May 16, 2016

Mr. Scott Besmer
Production Manager, Oilfield Services
KLJ Engineering
PO Box 1157
Bismarck, ND 58502

Dear Mr. Besmer:

Subject: Final Report Entitled “Evaluation of VISCOTAQ® Pipeline Wrap Performance Related to Crude Oil Exposure”; EERC Fund 20014

Please find attached the subject report prepared by the Energy & Environmental Research Center (EERC) detailing the pipeline wrap performance test results. It should be noted that final results not available at the time of initial review have been added in Section 5.0 Addendum.

If you have any questions or require clarification of any point, please contact me by phone at (701) 777-5050 or by e-mail at bkurz@undeerc.org.

Sincerely,

Bethany A. Kurz
Principal Hydrogeologist
Laboratory Analysis Group Lead

BAK/bjr

Attachment

c/att: Carolyn Nyberg, EERC

EVALUATION OF VISCOTAQ® PIPELINE WRAP PERFORMANCE RELATED TO CRUDE OIL EXPOSURE

Final Report

Prepared for:

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EERC DISCLAIMER

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EVALUATION OF VISCOTAQ® PIPELINE WRAP PERFORMANCE RELATED TO CRUDE OIL EXPOSURE

EXECUTIVE SUMMARY

This project was conducted to evaluate the performance of the VISCOTAQ® bell and spigot sealing system as a means of protecting rural water supplies from potential contamination in the event of a crude oil pipe leak at crossings with rural water supply pipes. The VISCOTAQ sealing system is a multilayer pipe wrap that can be applied over the joints of polyvinyl chloride (PVC) pipes to prevent exposure of the joint gaskets to crude oil, which can degrade the gasket material and result in leaks. To conduct this effort, a test apparatus was designed that included six jointed PVC pipes completely submerged in a saturated sand–crude oil mixture for a period of 8 months. Four of the six pipes were protected and wrapped with the VISCOTAQ sealing system, and two of the pipes were unwrapped. All six pipes were filled with distilled water, and an internal water pressure of 45 to 47 psi was maintained in four of the pipes throughout the testing, while two remained unpressurized.

Water samples from the pipes were collected periodically throughout the project and analyzed for total organic carbon as a first indicator of a hydrocarbon leak. Although organic carbon was detected in the water samples within the first month of testing, the levels were consistent among the six pipes, and additional testing confirmed that the organic carbon detected was from the pipe assembly materials and not from a crude oil leak. Following approximately 6 months of exposure, low levels of benzene, toluene, ethylbenzene, and xylenes (BTEX), a component of crude oil, were detected in the two pipes that were not protected or wrapped with the VISCOTAQ sealing system. Samples taken after 8 months of exposure confirmed the presence of BTEX in the same two pipes, and the levels approximately doubled, while no compounds were detected in the wrapped pipes.

Based on the experimental results obtained in this study, the VISCOTAQ sealing system appears to provide additional protection to bell and spigot joints of PVC pipes when exposed to crude oil for a period of 8 months, while the unprotected pipes showed evidence of a crude oil leak as early as 6 months.

EVALUATION OF VISCOTAQ® PIPELINE WRAP PERFORMANCE RELATED TO CRUDE OIL EXPOSURE

1.0 BACKGROUND

A major concern at the crossing of crude oil pipelines and rural water supply pipelines is the potential impact of a crude oil spill on the integrity of the polyvinyl chloride (PVC) pipe used for water pipelines in the unlikely event of an oil pipeline leak. One of the commonly used methods to provide an assumed layer of protection for water pipelines is to case them with additional PVC pipe at crude oil pipeline crossing points; however, a study conducted by South Dakota State University[1] to examine the impact of crude oil on the integrity of PVC and high-density polyethylene pipes and casing materials demonstrated that exposure of pipe joints to crude oil resulted in hydrocarbon permeation through the pipe joint gaskets within 5 to 9 weeks of exposure. The study results suggest that casing of PVC pipelines may not provide adequate protection in the event of an oil pipeline leak.

An alternative to casing of water supply pipelines may be the VISCOTAQ® bell and spigot sealing system. This self-adhesive wrap is designed to provide mechanical and chemical protection of PVC pipelines, which may provide a seal to prevent contact of crude oil with PVC joint gaskets. Appendix A contains a detailed product description. In order to test the performance of VISCOTAQ in preventing the degradation of PVC bell and spigot joint seals by hydrocarbon exposure, the Energy & Environmental Research Center (EERC) was subcontracted by KLJ Engineering to conduct bench-scale crude oil exposure testing of PVC joints that were unwrapped as well as wrapped with VISCOTAQ.

2.0 METHODS

2.1 Experimental Design

A detailed experimental design for evaluating the wrapped and unwrapped PVC joints was prepared by the EERC and provided to the project partners for review. This was to ensure that all interested parties were in agreement with the proposed approach, testing apparatus design, and test conditions. A detailed drawing of the testing apparatus is attached in Appendix B.

2.2 Testing Apparatus Assembly

Fabrication of the test box began in May 2015 at the EERC. It was constructed of 14-gauge, 304 stainless steel plate and ⅛" angle, and all plate materials were laser-cut. The six bell and spigot sample pipes were constructed of 6" PVC pipe with flanges and caps.

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[1] DeBoer, D.E., and Julson, D., 2012, Improving safety of crude oil and regional water system pipeline crossings: Final report to the Pipeline and Hazardous Material Safety Administration, Brookings, South Dakota, South Dakota State University.

Before inserting the pipes into the test apparatus, Chuck Holt, a representative from Pro-Kote Engineering and Supply, arrived at the EERC in early June to wrap four of the six pipes with the VISCOTAQ bell and spigot sealing system (Figure 1). Three of these were single-wrapped and one was double-wrapped. Once wrapped, the pipes were inserted in the box with the ends extending beyond the walls of the test apparatus, and each was fitted with a pressure gauge and valves for venting and sampling. Once the pipes were inserted, the box was reinforced and tested for leaks by filling it with water. The pipes were also flushed with water and tested for leaks. After the leak checks were completed, the pipes were flushed several times with tap water followed by distilled water and tested for total organic carbon (TOC) until levels were at background levels (<1 mg/L). They were then filled with distilled water. The water pressure in Pipes 1, 2, 4, and 5 was maintained at 45 to 47 psi, while there was no internal pressure in Pipes 3 and 6 (Figure 2). Each pipe contained approximately 21 liters of water. Below are descriptions of the six test pipes:

Pipe 1: Single wrap extending to the flanges on the internal walls of the box and sealed with silicone. Internal water pressure of 45 to 47 psi.

Pipe 2: No wrap. Internal water pressure of 45 to 47 psi.

Pipe 3: Single wrap with no silicone sealant. No internal water pressure.

Pipe 4: Double wrap with no silicone sealant. Internal water pressure of 45 to 47 psi.

Pipe 5: Single wrap with no silicone sealant. Internal water pressure of 45 to 47 psi.

Pipe 6: No wrap. No internal water pressure.

On June 11, 2015, the box was filled with a mixture of damp sand and Bakken crude oil. The sand and oil were added in layers by filling the box approximately one-third full with sand (Figure 3) and then pouring 20 gallons (four 5-gallon pails) of crude slowly over the sand, which was readily absorbed. This was followed by another layer of sand and 20 gallons of oil and then a final layer of sand and 15 gallons of oil (Figures 4 and 5). This resulted in a completely saturated sand–oil mixture (Figure 6). The test box was covered with a stainless steel plate lined with a Viton® gasket and bolted along the edges. A pressure release valve was installed in the center of the top cover, with a pipe vent to an exhaust fan.

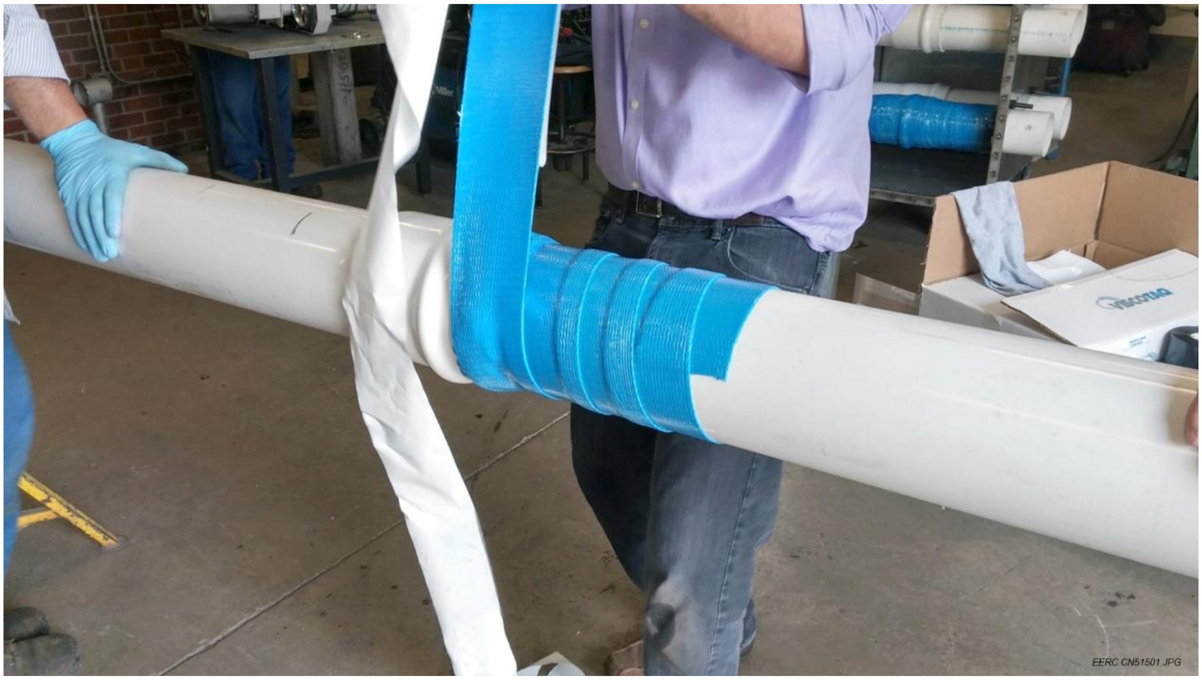

Figure 1. VISCOTAQ wrap being applied by Chuck Holt on June 2, 2015.

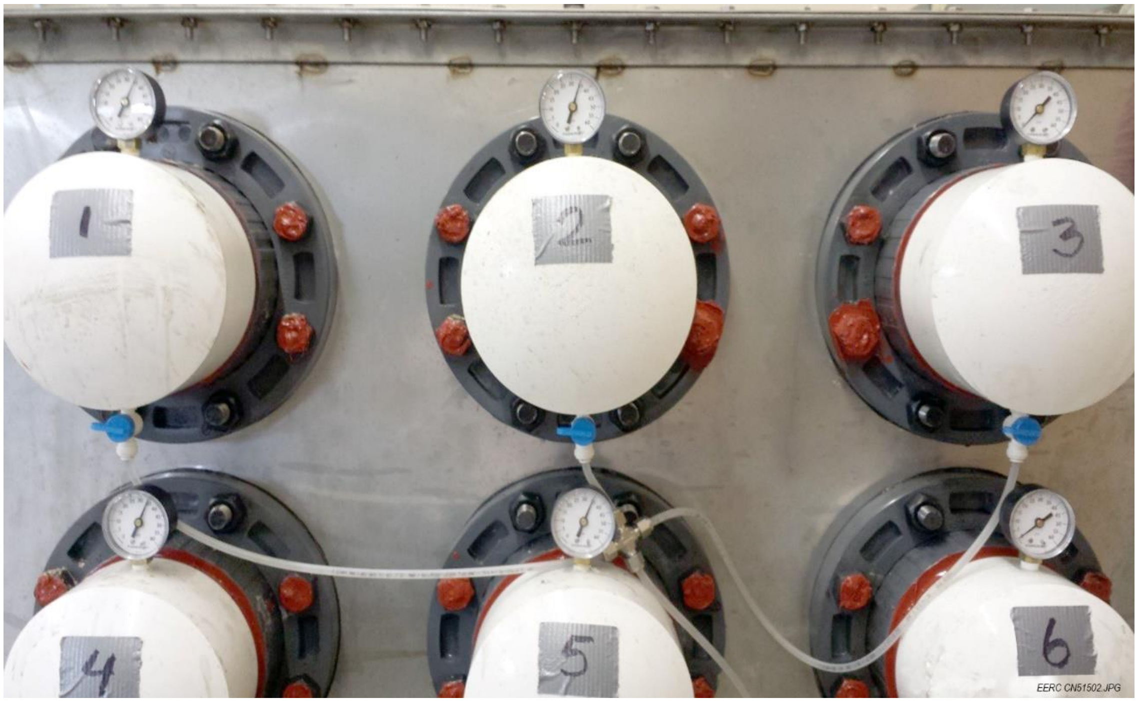

Figure 2. End view of pipes after internal water pressures were adjusted.

Figure 3. Top view of box after the first layer of sand was added.

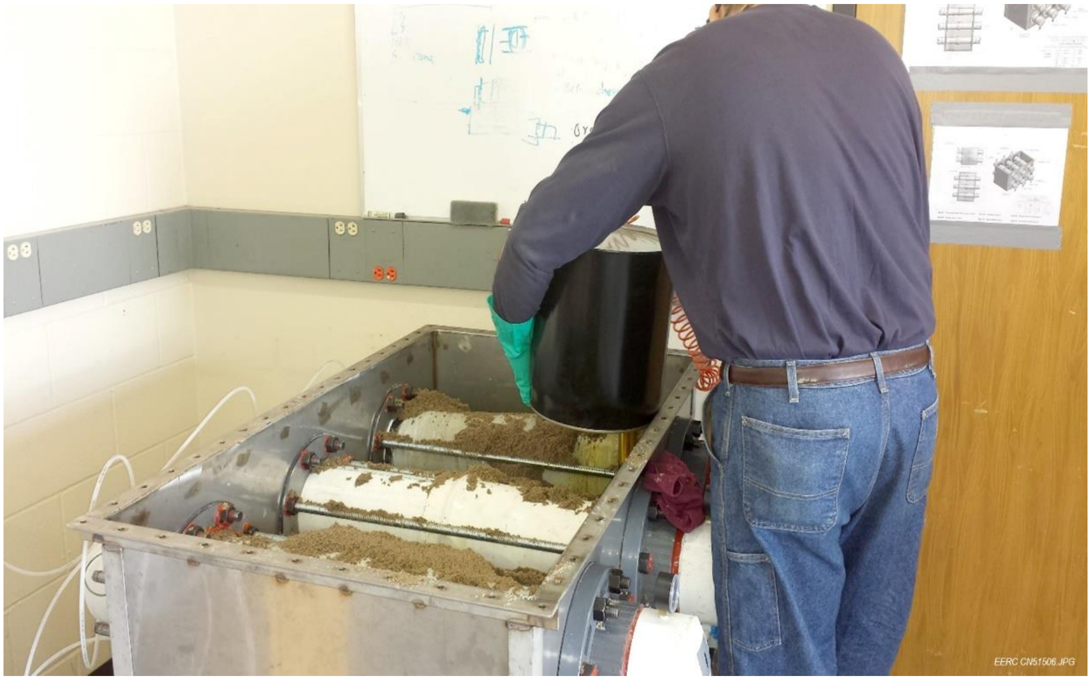

Figure 4. Second 20 gallons of crude oil being added.

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Figure 5. Final addition of crude oil.

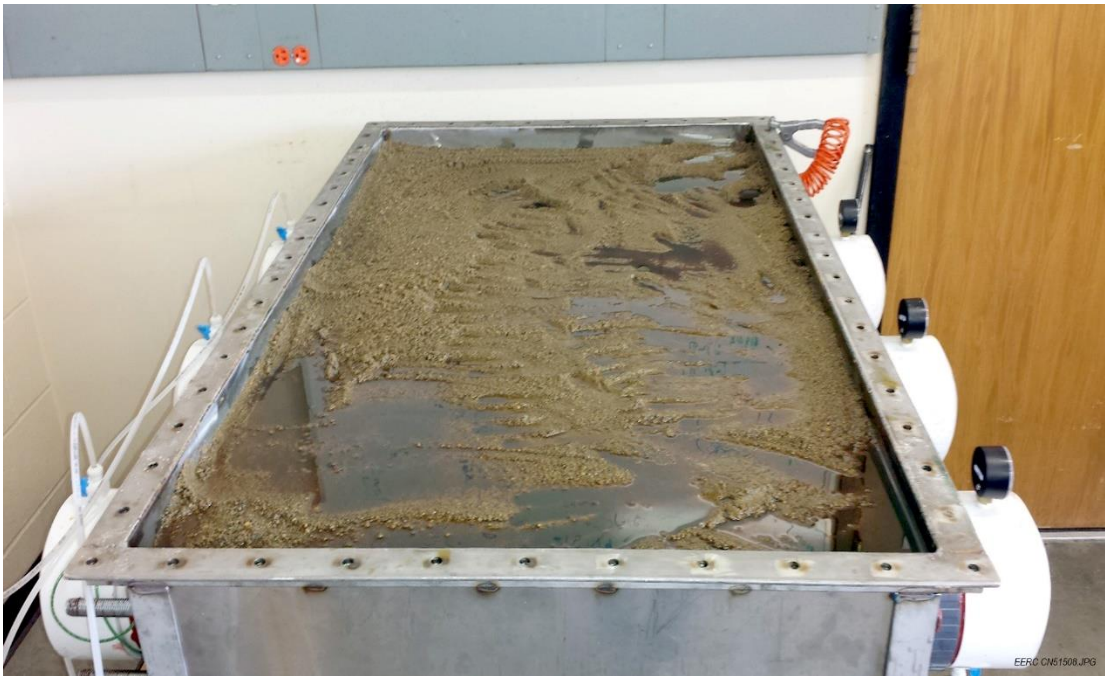

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Figure 6. View of the fully saturated sand before the top cover was applied.

2.3 Water Sampling and Analysis

2.3.1 Sampling Schedule

In the original scope of work, the pipe exposure experiment was scheduled to last for 6 months, with water samples collected from each of the pipes once a week for the first month, once every 2 weeks for the second and third months, and once every 3 weeks thereafter for a duration of 6 months. In accordance with this sampling schedule, the first water samples were collected on June 16, 2015 (Week 1), and the final samples were collected on November 30, 2015 (Week 25). However, since the test results did not show definitive signs of crude oil leaking into the pipes until the final sampling event, the proposed sampling was extended to include three more sampling events to take place in January, March, and April of 2016 at approximately 6-week intervals.

2.3.2 Analytical Methods

All water samples collected for this project were initially screened for organic carbon by analyzing for TOC using Standard Method 5310B as a first indicator of hydrocarbon breakthrough. Since the TOC results were low and remained relatively consistent over the course of 6 months, other analytical methods that were more specific in detecting hydrocarbon components were employed at different times throughout the testing. These included:

- Semivolatile petroleum hydrocarbons by U.S. Environmental Protection Agency (EPA) Method 8015B using a solvent extraction followed by gas chromatography–flame ionization detection (GC–FID). This method detects diesel range organics (DRO) or other hydrocarbons eluting between C10 and C28.

- Volatile petroleum hydrocarbons by EPA Method 8015B using purge and trap followed by GC–FID. This method detects gasoline range organics or other hydrocarbons eluting between C5 and C10.

- Volatile organic compounds by EPA Method 8260B using purge and trap followed by GC–mass spectrometry (GC–MS). This method detects benzene, toluene, ethylbenzene, and total xylenes (BTEX).

2.4 Tension Testing

One additional test was performed to evaluate the cohesiveness of the VISCOTAQ sealing system when used on rural water supply pipes that may be subject to expansive and compressive stress conditions as a result of seasonal temperature fluctuations in the subsurface. A jointed section of PVC pipe was wrapped with the VISCOTAQ sealing system, and an initial tension of 30 psi was applied to one end of the pipe, while the other end was fixed to a stable bracket (Figure 7). A spring gauge was used to monitor the tension on the pipe, and the gauge was checked regularly for the duration of the project. The tension remained at 30 psi, and no visible changes to the pipe or the VISCOTAQ pipe wrap were detected.

Figure 7. Wall-mounted pipe used for tension testing.

3.0 RESULTS AND DISCUSSION

3.1 TOC Results

The TOC results from the water samples collected during the 6-month exposure experiment (through Week 25) are presented in Table 1 and displayed in Figure 8. The results show a continual increase in the water from all pipes through Week 2. However, since the levels rose consistently in all samples, EERC staff believe that the increased organic carbon levels were likely because of one or more of the pipe assembly materials (i.e., cleaning solvent, primer, or glue) rather than a crude oil leak. To confirm this, all six pipe samples were screened for crude oil components by GC, and none were detected. A sample of the solvent used to clean the PVC pipe prior to adhering the end caps was also analyzed by GC, and the signature was consistent with that of the organic carbon within the water samples, confirming that the elevated TOC levels were from the cleaning solvent.

There was concern that the increasing levels of TOC from the pipe materials would mask the presence of low-level hydrocarbons from the crude oil if a leak were to occur. Therefore, to help minimize the contribution of these materials, it was decided that the pipes would be thoroughly flushed and refilled after each sampling event, followed by TOC analysis to confirm that levels returned to baseline concentrations of <1 mg/L. This procedure of draining and refilling with clean water after each sampling event is similar to that used in the South Dakota State University pipeline crossing study.[1] After the flushing was implemented in Week 3, the TOC levels

Table 1. TOC Results, mg/L

	Pipe 1	Pipe 2	Pipe 3	Pipe 4	Pipe 5	Pipe 6
Baseline	<1	<1	<1	<1	<1	<1
Week 1	7.0	7.4	7.5	7.5	9.5	9.0
Week 2	11.9	12.3	11.9	12.1	15.6	14.3
Week 3	2.9	4.3	2.3	2.7	4.7	3.1
Week 4	2.9	2.9	2.4	2.6	4.4	3.3
Week 6	4.2	4.4	3.9	4.7	5.8	4.8
Week 8	2.5	3.2	3.6	3.2	5.1	4.5
Week 10	1.9	2.8	2.1	2.5	3.4	2.9
Week 12	1.6	2.3	1.4	2.0	2.7	2.1
Week 15	2.2	2.2	1.7	2.1	2.9	2.7
Week 18	1.9	2.2	1.6	2.0	3.2	2.3
Week 21	2.1	2.0	1.3	1.2	2.8	1.2
Week 25	1.5	1.5	<1	1.2	2.0	1.7
Weeks 1–25 Average	3.5	4.0	3.4	3.7	5.2	4.4
Weeks 3–25 Average	2.4	2.8	2.1	2.4	3.7	3.0

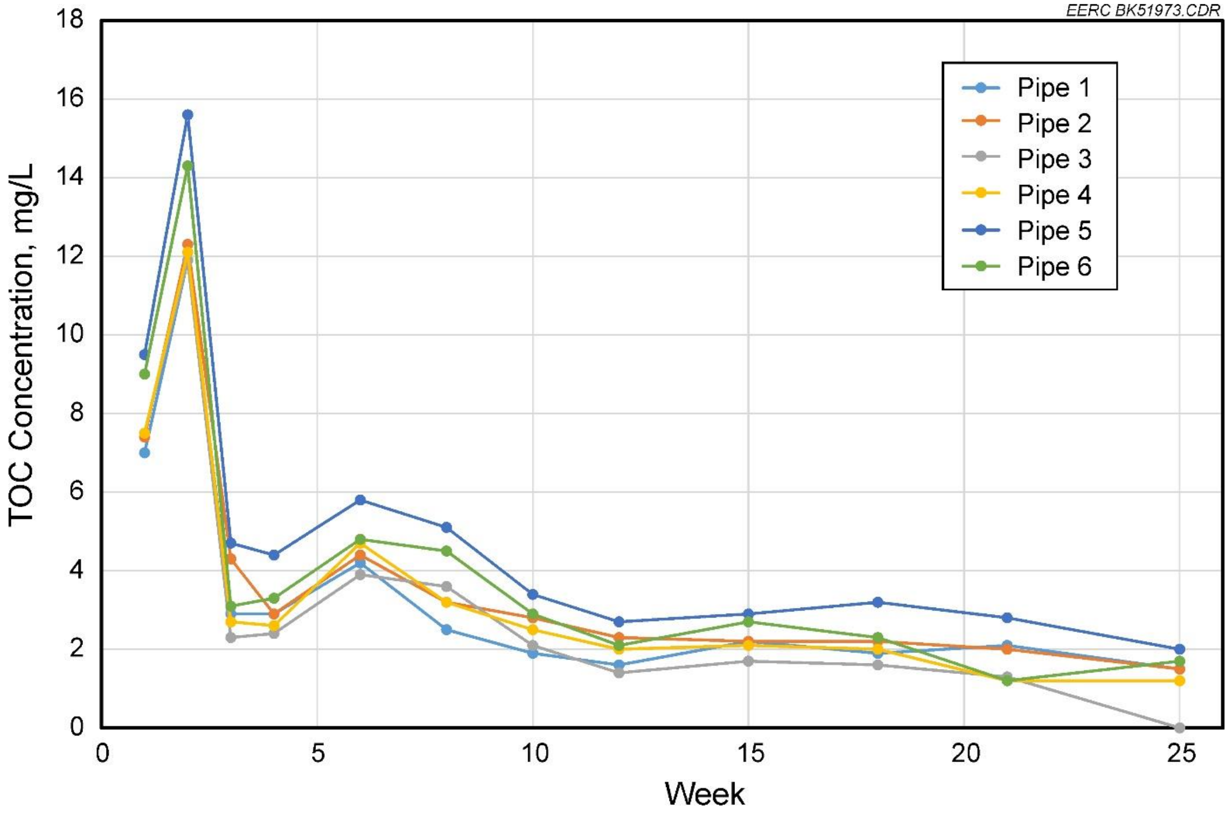


Figure 8. Comparison of TOC concentrations over the duration of the experiment.

remained relatively consistent for the remainder of the exposure experiment. With the exception of Pipe 6, the TOC levels during the last week of testing were actually lower than any of the previously reported results. Average TOC levels of the water were also calculated for each pipe (Table 1). Since the sampling conditions changed after Week 2 when the pipes were flushed, the averages were calculated separately for Weeks 3–25 and compared to the average including all weeks.

3.2 Total Petroleum Hydrocarbon Results

Although the GC screening that was done in Week 2 showed that the organic carbon levels seen in the TOC analysis were likely due to one or more of the pipe assembly materials, a second set of tests were done on samples collected in Week 4 to confirm this. The Week 4 samples were analyzed using EPA Method 8015B for total petroleum hydrocarbon (TPH)–DRO and total extractable hydrocarbons (TEH). These are also referred to as semivolatile petroleum hydrocarbons. The TPH–DRO results for all pipe samples were <0.3 mg/L. The TEH results ranged from 0.54 to 1.4 mg/L. However, after reviewing the chromatograms, it was confirmed that the peaks were caused by the pipe assembly materials and not a crude oil leak, and the results followed the same trend as the TOC results (Figure 9).

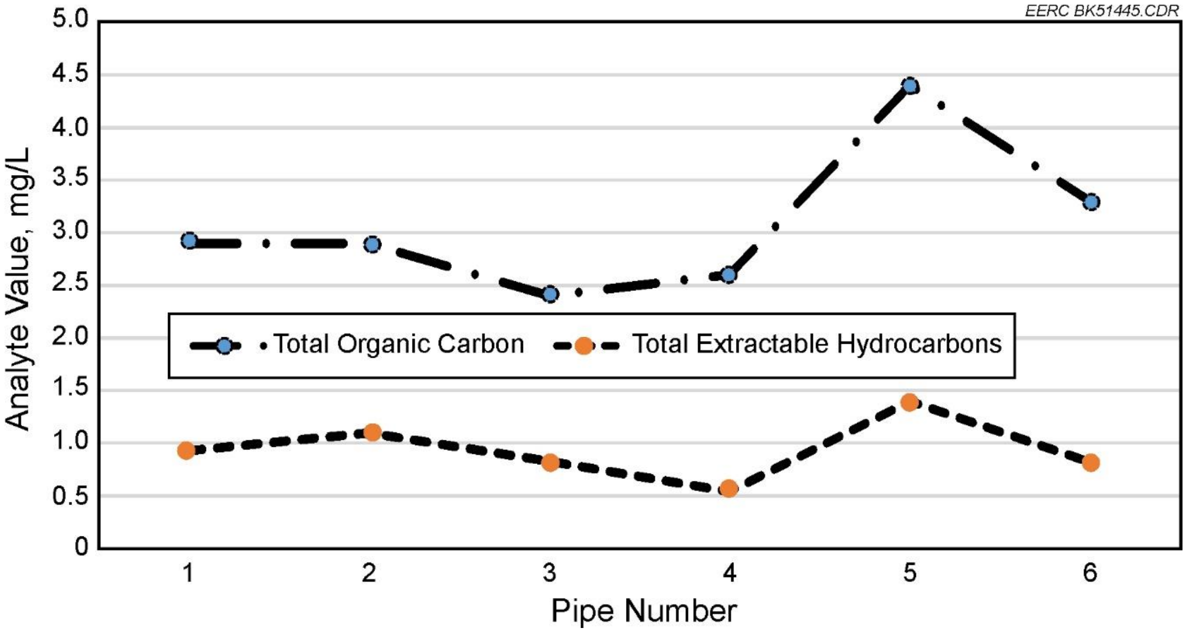


Figure 9. Comparison of TOC and TEH results for Week 4 samples.

As mentioned earlier, the TOC results showed little change over the course of the 6-month experiment. As a result, the last set of samples (collected at Week 25) were analyzed for volatile petroleum hydrocarbons and volatile organic compounds (BTEX) in addition to the regular TOC analysis and the TEH analysis performed on the Week 4 samples. The results of volatile and semivolatile petroleum hydrocarbon analysis by EPA Method 8015B are presented in Table 2. The data show that the semivolatile petroleum hydrocarbons were below or near the method reporting limit of 0.30 mg/L, ranging from nondetectable (ND) to 0.77 mg/L. The results of the volatile petroleum hydrocarbon using Method 8015B were above the method reporting limit of 0.02 mg/L, ranging from 0.386 to 0.606 mg/L. These results were similar to those from the sample set collected in Week 4 and were likely due to the organic chemicals used to assemble the PVC pipes, including tetrahydrofuran, acetone, butanone, and cyclohexane.

Table 2. Week 25 Volatile and Semivolatile Petroleum Hydrocarbon Results, mg/L

	Volatile Petroleum Hydrocarbons		Semivolatile Petroleum Hydrocarbons	
Sample ID	**Results**	**Reporting Limit**	**Results**	**Reporting Limit**
Pipe 1	0.606	0.02	0.31	0.3
Pipe 2	0.552	0.02	0.43	0.3
Pipe 3	0.443	0.02	ND	0.3
Pipe 4	0.386	0.02	0.34	0.3
Pipe 5	0.460	0.02	0.77	0.3
Pipe 6	0.603	0.02	0.43	0.3

3.3 BTEX Results

The most definitive and telling test results obtained from the Week 25 sampling were the BTEX results, which are presented in Table 3. The results for the Pipe 2 sample show BTEX compounds in concentrations significantly above the Method 8260B reporting limit of 0.0010 mg/L (1 ppb), and the sample from Pipe 6 shows benzene levels slightly above the reporting limit. All other pipe samples showed ND values. This may be significant since Pipes 2 and 6 are the only pipes that were not wrapped with the VISCOTAQ sealing system. The ratios of the individual BTEX compounds found in the Pipe 2 sample are reasonable for petroleum-derived BTEX, and their identification is likely correct since Method 8260B uses GC–MS, which is much more specific than the GC–FID used in Method 8015B. Since BTEX compounds are among the most water-soluble of crude oil components, it is possible they came from crude oil via small leaks in the two pipes that were not protected with the VISCOTAQ wrap (Pipes 2 and 6). The results also show slightly higher levels in the pressurized pipe vs. the nonpressurized pipe. There is no explanation for that at this point, and additional testing would have to be performed to determine if that was a consistent trend between pressurized and nonpressurized pipe.

Table 3. Week 25 BTEX Results, mg/L

Sample ID	Benzene	Toluene	Ethylbenzene	Total Xylenes
Pipe 1	ND	ND	ND	ND
Pipe 2	0.014	ND	0.0055	0.0023
Pipe 3	ND	ND	ND	ND
Pipe 4	ND	ND	ND	ND
Pipe 5	ND	ND	ND	ND
Pipe 6	0.0012	ND	ND	ND

3.4 Additional Testing for BTEX

Based on the test results from Week 25, it was determine that additional testing was warranted. The first of three additional sampling events took place at the end of January in Week 33, and the results show a similar trend to those from Week 25, with BTEX compounds showing up in the samples collected from the unwrapped pipes (2 and 6) and ND amounts in the wrapped pipe samples (Table 4). Additionally, the detectable amounts of BTEX compounds benzene, toluene, and xylenes appear to be increasing. The amount of benzene in both pipes approximately doubled; the amount of total xylenes in Pipe 2 also doubled, and a small amount of toluene showed up in Pipe 2 that was not present in the previous testing. However, the ethylbenzene that was detected in Pipe 2 in the previous sampling event was not present this time. Blanks that were analyzed with these samples were reported as ND for all compounds. It should be noted that the laboratory reporting limit for this method is 0.0010 mg/L.

Table 4. Weeks 25 and 33 BTEX Results, mg/L

	Benzene		Toluene		Ethylbenzene		Total Xylenes	
Sample ID	Week 25	Week 33	Week 25	Week 33	Week 25	Week 33	Week 25	Week 33
Pipe 1	ND	ND	ND	ND	ND	ND	ND	ND
Pipe 2	0.014	0.032	ND	0.012	0.0055	ND	0.0023	0.0054
Pipe 3	ND	ND	ND	ND	ND	ND	ND	ND
Pipe 4	ND	ND	ND	ND	ND	ND	ND	ND
Pipe 5	ND	ND	ND	ND	ND	ND	ND	ND
Pipe 6	0.0012	0.0021	ND	ND	ND	ND	ND	ND

4.0 SUMMARY

This report includes the experimental results of six water-filled PVC pipes with bell and spigot joints that were exposed to Bakken crude oil for a total of 33 weeks (8 months). Four of the pipe joints were protected with the VISCOTAQ bell and spigot sealing system, and two of them were unwrapped with no protection. Water samples from the pipes were collected periodically throughout the experiment and tested for TOC levels as a first indicator of hydrocarbon breakthrough. TOC was detected in all pipes early in the testing; however, it was confirmed that the organic carbon was from the pipe assembly materials and not from a crude oil leak. After approximately 6 months of exposure, minor concentrations of water-soluble crude oil components, including benzene, ethylbenzene, and xylenes, began to appear in the two pipes that were not protected or wrapped with the VISCOTAQ sealing system. Samples taken after 8 months of exposure confirmed the presence of BTEX in the same two pipes, and the levels approximately doubled, while no compounds were detected in the wrapped pipes.

These results indicate that for the 8-month exposure period during which this effort was conducted, the VISCOTAQ sealing system appears to be an effective mechanism to prevent the leakage of crude oil through bell and spigot pipe joints commonly used for rural water supply pipelines. Additional testing may be warranted to confirm the effectiveness of the VISCOTAQ sealing system over longer periods of exposure to crude oil. This method of protecting water supply pipelines at crossings with crude oil pipelines appears to be a more robust method of protection than current practices, since simply using a second PVC pipeline as a casing around the first PVC pipeline may extend the time it takes for oil to penetrate through the PVC joints, but not ensure that they will remain leak-proof for extended periods of time.

5.0 ADDENDUM

Table 5 reports final BTEX results that were still pending at the time the final project report was completed in March 2016. Since then, two additional sampling and analysis events took place: one on March 8 (Week 39), where the samples were collected but the analysis had not been completed at the time of reporting, and the second on April 19 (Week 45). The results from these two events were added to the BTEX results reported in Table 4 to clearly show that the BTEX components continued to increase in pipes not wrapped with the VISCOTAQ sealing system (No.s 2 and 6). It is also interesting to note that toluene, ethylbenzene, and total xylenes appeared in Pipe No. 6 during Weeks 39 and 45 but were not detected previously. These results represent a total exposure time of 10 months.

Table 5. Final BTEX Results, mg/L

	Benzene				Toluene				Ethylbenzene				Total Xylenes			
Sample ID	Week 25	Week 33	Week 39	Week 45	Week 25	Week 33	Week 39	Week 45	Week 25	Week 33	Week 39	Week 45	Week 25	Week 33	Week 39	Week 45
Pipe No. 1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Pipe No. 2	0.014	0.032	0.045	0.074	ND	0.012	0.019	0.026	0.0055	ND	0.0010	0.0012	0.0023	0.0054	0.0080	0.011
Pipe No. 3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Pipe No. 4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Pipe No. 5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Pipe No. 6	0.0012	0.0021	0.011	0.026	ND	ND	0.014	0.031	ND	ND	0.0024	0.0055	ND	ND	0.021	0.048

APPENDIX A

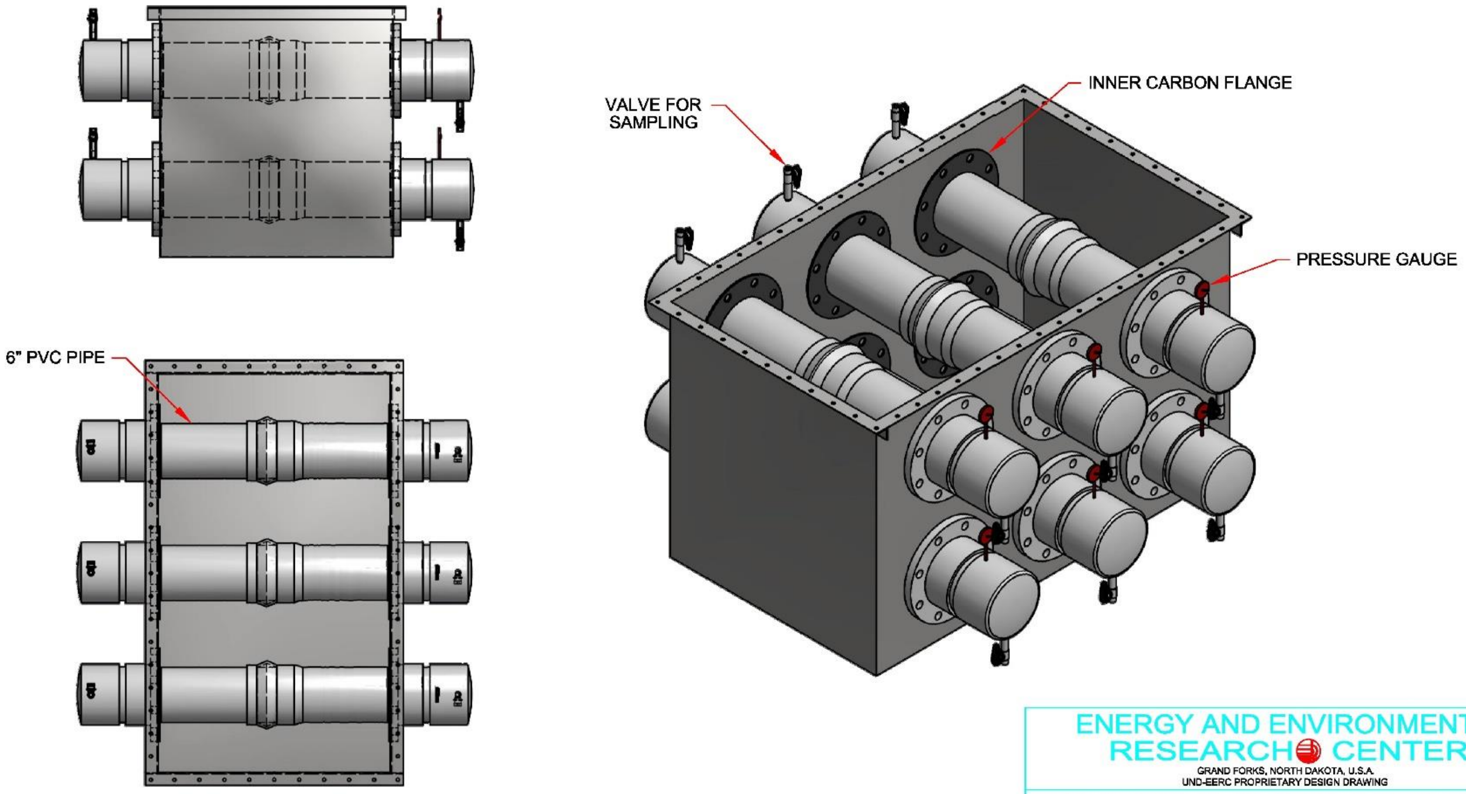
VISCOTAQ® PRODUCT DESCRIPTION

APPENDIX B

EERC TESTING APPARATUS DESIGN

5502

EERC BK51981.CDR



ENERGY AND ENVIRONMENTAL RESEARCH CENTER

GRAND FORKS, NORTH DAKOTA, U.S.A.
UND-EERC PROPRIETARY DESIGN DRAWING

PROPOSED PVC CONNECTION TEST, EERC

DRAWN BY: KG	ESS CHK: J. RICHTER	DATE: 2/24/15	W: 724 E: 642
DO NOT SCALE FROM DRAWING UNLESS SPECIFIED ALL DIMENSIONS IN INCHES	ENGR: J. RICHTER	DATE: 2/24/15	FUND NO: -
	CLIENT: -	DATE: -	REV: -
	EERC S.O. K. GROHS	DATE: 2/24/15	SCALE: 1:1
	MGR: B. KURZ	DATE: 2/24/15	SHEET 1 OF 1
	APVD: D. HAJICEK	DATE: 2/24/15	PRESSURE TEST DRAWING LEVEL: -
	FINAL ASSY: 2030	NEXT ASSY: -	DWG. NO: 5502

2014

Figure B-1. EERC testing apparatus design.