# 5.0 MICROFINE CEMENT GROUT LABORATORY TESTING AND RESULTS

## 5.1 Introduction

The purpose of the cement grout testing program was to determine if microfine cement grout mixes could be developed with the desired properties necessary for passive site remediation. Based on groutability ratios, microfine cements should be able to penetrate liquefiable formations, as discussed in Section 3.2. The rheological properties and stability characteristics are also important in determining the penetrability of microfine cement grouts. An additional consideration for passive site remediation is the set time. Extended set times will be required for microfine cement grouts to be suitable for this application. Therefore, the microfine cement grout testing program focused on determining the rheological properties, stability characteristics, and set times of different microfine cement grouts. If these results were favorable, strength testing of stabilized sands would also be necessary.

This chapter consists of a description of the testing program, followed by a presentation of the results. A summary of the results is given in Section 5.3.1 and is followed by a detailed presentation. Data for each of the mixes tested is presented in Appendix 1, Microfine Cement Grout Testing Results.

## 5.2 Microfine Cement Grout Testing Program

### 5.2.1 Properties Measured

The purpose of the cement grout testing program was to determine the stability, rheological and setting properties of microfine cement grouts. The stability characteristics measured included bleed and pressure filtration. As discussed in Chapter 3, bleed is the amount of free water that develops with time at the top of a grout column at rest, expressed as a percentage of the total volume. A grout is considered stable if the bleed is less than five percent after two hours. Pressure filtration is a measure of bleed under pressure. The pressure filtration coefficient is a measure of how much water is forced out of a sample under pressure in a given period of time. The rheological properties determined included viscosity and cohesion. The density of the grout was also measured in order to calculate the cohesion. Detailed descriptions of the materials and methods are presented in the following sections.

### 5.2.2 Materials

Three types of cement were used, including Type V Premium Ultrafine Cementitious Grout manufactured by U.S. Grout, Malad City, Utah; Rheocem-900 supplied by Master Builders Technologies, Cleveland, Ohio; and NewCem slag cement manufactured by Blue Circle Cement, Baltimore, Maryland. Type V Premium Ultrafine Cementitious Grout contains 55 percent pumice, 45 percent Type V Portland cement and 1.5 percent Disal Superplasticizer by weight of cement. The average particle size of the cement is 2.5 microns, 90 percent by weight is finer than 5 microns, and the Blaine fineness is 17,100 $cm^2/g$. Blaine fineness is a measure of the specific surface area of the cement particles per gram of cement. It is often used to distinguish between ordinary cement and microfine cement. Rheocem-900 has a Blaine fineness of 10,000 $cm^2/g$ and an average particle size of 3.2 microns. NewCem is ground granulated blast-furnace slag cement manufactured from iron blast furnace slag, with a Blaine fineness of 5200 $cm^2/g$. The NewCem supplied was not microfine cement, but this was not discovered until after the cement testing program was completed.

Three types of dispersants were used, all of which are classified as high-range, water-reducing superplasticizers. Disal Superplasticizer, manufactured by Handy Chemicals, is a polynaphthalene sulfonate sodium salt dispersant. It is supplied as a free flowing powder. Rheobuild 1000 and Rheobuild 2000, manufactured by Master Builders Technologies, are naphthalene sulphonate based and melamine based superplasticizers, respectively. Both are supplied as liquids.

The set-retarding admixture used was Delvo Stabilizer, manufactured by Master Builders Technologies. It is a two-component system comprising a stabilizer and an activator. The stabilizer is a modified, hydroxylated, carbosilicic acid that forms a barrier around the cement particles, preventing hydration (Gause and Bruce 1997). The activator is an inorganic alkaline that dissolves the barrier formed by the stabilizer. Only the stabilizing component of the system was used in this study.

Other admixtures that were used included bentonite, silica fume and welan gum. The bentonite used was Hydrogel, supplied by Wyo-Ben, Billings, Montana. The silica fume used was Rheomac SF100 Compacted Silica Fume supplied by Master Builders Technologies. Welan gum, manufactured by Monsanto, is a polysaccharide gum that is supplied as a free flowing powder. It was supplied by Master Builders Technologies.

### 5.2.3 Methods

#### 5.2.3.1 Mix Design

All of the mixes were designed with a water to cement ratio of 2. This ratio was selected to obtain a combination of low viscosity and high stability. The goal was to develop a stable grout with an apparent viscosity of less than 10 cP and a set time of 50 days. The base mixes consisted of water, dispersant, cement, and a set-retarding admixture. Two base mixes were made, one with Rheobuild 1000 and the other with Rheobuild 2000. Different combinations of stabilizing admixtures were then added to the base mix design. A list of the mixes is shown Appendix 1, Microfine Cement Grout Testing Results. Table A-1 lists all of the mix designs while Table A-2 contains the bleed, pressure filtration, Marsh viscosity, cohesion, wet density, and viscosity results. The 1000 series signifies use of Rheobuild 1000 dispersant, while the 2000 signifies use of Rheobuild 2000 dispersant. Unless otherwise noted, the cement used was Rheocem-900.

#### 5.2.3.2 Sample Preparation

The cement grouts were prepared using the following procedure:

1. The components of each mix were measured and stored in separate containers.

2. A bentonite slurry was made with a portion of the mix water using a Silverson L4RT high-speed shear mixer and hydrated for a period of at least 24 hours prior mixing the cement grout.
3. The welan gum was mixed with a portion of the mix water using a milkshake mixer.
4. The balance of the mix water was placed in the mixing container and the dispersant was added and mixed thoroughly using the Silverson high-speed shear mixer. The purpose of mixing the dispersant into the water prior to adding the cement is to encourage maximum coverage of the cement particles by the dispersant.
5. The cement was added to the mix water and mixed with the high-speed shear mixer for two minutes.
6. The bentonite slurry was added and mixed until thoroughly incorporated.
7. The welan gum slurry was added and mixed until thoroughly incorporated.
8. The silica fume was added and thoroughly incorporated.
9. The set-retarding admixture was added and thoroughly incorporated.
10. The mix was stored in 5-gallon plastic buckets for up to 40 days. The mix was discarded when it became obvious that it would not be suitable for passive site remediation

#### 5.2.3.3 Stability Testing

The bleed was measured by placing a 1000-milliliter sample of the grout in a graduated container and measuring the free water that accumulated at the top of the grout column. The bleed was measured several times during the first day after the grout was mixed and daily thereafter until it stabilized. After the bleed stabilized, it was measured weekly.

An API filter press was used to measure the pressure filtration coefficient. A sketch of the API filter press is shown in Figure 5-1. A 200- or 400-milliliter grout sample was placed in the chamber of the filter press. The chamber was placed securely in the frame and a pressure of 100 psi was applied for 10 minutes. The liquid extruded through the filter paper was collected and measured.

The pressure filtration coefficient was calculated according to the following formula:

$$K_{pf} = V_{fluid\ lost}/(V_{initial} * T^{1/2}) \quad (5\text{-}1)$$

where $K_{pf}$ = pressure filtration coefficient, minutes$^{-1/2}$

$V_{fluid\ lost}$ = volume of fluid extruded from sample, ml

$V_{initial}$ = initial volume in filter press chamber, ml

T = time in minutes

The smaller the pressure filtration coefficient, the more stable the grout.

5.2.3.4 Rheological Testing

As discussed in Section 3.1, stable cement grouts behave like Bingham fluids. Therefore, both the viscosity and cohesion need to be measured to provide a complete picture of the rheological behavior. The most common method of assessing the rheological behavior of cement grouts in the field is to measure the amount of time it takes one liter of grout to flow through a Marsh cone or funnel. This measurement is called the Marsh time or Marsh viscosity and gives an idea of the rheologic behavior of the grout. However, it does not provide a specific viscosity or cohesion measurement. Lombardi (1985) devised a cohesion meter that can be used in conjunction with the Marsh viscosity to determine the apparent viscosity of the grout. The plot used to determine the apparent viscosity given the cohesion and the Marsh viscosity is shown in Figure 5-2 (Lombardi 1985). Using the cohesion in millimeters and the Marsh viscosity in seconds, a value of viscosity divided by unit weight, or $\mu/\gamma$, is read off the chart in units of meter-seconds. This value is multiplied by the unit weight and converted centipoises (cP) to give the apparent viscosity. Lombardi (1985) compared the results obtained using this chart with results obtained using a rotational viscometer, which is a more precise method for determining the viscosity. He found that the values obtained using the chart compared favorably with results from the rotating viscometer.

The Marsh viscosity of the microfine cement grouts was measured using a Marsh cone or funnel. A picture of the Marsh funnel is shown in Figure 5-3. API Method RP 13B-1 was used to measure the Marsh viscosity. The spout of the funnel was covered with a fingertip and the funnel was filled to the bottom of the screen with grout. The amount of time required for exactly one liter of grout to exit the cone was recorded. This time is called the Marsh viscosity or Marsh time. The Marsh viscosity of water at 70 degrees Farenheit is 26+/- 0.5 seconds.

A Lombardi plate cohesion meter was used to measure the cohesion. A picture of the plate cohesion meter is shown in Figure 5-3. There is no ASTM standard the plate cohesion meter, but the dimensions are described in Weaver (1991). The meter was constructed of a thin steel plate 10 centimeters by 10 centimeters by about 3 millimeters thick. Grooves were cut into both sides of the plate to give it a rough surface. This allows grouts with some cohesion to stick to the plate when it is immersed in grout. A handle was welded to the side of the plate to aid in immersing the plate in the grout mixture. Since the meter is not standard, the value of cohesion obtained is relative to the base mix. While not absolute, this value can be used for comparing different grout mixes.

The procedure for measuring the cohesion follows.

1. The plate was weighed.
2. The plate was immersed in the grout, removed, and weighed again.
3. The weight of the grout that stuck on the plate was calculated by subtracting the initial weight from the final weight.
4. The weight was divided by the area of the plate to obtain the weight per unit area. The weight per unit area was divided by the specific weight of the grout to determine the thickness of grout that stuck to the plate. This thickness, when converted to millimeters, is the cohesion (Deere and Lombardi 1985).

The unit weight or “mud weight” of the grout was determined using a mud balance apparatus, which is shown in Figure 5-4. The unit weight was determined in accordance with the API Method RP 13B-1 and described in Weaver (1991).

1. The balance was placed on a level location bench and the balance cup was filled with grout.
2. The cap was carefully placed on the balance cup and rotated to expel the excess grout through the hole in the top of the cap. Excess grout was cleaned off the cup.
3. The beam was placed on the support and balanced by moving the rider along the graduated scale until the bubble was centered.
4. The unit weight was read at the side of the rider closest to the knife edge.

5.2.3.5 Measurement of Set Time

The set time was measured using a Soiltest pocket penetrometer. This method was used by Schwarz and Krizek (1992). Schwarz and Krizek (1992) arbitrarily defined the initial set as the time when the penetrometer read 0.5 tons/ft$^2$. The same definition was used for this study.

## 5.3 Microfine Cement Grout Testing Results

### 5.3.1 Summary

As noted earlier, the goal of the microfine cement grout testing program was to develop a stable grout with a viscosity of less than 10 cP and a set time of at least 50 days. Three different types of cement were used and a total of 46 mixes were made. All of the mixes had a water to cement ratio of 2. Two base mixes containing cement, dispersant, and a set-retarding admixture were used for baseline measurements. The remaining 44 mixes consisted of a base mix with different combinations of stabilizing admixtures or a different type of cement. A list of the mixes and the results of the testing are presented in Tables A-1 and A-2, respectively, of Appendix 1. A brief summary of the results is presented below and detailed results are presented in the following sections.

In general, the base mixes had the lowest apparent viscosities, but bled excessively and had the highest pressure filtration coefficients. While the viscosities of the base mixes were below 10 cP, the bleed exceeded 35 percent in 1 day. In contrast, the stabilized mixes had good or excellent stability, but poor rheological properties. Many mixes had less then 5 percent bleed for 2 or 3 days, but the apparent viscosity was often above 20 or 30 percent. In addition, the stabilized mixes tended to become less stable with time. Bleed often increased above 10 percent after several days. The set retarding admixture was successful in delaying the hydration for more than 50 days. In cases where the highest doses were used, the set was retarded for more than 100 days, when the test was terminated.

When additives were used singly, bentonite was excellent at reducing bleed, but did not improve the pressure filtration coefficient and caused a dramatic increase in the apparent viscosity. Alone, neither silica fume nor welan gum were very good at reducing bleed, but they reduced the pressure filtration coefficient and had no detrimental effect on the apparent viscosity and cohesion. When additives were used together, the best overall performance was achieved with the combination of bentonite and welan gum.

Although extended set times were obtained, microfine cement grouts were eliminated from further consideration due to their rheological and stability properties.

### 5.3.2 Stability Testing Results

The cement results below are presented for eight representative mixes. The components of the mixes are shown in Table 5-1. Mix 1-1000 was a base mix. Mixes 2-1000, 4-1000, and 7-10000 were selected because each contained only one stabilizer. Mixes 3-1000, 6-1000, 10-1000, and 15-1000 were chosen because each contained a combination of stabilizers. These mixes illustrate the range of behavior of all the mixes.

Plots of bleed with time are shown in Figures 5-5 and 5-6 for the representative mixes. The base mix bled 18 percent in 2 hours, 37 percent in 18 hours, and then leveled off at a value of 43 percent after 5 days. Of the mixes with a single stabilizer, bentonite was most successful in reducing the bleed while silica fume was least successful. Mix 2-1000 had 3 percent bentonite; bleed was reduced to less than 5 percent for about 1 day and then increased to about 14 percent after about

4 days. Mix 7-1000 contained 5 percent silica fume but the bleed was about 30 percent after 1 day and increased to 35 percent after 2 days. The mix with 0.1 percent welan gum (7-1000) bled 20 percent after 1 day and 25 percent after 2 days.

Table 5-1 Components of Representative Mixes

| Mix | Dispersant (% BWOC) | Set-Retarder (% BWOC) | Bentonite (% BWOC) | Welan Gum (% BWOC) | Silica Fume (% BWOC) |
|---|---|---|---|---|---|
| 1-1000 | 1.5 | 10 | - | - | - |
| 2-1000 | 1.5 | 10 | 3 | - | - |
| 3-1000 | 1.5 | 10 | 3 | - | 5 |
| 4-1000 | 1.5 | 10 | - | - | 5 |
| 6-1000 | 1.5 | 10 | - | 0.05 | 5 |
| 7-1000 | 1.5 | 10 | - | 0.1 | - |
| 10-1000 | 1.5 | 10 | 2 | 0.05 | - |
| 15-1000 | 1.5 | 10 | 1.5 | 0.1 | - |

When combinations of additives were used, those containing bentonite were most successful in reducing bleed. Mixes 3-1000, 10-1000, and 15-1000 maintained less than 5 percent bleed for up to 3 days. Mix 3-1000 contained 3 percent bentonite and 5 percent silica fume and had less than 5 percent bleed for 2 days and less than 10 percent bleed after 4 days. Mixes with a combination of bentonite and welan gum were also successful in reducing bleed to less than 5 percent for 2 to 3 days. As the percentage of bentonite decreased, the amount of bleed increased slightly, as demonstrated with Mixes 10-1000 and 15-1000. Mix 10-1000 contains 3 percent bentonite while mix 15-1000 contains 1.5 percent bentonite. The maximum bleed for Mix 10-1000 was 12 percent, while the maximum bleed for Mix 15-1000 was 13 percent. Combinations of silica fume and welan gum were least successful in reducing bleed. Mix 6-1000 contained 5 percent silica fume and 0.05 percent welan gum and bled more than 15 percent after 1 day and then leveled off at 23 percent bleed after 2 days. None of the grouts were able to maintain less than 5 percent bleed for longer than 3 or 4 days.

The pressure filtration results are shown in Table 5-2 for the representative mixes. The pressure filtration coefficient for Base Mix 1-1000 was initially 0.14 $min^{-1}$ but increased to 0.21 $min^{-1}$ after 7 days. Of the single-additive mixes, silica fume and welan gum both reduced the pressure filtration coefficient significantly. Bentonite alone lowered the coefficient only slightly. The best reductions in the pressure filtration coefficients were obtained when a combination of additives was used. However, as the mixes aged, the pressure filtration coefficients increased to about the same value as for the base mix.

Table 5-2 Pressure Filtration Coefficients for Representative Mixes

| Mix | Additive | $K_{pf}$ ($min^{-1}$) Initial | $K_{pf}$ ($min^{-1}$) 16 hours | $K_{pf}$ ($min^{-1}$) 2 – 4 days | $K_{pf}$ ($min^{-1}$) > 7 days |
|---|---|---|---|---|---|
| 1-1000 | None | 0.14 | - | - | 0.21 |
| 2-1000 | Bentonite | 0.11 | - | - | - |
| 4-1000 | Silica fume | 0.081 | - | - | - |
| 7-1000 | Welan gum | 0.093 | - | - | - |
| 3-1000 | Bentonite, silica fume | 0.11 | 0.089 | - | - |
| 6-1000 | Silica fume, welan gum | 0.084 | - | - | - |
| 10-1000 | Bentonite, welan gum | 0.074 | - | .097, 0.11 | 0.23 |
| 15-1000 | Bentonite, welan gum | 0.062 | 0.097 | 0.11 | 0.19 |

### 5.3.3 Rheological Testing Results

The rheological properties tested included the Marsh viscosity and cohesion. The Marsh viscosity and cohesion can be used together to determine an apparent viscosity for each mix (Lombardi 1985). The specific weight of the grout was also determined to use in calculating the cohesion. Marsh viscosity results for the eight representative mixes are shown in Figures 5-7 and 5-8. The results in Figure 5-7 are for mixes with a single additive while the results in Figure 5-8 are for mixes with a combination of additives. The initial Marsh viscosity of the base mix was 29 seconds. The Marsh viscosity of water is 26 +/- seconds.

Bentonite increased the viscosity from 29 to 39 seconds while silica fume had no effect on the initial value and welan gum caused a slight decrease from 29 to 28 seconds. The combination of silica fume and bentonite (Mix 3-1000) caused the Marsh viscosity to nearly double from 29 to 57 seconds. The combination of bentonite and welan gum (Mix 10-1000) caused the Marsh viscosity to increase from 29 seconds to 39 seconds of the mixes 10, 15, and 18 ranged from 30 to 51 seconds.

In general, the Marsh viscosity of the stabilized mixes increased rapidly in the first 24 hours and then decreased gradually over several days to a value close to or below the initial Marsh viscosity. The increase is attributed to initial hydration that is then stopped by the set retarder. The gradual decrease in viscosity with time is attributed to the gradual breakdown of the stabilizers.

There was a similar trend with respect to cohesion. Plots of cohesion versus time are presented in Figures 5-9 for mixes with single additives and in Figure 5-10 for mixes with a combination of additives. The cohesion of the base mix was very small. Mixes with bentonite had the largest increase in cohesion. The cohesion also showed a rapid increase during the first day and then gradually decreased over the course of several days.

The Marsh viscosity and cohesion were used to determine an apparent viscosity for each mix as shown in Figure 5-11. The apparent viscosity of the base mix is consistently 10 cP or less. The apparent viscosity of the stabilized mixes is generally between 10 and 20 cP, but the peak at 24 hours is between about 25 and 35 cP.

### 5.3.4 Differences in Cements Tested

Three different types of microfine cement were used. The first was Rheocem-900 microfine cement supplied by Master Builders Technologies. The second was Type V Premium Ultrafine cement supplied by US Grout. The third was NewCem slag cement supplied by Blue Circle cement. The properties of these cements were discussed in Section 5.2.2. There were significant differences in the behavior of the mixes formulated with different cements. Most of the mixes were made with Rheocem-900. However, NewCem slag cement was substituted for 80 percent of the microfine cement in two mixes. Slag cement hydrates more slowly than regular microfine cement. The purpose in substituting some slag cement for regular microfine cement was to extend the set time. If a long set time could be achieved using slag cement, the amount of set retarding admixture, which is expensive, could be decreased.

A comparison of results is shown in Table 5-3. The NewCem slag cement mixes bled excessively within one day, although the pressure filtration and Marsh viscosity results were better than comparable mixes that used all Rheocem-900. Ordinary grinds of cement typically have a much higher bleed than microfine cements. As noted earlier, the NewCem supplied was not a microfine grind, which was not realized until the test mixes bled excessively. Therefore, NewCem was not used for any other test mixes.

Table 5-3 Comparison of Results between Rheocem-900 and NewCem Slag Cement

| | Bleed 2 hours | Bleed 1 day | Apparent Viscosity | Pressure Filtration |
|---|---|---|---|---|
| Microfine 11-1000 | 1 | 5 | 14 | .059 |
| Slag 19-1000 | 34 | 34 | 7 | .081 |

A comparison of results between Rheocem-900 and Type V Premium ultrafine cement mixes is shown in Table 5-4. Ultrafine cement had slightly better rheological properties and comparable stability properties to microfine cement grouts. However, the ultrafine cement mixes set about twice as quickly as comparable mixes using microfine cement grouts.

Table 5-4 Comparison of Results between Rheocem-900 and Type V Premium Cements

| | Bleed 2 hours | Bleed 1 day | Apparent Viscosity | Pressure Filtration | Set Time |
|---|---|---|---|---|---|
| Microfine | | | | | |
| 4-1002-NS | 3 | 10 | 7 | .077 | 38 hours |
| 4-1002-D2.5 | 2 | 11 | 9 | .080 | 43 days |
| Ultrafine | | | | | |
| 5-1002-NS | 1 | - | 11 | 0.13 | 12 hours |
| 5-1002-D2.5 | 1 | 11 | 9 | 0.067 | 22 days |

### 5.3.5 Set Time Results

When no Delvo Stabilizer was added, the Rheocem-900 base mix with 2% dispersant set in about 40 hours. When Type V Premium ultrafine cement was used instead of Rheocem-900, the base mix without Delvo Stabilizer set in less than 13 hours. When 2.5% Delvo was used, the set was delayed for at least 43 days when Rheocem-900 was used and for about 20 days when Type V Premium ultrafine cement was used. When 10 percent Delvo Stabilizer was added, the set time of both cements was retarded for more than 100 days, when the test was terminated. It is not known whether these mixes will eventually set or if the hydration was prevented by the overdose of set retarding admixture.

## 5.4 Conclusions

The goal of the cement testing program was to develop a stable grout with and apparent viscosity of less than 10 cP and a set time of 50 days. The base mix met the viscosity requirement, but was unstable. Mixes with additives had better stability characteristics, but did not meet the viscosity requirement. Additionally, the stability of mixes with additives tended to decrease after several days. Set times of at least 43 days were measured; samples with the highest doses of set-retarding admixtures had not set when the test was terminated after 100 days. Based on stability and viscosity results, microfine cement was eliminated from further consideration as a potential stabilizer for passive site remediation.

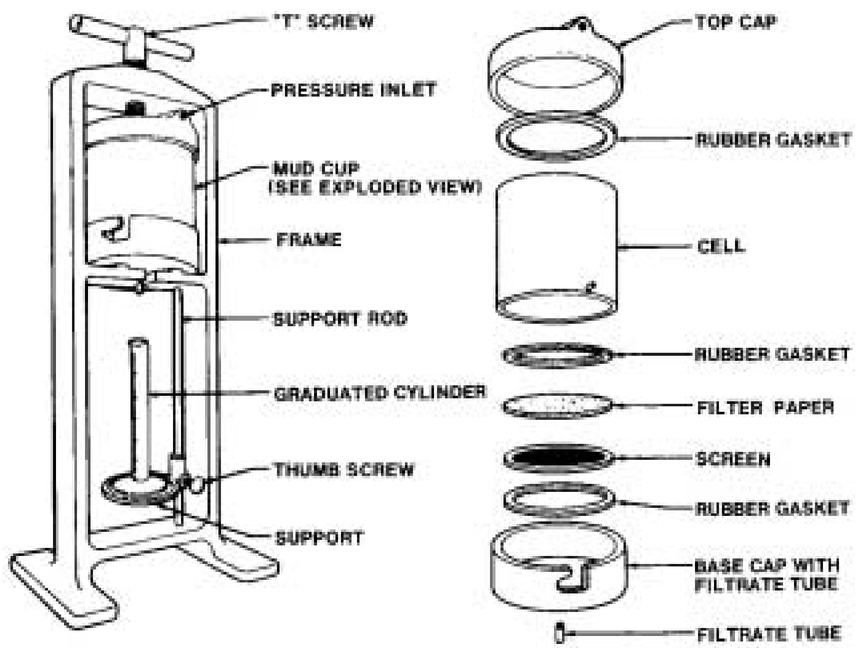



Figure 5-1 API Filter press and cell assembly, exploded view (After Fann 1995)

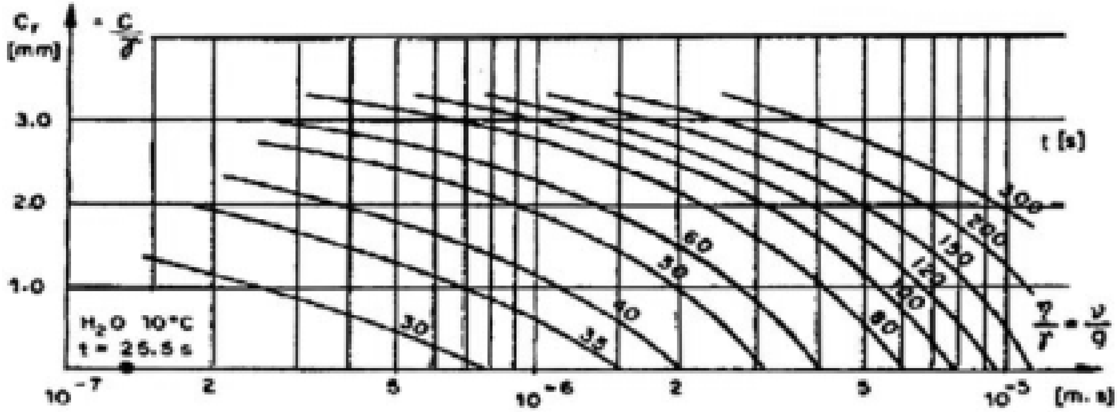



Figure 5-2 Chart to determine apparent viscosity from Marsh viscosity and cohesion (After Lombardi 1985)

Figure 5-3 Marsh funnel (left) and Lombardi plate cohesion meter (right)

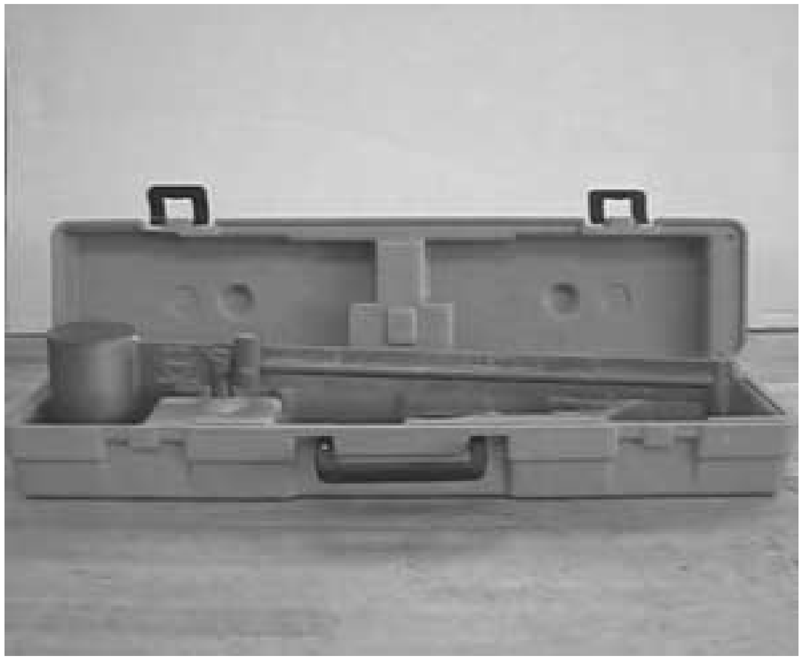
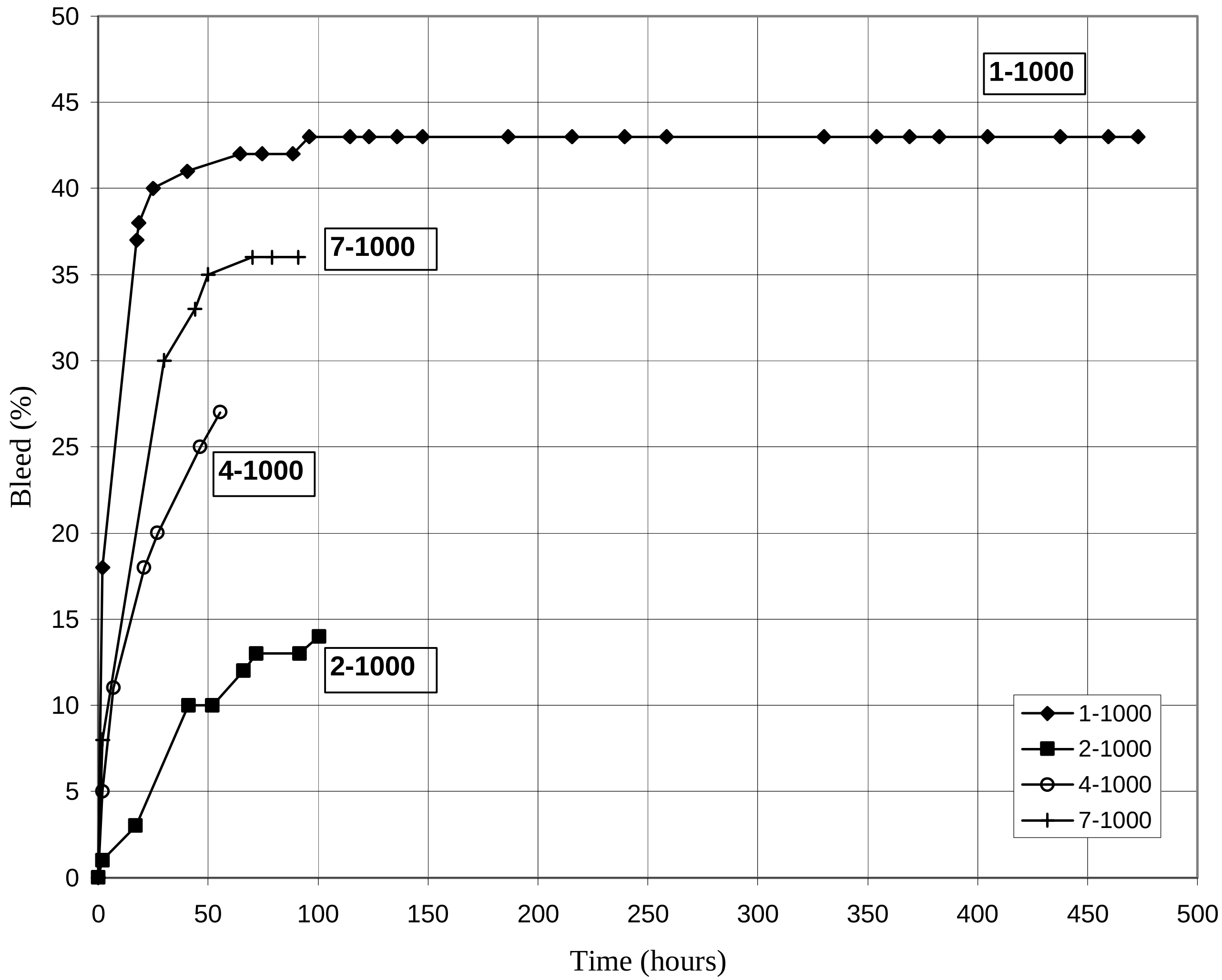


Figure 5-4 API mud balance




Mix 1-1000:
Base Mix
w/c = 2
Rheocem-900
1.5% RB-1000
10% Delvo

Mix 2-1000:
Base mix plus
3% bentonite

Mix 4-1000:
Base mix plus
5% silica fume

Mix 7-1000:
Base mix plus
0.1% welan gum

Figure 5-5 Bleed versus time, mixes with single stabilizer

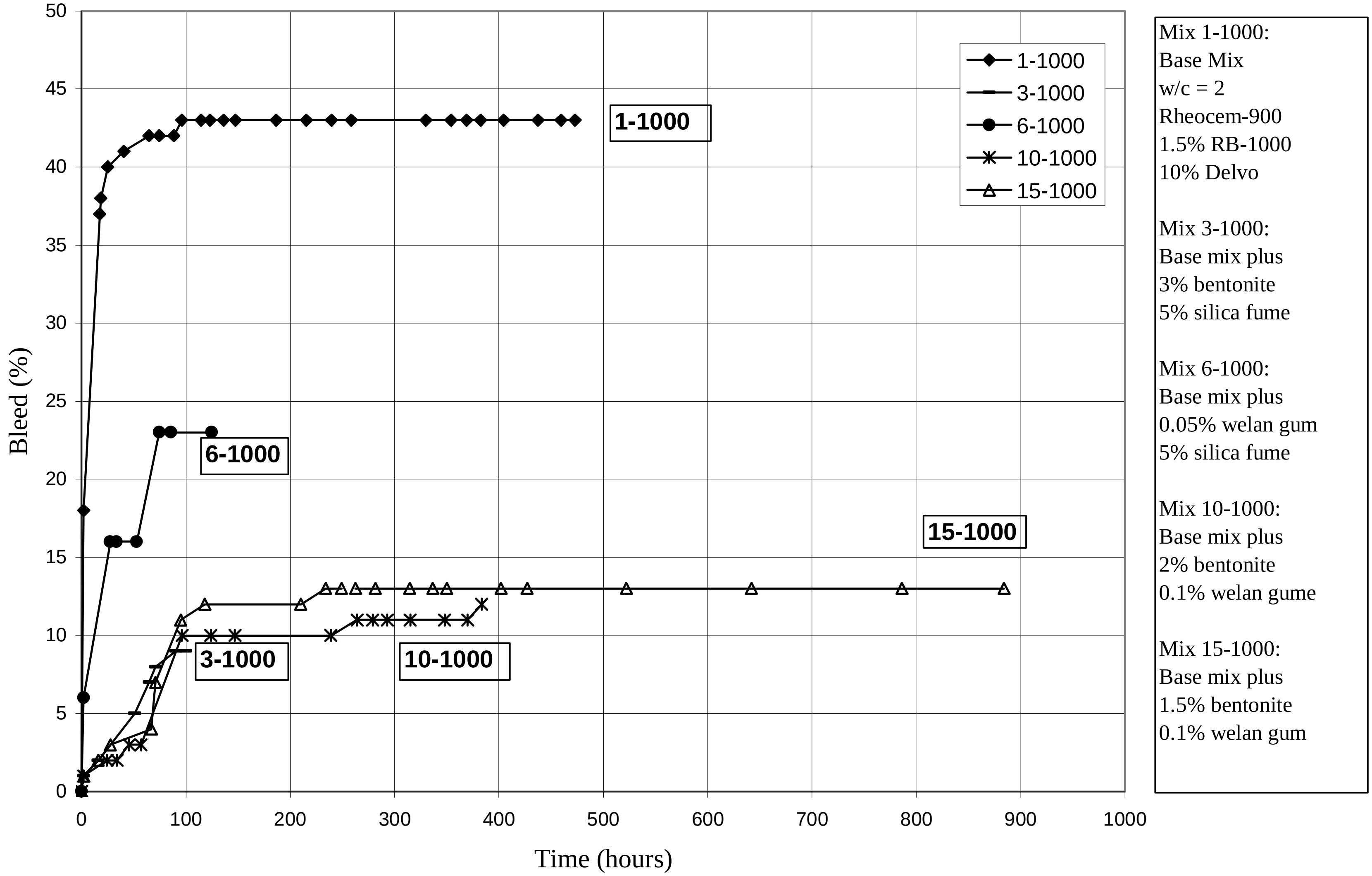



Figure 5-6 Bleed versus time, mixes with two stabilizers

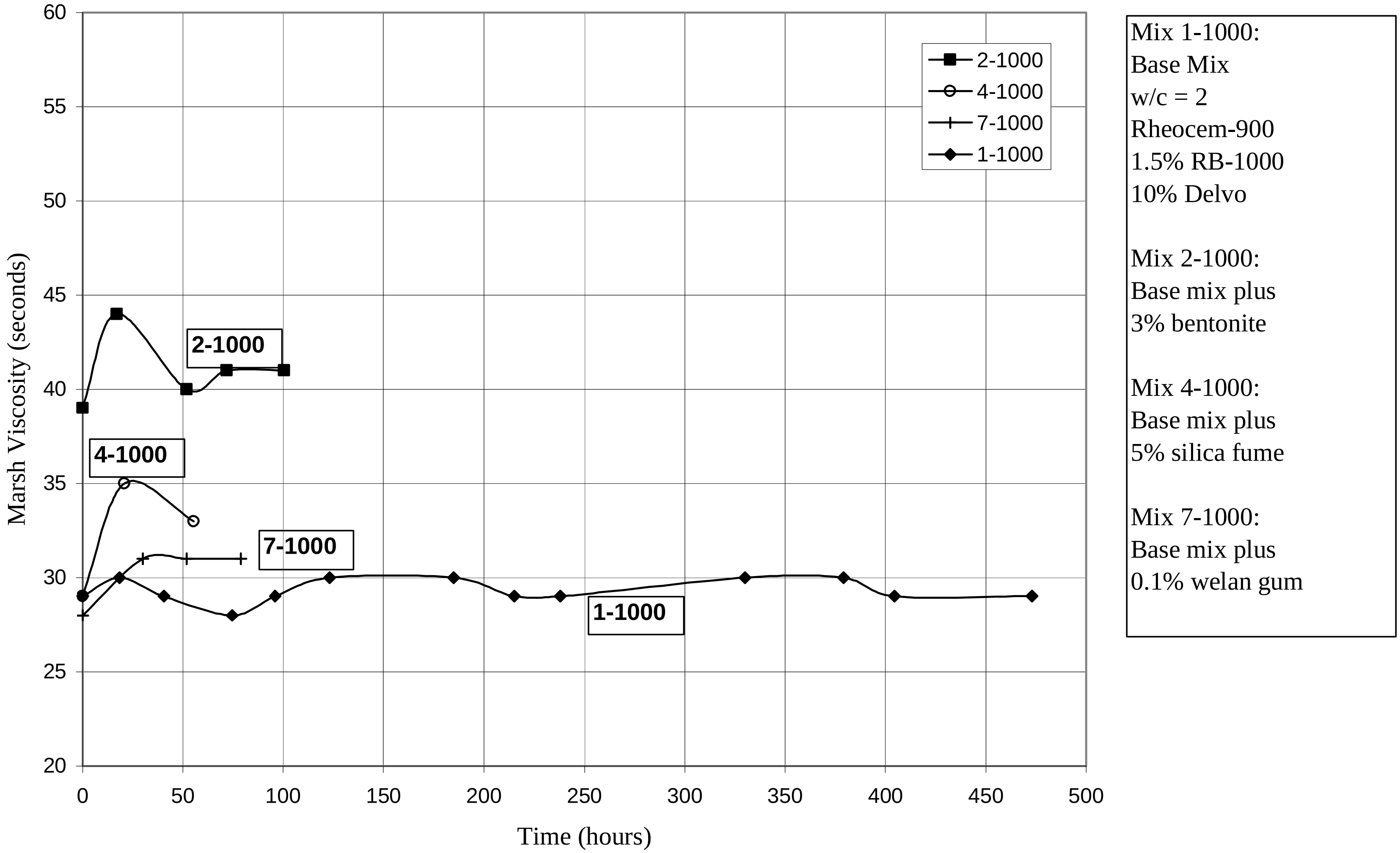



Figure 5-7 Marsh viscosity versus time, mixes with single stabilizers

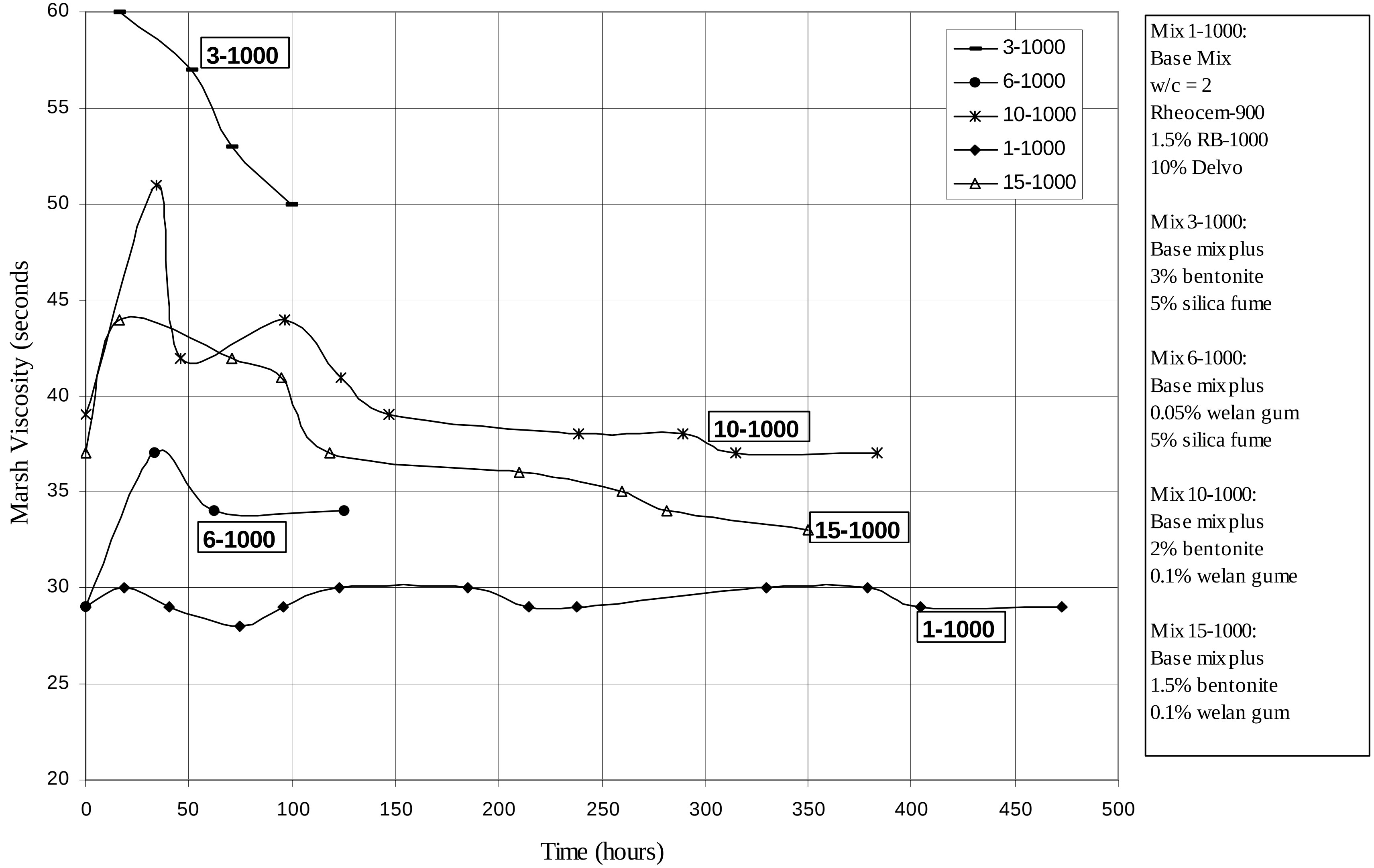



Figure 5-8 Marsh viscosity versus time, mixes with two stabilizers

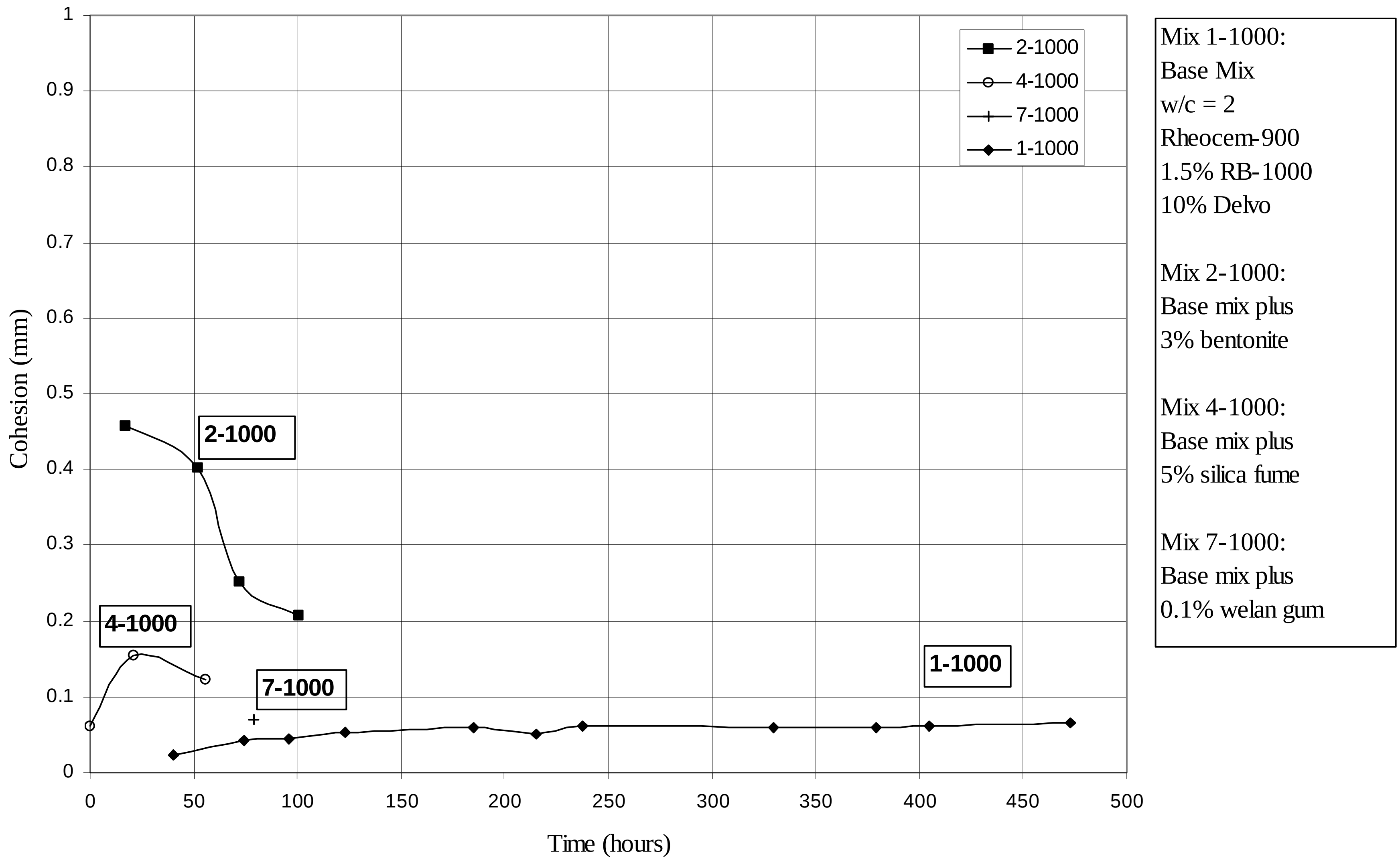



Figure 5-9 Cohesion versus time, mixes with single stabilizer

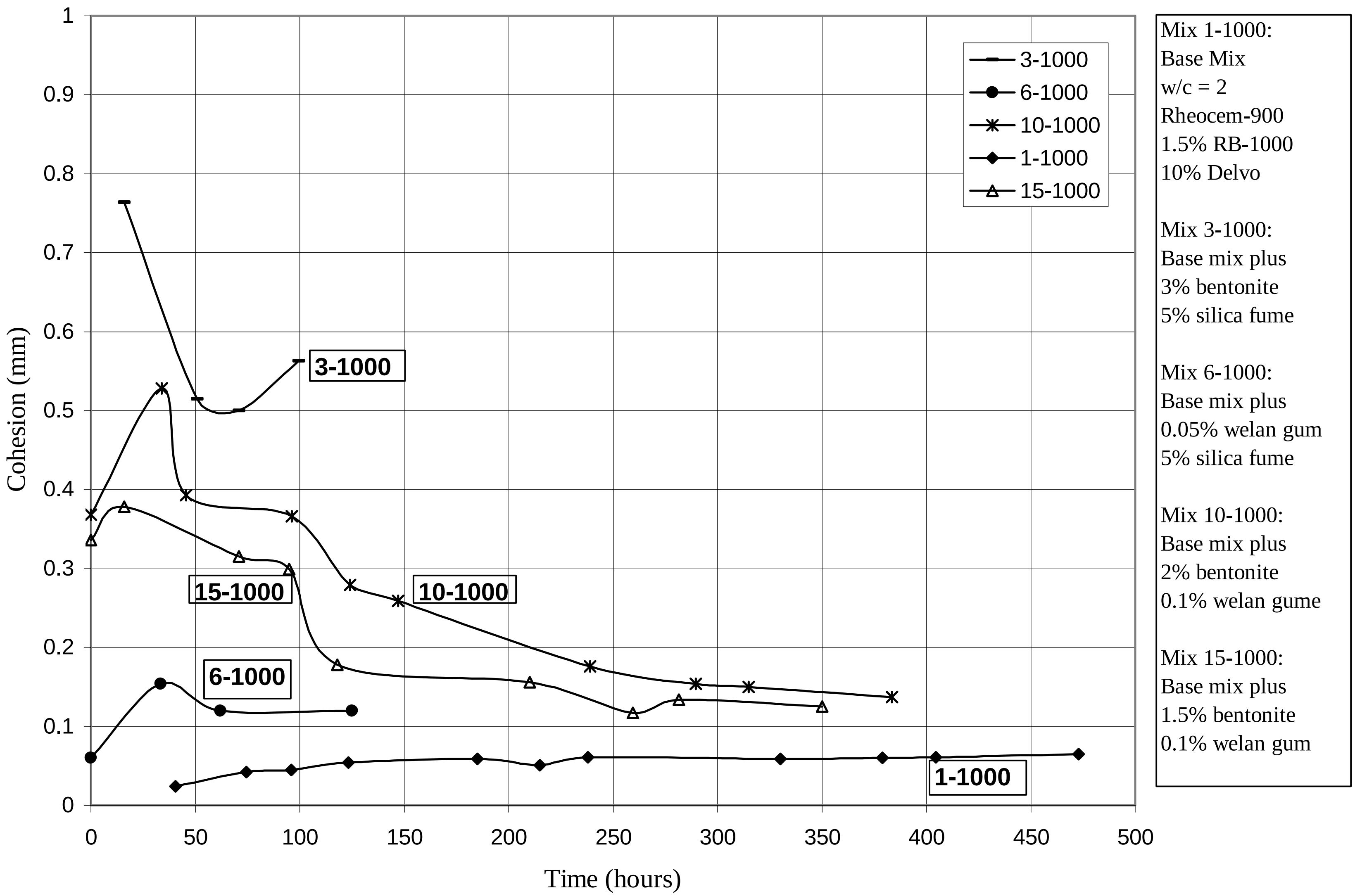



Figure 5-10 Cohesion vs. time, mixes with two stabilizers

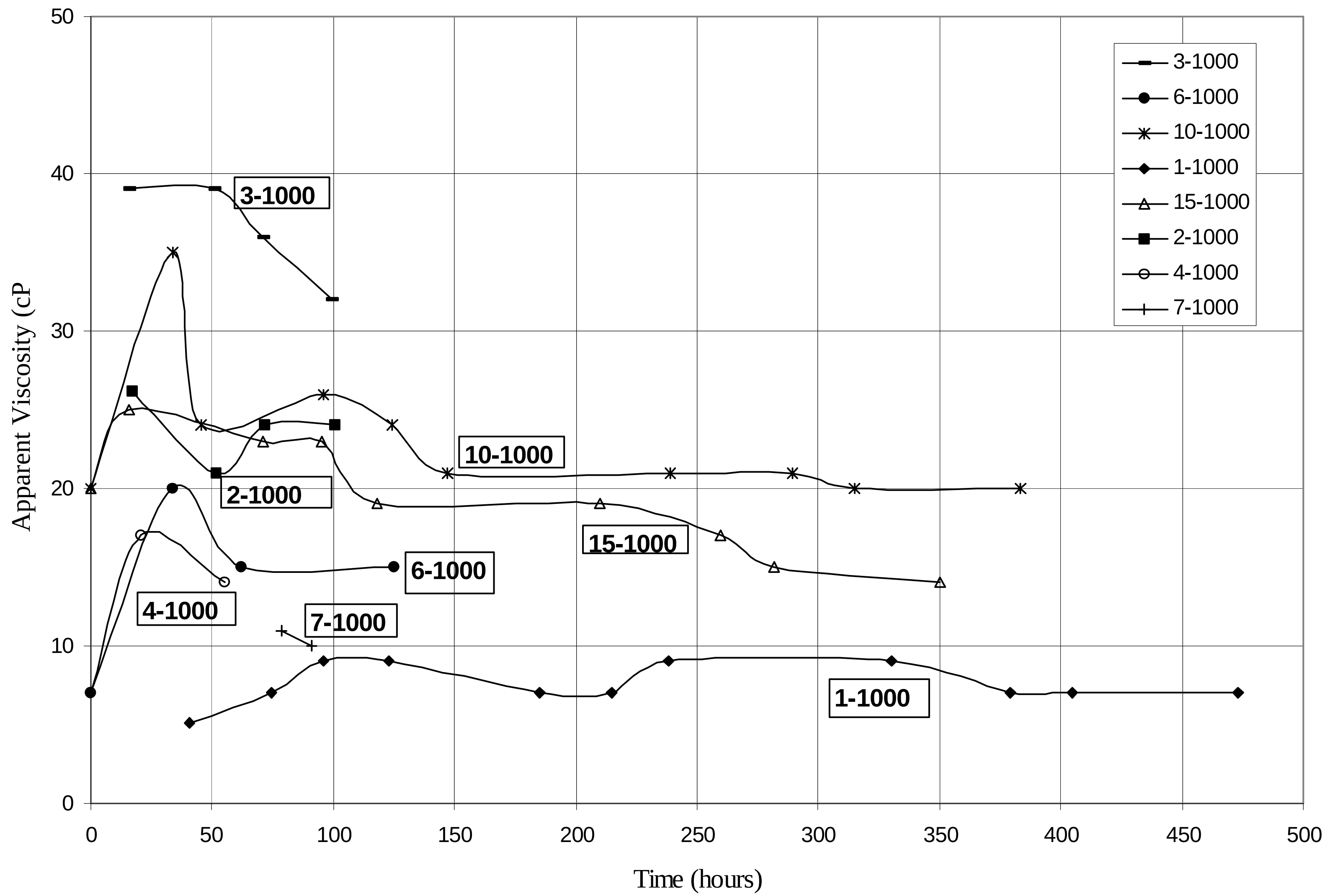



Figure 5-11 Apparent viscosity versus time, all mixes