bead was postioned in the hole and powdered tile raw material packed into the space around the thermocouple. This packed powder was wet with one to two drops of Ludox "AS" colloidal silica. After drying the thermocouples were firmly attached to the tile. Single thermocouples were located in the geometric center of the tile. When three thermocouples were placed on a tile they were aligned in a vertical row one inch on either side of the center thermocouple.

C. Solar Test Firings

Twelve solar firing tests were conducted. The commercial tiles were reported to be fired at approximately 1100^{0} C with an 18 minute dwell time in the furnace. During the first four tests it was discovered that temperatures of 1150^{0} C "burned out" the glaze color very rapidly so the matrix shown in Table I was set up to cover the expected usable temperature ranges.

1. Test Firing Number 1, 30 October 1981

This test was conducted to check out operation of the cavity. Small sections of unglazed tile were placed over the eight thermocouple positions in the base plate to prevent burn-out of the thermocouples. The test was begun at 1330 hours and the maximum temperature reached at 1553 hours.

TABLE I

MATRIX FOR SOLAR FIRED TILE TEST CONDITIONS

Time (min)	Temperature 1080° C	1100° C	1120° C
10	(*)	(3)	(9)
20	(10)	(11)	(8)
30	(7)	(6)	(5)

Table II shows maximum temperature reached at 1553 hours according to thermocouple positions in the base plate.

TABLE II

INDICATED TEMPERATURE VARIATION FOR
TEST RUN NUMBER I

Location	Temperature (°C)
N	735
NE	746
E	761
SE	707
S	729
SW	751
W	800
NW	751

Insolation 576 W/m^2.

Attempts were made to control temperature by raising the cavity off the base. Two turns of the crank which raised the cavity about one inch dropped the temperature approximately 50 degrees. The cavity was then raised as high as possible to be certain that stray radiation would not become a problem.

2. Test Firing Number 2, 1 November 1981

This test utilized a cone plaque on the south side as shown in Figure 6. Tile sections in the north, south, east and southwest directions were placed on trivets and the remaining tile on ceramic fiber pads. Unglazed tile in the southeast and southwest corners were instrumented with thermocouples. Unfortunately, the tile on the thermocouple in the southwest corner failed to function.

The thermocouple embedded in the unglazed tile in the southeast corner was used to control this firing. Control of temperature was initially attempted by raising the cavity, however, this caused rapid fluctuations in temperature due to wind blowing across the tiles. This resulted in thermal shock of the tile section in the southwest corner as can be seen in Figure 6.

The shutters were opened at 1251 and the temperature reached 1125^{o} C at 1303. At this point the cavity was raised 3/4 inch. The computer updated temperatures each ten seconds rather than continuously. Temperature peaked at 1137^{o} C at 13:03:20 and then fluctuated wildly over the next several updates as shown in Table III.

After lowering the cavity the shutters were closed and the tile temperature stabilized at 886^{o} C and then slowly cooled to 410^{o} C when the shutters were reopened at 1317:50. At this point the insolation had dropped from 750 W/m^2 to 470 W/m^2 due to some passing thin clouds. The insolation and

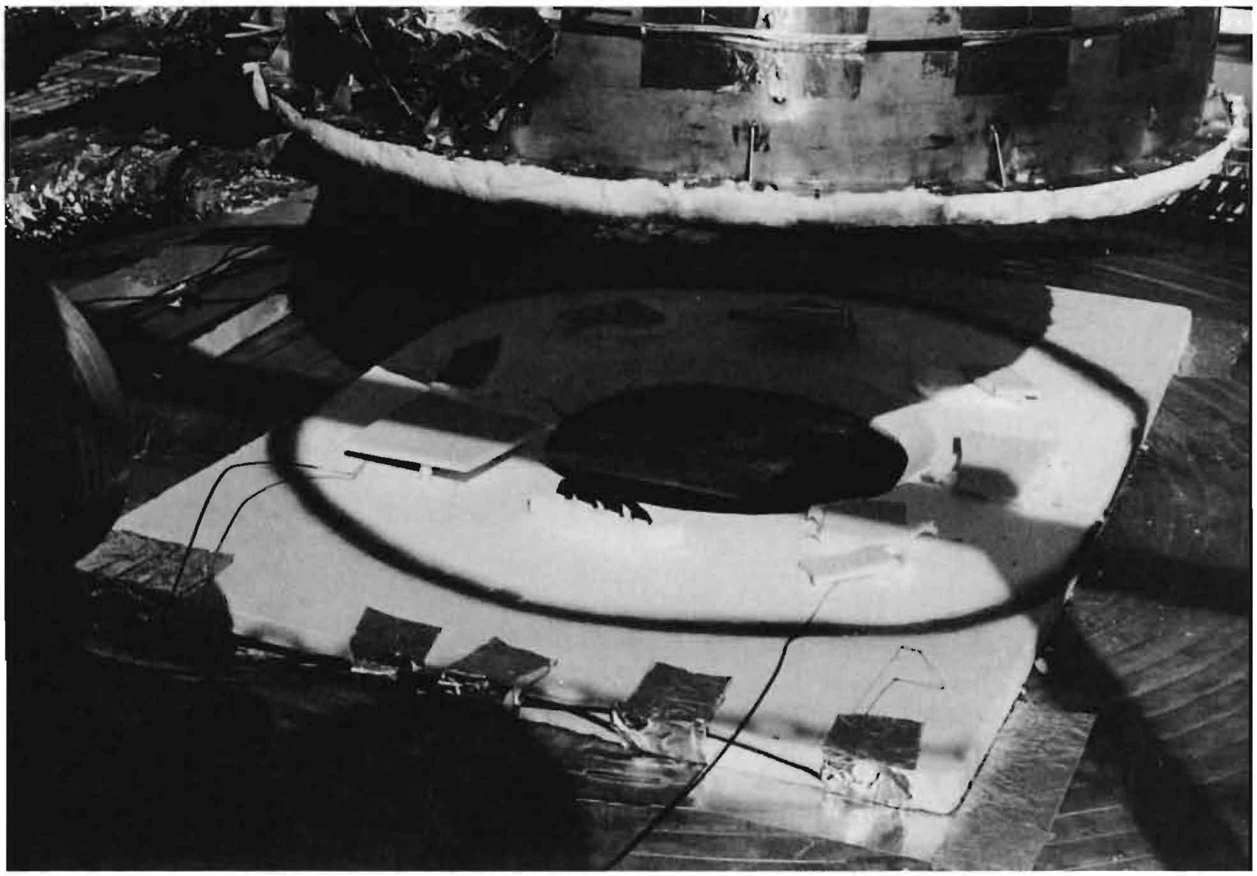

Figure 6. Tile Section and Pyrometric Cone Arrangement for Solar Test Firing Number 2 (View is to the North).

TABLE III

EFFECT ON TEMPERATURE OF RAISING CAVITY 3/4 INCH DURING RUN NUMBER 2

Time hr	(min)	(sec)	Thermocouple Read Out (°C)
13	03	20	1137
13	03	30	859
13	03	40	410
13	03	50	524
13	04	00	383
13	04	10	402
13	04	20	355
13	04	30	396
13	04	40	611
13	04	50	339
13	05	00	711
13	05	10	456
13	05	20	718
13	05	30	765
13	05	32	cavity lowered

temperature recovered slowly until the temperature reached 1101° C at 13:31:20. The temperature then fluctuated as shown in Figure 7 to a maximum of 1141° C. The shutters were closed at 13:41:40. Table IV gives time above stated temperature for this test.

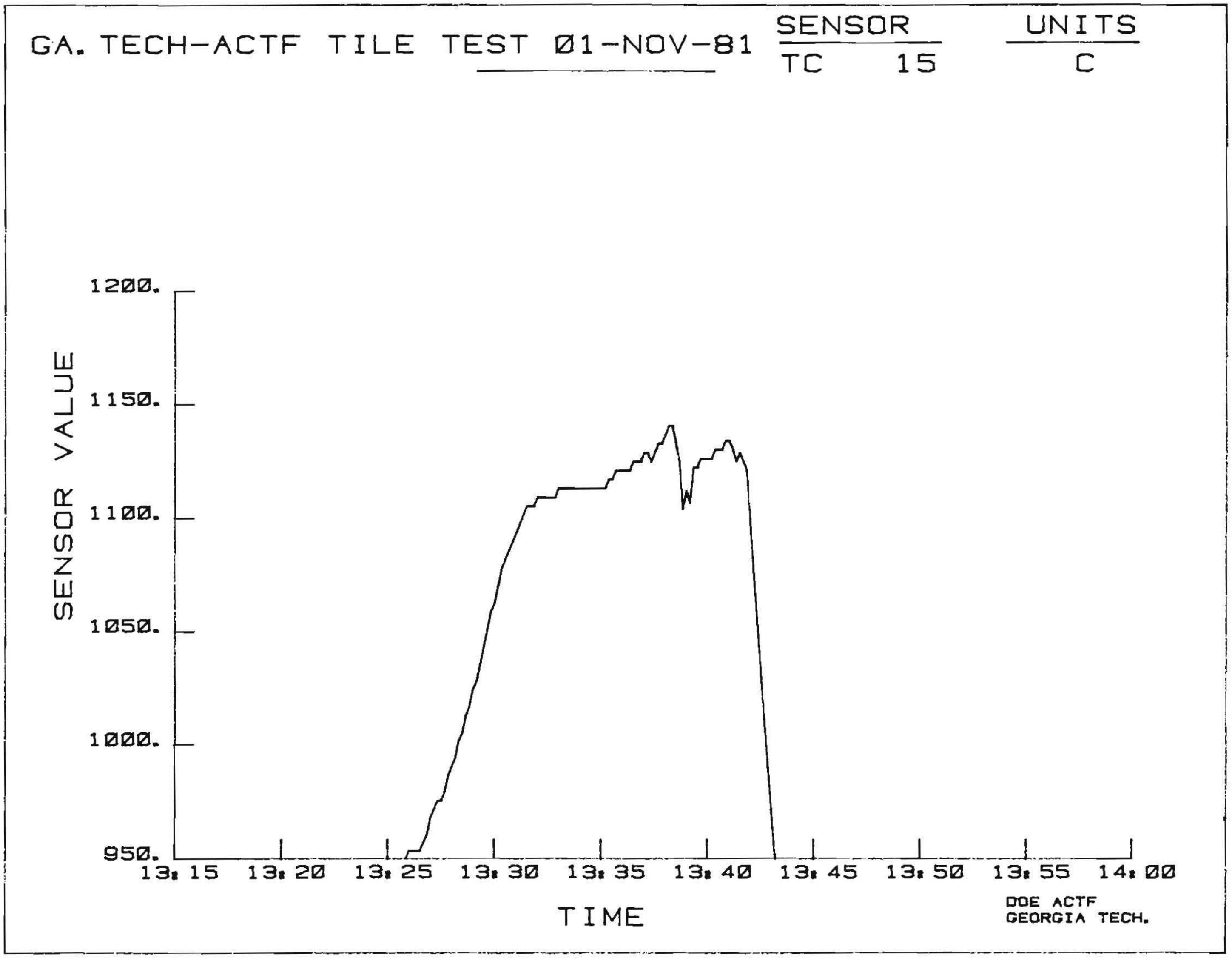

Figure 7. Temperature from Thermocouple in SW Tile, Firing Number 2

TABLE IV

TIME ABOVE SPECIFIED FIRING TEMPERATURE
FOR TEST NUMBER 2

Temperature (^{o}C)	Total Time (sec)
1140	20
1130	130
1120	380
1110	550
1100	680

Although the rate of temperature rise was much faster than that designated for large cones, the cones gave a resonable estimate of temperature since cone 01 was completely down. The temperature rating for cone 01 heated at 150^{0} C per hour is 1137^{0} C since these tile were heated at approximately 30 times this rate it can be assumed that the dark cones achieved temperatures in excess of 1150^{0} F.

3. Test Firing Number 3, 1 November 1981

This was the first fully instrumented test with thermocouples on all samples and using a mixture of glazed and unglazed tiles. Some tiles were placed on three point ceramic trivets which elevated the tile 3/8 inch above the base. Others were placed on 1/2-inch thick ceramic fiber pads and others were placed directly on the 3000 degree fiberboard base. Table V shows the type of tile, its location, and type of setting.

TABLE V

TILE POSITIONS FOR TEST NUMBER 3

Location	Tile Type	Setting	TC/No.
S	Unglazed	On Base	15
SW	Glazed	On Fiber Pad	16
W	Unglazed	On Base	18
NW	Unglazed	On Base	19
N	Glazed	Trivet	20
NE	Glazed	On Base	21
E	Unglazed	On Fiber Pad	22,23
SE	Unglazed	On Trivet	9

Due to intermittent cloudiness it was not possible to maintain the temperature continuously above 1100^{o} C for ten minutes as planned. A total of 7 minutes and 20 seconds above 1100^{o} C was achieved before the insolation dropped too low to maintain temperature. The thermocouple in the southeast unglazed tile was used for control. Time at temperature above 1100^{o} C for this position is shown in Table VI.

No temperature control was exercised on this run. The variable insolation controlled the temperature of the tiles in the cavity. The effect of emittance on the glazed and unglazed tiles can be clearly seen in Figures 8, 9, and 10. Figure 8 shows curves for the four tiles placed directly on the 3000 degree board base. Figure 9 shows the two tiles on trivets and the two tiles on ceramic fiber pads. Figure 10 shows two thermocouples in the East position tile. Thermocouple 22 was in the corner

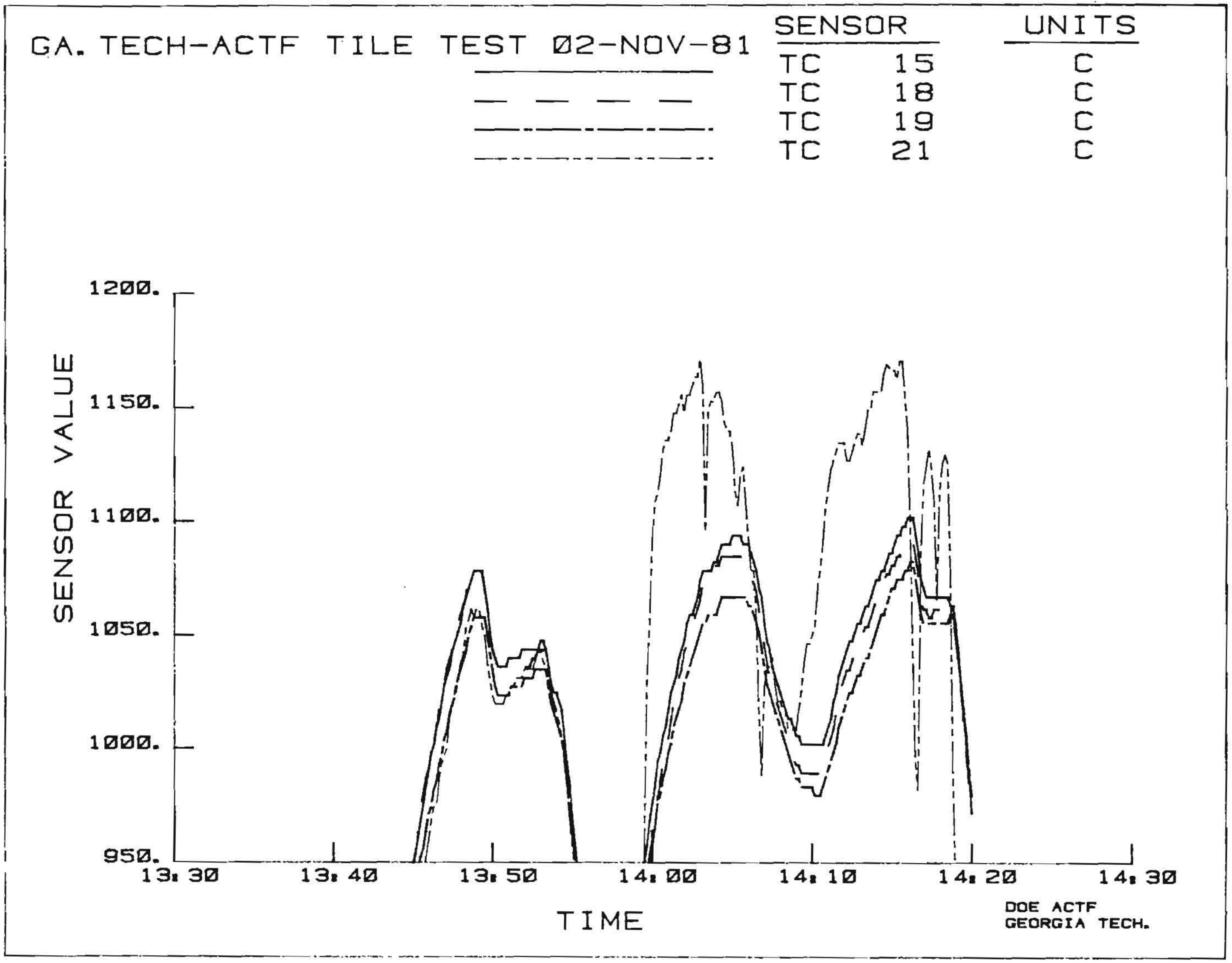

Figure 8. Temperature Variation in Four Tiles Placed Directly on 3000° Board Base, Firing Number 3 (Table V)

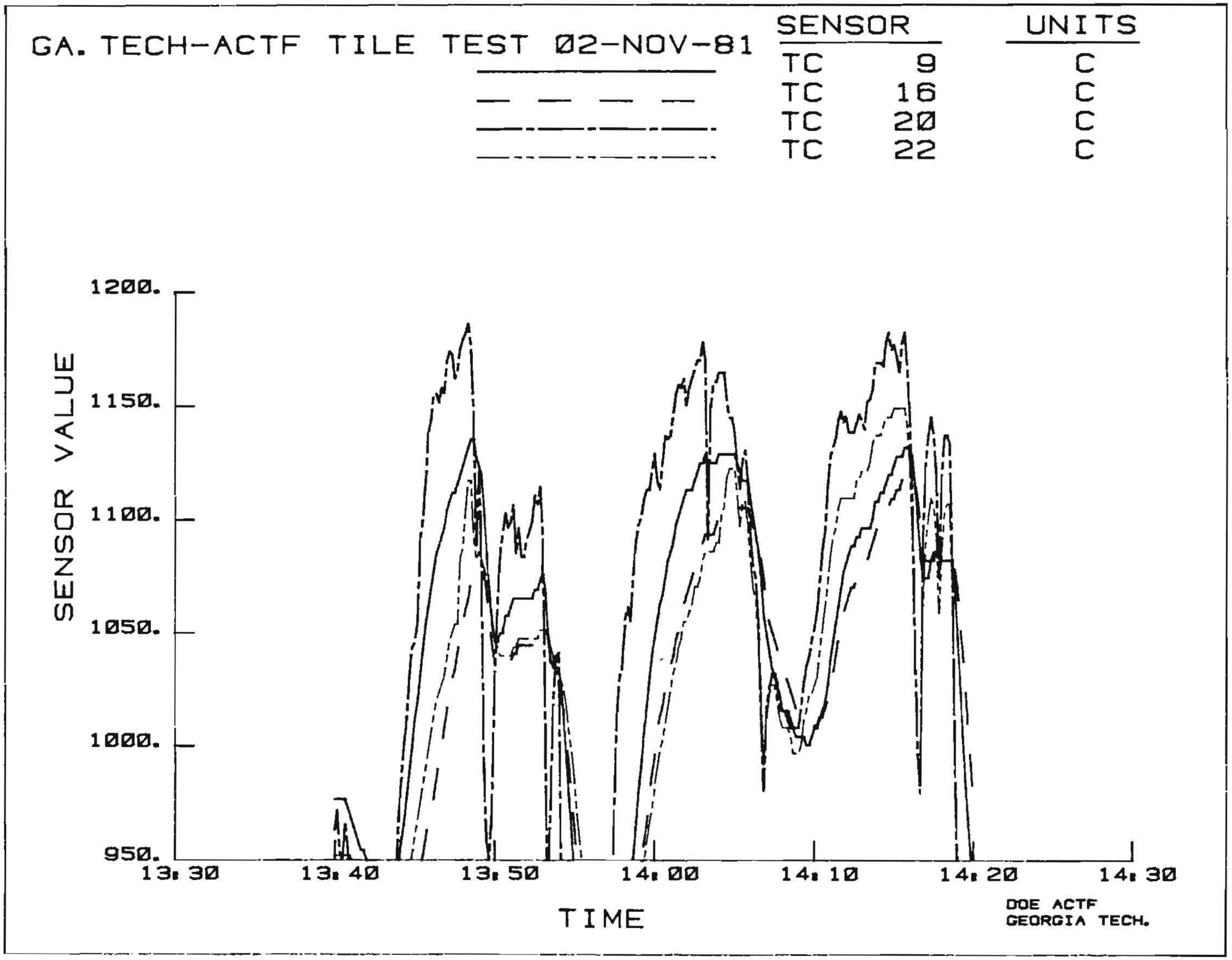

Figure 9. Temperature Variation for Glazed and Bisque Tiles on Trivets and on Ceramic Fiber Pads, Firing Number 3 (Table V)

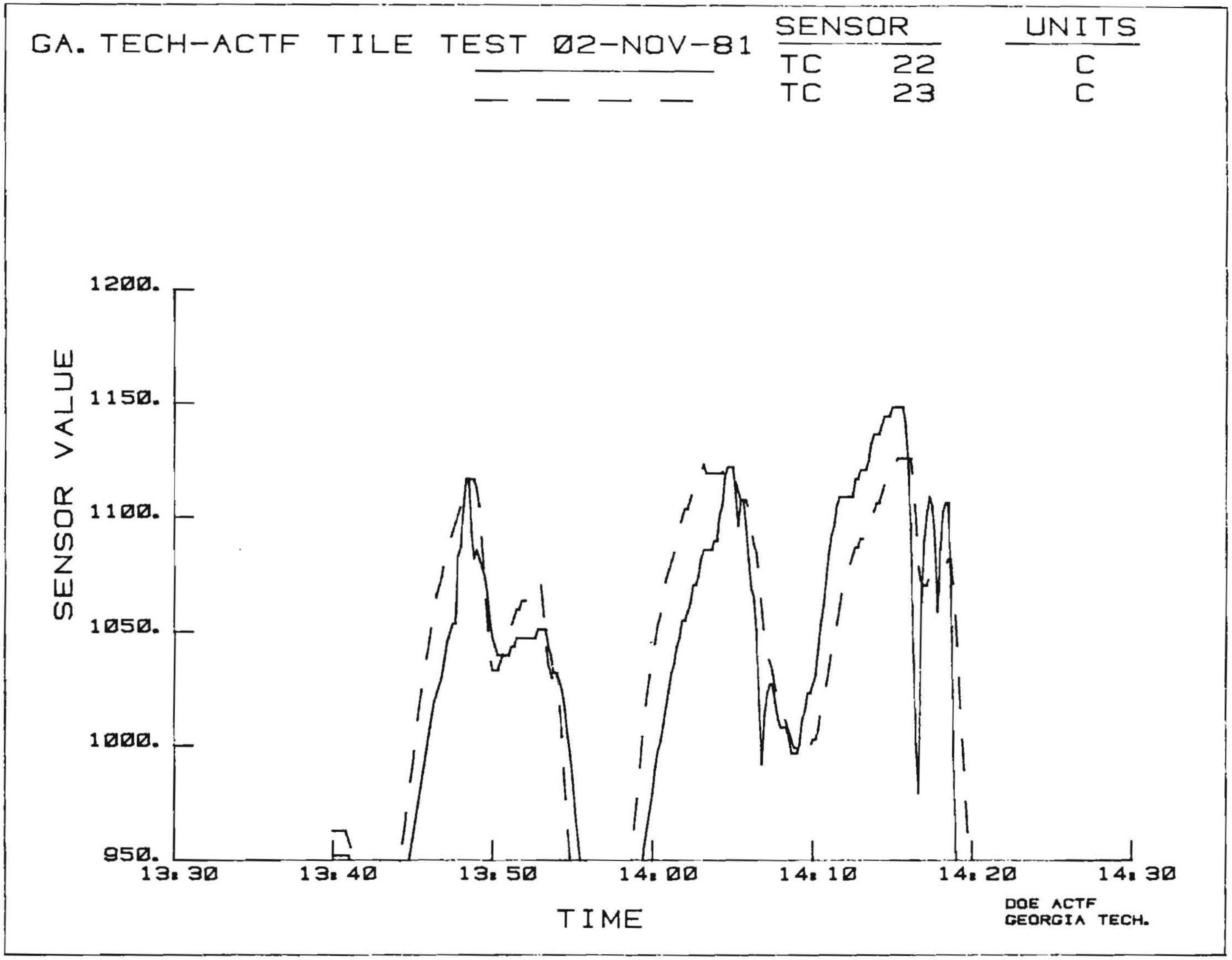

Figure 10. Variation of Temperature Within the Same Bisque Tile, Firing Number 3

TABLE VI

TIME AT TEMPERATURE FOR UNGLAZED TILE IN SOUTHEAST POSITION, FOR TEST NUMBER 3

Temperature	Time (sec)
1100	440
1110	360
1120	240
1130	30
1135*	20

*Maximum Recorded Temperature.

of the tile near the aperture and thermocouple 23 was in the geometric center of the tile.

Figure 11 shows the tiles after firing number 3. Unfortunately, the edge of the cavity caught some of the tiles when it was lowered after loading, preventing the glaze from maturing completely. This prompted a change in loading procedure. In the first three runs the tiles were placed in position from above the base. Beginning with run 4 the tiles were loaded through the aperture with the cavity down to be certain that the tiles were properly positioned.

4. Test Firing Number 4, 5 November 1981

Test number four was run on broken pieces of glazed tile with a thermocouple in the center of each. In addition, three cone plaques were placed on the base at the south, west and north positions. As can be seen

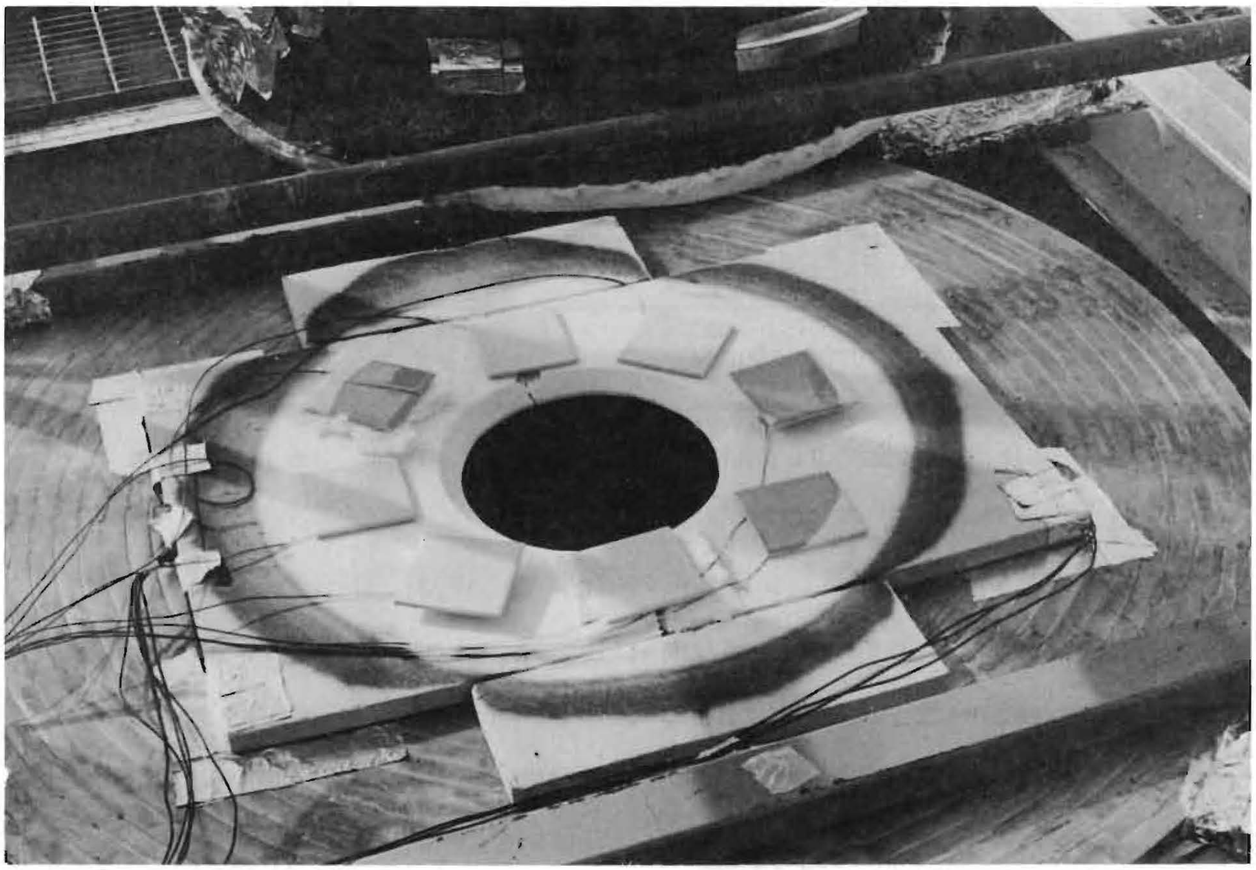

Figure 11. Tile Arrangement After Firing Number 3 (View to the West).

in Figure 12 all cones are down uniformly and Cone 1 has just started to bend indicating a temperature between 1137^{o} and 1154^{o} C at the 150^{o} C per hour heating rate. However, these tiles were subjected to heating rates in excess of 750^{o} C per minute on start up and because of very poor insolation conditions, Figure 13, there was considerable temperature fluctuation before and after reaching temperature. Control for this firing was on thermocouple 15 (south tile). Figure 14 is a plot of the south, southwest, west and northwest tiles. The other four tile positions were very similar on this run; the rate of rise was controlled for the first time by partially opening and closing the ACTF shutters below the focal zone. However, the thermal shock cracks as can be seen in Figure 12 indicate that the rate of rise was too rapid for these tile. The fact that the glaze flowed into the cracks indicate that thermal shock occurred upon heating rather than in cooling.

Table VII tabulates the time that the south tile position had an indicated temperature greater than 1100^{o} C.

5. Test Firing Number 5, 6 November 1981

This firing consisted of 4 glazed tile in the north, east, south and west positions and 4 unglazed tile in the northeast, southeast, southwest and northwest positions. This was the second test in which shutter control was used in an attempt to control rate of temperature rise. Initially this was anticipated to be a 1080^{o} C firing, but after one hour the maximum temperature reached was 1047^{o} C. A 69 minute cloudy period then developed which allowed the tile to cool back to 35^{o} C. During the first three minutes the indicated temperature increased 116^{o} C, 143^{o} C, and 138^{o} C

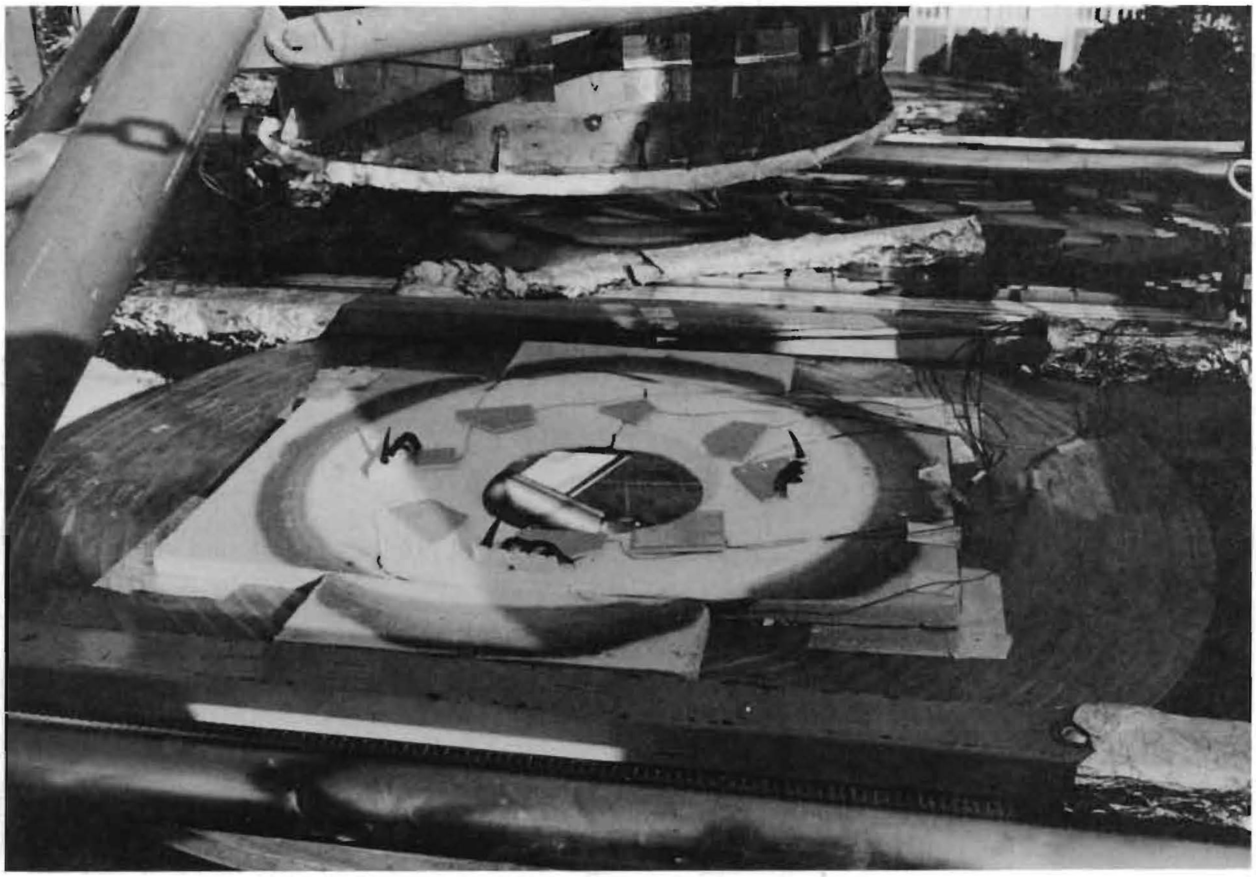

Figure 12. Tile Section and Pyrometric Cone Arrangement After Firing Number 4, (View is to the East).

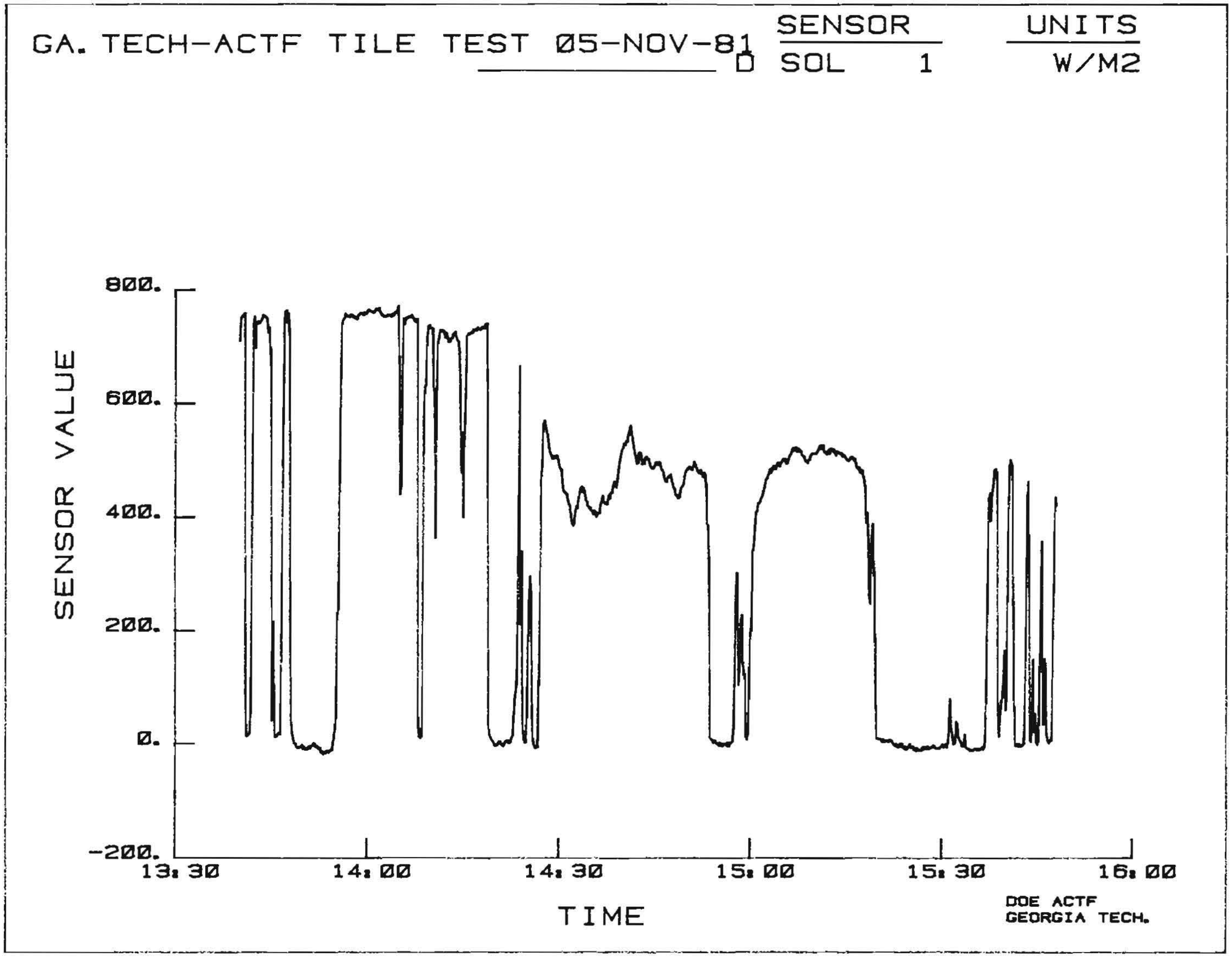

Figure 13. Insolation During Firing Number 4

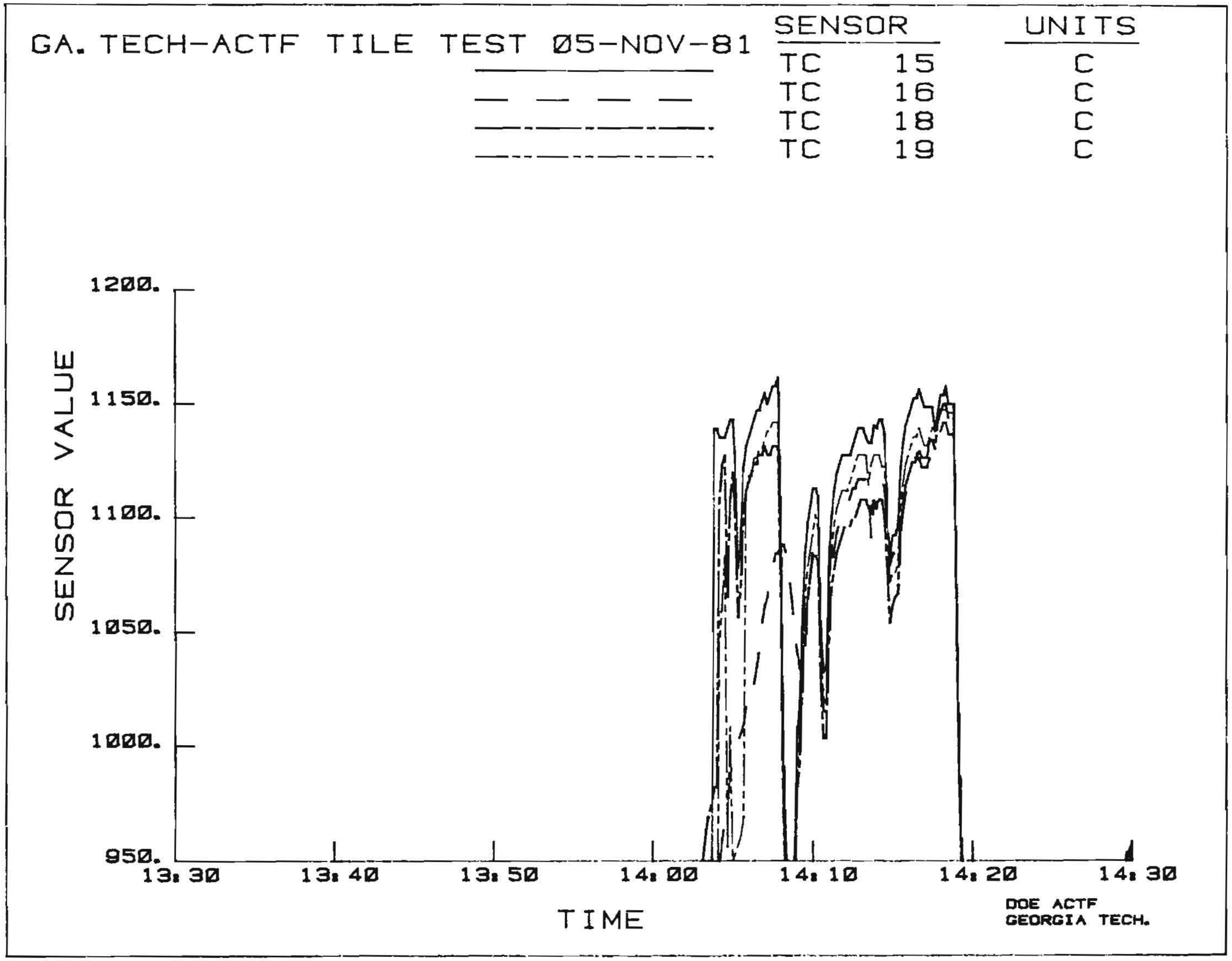

Figure 14. Temperature Variation for South, Southwest, West and Northwest Tiles in Firing Number 4

TABLE VII

TIME ABOVE TEMPERATURE FOR GLAZED TILE IN SOUTH POSITION, TEST NUMBER 4

Temperature (°C)	Time (sec)
1100	710
1110	660
1120	530
1130	340
1140	310
1150	130
1162*	10

*Maximum temperature.

respectively. This undoubtedly caused thermal shock cracking of all the glazed tiles and one of the unglazed tiles. The unglazed tile in the southeast position was used for control. Time at temperature above 1100° C is shown in Table VIII.

The minimum temperature during the 30 minute timing period was 1105° C for 30 seconds and the maximum was 1151°C for 20 seconds. This was the first firing with relatively close temperature control. Figure 15 clearly shows the effect of emittance for the two glazed and two unglazed tile in the south and west and in the southwest and northwest positions, respectively.

6. Test Firing Number 6, 6 November 1981

This firing was planned to control at 1100° C for 30 minutes. The tiles were arranged as shown in Table IX.

TABLE VIII

TIME ABOVE TEMPERATURE FOR GLAZED TILE IN WEST POSITION, TEST NUMBER 5

Temperature (^oC)	Time (sec)
1100	1920
1110	1660
1125	1240
1130	260
1140	40
1151*	20

*Maximum temperature.

TABLE IX

TILE POSITIONS FOR TEST NUMBER 6

Location	Tile Type	TC/No.
S	Unglazed	15
SW	Glazed	16
W	Glazed (Partial Tile)	18
NW	Unglazed (Partial Tile)	19
N	Glazed (Partial Tile)	20
NE	Unglazed	21
E	Glazed	22
SE	Unglazed	23

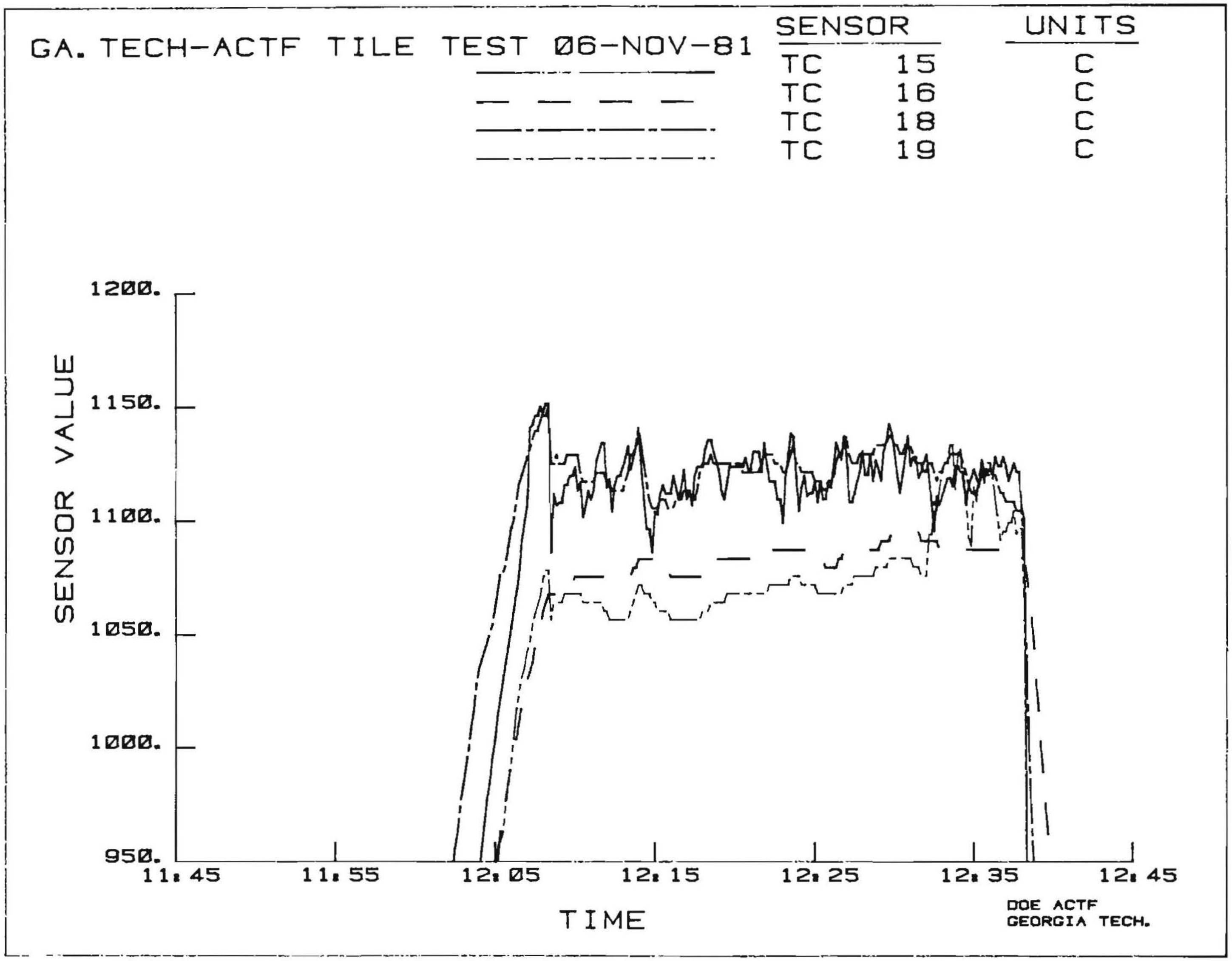

Figure 15. Temperature Variation for Glazed and Unglazed Tile in Firing Number 5, (TC 15 and 18 Glazed Tile; TC 16 and 19 Bisque Tile)

Control for this test was on the unglazed south tile. Indicated temperature increases in this unglazed tile per minute for the first three minutes were 197^{o} C, 232^{o} C, and 217^{o} C respectively. Indicated temperature increases for the first three minutes for the glazed tile next to it (SW position) were 80^{o} C, 87^{o} C, and 126^{o} C respectively. The effect of emittance and absorption are clearly seen from these data, however, no thermal shock cracking was present on any of these tiles after firing.

Since the attempt of this firing was to center temperature about 1100^{o} C total time at temperature for this firing is calculated beginning with 1080^{o} C in Table X.

TABLE X

TIME ABOVE TEMPERATURE FOR UNGLAZED TILE
IN SOUTH POSITION, TEST NUMBER 6

Temperature (^{o}C)	Time (sec)
1080	1590
1090	910
1100	680
1110	300
1120	20

Maximum temperature 1126^{o} C.

Temperature varied in the 20 minute firing from 1063 to 1126^{o} C. It was not possible to hold closer without damaging the ACTF shutters by overheating them. Temperature plots for the south and three west side tiles are shown in Figure 16.

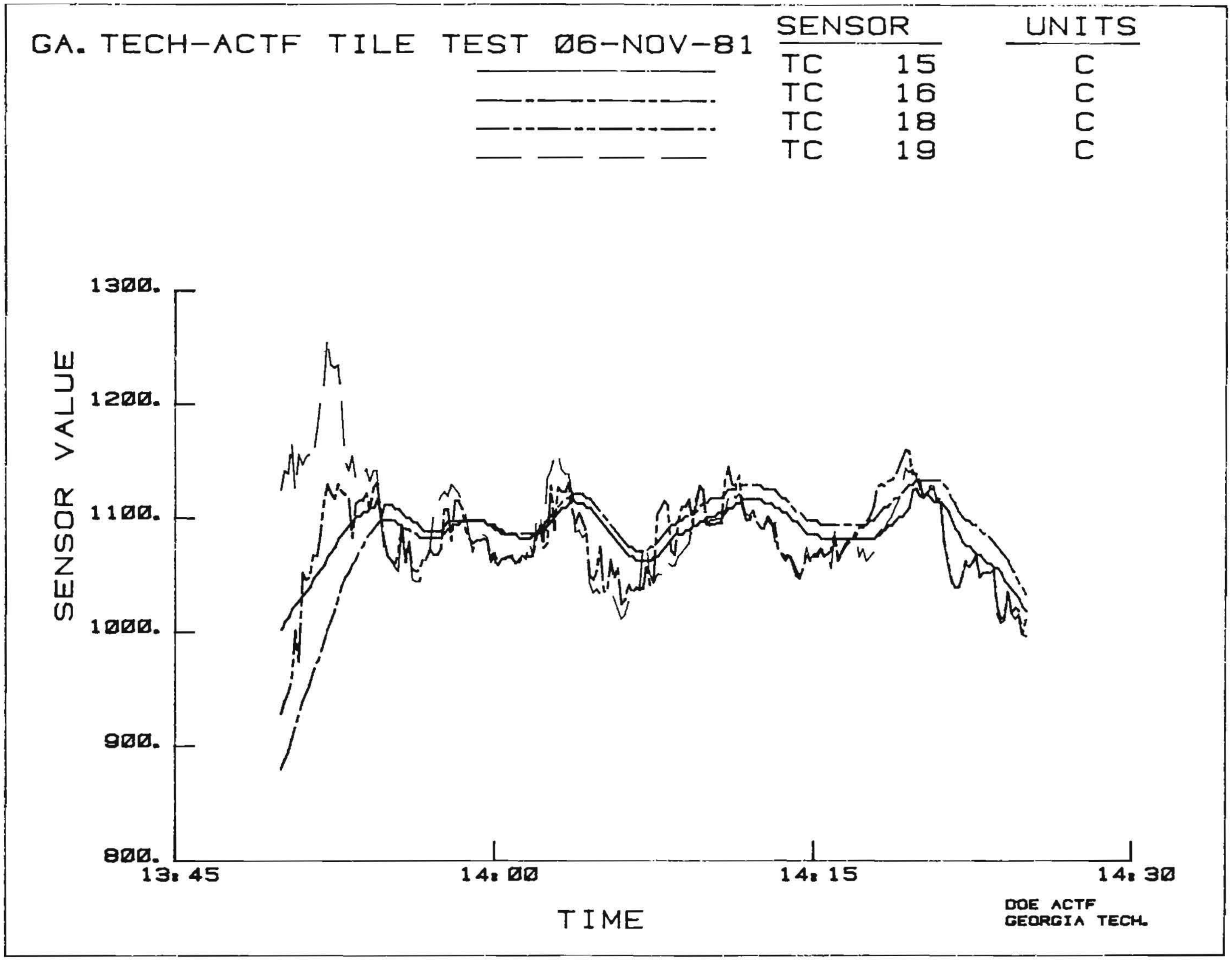

Figure 16. Temperature Variation for the South and Three West Side Tiles, Firing No. 6 (Table IX)

7. Test Firing Number 7, 7 November 1981

This test was conducted on the first cloudless day of the series. The intent of this test was to fire 30 minutes at a temperature of 1080^{0} C. The tiles for this test were arranged as shown in Table XI.

TABLE XI

TILE POSITIONS FOR TEST NUMBER 7

Location	Tile Type	TC/No.
S	Glazed	15
SW	Unglazed	16
W	Glazed (Partial tile)	18
NW	Glazed (Partial tile)	19
N	Unglazed (Partial tile)	20
NE	Glazed (Partial tile)	21
E	Unglazed	22
SE	Glazed	23

For this test control was on the east unglazed tile. Temperature increases for this tile per minute for the first three minutes were 25^{0} C, 99^{0} C, and 120^{0} C respectively. Temperature increases for the glazed tile in the northeast position were, 14^{0} C, 48^{0} C, and 68^{0} C respectively. However, even though the rates of increase were smaller than the previous test the glazed tiles cracked due to thermal shock.

Table XII gives total times above 1070 (minimum temperature during this firing).

TABLE XII

TIME ABOVE TEMPERATURE FOR UNGLAZED TILE IN EAST POSITION, TEST NUMBER 7

Temperature (^{o}C)	Time (sec)
1070	1800
1080	1050
1090	250
1097*	110

*Maximum temperature.

Control of temperature for this test was from -10^{o} (1070^{o} C) to + 17^{o} (1097^{o} C). Curves for temperatures of tiles on the northwest and for the three east positions are shown in Figure 17.

8. Test Firing Number 8, 7 November 1981

So as to have some samples not damaged by thermocouples, only part of these samples had thermocouples. The tile position and thermocouple locations are shown in Table XIII.

This firing was planned for 1120^{o} C for twenty minutes; control was on the center thermocouple in the southeast glazed tile. Increase in temperature per minute for the south unglazed tile and the southeast glazed tile for the first three minutes were respectively: 69^{o}, 149^{o}, 148^{o}, and 18^{o}, 62^{o}, and 71^{o} C. These tiles did not thermal shock during firing.

Table XIV shows total time above temperature during the 20 minute firing time for these tile.

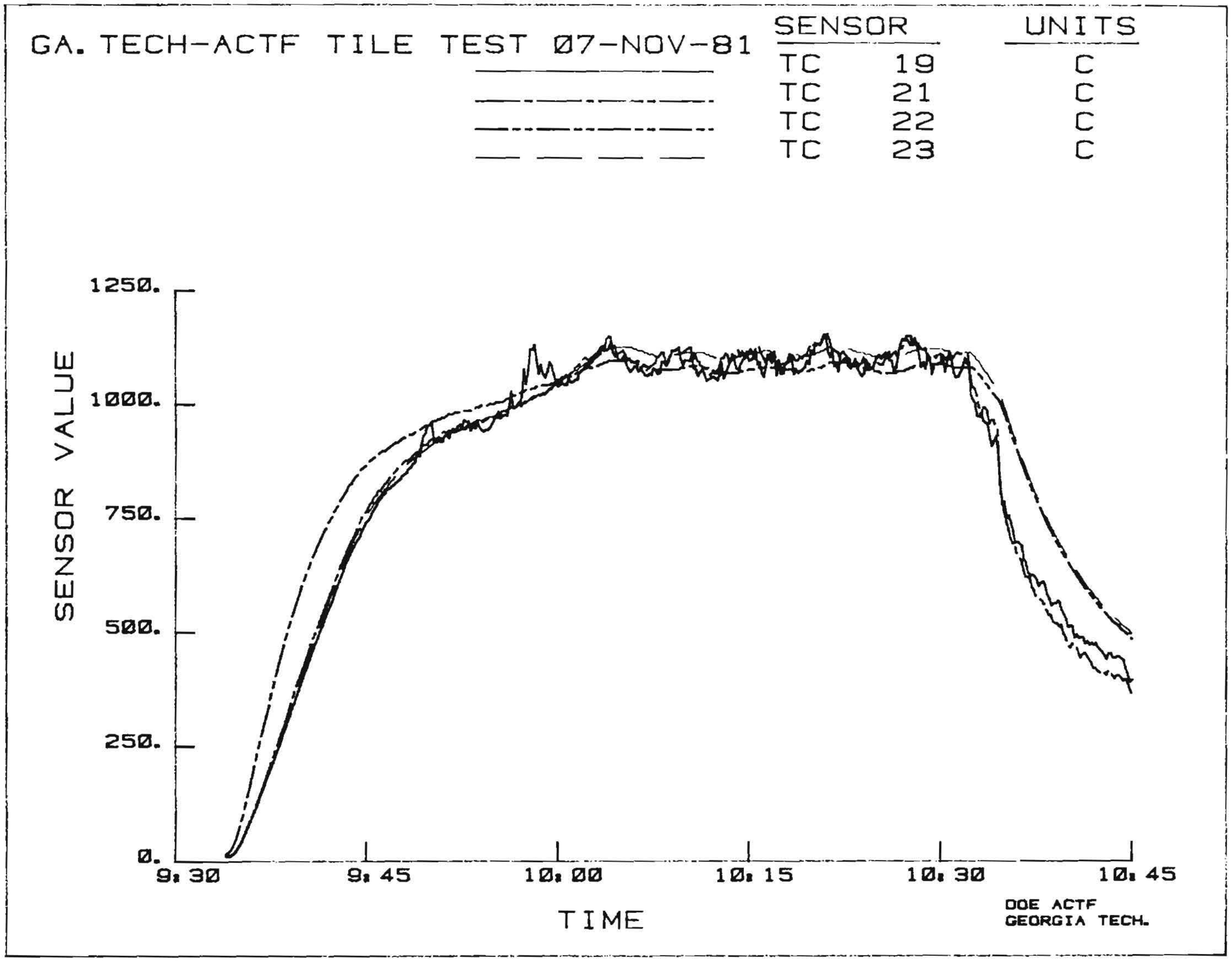

Figure 17. Temperature Variation for Northwest and Three East Positions in Firing Number 7 (Table XI)

TABLE XIII

TILE POSITIONS FOR TEST NUMBER 8

Location	Tile Type	TC/No.
S	Unglazed	15
SW	Glazed	None
W	Unglazed	None
NW	Glazed	19
N	Glazed	None
NE	Glazed (Broken)	None
E	Unglazed	20
SE	Glazed	21,22,23

TABLE XIV

TIME ABOVE TEMPERATURE FOR GLAZED TILE IN SOUTHEAST POSITION, TEST NUMBER 8

Temperature (^{o} C)	Time (sec)
1100	1200
1120	580
1130	140

Maximum temperature 1135^{o} C.

Control of temperature during the firing period was from -19^{o} (1101^{o} C) to + 15^{o} (1135^{o} C).

Two curves are presented for temperature. Figure 18 shows the firing temperatures for the one glazed and two unglazed tiles. Again the difference in absorption of the glazed and unglazed tiles can be seen.

Figure 19 shows the temperature distribution across the glazed tile in the southeast position. The large gradient across the tile from the aperture side to the cavity wall side should be noted.

9. Test Firing Number 9, 7 November 1981

This test had only four of the eight tiles instrumented with thermocouples, two of each type had thermocouples attached. The positions of the tiles are shown in Table XV.

TABLE XV

TILE POSITIONS FOR TEST NUMBER 9

Location	Tile Type
S	Unglazed
SW	Glazed
W	Unglazed
NW	Glazed
N	Unglazed
NE	Unglazed
E	Glazed
SE	Glazed

Temperature was controlled on the southeast glazed sample. Temperature increase on adjacent unglazed and glazed samples per minute for three minutes were respectively: 114^{o}, 148^{o}, 148^{o}, and 43^{o} 65^{o}, and 67^{o} C.

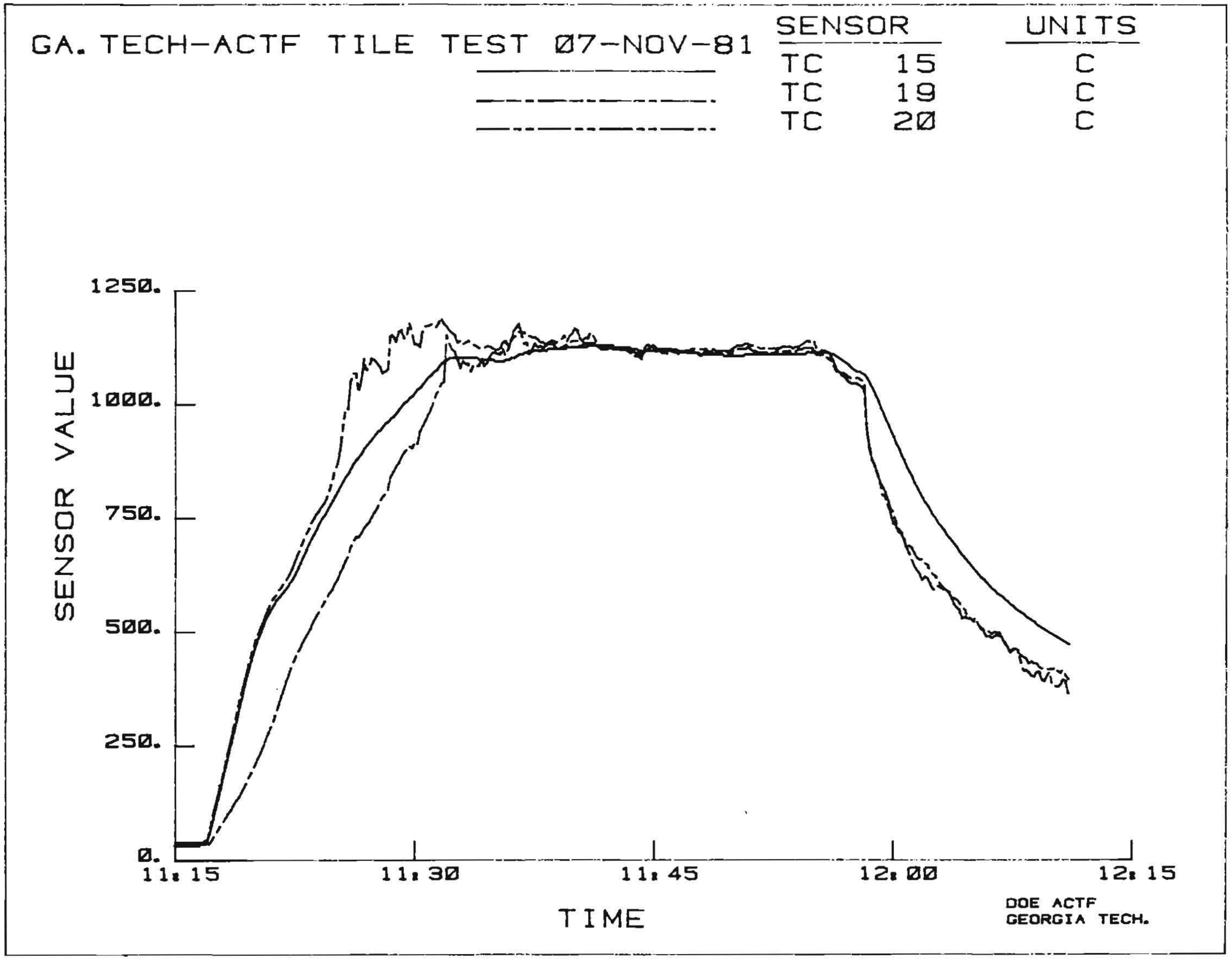

Figure 18. Temperature Variation for One Glazed and Two Unglazed Tiles in Firing Number 8 (Table XIII)

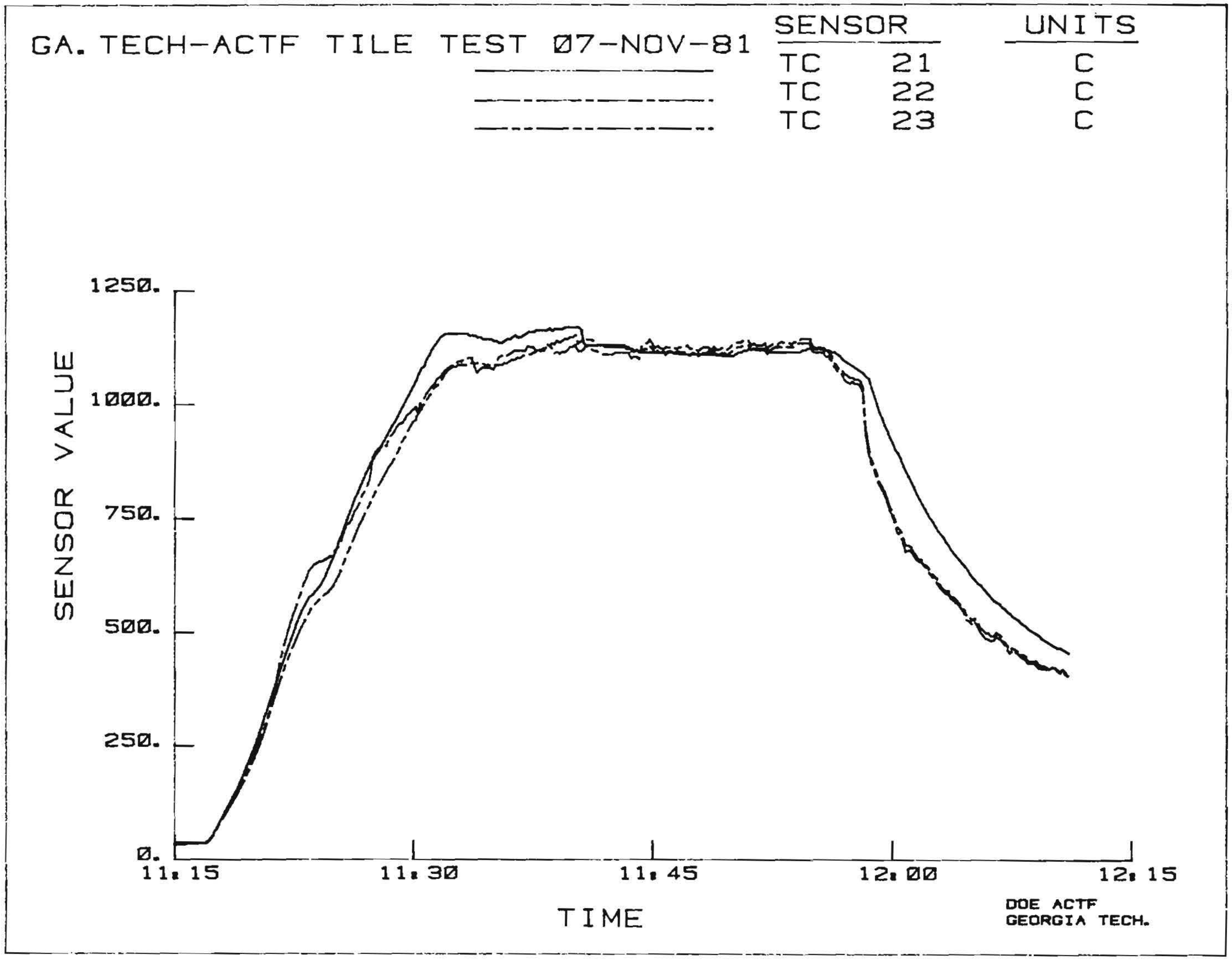

Figure 19. Temperature Variations in Southeast Glazed Tile, Firing Number 8

This run was designed for 1120° C for ten minutes, but temperatures behaved erratically as shown in Figure 20. A table of time above temperature beginning at 1100° C is shown in Table XVI.

TABLE XVI

TIME ABOVE TEMPERATURE FOR GLAZED TILE IN SOUTHEAST POSITION, TEST NUMBER 9

Temperature (°C)	Time (sec)
1100	560
1110	470
1120	380
1130	220
1140	110
1150	40
1160	20
1170	20

Maximum temperature 1176° C.

Control of temperature for this test was from -63° C (1057° C) to +56° C (1176° C). Although this was the greatest indicated variation in firing temperature, tile from this firing made the closest color match with the commercial tile.

10. Test Firing Number 10, 7 November 1981

This test utilized only three thermocouples all on glazed tile. The three thermocouples were on the southwest, northwest and southeast tile.

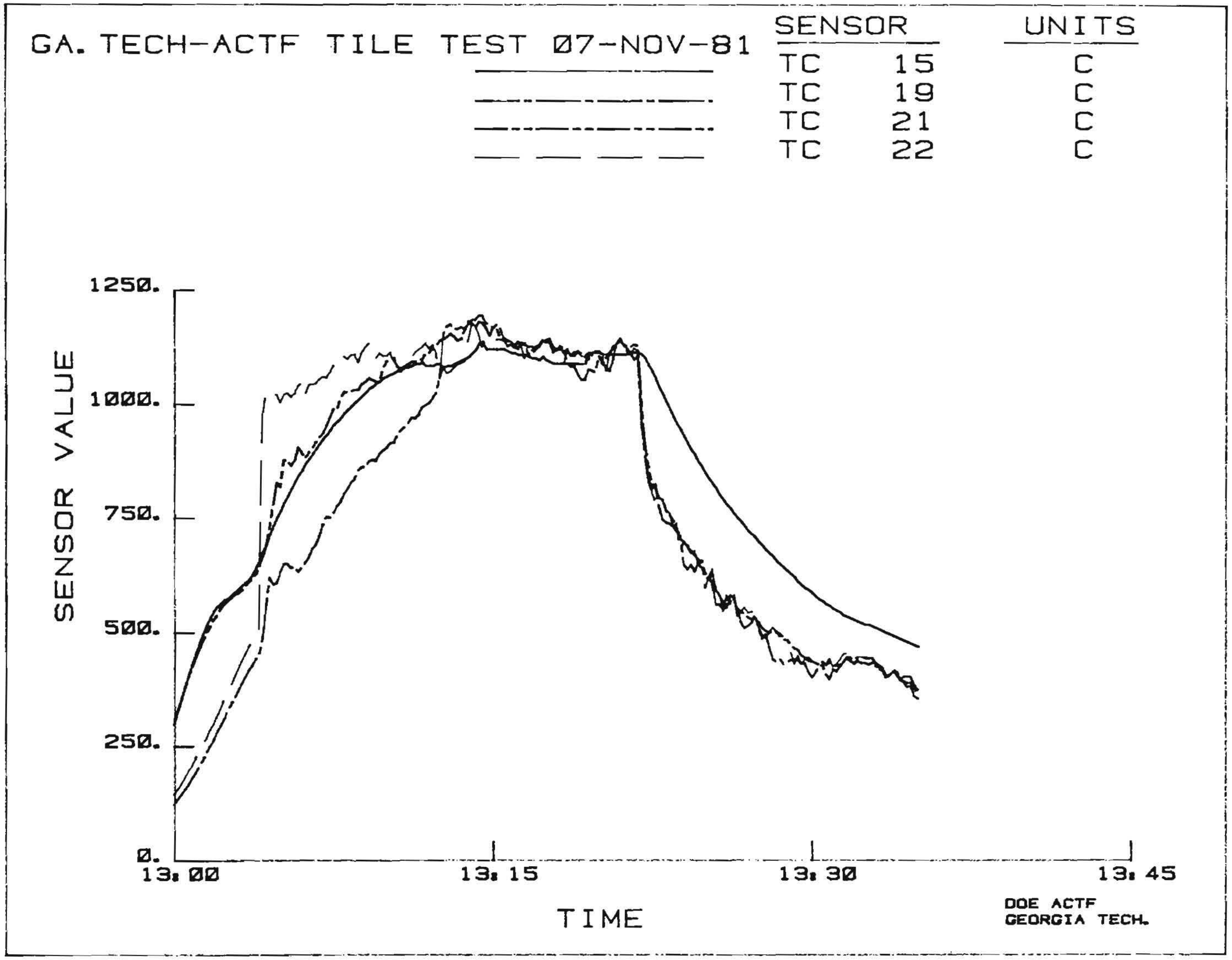

Figure 20. Temperature Variations for 4 Instrumented Tiles, Two Glazed and Two Bisque in Firing Number 9

Control for this firing was on the southeast tile. Unglazed tile were located in the north, west, and south positions. All other positions had glazed tile.

The intended firing conditions for these tile were 1080^{o} C for 20 minutes. The rate of temperature increase per minute for the first three minutes was: 45^{o}, 77^{o}, and 96^{o} C respectively. There was no indication of thermal shock after firing. The tiles reached firing temperature in 15 minutes and were held at temperature for 20 minutes before slow closing of the shutters to cool down the tiles at a rate similar to the heat up rate.

Table XVII shows the time at temperatures above 1065^{o} C for the 20 minute firing.

TABLE XVII

TIME ABOVE TEMPERATURE FOR GLAZED TILE IN SOUTHEAST POSITION, TEST NUMBER 10

Temperature (^{o}C)	Time (sec)
1065	1240
1075	780
1085	480
1095	190
1105*	50

*Maximum temperature.

Control temperature during this firing period varied from -14^{0} C (1066) to $+25^{0}$ C (1105). This firing produced good tile but they were slightly greener in color than the commercial fired bodies. The uniformity of firing of the glazed tile can be seen for the three instrumented tiles in Figure 21.

11. Test Firing Numbers 11 and 12, 7 December 1981

These test firings were conducted to determine the temperatures across the tiles from the side nearest the aperture to the side nearest the cavity wall. This was done by mounting three thermocouples in a tile, one in the geometric center and two in line with the center thermocouple located one inch on either side. Table XVIII gives the type of tile, position and thermocouples located on each for both firings 11 and 12.

TABLE XVIII

TILE POSITIONS FOR TEST NUMBERS 11 AND 12

Location	Tile Type	Thermocouple Numbers
SW	Glazed	11, 12, 13
W	Glazed	14
NW*	Unglazed	15, 16, 17
N	Unglazed	None
NE*	Glazed	18, 19, 20
NE	Unglazed	21
SE	Glazed	22
S	Unglazed	None

*Tile Positions Switched for Run 12.

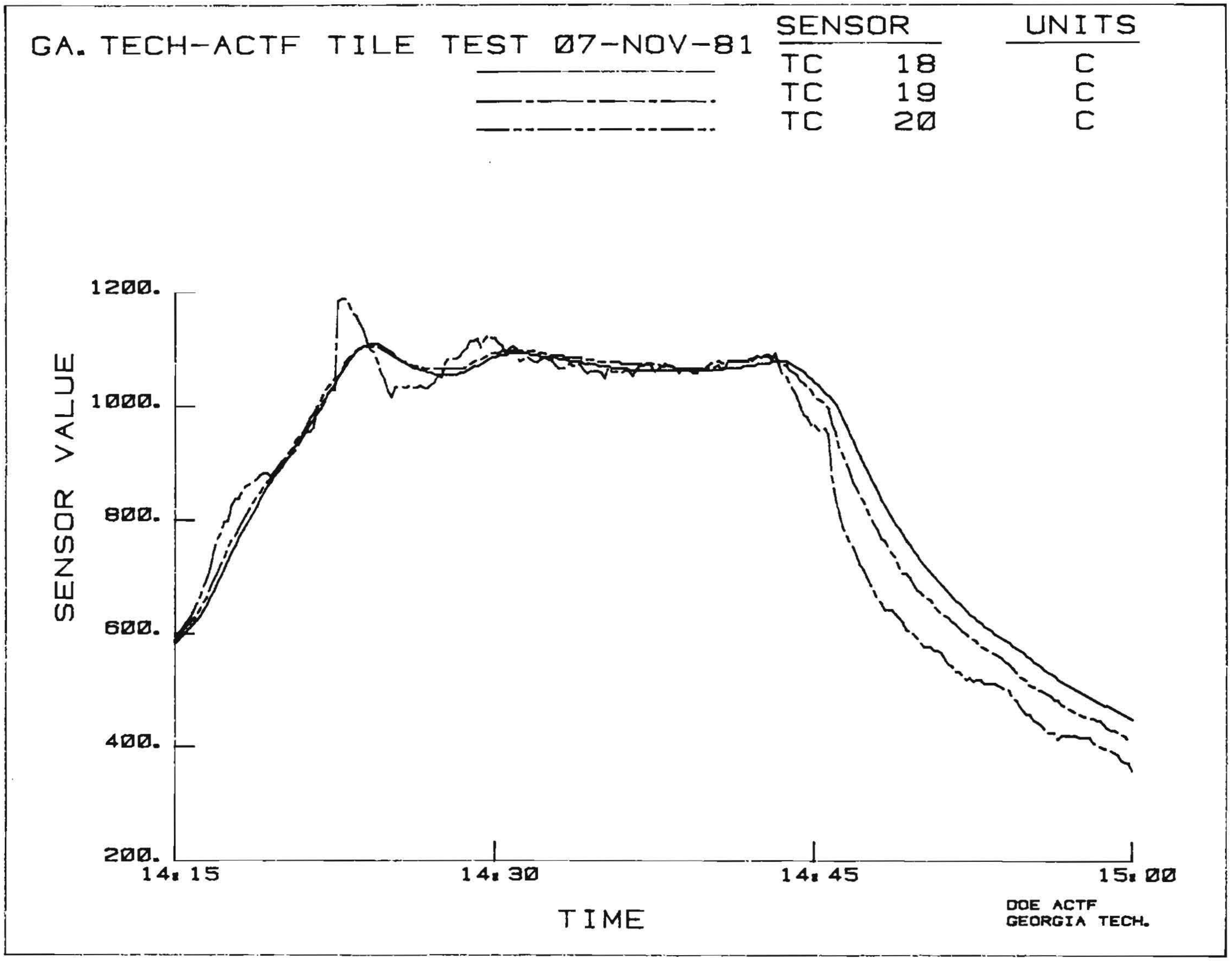

Figure 21. Temperature Variation of Glazed Tile in Southwest, Northwest and Northeast Positions, Firing Number 10

Temperature control for firing number 11 was 1100^{0} C for 20 minutes. Control was the center thermocouple on the glazed tile in the northeast position. A definite attempt was made to hold the initial rate of temperature increase to less than 50^{0} C per minute. This was accomplished with the glazed control tile with per minute temperature increases of 27^{0}, 40^{0}, and 35^{0} C for the first three minutes. The unglazed tile in the northwest position showed temperature increases of 54^{0}, 88^{0}, and 106^{0} C for the same period. There was, however, no evidence of thermal shock after firing. The tiles required 54 minutes to reach firing temperature due to a few small clouds. This run had less deviation from planned temperature control than any previous run.

Time above 1090^{0} C is shown in Table XIV.

TABLE XIV

TIME ABOVE TEMPERATURE FOR GLAZED TILE IN NORTHEAST POSITION, TEST NUMBER 11

Temperature (^{0}C)	Time (sec)
1090	1110
1100	300
1110	90
1120	40
1124*	10

*Maximum temperature.

Control was from -15^{0} (1085^{0} C) to $+24^{0}$ (1124^{0} C). Three sets of curves are presented in Figures 22, 23, and 24 for the two glazed tiles in

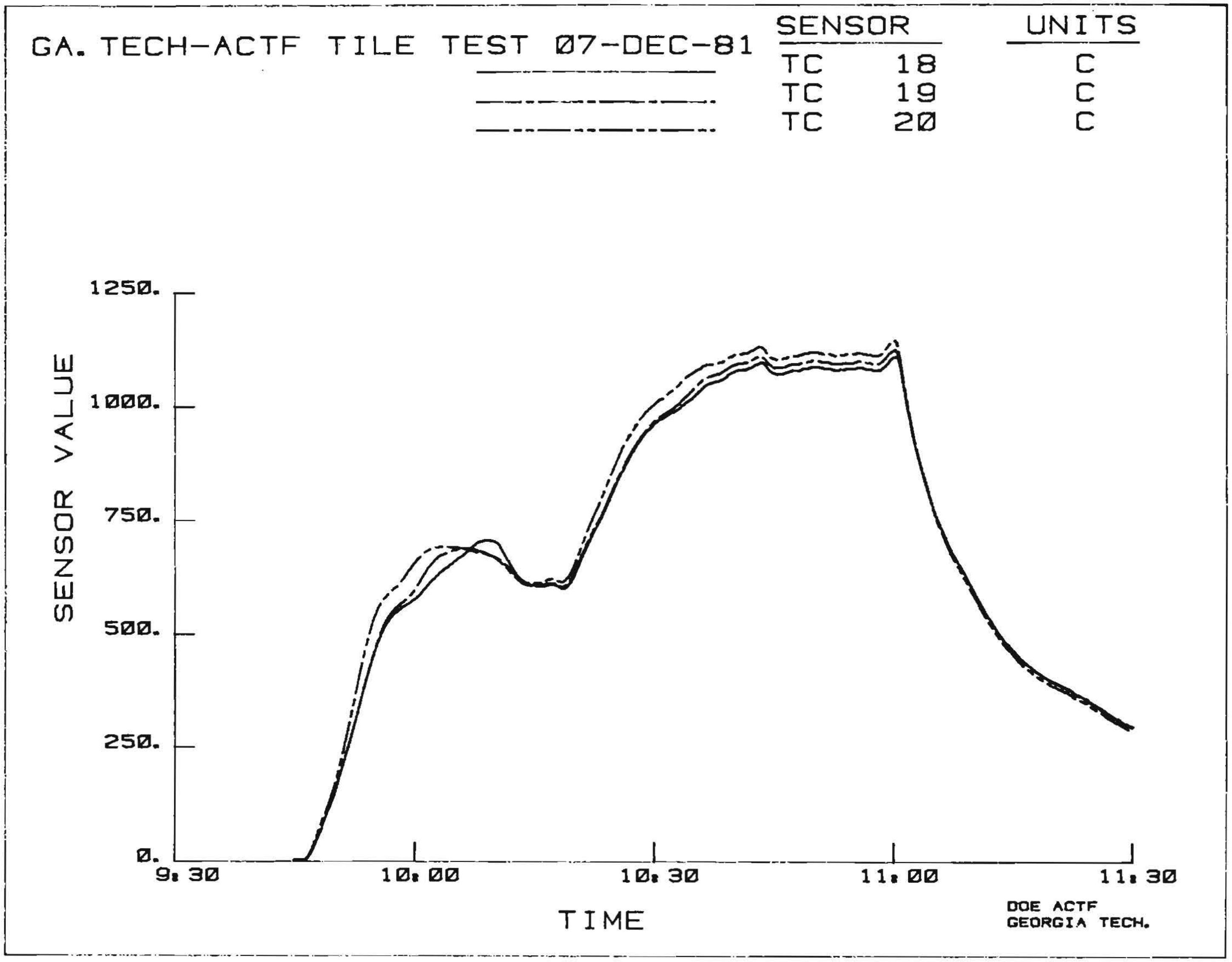

Figure 22. Temperature Variations in Glazed Tile in Northeast Position, Firing Number 11

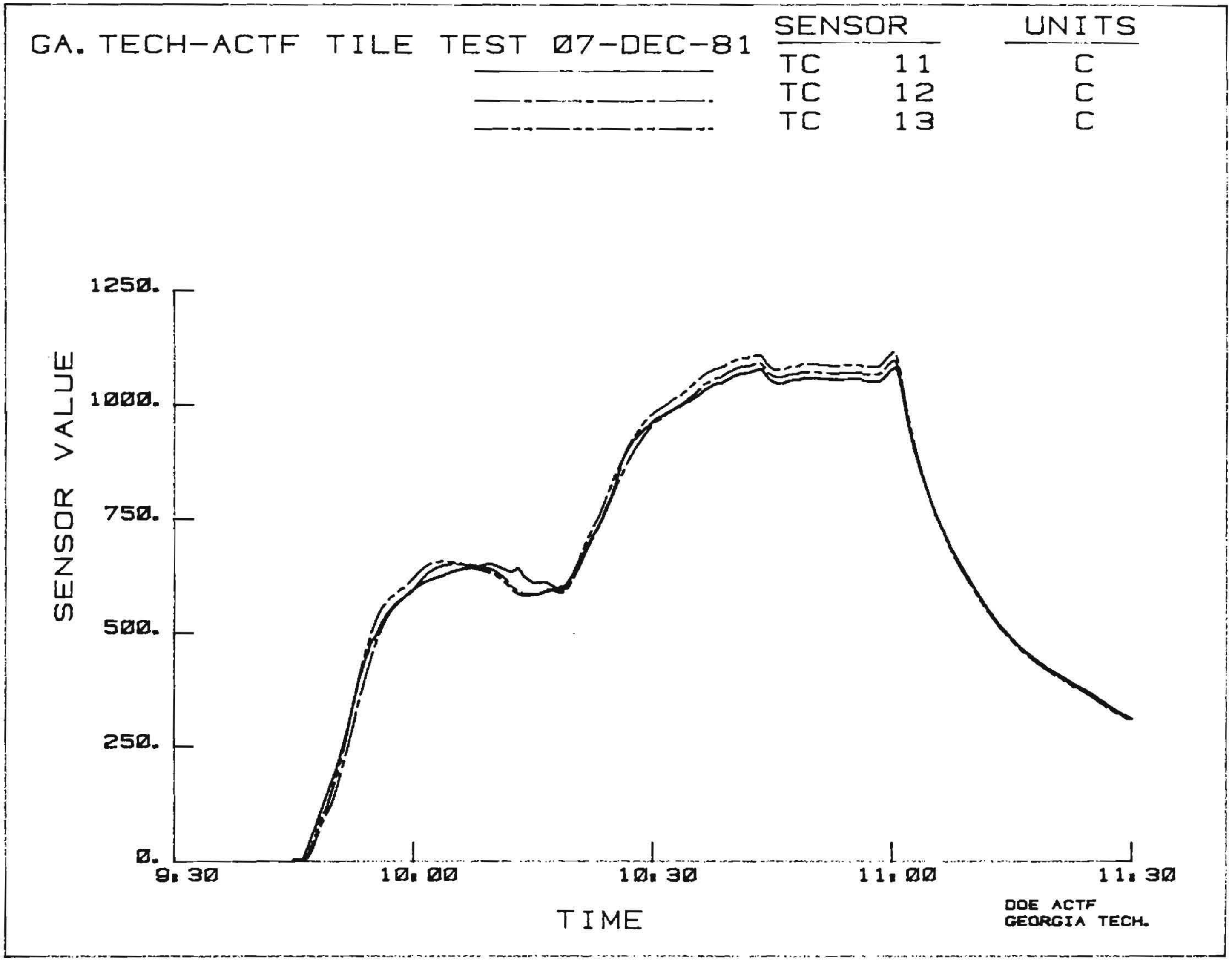

Figure 23. Temperature Variations in Glazed Tile in Southwest Position, Firing Number 11

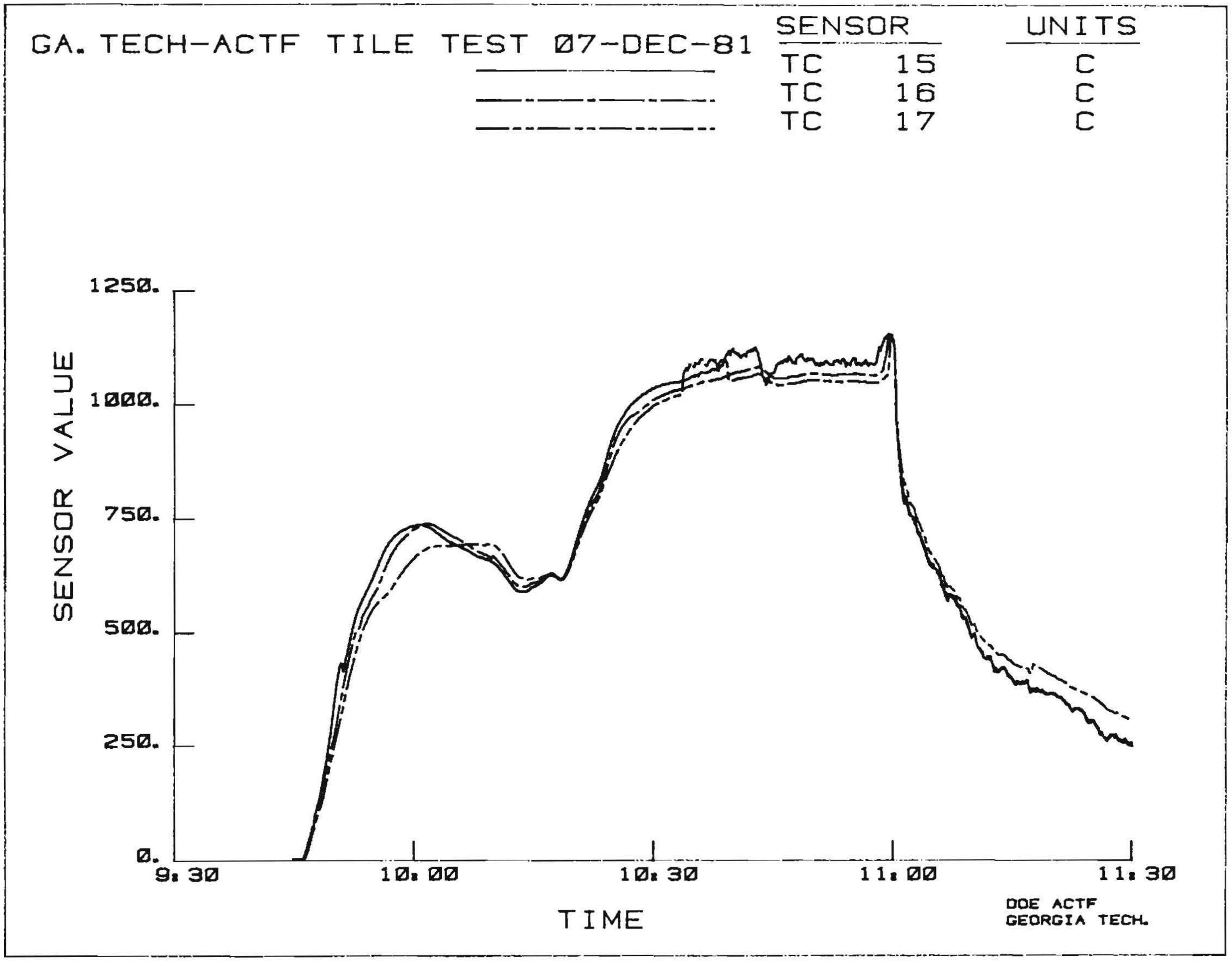

Figure 24. Temperature Variations in Bisque Tile in Northwest Position, Firing Number 11

the northeast and southwest positions and for the unglazed tile in the northwest position. The decrease in temperature from the aperture toward the cavity wall in each case is clearly shown.

Test number 12 was identical to number 11 except the planned firing temperature was 15 minutes at 1080^{0} C and the tiles in the northeast and northwest positions were switched so that control for firing 12 was on the unglazed tile in the northeast position. Rate of temperature increase for the first three minutes on the unglazed tile could not be held to less than 50^{0} C. Per minute rates were 132^{0}, 151^{0}, and 150^{0} C on the unglazed tile. The per minute rate of temperature increase for the glazed tile in the first three minutes of firing were: 46^{0}, 77^{0}, and 91^{0} C. However, even though the desired control was not achieved no thermal shock occurred. Time to reach firing temperature was 16 minutes. All thermocouple readings were erratic on this run so no table of times above temperature is given. Figures 25, 26, and 27 present data for the two glazed and one unglazed tile with three thermocouples each.

D. Evaluation of Unfired and Solar Fired Tiles

1. Elemental Content

The unfired tiles were examined by emission spectroscopy to provide a qualitative analysis of the elements present. A list of elements and relative strength of spectroscopic lines are shown in Table XV.

2. Volatile Content

The tile raw material was subjected to differential scanning calorimetry (DSC) and thermogravimetric analysis on a Perkin Elmer thermal

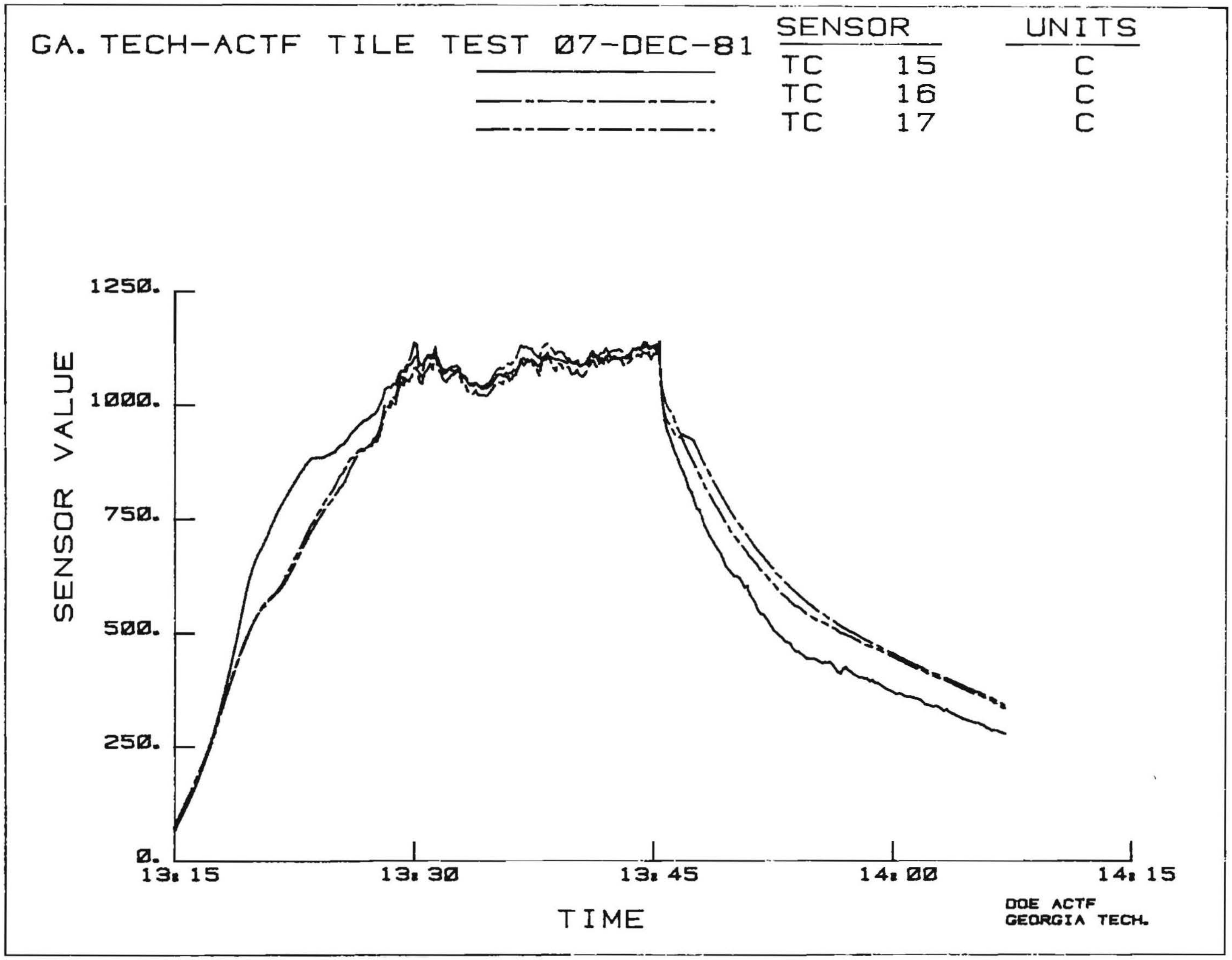

Figure 25. Temperature Variations in Glazed Tile in Northwest Position, Firing Number 12

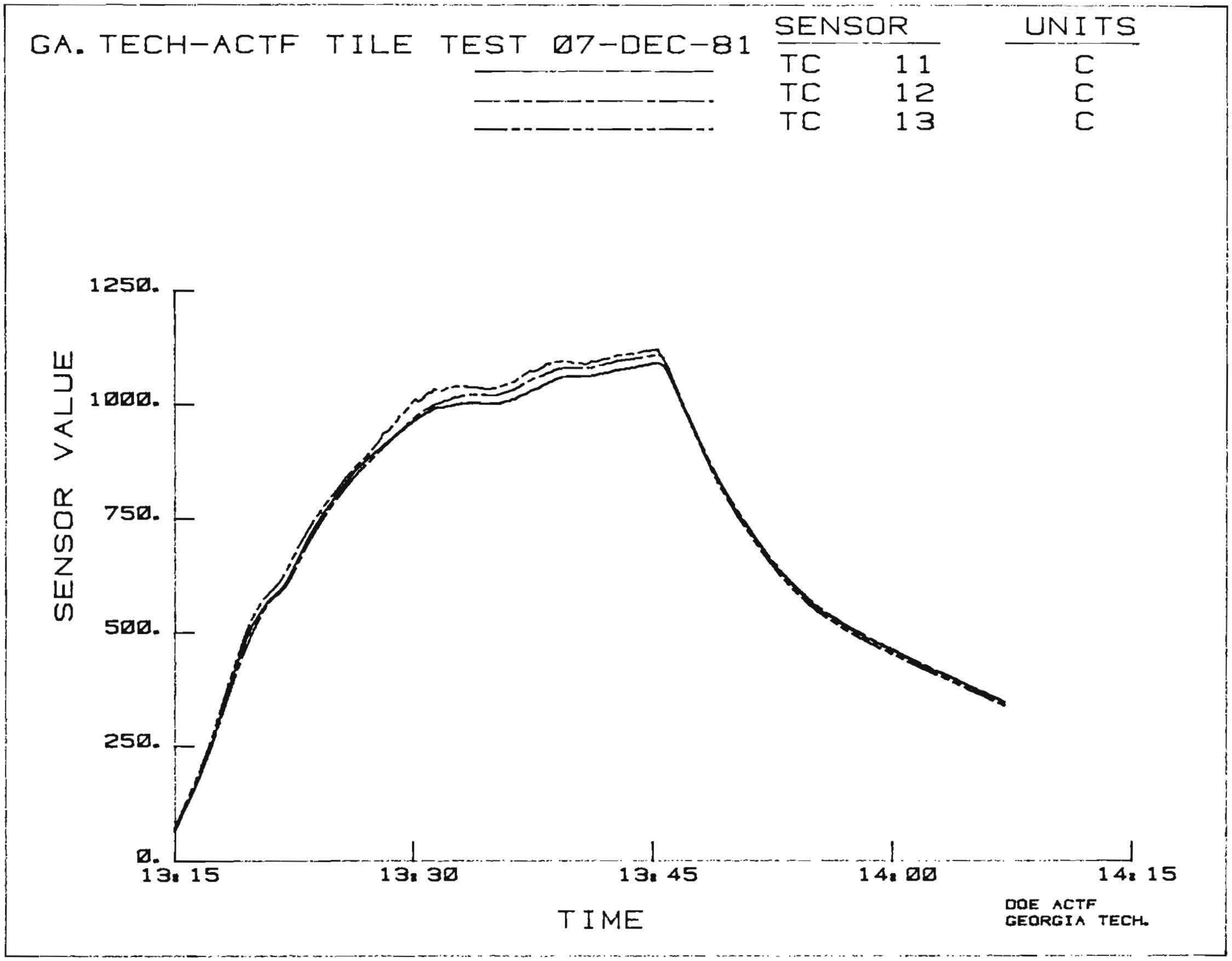

Figure 26. Temperature Variation In Glazed Tile in Southwest Position, Firing Number 12

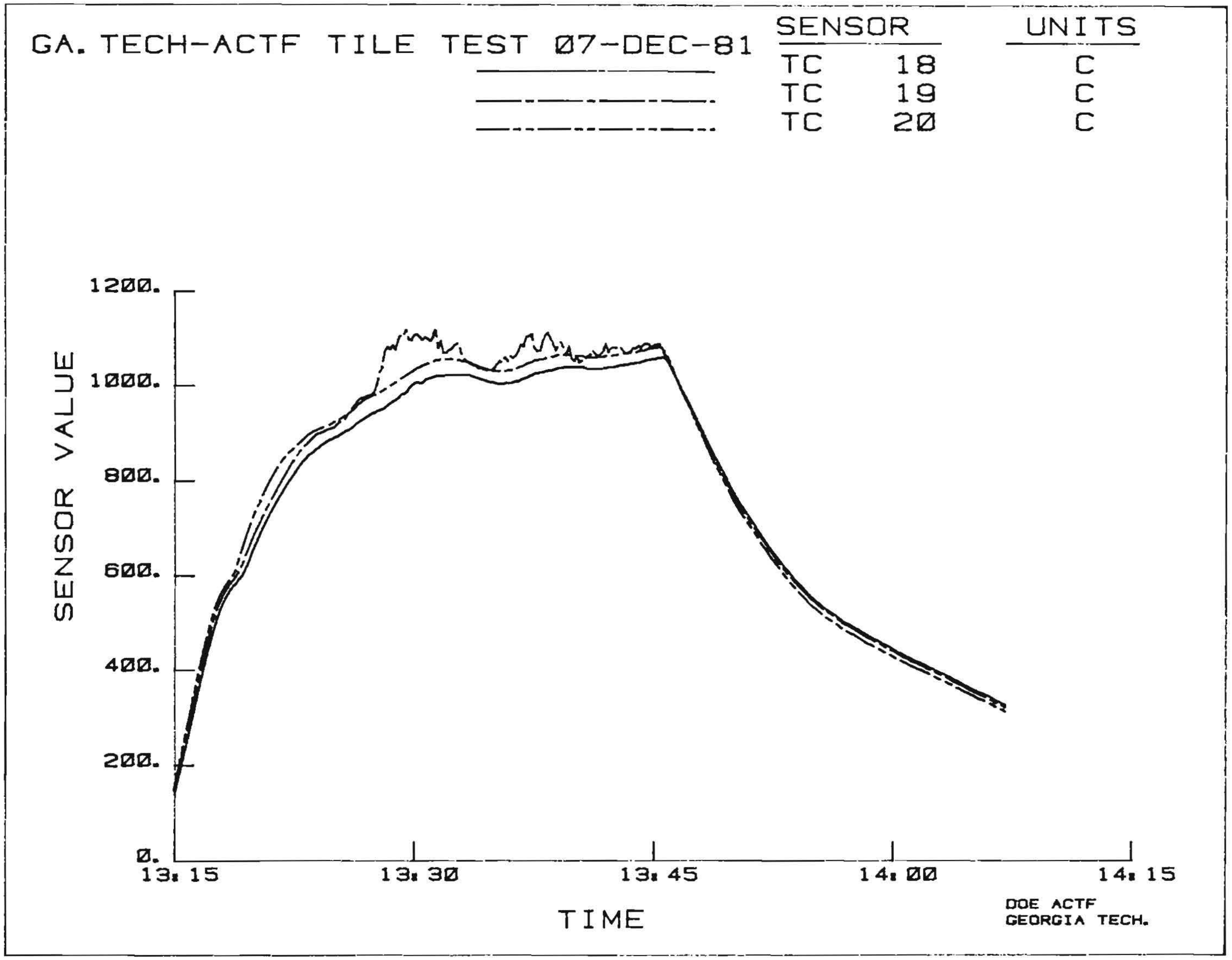

Figure 27. Temperature Variations in Bisque Tile in Northeast Position, Firing Number 12

TABLE XV

QUALITATIVE ELEMENTAL ANALYSIS OF
COMMERCIAL TILE BODY

Element	Line Density
Aℓ	Medium Strong
Ag	Trace
B	Weak
Ca	Strong
Cr	Faint
Cu	Weak
Fe	Medium
Ga	Faint
K	Weak
Mg	Very Strong
Mn	Medium
Na	Medium
Ni	Weak
P	Trace
Pb	Trace
Si	Very Strong
Ti	Weak
V	Faint

Increasing order of strength: Trace, Faint, Weak, Medium, Medium Strong, Strong, Very Strong.

analyzer. Both methods revealed a slow steady loss of volatile material over the temperature range of 500^{o} to 800^{o} C. The DSC method showed a volatile loss of 9.4 percent and required 865 mcal of energy to remove 1.60 mg of volatile material from a 17.02 mg sample.

The TGA was run at a 20^{o} C/minute heating rate. The average weight loss with respect to temperature is shown in Table XVI.

TABLE XVI

THERMOGRAVIMETRIC ANALYSIS RESULTS

Temperature (^{o}C)	Weight Percent Remaining
60	100
100	100
200	100
300	100
450	99.6
500	98.4
600	96.8
700	95.2
800	94.6
900	93.3
1000	93.3

Average Weight Loss 6.7 percent.

3. Percent Firing Shrinkage

The fired tiles were measured for shrinkage at the center and near both ends in two directions; perpendicular and parallel to the center-line axis running from the aperture to the cavity wall. The shrinkage parallel to this axis was essentially uniform, but measurements perpendicular to the axis indicated that shrinkage generally decreased in the direction from the aperture toward the cavity wall. Shrinkage data are given in Table XVII.

4. Warpage of Ceramic Tile

The flatness of the fired tile was determined using a device consisting of a heavy brass plate with a dial gauge mounted in the center. The plate is supported on three eccentric registry pins. When this device is placed on a flat surface and rotated four times through 90 degrees each rotation the readings remain at zero. For test specimens negative or positive readings are obtained. Negative readings indicate a concave surface. Each whole tile was measured with this device placed in four positions. The commercial fired tile were measured to serve as a reference standard. The data for these tests are shown in Table XVIII.

5. Visual Observations of Glazed Tile

(a) Firing No. 2. The tile in the north position had thermal shock cracks with the glaze flowing into them indicating failure while heating up. The glaze on the edge toward the cavity wall did not completely mature due to raising the cavity to control temperature. The east tile was the same as the north tile. The northeast tile was the same, but exhibited no thermal shock

TABLE XVII

PERCENT SHRINKAGE OF FIRED WALL TILE

Tile Designation	Perpendicular			Parallel		
	End	Middle	End	End	Middle	End
Commercial						
Glaze-1	1.41	1.36	1.36	1.43	1.50	1.41
Glaze-2	1.43	1.41	1.41	1.48	1.48	1.41
Glaze-3	1.48	1.52	1.48	1.41	1.39	1.41
Glaze-4	1.43	1.41	1.39	1.43	1.50	1.50
Glaze-5	1.50	1.41	1.39	1.43	1.50	1.43
Bisque-1	1.75	1.80	1.69	1.62	1.69	1.66
Bisque-2	1.78	1.87	1.66	1.73	1.82	1.78
Bisque-3	1.66	1.75	1.75	1.55	1.64	1.55
Bisque-4	1.71	1.87	1.75	1.69	1.69	1.69
Bisque-5	1.87	1.87	1.75	1.64	1.73	1.69
Firing 2						
Bisque-SE	1.27	1.18	1.15	1.15	1.15	1.02
Bisque-N	1.36	1.09	0.79	1.32	1.25	1.13
Glazed-NE	1.20	0.92	0.58	0.90	0.90	0.85
Glazed-N	1.04	0.92	0.67	0.92	0.74	0.83
Firing 3						
Bisque-S	1.57	1.22	0.35	1.32	1.15	1.25
Bisque-W	1.36	1.20	0.90	1.32	1.27	1.34
Bisque-NW	1.29	1.20	0.88	1.32	1.18	1.27
Bisque-E	1.29	1.41	0.97	1.39	1.20	1.41
Bisque-SE	1.48	1.29	0.79	1.36	1.39	1.45
Glaze-N	1.41	1.52	0.41	1.32	1.25	1.32
Glaze-NE	1.41	0.95	0.65	0.51	0.85	1.45

(Continued)

TABLE XVII (Continued)

PERCENT SHRINKAGE OF FIRED WALL TILE

Tile Designation	Perpendicular End	Perpendicular Middle	Perpendicular End	Parallel End	Parallel Middle	Parallel End
Firing 5						
Bisque-SW	1.43	1.29	1.15	1.39	1.29	1.34
Bisque-NW	1.32	1.18	1.02	1.22	1.22	1.13
Bisque-NE	1.29	1.25	1.22	1.27	1.27	1.27
Bisque-SE	1.45	1.39	1.20	1.41	1.36	1.36
Firing 6						
Bisque-S	1.43	1.27	1.41	1.43	1.43	1.22
Bisque-NE	1.27	1.32	1.20	1.32	1.29	1.29
Glaze-SW	1.41	1.22	1.04	1.32	1.36	1.41
Glaze-N	1.16	0.97	0.99	1.02	1.22	1.18
Glaze-E	1.20	1.04	0.90	1.25	1.18	1.13
Firing 7						
Bisque-SW	1.29	1.29	1.18	1.22	1.20	1.25
Bisque-E	1.34	1.22	1.06	1.22	1.13	1.25
Glaze-S	1.25	1.20	1.18	1.18	1.20	1.02
Glaze-SE	1.36	1.32	1.15	1.13	1.13	1.22
Firing 8						
Bisque-S	2.03	1.69	1.36	1.66	1.71	1.73
Bisque-W	1.41	1.57	1.46	1.41	1.43	1.29
Bisque-E	1.66	1.66	1.41	1.80	1.59	1.75
Glaze-SW	1.52	1.32	1.18	1.43	1.52	1.64
Glaze-NW	1.27	1.32	1.27	1.27	1.13	1.20
Glaze-N	1.36	1.25	1.18	1.43	1.52	1.64
Glaze-SE	1.94	1.62	1.32	1.80	1.55	1.71

(Continued)

TABLE XVII (Concluded)

PERCENT SHRINKAGE OF FIRED WALL TILE

Tile Designation	Perpendicular			Parallel		
	End	Middle	End	End	Middle	End
Firing 9						
Bisque-S	1.87	1.73	1.34	1.92	1.62	1.78
Bisque-W	1.48	1.36	1.62	1.29	1.41	1.34
Bisque-N	1.43	1.29	1.36	1.29	1.36	1.29
Bisque-NE	1.39	1.39	1.36	1.29	1.45	1.38
Glaze-SE	1.69	1.59	1.18	1.80	1.69	1.78
Glaze-E	1.39	1.66	1.25	1.64	1.27	1.50
Firing 10						
Bisque-S	1.66	1.50	1.18	1.82	1.45	1.73
Bisque-W	1.59	1.43	1.20	1.52	1.18	1.34
Bisque-N	1.43	1.32	1.36	1.27	1.29	1.36
Glaze-SW	1.80	1.64	1.34	1.59	1.34	1.57
Glaze-NW	1.39	1.15	1.11	1.09	1.11	1.13
Glaze-NE	1.20	1.36	1.32	1.32	1.06	1.18
Glaze-E	1.20	1.41	1.25	1.34	1.09	1.32

TABLE XVIII

RELATIVE FLATNESS OF COMMERCIAL AND SOLAR FIRED TILE

Tile Designation	Deviation (Inches) 0^{o}	90^{o}	180^{o}	270^{o}
Commercial Fired				
Bisque-1	-0.003	+0.003	-0.004	+0.003
Bisque-2	-0.004	0	-0.006	-0.001
Bisque-3	+0.003	0	+0.003	0
Bisque-4	-0.004	-0.002	-0.001	-0.002
Bisque-5	-0.001	-0.005	-0.001	-0.005
Glaze-1	-0.001	-0.002	-0.001	-0.002
Glaze-2	0	+0.002	0	+0.002
Glaze-3	+0.001	+0.001	+0.001	+0.001
Glaze-4	-0.001	-0.002	-0.001	-0.002
Glaze-5	+0.001	+0.001	+0.001	0
Firing 2				
Bisque-W	0	-0.006	-0.004	-0.003
Bisque-SE	-0.018	-0.018	-0.024	-0.020
Glaze-N	+0.006	+0.003	+0.006	+0.006
Glaze-NE	+0.015	+0.021	+0.016	+0.019
Firing 3				
Bisque-S	-0.007	-0.008	-0.010	-0.013
Bisque-W	-0.007	-0.001	-0.008	-0.002
Bisque NW	-0.005	-0.010	-0.004	-0.012
Bisque-E	-0.007	-0.007	-0.011	-0.013
Bisque-SE	-0.023	-0.017	-0.018	-0.015
Glaze-N	-0.002	-0.001	-0.008	+0.006
Glaze-NE	+0.004	+0.002	+0.003	+0.001

(Continued)

TABLE XVIII (Continued)

RELATIVE FLATNESS OF COMMERCIAL AND SOLAR FIRED TILE

Tile Designation	Deviation (Inches)			
	0^0	90^0	180^0	270^0
Firing 5				
Bisque-SW*	+0.002	+0.002	+0.001	+0.001
Bisque-NW*	+0.005	0	+0.004	+0.003
Bisque-NE*	+0.002	+0.002	+0.003	+0.002
Bisque-SE*	+0.004	+0.005	+0.004	+0.005
Firing 6				
Bisque-S*	-0.004	-0.004	-0.001	-0.004
Bisque-NE*	-0.003	-0.005	-0.002	-0.004
Glaze-SW	+0.013	+0.008	+0.013	+0.011
Glaze-N	+0.007	+0.005	+0.008	+0.006
Glaze-E*	+0.003	+0.003	+0.001	+0.002
Firing 7				
Bisque-SW*	+0.004	+0.004	+0.004	+0.005
Bisque-E*	+0.004	0	+0.003	0
Glaze-S	+0.002	+0.007	+0.003	+0.005
Glaze-SE	+0.028	+0.027	+0.028	+0.030
Firing 8				
Bisque-S*	-0.004	0	-0.005	-0.002
Bisque-W	-0.009	-0.021	-0.012	-0.017
Bisque-E*	-0.005	-0.002	-0.005	0
Glaze-SW	-0.011	-0.011	-0.013	-0.009
Glaze-NW*	-0.005	0	-0.003	0
Glaze-N	-0.008	-0.005	-0.009	-0.005
Glaze-SE	-0.002	-0.006	+0.005	+0.004

*Acceptable based on being in the range of flatness of the commercial tile.

(Continued)

TABLE XVIII (Concluded)

RELATIVE FLATNESS OF COMMERCIAL AND SOLAR FIRED TILE

Tile Designation	Deviation (Inches) 0°	90°	180°	270°
Firing 9				
Bisque-S*	-0.003	-0.002	-0.004	-0.005
Bisque-W	-0.017	-0.015	-0.015	-0.024
Bisque-N	-0.011	-0.007	-0.011	-0.007
Bisque-NE*	-0.005	-0.002	-0.005	-0.001
Glaze-SW	-0.016	-0.013	-0.018	-0.014
Glaze-NW*	-0.002	-0.001	-0.003	-0.001
Glaze-SE	-0.007	-0.007	-0.009	-0.008
Glaze-E	-0.016	-0.014	-0.017	-0.013
Firing 10				
Bisque-S	-0.038	-0.030	-0.037	-0.029
Bisque-W	-0.026	-0.034	-0.028	-0.030
Bisque-N	-0.018	-0.016	-0.018	-0.024
Glaze-SW	-0.004	-0.007	-0.005	-0.004
Glaze-NW*	+0.003	+0.001	0	0
Glaze-NE	-0.009	-0.008	-0.008	-0.008
Glaze-E	-0.008	-0.011	-0.008	-0.011
Glaze-SE	-0.008	-0.004	-0.009	-0.007

*Acceptable based on being in the range of flatness of the commercial tile.

cracks. This may have been due to the fact that this tile had no thermocouple attached thus, there were no stress risers.

(b) Firing No. 3. The glaze on all specimens was glossy, however, the outer edge of the cavity caught the tiles in the northern sector and prevented maturity of the glaze over approximately one-third of the tile area.

(c) Firing No. 4. All tiles showed severe thermal shock cracks and the glazed surface finish was matte on all tiles.

(d) Firing No. 5. All glazed tiles were severely thermal shocked in that all cracked into several pieces. The glaze over most of the surface on all tiles was very smooth except for approximately 1/8 inch along the surface nearest the cavity wall. These edges failed to mature.

(e) Firing No. 6. The surface of these tiles was not uniform in color. Surface was glossy on the sides nearest the aperture, but became matte in large areas over 1/2 the surfaces which were near the cavity wall. The matured parts of this glaze exhibited large pinholes.

(f) Firing No. 7. All glazed tiles exhibited thermal shock lines extending into the tiles approximately 1/2 inch on the edges nearest the cavity walls. All tile exhibited a low gloss surface. Thermal shock seems to have been caused by wind blowing under the edge of the cavity cooling the outer surfaces.

(g) Firing No. 8. This firing produced the tiles most comparable to the commercial fired tiles. Tile color was good and at least two tiles were comparable in flatness to the commercial tile. The major difference was there were more pinholes in the surface and the color tended to be slightly greener than the commercial fired tiles.

(h) Firing No. 9. Surface of all tiles were uniform in color and had a high gloss. These tiles exhibited more warping than firing 7 and there were more pinholes in the surface.

(i) Firing No. 10. Glazed surfaces were non-uniform with some glossy areas and some matte areas on all tiles. Time-temperature of this firing was insufficient for complete glaze maturity.

(j) Firing No's. 11 and 12. These firings occurred too late for ceramic evaluation and are not included in the visual comments because most had damage to the glaze surface prior to firing.

6. Transverse Strength

The tiles were cut into 1-1/4 inch wide strips and subjected to three point bending over a three inch span to determine modulus of rupture. The tiles were broken with the glazed surface down and in the case of the bisque tile the surface that would be glazed was down. The tile were broken on an Instron test machine; cross-head speed was 0.05 cm/minute. Since no more than two specimens were available from each tile and since there was insufficient time to break all tiles representative data have been grouped by firing number in Table XIX.

7. Porosity, Water Absorption, Bulk Density and Apparent Specific Gravity

The broken test bars from the bisque fired tiles were weighed dry, boiled in water, weighed wet and suspended in water according to the procedures

TABLE XIX

MODULUS OF RUPTURE FOR COMMERCIAL AND SOLAR FIRED TILE

Designation	Modulus of Rupture (psi)
Commercial	
Glazed 3a	5117
Glazed 3b	4690
Glazed Avg	4903
Bisque 4a	2944
Bisque 4b	2243
Bisque 5a	2265
Bisque 5b	2281
Bisque Avg	2433 ± 295
Firing 6	
Bisque Sm	3589
Bisque 5a	3495
Bisque SEa	3441
Bisque NEa	2382
Bisque NEm	1114
Bisque Avg	2804 ± 952
Glaze 6Na	4345
Glaze 6Em	3442
Glaze SWa	4100
Glaze Avg	3962 ± 381
Firing 7	
Bisque Ea	1750
Bisque Em	2566
Bisque SWa	2380
Bisque SWb	1909
Bisque Avg	2151 + 333

(Continued)

TABLE XIX (Continued)

MODULUS OF RUPTURE FOR COMMERCIAL AND SOLAR FIRED TILE

Designation	Modulus of Rupture (psi)
Glaze Sa	2716
Glaze SEa	2412
Glaze Avg	2564
Firing 8	
Bisque Sa	2853
Bisque Sm	3163
Bisque Ea	3399
Bisque Em	3436
Bisque Avg	3213 ± 233
Glaze Na	4893
Glaze Nd	3446
Glaze Avg	4194
Firing 9	
Bisque Sa	3778
Bisque Sb	3993
Bisque NEa	4248
Bisque NEm	3426
Bisque Avg	3861 ± 301
Glaze NWa	3473
Glaze NWm	3814
Glaze Avg	3644

(Continued)

TABLE XIX (Concluded)

MODULUS OF RUPTURE FOR COMMERCIAL AND SOLAR FIRED TILE

Designation	Modulus of Rupture (psi)
Firing 10	
Bisque Sa	5002
Bisque Sb	4380
Bisque Na	2398
Bisque Nb	2628
Bisque Avg	3602 ± 1114
Glaze Ea	3944
Glaze Em	5205
Glaze Avg	4575

specified in ASTM C-373. From these data the porosity, water absorption, bulk density and apparent specific gravity were calculated. These data for each group of tiles are presented in Table XX.

TABLE XX

POROSITY, WATER ABSORPTION, BULK DENSITY AND APPARENT SPECIFIC GRAVITY FOR COMMERCIAL AND SOLAR FIRED BISQUE WALL TILES

Tile Designation	Porosity	Water Absorption	Bulk Density	Apparent Specific Gravity
Commercial				
3	28.96	14.73	1.966	2.768
4a	28.81	14.61	1.972	2.770
4b	30.59	15.96	1.917	2.762
5a	30.02	15.75	1.906	2.723
Avg	29.60 ± 0.74	15.26 ± 0.60	1.940 ± 0.029	2.756 ± 0.019

(Continued)

TABLE XX (Continued)

POROSITY, WATER ABSORPTION, BULK DENSITY AND APPARENT SPECIFIC GRAVITY FOR COMMERCIAL AND SOLAR FIRED BISQUE WALL TILES

Tile Designation	Porosity	Water Absorption	Bulk Density	Apparent Specific Gravity
Firing 6				
Sa	27.03	13.46	2.007	2.751
Sm	26.96	13.42	2.009	2.751
NEa1	30.09	15.71	1.915	2.739
Nea2	29.11	14.95	1.948	2.747
NEm2	28.32	14.40	1.966	2.744
SEa1	28.06	14.23	1.972	2.739
SEa2	26.87	13.41	2.003	2.739
Avg	28.06 ± 1.13	14.23 ± 0.82	1.974 ± 0.033	2.744 ± 0.005
Firing 7				
Ea	30.93	16.12	1.919	2.778
Em	30.08	15.67	1.920	2.746
SWa	29.10	14.95	1.946	2.745
SWb	29.23	15.10	1.936	2.736
Avg	29.84 ± 0.74	15.46 ± 0.47	1.930 ± 0.011	2.751 ± 0.016
Firing 8				
Sa	29.70	15.42	1.526	2.740
Sm	28.28	14.43	1.960	2.732
Em	26.24	13.10	2.003	2.716
Ea	30.17	15.79	1.910	2.736
Avg	28.60 ± 1.53	14.69 ± 1.04	1.950 ± 0.041	2.731 ± 0.009

(Continued)

TABLE XX (Concluded)

POROSITY, WATER ABSORPTION, BULK DENSITY AND APPARENT SPECIFIC GRAVITY FOR COMMERCIAL AND SOLAR FIRED BISQUE WALL TILES

Tile Designation	Porosity	Water Absorption	Bulk Density	Apparent Specific Gravity
Firing 9				
Sa	25.24	12.51	2.017	2.698
NEa	27.82	14.09	1.975	2.736
NEm	29.53	15.27	1.933	2.743
Sm	25.89	12.93	2.002	2.702
Sb	28.94	14.94	1.437	2.726
Avg	27.48 $\pm$ 1.67	13.95 $\pm$ 1.08	1.973 $\pm$ 0.034	2.721 $\pm$ 0.018
Firing 10				
Sa	26.27	13.10	2.015	1.720
Sb	27.49	13.91	1.976	2.725
Na	28.92	14.81	1.953	2.747
Nb2	29.61	15.24	1.943	2.761
Nb1	29.96	15.55	1.927	2.751
Avg	28.45 $\pm$ 1.38	14.52 $\pm$ 0.90	1.961 $\pm$ 0.027	2.742 $\pm$ 0.016

8. Linear Thermal Expansion

Two specimens were measured for linear thermal expansion using an Orton dilatometer. One specimen was cut from a commercial fired bisque tile and one from the south specimen in firing number nine. These two thermal expansion runs are shown in Figures 28 and 29. The major difference in the two appear to be the amount of quartz in the specimen. The quartz inversion at 570^{0} C shows much more prominently in the commercial fired sample than in the solar fired sample.

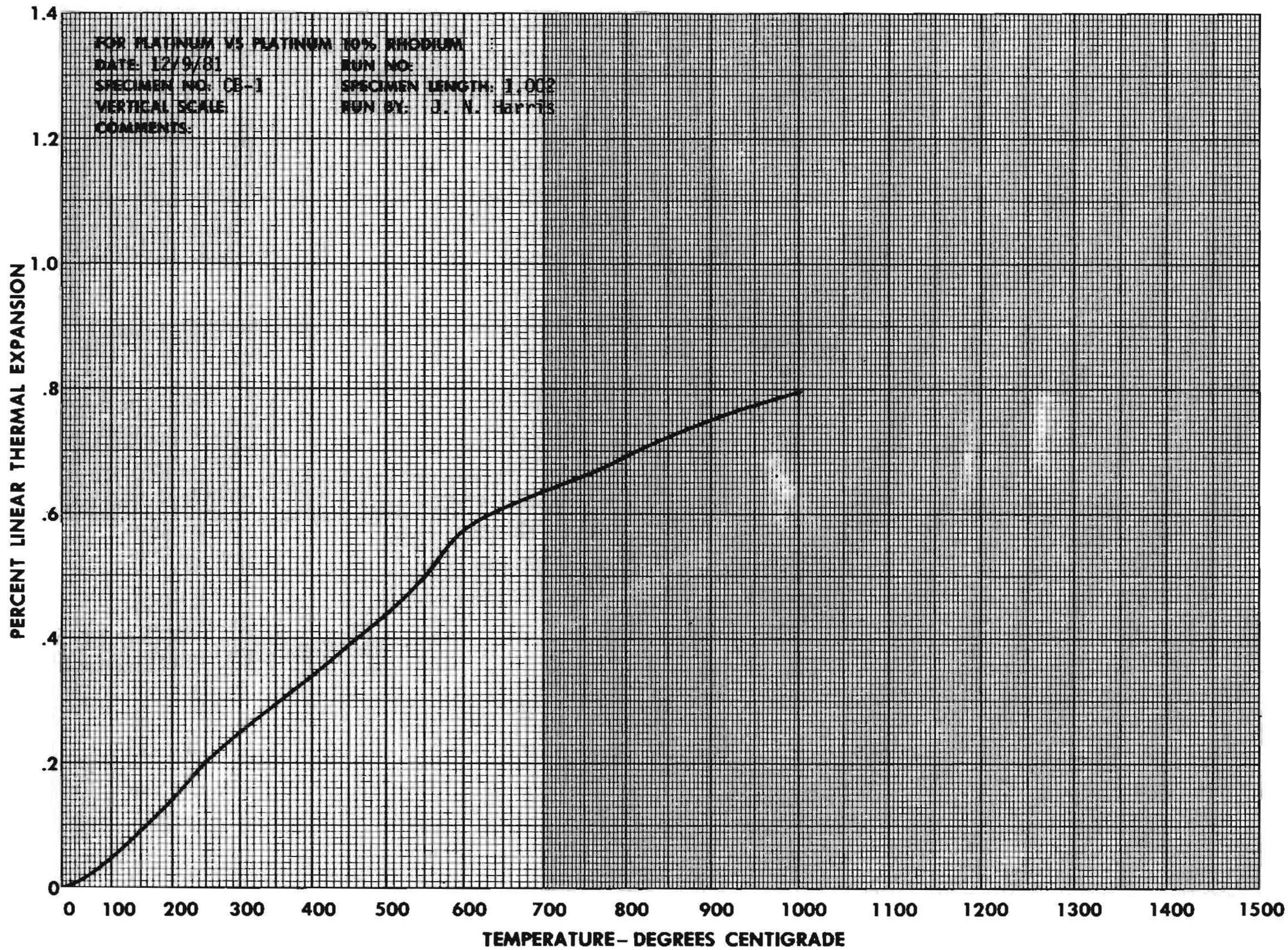

Figure 28. Linear Thermal Expansion of Commercial Fired Tile

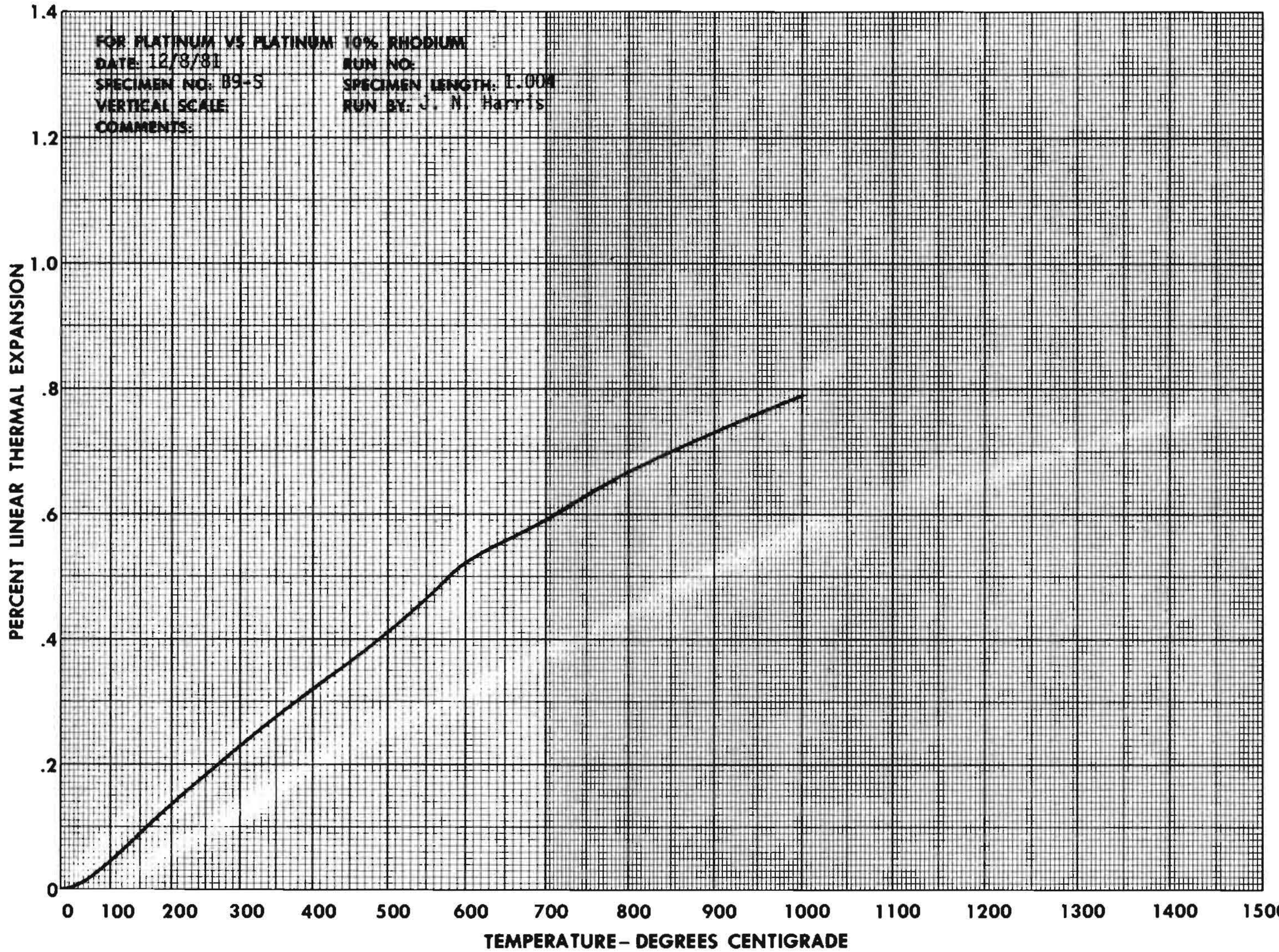

Figure 29. Linear Thermal Expansion of Solar Fired Tile , Firing 9, South Position

9. X-ray Diffraction Analysis

Three specimens were examined by x-ray diffraction analysis to determine crystalline forms present. One specimen was from the commercial bisque fired tile, the second specimen was from firing 9 and the third specimen from firing 10. Three major crystalline forms were identified in each tile body: quartz, $MgSiO_3$, and $CaSiO_3$. There was a very slight difference in the major quartz peak height between the samples from firings 9 and 10 but this difference was so slight as to to be within experimental error. The major quartz peak for the commercial sample went off scale when run at the same x-ray intensity as the two solar fired samples. This is consistent with the thermal expansion data.

10. Reflectance and Color Match

The reflectance transmission and color match was determined by running reflectance transmission values for commercial and solar fired glazed tile using a Bausch and Lomb Model 505 Spectrophotometer. Closeness of color match was determined by calculating trichromatic coefficients X and Y for each reflectance curve.

The reflectance transmission of a commercial fired tile was determined at three spots on the tile. These values all overlapped as shown in Figure 30. Reflectance transmission values for other selected solar fired tiles are shown in Figures 30-36.

The computed trichromatic values for illuminant C are shown in Table XXI. Based on these numbers the closest color match to the commercial fired tile was the northwest tile in firing 9. The poorest match of the measured tiles was the east tile in firing 10.

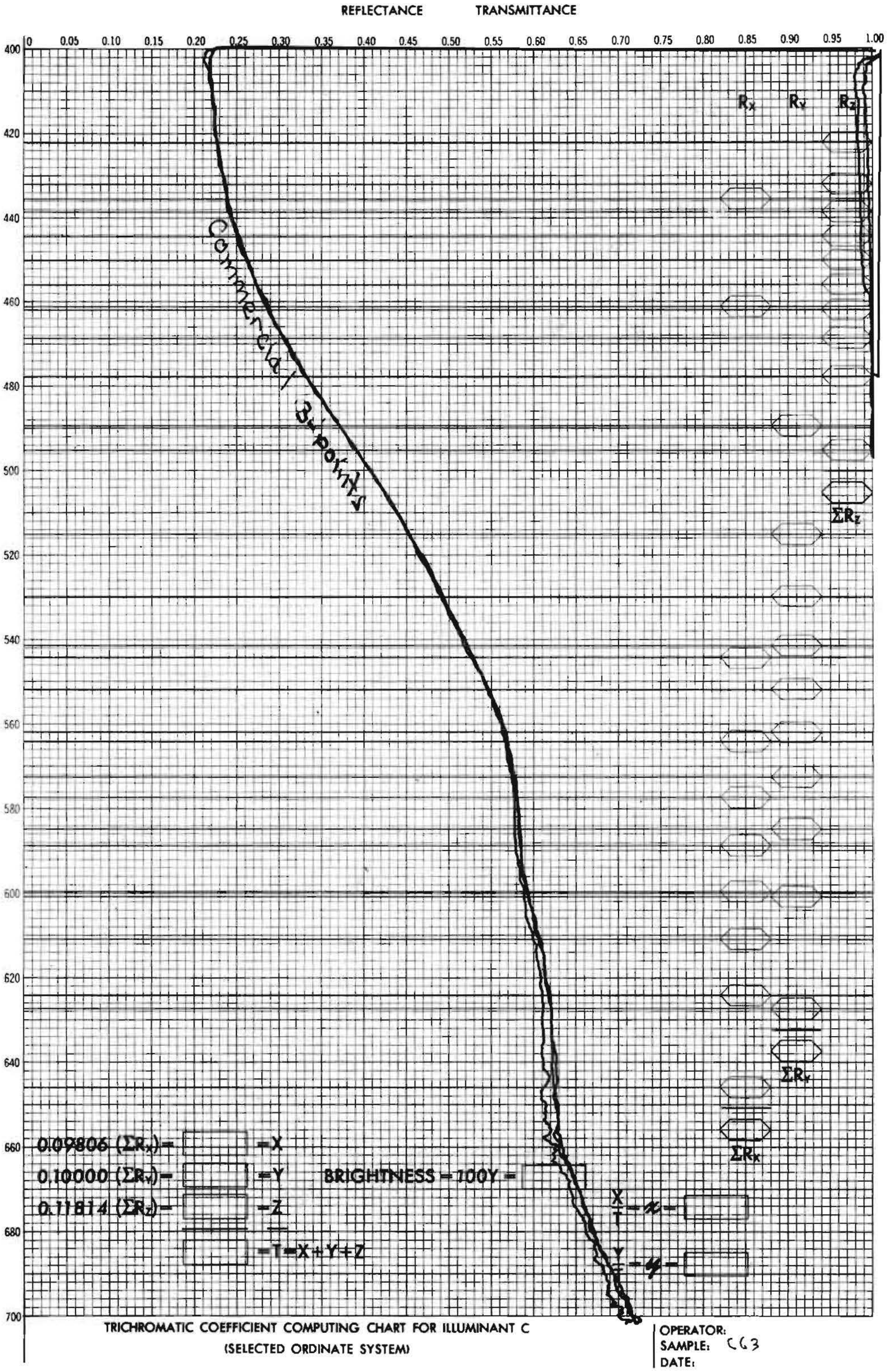

Figure 30. Reflectance/Transmittance Curve for Commercial Fired Glazed Tile . . . .

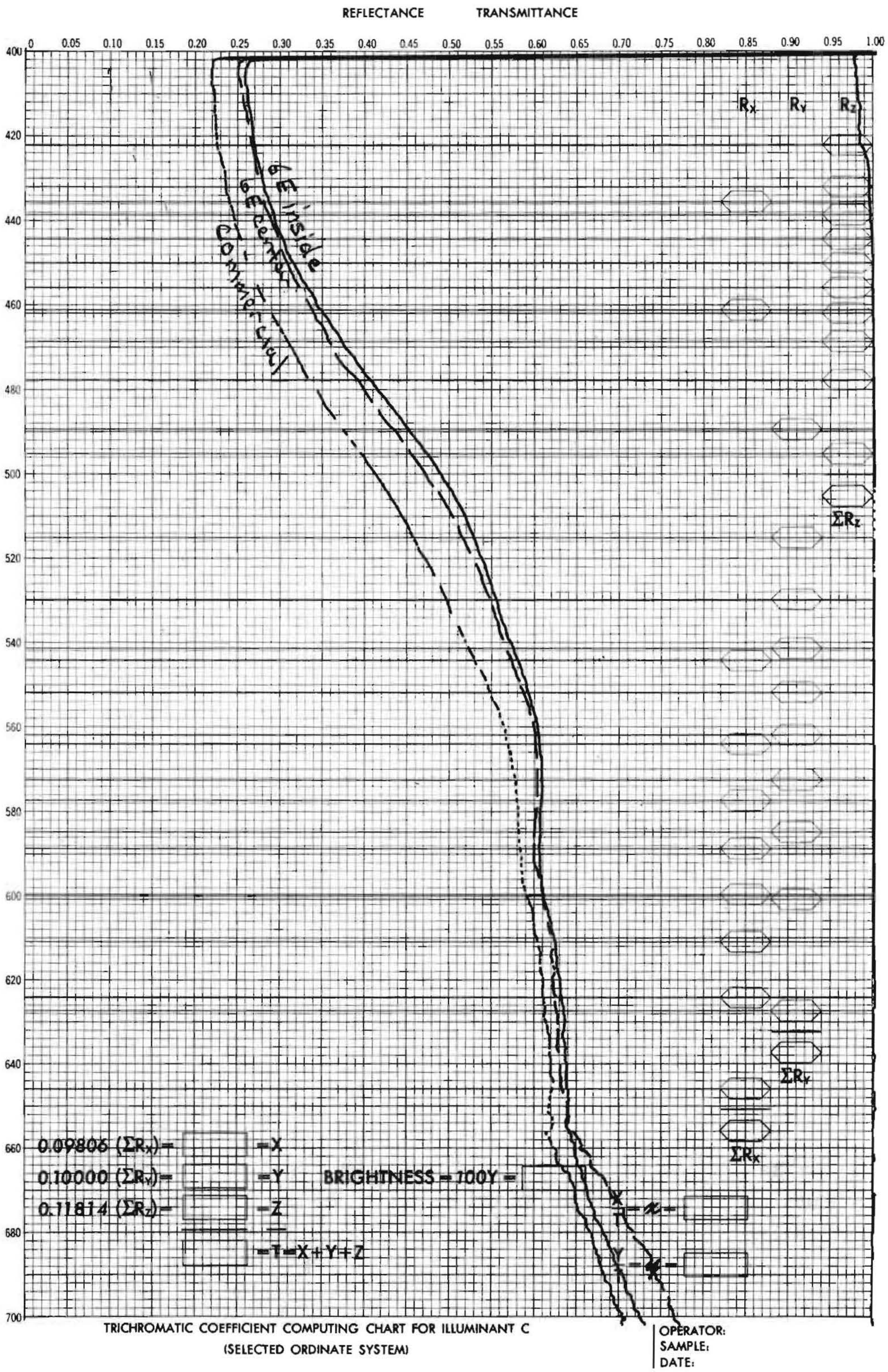

Figure 31. Reflectance/Transmittance Curve for East Tile, Firing Number 6

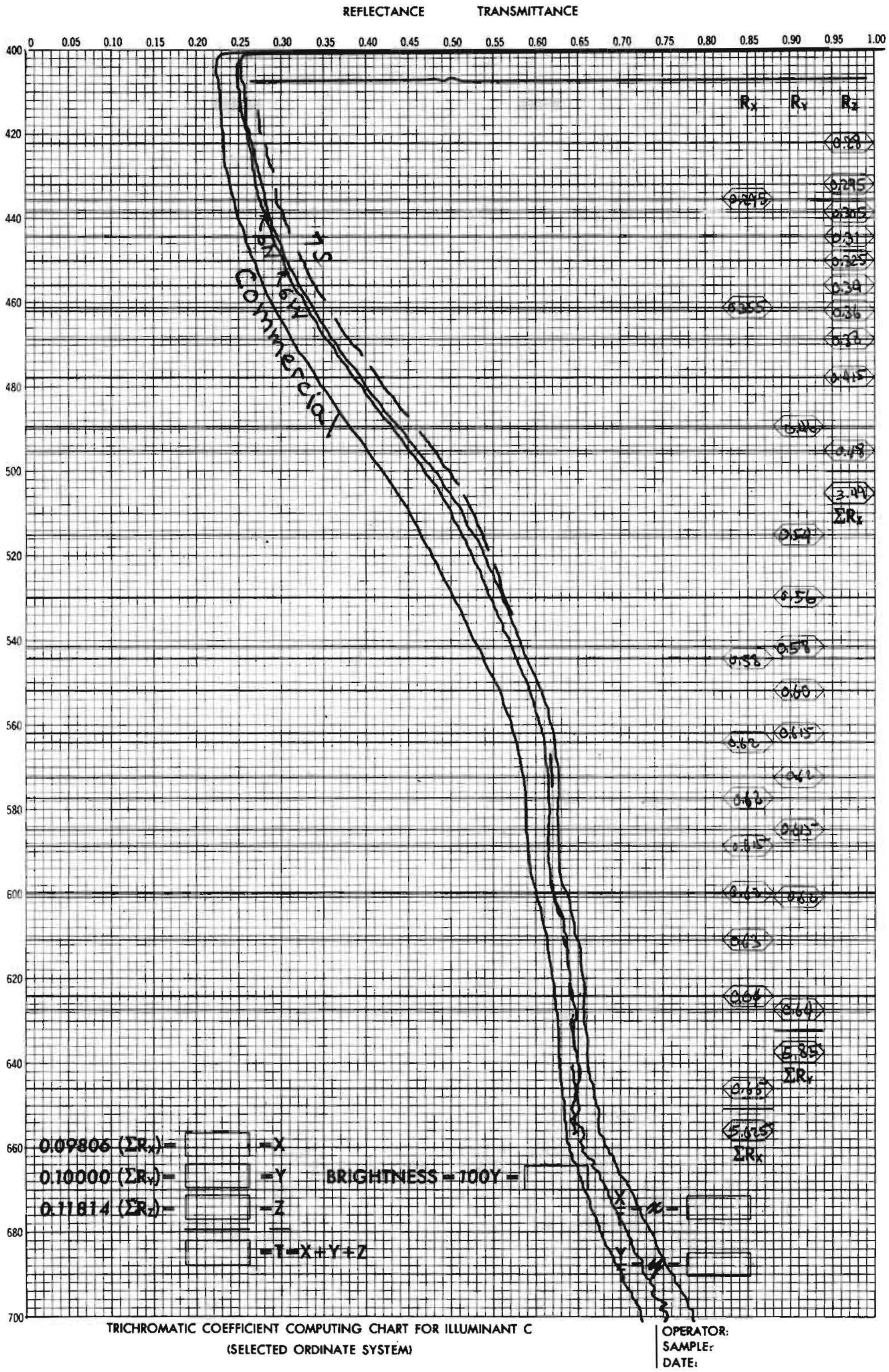

Figure 32. Reflectance/Transmittance Curves for South Tile, Firing Number 7 and North and West Tiles, Firing Number 6

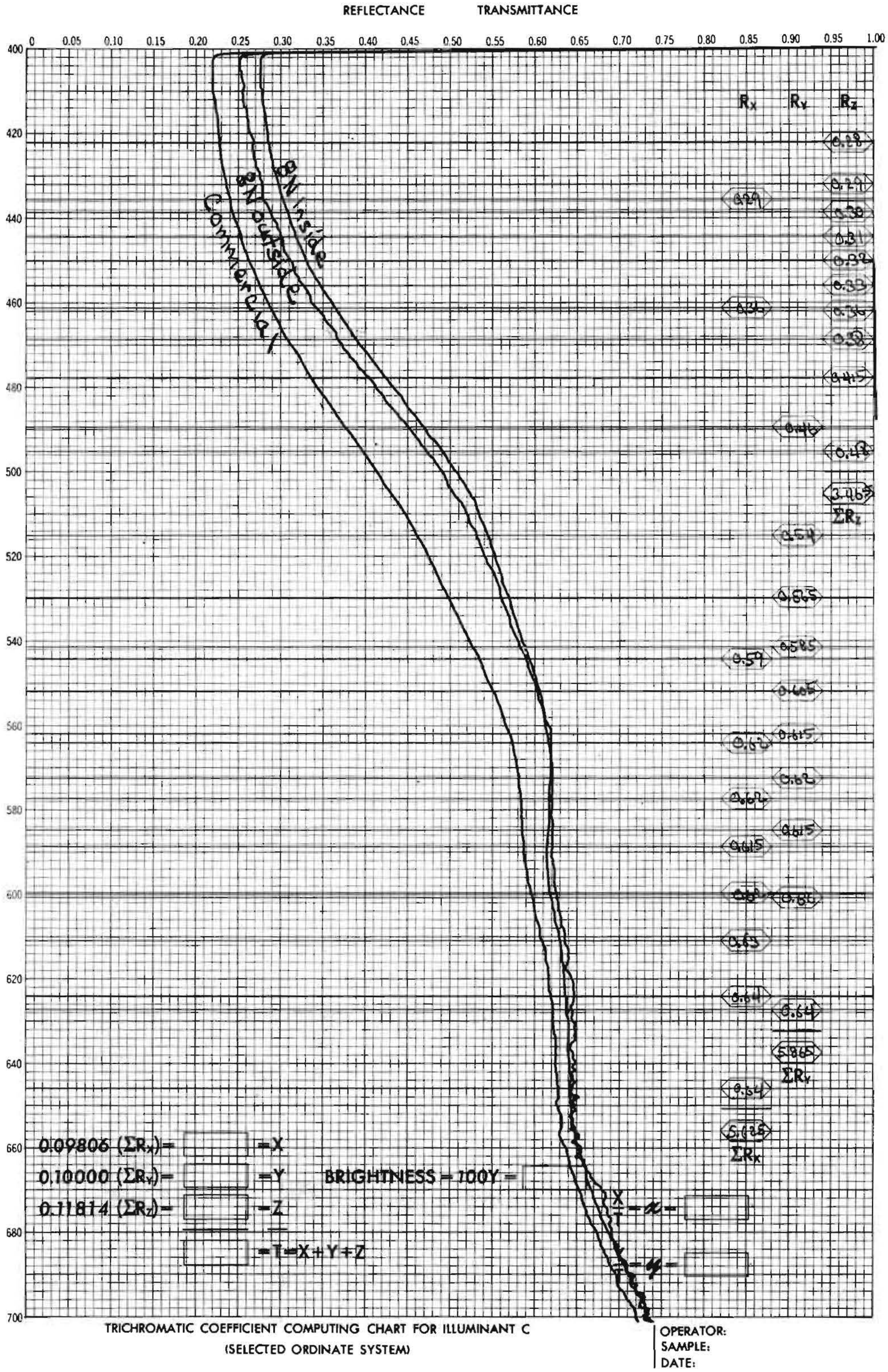

Figure 33. Reflectance/Transmittance Curve for North Tile, Firing Number 8

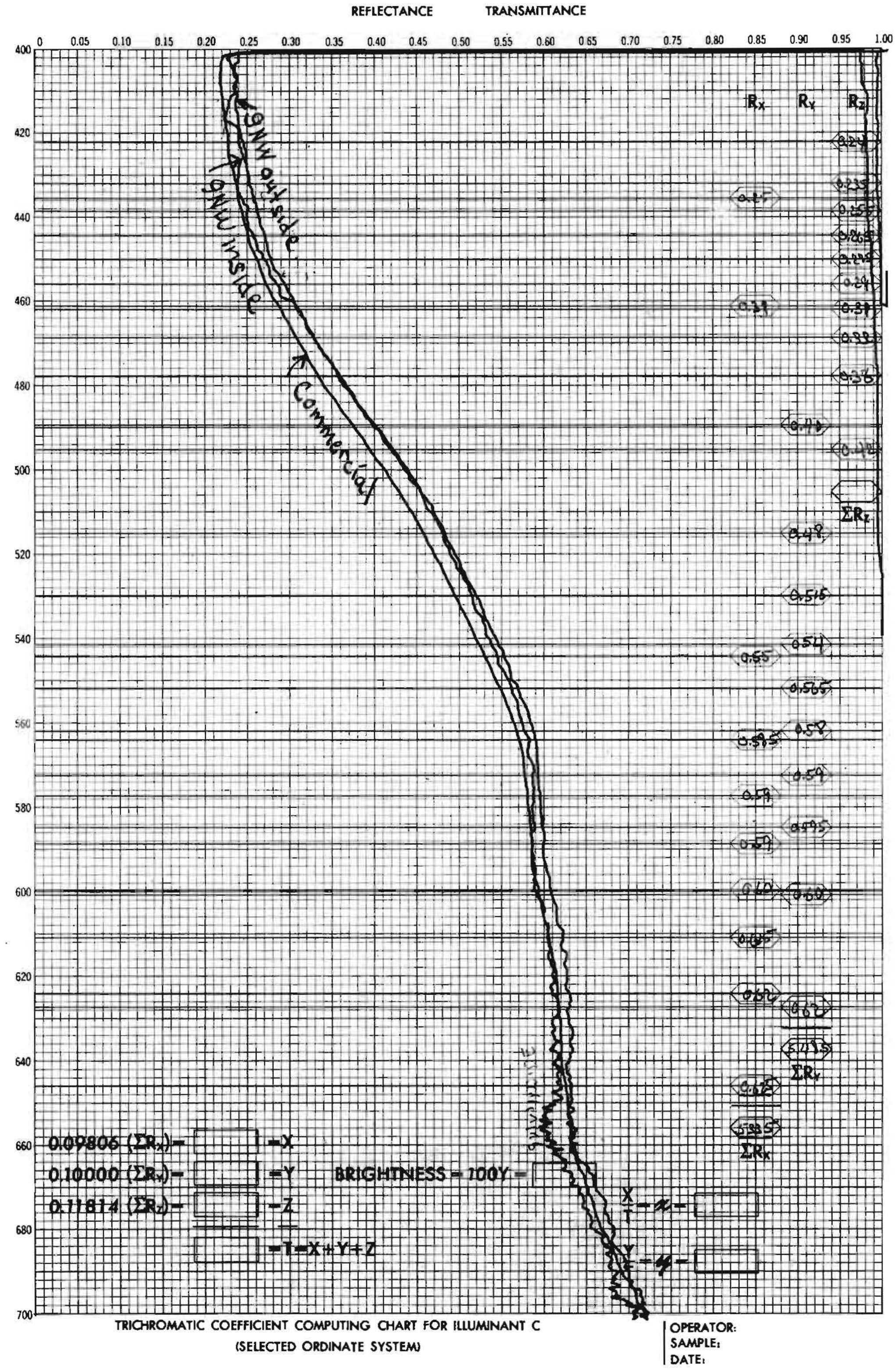

Figure 34. Reflectance/Transmittance Curve for Northwest Tile, Firing Number 9

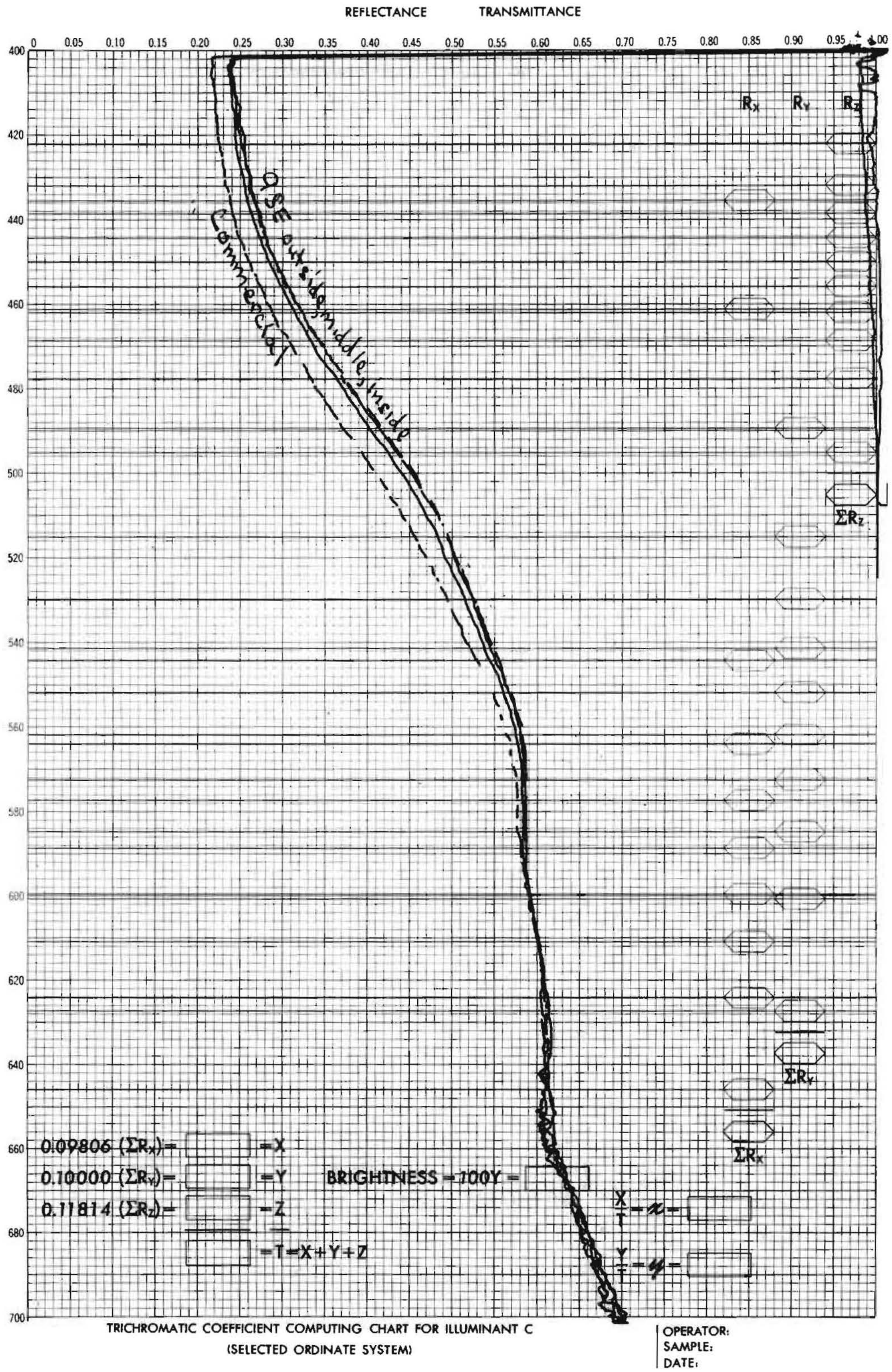

Figure 35. Reflectance/Transmittance Curve for Southeast Tile, Firing Number 9

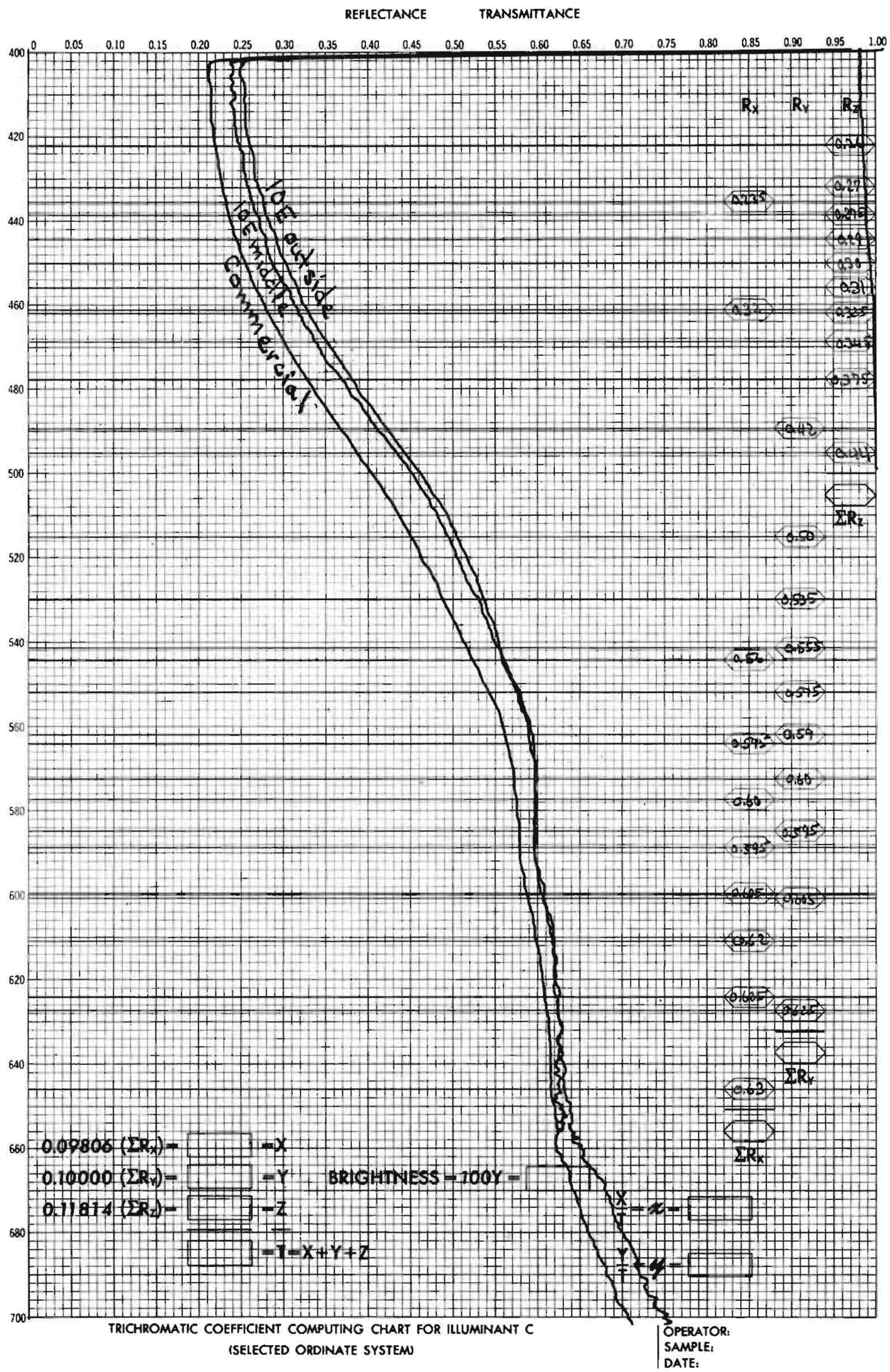

Figure 36. Reflectance/Transmittance Curve for East Tile, Firing Number 10

TABLE XXI

TRICHROMATIC COEFFICIENTS FOR ILLUMINANT C FOR COMMERCIAL AND SOLAR FIRED TILE

	X	Y
Commercial Fired Tile	0.3707	0.3882
East Tile, Firing 6	0.3571	0.3793
Average of North and West Tiles, Firing 6	0.3561	0.3777
North Tile, Firing 8	0.3565	0.3790
Northwest Tile, Firing 9	0.3675	0.3853
Southeast Tile, Firing 9	0.3626	0.3823
East Tile, Firing 10	0.3414	0.36134

IV. DISCUSSION

Ceramic wall tile can be fired by diffuse radiation reflected from a concentrated solar beam, however, there are a number of problems to be overcome that make the use of direct solar radiation unattractive for translating wall tile from the raw state to the final fired and glazed state. The greatest problem with this type of firing is keeping the tiles from warping. In the cavity system used the greatest amount of heat transfer occurs by radiation and the majority of thermal flux enters the top of the tile. As a result, the upper portions of the tile densify more than the lower portions, resulting in a convex surface. Elevating the tile on trivets did not allow enough secondary reflected radiation to get under the tile to provide uniform heating from the bottom. Also, the small support area of the trivet allowed the tiles to droop resulting in a convex warp. The flatest tile were achieved by firing directly on the 3000 degree board base.

An unanticipated problem was the non-uniform heating across the tile body. The heating was much greater on the tile body near the aperture than it was near the cavity wall. Also, heating of each tile in a single firing was not uniform. Two factors appeared to affect the temperature at individual positions within the cavity: (1) the position of the sun, and (2) the direction and velocity of the wind. Examination of all the temperature data from all the firings showed that the hottest position within the cavity corresponded to the position of the sun in the sky. That is, in the early morning the east and southeast positions were hottest, at mid-day the south position was hottest and in the afternoon it was the southwest and west positions. This was generally true for all firings, however, on days when the wind velocity was high, wind blowing into the aperture and between the cavity and base could cause the hottest point in the cavity to vary. As a result of these temperature variations the color and density of tile varied from position to position and also across individual tiles, as can be seen in many of the plots of temperature data.

The rate of temperature increase for glazed and bisque tile varied greatly on heat up due to differences in absorption/emittance of these surfaces; however, as firing temperature was reached and the glaze began to mature the recorded temperature differences between glazed and bisque tiles became much smaller.

Another major problem occurred in controlling heating and cooling rates. Initial attempts were made to control the heating rate by raising the cavity of the base, however, this resulted in cooling of the outer edges, preventing maturing of the glaze and/or causing thermal shock. On later firings the rate of temperature increase or decrease was controlled by slowly opening or closing the shutters of the ACTF as required. However, this control was limited because the shutters could not be left in the solar beam very long without overheating. A better arrangement would have been a water cooled iris type shutter which would completely close the aperture, however, neither the time or funds were available on this program to construct a shutter of this type.

Reasonably good color matches were obtained with the commercial glazed tile on some tiles in test firings 8 and 9, although visual observation indicated that all of the yellow glazed solar fired tile had a slightly greener hue. This is affirmed by the trichromatic values in Table XX. All of the solar fired tiles exhibited more pinholing than the commercial fired tile. The best firings as to color match and flatness of the tile were 8 and 9 where temperature control was attempted at 1120^{o} C for 20 and 10 minutes, respectively. It appears that the higher temperature was required to gain uniform maturity of the glaze over the entire tile surface.

The modulus of rupture data are difficult to interpret because of the small number of samples and the large scatter. The scatter is primarily due to ridges on the back of the tile resulting in non-uniform thickness. The most apparent data are the low values for firing 7. This may be attributed to the thermal shock during this firing resulting in micro-cracking of the test specimens, but the porosity data also show a lower bulk density for firing 7 which could have caused the lower modulus of rupture values.

The porosity data also show the large variation in densities achieved in the same firing, pointing out the non-uniformity of firing from position to position within the cavity.

The commercial tile have a lower modulus of rupture and bulk density and a higher porosity than the best solar fired tile indicating that the solar fired tile have been subjected to higher temperatures. This is borne out by the thermal expansion and x-ray diffraction data which show less crystalline quartz in the solar fired tile than in the commercial fired tile. This indicates that the solar fired tile reached higher temperatures quickly and were at the higher temperatures longer than the commercial fired tile. This may be attributed to the difference in methods of heat transfer between conventional and solar firing. The primary method of heating the solar fired tile is by relatively short wavelength radiation in the visible and near infrared. Very little heat transfer takes place by convection or conduction. As a result, the radiant heating penetrates in depth and brings the majority of the tile body to firing temperature very quickly. The opposite is true in conventional heating of wall tile where the majority of heat transfer takes place by convection. Any radiant heating that occurs is from much longer wavelength infrared. As a result the in-depth heating rate of the tile is considerably different.

Another problem that has not been covered with firing wall tile with direct solar radiation is the lack of temporary thermal storage. That is if a cloud covers the sun there can be a rapid fall in temperature resulting in cycling the temperature up and down several times before completing firing.

The negative points brought out in this discussion are not intended to discourage the use of solar energy as a source of process heat for ceramic products such as wall tile, however, another approach rather than direct firing with radiant energy should be considered.

The ACTF as presently configured could easly provide temperatures greater than 1300^{0} C in a cavity of the type used for this experiment. This radiant energy could be used to heat a brick or other ceramic refractory checker stove. With suitable fans and ducting, air could be blown across this "stove" to another chamber arranged for firing ceramic tile in a more conventional manner. By suitable valving of hot and cold air temperature zones of heat could be established at certain points in the firing chamber and tiles could be continually pushed through the firing zone in the same manner as with conventional tunnel kilns. Heat transfer would also be primarily by convection.

V. RECOMMENDATIONS

Any future program to use solar energy as process heat for ceramic parts such as wall tile should consider using the solar energy to heat a refractory air heat exchanger. The hot air would then be used to process the ceramic parts. Such a program could be conducted using the DOE Advanced Components Test Facility at the Georgia Institute of Technology and would be a natural follow-on to the work described in this report.