Evaluation Report of

InnerGeo – WaterReco's Hydrocarbon Extraction Vessel

Report Prepared for:



Analysis Performed by:



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Introduction

WaterReco's hydrocarbon extraction vessel was delivered to a well location in Frio County Texas producing from the Eagle Ford and adjacent formations on March 14, 2016. The vessel was connected to the discharge from the "heater-treater" and flow tested. Adjustments were made to the configuration over the first few days and on March 17, 2016 the samples of produced water were collected both entering and exiting the vessel. The vessel remained on location and was sampled again on March 28, 2016.

Hanby Environmental and Hanby PetroAnalysis were selected to provide testing services because we were able to measure total petroleum hydrocarbons (TPH) on-site using methods accepted by the EPA and several state agencies. The measurement of TPH concentration in water using this method captures all hydrocarbons that will partition to a solvent (carbon tetrachloride in this case) regardless to the hydrocarbons state of emulsification or availability as a free (non-aqueous phase) liquid. These are the set ups for both days of data collection.



Objectives

The primary objective of these tests is to collect sufficient TPH concentration values (data) in the produced water entering and exiting the vessel to evaluate the efficiency of the vessel to remove a significant portion of the hydrocarbons entrained in the produced water. Since inflow concentrations vary over time depending on well performance and the efficiency of other apparatus upstream of the test vessel (3-phase separator, heater-treater) there should be at least 30 samples collected (both in and out) to provide enough data to give statistically sound performance results.

Procedures

On-site testing was performed by Hanby Environmental and Hanby PetroAnalysis using our TPH in water field kit method and our Hanby Chemical Reaction Spectrophotometry ("CRS") method performed by our new Hydrocarbon ID device which is currently under development. A brief Hanby history is included at the conclusion of this report. These pictures are the two Hanby methods described.



Calibration standards of 10,000 ppm, 5,000 ppm, 2,500 ppm, 1,000 ppm, 500 ppm, 250 ppm, and 125 ppm were prepared on site using the extract solvent and oil collected from the test well. These standards were then processed using both Hanby methods to provide color comparison for estimating TPH concentrations on-site and to convert the on-site spectrophotometer measurement to TPH concentrations later, after completion of the field campaign.

The pictures below are from the calibration standards prepared on site. Following these are the spectral curves of these calibrations created by the spectrophotometer. The first set is the entire calibration range from 125 ppm to 10,000 ppm. The second set is just the lower end calibration from 125 ppm to 1,000 ppm. In regards to the spectral curve pictures, the only difference in the left and right pictures, are the pictures to the right are zoomed in to be able to better see the legend sample descriptions.





Inflow samples were acquired at Port 1 after purging an approximate one-liter volume and outflow samples were acquired at Port 6 using the same procedure (Figure 1 below). There were 33 samples collected and tested on March 17, 2016 over a period of 4.5 hours with 15 samples for Port 1 (Inflow) and 18 samples from Port 6 (Outflow). An additional 30 samples were collected and tested on March 28, 2016 over a three-hour period with 15 samples from Port 1 and 15 samples from Port 6.



Figure 1. WaterReco Vessel with arrows indicating the sampling ports 1 and 6.



The pictures below reflect both extraction methods.

The picture on the left reflects the Hanby Chemical Reaction Spectrophotometric method and the picture on the right is the Hanby TPH Water Field Test Kit method.



The following pictures are just examples of the types of results that are developed utilizing these methods. By obvious visual observation of these, you can see the high oil content or ppm oil in water concentration depicted by the dark results which are the inflow water to the vessel, followed by the significantly reduced ppm or oil in water concentration depicted by the much lighter results which are outflow water from the vessel. In the pictures below on the right, the first dark color has the script 1-10 and the next has the script 6-10. In these scripts, the first number indicates the port the sample was taken from and the second number is the test sequence number. So shown below in the example is test sequence number 10 and the dark result is from port 1 (inflow) and the light result is from port 6 (outflow). The same is the case for the following pictured results from sequence test number 13. This is followed by a group picture of multiple test sequences from sequence number 7 through 13.



These pictures are just examples of the analytical work in process.



Discussion

Spectrophotometer data were processed after field testing was complete using R-Project open-source statistical software. Spectrophotometer data consisted of absorbance values for wavelengths ranging from 347 nm to 902 nm in 0.27 nm increments or 2048 measurements per scan. Absorbance values in wavelengths below 370 nm were excluded to reduce noise present in the lower wavelengths. Each spectrophotometer scan was reduced to area under the curve (AUC) to best consolidate the results into a single measure of TPH concentration present in the water (Figures 2 below). These illustrate the difference in the results comparing the "area under the curve" of the calibrations of 250 ppm to 2,500 ppm. As depicted, as the concentration level of ppm increases, the resulting spectral curve shifts higher above the x-axis of wavelength, creating a larger area under the curve, providing the means to calculate the exact concentration level correlated back by the created calibrations that were created and analyzed of 125, 250, 500, 1,000, 2,500, 5,000 and 10,000 ppm. Additionally, note that the y-axis which represents light absorbance by the sample also increases as concentration level goes up. This can visually be seen in the results by high concentration ppm being dark in color and low concentration ppm being light in color. The dark results absorb more light when illuminated providing the higher absorption reading on the y-axis. So depicted below, in the low concentration of 250 ppm, the absorption reading across the entire spectral curve is below 0.6 and in the high concentration of 2,500 ppm, the absorption reading is closer to 0.8. Very easily you can see that the shaded area under the curve is significantly larger and higher in the case of the higher 2,500 ppm concentration.



AUC was then plotted against the known concentrations of each of the calibration standards and a best fit curve was found (Figure 3 below). A third degree polynomial curve with an R² value of 0.9945 (perfect fit is 1.0) was selected as the best fit. The formula for this curve was used to calculate TPH concentrations in the collected samples.



Results

Individual results for each sample tested can be found in Table 1 at the back of this report. Inflow concentrations of TPH ranged from 2,152 to 15,045 ppm oil in water with a median TPH concentration of 9,341 ppm (n=30). Outflow TPH concentrations ranged from 200 to 7,100 ppm oil in water with a median concentration of 1,556 ppm. Using median TPH values for Inflow and Outflow the reduction in TPH concentrations passing through the WaterReco vessel is approximately 83%.

In order to provide a visual representation of these results, based on the median figures, a review of the results reflected below in Table 1 and highlighted by bold and red scripts, it was determined that the sample results that most closely related to those of the median calculated figures, that sequence test number 20 from the first test series (3/17/16) and test sequence number 8 from the second series (3/28/16) most closely match these median figures. So reflected in the pictures below in test sequence number 20 from the first test series, the inflow from port 1 reflected a concentration of 9,995 ppm and the outflow from port 6 reflected a reduced concentration of 1,366 ppm.

Likewise, in test sequence 8 from the second test series, reflected inflow of 10,878 ppm and outflow of 2,381. In this second test series, to provide an addition reference point, also reflected in the pictures is a third result that was captured by the Hanby CRS method, whereby a 1 to 5 dilution was performed on the inflow sample extraction in order to show that even after being diluted by the solvent by a factor of 5, the resulting color developed was still darker than that of the outflow. These dilution results were omitted from the results table to keep from confusing the most prominently relevant direct results comparisons of the inflows vs. outflows.



Important to note regarding the design of our technology is that the solubility of oil in water is around 10,000 ppm, which is why that is the highest calibration that we prepared. When oil concentration in water is over 10,000 ppm, it floats in water, which is what you see in an oil spill. So our technology is designed to be focused on the lower end, whereby we can detect traces of oil in water down to single digit parts per billion. As can be observed throughout the results in this report, when the concentration of oil is around the 10,000 ppm concentration, the result is very dark to black in color. We designed our technology to have this as the upper concentration threshold, mainly because this is what is the most relevant to the past approaches that our technology has been utilized for; having been on every major oil spill response. Thus, higher concentration results readings do not make sense to perform. However, when higher concentrations are desired, it is simple math to back into the higher concentrations through performance of dilutions. As previously referenced, as a matter of interest, during our field testing and outside the scope of this project, we did perform dilutions on the second day series tests, to obtain an accurate estimate for "real" inflow concentration which is above the detection range of our conventional Hanby CRS Method of 10,000 ppm. We could easily adjust our method to performing the dilutions prior to taking readings and performing the simple math calculations into our conventional

method to increase the detection range, but for now we have not identified a larger enough market place to warrant these needs, so we elect to do required dilutions on an as needed basis.

In this case, utilizing the highest calibration we prepared of 10,000 ppm, which results in a very high absorbance reading of between 1 and 1.2 and by performing 1 to 5 dilutions on the sample extractions, we can calculate backing into the inflow concentration levels and get an accurate estimated idea for what the "actual" inflow concentrations were, but that is outside the scope of this project.

As an example point of reference, below is compared the second series sequence number 8 to the calibrations. Note the far right vial in the right picture being the 1 to 5 dilution performed. Visually, the color resulting from this 1 to 5 dilution falls in between the calibrations in the left picture of 1,000 ppm and 2,500 ppm, being only slightly lighter than the 2,500 ppm calibration color. Thus, it can be concluded that it is approximately 2,000 ppm after being diluted by factor of 5, thus multiply the estimated result of 2,000 ppm by this factor provides for the result prior to dilution of being 10,000 ppm (1%). This is also supported by the result in the first vial in the right picture being prior to dilution and it resulting visually in the same color as the first vial in the left calibration picture which is the 10,000 ppm (1%) calibration. This example supports the math that estimates the "actual" inflow concentrations above the conventional upper detection limit of 10,000 ppm (1%) which could be the scope of future project such it be warranted.



The following pictures are simply group combinations of all tests performed in the second series up to that particular analysis. So the first picture is through test sequence number 8 and the second picture is all tests performed that day through test sequence number 15. These are just additional visual points of reference of how the concentration levels reduced from inflow to outflow, but also reflect the variations based on the well flow control. However, as is reflected by the results table, it is easy to see the performance of the vessel which is the primary objective of this project.



Using Figure 4 below, being a statistical plot (box and whisker) comparing the TPH concentrations of the inflow to the vessel to that of the outflow, the effect of the vessel process can be seen graphically. The interquartile range (IQR), represented by the grey area in the boxes, can be viewed as a representation of the variability of the TPH concentrations. Note that the inflow has a much greater variability than the outflow. This may be related to the lack of control over flow rates through the vessel, since the well did not flow continuously but discharge oil, water, and gas intermittently over the test periods.



Conclusion

Over two test periods the vessel was able to reduce the TPH concentration of the produced water by 83%. The recovered oil was discharged to a separate vessel. This process provided additional oil production for the well and (although slightly) reduced the volume of fluids for disposal.

Hanby Environmental and Hanby PetroAnalysis were pleased to be a part of this validation process. In our opinion, this real time analysis was the only accurate and effective way to validate the performance of the vessel. In comparing to the alternative method of taking samples and sending them to a fixed laboratory, on top of being a sampling logistics headache, by the time the samples were received and analyzed by a fixed laboratory, the samples could be changing providing inaccurate analysis results. It is our professional opinion that real time analysis of samples provides the most accurate and reliable feedback. It was also this real time feedback that on the day prior to the first series of testing allowed for configuration changes to be made, adjusting the vessel to provide more reliable and more accurate performance results which mainly consisted of well control. This would not have been possible by sending samples to a fixed laboratory. It was this real time feedback that lead to the inclusion of a check value to help control flow and then to inspire the future modification of the further flow control measure of flow moving through a tank prior to moving toward the vessel to further reduced resulting variations. This was just a thought inspired by these real time feedback analysis results and is outside the scope of these results. It is the thought that by doing this on the next round of testing that variation should be further reduced.

Test Name	Area Under Curve	Concentration from Curve Fit #3 (ppm)	Port ID (1=In, 6=Out)	Test ID Number	Timecode
1 10 1 com	511.25	9619	1	10	2/17/2016 16:20
1-10-1.5dill	521 70	0227	1	10	3/17/2010 10:23
1-11-1.sam	521.79	9337	1	11	2/17/2010 10:43
1-12-1.sam	517.00	12264	1	12	2/17/2010 17:01
1-10-1.sam	500.39	11296	1	10	3/17/2010 18:12
1-17-1.5dill	540.05	11500	1	17	3/17/2010 10.20
1-10-1.5dm	545.00	11147	1	10	3/17/2010 10.59
1-19-1.sam	540.70	11219	1	19	3/17/2016 18:54
1-20-1.sam	530.88	9995	1	20	3/17/2016 19:10
1-21-1.sam	430.33	4265	1	21	3/1//2016 19:23
1-22-1.sam	552.98	11/24	1	22	3/1//2016 19:36
1-23-1.sam	589.91	15045	1	23	3/17/2016 19:52
1-24-1.sam	578.68	13976	1	24	3/17/2016 20:01
1-7-2.sam	543.72	10977	1	7	3/17/2016 15:26
1-8-1.sam	518.07	9076	1	8	3/17/2016 15:51
1-9-1.sam	571.40	13311	1	9	3/17/2016 16:12
6-10-1.sam	199.76	225	6	10	3/17/2016 16:33
6-11-1.sam	279.68	654	6	11	3/17/2016 16:47
6-12-1.sam	246.58	395	6	12	3/17/2016 17:03
6-13-1.sam	237.18	345	6	13	3/17/2016 17:17
6-14-1.sam	355.29	1874	6	14	3/17/2016 17:30
6-15-1.sam	418.79	3805	6	15	3/17/2016 17:45
6-16-1.sam	334.96	1445	6	16	3/17/2016 18:15
6-17-1.sam	380.64	2530	6	17	3/17/2016 18:28
6-18-1.sam	380.00	2512	6	18	3/17/2016 18:42
6-19-1.sam	401.08	3168	6	19	3/17/2016 18:56
6-20-1.sam	330.73	1366	6	20	3/17/2016 19:12
6-21-1.sam	303.38	932	6	21	3/17/2016 19:25
6-22-1.sam	299.78	884	6	22	3/17/2016 19:39
6-23-1.sam	350.36	1762	6	23	3/17/2016 19:54
6-24-1.sam	352.44	1808	6	24	3/17/2016 20:05
6-7-1.sam	361.30	2016	6	7	3/17/2016 15:30
6-8-1.sam	153.26	200	6	8	3/17/2016 15:54
6-9-1.sam	324.77	1260	6	9	3/17/2016 16:15
1-1-1.sam	529.60	9901	1	1	3/28/2016 12:40
6-1-1.sam	398.19	3071	6	1	3/28/2016 12:40
1-2-1.sam	416.98	3736	1	2	3/28/2016 13:10
6-2-1.sam	314.00	1085	6	2	3/28/2016 13:10

Table 1. TPH concentrations in the water samples collected

1-3-1.sam	521.91	9346	1	3	3/28/2016 13:25
6-3-1.sam	467.72	6017	6	3	3/28/2016 13:25
1-4-1.sam	394.72	2959	1	4	3/28/2016 13:35
6-4-1.sam	327.98	1316	6	4	3/28/2016 13:35
1-5-1.sam	366.74	2152	1	5	3/28/2016 13:45
6-5-1.sam	421.79	3921	6	5	3/28/2016 13:45
1-6-1.sam	392.00	2872	1	6	3/28/2016 14:00
6-6-1.sam	363.64	2074	6	6	3/28/2016 14:00
1-7-1.sam	525.10	9573	1	7	3/28/2016 14:15
6-7-1.sam	384.83	2653	6	7	3/28/2016 14:15
1-8-1.sam	542.46	10878	1	8	3/28/2016 14:30
6-8-1.sam	375.35	2381	6	8	3/28/2016 14:30
1-9-1.sam	500.53	7913	1	9	3/28/2016 14:40
6-9-1.sam	471.71	6229	6	9	3/28/2016 14:40
1-10-1.sam	500.80	7930	1	10	3/28/2016 14:55
6-10-1.sam	331.50	1380	6	10	3/28/2016 14:55
1-11-1.sam	445.23	4914	1	11	3/28/2016 15:05
6-11-1.sam	312.95	1069	6	11	3/28/2016 15:05
1-12-1.sam	482.74	6842	1	12	3/28/2016 15:15
6-12-1.sam	340.62	1556	6	12	3/28/2016 15:15
1-13-1.sam	382.92	2596	1	13	3/28/2016 15:30
6-13-1.sam	193.76	216	6	13	3/28/2016 15:30
1-14-1.sam	536.17	10392	1	14	3/28/2016 15:40
6-14-1.sam	223.95	289	6	14	3/28/2016 15:40
1-15-1.sam	468.13	6039	1	15	3/28/2016 15:55
6-15-1.sam	487.20	7100	6	15	3/28/2016 15:55

The Hanby Story

Our company has been around for the last 30 years as an environmental company. As Hanby Environmental we manufacture TPH Field Test Kits for the immediate analysis of water and soil samples. In a matter of 4 minutes for soil and 6 minutes for water, you will know the concentration level for the hydrocarbon or contaminate. This very economical quantification and qualification has proven to be an extremely valuable tool the world over on every major oil spill response and on remediation projects. By independent studies performed by the US EPA and US Army Corps of Engineers both found that the results per the Hanby method correlated to laboratory results within a variance of 10%, which is 2.5x better than the acceptable variance for field analysis of 25%.

The Hanby method is a visual colorimetric method utilizing the human eye to compare the sample results to the color calibration photos included in the kits with the color indicating the hydrocarbon or contaminant and the hue or lightness or darkness providing the concentration level thereof. More recently, rebranded under Hanby PetroAnalysis, we have developed a new portable and field ready instrument called the Hydrocarbon ID that standardizes the result reading effectively replacing the subjectivity of human eyes, with that of a computer eye by the use of a uv/vis spectrometer. Through this process, we put a lab in the field immediately obtaining results as accurate or more than that of a laboratory. The uv-vis spectrometer provides a unique identifying spectral curve and then an area under the curve calculation is made to get an exact concentration level. The unique spectral curve identifier is in line with DNA analysis and fingerprinting of hydrocarbons including crude oils.

Our chemical reaction produces robust colors by the excitement of aromatics that make up between 3-30% of a crude oils make up. Reading these results from samples of water, soil, cuttings, drilling mud/fluids or core samples, we are able to use the aromatics as the marker compounds to fingerprint the hydrocarbons including crude oils. This is a revolutionary new technology utilizing molecular spectroscopy in the field or at the wellhead. The numerous applications for this new technology are still being uncovered all the time.