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16. Abstract

Many entities currently use fly ash in portland cement concrete (PCC) pavements and structures. Although the body of knowledge is great concerning the use of fly ash, several projects per year are subject to poor performance where fly ash is named as the culprit. Generally the "bad" projects arise due to one of two common errors:

1. Poor understanding of what fly ash is and how it affects concrete pavement construction and performance or
2. A switch of fly ash sources midstream during the construction project.

The objective of this research was to identify tools available for quality control (QC) of as delivered class C fly ash. The main focus of the research was to identify penetration type devices and test procedures including the Iowa Set Time Test, Gillmore needle, and Vicat needle. Another focus of the investigation was the quick heat generation index test. For the first objective, three penetration type test devices were investigated including the Vicat needle, Gillmore needle, and the pocket penetrometer. Class C fly ash samples were obtained from about ten sources available to Louisiana Department of Transportation and Development (LADOTD). The second objective was to indentify if the quick heat generation test can identify small changes in class C fly ash whether that be a change in chemistry or a physical change in the fly ash fineness. Statistical modeling was used to determine if a relationship existed between the various initial and final set times and the maximum temperature of the fly ash paste and the fly ash chemistry and fineness

The Gillmore needle, Vicat needle and the pocket penetrometer yielded similar results when observing the times to initial and final set across the three test methods. Although the test methods pointed out significant differences in set times between buckets within a source, those differences were a non-issue when incorporating portland cement into the sample.

The temperature results showed that the test method is unable to be used as either a quality control or quality assurance device in characterizing class C fly ash. The statistical analysis results showed outliers within the sources, but further testing when incorporating portland cement showed these differences to be negligible in effect. A suitable correlation was found to exist between the calcium oxide and sulfur trioxide content and the maximum temperature of the fly ash temperature results.


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# Evaluation of Fly Ash Quality Control Tools

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June 2010

# ABSTRACT

Many entities currently use fly ash in portland cement concrete (PCC) pavements and structures. Although the body of knowledge is great concerning the use of fly ash, several projects per year are subject to poor performance where fly ash is named as the culprit. Generally the "bad" projects arise due to one of two common errors:

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# IMPLEMENTATION STATEMENT

Problems that arise during construction are generally due to one of three problems including: materials, construction methods or technique, and environment. The results of this study were to gain an understanding of and characterize the as-delivered class C fly ash in Louisiana. The characterization of the class C fly ash provided an excellent record of variation by source of chemistry and set times. For each source, however, this characterization could not be linked to the set times of class C fly ash. It is significant to note that the variations of fly ash as measured in this report are not an issue and no change in specifications is recommended. The current practice of monitoring the daily set time with a penetrometer in the field is considered a “best practice” and should be continued as a method to identify environmental and material variations in products containing portland cement and fly ash blends.

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# INTRODUCTION

Many entities currently use fly ash in PCC pavements and structures. Although the body of knowledge is great concerning the use of fly ash, several projects per year are subject to poor performance where fly ash is named as the culprit. Generally the "bad" projects arise due to:

1. Poor understanding of how fly ash affects concrete pavement construction and performance or
2. A switch of fly ash sources midstream during the construction project.

Although there may be several "bad" projects per year, there is general agreement that the use of class C fly ash has the following effects on concrete:

1. Improved workability and finishability.
2. Increased time of setting and has caused unpredictable change in time between initial and final set. (This is of particular concern for saw cutting operations.) The use of class C fly ash has also shown false or flash set tendencies in field construction operations.
3. Despite early strength reduction, beyond 7 days concrete incorporating fly ash tend to show increased overall strengths over portland cement concrete.
4. The use of fly ash has been shown to reduce early rate of heat generation.
5. Permeability is reduced in mature concrete and resistance to sulfate and chloride attack is improved.
6. Freeze thaw resistance, modulus of elasticity, and resistance to de-icing salts are all about the same as in ordinary portland cement concrete.
7. The use of fly ash and other Supplementary Cementitious Materials (SCMs) in concrete helps reduce permeability and thus reduces chloride penetration leading to reduced corrosion of reinforcing steel.

At the time of this report, there are pending decisions by the Environmental Protection Agency (EPA) that may have a large impact on the use of fly ash by LADOTD. It is anticipated that if the EPA moves forward with dual classification or other similar legislation, the chemical composition and cost of the resulting fly ash may change, and the results of this study may need to be re-visited.

## Literature Review

The majority of electricity produced in the United States is produced from the combustion of coal at coal-fired utilities. As a result, over 117 million tons of coal combustion byproducts are produced per year [*1*]. The American Coal Ash Association (ACAA) estimates that fly

ash comprise 68 million tons. The 68 million tons is broken down into the following categories and tonnages [*1*]:

- Bottom ash is approximately 18.7 million tons;
- Boiler slag totals approximately 2.5 million tons; and
- Other byproducts are approximated at 24.8 million tons.

The ACAA states that fly ash use continually grows, but less than 32 percent of coal combustion byproducts are recycled each year leading to a sluice pond or landfill disposal practices [*1*]. Of the fly ash being recycled, the widest application is as a partial replacement of cement in portland cement concrete.

**Chemical Properties and Reaction Mechanisms of Self-Cementing Fly Ash**

ASTM C618 [Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete] defines fly ash as the fine residue produced from the burning of ground or powdered coal [*2*]. Fly ash is collected from the flu gas of coal-fired boilers by the means of an electrostatic precipitator or bag house. Fly ash color may vary from tan to gray [*3*]. Self-cementing fly ash is produced from the burning of low sulfur, subbituminous, and lignite coals. Fly ash particles are typically spherical in nature and contain some crystalline as well as carbonaceous matter [*3, 4*]. Misra noted that a large percentage of fly ash is in the form of silica, alumina, ferric oxide, and calcium oxide [*3*]. Table 1 shows typical class C fly ash composition. ASTM C618 chemical requirements are also shown in Table 1.

ASTM C618 states, “A pozzolan is a material rich in silica and alumina that has little or non self-cementing properties, but will, in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties [*2*].”

Research states that the pozzolinity of fly ash is mainly dependent upon the fineness of the ash, amounts of silica and alumina, and the presence of moisture and free lime [*3, 4*]. Winkerton and Pamukcu also state that density, amount of carbon, temperature, and age also affect the rate of pozzolanic reaction [*5*].

**Table 1**
**Typical chemical composition of a class C fly ash and ASTM C 618 chemical requirements for a class C fly ash**

| Oxide | Self Cementing Fly Ash (% of Total Weight) | ASTM C 618 | LADOTD (AASHTO M295) |
|---|---|---|---|
| $SiO_2$ | 20-40 | Summation | Summation |
| $Al_2O_2$ | 10-30 | between 50% and | between 50% and |
| $FeO_3$ | 3-10 | 70% | 70% |
| CaO | 10-32 | | |
| MgO | 0.8-8 | | |
| $Na_2O$ | 0.5-6 | | |
| $K_2O$ | 0.5-4 | | |
| $TiO_2$ | 0.5-2 | | |
| $SO_3$ | 1-8 | Maximum of 5% | Maximum of 5% |
| LOI | 0-3 | Maximum of 5% | Maximum of 5% |

# OBJECTIVE

The objective of this research was to identify tools available for QC of as delivered class C fly ash. The main focus of the research was to identify penetration type devices and test procedures including the Iowa Set Time Test, Gillmore needle, and Vicat needle. Another focus of the investigation was the quick heat generation index test.

# SCOPE

For the first objective, three penetration type test devices were investigated including the Vicat needle, Gillmore needle, and pocket penetrometer. Class C fly ash samples were obtained from about 10 sources available to Louisiana Department of Transportation and Development (LADOTD). Statistical analysis was completed on the results from each bucket as well as each source.

The second objective was to indentify if the quick heat generation test can identify small changes in class C fly ash whether a change in chemistry or a physical change in the fly ash fineness. Statistical modeling was used to determine if a relationship existed between the various initial and final set times and the maximum temperature of the fly ash paste and the fly ash chemistry and fineness.

A third objective was added during the course of the study to examine the set time effects when fly ash with high variations in set time are added to cements with a 50 percent addition rate.

# METHODOLOGY

The methodology is divided into four sections. The first section details the chemical characterization of the class C fly ash. The second section discusses the test methods used for determining the set time of the fly ash samples; the third section details the coffee cup, or quick calorimetry, test; and the fourth section details the statistical analysis.

Each of the 10 sources of class C fly ash were tested for chemistry, set time, and coffee cup for two samples per week for a period of 10 weeks totaling 20 samples per source, more or less. The non-combined samples were taken from production by the ash companies and delivered to the LTRC for testing.

## Chemical Characterization

Each of the buckets of fly ash was chemically characterized according to ASTM C618 [Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete] [*2*]. This work was completed by the materials section of LADOTD. Results included fineness and chemical composition.

## Laboratory Determination of Set Time

The laboratory determination of set time involved three tests. The three tests included a modified ASTM C191 [Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle], modified ASTM C266 [Standard Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles], and the Iowa Set Time Test [*6, 7*].

ASTM C 191 and ASTM C 266 were modified to allow determination of set time of a class C fly ash [*6, 7*]. The test procedure was modified by changing the specified water content to a water content of 27.5 percent or a water/fly ash ratio of 0.275. Without this change, the fly ash would have set too quick to determine anything of value.

The Iowa Set Time Test procedure is as follows:

1. Weigh out approximately 500 grams of fly ash.
2. Weigh the proper amount of water for 27.5 percent water content.
3. Mix with a mixer that conforms to ASTM C305 on speed one for 10 seconds, and then switch to speed two and mix for 50 seconds using a wire whip [*8*].
4. Spread mixture evenly in a suitable size container and determine the penetration resistance of the mixture about every 5 minutes using a pocket penetrometer.
5. Plot the elapsed time versus the penetration resistance. Initial set is determined to be

the time at which the material exerts some penetration resistance, and the final set is determined to be when the penetration resistance is 4.5 tons per square foot.

Figure 1 and Figure 2 show the mixer and the Vicat needle apparatus used for determination of set time, respectively. Figure 3 and Figure 4 show the Gillmore needle and pocket penetrometer, respectively.

**Figure 1**
**Mixer used for the study**

**Figure 2**
**Vicat needle**

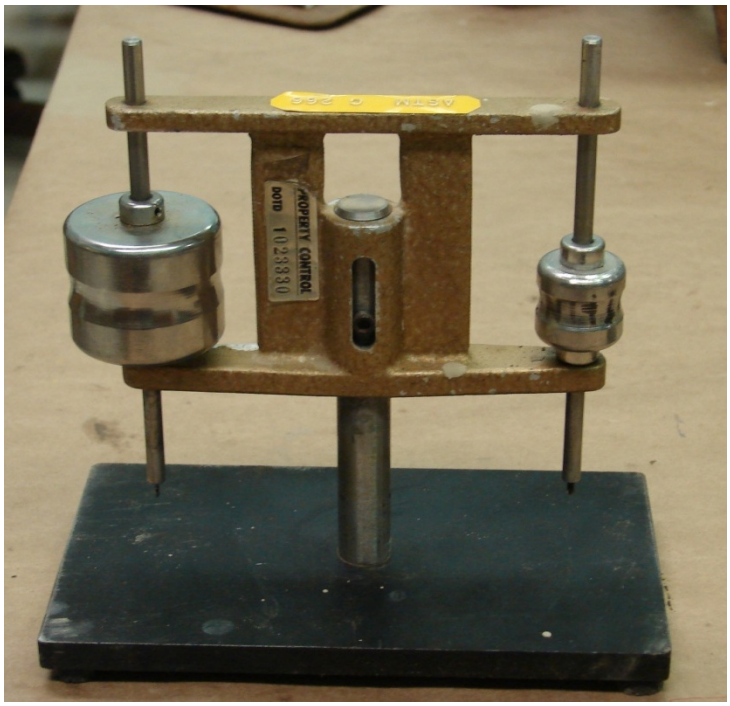


**Figure 3**
**Gillmore needle**

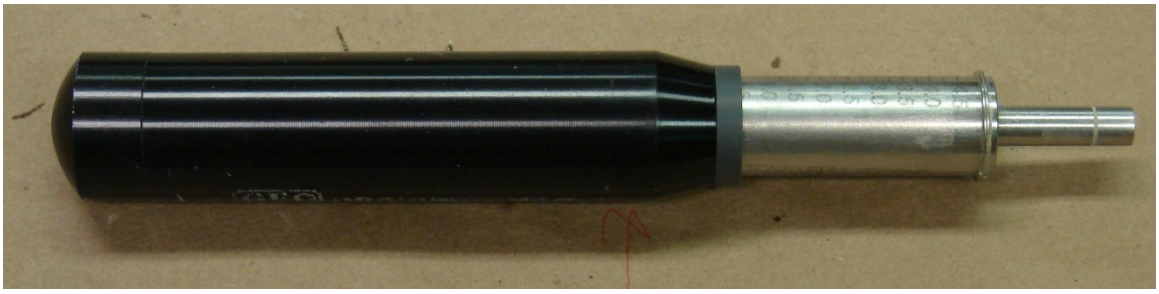

**Figure 4**
**Pocket penetrometer**

## Coffee Cup Test

The coffee cup test is a quick heat generation index test that determines the heat liberated due to hydration of cementitious materials. The test procedure is outlined below. More information on the coffee cup test can be found in Rupnow and Sandberg [*9, 10*]. Figure 5 shows the equipment used for the coffee cup test.

1. Obtain representative sample of fly ash and record the material temperature.
2. Bring the water and fly ash to 70˚F ± 0.5˚F.
3. Mix about 500 g of fly ash with 200 g of water:
    a. Vigorously shake the mixture for about 20 seconds in a 1-liter Nalgene bottle.
    b. Pour the paste slurry mixture into a styrofoam coffee cup
    c. Insert the t-wire thermocouple (temperature readings every 5 seconds) attached to a temperature data logger.
    d. Conduct the test until final set.
4. Download and plot the results with temperature on the y-axis and time on the x-axis.

**Figure 5**
**Laboratory test equipment used to conduct the quick heat generation test**

## Statistical Analysis

Statistical analysis of the results was completed using Excel and JMP 8.0 [*11*]. Excel was used to determine averages and standard deviations, and JMP 8.0 was initially used to determine significance both between samples within a source and between sources. JMP was also used to model the results to determine if a relationship existed between the set time, temperature data, fly ash chemistry, and fineness.

An attempt was made to use the t-test to compare the results between buckets from the same source. The results showed the variability of each bucket was too small, which led to each bucket being significantly different from the others for most cases. The research team then used ASTM E178 [Standard Practice for Dealing with Outlying Observations] to determine outliers [*12*].

# DISCUSSION OF RESULTS

This section is divided into four distinct sections. The first section details the chemical and physical characterization results; the second section details the set time results; the third section details the coffee cup results; and the fourth section details the modeling and statistical analysis results.

## Chemical Characterization

For each bucket from each source, a chemical analysis was conducted to determine the total oxides, silicon dioxide ($SiO_2$), sulfur trioxide ($SO_3$), calcium oxide (CaO), loss on ignition (LOI), and the fineness (percentage retained on the 325 sieve). Table 2 to Table 12 shows the chemical analysis results and fineness results for Sources 1 – 11. Note that not all sources had the same number of samples. Even though some sources provided more than the 20 samples, it was decided to test all that were provided to include in the test matrix.

**Table 2**
**Chemical characterization results for Source 1**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 58.0 | 33.0 | 25.0 | 1.8 | 6.0 | 29.4 | 1.1 | 0.1 | 19.0 |
| 2 | 59.9 | 33.4 | 26.5 | 2.4 | 0.0 | 32.3 | 1.1 | 0.2 | 19.4 |
| 3 | 56.9 | 31.1 | 25.8 | 2.4 | 6.1 | 29.6 | 0.1 | 0.2 | 15.2 |
| 4 | 58.6 | 33.1 | 25.4 | 1.9 | 6.1 | 28.6 | 1.2 | 0.2 | 19.0 |
| 5 | 58.6 | 33.4 | 25.2 | 1.9 | 6.0 | 28.9 | 0.9 | 0.2 | 17.7 |
| 6 | 58.6 | 32.3 | 26.3 | 1.9 | 6.0 | 28.4 | 0.7 | 0.2 | 14.4 |
| 7 | 59.3 | 32.6 | 26.8 | 1.9 | 5.9 | 28.6 | 0.9 | 0.2 | 16.7 |
| 8 | 59.0 | 32.5 | 26.5 | 1.9 | 5.6 | 28.6 | 0.8 | 0.1 | 16.4 |
| 9 | 58.1 | 31.4 | 26.7 | 2.1 | 5.9 | 28.9 | 1.0 | 0.3 | 15.6 |
| 10 | 58.4 | 33.7 | 24.8 | 2.1 | 5.9 | 28.5 | 0.9 | 0.2 | 19.6 |
| 11 | 55.8 | 30.3 | 25.5 | 1.6 | 4.2 | 33.3 | 0.9 | 0.2 | 16.0 |
| 12 | 56.4 | 30.0 | 26.4 | 1.5 | 4.2 | 33.2 | 0.4 | 0.2 | 14.3 |
| 13 | 58.7 | 31.3 | 27.4 | 1.2 | 3.9 | 32.0 | 0.9 | 0.2 | 16.8 |
| 14 | 57.1 | 31.0 | 26.1 | 1.3 | 4.2 | 33.3 | 0.9 | 0.2 | 18.2 |
| 15 | 58.9 | 32.5 | 26.4 | 2.3 | 6.0 | 28.4 | 0.9 | 0.0 | 15.2 |
| 16 | 58.3 | 32.2 | 26.0 | 2.2 | 6.1 | 29.0 | 1.2 | 0.0 | 19.8 |
| 17 | 55.6 | 31.6 | 24.0 | 2.4 | 6.7 | 30.7 | 1.3 | 0.2 | 15.7 |
| 18 | 58.6 | 32.0 | 26.6 | 2.2 | 6.0 | 28.6 | 1.3 | 0.2 | 16.1 |
| 19 | 56.9 | 30.3 | 26.5 | 2.8 | 6.2 | 29.6 | 1.1 | 0.2 | 13.4 |
| 20 | 58.3 | 32.1 | 26.2 | 2.6 | 6.0 | 28.8 | 1.1 | 0.2 | 15.1 |
| Average | 58.0 | 32.0 | 26.0 | 2.0 | 5.4 | 29.9 | 0.9 | 0.2 | 16.7 |
| Stdev | 1.2 | 1.1 | 0.8 | 0.4 | 1.5 | 1.8 | 0.3 | 0.1 | 1.9 |
| COV | 2.0 | 3.4 | 3.1 | 20.6 | 27.9 | 6.1 | 30.9 | 40.9 | 11.6 |

Note the results tend to show differences between sources. Of particular interest are the standard deviation results. Note that the standard deviations are very low for most measured properties. The coefficient of variation tends to be a little higher for the moisture content, LOI, and $SO_3$. This shows that, for the most part, the fly ashes tested are generally the same with little variation between each of the buckets for each source over about a 10-week period.

**Table 3**
**Chemical characterization results for Source 2**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 58.5 | 32.9 | 25.7 | 2.2 | 5.8 | 28.6 | 0.4 | 0.1 | 15.7 |
| 2 | 49.4 | 37.3 | 12.1 | 2.7 | 7.1 | 34.4 | 0.9 | 0.1 | 16.8 |
| 3 | 57.0 | 31.2 | 25.8 | 2.5 | 6.1 | 29.4 | 0.4 | 0.2 | 15.7 |
| 4 | 57.0 | 31.0 | 26.0 | 2.4 | 5.9 | 29.9 | 0.3 | 0.2 | 16.1 |
| 5 | 58.4 | 32.4 | 26.0 | 2.0 | 6.0 | 28.7 | 0.2 | 0.2 | 16.6 |
| 6 | 58.8 | 32.5 | 26.3 | 2.2 | 5.9 | 28.1 | 0.3 | 0.2 | 15.8 |
| 7 | 58.6 | 32.8 | 25.8 | 2.5 | 5.7 | 28.3 | 0.3 | 0.2 | 16.6 |
| 8 | 59.0 | 32.4 | 26.5 | 2.9 | 5.6 | 27.9 | 0.6 | 0.1 | 15.7 |
| 9 | 58.6 | 32.5 | 26.1 | 2.4 | 6.0 | 28.5 | 0.3 | 0.2 | 16.7 |
| 10 | 57.8 | 32.4 | 25.4 | 2.3 | 6.1 | 29.1 | 0.8 | 0.1 | 17.9 |
| 11 | 56.6 | 31.3 | 25.3 | 1.5 | 4.3 | 33.7 | 0.7 | 0.1 | 15.6 |
| 12 | 55.7 | 29.9 | 25.8 | 1.9 | 4.1 | 33.6 | 0.6 | 0.1 | 18.0 |
| 13 | 56.2 | 30.2 | 26.0 | 1.7 | 1.7 | 33.6 | 0.4 | 0.2 | 13.8 |
| 14 | 46.8 | 33.6 | 13.2 | 2.1 | 4.8 | 41.3 | 0.3 | 0.3 | 14.8 |
| 15 | 57.8 | 31.8 | 25.9 | 2.7 | 6.1 | 29.3 | 0.5 | 0.0 | 17.7 |
| 16 | 59.0 | 34.4 | 24.6 | 2.5 | 5.9 | 28.5 | 0.5 | 0.0 | 16.2 |
| 17 | 56.8 | 30.8 | 25.9 | 3.0 | 6.4 | 29.5 | 0.4 | 0.2 | 17.1 |
| 18 | 57.0 | 31.0 | 26.0 | 2.6 | 6.2 | 29.6 | 0.7 | 0.1 | 15.5 |
| 19 | 57.5 | 31.7 | 25.8 | 2.6 | 6.1 | 29.4 | 0.5 | 0.1 | 14.3 |
| 20 | 59.2 | 32.5 | 26.7 | 2.6 | 5.8 | 28.2 | 0.4 | 0.2 | 13.6 |
| Average | 56.8 | 32.2 | 24.5 | 2.4 | 5.6 | 30.5 | 0.5 | 0.1 | 16.0 |
| Stdev | 3.2 | 1.6 | 4.1 | 0.4 | 1.1 | 3.3 | 0.2 | 0.1 | 1.2 |
| COV | 5.6 | 5.1 | 16.7 | 16.2 | 20.4 | 10.8 | 39.8 | 52.4 | 7.8 |

**Table 4**
**Chemical characterization results for Source 3**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 65.6 | 38.2 | 27.8 | 0.0 | 4.5 | 24.9 | 0.3 | 0.1 | 15.4 |
| 2 | 65.5 | 37.4 | 28.0 | 1.2 | 4.5 | 24.4 | 0.2 | 0.2 | 15.0 |
| 3 | 65.4 | 36.2 | 29.2 | 1.1 | 4.2 | 24.6 | 0.3 | 0.2 | 14.4 |
| 4 | 65.0 | 37.1 | 27.9 | 0.0 | 4.7 | 25.6 | 0.2 | 0.2 | 14.7 |
| 5 | 64.8 | 36.9 | 27.8 | 1.2 | 4.5 | 24.9 | 0.2 | 0.2 | 14.8 |
| 6 | 64.8 | 37.1 | 27.7 | 1.2 | 4.5 | 24.9 | 0.2 | 0.1 | 15.0 |
| 7 | 63.6 | 36.7 | 26.9 | 0.8 | 3.0 | 28.9 | 0.4 | 0.1 | 14.9 |
| 8 | 64.0 | 35.6 | 28.4 | 0.0 | 3.1 | 29.2 | 0.4 | 0.1 | 13.9 |
| 9 | 65.3 | 36.7 | 28.6 | 0.0 | 3.1 | 28.4 | 0.3 | 0.1 | 14.1 |
| 10 | 65.3 | 36.7 | 28.6 | 0.7 | 2.9 | 27.2 | 0.3 | 0.1 | 14.7 |
| 11 | 63.9 | 36.1 | 27.9 | 0.7 | 0.0 | 28.5 | 0.2 | 0.2 | 13.1 |
| 12 | 65.9 | 38.0 | 27.9 | 1.3 | 4.6 | 24.4 | 0.3 | 0.0 | 13.5 |
| 13 | 64.6 | 36.6 | 28.0 | 1.3 | 5.1 | 24.8 | 0.4 | 0.0 | 13.8 |
| 14 | 67.2 | 38.8 | 28.4 | 0.0 | 4.5 | 24.4 | 0.3 | 0.0 | 14.4 |
| 15 | 68.3 | 40.7 | 27.7 | 1.0 | 4.3 | 22.4 | 0.3 | 0.0 | 15.9 |
| 16 | 35.6 | 35.6 | 27.0 | 1.6 | 4.8 | 26.5 | 0.3 | 0.0 | 16.9 |
| 17 | 65.0 | 36.6 | 28.3 | 1.4 | 4.6 | 24.9 | 0.3 | 0.0 | 14.7 |
| 18 | 67.7 | 38.5 | 29.2 | 0.0 | 4.5 | 23.7 | 0.3 | 0.0 | 13.7 |
| 19 | 66.4 | 37.9 | 28.5 | 1.2 | 4.6 | 23.9 | 0.3 | 0.0 | 15.3 |
| 20 | 64.8 | 36.0 | 28.8 | 1.3 | 4.5 | 25.1 | 0.2 | 0.0 | 15.6 |
| Average | 63.9 | 37.2 | 28.1 | 0.8 | 4.0 | 25.6 | 0.3 | 0.1 | 14.7 |
| Stdev | 6.8 | 1.2 | 0.6 | 0.6 | 1.1 | 1.9 | 0.1 | 0.1 | 0.9 |
| COV | 10.6 | 3.3 | 2.2 | 72.4 | 28.6 | 7.4 | 23.5 | 104.2 | 6.1 |

**Table 5**
**Chemical characterization results for Source 4**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 60.2 | 33.1 | 27.1 | 1.8 | 5.4 | 27.3 | 0.4 | 0.1 | 14.9 |
| 2 | 61.4 | 34.9 | 26.5 | 1.6 | 5.6 | 26.3 | 0.5 | 0.1 | 16.7 |
| 3 | 61.5 | 35.1 | 26.3 | 1.5 | 5.2 | 26.5 | 1.0 | 0.1 | 16.6 |
| 4 | 60.9 | 34.1 | 26.9 | 1.6 | 5.4 | 26.6 | 0.6 | 0.2 | 15.9 |
| 5 | 61.8 | 34.4 | 27.4 | 1.4 | 5.6 | 27.0 | 0.4 | 0.1 | 16.7 |
| 6 | 60.7 | 34.0 | 26.7 | 1.7 | 5.6 | 26.7 | 0.3 | 0.1 | 15.0 |
| 7 | 62.4 | 36.1 | 26.3 | 2.0 | 5.2 | 25.0 | 0.2 | 0.2 | 14.6 |
| 8 | 62.9 | 36.9 | 26.0 | 1.5 | 5.5 | 24.8 | 0.3 | 0.2 | 16.7 |
| 9 | 61.3 | 34.7 | 26.5 | 1.5 | 5.6 | 26.1 | 0.3 | 0.1 | 15.3 |
| 10 | 59.7 | 33.5 | 26.2 | 1.1 | 4.1 | 30.8 | 0.3 | 0.2 | 15.7 |
| 11 | 61.9 | 34.7 | 27.2 | 0.3 | 3.9 | 30.4 | 0.3 | 0.2 | 13.5 |
| 12 | 60.7 | 33.2 | 27.5 | 0.8 | 3.9 | 30.9 | 0.5 | 0.1 | 13.7 |
| 13 | 60.6 | 33.3 | 27.2 | 0.0 | 3.9 | 31.0 | 0.3 | 0.2 | 15.9 |
| 14 | 62.0 | 34.8 | 27.1 | 1.5 | 5.4 | 26.2 | 0.5 | 0.0 | 15.1 |
| 15 | 61.7 | 35.0 | 26.7 | 1.4 | 5.6 | 26.4 | 0.5 | 0.0 | 14.8 |
| 16 | 62.5 | 34.9 | 27.7 | 1.6 | 5.8 | 25.3 | 0.5 | 0.0 | 14.3 |
| 17 | 60.7 | 32.0 | 28.7 | 1.8 | 5.2 | 27.2 | 0.3 | 0.1 | 13.0 |
| 18 | 61.2 | 33.4 | 27.9 | 1.9 | 5.4 | 26.4 | 0.3 | 0.0 | 11.7 |
| 19 | 62.7 | 35.1 | 27.6 | 1.6 | 5.5 | 24.9 | 0.3 | 0.0 | 12.9 |
| 20 | 63.2 | 35.6 | 27.7 | 1.2 | 5.6 | 24.7 | 0.4 | 0.0 | 14.5 |
| Average | 61.5 | 34.4 | 27.1 | 1.4 | 5.2 | 27.0 | 0.4 | 0.1 | 14.9 |
| Stdev | 0.9 | 1.1 | 0.7 | 0.5 | 0.6 | 2.1 | 0.2 | 0.1 | 1.4 |
| COV | 1.5 | 3.3 | 2.5 | 36.5 | 12.5 | 7.7 | 42.5 | 79.5 | 9.4 |

**Table 6**
**Chemical characterization results for Source 5**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 62.0 | 35.7 | 26.3 | 2.0 | 5.3 | 26.1 | 0.5 | 0.2 | 17.6 |
| 2 | 60.7 | 33.7 | 27.1 | 2.5 | 5.3 | 26.9 | 0.5 | 0.2 | 12.7 |
| 3 | 60.4 | 32.7 | 27.6 | 1.5 | 3.6 | 30.4 | 0.2 | 0.2 | 18.4 |
| 4 | 61.0 | 33.7 | 27.3 | 1.5 | 3.4 | 29.8 | 0.7 | 0.2 | 20.9 |
| 5 | 58.9 | 32.5 | 26.4 | 1.7 | 3.9 | 31.1 | 0.9 | 0.2 | 18.6 |
| 6 | 60.4 | 33.4 | 27.0 | 1.7 | 0.0 | 32.7 | 0.4 | 0.2 | 21.3 |
| 7 | 62.6 | 36.6 | 26.0 | 2.1 | 5.2 | 25.8 | 0.5 | 0.0 | 18.9 |
| 8 | 61.9 | 36.2 | 25.7 | 2.5 | 5.2 | 26.0 | 0.5 | 0.0 | 19.9 |
| 9 | 61.1 | 35.0 | 26.1 | 2.4 | 5.7 | 26.5 | 0.5 | 0.0 | 20.2 |
| 10 | 62.1 | 35.1 | 27.0 | 2.5 | 5.0 | 25.9 | 0.5 | 0.0 | 14.9 |
| 11 | 63.8 | 36.1 | 27.7 | 2.3 | 4.6 | 25.5 | 0.6 | 0.0 | 20.0 |
| 12 | 61.4 | 34.5 | 26.8 | 2.7 | 5.1 | 26.7 | 0.3 | 0.0 | 15.4 |
| 13 | 62.2 | 34.8 | 27.4 | 2.5 | 4.8 | 26.3 | 0.3 | 0.0 | 21.0 |
| 14 | 62.8 | 35.6 | 27.2 | 2.4 | 4.9 | 25.7 | 0.0 | 0.0 | 15.6 |
| 15 | 58.1 | 32.5 | 25.7 | 3.0 | 5.4 | 28.4 | 0.4 | 0.2 | 20.4 |
| 16 | 60.2 | 33.1 | 27.1 | 2.4 | 5.8 | 26.4 | 0.4 | 0.1 | 16.4 |
| 17 | 64.4 | 37.0 | 27.4 | 1.8 | 4.8 | 24.0 | 0.2 | 0.2 | 18.3 |
| 18 | 62.2 | 35.2 | 26.9 | 2.3 | 5.0 | 25.8 | 0.6 | 0.2 | 18.1 |
| 19 | 65.4 | 36.2 | 29.2 | 1.9 | 4.6 | 24.1 | 0.4 | 0.2 | 15.3 |
| 20 | 59.8 | 33.8 | 26.0 | 2.4 | 5.8 | 27.3 | 0.3 | 0.2 | 15.5 |
| Average | 61.6 | 34.7 | 26.9 | 2.2 | 4.7 | 27.1 | 0.4 | 0.1 | 18.0 |
| Stdev | 1.8 | 1.4 | 0.8 | 0.4 | 1.3 | 2.3 | 0.2 | 0.1 | 2.5 |
| COV | 2.9 | 4.1 | 3.1 | 18.6 | 27.4 | 8.4 | 44.9 | 85.9 | 13.7 |

**Table 7**
**Chemical characterization results for Source 6**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 60.1 | 33.1 | 26.9 | 1.9 | 5.1 | 28.2 | 0.8 | 0.2 | 19.4 |
| 2 | 60.7 | 34.0 | 26.7 | 1.8 | 5.3 | 28.2 | 0.2 | 0.2 | 22.6 |
| 3 | 61.5 | 34.4 | 27.2 | 1.5 | 5.3 | 26.8 | 0.7 | 0.1 | 17.2 |
| 4 | 61.7 | 35.2 | 26.6 | 1.6 | 5.2 | 26.5 | 0.9 | 0.2 | 17.7 |
| 5 | 59.3 | 32.9 | 26.4 | 1.8 | 5.5 | 28.8 | 1.2 | 0.0 | 13.6 |
| 6 | 59.0 | 32.9 | 26.1 | 1.8 | 5.4 | 28.8 | 1.1 | 0.1 | 13.8 |
| 7 | 57.8 | 31.4 | 26.4 | 2.2 | 5.2 | 30.1 | 1.1 | 0.0 | 14.1 |
| 8 | 56.9 | 31.9 | 25.0 | 2.2 | 5.1 | 31.1 | 1.3 | 0.0 | 13.3 |
| 9 | 57.7 | 31.7 | 26.0 | 2.3 | 5.1 | 30.2 | 0.9 | 0.1 | 17.5 |
| 10 | 57.7 | 31.7 | 26.0 | 2.4 | 5.1 | 30.0 | 0.7 | 0.2 | 15.2 |
| 11 | 58.6 | 33.3 | 25.3 | 2.1 | 5.2 | 29.4 | 0.8 | 0.1 | 15.5 |
| 12 | 59.3 | 33.2 | 26.2 | 2.0 | 5.2 | 28.8 | 0.9 | 0.1 | 15.8 |
| 13 | 59.4 | 32.8 | 26.6 | 1.9 | 5.4 | 28.5 | 0.9 | 0.1 | 17.0 |
| 14 | 58.8 | 32.4 | 26.4 | 2.0 | 5.4 | 28.9 | 1.2 | 0.1 | 17.1 |
| 15 | 57.6 | 32.0 | 25.5 | 2.3 | 5.5 | 29.8 | 0.8 | 0.1 | 16.0 |
| 16 | 57.0 | 32.1 | 24.9 | 2.3 | 5.4 | 30.4 | 0.8 | 0.1 | 17.9 |
| 17 | 58.3 | 33.0 | 25.4 | 2.2 | 4.9 | 29.8 | 0.8 | 0.1 | 15.8 |
| 18 | 59.0 | 33.6 | 25.4 | 2.2 | 5.0 | 29.7 | 0.8 | 0.1 | 15.7 |
| 19 | 60.0 | 33.2 | 26.8 | 2.0 | 5.0 | 28.7 | 0.8 | 0.1 | 17.0 |
| 20 | 60.3 | 33.7 | 26.6 | 1.8 | 5.0 | 28.0 | 0.9 | 0.1 | 17.3 |
| Average | 59.0 | 32.9 | 26.1 | 2.0 | 5.2 | 29.0 | 0.9 | 0.1 | 16.5 |
| Stdev | 1.4 | 1.0 | 0.7 | 0.2 | 0.2 | 1.2 | 0.2 | 0.1 | 2.2 |
| COV | 2.3 | 3.0 | 2.5 | 12.4 | 3.4 | 4.0 | 26.7 | 57.6 | 13.1 |

**Table 8**
**Chemical characterization results for Source 7**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 62.9 | 35.1 | 27.8 | 1.3 | 4.7 | 27.4 | 0.5 | 0.2 | 18.4 |
| 2 | 63.9 | 35.6 | 28.3 | 1.3 | 4.8 | 26.4 | 0.5 | 0.2 | 13.2 |
| 3 | 60.9 | 33.0 | 27.9 | 1.8 | 4.6 | 26.4 | 0.5 | 0.2 | 15.0 |
| 4 | 61.5 | 34.3 | 27.2 | 0.9 | 3.1 | 30.2 | 0.4 | 0.2 | 19.3 |
| 5 | 63.3 | 34.0 | 29.2 | 1.1 | 3.0 | 28.4 | 0.3 | 0.3 | 18.2 |
| 6 | 61.0 | 32.5 | 28.5 | 0.0 | 3.4 | 31.5 | 0.3 | 0.2 | 18.1 |
| 7 | 62.4 | 33.3 | 29.1 | 1.5 | 5.1 | 26.6 | 0.6 | 0.0 | 17.7 |
| 8 | 62.8 | 35.1 | 27.8 | 1.4 | 4.9 | 26.8 | 0.7 | 0.0 | 15.7 |
| 9 | 63.5 | 34.6 | 28.9 | 1.5 | 5.1 | 25.7 | 0.5 | 0.0 | 14.7 |
| 10 | 62.7 | 33.7 | 29.0 | 1.7 | 4.7 | 26.5 | 0.7 | 0.1 | 16.0 |
| 11 | 63.9 | 25.4 | 28.6 | 1.4 | 4.5 | 26.2 | 0.5 | 0.1 | 20.1 |
| 12 | 63.3 | 35.6 | 27.6 | 1.5 | 4.7 | 26.3 | 0.6 | 0.2 | 20.8 |
| 13 | 58.5 | 31.4 | 27.0 | 2.2 | 4.8 | 29.8 | 0.6 | 0.1 | 14.0 |
| 14 | 66.2 | 38.8 | 27.4 | 1.3 | 4.2 | 24.1 | 0.3 | 0.2 | 15.9 |
| 15 | 62.0 | 35.8 | 26.3 | 1.7 | 4.6 | 27.3 | 0.5 | 0.2 | 20.0 |
| 16 | 64.8 | 37.8 | 27.0 | 1.3 | 5.1 | 24.3 | 0.6 | 0.2 | 20.9 |
| 17 | 59.8 | 33.4 | 26.4 | 1.7 | 4.8 | 28.9 | 0.8 | 0.1 | 15.5 |
| 18 | 63.0 | 35.9 | 27.1 | 1.5 | 5.0 | 25.7 | 0.7 | 0.1 | 14.7 |
| 19 | 62.3 | 35.9 | 26.4 | 1.4 | 5.0 | 27.2 | 0.4 | 0.1 | 14.1 |
| Average | 62.6 | 34.3 | 27.8 | 1.4 | 4.5 | 27.1 | 0.5 | 0.1 | 17.0 |
| Stdev | 1.7 | 2.8 | 0.9 | 0.4 | 0.7 | 1.9 | 0.1 | 0.1 | 2.5 |
| COV | 2.8 | 8.1 | 3.4 | 31.4 | 14.4 | 7.0 | 27.5 | 58.9 | 14.7 |

**Table 9**
**Chemical characterization results for Source 8**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 72.9 | 30.8 | 42.1 | 1.1 | 3.7 | 18.9 | 0.4 | 0.2 | 13.9 |
| 2 | 66.4 | 36.6 | 29.7 | 1.2 | 4.4 | 23.7 | 0.4 | 0.1 | 12.9 |
| 3 | 63.8 | 36.2 | 27.6 | 1.2 | 5.0 | 25.2 | 0.1 | 0.2 | 13.9 |
| 4 | 63.8 | 36.5 | 27.3 | 1.2 | 5.0 | 25.4 | 0.4 | 0.1 | 13.7 |
| 5 | 65.0 | 38.2 | 26.8 | 1.1 | 4.9 | 24.4 | 0.2 | 0.1 | 13.6 |
| 6 | 64.3 | 38.4 | 25.9 | 1.2 | 4.9 | 25.1 | 0.4 | 0.1 | 13.3 |
| 7 | 64.2 | 34.4 | 29.8 | 0.0 | 0.0 | 31.4 | 0.3 | 0.1 | 13.6 |
| 8 | 64.0 | 35.2 | 28.8 | 0.0 | 3.2 | 28.4 | 0.3 | 0.1 | 10.2 |
| 9 | 65.8 | 36.9 | 28.9 | 0.0 | 3.2 | 27.3 | 0.3 | 0.1 | 15.5 |
| 10 | 56.8 | 43.0 | 13.9 | 0.0 | 4.2 | 34.1 | 0.5 | 0.1 | 15.7 |
| 11 | 70.5 | 41.8 | 28.7 | 0.0 | 4.4 | 21.1 | 0.4 | 0.0 | 18.1 |
| 12 | 70.4 | 41.2 | 21.5 | 0.0 | 4.3 | 21.5 | 0.3 | 0.0 | 18.1 |
| 13 | 67.6 | 39.6 | 28.0 | 0.0 | 4.9 | 23.5 | 0.3 | 0.0 | 14.2 |
| 14 | 66.9 | 39.1 | 27.8 | 0.0 | 5.1 | 23.9 | 0.4 | 0.0 | 13.2 |
| 15 | 67.1 | 39.1 | 28.0 | 1.1 | 4.5 | 23.3 | 0.3 | 0.1 | 17.6 |
| 16 | 64.9 | 37.2 | 27.2 | 1.3 | 4.8 | 24.6 | 0.4 | 0.1 | 17.8 |
| 17 | 65.4 | 39.4 | 26.0 | 0.0 | 5.5 | 25.5 | 0.4 | 0.0 | 14.5 |
| 18 | 66.1 | 39.2 | 26.9 | 0.7 | 5.1 | 24.4 | 0.3 | 0.0 | 15.0 |
| 19 | 67.0 | 39.2 | 27.8 | 0.0 | 4.9 | 24.2 | 1.7 | 0.0 | 13.1 |
| 20 | 61.4 | 34.9 | 26.5 | 0.9 | 5.3 | 28.9 | 0.4 | 0.0 | 13.3 |
| 21 | 63.4 | 36.7 | 26.7 | 1.2 | 5.1 | 25.7 | 0.2 | 0.1 | 17.5 |
| 22 | 63.8 | 37.2 | 26.6 | 1.3 | 5.1 | 25.8 | 0.4 | 0.1 | 17.3 |
| 23 | 63.7 | 36.5 | 27.3 | 1.5 | 4.8 | 26.5 | 0.3 | 0.0 | 12.2 |
| 24 | 64.0 | 36.1 | 27.9 | 1.4 | 4.8 | 25.5 | 0.3 | 0.0 | 11.6 |
| 25 | 63.9 | 37.0 | 26.9 | 1.3 | 4.9 | 25.6 | 0.4 | 0.0 | 14.2 |
| 26 | 64.7 | 37.3 | 27.4 | 1.3 | 4.8 | 25.0 | 0.9 | 0.0 | 14.9 |
| Average | 65.3 | 37.6 | 27.4 | 0.7 | 4.5 | 25.3 | 0.4 | 0.1 | 14.6 |
| Stdev | 3.1 | 2.5 | 4.3 | 0.6 | 1.1 | 3.0 | 0.3 | 0.1 | 2.1 |
| COV | 4.7 | 6.7 | 15.6 | 83.1 | 24.0 | 12.0 | 72.2 | 103.6 | 14.4 |

**Table 10**
**Chemical characterization results for Source 9**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 60.3 | 33.8 | 26.5 | 1.8 | 5.1 | 28.5 | 0.6 | 0.0 | 16.9 |
| 2 | 59.9 | 33.8 | 26.1 | 1.8 | 5.0 | 28.8 | 0.5 | 0.0 | 16.8 |
| 3 | 58.8 | 33.2 | 25.7 | 1.9 | 5.1 | 29.7 | 0.4 | 0.0 | 16.9 |
| 4 | 58.9 | 32.9 | 26.0 | 1.8 | 5.2 | 29.7 | 0.6 | 0.0 | 16.0 |
| 5 | 59.2 | 33.2 | 26.1 | 1.9 | 5.1 | 29.4 | 0.5 | 0.0 | 16.4 |
| 6 | 59.2 | 33.2 | 26.0 | 1.9 | 5.2 | 29.3 | 0.5 | 0.0 | 16.9 |
| 7 | 59.7 | 34.1 | 25.6 | 2.1 | 5.1 | 28.8 | 0.5 | 0.0 | 17.3 |
| 8 | 59.6 | 34.5 | 25.1 | 1.9 | 5.4 | 28.5 | 0.5 | 0.1 | 15.5 |
| 9 | 58.9 | 33.6 | 25.4 | 2.0 | 5.1 | 29.5 | 0.8 | 0.1 | 17.3 |
| 10 | 60.5 | 34.0 | 26.5 | 1.6 | 5.3 | 28.1 | 0.6 | 0.1 | 17.0 |
| 11 | 66.1 | 39.9 | 26.2 | 1.8 | 0.0 | 27.3 | 0.7 | 0.0 | 17.3 |
| 12 | 58.2 | 32.9 | 25.3 | 2.3 | 5.3 | 29.4 | 0.6 | 0.0 | 17.4 |
| 13 | 60.2 | 34.3 | 25.9 | 1.4 | 5.3 | 29.1 | 0.5 | 0.0 | 12.5 |
| 14 | 58.5 | 32.6 | 25.9 | 1.9 | 5.4 | 29.5 | 0.5 | 0.0 | 13.5 |
| 15 | 59.6 | 33.0 | 26.6 | 1.9 | 5.1 | 28.8 | 0.4 | 0.0 | 17.6 |
| 16 | 58.2 | 33.4 | 24.8 | 2.2 | 4.9 | 30.1 | 0.5 | 0.0 | 17.5 |
| 17 | 58.1 | 32.2 | 26.0 | 1.9 | 5.1 | 30.4 | 0.5 | 0.0 | 14.3 |
| 18 | 59.3 | 33.3 | 26.1 | 1.6 | 5.3 | 29.1 | 0.4 | 0.0 | 11.5 |
| 19 | 58.9 | 32.8 | 26.1 | 1.7 | 5.4 | 29.3 | 0.5 | 0.0 | 12.6 |
| 20 | 58.3 | 32.2 | 26.1 | 1.8 | 5.5 | 29.6 | 0.4 | 0.0 | 13.9 |
| Average | 59.5 | 33.6 | 25.9 | 1.9 | 4.9 | 29.1 | 0.5 | 0.0 | 15.8 |
| Stdev | 1.7 | 1.6 | 0.5 | 0.2 | 1.2 | 0.7 | 0.1 | 0.0 | 2.0 |
| COV | 2.9 | 4.8 | 1.8 | 10.9 | 23.7 | 2.4 | 19.4 | 244.2 | 12.4 |

**Table 11**
**Chemical characterization results for Source 10**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 81.0 | 53.7 | 27.2 | 0.5 | 2.9 | 11.4 | 0.1 | 0.2 | 25.0 |
| 2 | 80.4 | 52.9 | 27.5 | 0.5 | 3.0 | 12.2 | 0.2 | 0.3 | 21.6 |
| 3 | 81.1 | 53.6 | 27.5 | 0.5 | 2.8 | 11.5 | 0.3 | 0.2 | 22.1 |
| 4 | 80.8 | 53.1 | 27.6 | 0.6 | 0.0 | 14.4 | 0.2 | 0.2 | 24.6 |
| 5 | 79.3 | 53.0 | 26.3 | 0.6 | 3.1 | 12.9 | 0.3 | 0.1 | 25.1 |
| 6 | 79.3 | 52.3 | 27.0 | 0.6 | 3.2 | 13.6 | 0.2 | 0.2 | 21.6 |
| 7 | 77.4 | 48.4 | 29.0 | 0.6 | 3.5 | 14.3 | 0.2 | 0.3 | 25.7 |
| 8 | 81.2 | 52.6 | 28.6 | 0.5 | 2.9 | 11.5 | 0.1 | 0.2 | 23.4 |
| 9 | 77.7 | 50.2 | 27.6 | 0.4 | 2.3 | 16.0 | 0.3 | 0.2 | 22.4 |
| 10 | 79.9 | 50.8 | 29.0 | 0.4 | 0.0 | 15.9 | 0.1 | 0.2 | 21.7 |
| 11 | 77.8 | 47.5 | 30.4 | 0.2 | 2.4 | 16.3 | 0.2 | 0.2 | 20.6 |
| 12 | 78.7 | 49.1 | 29.6 | 0.3 | 2.3 | 14.8 | 0.3 | 0.2 | 22.4 |
| 13 | 80.4 | 51.9 | 28.5 | 0.3 | 2.0 | 13.7 | 0.1 | 0.2 | 25.4 |
| 14 | 81.5 | 54.4 | 27.2 | 0.2 | 1.9 | 13.5 | 0.1 | 0.2 | 22.7 |
| 15 | 79.3 | 51.4 | 27.9 | 0.6 | 3.2 | 13.5 | 0.4 | 0.1 | 22.4 |
| 16 | 81.3 | 49.1 | 32.2 | 0.8 | 2.8 | 12.1 | 0.3 | 0.2 | 17.2 |
| 17 | 79.2 | 55.0 | 24.2 | 0.6 | 3.2 | 13.0 | 0.3 | 0.1 | 23.8 |
| 18 | 79.2 | 54.2 | 25.0 | 0.6 | 3.3 | 13.1 | 0.4 | 0.1 | 21.9 |
| 19 | 77.7 | 50.3 | 27.4 | 0.7 | 3.2 | 14.3 | 0.2 | 0.1 | 22.3 |
| 20 | 78.7 | 51.6 | 27.1 | 0.7 | 3.3 | 13.8 | 0.2 | 0.0 | 23.7 |
| 21 | 79.1 | 52.8 | 26.3 | 0.7 | 3.1 | 13.6 | 0.3 | 0.0 | 18.6 |
| Average | 79.6 | 51.8 | 27.8 | 0.5 | 2.6 | 13.6 | 0.2 | 0.2 | 22.6 |
| Stdev | 1.3 | 2.1 | 1.7 | 0.2 | 1.0 | 1.4 | 0.1 | 0.1 | 2.1 |
| COV | 1.6 | 4.0 | 6.3 | 32.0 | 37.4 | 10.5 | 41.8 | 47.7 | 9.4 |

**Table 12**
**Chemical characterization results for Source 11**

| Bucket Number | Total Oxides (%) | $SiO_2$ (%) | Ammonium Hydroxide Group (%) | $SO_3$ (%) | MgO (%) | CaO (%) | LOI (%) | Moisture Content (%) | Fineness (%325) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 63.8 | 36.0 | 27.8 | 2.2 | 4.5 | 25.0 | 0.6 | 0.5 | 16.6 |
| 2 | 61.3 | 34.6 | 26.7 | 2.6 | 4.6 | 26.8 | 0.6 | 0.4 | 16.1 |
| 3 | 60.6 | 33.8 | 26.8 | 2.7 | 4.8 | 27.6 | 0.4 | 0.7 | 16.2 |
| 4 | 61.7 | 34.8 | 26.8 | 2.5 | 4.7 | 26.5 | 0.5 | 0.6 | 16.3 |
| 5 | 60.8 | 33.2 | 27.7 | 1.7 | 3.1 | 29.9 | 1.0 | 0.1 | 17.2 |
| 6 | 61.6 | 33.4 | 28.2 | 1.9 | 3.1 | 29.5 | 1.3 | 0.1 | 17.4 |
| 7 | 66.3 | 40.0 | 26.3 | 2.1 | 4.2 | 23.0 | 0.6 | 0.3 | 16.7 |
| 8 | 60.5 | 33.8 | 26.8 | 2.6 | 4.8 | 27.2 | 0.5 | 0.4 | 17.1 |
| 9 | 60.1 | 32.6 | 27.5 | 2.6 | 4.7 | 28.1 | 0.7 | 0.4 | 18.3 |
| 10 | 64.0 | 35.6 | 28.4 | 2.1 | 4.4 | 25.0 | 0.6 | 0.3 | 16.9 |
| 11 | 64.1 | 34.5 | 29.6 | 2.2 | 4.2 | 25.2 | 0.7 | 0.3 | 16.4 |
| 12 | 66.3 | 36.1 | 30.2 | 2.3 | 0.0 | 26.6 | 0.5 | 0.4 | 16.7 |
| Average | 62.6 | 34.9 | 27.7 | 2.3 | 3.9 | 26.7 | 0.7 | 0.4 | 16.8 |
| Stdev | 2.2 | 2.0 | 1.2 | 0.3 | 1.4 | 2.0 | 0.2 | 0.2 | 0.6 |
| COV | 3.6 | 5.6 | 4.4 | 13.7 | 34.9 | 7.4 | 37.5 | 47.1 | 3.7 |

## Set Time

For each bucket from each source, set time tests were conducted in triplicates as mentioned in the methods section. Appendix A shows the individual set time graphs for the Vicat needle and pocket penetrometer for each source and each bucket. Note that if a graph is not provided in the Appendix for a certain bucket number, then there was no recordable set time for that material after 240 minutes of testing.

After completing the set time testing, the initial and final set times were plotted for each bucket number to determine correlations. Figure 6 to Figure 8 shows the initial and final set times for the Gillmore needle, Vicat needle, and pocket penetrometer for Source 1, respectively. The remaining figures for Sources 2 to 9 can be found in Appendix B. Note that if the set time shows 240 minutes on the figure, that means that the time to set was greater than 240 minutes.

Note the strong correlation between the initial and final set times for each test. Note that the Gillmore needle and the Vicat needle tend to give nearly the same set time results for each bucket. The pocket penetrometer, or Iowa set time test, tends to give shorter times to initial and final sets for each bucket. The author believes that this is due to the much increased surface area of the testing apparatus of the pocket penetrometer compared to those of the Vicat and Gillmore needles. These findings proved consistent throughout all sources tested.

Of particular interest are the results for bucket number 17. Note the increased set time at bucket number 17 for all set time tests. The results show an outlier for the sample that will be discussed in the following modeling and statistics section.

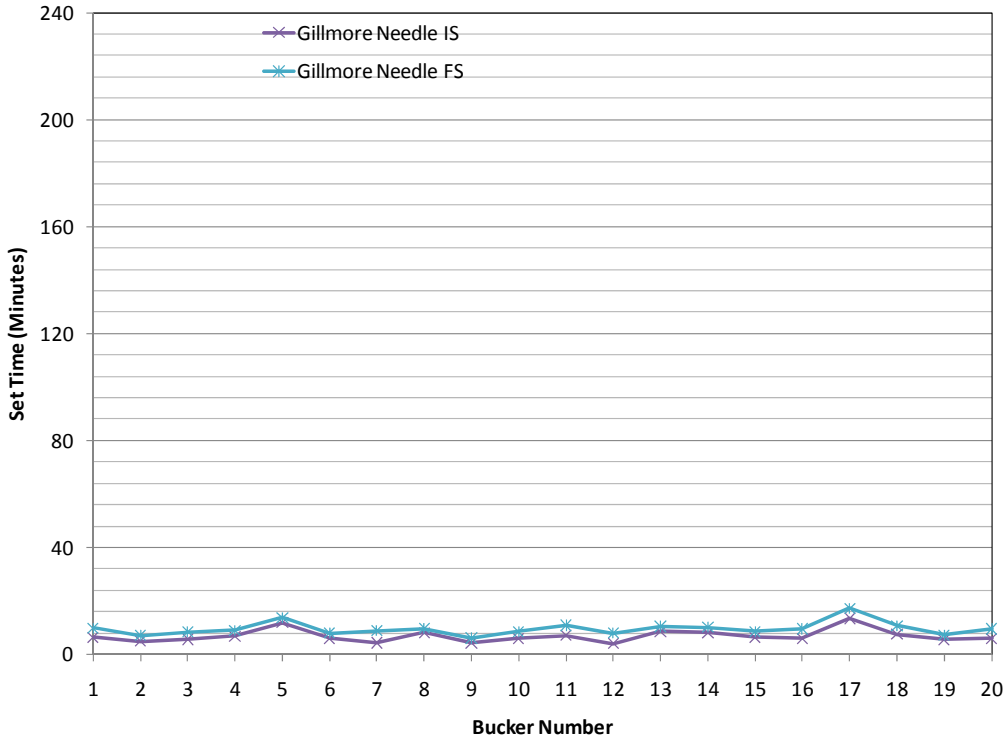


**Figure 6**
**Gillmore needle initial and final set results versus bucket number for Source 1**

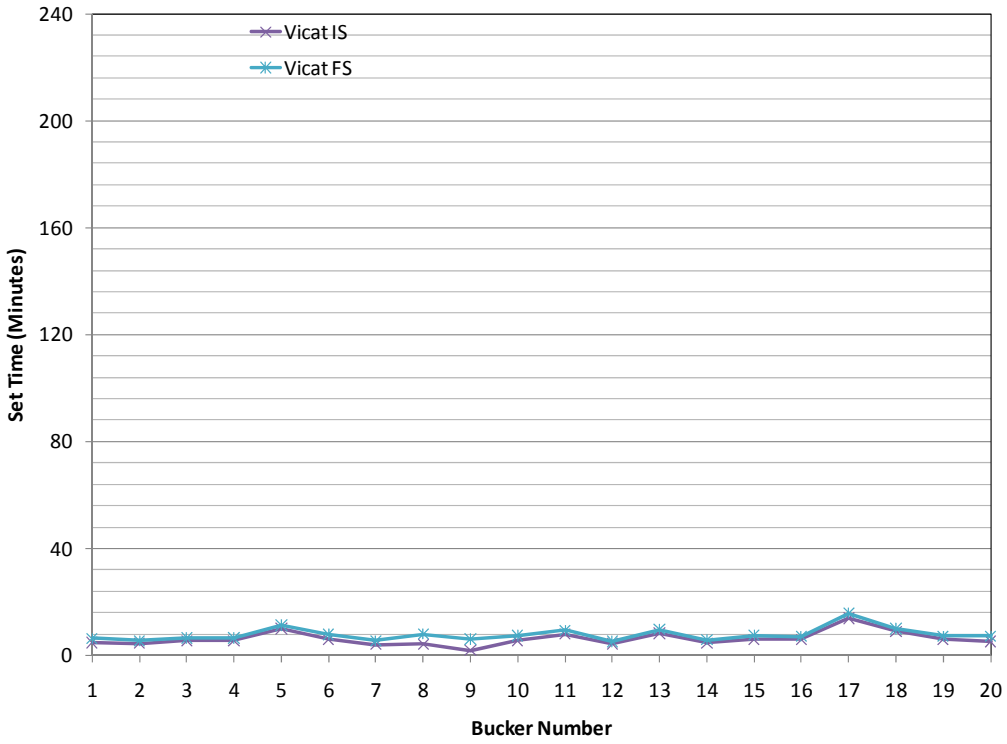


**Figure 7**
**Vicat needle initial and final set results versus bucket number for Source 1**

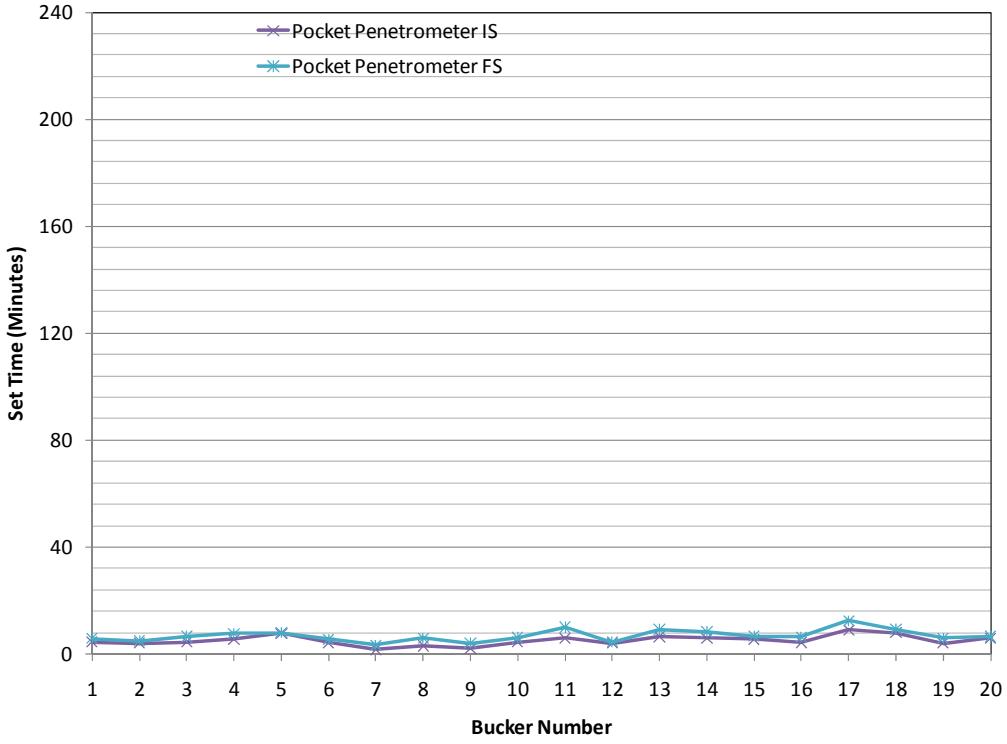


**Figure 8**
**Pocket penetrometer initial and final set results versus bucket number for Source 1**

Figure 9 to Figure 11 show the set time results for Source 8. Note the dramatic differences in set times for all tests. It is also important to note the differences when compared to the results from Source 1. The set time test results from Source 8 indicate variability, especially when compared to the set time test results from Source 1. After consultation with fly ash and cement suppliers, it was decided to further explore the effects of fly ash and cement on the set times.

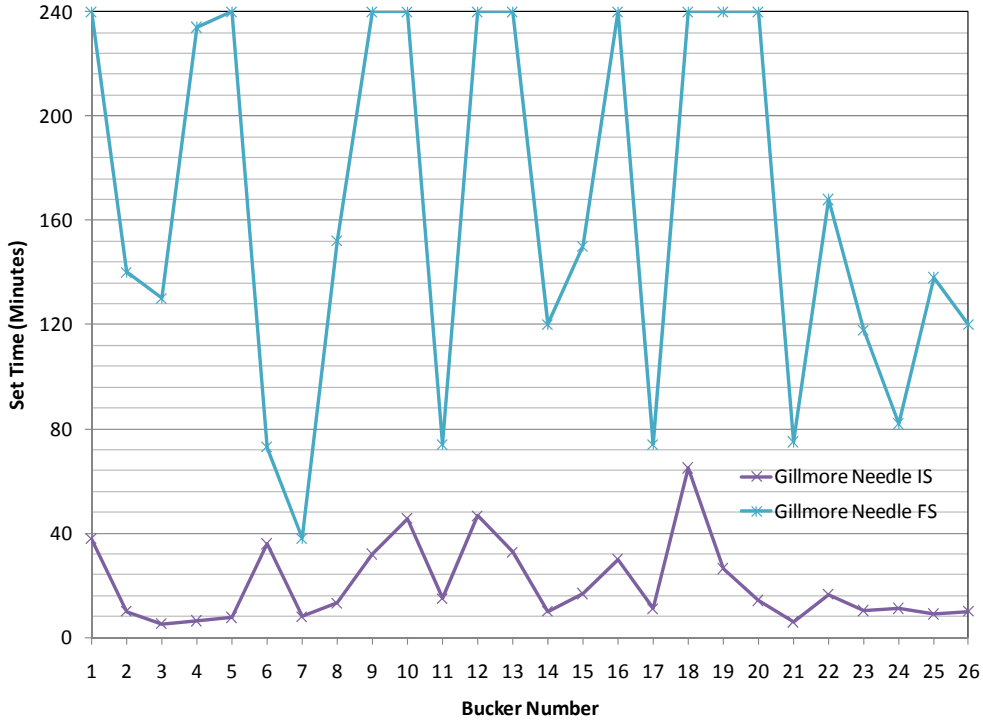


**Figure 9**
**Gillmore needle initial and final set results versus bucket number for Source 8**

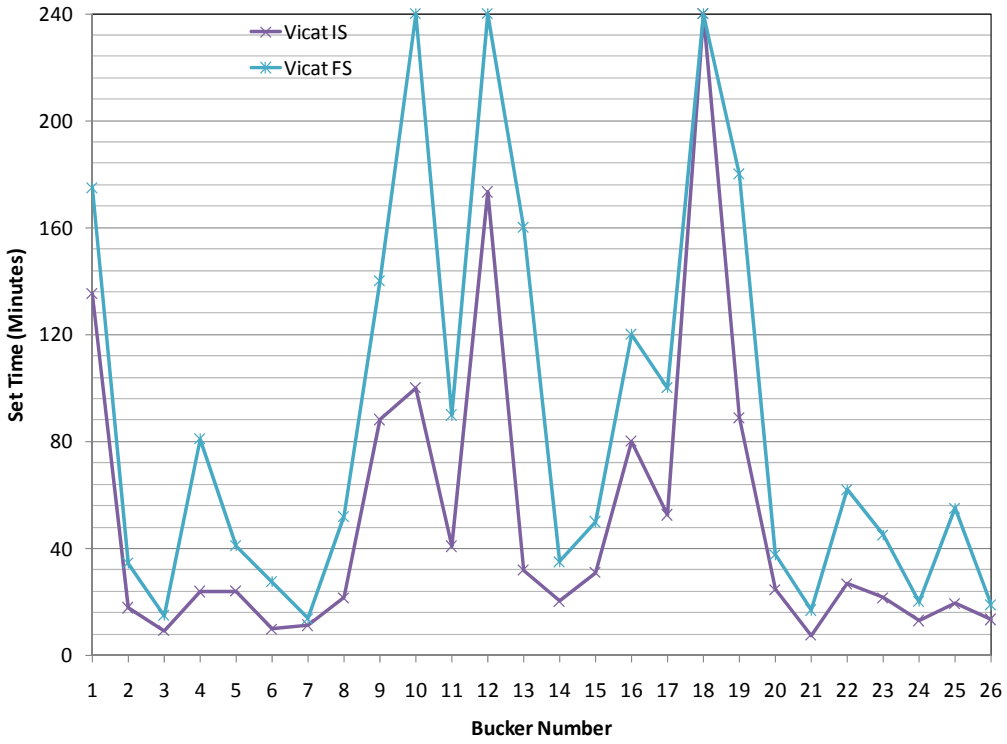


**Figure 10**
**Vicat needle initial and final set results versus bucket number for Source 8**

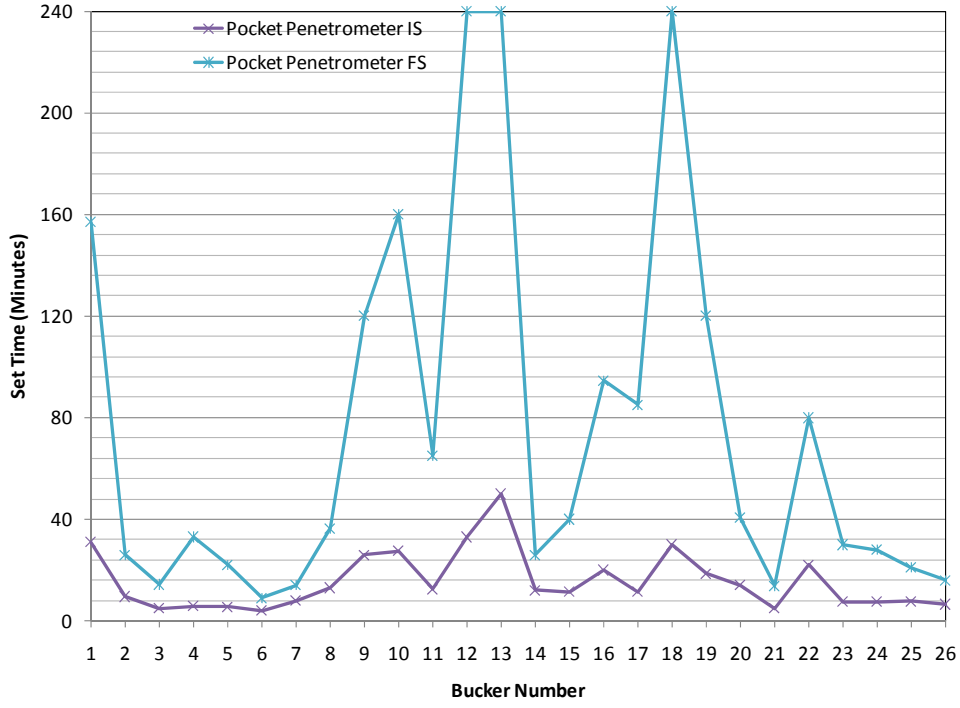


**Figure 11**
**Pocket penetrometer initial and final set results versus bucket number for Source 8**

Upon further discussion, a test plan was developed to test select buckets from two sources of ash tested herein. The sources selected were Source 5 and Source 8. Source 5 was regarded as an average of the ashes tested, and Source 8 was regarded as a longer setting time ash of those tested for this project. The Vicat set time method was used and the mix design was produced as noted in the methodology section with the exception that 50 percent of the fly ash was replaced with type I/II portland cement by mass. The results for Source 5 and 8 are shown in Figure 12 and Figure 13, respectively.

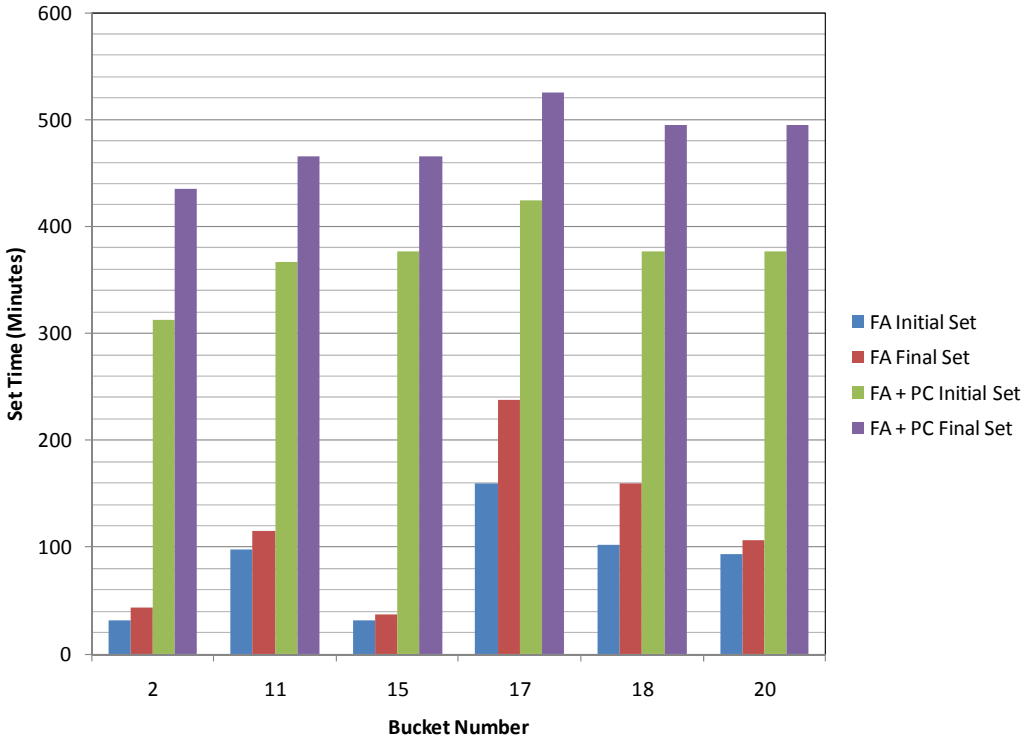


**Figure 12**
**Comparison of Vicat initial and final set values for fly ash and 50 percent fly ash – 50 percent cement combinations from Source 5**

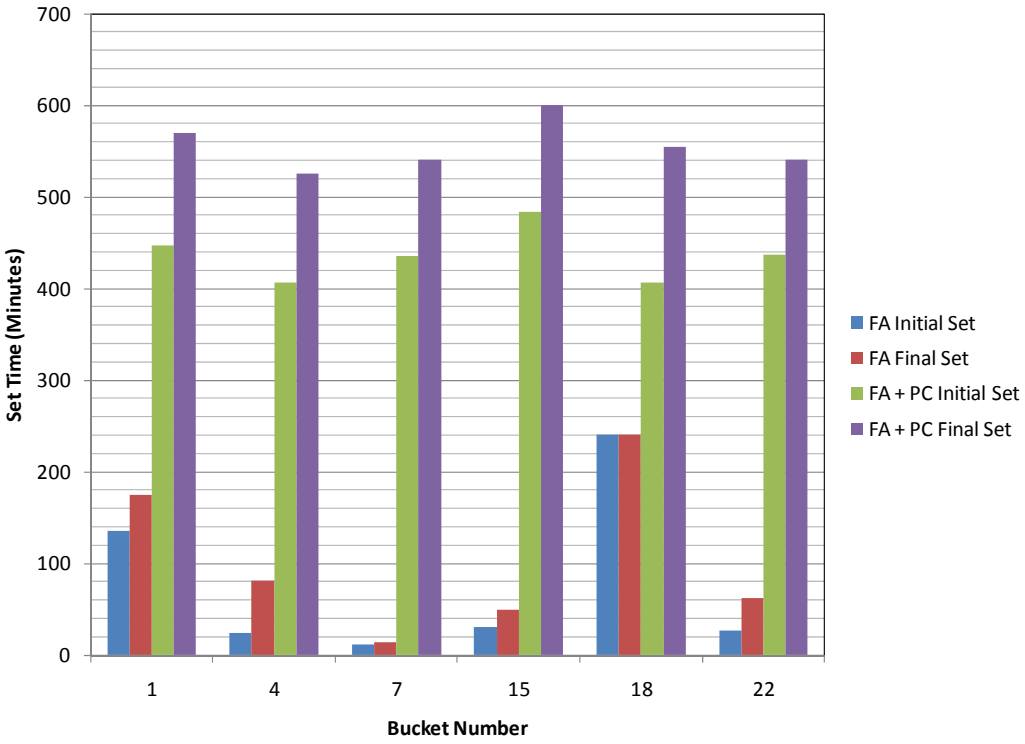


**Figure 13**
**Comparison of Vicat initial and final set values for fly ash and 50 percent fly ash – 50 percent cement combinations from Source 8**

The portland cement (PC) fly ash (FA) combined set time results showed that the perceived variability of the fly ash is not an issue. Note the results from Source 8 showed significant differences in the final set times between the buckets for the fly ash only samples. When comparing the results of the 50 percent fly ash / 50 percent cement samples, the final set values all fall within 80 minutes of each other. This indicates that at an increased replacement of fly ash, more than currently allowed under LADOTD specifications, does not lead to erratic set times of the portland cement concrete mixture. The same results are also validated when examining the results from Source 5.

The set time results of the fly ash only mixtures indicate that the test procedures may flag changes in chemistry or physical properties. The reality is that fly ash is a unique material with both a glassy and crystalline phase that can vary from particle to particle. The results shown in Figure 12 and Figure 13 indicate that this variability is not a problem at a 50 percent portland cement replacement level, suggesting a lesser issue at LADOTD's accepted 20 percent replacement level.

## Coffee Cup

The coffee cup test was conducted on each bucket from each source. Figure 14 to Figure 24 show the range of coffee cup results for all 11 sources. The general shape of the temperature curves are expected due to the hydration of free lime, sulfates, and tricalcium aluminate. Note the variance within each source and the variation between sources. The large variation between sources is to be expected. The author believes that the variation between samples within the same source is in part to several mechanisms including: hydration characteristics of the fly ash sample, subtle mixing differences, and differences of the individual fly ash particles within each sample.

The general grouping of temperature curves for Source 1 are shown in Figure 14. Of particular interest are the results for Bucket 14. The temperature results indicate that this particular bucket is dramatically different from the other buckets, but the set time results shown in Figure 6 to Figure 8 indicate otherwise. This phenomenon was prevalent throughout the temperature and set time testing.

Other typical trends are shown in Source 2 and Source 4, see Figure 15 and Figure 17. The trends for Source 2 and 4 show a wide range of maximum temperatures and time to maximum temperatures. Note the set times for each of the two sources were consistent between buckets. Figure 23 shows the temperature results for a high calcium class F ash that was tested for this study. Note the temperature curves do not show any appreciable temperature gain due to hydration. This result is expected due to this particular fly ash not exhibiting an initial or final set time.

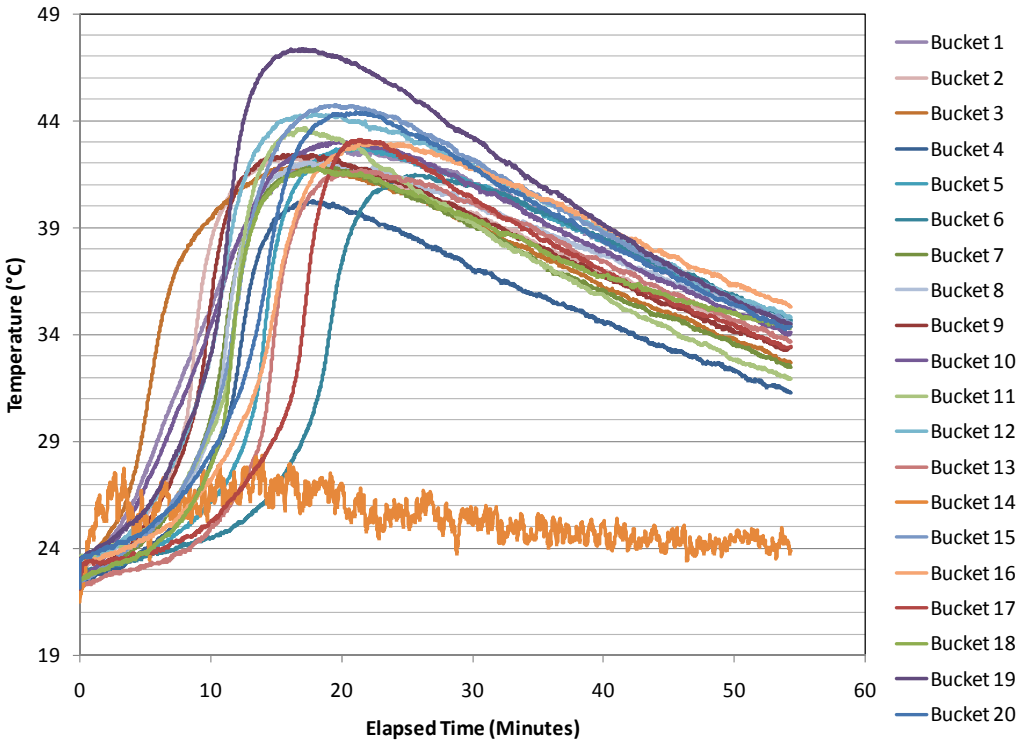


**Figure 14**
**Coffee cup results for Source 1**

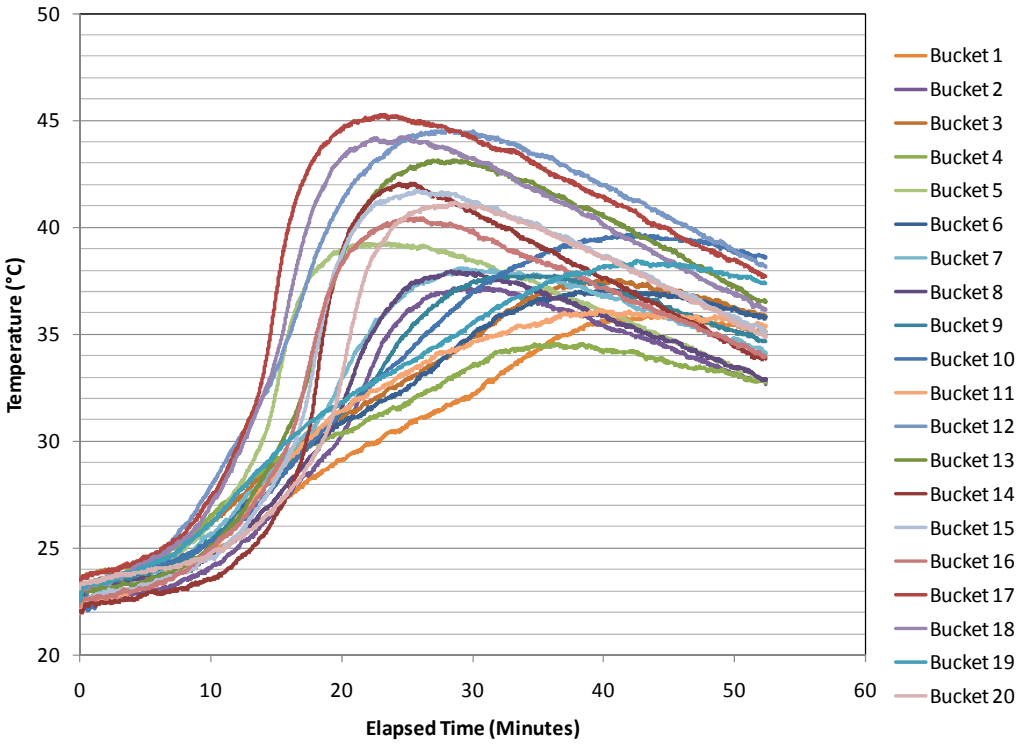


**Figure 15**
**Coffee cup results for Source 2**

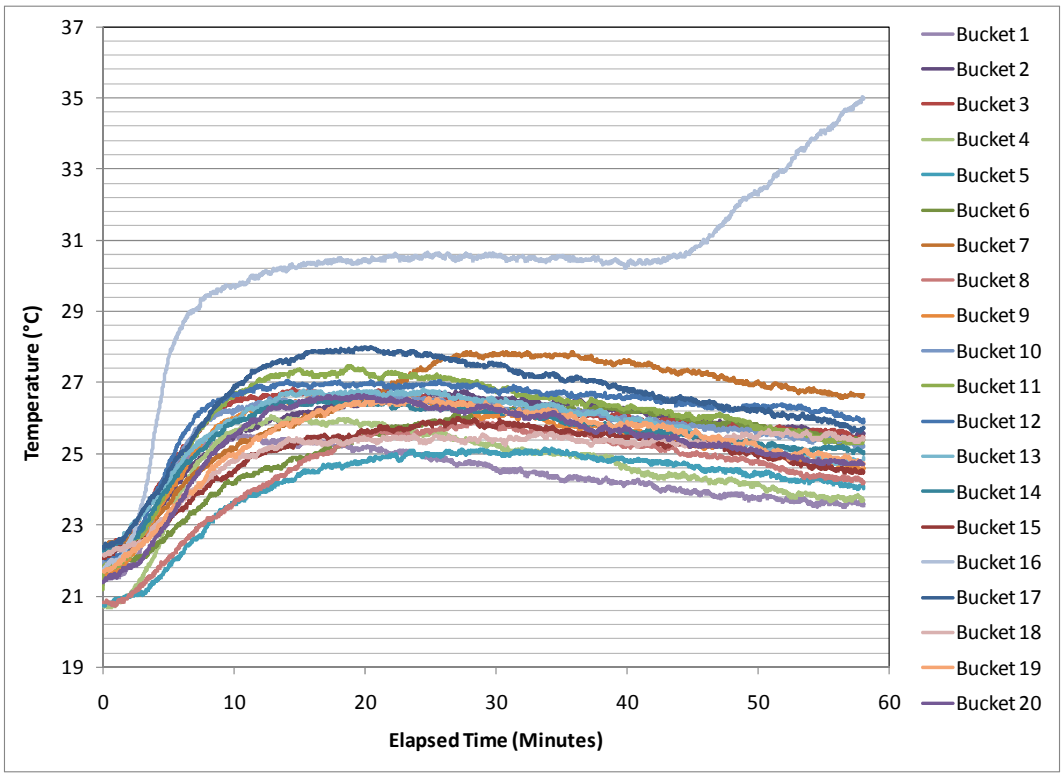


**Figure 16**
**Coffee cup results for Source 3**

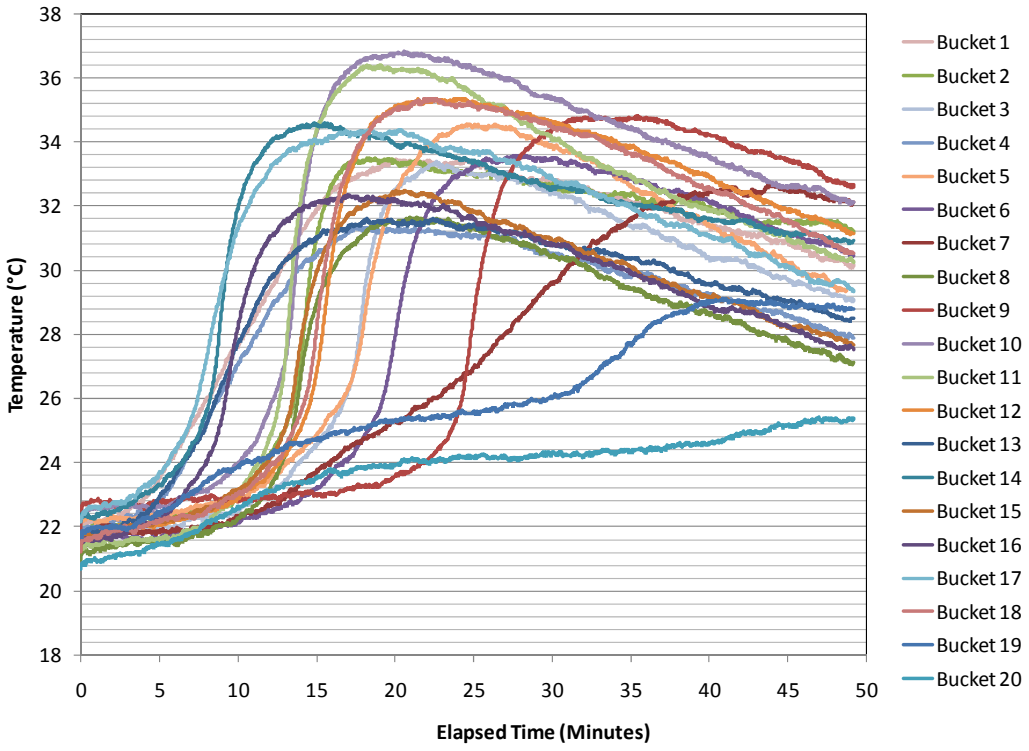


**Figure 17**
**Coffee cup results for Source 4**

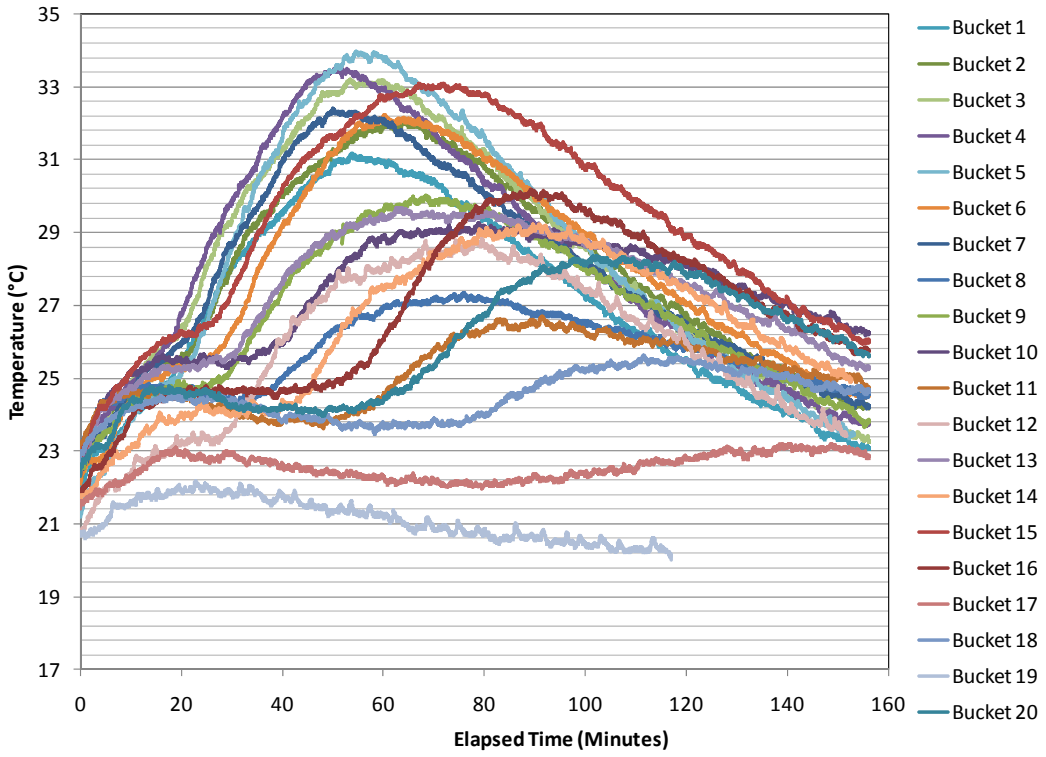


**Figure 18**
**Coffee cup results for Source 5**

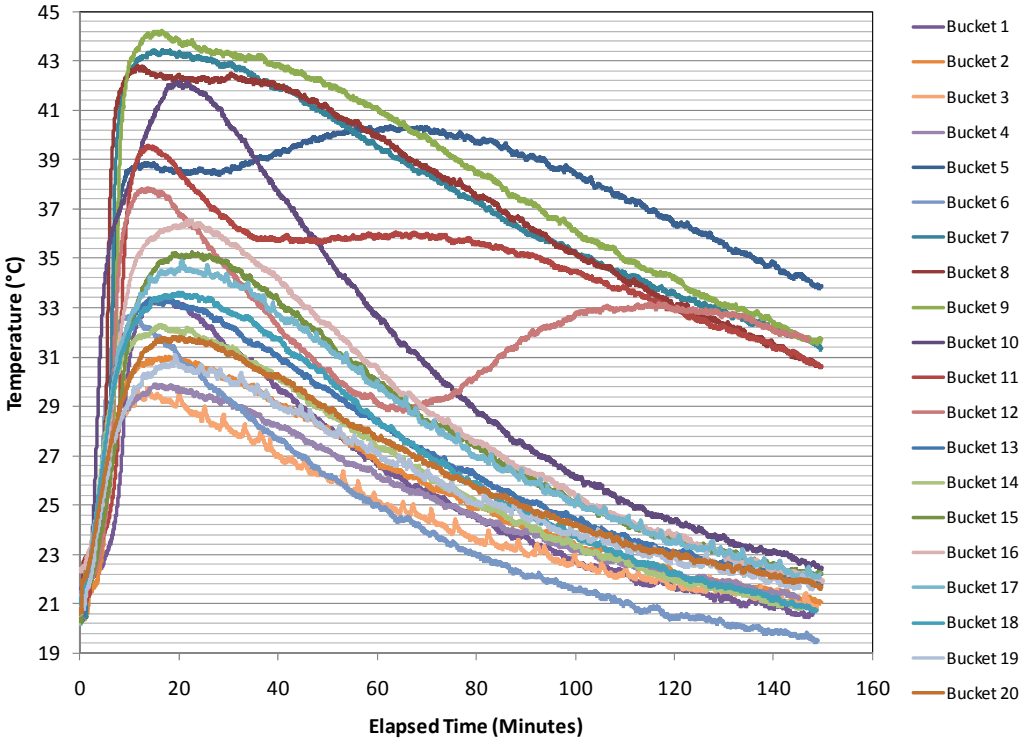


**Figure 19**
**Coffee cup results for Source 6**

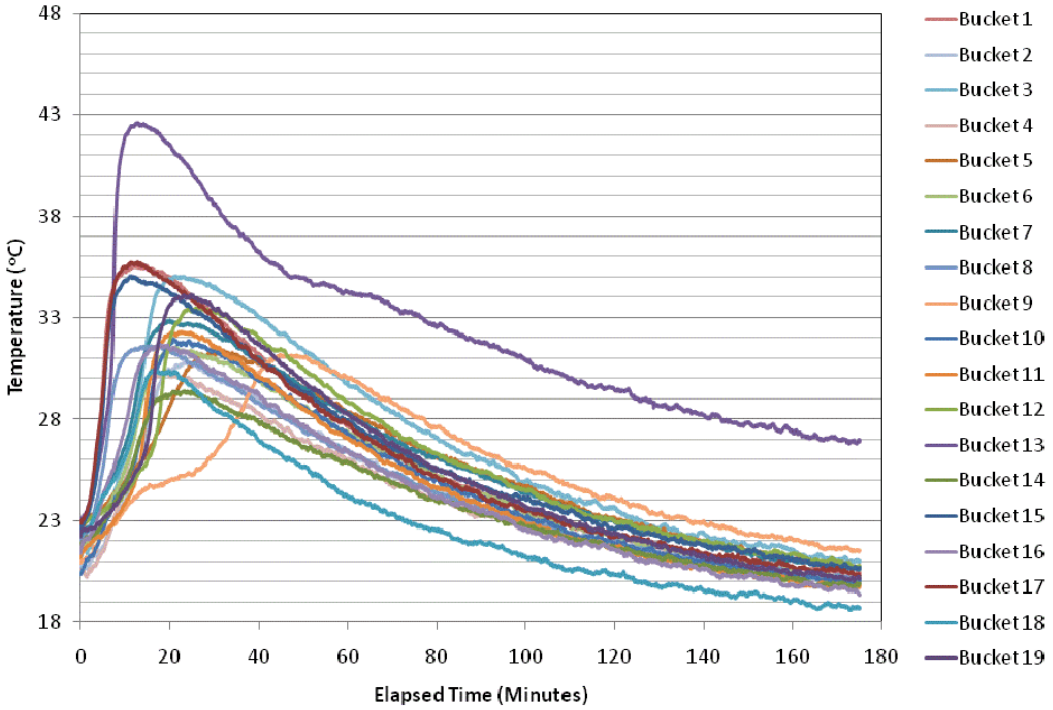


**Figure 20**
**Coffee cup results for Source 7**

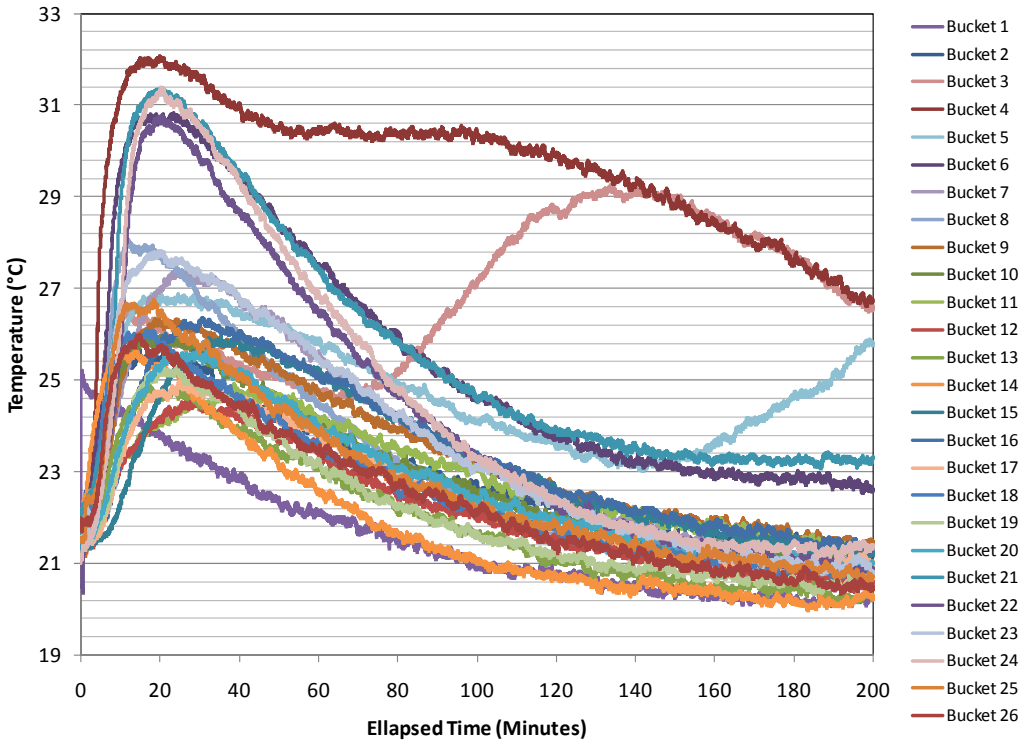


**Figure 21**
**Coffee cup results for Source 8**

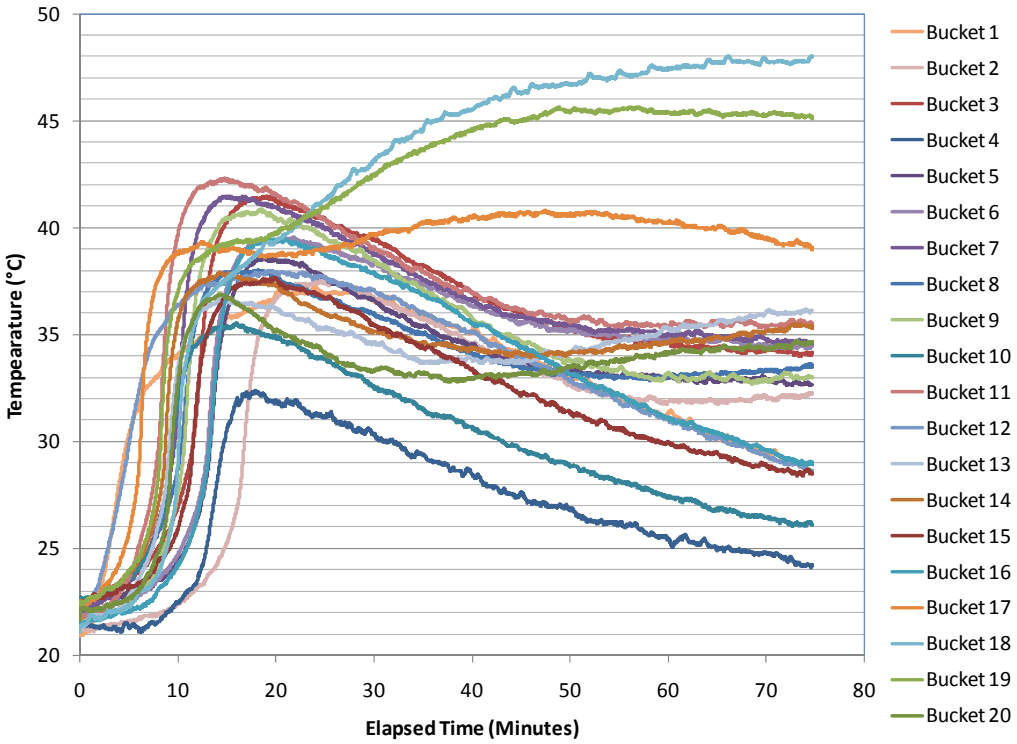


**Figure 22**
**Coffee cup results for Source 9**

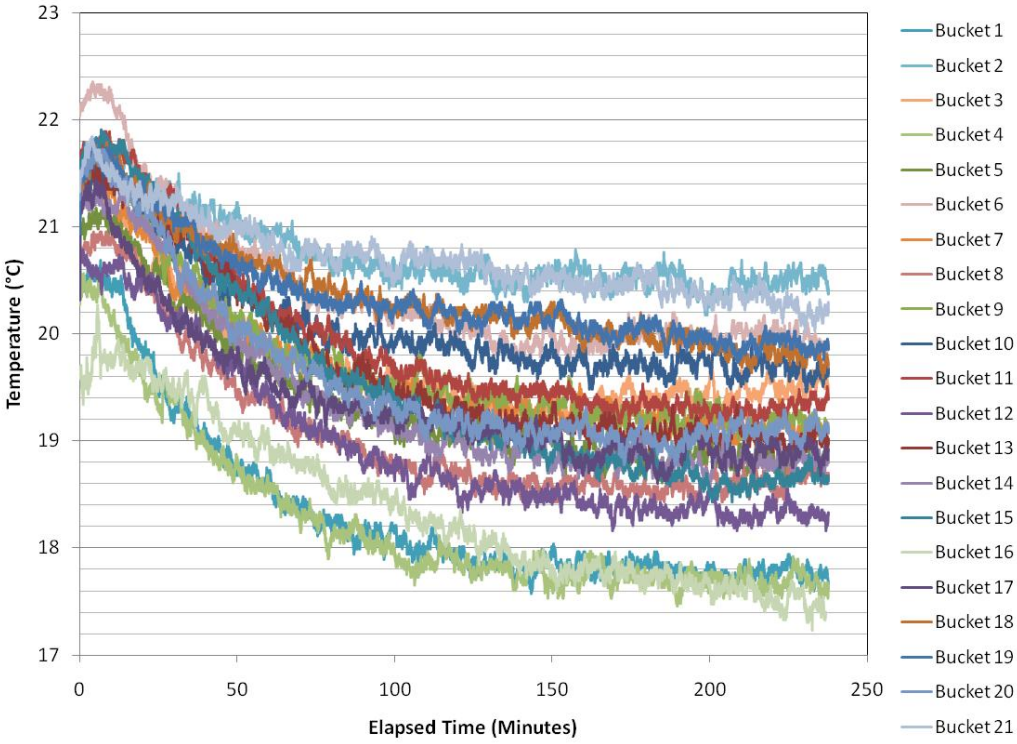


**Figure 23**
**Coffee cup results for Source 10**

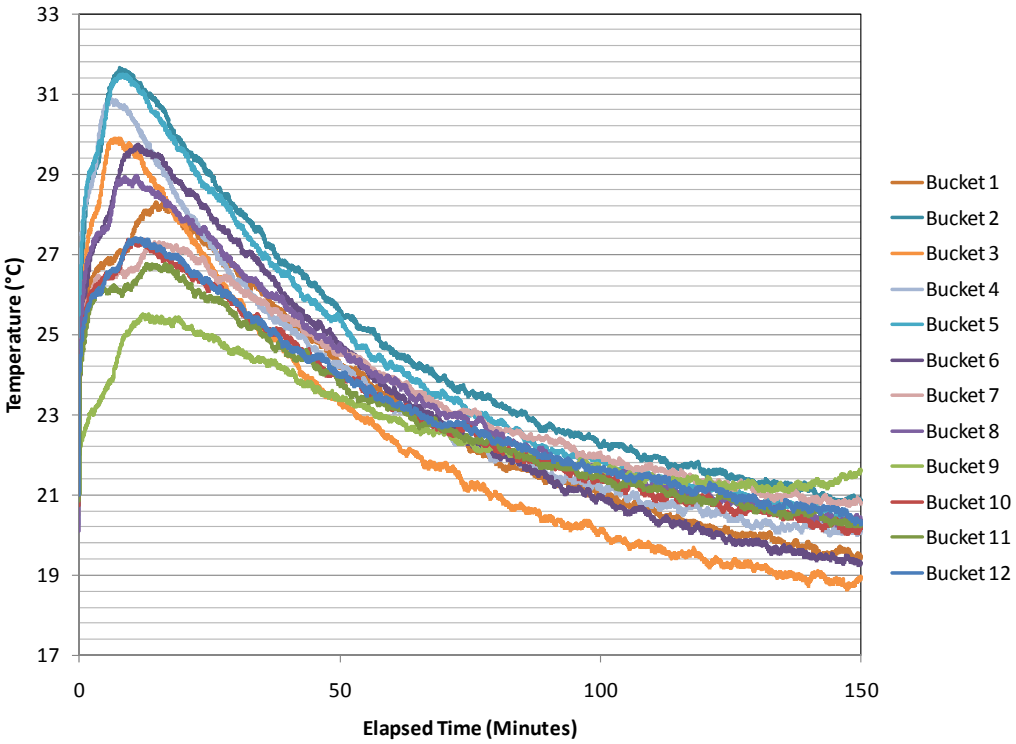


**Figure 24**
**Coffee cup results for Source 11**

It is accepted that as the glassy and crystalline phases of the individual fly ash particles change, the behavior of that individual particle changes as well. The temperature results shown here denote this fact and show that hydration of class C fly ash is a very complex and intriguing phenomenon. The coffee cup results for Source 8 (see Figure 21) lend themselves to this explanation. The author hypothesized that a relationship between the temperature, chemistry, fineness, and set time would be determined and that those relationships would be good. The individual set times were longer for several of the buckets from Source 8 with increased temperature curves, running counterintuitive to conventional thought being a larger temperature curve generally leads to shorter set times. This result led the author to further examine the results in combination with portland cement and to further develop the statistical analysis as noted in the following section.

## Statistical Analysis

This section is divided into two sections. The first section deals with the determination of outliers and the determination of significant differences between buckets within a source. The second section details the extensive modeling effort put forth to determine if relationships exist between the various measured chemical and physical parameters and both

the set time and coffee cup results. Chemical properties determined from X-Ray Fluorescence (XRF) and fineness measurements are the chemical and physical parameters used in the model, respectively. The set times are the initial and final set times from the various tests conducted. The temperature results were reduced to two parameters including the maximum temperature and the time to maximum temperature.

**Determination of Statistical Significance**

JMP 8.0 software was originally used to determine significance at an alpha level of 0.05 [*11*]. The results of the t-test showed that each sample (i.e., bucket) was significantly different from the others within the same source. The research team examined the results more closely and determined that this was due to the very low variability in the measured set time results. The variability of the set time results was very low, on the order of less than 30 seconds to 3 minutes, between set time samples produced from the same mixing batch. This low variability produced little overlap between samples tested from each bucket.

The research team then used ASTM E178 to determine test results that were outliers [*12*]. A significance level of 0.05 was used in the analysis. The results were then much more realistic due to the methodology of comparing the average of the results for each bucket against the mean results for all samples of the source.

Table 13 to Table 23 show the results for the determination of outliers for all sources, the average, standard deviation, and the coefficient of variation for each source. Note the low number of outliers for Source 9 and Source 6. The results show that the class C fly ashes used in the state of Louisiana are generally uniform and the variations that occur are of no concern especially when being added to portland cement concrete as shown in set time section of the discussion.

Care should be exercised when interpreting the data from Source 11. Because of a very low number of actual samples exhibiting a set time and the removal of samples that did not exhibit a set time from the mean and standard deviation calculations, the statistical analysis showed all samples that did not set to be outliers. The author believes that the opposite is true and the samples that actually set are the outliers in this case.

The results also show varying degrees of variability of the individual sources when comparing the coefficients of variation (COV). The COVs range from 15% to 135% depending upon the source and the test method. Note that as the variability increases, the likelihood of producing an outlier becomes smaller.

**Table 13**
**Average set time results for all set time tests for Source 1**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 6.33 | 9.82 | 4.72 | 6.33 | 4.50 | 5.83 |
| 2 | 4.90 | 7.05 | 4.53 | 5.50 | 4.00 | 5.00 |
| 3 | 5.68 | 8.43 | 5.52 | 6.50 | 4.50 | 6.58 |
| 4 | 6.83 | 8.98 | 5.50 | 6.50 | 5.50 | 7.67 |
| 5 | 11.88 | 13.73 | 9.95 | 11.33 | 7.67 | 8.00 |
| 6 | 6.00 | 8.00 | 5.97 | 7.83 | 4.25 | 5.50 |
| 7 | 4.42 | 8.90 | 4.06 | 5.50 | 1.75 | 3.33 |
| 8 | 8.19 | 9.66 | 4.17 | 8.00 | 3.00 | 6.00 |
| 9 | 4.39 | 6.08 | 1.85 | 6.05 | 2.00 | 4.00 |
| 10 | 6.00 | 8.50 | 5.57 | 7.33 | 4.50 | 6.17 |
| 11 | 7.18 | 10.83 | 8.00 | 9.45 | 6.00 | 10.00 |
| 12 | 4.12 | 8.00 | 4.31 | 5.25 | 4.00 | 4.50 |
| 13 | 8.52 | 10.43 | 8.28 | 9.66 | 6.50 | 9.13 |
| 14 | 8.06 | 10.18 | 4.65 | 5.84 | 6.00 | 8.25 |
| 15 | 6.33 | 8.60 | 5.91 | 7.50 | 5.50 | 6.67 |
| 16 | 5.92 | 9.57 | 5.91 | 7.17 | 4.34 | 6.33 |
| 17 | 13.50 | 17.25 | 13.97 | 15.75 | 9.00 | 12.50 |
| 18 | 7.58 | 10.75 | 8.98 | 10.00 | 8.00 | 9.33 |
| 19 | 5.50 | 7.25 | 5.90 | 7.25 | 4.00 | 6.00 |
| 20 | 6.00 | 9.50 | 5.28 | 7.25 | 6.00 | 6.50 |
| Average | 6.87 | 9.58 | 6.15 | 7.80 | 5.05 | 6.86 |
| stdev | 2.36 | 2.44 | 2.62 | 2.49 | 1.87 | 2.20 |
| cov (%) | 34.43 | 25.52 | 42.65 | 31.90 | 36.94 | 32.03 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 14**

**Average set time results for all set time tests for Source 2**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 19.16 | 27.38 | 17.00 | 21.00 | 15.00 | 21.67 |
| 2 | 16.60 | 30.45 | 17.05 | 21.50 | 14.00 | 22.50 |
| 3 | 17.45 | 30.25 | 17.12 | 20.50 | 12.00 | 21.67 |
| 4 | 15.08 | 39.72 | 15.99 | 24.50 | 12.00 | 27.00 |
| 5 | 16.00 | 18.00 | 13.49 | 14.58 | 12.00 | 15.00 |
| 6 | 19.25 | 24.00 | 19.97 | 22.10 | 13.67 | 20.50 |
| 7 | 11.83 | 20.00 | 13.58 | 15.75 | 10.00 | 16.00 |
| 8 | 16.75 | 28.08 | 16.39 | 20.00 | 13.50 | 20.83 |
| 9 | 17.54 | 38.35 | 19.74 | 25.00 | 13.00 | 27.00 |
| 10 | 18.00 | 21.50 | 15.41 | 18.00 | 13.00 | 17.67 |
| 11 | 14.02 | 21.68 | 13.41 | 15.50 | 10.00 | 15.00 |
| 12 | 15.97 | 32.13 | 14.21 | 19.00 | 10.00 | 21.00 |
| 13 | 14.80 | 19.38 | 13.34 | 15.50 | 12.00 | 15.00 |
| 14 | 13.78 | 17.10 | 12.52 | 14.25 | 11.00 | 14.00 |
| 15 | 12.50 | 17.00 | 14.29 | 17.00 | 10.25 | 14.17 |
| 16 | 14.33 | 24.66 | 14.22 | 16.50 | 11.50 | 17.33 |
| 17 | 13.50 | 17.88 | 10.96 | 14.00 | 11.00 | 14.00 |
| 18 | 10.00 | 14.46 | 11.92 | 14.00 | 9.00 | 10.50 |
| 19 | 9.00 | 15.00 | 9.11 | 11.00 | 7.33 | 10.00 |
| 20 | 16.50 | 21.00 | 17.73 | 20.50 | 14.00 | 18.75 |
| Average | 15.10 | 23.90 | 14.87 | 18.01 | 11.71 | 17.98 |
| stdev | 2.80 | 7.36 | 2.79 | 3.80 | 1.93 | 4.77 |
| cov (%) | 18.54 | 30.78 | 18.77 | 21.10 | 16.44 | 26.51 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 15**

**Average set time results for all set time tests for Source 3**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 8.80 | 98.80 | 19.69 | 60.00 | 9.00 | 36.00 |
| 2 | 10.00 | 99.00 | 16.27 | 55.00 | 2.50 | 28.00 |
| 3 | 5.92 | 95.83 | 16.04 | 44.00 | 5.00 | 23.00 |
| 4 | 10.08 | 114.00 | 21.58 | 50.00 | 9.50 | 35.00 |
| 5 | 12.72 | 240+ | 34.40 | 90.00 | 9.00 | 47.00 |
| 6 | 11.77 | 136.00 | 20.98 | 58.00 | 8.00 | 38.33 |
| 7 | 21.19 | 105.00 | 27.80 | 50.00 | 12.00 | 34.50 |
| 8 | 12.25 | 123.65 | 20.46 | 42.00 | 10.00 | 27.08 |
| 9 | 9.50 | 131.00 | 17.66 | 40.00 | 8.00 | 23.25 |
| 10 | 12.00 | 133.50 | 34.83 | 80.00 | 8.00 | 54.00 |
| 11 | 16.00 | 135.00 | 29.29 | 56.00 | 11.50 | 45.00 |
| 12 | 10.83 | 147.00 | 21.34 | 52.00 | 7.00 | 35.00 |
| 13 | 27.00 | 240+ | 28.99 | 95.00 | 13.00 | 98.00 |
| 14 | 9.25 | 124.00 | 16.52 | 50.00 | 7.50 | 32.00 |
| 15 | 13.22 | 240+ | 32.77 | 91.00 | 9.75 | 48.50 |
| 16 | 6.28 | 31.12 | 7.81 | 10.00 | 6.00 | 10.00 |
| 17 | 8.83 | 76.00 | 11.50 | 25.00 | 6.50 | 13.50 |
| 18 | 67.61 | 240+ | 73.92 | 100.00 | 41.00 | 100.00 |
| 19 | 9.03 | 17.63 | 14.41 | 38.00 | 5.00 | 19.33 |
| 20 | 19.50 | 93.00 | 46.90 | 69.00 | 13.00 | 75.00 |
| Average | 15.09 | 131.03 | 25.66 | 57.75 | 10.06 | 41.13 |
| stdev | 13.39 | 64.68 | 14.71 | 23.69 | 7.78 | 24.69 |
| cov (%) | 88.75 | 49.37 | 57.35 | 41.02 | 77.29 | 60.03 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 16**

**Average set time results for all set time tests for Source 4**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 29.00 | 42.00 | 27.81 | 32.33 | 27.00 | 30.00 |
| 2 | 10.00 | 15.00 | 10.01 | 10.75 | 8.00 | 10.00 |
| 3 | 8.97 | 11.92 | 8.47 | 9.50 | 7.00 | 9.50 |
| 4 | 25.00 | 41.00 | 26.91 | 31.00 | 23.50 | 26.50 |
| 5 | 9.35 | 16.00 | 10.47 | 11.50 | 9.50 | 11.50 |
| 6 | 9.62 | 13.43 | 9.97 | 11.00 | 9.00 | 10.25 |
| 7 | 39.00 | 54.00 | 39.16 | 49.00 | 34.00 | 40.83 |
| 8 | 26.00 | 48.00 | 26.05 | 33.33 | 23.00 | 29.50 |
| 9 | 19.00 | 26.50 | 20.14 | 22.67 | 20.00 | 22.33 |
| 10 | 8.22 | 12.12 | 8.47 | 9.50 | 7.00 | 9.50 |
| 11 | 10.10 | 15.40 | 10.46 | 11.50 | 9.00 | 11.00 |
| 12 | 8.22 | 13.37 | 9.45 | 11.00 | 8.00 | 10.50 |
| 13 | 7.12 | 12.10 | 6.98 | 8.75 | 6.50 | 8.50 |
| 14 | 15.58 | 43.88 | 13.40 | 16.50 | 11.00 | 18.67 |
| 15 | 8.90 | 15.63 | 9.17 | 10.75 | 7.00 | 9.67 |
| 16 | 42.00 | 60.00 | 43.56 | 55.00 | 27.00 | 44.83 |
| 17 | 16.50 | 26.15 | 19.85 | 21.50 | 9.50 | 21.00 |
| 18 | 6.66 | 11.20 | 7.27 | 8.25 | 7.00 | 8.50 |
| 19 | 43.00 | 63.00 | 35.67 | 42.00 | 44.00 | 50.50 |
| 20 | 23.47 | 40.45 | 26.44 | 33.00 | 17.25 | 29.00 |
| Average | 18.29 | 29.06 | 18.49 | 21.94 | 15.71 | 20.60 |
| stdev | 12.11 | 18.03 | 11.61 | 14.65 | 10.81 | 13.23 |
| cov (%) | 66.24 | 62.05 | 62.79 | 66.78 | 68.81 | 64.21 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 17**

**Average set time results for all set time tests for Source 5**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 37.05 | 85.28 | 48.16 | 60.00 | 31.00 | 54.00 |
| 2 | 26.27 | 50.75 | 31.61 | 43.00 | 15.00 | 37.50 |
| 3 | 35.05 | 71.08 | 46.15 | 54.00 | 31.00 | 50.00 |
| 4 | 32.88 | 69.50 | 39.75 | 45.00 | 26.50 | 46.50 |
| 5 | 29.06 | 33.55 | 35.87 | 43.00 | 25.00 | 43.00 |
| 6 | 38.15 | 56.05 | 45.08 | 51.00 | 34.25 | 52.00 |
| 7 | 38.36 | 105.00 | 54.00 | 71.16 | 32.00 | 51.00 |
| 8 | 54.60 | 121.00 | 44.30 | 85.00 | 44.00 | 82.00 |
| 9 | 43.00 | 83.50 | 53.41 | 62.50 | 42.50 | 63.00 |
| 10 | 40.17 | 76.00 | 51.11 | 60.00 | 37.00 | 62.00 |
| 11 | 78.00 | 146.00 | 97.89 | 115.00 | 65.00 | 111.00 |
| 12 | 48.00 | 94.00 | 57.30 | 63.83 | 45.00 | 68.58 |
| 13 | 42.55 | 60.56 | 52.20 | 64.67 | 45.00 | 78.00 |
| 14 | 60.28 | 120.37 | 61.06 | 68.67 | 60.00 | 95.00 |
| 15 | 25.92 | 46.35 | 31.87 | 36.50 | 22.00 | 36.00 |
| 16 | 70.50 | 118.50 | 78.62 | 91.00 | 60.50 | 85.00 |
| 17 | 163.00 | 240.00 | 159.83 | 238.00 | 105.00 | 185.00 |
| 18 | 87.00 | 180.27 | 102.12 | 160.00 | 70.00 | 130.00 |
| 19 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 20 | 86.50 | 163.00 | 93.56 | 106.00 | 72.00 | 100.00 |
| Average | 54.54 | 101.09 | 62.31 | 79.91 | 45.41 | 75.24 |
| stdev | 32.62 | 52.39 | 31.77 | 48.64 | 22.03 | 37.19 |
| cov (%) | 59.81 | 51.82 | 50.98 | 60.87 | 48.51 | 49.42 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 18**

**Average set time results for all set time tests for Source 6**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 12.65 | 28.47 | 12.28 | 15.00 | 8.50 | 17.50 |
| 2 | 6.00 | 15.08 | 7.45 | 9.25 | 5.00 | 8.50 |
| 3 | 38.00 | 76.50 | 49.88 | 67.00 | 15.00 | 60.00 |
| 4 | 5.85 | 15.25 | 7.17 | 8.75 | 4.00 | 8.50 |
| 5 | 2.38 | 8.78 | 2.60 | 4.50 | 2.00 | 4.00 |
| 6 | 3.40 | 6.28 | 3.44 | 4.25 | 2.50 | 3.75 |
| 7 | 2.12 | 4.45 | 2.68 | 3.25 | 2.00 | 3.00 |
| 8 | 2.50 | 4.35 | 1.81 | 3.50 | 1.00 | 3.50 |
| 9 | 2.00 | 4.53 | 3.03 | 3.75 | 2.50 | 3.50 |
| 10 | 2.60 | 3.83 | 3.44 | 4.00 | 2.00 | 3.00 |
| 11 | 3.50 | 5.75 | 3.44 | 4.00 | 2.75 | 3.75 |
| 12 | 2.93 | 6.68 | 3.22 | 4.00 | 2.50 | 4.00 |
| 13 | 6.37 | 10.77 | 5.88 | 8.75 | 5.00 | 7.00 |
| 14 | 5.60 | 10.88 | 5.73 | 7.00 | 4.00 | 7.50 |
| 15 | 4.00 | 5.77 | 4.45 | 5.25 | 3.50 | 4.50 |
| 16 | 4.50 | 6.50 | 4.43 | 5.00 | 3.50 | 5.00 |
| 17 | 3.00 | 5.55 | 3.19 | 3.75 | 2.50 | 4.50 |
| 18 | 2.17 | 4.80 | 2.68 | 3.25 | 2.00 | 3.25 |
| 19 | 5.45 | 14.00 | 5.56 | 8.00 | 2.00 | 7.25 |
| 20 | 5.00 | 12.00 | 6.52 | 7.75 | 4.00 | 6.67 |
| Average | 6.00 | 12.51 | 6.94 | 9.00 | 3.81 | 8.43 |
| stdev | 7.91 | 16.16 | 10.39 | 13.97 | 3.10 | 12.59 |
| cov (%) | 131.89 | 129.20 | 149.65 | 155.18 | 81.32 | 149.23 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 19**

**Average set time results for all set time tests for Source 7**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 6.75 | 15.00 | 6.84 | 8.00 | 6.00 | 9.17 |
| 2 | 21.35 | 21.92 | 11.49 | 13.25 | 9.75 | 15.00 |
| 3 | 9.90 | 14.00 | 10.91 | 11.75 | 9.00 | 12.00 |
| 4 | 8.78 | 19.67 | 9.46 | 10.25 | 8.00 | 10.50 |
| 5 | 20.72 | 31.58 | 20.26 | 23.00 | 16.00 | 22.00 |
| 6 | 11.00 | 24.62 | 12.31 | 13.25 | 10.00 | 15.00 |
| 7 | 12.70 | 21.35 | 11.42 | 13.00 | 10.00 | 12.67 |
| 8 | 8.88 | 23.43 | 8.27 | 10.00 | 7.00 | 9.67 |
| 9 | 24.00 | 35.90 | 26.40 | 31.00 | 20.00 | 40.67 |
| 10 | 16.90 | 35.77 | 14.63 | 16.50 | 14.00 | 17.33 |
| 11 | 11.92 | 73.50 | 14.03 | 17.50 | 9.00 | 14.50 |
| 12 | 12.50 | 42.50 | 10.40 | 13.00 | 10.00 | 13.00 |
| 13 | 4.83 | 7.50 | 5.20 | 7.00 | 4.00 | 5.50 |
| 14 | 15.97 | 45.15 | 19.38 | 25.00 | 13.00 | 26.00 |
| 15 | 12.00 | 133.50 | 8.38 | 80.00 | 8.00 | 54.00 |
| 16 | 11.67 | 24.62 | 13.32 | 15.00 | 9.50 | 14.50 |
| 17 | 8.25 | 16.62 | 7.12 | 9.25 | 6.00 | 8.00 |
| 18 | 14.60 | 29.05 | 14.12 | 17.50 | 11.00 | 16.00 |
| 19 | 8.00 | 20.15 | 7.40 | 10.00 | 7.00 | 9.50 |
| Average | 12.67 | 33.46 | 12.18 | 18.12 | 9.86 | 17.11 |
| stdev | 5.17 | 28.33 | 5.28 | 16.19 | 3.79 | 11.89 |
| cov (%) | 40.80 | 84.65 | 43.38 | 89.36 | 38.41 | 69.49 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 20**

**Average set time results for all set time tests for Source 8**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 37.78 | 240+ | 135.23 | 175.00 | 31.00 | 157.00 |
| 2 | 10.00 | 140.00 | 17.85 | 34.50 | 9.50 | 26.00 |
| 3 | 5.28 | 130.00 | 9.26 | 15.00 | 5.00 | 14.33 |
| 4 | 6.35 | 234.00 | 23.87 | 81.00 | 5.75 | 33.00 |
| 5 | 7.68 | 240+ | 24.07 | 41.00 | 5.50 | 22.00 |
| 6 | 36.00 | 73.00 | 9.92 | 27.50 | 4.00 | 9.00 |
| 7 | 8.00 | 38.00 | 11.35 | 13.83 | 8.00 | 14.00 |
| 8 | 13.25 | 152.00 | 21.63 | 52.00 | 13.00 | 36.33 |
| 9 | 32.08 | 240+ | 88.18 | 140.00 | 26.00 | 120.00 |
| 10 | 45.63 | 240+ | 100.00 | 240+ | 27.50 | 160.00 |
| 11 | 15.00 | 74.00 | 40.78 | 90.00 | 12.50 | 65.00 |
| 12 | 46.70 | 240+ | 173.33 | 240+ | 33.00 | 240+ |
| 13 | 32.82 | 240.00 | 31.92 | 160.00 | 50.00 | 240+ |
| 14 | 10.00 | 120.00 | 20.29 | 35.00 | 12.00 | 26.00 |
| 15 | 16.83 | 150.00 | 31.00 | 50.00 | 11.50 | 40.00 |
| 16 | 30.00 | 240+ | 80.07 | 120.00 | 20.00 | 94.50 |
| 17 | 11.00 | 74.00 | 52.63 | 100.00 | 11.50 | 85.00 |
| 18 | 65.00 | 240+ | 240+ | 240+ | 30.00 | 240+ |
| 19 | 26.35 | 240+ | 88.95 | 180.00 | 18.50 | 120.00 |
| 20 | 14.25 | 240+ | 24.67 | 37.67 | 14.00 | 40.50 |
| 21 | 6.02 | 75.00 | 7.49 | 17.00 | 5.00 | 13.83 |
| 22 | 16.50 | 168.00 | 26.75 | 62.00 | 22.00 | 80.00 |
| 23 | 10.25 | 117.83 | 21.59 | 45.00 | 7.50 | 30.00 |
| 24 | 11.15 | 82.00 | 13.00 | 20.10 | 7.50 | 28.00 |
| 25 | 9.00 | 138.00 | 19.36 | 55.00 | 7.75 | 21.00 |
| 26 | 10.05 | 120.00 | 13.47 | 19.00 | 6.50 | 16.00 |
| Average | 20.50 | 125.05 | 43.47 | 68.29 | 15.56 | 54.41 |
| stdev | 15.63 | 55.14 | 43.23 | 53.03 | 11.39 | 46.84 |
| cov (%) | 76.24 | 44.10 | 99.45 | 77.66 | 73.21 | 86.09 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 21**

**Average set time results for all set time tests for Source 9**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 3.08 | 6.03 | 3.36 | 4.25 | 2.50 | 3.67 |
| 2 | 3.17 | 5.38 | 3.34 | 4.17 | 2.00 | 3.00 |
| 3 | 3.75 | 7.28 | 3.98 | 4.67 | 3.00 | 4.08 |
| 4 | 2.70 | 4.95 | 2.93 | 3.50 | 2.50 | 3.25 |
| 5 | 2.75 | 4.43 | 3.15 | 3.67 | 2.50 | 3.25 |
| 6 | 2.46 | 4.87 | 2.94 | 3.50 | 2.50 | 3.25 |
| 7 | 3.88 | 5.38 | 3.40 | 3.92 | 3.00 | 4.50 |
| 8 | 2.58 | 4.48 | 3.17 | 3.75 | 2.50 | 3.25 |
| 9 | 2.97 | 4.40 | 3.13 | 3.75 | 2.50 | 3.00 |
| 10 | 3.75 | 5.67 | 3.97 | 4.75 | 3.25 | 4.50 |
| 11 | 4.92 | 6.93 | 3.70 | 4.50 | 3.50 | 5.17 |
| 12 | 4.22 | 7.62 | 4.87 | 5.75 | 3.00 | 5.00 |
| 13 | 2.62 | 6.67 | 3.14 | 3.75 | 2.25 | 3.50 |
| 14 | 4.00 | 8.08 | 3.64 | 4.50 | 3.25 | 4.50 |
| 15 | 3.50 | 6.00 | 3.94 | 4.50 | 2.50 | 4.50 |
| 16 | 2.78 | 4.07 | 3.11 | 3.75 | 2.50 | 3.50 |
| 17 | 3.00 | 6.08 | 2.85 | 3.50 | 2.50 | 3.50 |
| 18 | 6.35 | 7.50 | 6.36 | 7.00 | 5.92 | 6.62 |
| 19 | 3.93 | 17.00 | 5.78 | 11.00 | 3.25 | 8.50 |
| 20 | 2.93 | 5.42 | 3.24 | 3.75 | 2.50 | 3.50 |
| Average | 3.47 | 6.41 | 3.70 | 4.60 | 2.87 | 4.20 |
| stdev | 0.94 | 2.76 | 0.94 | 1.73 | 0.82 | 1.36 |
| cov (%) | 27.21 | 43.00 | 25.53 | 37.68 | 28.47 | 32.31 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 22**

**Average set time results for all set time tests for Source 10**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 2 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 3 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 4 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 5 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 6 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 7 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 8 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 9 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 10 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 11 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 12 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 13 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 14 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 15 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 16 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 17 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 18 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 19 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 20 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| Average | N/A | N/A | N/A | N/A | N/A | N/A |
| stdev | N/A | N/A | N/A | N/A | N/A | N/A |
| cov (%) | N/A | N/A | N/A | N/A | N/A | N/A |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Table 23**

**Average set time results for all set time tests for Source 11**

| | Gillmore Needle | | Vicat Needle | | Pocket Penetrometer | |
|---|---|---|---|---|---|---|
| Bucket Number | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) | Initial Set (Minutes) | Final Set (Minutes) |
| 1 | 240+ | 240+ | 240+ | 240+ | 66.00 | 240+ |
| 2 | 130.00 | 240+ | 214.86 | 240+ | 30.00 | 240+ |
| 3 | 240+ | 240+ | 135.00 | 160.00 | 71.00 | 230.00 |
| 4 | 89.00 | 240+ | 240+ | 240+ | 17.00 | 210.00 |
| 5 | 170.00 | 240+ | 240+ | 240+ | 55.00 | 240+ |
| 6 | 178.00 | 240+ | 240+ | 240+ | 25.00 | 240+ |
| 7 | 240+ | 240+ | 240+ | 240+ | 51.00 | 240+ |
| 8 | 240+ | 240+ | 240+ | 240+ | 38.00 | 240+ |
| 9 | 240+ | 240+ | 240+ | 240+ | 98.00 | 240+ |
| 10 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 11 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| 12 | 240+ | 240+ | 240+ | 240+ | 240+ | 240+ |
| Average | 141.75 | N/A | 174.93 | 160.00 | 50.11 | 220.00 |
| stdev | 40.96 | N/A | 56.47 | N/A | 25.70 | 14.14 |
| cov (%) | 28.89 | N/A | 32.28 | N/A | 51.29 | 6.43 |

*Note each set time test result is an average of three tests

†Note that outliers are shaded gray

‡Results noted as 240+ indicate samples that never set after 240 minutes

**Statistical Modeling**

JMP 8.0 software was used in determining suitable models [*11*]. Input variables and their respective units are shown in Table 24. Response variables and their respective units are shown in Table 25. Note that attempts at simple linear regression modeling using one variable produced very low correlation values. To obtain a statistically significant model with the least number of variables, stepwise regression techniques were used alongside their respective residual plots to determine significance of the various input variables.

**Table 24**
**Input variables and units for statistical modeling**

| Variable | Unit(s) |
|---|---|
| Silicon Dioxide | % |
| Ammonium Hydroxide Group | % |
| Sulfur Trioxide | % |
| Magnesium Oxide | % |
| Calcium Oxide | % |
| Fineness | % Retained on the 325 Sieve |

**Table 25**
**Response variables and units for statistical modeling**

| Response Variable | Unit(s) |
|---|---|
| Gillmore Initial Set | Minutes |
| Gillmore Final Set | Minutes |
| Vicat Initial Set | Minutes |
| Vicat Final Set | Minutes |
| Pocket Penetrometer Initial Set | Minutes |
| Pocket Penetrometer Final Set | Minutes |
| Maximum Temperature | °C |
| Time to Maximum Temperature | Minutes |

Numerous attempts to obtain suitable models were attempted. The author was unable to find suitable models (i.e., $R^2$ values were less than 0.5) to describe the initial and final set behavior and the time to maximum temperature for the data set using the input parameters defined in Table 24.

**Least Squares Regression Analysis Equation for Maximum Temperature**

Equation (1) shows the regression equation for the maximum temperature. Note the good correlation coefficient of 0.6255.

Maximum Temperature = $-.406SiO_2$ + 0.236Ammonium Hydroxide + $2.56SO_3$ + .941MgO + 1.162CaO - .209Fineness + 4.015

**Stepwise Regression Analysis Equation for Maximum Temperature**

Equation (2) shows the stepwise regression analysis results for the maximum temperature. Note the slight reduction of the correlation coefficient from 0.6255 to 0.5798. Although the correlation coefficient has been reduced, the model makes sense due to the parameters of calcium oxide and sulfur trioxide. The addition of water to both of these compounds creates an exothermic chemical reaction giving off measurable heat.

Maximum Temperature = $3.612SO_3$ + 1.159CaO - 4.006

The author believes the lack of modeling results show that hydration of self cementing fly ash is a complex, poorly understood problem that can be addressed by further research. That being said, the author believes that the tricalcium aluminate content of the fly ash may be able to better define the set time and hydration characteristics. The drawback of using the tricalcium aluminate content is accurately quantifying or determining it. The tricalcium aluminate of fly ash can only be determined through semi-quantitative x-ray diffraction techniques that can be costly and very time consuming. With this knowledge, the author recommends that if the Department wishes to further define the set time characteristics, a smaller x-ray diffraction testing regime be developed to determine the role of tricalcium aluminate in the set time or hydration process of class C fly ash. However the author does not recommend this expense at this time.

# CONCLUSIONS

The results of this study warrant the following conclusions. The Gillmore needle, Vicat needle, and the pocket penetrometer yielded similar results when observing the times to initial and final set across the three test methods; therefore, any of these devices may be used to determine set time. Although all test methods pointed out significant differences in set times between buckets within a source, those differences were mitigated when incorporating portland cement into the sample. In other words, blending fly ash with portland cement normalized the set time of the fly ash, even from a source exhibiting high variability in set times when incorporated at 50 percent.

The temperature results showed that the coffee cup test method is unable to be used as either a quality control or quality assurance device in characterizing class C fly ash.

The statistical analysis results showed outliers within the sources, but further testing when incorporating portland cement showed these differences to be negligible.

A suitable correlation was found to exist between the calcium oxide and sulfur trioxide content and the maximum temperature of the fly ash temperature results.

# RECOMMENDATIONS

The results of this study indicate that the hydration of class C fly ash is a complex phenomenon that cannot be fully described by the tests used in this study. It is recommended that if the Department wants to further define the relationship between fly ash chemistry and fly ash set time, another study be undertaken to look at the tricalcium aluminate content and its role in hydration characteristics of class C fly ash.

The results of this study also show that the current practice of requiring field set time tests to be conducted in the field are adequate and should be continued in quantifying field variation of set time.

# ACRONYMS, ABBREVIATIONS, & SYMBOLS

| | |
|---|---|
| ACAA | American Coal Ash Association |
| COV | Coefficient of variation |
| EPA | Environmental Protection Agency |
| FA | Fly ash |
| FHWA | Federal Highway Administration |
| LADOTD | Louisiana Department of Transportation and Development |
| LTRC | Louisiana Transportation Research Center |
| LOI | Loss on ignition |
| PC | Portland cement |
| PCC | Portland cement concrete |
| QC | Quality control |
| SCM | Supplementary cementitious material |
| XRF | X-ray fluorescence |

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# APPENDIX A

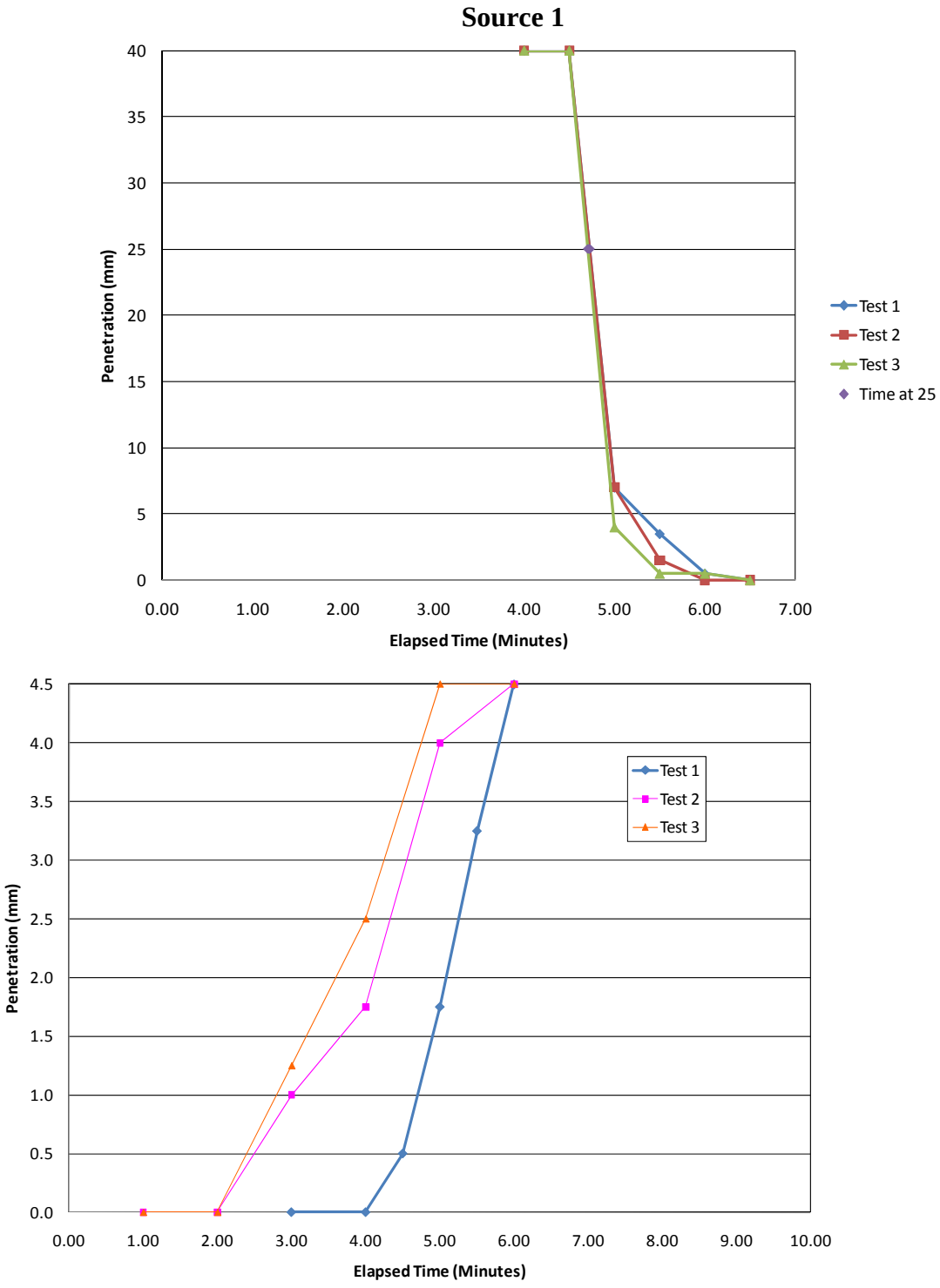



**Figure 25**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 1**

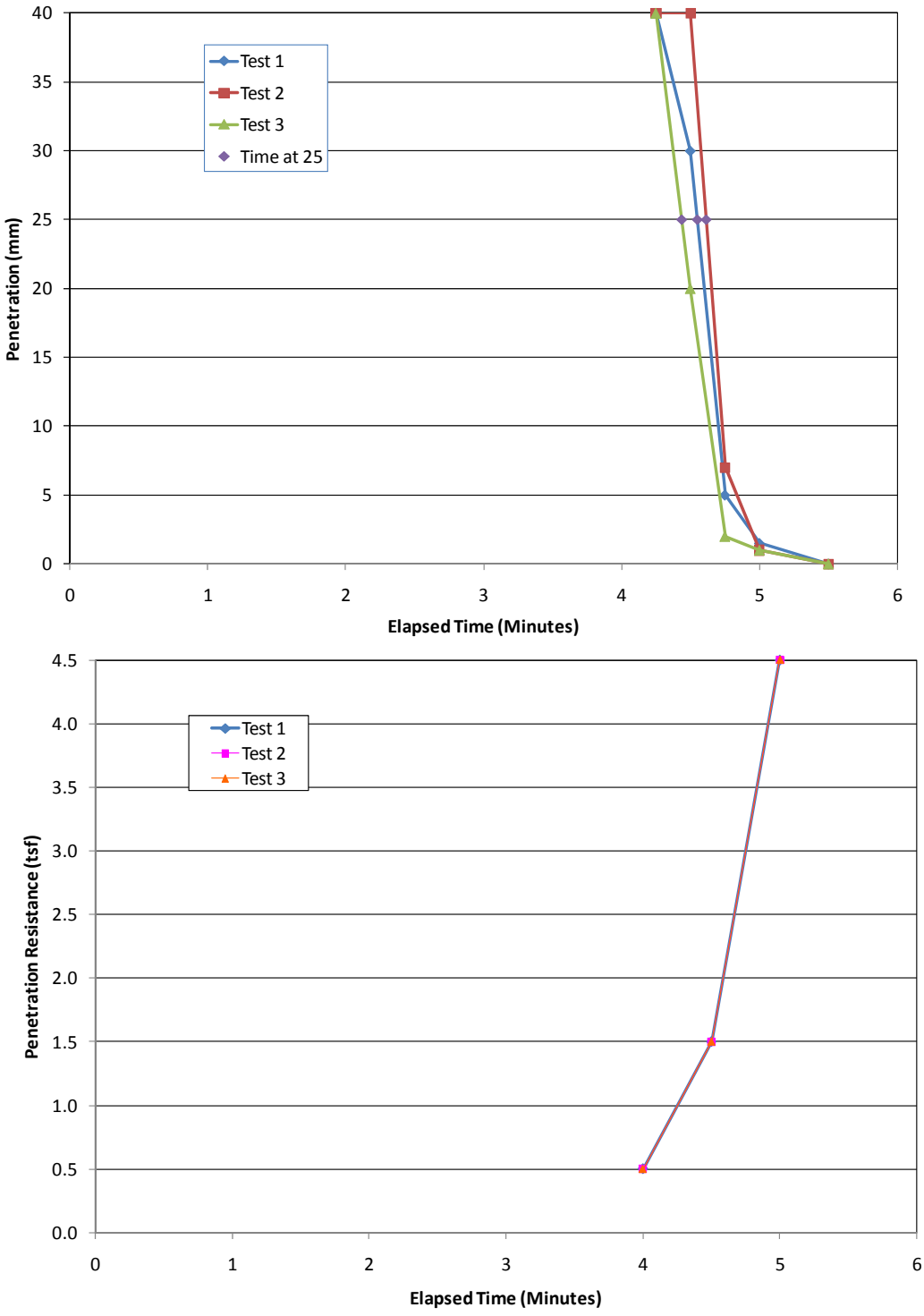
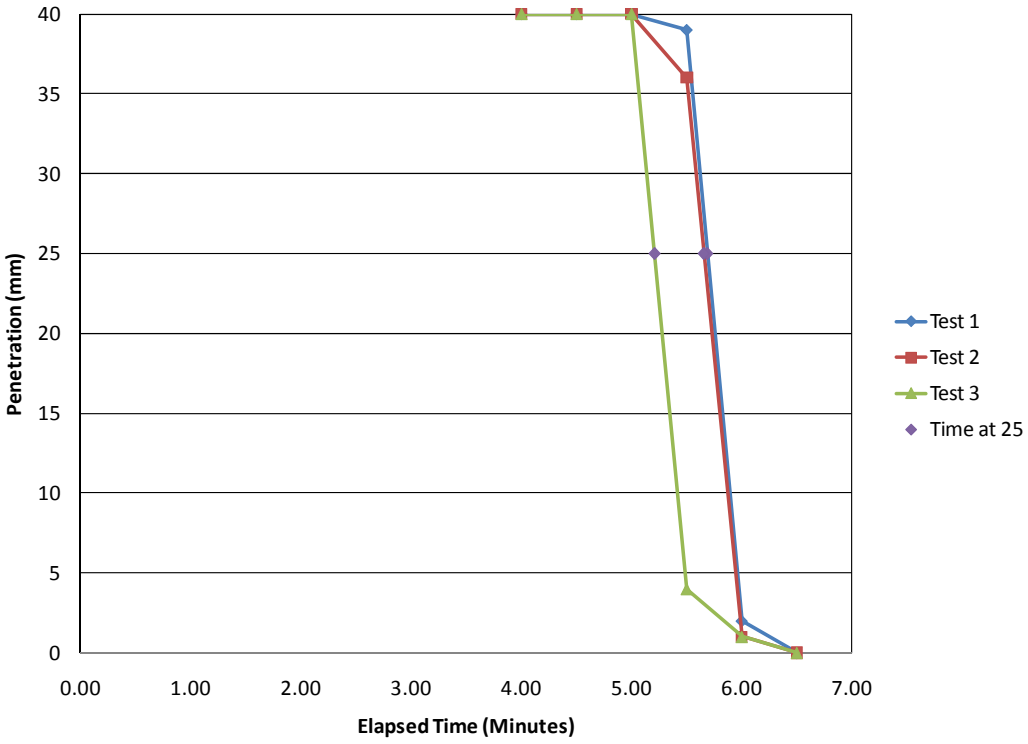



**Figure 26**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 2**



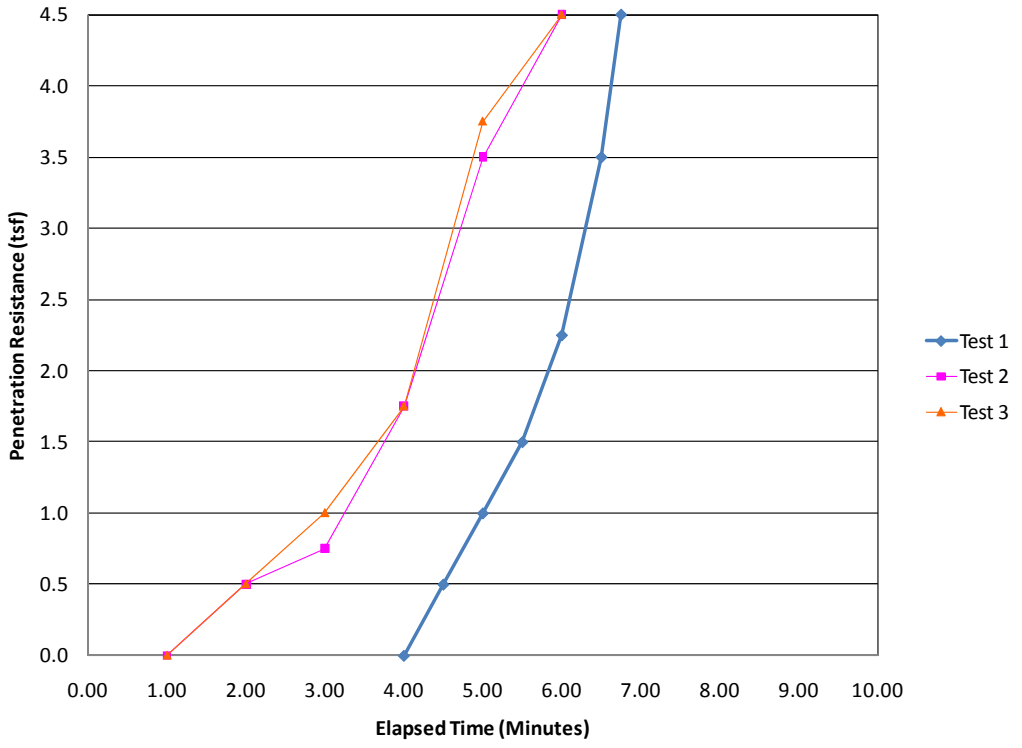
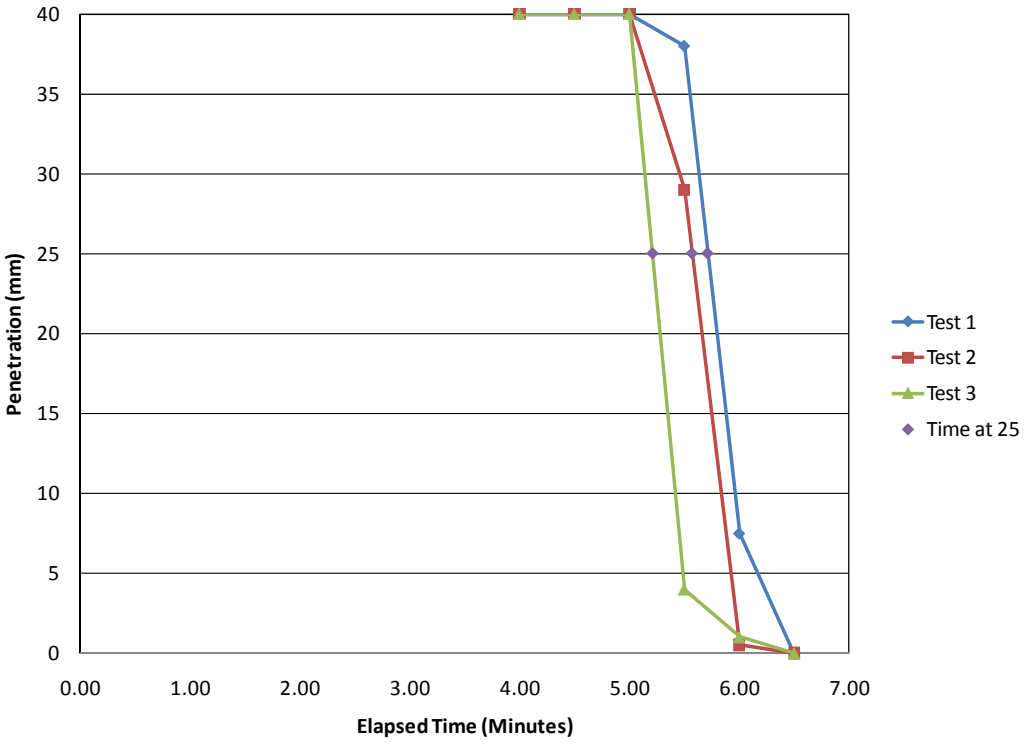




**Figure 27**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 3**



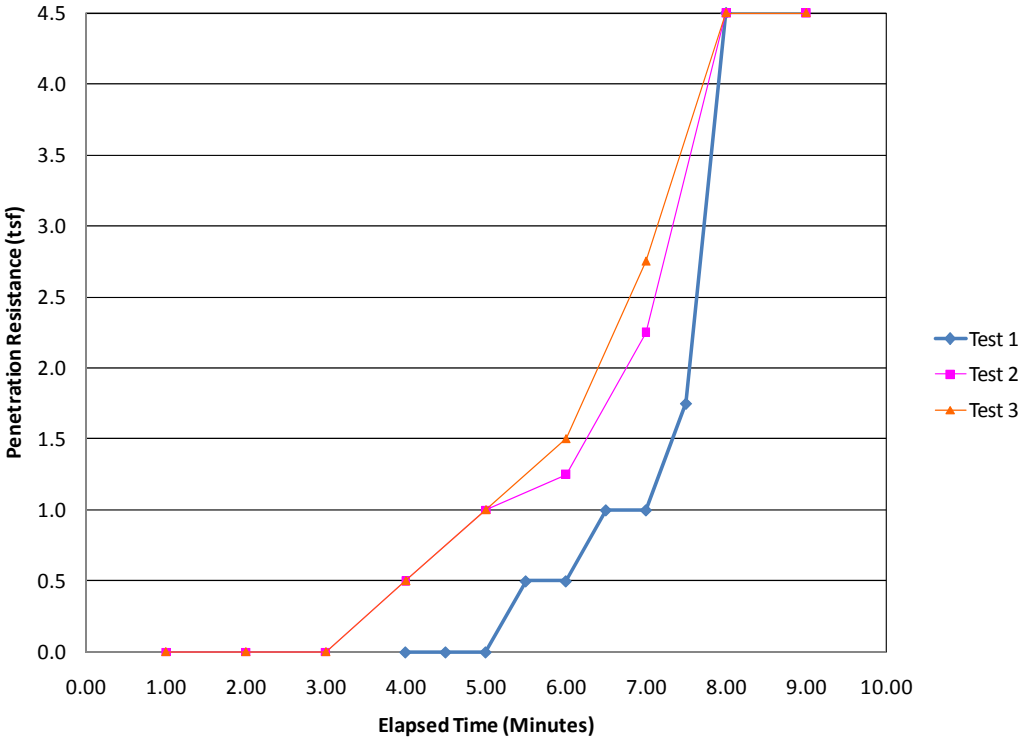
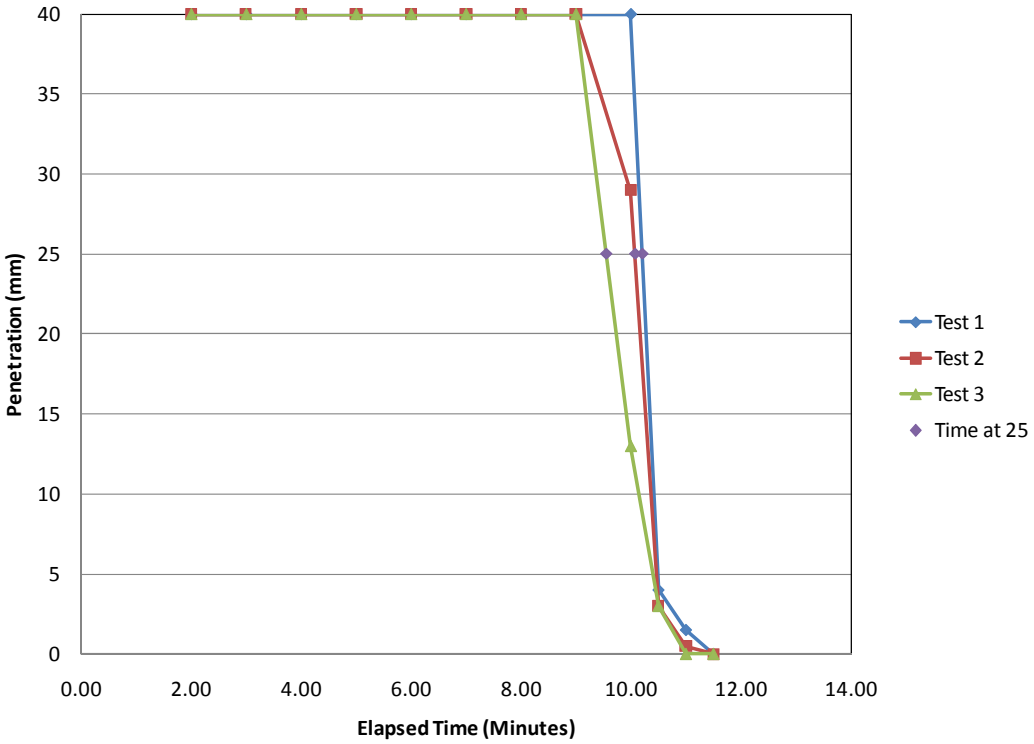




**Figure 28**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 4**



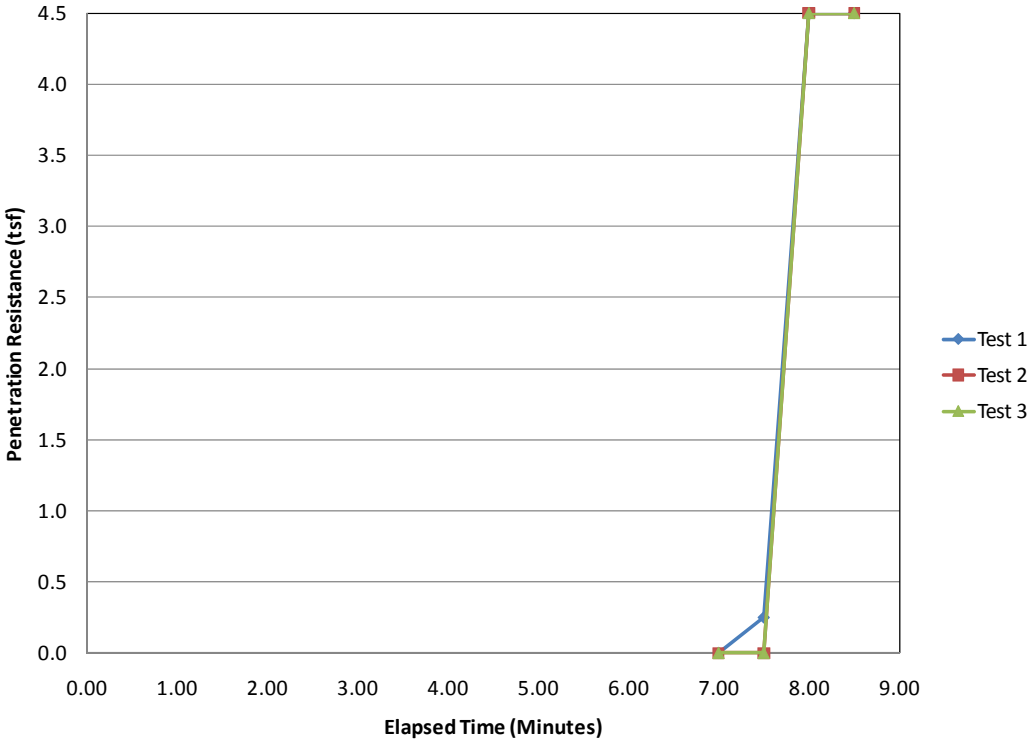




**Figure 29**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 5**

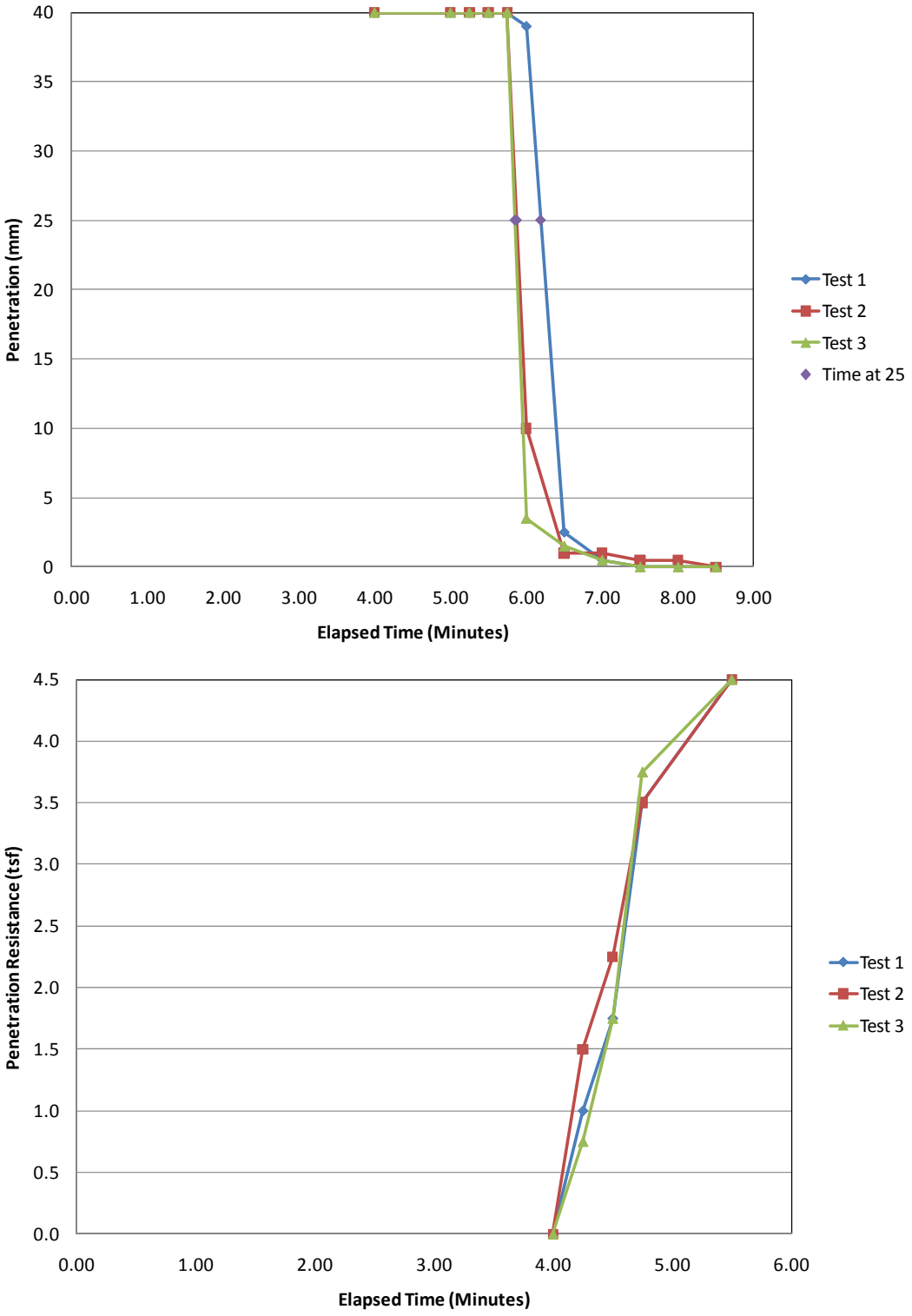
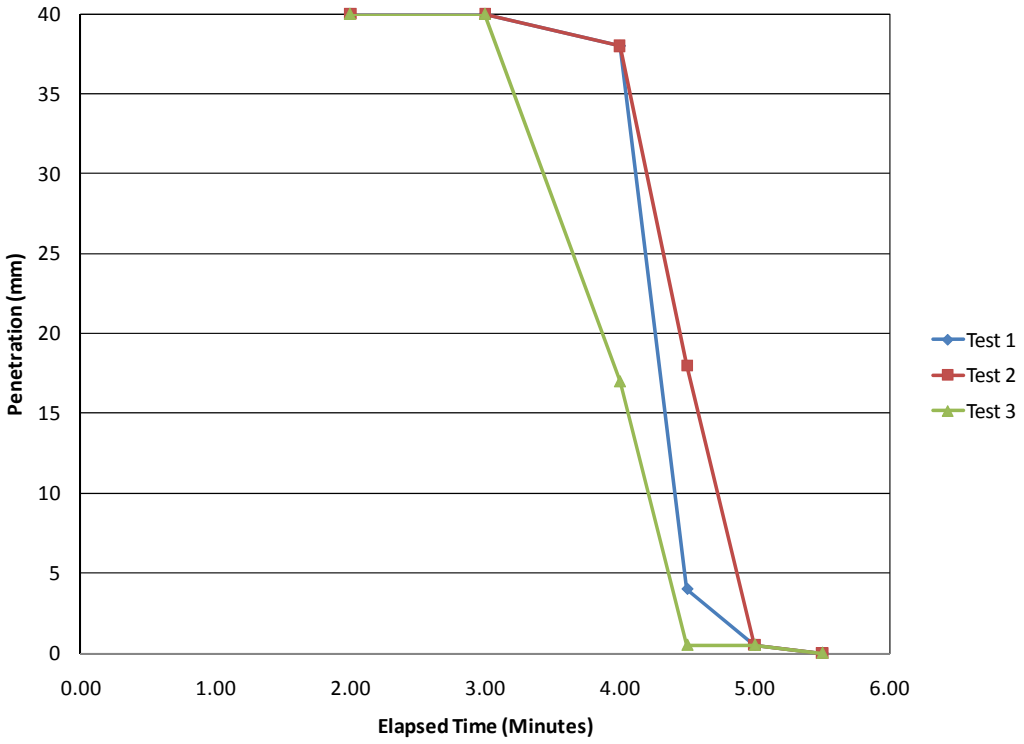



**Figure 30**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 6**



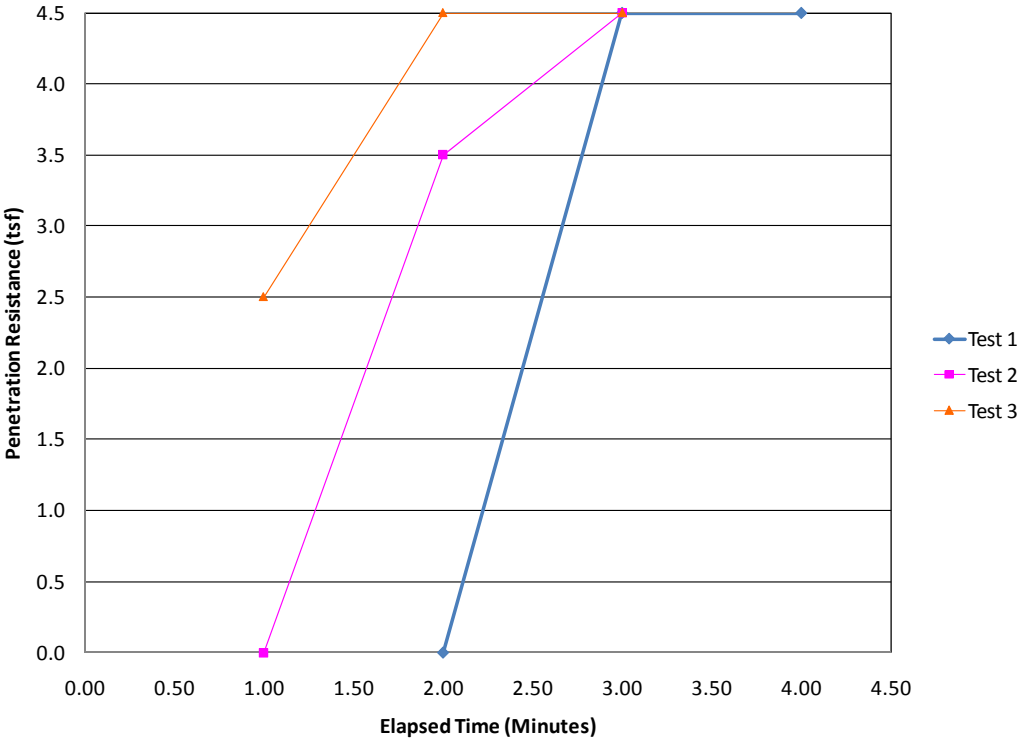
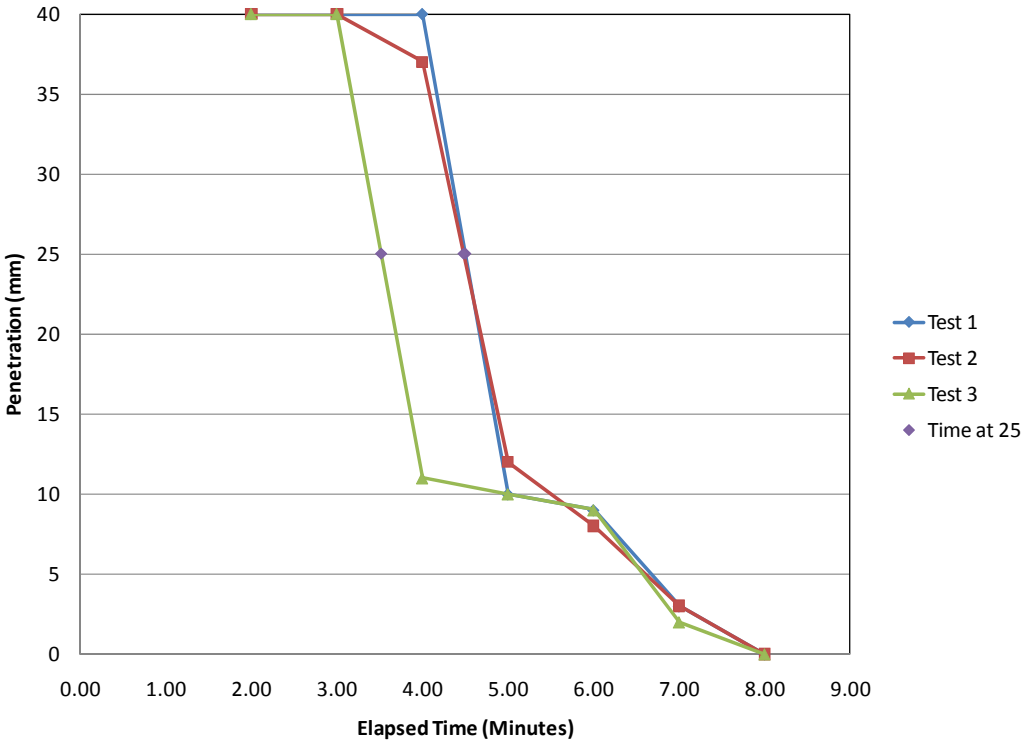




**Figure 31**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 7**



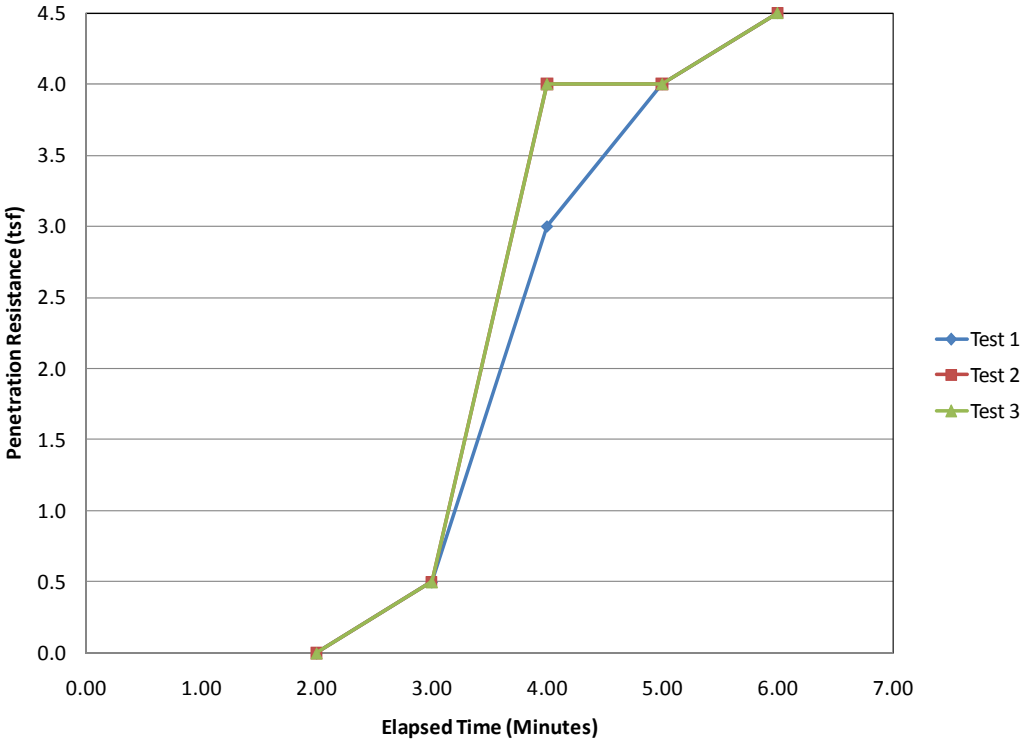
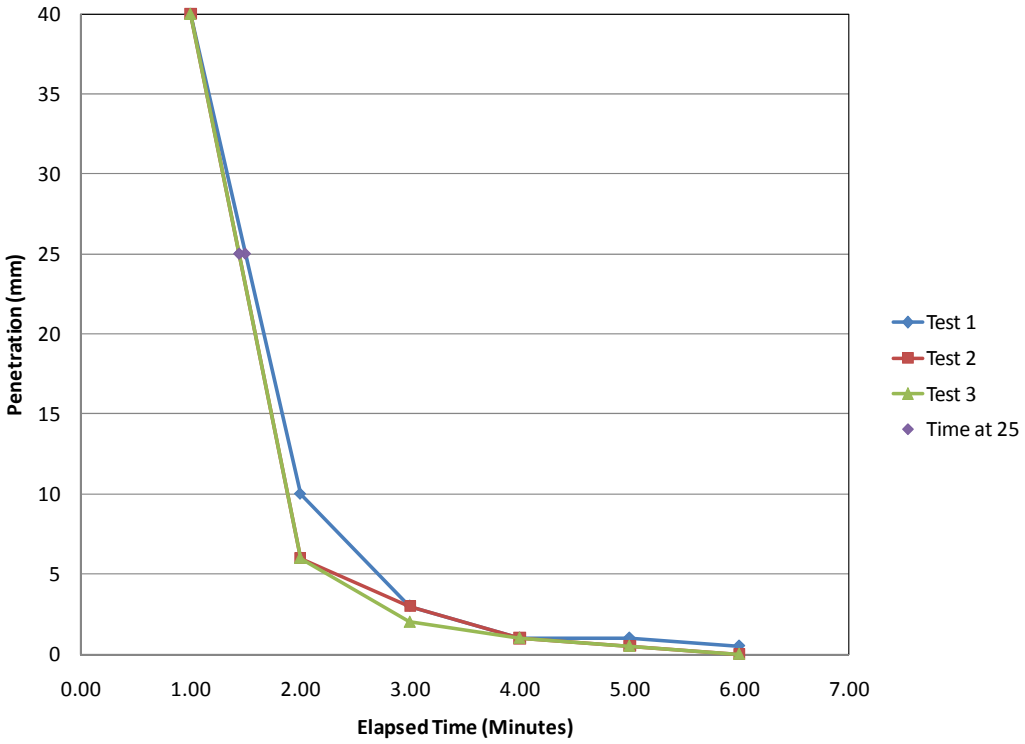




**Figure 32**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 8**



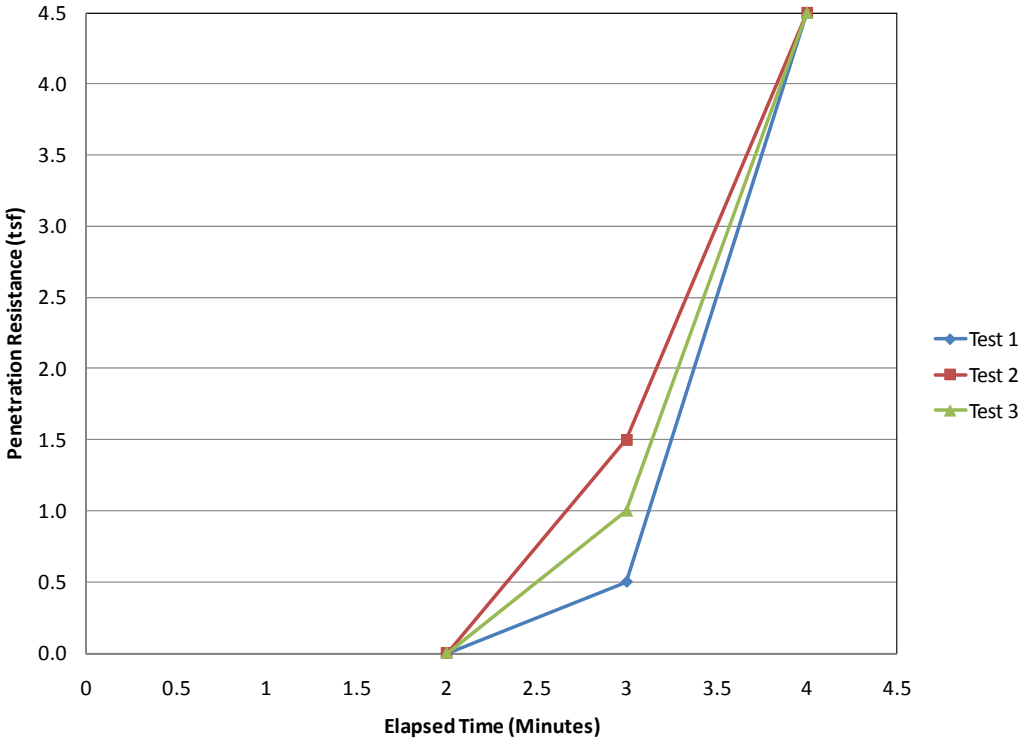
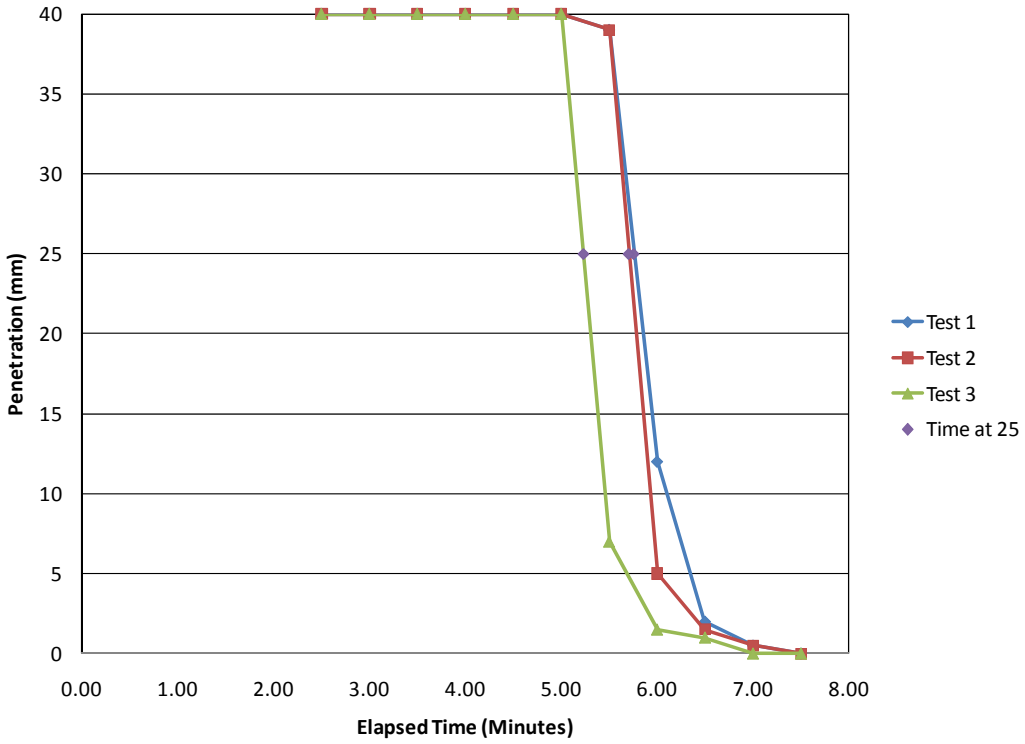




**Figure 33**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 9**



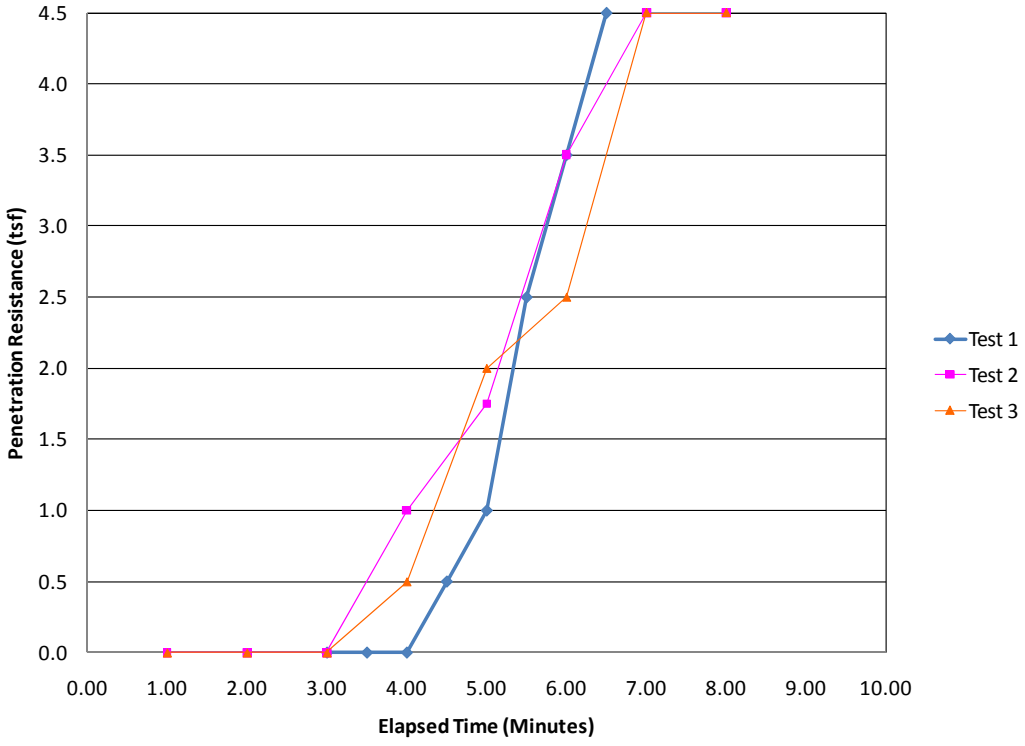




**Figure 34**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 10**

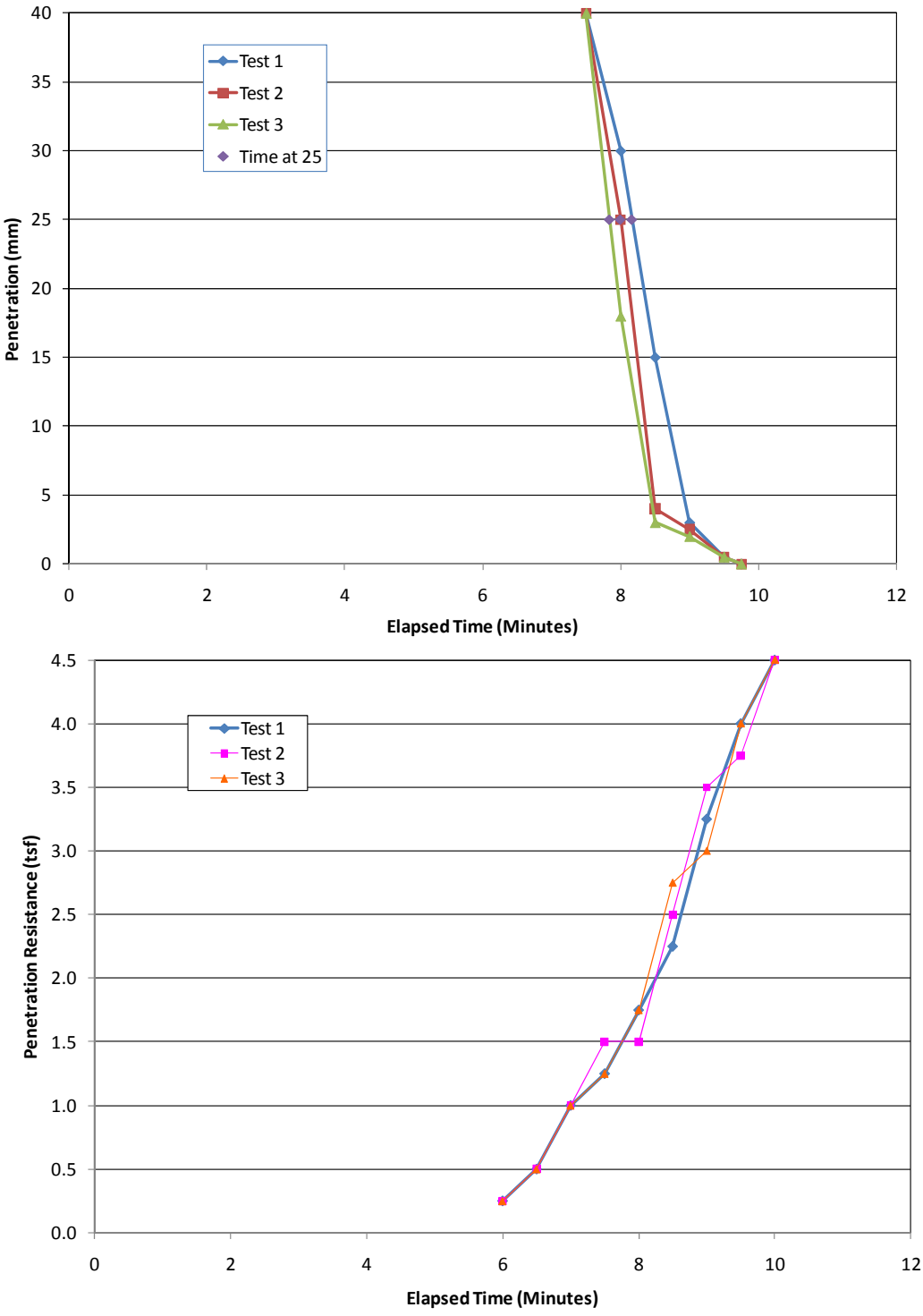



**Figure 35**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 11**

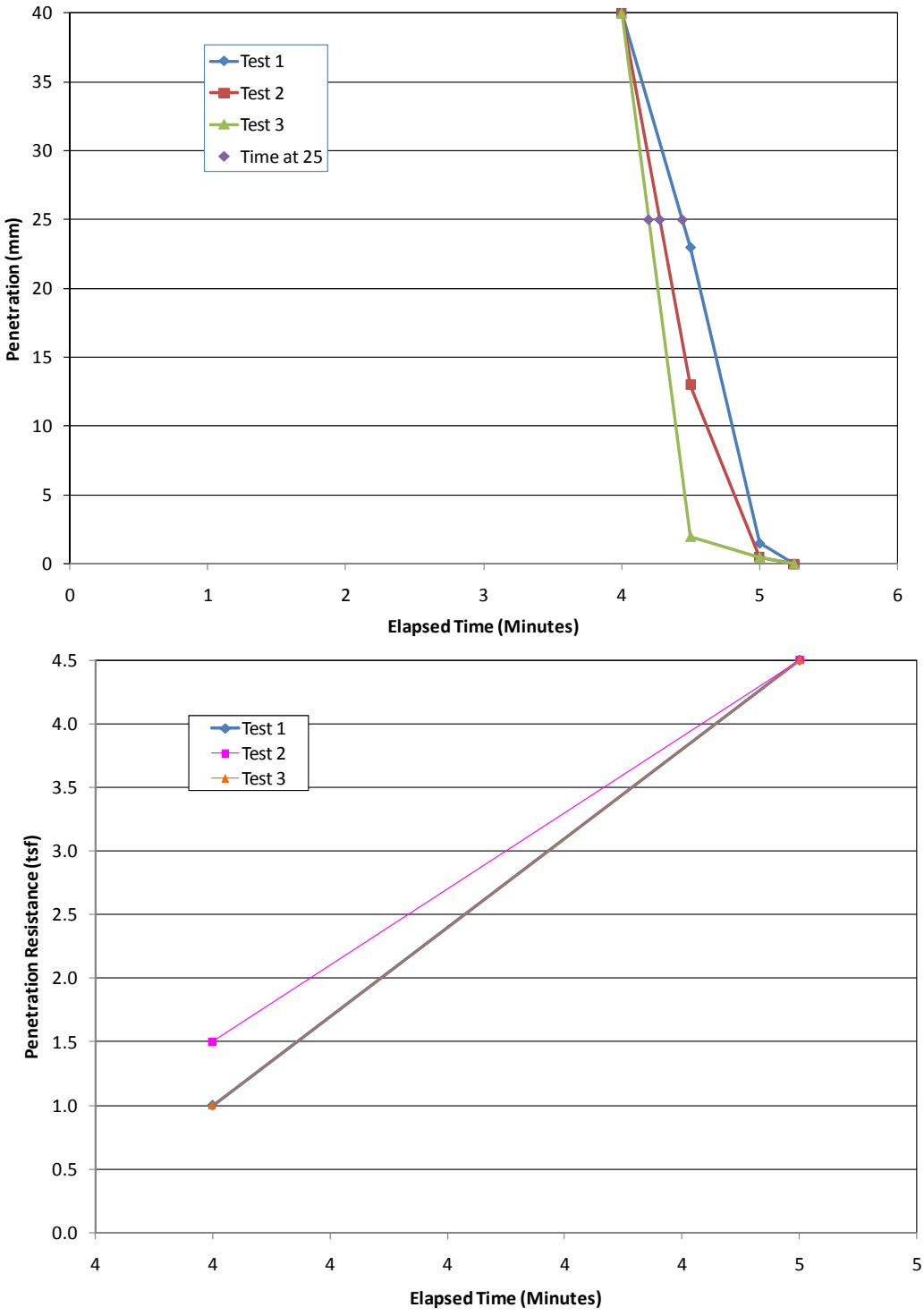
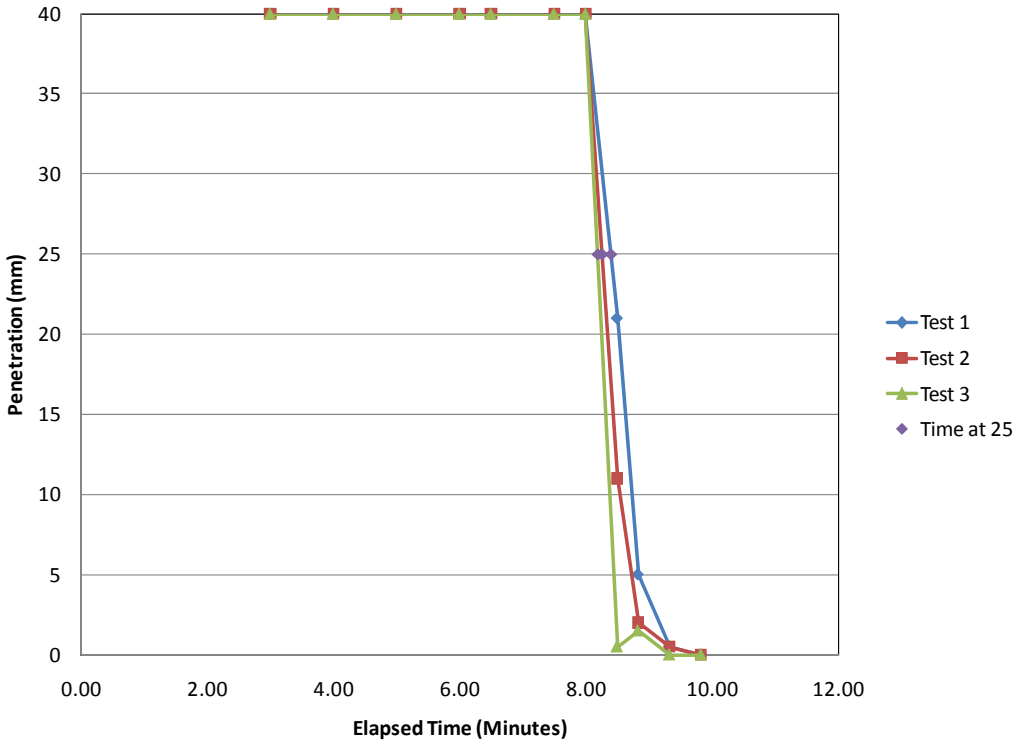



**Figure 36**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 12**



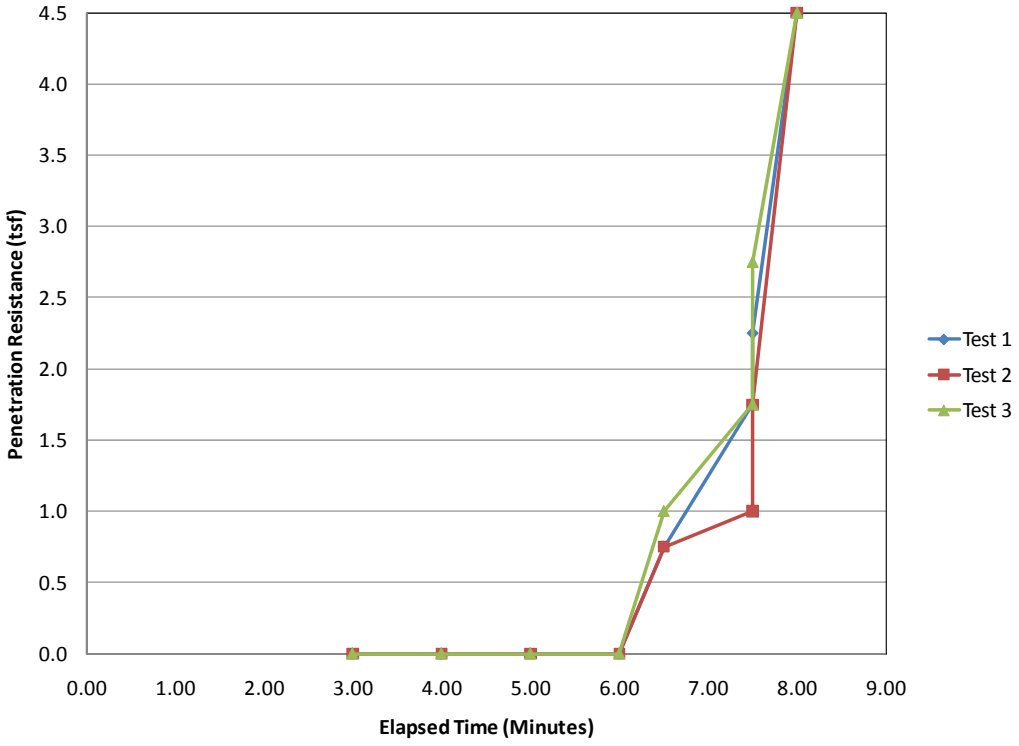
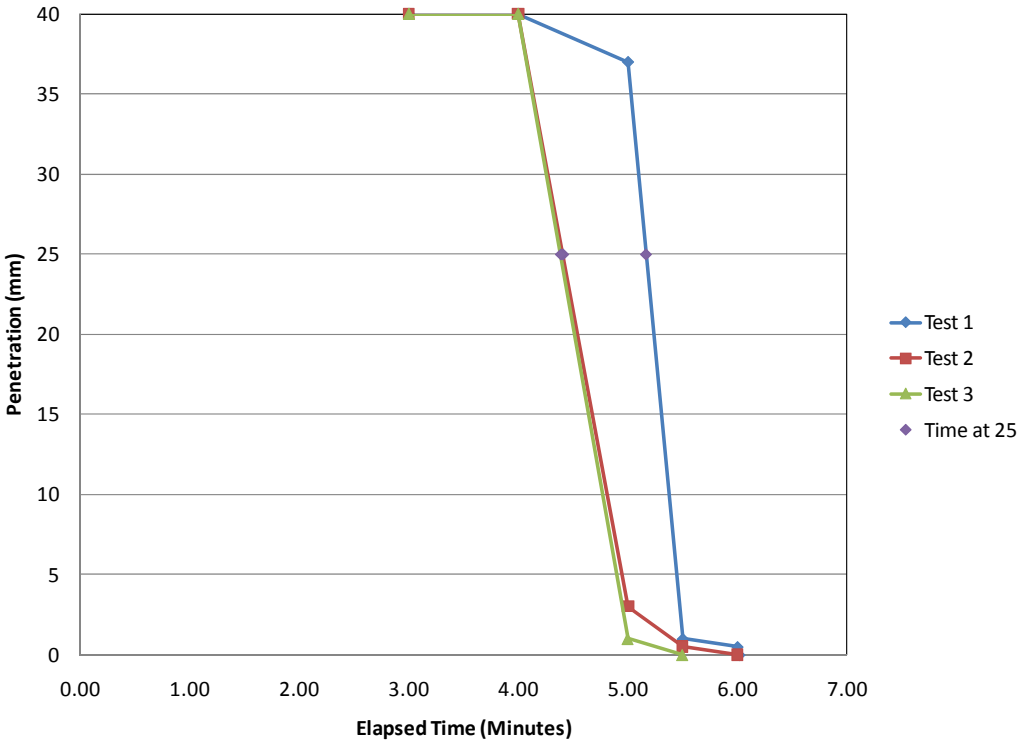




**Figure 37**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 13**



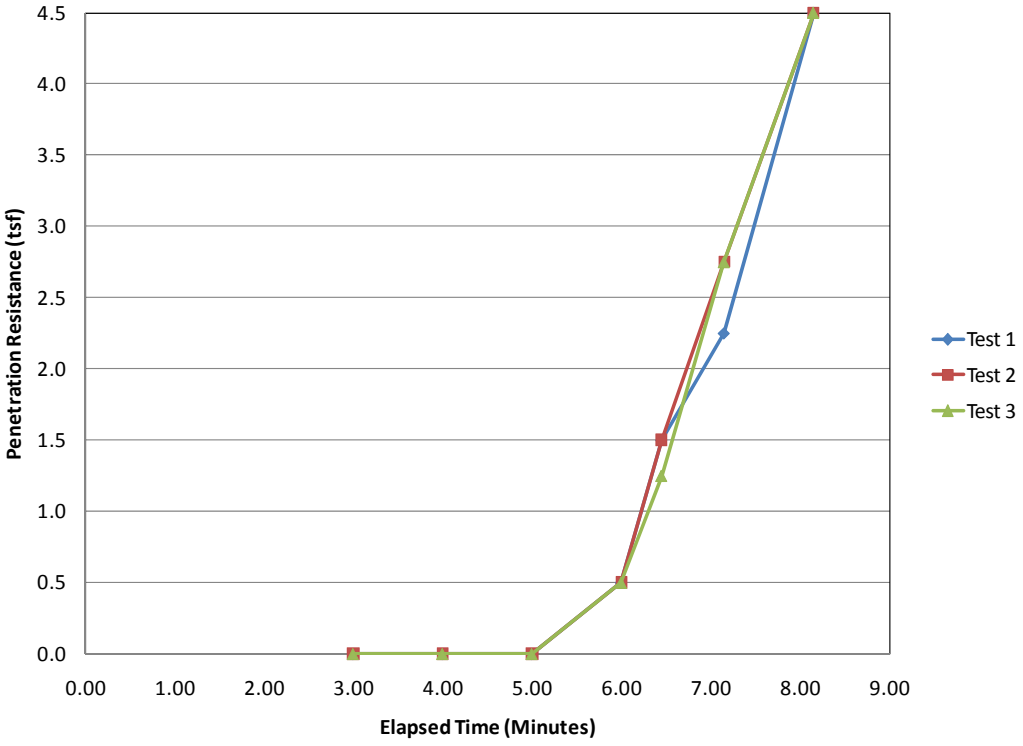
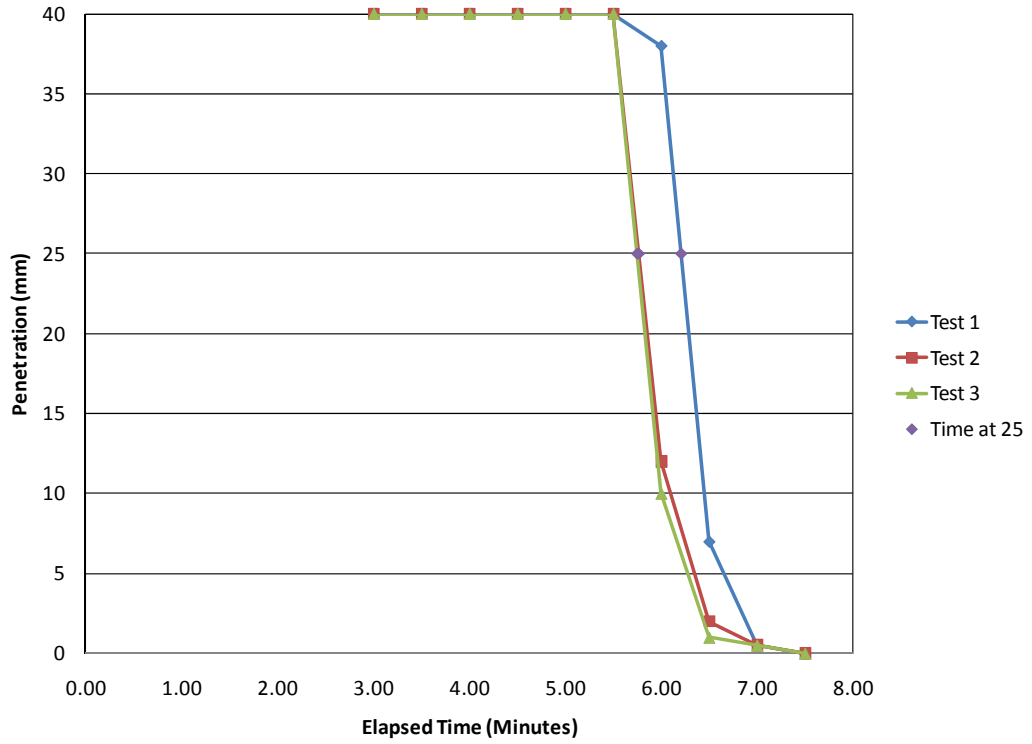




**Figure 38**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 14**



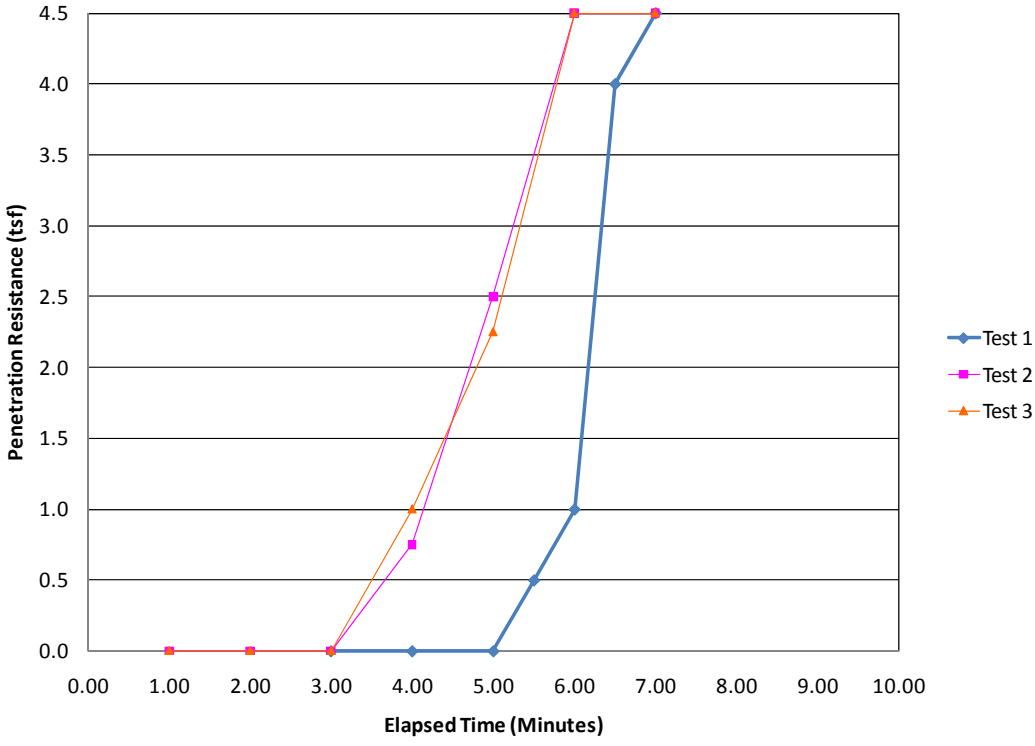
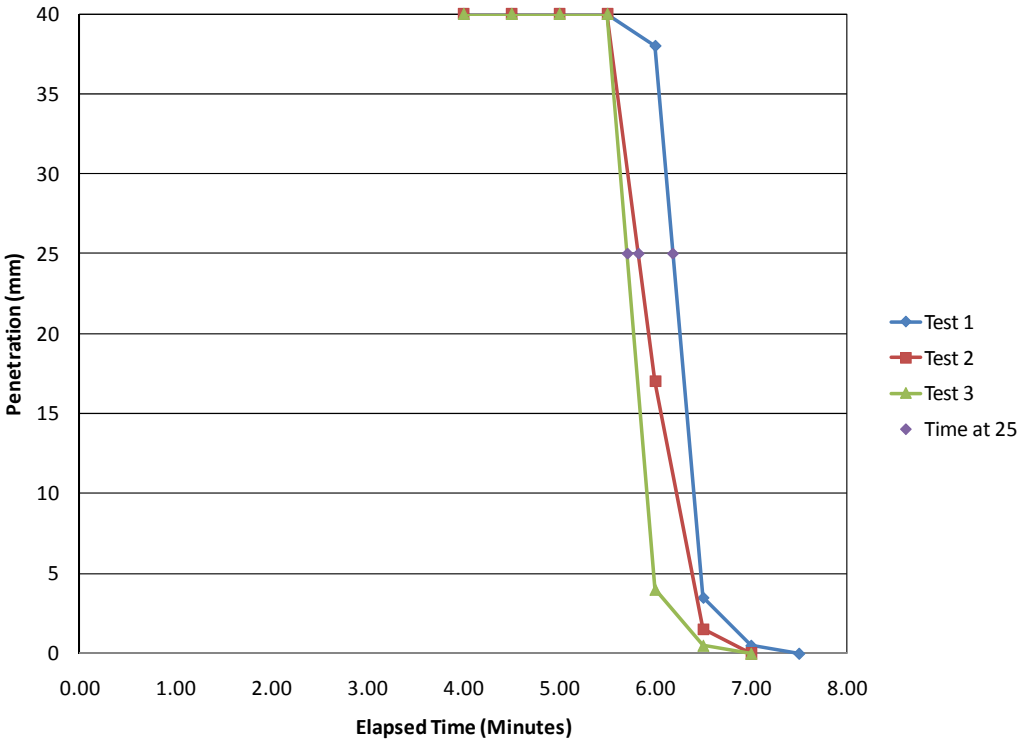




**Figure 39**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 15**



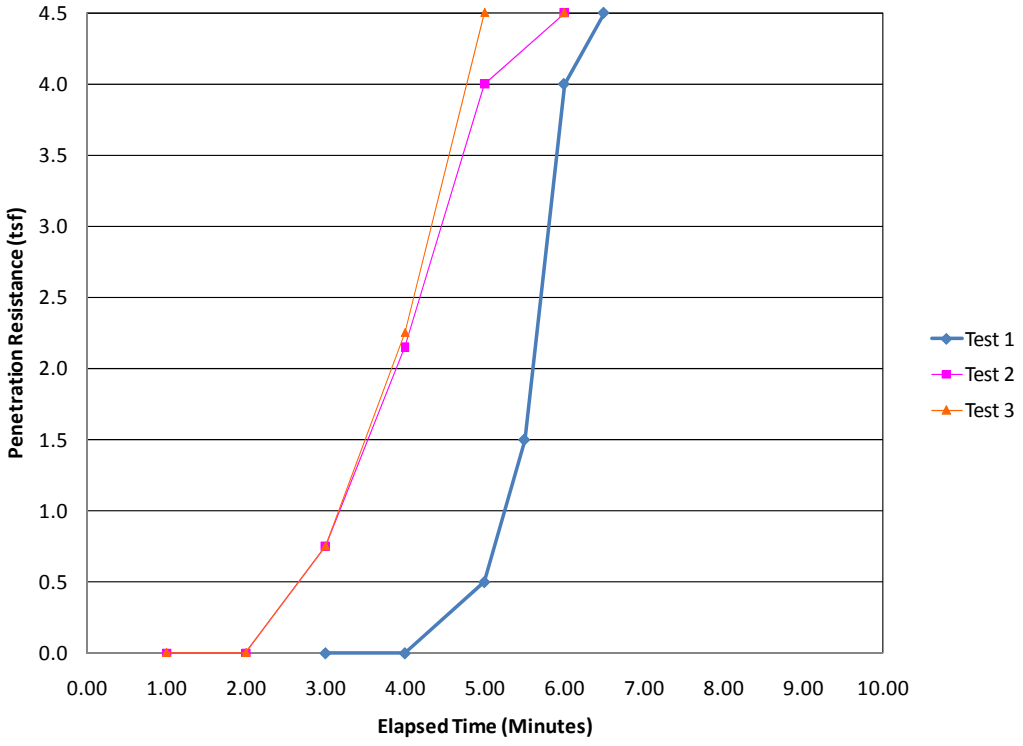
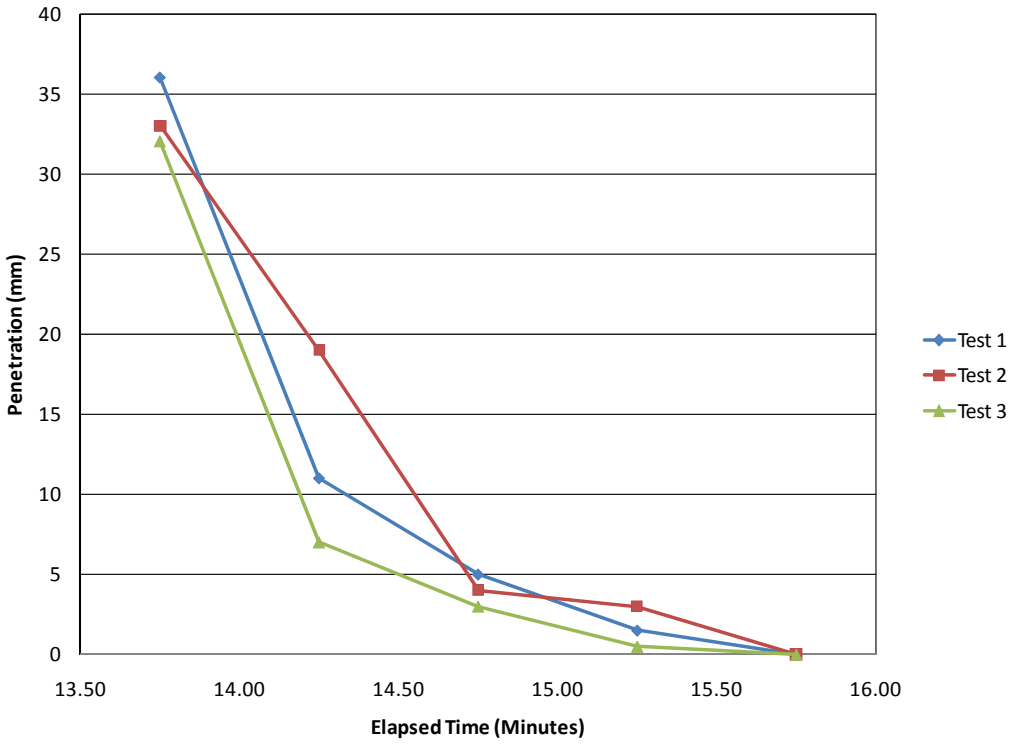




**Figure 40**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 16**



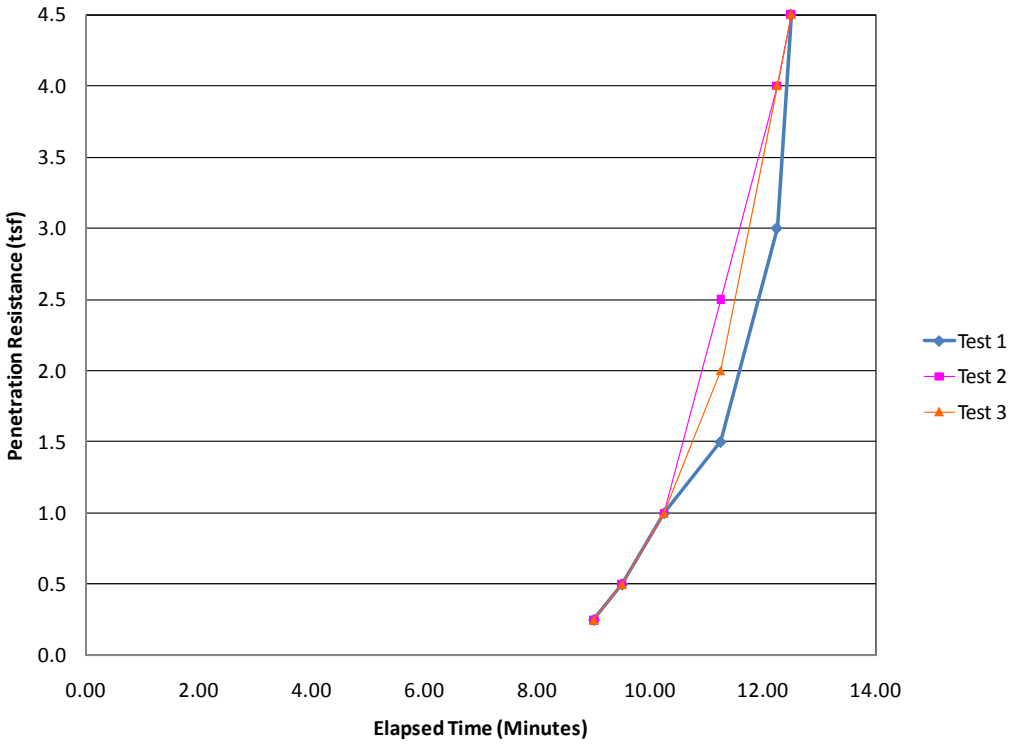
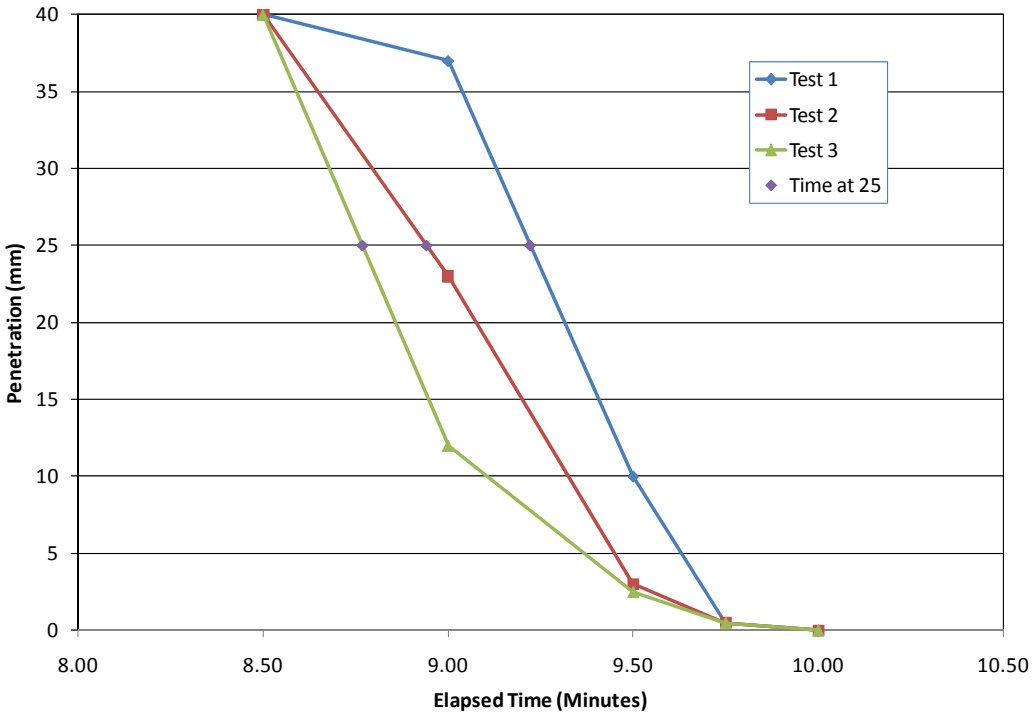




**Figure 41**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 17**



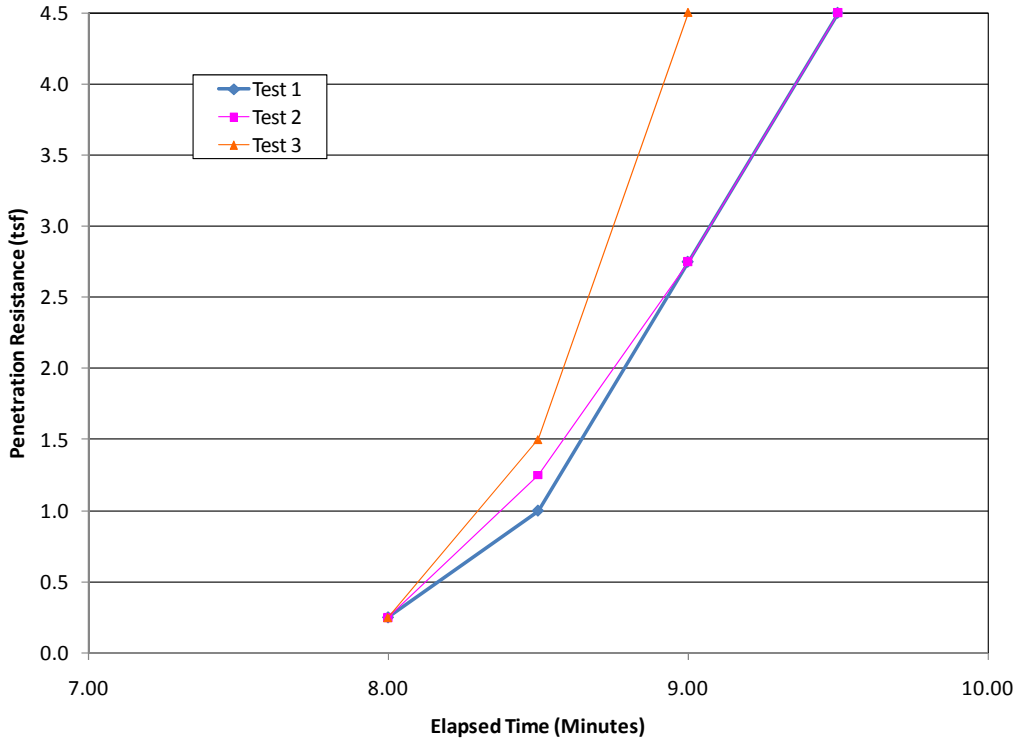




**Figure 42**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 18**

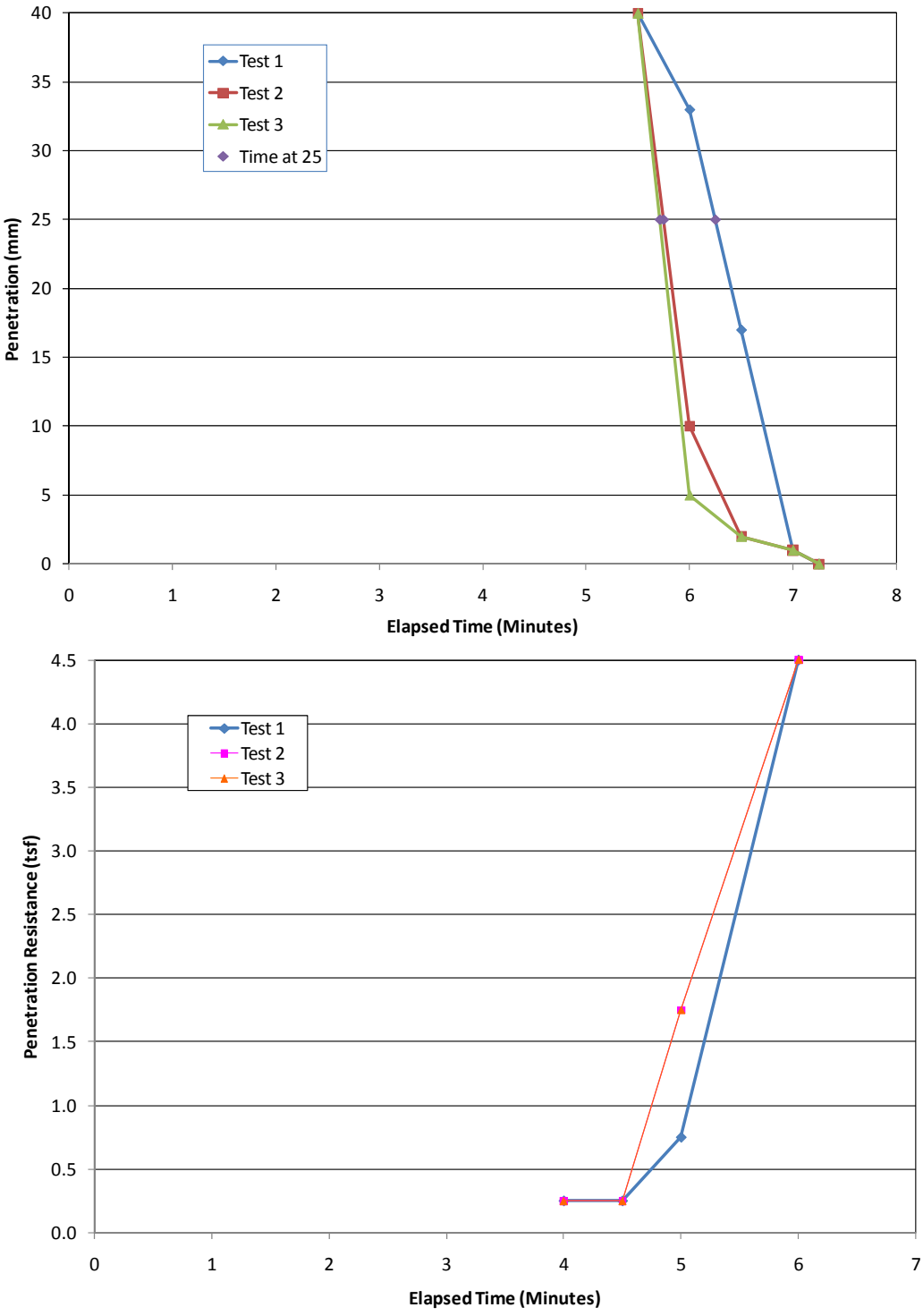



**Figure 43**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 19**

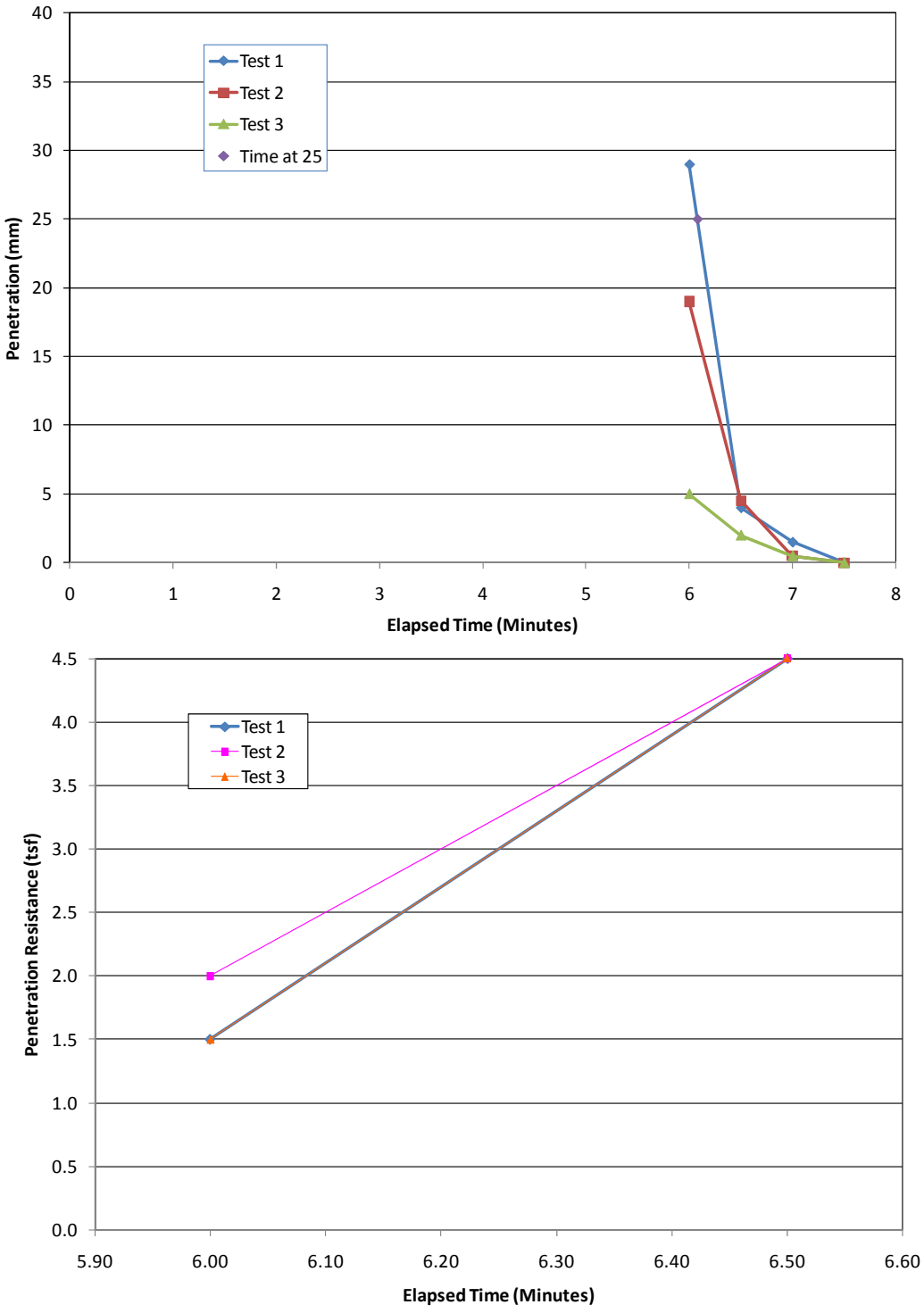



**Figure 44**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 1 Bucket 20**

## Source 2

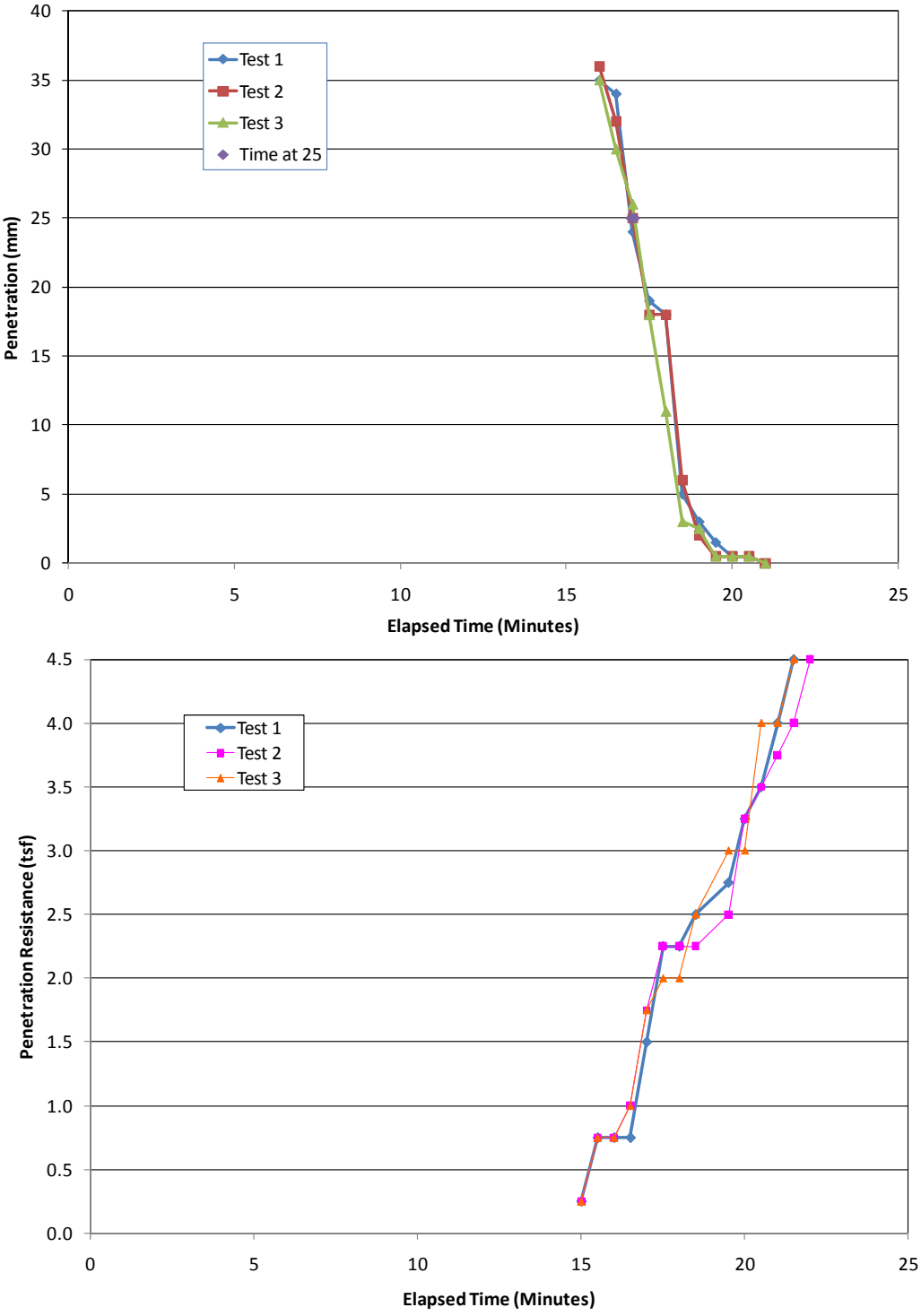



**Figure 45**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 1**

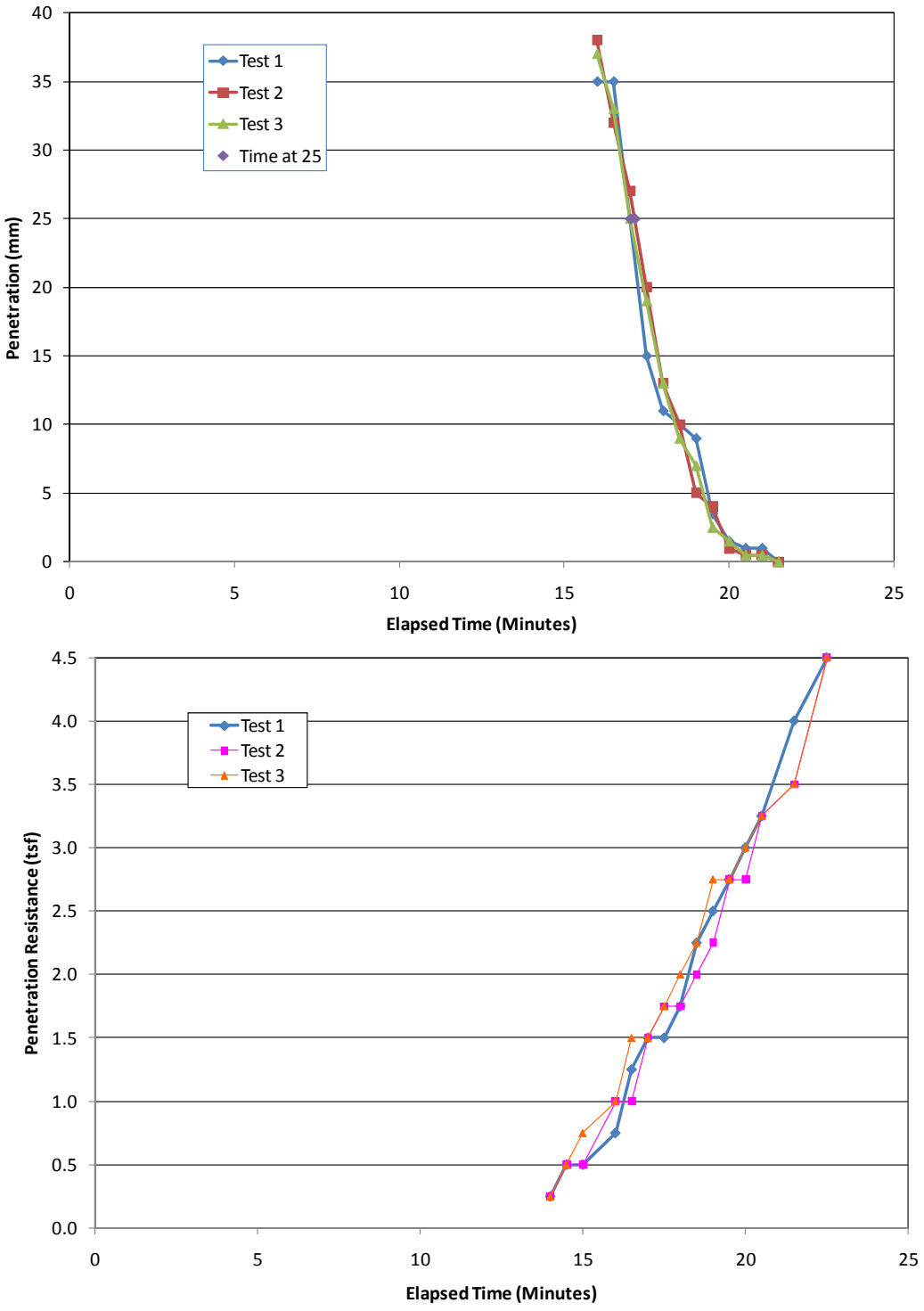



**Figure 46**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 2**

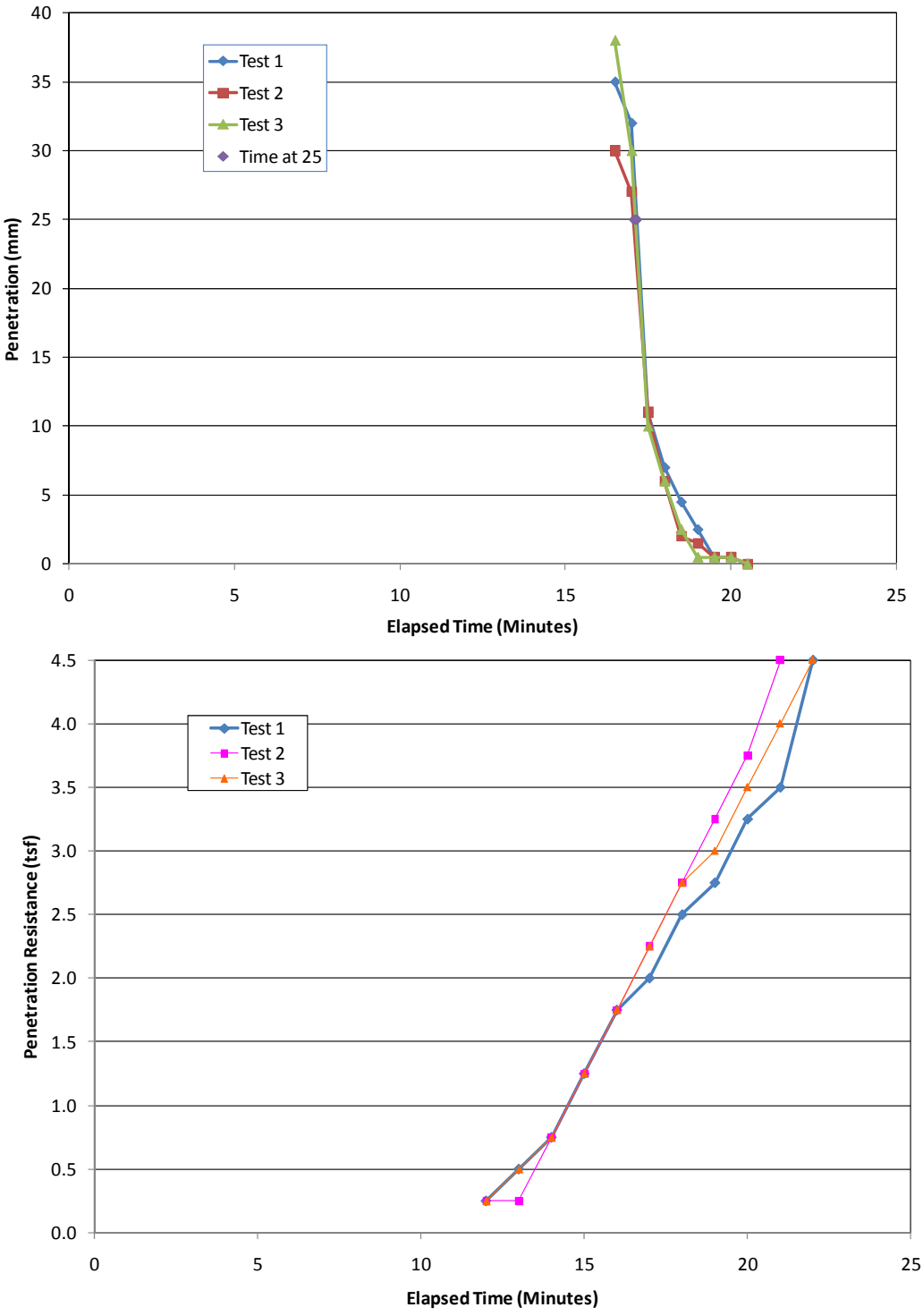



**Figure 47**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 3**

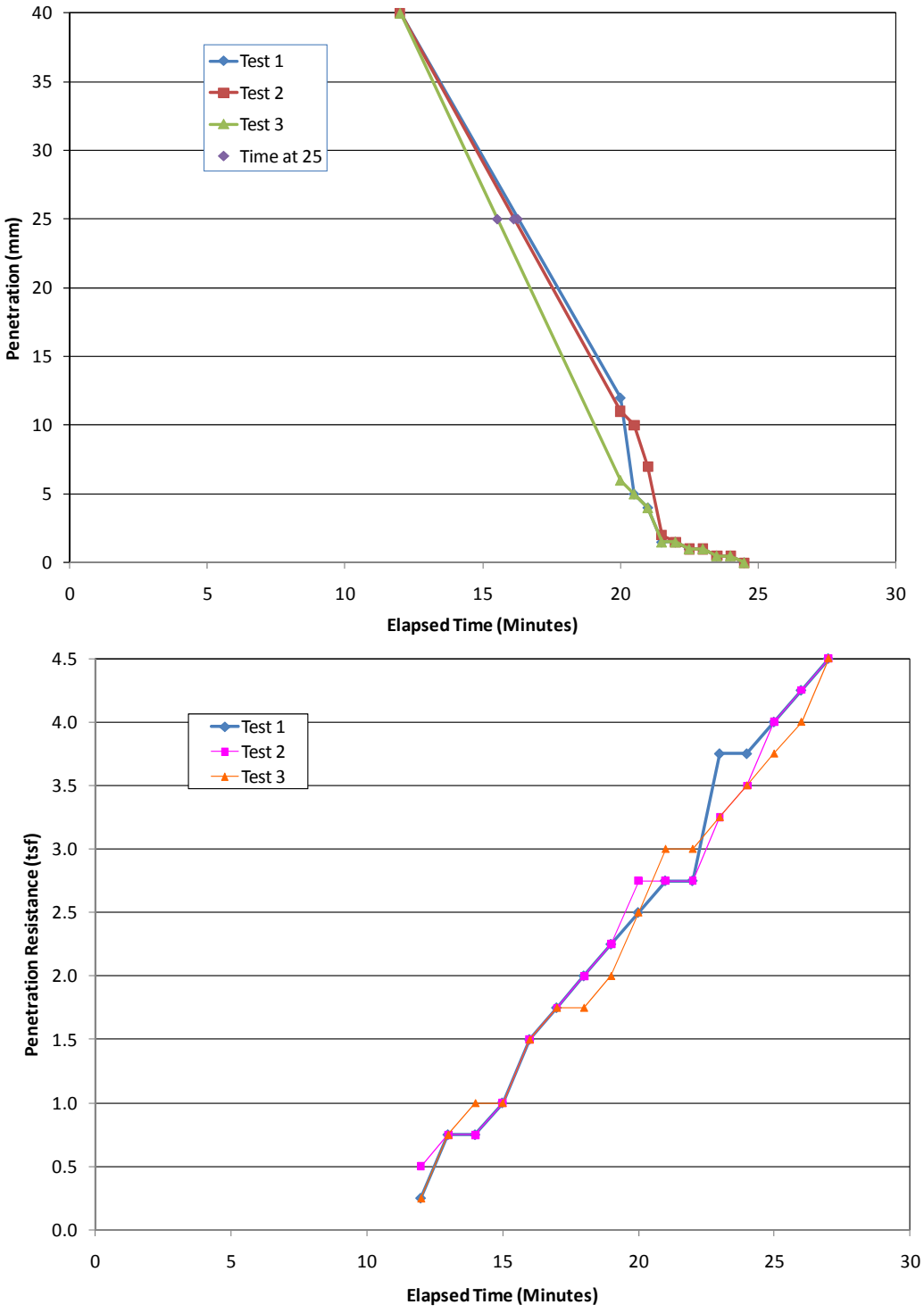
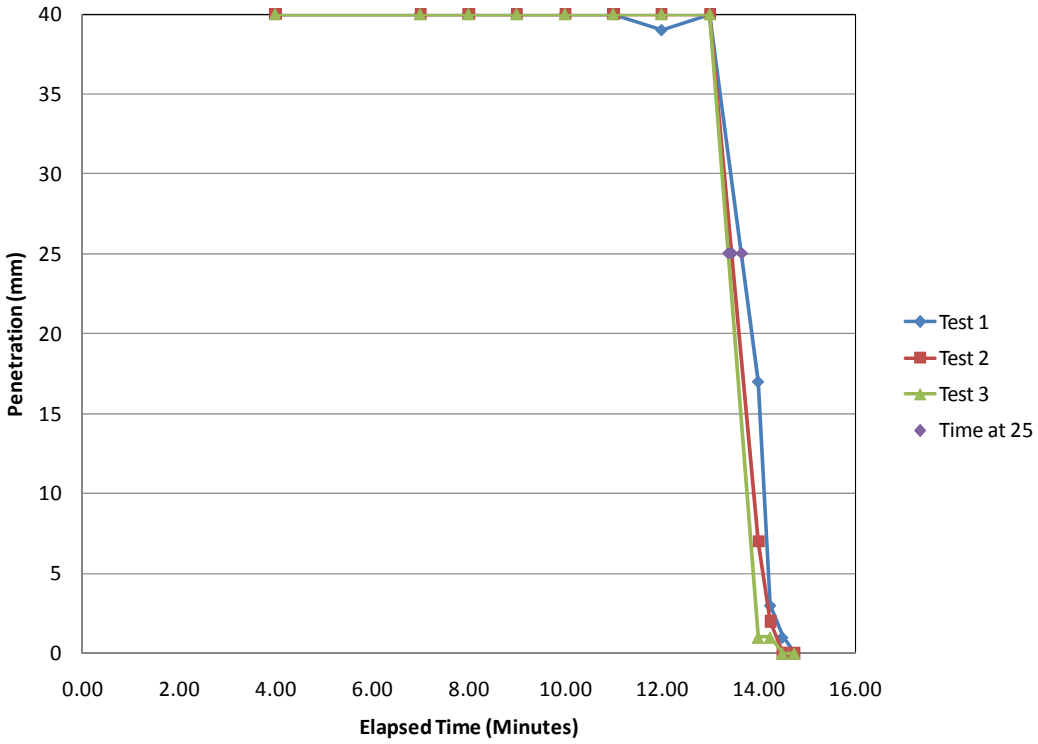



**Figure 48**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 4**



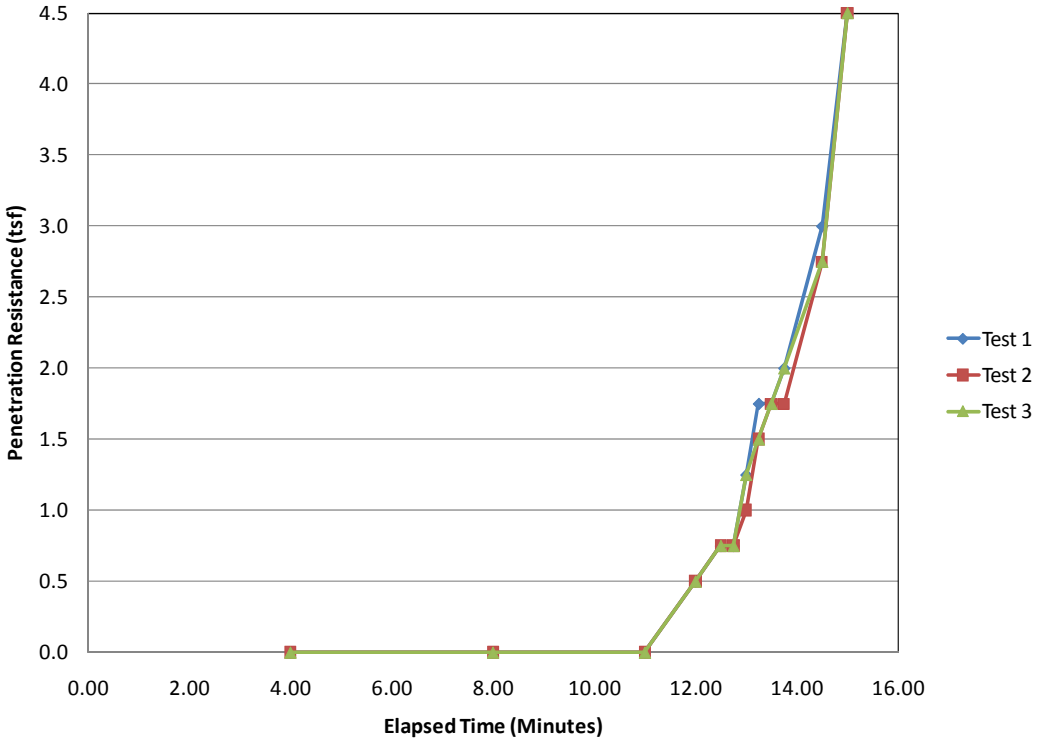
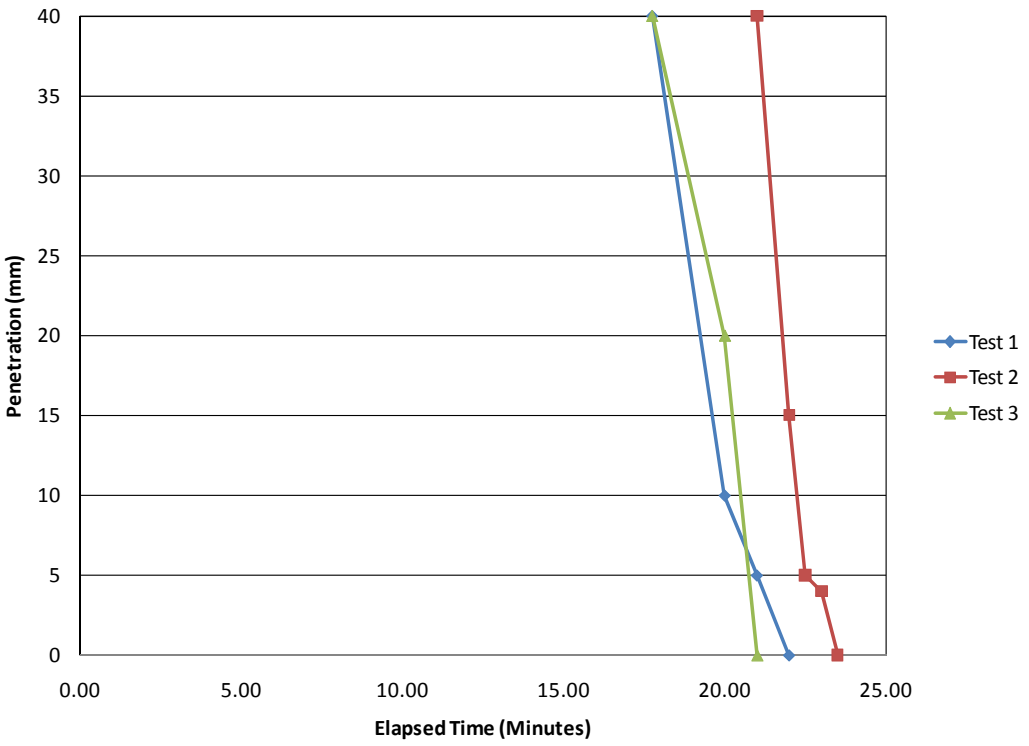




**Figure 49**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 5**



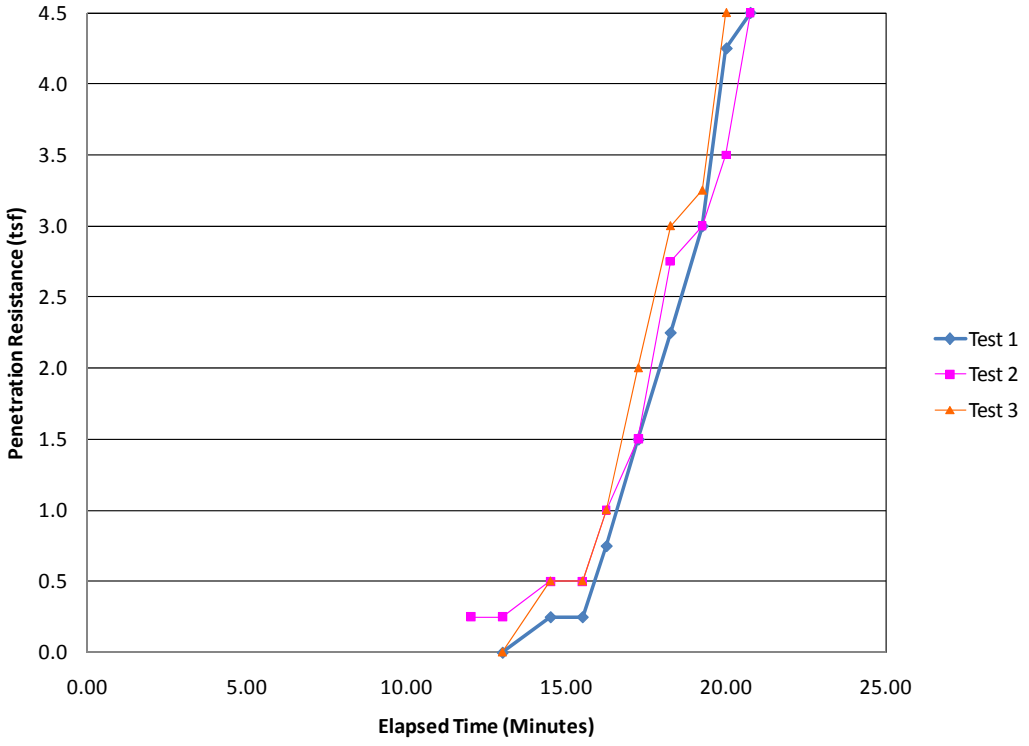




**Figure 50**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 6**

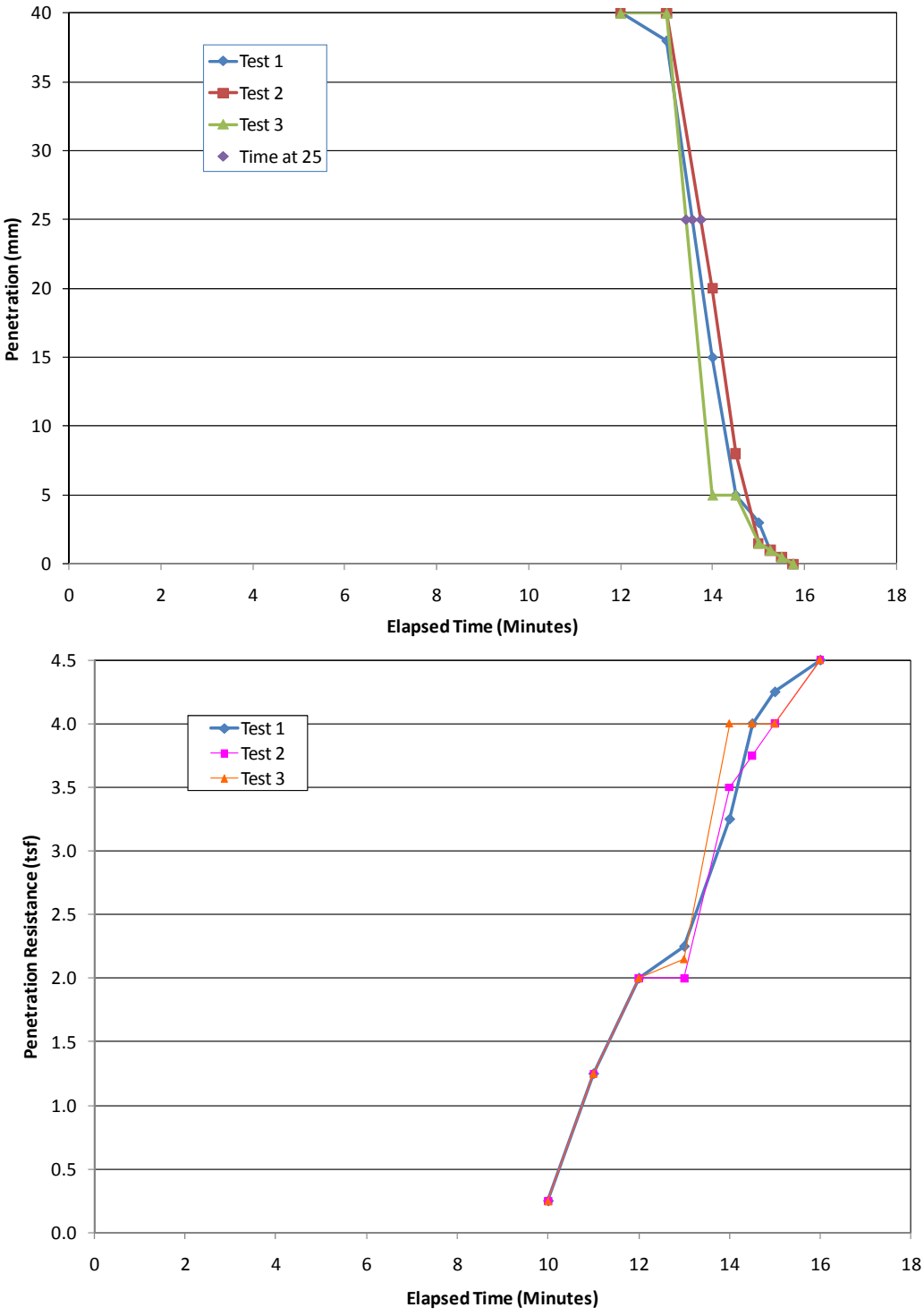



**Figure 51**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 7**

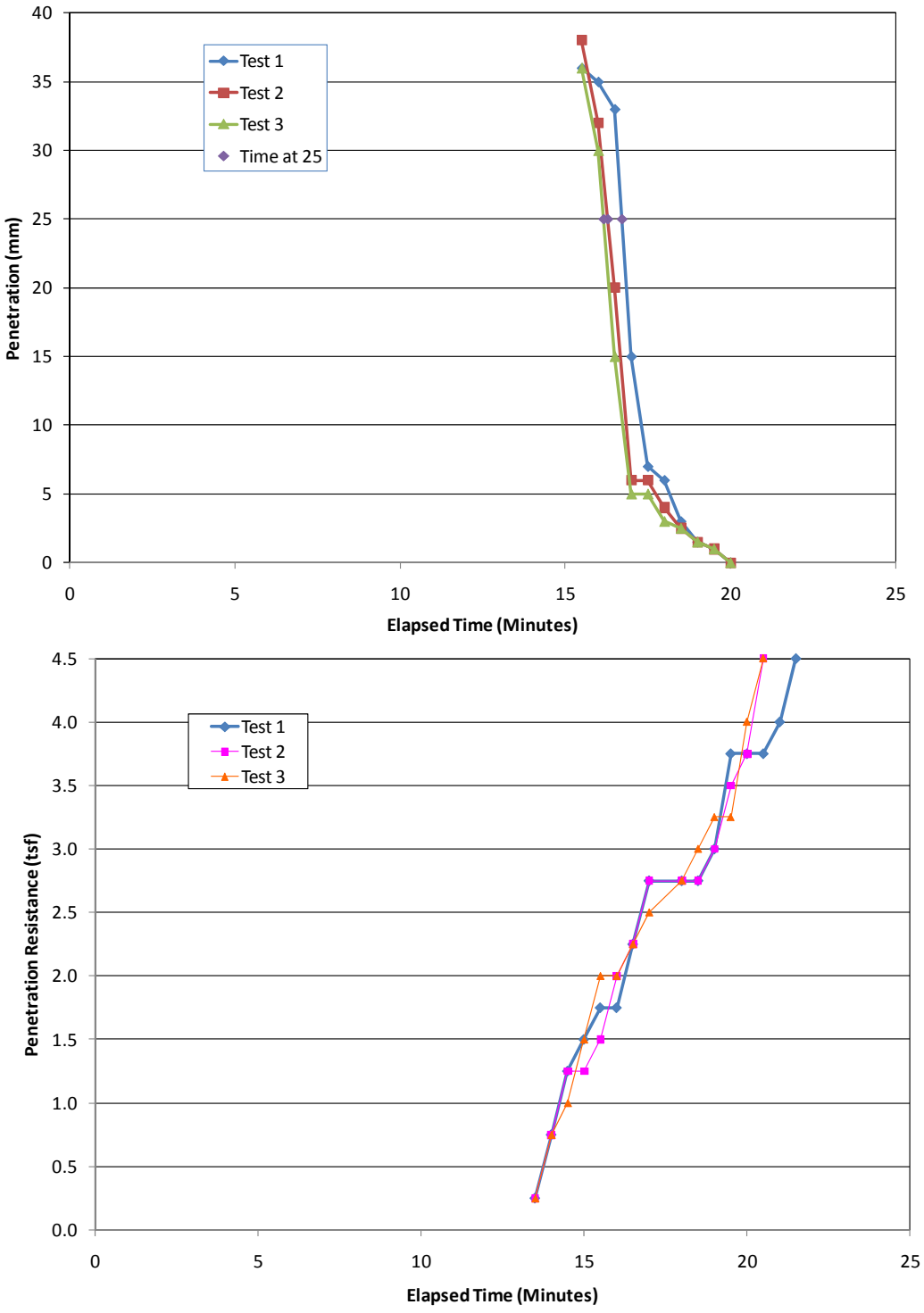



**Figure 52**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 8**

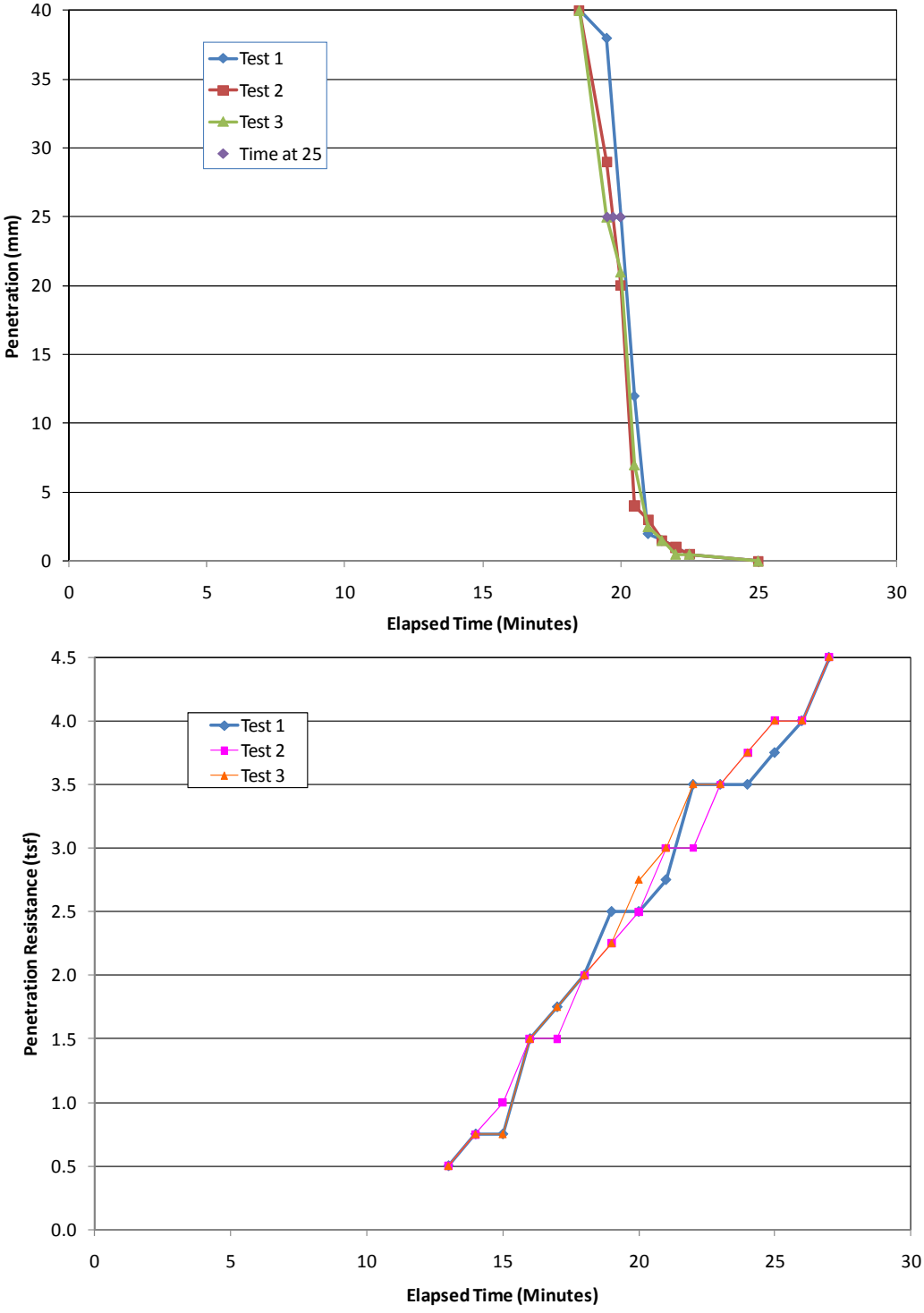



**Figure 53**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 9**

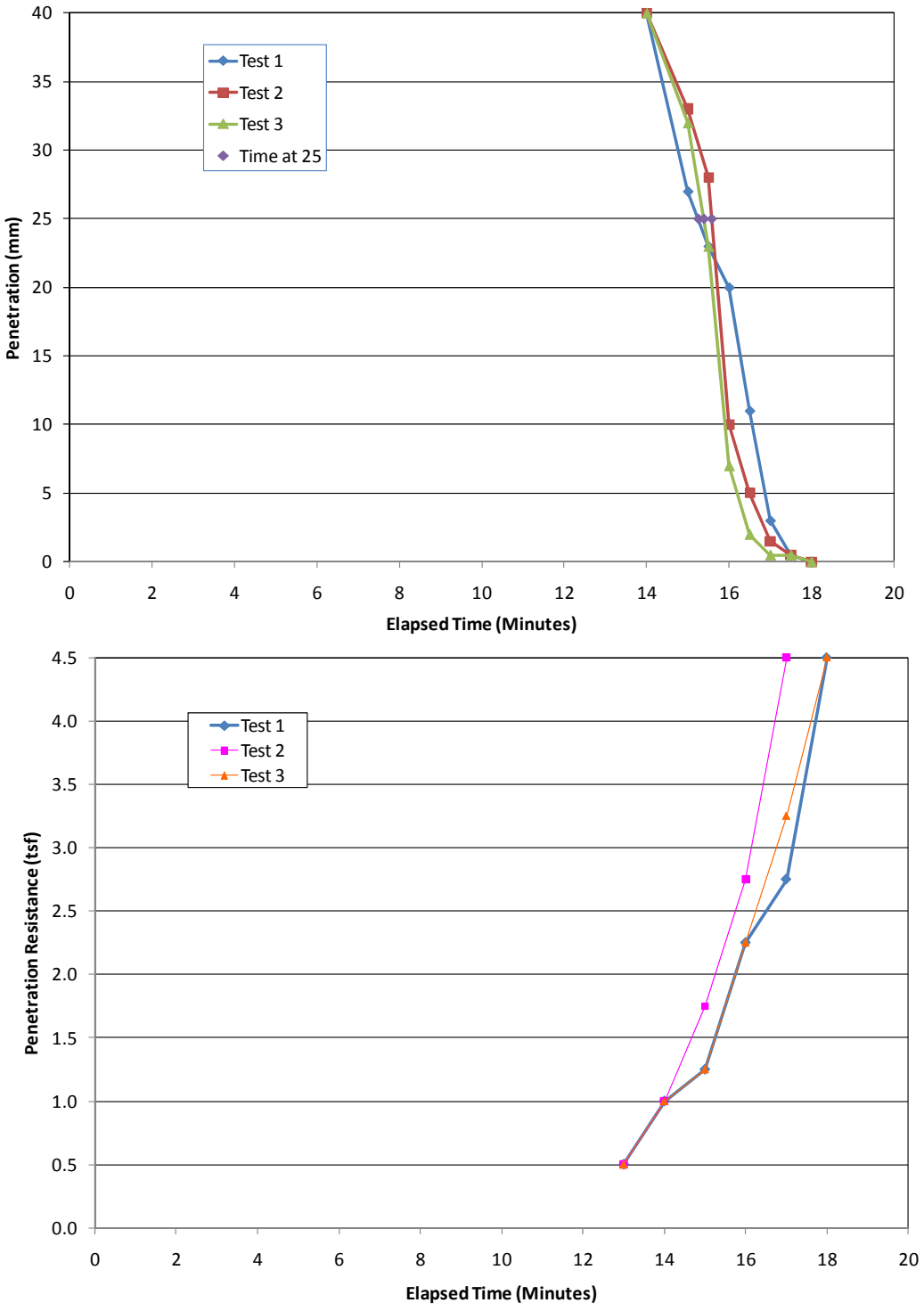
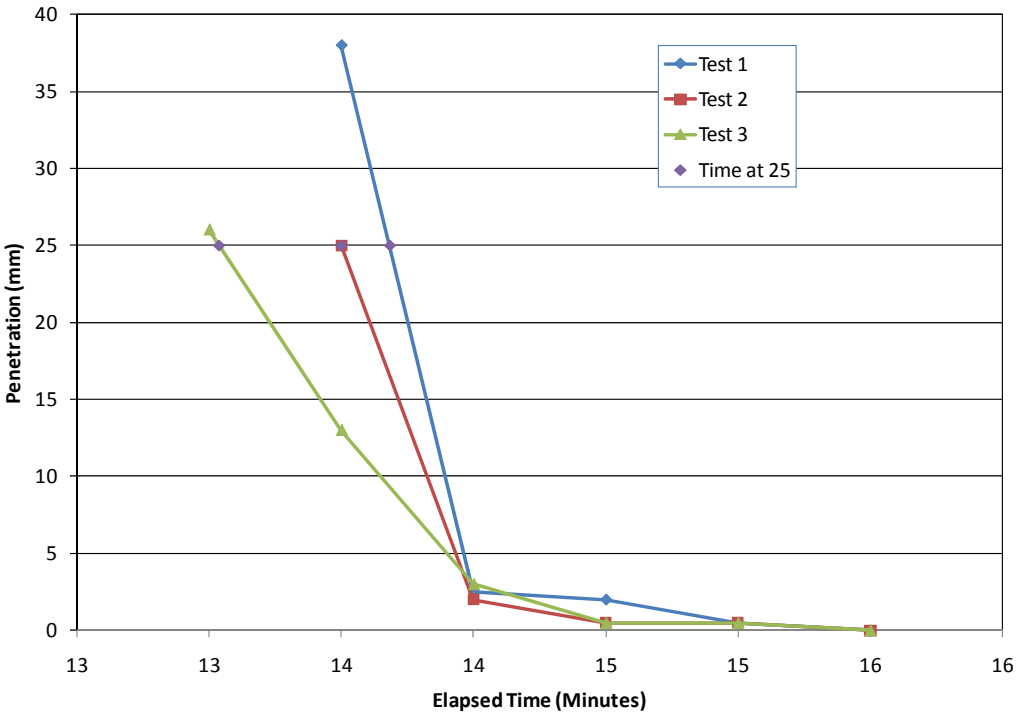



**Figure 54**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 10**



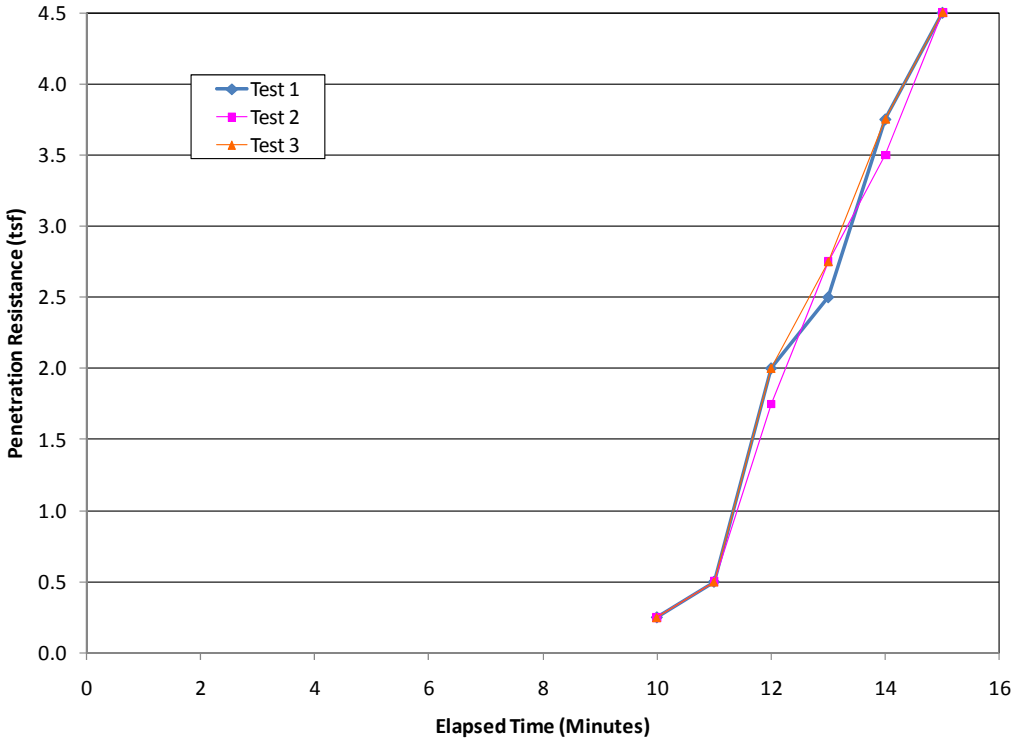




**Figure 55**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 11**

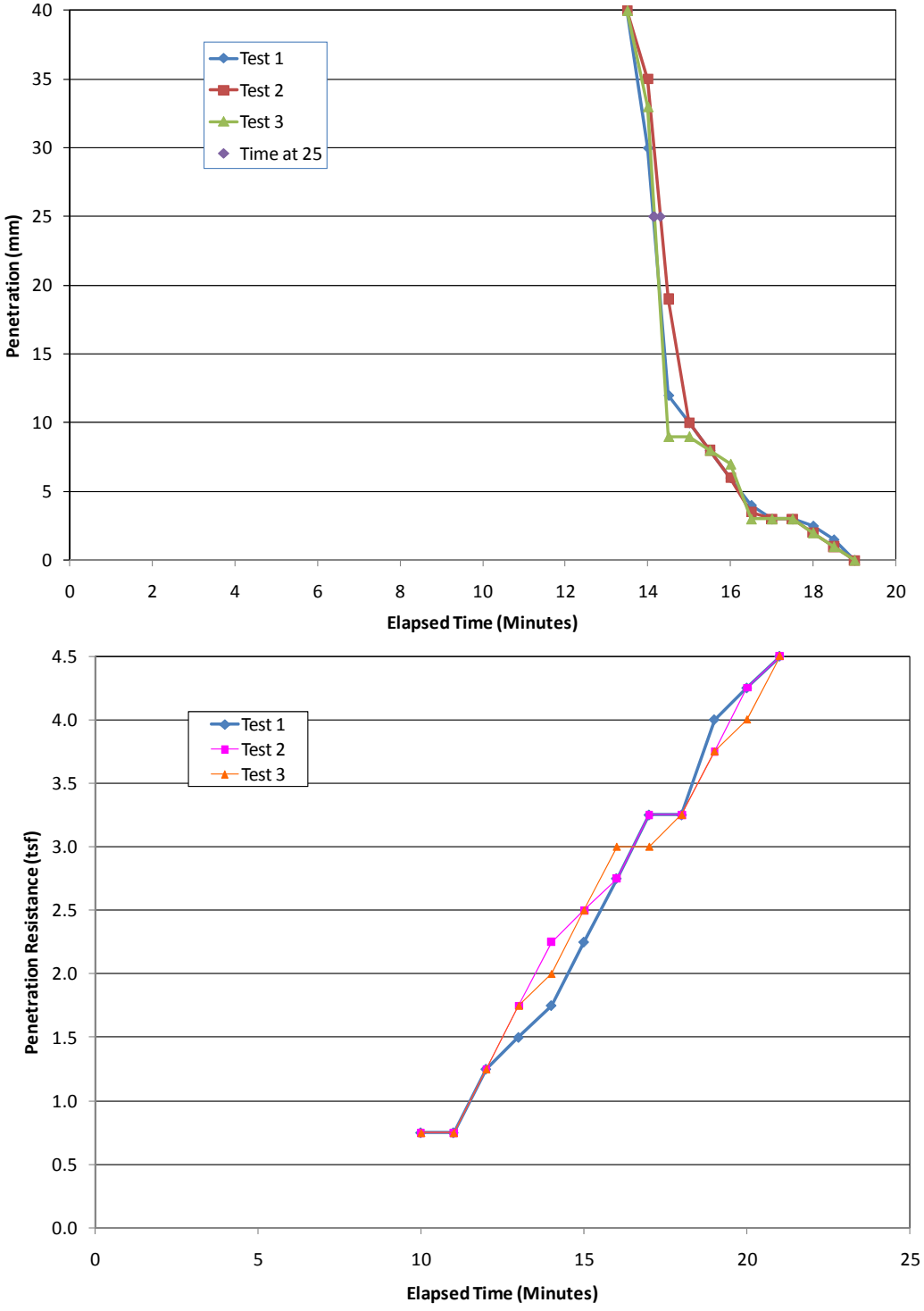
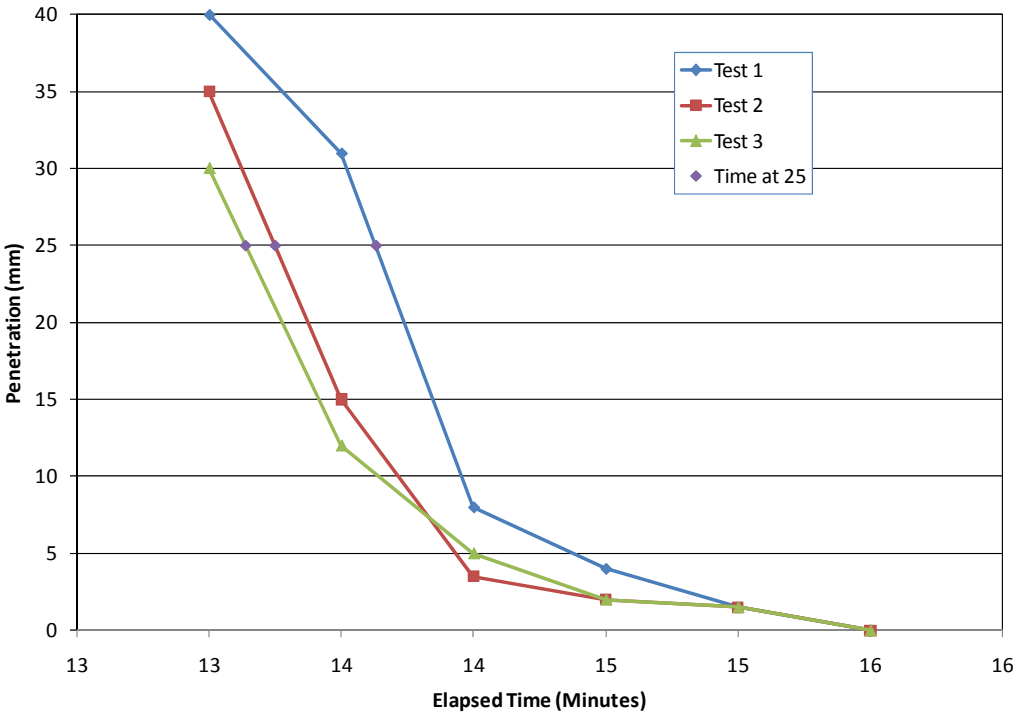



**Figure 56**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 12**



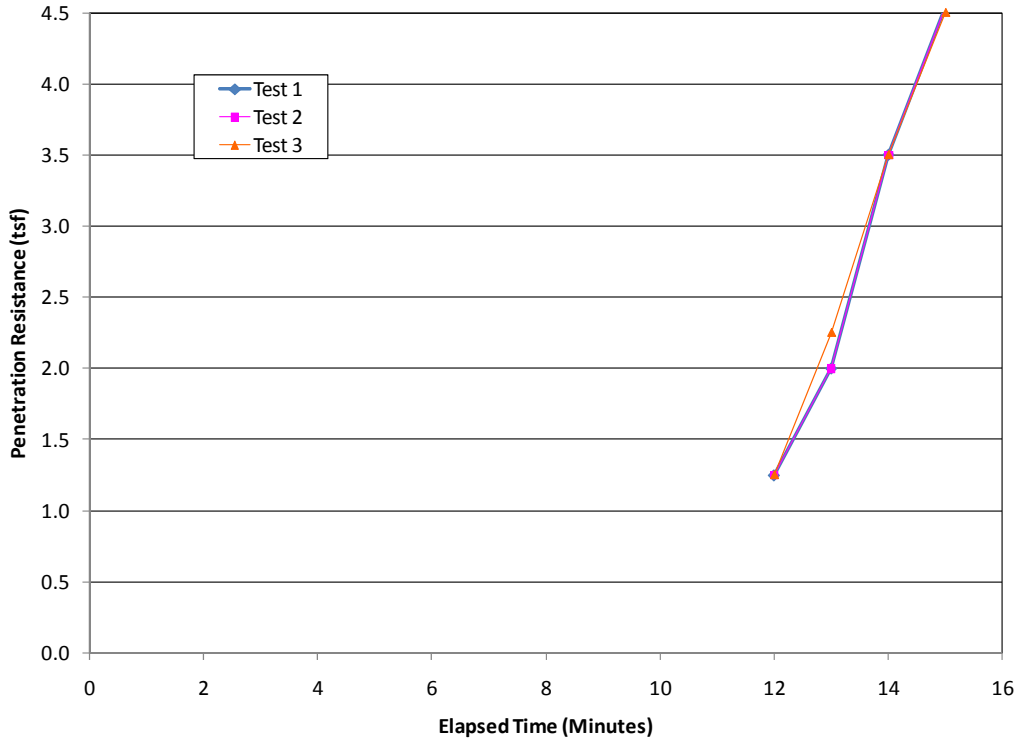




**Figure 57**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 13**

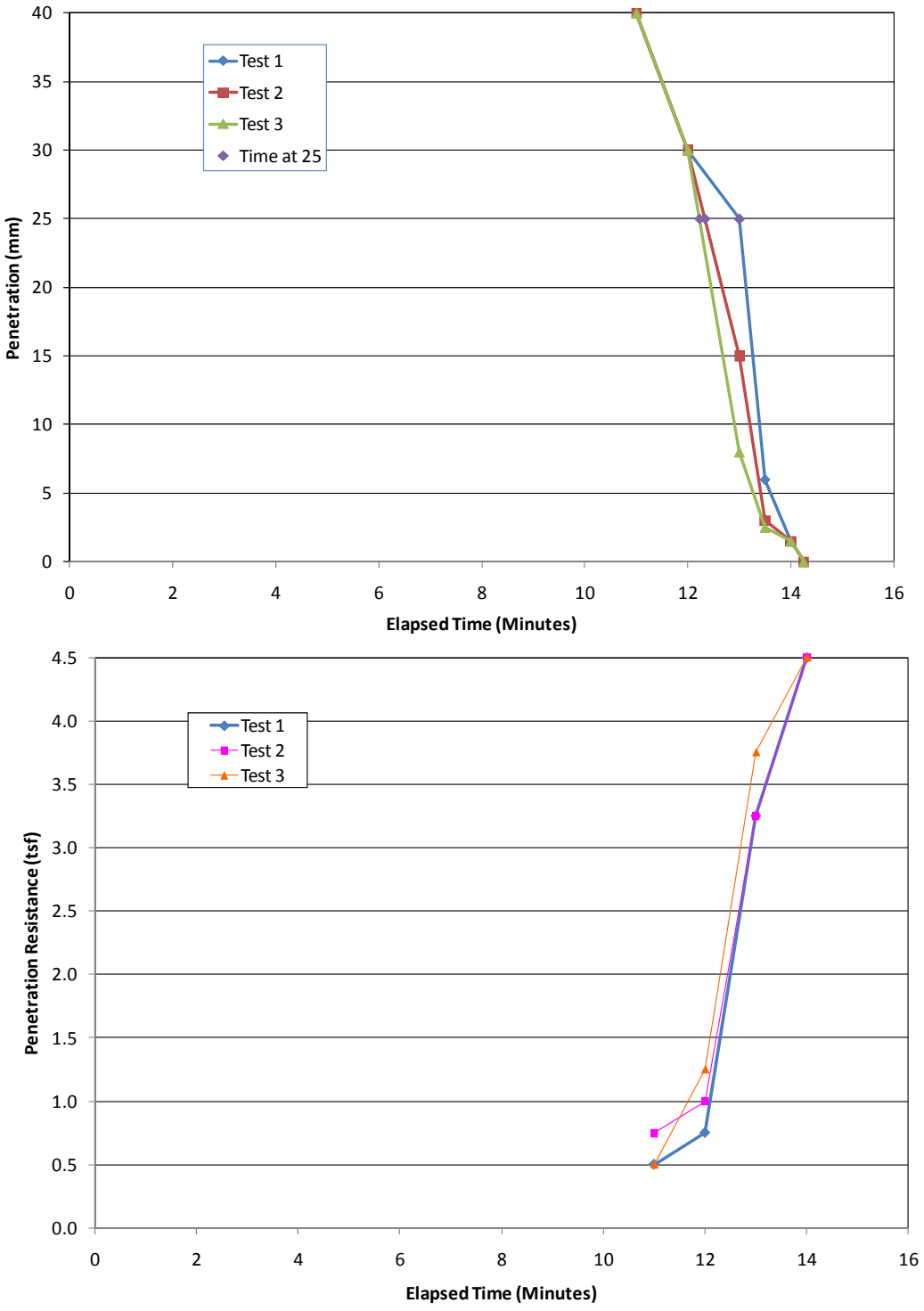
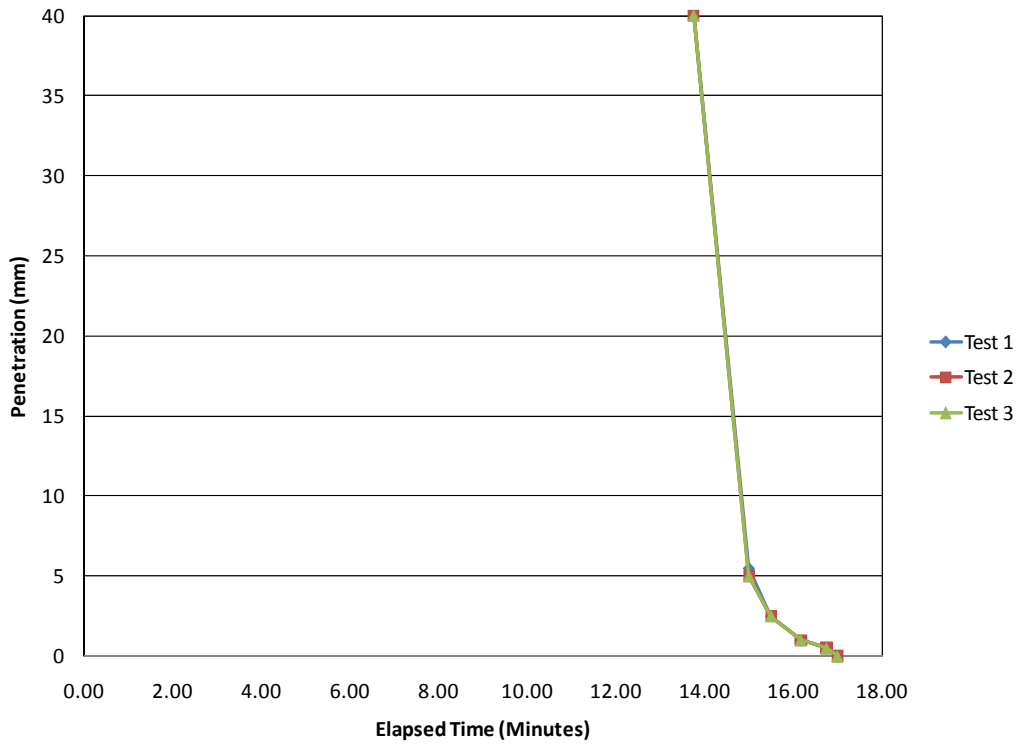



**Figure 58**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 14**



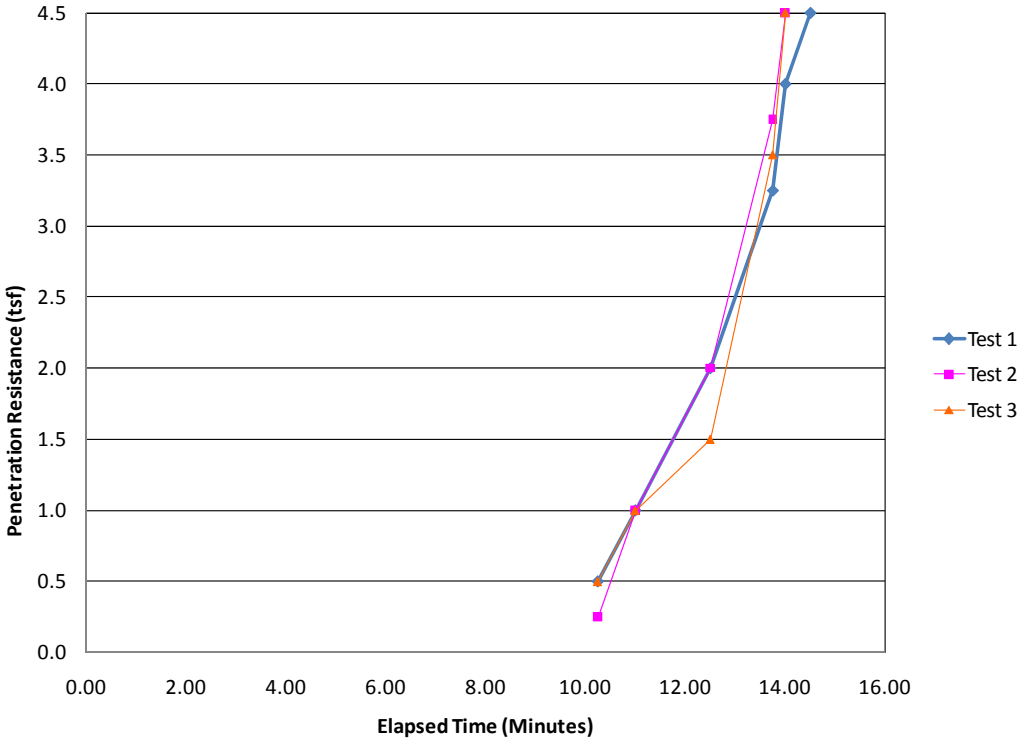




**Figure 59**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 15**

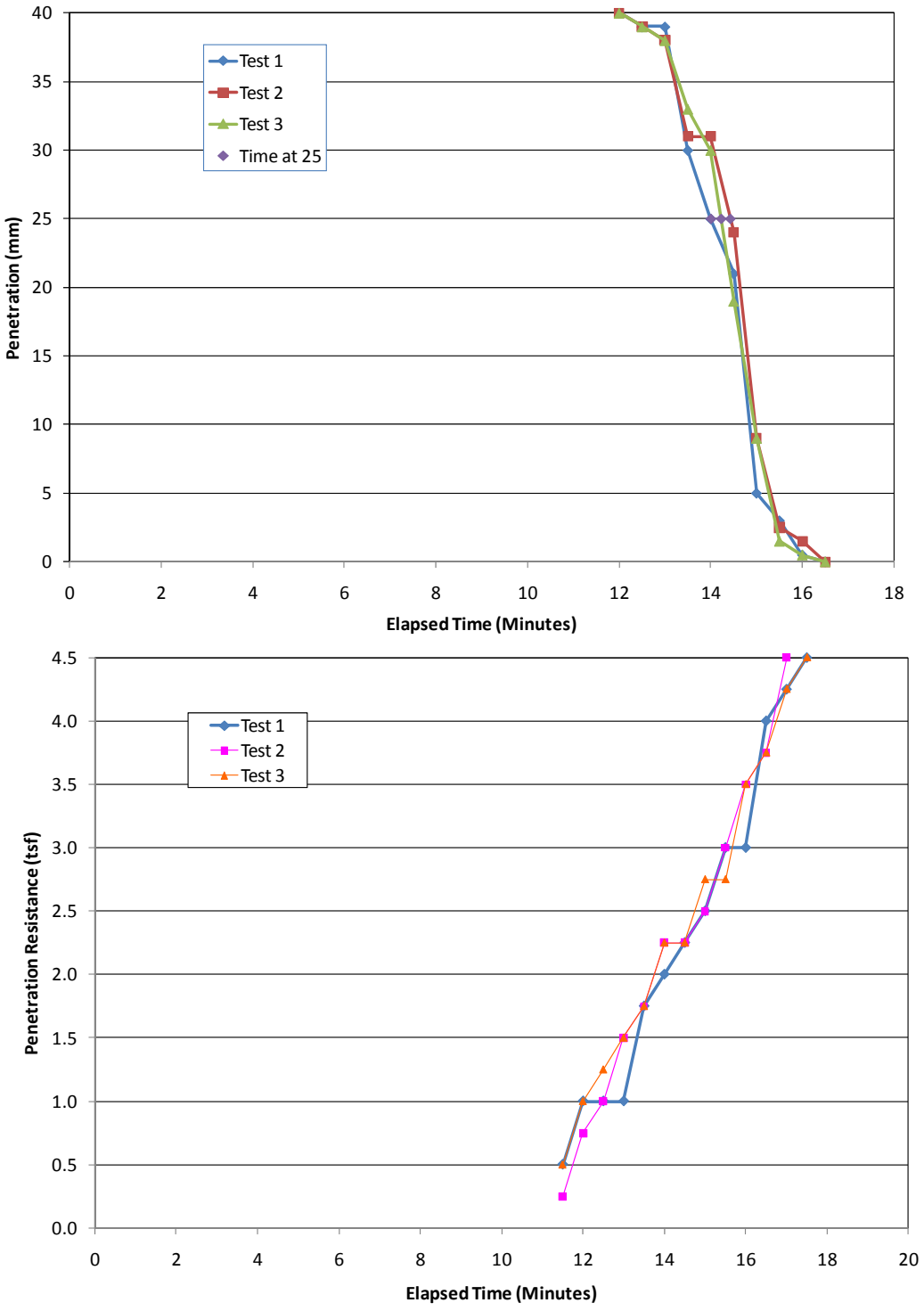



**Figure 60**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 16**

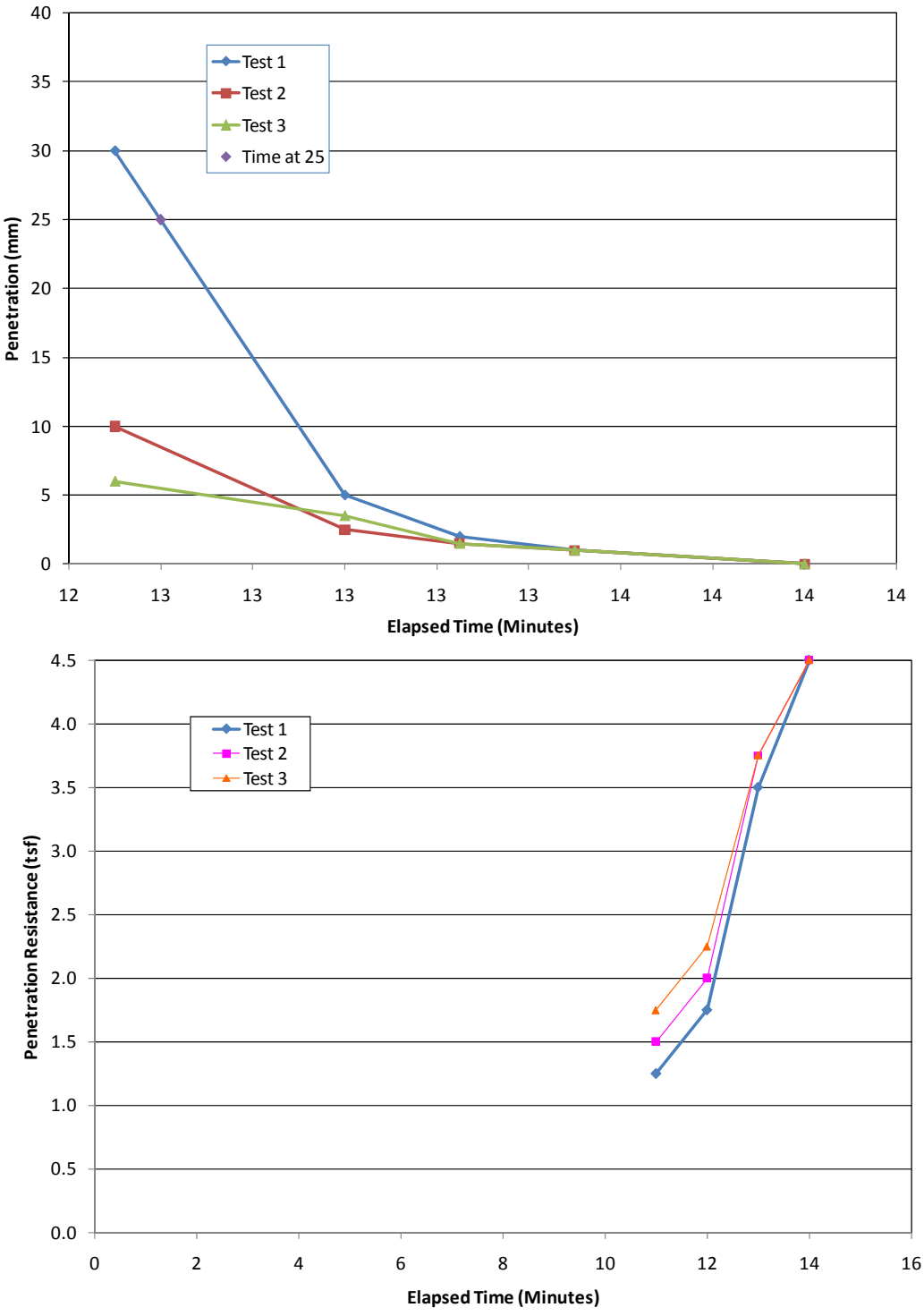
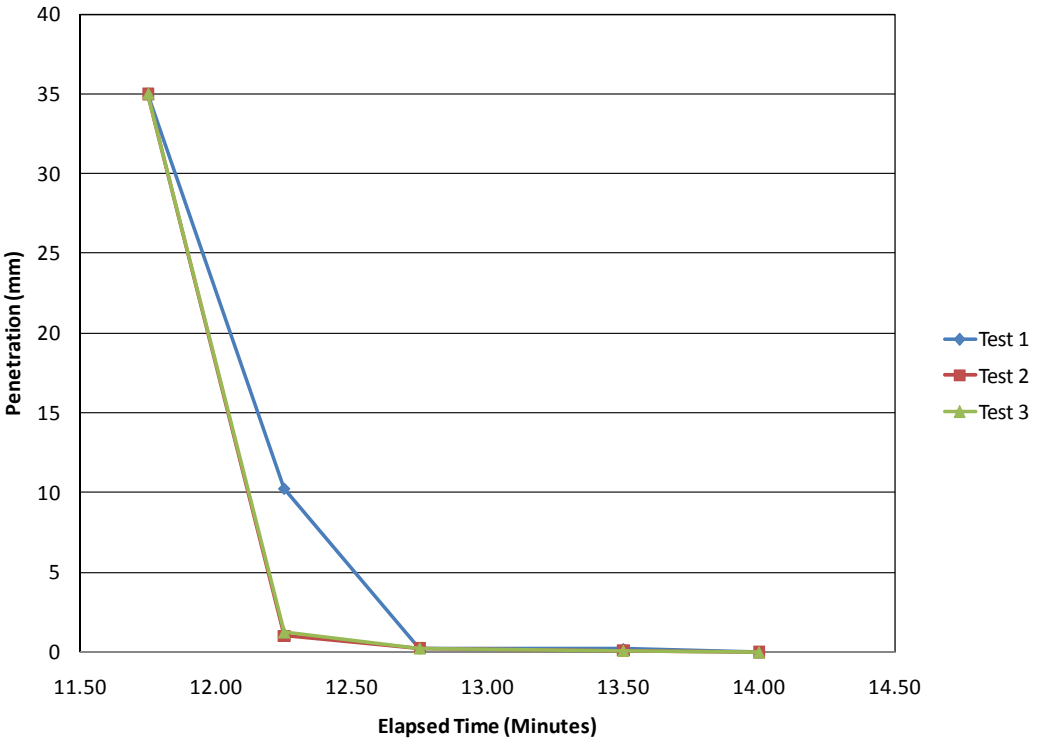



**Figure 61**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 17**



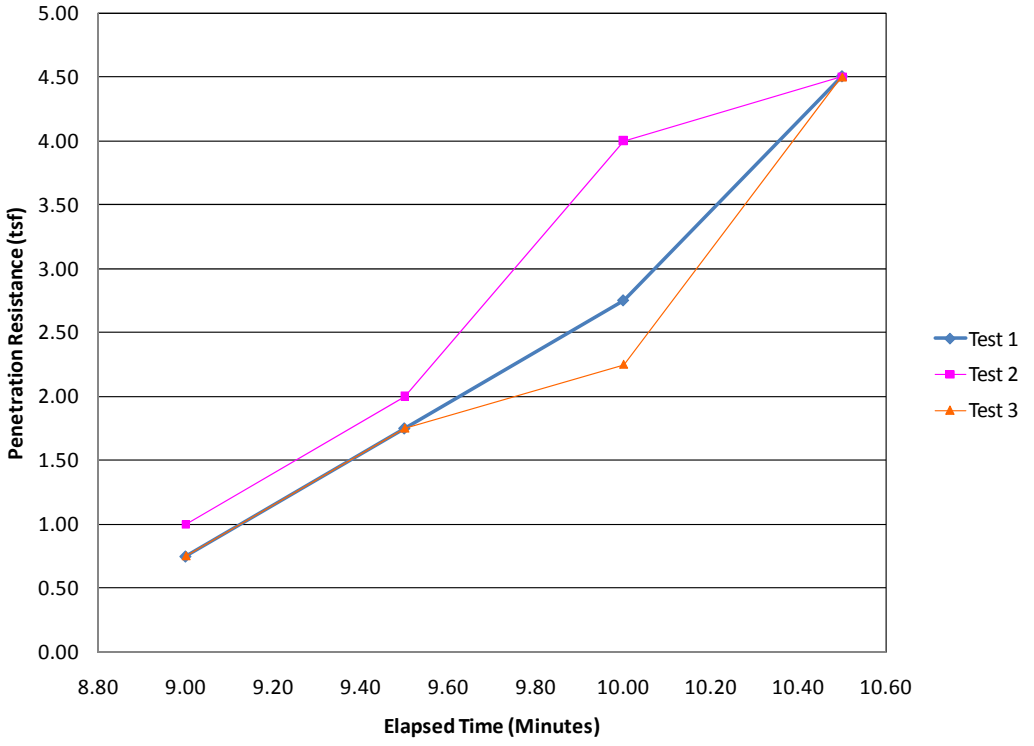
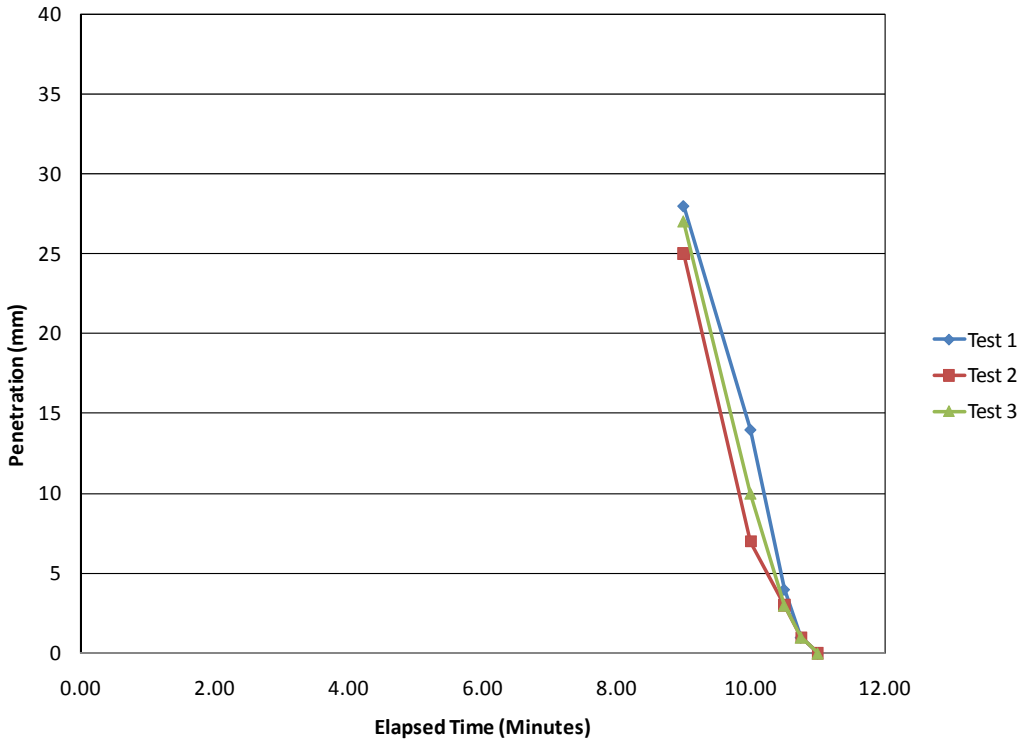




**Figure 62**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 18**



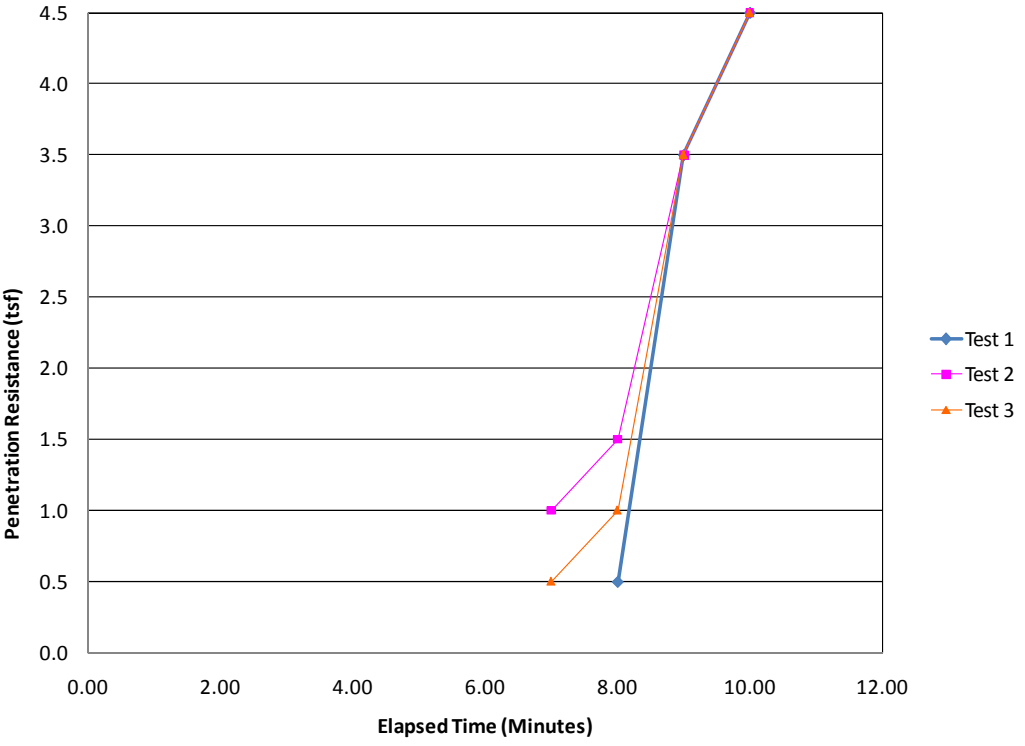
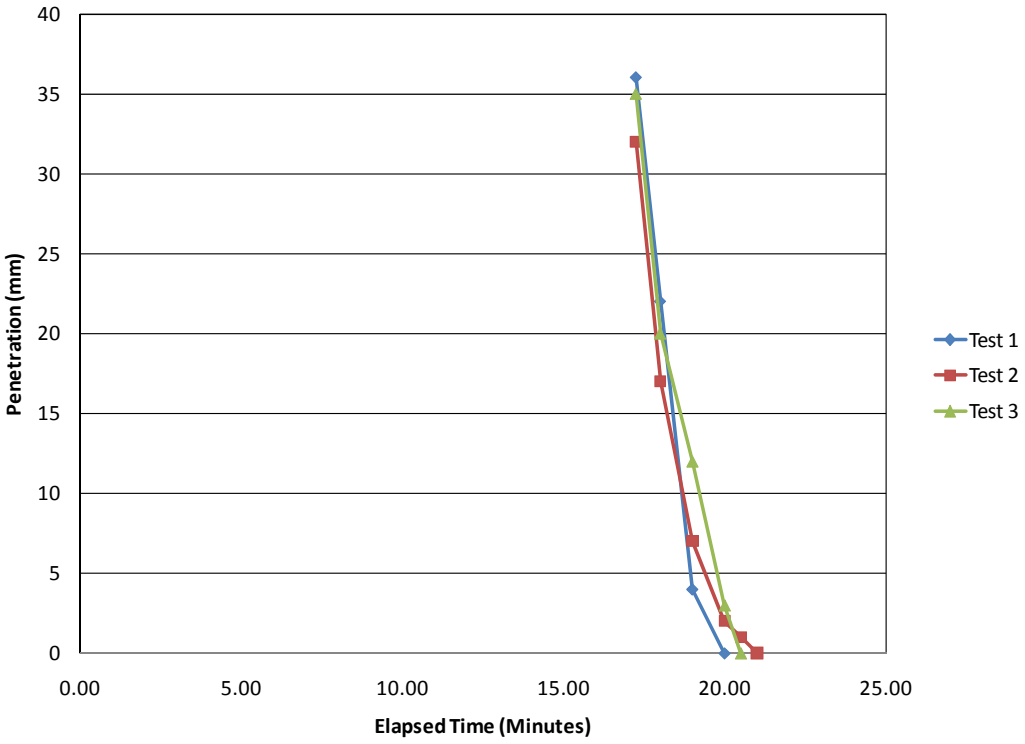




**Figure 63**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 19**



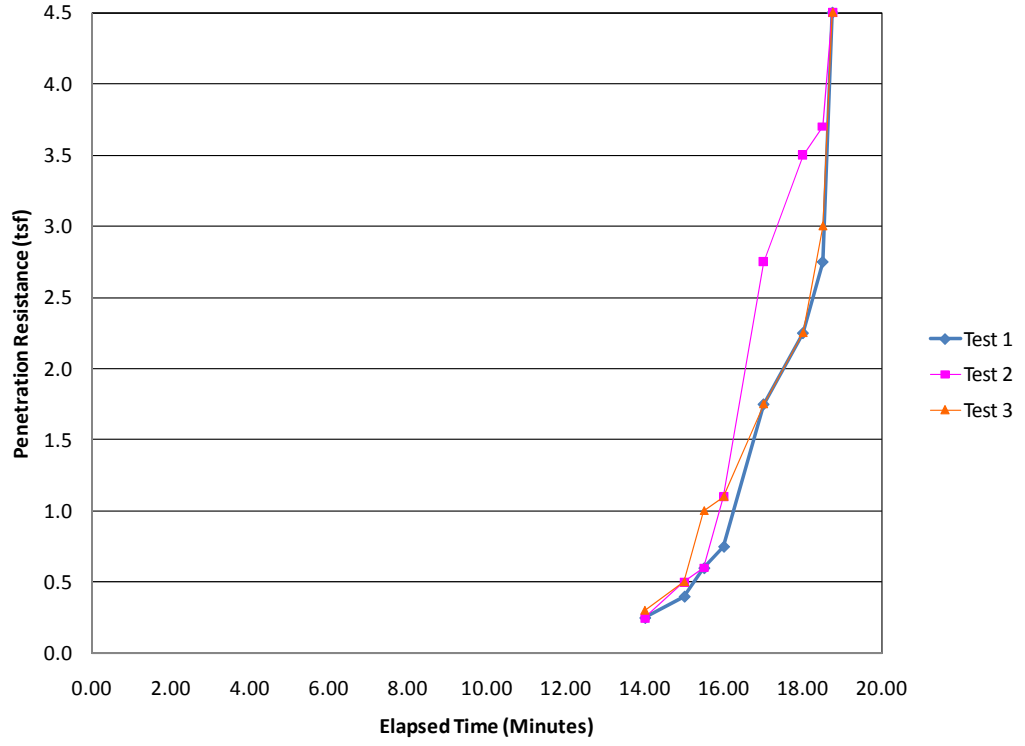
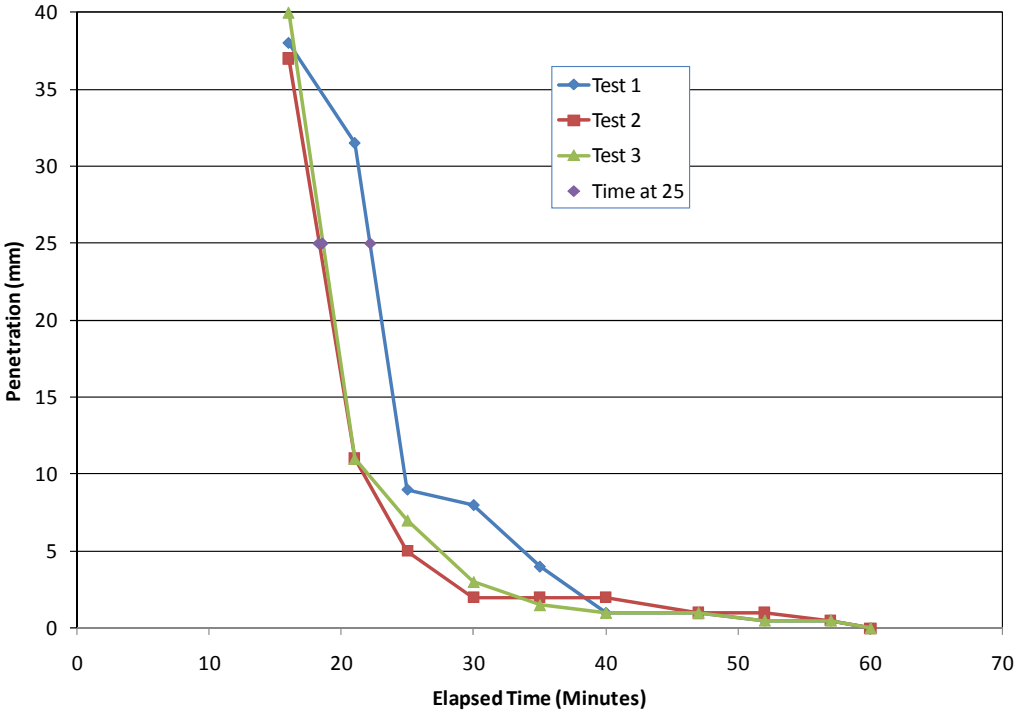




**Figure 64**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 2 Bucket 20**

## Source 3



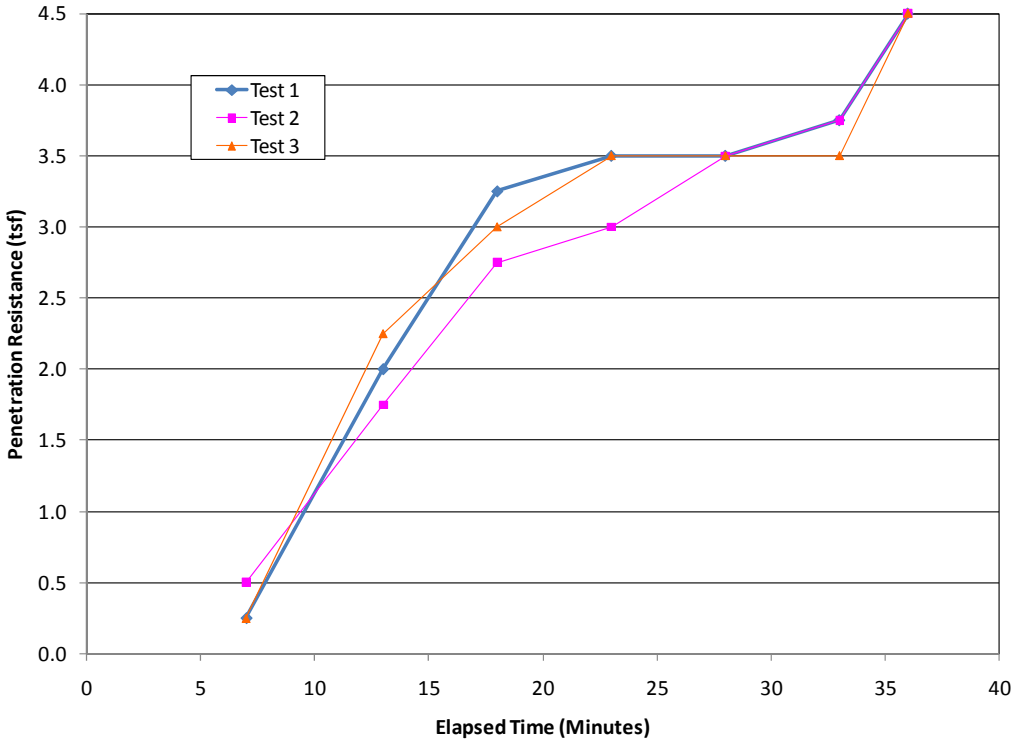




**Figure 65**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 1**

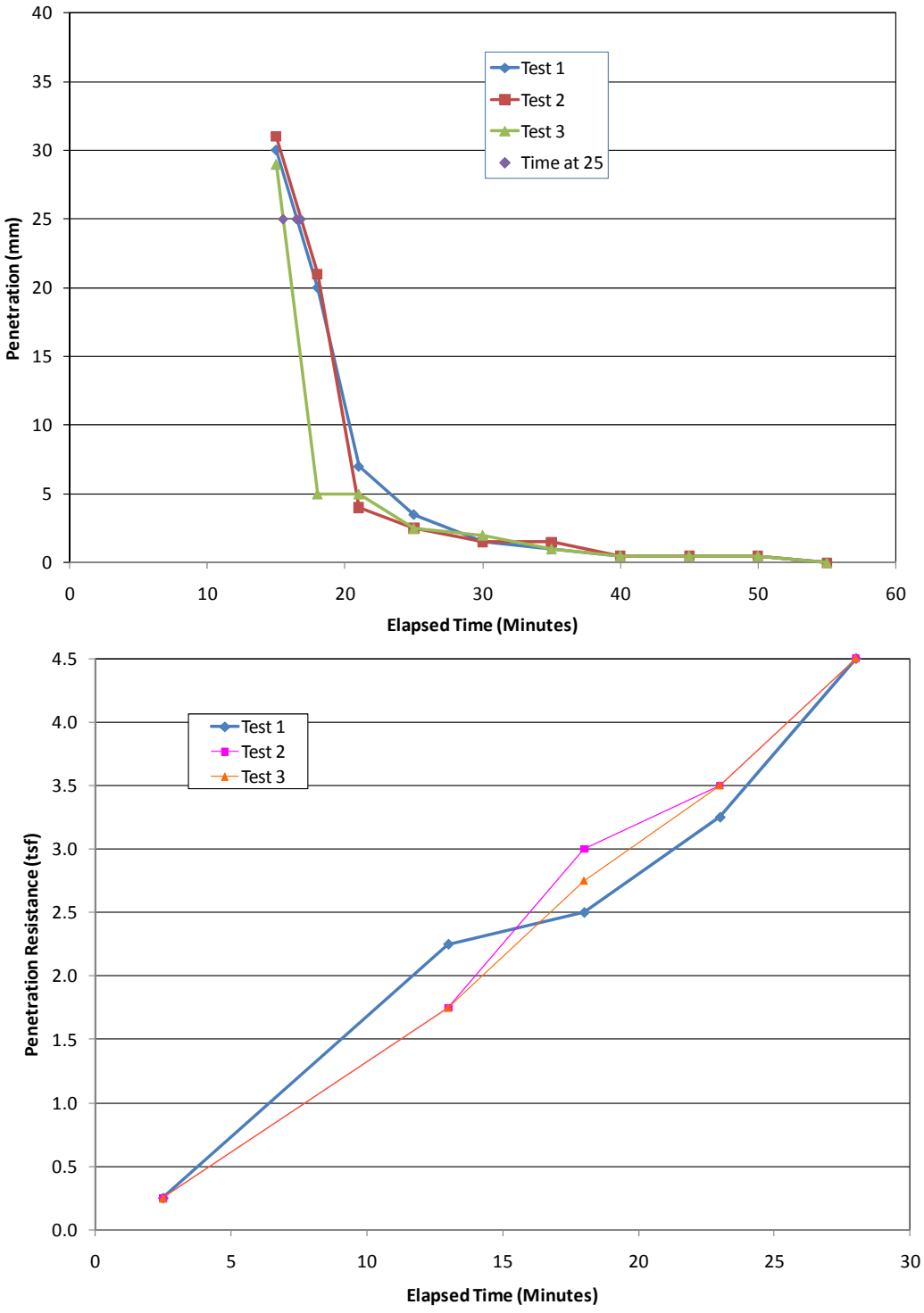
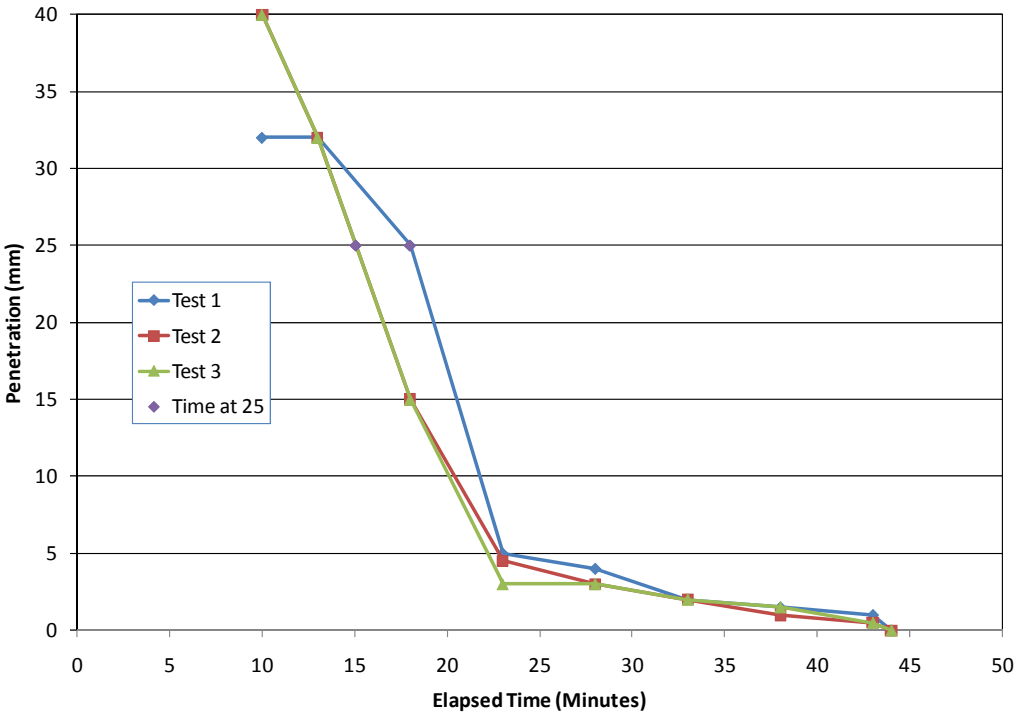



**Figure 66**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 2**



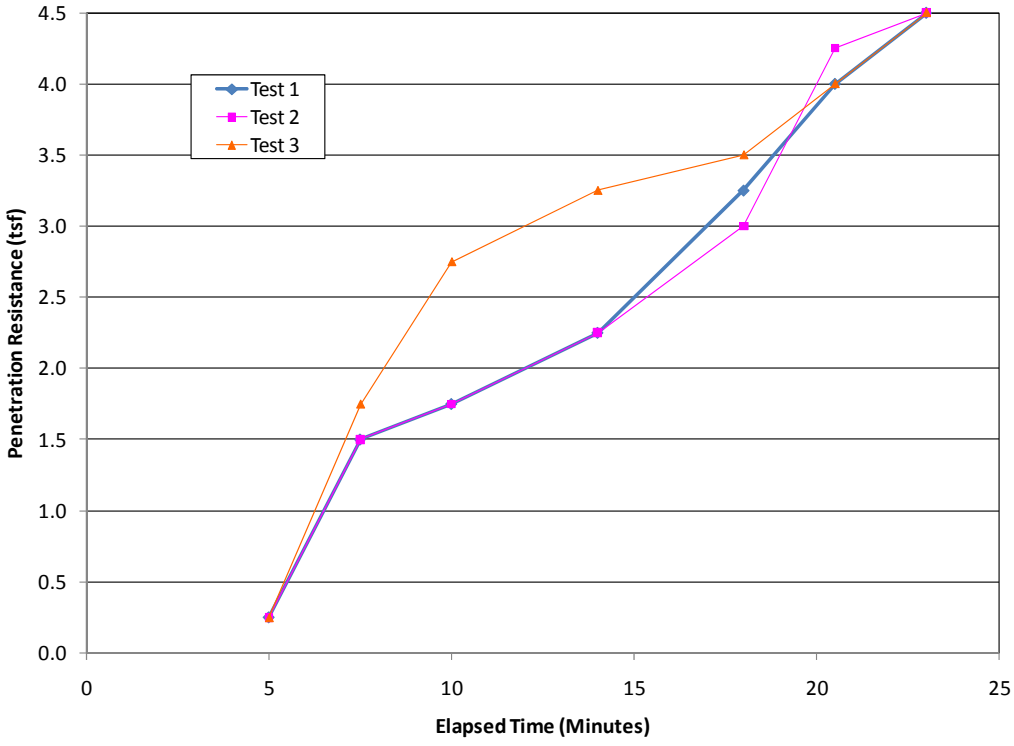




**Figure 67**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 3**

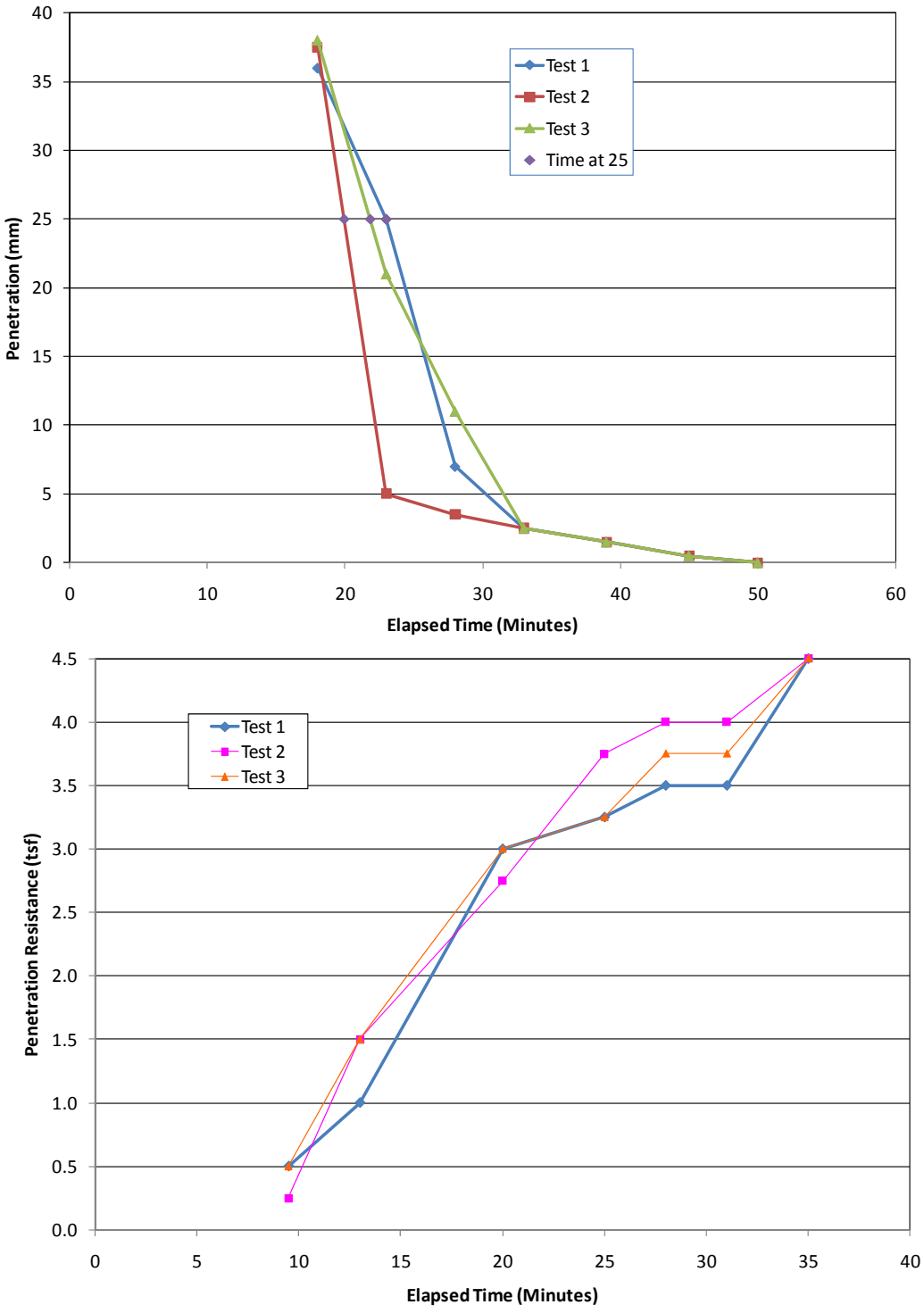
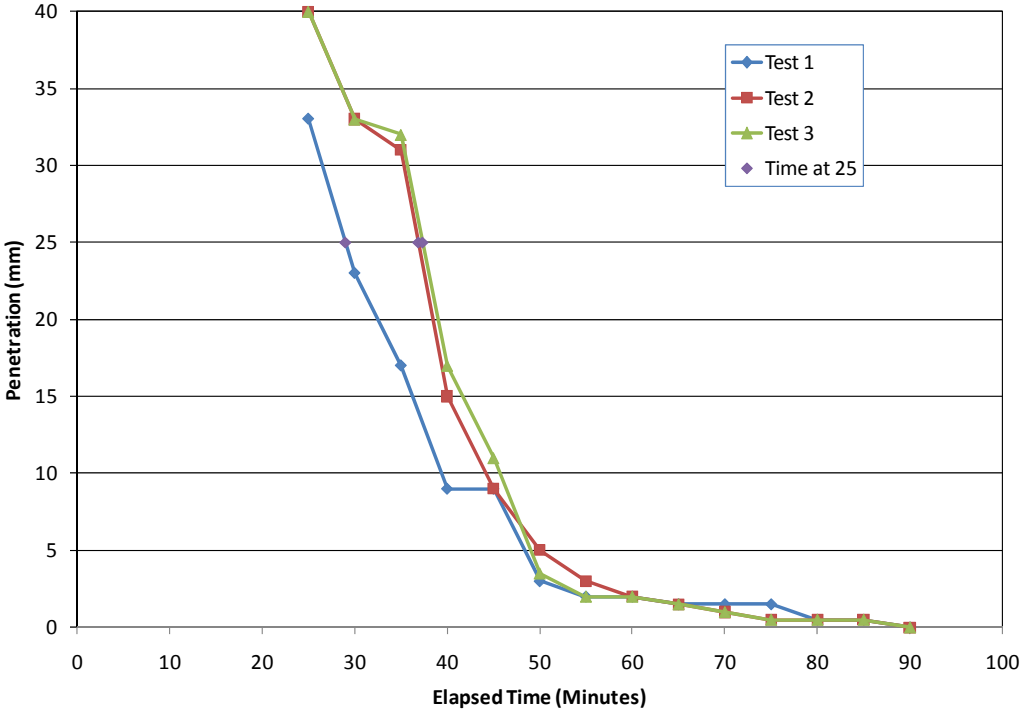



**Figure 68**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 4**



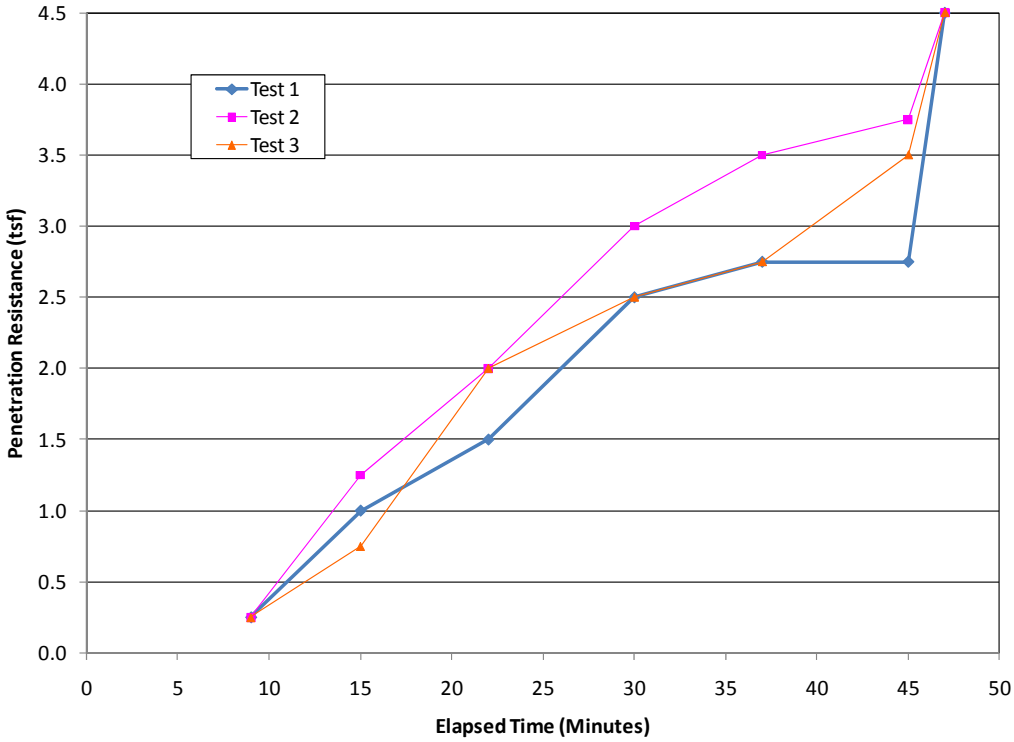




**Figure 69**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 5**

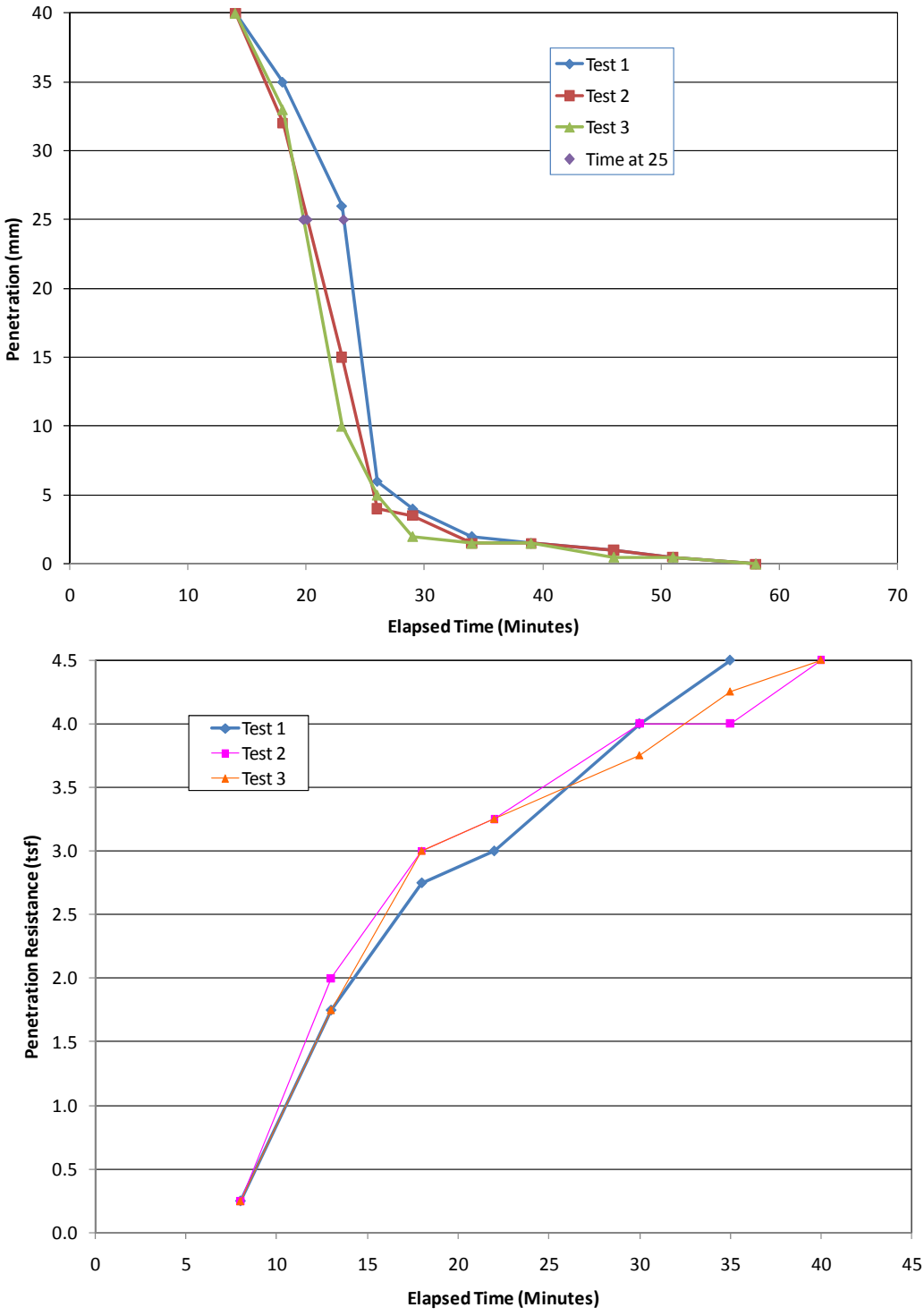



**Figure 70**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 6**

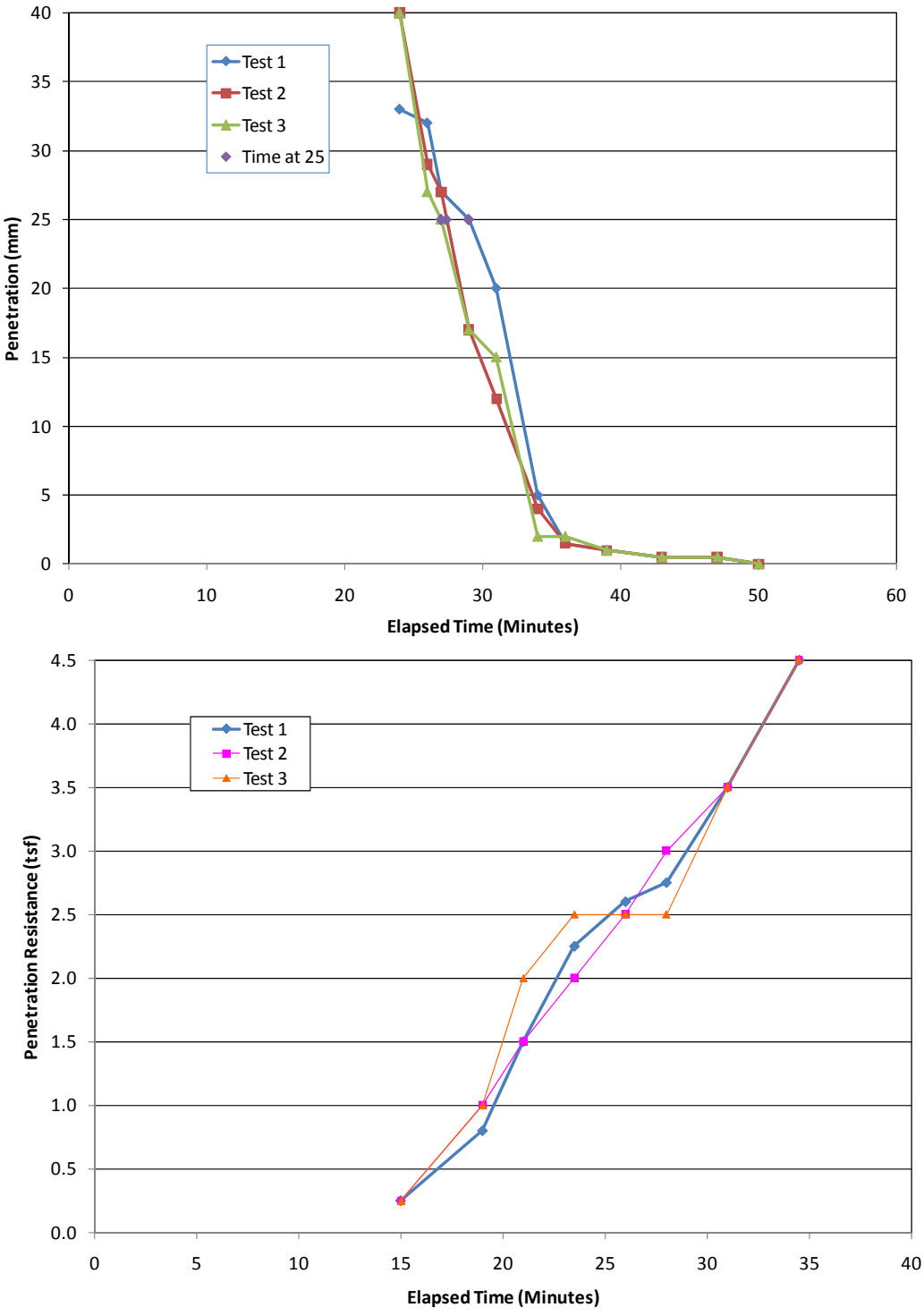



**Figure 71**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 7**

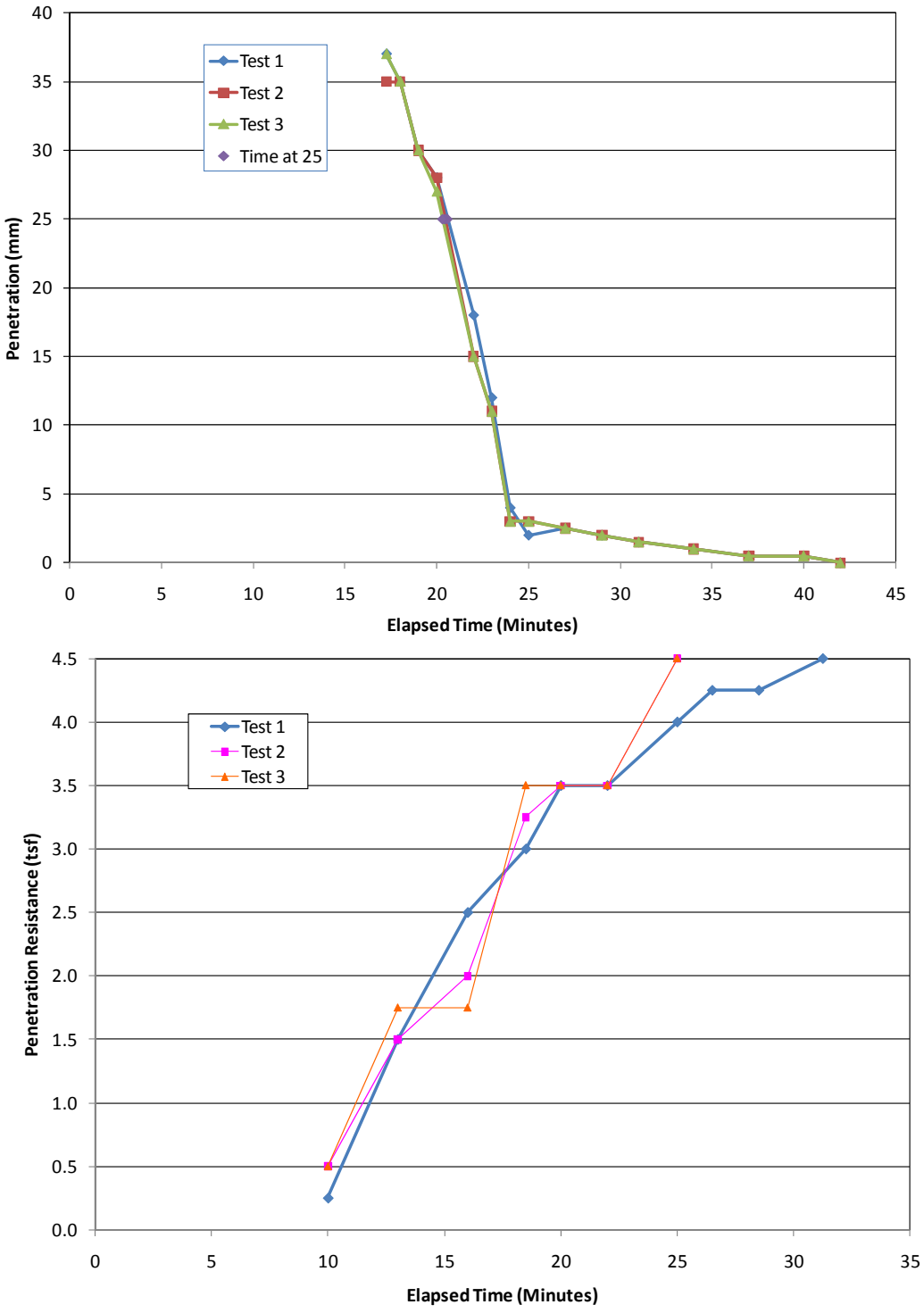



**Figure 72**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 8**

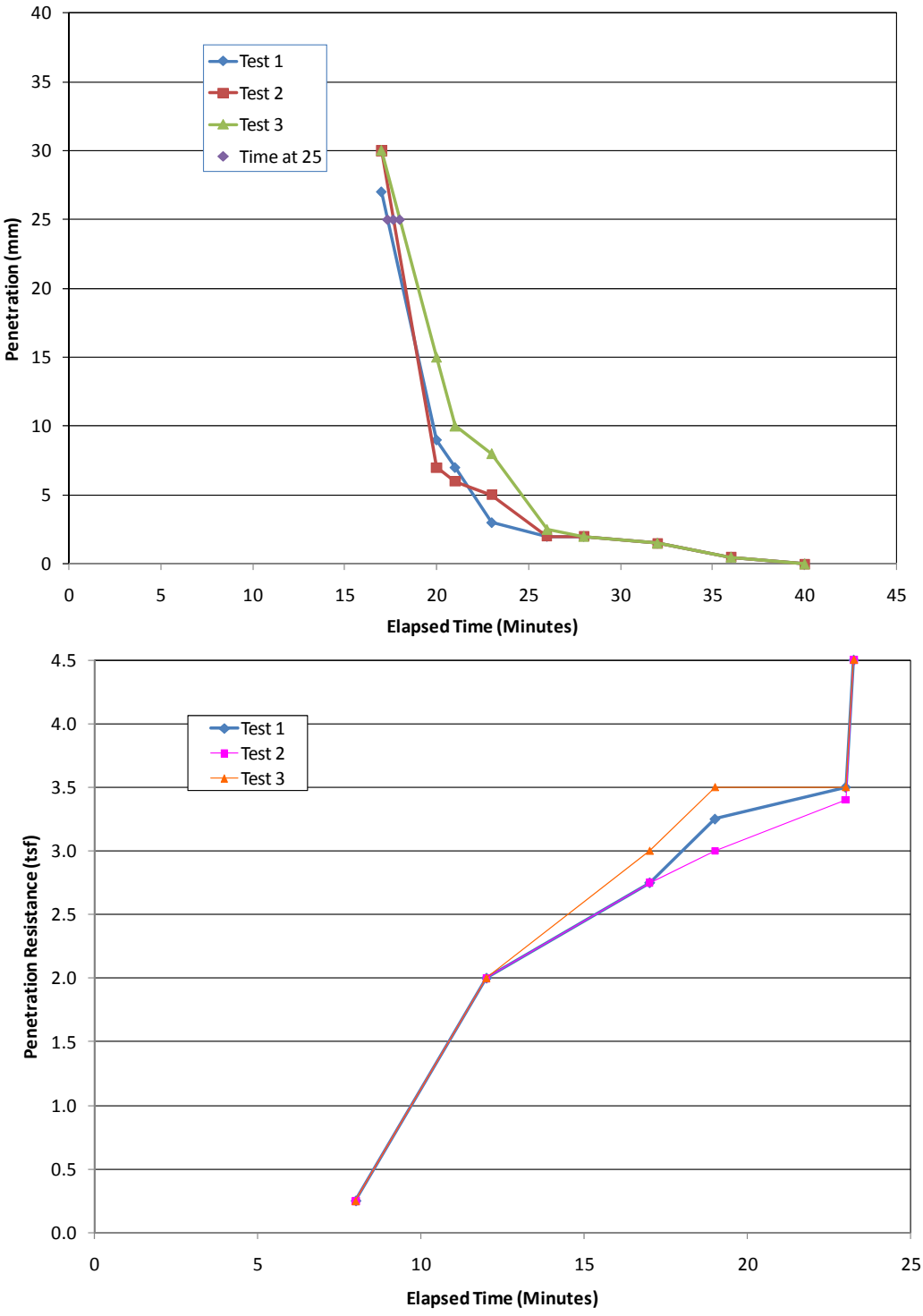



**Figure 73**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 9**

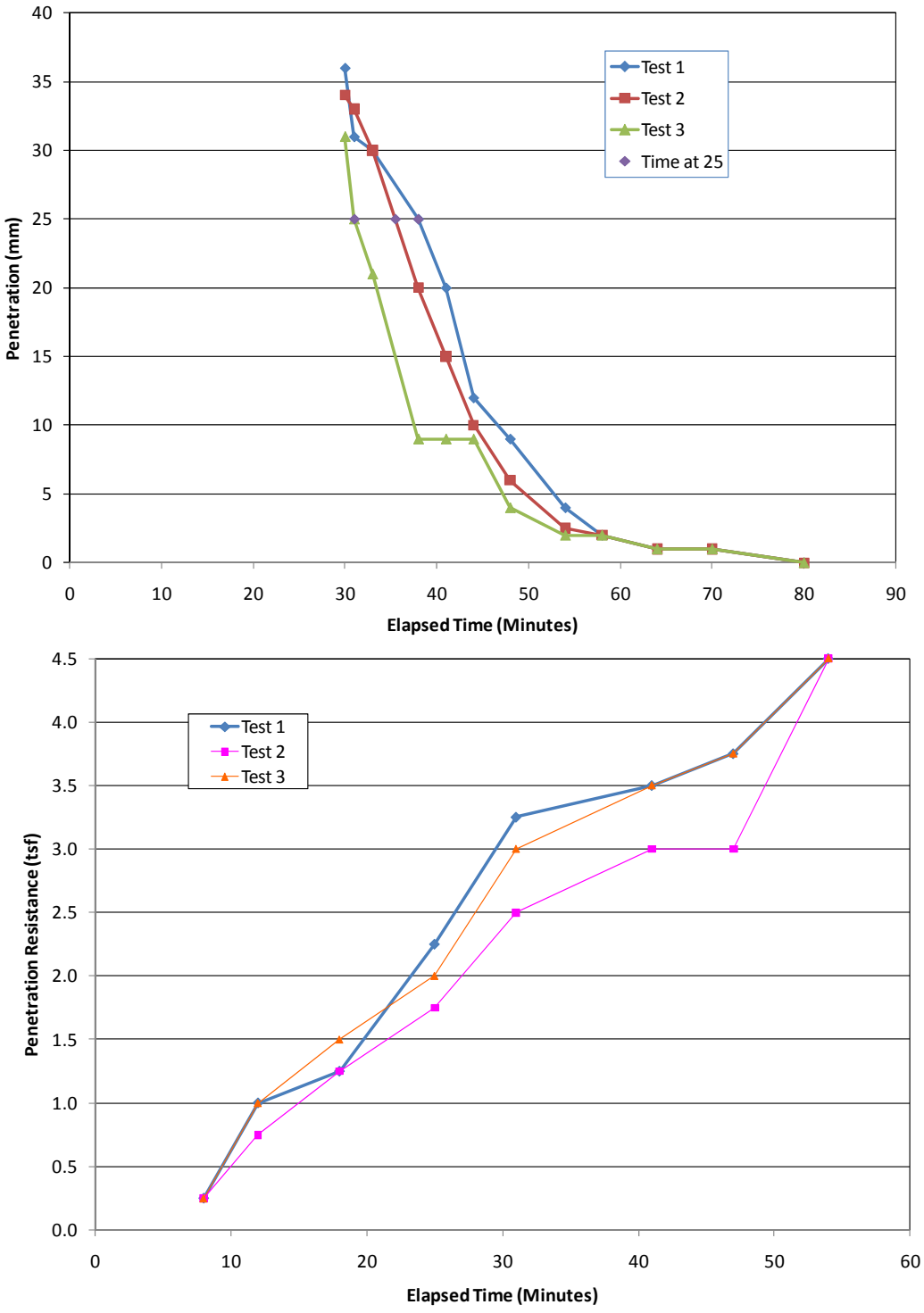
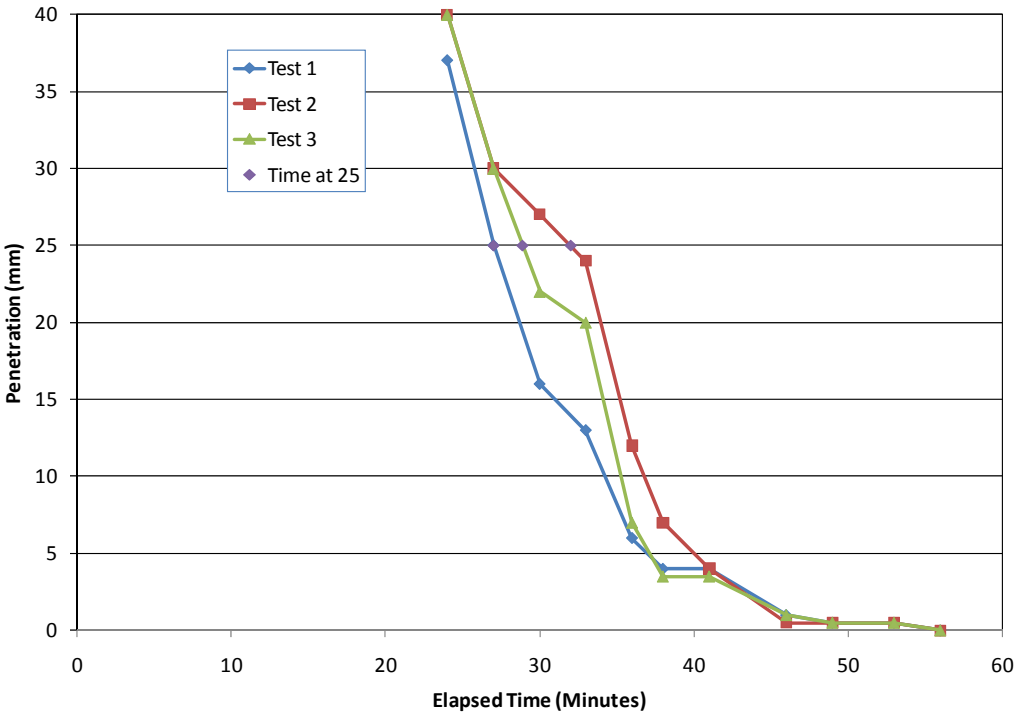



**Figure 74**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 10**



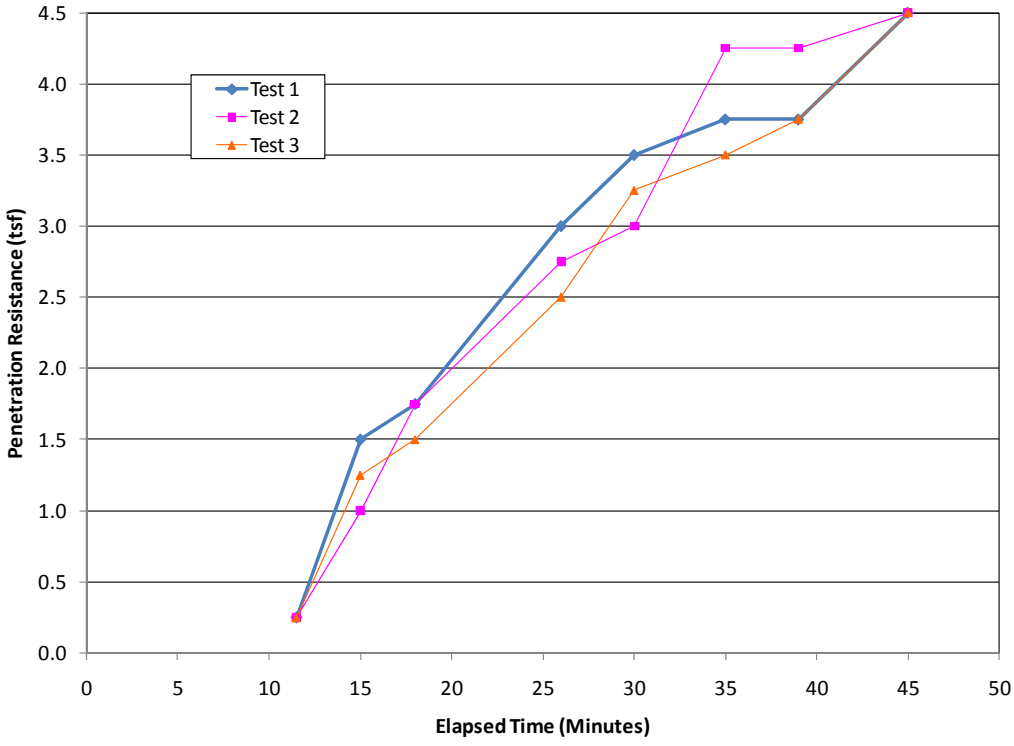




**Figure 75**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 11**

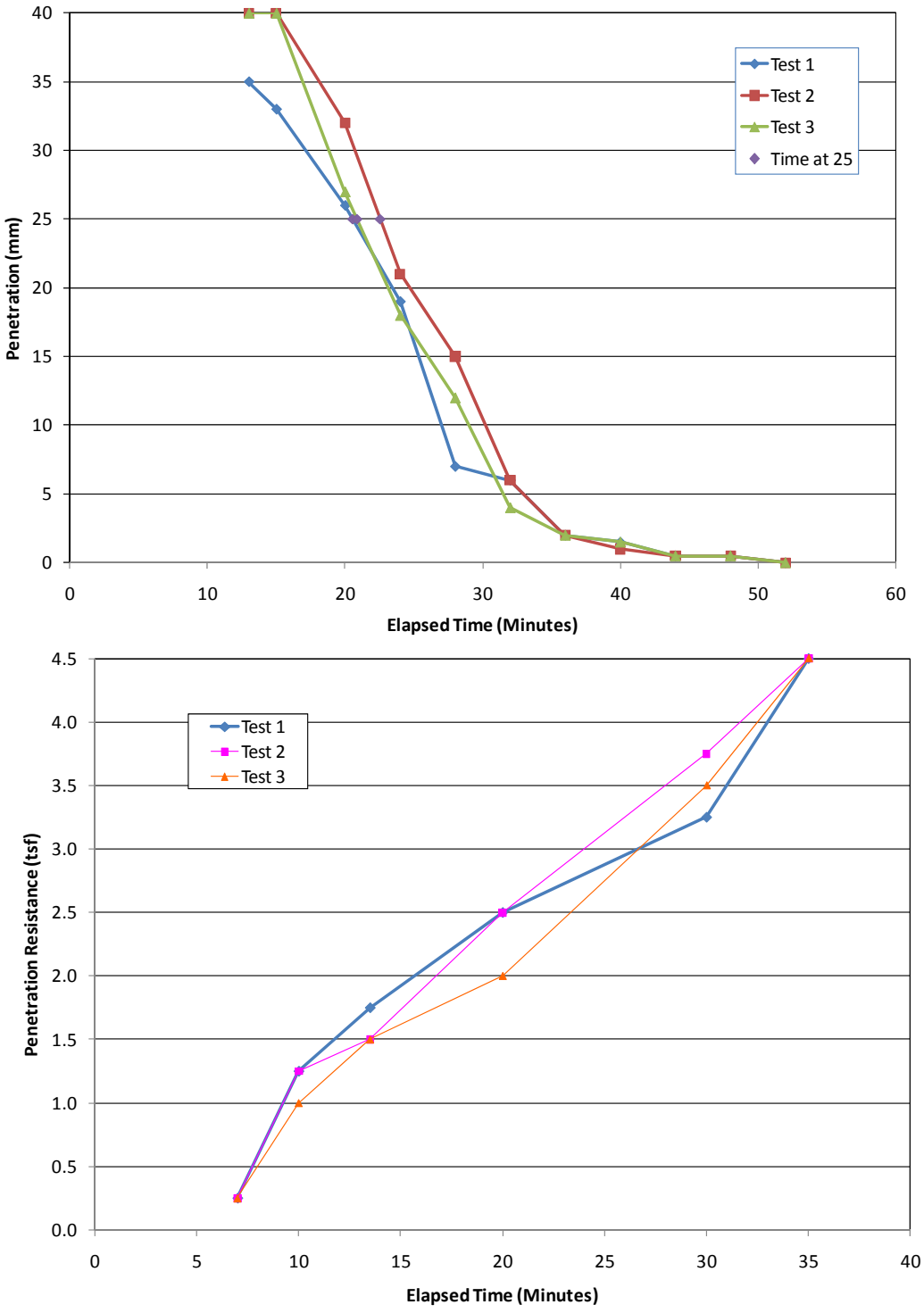
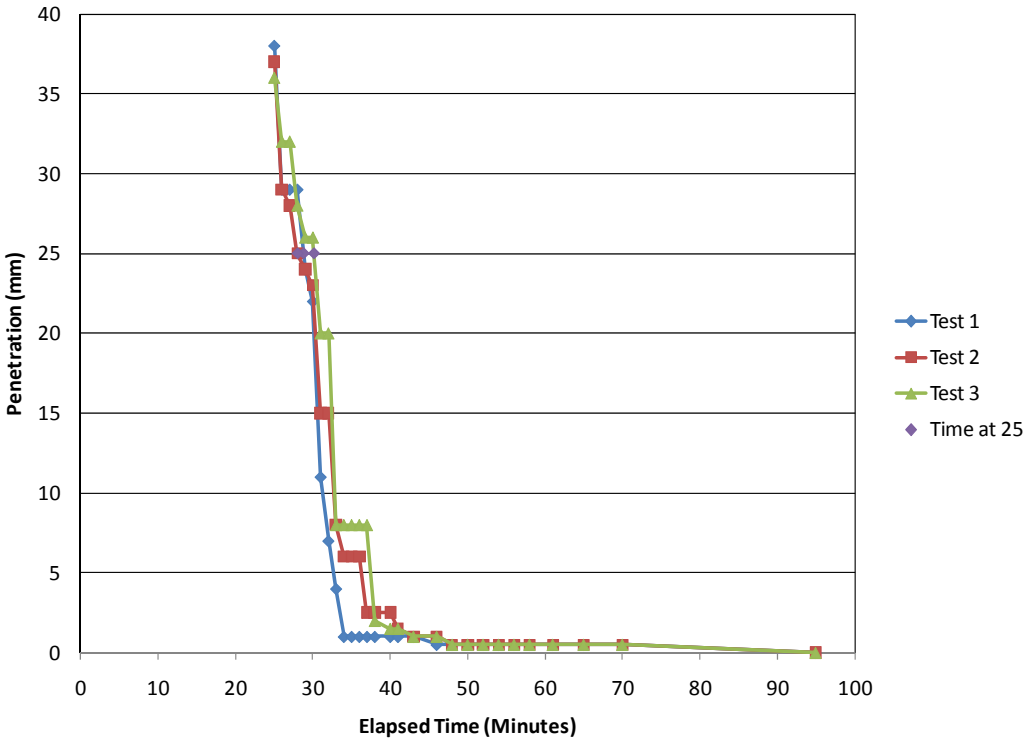



**Figure 76**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 12**



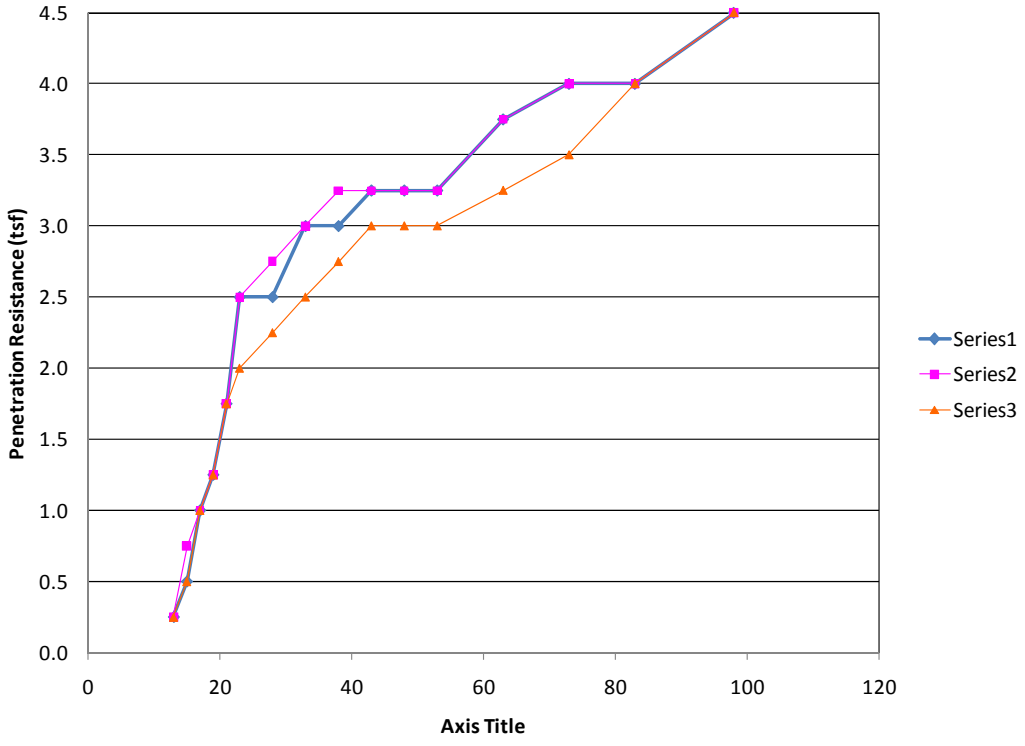




**Figure 77**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 13**

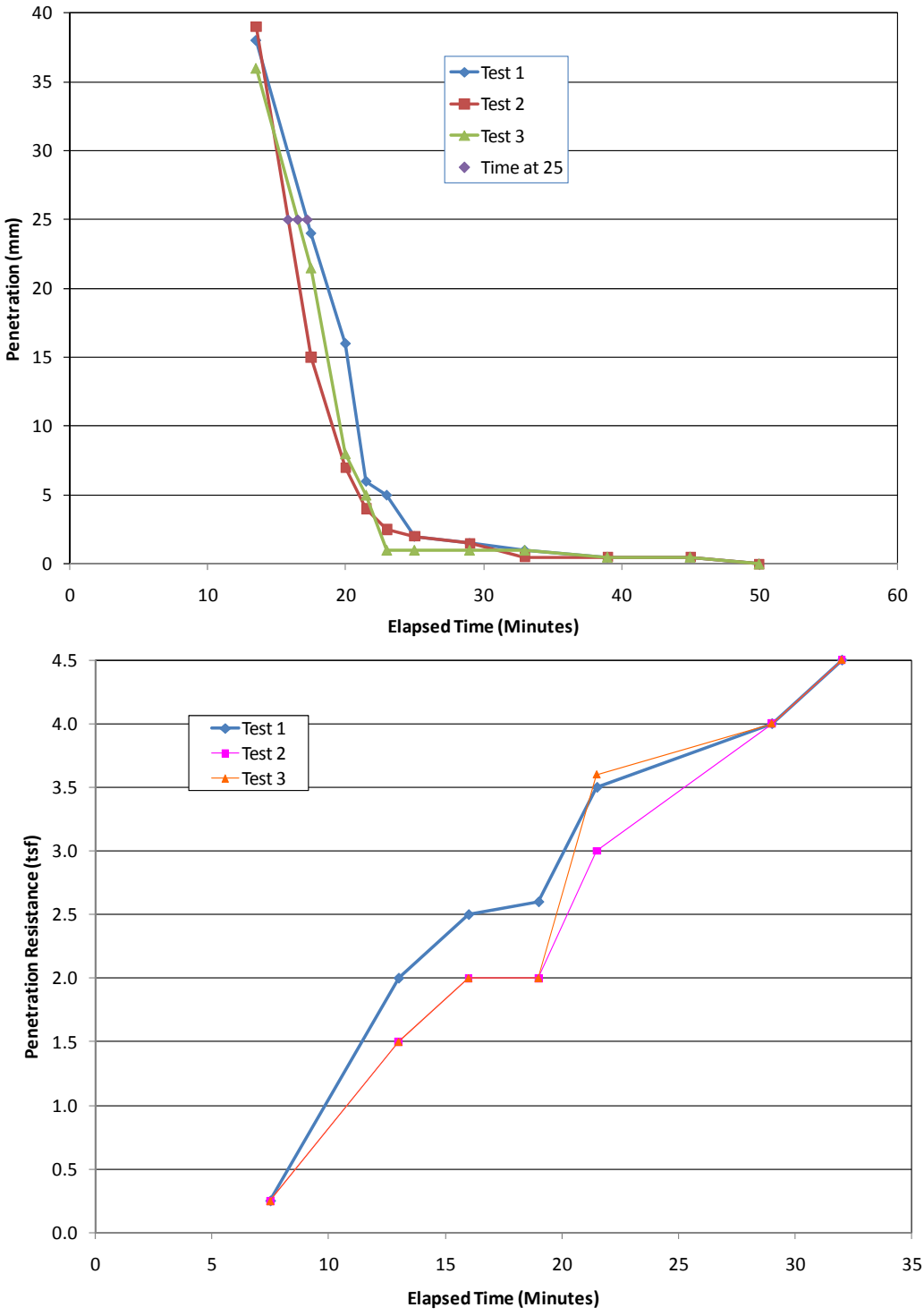
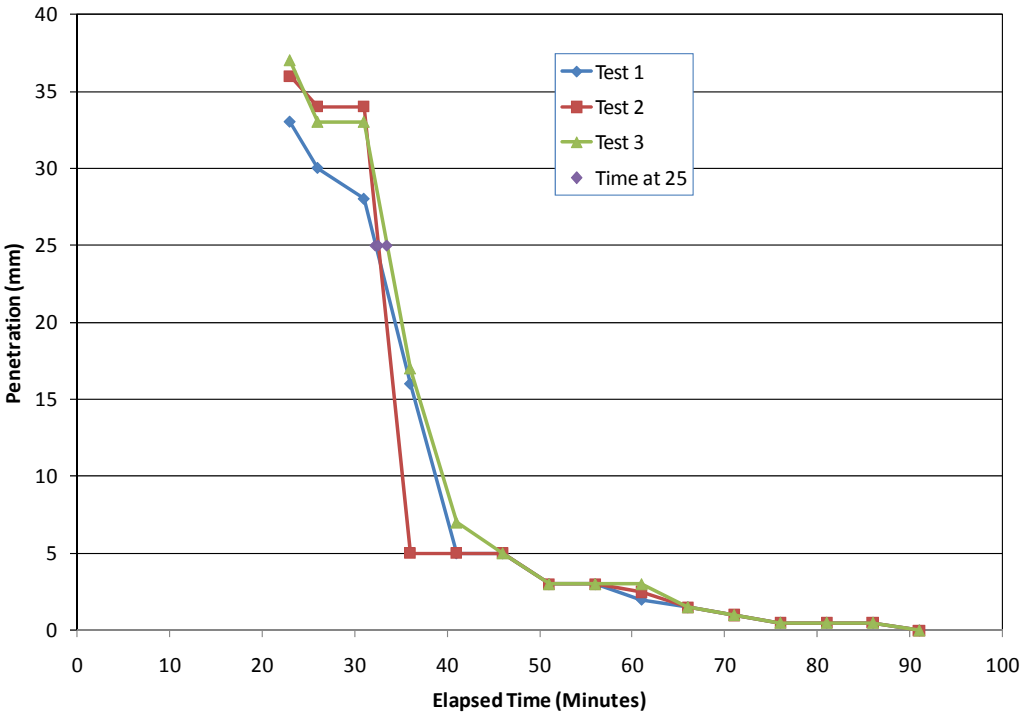



**Figure 78**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 14**



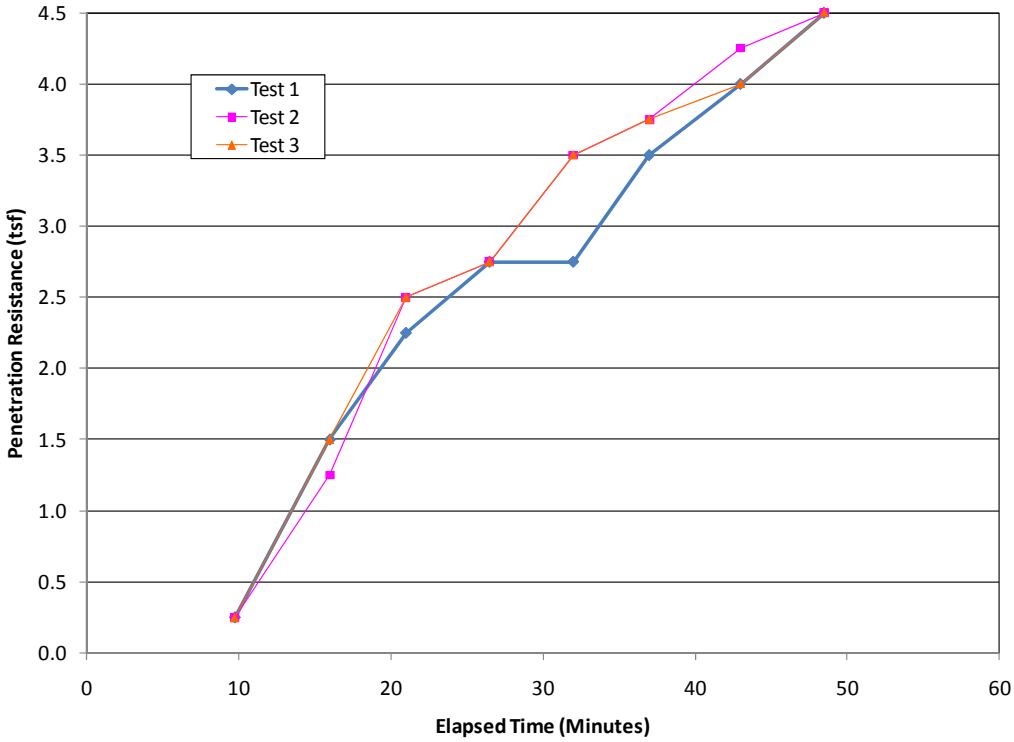




**Figure 79**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 15**

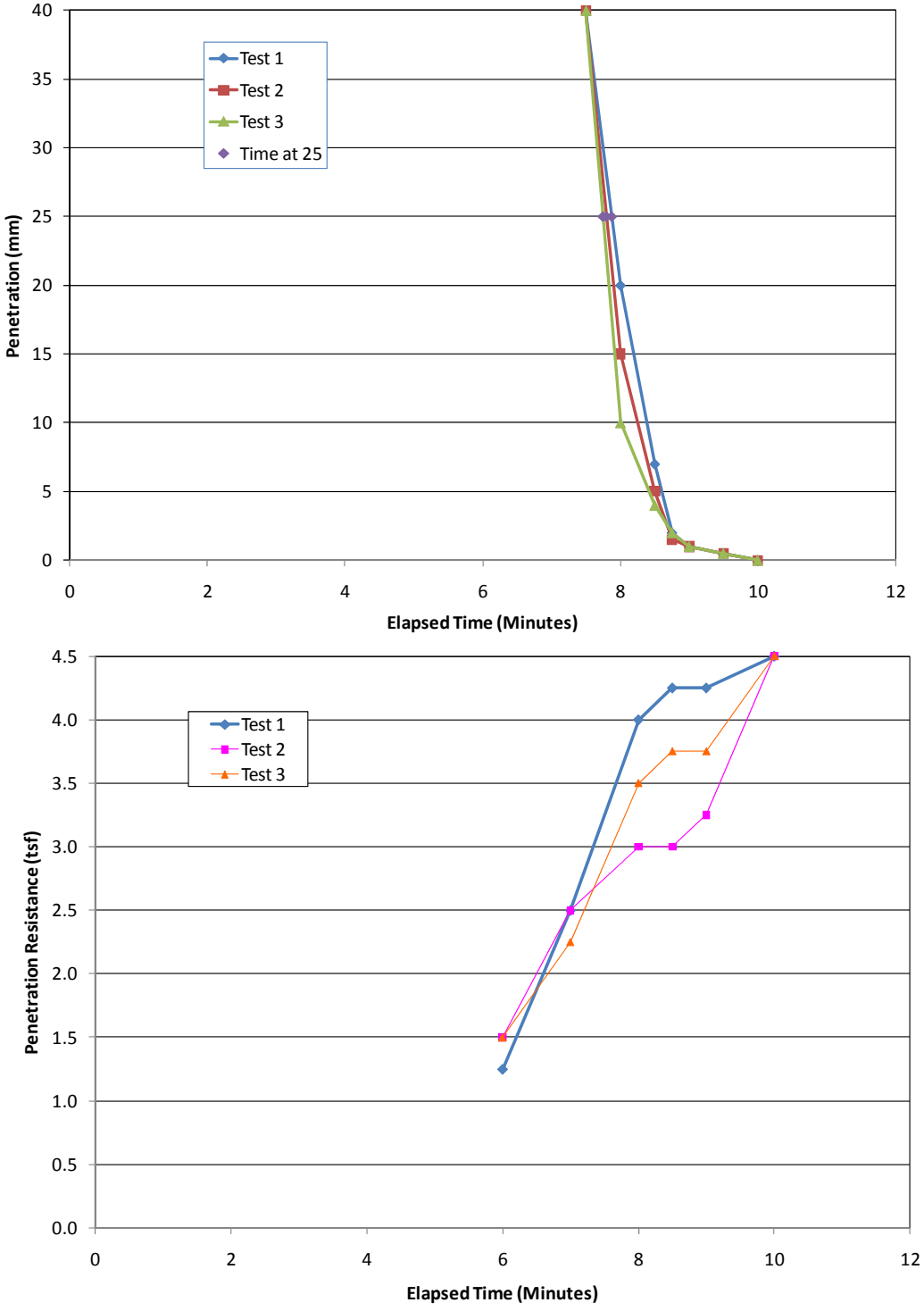



**Figure 80**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 16**

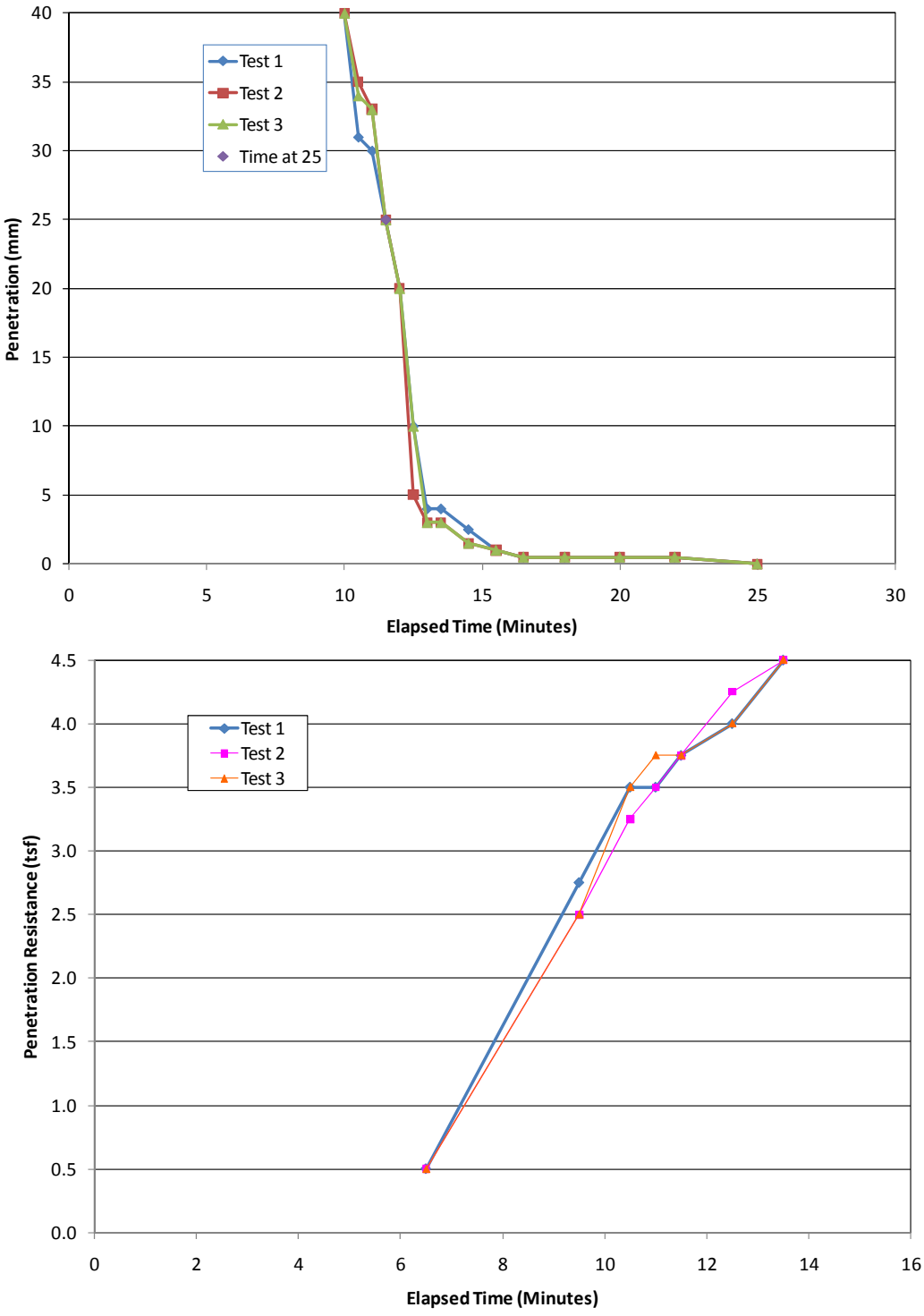



**Figure 81**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 17**

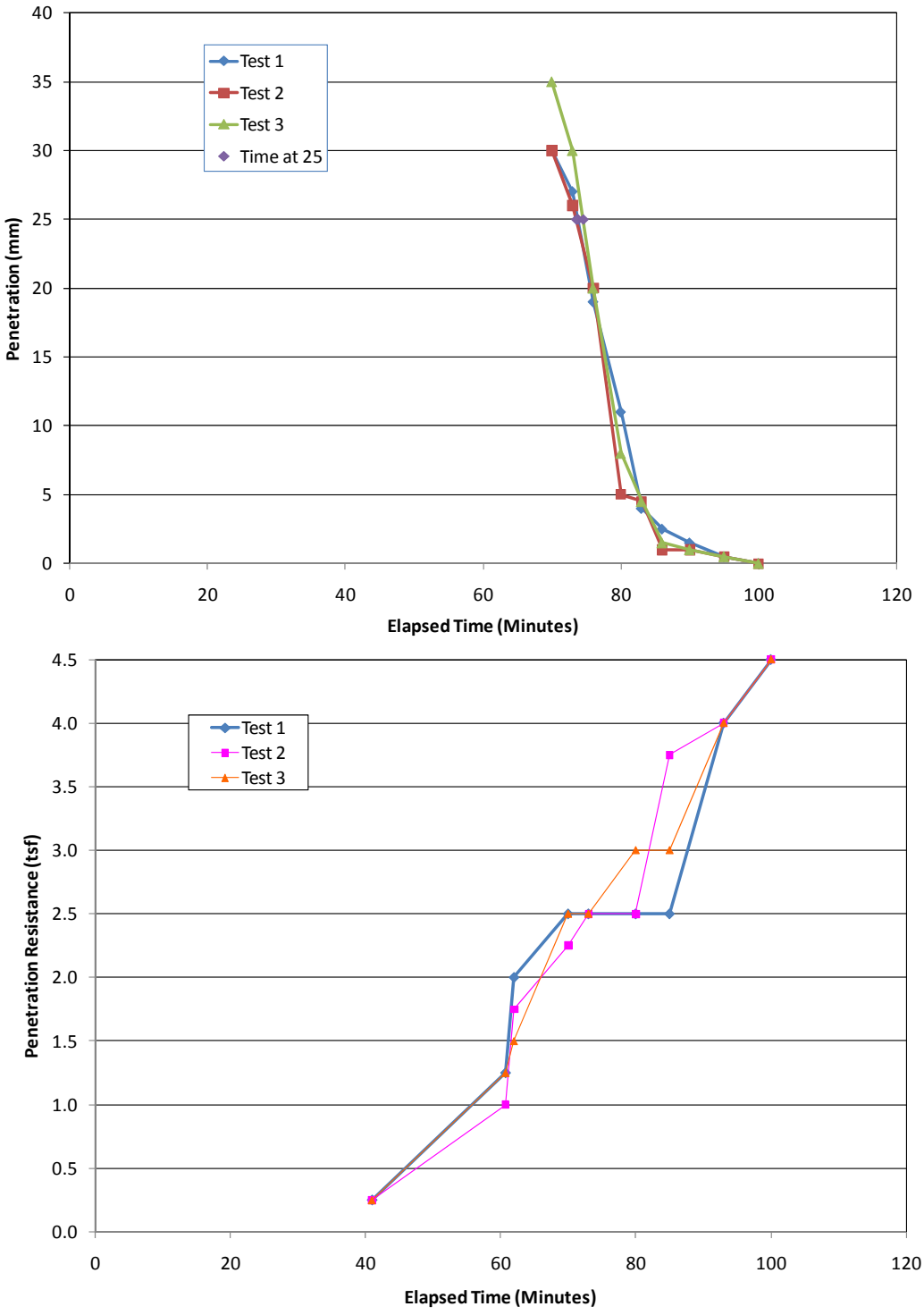



**Figure 82**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 18**

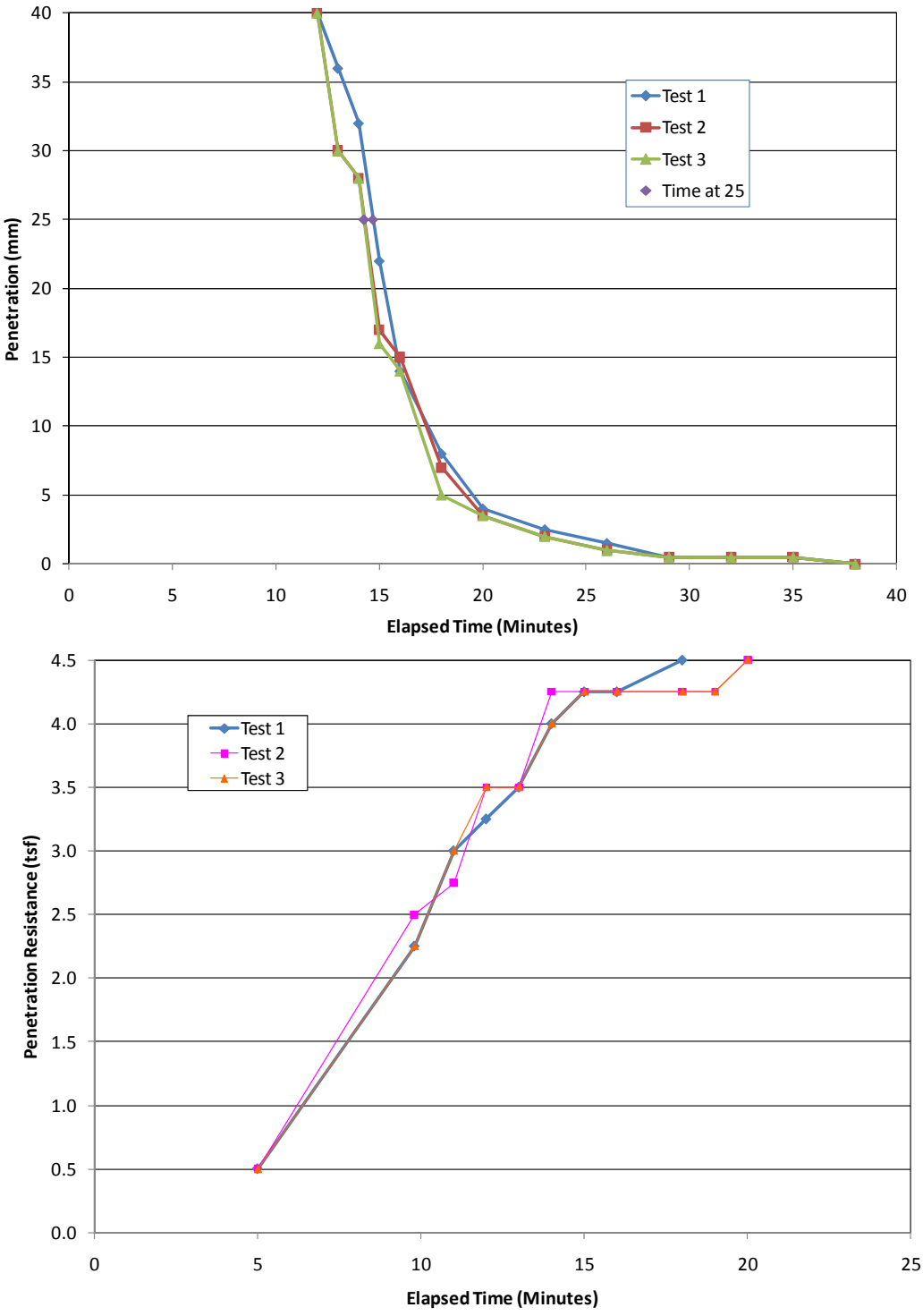



**Figure 83**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 19**

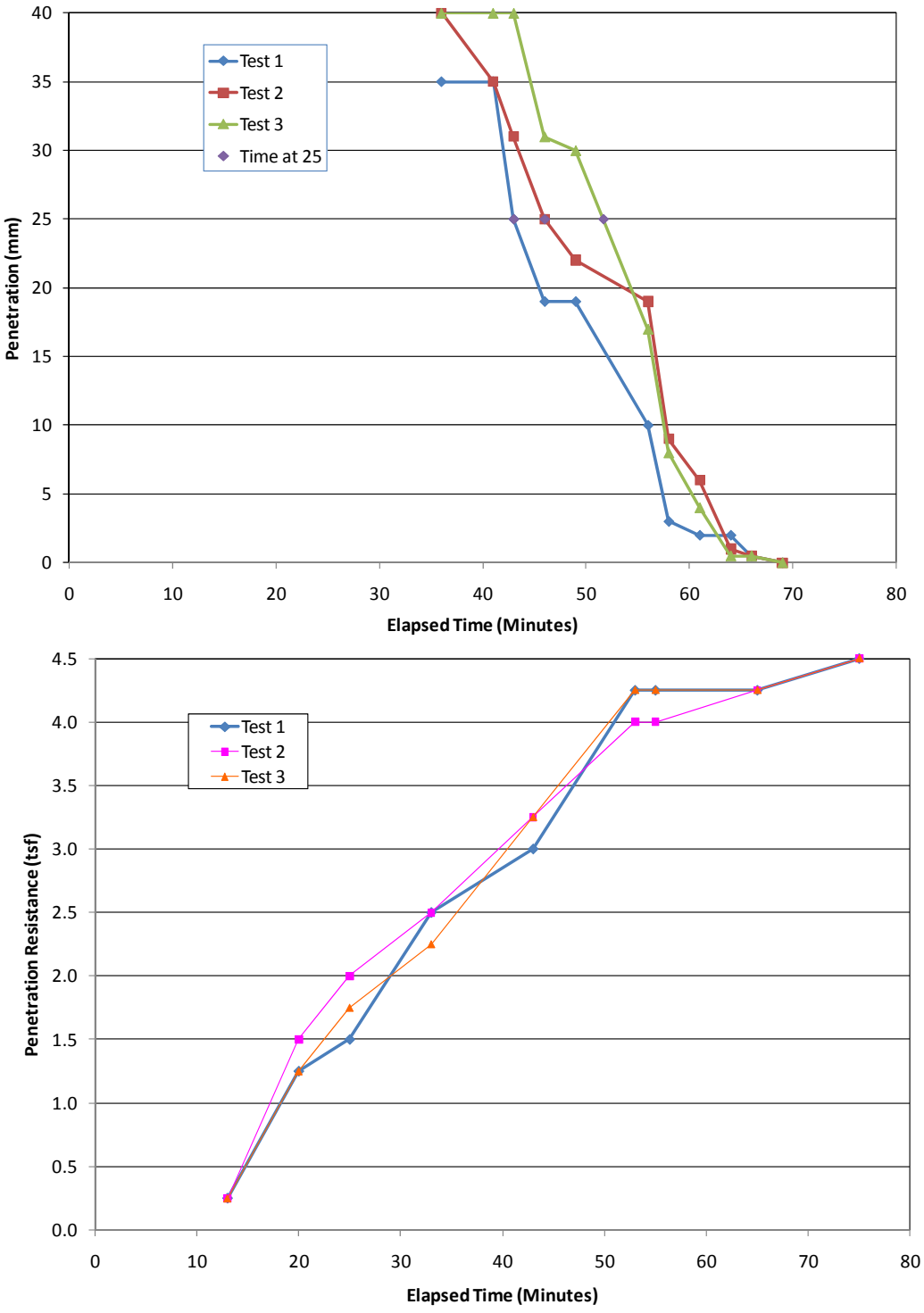



**Figure 84**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 3 Bucket 20**

## Source 4

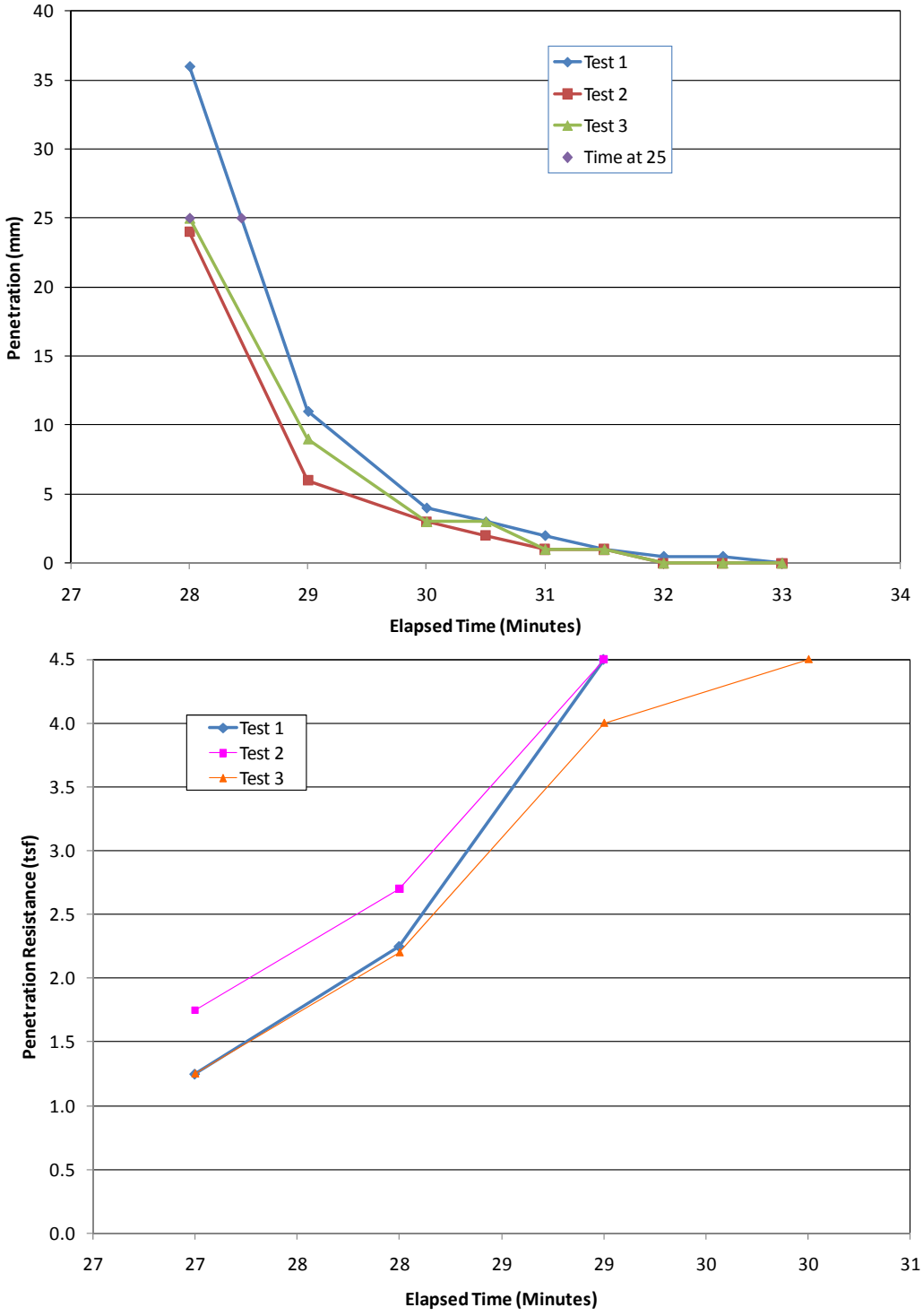



**Figure 85**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 1**

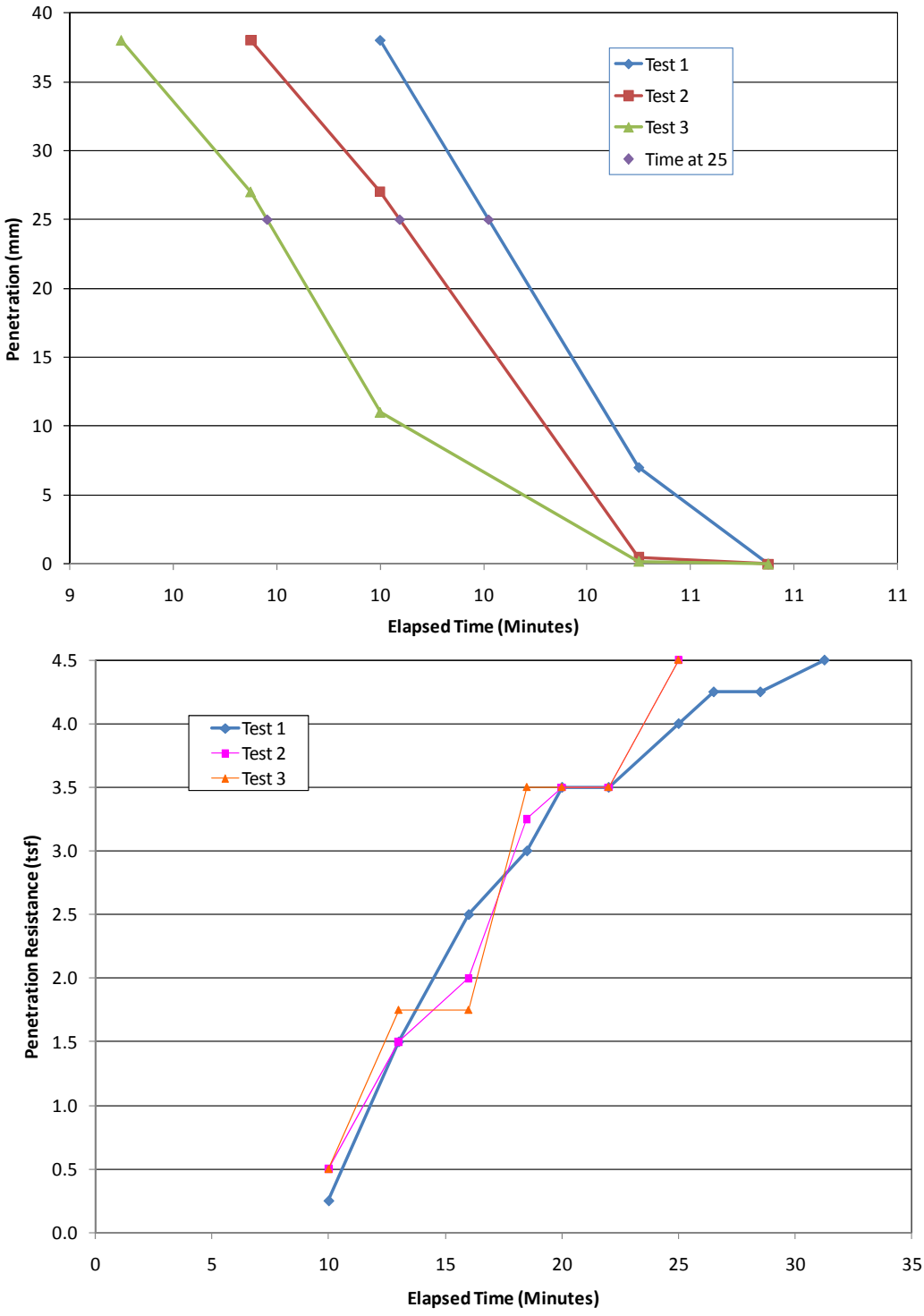
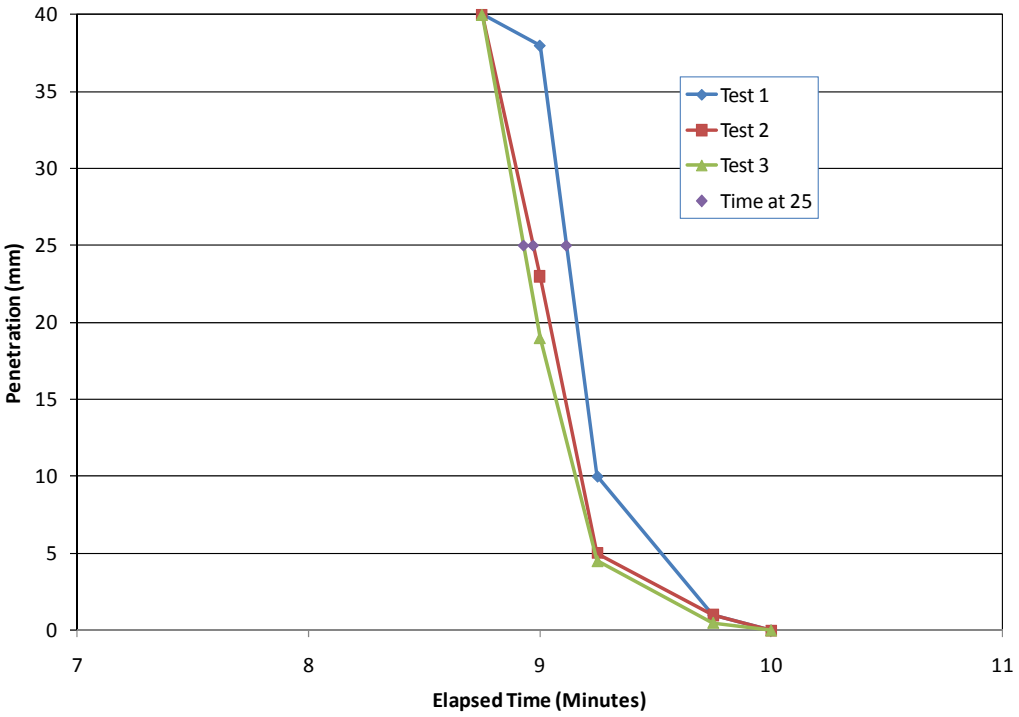



**Figure 86**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 2**



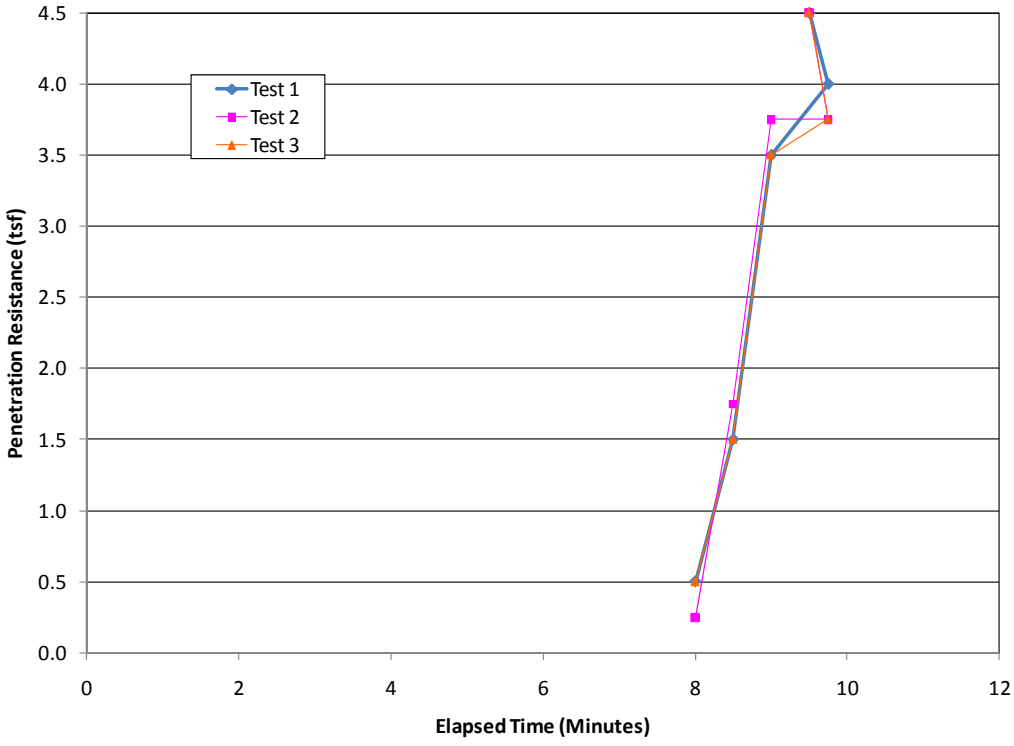




**Figure 87**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 3**

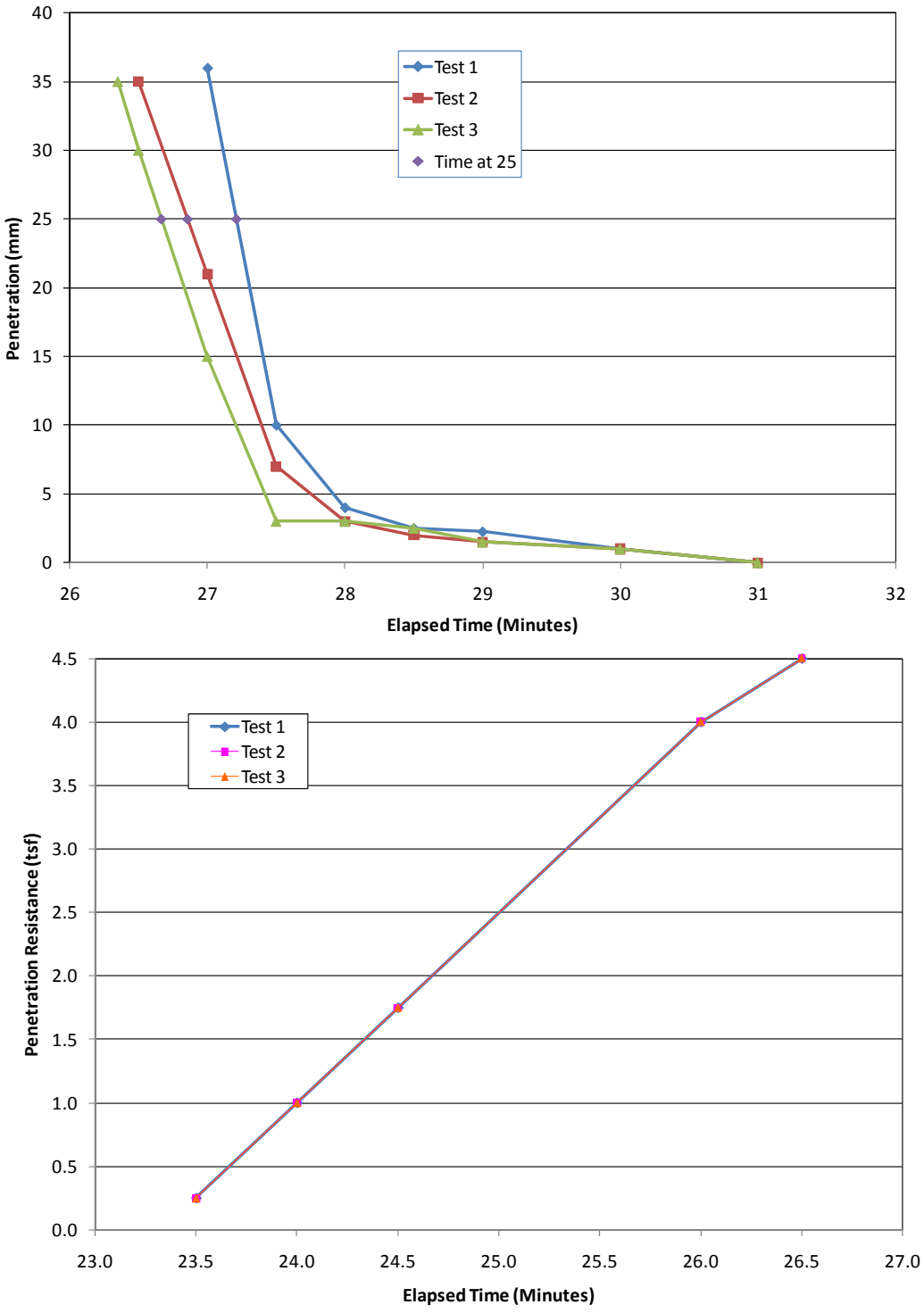



**Figure 88**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 4**

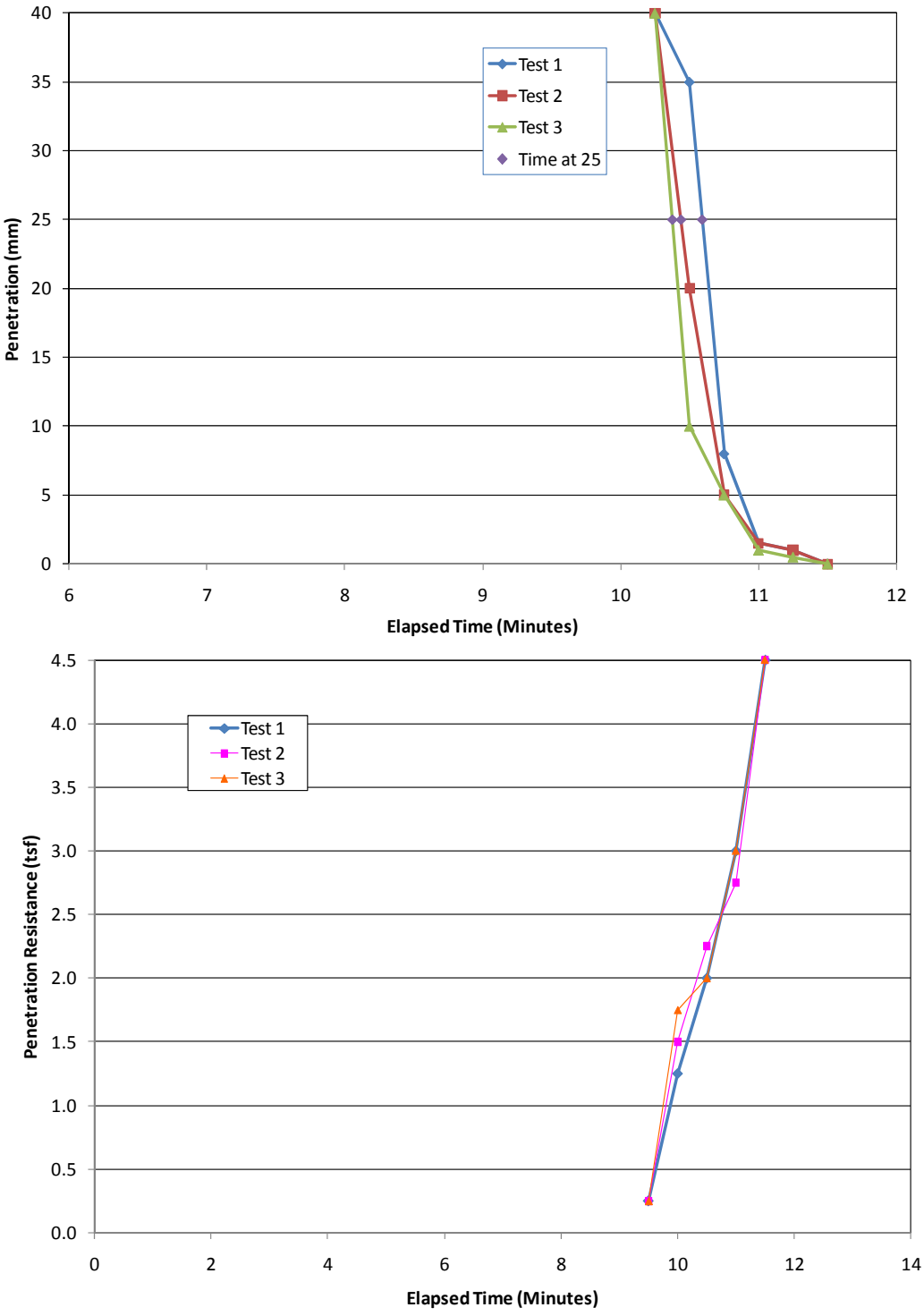
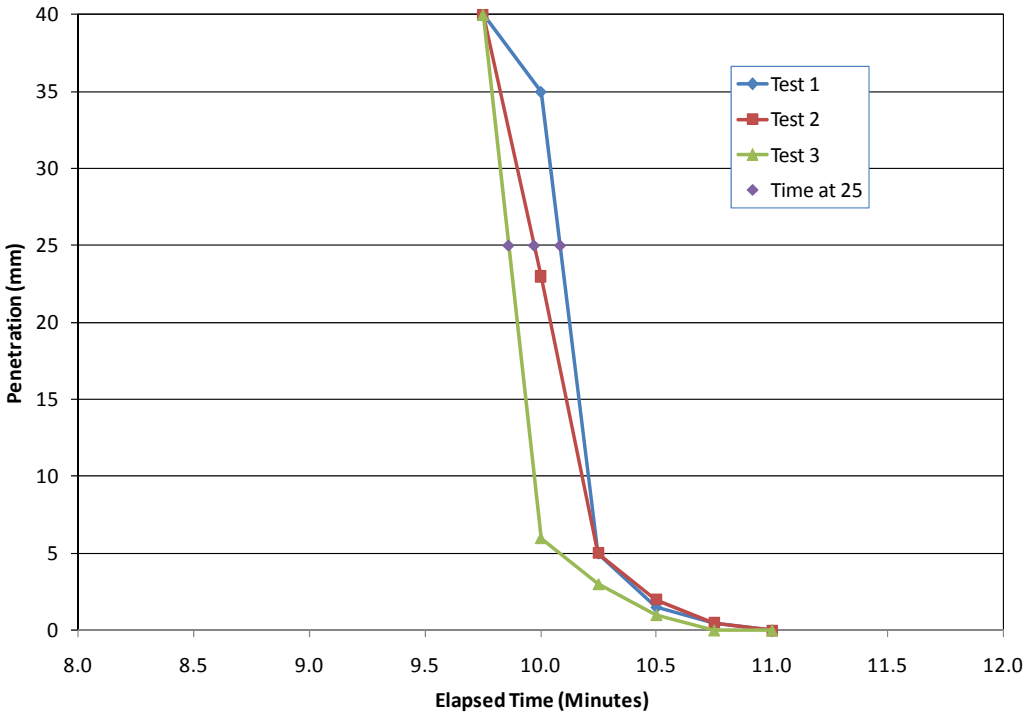



**Figure 89**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 5**



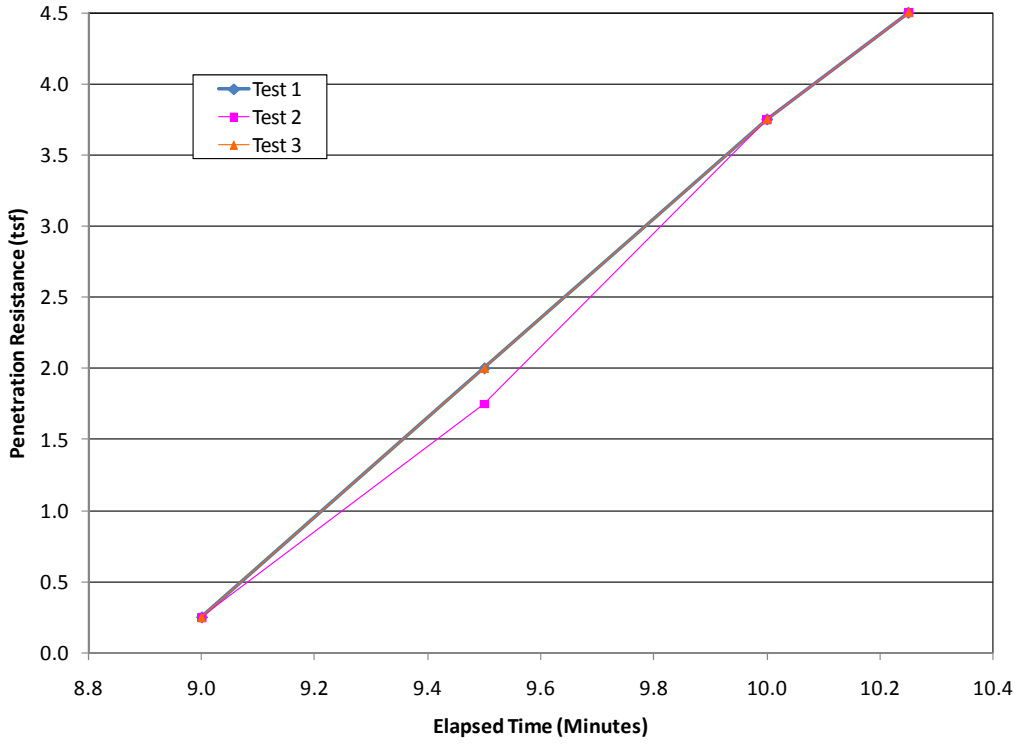
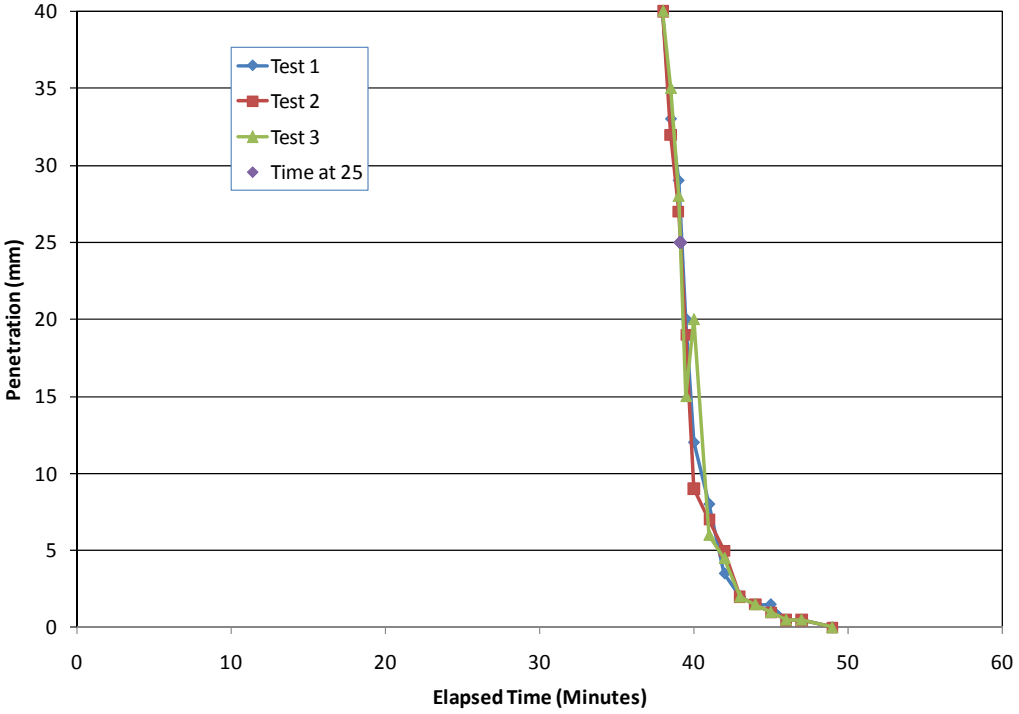




**Figure 90**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 6**



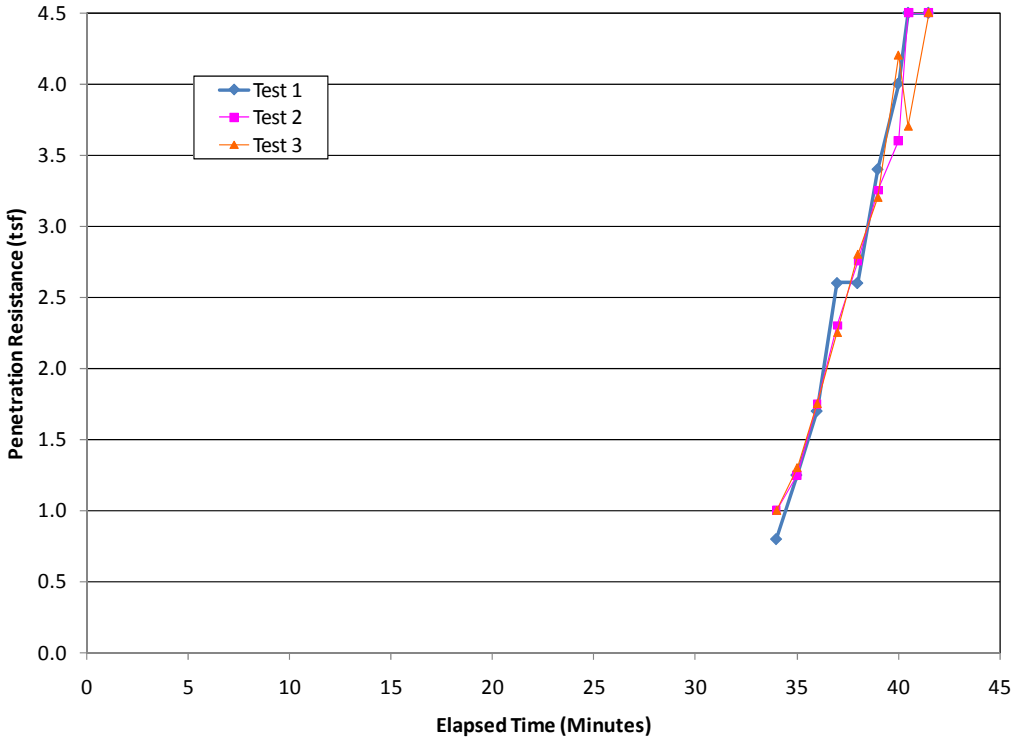




**Figure 91**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 7**

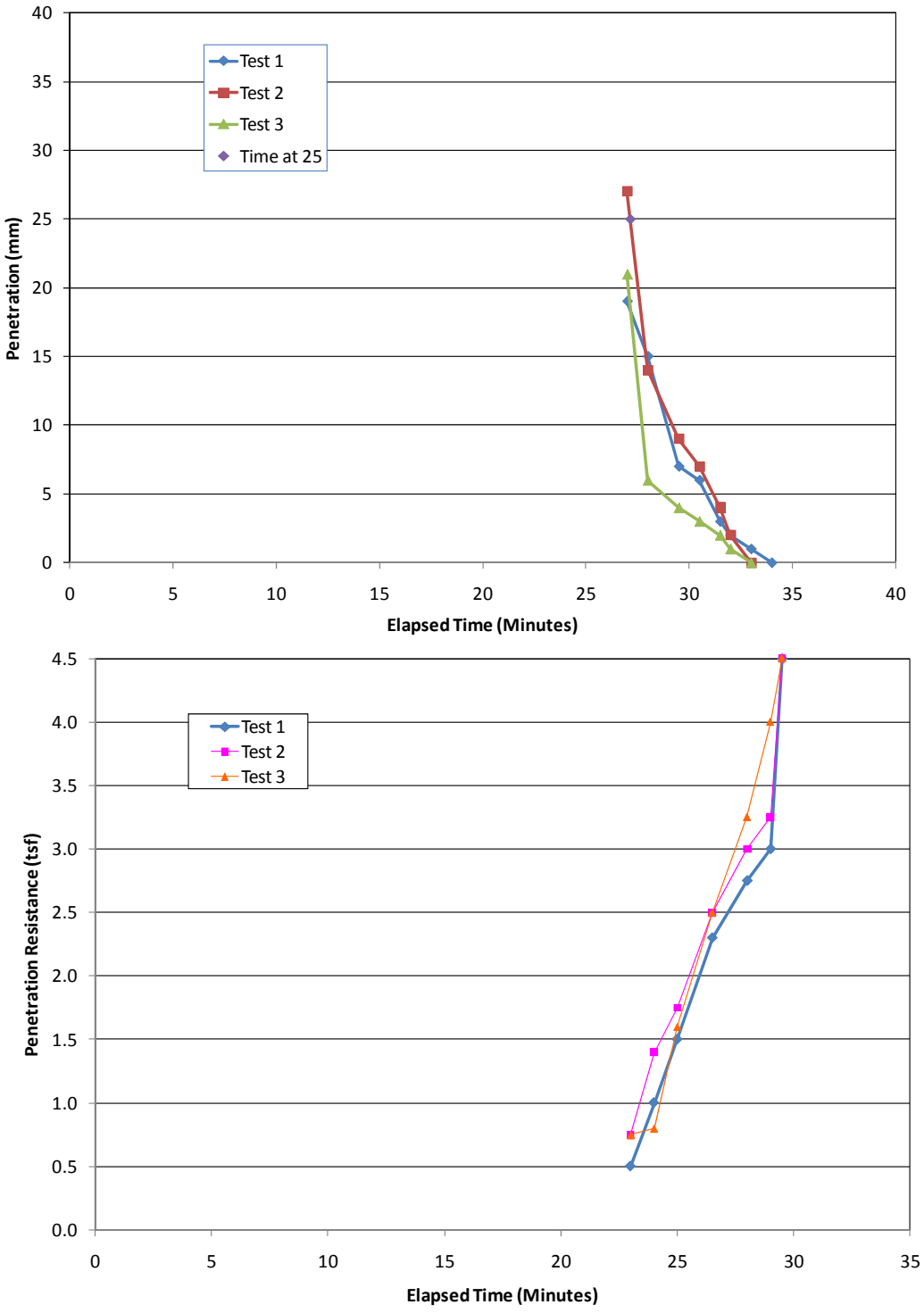



**Figure 92**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 8**

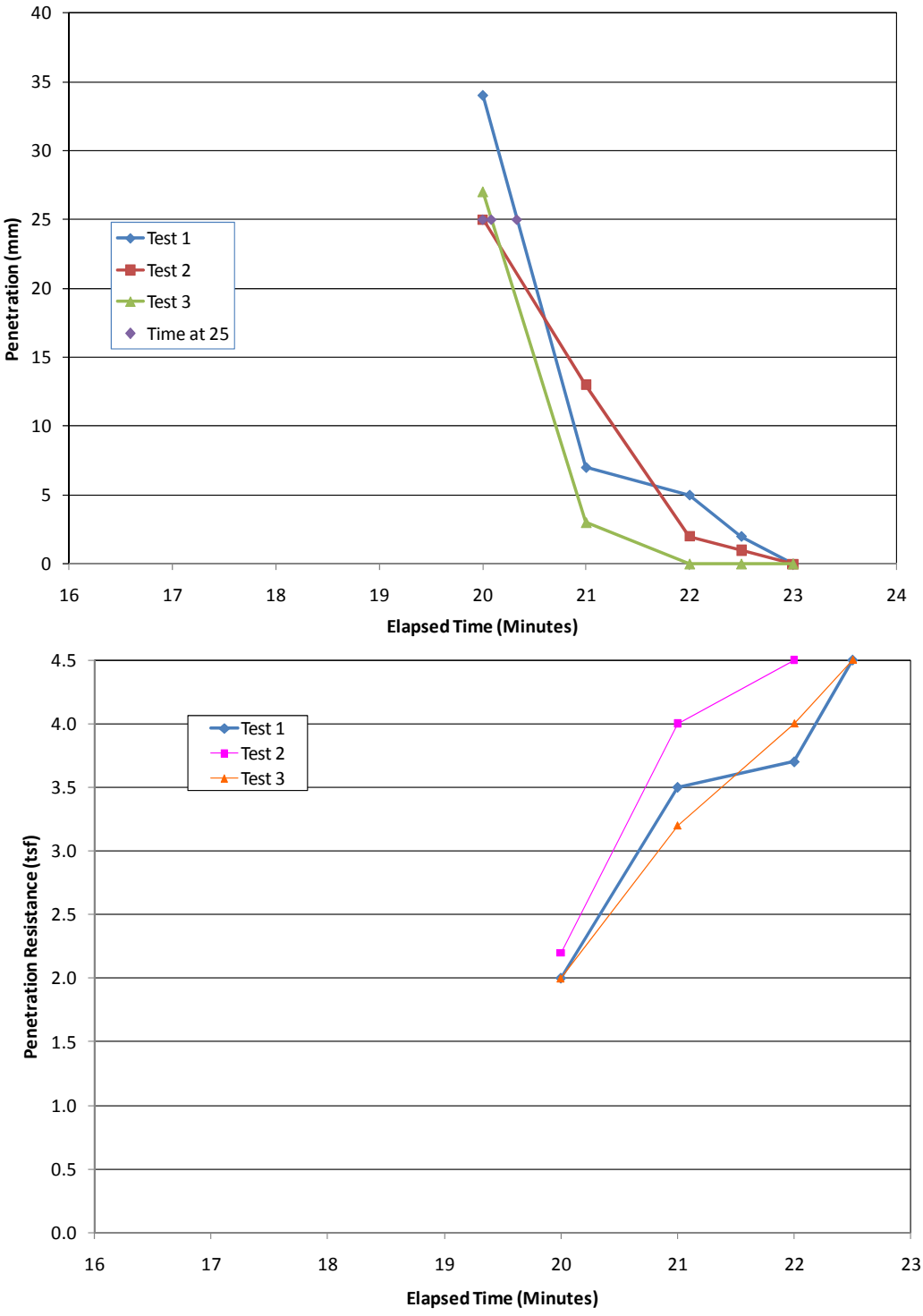



**Figure 93**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 9**

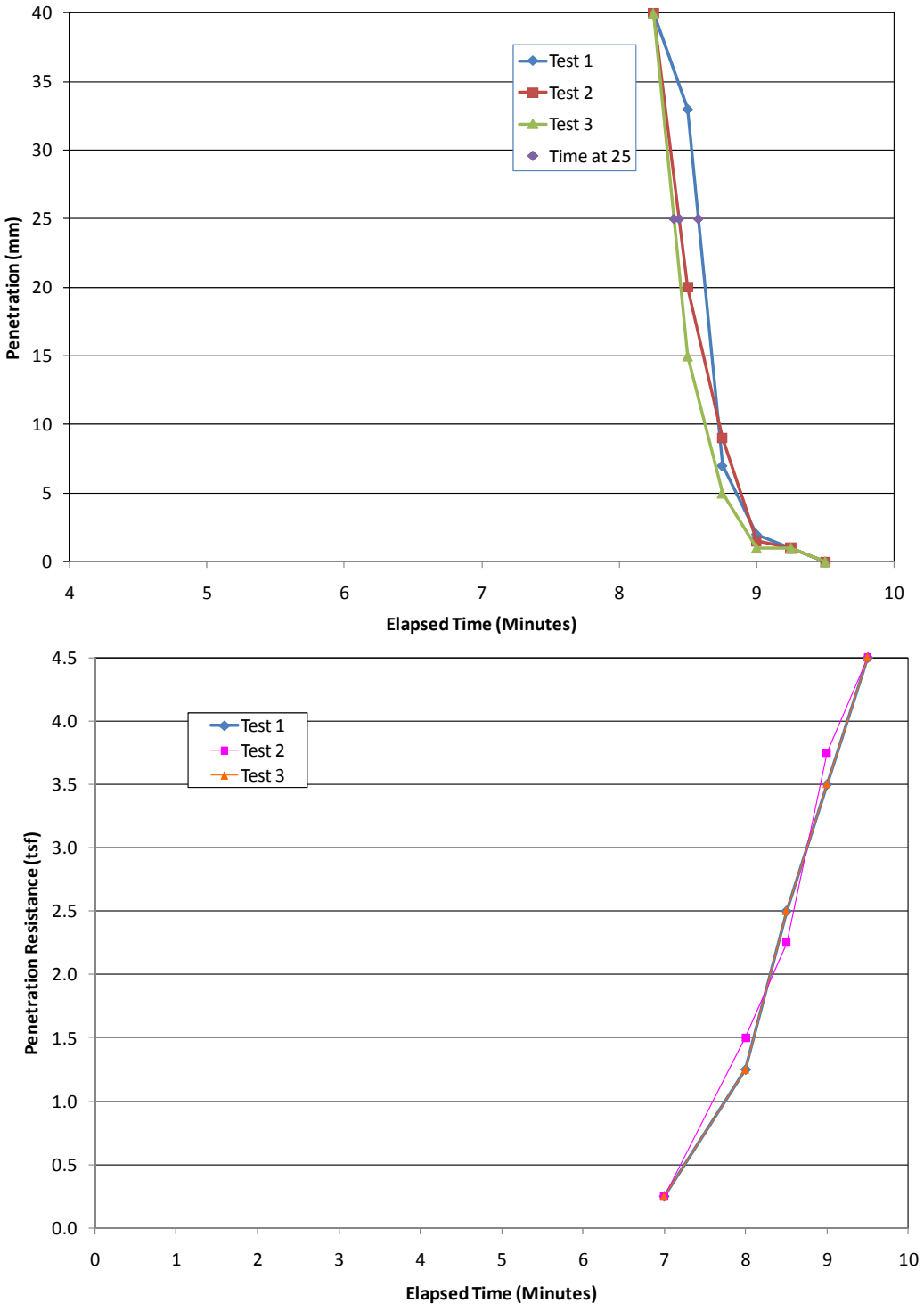
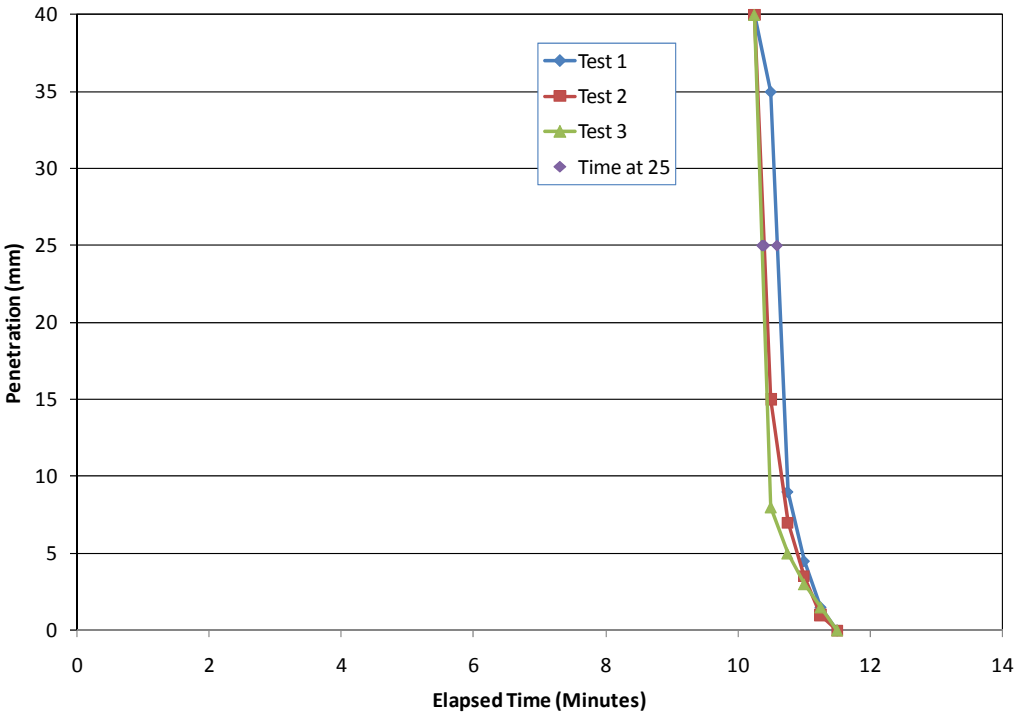



**Figure 94**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 10**



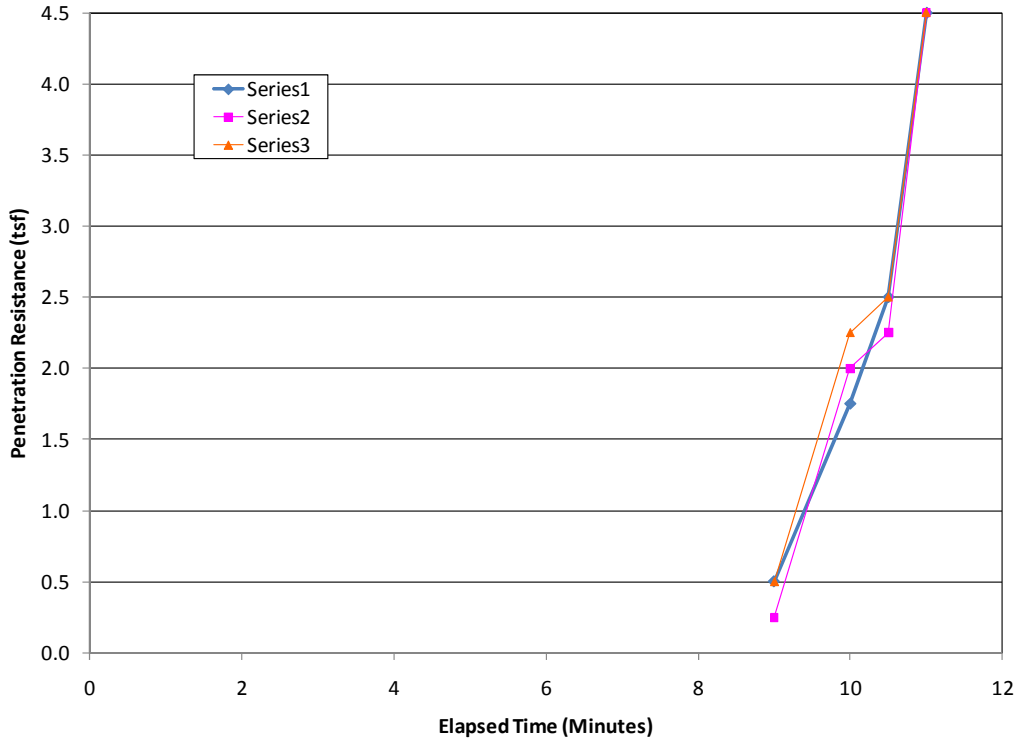




**Figure 95**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 11**

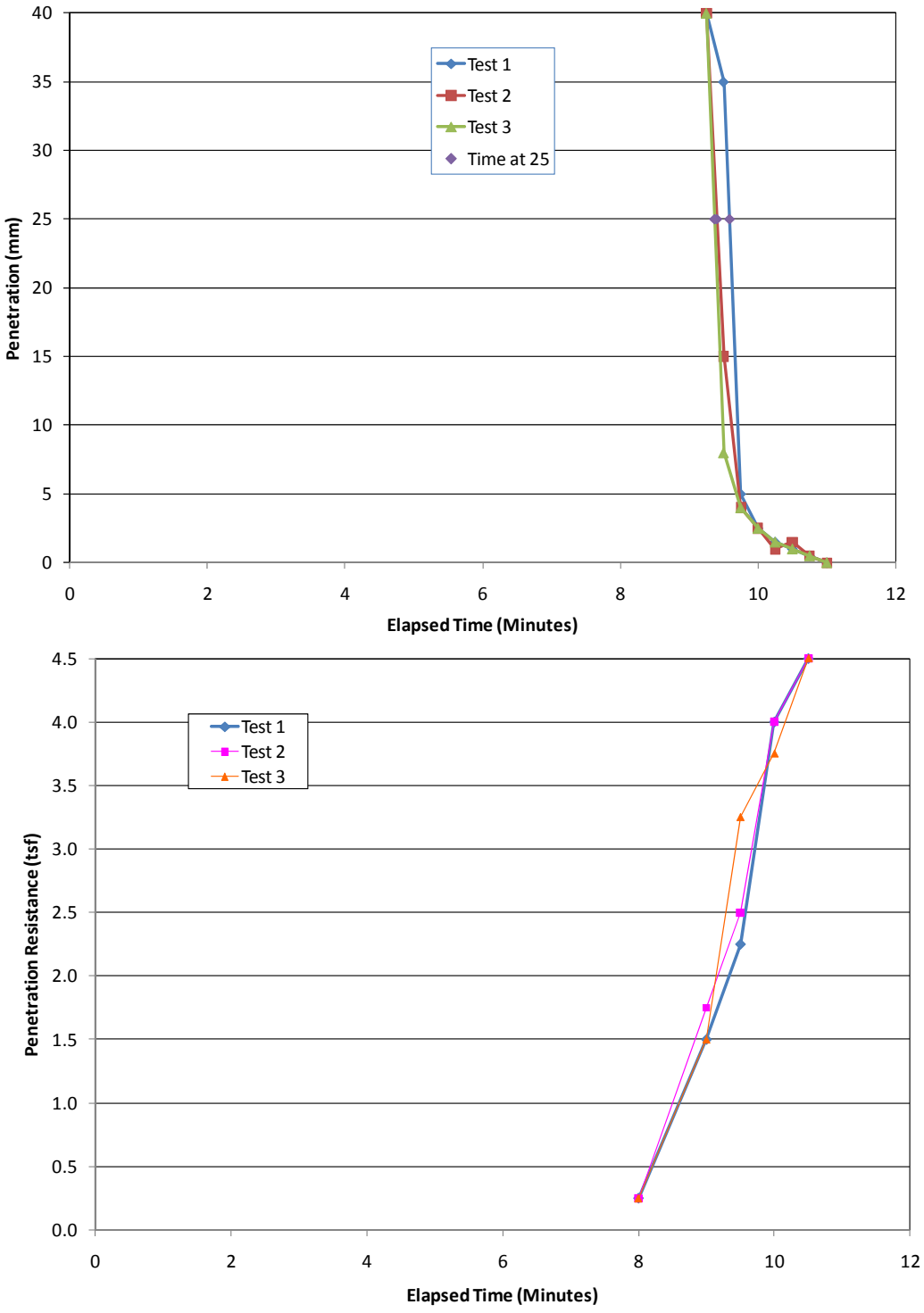



**Figure 96**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 12**

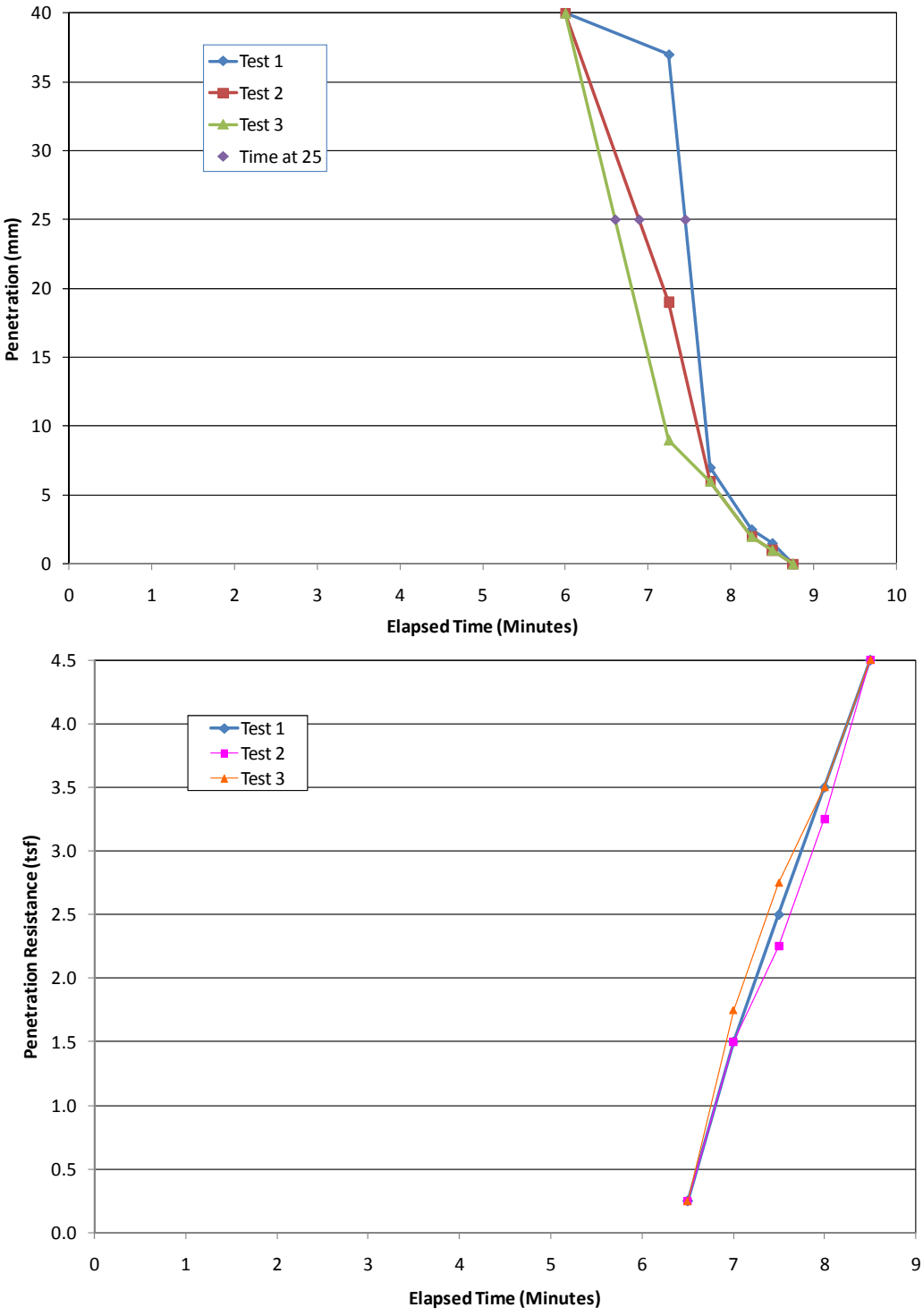
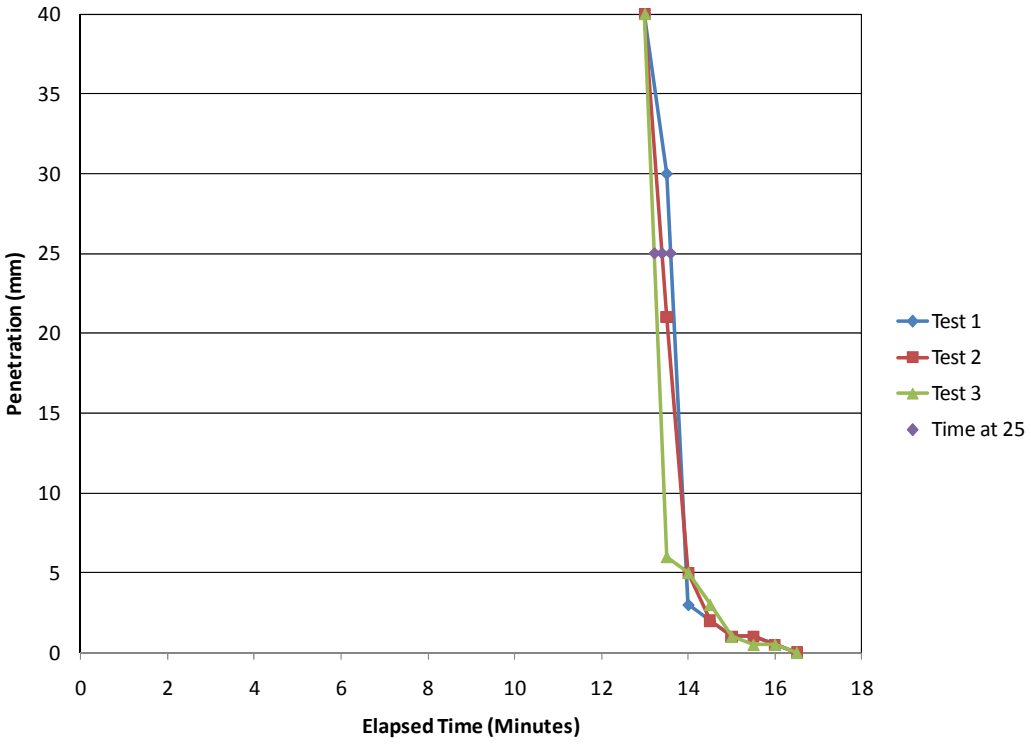



**Figure 97**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 13**



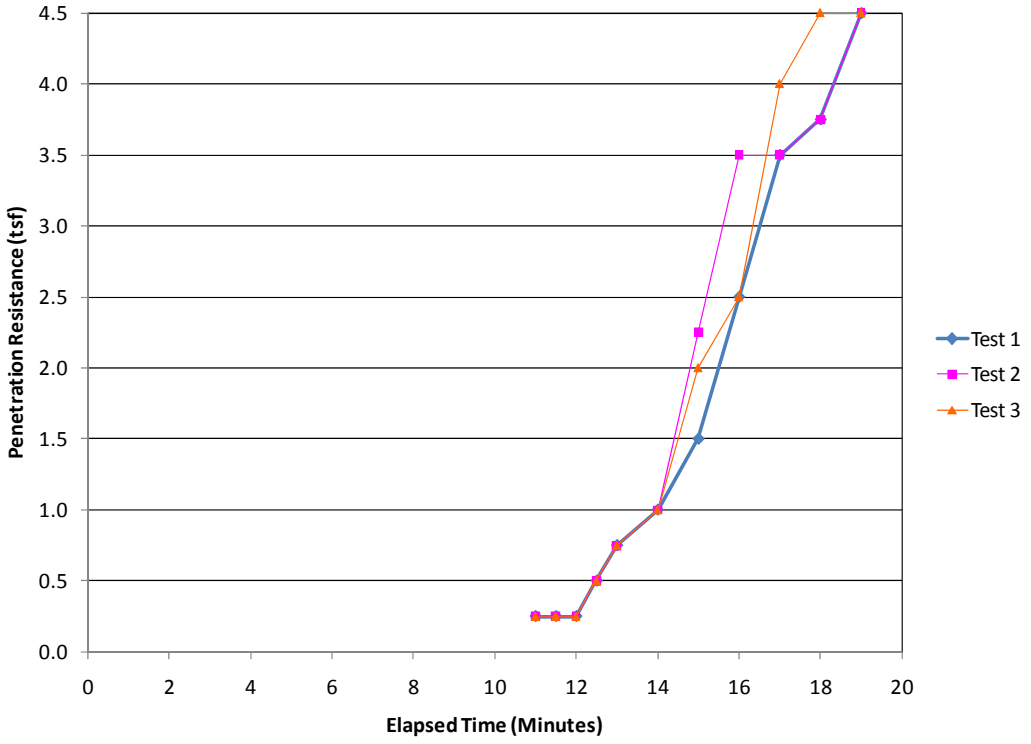
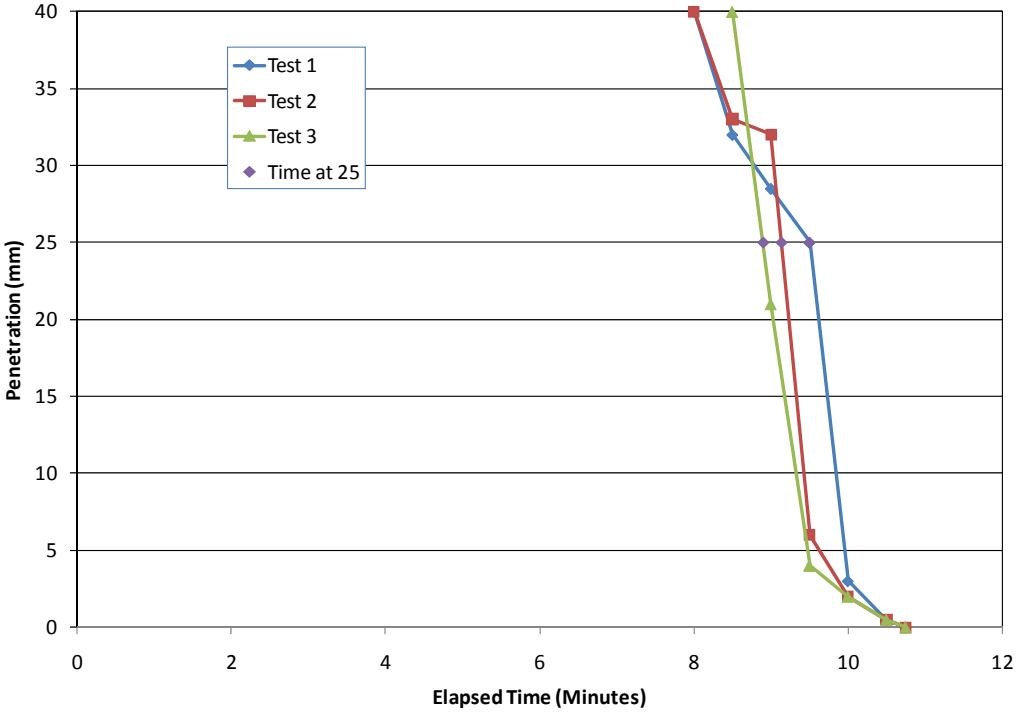




**Figure 98**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 14**



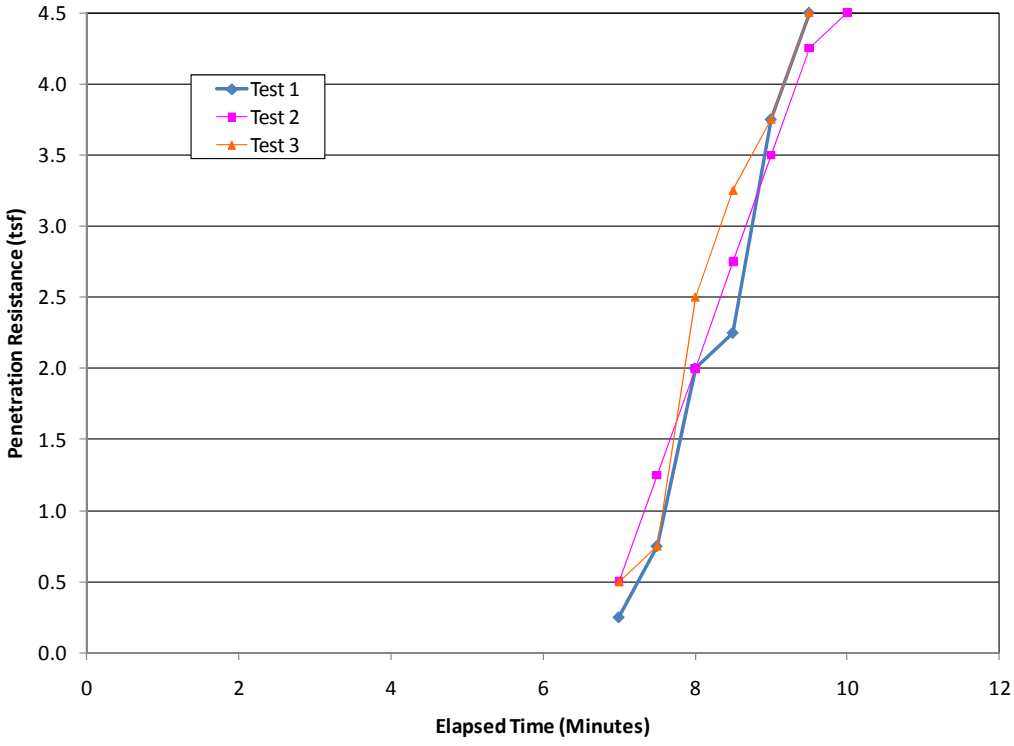




**Figure 99**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 15**

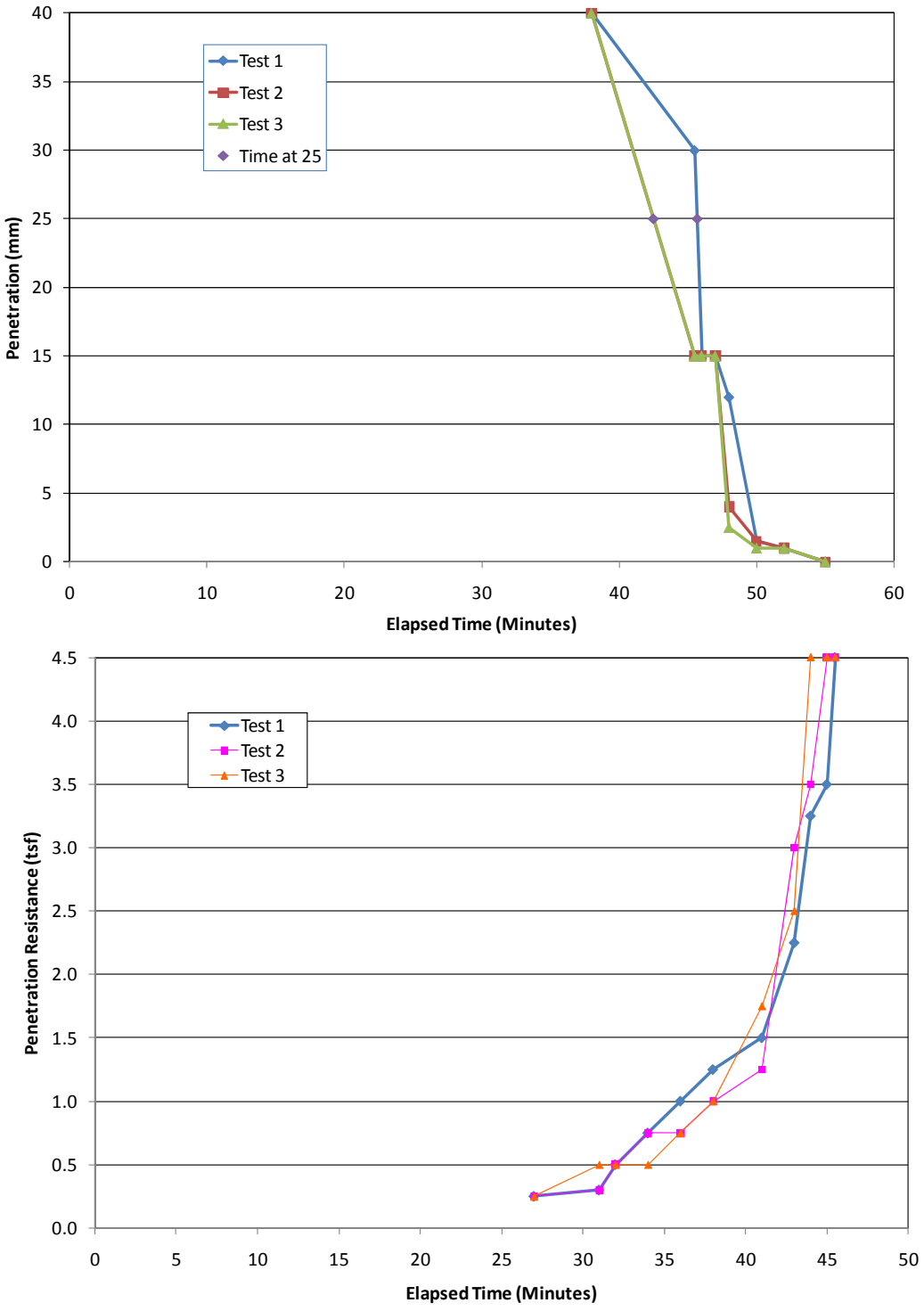
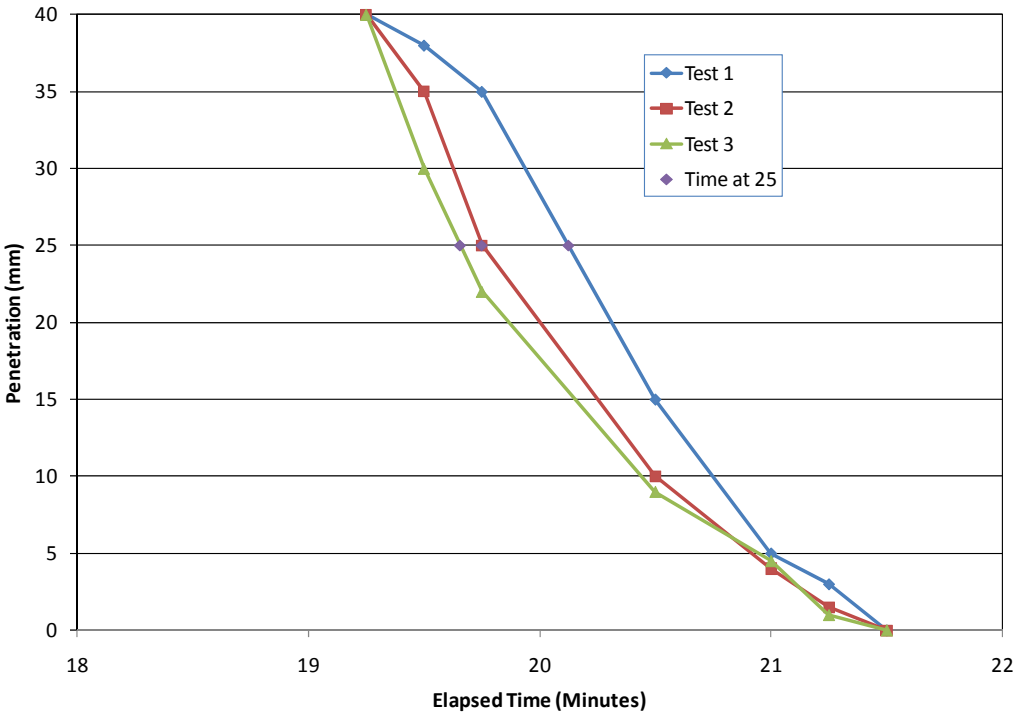



**Figure 100**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 16**



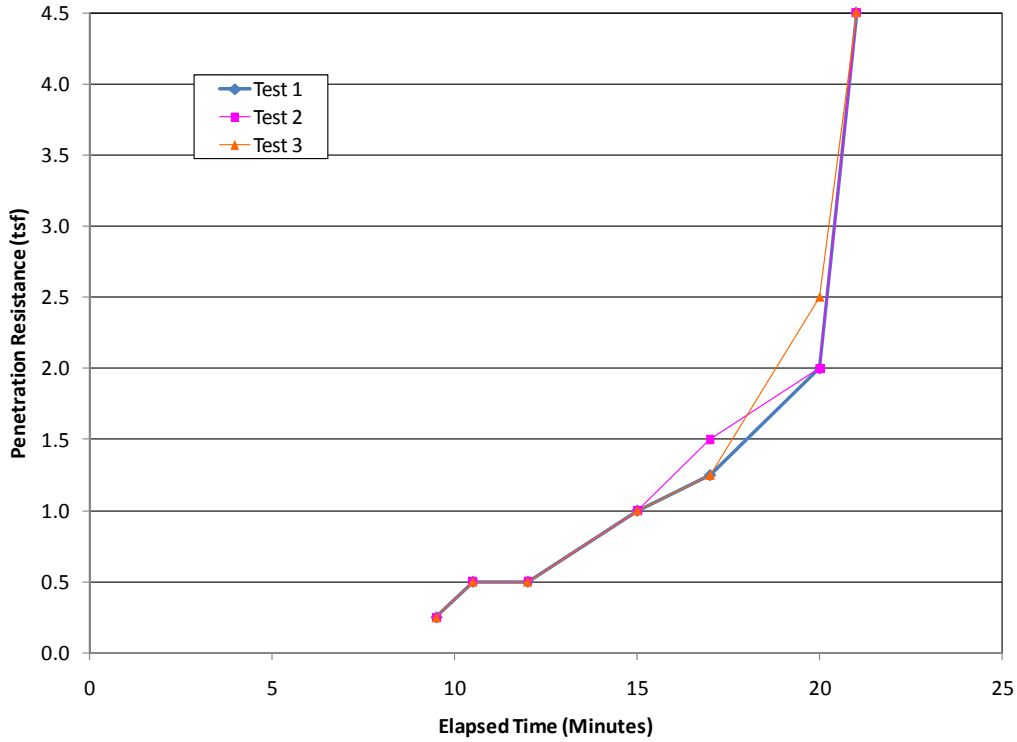




**Figure 101**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 17**

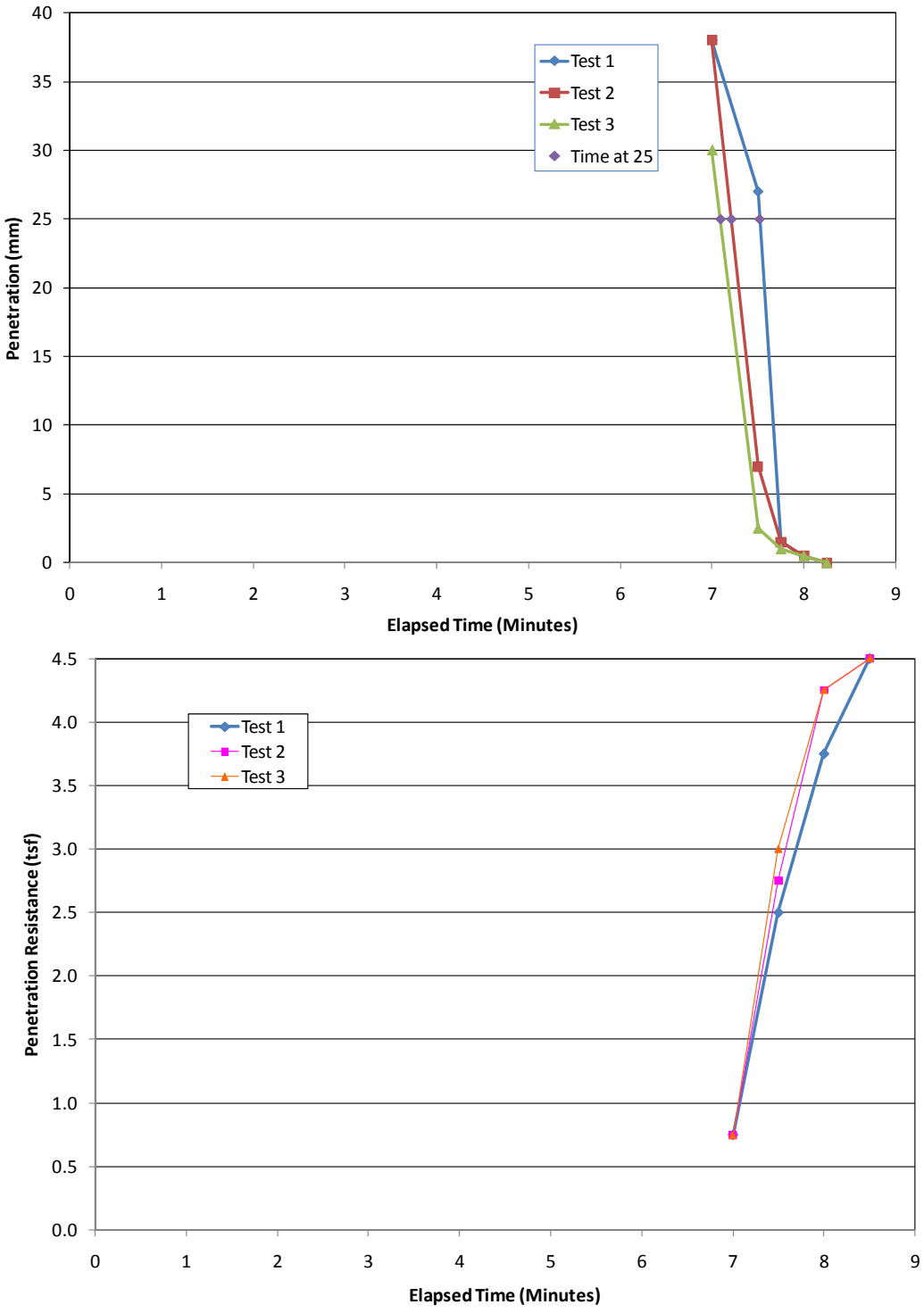



**Figure 102**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 18**

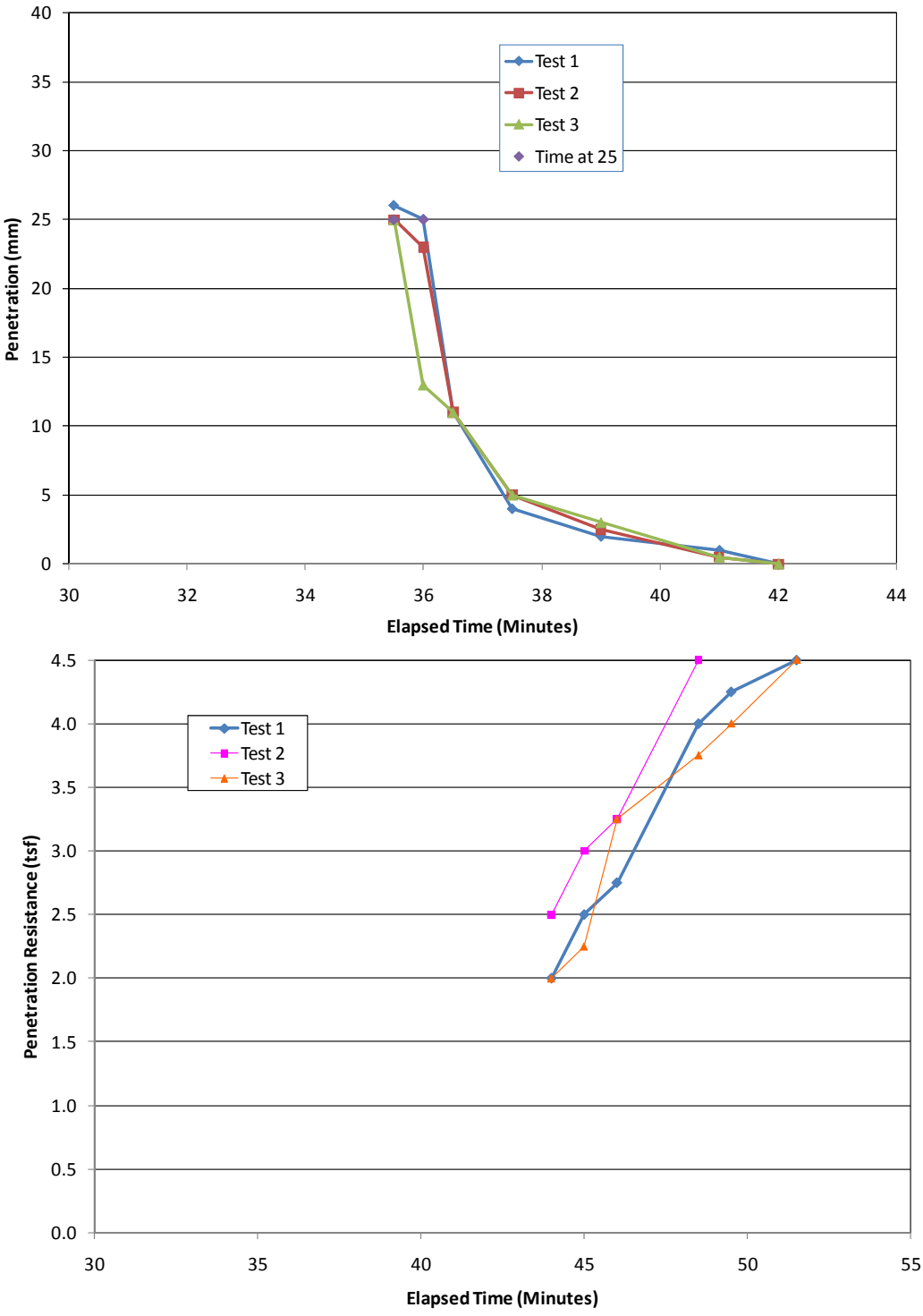



**Figure 103**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 19**

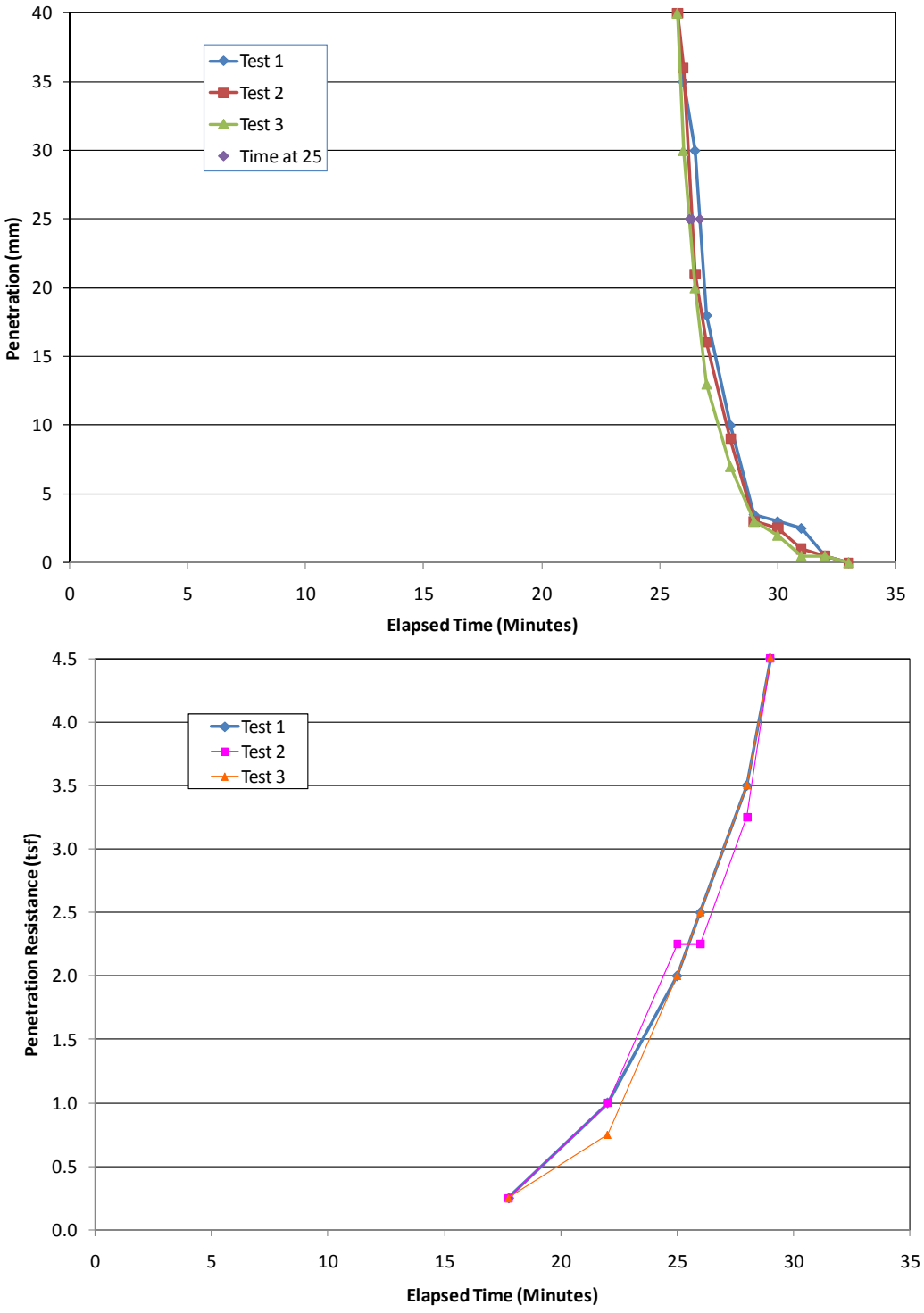



**Figure 104**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 4 Bucket 20**

## Source 5

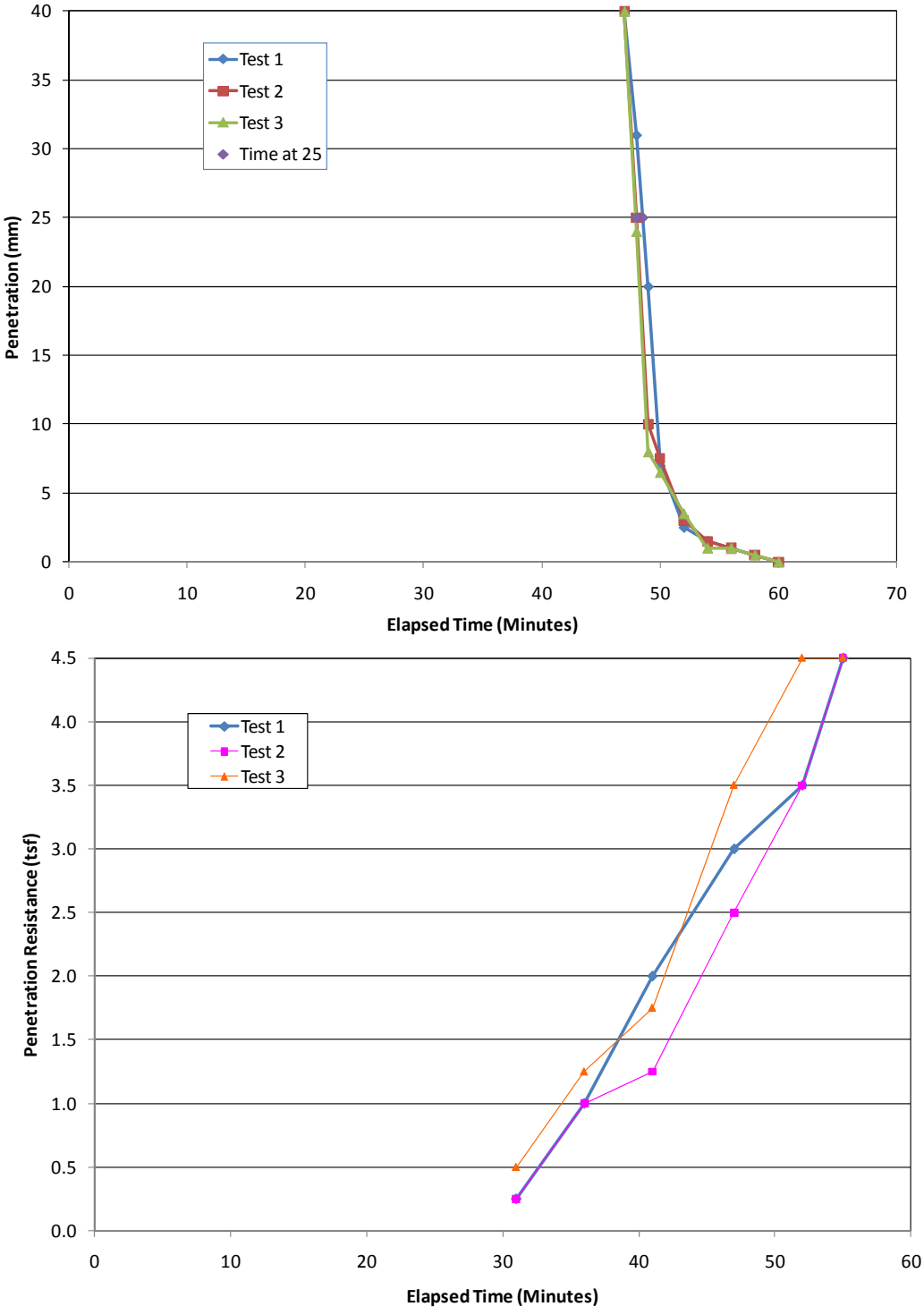



**Figure 105**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 1**

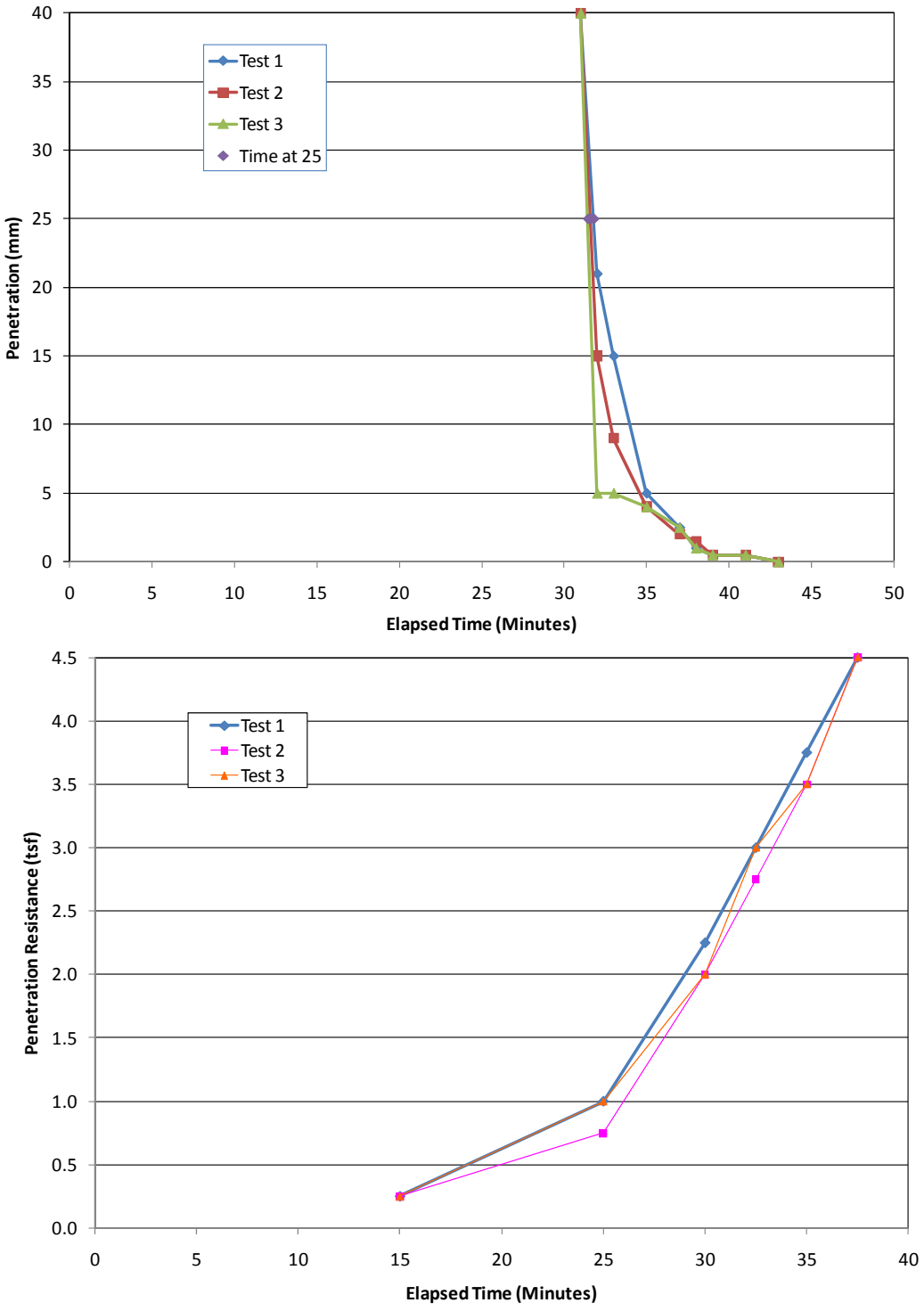



**Figure 106**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 2**

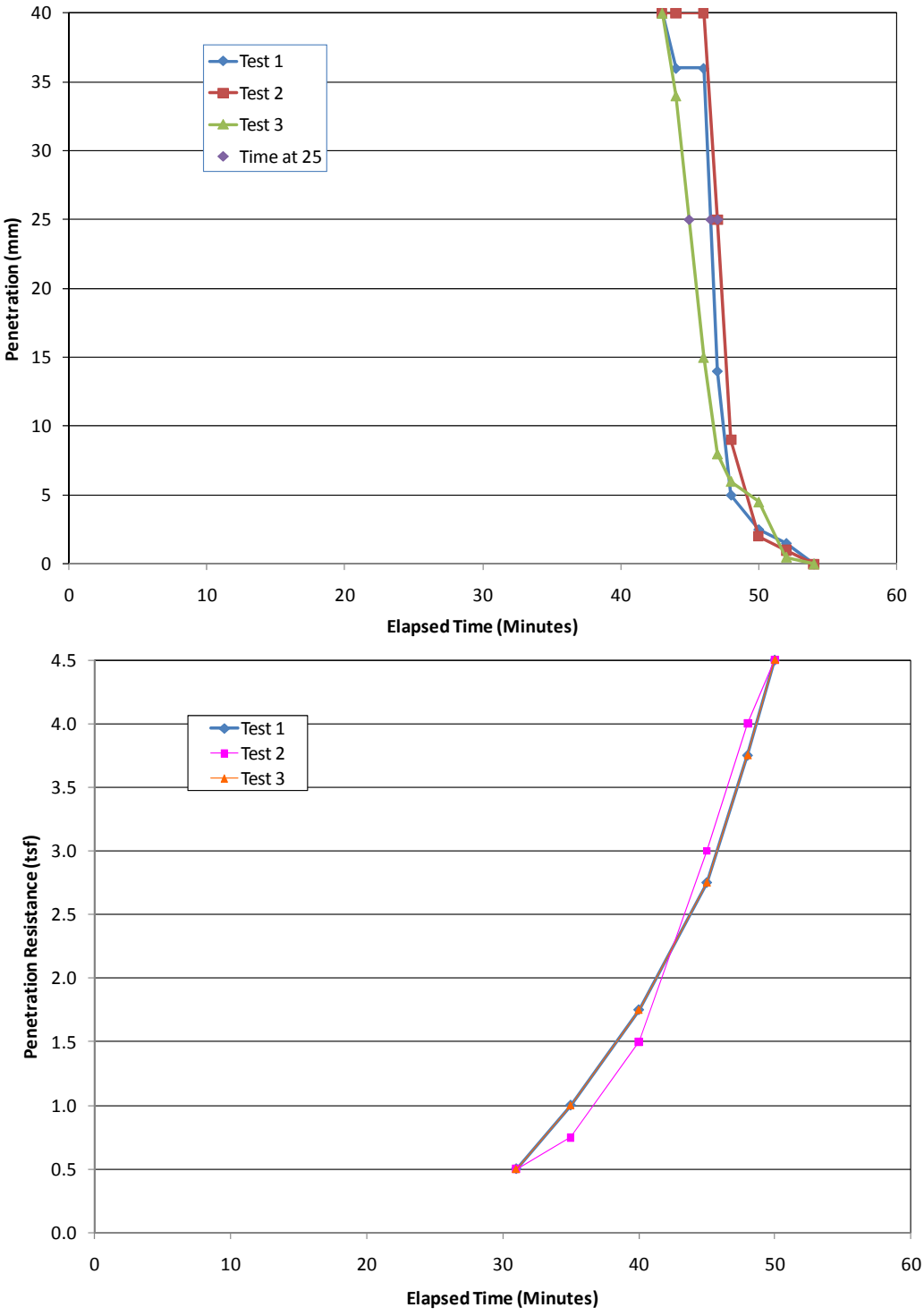



**Figure 107**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 3**

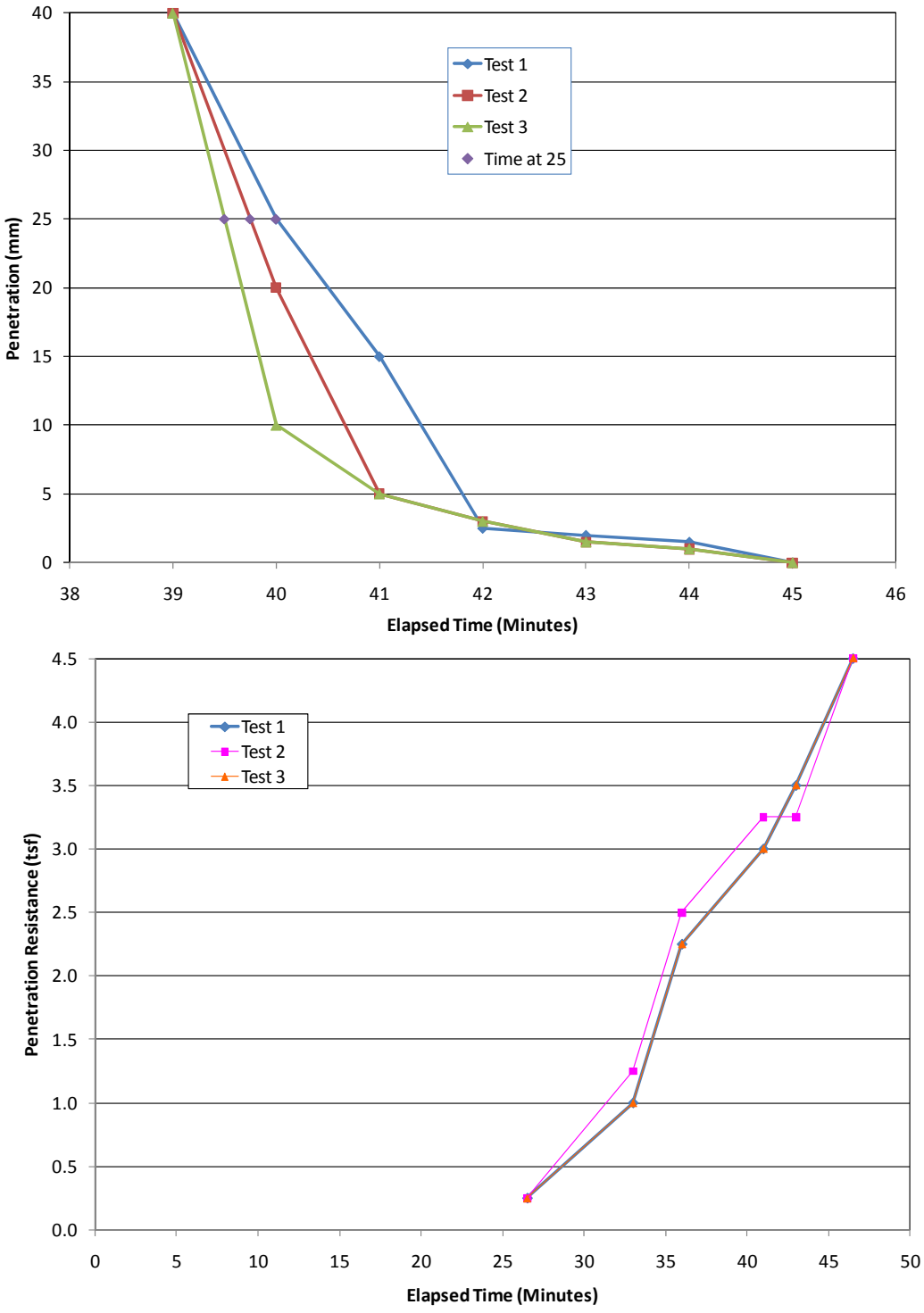



**Figure 108**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 4**

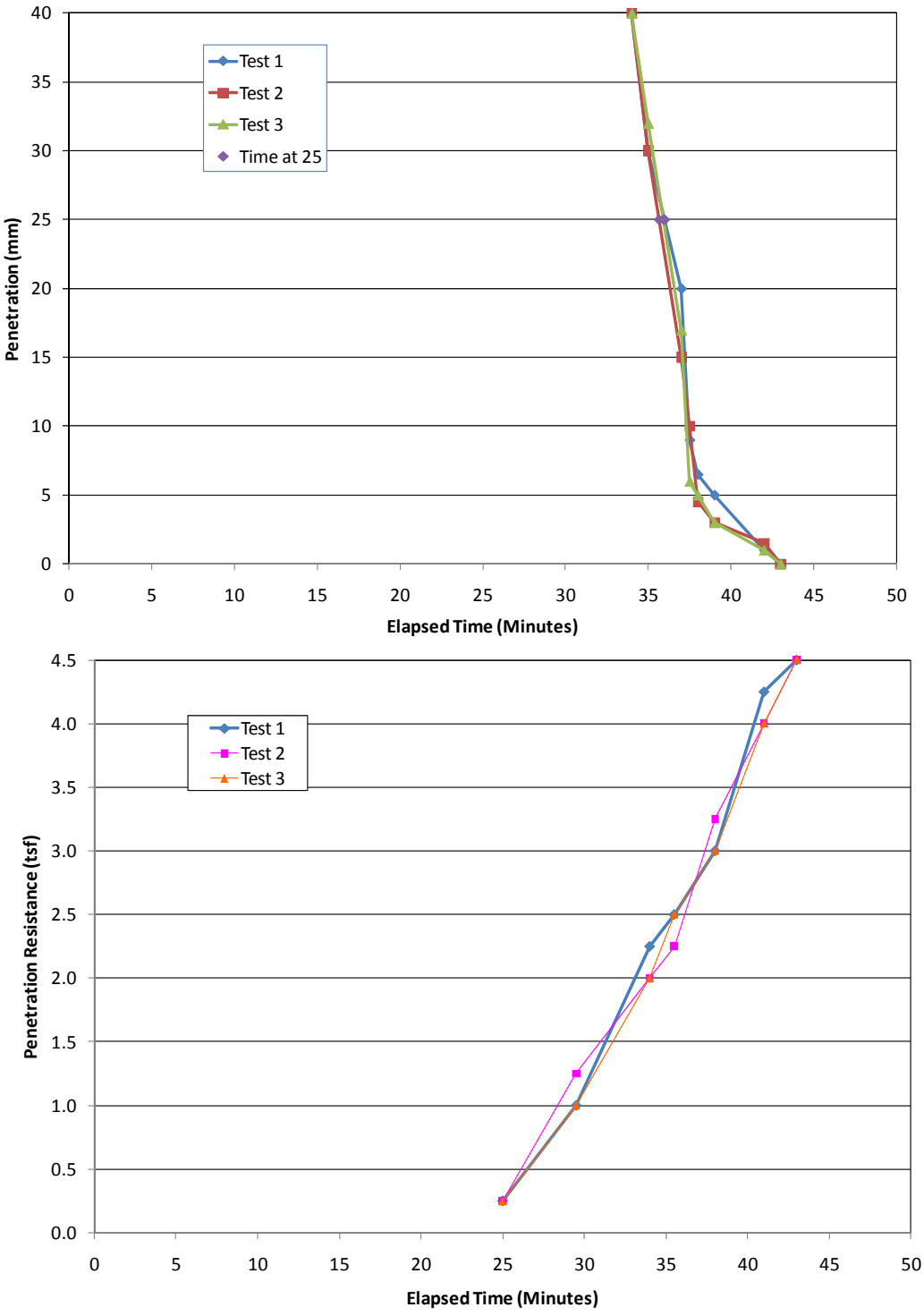



**Figure 109**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 5**

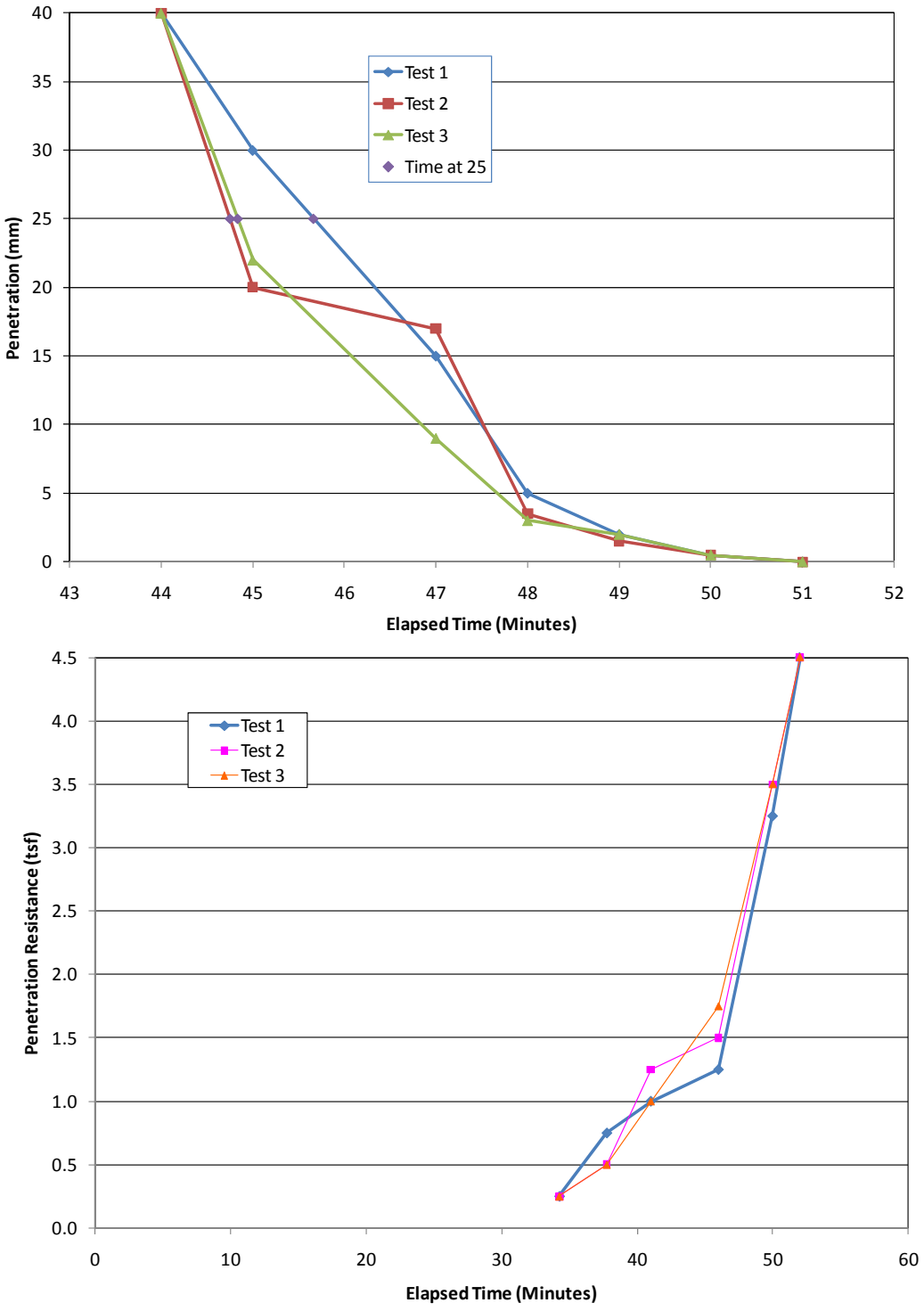



**Figure 110**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 6**

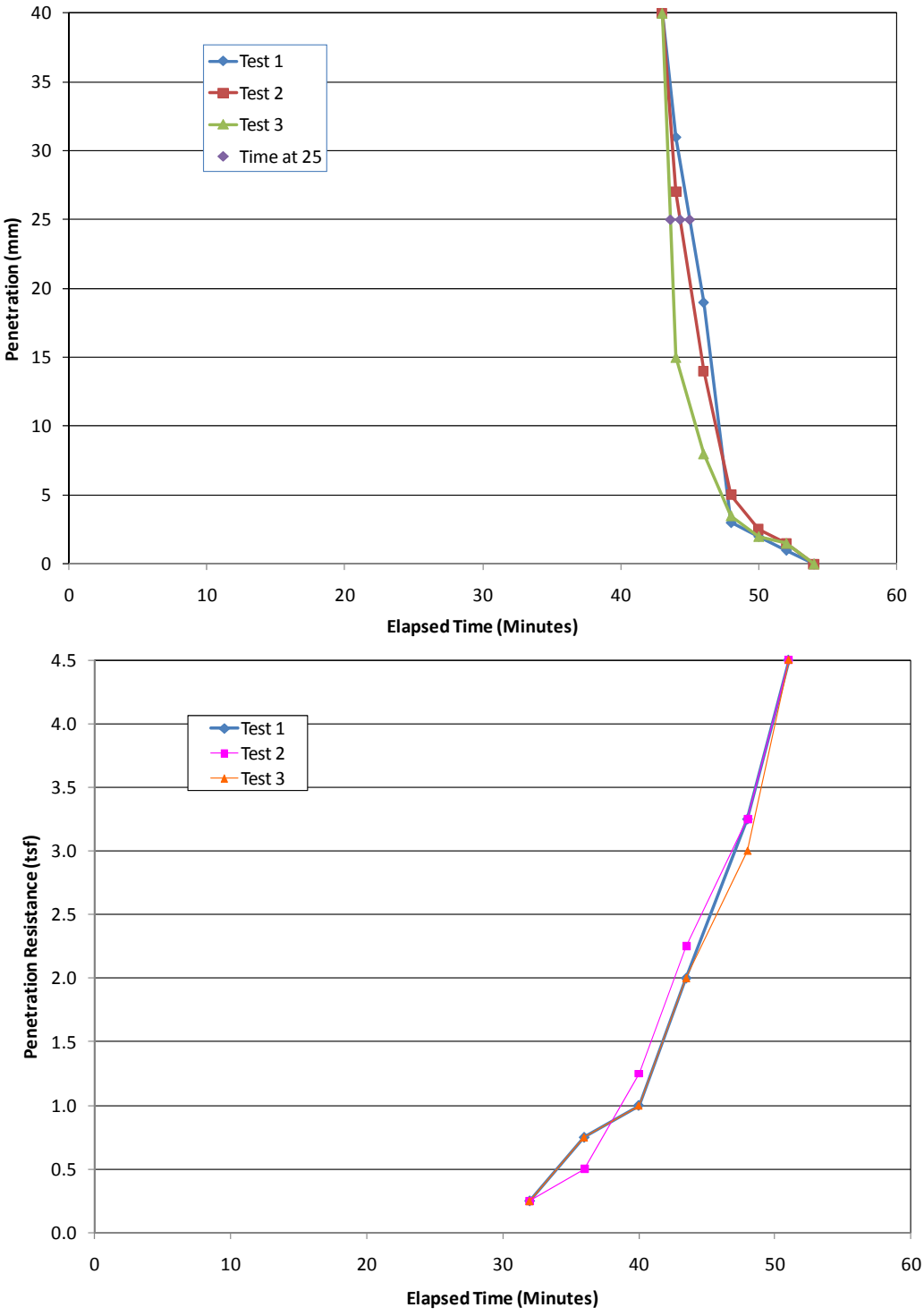



**Figure 111**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 7**

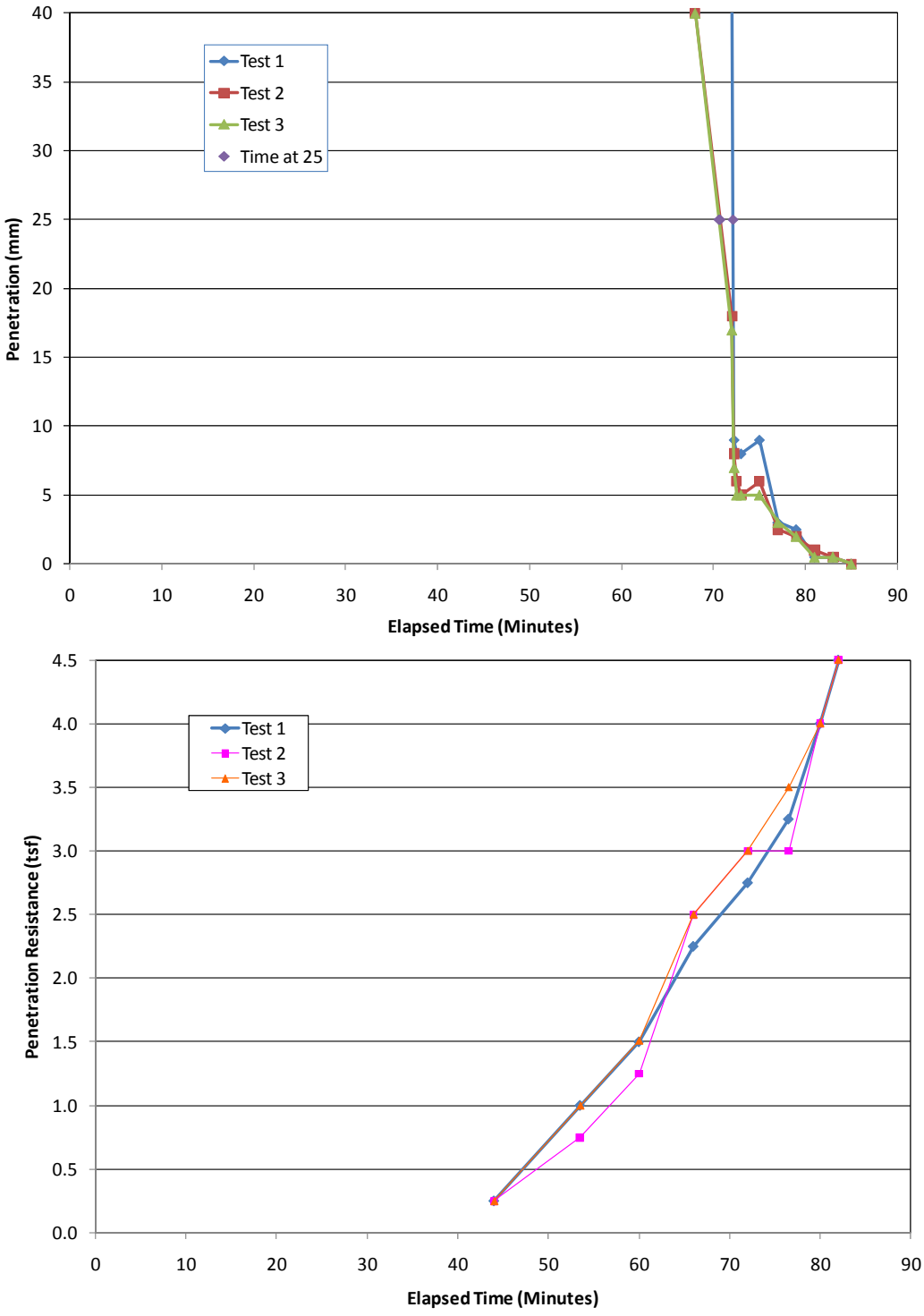



**Figure 112**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 8**

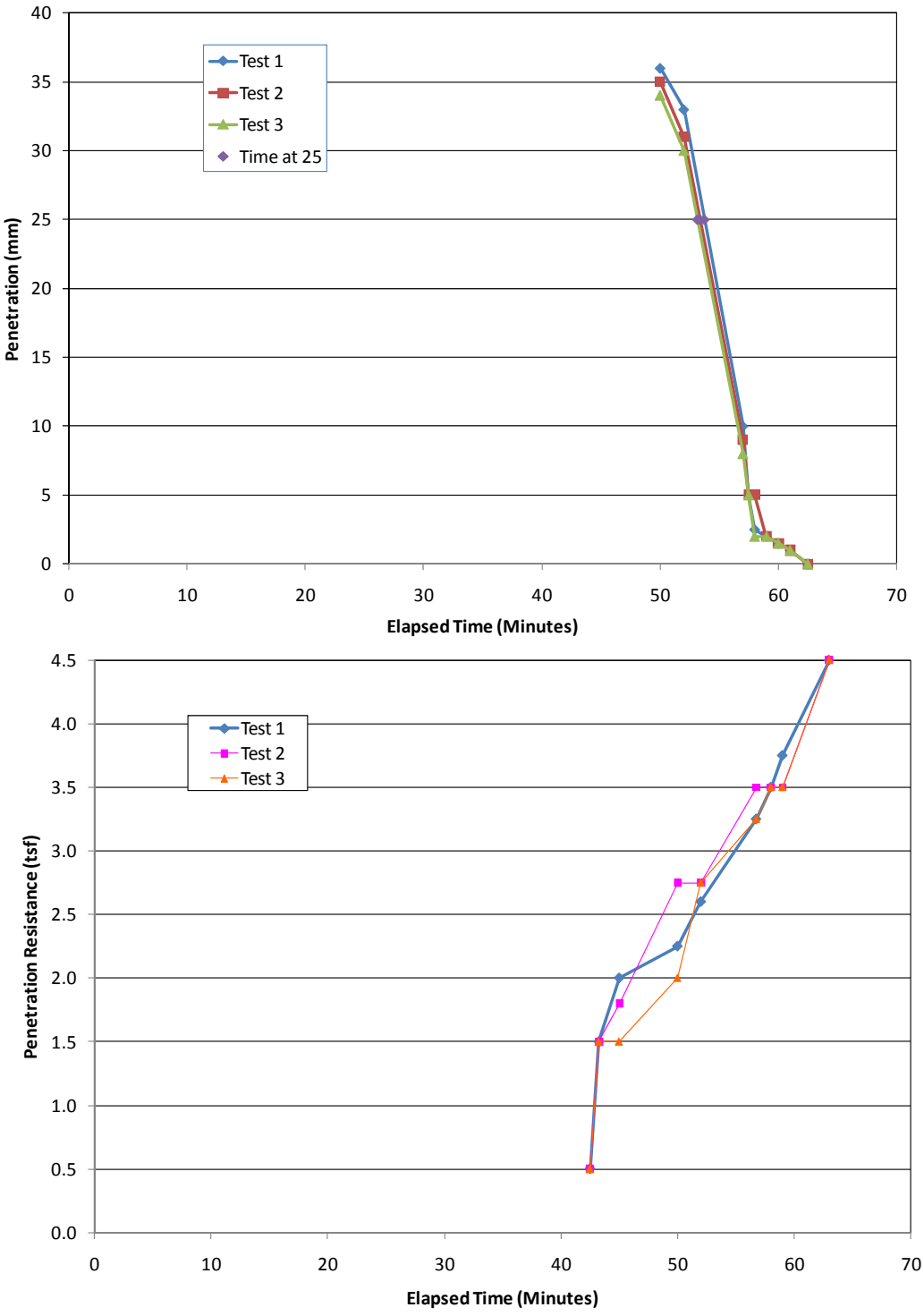



**Figure 113**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 9**

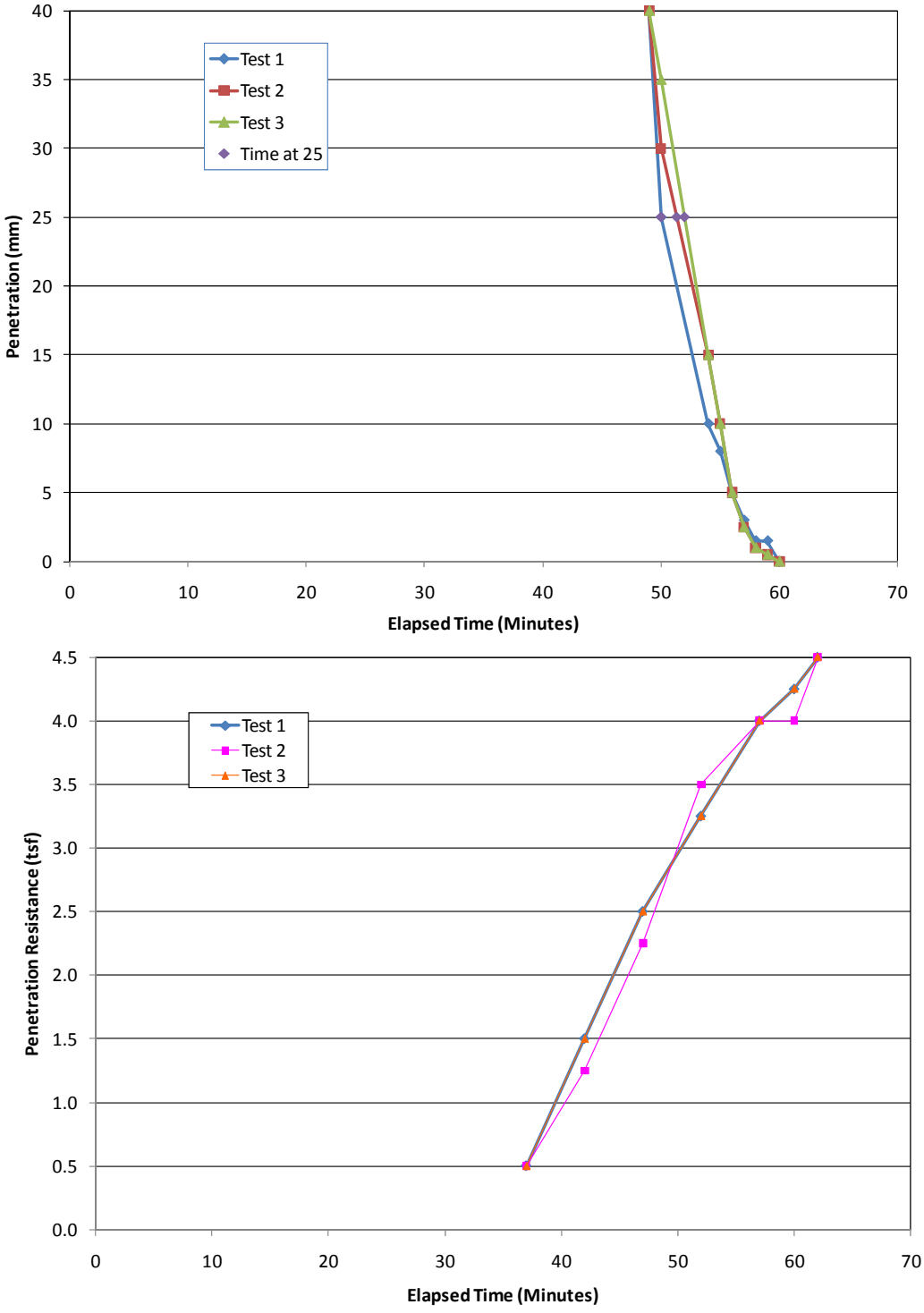
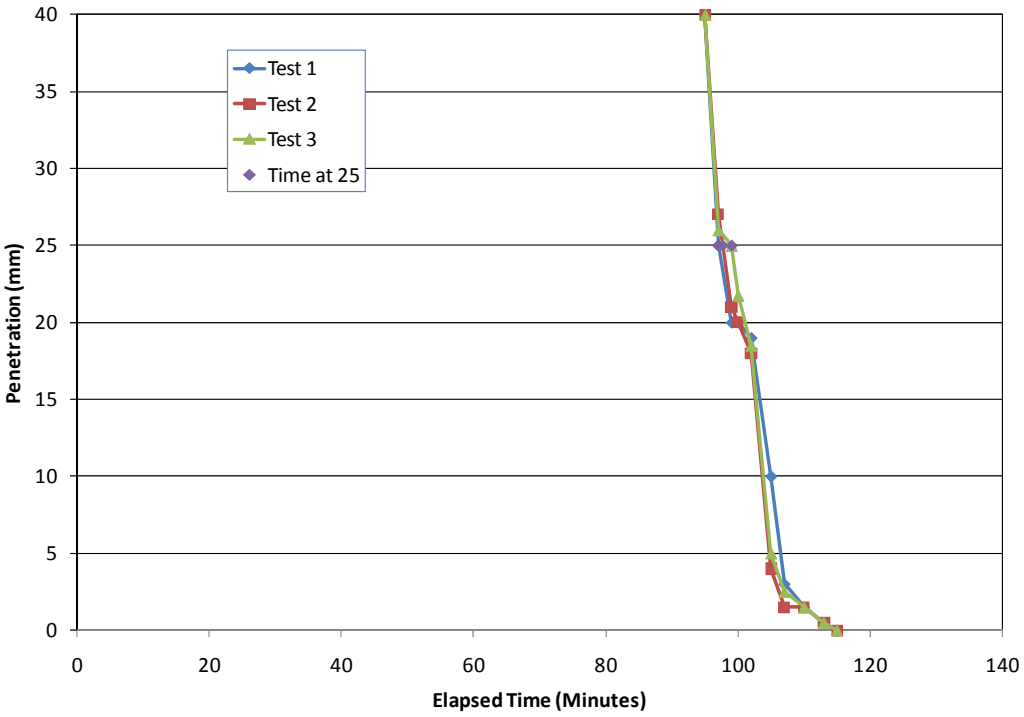



**Figure 114**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 10**



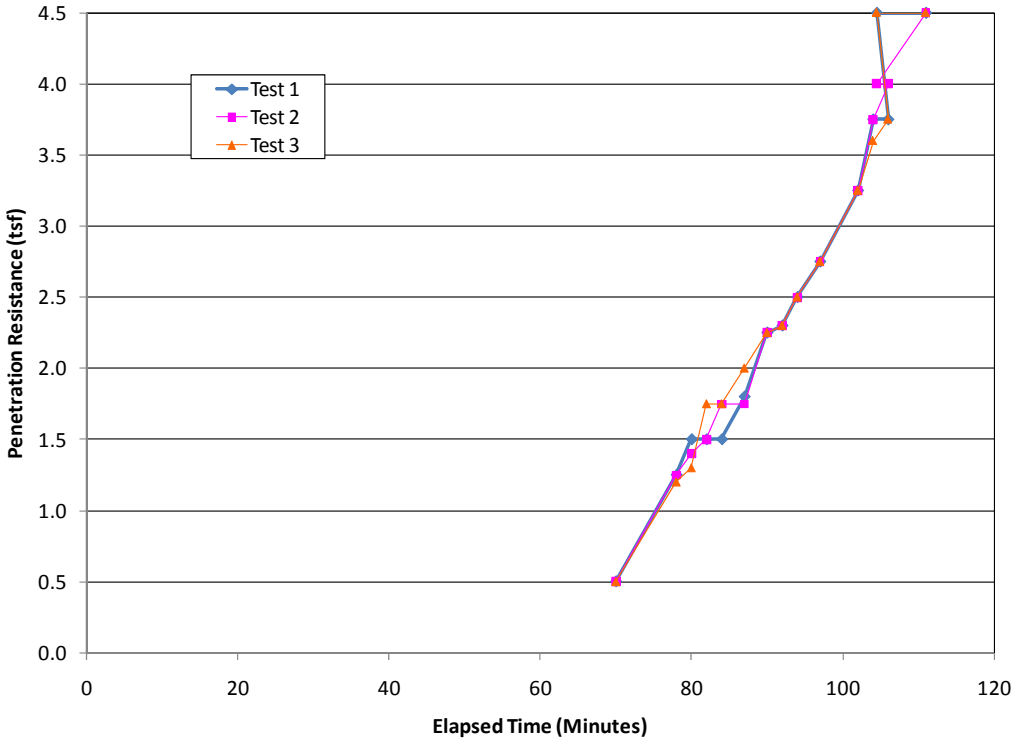




**Figure 115**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 11**

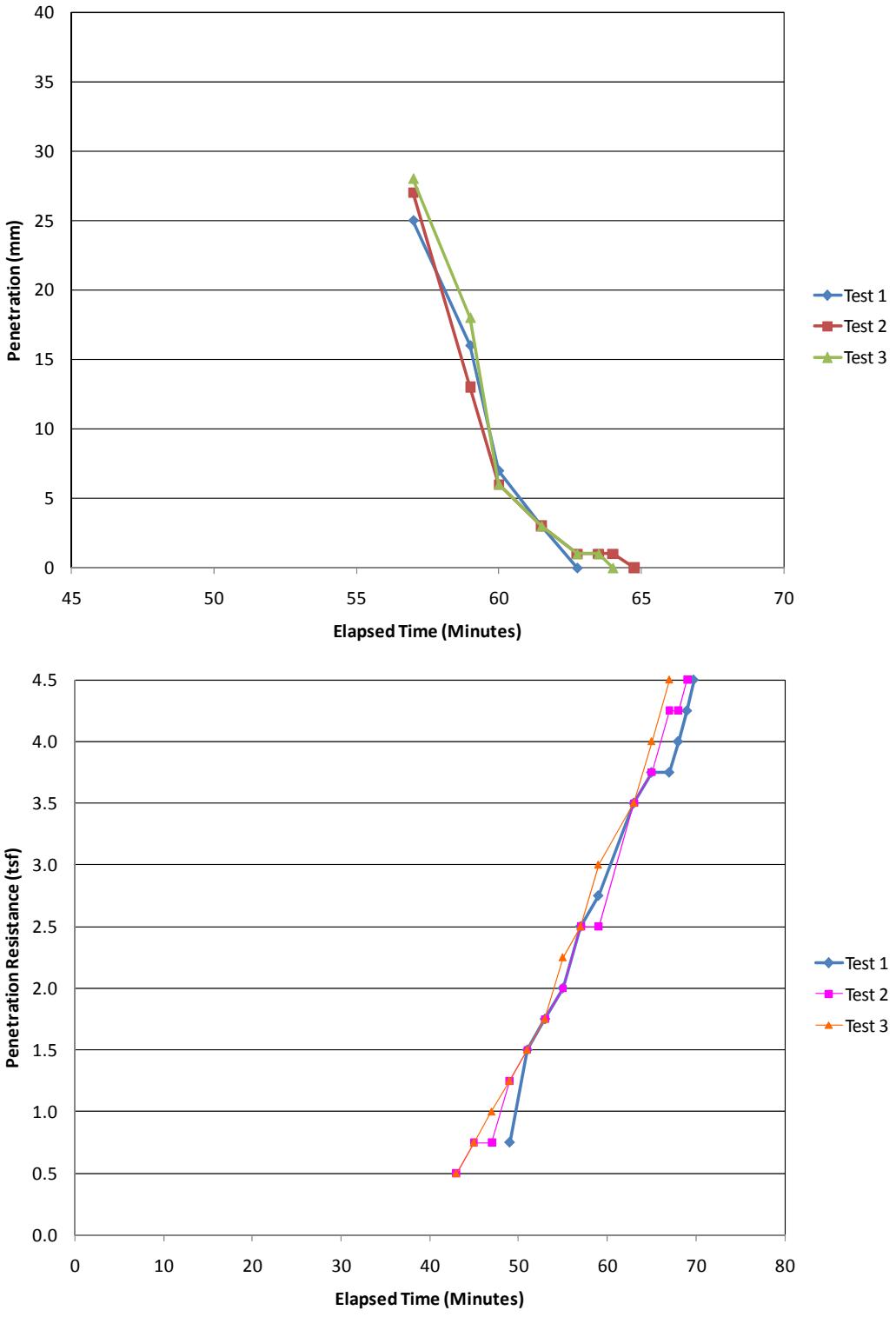
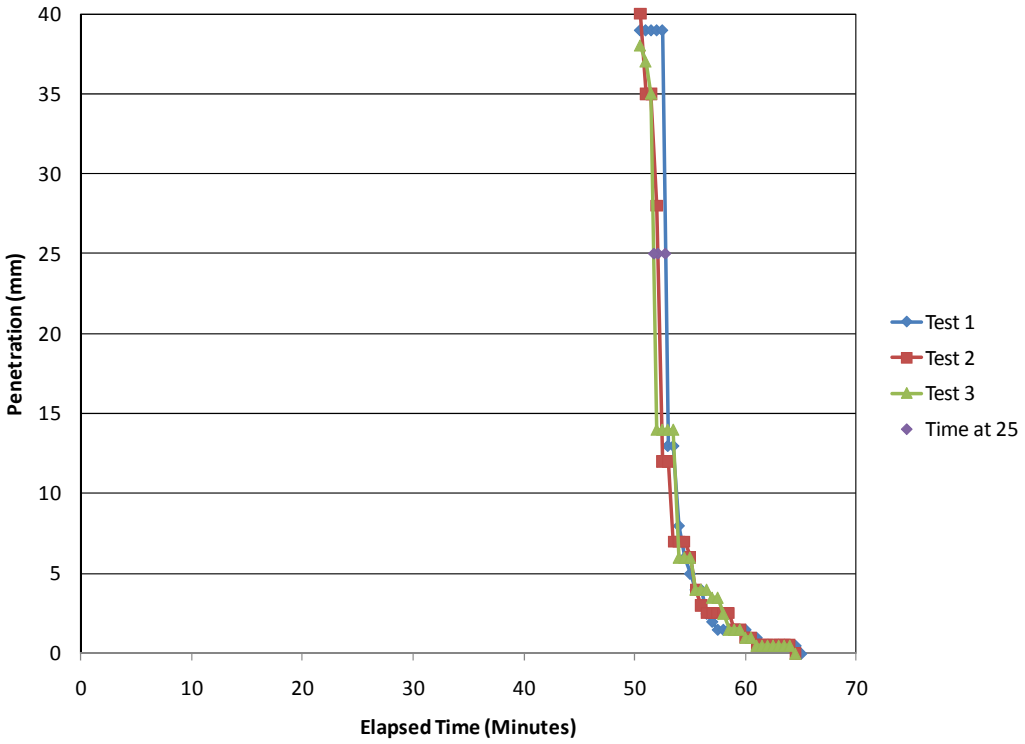



**Figure 116**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 12**



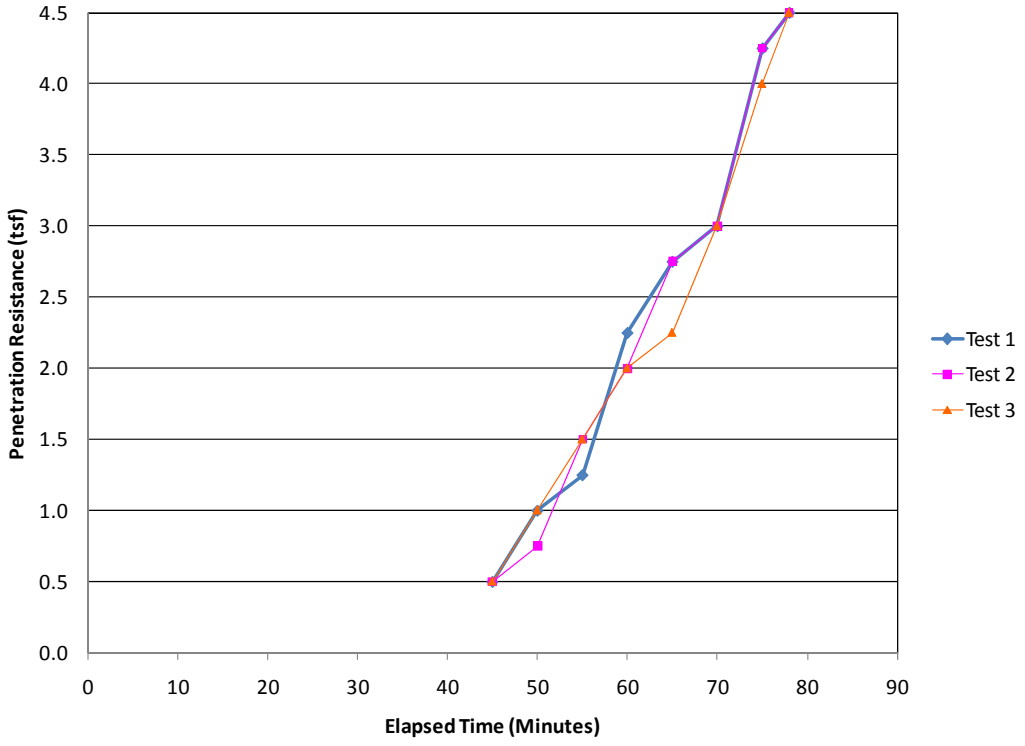
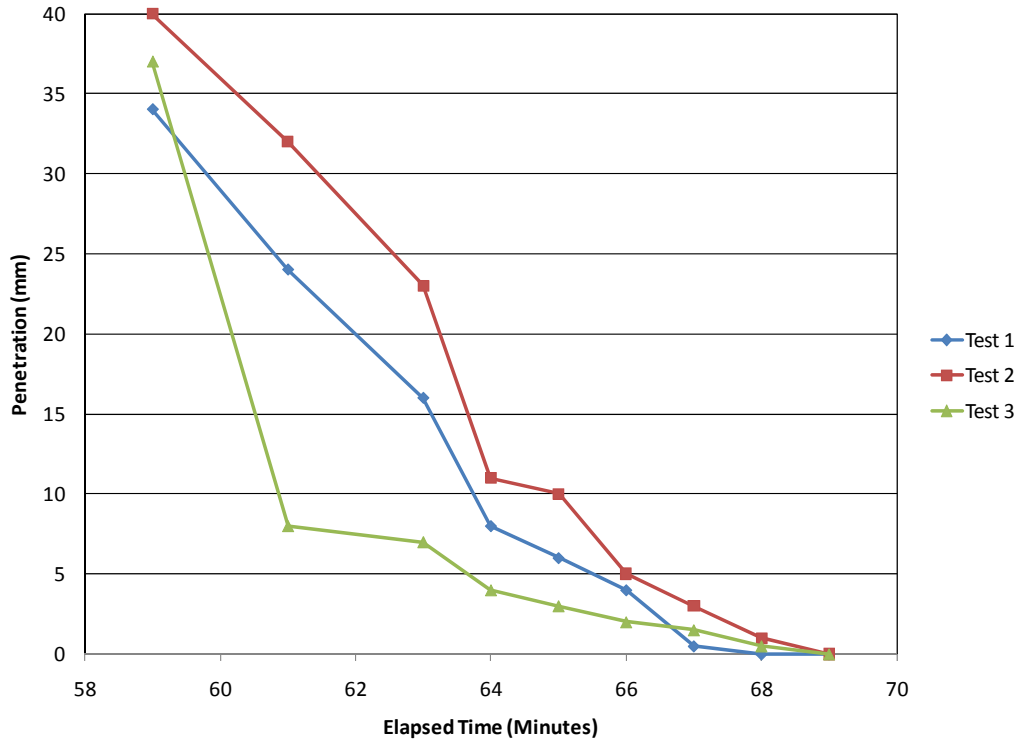




**Figure 117**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 13**



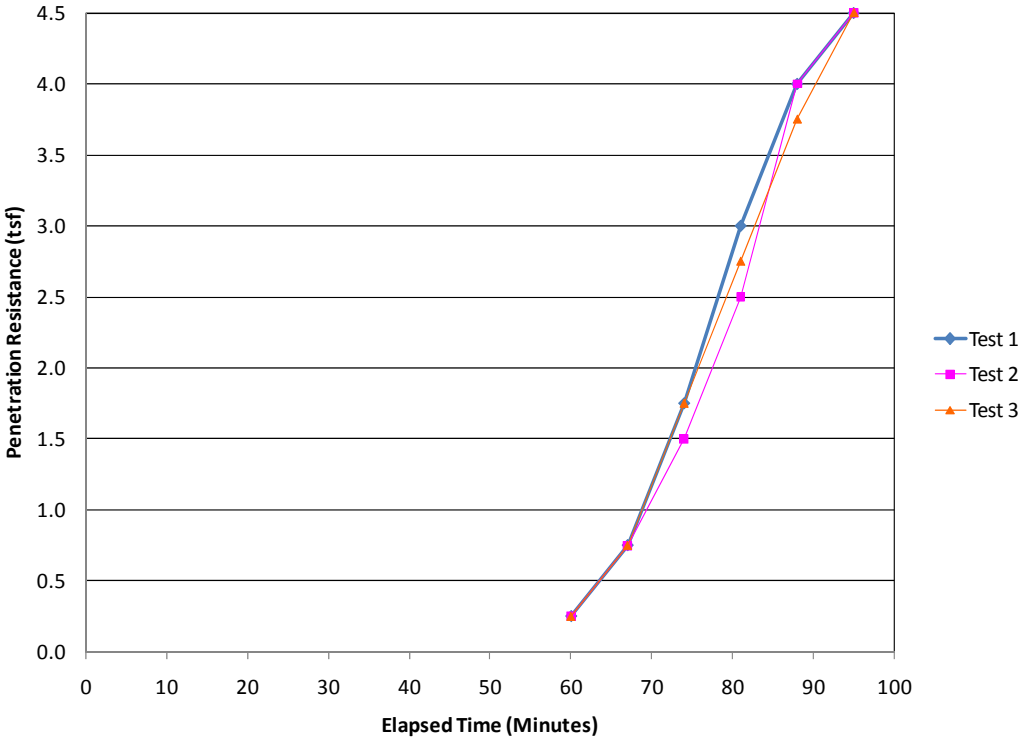




**Figure 118**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 14**

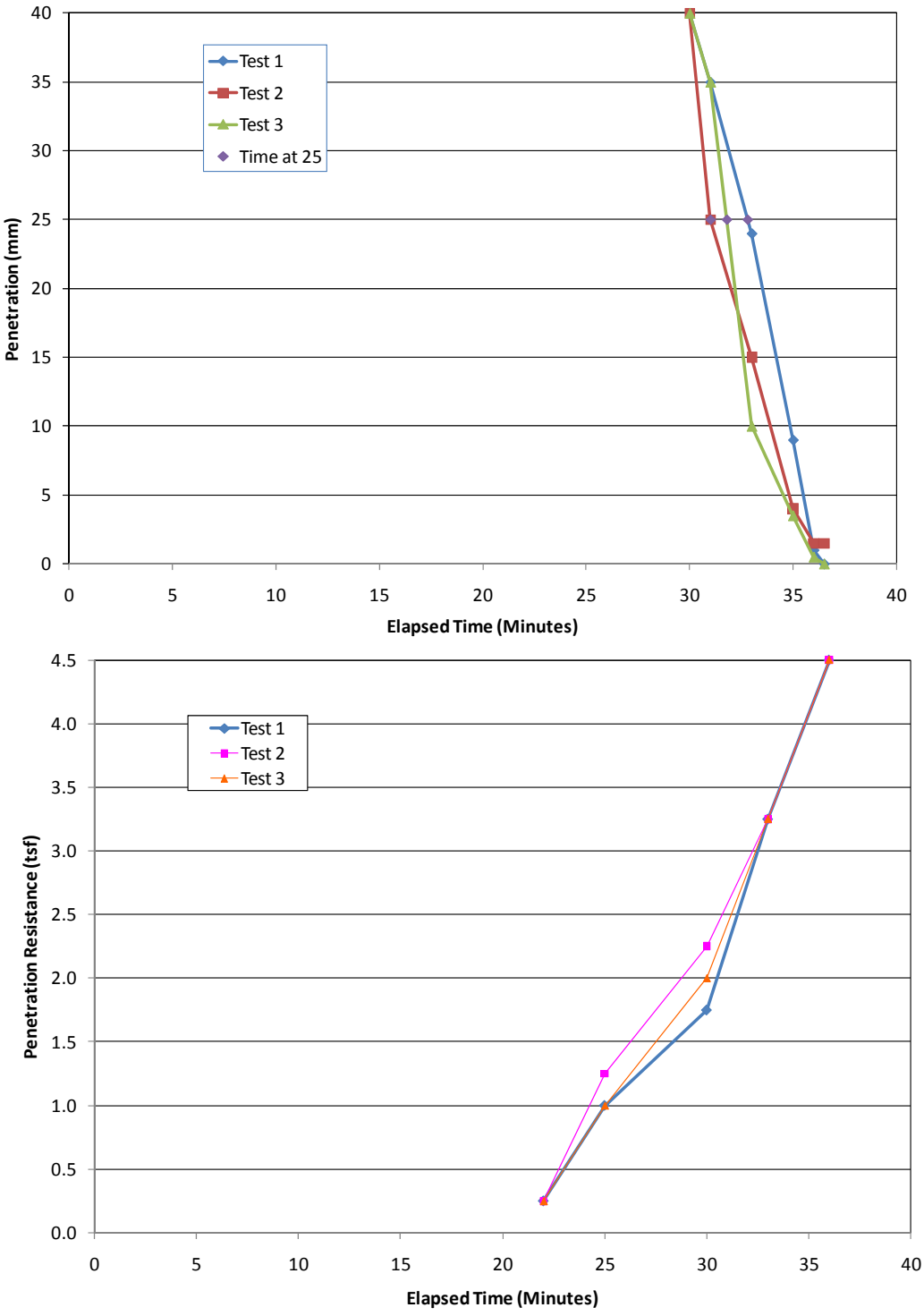



**Figure 119**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 15**

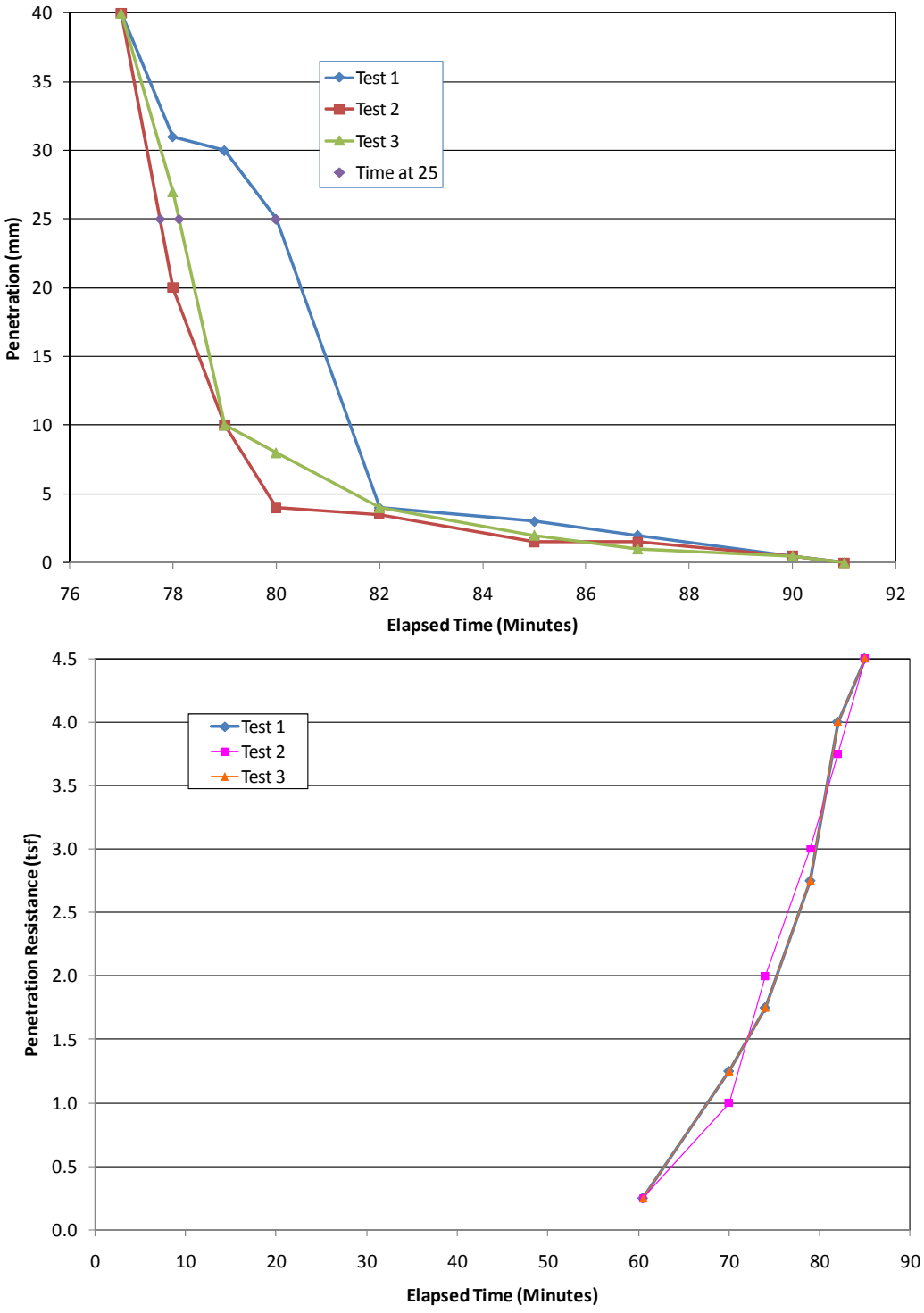



**Figure 120**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 16**

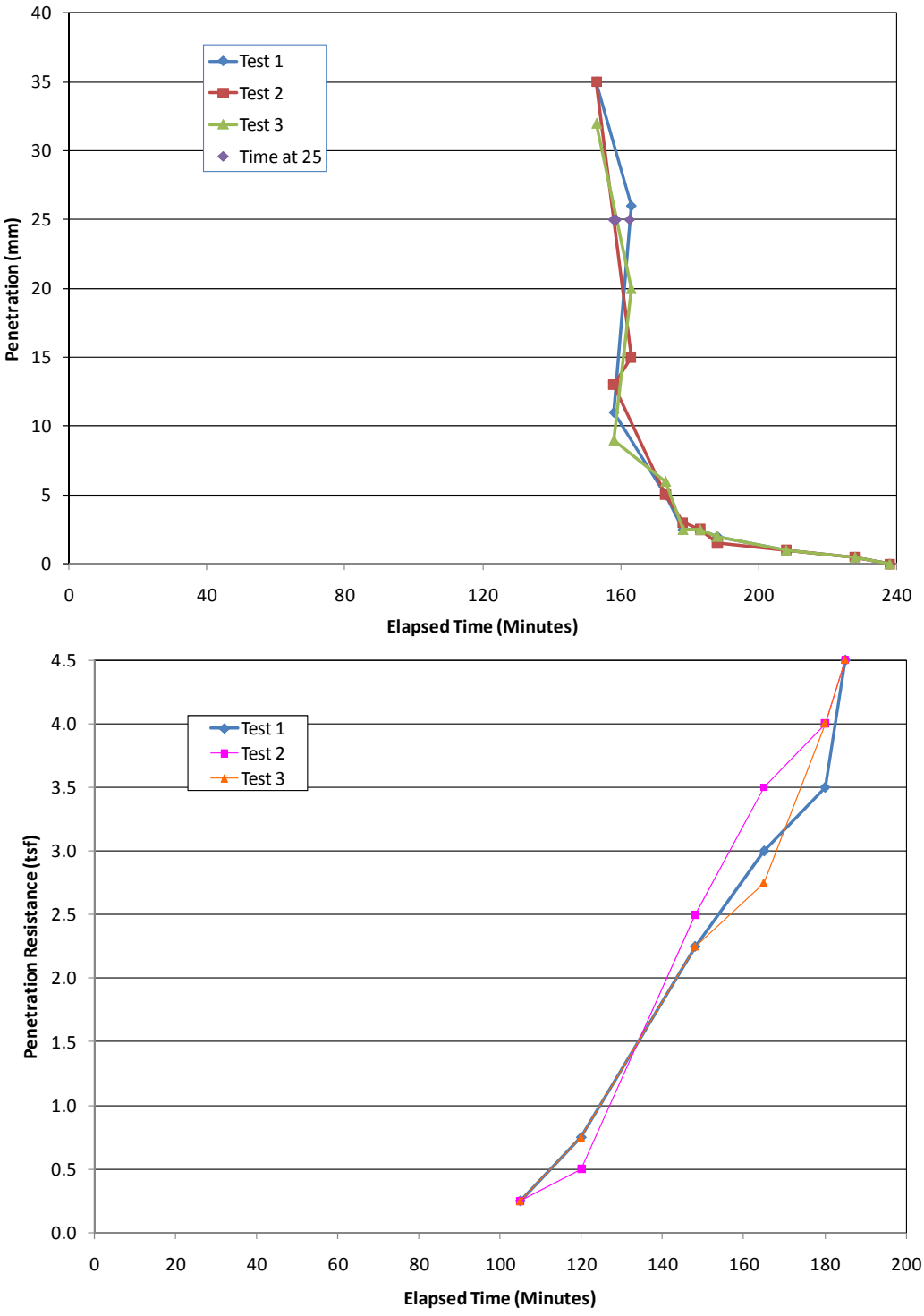
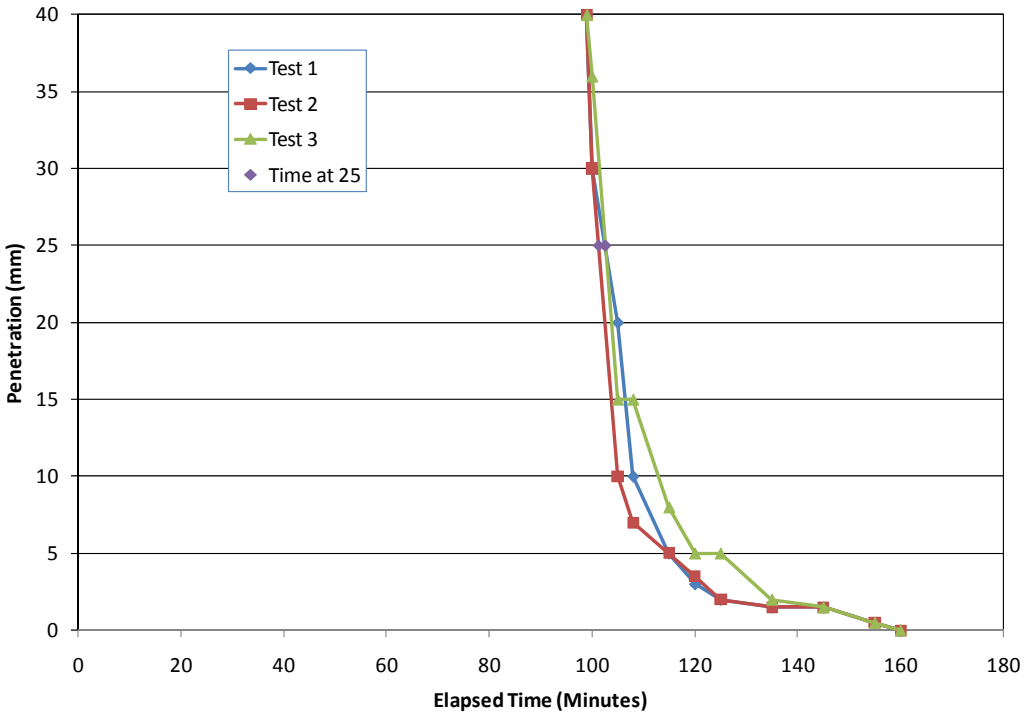



**Figure 121**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 17**



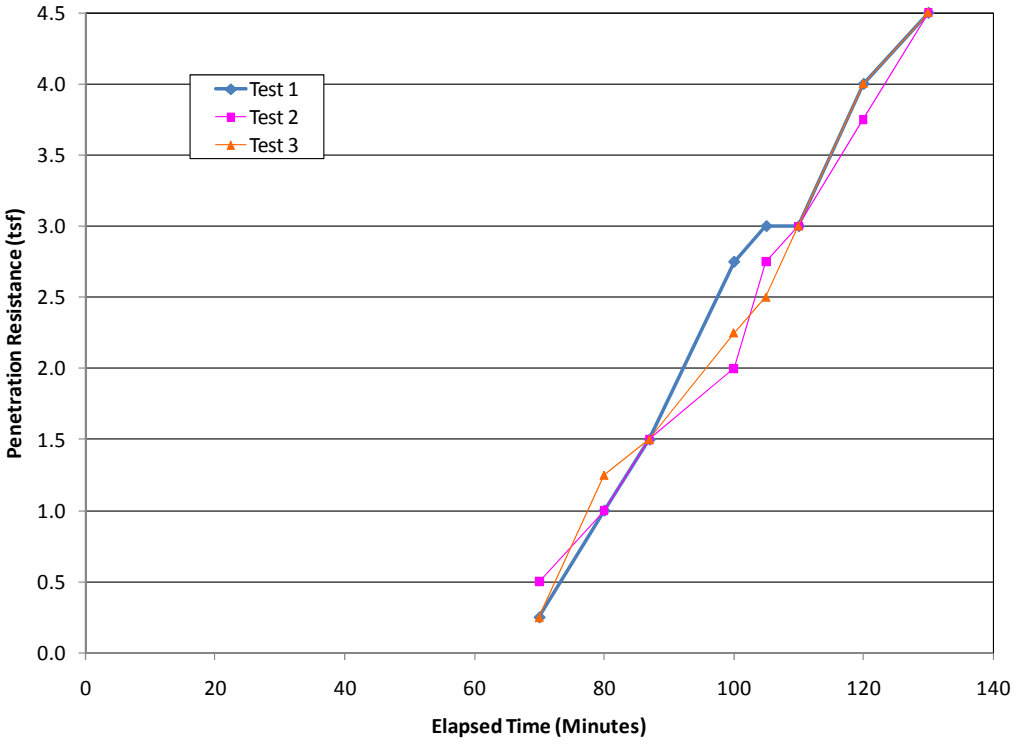




**Figure 122**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 18**

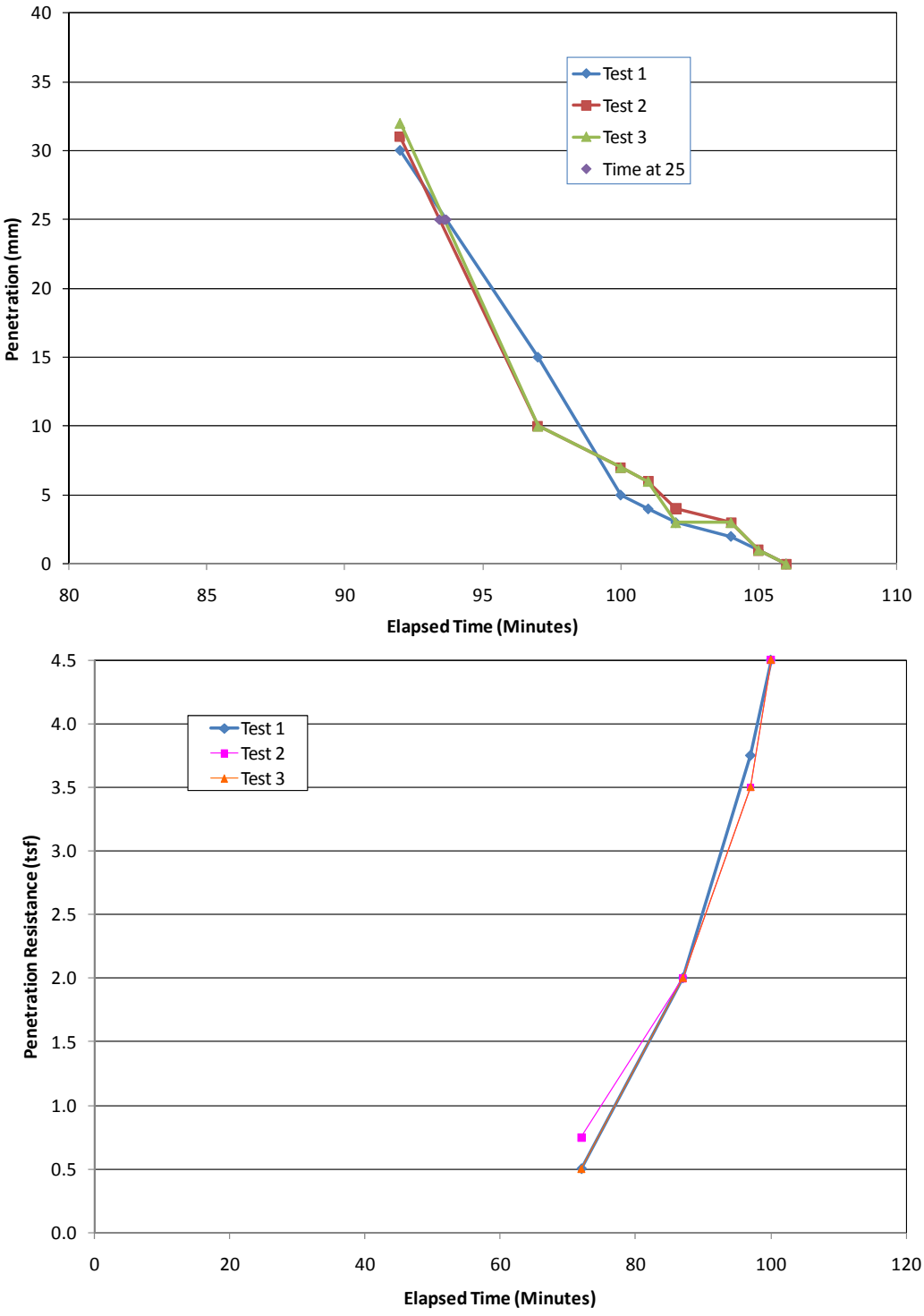



**Figure 123**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 5 Bucket 20**

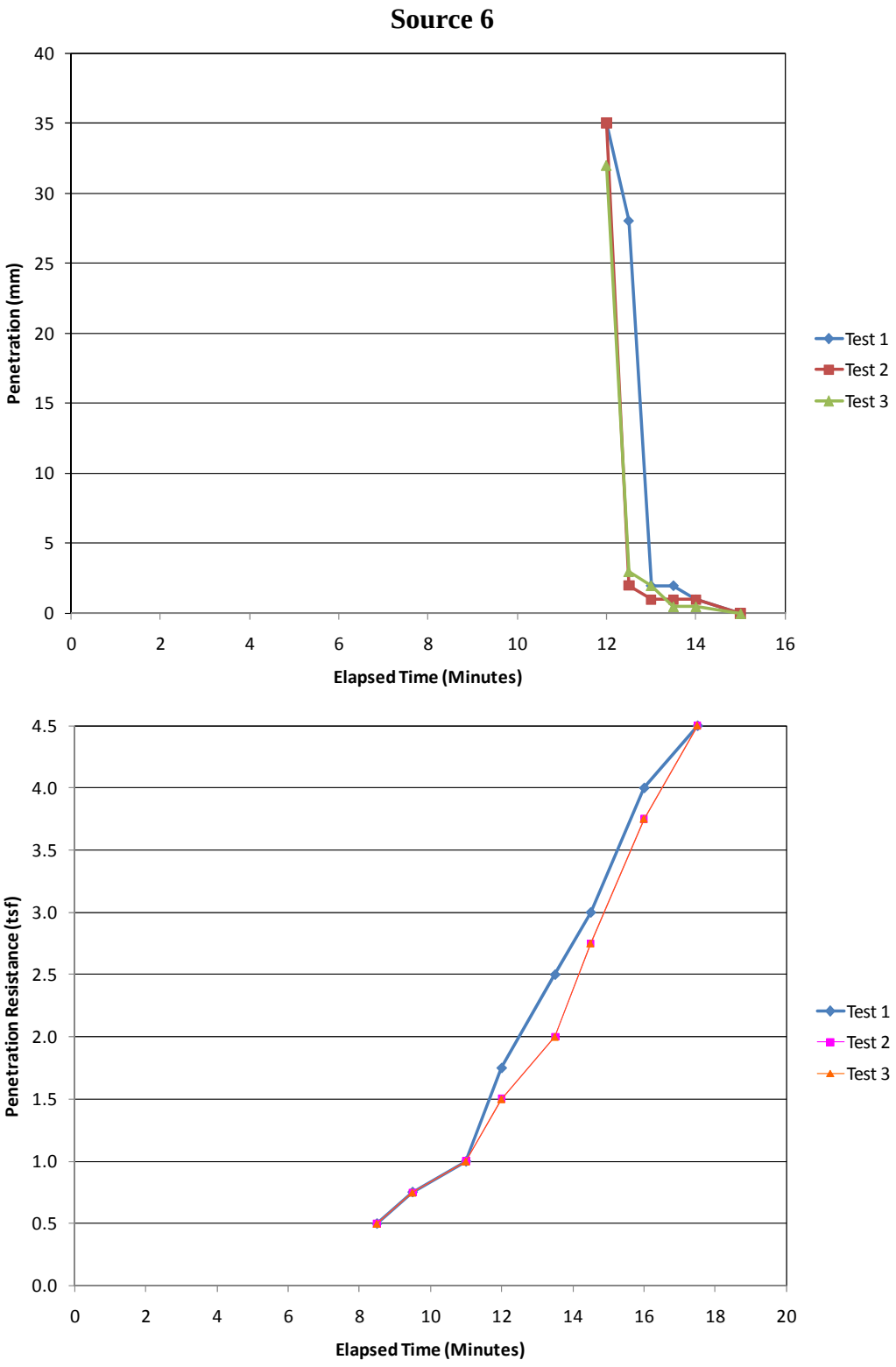
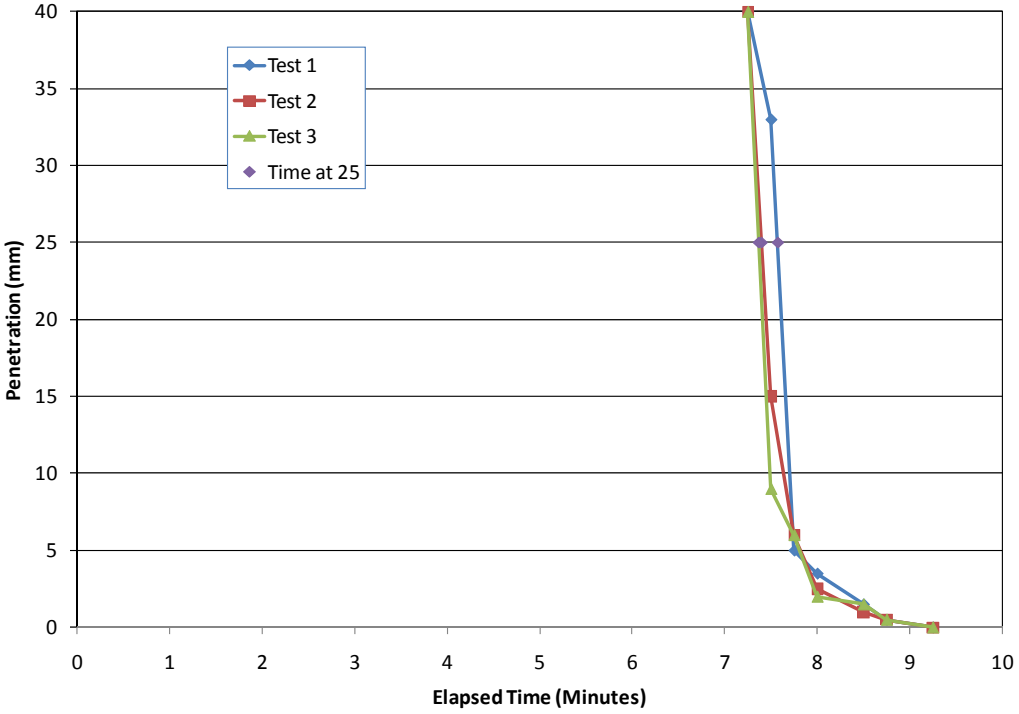



**Figure 124**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 1**



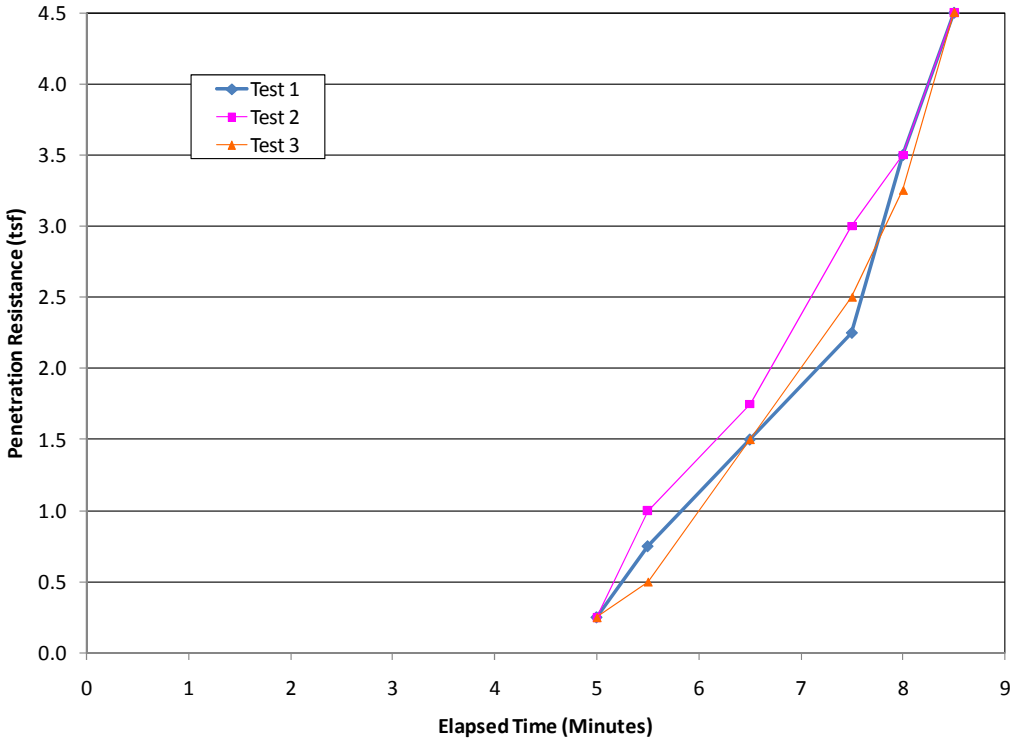




**Figure 125**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 2**

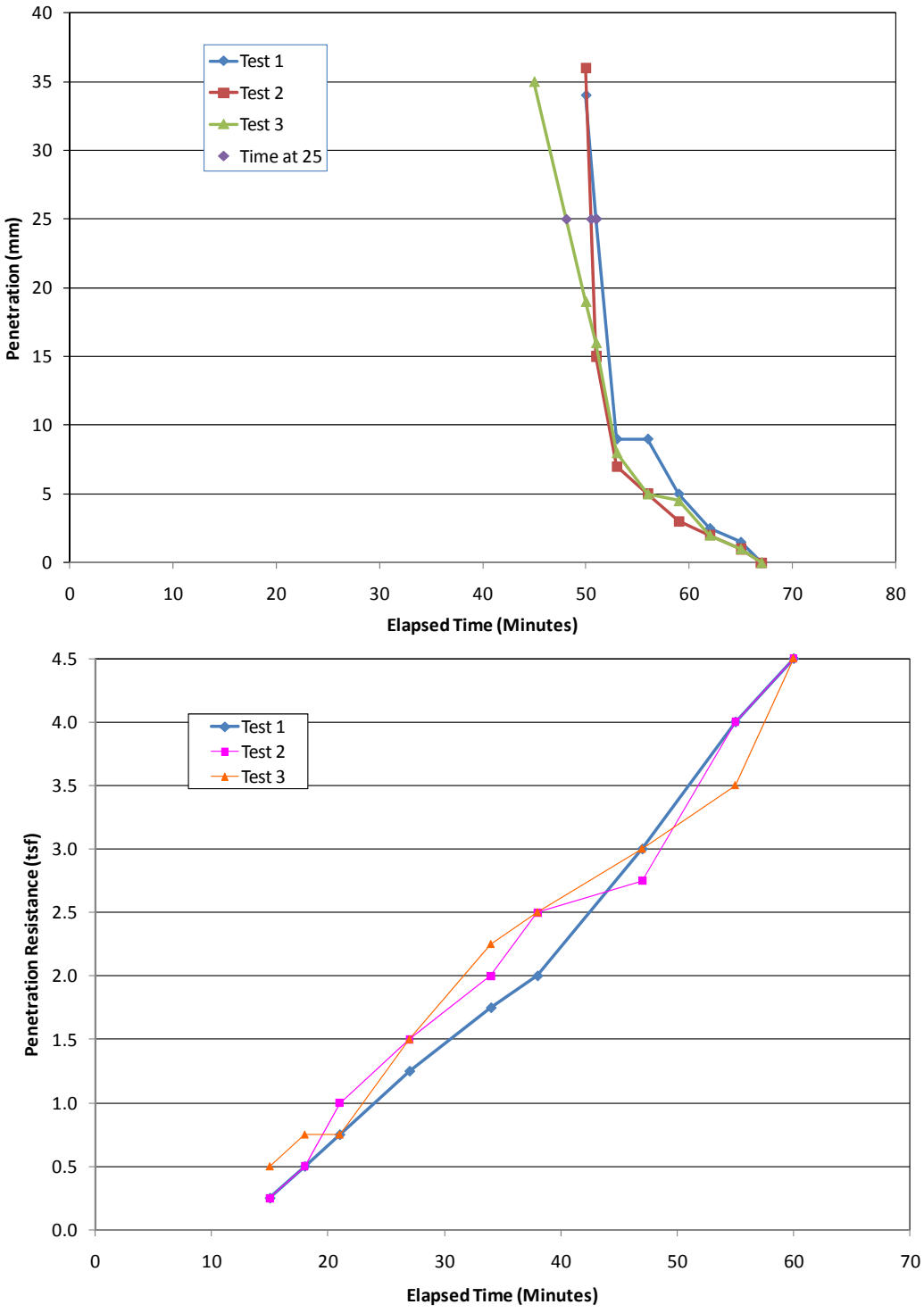
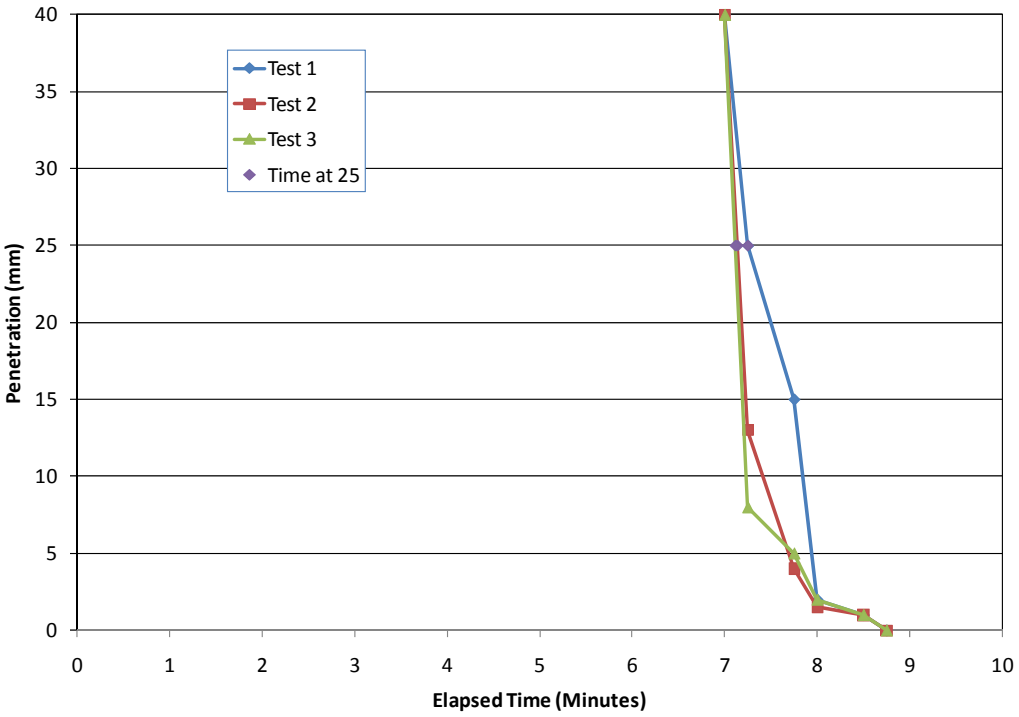



**Figure 126**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 3**



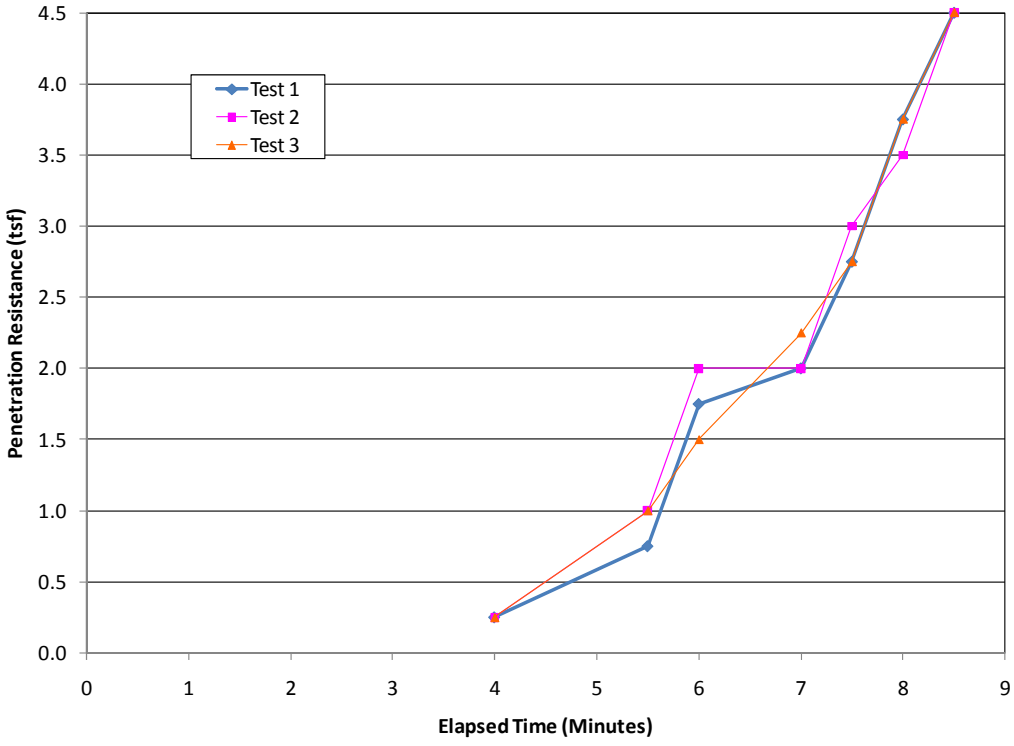




**Figure 127**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 4**

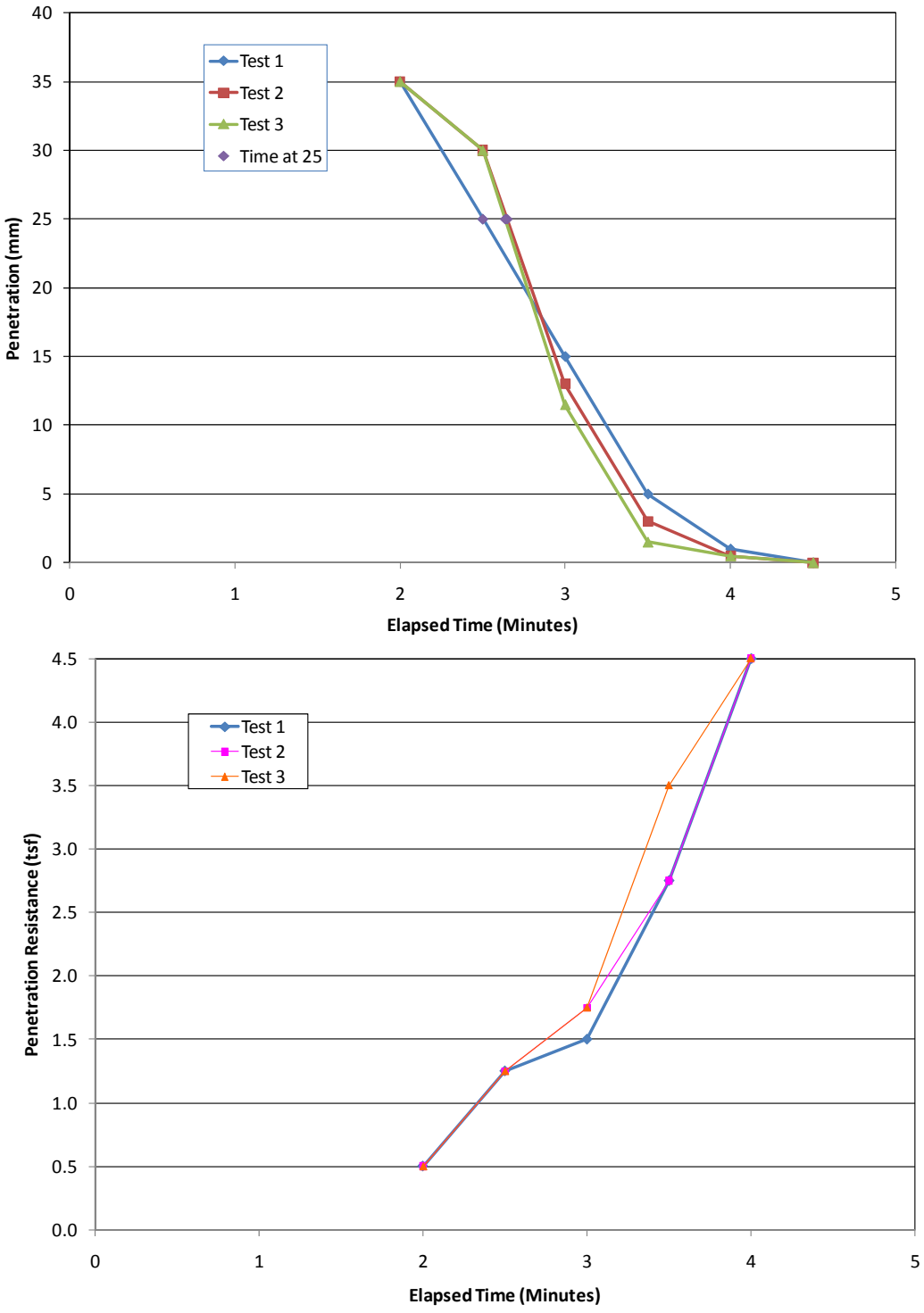



**Figure 128**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 5**

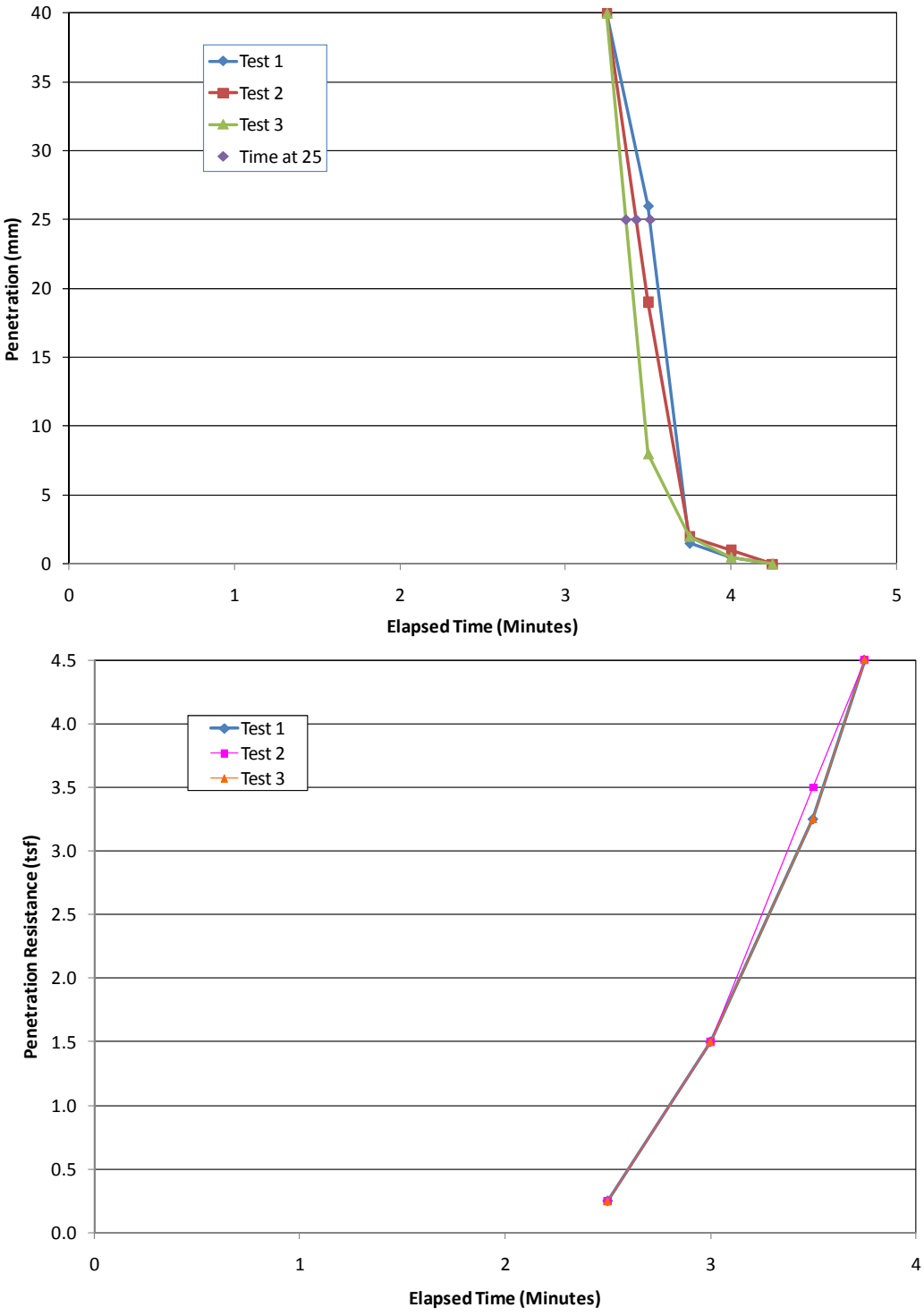



**Figure 129**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 6**

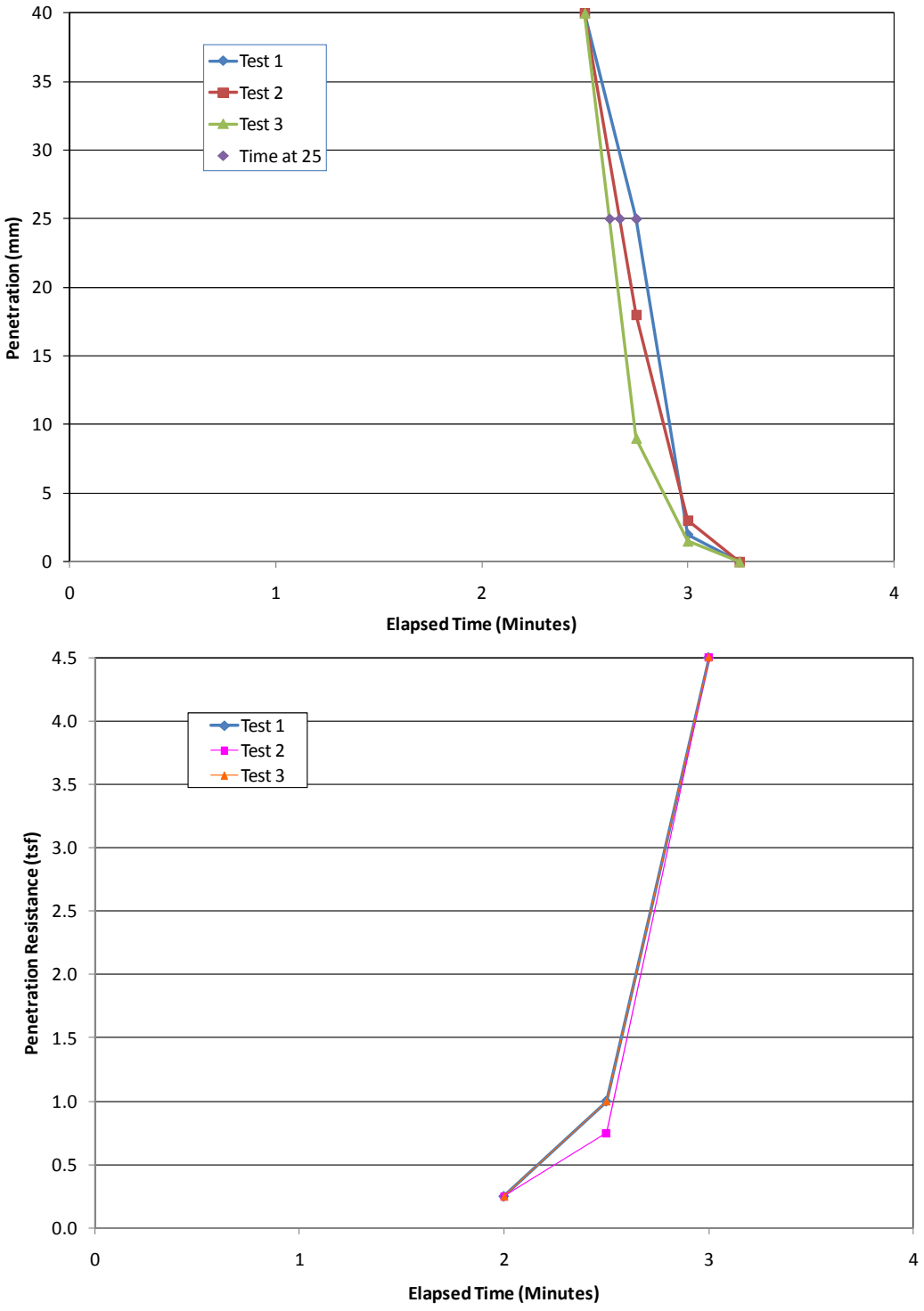



**Figure 130**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 7**

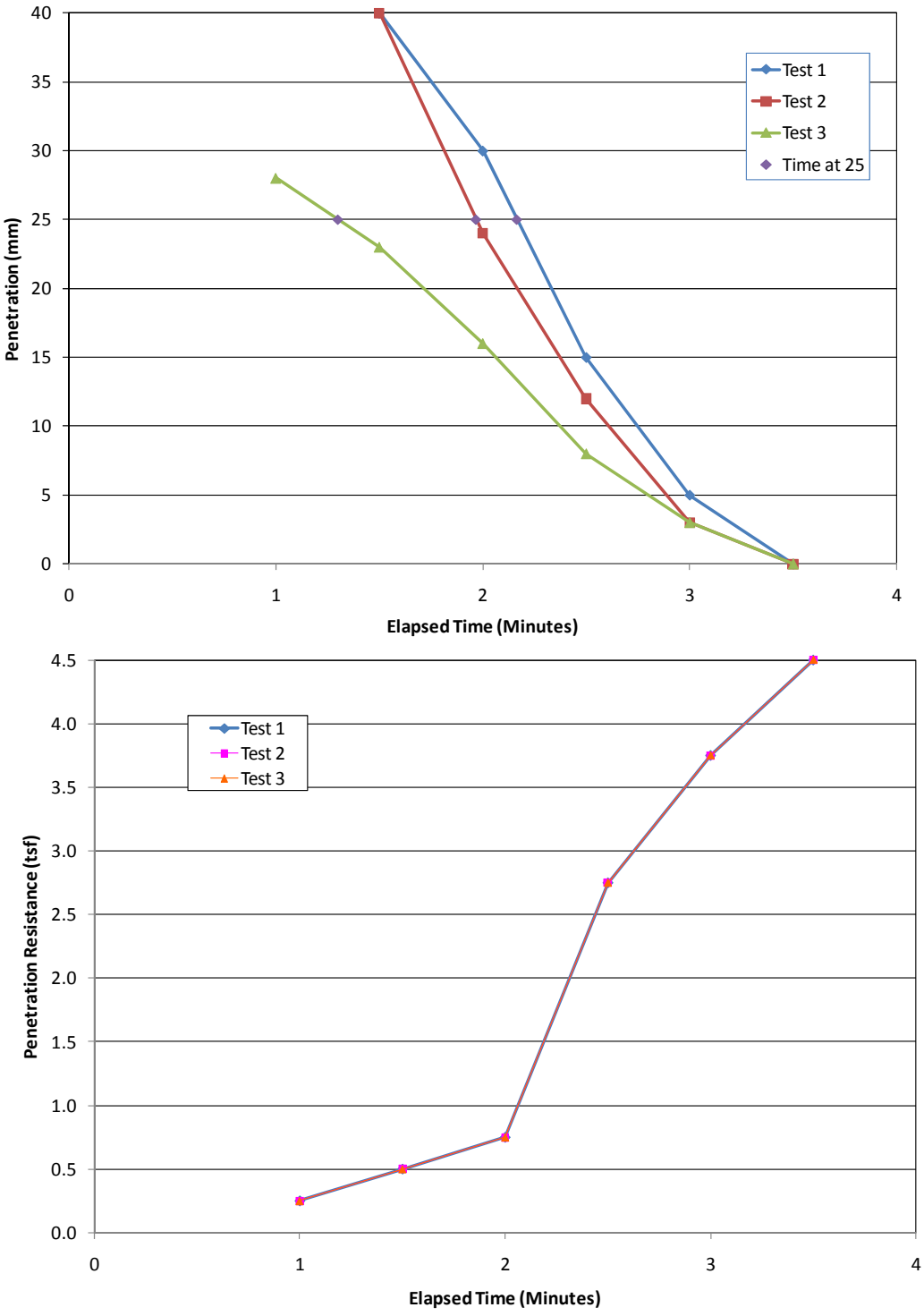



**Figure 131**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 8**

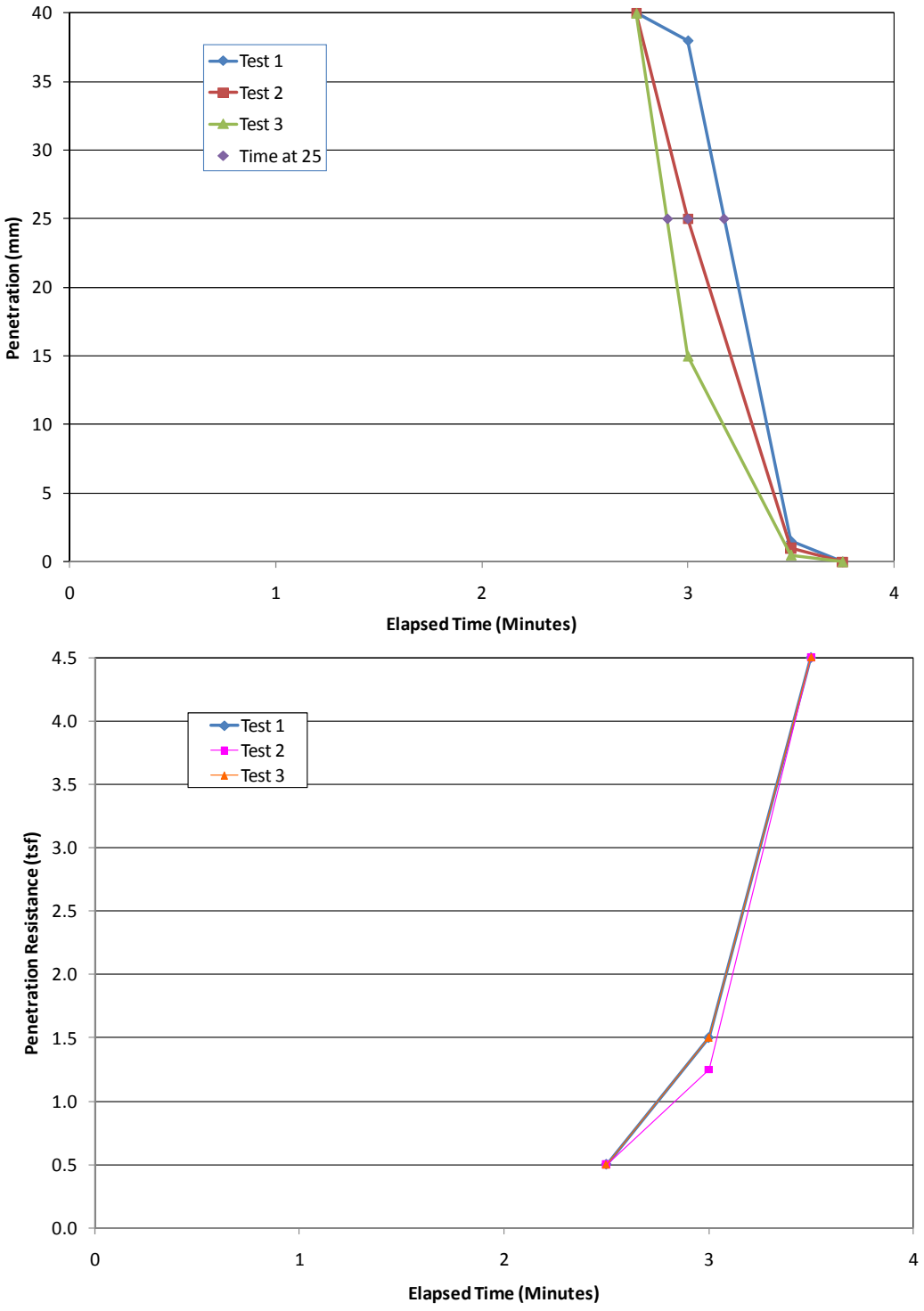



**Figure 132**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 9**

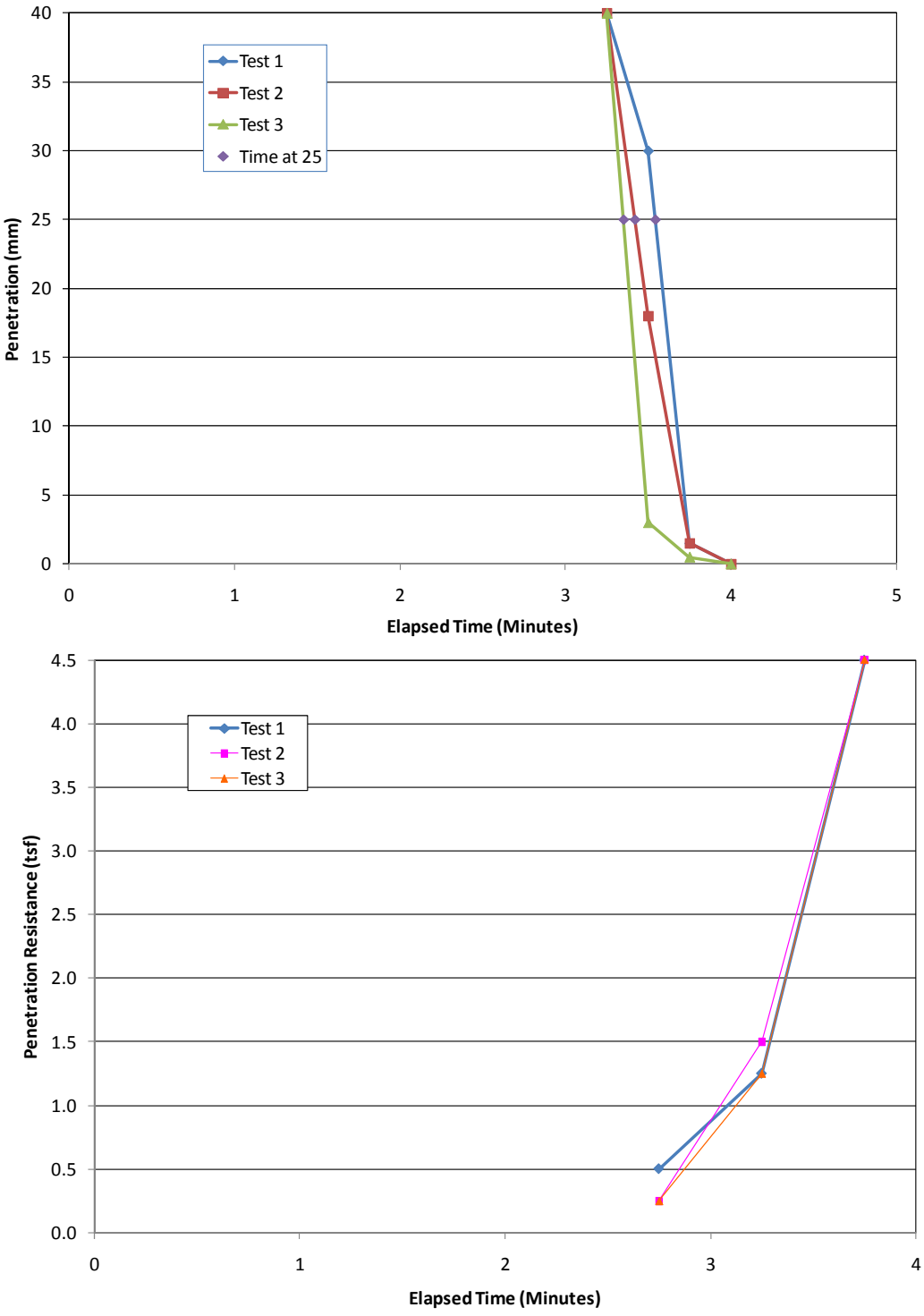



**Figure 133**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 10**

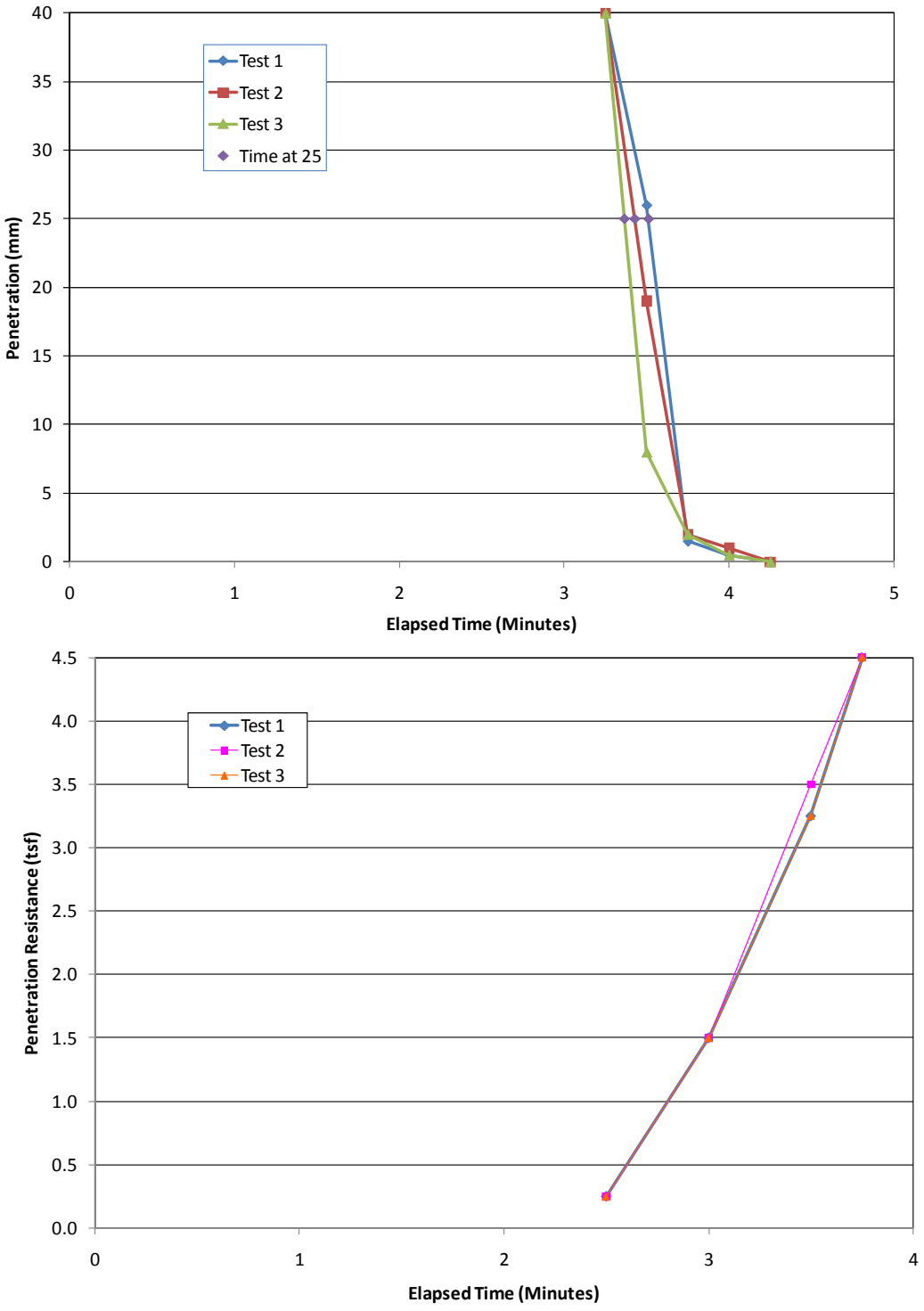



**Figure 134**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 11**

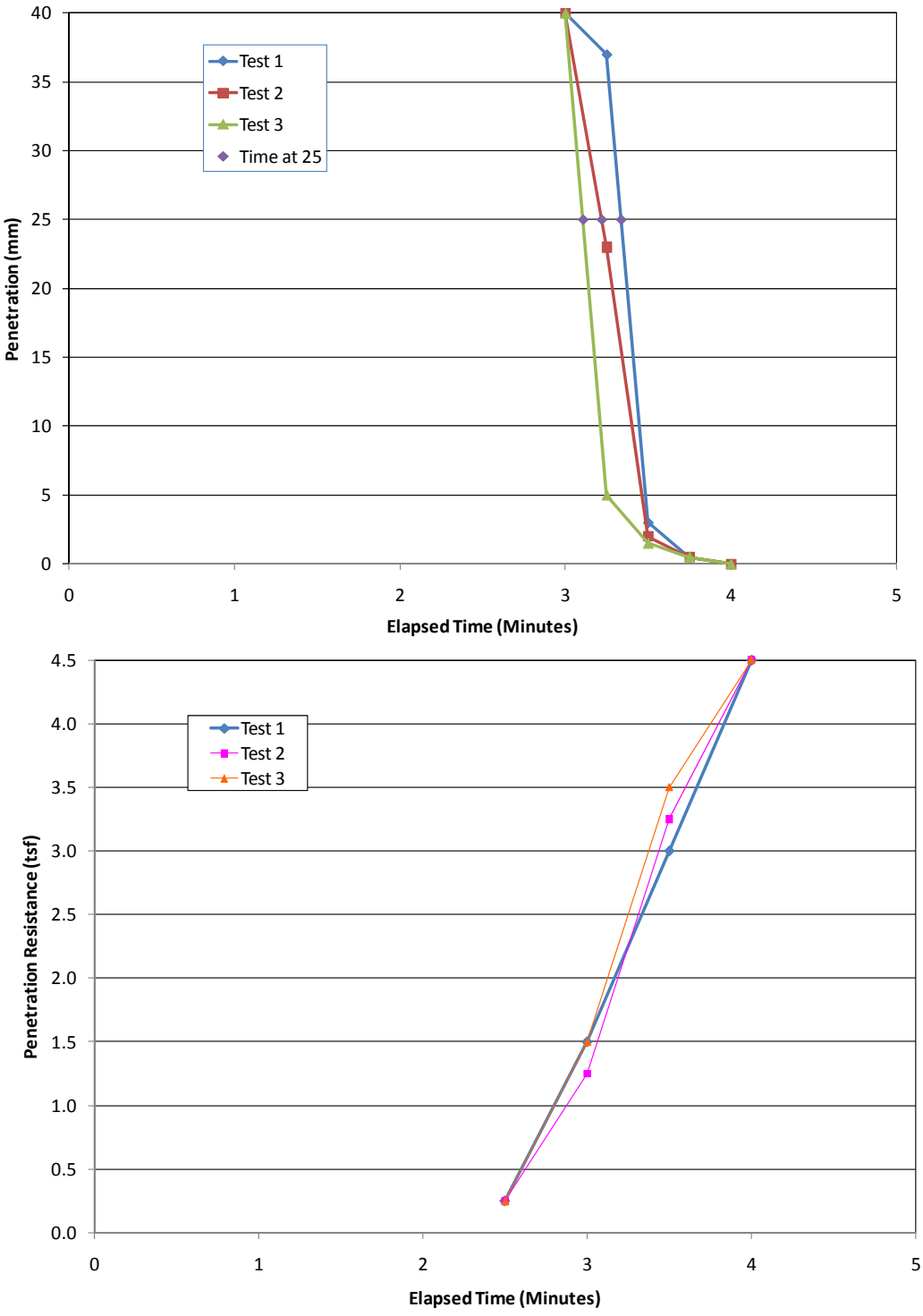



**Figure 135**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 12**

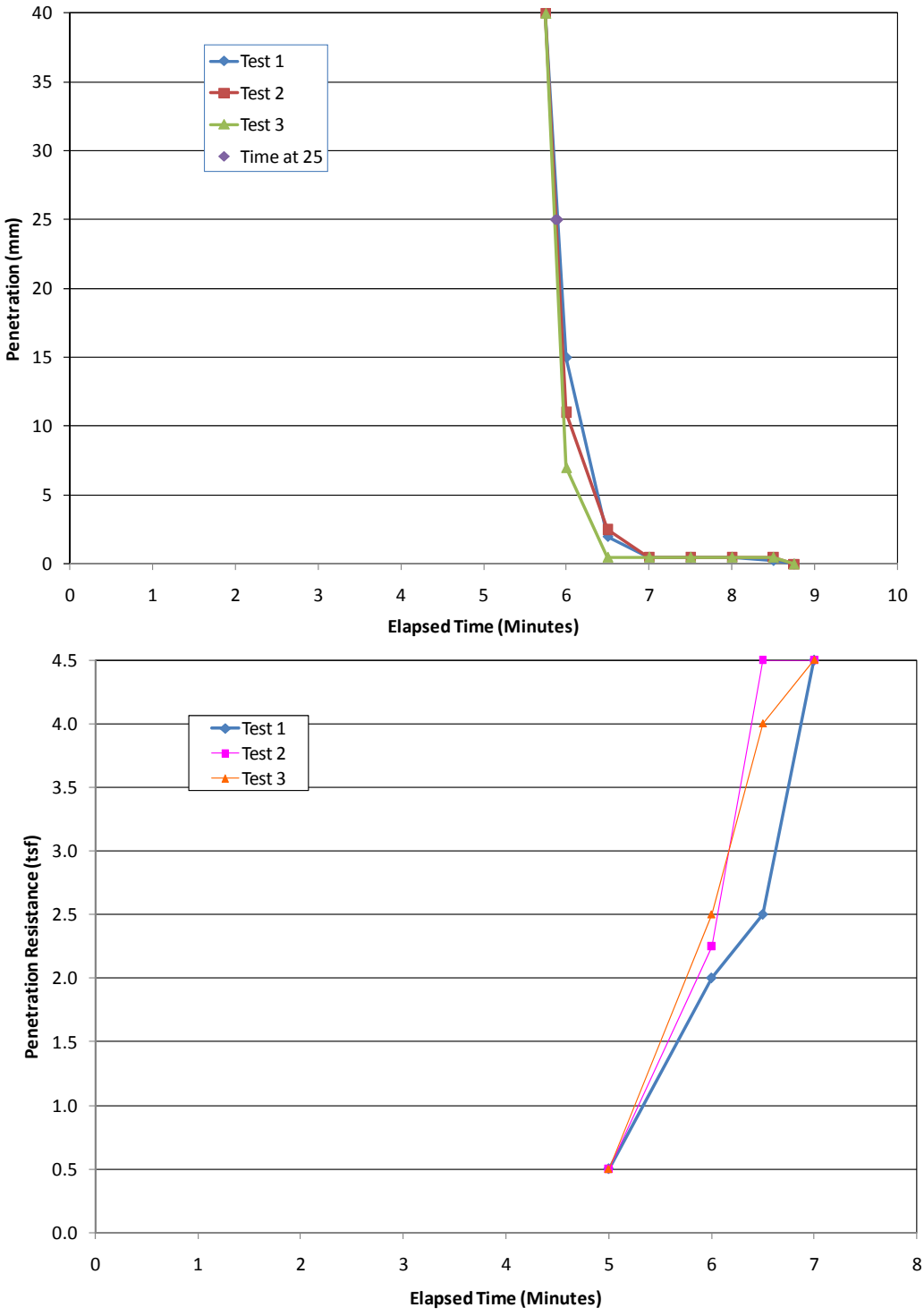



**Figure 136**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 13**

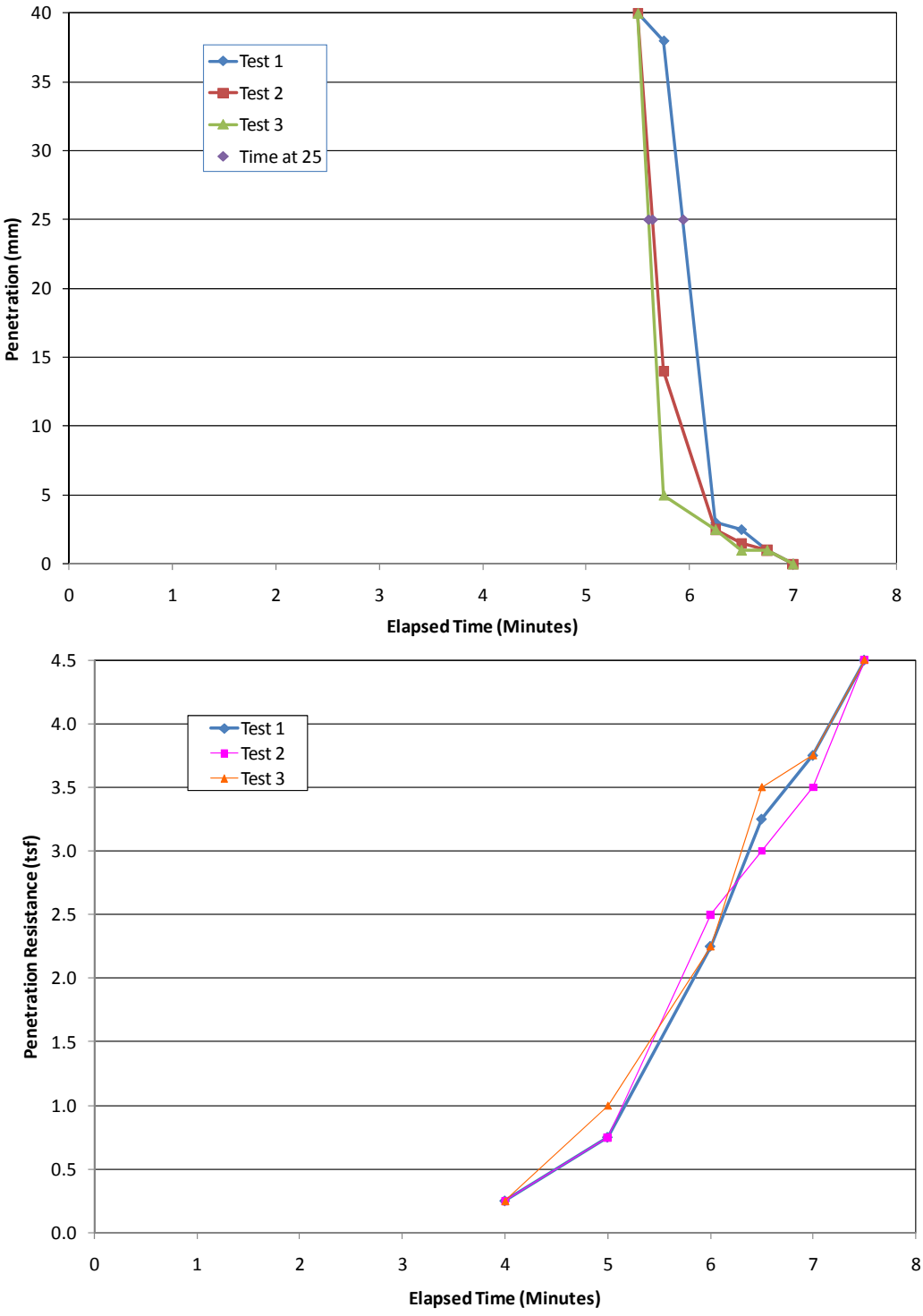



**Figure 137**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 14**

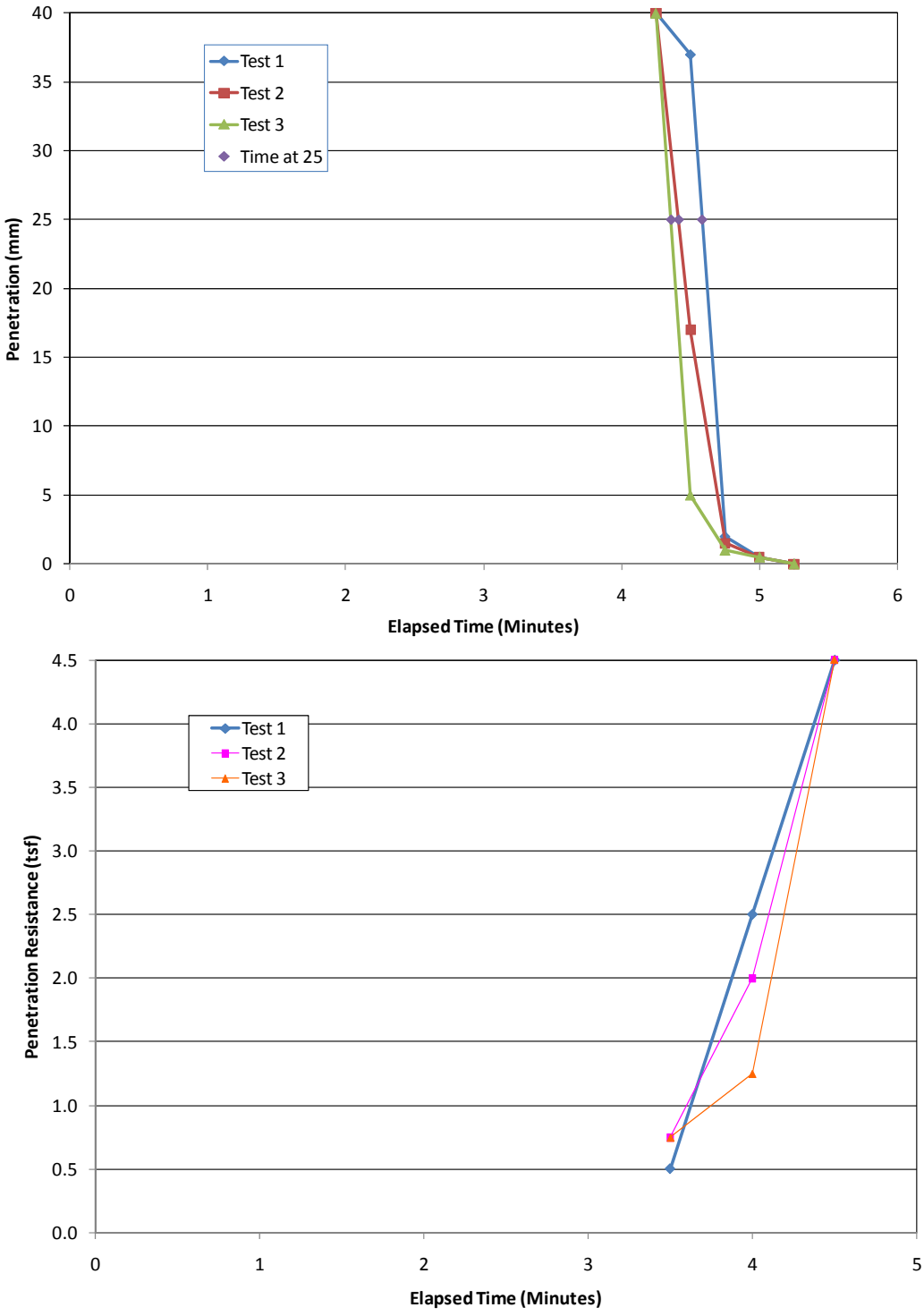



**Figure 138**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 15**

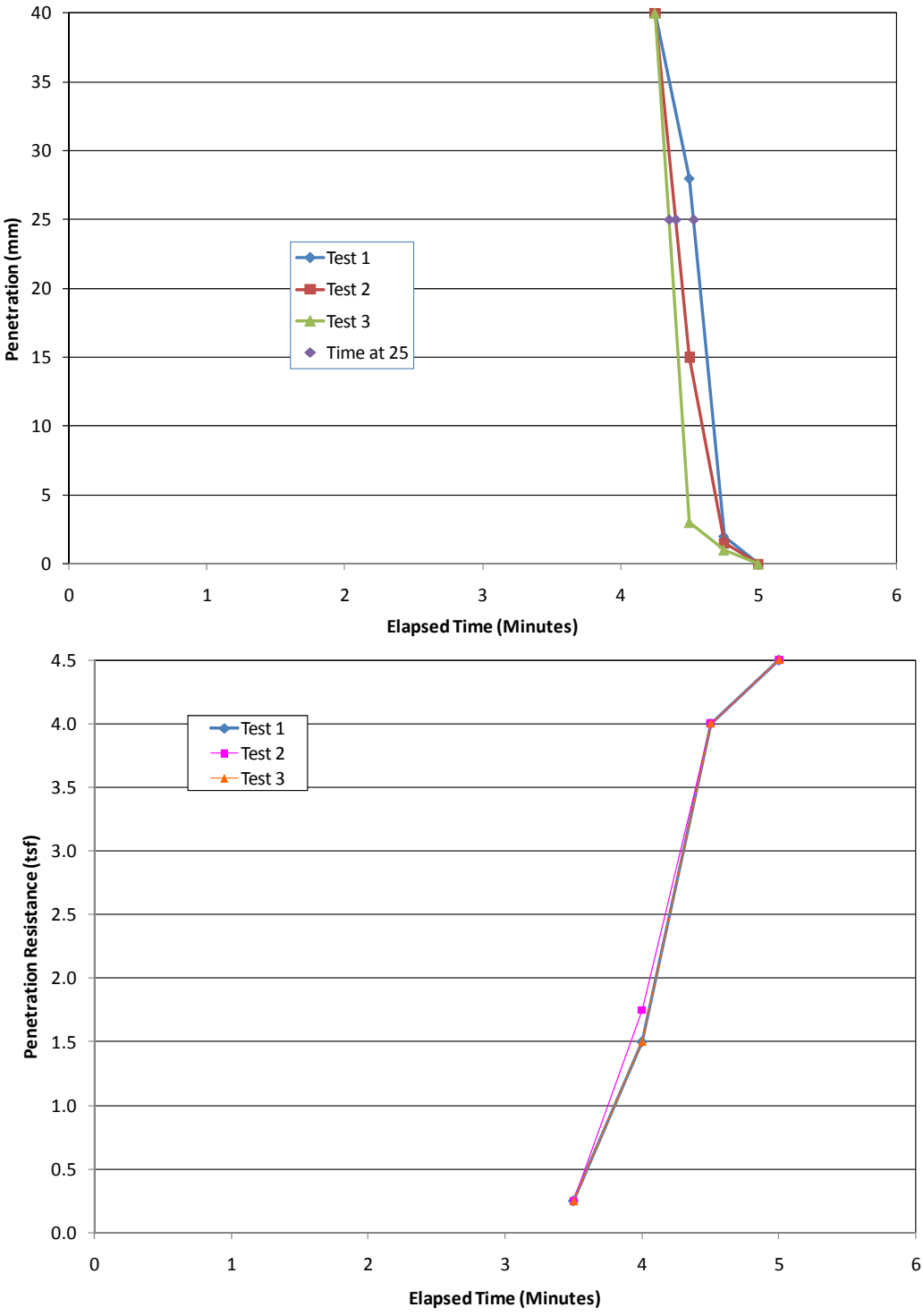



**Figure 139**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 16**

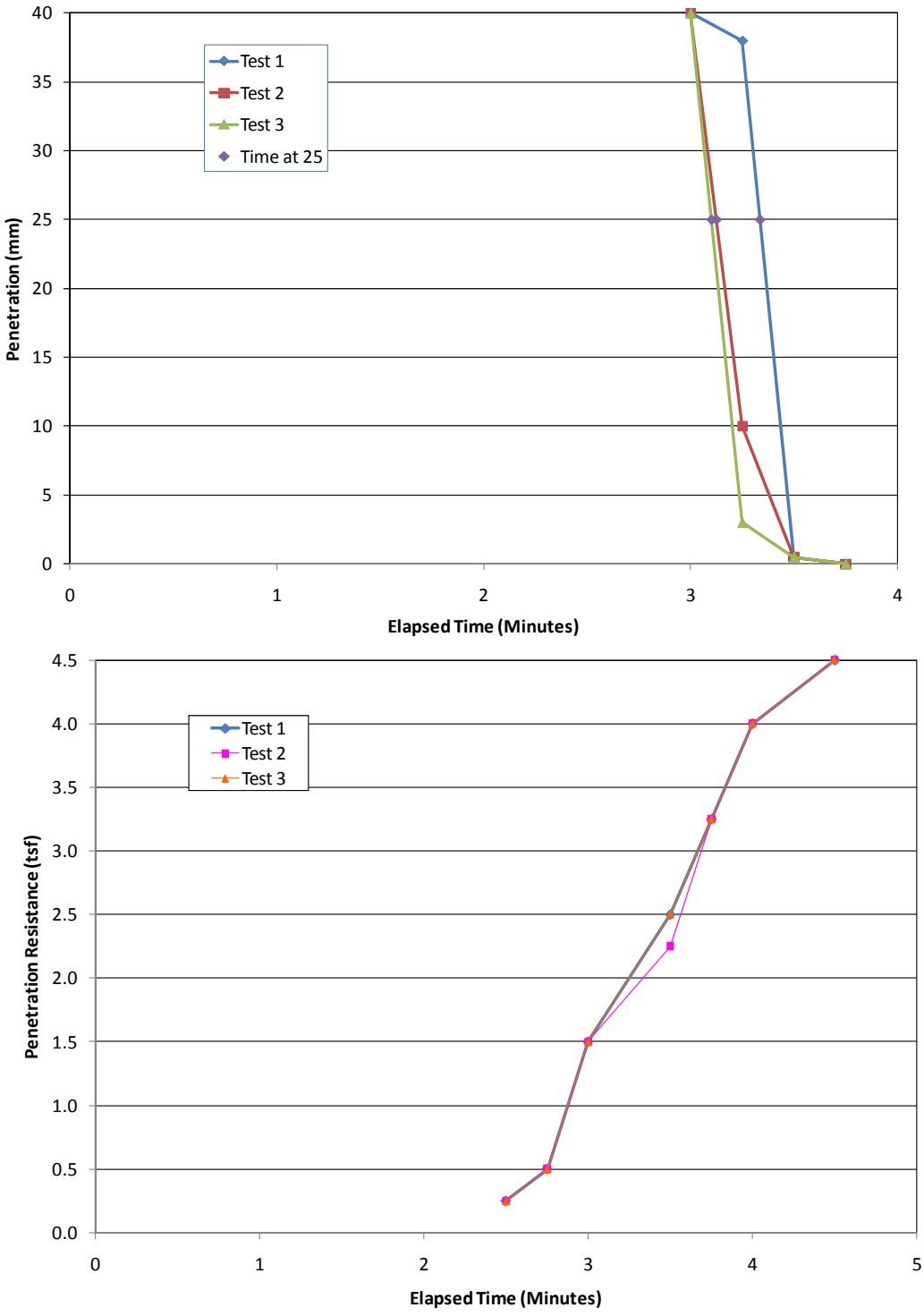



**Figure 140**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 17**

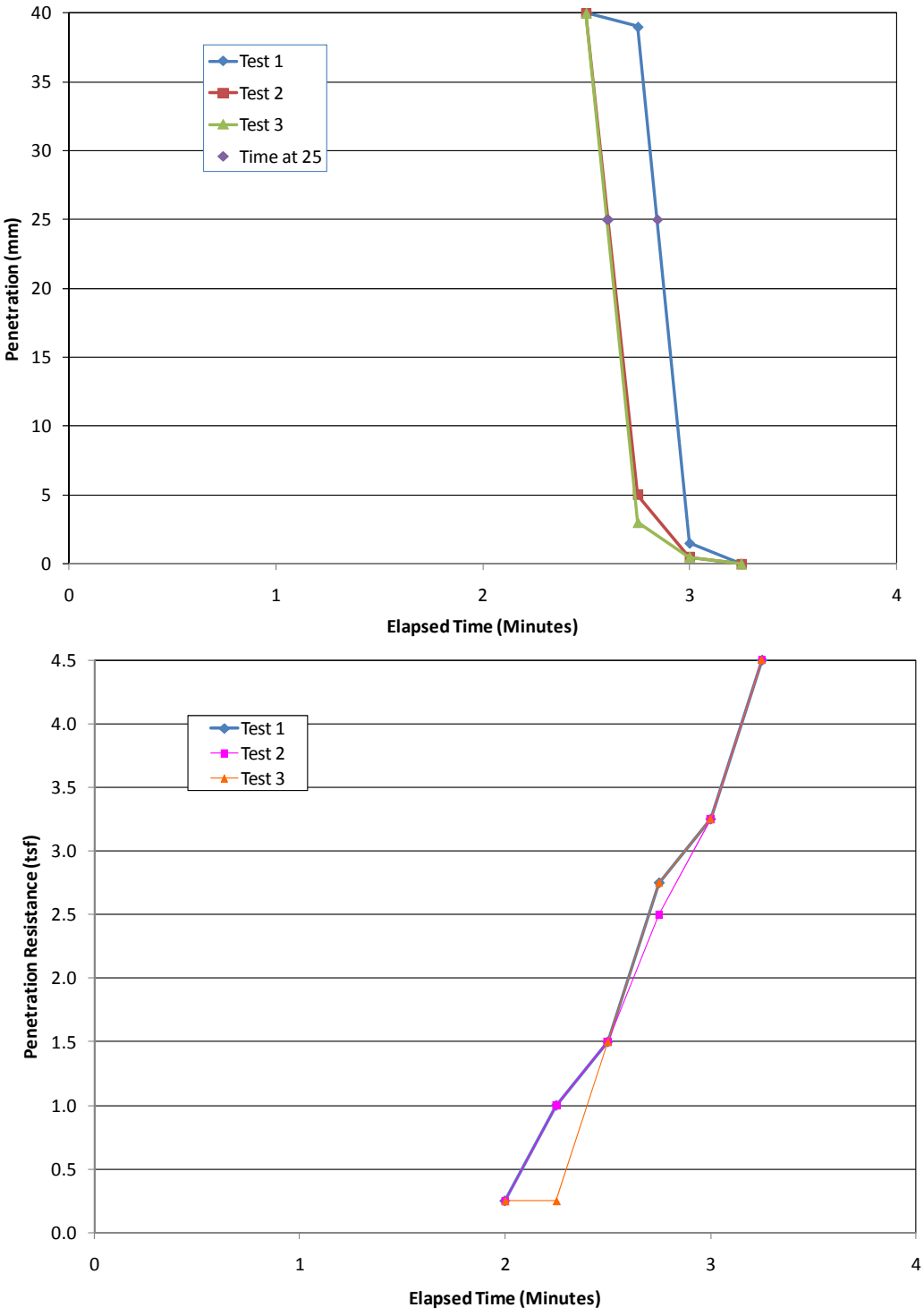



**Figure 141**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 18**

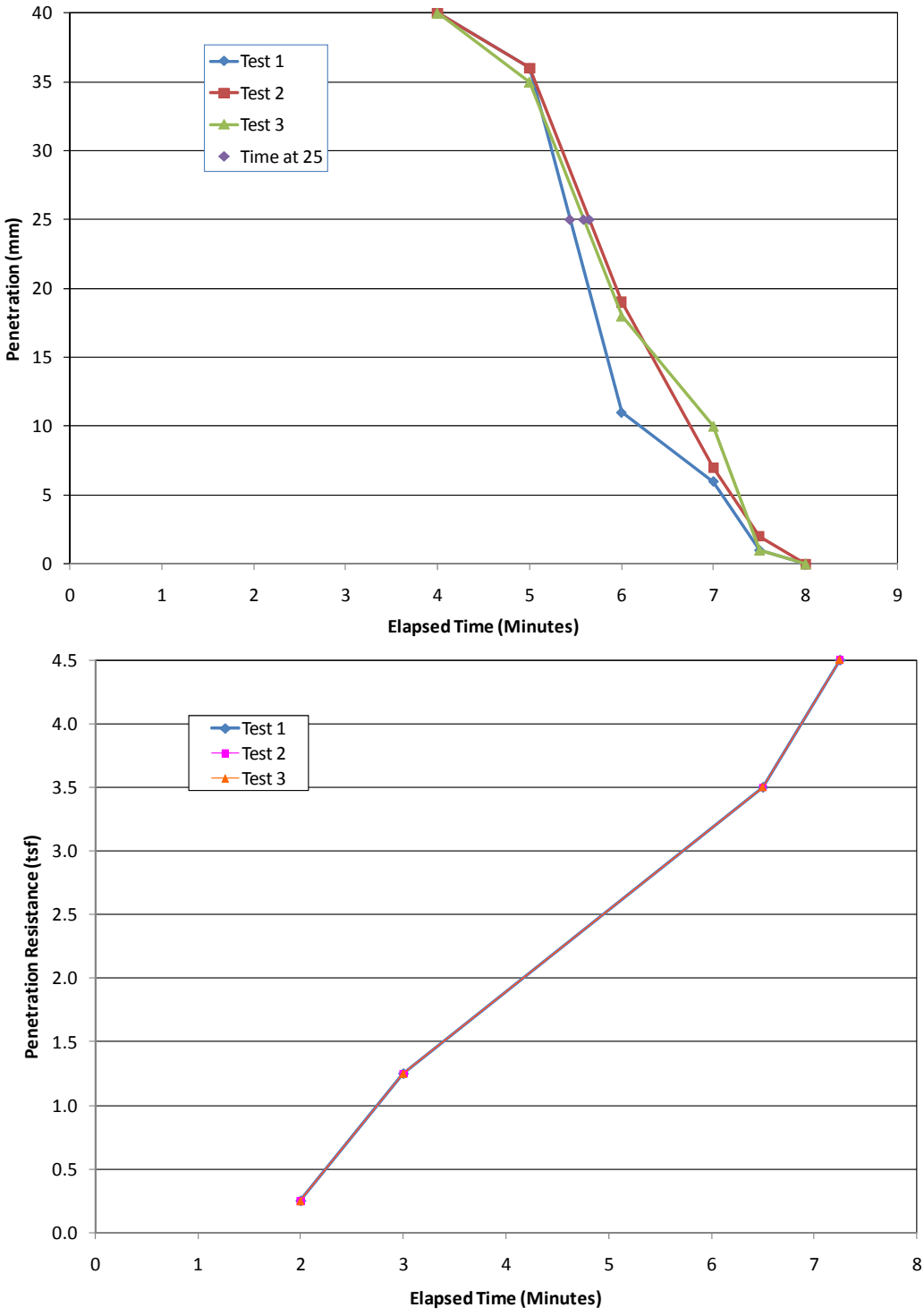



**Figure 142**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 19**

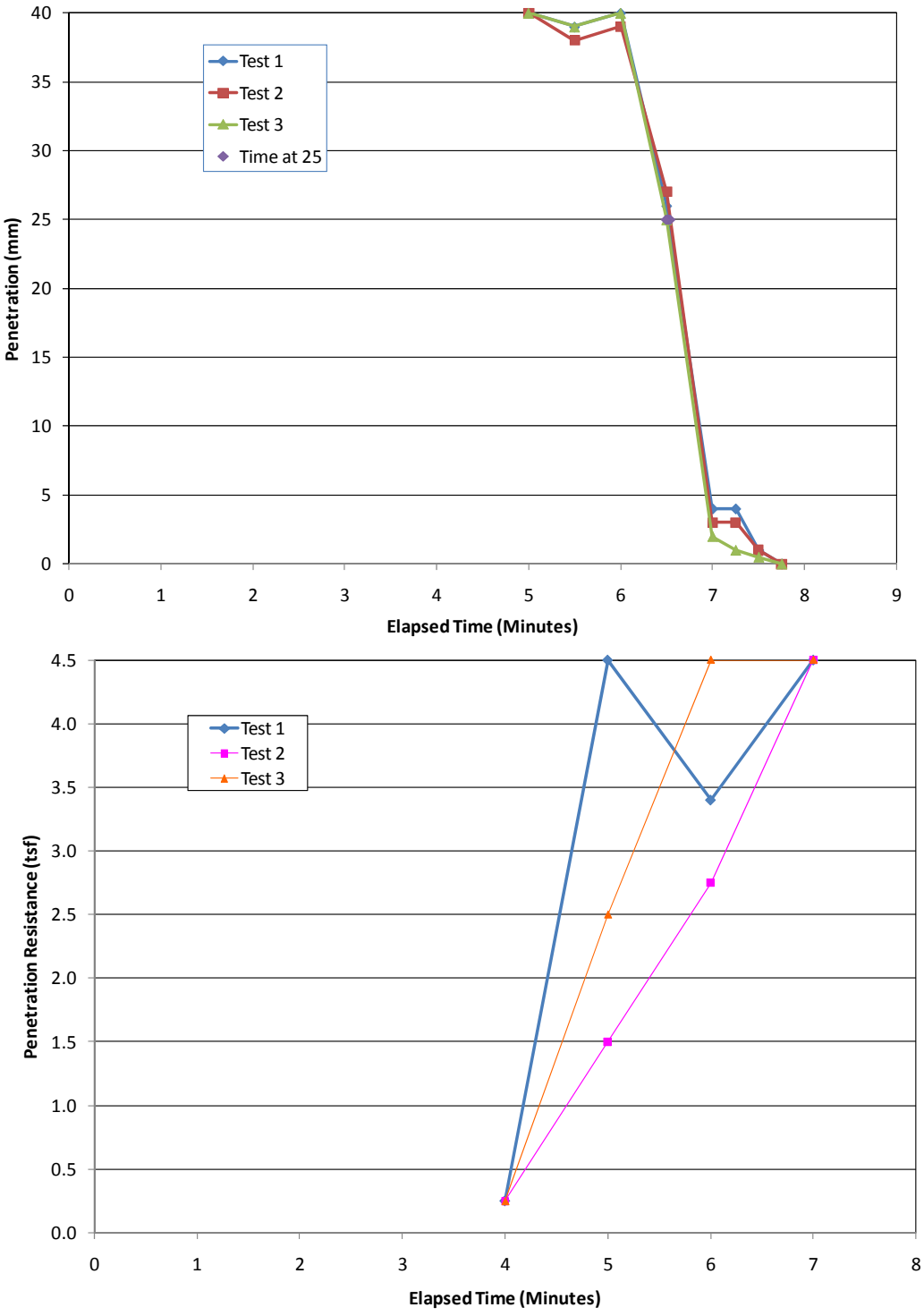



**Figure 143**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 6 Bucket 20**

## Source 7

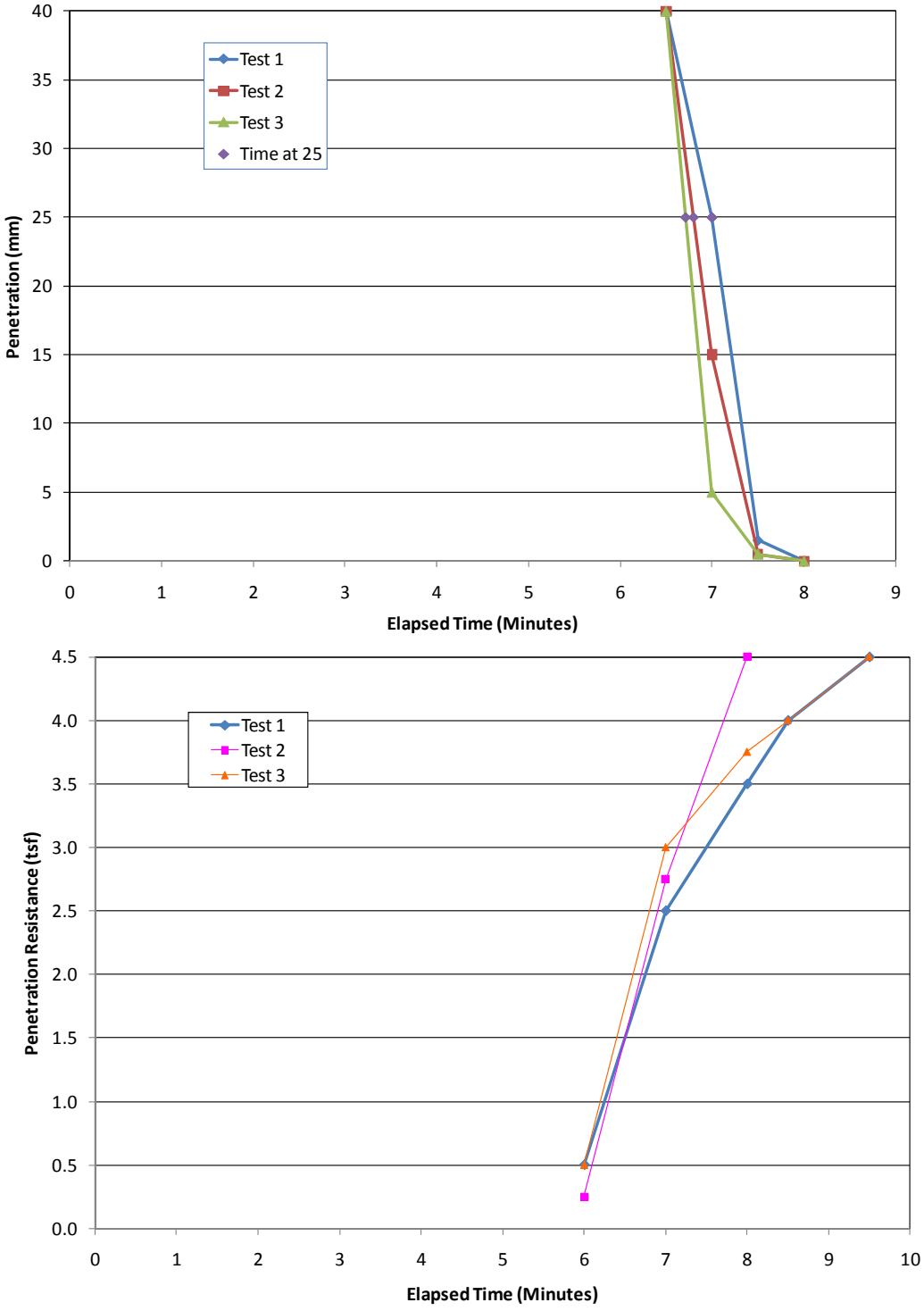



**Figure 144**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 1**

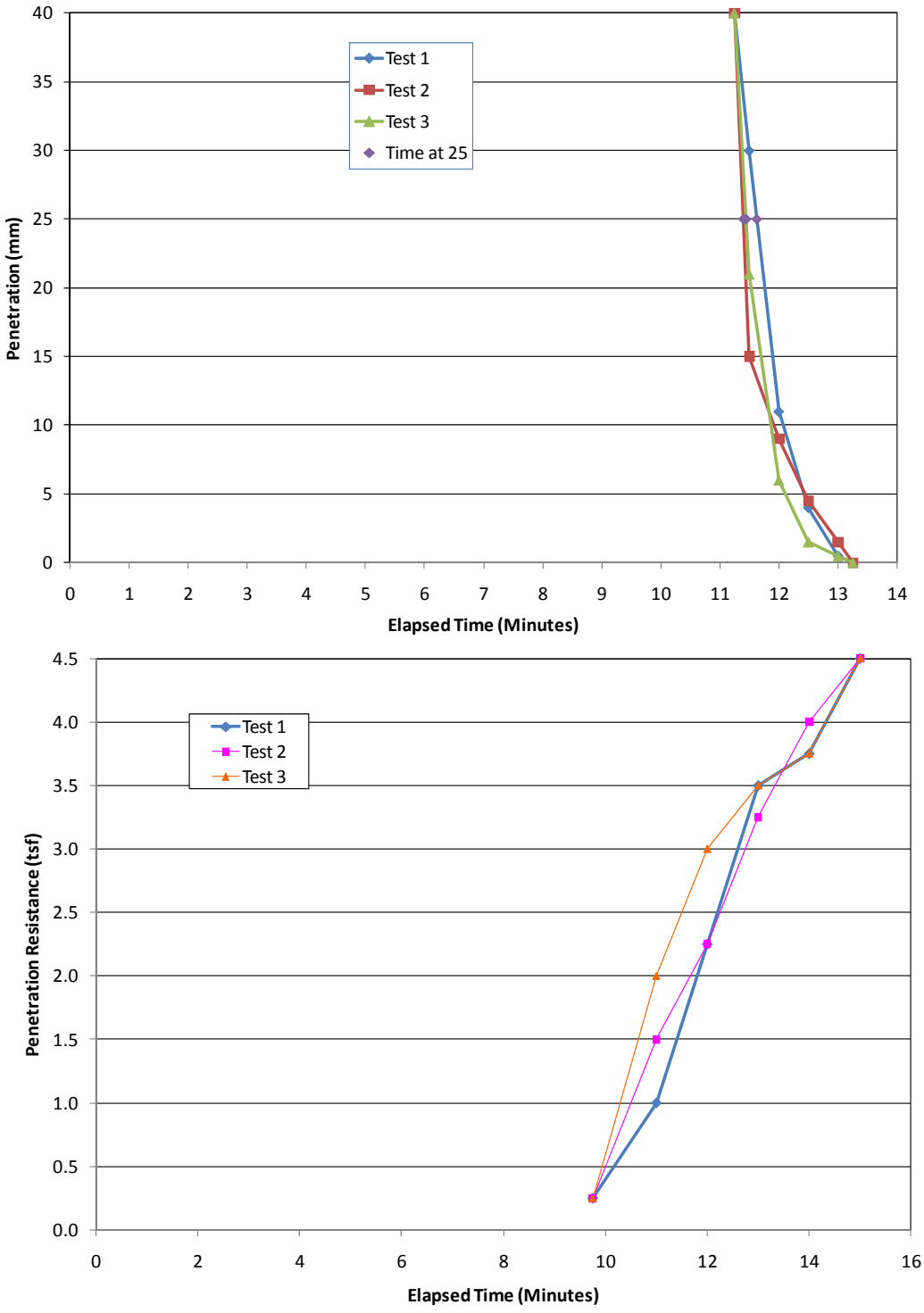



**Figure 145**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 2**

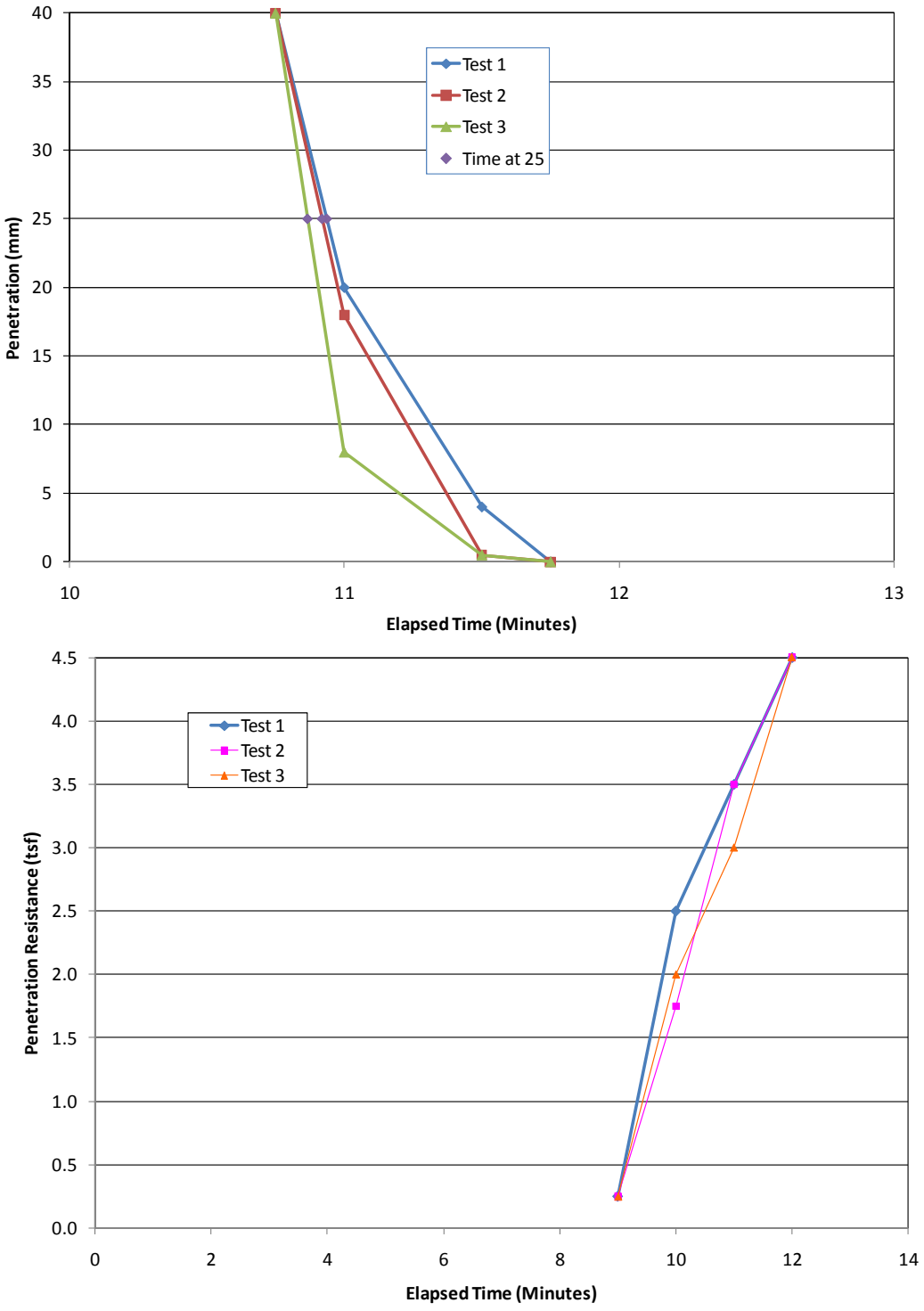



**Figure 146**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 3**

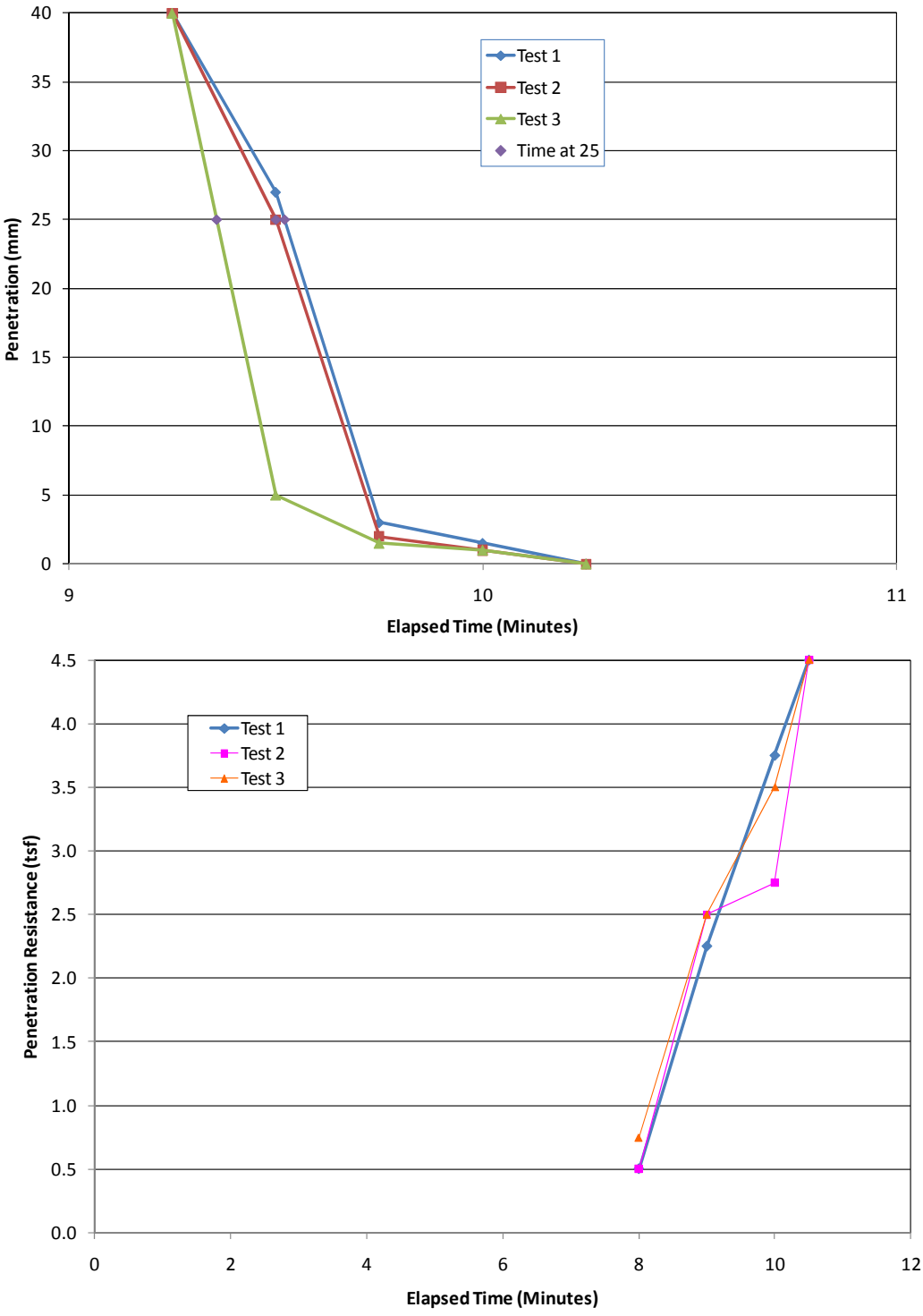



**Figure 147**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 4**

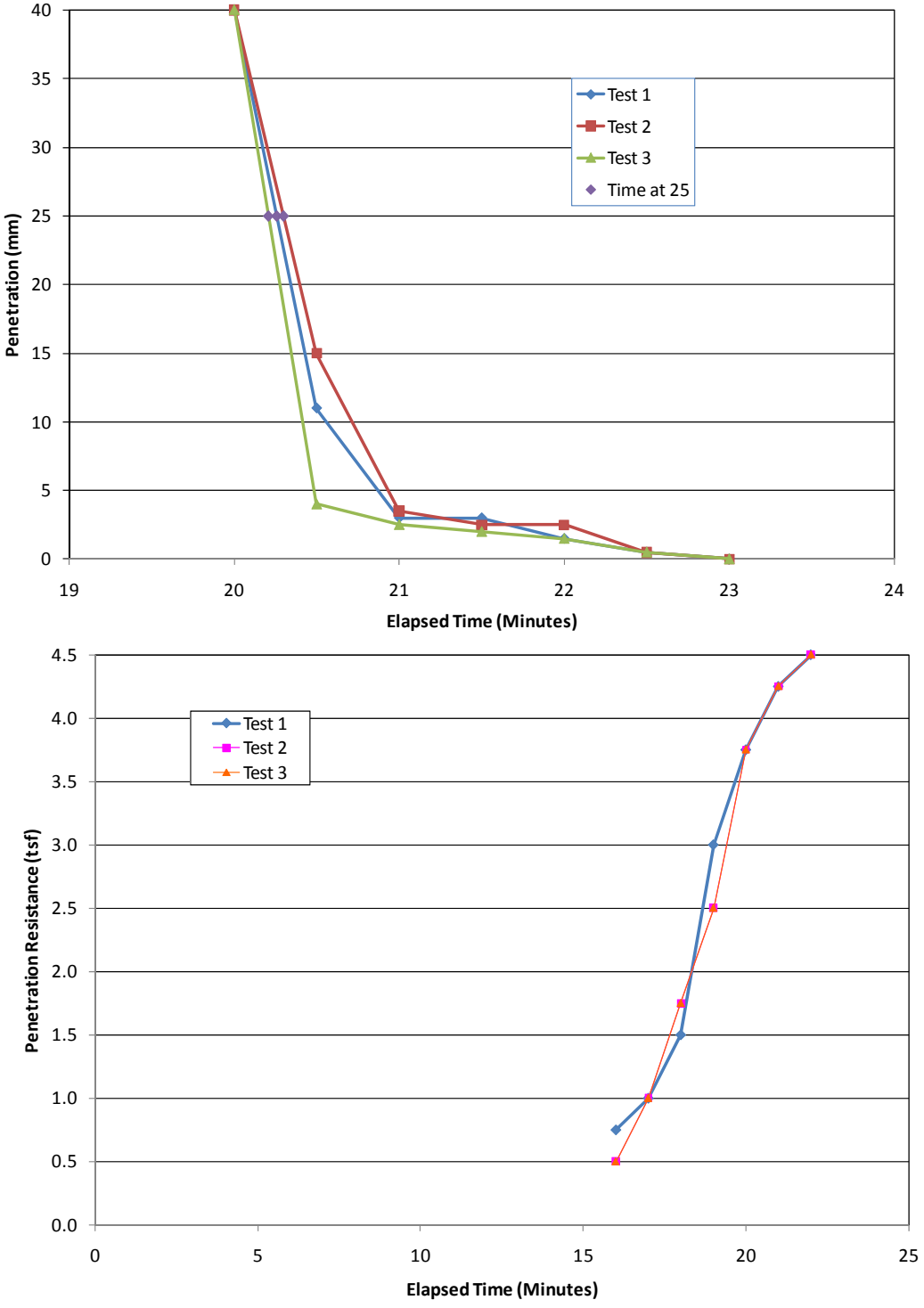



**Figure 148**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 5**

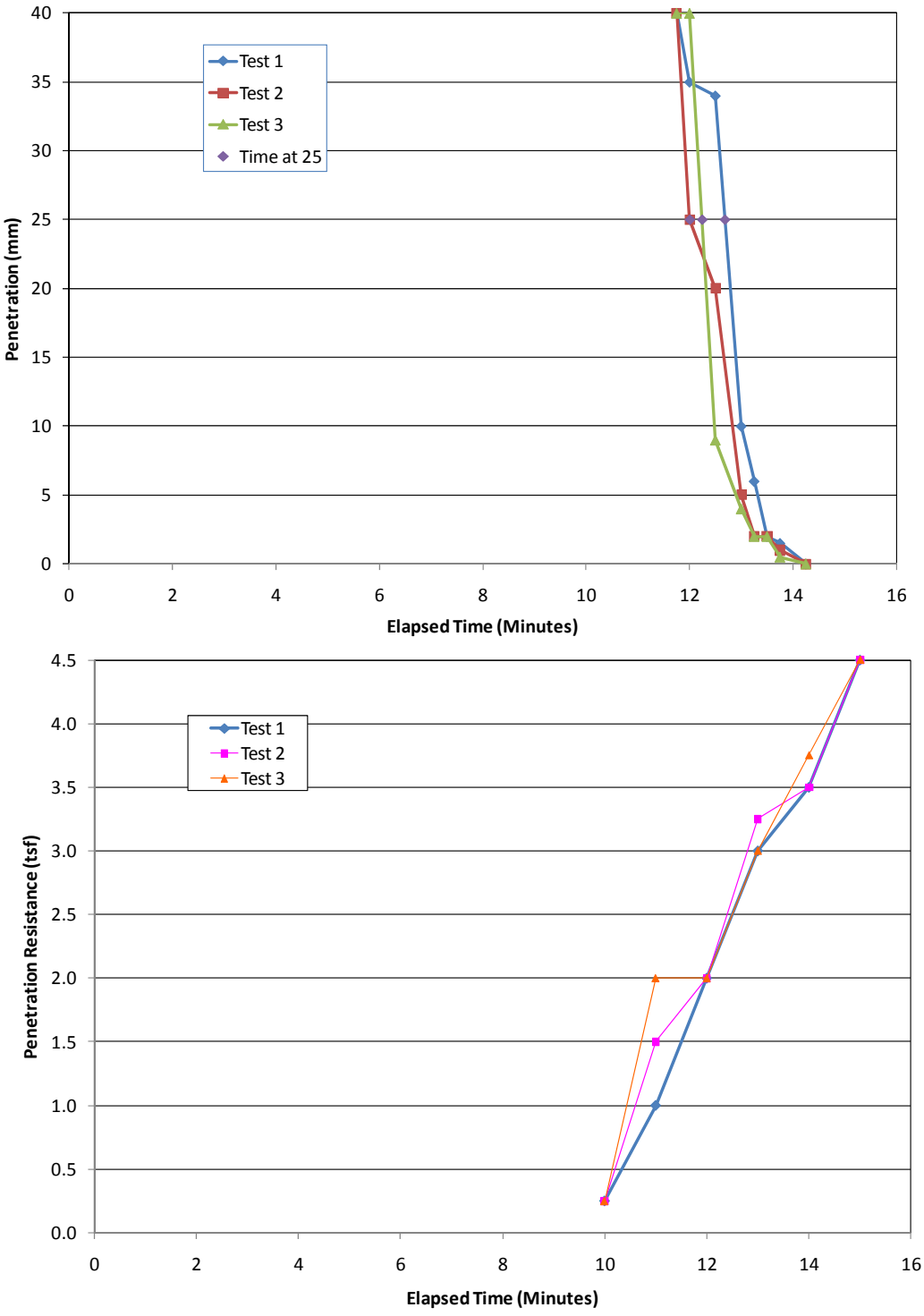



**Figure 149**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 6**

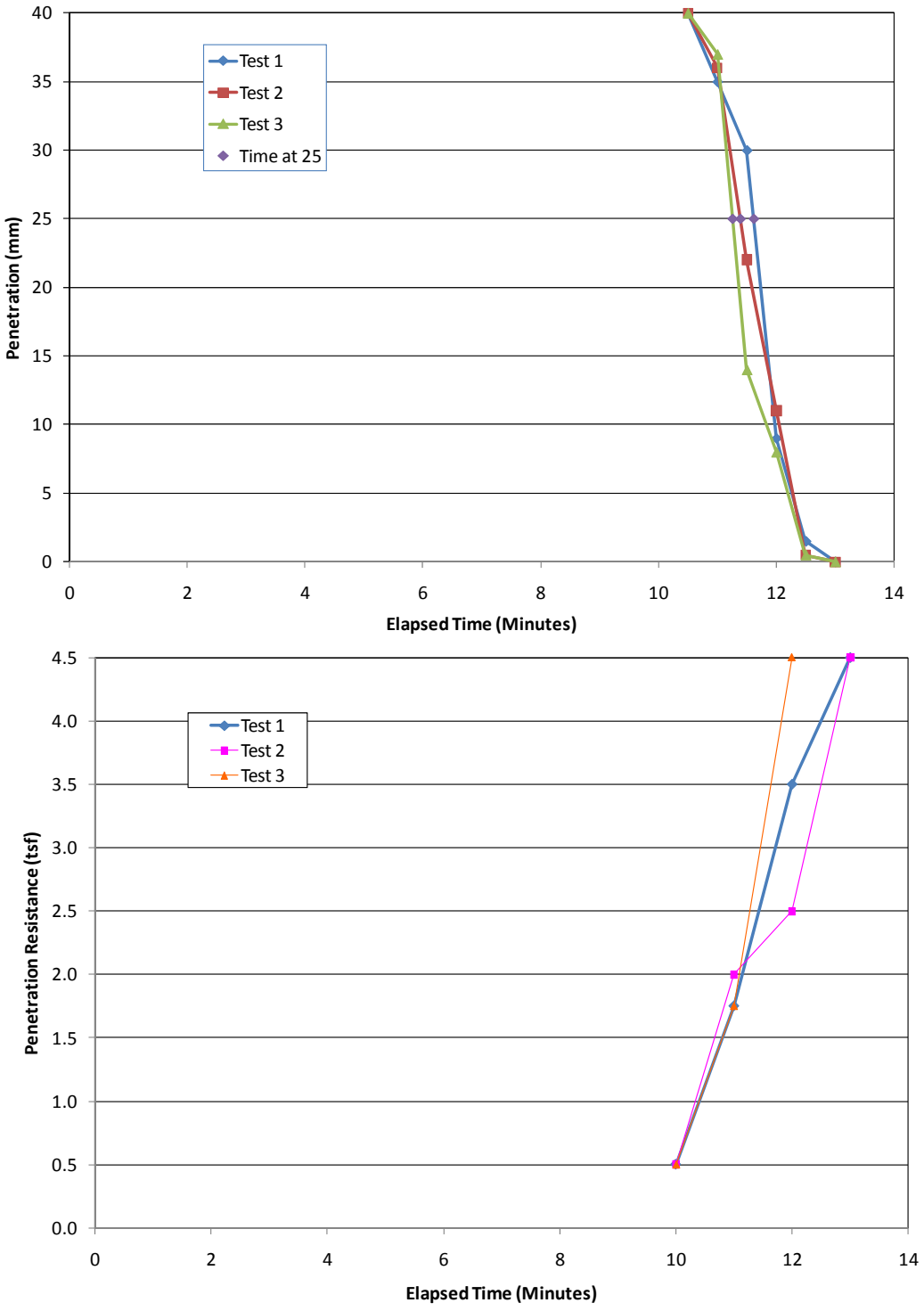



**Figure 150**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 7**

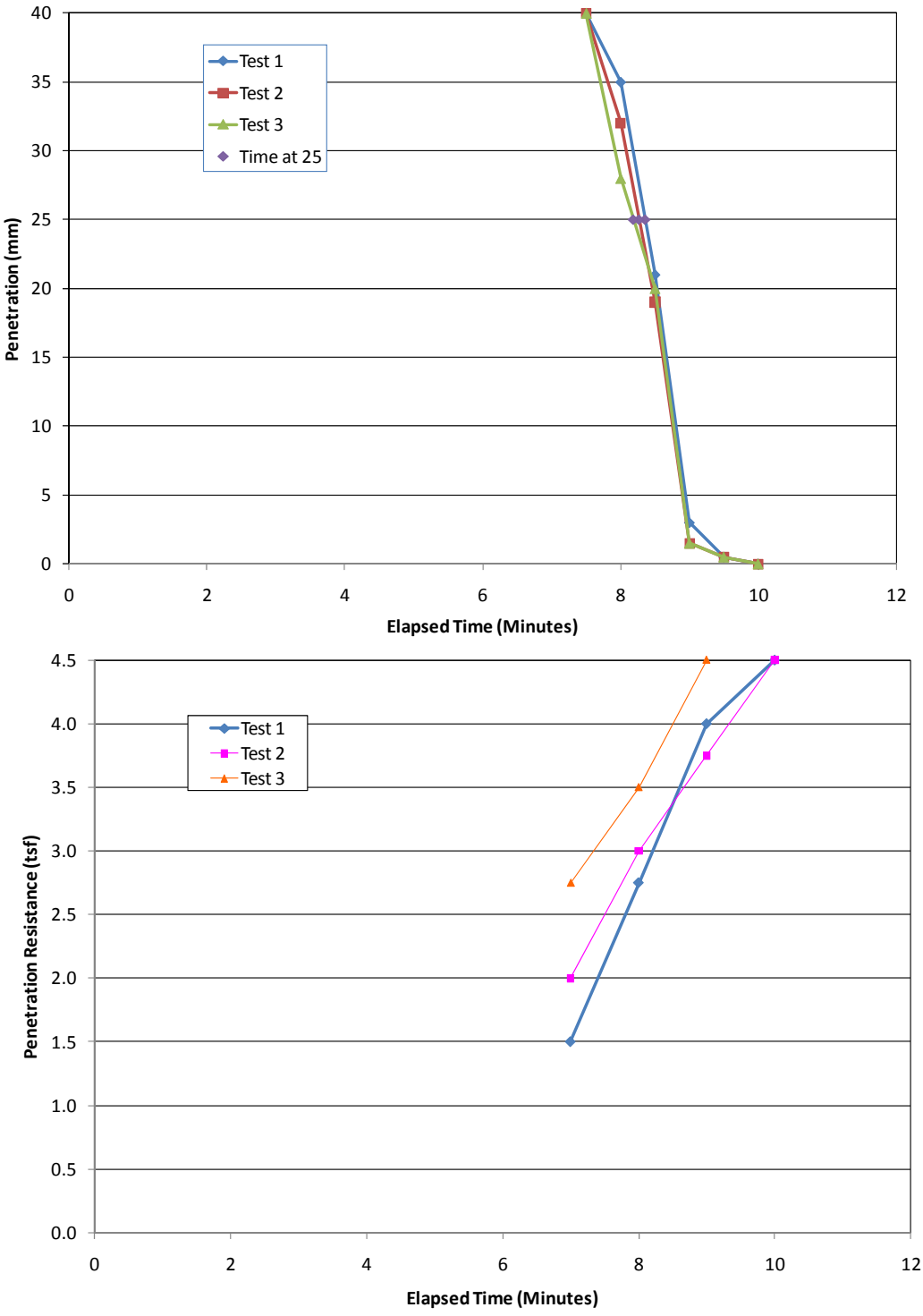



**Figure 151**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 8**

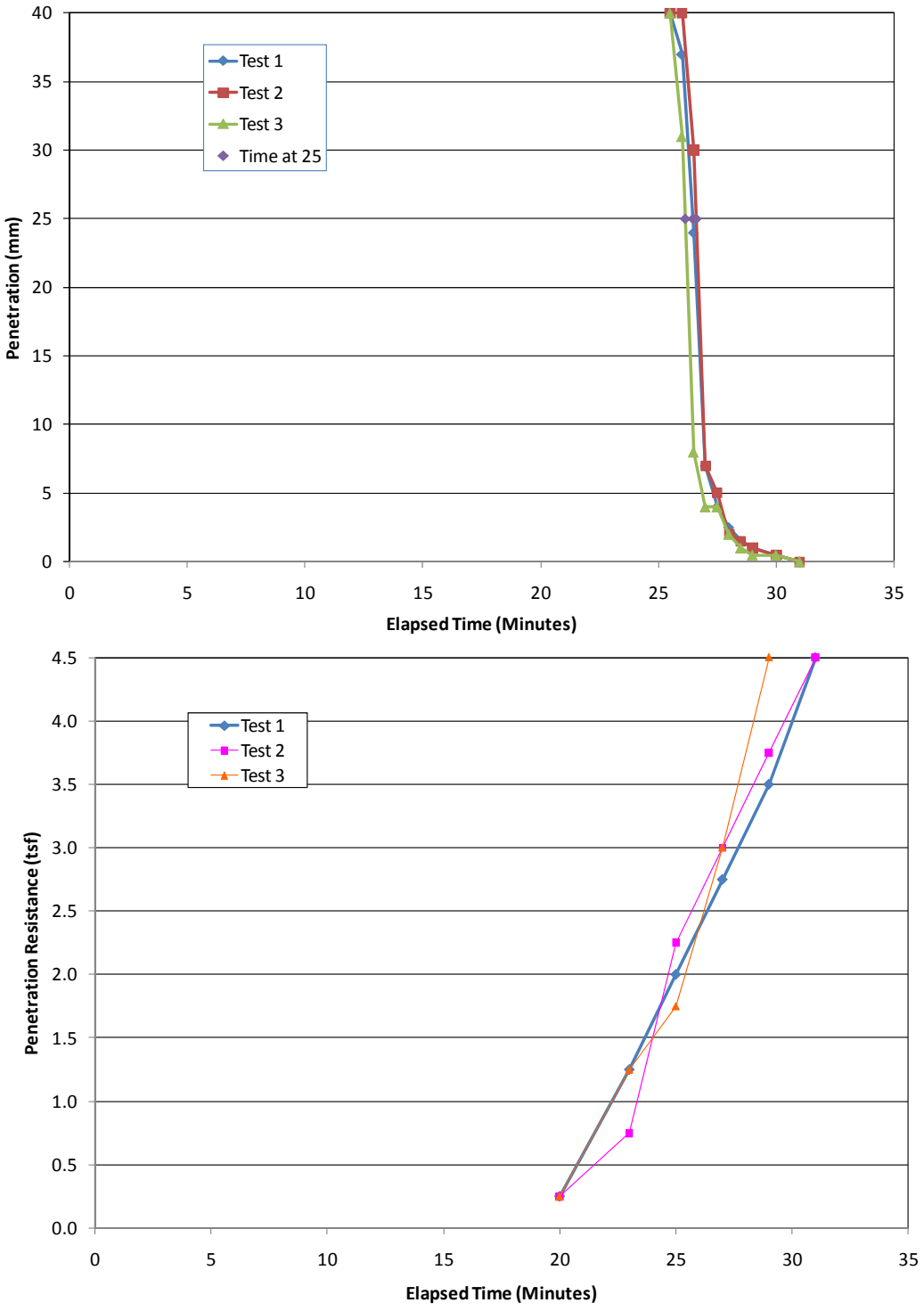
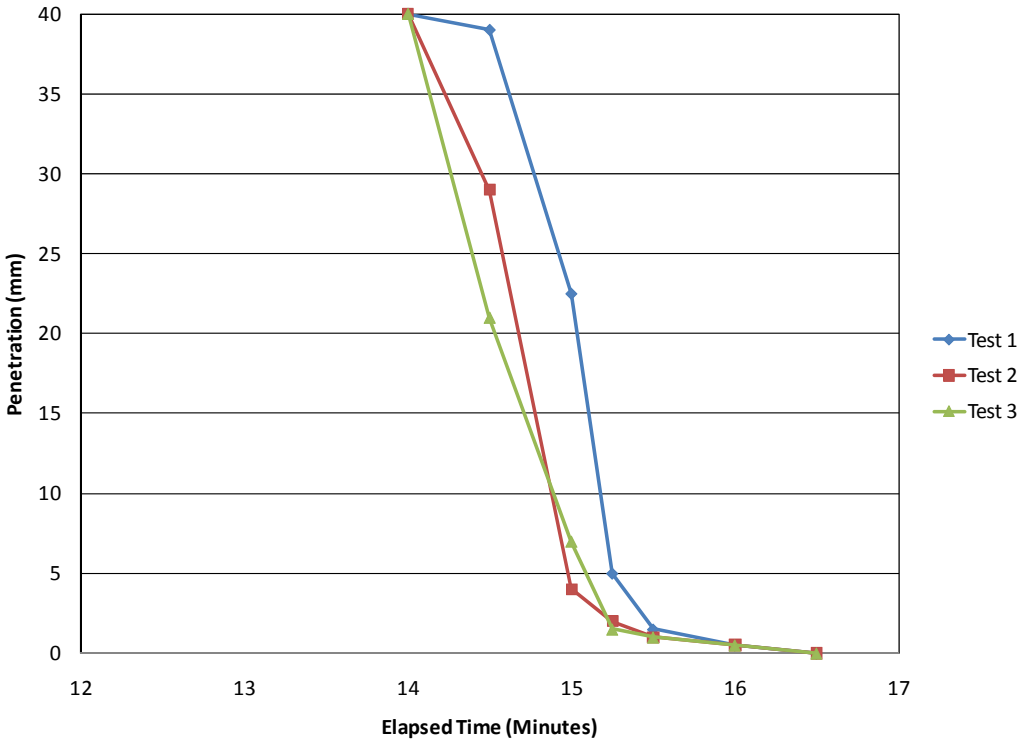



**Figure 152**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 9**



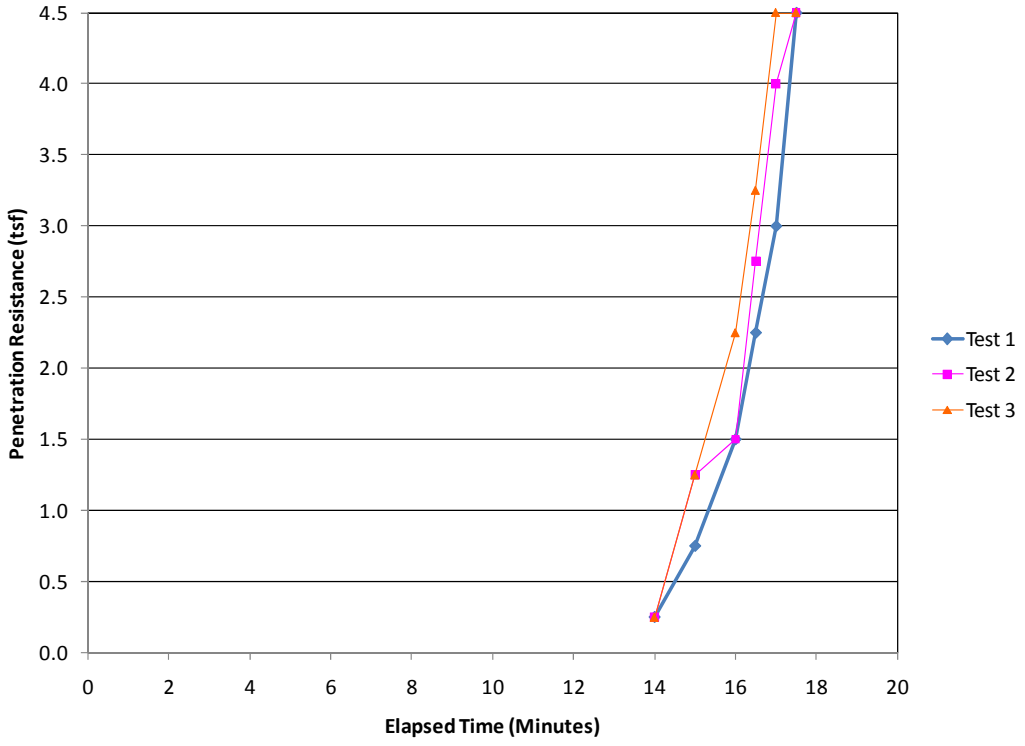




**Figure 153**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 10**

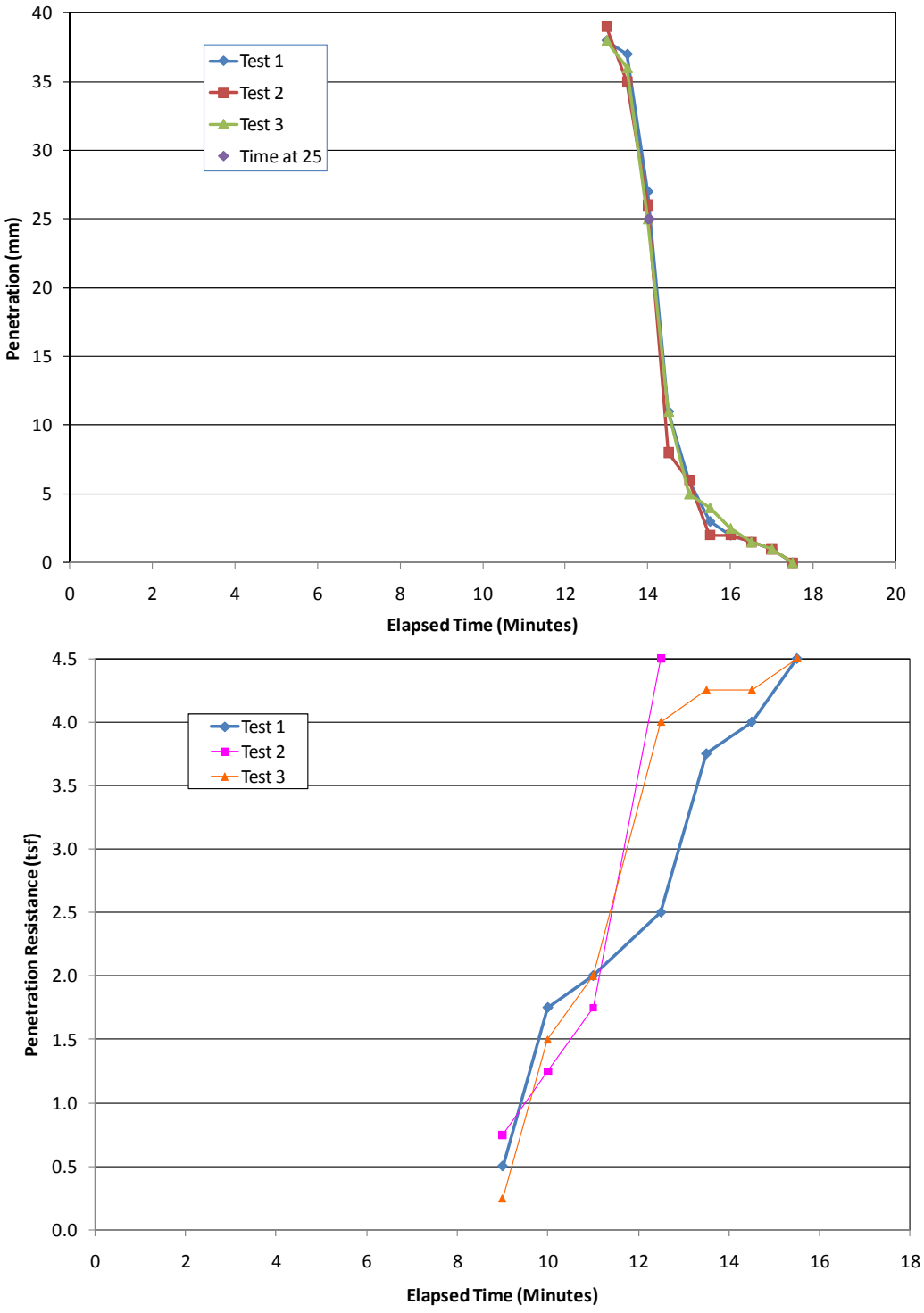



**Figure 154**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 11**

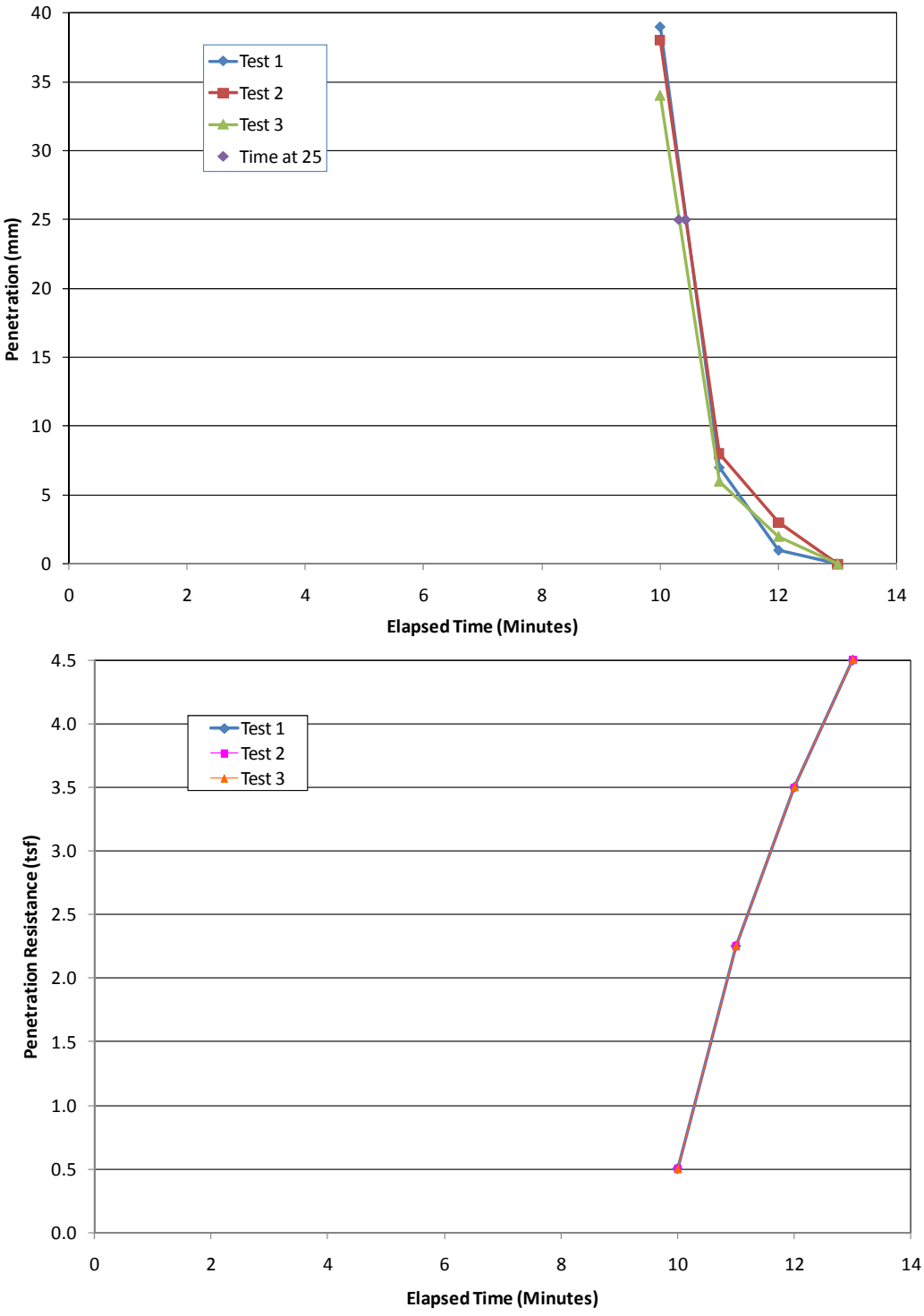



**Figure 155**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 12**

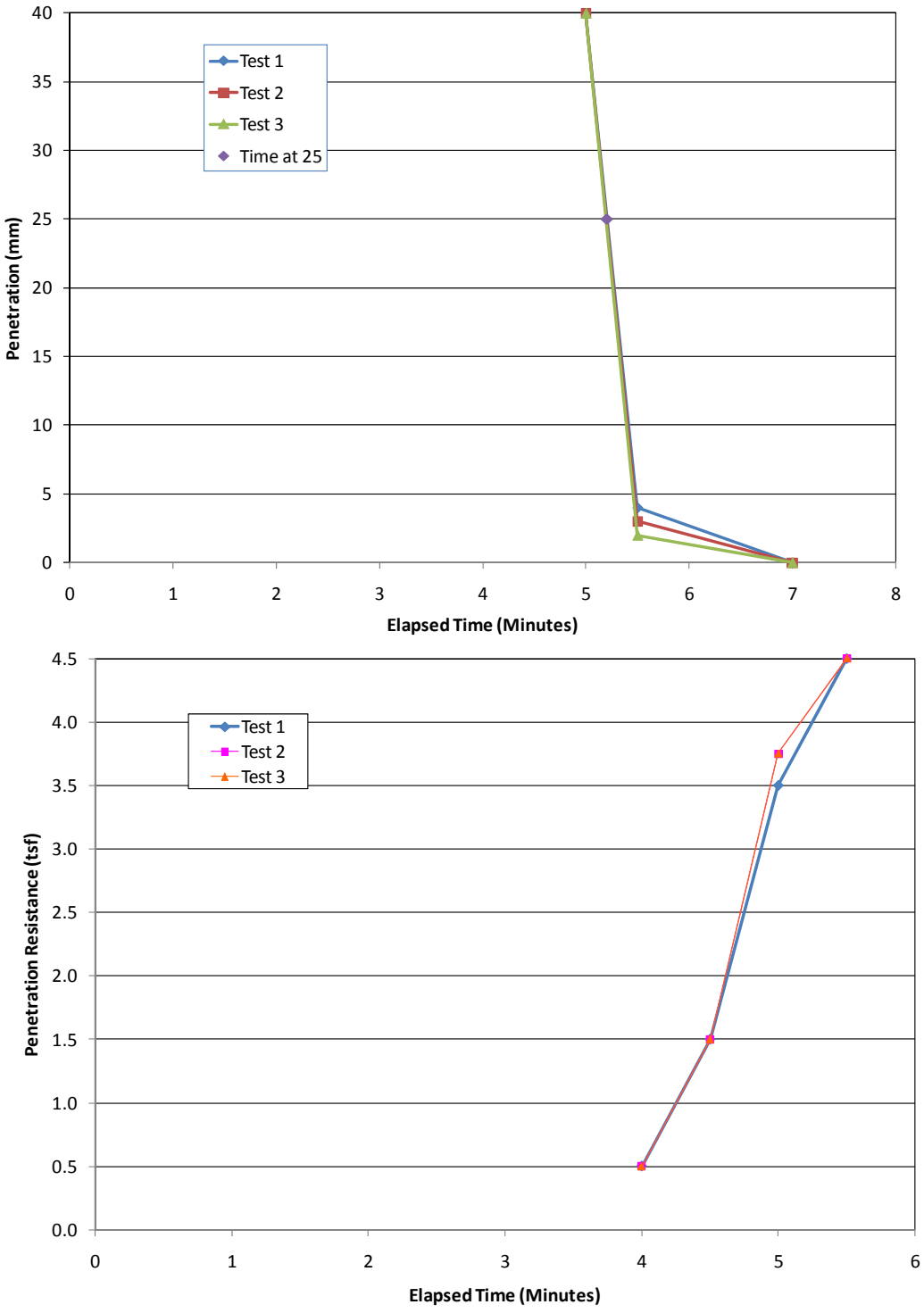
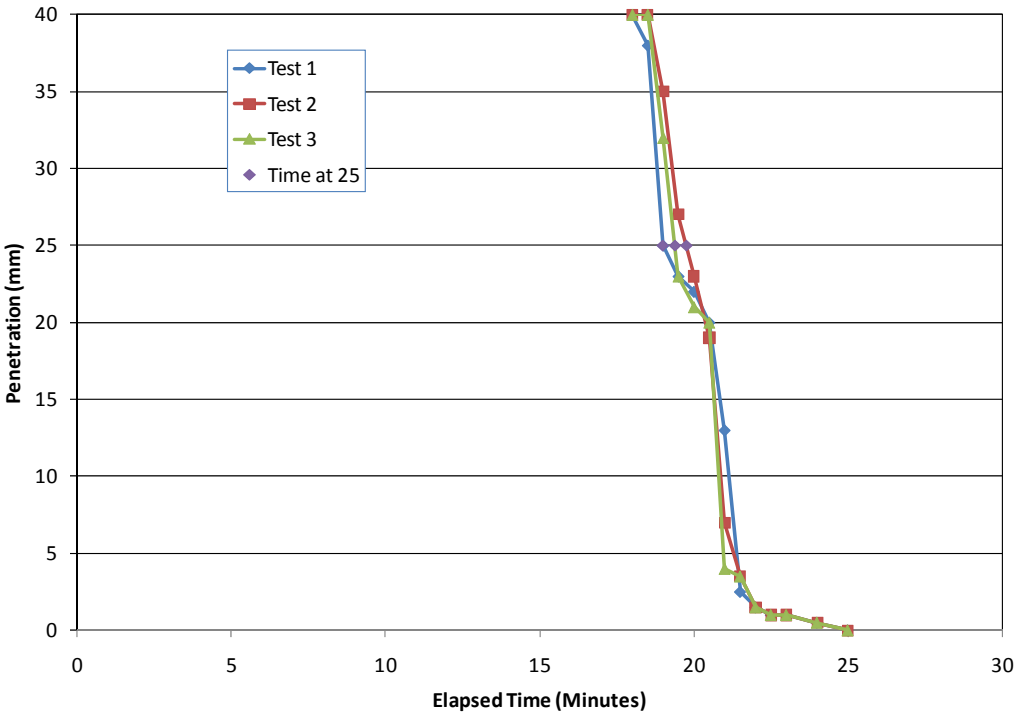



**Figure 156**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 13**



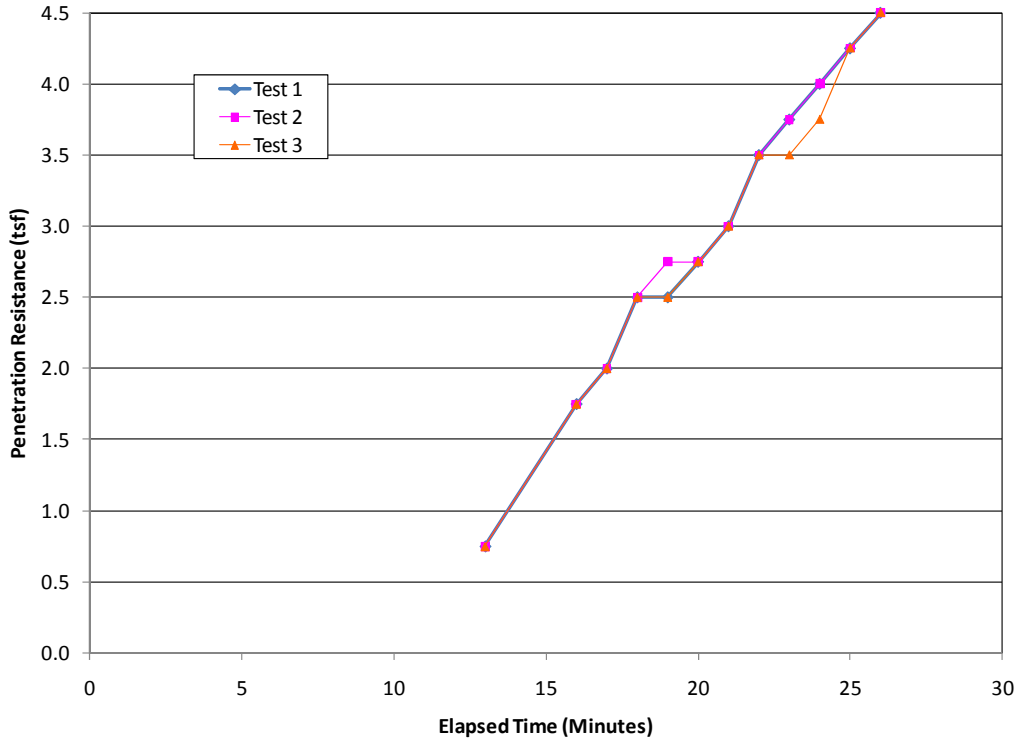




**Figure 157**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 14**

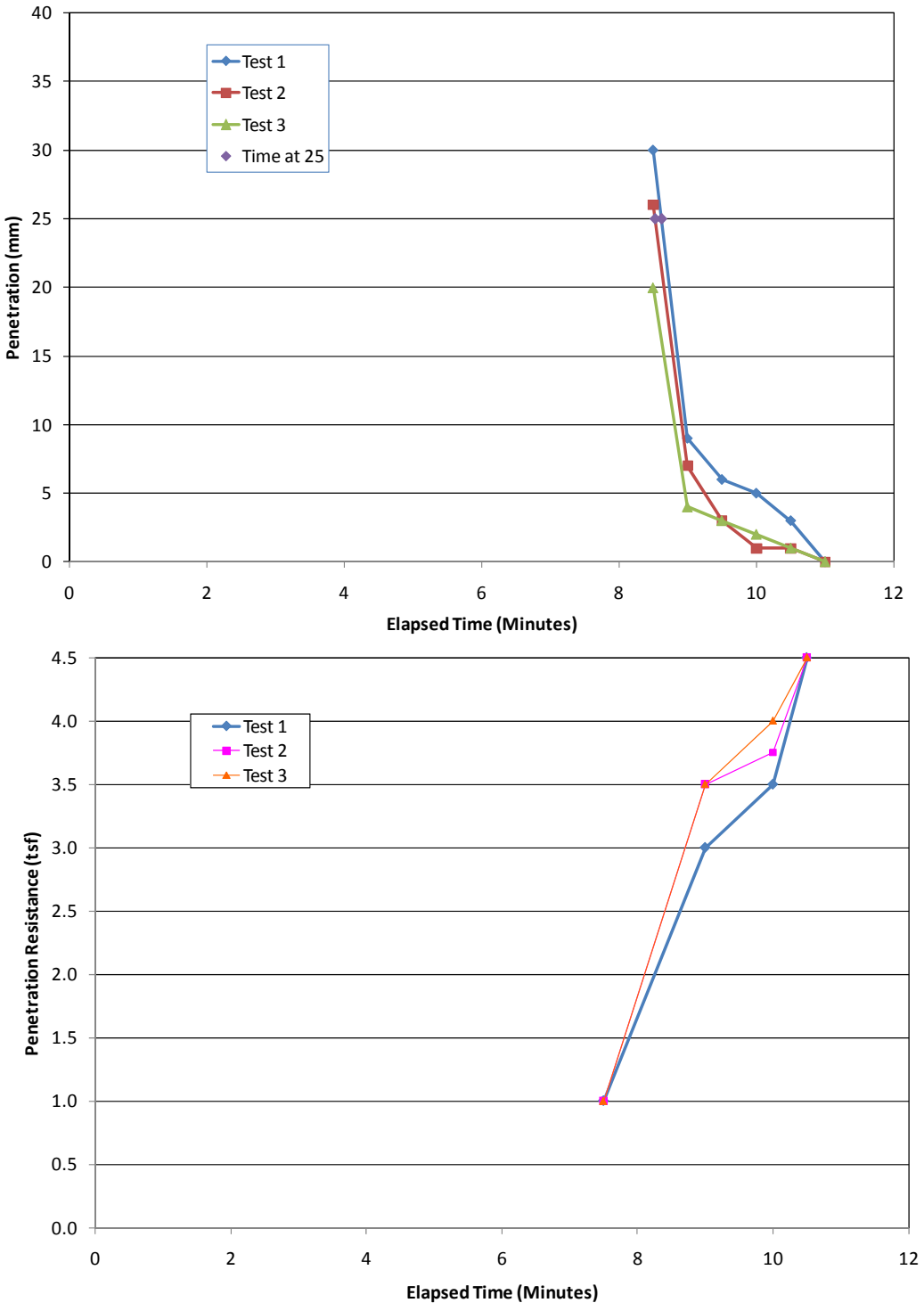



**Figure 158**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 15**

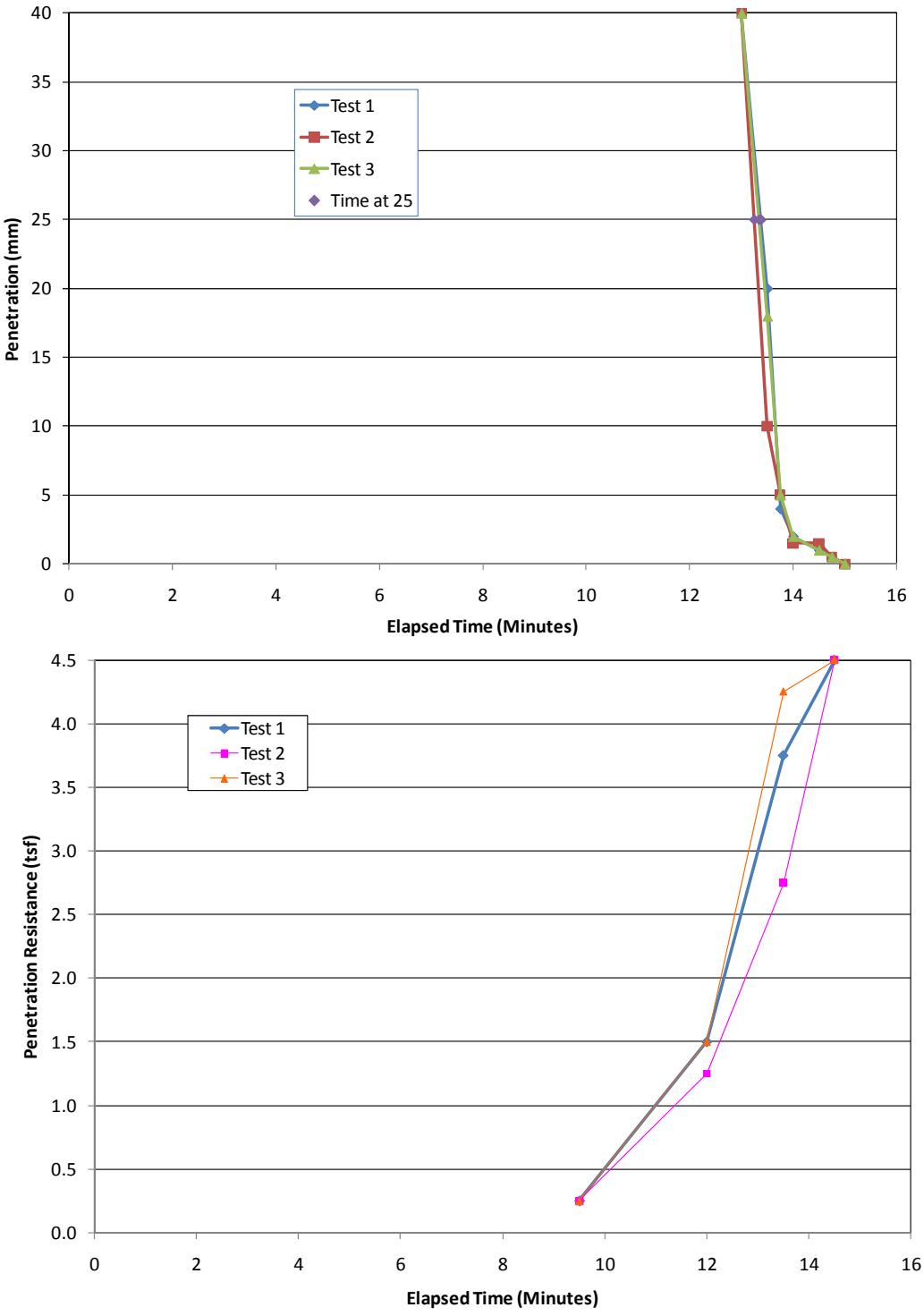



**Figure 159**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 16**

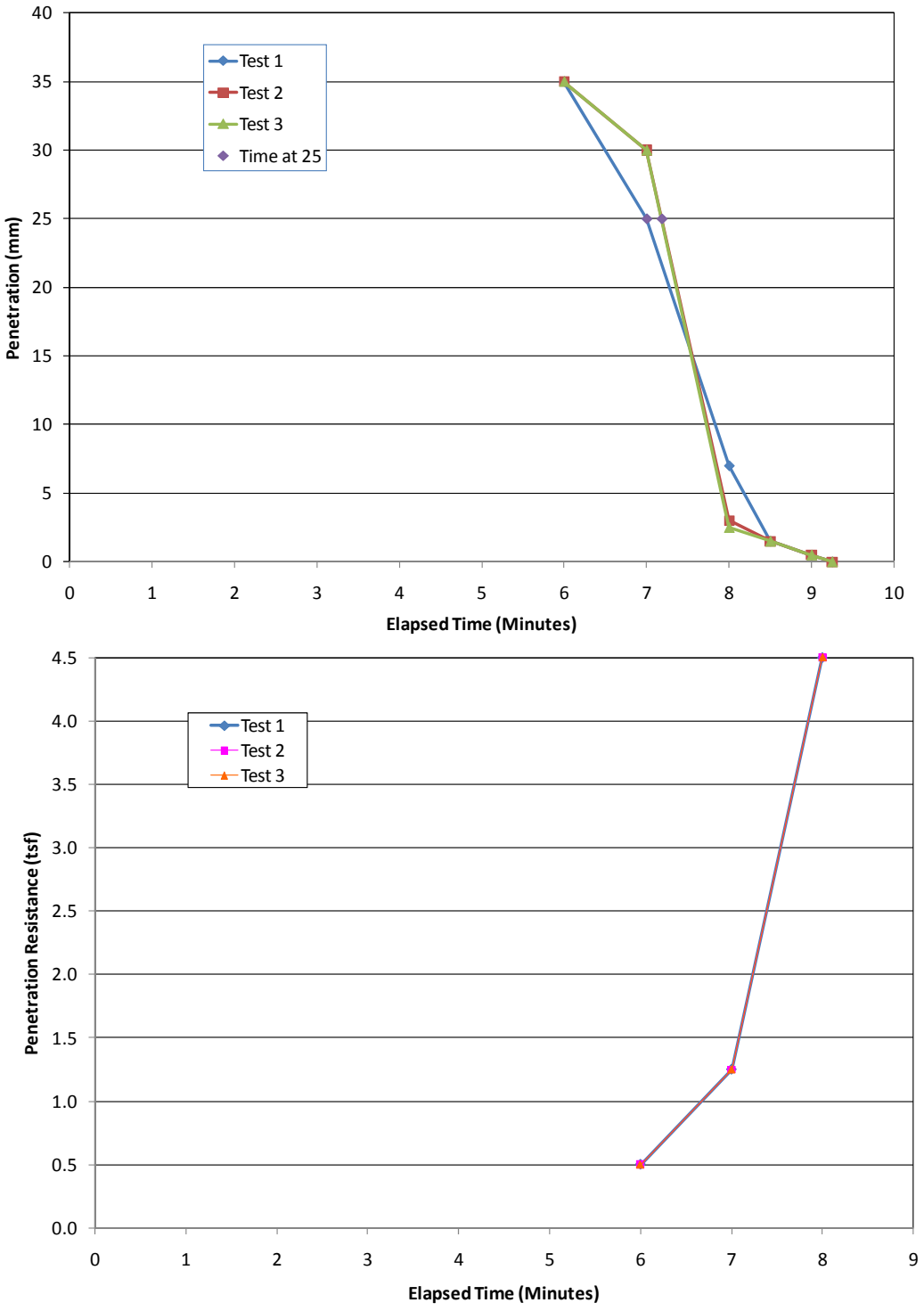
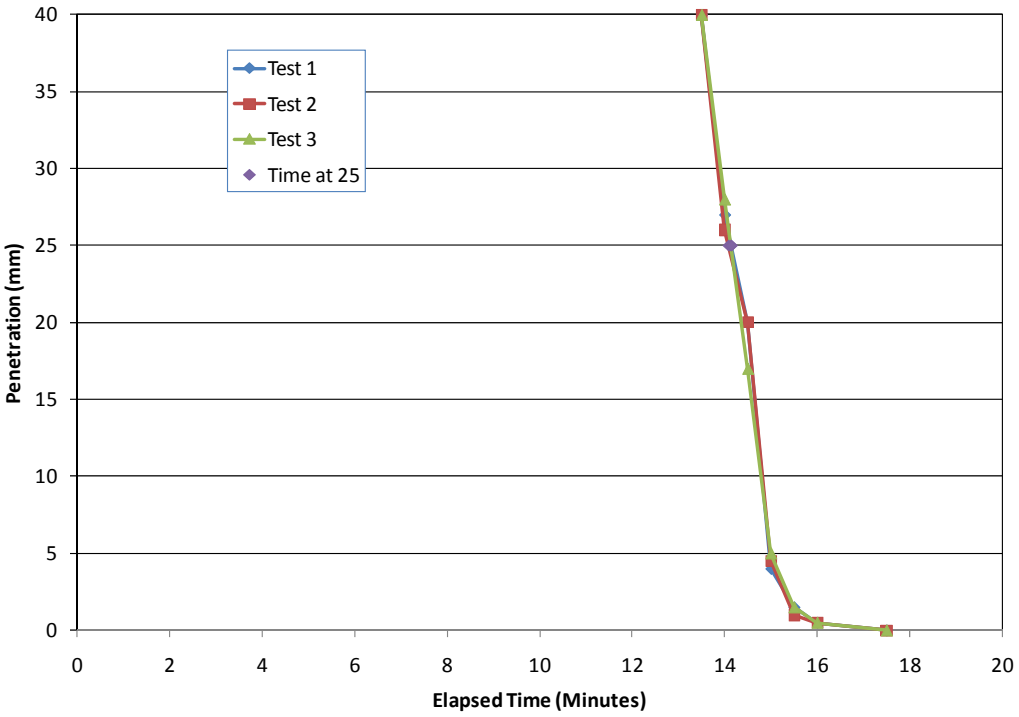



**Figure 160**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 17**



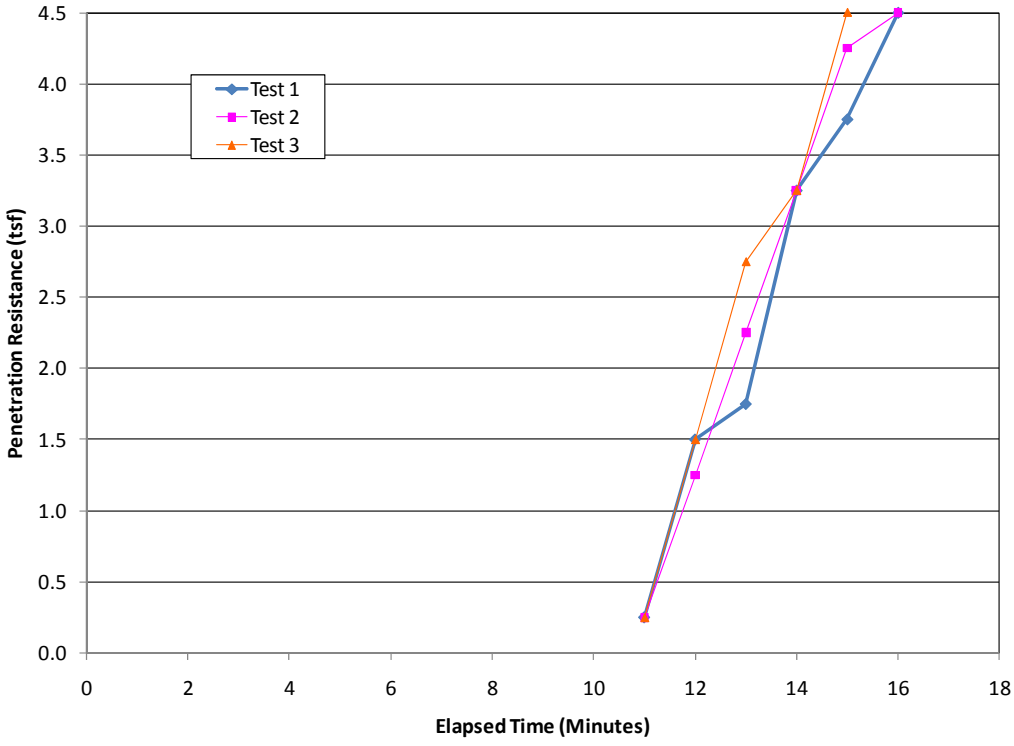




**Figure 161**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 18**

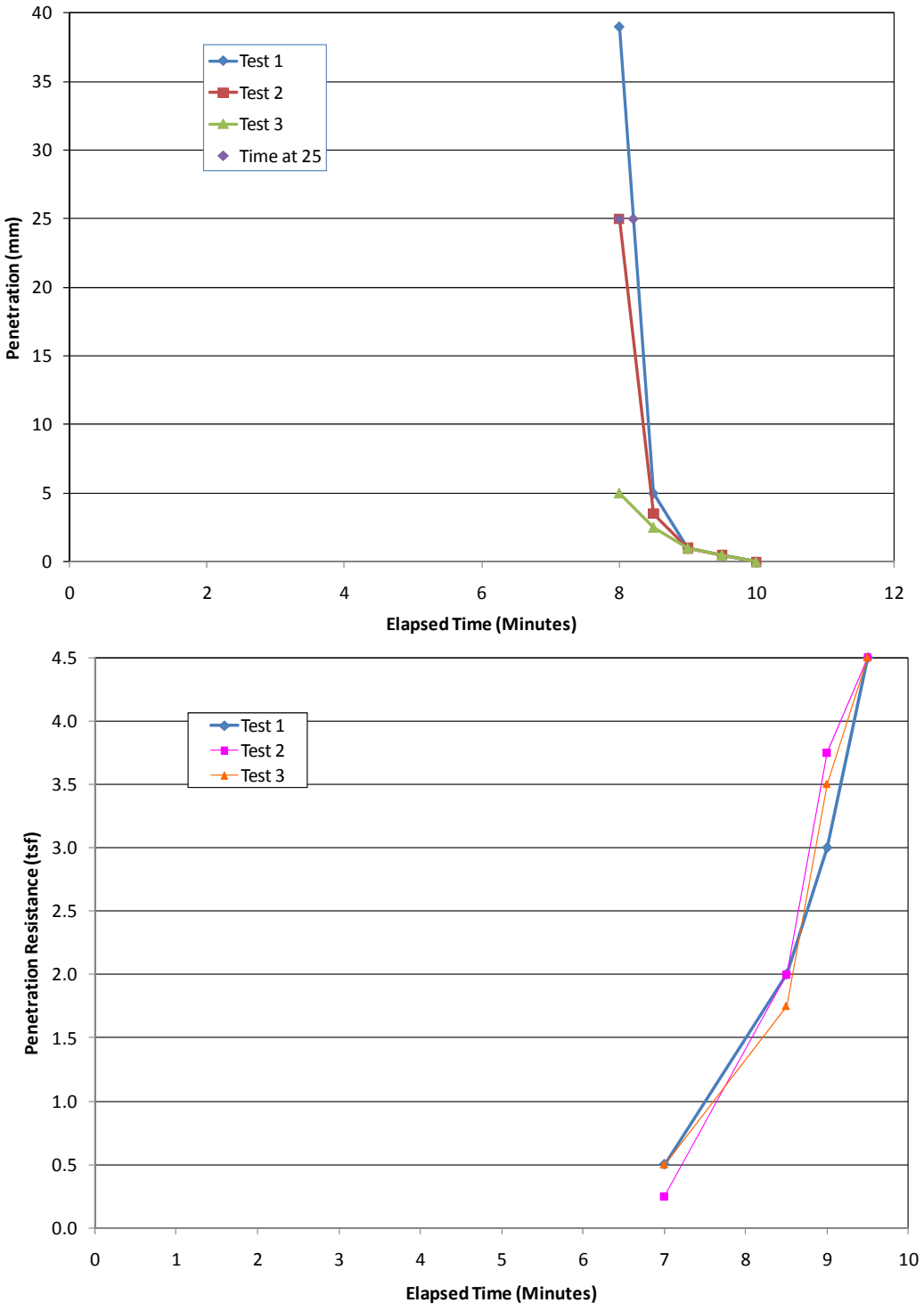



**Figure 162**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 7 Bucket 19**

## Source 8

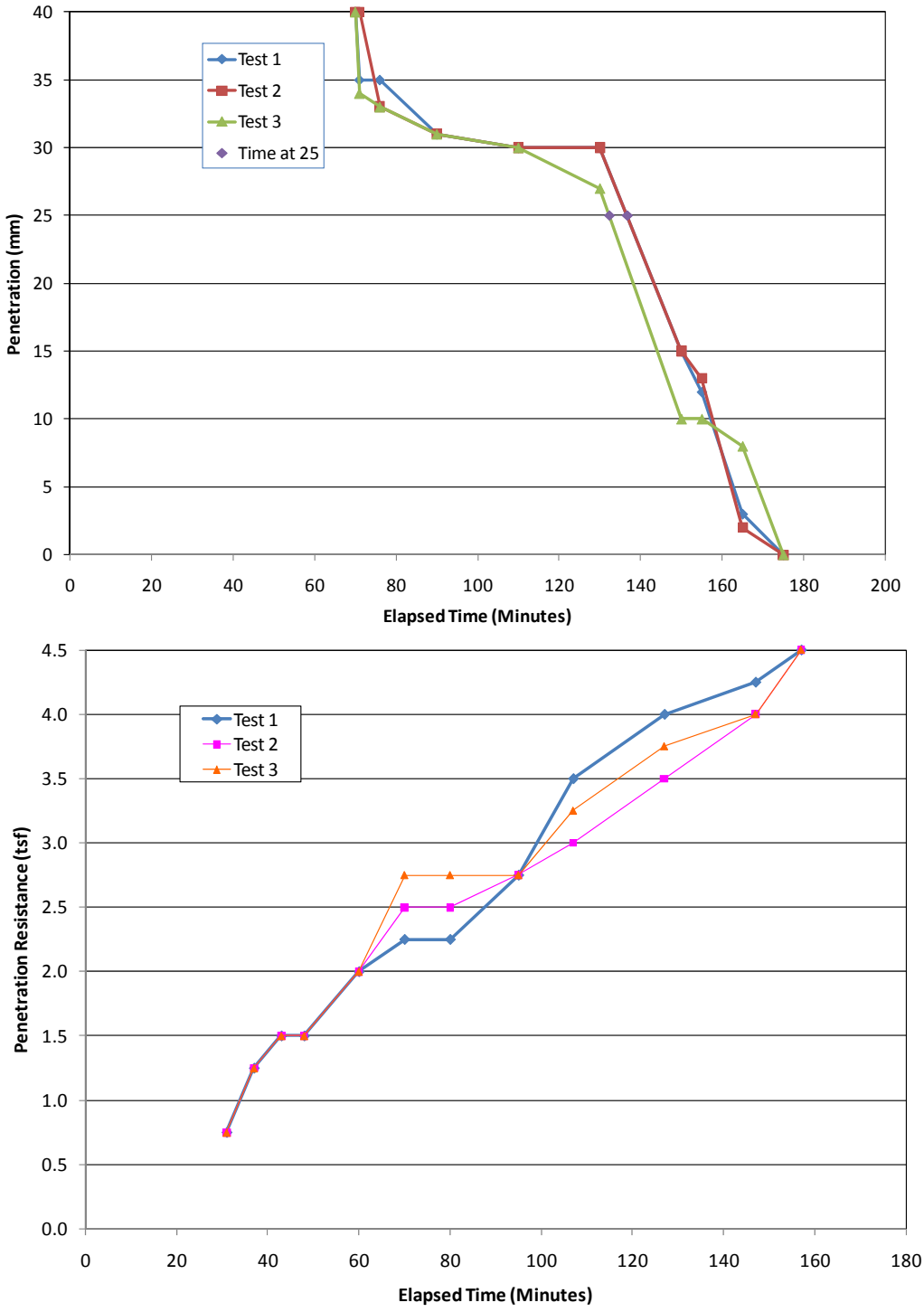



**Figure 163**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 1**

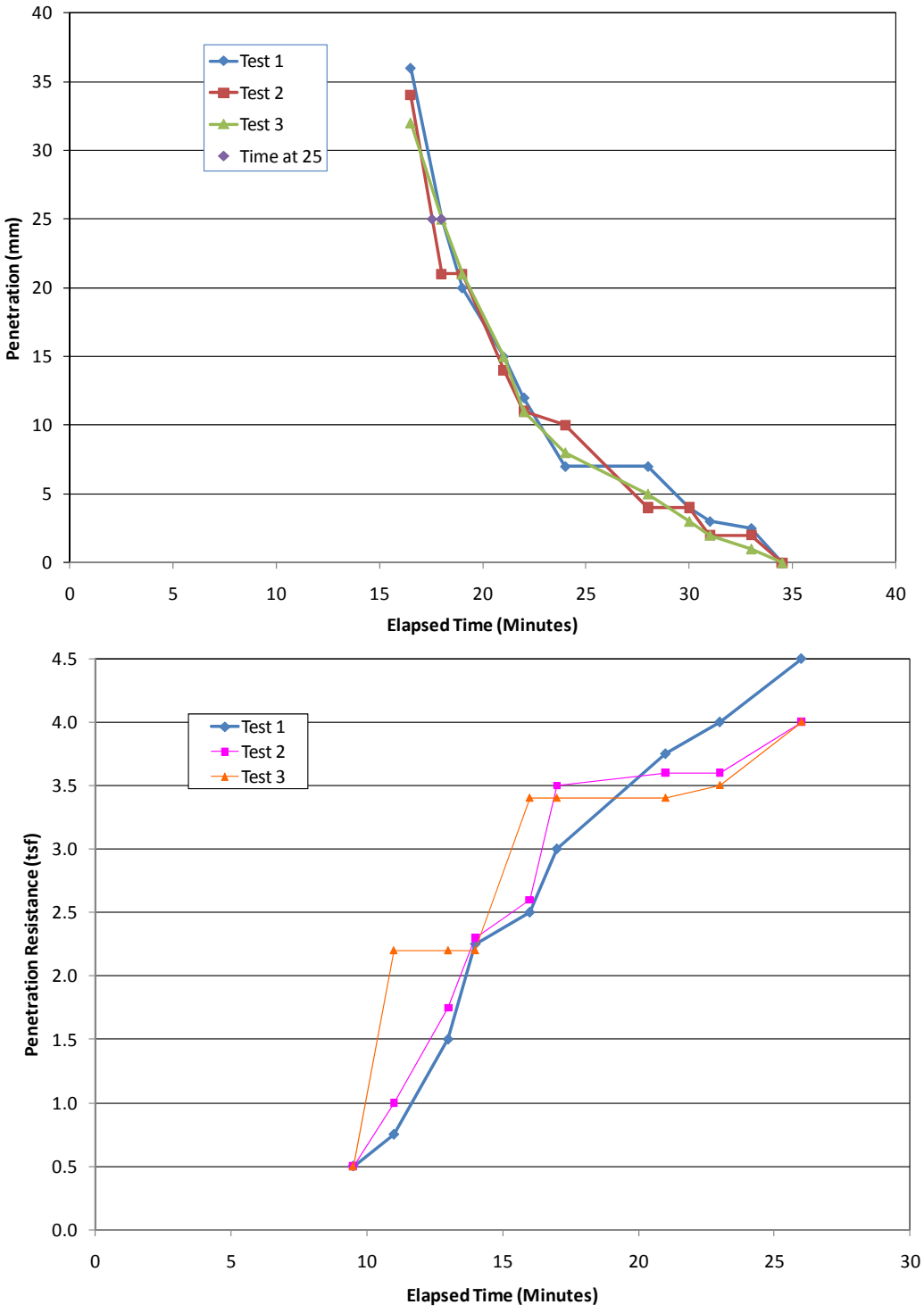



**Figure 164**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 2**

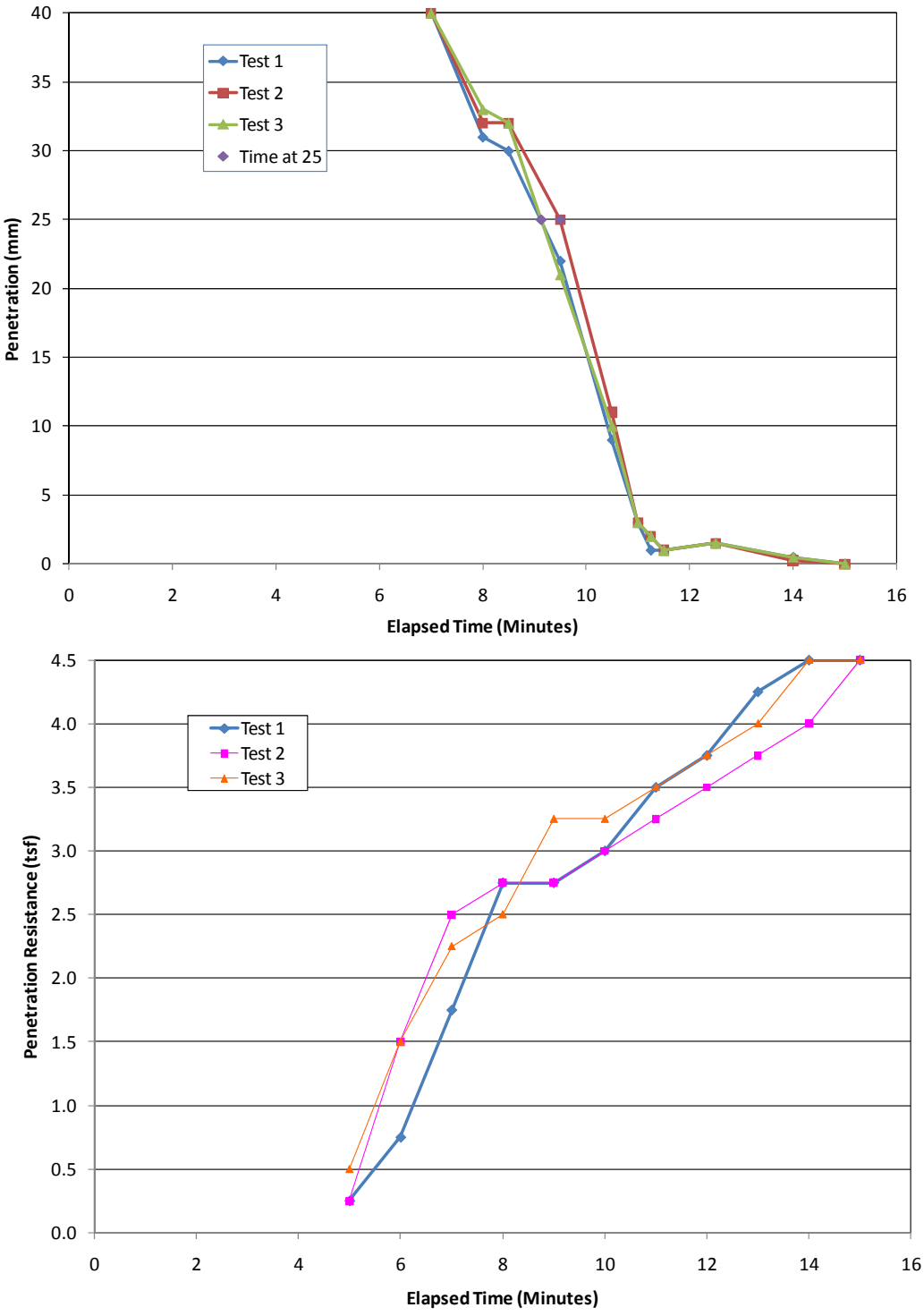



**Figure 165**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 3**

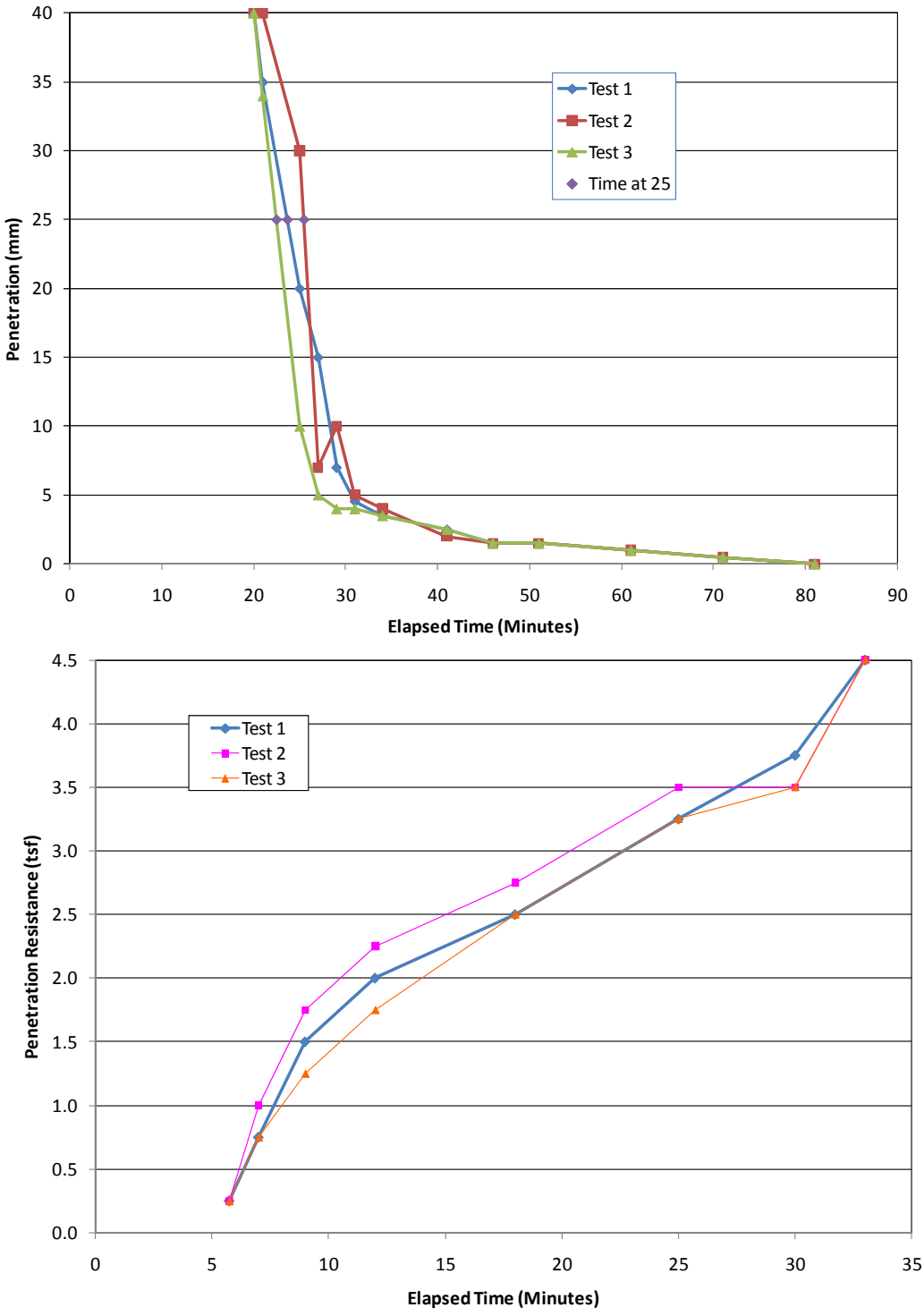



**Figure 166**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 4**

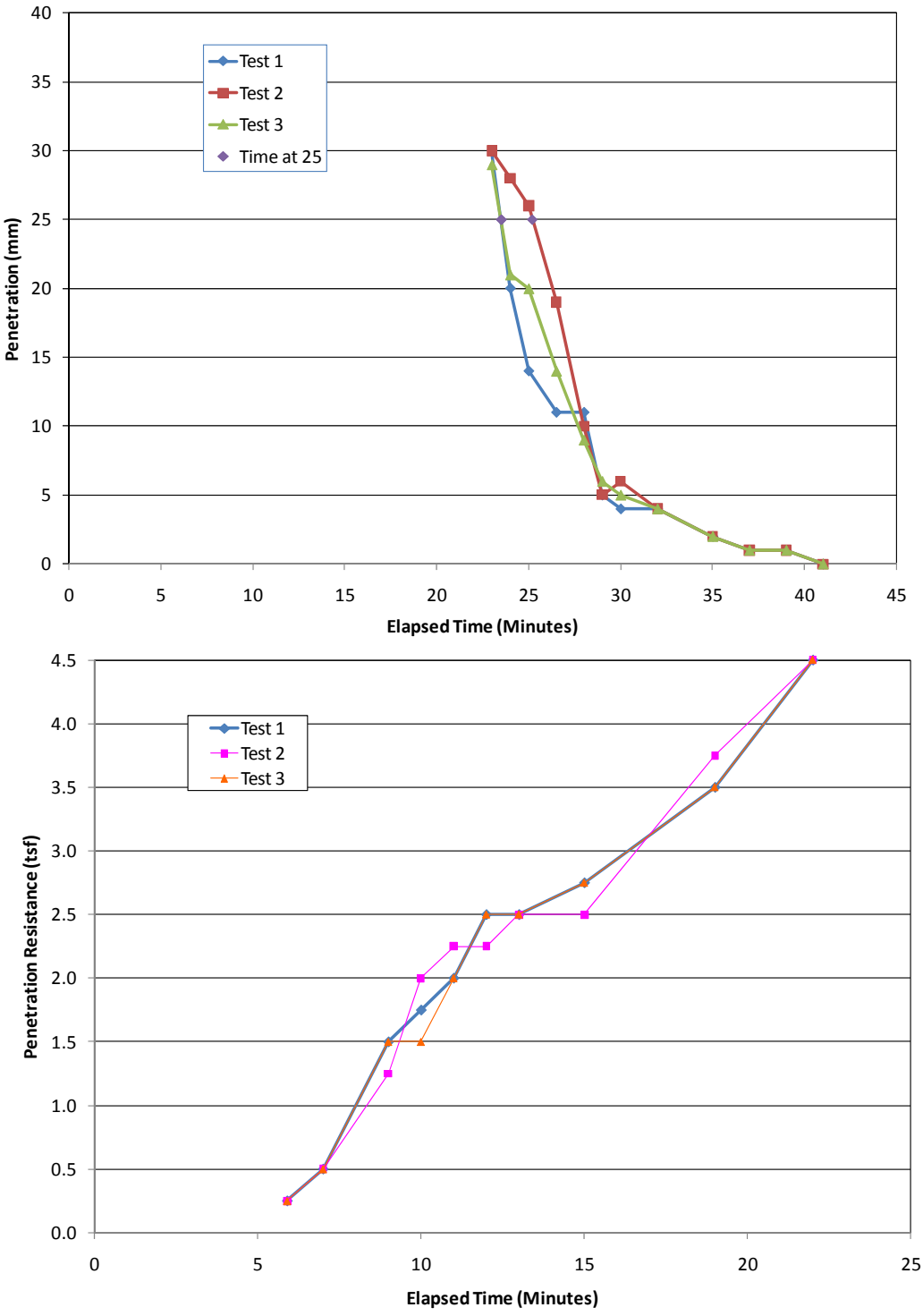



**Figure 167**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 5**

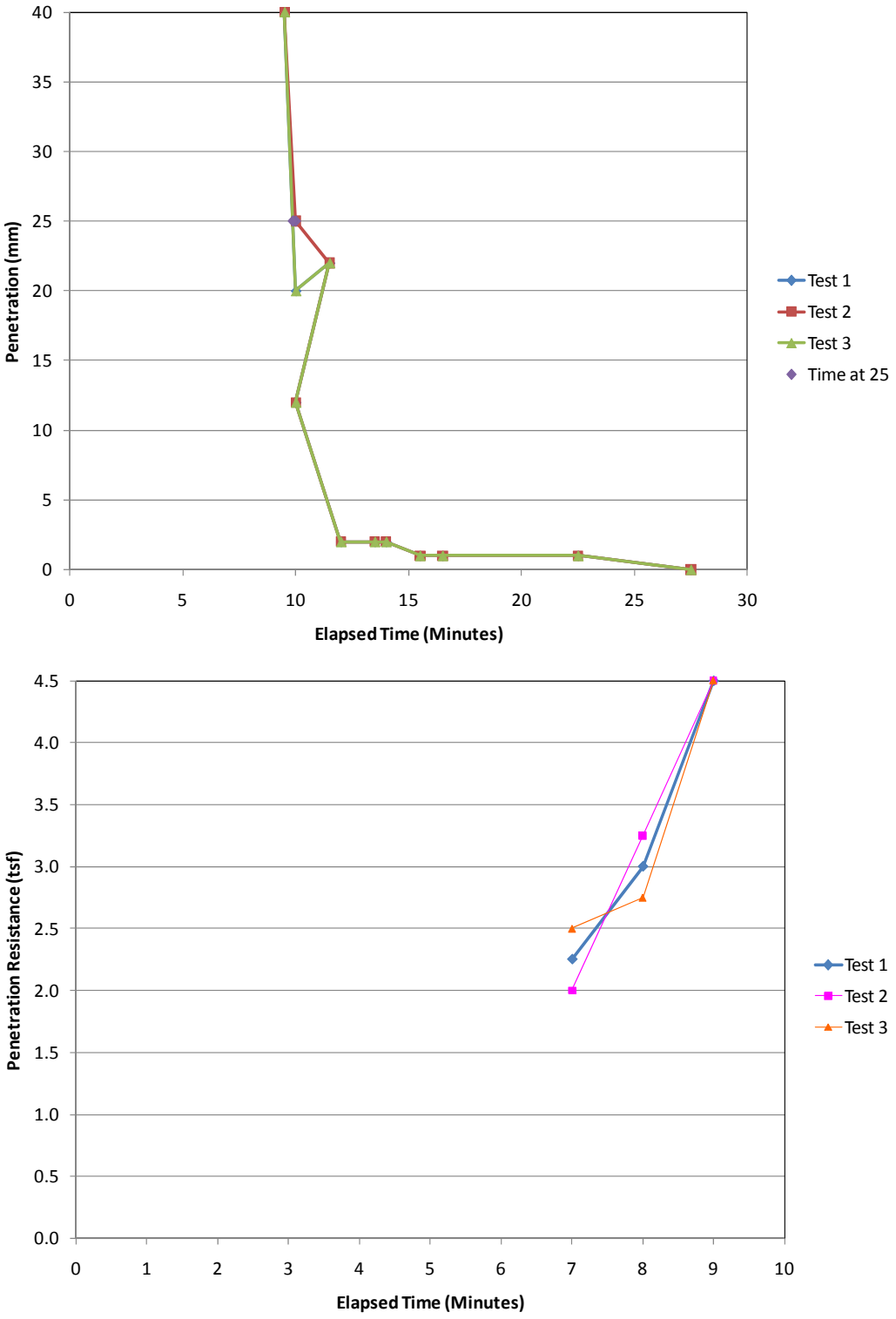



**Figure 168**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 6**

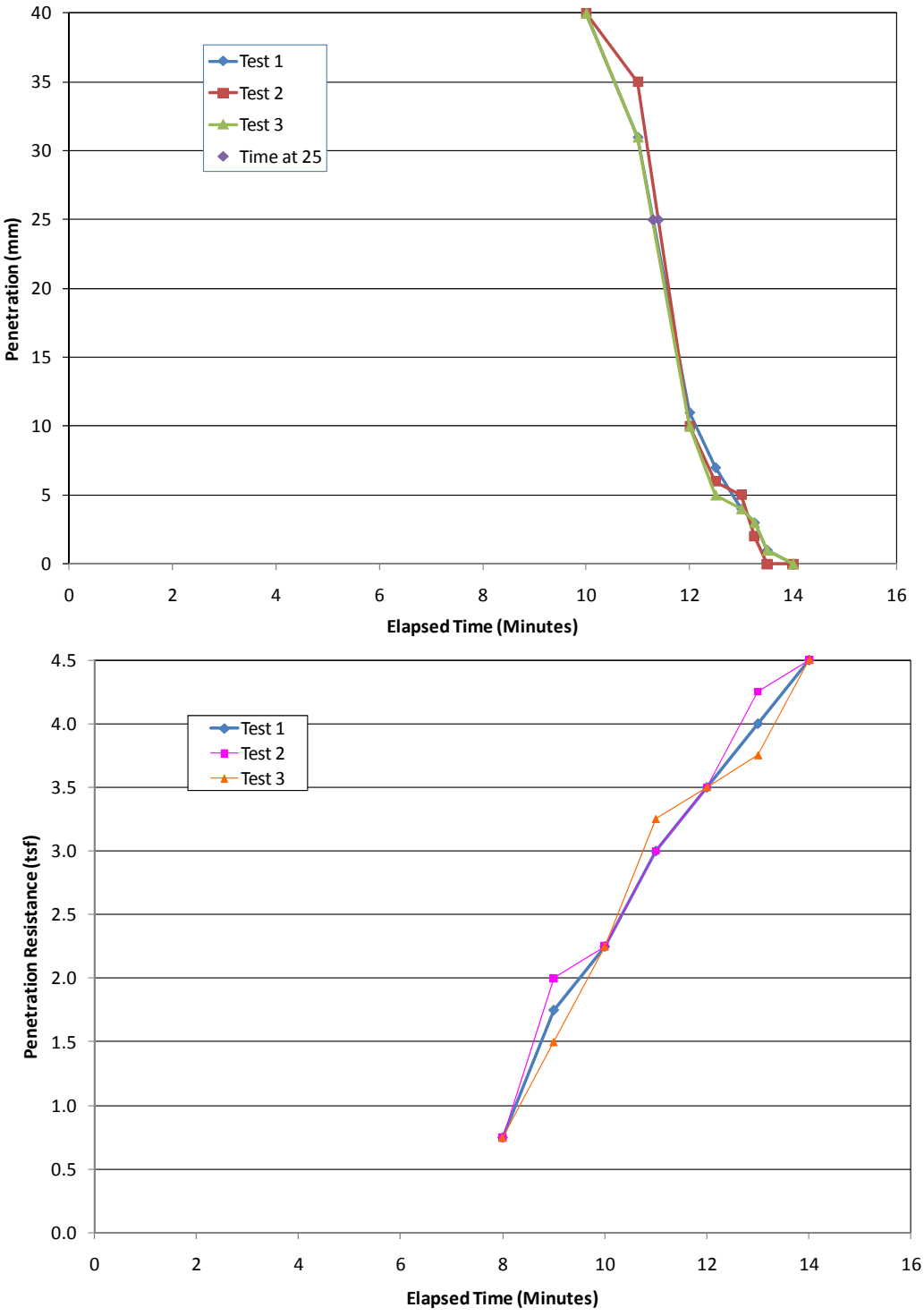



**Figure 169**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 7**

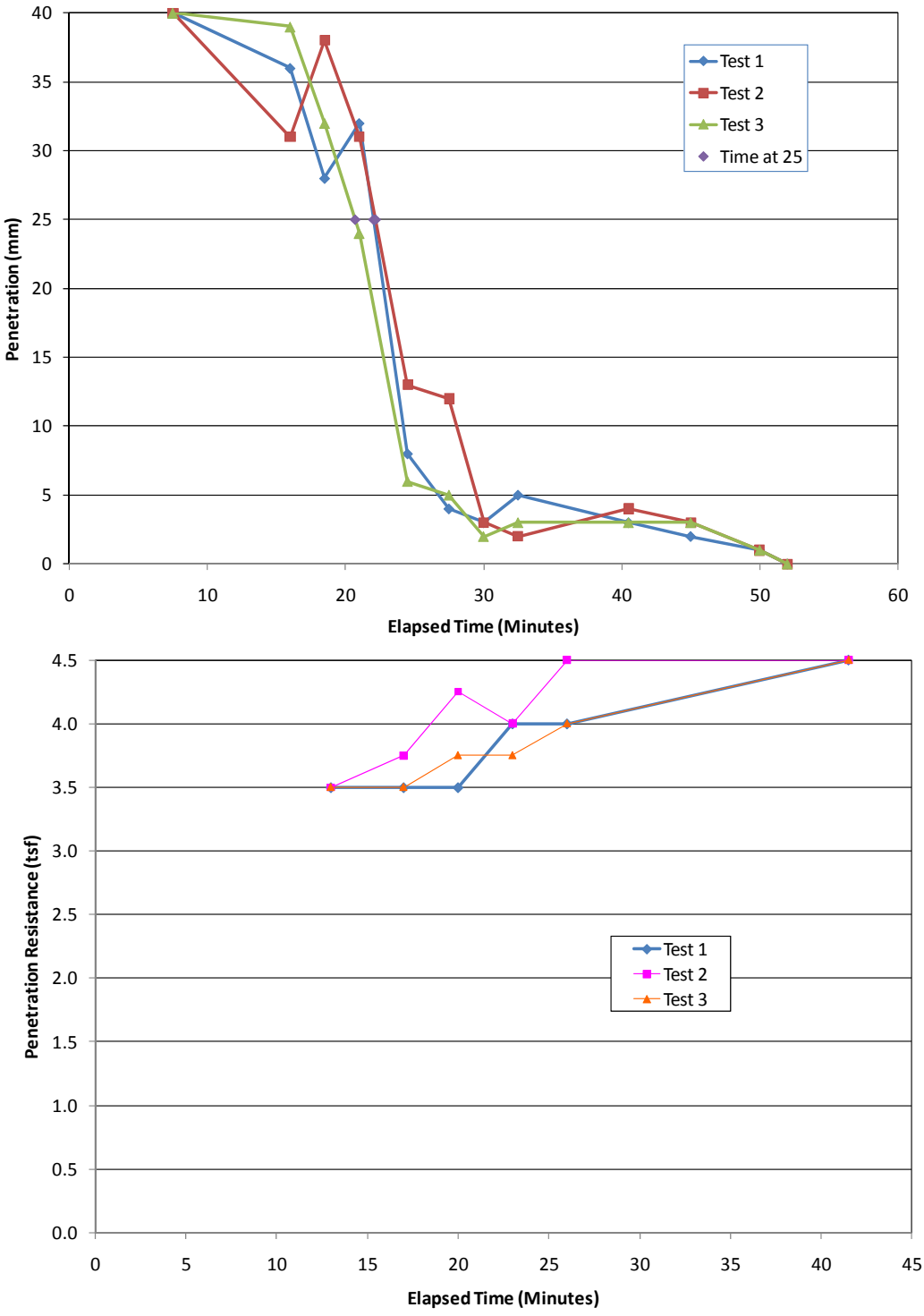



**Figure 170**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 8**

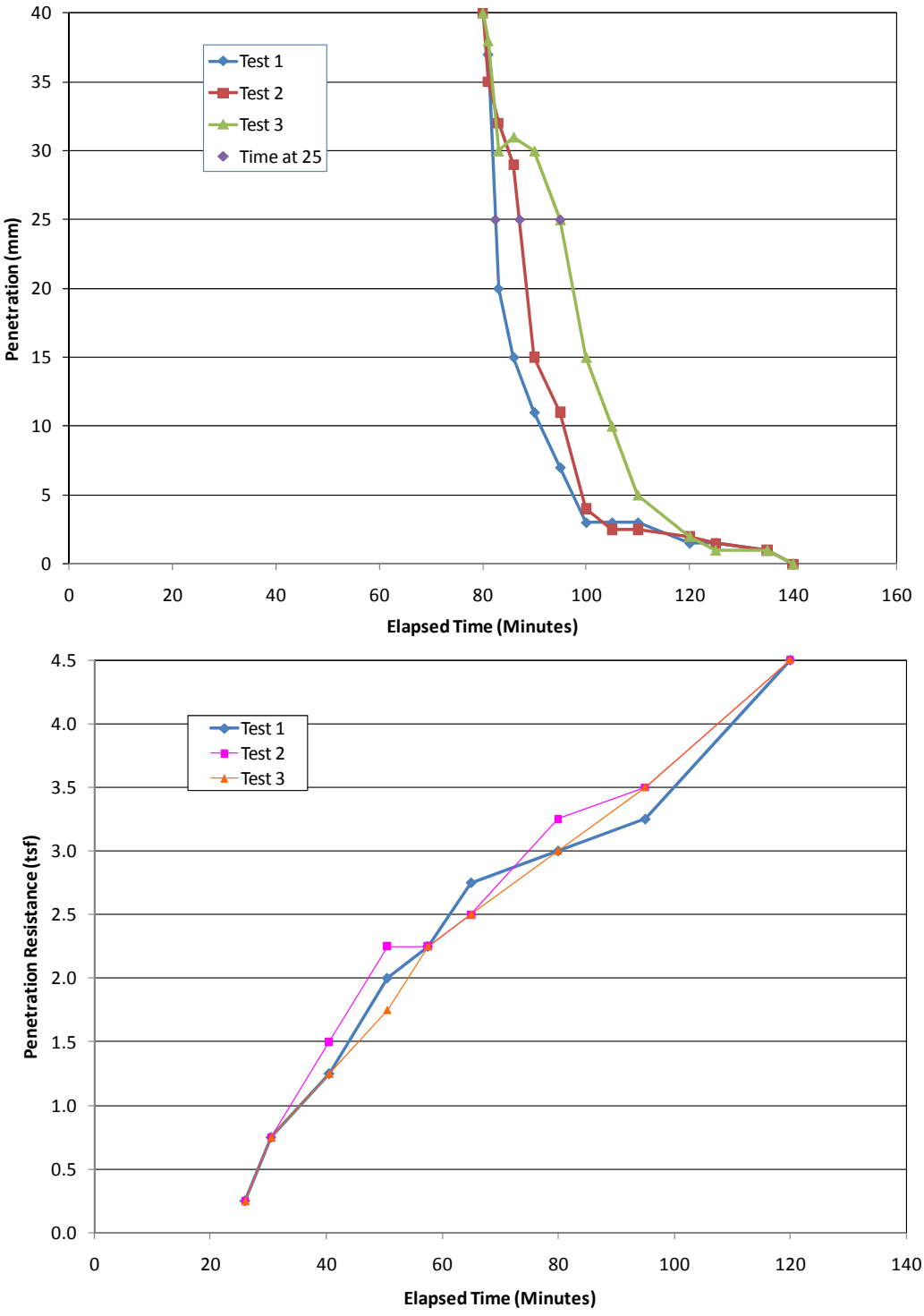
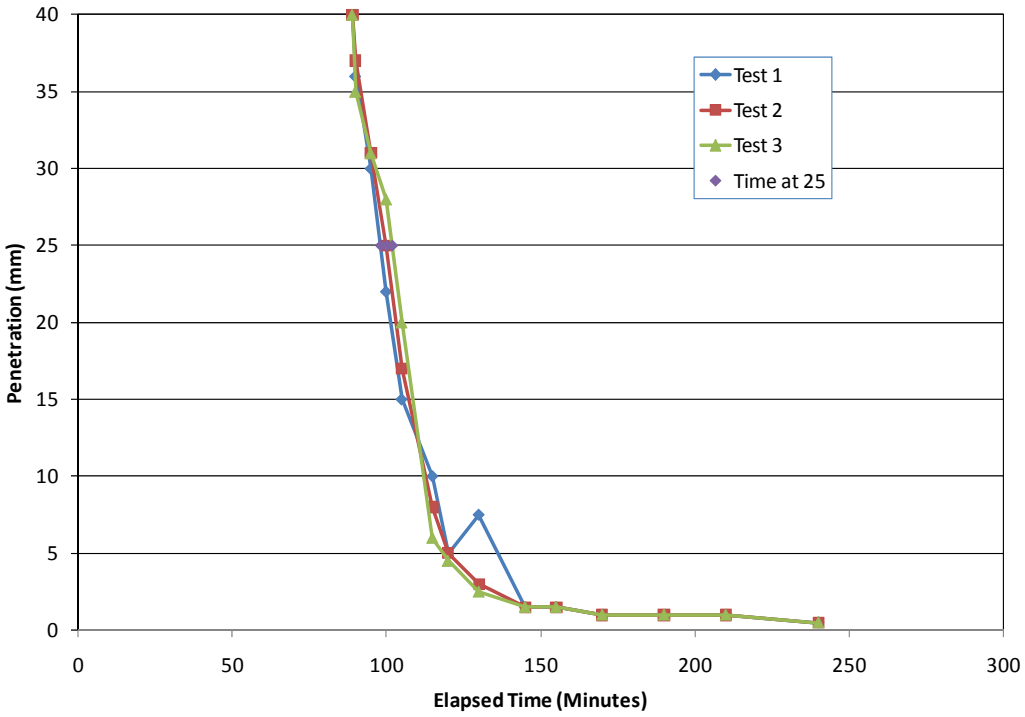



**Figure 171**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 9**



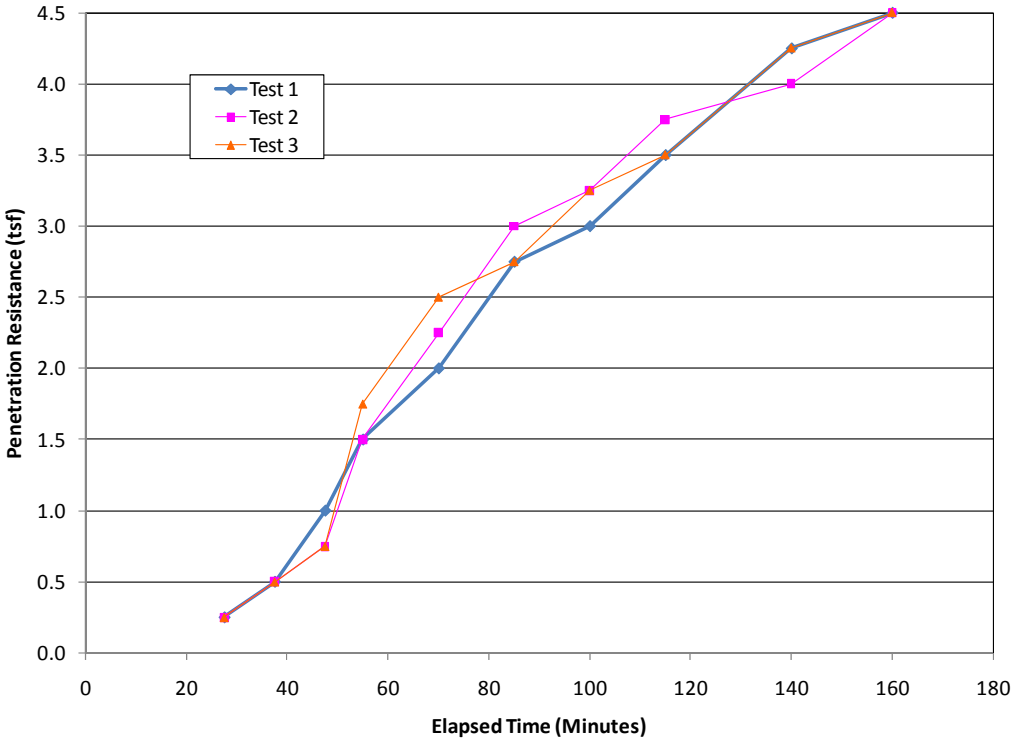




**Figure 172**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 10**

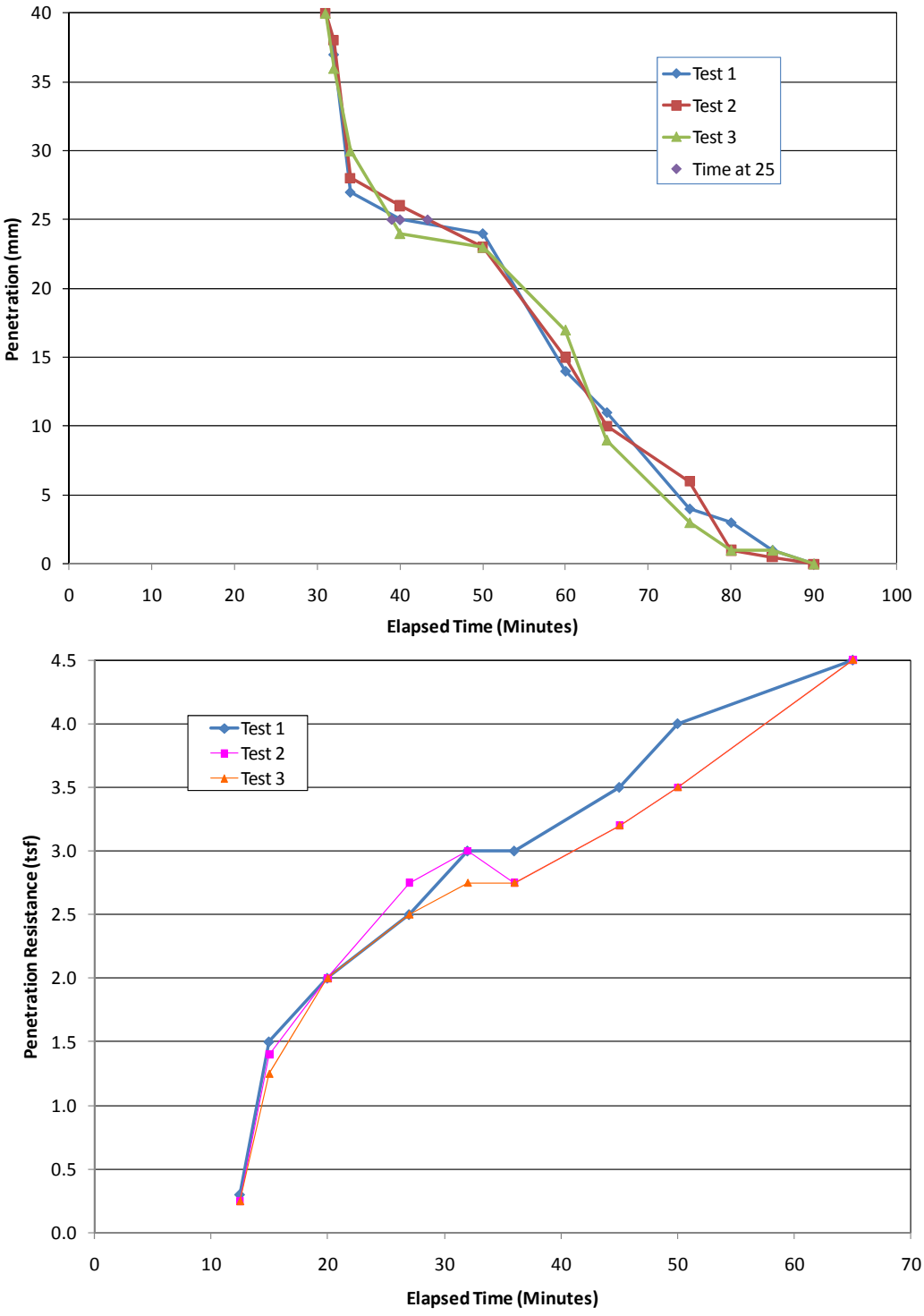



**Figure 173**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 11**

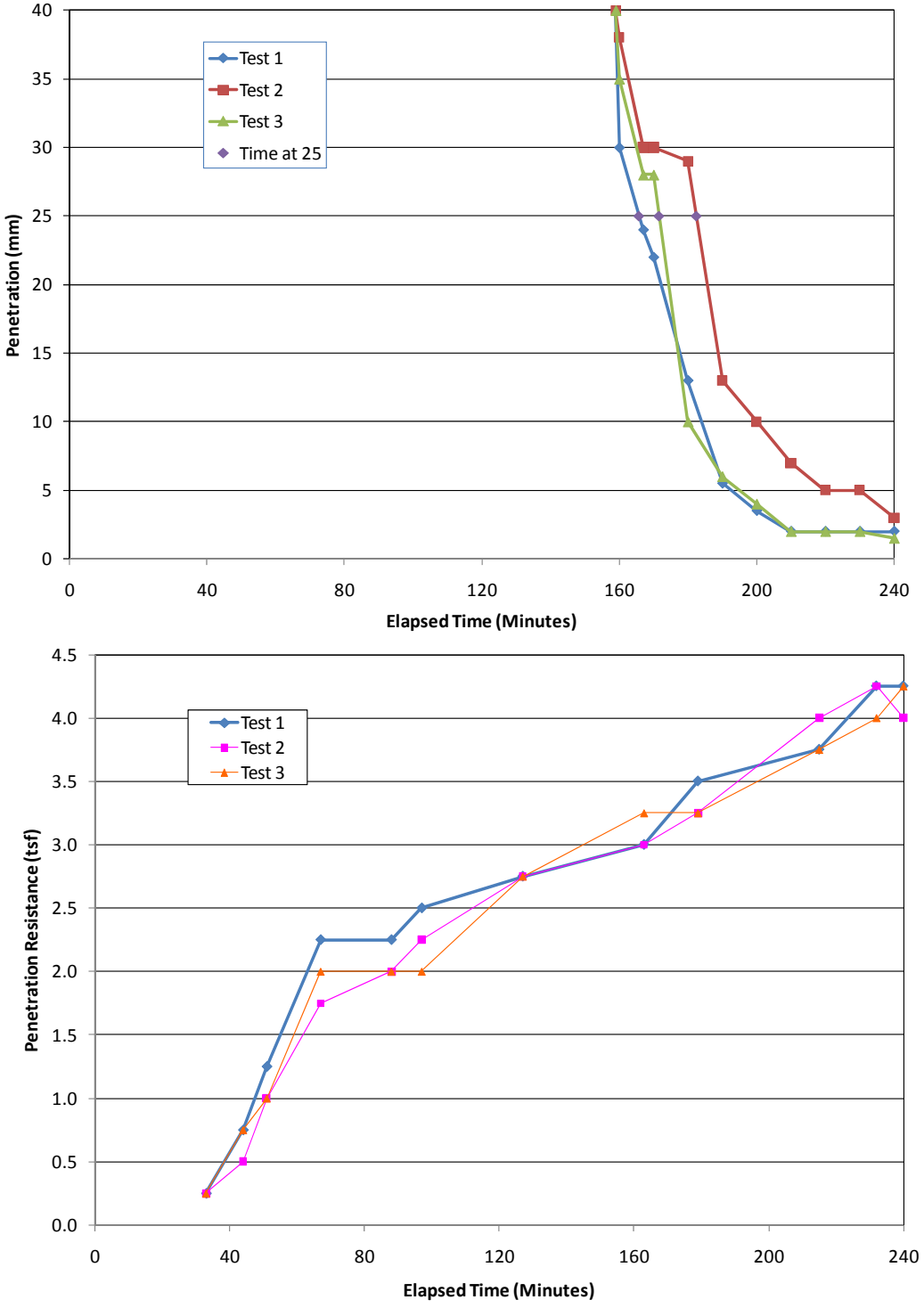
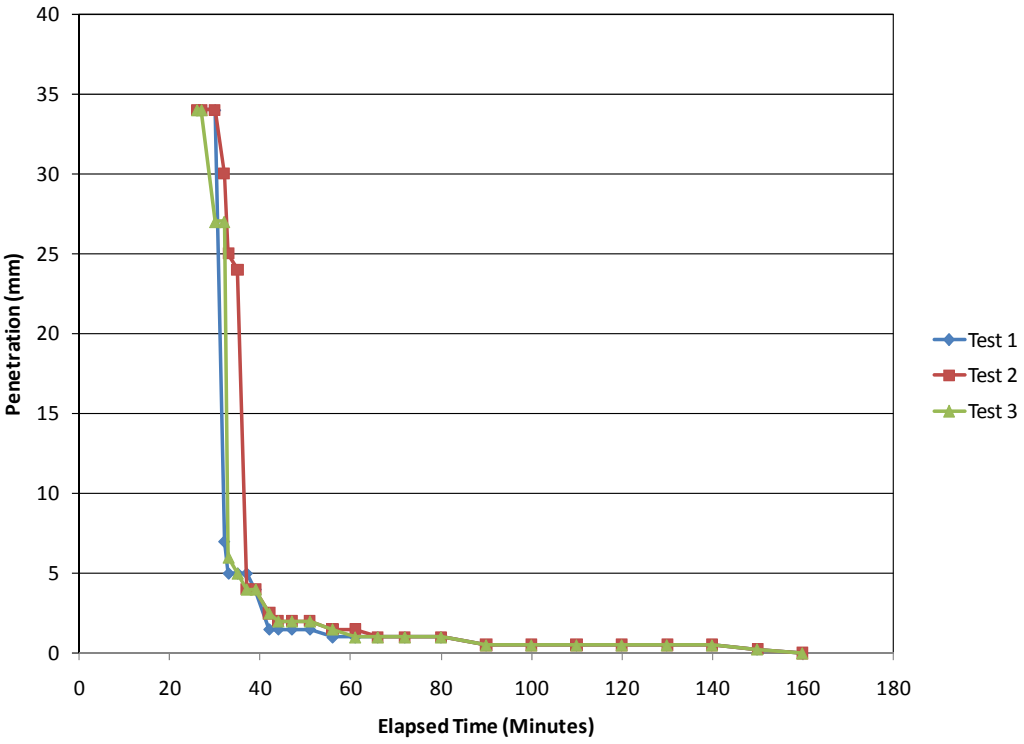



**Figure 174**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 12**



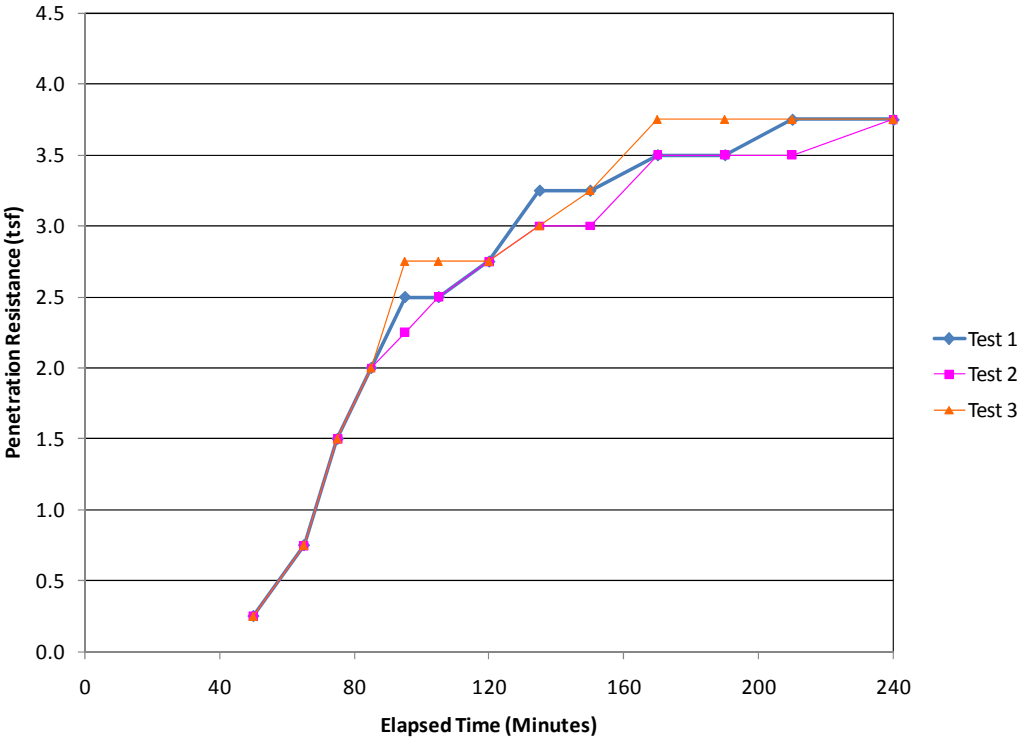




**Figure 175**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 13**

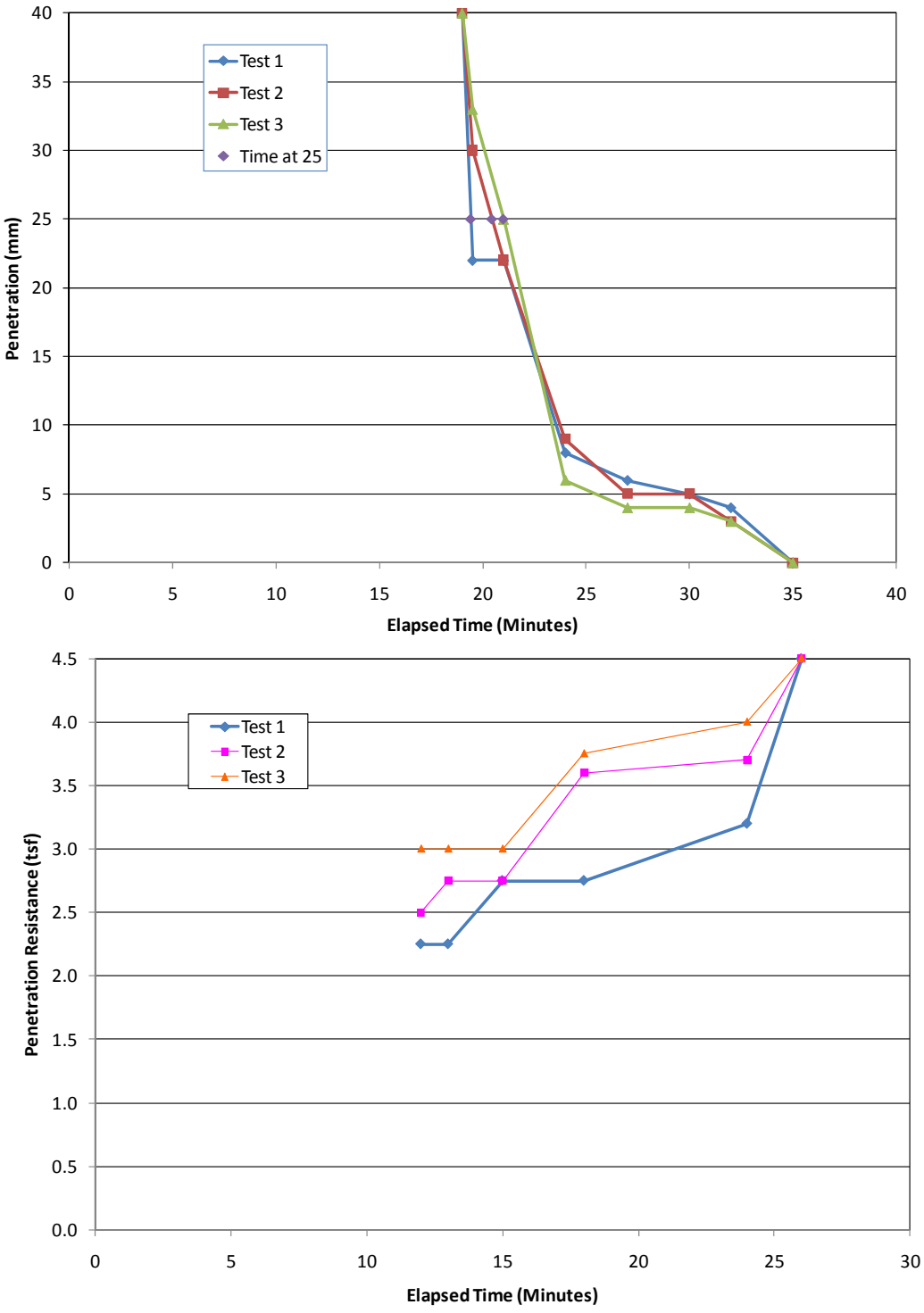
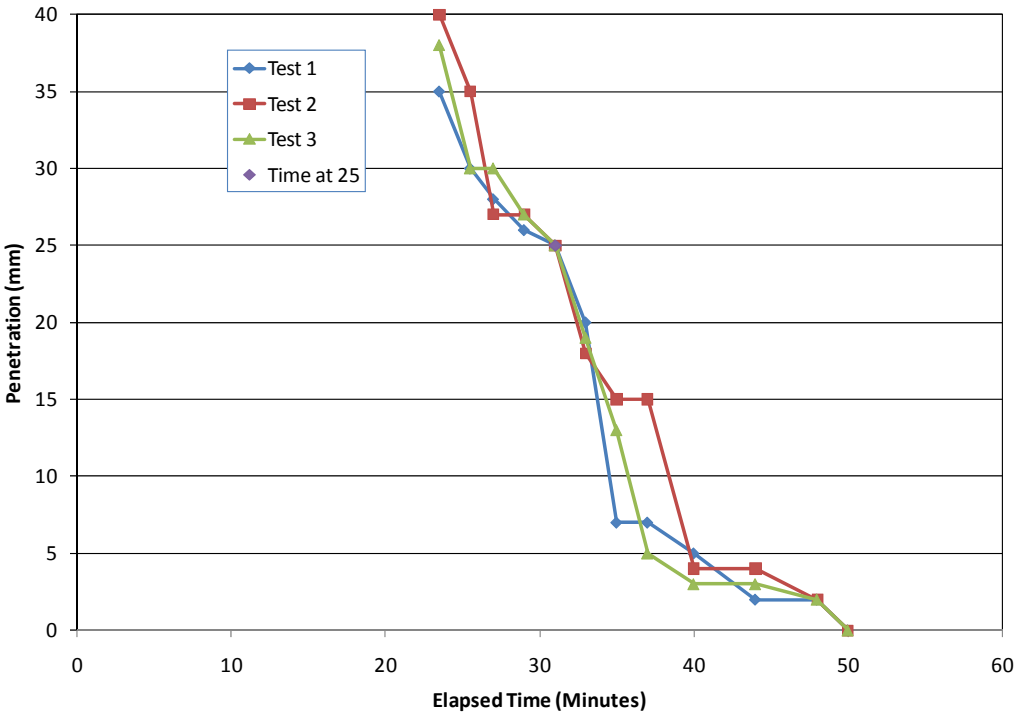



**Figure 176**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 14**



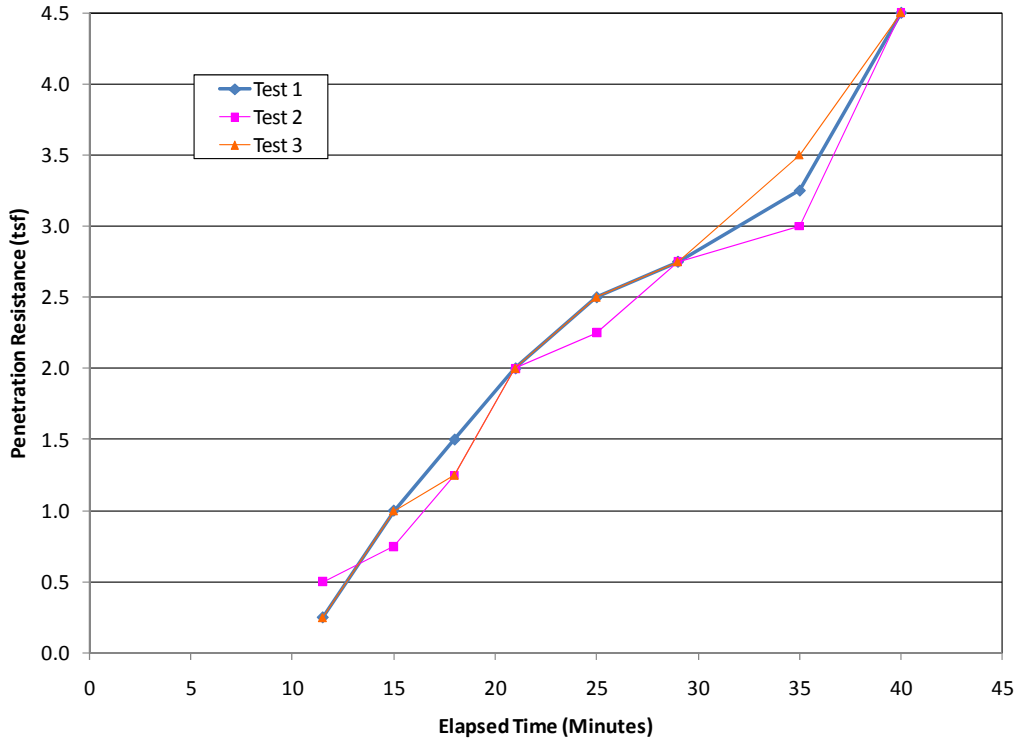
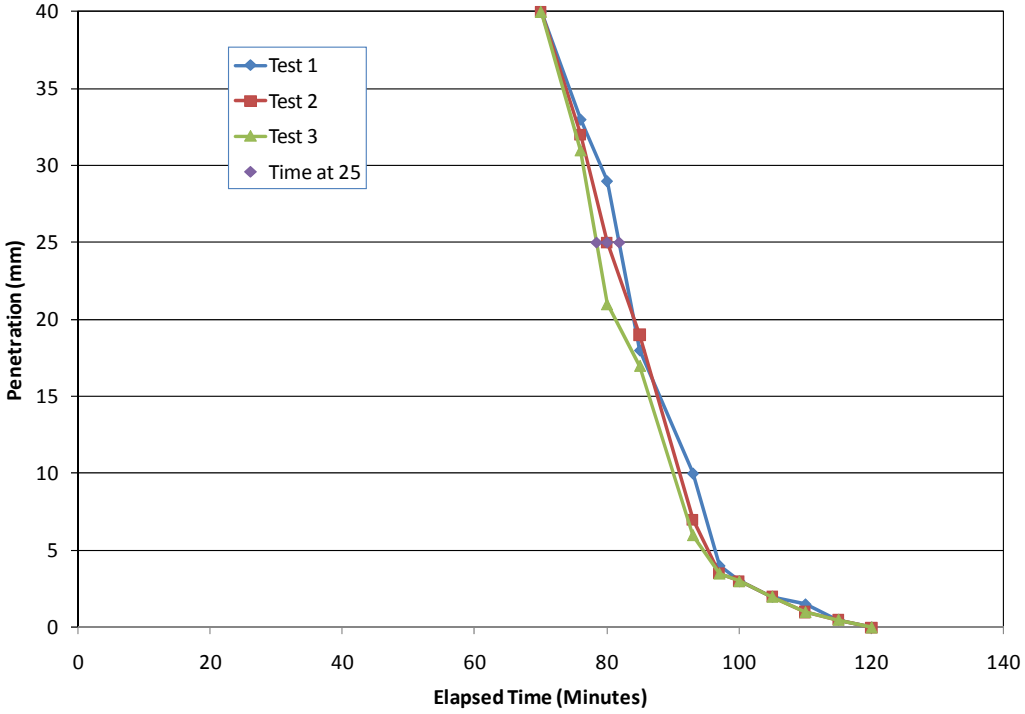




**Figure 177**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 15**



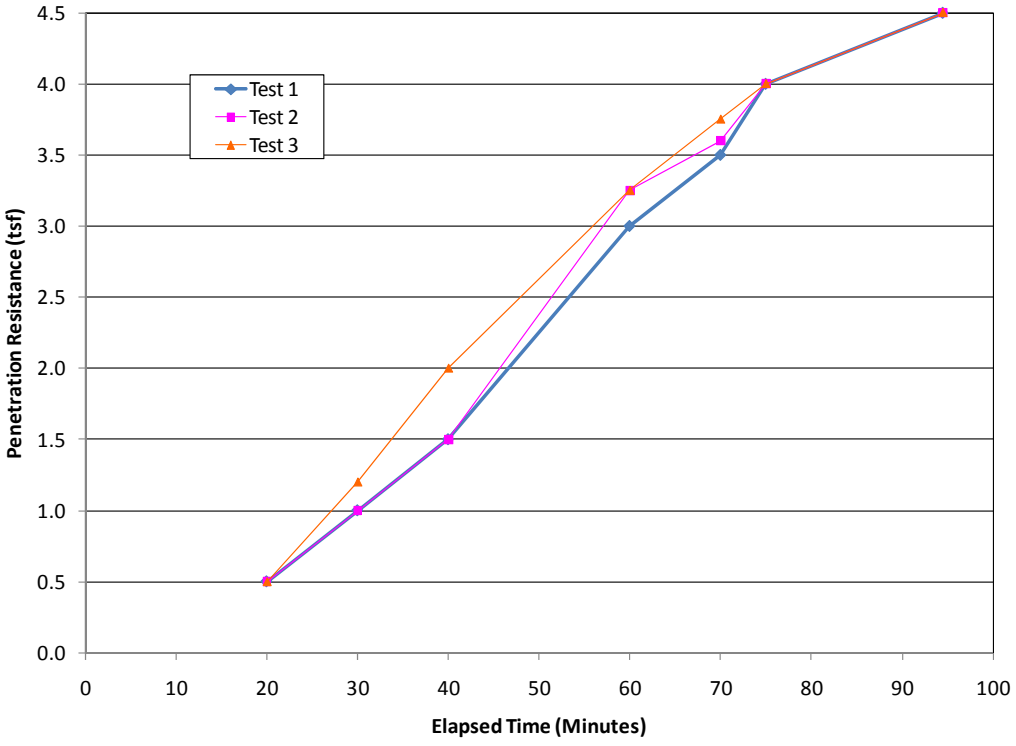
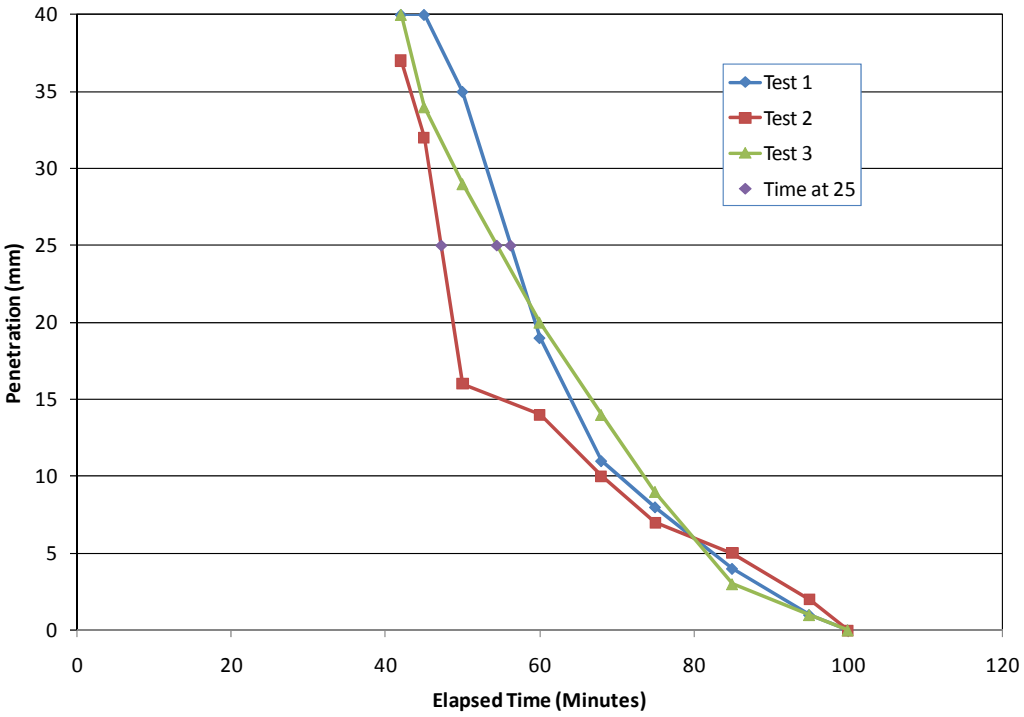




**Figure 178**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 16**



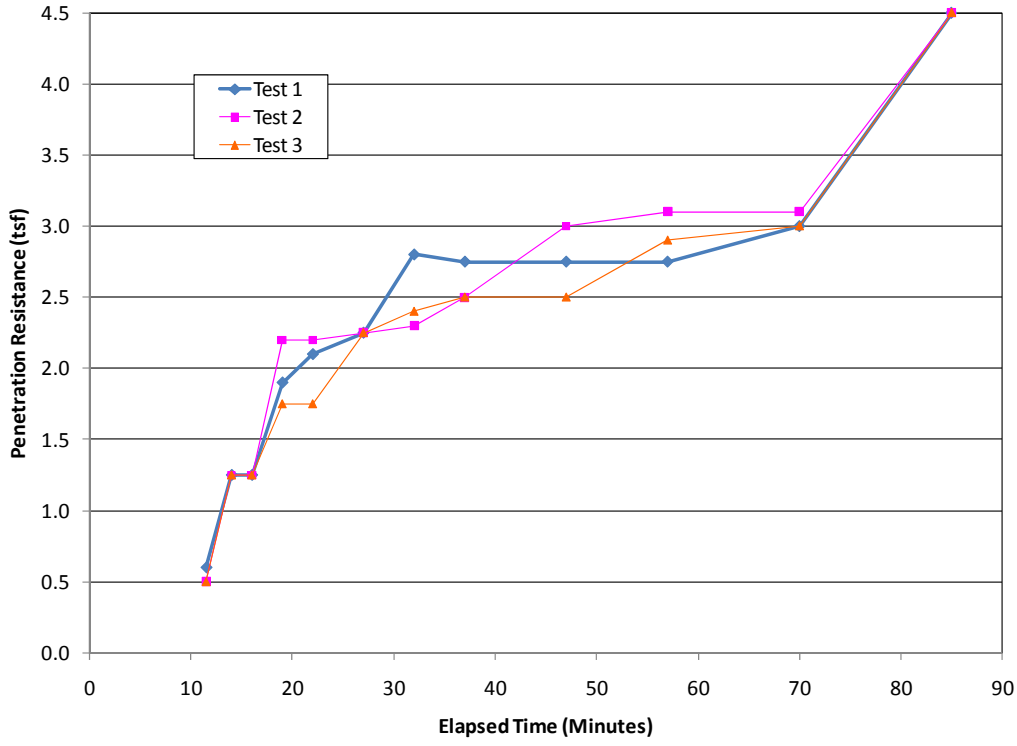




**Figure 179**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 17**

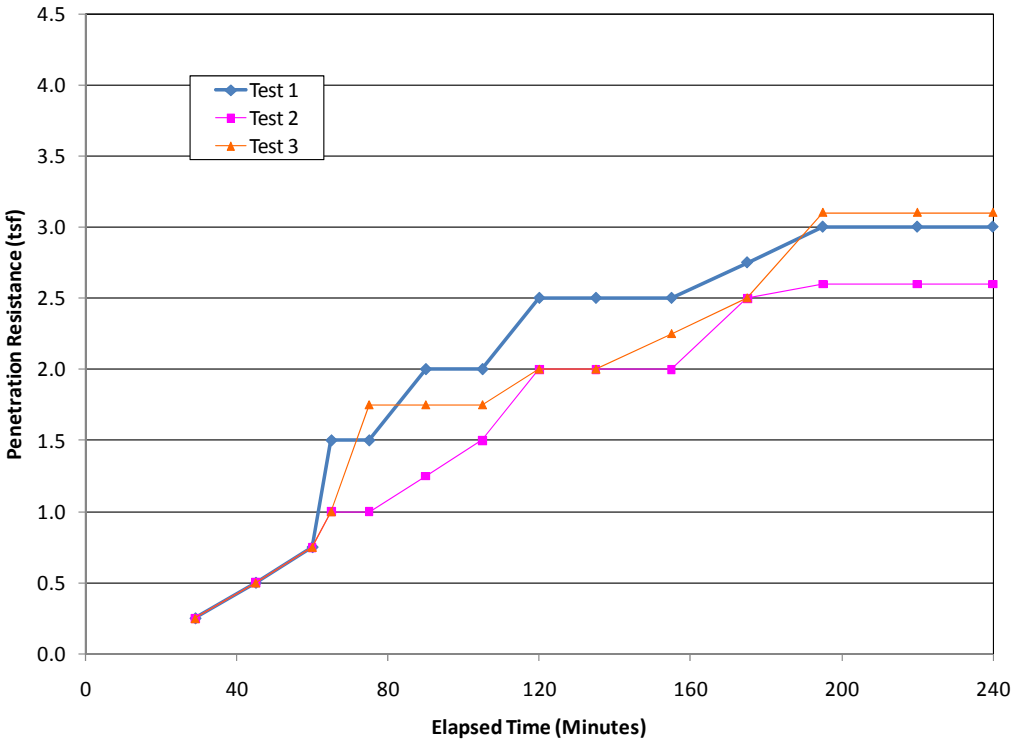



**Figure 180**
**Pocket penetration resistance graph for Source 8 Bucket 18**

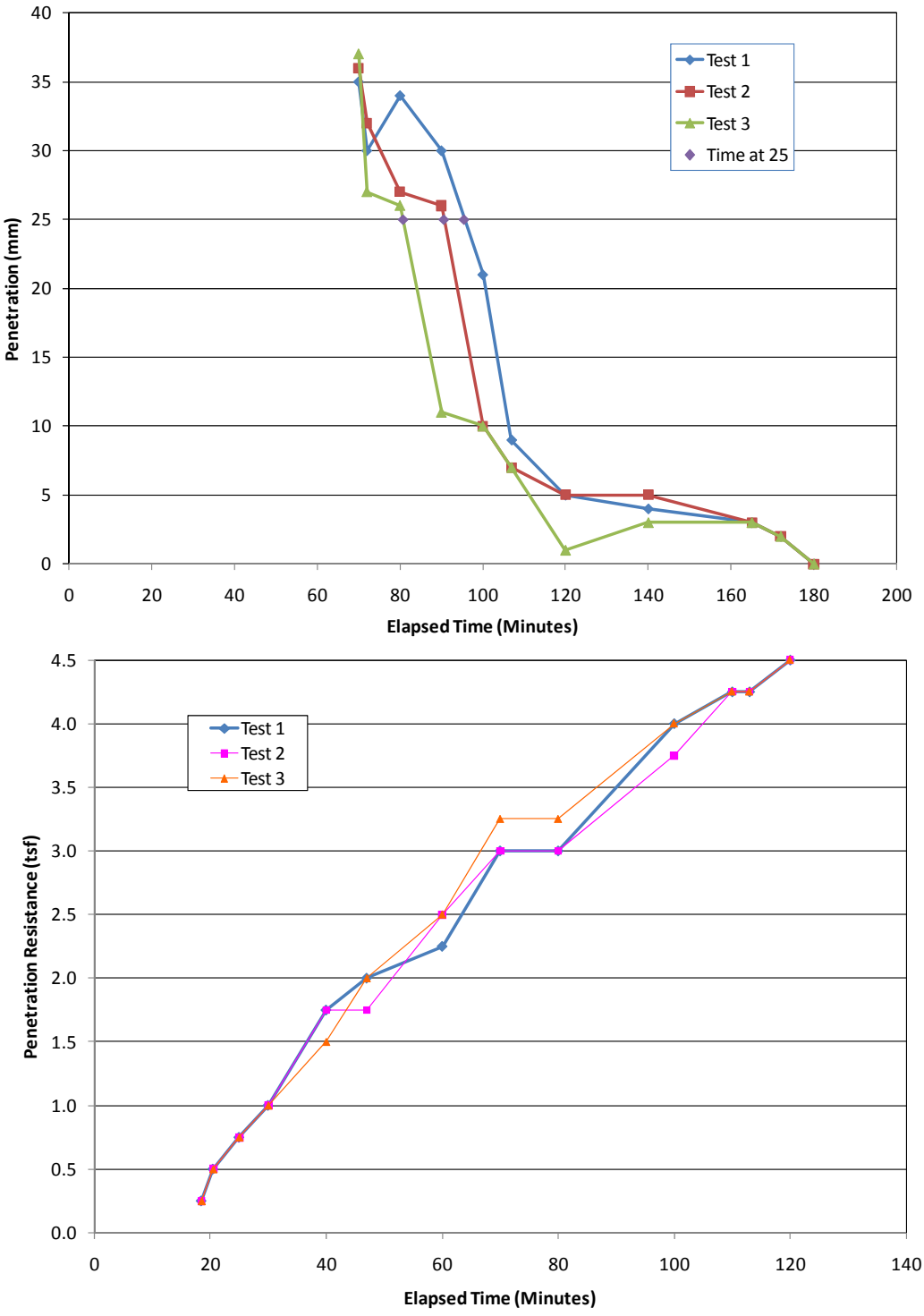



**Figure 181**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 19**

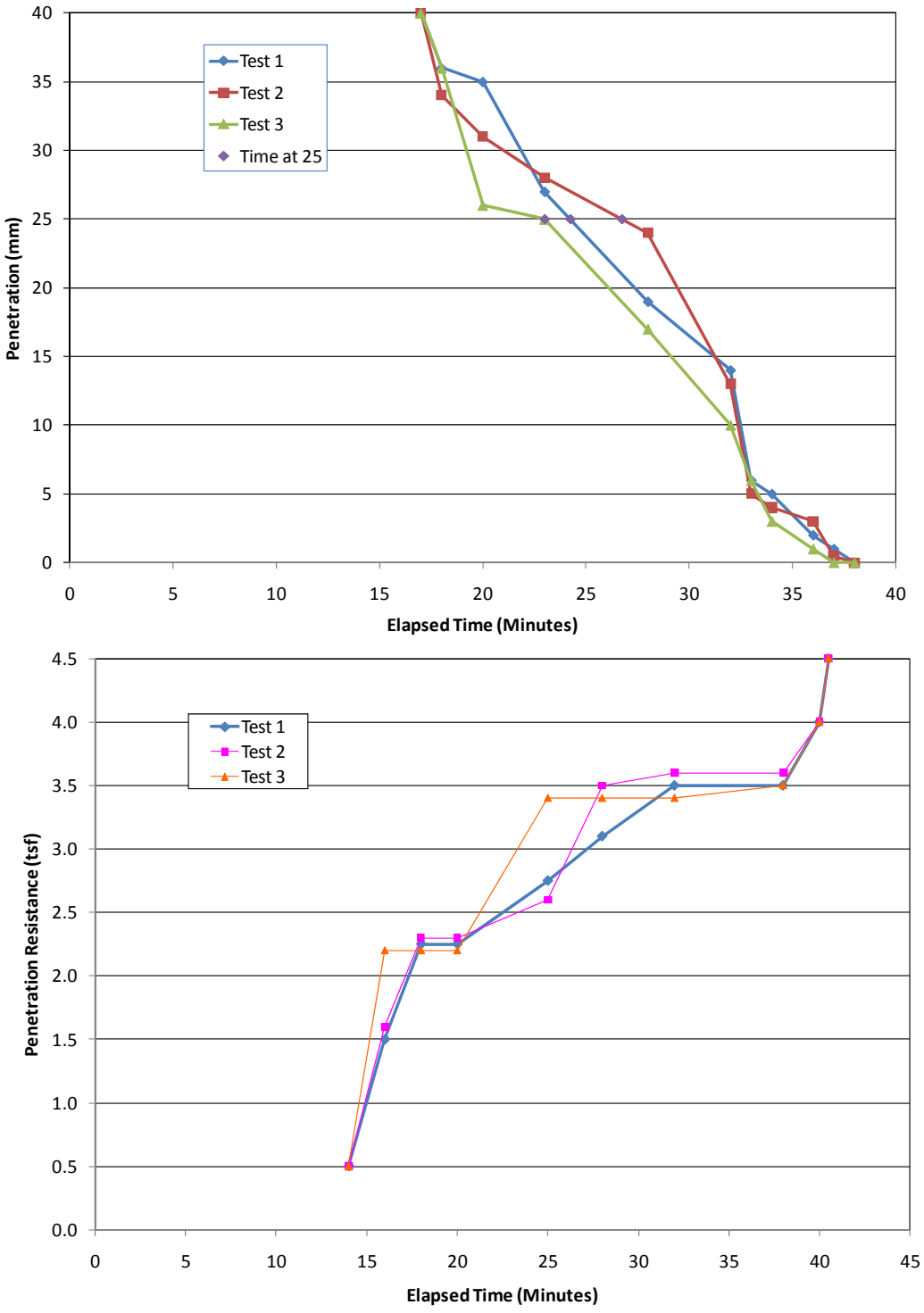
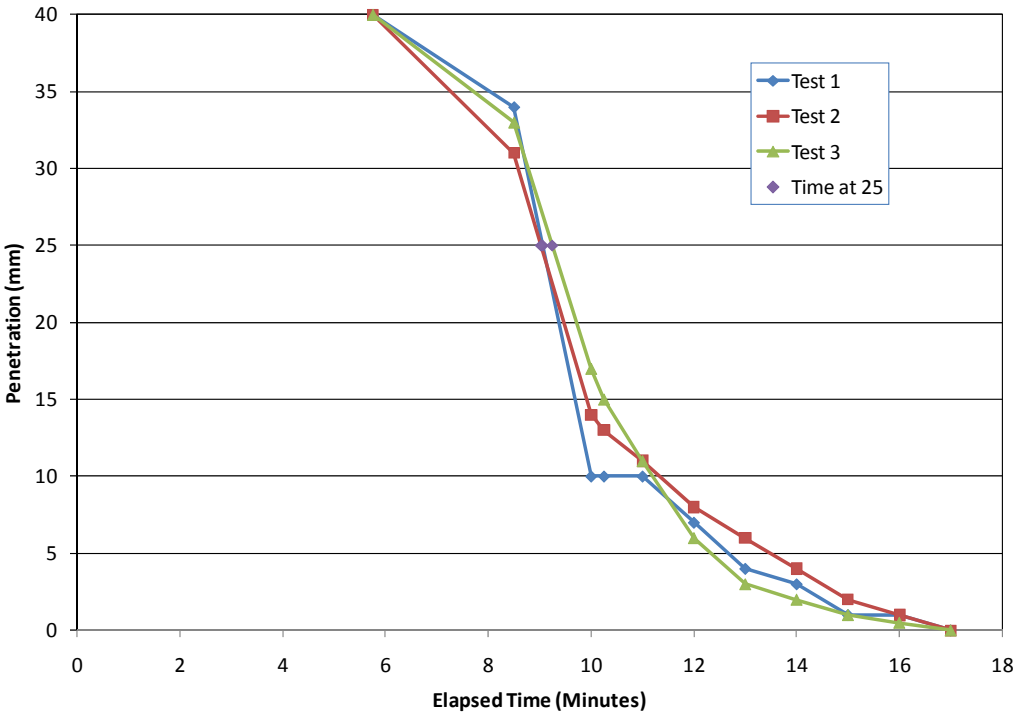



**Figure 182**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 20**



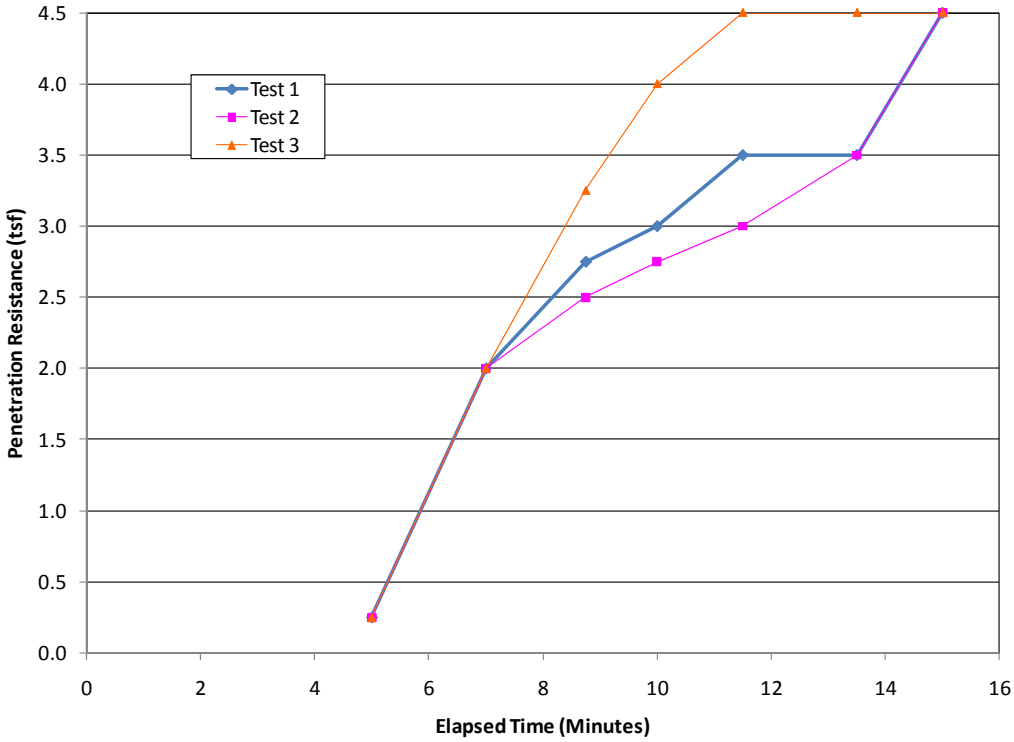




**Figure 183**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 21**

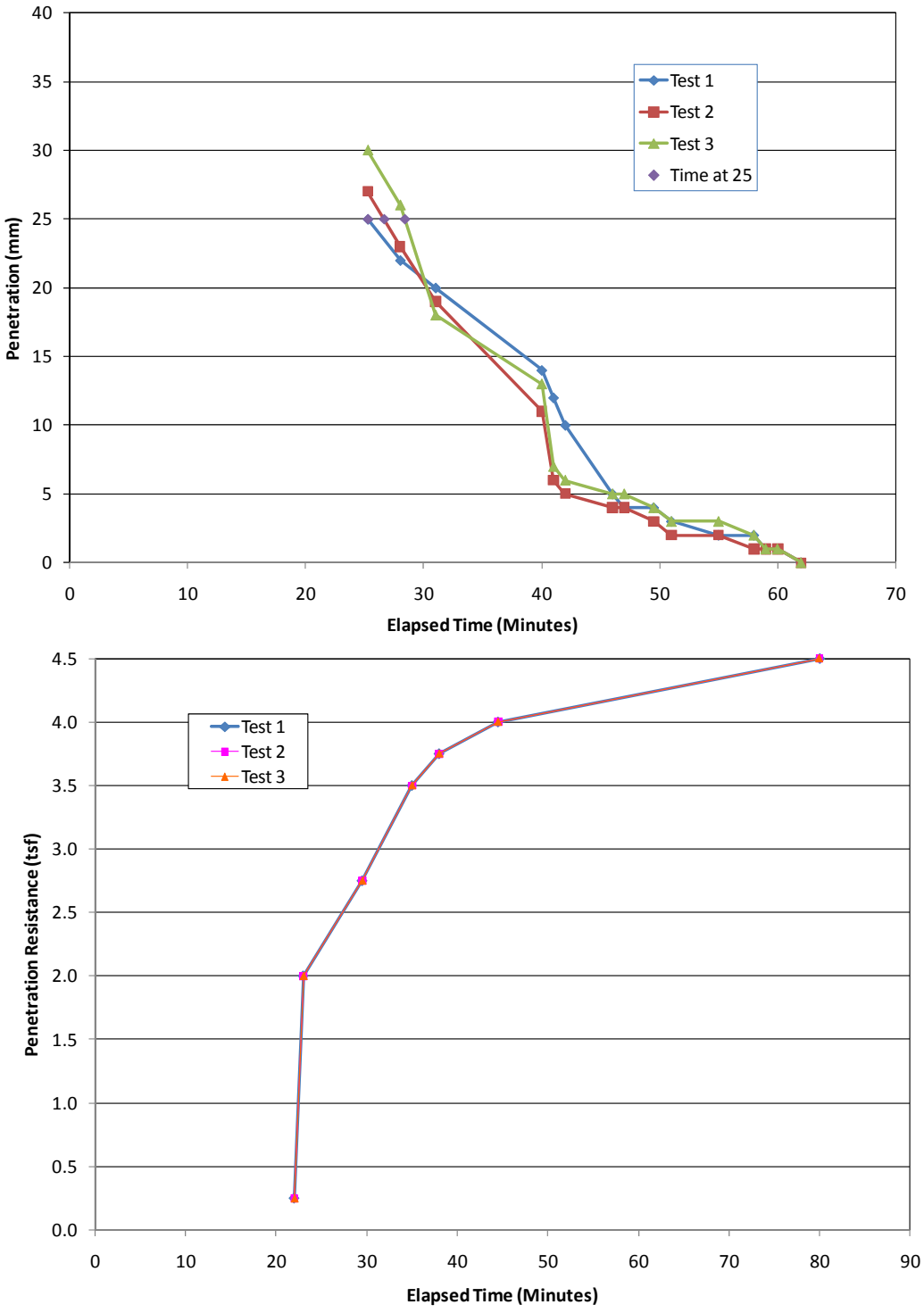
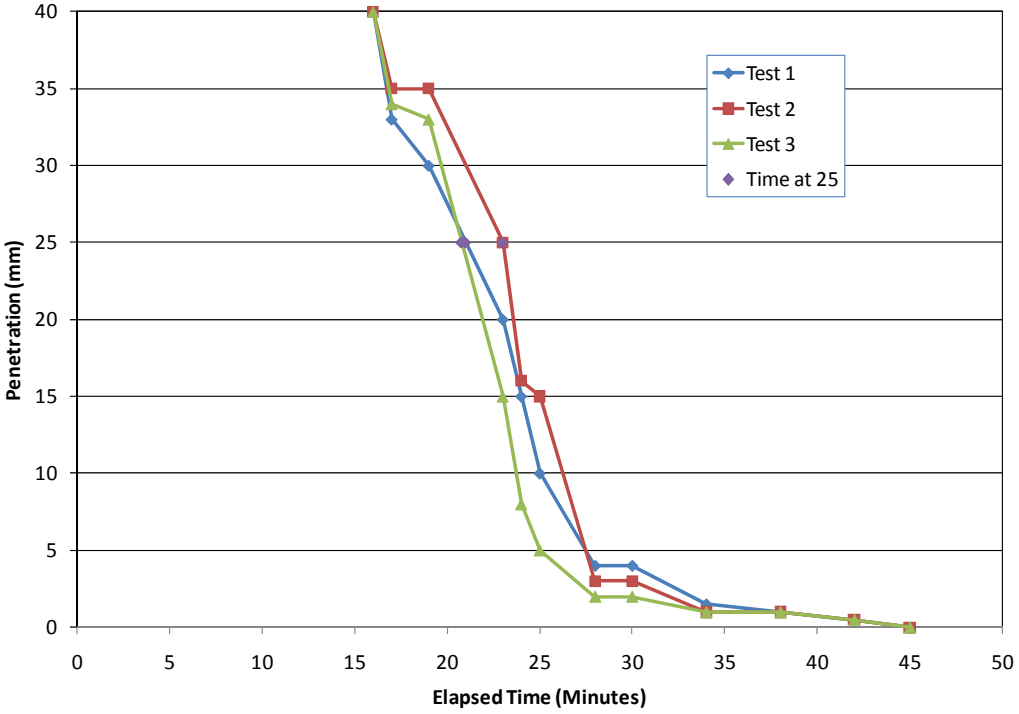



**Figure 184**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 22**



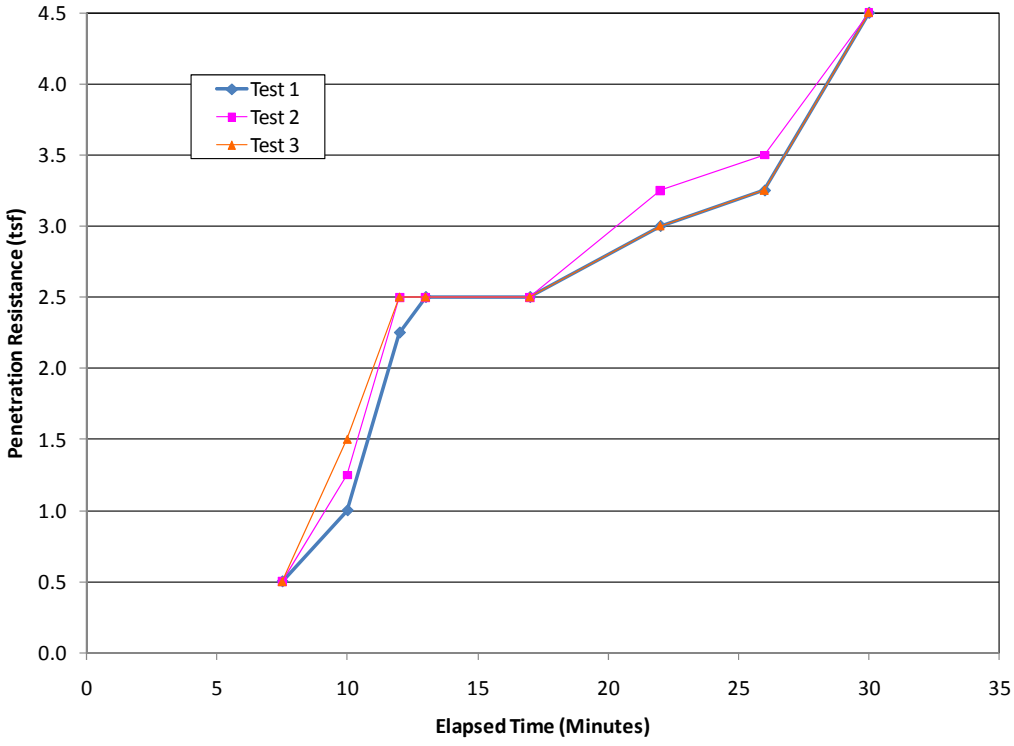




**Figure 185**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 23**

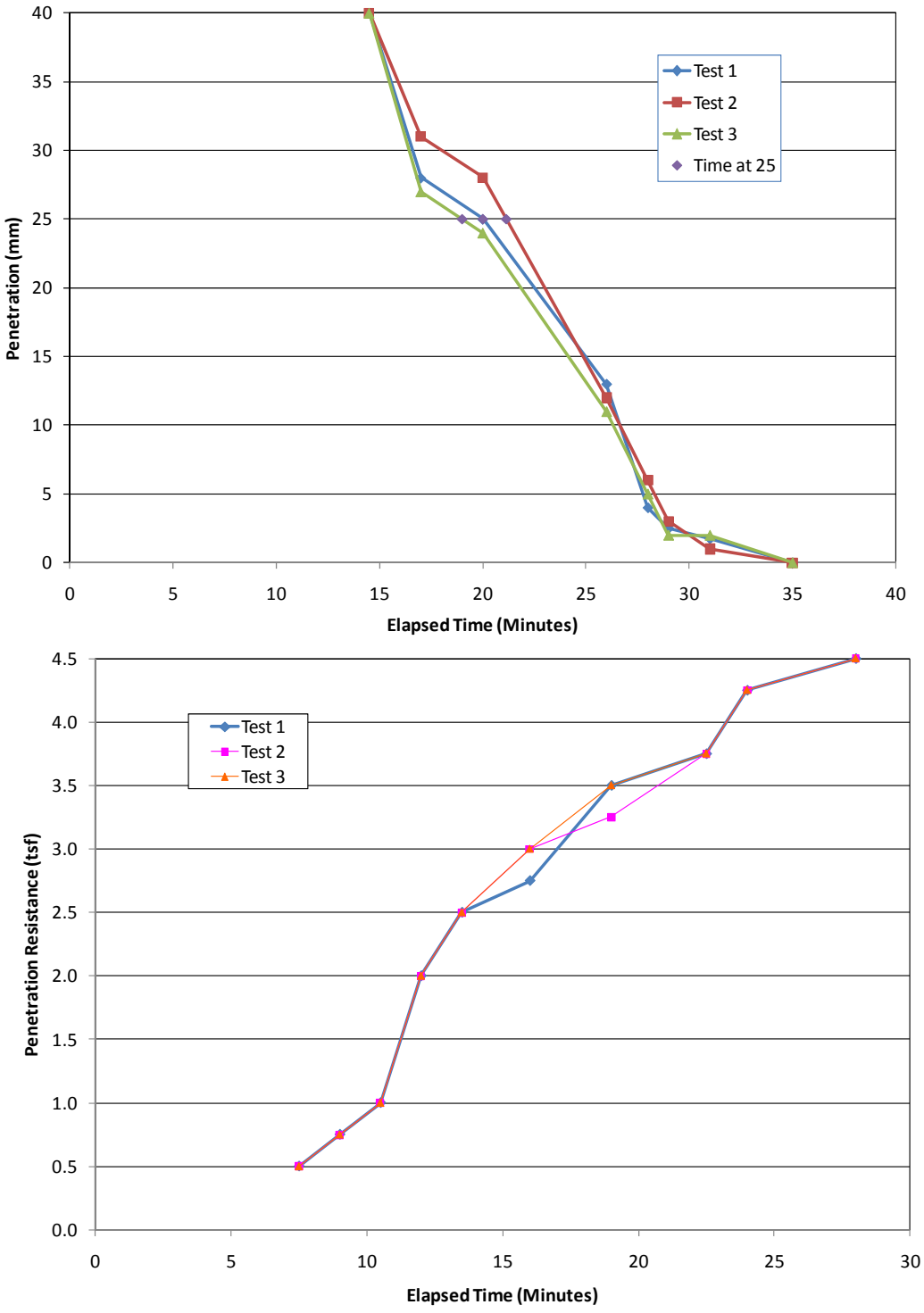
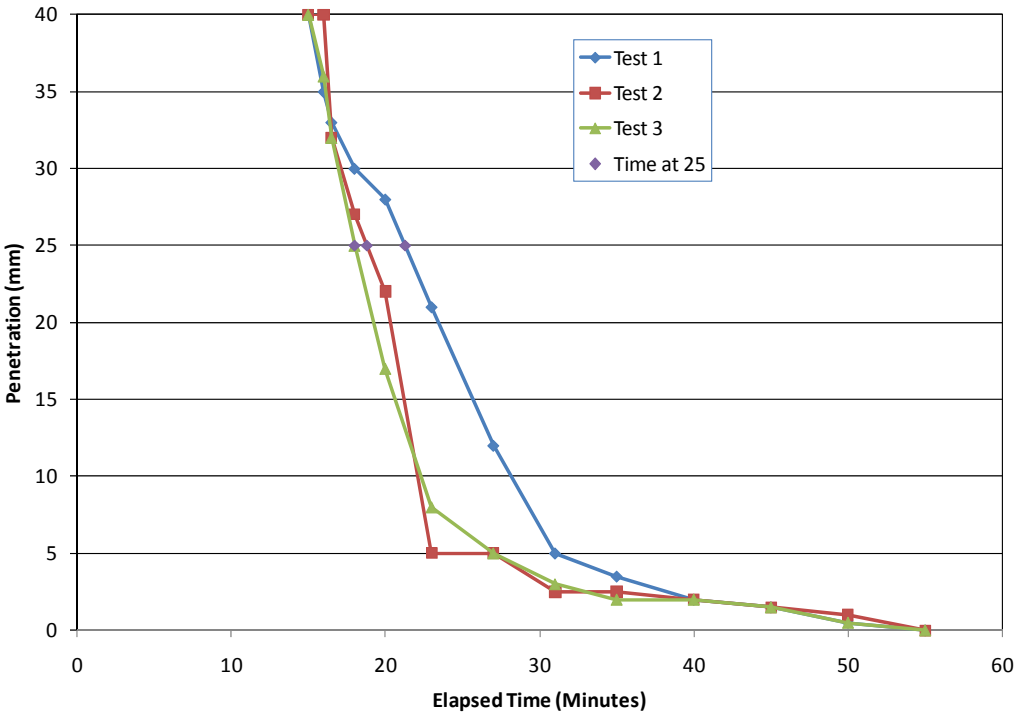



**Figure 186**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 24**



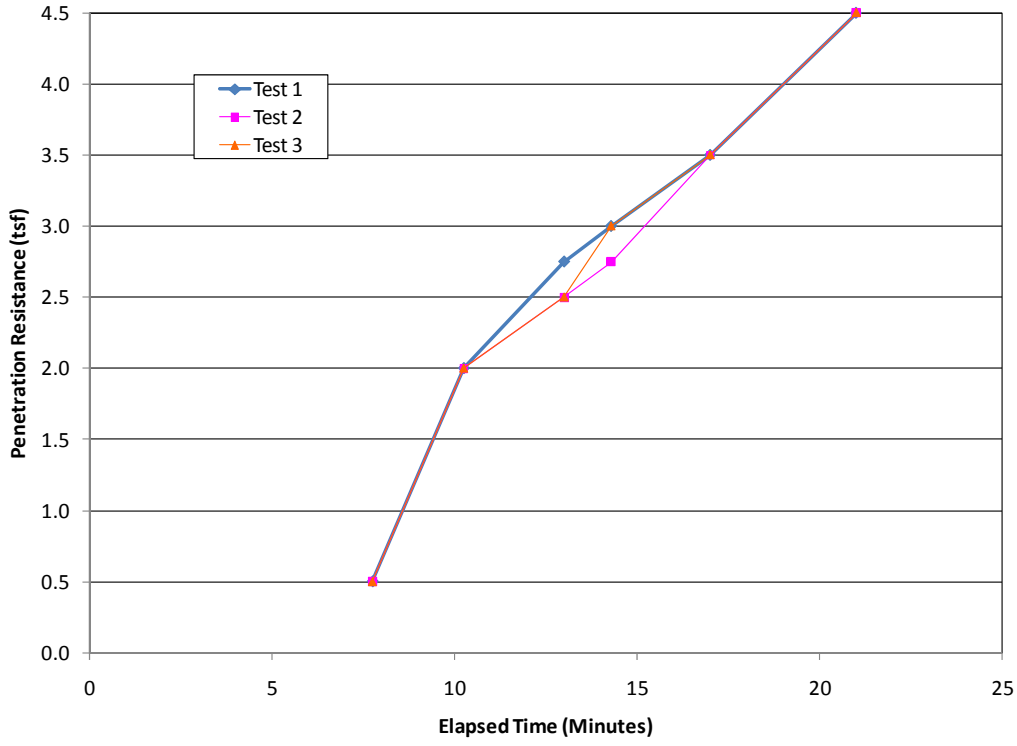




**Figure 187**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 25**

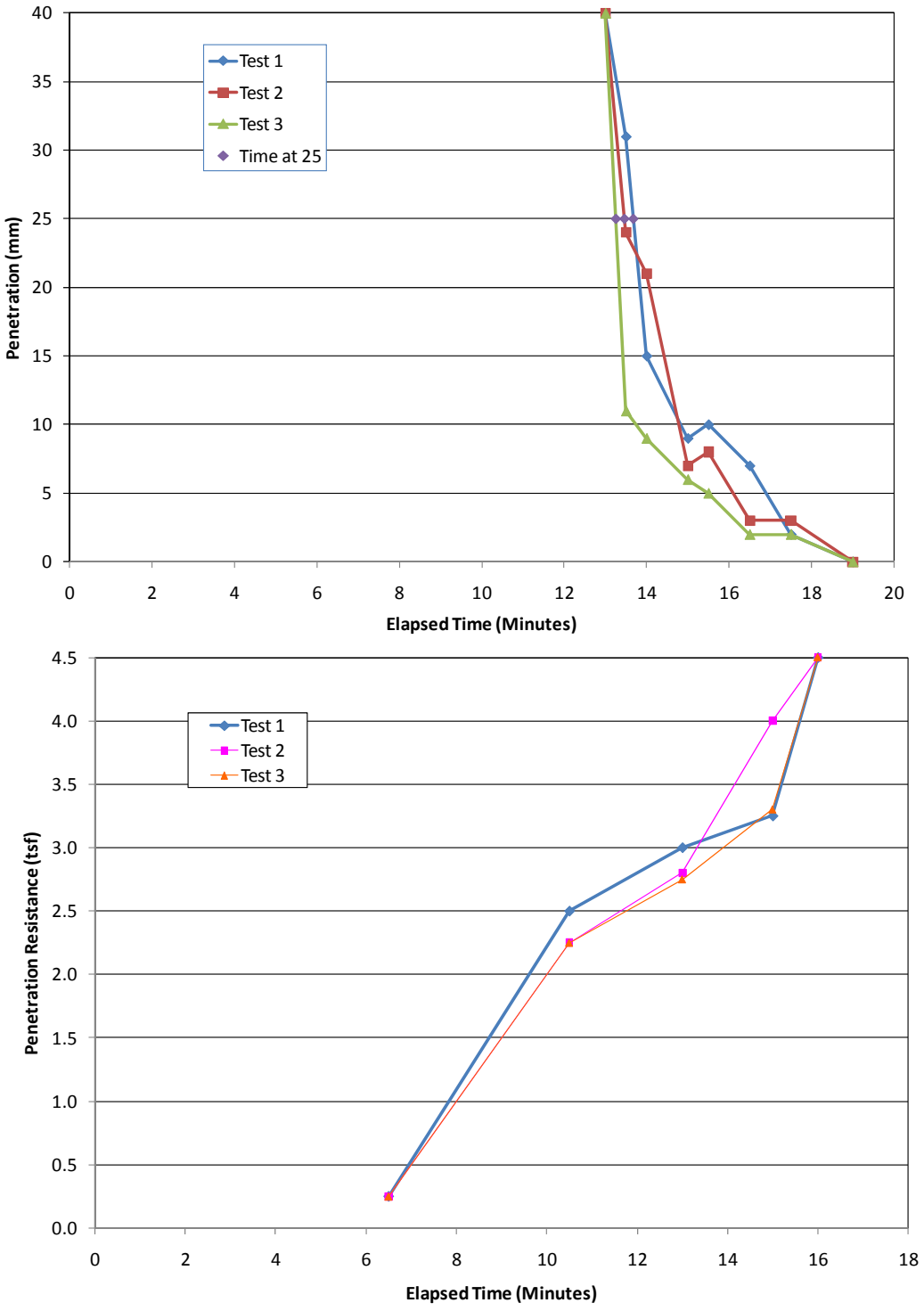



**Figure 188**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 8 Bucket 26**

## Source 9

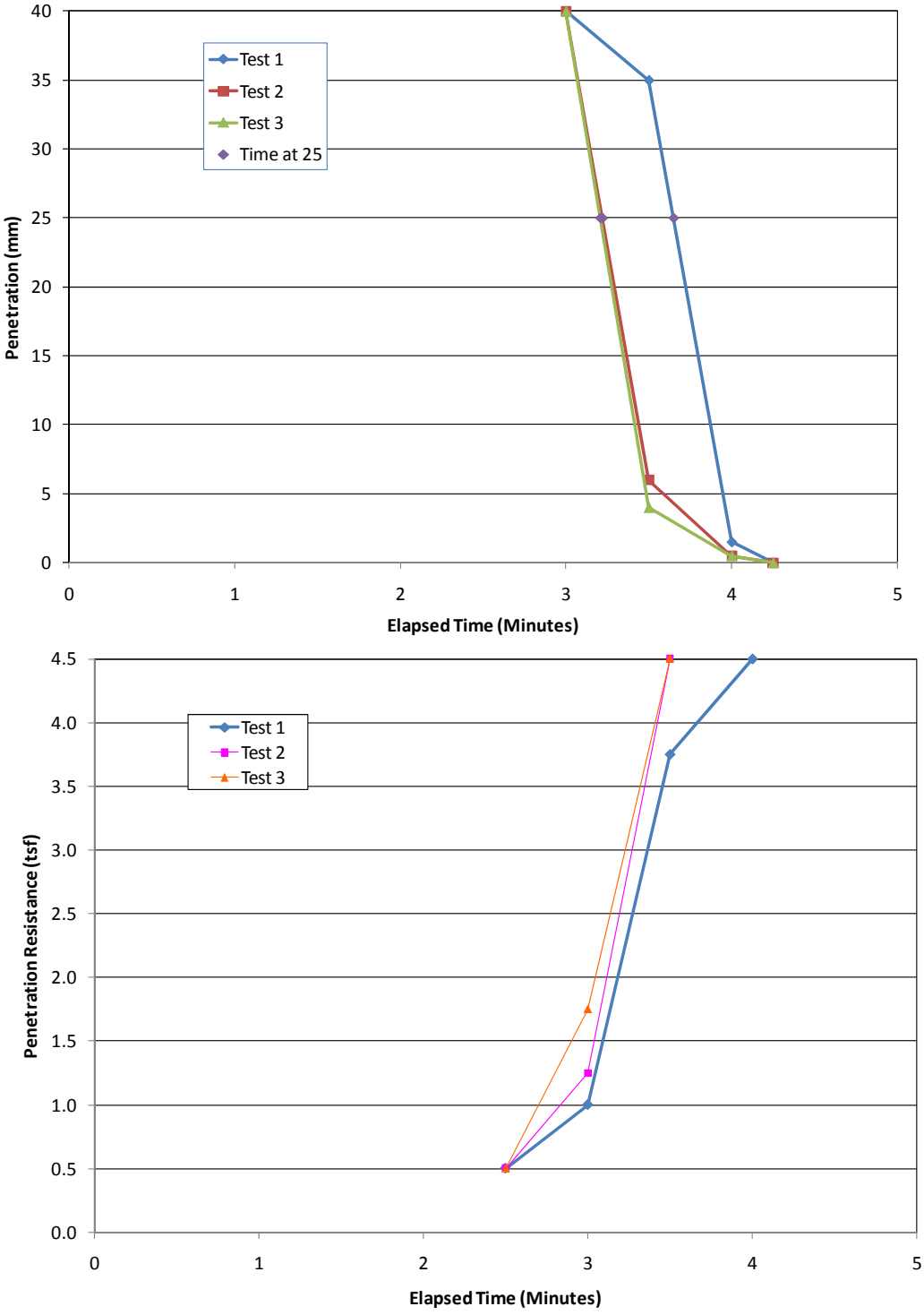



**Figure 189**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 1**

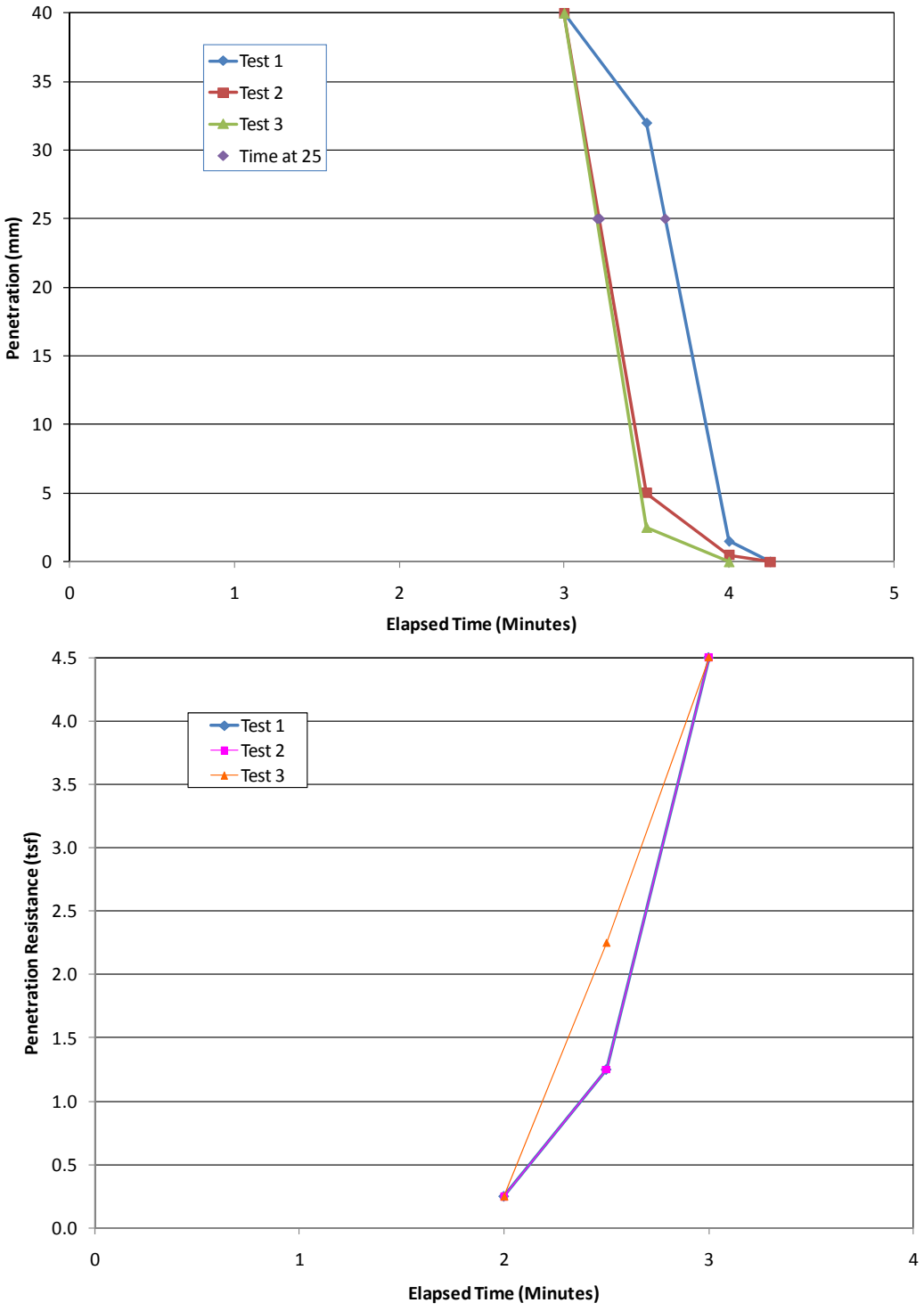



**Figure 190**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 2**

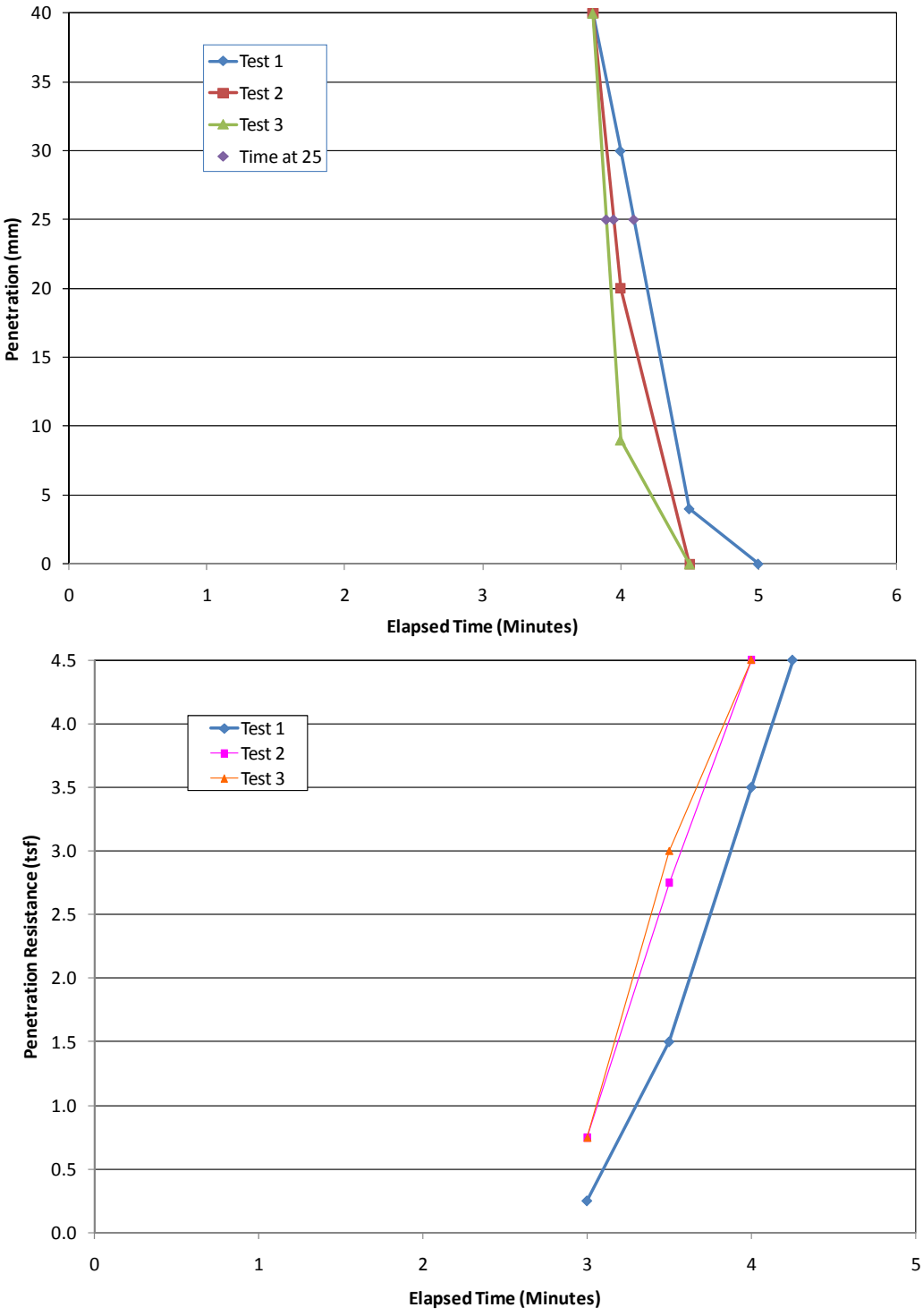



**Figure 191**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 3**

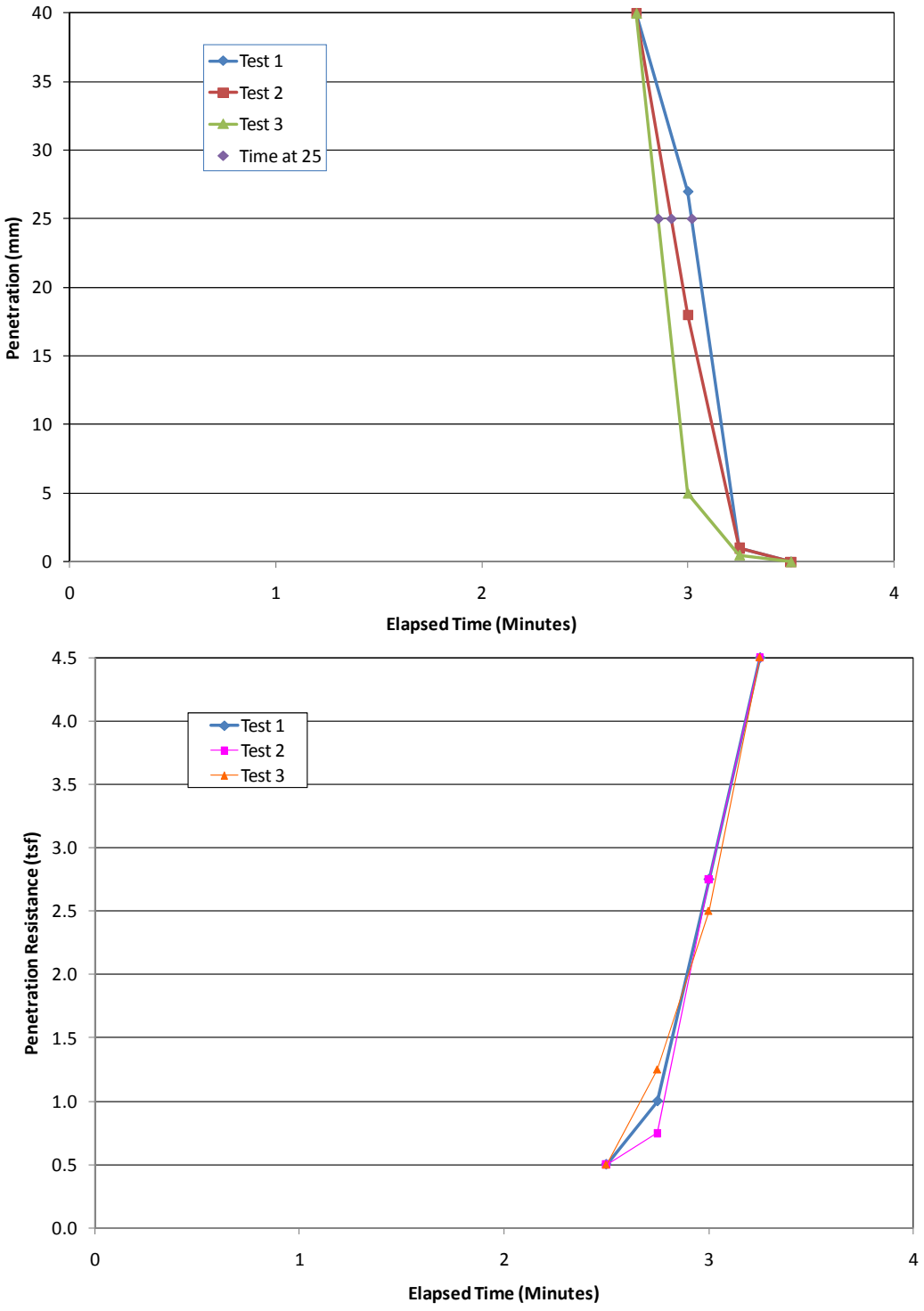



**Figure 192**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 4**

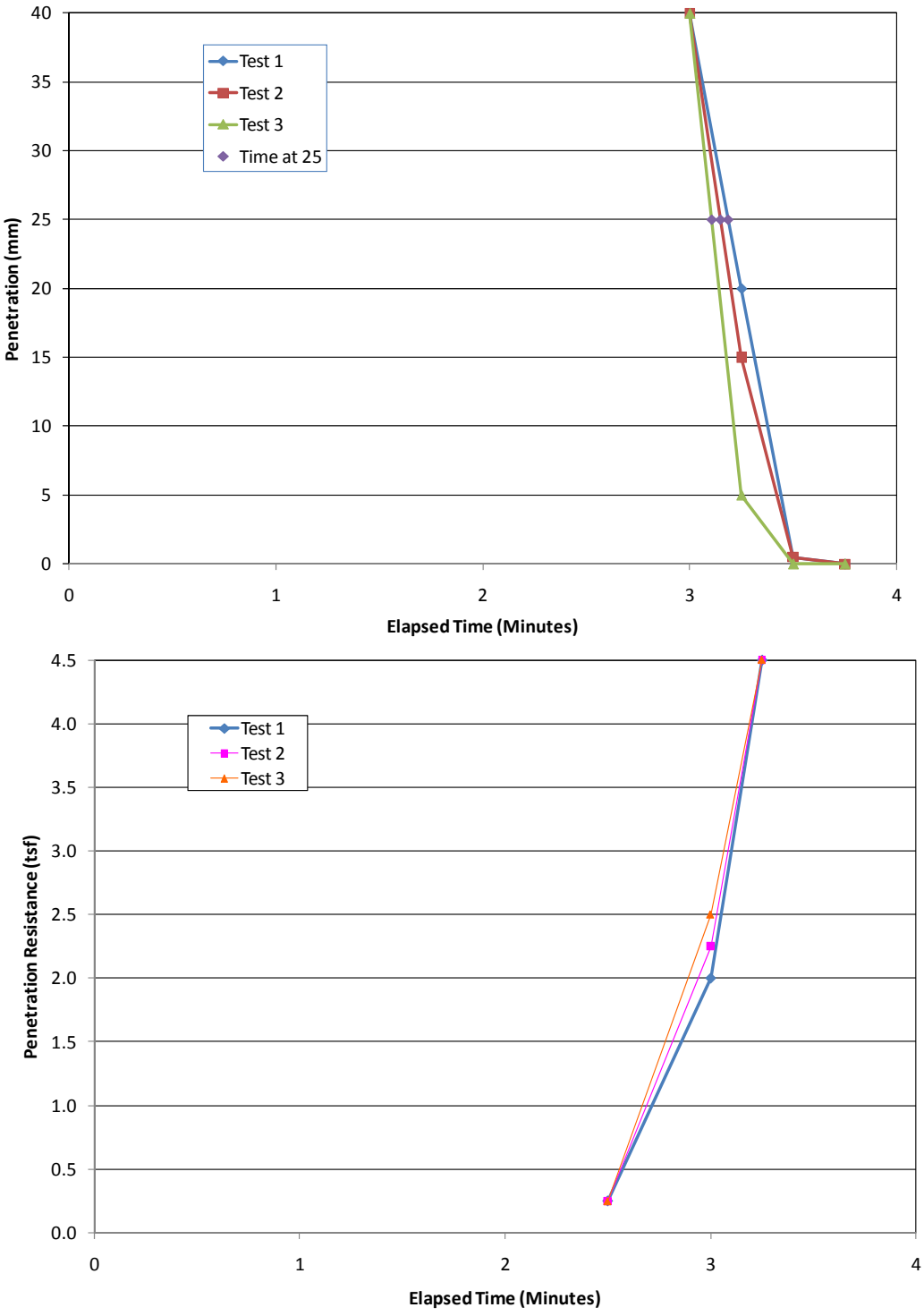



**Figure 193**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 5**

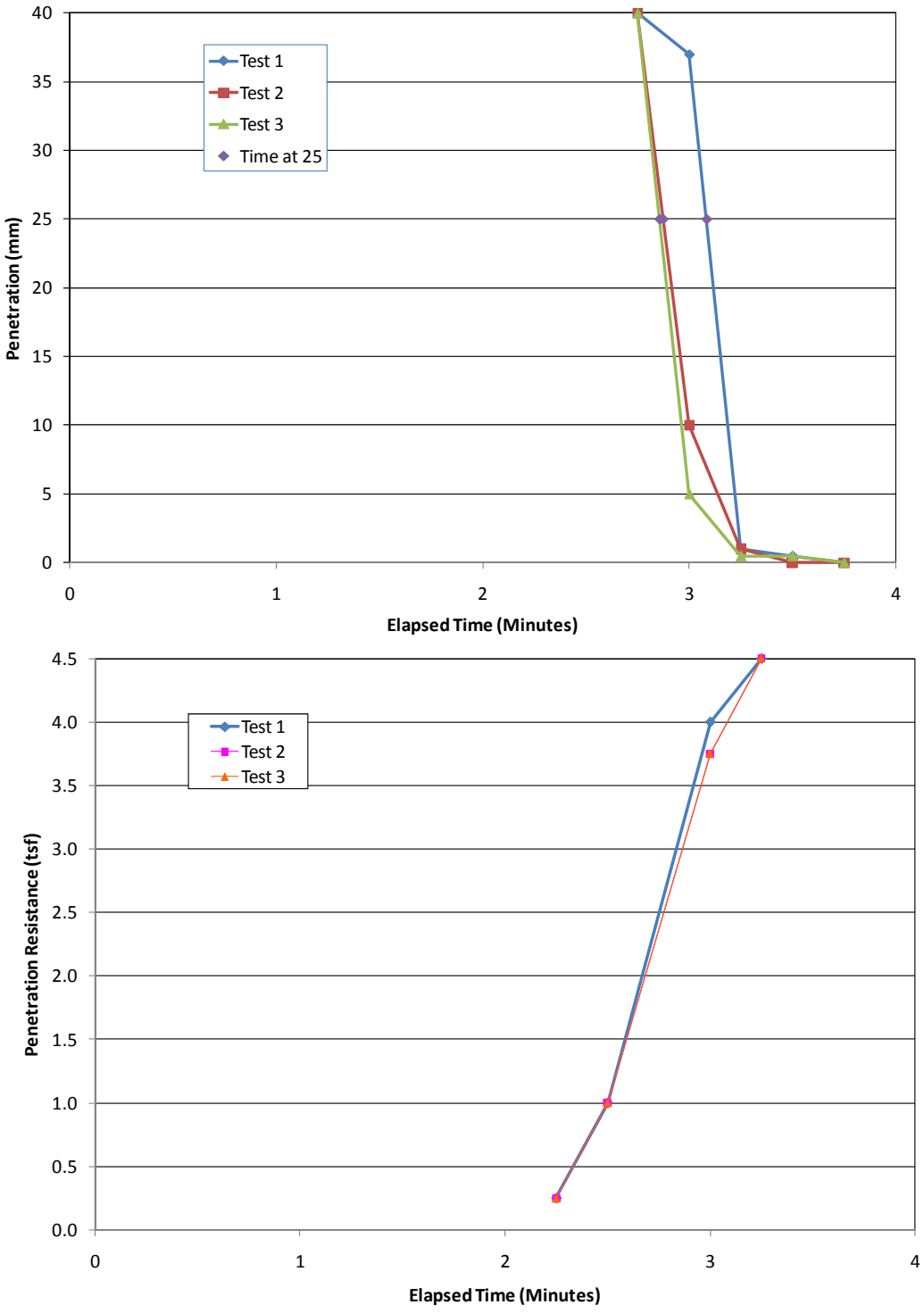



**Figure 194**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 6**

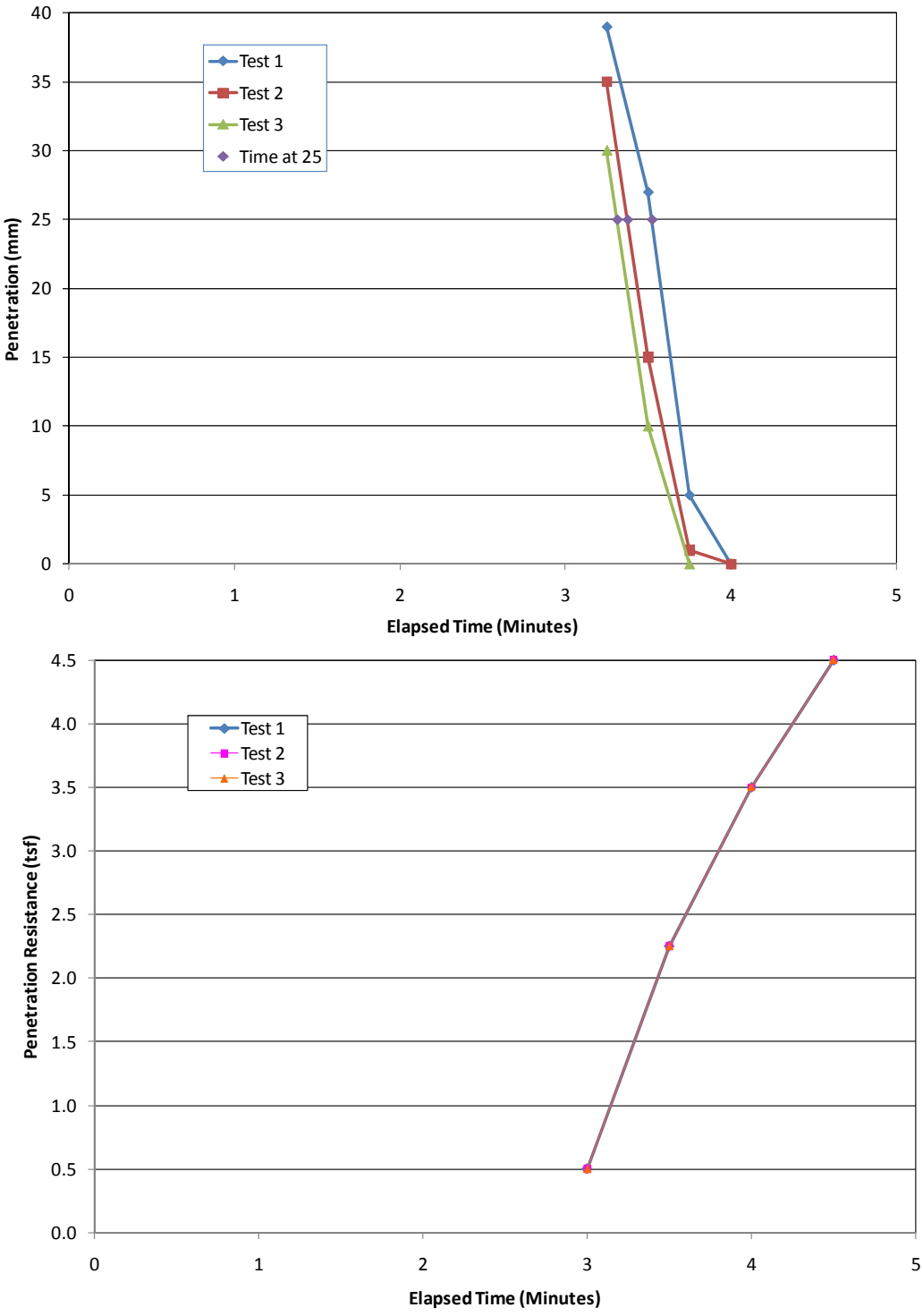



**Figure 195**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 7**

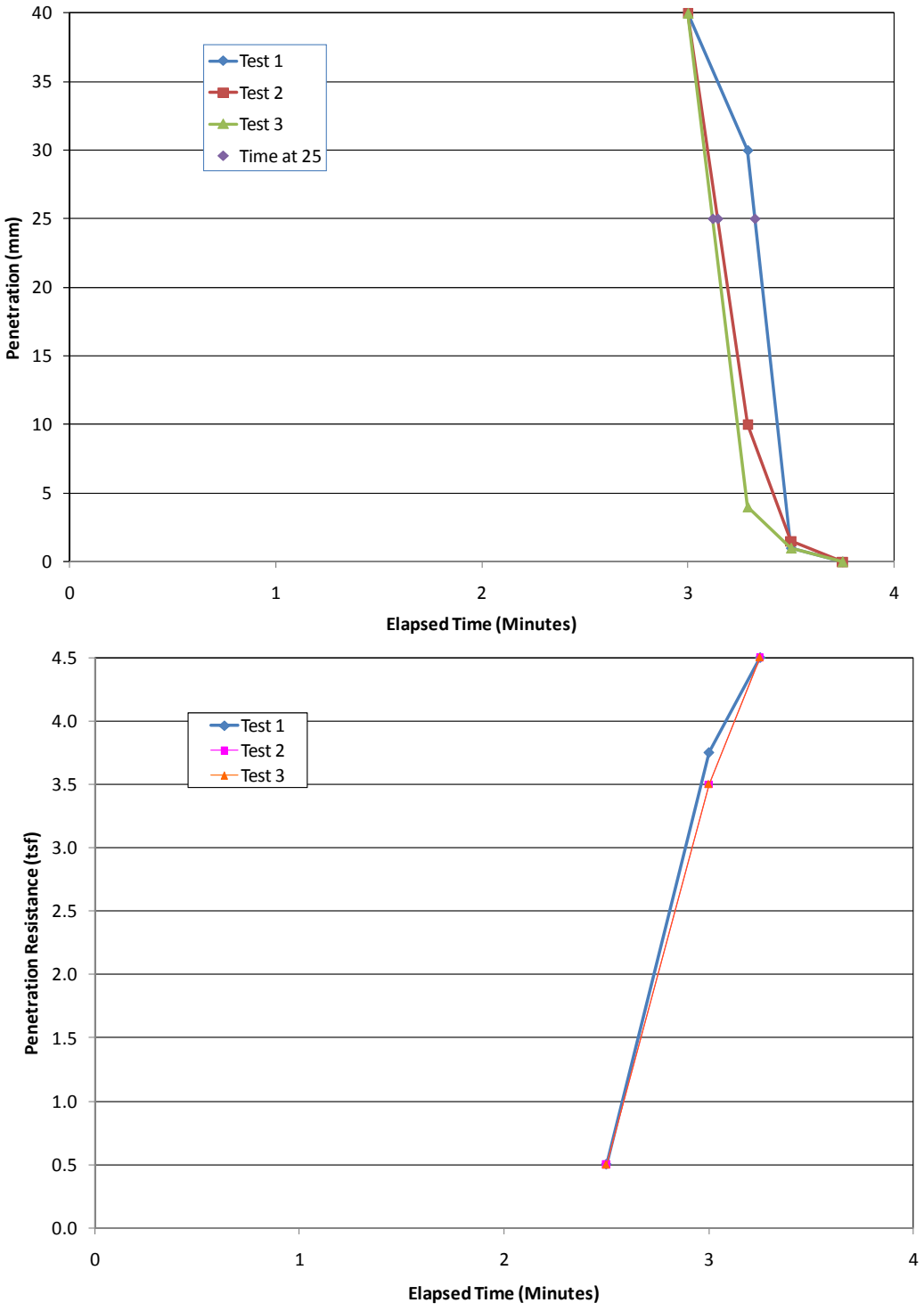



**Figure 196**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 8**

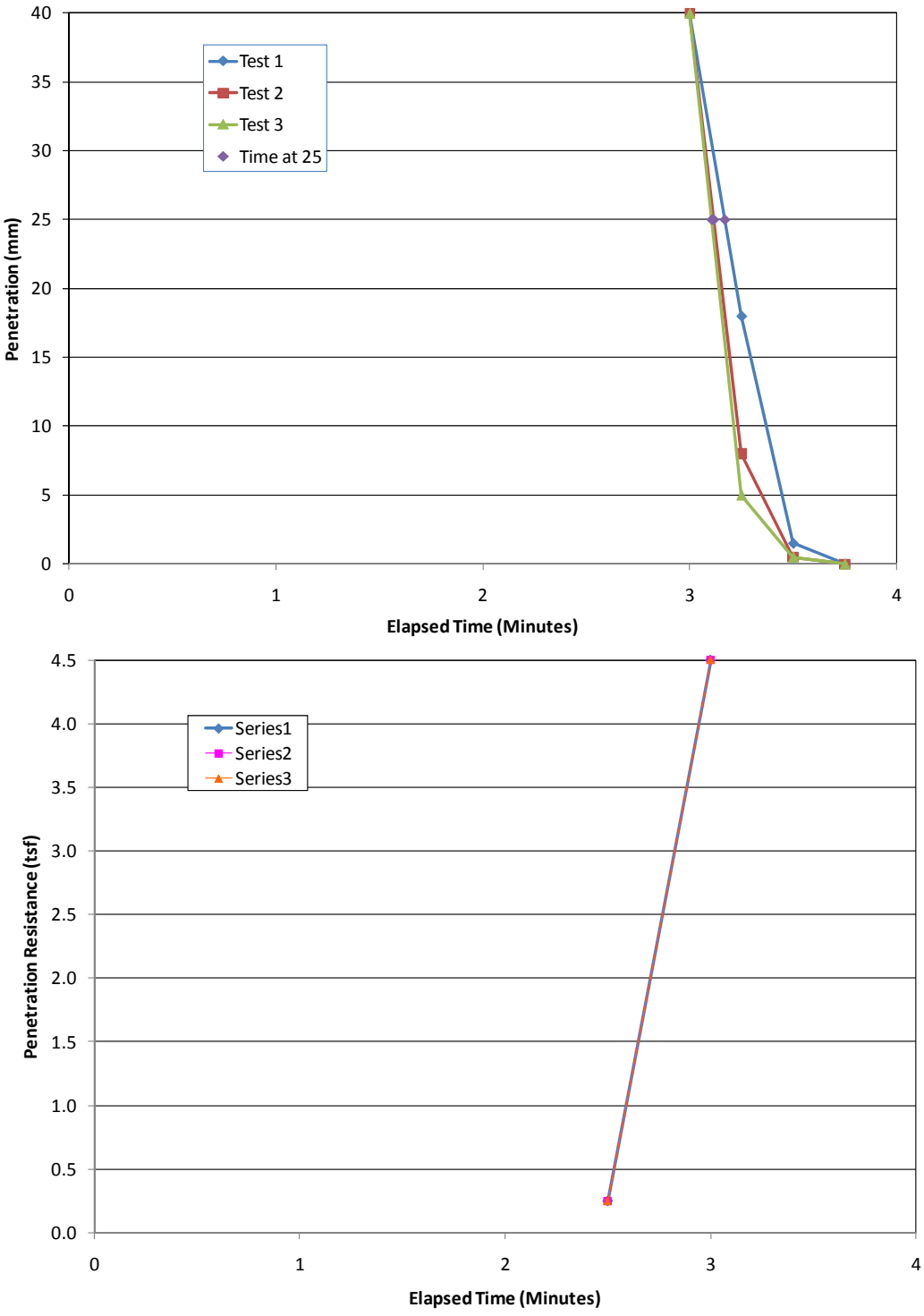



**Figure 197**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 9**

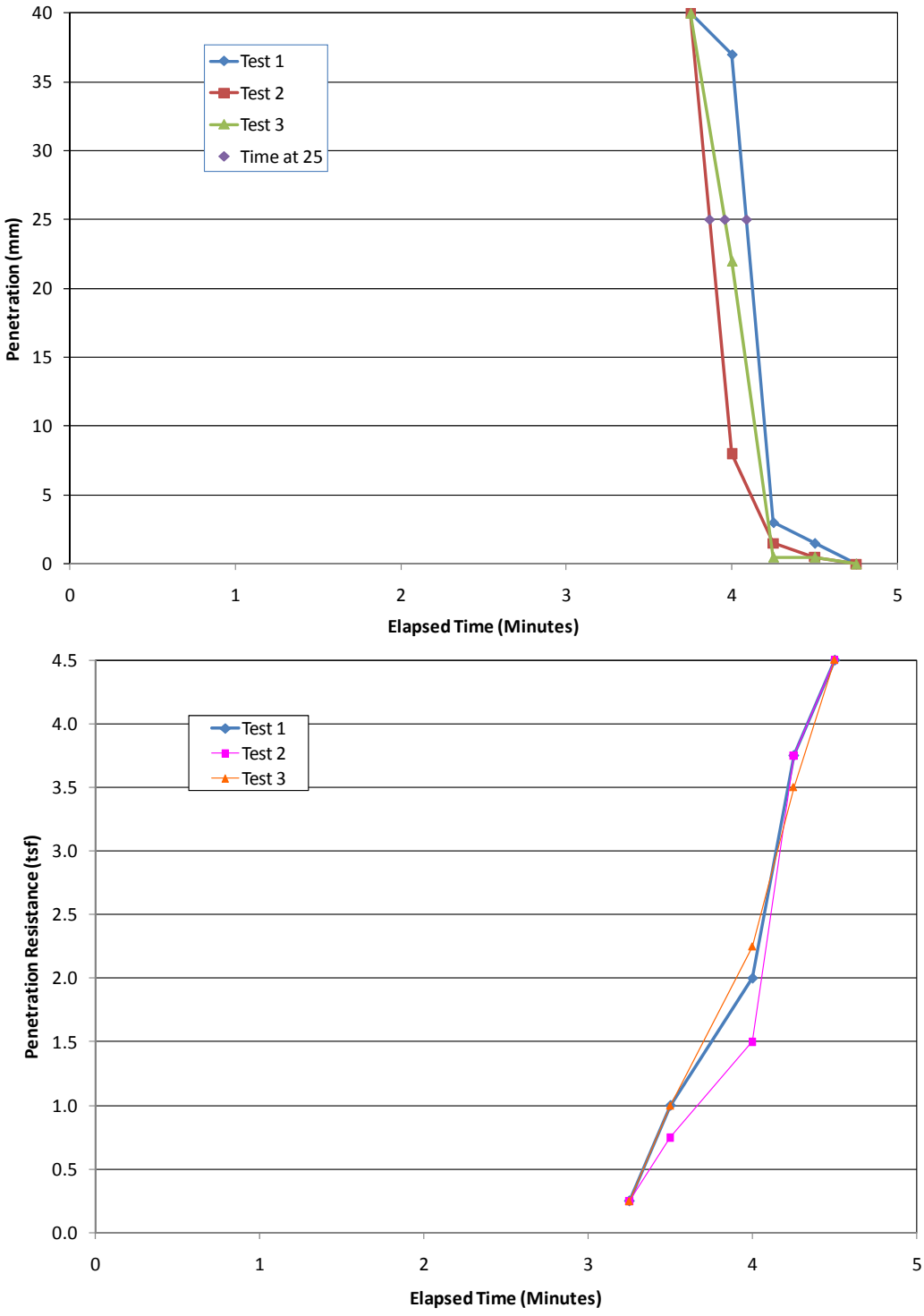



**Figure 198**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 10**

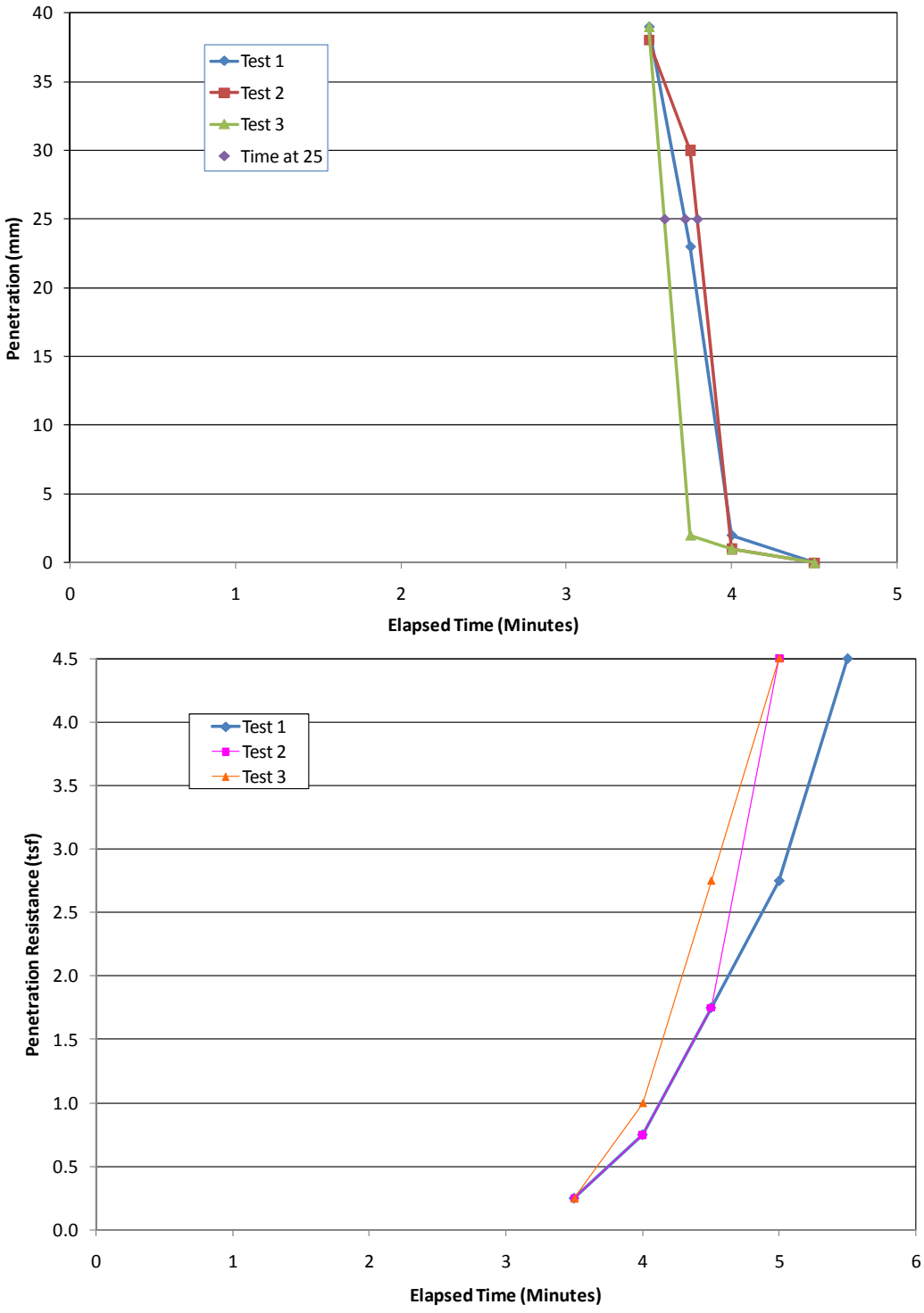



**Figure 199**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 11**

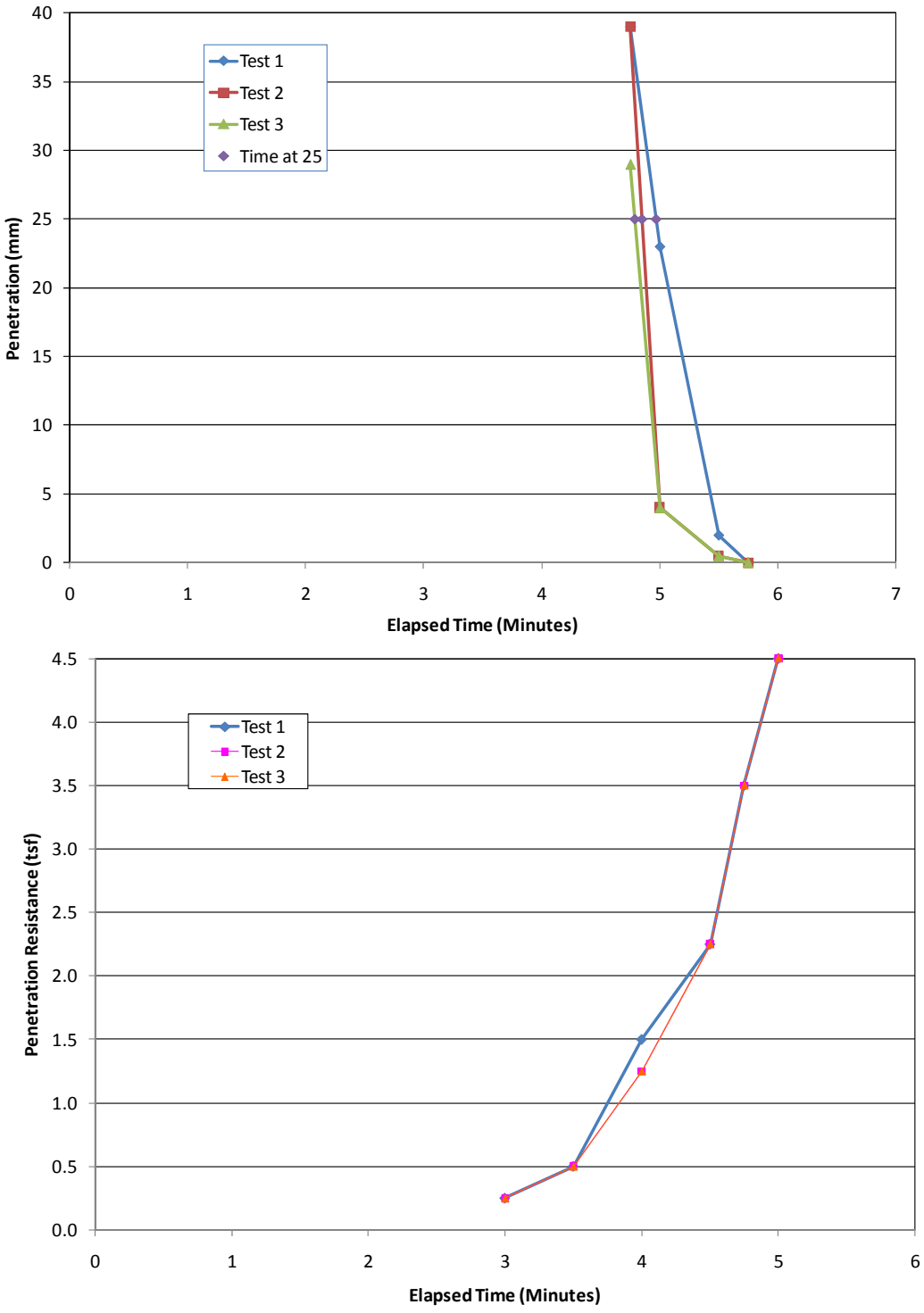



**Figure 200**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 12**

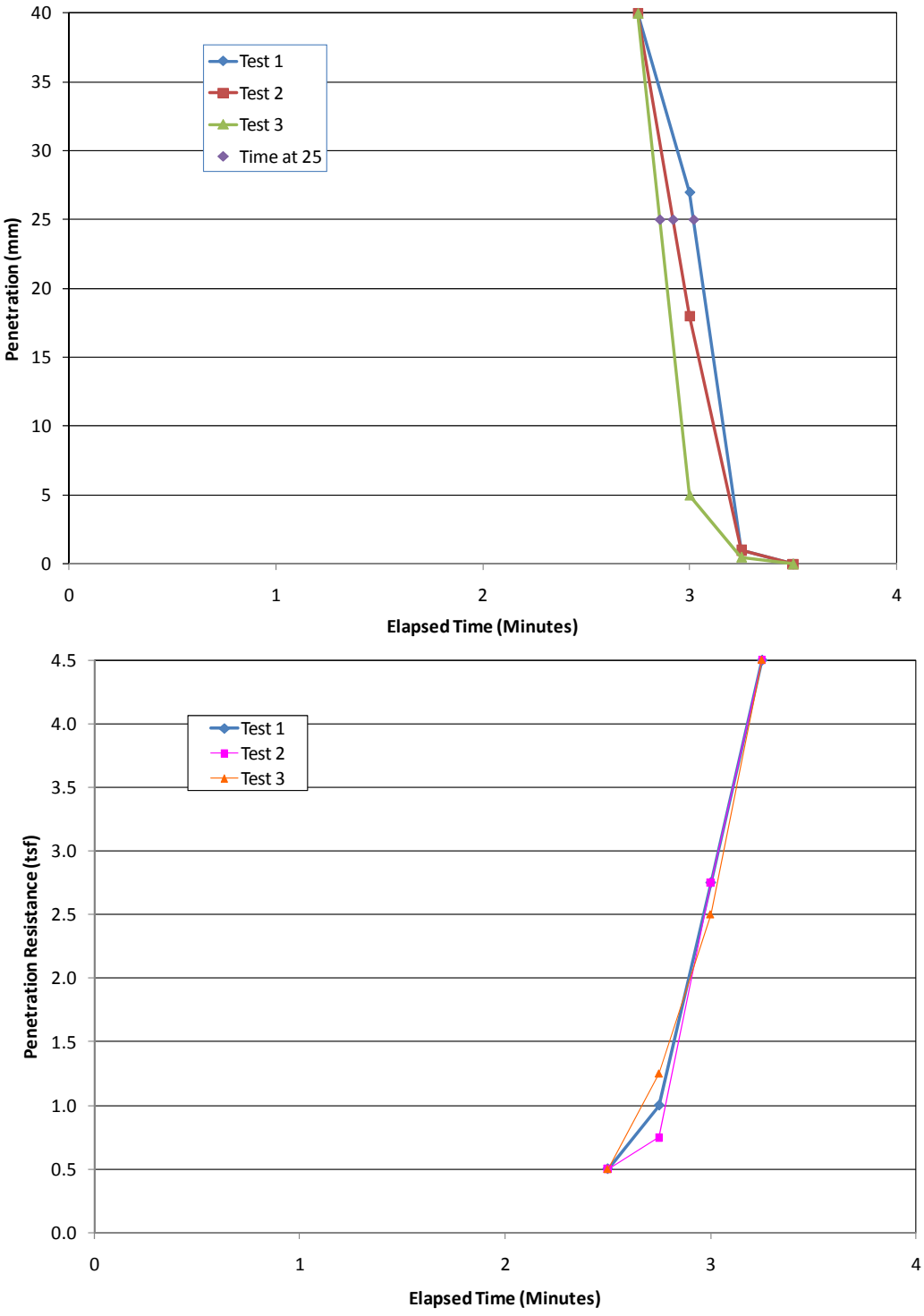



**Figure 201**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 13**

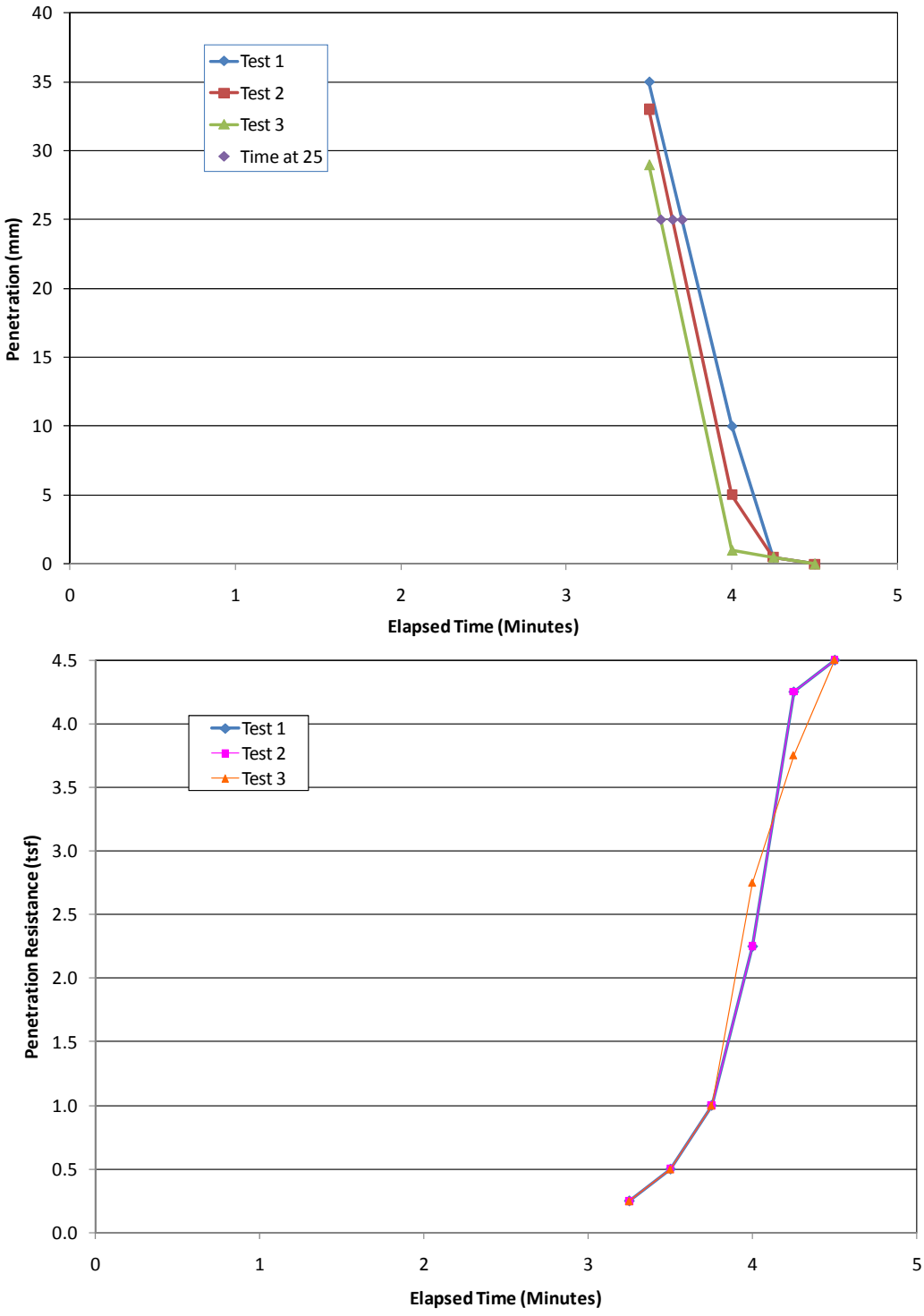



**Figure 202**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 14**

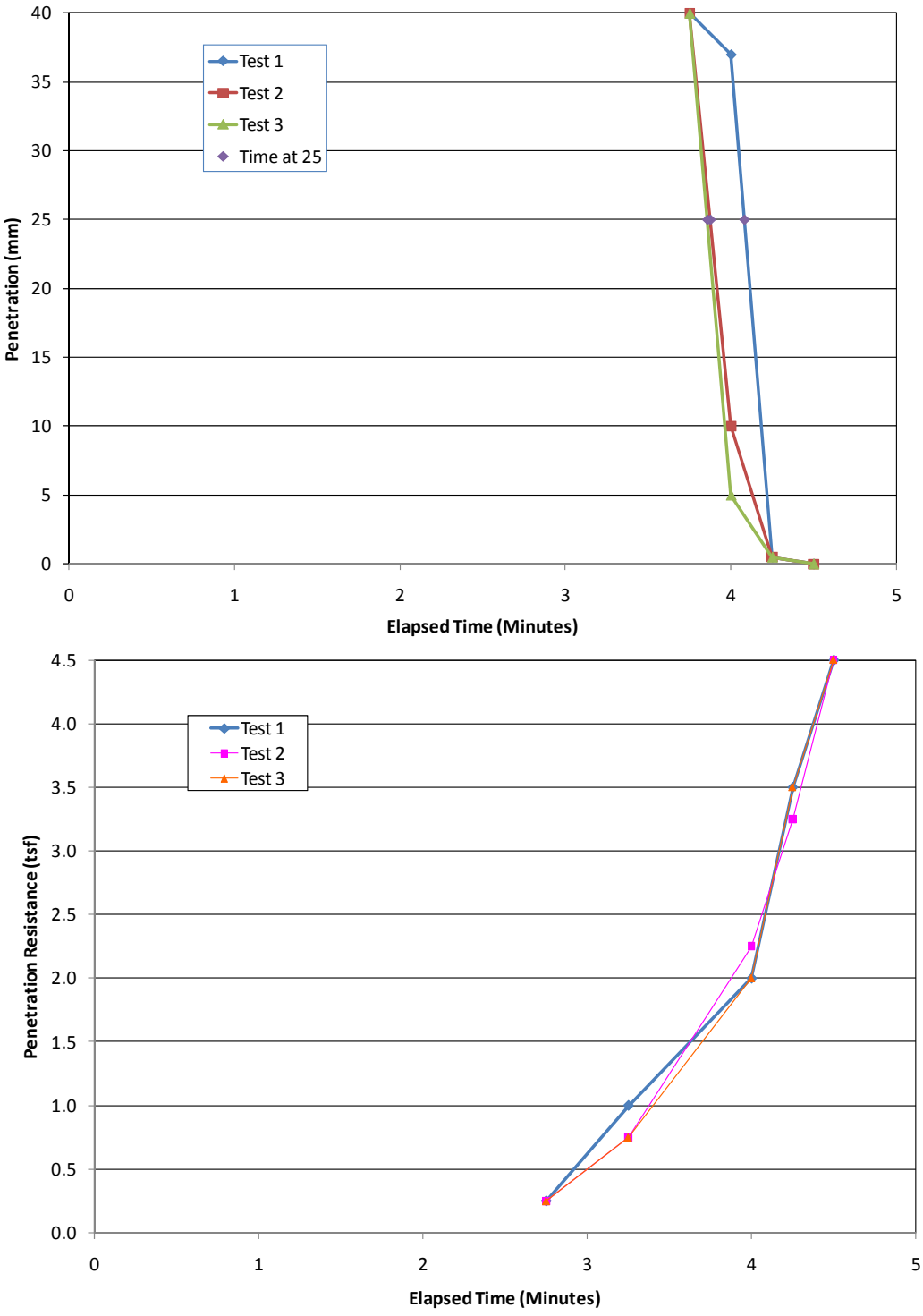



**Figure 203**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 15**

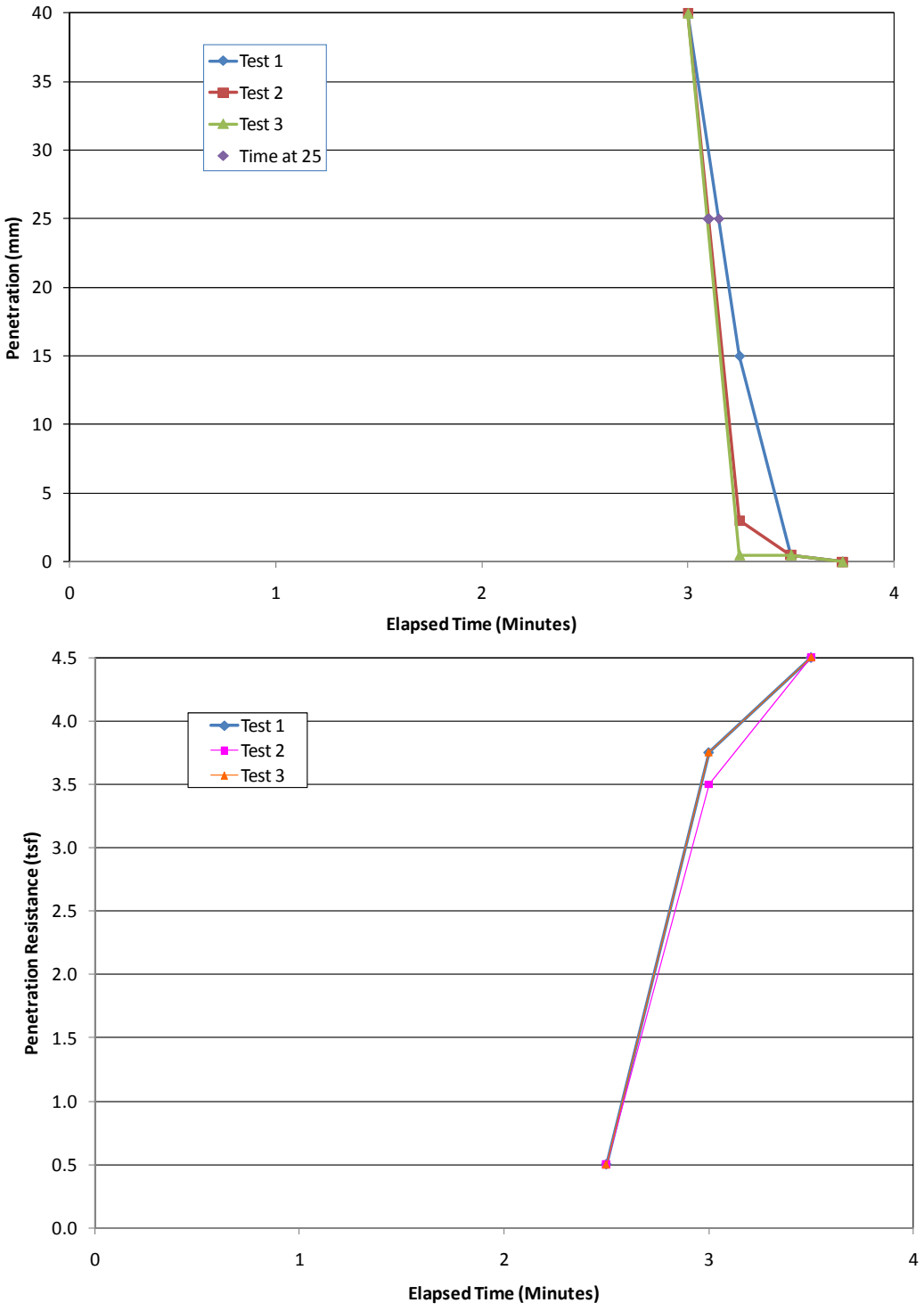
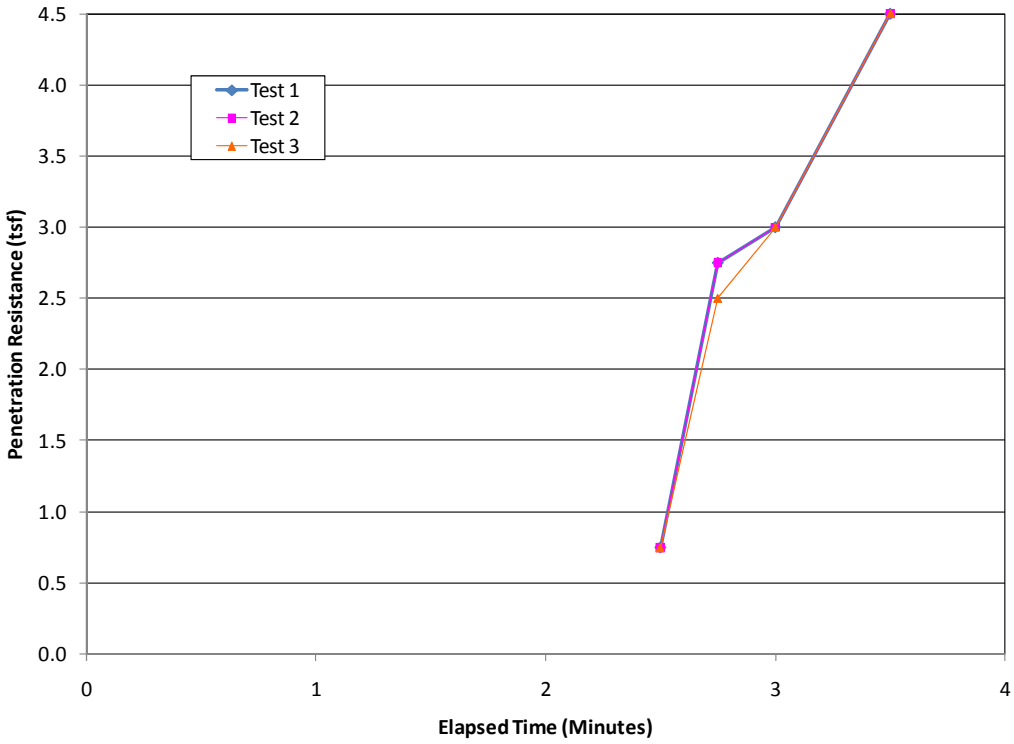



**Figure 204**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 16**



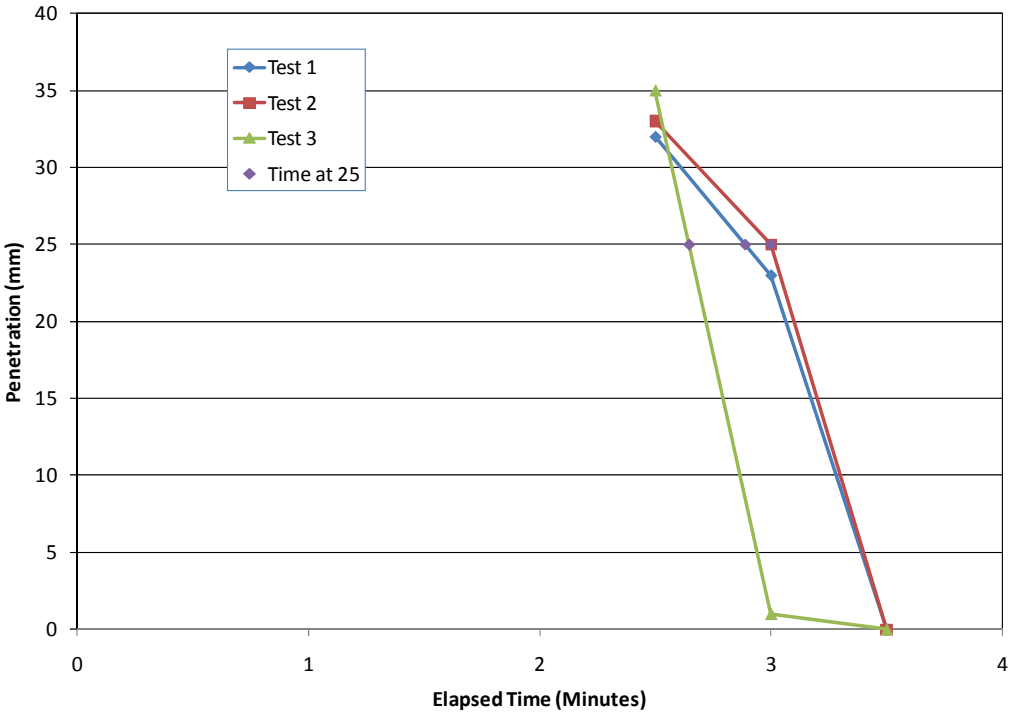




**Figure 205**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 17**

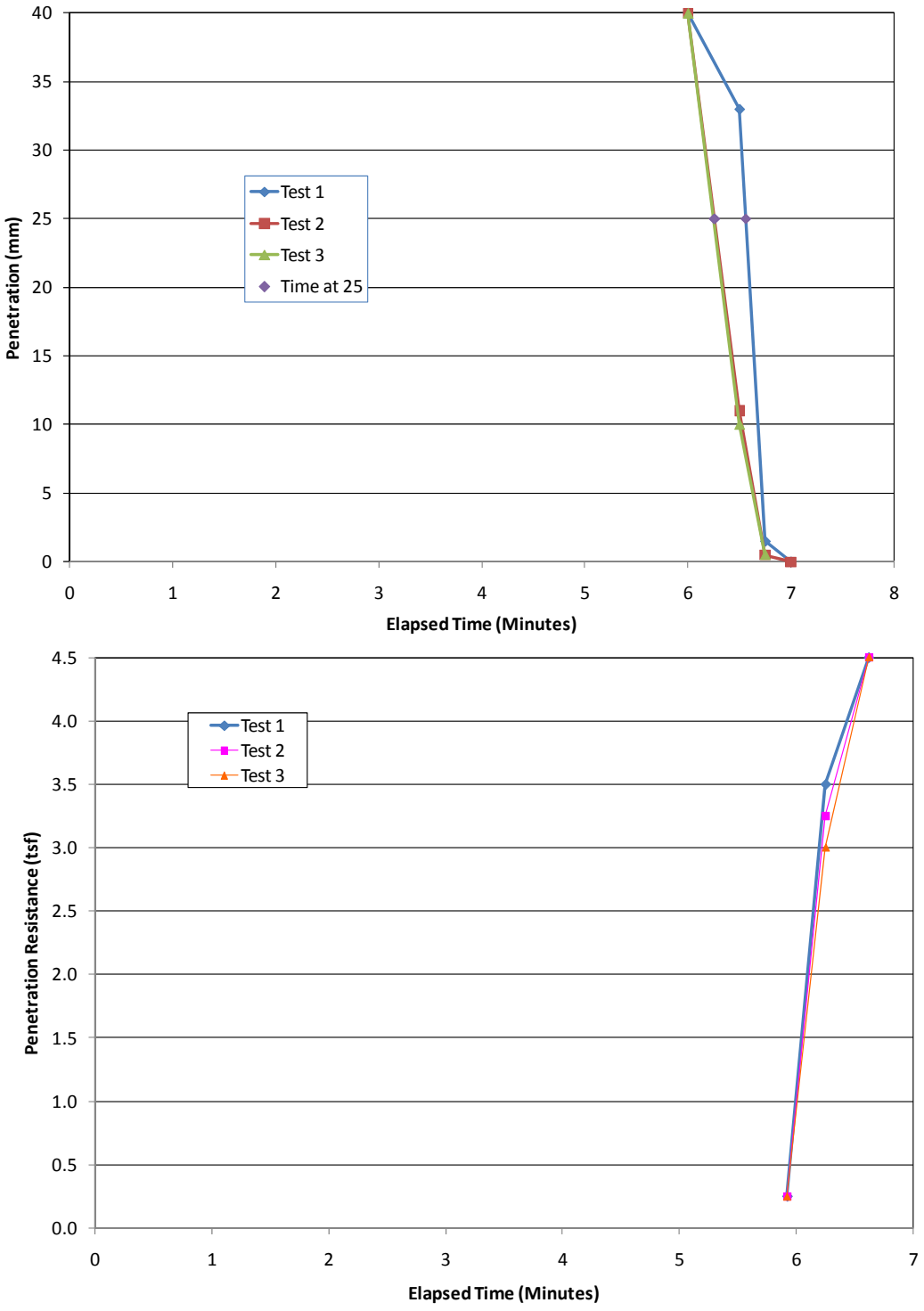



**Figure 206**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 18**

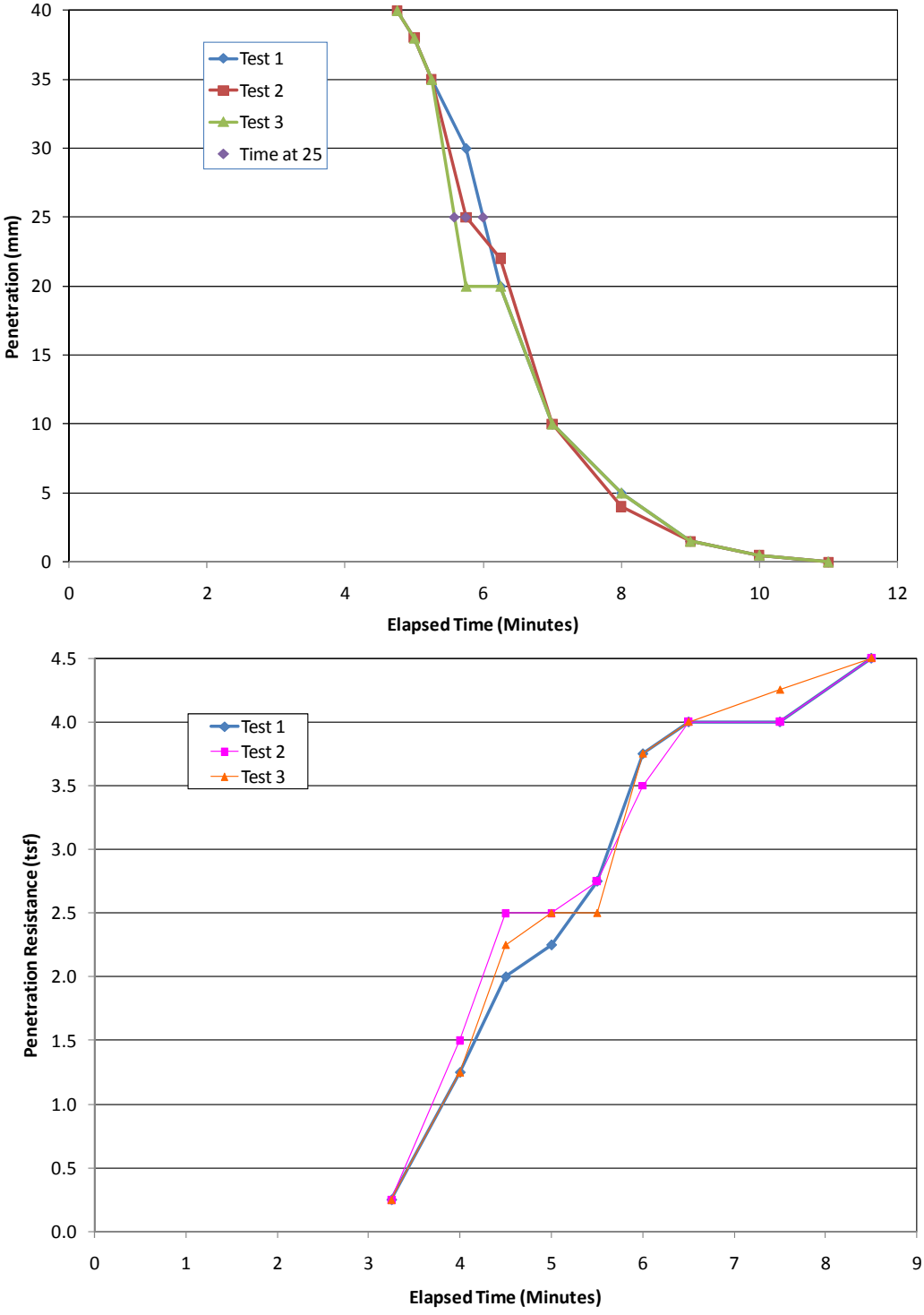



**Figure 207**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 19**

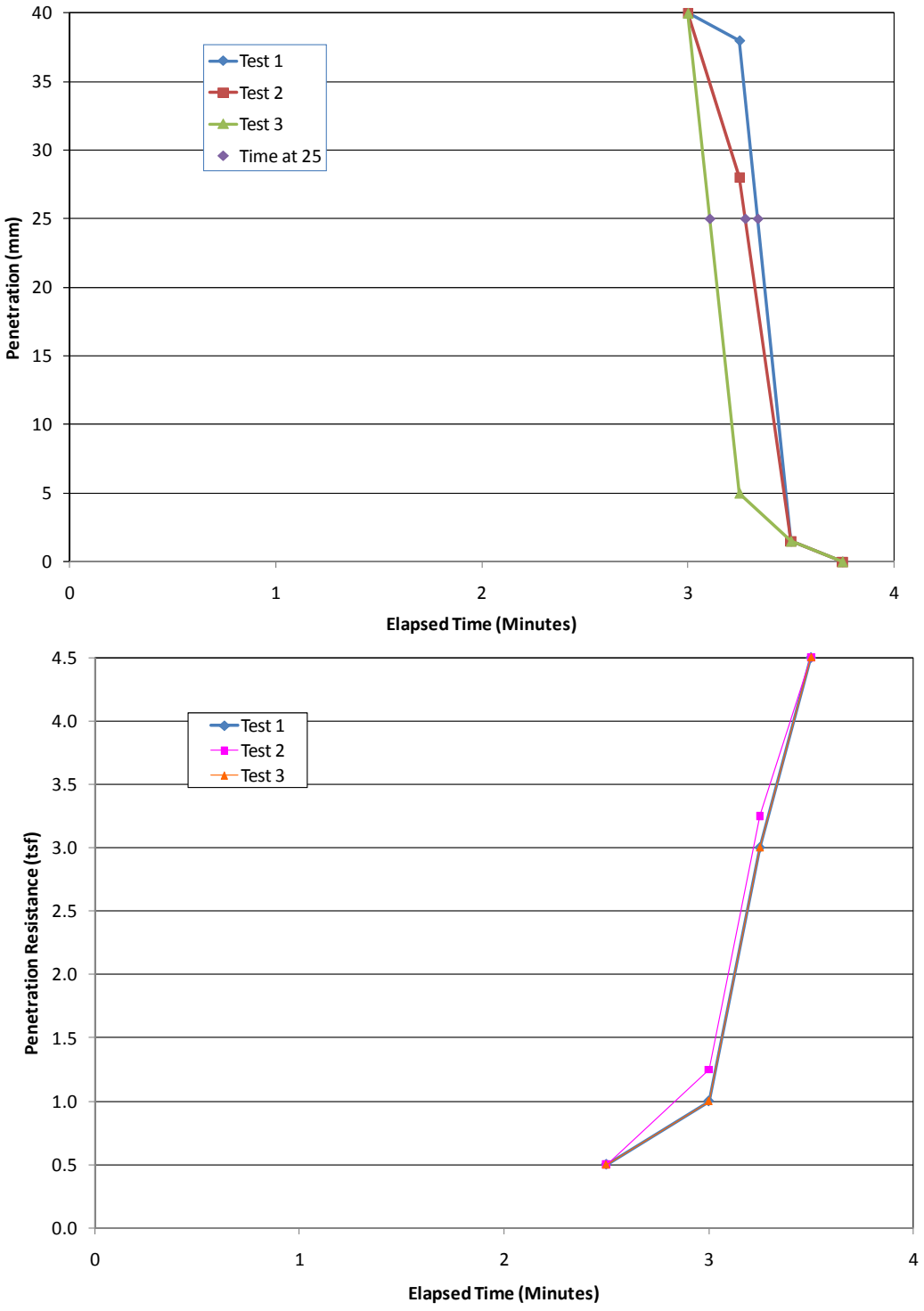



**Figure 208**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 9 Bucket 20**

**Source 11**

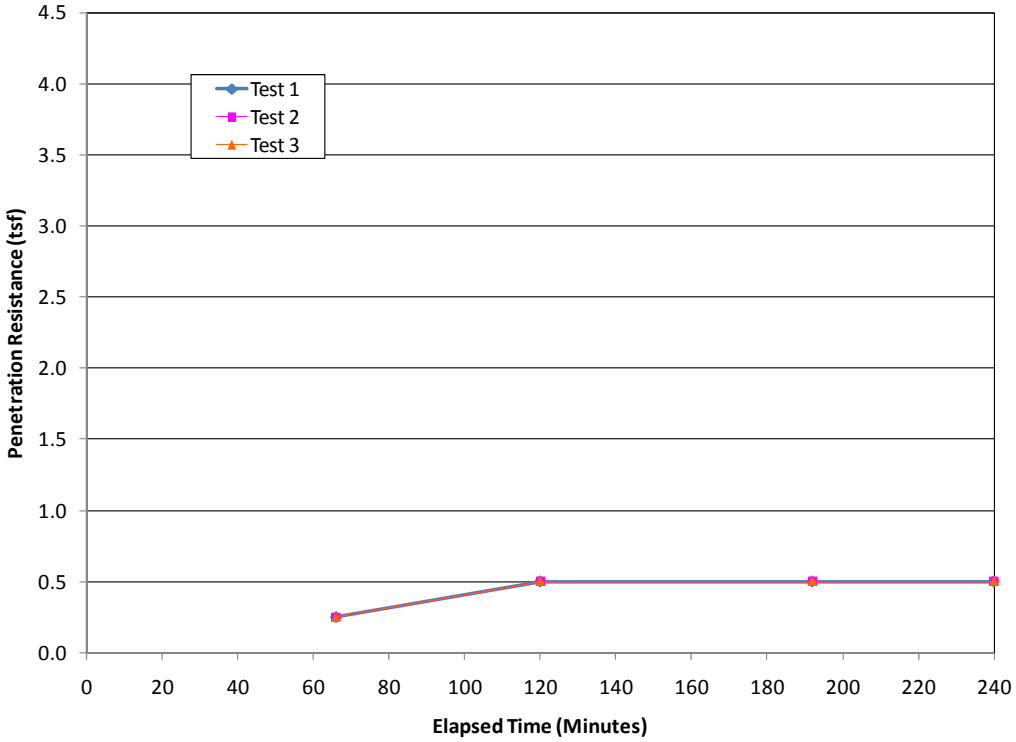
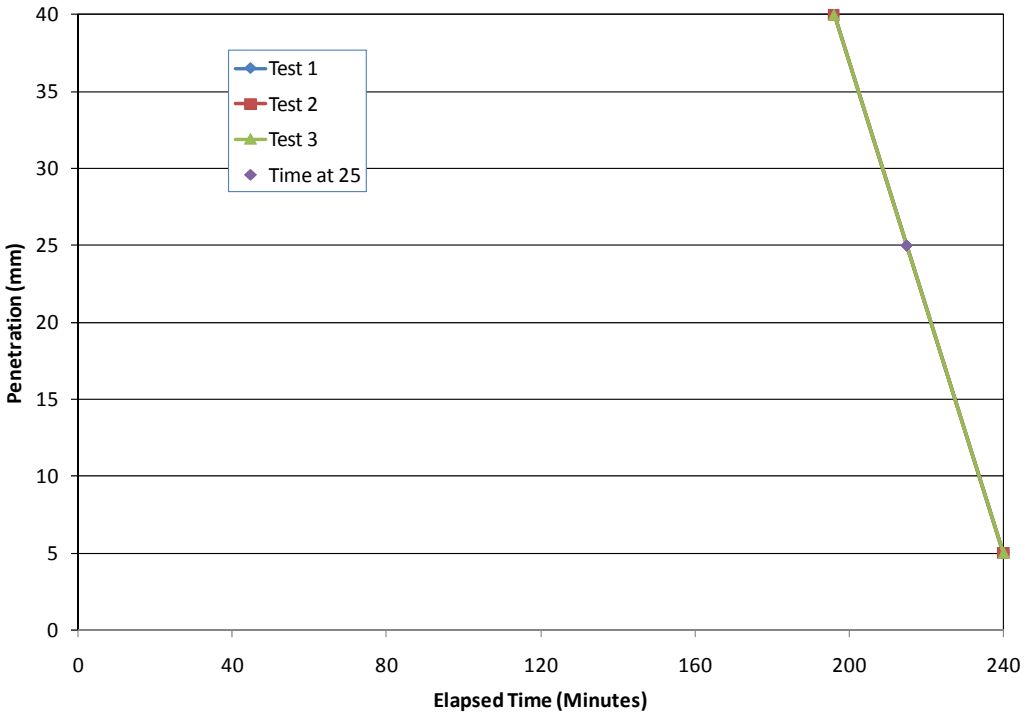



**Figure 209**
**Pocket penetration resistance graph for Source 11 Bucket 1**



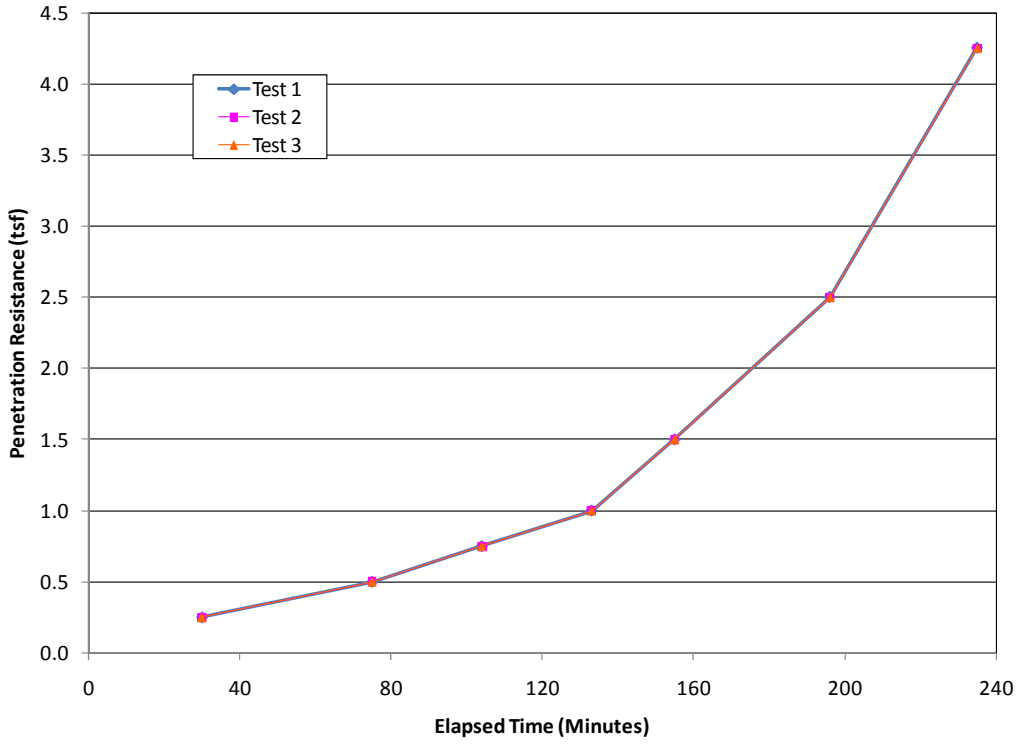




**Figure 210**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 11 Bucket 2**

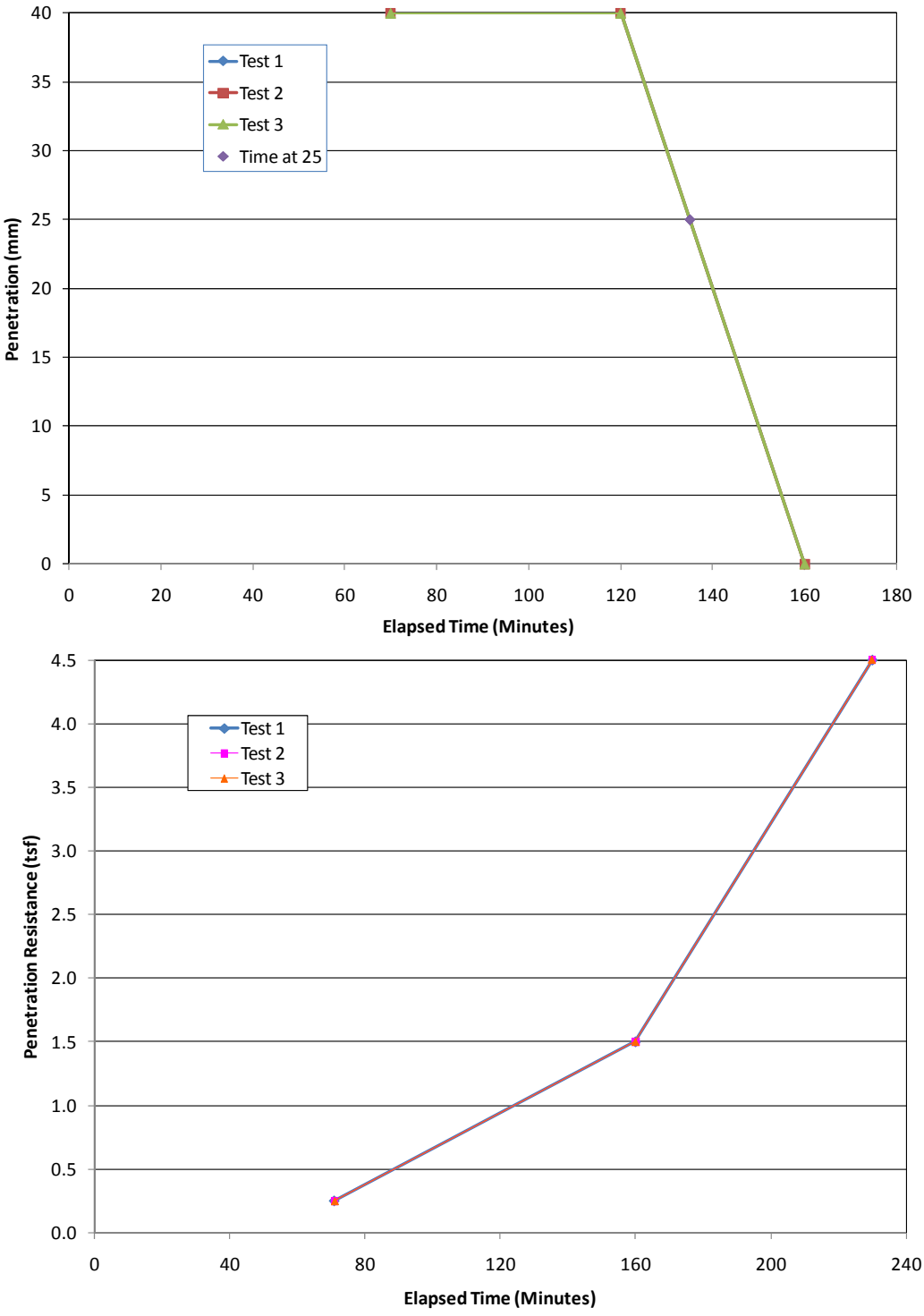



**Figure 211**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 11 Bucket 3**

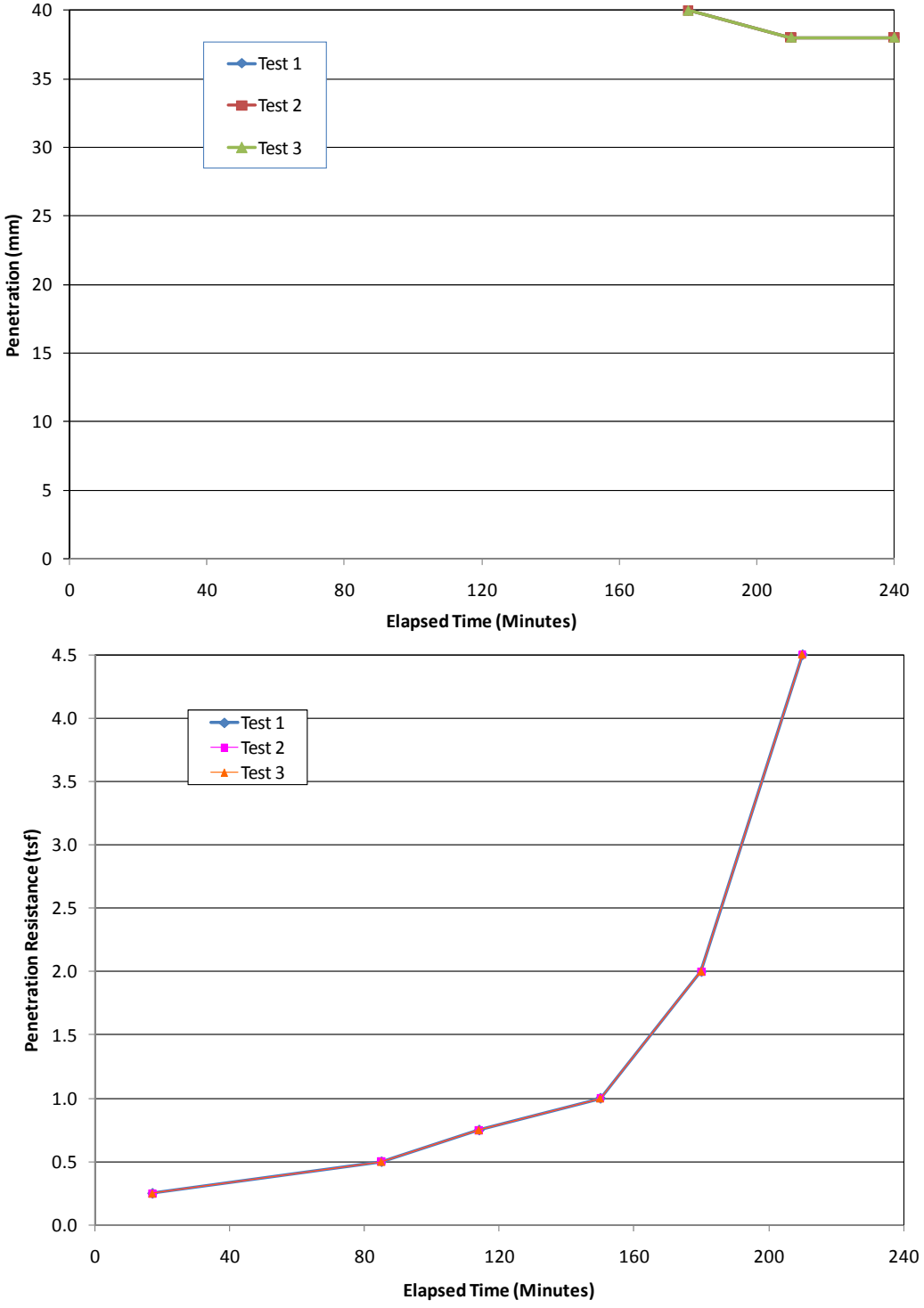



**Figure 212**
**Vicat (top) and pocket penetration resistance (bottom) graphs for Source 11 Bucket 4**

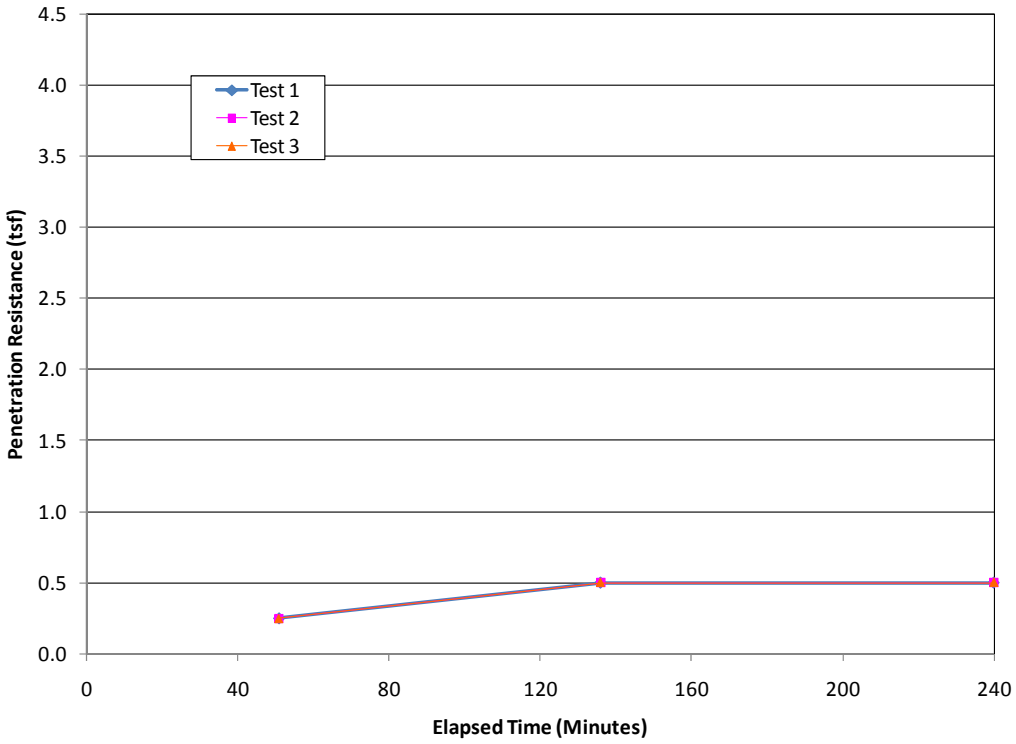



**Figure 213**
**Pocket penetration resistance graph for Source 11 Bucket 7**

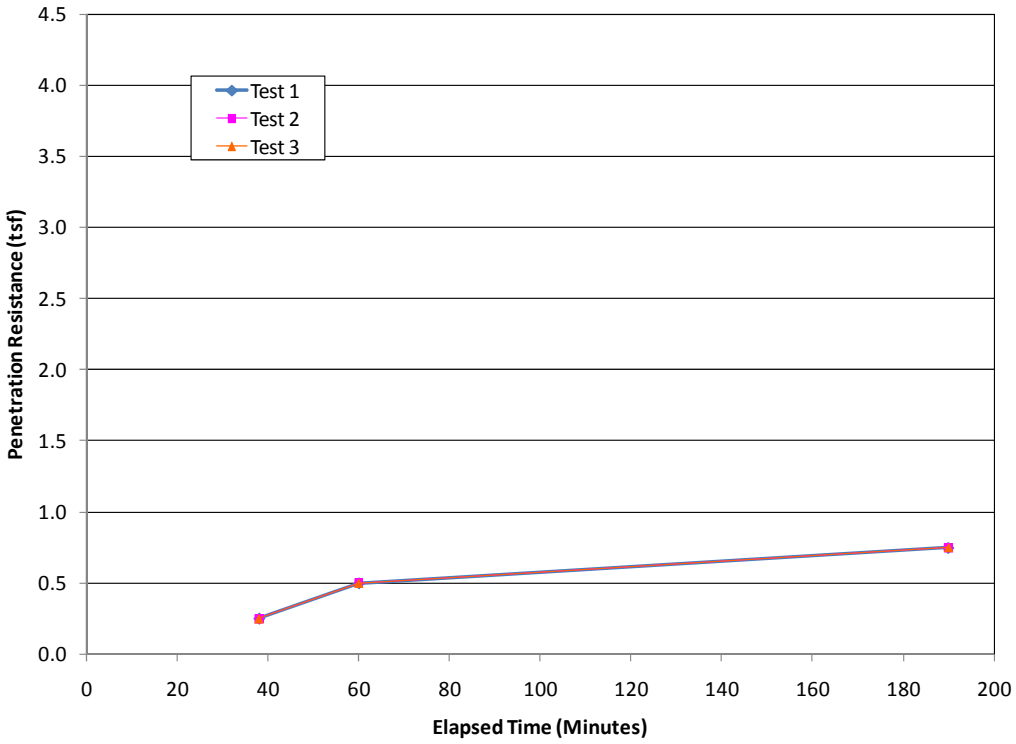



**Figure 214**
**Pocket penetration resistance graph for Source 11 Bucket 8**

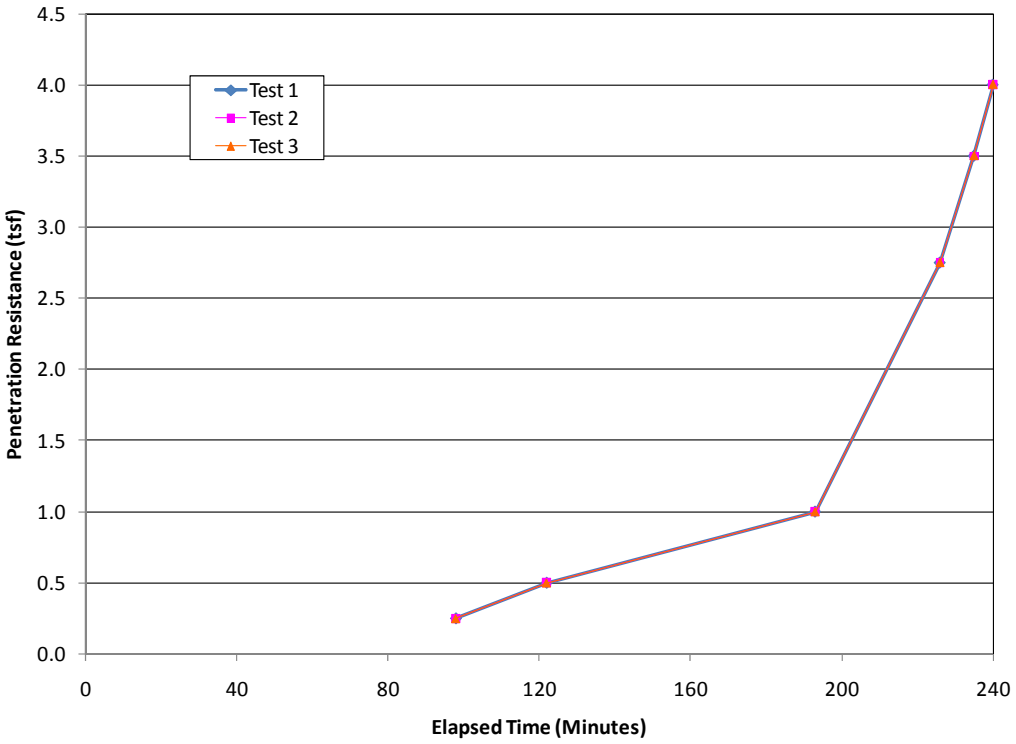



**Figure 215**
**Pocket penetration resistance graph for Source 11 Bucket 9**

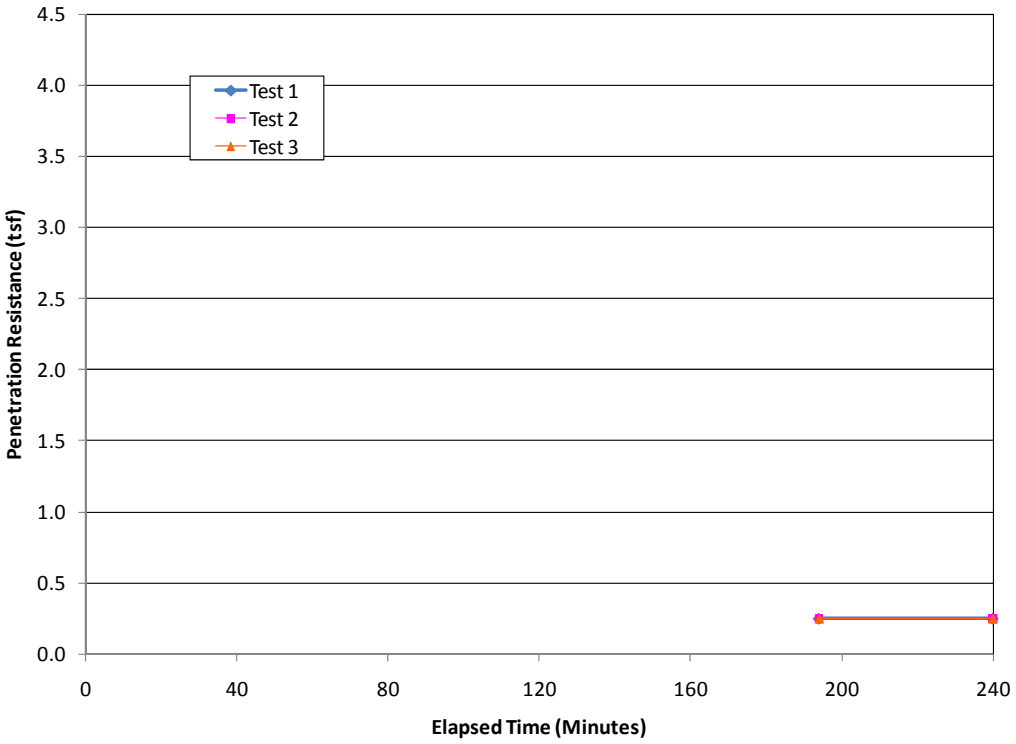



**Figure 216**
**Pocket penetration resistance graph for Source 11 Bucket 10**

# APPENDIX B

## Source 2

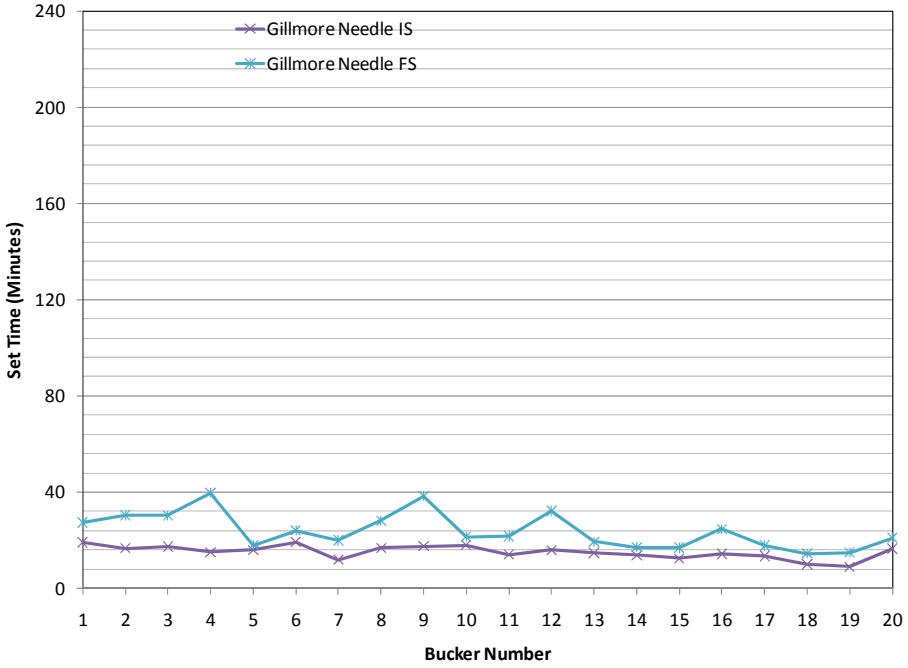



**Figure 217**
**Gillmore needle initial and final set results versus bucket number for Source 2**

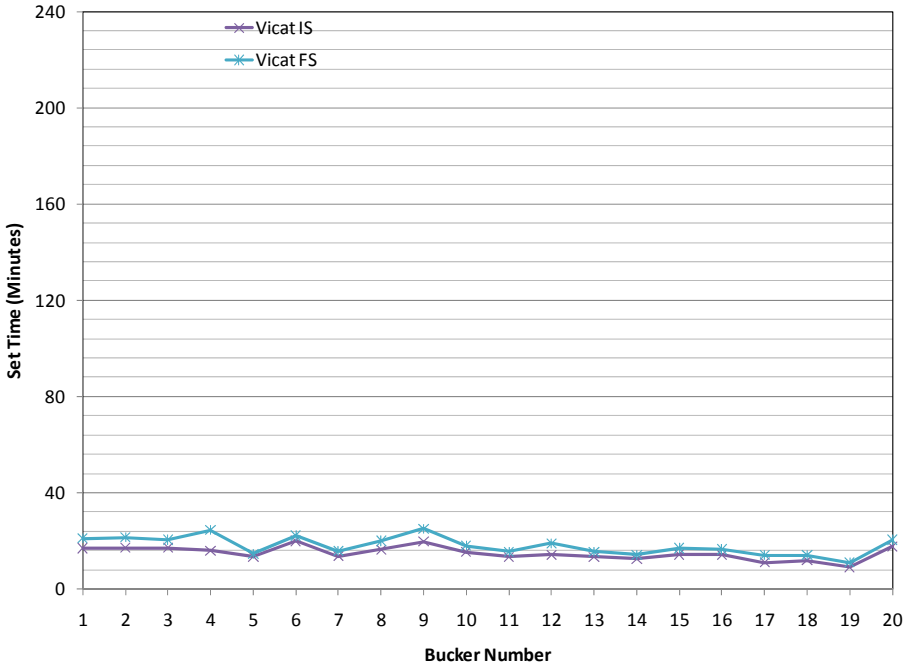



**Figure 218**
**Vicat needle initial and final set results versus bucket number for Source 2**

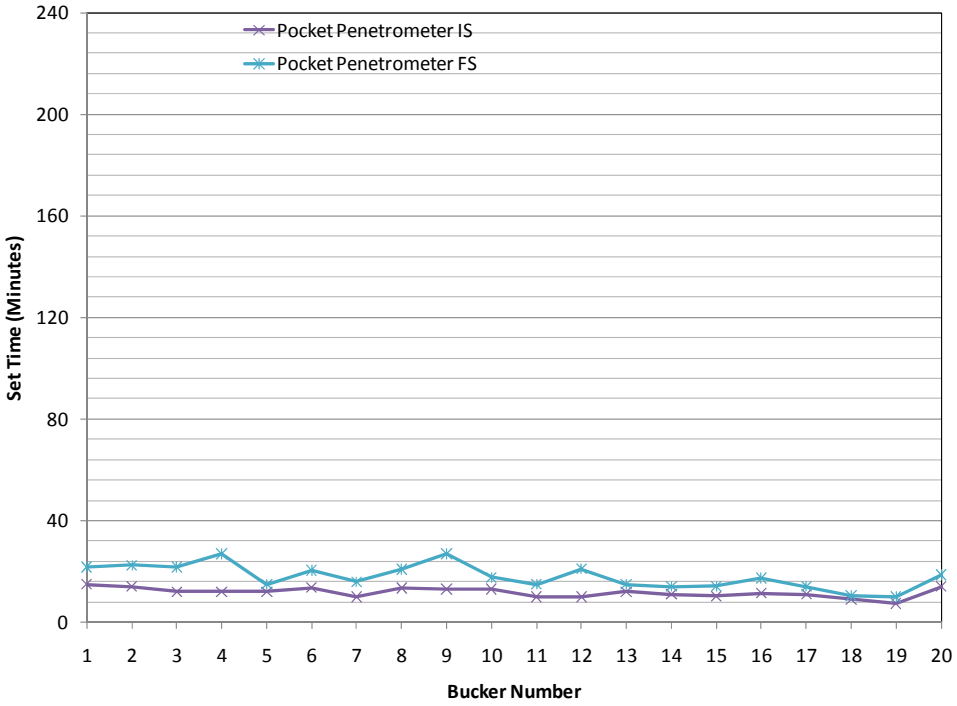



**Figure 219**
**Pocket penetrometer initial and final set results versus bucket number for Source 2**

**Source 3**

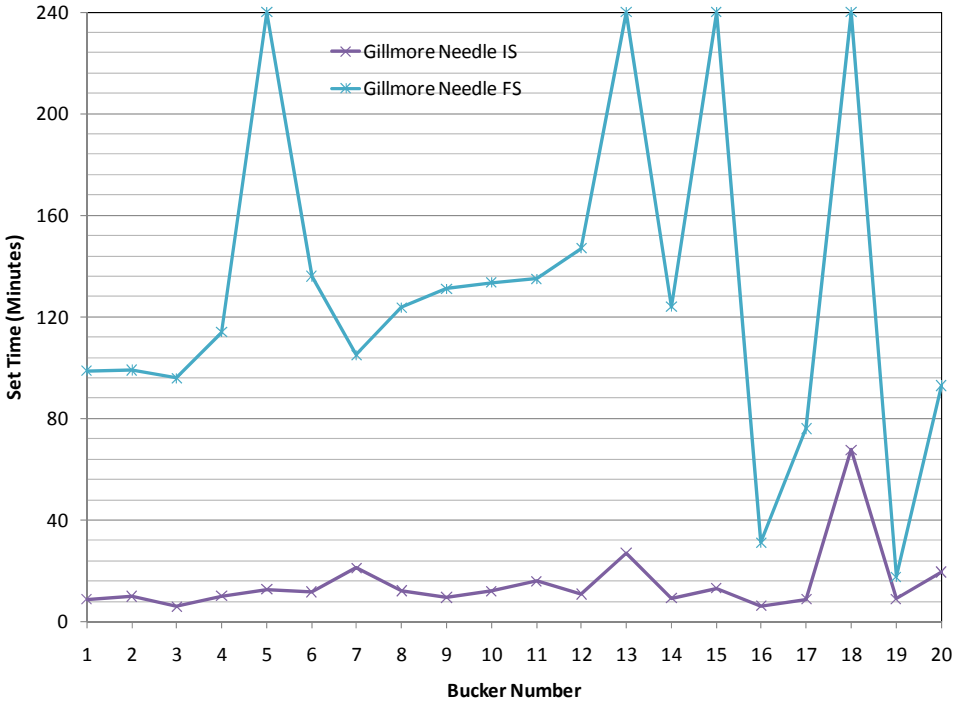



**Figure 220**
**Gillmore needle initial and final set results versus bucket number for Source 3**

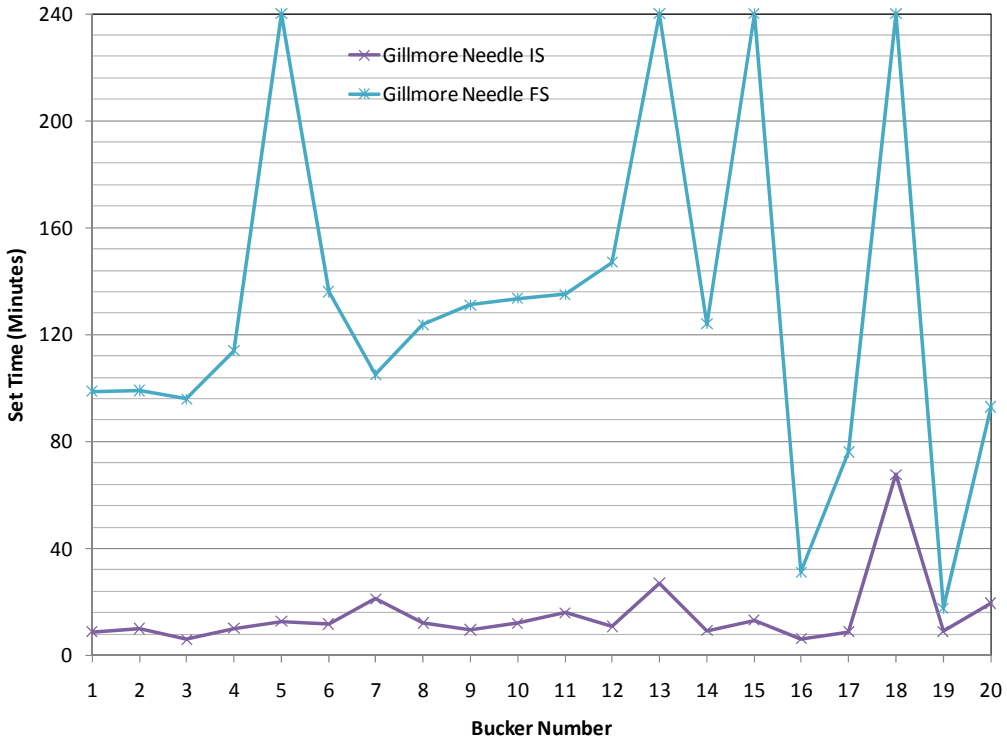



**Figure 221**
**Vicat needle initial and final set results versus bucket number for Source 3**

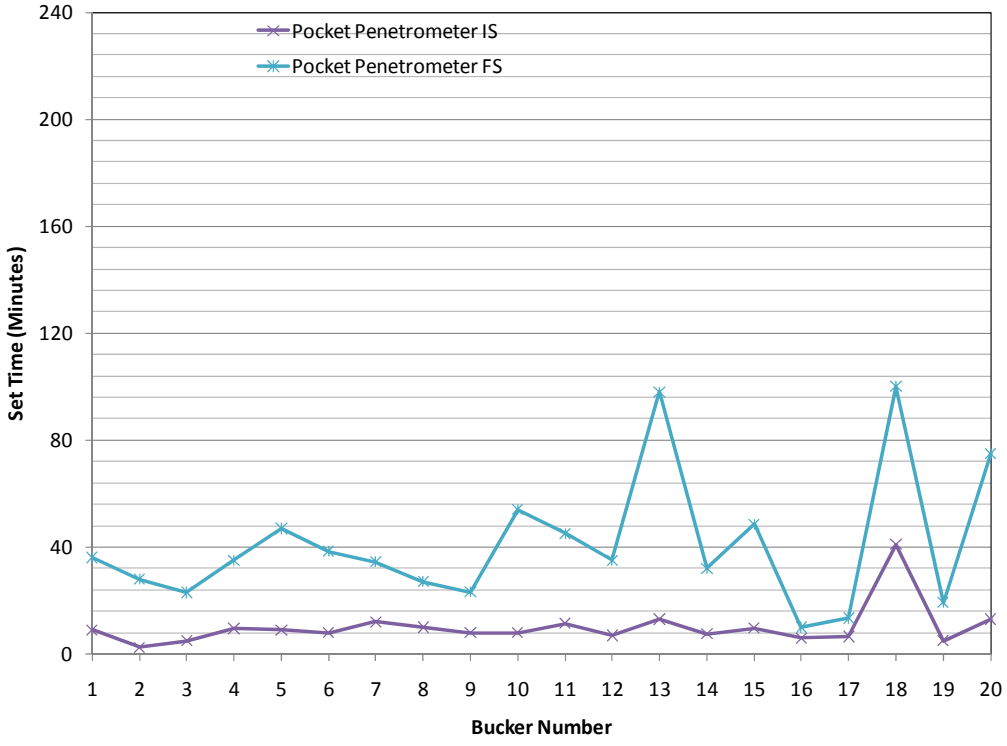



**Figure 222**
**Pocket penetrometer initial and final set results versus bucket number for Source 3**

**Source 4**

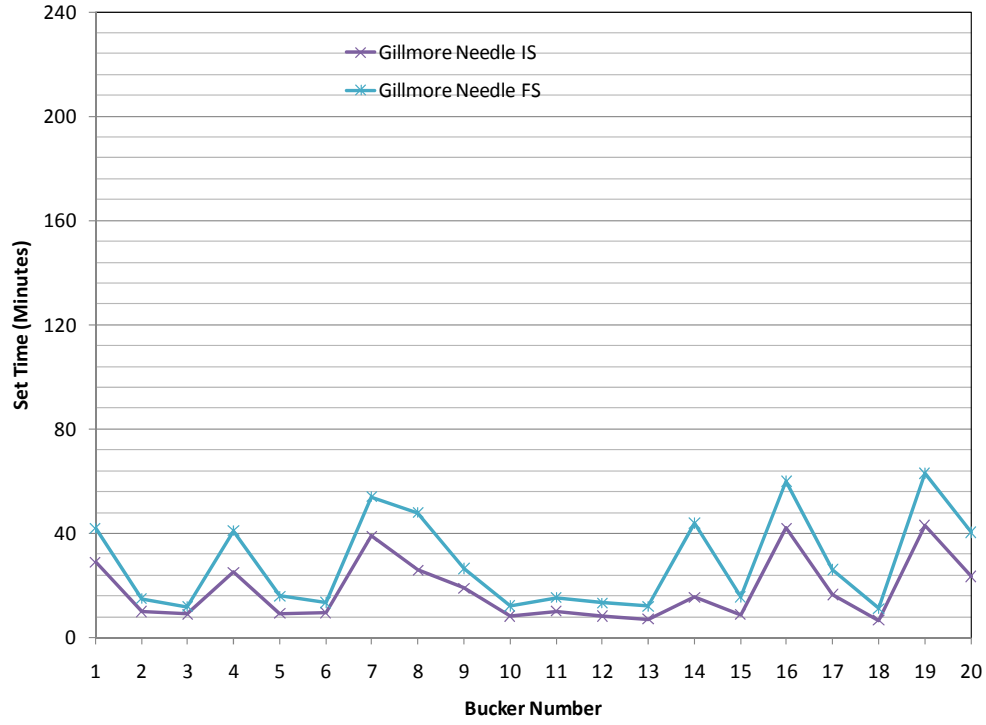



**Figure 223**
**Gillmore needle initial and final set results versus bucket number for Source 4**

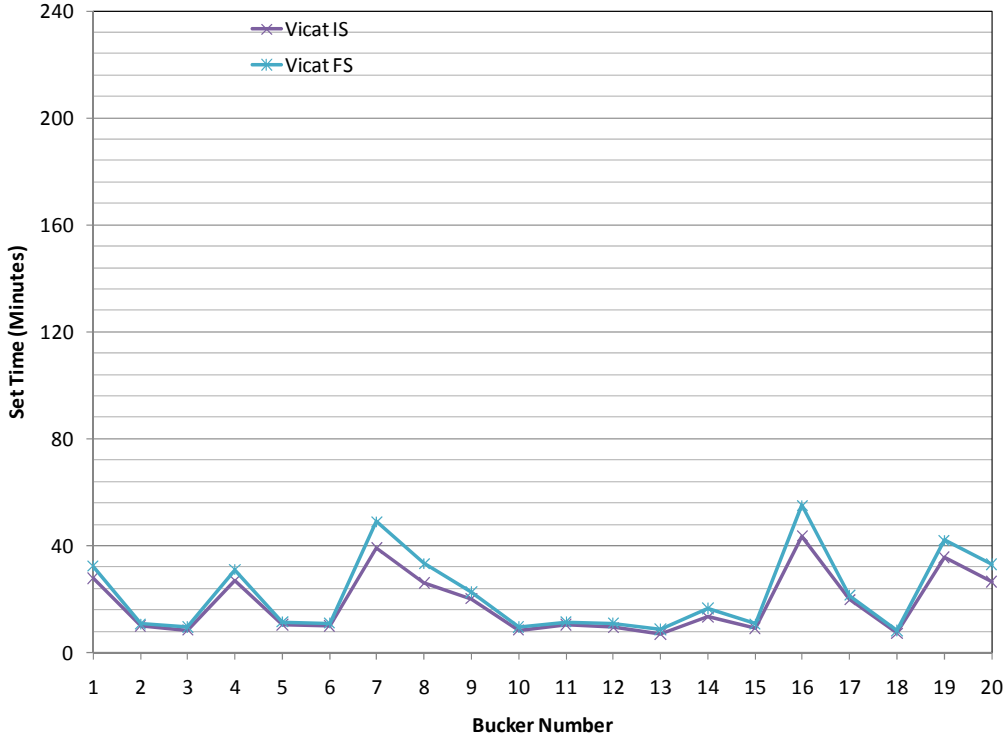



**Figure 224**
**Vicat needle initial and final set results versus bucket number for Source 4**

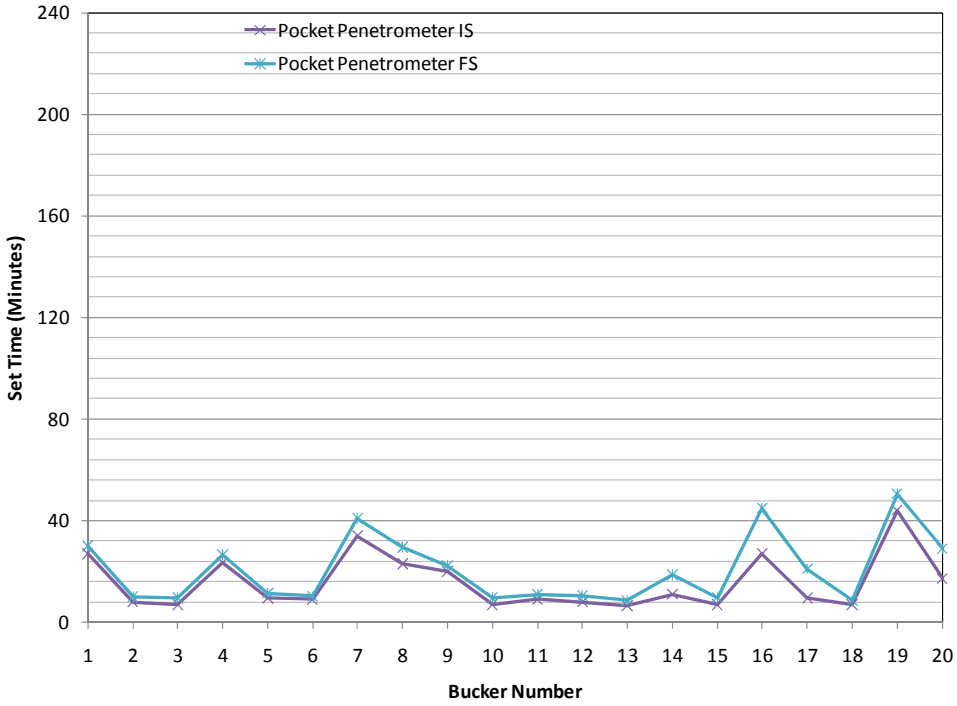



**Figure 225**
**Pocket penetrometer initial and final set results versus bucket number for Source 4**

**Source 5**

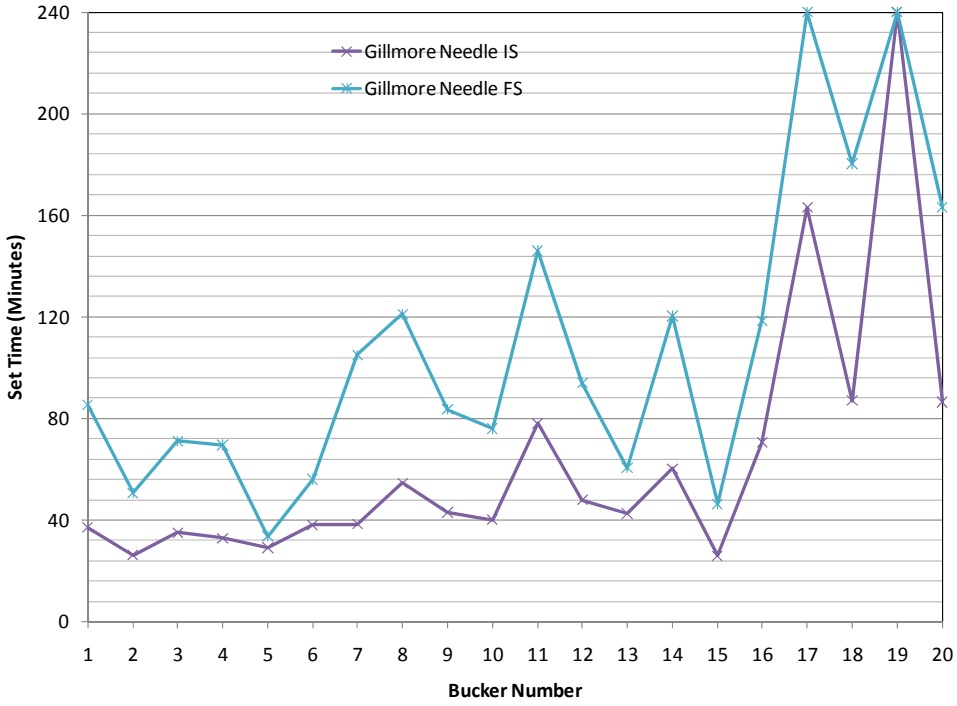



**Figure 226**
**Gillmore needle initial and final set results versus bucket number for Source 5**

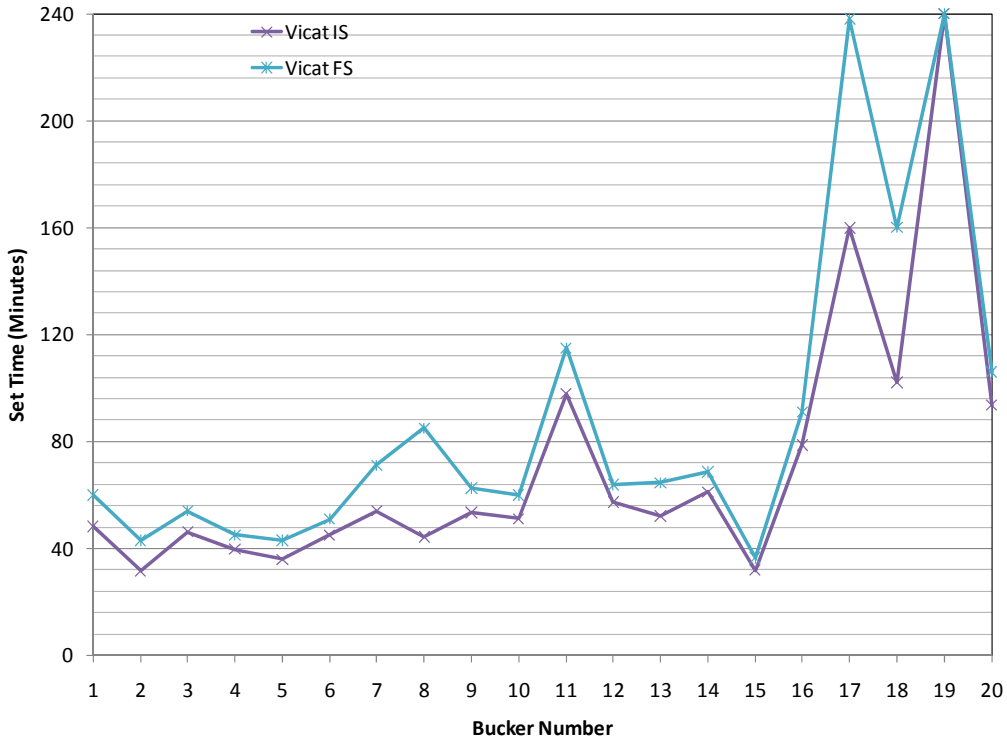



**Figure 227**
**Vicat needle initial and final set results versus bucket number for Source 5**

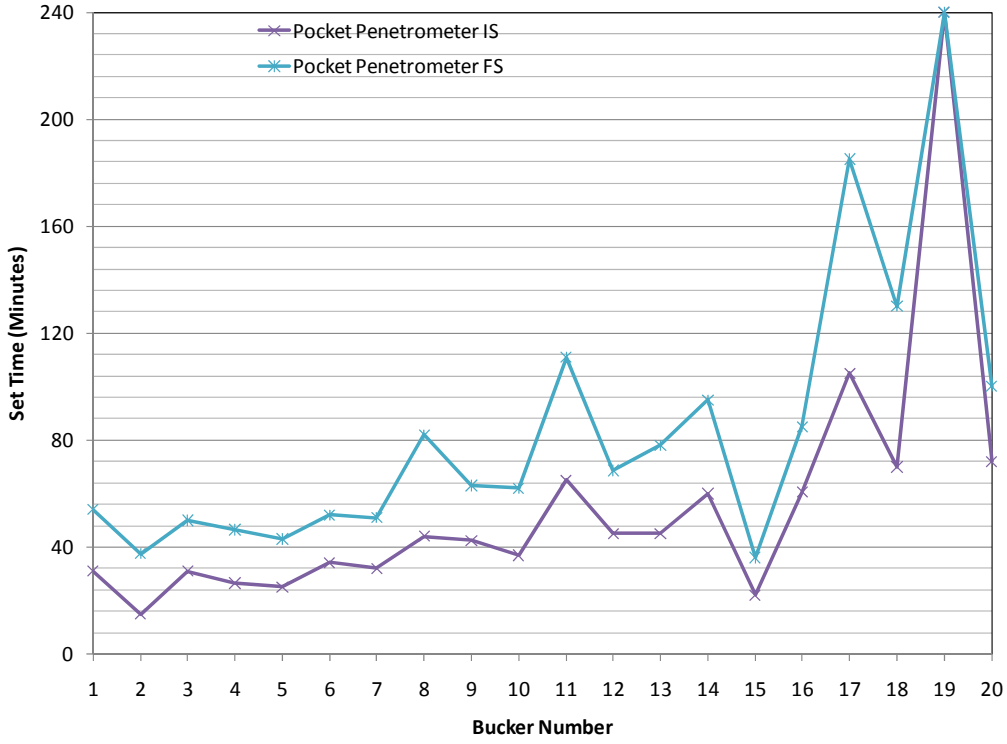



**Figure 228**
**Pocket penetrometer initial and final set results versus bucket number for Source 5**

**Source 6**

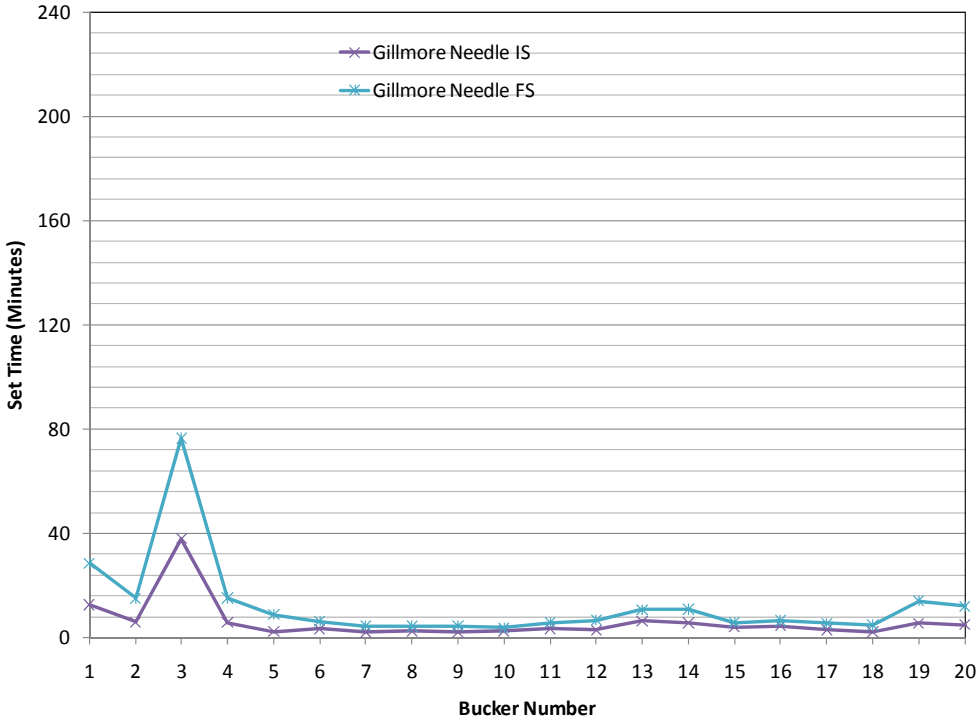



**Figure 229**
**Gillmore needle initial and final set results versus bucket number for Source 6**

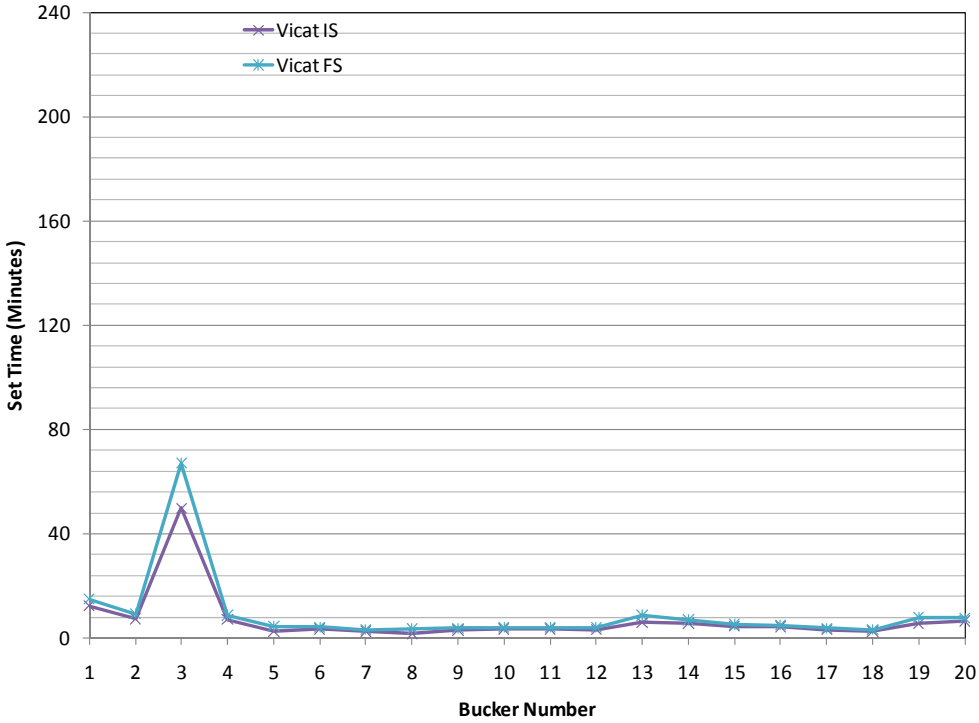



**Figure 230**
**Vicat needle initial and final set results versus bucket number for Source 6**

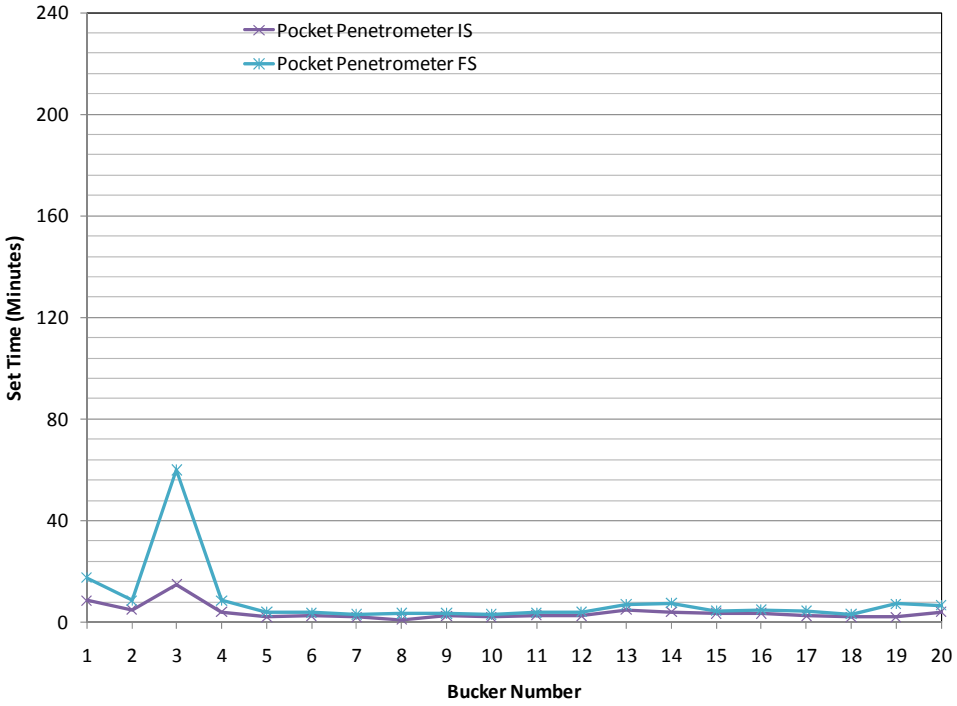



**Figure 231**
**Pocket penetrometer initial and final set results versus bucket number for Source 6**

**Source 7**

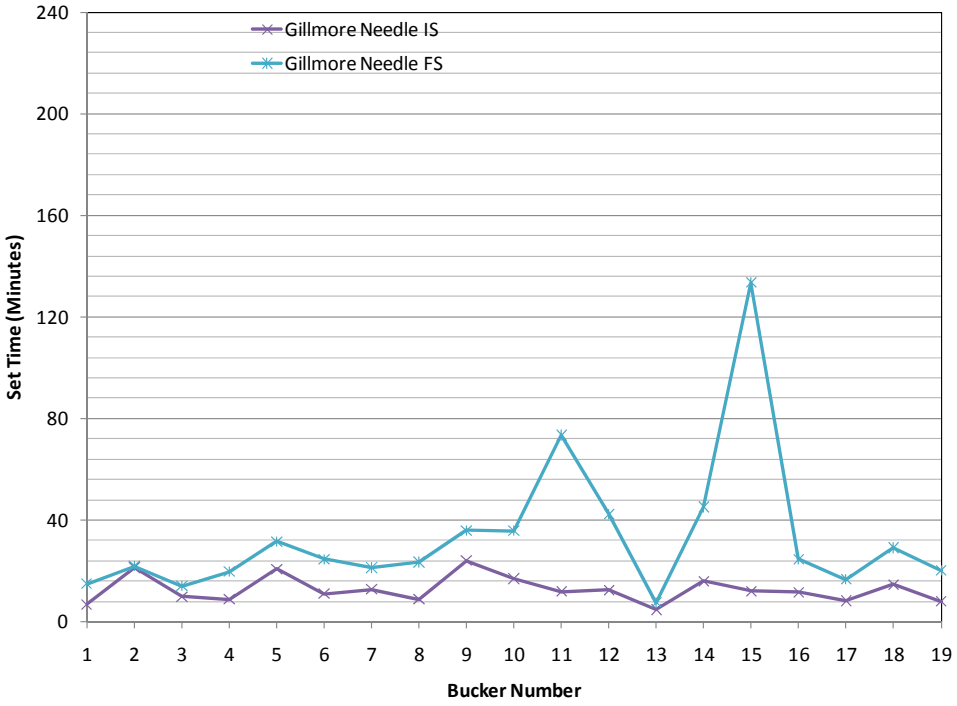



**Figure 232**
**Gillmore needle initial and final set results versus bucket number for Source 7**

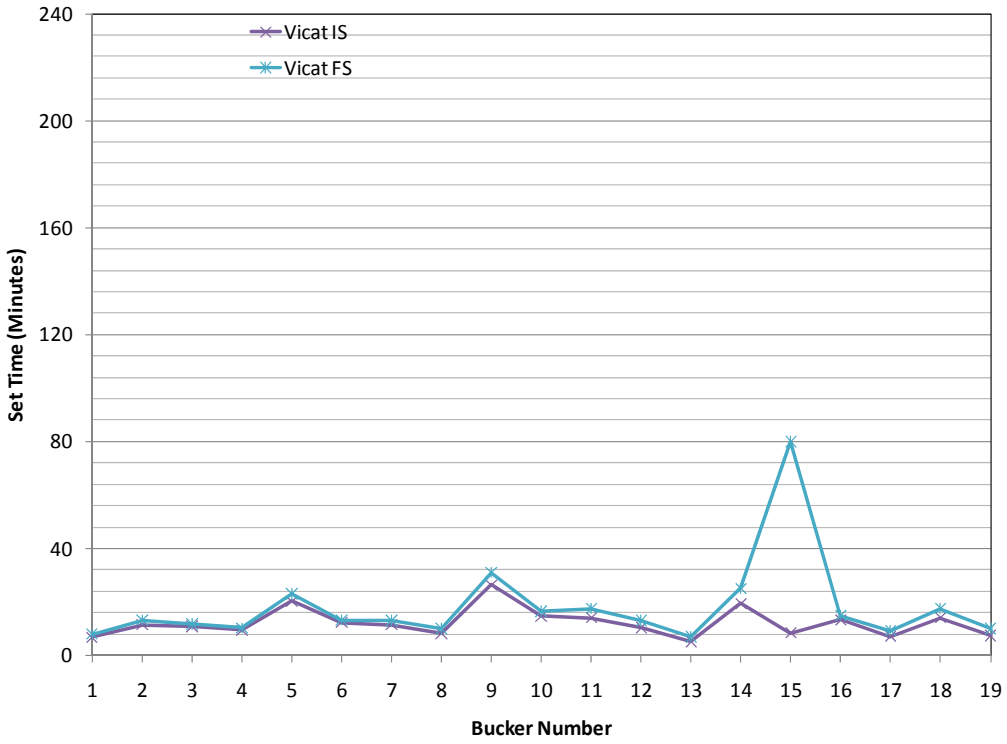



**Figure 233**
**Vicat needle initial and final set results versus bucket number for Source 7**

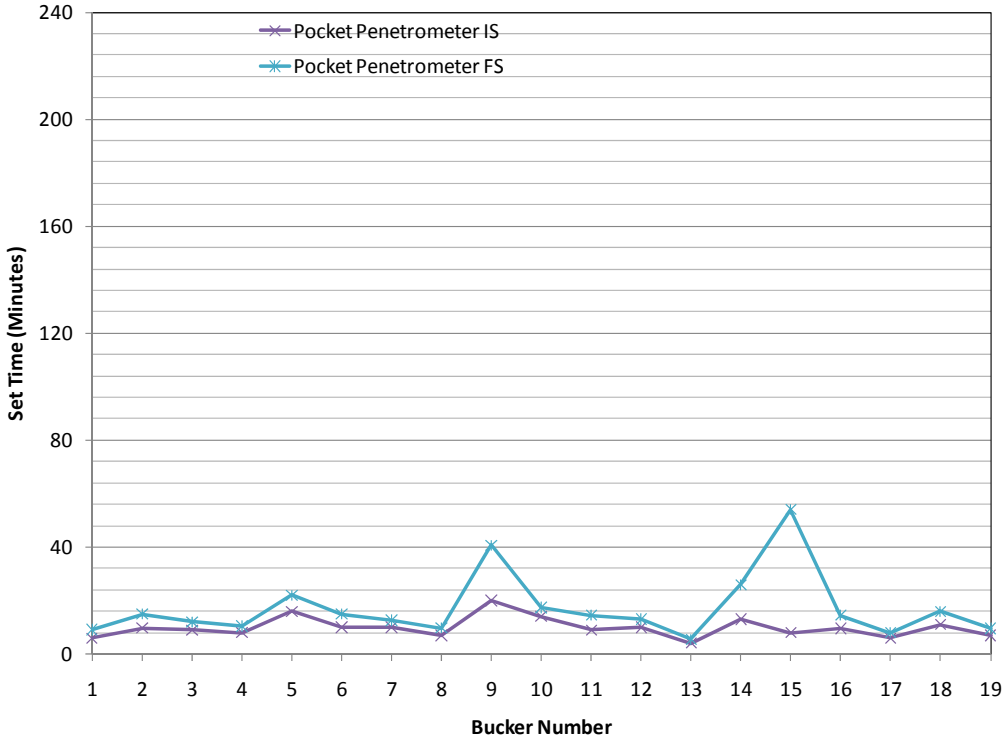



**Figure 234**
**Pocket penetrometer initial and final set results versus bucket number for Source 7**

**Source 9**

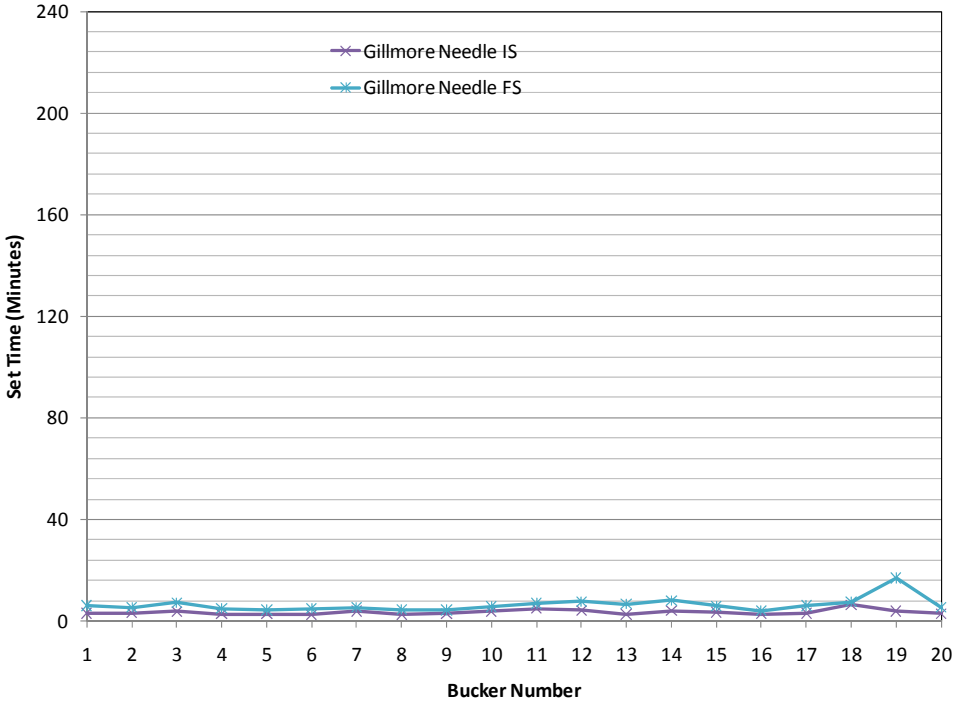



**Figure 235**
**Gillmore needle initial and final set results versus bucket number for Source 9**

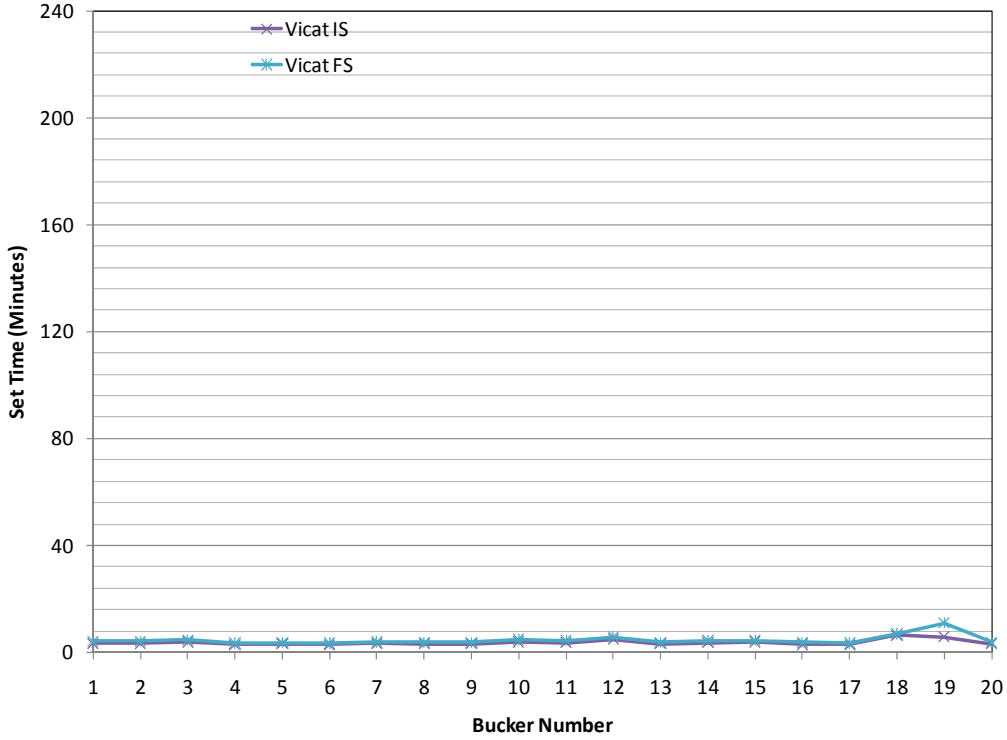



**Figure 236**
**Vicat needle initial and final set results versus bucket number for Source 9**

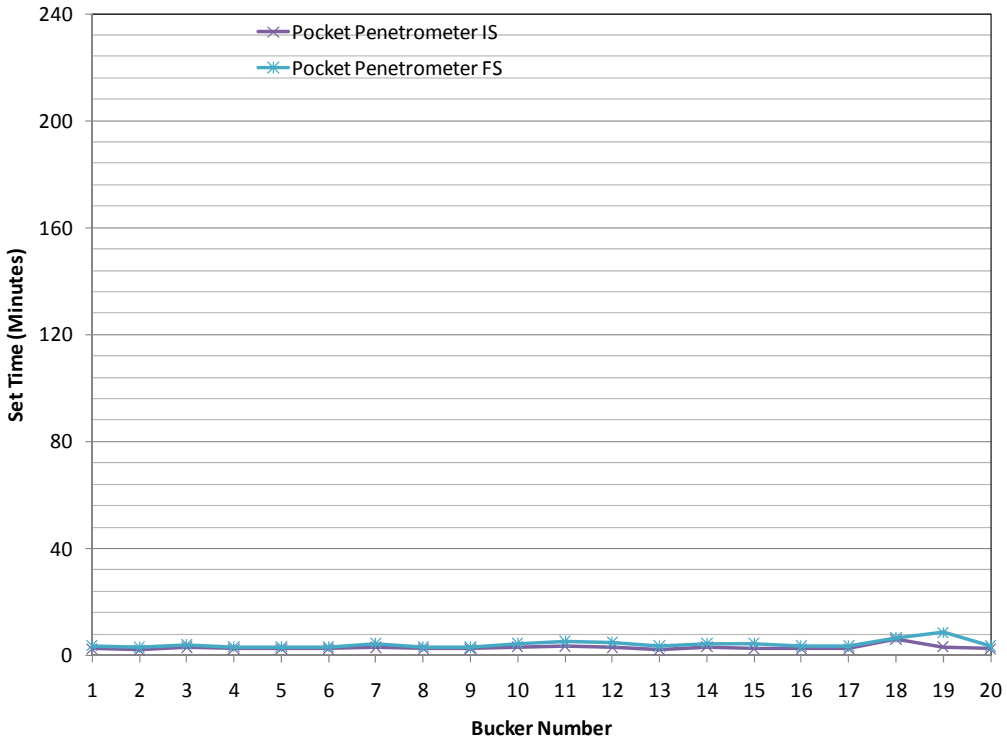



**Figure 237**
**Pocket penetrometer initial and final set results versus bucket number for Source 9**