wilks
simple analytical solutions

InfraCal® TOG/TPH Analyzer

Model HATR-T2 and CH

User's Guide

InfraCal is a registered trademark of Wilks Enterprise, Inc.
Copyright 2008 Wilks Enterprise, Inc., East Norwalk, CT

www.WilksIR.com

Rev 4.3, November 2009

Table of Contents

1. InfraCal TOG/TPH Analyzer Overview 4

1.1. Introduction 4
1.2. Basic measurement concept 4
1.3. Analyzer description 4
1.3.1. Front operating panel 5
1.3.2. Back panel 5
1.3.3. Description of the push button controls 6
1.4. Analyzer Features 6
1.4.1. Internal Calibration: 6
1.4.2. External Communication: 6
1.4.3. Recall Function/Averaging Results 6
1.4.4. Printing the Result 6

2. Getting Started 7

2.1. Installation 7
2.1.1. Location 7
2.1.2. Power Requirements 7
2.1.3. Warm up time 7
2.2. Zeroing the Analyzer 7
2.2.1. Establishing Zero 7
2.2.2. Zero Check 8

3. Analyzer Calibration 8

3.1. Data Presentation 8
3.2. Considerations for Calibration Standards 8
3.2.1. Calibration correlated to an Alternate Method 8
3.3. Preparing Calibration Standards 9
3.3.1. Preparing Volumetric Standards 9
3.3.2. Preparing Gravimetric Standards 10
3.4. Calibrating in the Analyzer 11
3.4.1. Selecting the Calibration Mode 11
3.4.2. Setting the evaporation timer 11

Timer Programming 11

Overriding the Timer 12

3.4.3. Collecting calibration data 12
3.4.4. Entering Calibration Data into Edit program 12
3.4.5. User Calibration Mode Procedure: 13
3.4.6. Calibration printing 13

4. Analyzing an Oil Sample 13

4.1. Analyzer Pre-Check 13
4.2. 10 to 1 Extraction Procedure for oil in water 13
4.2.1. Supplies needed for Extraction in water 13
4.2.2. Considerations: 14
4.2.3. Total Oil and Grease (TOG) Extraction from Water for Solvents Heavier than Water (ie: Vertrel MCA) 14
4.2.4. Total Oil and Grease (TOG) Extraction from Water for Solvents Lighter than Water (ie: Hexane) 15
4.2.5. Total Petroleum Hydrocarbon (TPH) Extraction from Water 16
4.3. 1 to 1 Extraction Procedure for oil in soil 16
4.3.1. Supplies needed 16
4.3.2. Total Oil and Grease (TOG) Extraction from Soil 17

4.3.3. Total Petroleum Hydrocarbon (TPH) Extraction from Soil 17
4.4. Dilution Procedures 17
4.4.1. 10:1 dilution 18
4.5. Averaged Results Display 18

5. Detailed Sample Stage Description 18

5.1. IR Model HATR-T2 platform sample stage description 18
5.1.1. IR platform (model HATR-T2) measurement concept 19
5.1.2. IR platform considerations 20
5.2. IR card reader sample stage description 20
5.2.1. Card reader (model CH) measurement concept 21
5.2.2. IR Card reader considerations 22

6. Analyzer Specifications 23

6.1. External power requirements: 23
6.2. Physical: 23
6.3. Environmental: 23
6.4. Electrical 24
6.5. Calibration: 24

7. Analyzer Communications Interface 24

7.1. Abstract 24
7.2. Physical connection 25
7.3. Communications port setup parameters 25
7.4. Operation 25
7.5. Data logging 26
7.6. Remote zero balance control 26
7.7. Remote calibration control 26

8. Service and Customer Support 27

Appendix A: Alternate Data Presentation 28
Appendix B: Correlation to an Alternate method 29
Appendix C: Solvent Options 30

List of Figures

Figure 1 - The InfraCal TOG/TPH Analyzer: Front View of Analyzer 4
Figure 2: The Display and Control Panel 5
Figure 3: The TOG/TPH Analyzer: Rear View 5
Figure 4: The TOG/TPH Analyzer Model HATR-T2 Sample Stage: Front View 19
Figure 5: The TOG/TPH Analyzer Model HATR-T2: Top View 19
Figure 6: Measurement of IR Absorption with an IR Platform 20
Figure 7: Model CH Sample Stage 21
Figure 8: The TOG/TPH Analyzer Model CH: Top View 21
Figure 9: Measurement of IR Absorptions with an IR Card Reader 22
Figure 10: The IR Sample Plate 22

1. InfraCal TOG/TPH Analyzer Overview

1.1. Introduction

The InfraCal TOG/TPH Analyzer is designed to measure solvent extractable material (hydrocarbons or oil and grease) by infrared determination in water or wastewater. Models HATR-T2 and CH are designed to correlate with EPA Method 1664 with hexane extraction and gravimetric determination. The HATR-T2 and CH have an evaporation step to accommodate solvents containing hydrocarbons, such as hexane or Vertral MCA. The HATR-T2 uses a cubic zirconia crystal on which the oil film is deposited and measured by internal reflection. The model CH uses a reusable IR sample plate with a sapphire window on which the oil is deposited and measured by transmission. Hydrocarbons with boiling points lower than the extracting solvent used will be lost in the evaporation process.

A dual detector is used in the TOG/TPH Analyzer to measure hydrocarbon concentrations at 3.4 micrometers (2940cm^{-1}) with a reference at 2.7 micrometers (3700cm^{-1}).

1.2. Basic measurement concept

The InfraCal TOG/TPH Analyzer makes use of the fact that hydrocarbons such as oil and grease can be extracted from water or soil through the use of an appropriate solvent and extraction procedure. The extracted hydrocarbons absorb infrared energy at a specific wavelength and the amount of energy absorbed is proportional to the concentration of oil and grease in the solvent. The analyzer can be calibrated to read out directly in the desired units.

1.3. Analyzer description

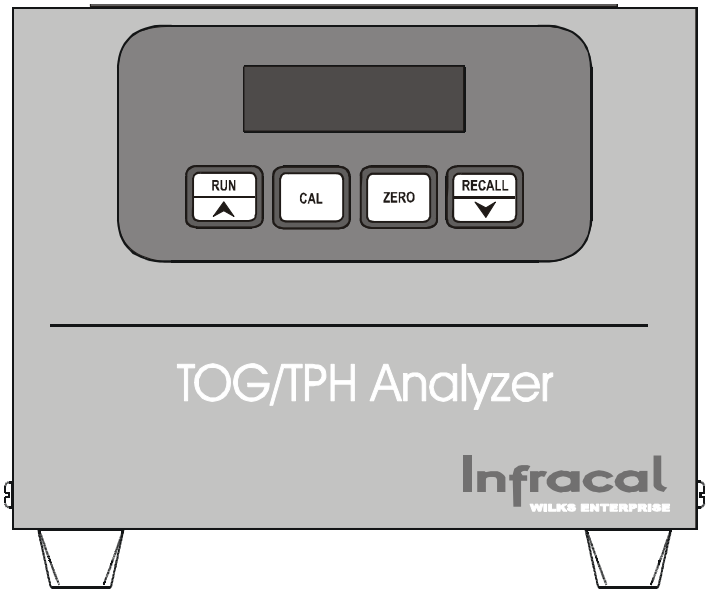

Figure 1: The InfraCal TOG/TPH Analyzer: Front View of Analyzer

1.3.1. Front operating panel

The front panel consists of a 4 digit LED display and four labeled, touch-sensitive push button controls as illustrated in Figure 2. The LED display remains illuminated while the analyzer is plugged in (switched on). When the instrument is on and not in use, the display may either show the result of the last analysis, or it may show ***idLE***.

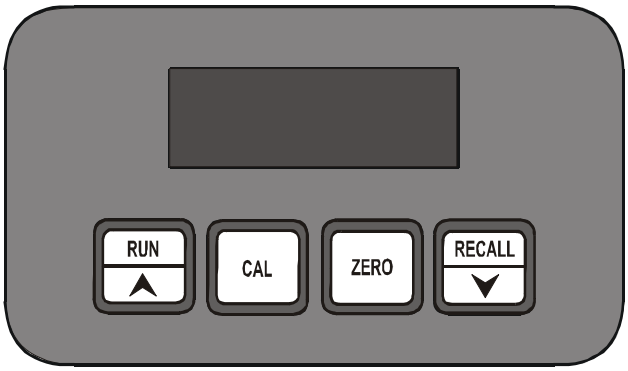

Figure 2: The Display and Control Panel

1.3.2. Back panel

The main power socket for the 12 Volt power supply is located on the back panel. The back panel also provides a standard nine pin, female DB9 connector for serial (RS232-C) data communications with the analyzer. This requires the use of a standard straight through serial data cable. See Appendix C for details of data communications with the TOG/TPH Analyzer. The CAL lockout switch deactivates the front panel CAL button to keep the internal calibration table from being inadvertently changed or turned off. For calibration, the switch is ON (I). After calibration the switch may be moved to the locked position (O). The back panel also contains the CE Mark designation indicating compliance with the codes for operation within the European Community countries, and the analyzer serial number. Make a permanent note of the serial number, and quote this when contacting Wilks Enterprise with a service or warranty related issue.

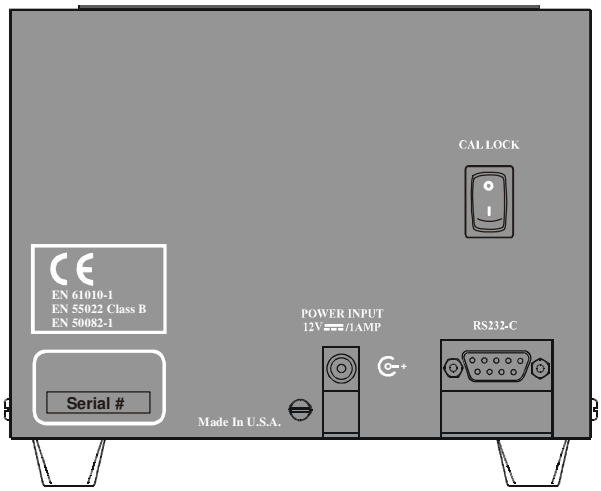

Figure 3: The TOG/TPH Analyzer: Rear view

1.3.3. Description of the push button controls

RUN - initiates sample analysis (Section 4). Also used in CAL to record a calibration sample (Section 3).

UP arrow control - used to increase numerical values used in the CAL function (Section 3).

CAL – Hold for 2 seconds to select calibration type (***uSEr, Edit*** or ***oFF***). Also used to generate a new user calibration. Quickly press and release to print the last result.

ZERO - Hold for 2 seconds to zero balance the instrument (***bAL*** appears on display during operation (Section 2). Also used to exit CAL. Quickly press and release to print the current calibration table.

RECALL - Quickly press and release to recall up to the last ten results (recall mode, section 4.5) or to display the average (averaging mode, section 4.5). Hold for 2 seconds to reset the printer sequence number.

DOWN arrow control - used to decrease numerical values used in the CAL function (Section 3)

1.4. Analyzer Features

1.4.1. Internal Calibration:

The InfraCal TOG/TPH Analyzer reads in relative absorbance units that are proportional to concentration. An internal microprocessor allows the user to enter a calibration in order to read in the desired units. The Analyzer contains three different user selectable calibration modes. These are ***oFF***, ***uSEr*** or ***Edit***. Section 3 explains the calibration functions in detail.

1.4.2. External Communication:

The InfraCal TOG/TPH Analyzer supports communications to a PC, printer or controller via an RS-232C asynchronous serial communications port. This capability allows for collection of sample measurement data and instrument control by a host computer. The RS-232 interface also allows for data transmission into an excel spreadsheet on a PC. It also allows for multiple calibration tables if more than one table is being used with a single instrument. Specification details for communication parameters are in Section 7.

1.4.3. Recall Function/Averaging Results

The analyzer has the ability to store ten results for use with the averaging function or for local recall and display (see Section 4.5).

1.4.4. Printing the Result

An optional printer can be connected to the analyzer through the RS-232C port located on the back. To print the result, momentarily press and release the **CAL** button. Note that the Cal Lock switch must be in the (I) position. The result is printed on one line. The first number printed is a 5-digit sequence number. The sequence number is followed by the result. The remainder of the line contains the date, time and day of the week. To reset the print sequence number, unplug the printer and plug it back in. The next result will print as sequence number 000001.

2. Getting Started

2.1. Installation

2.1.1. Location

The InfraCal TOG/TPH Analyzer may be installed virtually anywhere. It is not affected by vibration and it can operate over a broad range of ambient temperatures (40° F, 4°C to 110°F, 45°C). Model HATR-T2 must be level. A bubble level and leveling feet are provided to level the analyzer.

2.1.2. Power Requirements

The analyzer is powered from a 12 volts d.c. power source. A standard 12 volt power supply is provided with the analyzer, and this may be operated from any grounded a.c. outlet (line power requirements: 100 - 250 VAC, 50-60 Hz, 0.5-0.3 amps). When operating, the TOG/TPH Analyzer consumes approximately 8 watts (0.67 amps).

For field use, the instrument may be connected to other sources of 12 volt d.c. power, such as an external battery pack or the cigarette lighter output of an automobile (contact Wilks Enterprise for details).

Plug in the external 12 volt supply to the power connector at the rear of the instrument. When plugged in, the instrument display will show ***init*** for a short time. Once the power-on initialization is complete, the instrument displays ***idLE***. The TOG/TPH Analyzer is now ready for use.

Note: *the connector is polarized with the center pole positive. Failure to use the correct power supply or the correct cable can result in permanent damage to the analyzer and may invalidate the warranty.*

The InfraCal internal memory, which retains the factory and user calibration tables could be erased from a voltage spike or surge on the 120-220VAC line. The use of a surge protection device between the user's AC line and the 12V DC power supply is recommended to prevent the loss of calibration tables.

2.1.3. Warm up time

For normal operation, it is recommended that the instrument be allowed to warm up for 1 hour prior to use. However, the analyzer is sufficiently stable after 15 minutes, and meaningful measurements may be obtained at this time. If the analyzer is used under the 1 hour warm-up time, frequent checking of the zero is recommended for best results. The longer warm-up time is recommended prior to making critical measurements and when performing analyzer calibration. The TOG/TPH Analyzer draws very little power and, if used daily, it can be left on at all times (unless operated from an external battery pack).

2.2. Zeroing the Analyzer

For initial set-up, establish zero for the analyzer using the following procedure. Once a zero has been established, subsequent zero checks should use the zero check procedure described in section 2.2.2.

2.2.1. Establishing Zero

Ensure that the HATR-T2 trough plate or IR sample plate is clean. Clean the HATR-T2 trough plate or IR sample plate with solvent after each use. To clean the HART-T2 trough plate, add enough solvent to cover the HATR crystal. Wipe slowly across the crystal in one direction with a clean anti-static wipe to dry surface. Rubbing back and forth on the crystal tends to spread any residual oil rather than removing it. If the previous sample had high TOG or TPH levels, this procedure may need to be repeated two or three times to remove hydrocarbon residues.

Check zero (see section 2.2.2).

- For Model CH, place the clean IR sample plate in the sample stage.
- Press and hold the **ZERO** button until the display reads ***bAL***. Release the button. A multiplier value to 3 decimal places will be displayed when zero is established. The actual value is only of interest when reporting problems to the factory.
- Press **RUN**. The display should read 00 ± 02. If not, repeat the zero process.

2.2.2. Zero Check

The zero value is retained in permanent memory and is restored each time the instrument is powered up. It is recommended that the zero be checked and (if necessary) reestablished, on a daily basis.

- To check the zero value, press the **RUN** button. (For Model CH, first place the clean IR sample plate in the sample stage).
- If the result is not ± 02, re-clean the trough plate or IR sample card.
- Check zero again.
- If the result is not ± 02, reestablished the zero as described in 2.2.1

---

3. Analyzer Calibration

3.1. Data Presentation

The standard display format for the TOG/TPH Analyzer is relative absorbance (***AbS***). This is the format the analyzer is preset for unless the customer specified otherwise. Other formats are available, and these may be set for specific applications. Details for other formats are in Appendix A – Alternate Data Presentation Modes.

3.2. Considerations for Calibration Standards

There are five choices for calibration.

1. Prepare your own standards. Standard preparation is described in detail in section 3.3.
2. Purchase pre-prepared standards from Wilks Enterprise.
3. Calibrate to an alternate laboratory method.
4. Non-certified factory calibration. Wilks Enterprise can provide the InfraCal TOG/TPH Analyzer with a non-certified 5-point factory calibration using either Freon or Hexane calibration standards
5. Certified laboratory calibration. Wilks Enterprise can recommend a certified laboratory for analyzer calibration.

3.2.1. Calibration correlated to an Alternate Method

Each type of oil and grease analysis sees different physical properties and will sometimes give different results from each other. The InfraCal TOG/TPH Analyzer can be calibrated against an alternate method rather than with prepared standards. For calibration against an alternate method obtain duplicate samples or if possible, test the same sample with the InfraCal and the alternate method. Data collected for this

purpose should be obtained with the calibration in the ***oFF*** mode. With a minimum of 10 data points, make a graph with InfraCal relative absorbance data vs. the alternate method values. Select 3 to 5 data points within the desired measurement range of operation and enter these calibration points into the InfraCal memory using the **Edit** program that is described in section 3.6.

Note: *Data can be collected with the calibration from standards programmed into the* ***Edit*** *program with the edit program on. Refer to Appendix B for the calculations needed to correlate the alternate method values to the calibrated InfraCal TOG/TPH values for new values to enter into the Edit program.*

3.3. Preparing Calibration Standards

Standards can be made gravimetrically (mg/L for water, mg/kg for soil) or volumetrically (ppm). Customers can prepare their own standards using a non-volatile (heavy weight) oil such as "3-in-1" or an actual oil sample. Calibration standards should cover the desired range for the analysis. An ideal calibration set contains three to five samples. A maximum of 20 samples can be used.

The calibration curve for oil and grease is typically linear up to 3000 ppm (300 ppm for oil in water with a 10:1 extraction ratio). Above 4000 ppm (or 400 ppm for oil in water samples) the curve flattens out. Because the InfraCal TOG/TPH Analyzer performs a point-to-point calibration, samples above the linear range will be accurate up to the highest calibration standard. It is best to calibrate to 4000 ppm (400ppm for 10:1 extraction ratios) and dilute the extract for samples above this range. See section 4.4 for dilution procedures. The lowest concentration standard should measure at least 10 on the InfraCal in the ***oFF*** mode for calibration. All successive samples should be spaced apart by at least 10 absorption units.

3.3.1. Preparing Volumetric Standards

Supplies needed for volumetric calibration

125-ml Teflon wash bottle

10-ml graduated cylinder

40-ml vials with Teflon lined caps (at least 3 for holding standards)

100 microliter syringe

Solvent (see Appendix C for solvent options)

Calibration oil

Below is a chart for mixing volumetric standards. For water analysis with an extraction ratio of 10:1, the oil and grease is concentrated 10 times in the solvent. The actual value of the standard is divided by 10 in order to match the concentrated value of the extract. The water analysis column below makes this correction. For standards below 1000 ppm (100 ppm for water samples), it is best to mix a concentrated stock solution to be diluted for lower standards.

For example, the desired range for a particular analysis is typically 10 ppm with values up to 100 ppm in water.

1. Prepare a stock solution for 100 ppm for water analysis. (10 ul oil in 10 ml of solvent).
2. Pour half (5 ml) into a 10-ml graduated cylinder.
3. Fill with solvent up to 10 ml. This will cut the 100 ppm standard in half to be 50 ppm.
4. Repeat this again with the 50 ppm standard for a 25 ppm standard.

This makes three standards (100 ppm, 50 ppm and 25 ppm) within the operating range of 10 – 100 ppm.

Actual ppm (1:1) Extraction (soil analysis)	Corrected ppm for 10:1 extraction ratio (water analysis)	Oil	Solvent
4000	400	40ul	10 ml
3000	300	30ul	10 ml
2000	200	20ul	10 ml
1000	100	10 ul	10 ml

Note: *To convert ppm to mg/l, multiple by the specific gravity of the oil used for the calibration standard (for 3-in-1 Oil or 30W motor oil, use 0.865.) by the ppm value to get a mg/l value*

3.3.2. Preparing Gravimetric Standards

Supplies needed for gravimetric calibration

125-ml Teflon wash bottle

10-ml graduated cylinder

10-ml volumetric

40-ml vials with Teflon lined caps (at least 3 for holding standards)

Solvent (see Appendix C for solvent options)

Calibration oil

Analytical Balance that reads to .001 gram

Prepare a stock solution and make the appropriate dilutions to cover analysis range. For water analysis with an extraction ratio of 10:1, the oil and grease is concentrated 10 times in the solvent**.** *The actual value of the standard is divided by 10 in order to match the concentrated value of the extract.*

1. Weigh about 0.1 gram of oil in a 10-ml volumetric flask.
2. Record the exact weight.
3. Fill with solvent up to the 10 ml line.

Calculate the exact concentrations as shown below:
Oil weight x 10,000 = mg/l (multiply grams x 1000 to equal mg and
divide 10 ml by 100 to equal liters = 0.1. 1000 divided by 0.1 = 10,000)

For an oil weight of 0.110 gram in 10 ml, the Stock solution will be:
0.110g x 10,000 = 1,100 mg/l

Using the above hypothetical stock solution, an example of dilutions for the desired range for a particular analysis that is typically 10 mg/l with values up to 100 mg/l in water could be as follows.

Stock solution 1,100 mg/l divided by 10 (for a 10:1 extraction in water) = 110 mg/l

1. Pour half of the stock solution (5 ml) into a 10 ml graduated cylinder.
2. Fill with solvent up to 10 ml. This will cut the 110 mg/l standard in half to be 55 mg/l.
3. Repeat this again with the 55 mg/l standard for a 27.5 mg/l standard.

This makes three standards (110 mg/l, 55 mg/l and 27.5 mg/l) covering the operating range of 10 – 100 mg/l for oil in water analysis.

3.4. Calibrating in the Analyzer

The Analyzer contains three different user modes in the calibration. These are ***uSEr, Edit*** **or** ***oFF.*** If a factory calibration is installed there will be a fourth mode, ***FACt***.

In the ***oFF*** mode the instrument measures levels in arbitrary absorption units that are proportional to concentration levels. Higher values indicate increased levels of hydrocarbons. This mode should be used to collect "raw" data for the ***Edit*** mode or for comparing to an alternate method.

The ***Edit*** mode allows the user to edit an existing calibration table or to create one from scratch, using absorption values obtained in the ***oFF*** mode. In this mode, standards (or samples used to calibrate to an alternate laboratory method) may be run several times and an average can be entered into the table.

The ***uSEr*** mode enters the calibration at the time the standards are presented to the analyzer.

3.4.1. Selecting the Calibration Mode

- Press the **CAL** button for two seconds, until ***CAL*** appears on the display. Press the **RECALL** button to display the active table, one of ***uSEr, Edit*** or ***oFF***. Press and release the **RECALL** button repeatedly until the desired mode is displayed.
- Press the **ZERO** button to exit the calibration mode. ***idLE*** will be displayed.

3.4.2. Setting the evaporation timer

Overview

The InfraCal TOG/TPH Analyzer contains a built in, user programmable, evaporation timer intended for use with model HATR-T2. For model CH, the evaporation time can be measured using the timer, although the IR plate needs to be manually placed into the holder for the ***run*** time.

Timer Programming

The CAL lockout switch must be in the on (I) position to set the timer (see figure 3, section 1.3.2). Press and hold the **RUN** button until the current timer value is displayed. The value is displayed as 1 or 2 digits in minutes and 2 digits in seconds, separated by a period (.). Release the **RUN** button once the current value (initially 0.00) is displayed. Use the up-arrow and down-arrow keys to scroll the timer to the desired value. Five minutes (5.00) is recommended. The optimum time will vary with the type of oil and atmospheric conditions at the point of use. To zero the timer during programming, press the **ZERO** button. Once the desired time has been programmed press the **CAL** button. The display will read ***idLE***.

Timer Operation

The timer is disabled when programmed to zero (0.00). When the timer is non-zero, it is invoked during the normal **RUN**, **ZERO** and **CAL** functions.

Press and release **RUN** and the timer value is displayed. The timer will count down one second at a time. The dot separating minutes and seconds flashes to indicate the timer is counting. Once the timer reaches zero the display will read ***run*** during the sample measurement cycle. The result is displayed on completion.

The **ZERO** function is initiated by pressing and holding the **ZERO** button until the timer value is displayed. The timer will count down as described above and ***bAL*** will then be displayed. On completion the balance result is displayed.

The timer is also invoked during calibration, each time the **RUN** button is pressed to analyze a sample.

Overriding the Timer

For a zero check or to do a second reading on an evaporated sample, the timer may be overridden. To override the timer, press and release the **RUN** button twice and the analyzer will go directly into the measurement cycle.

3.4.3. Collecting calibration data

Calibration data can be collected from standards (section 3.3) or from actual samples compared to an alternate oil and grease measurement method (section 3.2.2). If the data is to come from actual samples, skip to section 4 for extraction procedures.

1. Allow the analyzer to warm up at least one hour.
2. Always zero the Analyzer prior to calibration or collecting data for calibration analysis (see section 2.2).
3. Make sure the calibration is in the ***oFF*** mode (section 3.4.1).
4. Set the timer for five minutes (3.4.2).
5. Eject 50 microliters of standard using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR sample plate into sample stage and press run. (Press twice if the timer is preset to override the timer).
6. Record the result on the table below (AO1). Record the standard value in CO1. Standards may be run several times and an average can be entered on the table below.
7. Repeat for the remaining standards.

Note: *The results can plotted graphically as a calibration curve. The resulting plot can be used to prepare a reference chart for users who prefer not to use the analyzer's internal calibration or for data points to be edited into calibration.*

Relative absorbance Versus Calibration Standard or Alternate Method Table

(Relative absorbance Value)		(Calibration Standard in Desired Units)	
A01 =	_____	C01=	_____
A02 =	_____	C02 =	_____
A03 =	_____	C03 =	_____
A04 =	_____	C04 =	_____
A05 =	_____	C05 =	_____
↓		↓	
A20 =	_____	C20 =	_____

N (Number of calibration points or calibration standards) = _____

3.4.4. Entering Calibration Data into Edit program

1. Press the **CAL** button for two seconds, until ***CAL*** appears on the display. Press **RECALL** until ***Edit*** is displayed.

2. Press **CAL**. The display will read ***n*=** for a short time, followed by the number of entries currently in the calibration table. Scroll to the desired number of entries (0 - 20). *Selecting 0 will erase any existing calibration table.*

3. Press **CAL** to proceed. The display will read ***A01*=** for a short time followed by the current absorption value for the first calibration table entry. Scroll this to the desired value from the "relative absorbance versus calibration standard" table.

4. Press **CAL** again and the display will read ***C01*=** followed by the current analyzer concentration value for the entry. Scroll this to the desired value from the table. Continue to press **CAL** to step through all absorption and concentration values for the table size (***n*=**) entered. Once all entries have been created press **CAL** and the display will read ***idLE***.

3.4.5. User Calibration Mode Procedure:

Note that this is not the preferred method to use for calibration of the InfraCal Analyzer. If you would like to use this method, please contact tech@wilksir.com for instructions.

3.4.6. Calibration printing

With the optional printer, the current calibration table can be printed by momentarily pressing and releasing the **ZERO** button when the analyzer is idle. The first line indicates which calibration is active followed by the date and time. The second line gives the headings for the calibration table that follows. **ABS** represents absorption and **CON** represents concentration. The table headings are followed by the balance value. One additional line is printed for each calibration table entry. The absorption and concentration values are given.

---

4. Analyzing an Oil Sample

These extraction procedures are a simplified version of ASTM and EPA methods. The ASTM or EPA extraction methods may be used if desired.

4.1. Analyzer Pre-Check

1. For calibration, allow the analyzer to warm-up and stabilize for one hour

2. Ensure that the HATR-T2 trough plate or IR sample plate is clean. (section 2.2.1) Use clean solvent to rinse the HATR-T2 trough plate or IR sample plate after each use (see section 5.1.3 for additional cleaning options).

3. Perform a zero check (section 2.2.2)

4. Make sure glassware and sample containers are clean.

4.2. 10 to 1 Extraction Procedure for oil in water

4.2.1. Supplies needed for Extraction in water

125-ml Teflon wash bottle

100-ml stoppered graduated cylinder or Sample bottle graduated in mL (ie: 125-ml graduated bottles)

125-ml stopper separatory funnel or sample bottles with septa caps (for solvents heavier than water, ie: Vertrel MCA)

Hydrochloric (HCl) or sulfuric acid (H_2SO_4)(dilute with water 1:1)

pH indicator strips or pH meter

10-ml or 25-ml Graduated cylinder (depending on sample size)

Sodium sulfate (Na_2SO_4), ACS, granular anhydrous (For TOG only, not necessary for TPH readings)

Glass funnel

Whatman 40 filter paper, 11cm, or equivalent

Silica gel (for TPH), anhydrous, 75-150 micrometers

Disposable polyethylene pipette or equivalent

100-microliter syringe or pipette

5-mL syringe (for use with prescription bottles)

Solvent (see Appendix C for solvent options)

4.2.2. Considerations:

- Make sure glassware for used for analysis is clean. Any residual hydrocarbons in the glassware and sampling containers will be extracted and added to the TOG or TPH reading. To check the glassware, rinse with solvent then test the solvent rinse by ejecting 50 microliters onto the HATR-T2 trough plate or IR sample plate. A reading above 04 shows contaminated glassware.
- Oil and grease tends to adhere to the surfaces it comes in contact with. Use the entire sample collected. Either mix the solvent and sample in the sample collection container or rinse the sample collection container with a portion of the solvent to be used for extraction.
- The mechanical details of the extractions are a little different depending on whether the solvent is lighter than water (hexane) or is heavier than water (Vertrel MCA) but in all other respects the methods are identical.

NOTE: *Any hydrocarbons in the sample that have a lower boiling point than the extracting solvent will be lost in the evaporation process.*

4.2.3. Total Oil and Grease (TOG) Extraction from Water for Solvents Heavier than Water (ie: Vertrel MCA)

1. Pour measured sample into separatory funnel. Rinse the sample collection container and graduated cylinder with a portion of the solvent to be used for extraction. If using a graduated bottle with a septa cap, solvent can be mixed directly in the bottle without using the separatory funnel.
2. Adjust the pH to less than 2 with Hydrochloric acid or Sulfuric Acid (typically 3-5 drops depending on buffers in sample).

3. Add one tenth of the sample size of solvent to the sample collection container to rinse interior surfaces and cap. (With the 177-ml prescription bottle, it is convenient to collect 140 ml of sample and add 14 ml of solvent). Pour this solvent into separatory funnel containing sample.
4. Shake the separatory funnel or prescription bottle vigorously for 2 minutes with periodic venting to release excess pressure.
5. Allow the phases to separate.
6. Place a filter paper in a filter funnel and add approximately 1 gram (1Tablespoon) of sodium sulfate.
7. Drain the solvent (lower) layer from the separatory funnel through the sodium sulfate into a clean container (10-mL graduated cylinder can be used). With the prescription bottle, invert the bottle so that the solvent layer fills the neck. Using a 5 ml syringe withdraw 4-5 mL of the solvent layer and deliver through the sodium sulfate into a clean container.

Note: *Use of the sodium sulfate is necessary to prevent water from interfering with the analysis. With totally hydrophobic solvents, this step may be skipped. It is not necessary to collect all of the solvent but it is necessary to preclude water to prevent caking of the sodium sulfate.*

8. Eject 50 microliters of solvent extract using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR sample plate into sample stage and press run. (Press twice if the timer is preset to override the timer).

4.2.4. Total Oil and Grease (TOG) Extraction from Water for Solvents Lighter than Water (ie: Hexane)

1. Pour sample into a stopper graduated cylinder and measure. Rinse the sample collection container with a portion of the solvent to be used for extraction. If using a graduated bottle solvent can be mixed directly in the bottle. Adjust the pH to less than 2 with Hydrochloric acid or Sulfuric Acid (typically 3-5 drops depending on buffers in sample).
2. Add one tenth of the sample size of solvent to the sample collection container to rinse interior surfaces and cap. (With the 125-ml graduated bottle, it is convenient to collect 100 ml of sample and add 10 ml of solvent). Pour this solvent into graduated cylinder containing sample.
3. Shake the graduated cylinder or graduated bottle vigorously for 2 minutes with periodic venting to release excess pressure.
4. Allow the phases to separate.
5. Place a filter paper in a filter funnel and add approximately 1 gram (1Tablespoon) of sodium sulfate.
6. Using a disposable polyethylene pipette withdraw 4-5 mL of the solvent layer and deliver through the sodium sulfate into a clean container.

Note: *Use of the sodium sulfate is necessary to prevent water from interfering with the analysis. With totally hydrophobic solvents, this step may be skipped. It is not necessary to collect all of the solvent but it is necessary to preclude water to prevent caking of the sodium sulfate.*

7. Eject 50 microliters of solvent extract using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR

sample plate into sample stage and press run. (Press twice if the timer is preset to override the timer).

8. If the result is above the calibration range, see section 4.4 for dilution procedure.

4.2.5. Total Petroleum Hydrocarbon (TPH) Extraction from Water

The difference between TPH (Total Petroleum Hydrocarbon) and TOG (Total Oil and Grease) is the polar organics are removed from the extract using silica gel. The remaining hydrocarbons are the non-polar components considered to be TPH.

1. Follow the above procedure steps 1-5 for solvents heavier than water and 1-4 for solvents lighter than water. (The Sodium Sulfate procedure is not necessary as the silica gel will remove any water in the solvent.)
2. Place a filter paper in a filter funnel and add approximately 1 teaspoon of silica gel.
3. Pour extract from container though the silica gel into a clean container.
4. Eject 50 microliters of solvent extract using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR sample plate into sample stage and press run. (Press twice if the timer is pre-set to override the timer).
5. If the result is above the calibration range, see section 4.4 for dilution procedure.

4.3. 1 to 1 Extraction Procedure for oil in soil

4.3.1. Supplies needed

40-mL vials with Teflon-faced caps

10-mL or 25-mL Graduated cylinder (depending on sample size)

Sodium sulfate (Na_2SO_4), ACS, granular anhydrous (For TOG Only, not necessary for TPH readings)

Glass funnel (for TPH)

Whatman 40 filter paper, 11cm, or equivalent (for TPH)

Silica gel (for TPH), anhydrous, 75-150 micrometers

50-microliter syringe or pipette

Disposable polyethylene disposable pipette or equivalent

Plastic air syringe with filter frit and plunger (or equivalent)

Sample spatula

Solvent (see Appendix C for solvent options)

Analytical balance that reads to .1 gram

4.3.2. Total Oil and Grease (TOG) Extraction from Soil

1. Collect a soil sample directly in a washed and weighed (to the nearest 0.1 gram) EPA/VOA 40 ml vial. The sample should be about ¾ of the volume of the vial.
2. Weigh the sample to the nearest 0.1 gram, subtracting the tare weight of the vial.
3. If the sample is wet and clumpy, add up to 5 grams of sodium sulfate. Use the spatula to break up the clumps.
4. Add the same amount of solvent in mililiters as the soil sample weight in grams (do not include the weight of the sodium sulfate). ie: for 11.2 grams of soil, add 11.2 ml of solvent. This will give a 1:1 extraction ratio.
5. Cap the vial with the Teflon side of the liner toward the sample. Shake vigorously for 2 minutes.
6. Pour the solvent into the plastic air syringe with filter frit, leaving as much of the soil in the vial as possible.
7. Place the plunger into the air syringe force the solvent through the filter frit into a clean container.
8. Eject 50 microliters of solvent extract using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR sample plate into sample stage and press run. (Press twice if the timer is preset to override the timer).
9. If the result is above the calibration range, see section 4.4 for dilution procedure.

4.3.3. Total Petroleum Hydrocarbon (TPH) Extraction from Soil

The difference between TPH (Total Petroleum Hydrocarbon) and TOG (Total Oil and Grease) is the polar organics are removed from the extract using silica gel. The remaining hydrocarbons are the non-polar components considered to be TPH.

1. Follow the above procedure steps 1-6. (For Step 3, Silica Gel may be used in place of the sodium sulfate.)
2. Place a filter paper in a filter funnel and add approximately 1 teaspoon of silica gel.
3. Pour extract from container though the silica gel into a clean container.
4. Eject 50 microliters of solvent extract using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR sample plate into sample stage and press run. (Press twice if the timer is preset to override the timer).
5. If the result is above the calibration range, see section 4.4 for dilution procedure.

4.4. Dilution Procedures

If the sample reading is above the highest calibration point, a dilution must be performed to bring it into the measurement range.

4.4.1. 10:1 dilution

1. Pour 1 ml of solvent extract into a 10-mL graduated cylinder
2. Add 9 mL of solvent for a 10 to 1 dilution.
3. Mix and eject 50 microliters of solvent extract using a pipette or syringe onto the center of the HATR-T2 plate and press **RUN**. For the CH Model remove the sample plate and lay on a flat surface. Eject the standard from the pipette or syringe onto the center of the plate and allow the solvent to evaporate. (The evaporation time can be measured using the programmable timer.) Insert the IR sample plate into sample stage and press run. (Press twice if the timer is preset to override the timer).
4. Add a zero to the result on the InfraCal's digital display and record your reading, i.e. if the result is 465, the extract value after dilution is 4650 ppm.
5. This procedure may be repeated if the extract is still not within the calibration range. Add two zero's to the InfraCal's digital display if two dilutions are performed.

4.5. Averaged Results Display

The InfraCal Analyzer can display the average of up to ten sample measurements. To use the averaging mode, use the following procedure:

- Momentarily press the **RECALL** button once and ignore the result displayed.
- Analyze up to ten replicate samples using the measurement procedure described above.
- Momentarily press the **RECALL** button to display the average.

The next sample measurement will then start a new average accumulation.

The Analyzer alternatively can be configured to recall the last 10 measurements (from newest to oldest) in a circular fashion. First the TOG/TPH Analyzer must be switched from the average mode (factory default) to the recall mode as described below. Once the recall mode is selected momentarily press the **RECALL** button repeatedly to display the previous results.

The TOG/TPH Analyzer recall mode can be switched by pressing the ZERO button first, immediately followed by the RECALL button and holding both buttons for two seconds. The display will read *rCL* when switched to the recall mode. Repeat the procedure to return to average mode. The display will read ***Ag***.

---

5. Detailed Sample Stage Description

5.1. IR Model HATR-T2 platform sample stage description

The InfraCal TOG/TPH Analyzer Model HATR-T2 is supplied with a removable IR Platform sample stage with an integrated optics sensing system. The sample stage includes the infrared source (modulated) and detector system, positioned such that an elliptical energy beam is transmitted through the IR Platform crystal and focused directly on the detector-sensing window.

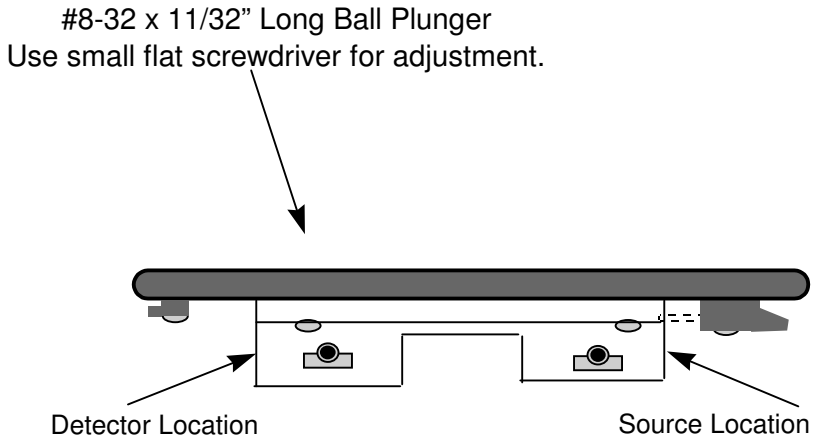

Figure 4: The InfraCal TOG/TPH Analyzer Model HATR-T2 Sample Stage: Side View

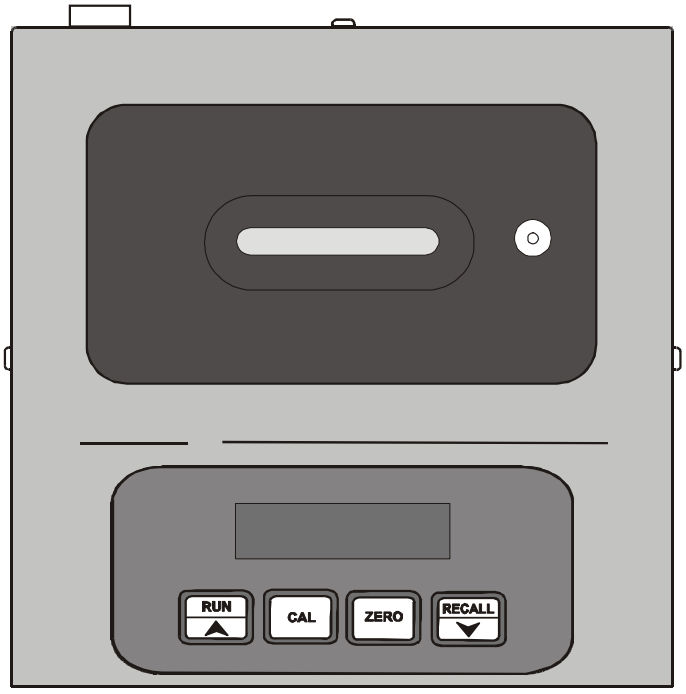

Figure 5: The TOG/TPH Analyzer Model HATR-T2: Top View

5.1.1. IR platform (model HATR-T2) measurement concept

Model HATR-T2 makes use of the fact that hydrocarbons such as oil and grease can be extracted from water or soil through the use of an appropriate solvent. The extracted hydrocarbons absorb infrared energy at a common IR wavelength and the amount of energy absorbed is proportional to the concentration of the oil/grease in the solvent. This can be directly calibrated or converted to the amount of oil in the original sample.

The InfraCal TOG/TPH Analyzer, Model HATR-T2, is designed as an equitable substitute for EPA Method 1664 for hexane extraction and gravimetric determination. A fixed volume of the extract is placed directly in an IR Platform trough with a known path length and the solvent is allowed to evaporate. When the IR Platform is placed on the sample stage an IR beam is internally reflected down the ATR crystal and the output is focused directly on the dual detector package. Since there is an evanescent wave that penetrates the sample at each internal reflection point, energy is absorbed at the analytical wavelength by the sample.

The energy collected at the analytical wavelength (I_A), is reduced when compared to the energy collected at the reference wavelength (I_R). The oil concentration is determined by a calculation of the logarithm of the ratio of the light transmission at the reference wavelength to the light transmission at the analytical wavelength (Beer-Lambert law) as shown in Figure 6. The Beer-Lambert law assumes a linear relationship between absorbance and concentration. Deviations from linearity are determined by obtaining absorbance values from known samples and an internal point-to-point calibration table is prepared (Section 3) so that actual concentration is directly presented on the display. If the concentration ratio used during extraction (typically 10 parts sample to 1 part hexane for liquid samples and 1:1 for soil samples) is taken into account during calibration the display will read directly in the desired units.

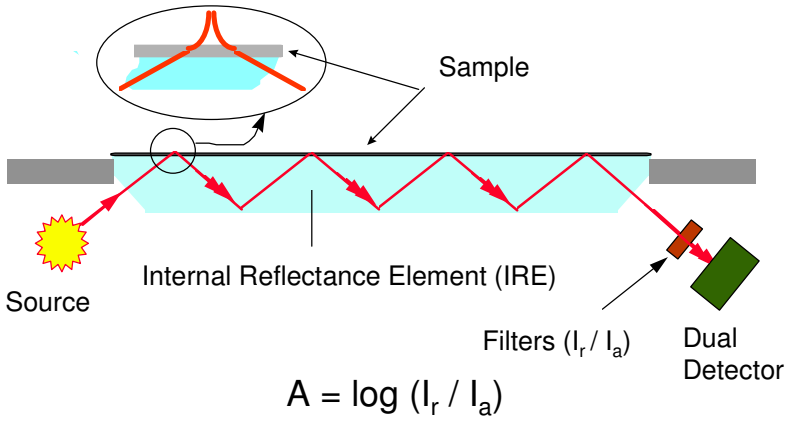

Figure 6: The Measurement of IR Absorption of an Oil Sample with a IR Platform

5.1.2. IR platform considerations

1. Only use IR Platforms supplied by Wilks Enterprise, Inc. made with infrared transmitting cubic zirconia ATR crystals. *Note: Changing IR Platforms may require analyzer re-calibration due to variances in IR Platform crystal characteristics.*
2. For best reproducibility, use precision fixed volume pipettes or syringes.
3. Always clean the IR Platform crystal with HPLC or Spectrophotometric grade hexane or other suitable solvent that leaves no residue.
4. The InfraCal Model HATR-T2 must be placed on a flat surface. The bubble level and leveling feet are provided for this purpose.
5. Repeatability and drift problems occur when the surface becomes contaminated with foreign matter or a charge buildup occurs. These conditions can cause the sample to dry in a non-uniform way or for the oil to migrate over time once the hexane has evaporated. These problems can be corrected as follows:

Clean the trough plate with methyl alcohol. Fill the trough with enough methyl alcohol to cover the crystal. Allow the crystal to soak for 10 seconds. Dry the crystal with an anti-static wipe, or a foam swab intended for use with alcohol. Check the zero value and re-zero if necessary (Section 2.2). This can be done each time a new sample is to be analyzed, immediately prior to sample application.

5.2. IR card reader sample stage description

Model CH is supplied with a removable IR Card Reader sample holder and an integrated optics sensing system. The sample stage includes the infrared source (modulated) and detector system, positioned such that an elliptical energy beam is transmitted through the IR Card Reader crystal and focused directly on the detector-sensing window.

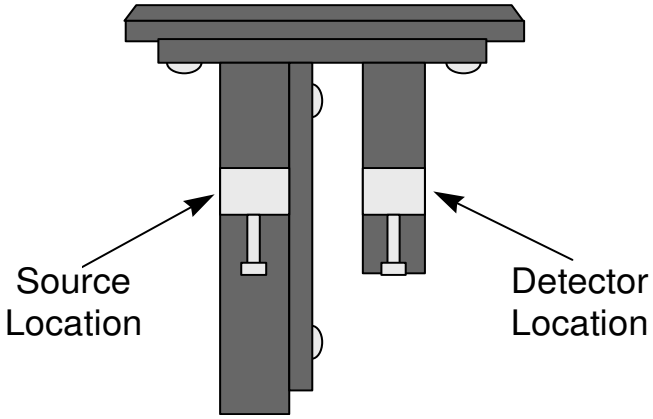

Figure 7: The InfraCal TOG/TPH Analyzer Model CH Sample Stage: Side View

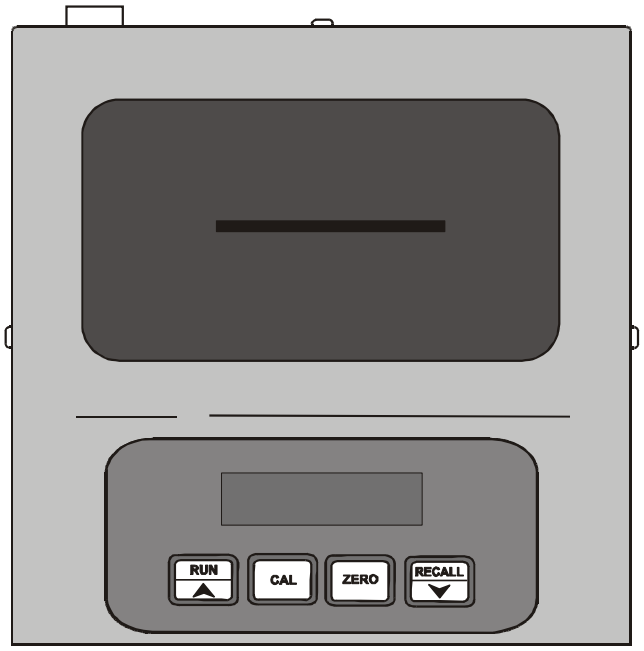

Figure 8: The InfraCal TOG/TPH Analyzer Model CH: Top View

5.2.1. Card reader (model CH) measurement concept

Model CH makes use of the fact that hydrocarbons such as oil and grease can be extracted from water or soil through the use of an appropriate solvent. The extracted hydrocarbons absorb infrared energy at a common IR wavelength and the amount of energy absorbed is proportional to the concentration of the oil/grease in the solvent. This can be directly calibrated or converted to the amount of oil in the original sample if the ratio of solvent to water is carefully controlled.

The InfraCal TOG/TPH Analyzer, Model CH, is designed as an equitable substitute for EPA Method 1664 for hexane extraction and gravimetric determination for higher levels of TOG or TPH. Since hexane is not transparent at the analytical wavelength used for hydrocarbon absorption, a fixed volume of the extract is placed directly in the IR Sample Plate and the hexane is allowed to evaporate. When the IR Sample Plate is placed into the sample stage, IR energy is focused from the source through the sapphire window on to the dual detector package, Figure 9.

The energy collected at the analytical wavelength (I_A), is reduced when compared to the energy collected at the reference wavelength (I_R). The oil concentration is determined by a calculation of the logarithm of the ratio of the light transmission at the reference wavelength to the light transmission at the analytical wavelength (Beer-Lambert law) as shown in Figure 6. The Beer-Lambert law assumes a linear relationship between absorbance and concentration. Deviations from linearity, if any, can be determined by obtaining absorbance values from known samples. This data can be used to prepare an internal point-to-point calibration table (Section 3) so that actual concentration is directly presented on the display. If the concentration ratio used during extraction (typically 10 parts sample to 1 part hexane for liquid samples and 1:1 for soil samples) is taken into account during calibration, the display will read directly in the desired units.

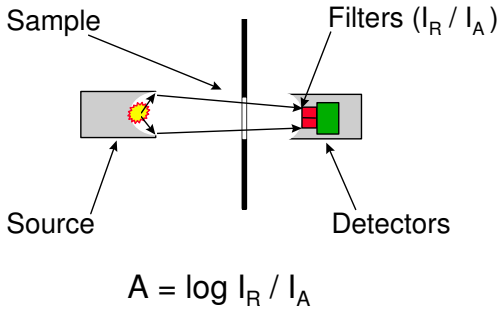

Figure 9: The Measurement of IR Absorption of an Oil Sample with a IR Card Reader

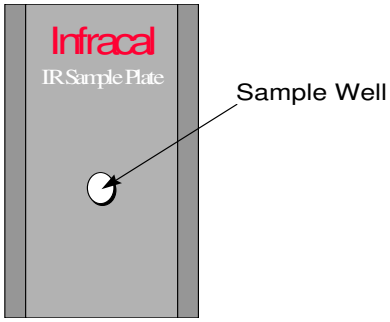

Figure 10: The IR Sample Plate

5.2.2. IR Card reader considerations

1. Only use IR sample plates supplied by Wilks Enterprise, Inc. made with infrared transmitting sapphire windows.
2. For best reproducibility, use precision fixed volume pipettes or syringes.
3. Always clean the IR sample plate window with HPLC or Spectrophotometric grade hexane or a suitable solvent that leaves no residue. Hold the plate vertically, flow a small amount of solvent down the plate and shake to ensure all oil is removed and that the solvent is dry. For best results clean with methyl or ethyl alcohol and dry with an anti-static wipe.
4. Differences in transmission characteristics between plates may introduce quantitative errors when multiple plates are used. In this case, check each clean IR sample plate for zero value after zeroing on one of the plates. If the zero values are different, zero to each plate prior to applying a sample for measurement.
5. Place the IR plate on a flat surface when applying a sample.

6. Repeatability and drift problems occur when the window becomes contaminated with foreign matter or a charge buildup occurs. These conditions can cause the sample to dry in a non-uniform way or for the oil to migrate over time once the hexane has evaporated. These problems can be corrected as follows:

Clean the sapphire plate with a solvent such as methyl alcohol. Remove the sapphire plate from the analyzer and fill the window with enough methyl alcohol to cover the surface. Allow the plate to soak for 10 seconds. Dry the window with an anti-static wipe, cotton ball or a swab intended for use with alcohol. Check the zero value and re-zero if necessary (Section 2.2). This can be done each time a new sample is to be analyzed, immediately prior to sample application.

---

6. Analyzer Specifications

6.1. External power requirements:

The InfraCal TOG/TPH Analyzer operates off external 12-volt power. The power sources can be either regulated DC power supplies or an external battery. This power can be provided by the user or by Wilks Enterprise, Inc. The suggested minimum requirement specifications for the 12 volt power source applied to the analyzer are described below:

Wall Supply Specifications:
Input: 100-250 VAC, 50-60 Hz, 0.5A
Output: 12 VDC, ±1%, 25 Watts

Battery Supply Specifications:
Output: 14 VDC Maximum, 11 VDC Minimum

Load Specifications:
1.5 Amperes Peak

6.2. Physical:

Dimensions:	6.5 in. x 6.5 in. x 5 in. (165 mm x 165 mm x 127 mm)	
Weight:	4.5 lb. (2.0 kg.)	
Control:	Display (output)	4 digit 7 segment red LED, 5/8 in. character height
	User (input)	4 multi-function push-button switches
Connectors:	Power --	Switchcraft Model 760 plug or equivalent
	Communications --	9-pin D-Sub, female

6.3. Environmental:

Temperature:	Non-operating --	0°F (-18°C) to 125°F (52°C)
	Operating --	40°F (4°C) to 110°F (45°C)
Humidity:	Relative --	10% to 60% non-condensing

6.4. Electrical

Noise:	Rejection --	60 dB minimum
Drift:	Short term Ambient --(< 1 Hr.)	± 0.3% of full scale
	Long term Ambient --	(> 1 Hr.) ± 0.1% of full scale
	Temperature --	± 0.03% of full scale per degree C
	Repeatability --	± 0.1% of full scale
Response:	On Delay --	5, 10, 15 or 20 second factory-set intervals
	Measure Time --	5 seconds
	Modes --	Local control or remote PC control
Resolution:	Conversion --	16 Bits (0.0015%)
Ranging:	Digital Ranging --	256 step automatic ranging
	Analog Range --	0 to 4.096 volts
	Answer Range --	Absolute; 00 to 9999
		Percent; 0.0 to 100.0%
		Decimal; .00 to 99.99
	Measure Range --	Dependant on sample concentration ratio
Measurement Accuracy:		± 1% of full scale
Measurement Repeatability:		± 0.1%, ±1 digit
Memory:	Non-volatile memory for calibration and configuration data	

6.5. Calibration:

- Electronic zero balance adjustment
- Up to 20 point curve fitting calibration
- Modes: Edit Table
  Off
  Factory (special order)

---

7. Analyzer Communications Interface

7.1. Abstract

The InfraCal TOG/TPH Analyzer supports communications to a PC or other host via an RS-232C asynchronous serial communications port. This capability allows for collection of sample measurement data and instrument control by a host computer. The host can also maintain calibration tables and download them to the instrument as required. This is particularly useful when more than one table is being used with a single instrument. Specification details for interfacing user customized host software are as follows:

7.2. Physical connection

The InfraCal TOG/TPH Analyzer is connected to the external device via the 9-pin female DB9 connector located on the rear panel in the lower left-hand corner. The InfraCal TOG/TPH Analyzer operates as a DCE device. To connect to a PC, a standard straight through 9 pin cable can be used, but only 3 wires are required.[1] The required signals are Transmit Data (TXD), Receive Data (RXD) and Ground (GND). The pinout is as follows:

Function	Pin
RXD	3
TXD	2
GND	5

[1] *Systems with serial numbers lower than 10200 require a null modem cable or null modem adapter.*

7.3. Communications port setup parameters

The port setup required by the InfraCal TOG/TPH Analyzer is:

- 9600 baud
- 8 data bits, 1 stop bit
- No parity

7.4. Operation

The TOG/TPH Analyzer accepts ASCII commands from the host and returns data as a response to certain commands or, in datalogging mode, on completion of a measurement cycle. All commands are two characters in length. Certain commands have parameters that follow the command. Parameters are separated by commas. All commands are terminated by a carriage return character. All data responses are comma separated ASCII fields, terminated by a carriage return character. The first field indicates the result type; the remaining fields are the result. Result types are 'B' for balance results, 'R' for run results or 'C' for calibration data. The result format is determined by the presentation mode and is identical to the LED display data. The Read Display Mode command returns a two character mode code. Alphabetic characters can be sent in upper or lower case. Response data is always upper case.

Command Set

Command	Description	Response Examples
RB	Read balance	B,1.025 B,0.865
RR	Read displayed result	R,27.5 R,315 R,1.873
RU	Run (same as RUN button)	None
RA	Run & display uncalibrated	result None
BA	Balance (same as Zero switch)	None
LR	Enable results data logging	None
DR	Disable results data logging	None
RM	Read display mode	MA, MP, MD or MR
MA	Set display mode to absolute	None
MP	Set display mode to percent	None
MD	Set display mode to decimal	None
MR	Set display mode to ratio	None
WB,<params.>	Set balance data	None
RC,<params.>	Read calibration table	See detailed description
WC,<params.>	Set calibration table	None

CM	Read Calibration Mode	CD, CE or CF
CD	Disable Calibration	None
CE	Enable User Calibration	None
CF	Enable Factory Calibration	None
ES	Return error status	E,0 E,2
RE	System reset	None
ID	Return firmware ID	2.02.06

7.5. Data logging

Data logging provides results output at the end of each RUN or BALANCE cycle. The results are output when data logging is enabled both for functions initiated from the instrument control panel and functions initiated by the host. The format of the data returned after a RUN cycle is as shown for the RR command and is determined by the display mode. The format of the data returned after a BALANCE cycle is as shown for the RB command. The RA command allows the host to initiate a run cycle and data log a result that is not adjusted by the calibration procedure. This can be used by an intelligent host resident calibration table generator.

7.6. Remote zero balance control

The instrument zero balance can be controlled via the communications port. The RB, WB and BA commands provide the necessary controls. This feature can be used to store multiple zero values for different operating conditions. This feature combined with the calibration controls described in the next section can be used to maintain multiple calibration curves when using the instrument for multiple applications.

The RB command will retrieve the current zero balance data. The WB command can then be used to reset the current zero balance to a previously recorded value. The WB data field is identical in format to the data returned in response to the RB command.

The BA command can be used to initiate a zero balance function under remote control. The operation is identical to initiating a zero balance from the instrument control panel. The user must insert the zero sample in the instrument prior to issuing this command. If data logging is enabled the result will be returned on completion of the function. The data format is identical to the RB command response. The result can optionally be read with the RB command if data logging mode is not used.

7.7. Remote calibration control

Calibration data can be retrieved or set under remote control. Due to the complexity of the calibration function (and the need to utilize multiple calibration standards) initial calibration can only be performed from the instrument panel. Another approach is to use the host to generate one or more calibration curves from uncorrected data log results collected with the RA command. This technique is extremely useful if the user desires to generate a calibration curve based on an average result of several measurements from a lot of each calibration standard.

The RC command is used to retrieve the current calibration table. A Calibration table consists of zero to twenty entries. The RC command can take the following forms:

RC	Read entire calibration table
RC,0	Read calibration table size
RC,n	Read a single calibration table entry, where n is the entry number.

The RC,0 command response is C,n where n is the number of calibration table entries from 0 to 20. If 0, the instrument is not calibrated. Otherwise, n is the number of calibration table entries.

The RC,n command response is C,n,x,y where n is the entry number as received, x is the raw measurement data as it appears on the display during calibration and y is the actual value as set by the user during calibration. The format is determined by the display mode (absolute, percent, or decimal). Calibration commands should not be used when in ratio mode since ratio mode does not use a calibration table. An RC command requesting data for a table entry number greater than the current table size returns erroneous data.

The RC command with no arguments returns the complete calibration table, one entry at a time starting with the table size information. The individual entries are then returned in numerical order up to the number of entries.

Read Calibration Table Example

Assume the instrument is calibrated in the absolute mode using three standards. Assume the calibration results were as follows:

Entry	Measured	Actual
1	15	30
2	26	50
3	33	70

The RC command will return the following:

C,0,3
C,1,15,30
C,2,26,50
C,3,33,70

The RC,0 command will return C,0,3

The RC,2 command will return C,2,26,50

The WC command can be used to download calibration table data based on previously uploaded data or as determined by a host program. The command format is WC,n,x,y where the parameters are identical in format to the RC command. The parameters must match the current display mode. When using the WC command the table size and all necessary table entries should always be downloaded. Once all table entries have been downloaded the table size should be set.

Write Calibration Table Example

To download the calibration table described in the previous example, send the following commands:

WC,1, 15,30
WC,2, 26,50
WC,3, 33,70
WC,0,3

---

8. Service and Customer Support

Your InfraCal® TOG/TPH Analyzer may have been purchased either directly from Wilks Enterprise or from a local dealer or representative. In cases of any difficulties or problems associated with operation you are recommended to contact your local representative or dealer first. If the analyzer was purchased

direct, or if there are problems that are unanswered by the local representative, or you have a technical question relative to the operation of the instrument or relative to the analysis, please contact Wilks Enterprise at the contact address provided below:

Customer Services Department
Wilks Enterprise, Inc.
25 Van Zant Street,Suite 8F
East Norwalk, CT 06855
USA

Telephone: (203) 855-9136
FAX: (203) 838-9868
E-mail:tech@WilksIR.com

Service and Repair

During the warranty period, Wilks Enterprise, Inc. offers free factory service for all failures that occur from normal instrument usage. The user is only required to cover the cost of shipping the instrument to the factory. After the warranty period, the user is required to cover the factory's cost of servicing plus all shipping charges. Normal one week turn around is offered for all InfraCal instruments that are returned to the factory for service. For users requiring faster service times, Wilks Enterprise also offers an advance replacement program that can respond to a user's needs with instrument replacement typically in less than 24 hours. For extended service contracts, advanced replacement programs, factory service charges or sample system installation procedures, please contact Wilks Enterprise, Inc. for details.

Appendix A: Alternate Data Presentation

The standard display format for the TOG/TPH Analyzer is relative absorbance (***AbS***) It provides relative absorbance value. Other formats are available, and these may be set for specific applications.

NOTE: *The TOG/TPH Analyzer must be calibrated in the selected data presentation mode. Changing the data presentation mode requires re-calibration.*

The following are the different data presentation modes available of the InfraCal analyzer:

Percent Mode (*PCt*): Calculated values are displayed to a single decimal place (0.0).

Decimal Mode (*dEC*): Calculated values are displayed to two decimal places (.00)

Absorption Mode (*AbS*): An arbitrary scale related to the raw absorption of the sample (00).

NOTE: *Inserting a decimal point does not change the raw relative absorbance value displayed for a given sample. ie: an Abs reading of 25 becomes 2.5 (pct) or .25 (dec).*

Ratio Mode (*RAt*): A threshold based scale where a value defining an acceptable limit for maximum or minimum acceptable concentration is set to the value of 1.000. All values less than 1.000 indicate that the concentration is less than the threshold, while all values greater than 1.000 indicate that the concentration is greater than the threshold.

The TOG/TPH Analyzer display format can be switched between modes by pressing and holding both the **CAL** and **ZERO** buttons for two seconds. Each time the **CAL** and **ZERO** buttons are pressed the display mode changes. Release both buttons and repeat until the desired mode is displayed. The display will read: ***AbS*** for absorption mode, ***PCt*** for percent mode, ***dEC*** for decimal mode, and ***RAt*** for ratio mode. Press **RUN** to exit the data presentation mode one the desired mode is displayed.

Appendix B-Correlation to an Alternate method

If values for sample analysis were taken with the Edit program on and calibration data in the Edit program, The following can be used to calculate revised calibration points to re-enter into the Edit program.

Rule Of Thumb:

- If the laboratory's analysis is greater in concentration than the Field analyzer's analysis, use a multiplier with the field instrument.
- If the laboratory's analysis is less in concentration than the Field analyzer's analysis, use a divider with the field instrument.

Example 1:

Assume the sample analysis on the **Wilks InfraCal** read as follows:
SAMPLE A = 25 ppm
SAMPLE B = 13 ppm
SAMPLE C = 11 ppm
Assume the alternate method to analyze the duplicate samples read as follows:
SAMPLE A= 63 ppm
SAMPLE B= 33 ppm
SAMPLE **C**= 28 ppm

In this case the alternate method read higher Oil and Grease concentrations than the Wilks InfraCal Analyzer. Therefore the difference must be determined by dividing the lowest number into the highest number. **The instrument must be made to read higher.** A multiplier must be determined as shown in the example below.

InfraCal results:
25 + 13 + 11 = 49 ppm

Alternate Method Results
63 + 33 + 28 = 124 ppm

124 divided by 49 **= 2.5 multiplier**

The multiplier for this example is 2.5. Therefore **multiply** the value from the "Calibration Standard in Desired Units"(CO1, CO2…) column in the *Relative absorbance Versus Calibration Standard or Alternate Method Table* (section 3.4.2) by 2.5. This will correlate the values to an alternate method. The new Calibration Standard values can be re-entered into **Edit** program (see section 3.4.3).

Example 2:
Assume the sample analysis on the **Wilks InfraCal** read as follows:
SAMPLE A = 63 ppm
SAMPLE B = 33 ppm
SAMPLE C = 28 ppm
Assume the analysis using the **alternate method** to analyze the duplicate samples read as follows:
SAMPLE A= 25 ppm
SAMPLE B= 13 ppm
SAMPLE **C**= 11 pip

In this case alternate method read higher Oil and Grease concentrations than the Wilks InfraCal Analyzer. Therefore the difference must be determined by dividing the lowest number into the highest number. **The instrument must be made to read lower.** A **divider** is needed instead of a **multiplyer.** As in example 1 divide the lowest number into the highest number to get the correlation factor.

124 divided 49 = **2.5 divider**

The divider for this example is 2.5. Therefore **divide** the value from the "Calibration Standard in Desired Units"(CO1, CO2…) column in the *Relative absorbance Versus Calibration Standard or Alternate Method Table* (section 3.4.2) by 2.5. This will correlate the values to an alternate method. The new Calibration Standard values can be re-entered into **Edit** program (see section 3.4.3).

Appendix C: Solvent Options

Hexane A volatile hydrocarbon that is specified for EPA Method 1664. Because it is a hydrocarbon, it must be evaporated prior to making the infrared measurement to determine TOG or TPH levels in a sample, which takes approximately 3 - 5 minutes. Since hexane is lighter than water, the solvent, after extraction will rest on top, making it easy to deliver a measured amount of extract to the sample platform or plate with a 50 microliter auto pipette without losing any of the sample.

Pentane A volatile hydrocarbon preferred in Scandinavian countries over Hexane. Similar in performance and use to hexane although it has a lower boiling point and therefore will retain more of the low boiling point components. Pentane is lighter than water and like hexane will rest on top after the extraction.

Vertrel MCA A volatile HFC solvent that has been used primarily as a replacement solvent for Freon in cleaning operations. Because of its hydrocarbon absorption, like hexane it must be evaporated prior to making the infrared measurement to determine TOG or TPH levels. Recent tests have indicated that Vertrel MCA is a suitable solvent to use instead of hexane. It reaches a stable level much quicker than hexane (1 ½ - 2 ½ vs. 3 - 5 min.) and because of its lower boiling point, fewer light-end volatiles components are lost in the evaporation process. Vertrel MCA has no ozone depleting potential, is non-flammable and has low toxicity. As Vertrel MCA is heavier than water, the solvent after extraction will rest on the bottom. A microliter glass pipette can be used when preparing a sample; however, because of the weight of Vertrel, the extract should be delivered to the sample platform or plate with a 50 microliter syringe, rather than a pipette for analysis.