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Leak Detectors for Organic Rankine Cycle Power Plants: On-Line and Manual Methods

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October 1984

**Prepared for the U.S. Department of Energy
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**Pacific Northwest Laboratory
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SUMMARY

Two leak detector systems have been designed, built, and tested at a binary-cycle (organic Rankine cycle) geothermal plant. One system is capable of detecting water in hydrocarbon streams down to 100 ppm liquid water in liquid isobutane. The unit first cools and/or condenses the hydrocarbon sample stream in a small heat exchanger. The cooled liquid stream flows to a large settling chamber where the water and isobutane separate because of density differences. Any water present is collected in a pipe and automatically dumped using a solenoid operated valve when the level reaches a certain point. The magnitude of the leak is estimated from the frequency at which the solenoid operated valve opens and closes, i.e. the amount of water collected in a known period of time is directly related to the number of dump cycles.

The second system can detect the presence of isobutane in water or brine streams down to 2 ppm liquid isobutane in liquid water or brine. The unit first cools the liquid stream if necessary then reduces the pressure in an expansion chamber so the hydrocarbon will vaporize. In brine streams flashed CO_2 carries the hydrocarbon to a non-dispersive infrared analyzer (NDIR). (In cooling water streams a nitrogen carrier gas is used to transport the hydrocarbon to the analyzer). The NDIR has been modified to be highly selective for isobutane. One can estimate the size of a leak knowing the total gas-to-liquid ratio entering the leak detection system and the concentration of hydrocarbon in the gas phase.

Four of the leak detector systems will be installed in the Heber Geothermal Demonstration Plant at Heber, California. Two will be on the hydrocarbon system, one on the brine system, and one on the cooling water system.

ORGANIZATION OF THE REPORT

This report describes three leak detection instruments which operate continuously. The leak detectors have many similarities but some subtle differences. For consistency, the sections describing each instrument (Sections II, III and IV) are organized in like fashion. Roman numerals are included in each sub section heading as an aid for the reader. That is:

- II.A. Design - refers to the hydrocarbon into brine leak detector.
- III.A. Design - refers to the hydrocarbon into cooling water detector.
- IV.A. Design - refers to the water/brine into hydrocarbon leak detector.

Section I is Introductory material while Section V describes simple back-up techniques for detecting leaks manually.

I. ORGANIC RANKINE CYCLE POWER PLANTS AND LEAK DETECTION

Rankine cycle power plants operate on a closed cycle in which heat is transferred from a high temperature reservoir to a low temperature sink while performing useful work. Leaks in this cycle cause the loss of working fluid and/or corrosion of the power plant. Leaks also result in brine contamination which can make the brine unacceptable for reinjection. A severe leak can reduce the efficiency of the cycle to the extent of creating a financial operating loss. Pacific Northwest Laboratories undertook the development of continuous monitoring techniques to protect Rankine cycle plants from losses of working fluid.

The location of these continuous monitors on an organic Rankine cycle is described below and shown schematically in the accompanying figure, using a geothermal reservoir as the high temperature heat source (Figure 1).

I.A. ORGANIC LEAKAGE INTO WATER/BRINE

The leakage of organic working fluid into either the geothermal fluid or the cooling water indicates a potential financial loss and probably foreshadows greater leaks in the future. The continuous monitor for this application is a cooled low pressure separator where gases come out of solution and are flowed to a continuous organic gas monitor. On the geothermal side there is enough inherent gas to sweep out the organic into the gas phase, concentrate it, and permit excellent sensitivities. For example, if a brine contains 500 ppm non-condensable gas and a leak were contributing 1 part per million (ppm) into the working fluid, the off-gas from this brine could consist of ~0.2% (~2,000 ppm) leaked working fluid. On the cooling water side there is no inherent dissolved gas so a nitrogen purge sweeps the organic into the gas phase and to a gas monitor. This instrument is planned to monitor the water flows downstream of the heat exchangers and condensers thereby covering leaks at both ends of the Rankine cycle.

BINARY POWER CYCLE LEAK DETECTOR LOCATIONS

Figure 1. Binary Power Cycle - Leak Detector Locations

I.B. WATER LEAKAGE INTO ORGANIC WORKING FLUID

In certain locations, and under some operational modes (e.g., startup), the normal pressure differential may be reversed to permit water to enter the organic working fluid and cause corrosion or turbine damage. The continuous monitor for this application is one in which a side stream of liquid hydrocarbon is pulled and flowed through a separator where the more dense water collects in a small diameter bottom section. In this lower section a capacitance level probe detects the (rising) level of water. When water reaches a set level a valve opens automatically and dumps the aqueous phase. The capacitance probe signal can be used to monitor a slow leak; the number of automatic dumps per hour can monitor a large leak. The instrument therefore has a large dynamic range capability. Laboratory tests on a simulated leak indicated that the instrument should have sensitivity to the 5 ppm level for insoluble organic fluids*. It was experimentally determined that vanes were necessary inside the separator to improve the efficiency of the water/hydrocarbon separation and therefore the overall efficiency.

This instrument could also be used in locations where gaseous hydrocarbons are present through the use of a flow cooler/condenser prior to the separator. The effectiveness of components such as a knock out drum might also be monitored by an instrument like this.

*Actual detection limit depends on the solubility of the organic fluid in water at the separator operating conditions.

II. HYDROCARBON WORKING FLUID INTO BRINE LEAK DETECTOR

The leak detector is a prototype instrument which has been calibrated and tested on one geothermal plant site. The leak detector is composed of a gas-liquid separation module and a gas phase composition monitoring instrument.

II.A. DESIGN

II.A.1. Principles

The isobutane* is separated from the brine by cooling the stream and lowering the pressure to near ambient conditions. Isobutane vaporizes and separates from the brine in an expansion chamber of the separation module. Isobutane is swept from the expansion chamber by CO₂ which is also released from the brine.

The gas stream passes on to a gas conditioning train and then to a non-dispersive infrared (NDIR) analyzer. The NDIR unit has been modified to be a highly selective detector for isobutane. The modification minimizes interference from methane which is found in many geothermal brines. The analyzer is equipped with an automatic zero/automatic span module which calibrates the instrument at operator specified intervals.

II.A.2. Details

Design details in this report are descriptive only. An operating manual is available (2). It gives detailed dimensions, calibration procedures and controller settings for proper operation in a power plant. A schematic diagram for the leak detector is shown in Figure 2.

*The hydrocarbon working fluid for testing the instrument was essentially pure isobutane. The leak detector should work equally well on mixtures of isobutane and isopentane such as those to be used in the Heber demonstration plant.

Figure 2. Hydrocarbon in Brine Leak Detector System Schematic

The leak detector itself samples a process stream continuously. The sampling rate is nominally 0.2 gallons per minute (gpm). The liquid is cooled when necessary; then the pressure is reduced to near ambient so the isobutane will vaporize. Gases are separated from the liquid in an expansion chamber. Gases flow on through a series of water traps and filters then to an NDIR analyzer. Liquid is drained out the bottom of a collection tube and sent to an atmospheric sump.

As plant operating conditions change, flow through the leak detector will vary also. Thus a control system is used to ensure some liquid is always in the unit but none flows to the gas analyzer. (Small amounts of liquid carry over in the gas stream are expected, but will be removed in the traps and filters). The level of liquid in the collection tube is sensed by a capacitance probe. Total volume of liquid in the system is determined by settings on a universal transmitter and two Setcon controllers. The controllers operate on the signal from the capacitance probe. The first controller actuates solenoid valves which admit liquid to the detector or drain liquid out of the collection tube. The second controller prevents accidental flooding of the separation chamber. If the liquid level is too high, this controller shuts a valve which stops liquid flow to the separation module. It also opens a second drain valve so the excess liquid can be dumped rapidly. Gas released from the brine occupies most of the collection vessel's volume. It is maintained a few psi above ambient by a back pressure regulator on a vent line. The slight overpressure is needed to ensure constant flow to the NDIR gas analyzer.

The gas analyzer is highly selective but not specific for isobutane. As shown in Figure 3, the analyzer produces infrared radiation from two separate energy sources. Once produced, this radiation is beamed separately through a chopper which interrupts it at 10 Hz. The radiation then passes through optical filters to reduce background interference from other infrared-absorbing components.

Figure 3. Infrared Sources and Cells

The infrared beams pass through two cells; one a reference cell containing a non-absorbing background gas, the other a sample cell containing a continuous flowing sample.

During operation, a portion of the infrared radiation is absorbed by the component of interest in the sample, with the percentage of infrared radiation absorbed being proportional to the component concentration. The detector is a "gas microphone" which operates on the Luft principle. It converts the difference in energy between sample and reference cells to a capacitance change. This capacitance change, proportional to component concentration, is amplified and indicated on a meter. It also produces a 4-20 ma signal for an external monitoring device. A non-dispersive infrared analyzer is generally sensitive to all hydrocarbons in a gas stream since it focuses on C-H vibration wavelengths. This modified unit focuses instead on C-C vibrations. That modification desensitizes the unit to methane which also exists in off-gases from geothermal brines. The modified analyzer is about 60 times more sensitive to isobutane than methane. Still the naturally occurring hydrocarbons in the non-condensable fraction of brines will elicit a small non-zero response from the NDIR analyzer. However, as long as the natural hydrocarbon background remains constant, this zero displacement should remain constant. The background level is expected to change as new wells come on line but will be fairly constant with time for a given well.

An auto-zero/auto-span accessory for the NDIR analyzer minimizes operator attention required for the unit. At some pre-selected time interval, the module alternately routes nitrogen, then a 100%-of-scale gas through the NDIR. (NOTE: The present unit has two scales; one is 2% isobutane maximum, the other is 10%). The automatic standardization procedure adjusts output from the zero/span module accordingly. This feature compensates for drift in the NDIR analyzer. Output

from the zero-span unit is a 4-20 ma signal: 4 ma = 0%, 20 ma = 100%. The signal is to be processed by a central computer. A built-in sample-and-hold amplifier can hold the latest sample value during the calibration sequence. Then the computer will not mistake the calibrations as alarm conditions. When geothermal brine is passing through the detector, the volume of gas released will be quite temperature dependent. Higher separator temperatures will give off more carbon dioxide and dilute the isobutane. conversely, at low temperatures, less gas is evolved and the measured isobutane concentration could approach alarm conditions. Therefore, it is recommended the temperature of brine into the separator be controlled to within a constant $\pm 5^{\circ}\text{F}$ using the built-in heat exchanger.

An additional type of hydrocarbon detector was tested for the determination of non-methane hydrocarbons (i.e. working fluid) in the non-condensable gas (NCG) identified in Table 1. This device was a catalytic hot wire detector. By adjusting the voltage on the hot wire catalytic element the manufacturer believed that the unit could be made to distinguish between methane (from the NCG) and the higher hydrocarbons (from working fluid leaks). The instrument manufacturer, however, showed that this performance was unachievable in the specified gas mixture. The reason for the failure was attributed to the presence of H_2 in the non-condensable gas; the combustion of this H_2 heated up the catalytic element independently of the applied voltage and lead to a lack of discrimination between C_1 and C_N hydrocarbons. In other gas compositions this simple and inexpensive unit might work well as a detector.

Table 1. Non-Condensable Gas Composition Used for Tests

<u>GAS</u>	<u>MOLE (%)</u>
CO ₂	60
N ₂	28
CH ₄	8.2
H ₂	1.6
H ₂ S	0.4
C ₂ H ₆	0.1
C _n H _{2n+2}	< 0.1

II.B. PERFORMANCE

Testing of the leak detector was accomplished using the piping arrangement shown in Figure 4. The brine source was a sample stream from the exit line of a reinjection pump in an operating geothermal plant. Sample stream flow rate from the reinjection pump was kept constant at 25 gallons per minute. Isobutane concentration to the leak detector was varied by changing the flow rate of isobutane into the brine.

Isobutane was injected into the brine stream using a small positive displacement pump. The small pump forced water into a 1 gallon cylinder filled with liquid isobutane. As water entered the cylinder, isobutane of equal volume was injected into the brine. The cylinder of isobutane was kept in a cool water bath to minimize the effects of ambient temperature changes. The density of isobutane changes slightly with temperature. Thus as ambient temperatures increased, the weight of isobutane injected could vary even though the volume was kept constant. This seemingly slight correction became important when detection limits for the instrument had to be established. Keeping the isobutane cylinder in the water bath kept the mass injection rate more nearly constant.

Figure 4. Leak Detector Test Stand Piping Diagram

II.B.1. Efficiency of Separating Working Fluid as Vapor From Brine

The leak detector itself works best when the brine flow is near 0.5 gpm. (That number was established experimentally). Sample port orientation and sample line configurations are very important. Liquid isobutane is lighter than brine and rises rapidly to the top of pipes. It will also collect in any vertical dead zones. Any sample port should be at the top of the brine line being sampled. The sample lines from the brine source to the leak detector should flow full at all times.

Table 2 shows representative recoveries of isobutane as concentrations in the brine changed during field tests.

Table 2. Isobutane Recovered in the Leak Detector

<u>iC₄ Injected</u> <u>ppm</u>	<u>Gas/Liquid Ratio</u> <u>l/kg</u>	<u>iC₄ Recovered</u> <u>%</u>
23	0.34	84
10	0.32	86
8	0.31	117
5	0.32	43

The greater than 100% recovery was attributed to thermal effects which were discussed earlier. The 43% recovery at 5 ppm is questionable. The pump for delivering the water used a stroke of only .025 inches and delivered only 8.4 cc of water per hour. This was considered the very lowest flow at which it could operate. There is some question about the pump delivering at a constant rate for the time required to flush the system and stabilize at a new reading (4 hours).

II.B.2. Sensitivity of Detecting Working Fluid in Vapor State

Separator off-gas from an East Mesa Geothermal brine was analyzed by gas chromatography and was found to have the following approximate composition:

Table 3. Gas Composition of an East Mesa Geothermal Brine

CO ₂	53%
N ₂ + H ₂	25%
H ₂ O (vapor)	variable
CH ₄	9%
C ₂ H ₆	900 ppm (vol/vol)
C ₃ H ₈	130 ppm
iso C ₄ H ₁₀	13 ppm
n C ₄ H ₁₀	17 ppm

The flowing brine was spiked with isobutane upstream of the separator and the separator off-gas was continuously monitored by a Beckman Model 864 non-dispersive infrared analyzer (NDIR). Periodically, the exit gas of the NDIR was injected into a Perkin-Elmer Model 910 Gas Chromatograph (GC). Comparison of NDIR response and isobutane content of off-gas as determined by GC is shown in Figure 5. Note the good correlation and near-theoretical sensitivity. Also the NDIR analyzer response to isobutane is highly selective but not specific. The naturally occurring hydrocarbon gases in the noncondensable gas fraction of brine elicit a background, non-zero response from the NDIR analyzer. The main contributor to the background response was found to be methane. Laboratory tests prior to actual field testing indicated that 100% methane elicited an NDIR response equivalent to 1.5% isobutane. The actual 9% methane in the separator off-gas is therefore expected to give an

Figure 5. NDIR Response vs. Gas Chromatograph Response

NDIR response equivalent to 0.14% isobutane. Note that the intercept of the NDIR analyzer calibration curve (Figure 5) is 0.16% isobutane--very close to the calculated methane background response.

II.B.3. Overall Detection Limit and Operational Conditions to Achieve It

In order to develop a predictive relationship between isobutane content of brine and concentration of isobutane in separator off-gas several operating parameters must be assumed or defined. The relevant parameters are: (1) separator recovery efficiency which is herein defined as the fraction of isobutane in brine that is stripped into the gas phase and (2) the gas/liquid ratio characteristic of the sampled brine.

Separator recovery efficiency was discussed in Section II.B.1. Gas/liquid ratio (G/L) is a function of separator operating temperature and pressure.

Derivation of Relationship Between Isobutane Content of Brine and Separator Off-Gas

Let X = isobutane content of brine (mg iC_4 /kg brine, i.e. ppm)

Y = % isobutane (vol/vol) in separator off-gas

α = separator recovery efficiency ($0 \leq \alpha \leq 1$)

R = gas/liquid ratio (liters gas evolved/kg brine)

Also, a molar volume of gas at ambient desert conditions of 25 L/mol is assumed.

It can be shown that:

$$Y/X = 0.043 (\alpha/R)$$

An analysis of the noncondensable gas fraction of Heber brine done by Fluor Power Services, Inc. found 48.5 ppm (wt/wt) noncondensable gas having a calculated molecular weight of 36.1 g/mol. This data yields a value of $R = 0.034$. Assuming a conservative value of $\alpha = 0.5$, one calculates that:

$$Y = 0.63X$$

That is, 1 ppm isobutane in brine will yield 0.63% (vol/vol) isobutane in gas stream to the NDIR analyzer.

San Diego Gas and Electric chose 10 ppm isobutane in brine as the upper tolerable limit. Therefore an alarm level of 6% isobutane in separator off-gas from Heber geothermal brine is called for.

II.B.4. Calculated Performance for Plant Use

The ultimate use of the leak detector system has never been to quantitatively analyze for hydrocarbon leaks. It is merely an alarm device. Economic considerations by San Diego Gas and Electric Company established that the unit must be able to detect at least as little as 10 ppm isobutane in the brine. Also, restrictions were set by the Chevron Company on the total hydrocarbon which can be present in the reinjected brine. Thus, operators must be alerted any time hydrocarbon leaks develop.

If semi-quantitative information is desired the liquid-to-gas ratio must be established as a function

of temperature for the brine being sampled. Then the ppm isobutane in the brine can be calculated using the formulas given in the previous section. The type of curve needed is shown in Figure 6. Obtaining the liquid to gas ratios requires collecting gas and brine simultaneously. The gas can be collected in an inverted graduated cylinder initially filled with water.

Figure 6. Gas-to-Liquid Ratio as a Function of Temperature

III. HYDROCARBON WORKING FLUID INTO COOLING WATER LEAK DETECTOR

III.A. DESIGN

III.A.1. Principles

The leak detector is composed of a gas-liquid separation module and a gas phase composition monitoring instrument. A schematic of the leak detector is shown in Figure 7.

The isobutane is separated from the water by lowering the pressure to near ambient conditions. Isobutane vaporizes and separates from the water in an expansion chamber of the separation module. The cooling water stream must be purged with nitrogen to sweep out the isobutane.

The nitrogen and isobutane gas stream passes on to a gas conditioning train and then to a non-dispersive infrared (NDIR) analyzer. The analyzer is equipped with an automatic zero automatic span module which calibrates the instrument at operator specified intervals.

III.A.2. Rationale for Choice

Three distinctly different analyzers were considered: gas chromatograph, combustible gas analyzer, and non-dispersive infrared analyzer (NDIR). In principle, each of these analyzers will work. Practical considerations and plant operator (San Diego Gas & Electrical Company) preference led to choosing the NDIR analyzer.

Combustible gas analyzers can be subject to drifting baseline and may have marginal sensitivity in some

Figure 7. Flow Diagram for Hydrocarbon in Cooling Water Detector

possible gas composition ranges. Process chromatographs, operating with flame ionization detectors, require frequent operator attention for maintenance, compressed gas bottle change out, etc.

The NDIR analyzer fitted with an automatic zero/span module will operate with little or no operator attention for months at a time.

The gas analyzer used for testing is identical to the one described in Section II.A.2 of this report. Since methane interference is not a problem in cooling water, other hydrocarbon detectors could also be used.

Nitrogen carrier gas must be used to sweep any isobutane to the gas analyzer. Since nitrogen is nearly insoluble in water at any temperature (and near ambient pressure), temperature control is not important. Maintaining a constant nitrogen flow is critical however.

III.B. PERFORMANCE

III.B.1. Efficiency of Separating Working Fluid as Vapor From Water

Unlike previous tests on geothermal brine samples, an air or nitrogen sparge must be incorporated into the separator vessel since there are no inherent gases evolving from the liquid phase to sweep isobutane to the gas phase detectors. Thus additional operating parameters, beyond those found in the brine leak detector, e.g., air/water flow ratio, were evaluated. Fortunately, the gases sparged from cooling water are much lower in background hydrocarbon content so that the detection instrument need not be as selective for

isobutane as is the case for brine streams. In fact, a simple nonselective combustible gas analyzer served adequately in short term tests.

The separator recovery efficiency (SRE) was measured in order to establish the sensitivity and quantitative performance of the leak detector. This was done by injecting controlled amounts of gaseous isobutane into the cooling water stream prior to the separator and relating that to the amount measured by the gas phase detector. The difficulty of continuously injecting a precisely known amount of isobutane accounts for the scatter of the individual test results. Although individually scattered, these results, when averaged (Table 4), indicate a fairly uniform recovery rate.

Table 4. Efficiency of Removing Isobutane to the Vapor Phase From Cooling Water Flow

<u>Test Series</u>	<u>Number of Tests</u>	<u>Range of iC₄ Content of Water (ppm)</u>	<u>Average Isobutane Separator Recovery Efficiency (%)</u>	<u>SRE Standard Deviation %</u>
A	3	9-31	52	5.5
B	3	47-77	45	12.5
C	4	6-10	46	11.3
D	6	43-77	50	15

Separator Recovery Efficiency (SRE)

SRE as a function of isobutane content of cooling water is roughly 50%, and a 50% SRE value will be used for illustrative calculation purposes below. The test data indicates that the sparge air/cooling water flow ratio does not effect the SRE.

Gas Sparge Rate Limitations

On one hand, the lowest possible sparge gas flow rate is desirable in order to maximize the isobutane content of the off-gas for a given concentration of iC_4 in cooling water. That is, the smallest practical value of sparge gas/cooling water flow rate ratio is desired. In practice, however, too low a sparge gas flow rate will lead to a "negative" pressure in the separator compartment during the "dump" phase of the programmed fill-dump cycle of separator operation. Therefore, a compromise in sparge gas flow rate is needed. A flow rate of approximately 0.5 L/min was found to be sufficient to prevent "negative" pressure during the dump phase. Of course, modification of the dump valving and plumbing could change the situation. That is, larger diameter tubing and valve ports in the dump plumbing would allow faster emptying, and thus require a higher sparge gas flow rate to maintain positive pressure in the separator compartment.

III.B.2. Sensitivity of Detecting Working Fluid in Vapor State

A Beckman Model 864 NDIR analyzer fitted with a 0 to 500 ppm* isobutane scale range has the required sensitivity. The next section outlines the calculation of expected sensitivity requirements.

III.B.3. Overall Detection Limit and Operational Conditions to Achieve It

In this section, an equation is derived for the overall detector sensitivity, i.e., ppm (vol/vol) isobutane in the off-gas stream as a function of isobutane content of cooling water, cooling water flow rate, separator recovery efficiency, and sparge gas flow rate.

*The 500 ppm is a "best guess" for a reasonable maximum on the Heber brine. The number is used for illustrative purposes on some sample calculations which follow.

Let X = ppm iC_4 in cooling water (mg/L)

F_1 = flow rate of cooling water into separator
(L/min)

α = separator recover efficiency ($0 \leq \alpha \leq 1$)

F_2 = flow rate of sparge gas (L/min)

Then $\alpha F_1 X$ = mg iC_4 entering gas phase/min

$$\frac{\alpha F_1 X}{F_2} = \text{mg } iC_4 / \text{Liter of off-gas } F_2$$

Now: molar volume of gas at ambient conditions
(1 atm, 90°F) is approximately 25 L/mol and molecular
weight of isobutane is 58 gm/mol.

Thus: each liter of off-gas contains approximately
40 mmol and the mole fraction of isobutane is given
by:

$$\frac{\alpha F_1 X}{(58)(40)F_2}$$

For gases at separator operating conditions, mol
fractions are synonymous with volume fractions.
Converting to ppm (vol/vol) isobutane in off-gas, one
obtains:

$$Y(\text{ppm (vol/vol) } iC_4 \text{ in gas phase}) = 431 \frac{\alpha F_1}{F_2} X$$

$$\text{or } X = 2.32 \times 10^{-3} \frac{(F_2)Y}{\alpha F_1}$$

The equation above relates the analytical instrument
detected quantity, Y (ppm iC_4 (vol/vol)) to the isobu-
tane content of the sampled cooling water X , mg(iC_4)/L
water.

III.B.4. Calculating Plausible Alarm Conditions

At a very large Rankine cycle plant like the Heber Geothermal Demonstration Plant (65,000 KWE gross) large cooling water flows of 65 million pounds per hour will be used. If we have an on-line instrument in use we can calculate its sensitivity to leaks as described below. As an example calculation, using the value of 0.5 for α -separator recovery efficiency, F_1 -water flow rates-(0.3 L/min), and F_2 -sparge gas flow rates-(0.5 L/min), one calculates $X = 0.0077Y$.

Viewed in the opposite sense, 1 ppm iC_4 in cooling water is expected to yield approximately 130 ppm (vol/vol) iC_4 in the off-gas stream if the same values for parameters (α , F_1 , and F_2) are used.

If overall sensitivity is viewed (correctly) as $\frac{\Delta Y}{\Delta X}$, it is clear that the sparge gas/water flow rate ratio (F_2/F_1) influences sensitivity. Higher gas to liquid ratios lead to diminished sensitivity as does smaller α . Once α and F_2/F_1 are established, however, the overall detector sensitivity is uniquely defined and an appropriate alarm level can be set on the analytical detection instrument.

So if the instrument has a full scale range of 0-500 ppm and an automatic zero adjustment then the alarm point could be set at a value corresponding to a desired sensitivity (Table 5).

Table 5. Alarm Setting vs. Leak Sensitivity

<u>Alarm Setting (% Full Scale)</u>	<u>Alarm Setting (C₄ ppm)</u>	<u>ppmC₄ in Cooling Water</u>	<u>C₄ Plant Loss pounds/hour (6.5 x 10⁷ Multiplier)</u>
5%	25	.19	12
10%	50	.39	25
20%	100	.77	50
40%	200	1.5	100

Instrument Flow Conditions as Described in Text. 500 ppm is Full Scale.

IV. WATER/BRINE INTO HYDROCARBON WORKING FLUID LEAK DETECTOR

The leak detector is a prototype instrument which has been calibrated and tested at one geothermal plant site. The leak detector when properly installed with appropriate associated equipment will detect the presence of water in a flowing isobutane stream. It gives information allowing the size of a leak to be estimated, at least to an order of magnitude.

IV.A. DESIGN

IV.A.1. Principles

The water/isobutane stream must be liquid while flowing through the instrument. Water separates from the isobutane by gravity alone in a large expansion chamber. The separated water collects in an annulus between a 1-1/4-inch pipe and a capacitance probe. The capacitance probe senses when the collection chamber is "full" and actuates a solenoid valve. The solenoid valve allows the collected water to drain. Simultaneous with the actuation of the solenoid valve a mechanical relay is closed. Closing the relay triggers a 3 volt pulse (adjustable from 1 to 5 volts) which is sent to an electronic counter. A second relay actuates a mechanical counter on the instrument. This mechanical count is a running total of the number of times the solenoid valve has opened and drained water.

A system capable of generating signals for computer monitoring and/or automatic alarming requires an electronic counter as an accessory for the leak detector instrument. A Himmelstein System 6 Pulse Measurement System® was tested with this first prototype leak detector.

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The System 6 is configured to monitor total counts as one channel of information and timed counts as a second channel for each leak detector. (The chosen instrument is easily expandable to monitor three leak detectors). Both channels get input from the 3 volt pulse triggered by opening the solenoid valve. The Total channel keeps track of total counts since the last manual reset or the last power failure. The timed counts is the number of solenoid valve openings which occurred in a pre-set time interval. Only the Total channel generates a 4-20 ma signal proportional to the number of counts. Both channels can generate alarms as desired by the user.

Once the leak detector is calibrated, the user will know how much water is collected and then dumped when the solenoid valve opens. He will also know the flow of hydrocarbon through the leak detector during operation. The System 6 counts the number of dumps. With this information, the user can estimate the size of a leak to at least an order of magnitude. The complete package is not designed to give accurate quantitative information. Its usefulness is in detecting changes from normal operating conditions.

IV.A.2. Design Details

Items in this section refer mainly to the leak detector itself and are qualitative in nature. Detailed features of the Himmelstein System 6 are described in the manufacturer's manuals. An operating manual (1) gives detailed dimensions, calibration procedures and controller settings for the leak detector module.

A schematic flow diagram for the instrument is shown in Figure 8.

Figure 8. Water Leak Detector Flow Diagram

When necessary, the hydrocarbon stream is first cooled to liquefy it. (Some streams which are liquid already, do not pass through a cooler). The liquid mixture is fed to an expansion chamber where water separates by gravity. A thermocouple at the entrance to the expansion chamber monitors temperature there. Good operating practice says this temperature should be kept fairly constant. Controlling cooling water flow rates is the easiest way to control this temperature. A pressure gauge at the inlet to the instrument reads system pressure for the instrument. Knowing temperature and pressure of the system, one can determine if the hydrocarbon is liquid or not.

Flow through the instrument is controlled using manual valves. An armored style rotameter with a magnetic follower is the flow indicator. Flow through the system is not critical but certain ranges are optimal. (Flows greater than 0.2 gpm hydrocarbon but less than 0.7 gpm work quite well). Higher flow rates (>1 gpm hydrocarbon) result in reduced separator efficiency. Low flows allow water to collect in the piping and result in a slugging effect at the collector. This complicates interpretation of the information from the instrument.

Hydrocarbon which is sampled from the plant is returned and re-used in the plant. It is not vented or sent to a sump. Collected water can be combined with the exit hydrocarbon or sent to a sump. The instrument is not meant to be a clean-up device, so no special disposal procedures were developed for the water.

IV.A.3. Water Detection and Measurement

The instrument detects water because it is a much better electrical conductor than liquid hydrocarbon. Water which is separated from the hydrocarbon collects in an annulus formed by a capacitance probe inside a pipe. The capacitance probe shows readings between 0 and 100% as the annulus fills with water. The 100% and 0% points are arbitrary and user adjustable at the universal transmitter.

The Setcon controller determines the volume of liquid which is dumped when the solenoid valve is opened. The dumped volume is always less than or at most equal to the collected volume. The "open" and "close" settings for the solenoid valve are set at some percentage value associated with the scale set by the universal transmitter. As an example one could choose to dump when the chamber was 75% full then start refilling when the annulus was still 10% full. Choosing values other than 0 and 100% alleviates problems associated with overshoots if the system is dumping and filling rapidly. When the system reaches its "dump" set point, the solenoid valve opens. This advances a mechanical counter 1/2 count. Simultaneously it actuates a relay which closes a circuit to send a 3 volt pulse to an electronic counter. When the water has emptied to the "fill" set point, the solenoid valve closes and the mechanical counter advances another 1/2 count. This feature of moving 1/2 count at actuation and a second 1/2 count at de-actuation is useful during debugging to tell where the system is in its cycle.

IV.A.4. Himmelstein System 6

This commercially available electronic counter was purchased to provide interface information for a process computer and operating alarms. The System 6 accepts a 1 to 5 volt d.c. pulse input and sends out a 4-20 ma signal proportional to the total number of pulses it receives. The pulse input is generated by closing of a mechanical relay whenever the solenoid drain valve opens. This mechanical closure can generate enough electrical noise to cause multiple counts. A simple resistance-capacitance filter is installed on the inlet signal line to minimize the noise problem. The 3 volt signal recommended for this configuration is generated by a solid state regulated power supply which is powered continuously. Only its 3-volt output is turned on and off. Early attempts to cut a.c. power on and off to the power supply resulted in high noise levels and multiple counts even with an electrical filter.

The leak detector as connected to the System 6 uses two totalizers, a timer, a dual digit limit and a D/A converter. One totalizer counts total number of dumps from the previous manually re-set time. An alarm can be set off this totalizer. A second totalizer monitors number of dumps in a user selected time interval. It also has an associated alarm. The alarm settings and time interval are user selectable.

Total counts give indications of long term leakage rates. The timed counts track transients which might occur during start-up and shut-down of a plant.

If the hydrocarbon does get contaminated during a shut down, this leak detection system can be used to monitor effectiveness of any clean-up procedures too. As the system gets cleaner, the number of counts per day will decrease to some steady-state point (hopefully zero).

IV.A.5. Solids Filtration System

The leak detector contains a filter housing through which hydrocarbon can be routed. The housing is pressure rated and uses a single filter 47 mm in diameter. A 0.5 micron filter is recommended for most applications. A separate flow meter is located in the filter loop so hydrocarbon rates through the filter can be set.

Normally the filter is not used. It has been added to the system so solids samples can be collected when desired.

IV.B. PERFORMANCE

IV.B.1. Efficiency of Separating Entrained Water

In most cases the pressure differential between the hydrocarbon working fluid and the aqueous streams (high temperature heat source and low temperature heat sink) will be such that heat exchanger leaks would be characterized by leaks out of the hydrocarbon system, rather than leaks into the system. However, under some operational modes, such as a start-up situation, the pressure differential can be reversed and may lead to water inleakage.

Laboratory Simulation

In order to verify the concept of using a density separation of two phases, a laboratory mock-up of the phase separator was constructed and tested. A high density oil injected into a flowing stream of water was used to simulate leakage into the Rankine cycle working fluid. This maintained the same density differential that would be present in the actual plant situation although both phases were more dense in absolute terms. The laboratory tests showed excellent recovery and separation (Figures 9 and 10).

Field Testing

Tests of the leak detector designed for field use, however, showed significantly poorer performance in actual plant use. In this instance known volumes of water were injected into the liquid isobutane working fluid over a period of time in order to simulate in-leakage of water.

The testing of this instrument in the field was done under two different, but complimentary philosophies regarding its final use. The initial testing was done with the interpretation that the detector needed to be able to detect leaks as quickly as possible (indicated by the number of dumps which would show up in the control room) since the water inleakage may be only transitory during startup. This implied that maximizing the rate of water separation (measured as volume/time) was the way to establish optimum instrument operational settings. However this required extensive testing at a range of flow rates and water contents and proved impractical. The testing was completed with the philosophy that the instrument was needed to detect leaks as small as possible rather

CUMULATIVE SEPARATOR RECOVERY

Figure 9. Laboratory Results on Oil/Water Mixture in Mock-Up Separator

LABORATORY PERFORMANCE FOR SEPARATING WATER/HYDROCARBON MIXTURES

Figure 10. Laboratory Performance Using Oil/Water Mixture in Mock-Up Separator

than as quickly as possible. This meant the instrument was to maximize the separated water as a fraction of the water brought into the leak detector (measured as separator recovery efficiency, %). This dictated the instrument operate at its lowest flow rate allowing "quiescent" conditions for maximum separation of water.

Figure 11 shows the type of results obtained during early testing of the device; the diminished separation performance compared to the laboratory mock-up tests is evident. In order to minimize turbulence at the water/isobutane interface in the detector two modifications were adopted:

- The operating range of the water well was lowered to physically move the water-butane interface away from the turbulence at the top of the separator.
- The flow was reduced to a minimum to reduce turbulence during the test.

Early tests had shown that the temperature of the isobutane in the instrument had varied with the temperature of the cooling water and had ranged from 78°F to 101°F during a seven week period in the spring as the ambient temperature trended upward. Since there would be a change in the water solubility with temperature, additional care was taken to stabilize the cooled isobutane temperature in the instrument during the final testing.

During the second field test, the unit was calibrated using the set-up shown in Figure 12. The first step was to calibrate the level indicator under ambient

Figure 11. Early Testing Results

Figure 12. Calibration Setup

pressure in air and then under isobutane line pressure. All the calibration numbers are arbitrary and depend on the user selected set points for filling and dumping the collected water. The collection efficiencies, however, are independent of these settings and should be reproducible in another plant.

Figure 12 shows the calibration data which was used in calculating recovery efficiencies to be discussed later on. Water was injected into the leak detector using a funnel when the system was at ambient pressure. A tygon tube sight glass showed liquid levels in the detector as water was added. The liquid level was then related to percent readings on the liquid level indicator (Drexelbrook meter). This ambient check is quick and easy to do so it is a worthwhile standardization check which ought to be performed periodically. However, absolute readings will change once the system is pressurized with isobutane.

A more meaningful calibration can be done with the system on line and isobutane flowing through the leak detector module. This requires a high pressure positive displacement pump with a 2 cc/min capacity. One model is manufactured by Eldex laboratories in Menlo Park, CA. This calibration requires stopping normal water drain lines and dumping into a graduated cylinder. The steps are to first fill the detector with water to its normal dump set point. Then by slowly draining the water into the graduated cylinder and recording the corresponding Drexelbrook readings, one can prepare a plot similar to the calibration shown in Figure 13. Now one can relate the meter readings directly to the amount of water collected. A comparison should be made between the amount of water added and that actually collected. Figure 14

Figure 13. Calibration of Level Indicator for Water Leak Detector

Figure 14. Water Recoveries in Leak Detector

shows that for the unit tested, roughly 60% of the water injected was actually collected in the leak detector. This was true for a wide range of water concentrations above 300 ppm but only for a narrow range of isobutane flows through the detector. Flows of isobutane had to be in the 0.5 to 0.7 gpm range to get consistent results. At higher velocities the recoveries dropped off rapidly. The explanation is separation depends on residence time in the detector chamber. At high isobutane velocities the water has insufficient time to settle and is carried out with the isobutane.

Detection Limit

With an isobutane flow of 0.5 gpm flowing through the leak detector a water concentration of 100 ppm was still detectable. This is close to the solubility limit for water in isobutane at the normal operating temperature of 65 to 70°C for the instrument. This unit will not detect dissolved water.

Calculated Performance

The primary function of the leak detector is to announce the presence of water regardless of the size of a leak. It is an alarm device and not a quantitative instrument.

The order of magnitude of a leak can be calculated if four things are known.

1. Volume of water dumped per cycle..in cm^3 (V)
2. Number of dump cycles per hour (N)
3. Flow of isobutane in the stream being sampled #/hr (I)
4. Recovery efficiency for the separator (e)

The water leak rate in #/hr (L) is given by

$$L = (V*N/454)/I)/e$$

V. MANUAL METHODS FOR DETECTING LEAKS

The foregoing sections discussed the detection of leaks using on-line instruments for continuous monitoring. In some plants the operator may be less concerned and check for leaks on a more infrequent basis. This section describes techniques that the authors have found to be usable.

V.A. GAS BUBBLE ANALYSIS (DETECTS HYDROCARBON LEAKAGE INTO BRINE)

V.A.1. Summary

The most common leak will probably be the leakage of hydrocarbon working fluid into the brine rather than the reverse because of the usual pressure differential between the two sides of the heat exchanger. The gas bubble analysis method works on the basis of hydrocarbon gas being carried into the vapor phase as the pressure is reduced. This bubble is then analyzed for hydrocarbon trace levels using a gas chromatograph. The on-line instrument described earlier evolved from the principles of this method.

V.A.2. Equipment

The following hardware is necessary:

- a sample port
- cooling coils (condenser)
- glass gas sample bulb
- gas chromatograph with a detector especially sensitive to hydrocarbons (flame ionization detector)

Probably any valved access to the brine flow could be used as a sample port, but the highest sensitivity to detecting leaked hydrocarbon must be from a valved port near the top of the flow. Hydrocarbon - if it is present as liquid droplets - is less dense than brine and will rise.

Any cooling coils or condenser will suffice if the brine temperature can be brought down to 20-30°C. Dual cooling coils (Kindle and Woodruff, 1981) have worked well in geothermal service and are those specified in ASTM Standard Specification E947, "Specification for Sampling Single-Phase Geothermal Liquid or Steam for Purposes of Chemical Analysis."

The gas sample bulb should be made of glass or other transparent material so that the gas bubble can be seen. The only other functional requirement is that the gas pocket be accessible via a syringe. A normal septum can accomplish this.

A gas chromatograph will serve as the analysis tool. Of the two common types of gas chromatographs the flame ionization detector is far more sensitive to hydrocarbons than the thermal conductivity detector. Since the purpose of the leak detection procedure is to detect small leaks before they can impact overall plant performance, the flame ionization detector gas chromatograph is recommended.

V.A.3. Procedure

The following steps comprise this method:

1. Add water and ice to the containers surrounding the cooling coils; with a two coil method ice is needed only around the second coil.
2. Start the sample stream flowing by regulating it downstream of the cooling coils to achieve a particular temperature (a temperature in the 20-30°C range is recommended).
3. Attach the gas sample bulb to the sample stream with a flexible hose and let it fill with cooled brine to eliminate all air.

4. Invert the gas sample bulb and let a gas bubble form as the brine is displaced.
5. When the gas bubble fills the gas sample bulb - or after a certain length of time-close the valves and disconnect the gas sample bulb sample line.
6. Use a syringe to withdraw a sample from the gas sample bulb and inject it into the gas chromatograph. Analyze it for the working fluid.
7. Compare the result to previously determined values or a value from brine before the heat exchanger (or suspected leak location). Be aware that if the cooled brine temperature varies from measurement to measurement there will be scatter in the results - even if the hydrocarbon content is unchanged - because of different gas evolution behavior at different temperatures.

V.B. WATER IN HYDROCARBON

V.B.1. Summary

As mentioned in the gas bubble analysis summary section, the most probable leak situation would be hydrocarbon into either brine or cooling water due to the usual operating conditions pressure differential. However, there are brief periods during startup when the normal pressure differential would be reversed leading to possible brine or cooling water intrusion into the hydrocarbon working fluid. A manual method for sampling the hydrocarbon working fluid and analysis of its water content is described below.

V.B.2. Equipment

The sampling apparatus consists of an insertion probe which is inserted into the main isobutane flow through a flange and gate valve arrangement. Downstream from the probe the sampling apparatus consists of 2 valves, a removeable 30 ml sample bomb, flow meter, pressure guage, regulating valve and low pressure return line. For sampling high temperature working fluid, a cooling coil immersed in an ice water bath is incorporated between the probe and the valves.

V.B.3. Procedure

Allow the pressurized hydrocarbon to flow through the sampling apparatus, controlling the flow and back-pressure with the regulating valve. Controlling the flow in this manner insures that the whole sample train contains only liquid phase hydrocarbon. After at least 3 sample bomb volumes of hydrocarbon liquid have flowed through the system, close all valves starting with the most downstream one first and proceed upstream.

Remove sample bomb from system and record its weight while full. Introduce the sample bomb contents into a Karl Fischer aquametry titration vessel containing dry methanol, by means of a 0.015" ID Teflon tube inserted through a rubber septum. Allow the sample bomb contents to bubble through the methanol at such a rate that 20-30 minutes are required to deliver the total sample. Titrate with standardized Karl Fischer reagent according to apparatus manufacturer's recommendations. Reweigh the now empty sample bomb. Calculate the water content of the hydrocarbon working fluid sample from the volume of titrant used, known titer of KF reagent, and weight of sample used.

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