API HIGH WATER CONTENT PHASE II
ANALYTICAL TEST METHODS

Final Report

5/05/05

1.0 Purpose, Objectives and Summary of the Laboratory Tests

Executive Summary

The primary objective of these tests was to validate and document test analytical methods in order to support the drafting of an API Recommended Practice for the Analysis of High Water Content Samples Between 2-50%.

The purpose of the testing was to qualify various methods' applicability with respect to the range of water content.

The test results (all test results in spreadsheet appendix) included herein are as follows:

- **Graduated Cylinder Method** using standard 500 ml and 1000 ml cylinders depending on sample size.
- **Centrifuge Method** (ASTM D 4007) using standard 100 ml crude samples and calibrated, 8" conical shaped tubes (with 1% minimum increment);
- **Distillation Method** (ASTM D 4006) using reduced sample size (gravimetrically measured, 10 ml approximate).
- **Pre-Separation Graduated Cylinder Method** which consists of draining free water from the 1000 ml graduated cylinder with pet cock and determining water content of the remaining oil phase. The water content of the residue or oil phase is determined by Distillation, Centrifuge or Karl Fischer Titration (ASTM D 4377) methods and added to the free water.
- A series of **Baseline Tests** on the two crude oils to determine API Gravity, Viscosity, Volumetric Shrink Factor and Water Content by Distillation, Centrifuge and Karl Fischer Titration.

The following tests were attempted and abandoned due to analytical problems:

- Heavy oil tests with the Graduated Cylinder Method was suspended for highest water content samples due to time requirements for oil/water interface (clear break);

- Centrifuge tests with reduced sample size in order to increase resolution through readability of scale (interface readings in conical section) was abandoned due inability to obtain more accurate results than with the standard centrifuge volume;
- Distillation Method (ASTM D 95) with standard, 200 ml crude oil sample and 20 ml trap with pet cock drain. The increased amount of water in the sample causes violent boiling problems with subsequent condenser blow out;
- Pre-Separation Graduated Cylinder Method of determining water content of the remaining oil phase by distillation (ASTM D 4006) was abandoned for low water content/large oil phase samples due to inherent problems with obtaining a representative aliquot sample from the larger oil phase sample.

In order to eliminate the possibility of skewed results, the reference blend water content was not determined until the test result was recorded.

All of the above test measurements except for the reduced volume distillation method were intended to be conducted in the field volumetrically. The reduced volume distillation method is intended to be used as referee/onshore lab method so that weigh systems may be utilized.

All tests were conducted at Southern Petroleum Lab's ASTM facility located near the South Loop in Houston. For consistency, all measurements were made and readings by SPL ASTM Lab Manager, Tom Benz. A special acknowledgement goes to Tom for his diligence and care in preparing the samples, sound advice and objectivity.

2.0 Reference Water Content Determination

All of the reference blends were determined gravimetrically using calibrated weigh scales and laboratory densitometer.

The laboratory density meter is a Parr Model DMA 48 with the following specifications:

- Range: 0-3 gm/cm^3
- Accuracy: +/-1 x 10^{-4} g/cm^3
- Temperature Range: -10 to +70^{o}C with accuracy of +/- 0.1^o C

The weigh scales used for determining the mass of each component and glassware used are as follows:

Weigh Scale Devices

Manufacturer	Model	Calibrated Range	Reading/Resolution
Ohaus	AS200	0.5-150 g	0.1 g
Mettler	PJ6000	10-5000 g	1 g
Mettler	PE24	100-10000 g	10 g

The above weigh scales were calibrated last on Feb 9th 2005 with zero reading error over the above calibrated range after service.

In order to minimize uncertainty, the most appropriate/accurate weigh scale was used for each blend component

Reference Blend Uncertainty

Based on the above, the estimated uncertainty of the reference blends was typically between +/- 0.1 – 0.2% water content.

However, as in all testing programs some test points were compromised due to inherent testing circumstances. The inherent problem referred to herein was evident during the distillation method test whereby a sample size of 10 ml was required. As can be readily realized from the table above 'Weigh Scale Device', the resolution with a minimum of +/- 0.1 gram, it is physically impossible to make an accurate (less than 0.1% uncertainty) reference blend of only 10 ml and therefore, cannot be obtained. Therefore, the larger reference blends were made (200-500 ml), homogenized and then sub-sampled to about 10 ml. For the higher water content blends, it is believed that some water falls out before a representative sub-sample is extracted. The blending and sub-sampling accuracy problem is probably best served with the use of a dynamic mixing system whereby the sub-sample is taken from a lab type mixing system (variable pump with suction on the bottom of the container, static mixer and take-off tubing

for direct sub-sampling of aliquot prior to return to container) over the available lab high shear insertion mixer (see appendix for additional information).

2.1 Reference Blending Procedure.

Briefly, the procedure for preparing each test point blend was as follows:

1. Clean and weigh the glassware to be used.
2. Pre-wet the oil transfer glassware (typically one quart jars) to minimize any non-representative clinage.
3. Heat the transfer glassware to 60^{o} C.
4. Heat oil and water samples to 60^{o} C.
5. Combine and homogenize the blend.

2.2 Reference Blend Calculations.

The reference blend calculations are based on gravimetric measurements of oil and water and converted to volume by dividing by the respective density at 60^{o} C.

Reference Water Content (vol/vol%) = $[(\text{Volume}_{W@60C})/(\text{Total Volume}_{W+O\ @\ 60C})] \times 100$

Where:

Volume Water @60^{o} C = $(\text{Mass}_{W}/\rho_{W@\ 60C})$

Volume Oil @60^{o} C = $(\text{Mass}_{o}/\rho_{o@\ 60C})$

Total Volume= Volume Oil @60^{o} C + Volume Water @60^{o} C

$$= [(\text{Mass}_{W}/\rho_{W}) + (\text{Mass}_{O}/\rho_{O})]$$

The final reference water content is adjusted by adding the baseline water content for the respective test method, if appropriate.

3.0 Estimated Uncertainty of the Reference Water Content

The target objective for uncertainty for each reference blend was to be a factor of ten better than the expected field results of +/- 1% or in other words, < +/- 0.1%. The overall test objective was to minimize all known biased errors such as volumetric shrinkage due to loss of entrained gases and glass clinage whereby higher concentrations of water may tend to remain in any transfer glassware. In order to minimize resolution of the weigh scale or gravimetric devices, the equipment was calibrated and the scale with the best resolution was used, depending on the weight requirement.

Although it may be possible to develop a rigorous statistical evaluation for a precision or uncertainty statement based on the uncertainty or resolution of the densitometer and weigh scales, it may not be entirely realistic or appropriate as there are other factors that should be included which are not readily available for evaluating these factors. The primary reason for this is there are inherent, non-measurable, non-representative losses in the blending process such as vapor losses (shrinkage) and glassware clinage. All possible efforts were made to minimize these errors due in shrinkage and clinage.

The only ascertainable uncertainty is in the gravimetric equipment (digital weigh scales and digital density determination) and baseline water content. The weigh scales were calibrated on Feb 9th with essentially no error as left over the entire range (see the table above). The baseline water content for both crudes and all test methods was relatively low with the maximum water content value of 0.06% (range of 0.05-0.06% for all three test methods) for the heavy oil when determined by the Karl Fisher Method. The probable error due to baseline uncertainty is minimal and estimated to be less than +/- 0.02%.

Based on the above laboratory precautions, weigh scale devices, observations and test results, the estimated uncertainty of the calculated water content of the reference blends is less than +/- 0.1% (including sample transfers and handling) for all test methods and water content test points.

4.0 General Sample Handling Issues:

4.1 Volumetric Shrinkage of Test Samples.

As samples are heated to 60 C for most tests and the crude oil volumetric shrinkage (due to loss of light ends) is critical in high water content samples, tests were conducted on both oil samples. Both oils demonstrated some volumetric shrinkage due to entrained gas when samples were taken at 68 F, heated to 140 F for one hour and then reduced back to 68 F. As a result, for these tests, all initial sample measurements were taken at 140 F (60C). Due to possible shrinkage in water from dissolved CO2, water will also be measured at 140 F (60C).

4.2 Sample Transfer.

All transfer glassware was wetted prior to use to minimize losses. There was concern that results may be biased slightly low due to inherent losses in glassware transfers as higher concentrations of water may remain in transfer glassware. However, the data did not reflect this assumption. This problem was minimized by washing the mixing/transfer glassware with the measured solvent prior to adding to the sample.

4.3 Sample Mixing.

The typical lab mixer, a high speed (13,000-22,000 rpm) shearing device which is inserted into a cylinder or jar is not adequate for samples with high water content or sample sizes greater than about one liter (see Appendix section on Lab Mixer).

5.0 Graduated Cylinder Method

5.1Test Notes

All actual reference blends were gravimetrically measured.

The glassware was verified/calibrated prior to testing and corrections applied to final reading results. No baseline data was applied as no graduated cylinder baseline tests were conducted.

The lowest test point (around 2% water content) was not included as the interface would be below first graduation in a 500 ml cylinder. Therefore, lowest test point is around 5%. Each test blend was pre-heated (oil and water to 60C) prior to measurement/blending for test precision.

The test readings were taken approximately five (5) minutes after sample achieving desired temperature of 60C.

5.2 Test Findings.

All readings were estimated between graduations to closest whole ml (500 ml has 5 ml graduations and 1000 ml cylinder has 10 ml graduations).

Glassware calibration is required for best precision.

A good break was observed at oil/water interface for all measurements for light oil and heavy oil after 24 hours.

Total volume readings (top of oil phase) were adjusted for the meniscus.

The graduated cylinder method is best suited for light oil with relatively high water content (>30-35 API)/condensate/>5% water content/<10 cP@100F).

Vibration did not significantly enhance separation.

Validation methods will be required for graduated cylinder tests in order to ensure all water has dropped out of oil phase prior to reading interface.

5.3 Sample Mixing.

Both sample inversion mixing method and high speed shear mixer used to determine possible emulsion problems. Shear mixer at 22,000 RPM creates tight emulsion and requires additional time to break for both oils.

5.4 Graduated Cylinder Test Results

Graduated Cylinder Method Results for Light Oil

Reference (%)	Reading (%)	Difference/Error (%)
5.74	5.47	-0.27
11.63	11.40	-0.23
19.29	18.80	-0.49
33.24	32.44	-0.8
47.4	47.39	-0.01

Average Error: - 0.36% (without shear mixing)
Error Range: -0.8 to -0.01%

Graduated Cylinder Method Results for Heavy Oil

Reference (%)	Reading (%)	Difference/Error (%)
5.68	5.15	-0.53
11.66	11.51	-0.15

Average Error: - 0.34% (after 24 hours)

5.5 Graduated Cylinder Method Summary:

1. Obtained good results on light oil tests over entire range with an average error of - 0.36% and error range of -0.8 to -0.01%;
2. Obtained good results but limited data on heavy oil due to time constraints as 24+ hours were required for even the lowest water content tested.
3. For both water content tests, the heavy oil will not separate in the predetermined time of five minutes. The heavy oil has to remain at least 24 hours with heat in order to break the emulsion regardless of mixing method. Therefore, heavy oil tests were suspended due to test time requirements with probable exclusion of heavy oil for graduated cylinder method.
4. Due to increased resolution, the 500 ml graduated cylinder should yield more accurate results than the 1000 ml cylinder.
5. All test results were within +/- 1% of the reference blend.

6.0 Centrifuge Method

6.1 Test Notes.

All reference blends were gravimetrically measured for best precision.

All results within +/- 1% of reference blend except for one at 1.03%.

Approximately 2 ml of demulsifier added to each tube before centrifuging in order to reduce viscosity and enhance separation.

All blends were prepared in 100 ml batches with entire quantity equally split into the two tubes so that results/readings are the combination of the two tubes. Therefore, no repeatability tests are available herein.

Reference blend for the heavy oil sample includes a baseline value of 0.05% S&W.

Reference blend for light oil has 0.0% S&W baseline correction

Tube # 1 Actual	Tube # 1 Reading	Tube # 2 Actual	Tube # 2 Reading
1 ml	1.2 ml	1 ml	1.2 ml
2 ml	2.3 ml	2 ml	2.2 ml
3 ml	3.2 ml	3 ml	3.2 ml
4 ml	4.1 ml	4 ml	4.1 ml
5 ml	5.0 ml	5. ml	5.0 ml
6 ml	6.0	6 ml	6.0 ml
10 ml	10.0	10 ml	10.0
15 ml	15.0	15 ml	15.0
20 ml	20.0	20 ml	20.0
25 ml	25.0	25 ml	25.0
30 ml	30.5	30 ml	30.5
35 ml	35.0	35 ml	35.5
40 ml	41.0	40 ml	40.5
45 ml	46.0	45 ml	46.0
50 ml	51.0	50 ml	51.0

These verifications were conducted by adding water measured with a buret and backed up with gravimetric methods and then the centrifuge tube was corrected for the ‘test result’.

6.2 Reduced Volume Centrifuge Method Tests

Two tests were conducted to determine if a reduced sample volume would enhance results. The test points selected were 20 and 40% water content as a sample reduction of the right proportion would yield a reading in the conical section of the tube (possible increased resolution due to readability of scale). The 20% blend was reduced by one-half and the 40% blend was reduced one-fourth in order to possibly improve resolution so that the interface readings are in conical section of the tube.

The sample size reduction tests were conducted on light oil by reducing the 20 % concentration sample size by 50% or 50 to 25 ml, then doubling the readings/results.

Reference 18.49% Readings 2 x 9.5 or 19.00% Error = -0.51%

The 40 % concentration light oil sample size was reduced by 25% or 50 ml to 12.5 ml then, the readings were multiplied by 4 for final readings/results.

Reference: 38.87 Reading 4 x 10.75 or 43%. Error = +4.13%

These tests were abandoned as the precision is less than the standard volume. This method is not recommended for the RP.

6.3 Sample Mixing

All blends used inversion mixing methods only. After testing, a high water content, heavy oil sample was remixed using high speed shear mixer to determine if a tight emulsion causes inherent centrifuge method problems. Three centrifuging sessions was required to break the emulsion.

The test was conducted by re-mixing the highest concentration (49.5%), heavy oil sample with the high shear mixer (19,000 rpm for one minute) to see if tight emulsion issues are critical problems for high water/heavy oils.

The initial centrifuge after shear mixing and adding 2 ml demulsifier yielded an oil/water interface at 3%.

The sample was allowed to sit overnight, then 2 ml demulsifier was added and centrifuged. Readings: 73%oil/12% emulsion/15% water.

Again, added 2 ml demulsifier and centrifuged a third time.

Reading: 49.50% or same as original.

6.4 Centrifuge Test Results

Centrifuge Method Results for Light Oil

Reference (%)	Reading (%)	Difference/Error (%)
2.27	2.10	-0.17
5.58	5.6	+0.02
11.76	12.25	+0.49
19.47	20.50	+1.03
36.03	35.50	-0.53
41.03	41.5	+0.47
49.51	50.0	+0.49

Average Error: + 0.25%
Error Range: -0.53 to +1.03%

Centrifuge Method Results for Heavy Oil

Reference (%)	Reading (%)	Difference/Error (%)
2.35	2.30	-0.05
5.87	5.60	-0.27
12.24	12.50	+0.26
19.42	19.25	-0.17
37.83	38.50	+0.67
41.25	41.50	+0.25
48.98	49.50	+0.52

Average Error: + 0.17%
Error Range -0.27 to +0.67%

6.4 Centrifuge Method Summary

1. Excellent results were obtained on both light and heavy oil samples over the entire test range by the centrifuge method using the standard tube fill volume of 50 ml. Average errors of +0.25 and +0.17% on light and heavy crude oils respectively. Test results range from - 0.53 to + 1.03% and - 0.27 to + 0.67% on light and heavy crude oils respectively.
2. Regardless of sample emulsion due to shear mixing, accurate results may be obtained by additional demulsifier/heat/centrifuge.
3. RP should state that if an emulsion exists, add demulsifier and centrifuge again until no emulsion layer is detected.
4. The RP should note that a vertical test tube stand is required for accurate readings.
5. Sample reduction did not improve results.

6. Sample volume measurement will be critical so that foaming and volumetric shrinkage due to light end loss should be avoided, if possible.

7.0 Distillation (ASTM D 4006) Method

7.1 Test Notes.

All reference blends were gravimetrically determined. Each sample was blended into a glass jar (4 oz or 120 ml), homogenized completely at 16,000 rpm for one minute so that no water will fall out the during the aliquot transfer process.

All sample sizes reduced to about 10 ml using light (plastic) cup for gravimetric determination. The sample cup is weighed with sample and after sample is removed so that the sample mass into the flask is precisely determined by difference. All samples had pre-heated oil and water to 60C prior to measurement/blending for test precision

The pre-calibrated five (5) ml trap was used in all tests. Trap readings were approximately at room temperature (68-75 F).

7.2 ASTM D 95 Method

The ASTM D95 distillation method uses 20 ml trap with pet cock to drain excess water and standard 200 ml sample. A farily low water content sample, 11% boiled violently and erupted the condenser packing. The D 95 tests were abandoned.

7.3 Distillation (ASTM 4006) Test Results

Distillation Method Results for Light Oil

Reference (%)	Reading (%)	Difference/Error (%)
11.87	11.925	+0.055
18.937	19.1	+0.163
41.795	40.925	-0.87

Average Error: - 0.22%
Error Range: -0.87 to +0.163%

Distillation Method Results for Heavy Oil

Reference (%)	Reading (%)	Difference/Error (%)
12.166	12.075	+0.091
19.238	19.3	+0.062
41.665	39.90	-1.765

Average Error: - 0.53%
Error Range -1.765 to +0.091%

7.4 Distillation Method Summary

1. This method is considered to be the most precise for any crude gravity or viscosity although the data above 25% water content does not reflect this statement. The problem with the higher water content test points were not an inherent problem with the distillation method but in obtaining a representative aliquot or sub-sample as both results were low indicating that some water started to drop out before the aliquot was removed from the larger blended sample. However, if samples are obtained directly from a circulating system such as a Lab Mixer, improved results for the higher water contents are expected.
2. Excellent results on both light and heavy oil distillation (ASTM D 4006) below 25% water content. Average errors of -0.21 and -0.53% on light and heavy crude oils, respectively. Test results range from - 0.87 to + 0.16% and – 1.73 to + 0.09% on light and heavy crude oils, respectively.
3. The ASTM D 4006 method should be the referee method for onshore lab testing as it is not affected by viscosity, gravity and emulsion.

8.0 Pre-Separation-Graduated Cylinder Method

8.1 Test Notes:

Glassware was calibrated and corrections applied.

Only light oil tests were conducted due to time constraints and heavy oil viscosity problems are the same as above in the standard graduated cylinder tests as the increased viscosity prevents the water from falling from the oil phase.

Test apparatus was a standard, 1000 ml graduated cylinder with drain pet cock near bottom with verified volume for corrected readings.

8.2 Pre-Separation-Graduated Cylinder Method Procedure:

Mix sample using gravimetric techniques for reference blend.

Homogenize using inversion techniques.

Pour sample into 1000 ml cylinder with drain pet cock in water bath at 60C.

Read total volume with meniscus and glassware calibration corrections.

No demulsifier added.

After 4 minutes at 60C, draw off free water into calibrated 500 cylinder and determine free water volumetrically.

Remove and homogenize oil phase with shear mixer at 24,000 rpm for one minute

Analyze oil phase/residue using one of the following three analytical methods:

Centrifuge
Distillation (100 ml sample using volumetric measuring techniques)
K-F

Then, compute total water content by adding free water + [(water content fraction from cent/distillation/K-F) x oil phase volume]. The water content is then divided by the sample total volume to yield sample water content % which may be compared to the reference.

Example:

Reference Blend
Oil volume = 699.88 ml
H2O volume = 175.6 ml
Total Sample 875.48 ml

Therefore the reference blend is (175.6/875.48) or 20.06% + baseline from appropriate test method.

Apply the baseline of 0.05% for all three methods to the remaining oil residue portion of 80% of 0.05 x 80% for resulting baseline adder of 0.04%.

Corrected Reference = 20.10%

Total volumetric reading in the graduated cylinder was 873 ml for the observed total. Subtract 1.5 for calibration correction for corrected 871.5 ml total volume.

Free water in 250 ml graduated cylinder = 54 observed corrected for -2 or resulting corrected free water removed of 52 ml.

Homogenized oil phase and tested 2 ways to get remaining water to add to the 52 ml free water.

$Reading_{centrifuge}$: 14.5% x (871.5 - 52 = 819.5) = 118.8 ml water

Total Water Content by centrifuge = [(52 + 119)/871.5] x 100 = 19.62%

$Reading_{K\text{-}F}$: 16.99% and 16.02% or average K-F value of 16.5% x (871.5-52 = 819.5) = 135.2 ml

Total Water Content by K-F = (52 + 135.2)/871.5 = 21.48%

8.3 Pre-Separated Graduated Test Results

Results for Light Oil

Reference (%)	Reading (%)	Difference/Error (%)
50.295	50.28 by centrifuge	-0.015
50.295	50.34 by distillation	+0.056
50.295	50.58 by K-F	+0.285
20.10	19.62 by centrifuge	-0.48
20.10	21.48 by K-F	+1.38

8.4 Pre-Separation Graduated Cylinder Method Summary

1. The distillation method of water content determination from the oil phase is hampered by problems with obtaining a representative aliquot from increased sample size at low water content as described above.
2. The K-F method is less precise for water content determination of the oil phase sample if it has relatively high water content due to decreased resolution from the multiplying of readings.
3. The most accurate determination of the water content in the oil phase is by the centrifuge method.
4. Pre-Separation-Graduated Cylinder Tests had some good results but limited to light oil (same reason as stated above for heavy oil – no break for 24+ hours).
5. The best results are achieved when using centrifuge for oil phase testing as there are inherent sampling problems with distillation and resolution with the K-F Method if the residue sample has higher water content.
6. May be suitable for offshore applications using only volumetric measurement techniques.
7. Not acceptable for viscous crudes.

9.0 RP Learning Summary:

- All glassware must be gravimetrically calibrated prior to use. Corrections must be applied when using these glassware components
- Graduated Cylinder and Pre-Separation Graduated Cylinder Methods are best suited for light oils, with API gravity > 35^{o}.
- Both Graduated Cylinder Methods are dependent on viscosity and time as heat and vibration do not significantly enhance (speed up) results.
- Allowing sufficient time, (24 hours or more), the Graduated Cylinder Method will work for heavy oils. However, the residue oil phase should be analyzed for water.
- The Pre-Separation Graduated Cylinder Method may be preferred over the Graduated Cylinder Method as it ensures all of the water is analyzed.
- For the centrifuge method, regardless of sample emulsion due to shear mixing, accurate results may be obtained by adding demulsifier, heating and re-centrifuging. RP should state that if a lighter colored emulsion layer is evident, keep adding demulsifier and spinning until no emulsion is present. Do not estimate water fraction in emulsion.
- RP should note that vertical test tube stand is critical for accurate centrifuge readings.
- Volume of sample in centrifuge tube will be a critical uncertainty issue as the water fraction increases. Foaming and light end loss volumetric shrinkage will contribute to this problem. RP should stress that at high water content, the centrifuge sample size is critical and must be accurate as the difference between 48 ml and 50 ml of sample at 50% water content is a 1% error.
- The distillation method (ASTM D 4006) with reduced sample size to 10 ml (gravimetrically measured) should be the onshore referee method.
- The ASTM D 95 method should not be included in the RP.

Appendix A

Sample Circulating System or Lab Mixer

As described above, there are sample handling problems with high water content samples that are not necessarily issues with lower water content samples. This problem is discussed above when reducing sample sizes to 10 ml for distillation or even smaller for K-F. This issue is referred to in ASTM D 4006 section 7.1.2.1.

The problem is a function of:

1. Sample size relative to aliquot size.
2. Water content fraction.
3. Oil gravity and viscosity.

If the aliquot is taken directly into the analytical receptacle glassware (such as centrifuge tubes or graduated cylinder whereby the entire amount is analyzed) from a properly designed and operated container circulation system, this requirement does not exist. However, if the sample size is larger than the required sample to be analyzed such as an analytical discrepancy offshore with a high water cut sample, a 1-2 gallon sub-sample is shipped to a 3rd party lab for 'referee' analysis. In this case, when the sample arrives at the lab, it has probably segregated into two phases (oil and water). Then, the laboratory is faced with re-mixing the entire sample or measuring free water and then homogenization of the oil phase. This mixing is typically conducted by the lab using a high speed (variable from 13,000-20,000+ RPM) shear mixer which is inserted into the sample container. The high speed shear lab mixer may not be capable of sufficiently homogenizing the entire high water content sample greater than about one liter for representative aliquot extraction such as 2 x 50 ml for centrifuge or 10 ml for distillation.

Recommended Solution: A sample container and mixing system consisting of a 1-3 gallon sample container, variable speed pump (speed based on viscosity/volume/gravity) and ¼"-1/2" circulation tubing with static mixer and sample extraction connection (Welker MSTCC Mixing skid and TCC-3 container or similar apparatus from Jiskoot. The entire sample is emptied into the container, mixed the proper amount of time and aliquots are then removed from the circulating system.