



# FLUITEC CONSULTATIVE SERVICES

## Fluid Failure Investigation

Petrochemical Plant  
Centrifugal Compressor

# EXAMPLE REPORT

**Customer** Petrochemical Plant (Example Report 1)  
**Subject** Centrifugal Compressor Oil Failure  
**Principal Analyst** Fluitec SME  
**Date** January 20XX

## BACKGROUND

In-service fluids were submitted for study to better understand the fluid's failure causes and determine how the reliability of the system could be improved. There was a comment about the fluid "burning" the skin when first taken from the sump. The fluid came from a swing compressor that operates between  $N_2$  and  $O_2$ . It was a Centrifugal Compressor, operating with an axial and process screw. There is a distance between the process gas section and the fluid sump that is exposed to the air. This operation is to keep the fluid separate from the working gas.

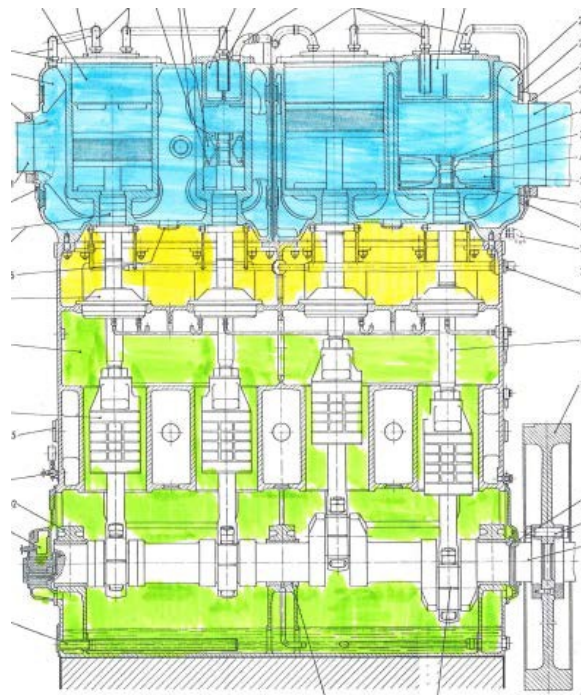


Fig. 1 Operational system Design

## INVESTIGATION

The fluid sample was initially studied by FTIR spectroscopy to learn the chemistry of the fluid. This fluid was defined as Brand X ISO VG 150. A new fluid sample for this fluid was not available, so it was compared with Brand Y and Brand Z which have similar formulations provided by the same fluid manufacturer.

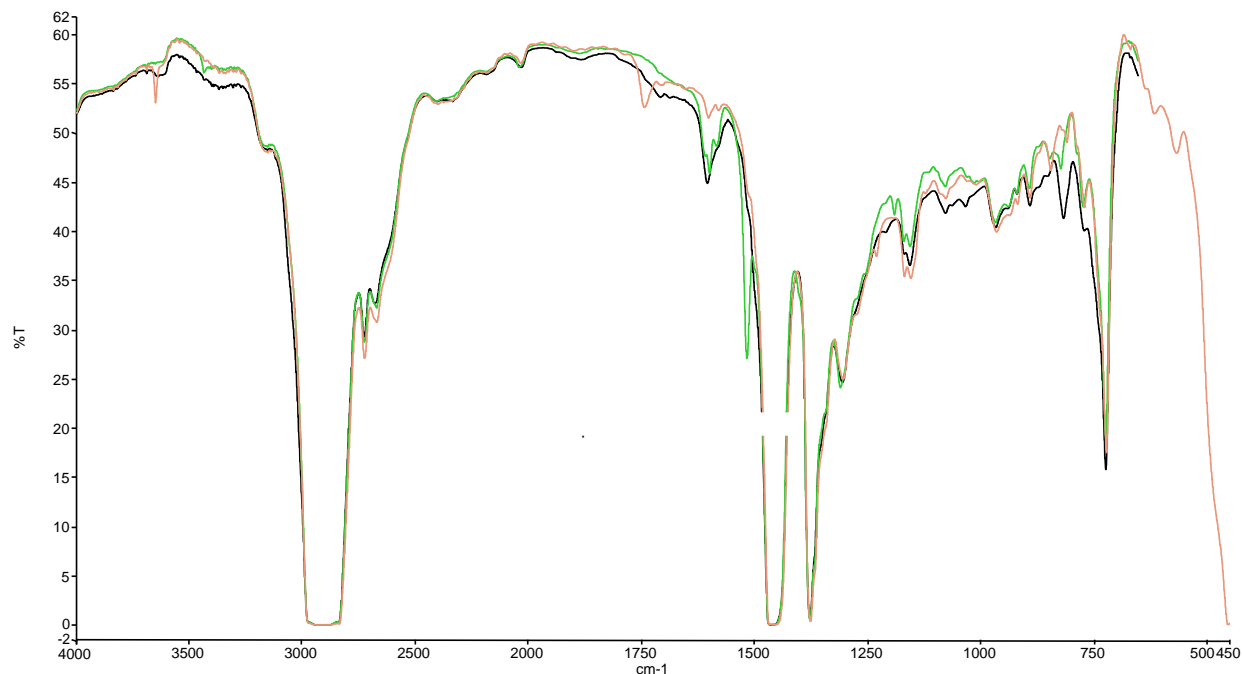


Fig.2 FTIR Spectra - In-service fluid compared against Brand Y and Z Fluids.

A summary of these infrared spectra indicates:

- The in-service fluid contains aromatic material at  $1600\text{ cm}^{-1}$ , indicating it is a Group I type base stock.
- There is no ester antifoam as seen in the Brand Y formulation.
- Degradation products exist at in the  $1700\text{ cm}^{-1}$  region – indicating oxidation has been occurring.
- The in-service fluid has a smaller aromatic amine content compared to the new Brand Z reference.

Traditional analyses of elemental, particle count and RULER show that the fluid is in severe condition.

Elemental analyses are obtained on a sample to determine the ingress of metals indicating potential wear problems. Metals like silicon and sodium are ingress metallic contaminants. Metals like iron, chromium and copper are internally generated contaminants indicating wear. Component wear may be the result of ingress metallic contaminants causing the wear, or varnish deposits creating accelerated wear conditions.

This sample showed wear elements of iron, copper and tin and ingress elements of silicon, calcium and sodium. The 18ppm of iron is a serious indication of wearing issues. The 22ppm of silicon is also critical. These both are recommended to be below 5ppm. The level of dirt ingress suggests that it is contributing to increased wear levels.

Element	Conc(ppm)
Iron	18
Copper	2
Lead	0
Aluminum	0
Tin	2
Nickel	0
Chromium	0
Titanium	0
Vanadium	0
Silver	0
Silicon	22
Boron	0
Calcium	4
Magnesium	0
Phosphorus	1
Zinc	4
Barium	0
Molybdenum	0
Sodium	4
Potassium	0
<b>ISO Code</b>	24/ 22/ 19
>4 Micron	88536
>6 Micron	34432
>14 Micron	2626
>50 Micron	115
>100 Micron	7
<b>TAN</b>	0.09

A measure of fluid contamination can be determined by Particle Count, reported according to ISO 4406-99. This test measures the “hard” particles in the fluid. Hard particles can be the root cause of wear problems, filter plugging and valve sticking. In this application, one wants to see particle count values in the 17/15/12 range. An ISO Particle code of 24/22/19 is extremely high for this application. This is critical, especially when detecting particles above 50 and 100 microns. Contaminant ingress is a likely source of premature component wear.

The TAN (Acid Number) for this fluid does not support the “burning” issue observed. This indicates the issue has dissipated before the sample was received. The observation was also tested when the sample arrived and not confirmed. This would indicate the burning was caused by something in the fluid that was no longer present – either reacted or evaporated (like a gas; however there was no statement of eye irritation).

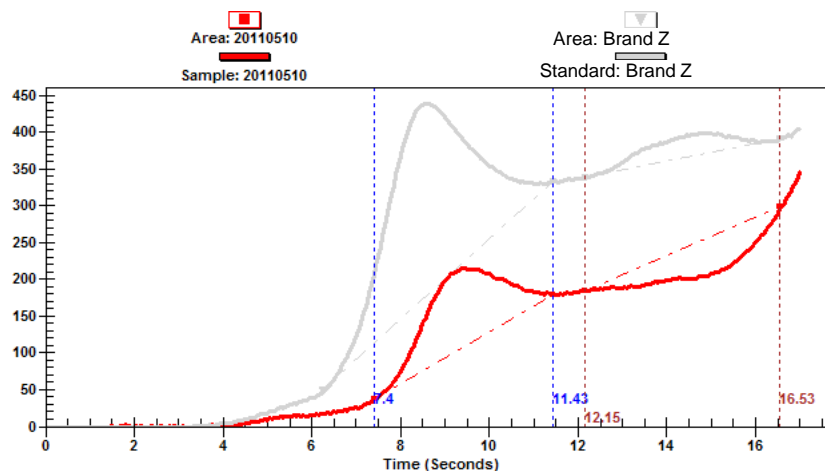
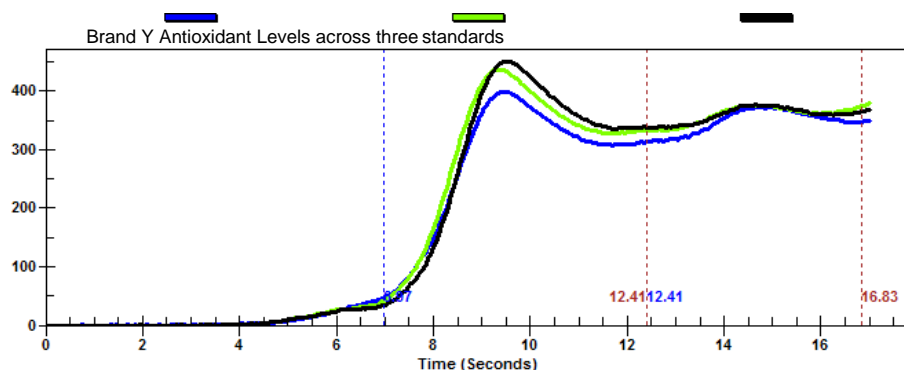
Linear Sweep Voltammetry (RULER) - ASTM D6971 has been developed to measure antioxidant health. This technique plots the aromatic amine and hindered phenol antioxidants concentration against a new oil

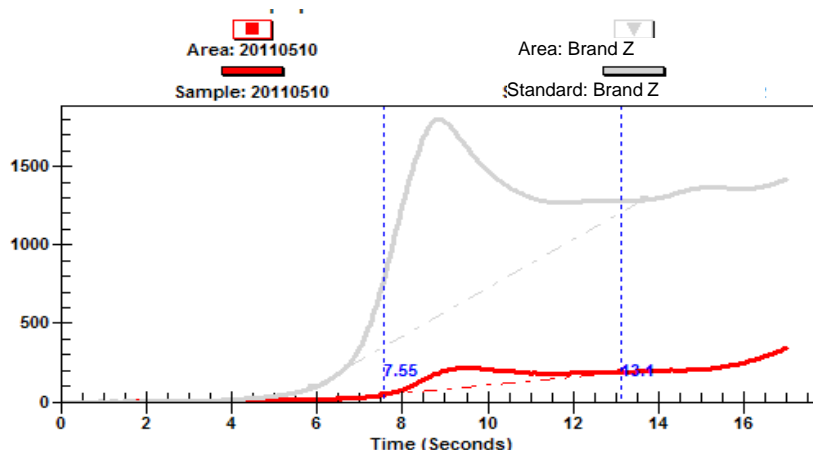
reference sample to determine the relative concentration. The RULER results are shown in the table below. Since an example of the new fluid was not available. Both Brand Y and Brand Z were compared. The practice of using the same formulation for multiple grades is also very common as shown by the graph of the ISO 32, 46 and 68 fluids. Thus the comparison of 32 and 150 vis-grades will yield similar information for comparisons.

With reference to either comparison, the in-service fluid has serious antioxidant depletion. A fluid with antioxidant levels below 50% amine is often alarmed and signals that the fluid is at risk of oxidizing. The low level of antioxidants is consistent with the observation in the FTIR spectrum of oxidation materials being formed in the 1700 cm<sup>-1</sup> region, indicating that indeed the fluid is oxidizing at the current level of antioxidants.

Table RULER Results

	Brand Y	In-Service Fluid	% Peak
(RUL1) Amine	12150.82	4415.01	36.3%
(RUL2) Phenol	1340.68	-2662.95	0%
<b>Total RUL:</b>	<b>75038.4</b>	<b>38512</b>	<b>51.3%</b>
	Brand Z	In-Service Fluid	% Peak
(RUL1) Amine	72929.63	6283	8.6%
<b>Total RUL:</b>	<b>273565.5</b>	<b>38512</b>	<b>14.1%</b>





Membrane Patch Colorimetry or MPC (ASTM D7843) is a technique of purposely isolating and measuring the specific degradation by-products responsible for sludge and varnish formation. MPC is an industry accepted predictive tool that can identify a lubricant’s potential to produce varnish. Visual and Spectrophotometric determinations assess the relative amount of compounds that absorb in specific color regions – thus comparing with a standard ASTM color number. The evaluation technique used can directly correlate the insoluble level to the varnish potential of the fluid. In the test fifty milliliters of oil sample is diluted with equal portion of petroleum ether to polarize and coagulate the oxidation by-products. The solution is then filtered through a 0.45 micron nitro-cellulose patch.

When this test was applied to this fluid, it plugged the filter with only 10 mls of the solution passing through the membrane. No more fluid would pass. This is an indication of critical level particles in the fluid. The varnish level on the patch (MCP = 63.4 dE) is also in what is considered as critical varnish potential.



The fluid being analyzed to this point was that fluid taken off the top of an unshaken sample of the fluid. This high level of particles is therefore from fluid that has been allowed to settle for several weeks before sampling. Heavy (large) particles would have settled by this time and all the filtration issues are from very small particles that remain suspended in the fluid. It was observed that there was about 1/16-inch of solids at the bottom of the sample bottle. This varnish material was isolated for characterization.

An isolation procedure for varnish samples followed by their characterization was developed. This procedure allowed the separation of the varnish to be achieved directly from the in-service lubricant fluids.

The isolated sample was then cleaned of the in-service fluid and submitted to characterization by FTIR spectroscopy. This procedure was developed to aid in root cause investigation studies. The picture below shows pictures of the varnish samples isolated from this unshaken sample.



A comparison of the material isolated from the bottom of the vessel and that isolated using the varnish isolation procedure from the top of the fluid was obtained using FTIR spectral analyses.

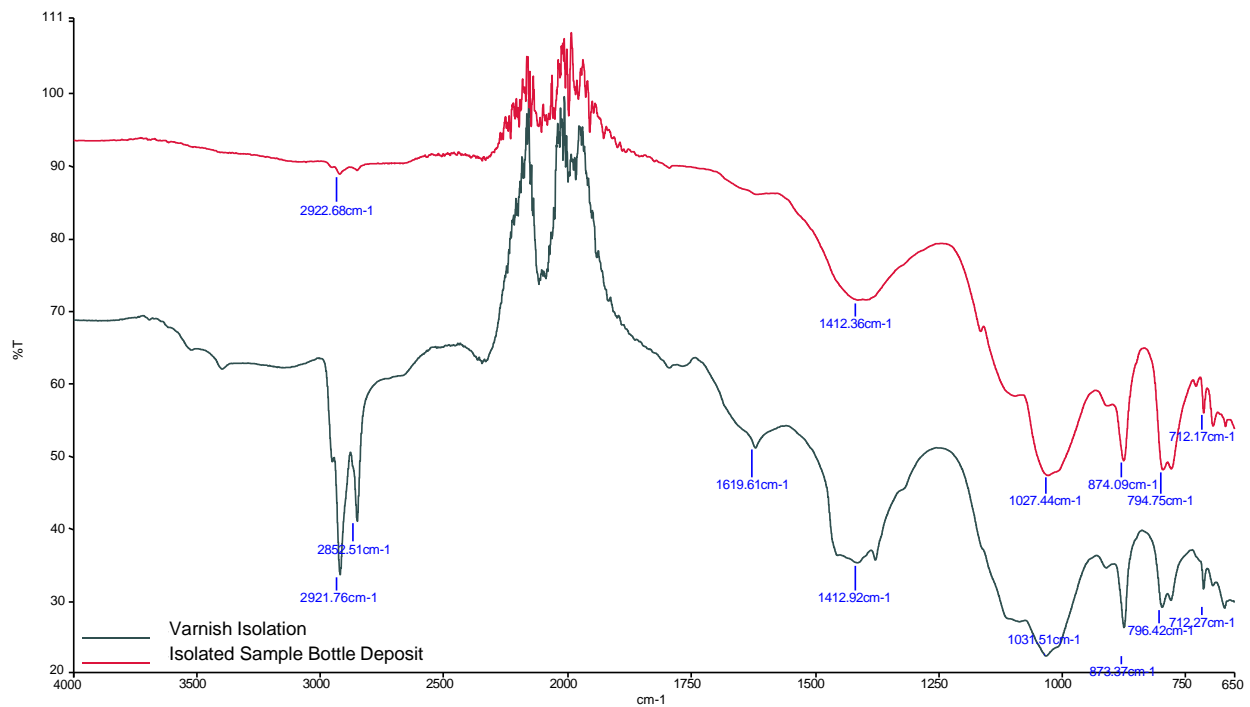


Fig. 6 FTIR spectra of isolated varnishes.

This figure shows that the two materials are very similar. The only difference is that the fluid varnish had a minor portion of some organics present. These organics can be defined as thermal-oxidative varnish by the presence of thermal-oxidative materials in the 1700-1600 cm<sup>-1</sup> region. This indicates one of the mechanisms of the organic varnish production is related to fluid over-heating in the equipment along with the low levels of antioxidant protection.

The major portion of both varnish samples is seen as that isolated from the bottom of the sample. This varnish material can be defined as inorganic in nature. This was determined because the sample does not contain any C-H bonds (observed in the 3000-2800  $\text{cm}^{-1}$  region). Comparing this sample with known references materials, suggests its likely chemistry.

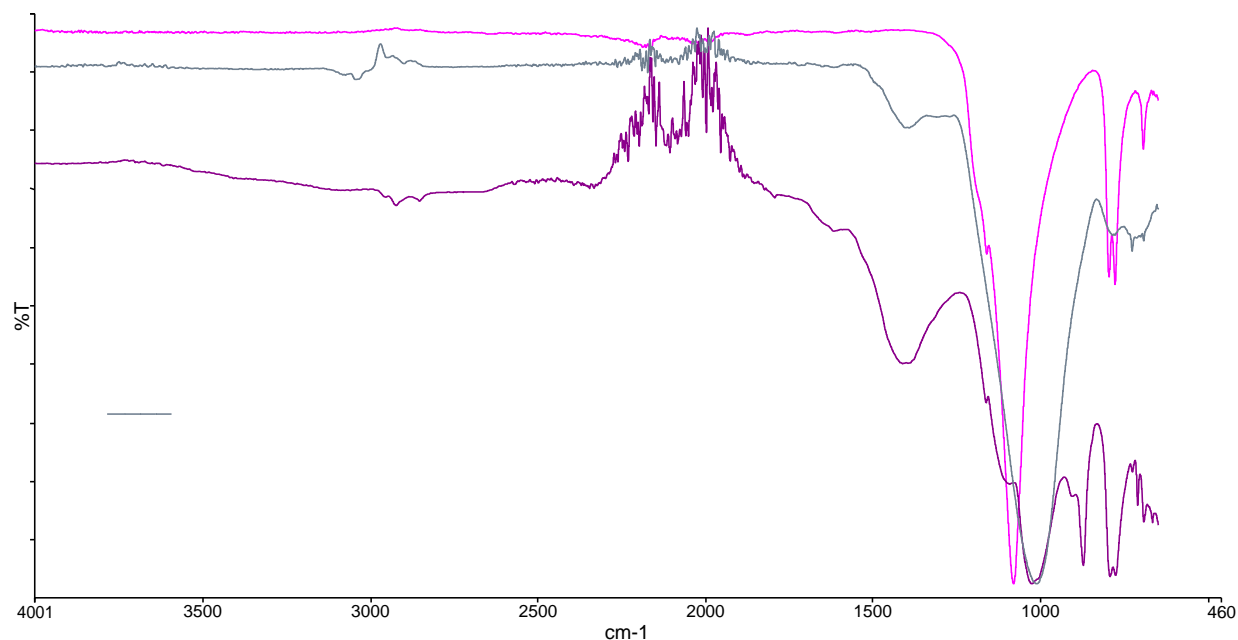


Fig. 7 FTIR spectra of isolated varnish compared against Silicates

This spectra comparison shows that the major portion of the varnish is silicate. Silicates can come from minerals ingress like silica, fuller's earth or sand. They can also come from waste-gas combustion products – which contain silicates and chloro-silicates. It should be noted that chloro-silicates will react with time to become the silicates observed. They also could cause the observed burning of the skin observed by the customer. One would not expect these chloro-silicates to still be present when the sample was received since their reactivity is too high. There is no evidence of chloro-silicates in the fluid or in the isolated varnish. Due to the “burning” observation however this source needs to be further investigated.

The remainder of the varnish can be identified by comparing it with carbonates.



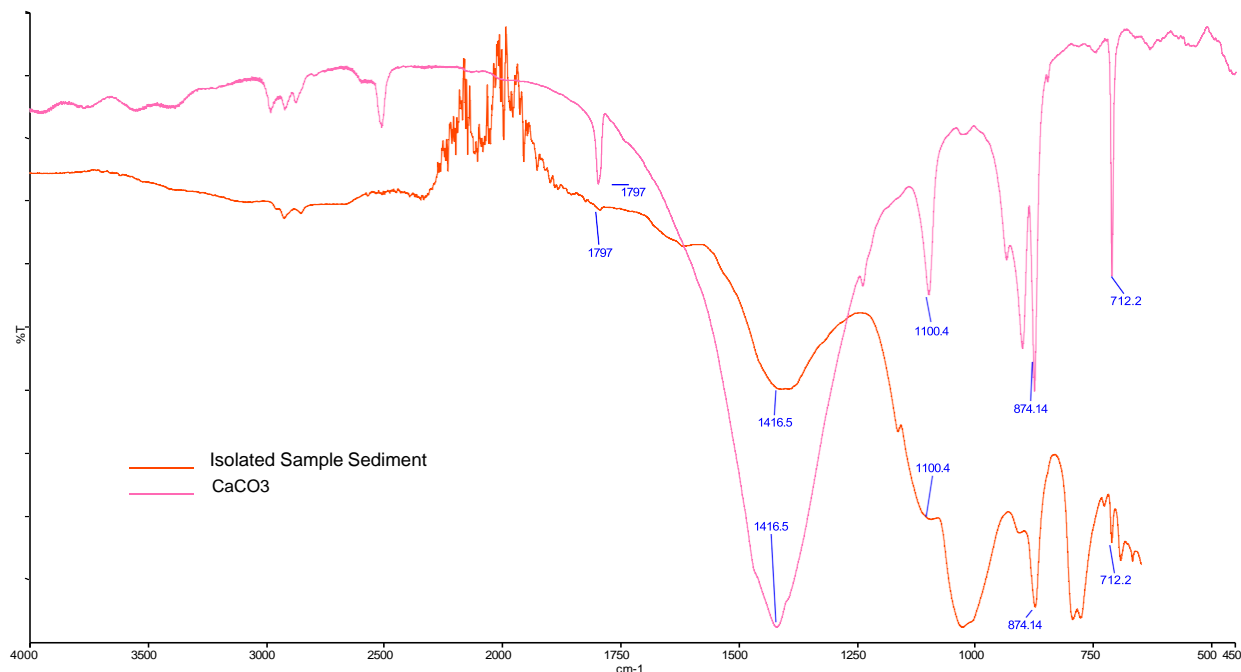


Fig. 8 FTIR spectra of isolated varnish compared against carbonate

Carbonates can come from the detergent packages (measured by TBN) of some crankcase fluids, CO<sub>2</sub> compressor gas ingress and minerals like calcite and bentonite. The fluid shows calcium, sodium and silicon which are the make-up of several of these minerals potential for the carbonates and silicates.

## CONCLUSION

Based on the sample submitted, the fluid is in severe condition warranting action. These findings are based on the following points:

- The antioxidants are at a low level.
- There is evidence of fluid oxidation
- The particle count is very high, with visible sediment in the bottom of the vessel. Filtration of the fluid through a membrane causes quick blinding of the membrane. All of these factors indicate a very high level of contamination that is likely contributing to premature component wear.
- The varnish and sediment from this fluid is primarily a combination of silicates and carbonates with enough organic thermal-oxidative material to add in as the binder. These carbonates and silicates are not something typically observed in fluids such as this. The inorganics are likely to be minerals – like calcite, bentonite, silica and sand.

There are multiple possibilities to reducing the high level of contaminants in the fluid and accelerated thermal failure of the fluid. Through phone consultation and site visitation, we can better determine these fluid failure sources to arrive at the optimum mitigation strategy to solve this problem.