Contents

Part 1

Before You Begin

Part 2

Operating the QFT2™ System

Part 3

Equipment and Supplies

Part 4

Safety and Health Information

Part 5

Turner Designs Fluorometer

Chapter 1

Overview of QFT2™ Technology

This chapter provides basic information about the QFT2™ system—the principles behind it as well as its development, applications, benefits, and limitations. If you are not yet familiar with QFT2™, you may find it useful to read the information in the following sections before proceeding in the guide:

- **What is QFT2™ Technology?**
- **What Are the Advantages of QFT2™?**
- **Example Applications of QFT2™**
- **Terms and Abbreviations**
- **Related References**
- **QFT2™ on CD-ROM**

What is QFT2™ Technology?

QFT2™ (Quantitative Fluorescence Technique 2) is a patented and licensed process developed by the Surface Logging Group in Texaco Upstream Technology. The QFT2™ process was developed to provide an objective, instrument-based fluorescence measurement that gives accurate, quantitative values representative of the amount and character of the oil in the formation.

QFT2™ is designed for rig-site use (usually from the mud logging unit) and does not involve extensive training. Formation samples are collected during the drilling operation and analyzed to determine oil concentration and character. The size of the sample interval depends on the importance of the formation. In zones of interest, sample intervals of 5 to 10 ft are normally collected.

The QFT2™ process involves preparing samples for analysis by spin drying, light grinding, and extraction of 0.5 g solids with 5 ml of either heptane (dry samples) or isopropyl alcohol (wet samples). Extract emissions are acquired at two wavelengths using a Turner Designs Model 10-AU fluorometer.

The Model 10-AU fluorometer with a laptop PC running the QFT2™ Data Logger Program is shown in Figure 1. This fluorometer is a ruggedized instrument designed for field use. It provides calibration features and a digital readout by means of an internal microprocessor.

Figure 1
Turner Designs Model 10-AU Dual Filter Fluorometer and Laptop PC Running the QFT2™ Data Logger Program

The QFT2™ Data Logger Program is used to record the readings and convert them to estimated Weight Percent Oil (Wt % Oil) and API gravity. Results are accumulated in a data file, which can be exported to log plotting programs. QFT2™ data are usually plotted adjacent to gas data on the mud log to aid in interpreting both.

When you interpret QFT2™ results in combination with other data (MWD or wireline), you can obtain significantly enhanced formation evaluation results. QFT2™ provides direct physical evidence of oil presence that can be overlooked by conventional methods, especially in low resistivity / low contrast pay.

How the System Was Developed

Since the industry first began drilling for oil, the driller has monitored formation samples to locate hydrocarbon zones. In the 1920s, the presence of light hydrocarbon gases in the mud was used as an indicator of oil and gas in the formation being drilled. In the 1930s, it was observed that many crude oils fluoresce when viewed with a broad-spectrum UV light. By the late 1930s, the use of the familiar black box to visually evaluate drill cuttings for oil content had become a standard mud logging technique. This technique,

which is still in use today, has long been recognized as highly subjective and inconsistent for detecting oil.

In 1985, Texaco began research to improve the value of mud logging. One of the first results of this effort was a patented technique for more accurately determining the amount of oil in the drill cuttings by measuring fluorescence. This technique, called the Quantitative Fluorescence Technique or QFT™, uses a sensitive portable fluorometer to provide quantitative values representative of the oil content of the cuttings.

The principle advantages of the original QFT™ method over visual UV-box fluorescence detection are twofold. First, QFT™ is a standardized process that yields an objective numeric value suitable for continuous logs. In contrast, use of the UV-box is operator dependent and necessarily involves qualitative, subjective judgments.

Second, oils fluoresce most strongly at UV wavelengths outside the visible region. In particular, light oils fluoresce only weakly in the visible range and are detectable with the UV-box only when present in large quantities. QFT™ measurements are made in the UV region, outside the visible region where crude oils exhibit peak fluorescence. The emission wavelength used in the original QFT™ method was selected for good sensitivity to both light and heavy oils.

When you can obtain representative samples, either while drilling or from cores, QFT™ technology provides a more direct measurement of oil content than you can obtain from wireline logs. The term “surface logging” has been introduced to represent new approaches to gas and oil detection that are now proven alternatives to older methods.

An early QFT™ improvement was the development of a solvent system that allowed for the analysis of wet cuttings. The original QFT™ analysis required that samples be dry. To avoid loss of volatiles, the drying process cannot use high temperatures. Thus, during periods of rapid drilling, many samples in various stages of dryness could accumulate in the logging unit. An alternative extraction technique using isopropyl alcohol (IPA) was developed to enable analysis of wet samples. This method reduces loss of volatiles while allowing the mud logger to keep pace with the drilling.

What Are the Advantages of QFT2™?

The original QFT™ process, released in the late 1980s, used a single point fluorescence measurement to determine oil concentration. This method was

quantitative in that it yielded a reproducible numeric value. The relationship of this value to actual oil quantity, however, was dependent on the type of oil present. Oils vary in their intrinsic fluorescence at any specific wavelength, so that each oil will have its own calibration curve. Thus, it was not possible to relate field QFT™ measurements to specific oil quantities because the type of oil was not known.

To overcome the limitations of the original QFT™ method, Texaco developed Quantitative Fluorescence Technique 2 (QFT2™), an enhanced fluorescence method for detecting and characterizing oil from drill cuttings, cores, and environmental samples. The QFT2™ system consists of a dual filter fluorometer to measure fluorescence intensities at two emission wavelengths and the QFT2™ Data Logger Program. The QFT2™ Data Logger Program monitors and records the emission signal via a serial data link. The readings are converted using an iterative calculation to give estimated Wt % Oil and API gravity. This information is stored in a usable file format. You will find a copy of this program on the diskette inside the back cover of this guide.

The QFT2™ Data Logger Program also calculates UV-absorption loss corrections and uses this data to warn you when sample extract dilution is required. The Model 10-AU fluorometer and a laptop PC running the QFT2™ Data Logger Program are shown in Figure 1. An example of the program's calculations screen, activated by the operator once readings have been acquired, is shown in Figure 2.

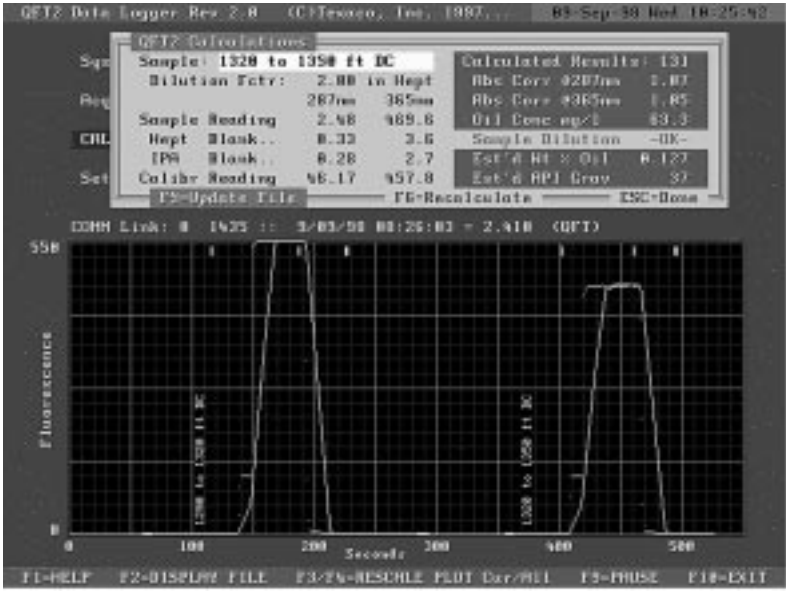


Figure 2
QFT2™ Data Logger Program Calculations Screen

The QFT2™ system provides the following important advantages over previous rig-site fluorescence techniques:

- Reproducible numeric results rather than the subjective evaluations of visual fluorescence methods
- Greatly improved sensitivity over visual methods, particularly for light oils and condensates
- Immediately useful Wt % Oil and API gravity results instead of relative fluorescence values or qualitative ratings
- Minimum response to troublesome cuttings contaminants (e.g., pipe dope)
- Computerized data acquisition, calculations, and data file

QFT2™ represents an advance over previous rig-site fluorescence methods, providing reproducible values in terms meaningful to log analysts and petroleum engineers. Keep in mind, however, that QFT2™ results are based on empirical trends that exhibit considerable scatter. QFT2™ is most valuable when used as a hydrocarbon survey tool rather than as a substitute for more accurate laboratory analysis.

The ability to interpret QFT2™ results is greatly enhanced when reliable gas data are available. Interpreting QFT2™ data in combination with gas readings provides more information than supplied by either measurement alone. Using QFT2™ in combination with gas data will enable you to make more informed decisions, particularly when wireline logs are either not available or difficult to interpret.

Example Applications of QFT2™

When you can obtain representative formation samples, QFT2™ provides a more direct measurement of formation oil content than do conventional techniques. When you use QFT2™ in combination with Texaco's Quantitative Gas Measurement (QGM™) System, it is a powerful tool for identifying hydrocarbons in zones that are difficult to evaluate using wireline or logging-while-drilling (LWD) data alone. If you rely solely on wireline and LWD tools, you can miss hydrocarbon bearing sand intervals containing dispersed clay or thin shale laminations. While resistivity values are suppressed by dispersed or laminated shale in the formation, QFT2™ (oil) and QGM™ (gas) values (which are a direct measure of hydrocarbons present in the formation), are unaffected by shale content.

The following examples provide a quick look at the application and results of QFT2™ analysis performed on several exploration wells. Each example shows QFT2™ data compared with laboratory analysis of fluid samples and other well evaluation data.

QFT2™ Correlates with Other Logging Tools

QFT2™ is useful for correlating with open hole and MWD logs. Figure 3 shows resistivity and surface logging data through the pay zone from a Gulf of Mexico well. Wet QFT2™ samples were run every 30 ft. In general, both normalized gas and QFT2™ values trend with wireline data. The QFT2™ estimated oil gravity is constant throughout the zone, which indicates a uniform oil type. Sample extracts gave an estimated gravity of 32° API in comparison with a laboratory value of 33° API obtained from a well test sample.

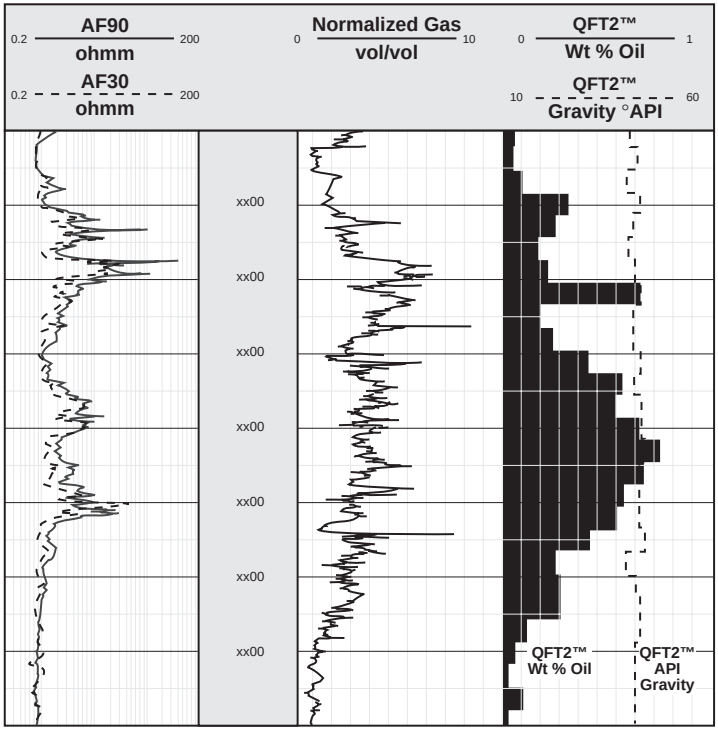


Figure 3
QFT2™ Correlates with Other Logging Tools

QFT2™ Correlates with Sidewall Core Analysis

QFT2™ is also useful for correlating with sidewall core data. Figure 4 shows wireline data and QFT2™ results on sidewall cores taken through a pay zone in another offshore well. Both sets of data indicate a dual lobe sand. Results

from X-Ray diffraction analysis indicate an intermediate shale interval. In both intervals, QFT2™ Wt % Oil increases with increasing resistivity.

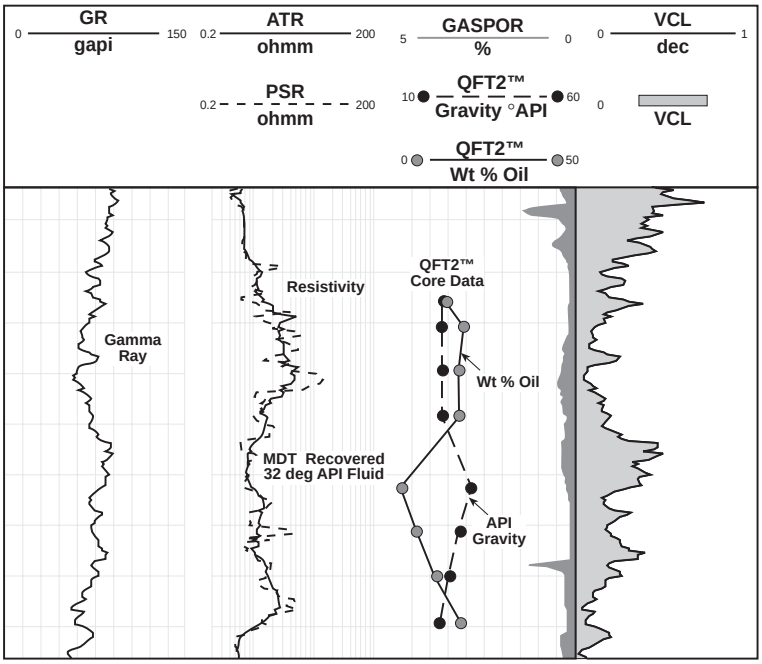


Figure 4
QFT2™ Correlates with Sidewall Core Analysis

The QFT2™ estimated API gravity clearly shows two distinct zones. In the upper zone, the oil character is constant at an estimated gravity of 24° API. Laboratory analysis of an MDT (Modular Dynamic Tester) fluid sample from the lower zone gave an oil gravity of 32° API, which matched the QFT2™ estimated gravity (31° API) from the sidewall core taken at that depth. However, QFT2™ data indicated a trend to heavier oil (24° API) deeper in the zone not typical of the MDT sample. Later well tests showed problems with asphaltene deposition in the lower zone due to the heavy oil predicted by QFT2™.

QFT2™ Discriminates Oil and Gas

Figure 5 shows an example of QFT2™ data integrated with surface gas and open hole log data from a deepwater well. Quantitative Gas Measurement (QGM™) normalized gas values greater than 5 (shown in gray) indicate a show. There are two show zones on the log. The gamma ray indicates sand with a corresponding increase in resistivity, normalized gas values, and QFT2™ Wt % Oil in both intervals. Although gas was observed on wireline data, the presence of oil was required to make the discovery economic.

In the upper zone, QFT2™ indicates 40° API gravity oil, and QGM™ shows significant quantities of gas and correspondingly high GOR values. Primary production from this zone was gas. In the lower section, QFT2™ estimated 30° API gravity oil coincident with a drop in GOR values from QGM™ data. Primary production from this zone was oil.

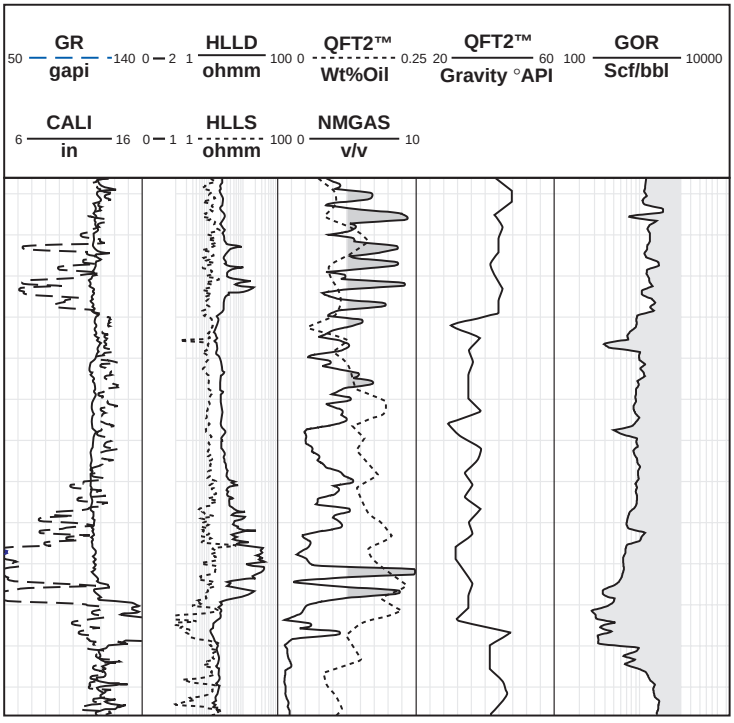


Figure 5
QFT2™ Discriminates Oil and Gas

QFT2™ Indicates Oil in Low Resistivity / Low Contrast Pay Zones

QFT2™ is an accurate and reliable tool for detecting liquid hydrocarbons. It has helped identify oil-bearing zones in intervals that are difficult to evaluate using wireline data alone.

Figure 6 shows an example of a low resistivity/low contrast pay zone in a deepwater well. The resistivity from MWD was less than 2 ohm-m. The gamma ray (GR) showed little sand development, and no gas crossover was observed. The QFT2™ Wt % Oil increased coincident with the normalized gas. Estimated fluid gravity from QFT2™ was 39° API. The zone was subsequently completed and produced 21 MMscf/d of gas and 3800 bbl/d of 46° API gravity oil.

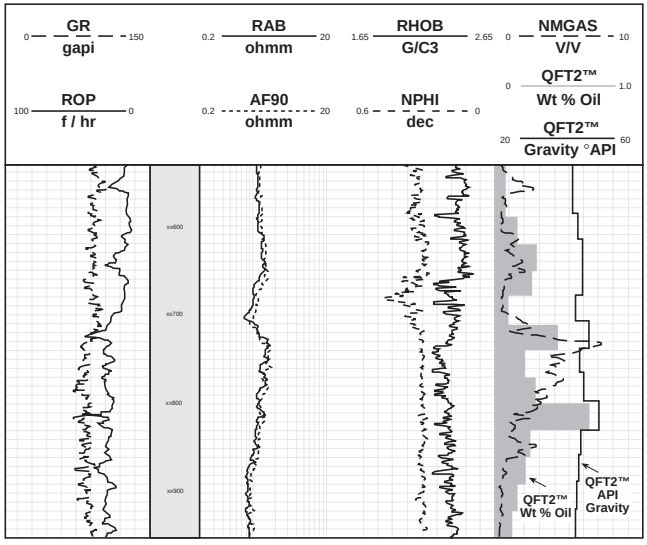


Figure 6
QFT2™ Indicates Oil in Low Resistivity/Low Contrast Pay Zones

QFT2™ Enhances Ability to Predict Immobile Oil

Figure 7 shows an example of QFT2™ Wt % Oil converted to volume percent oil (oil porosity) and plotted with hydrocarbon porosity from wireline data. (For more information on determining Wt % Oil, see *Interpreting Weight Percent Oil Values* in Chapter 3.) The results are integrated with resistivity and gamma ray. The estimated oil concentration, measured by the QFT2™ method, is equivalent to the estimated hydrocarbon porosity. This indicates very little flushing of the oil from the cuttings during the drilling process, which suggests immobile oil or a tar mat. A subsequent drillstem test produced no oil.

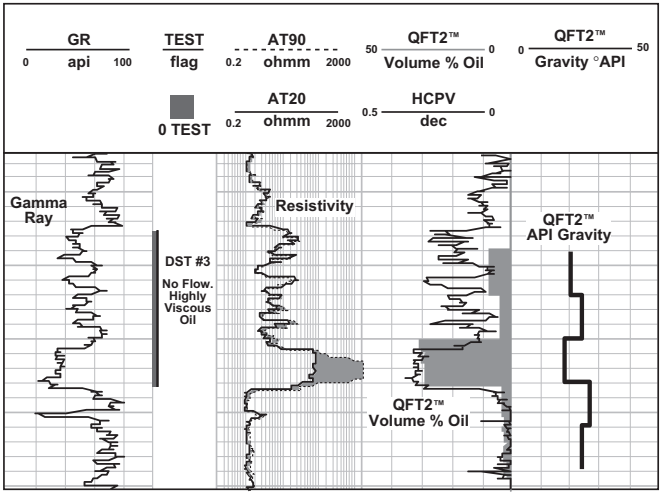


Figure 7
QFT2™ Enhances Ability to Predict Immobile Oil

QFT2™ Indicates Producible Oil

Figure 8 shows QFT2™ Wt % Oil converted to oil porosity and plotted with hydrocarbon porosity from wireline data. The hydrocarbon porosity predicts more hydrocarbons than the volume percent oil shown by the QFT2™ method. This indicates flushing of the hydrocarbons during the drilling process, which suggests moveable oil. A subsequent drillstem test in the lower section of the zone produced 1600 bbl/d of oil.

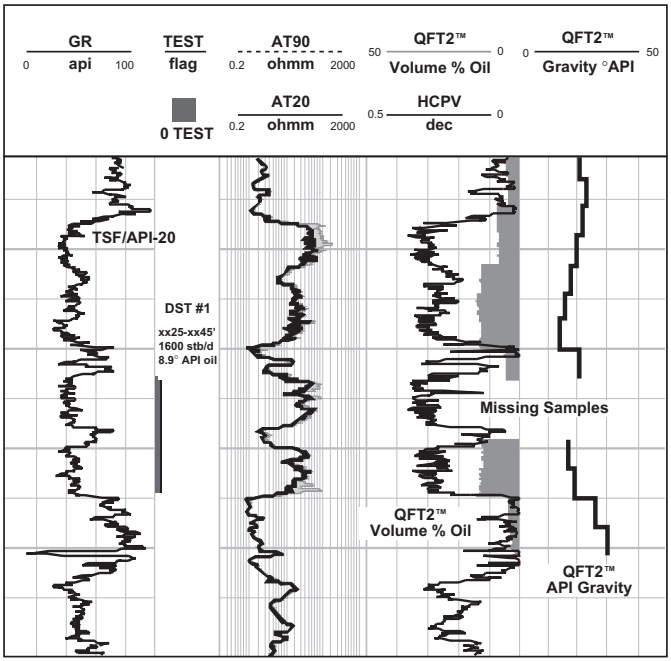


Figure 8
QFT2™ Indicates Producible Oil

Terms and Abbreviations

The key terms, abbreviations, and symbols used in this guide are defined in this section. For additional nomenclature, see Chapter 6. To operate the QFT2™ system and use the data it generates, you should first become familiar with the following key terms:

Absorbance Correction The factor applied to QFT2™ values to correct for UV absorption losses.

Fluorescence

Molecular absorption of UV radiation causes an electronic transition to a higher energy orbital. The electrons return rapidly (10 nanoseconds) to ground state, emitting energy in the form of light.

Normalized Gas

The ratio of the total volume of gas measured at surface conditions to the volume of cuttings drilled over the interval (V_s/V_c). The normalized gas value is useful because it gives a direct indication of how much surface (pipeline) gas is available from the show interval.

Oil Density

The mass of oil per unit volume. To convert Wt % Oil to the more commonly used Volume % Oil (oil porosity), multiply by the rock bulk density, and divide by the oil density. Oil density is calculated from API gravity using the following equation:

$$\text{Oil Density} = \frac{141.5}{(131.5 + \text{API Gravity})}$$

where:

Oil Density = g/cc

Oil Porosity

The volume percent oil (Vol % Oil).

Weight Percent Oil

Formation oil content calculated from the estimated oil concentration and the specified Dilution Factor. The standard deviation for this calculation based on QFT2™ analysis of 41 oils ranging from 20^o to 70^o API gravity corresponds to a factor of 1.7 uncertainty in actual oil content.

The abbreviations and symbols used in this guide are shown in Table 1 and Table 2.

Table 1
Abbreviations for Key Terms

Table 1. Abbreviations for Key Terms

Term	Abbreviation
Degrees API	^{o} API
Dilution Factor	DF
Drillstem Test	DST
Gas-Oil Ratio	GOR
Instrument Correction Factor	Instr. Corr. Factor
Lost Circulation Material	LCM
Measurement-While-Drilling	MWD
Million Standard Cubic Feet	MMscf
Modular Dynamic Tester	MDT
Observed Oil / Standard	Obsd Oil / Stnd
Polycrystalline Diamond Compact	PDC
Quantitative Fluorescence Technique™	QFT™
Quantitative Gas Measurement™	QGM™
SARA	Saturates, Aromatics, Resins, and Asphaltenes
Thousand Standard Cubic Feet	Mscf
Total Depth	TD
Volume % Oil	Vol % Oil
Weight % Oil	Wt % Oil

Table 2
Symbols

Table 2. Symbols

Fraction	Prefix	Symbol
10^{-1}	deci	d
10^{-2}	centi	c
10^{-3}	milli	m
10^{-6}	micro	μ
Quantity	**Units**	**Symbol**
Density	gram/cm^3	g/cc
Length	feet	ft
	meter	m
	millimeter	mm
	nanometer	nm
Mass	gram	g
Volume	milliliter	ml

Related References

For more information on related topics, you may wish to consult the following selected reference publications:

- Delaune, P.L., *Surface Techniques to Measure Oil Concentration While Drilling*, presented at the SPWLA 33rd Annual Logging Symposium, Oklahoma City, Oklahoma USA, June 14-17, 1992.
- DeLaune, P.L. and Spilker, K.K., Texaco Upstream Technology and Wright, A.C., Consultant, *Enhanced Wellsite Technique for Oil Detection and Characterization*, SPE 56802 presented at the SPE Annual Technical Conference and Exhibition, Houston, Texas USA, October 3-6,1999.
- Shugar, G. and Dean, J., *The Chemist's Ready Reference Handbook*, McGraw-Hill, l990.

- Shugar, G. and Ballinger, J., *Chemical Technician's Ready Reference Handbook*, 3rd edition, McGraw-Hill, 1990.
- *Turner Designs Model 10-AU-005 Field Fluorometer User's Manual*, Turner Designs, Sunnyvale, CA, 1992.

QFT2™ on CD-ROM

For additional information on Texaco's QFT2™ technology, request a copy of the QFT2™ interactive CD-ROM. This CD contains:

- Overview of the QFT2™ system
- Interactive version of the *QFT2™ User's Guide*
- Video showing set-up and calibration of the QFT2™ system
- Tutorial on preparing QFT2™ samples for analysis
- Tutorial on installing the QFT2™ Fluorometer Retrofit Kit
- Additional QFT2™ resources

For a copy of the QFT2™ CD-ROM, contact:

Texaco Upstream Technology
QFT™ Licensing
3901 Briarpark
Houston, TX 77042
713.954.6000

■■■■■

Chapter 2

The Basics of the QFT2™ System

Whether you are using the QFT2™ system for the first time or you just want to learn more about it, you may find it helpful to start with the basics. This chapter explains the general theory underlying the QFT2™ technique, provides an overview of the sampling and analysis process, and a gives you a quick look at how to interpret results and sensitivity of readings. The chapter contains the following sections:

- **How QFT2™ Works**
- **Overview of the QFT2™ Process**

How QFT2™ Works

The QFT2™ technique is based on the fact that the intensity of oil fluorescence is linearly proportional to the amount of crude oil present in a dilute sample. Oil fluorescence arises from the aromatic molecules which are present in all whole oils. The fluorescence process is shown schematically in Figure 9. Molecular absorption of UV energy causes an electronic transition to a higher energy orbital. Since the equilibrium bond lengths change during this process, the excited molecule will have excess vibrational energy which is rapidly lost through collisions with other molecules. After about 10 nanoseconds the electron returns to its ground state orbital, again with excess vibrational energy, by emitting longer wavelength radiation. While excited, the molecule has undergone about 10,000 collisions, so that the emitted radiation is scattered in all directions. Because the emission radiation differs in wavelength and direction from the excitation radiation, fluorometers can easily be designed to discriminate against detection of the incident beam and thus achieve high sensitivity. Figure 10 shows the general optical layout employed in the Turner Designs Model 10-AU fluorometer.

Figure 9
Relationship Between Absorption and Fluorescence Spectra

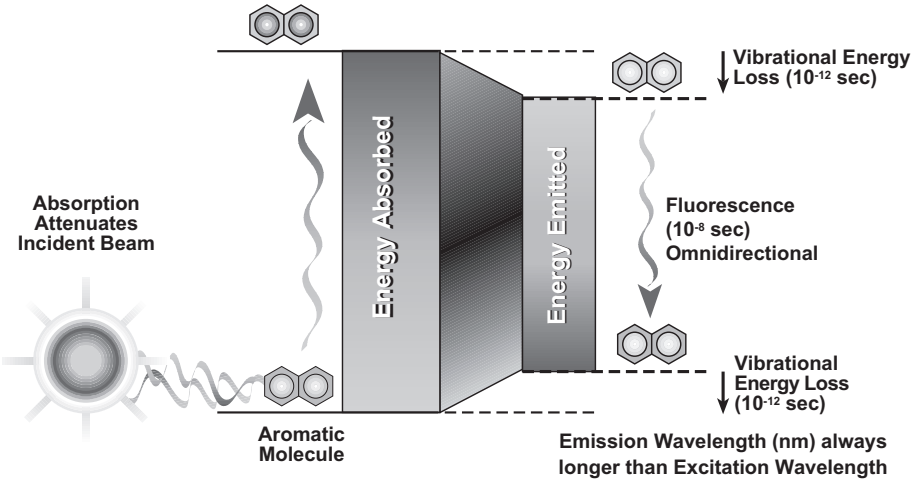
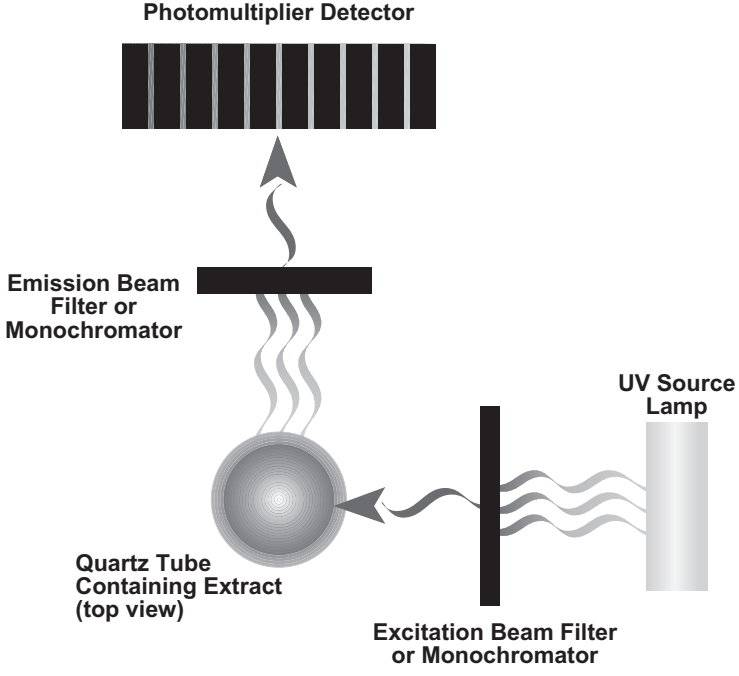


Figure 10
Schematic of Fluorometer Design



Fluorescence and Oil Maturity

As oils mature, their aromatic (fluorescent) content decreases relative to their saturates (non-fluorescent) content. In addition, the proportions of the various aromatic components undergo modification. Figure 11 and Figure 12 show absolute and normalized emission curves which demonstrate how these trends are reflected in oil fluorescence. The spectra were obtained with a spectrometer using the excitation wavelength employed by QFT2™. The QFT2™ technique is based on the fact that multi-ring aromatics decrease as the API gravity increases. The two emission wavelengths used by QFT2™ measure fluorescence from single-ring (short wavelength "A") and three-ring (long wavelength "B") aromatics, as shown in Figure 12. The A/B intensity ratio (A/B ratio) varies with the proportion of single-ring to three-ring compounds and thus increases with oil maturity. Although the fluorescence of single-ring aromatic molecules is weak relative to the fluorescence of multi-ring aromatic structures, it was found that short and long wavelength selection provides the best correlation with oil type by a significant statistical margin.

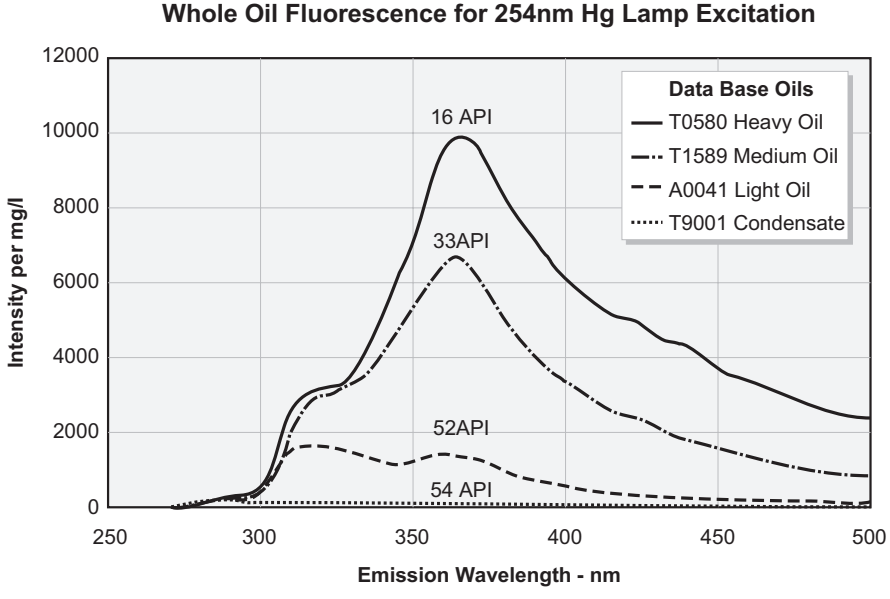


Figure 11
Whole Oil Fluorescence per (mg/l) in Heptane

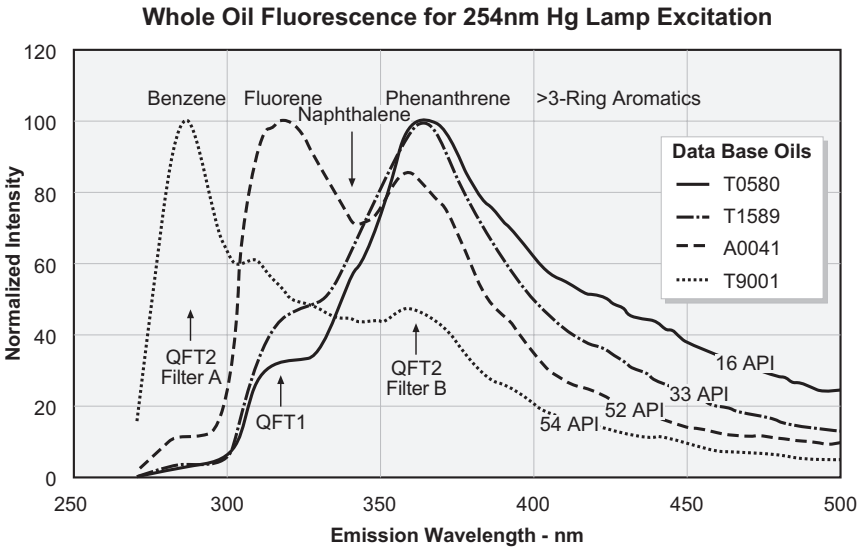


Figure 12
Whole Oil Normalized Fluorescence in Heptane

How QFT2™ Was Calibrated

When oils from diverse sources are analyzed, both oil gravity and Wt % Oil vary systematically with the A/B ratio. The QFT2™ method was developed from fluorescence spectra analysis of a world-wide suite of oils with gravities ranging from 10^{o} to 70^{o} API using both n-heptane and IPA as oil solvents. For oils having gravities of at least 20^{o} API, the standard deviation for the Wt % Oil calculation corresponds to a factor of 1.7 uncertainty in actual oil content when using either solvent. Analysis of the oils with gravities greater than 20^{o} API showed somewhat lesser accuracy when using heptane and erratic results for IPA (because of limited solubility). Similarly, for both solvents the standard deviation for the API gravity estimation is about $\pm 8^{o}$ API for oils having gravities of at least 20^{o} API. The heavier oils gave more variable, high estimates. The effect was most pronounced when using IPA.

Figures 13 through 16 show plots of the database oil measurements along with the resulting linear regression trend lines used in QFT2™ calculations. In all cases, the A/B ratio (designated by the letter R) is transformed as noted on the horizontal axes of the plots for linearization purposes. Only oils with gravities of at least 20^{o} API were included in the regression analyses. As these figures make clear, the measured values exhibit considerable scatter about the trend lines so that QFT2™ should always be regarded as providing *estimates* for oil gravity and content rather than firm values. QFT2™ is not a substitute for laboratory analysis of critical samples. QFT2™ is nonetheless valuable as a screening tool for detecting oil shows and oil type variations.

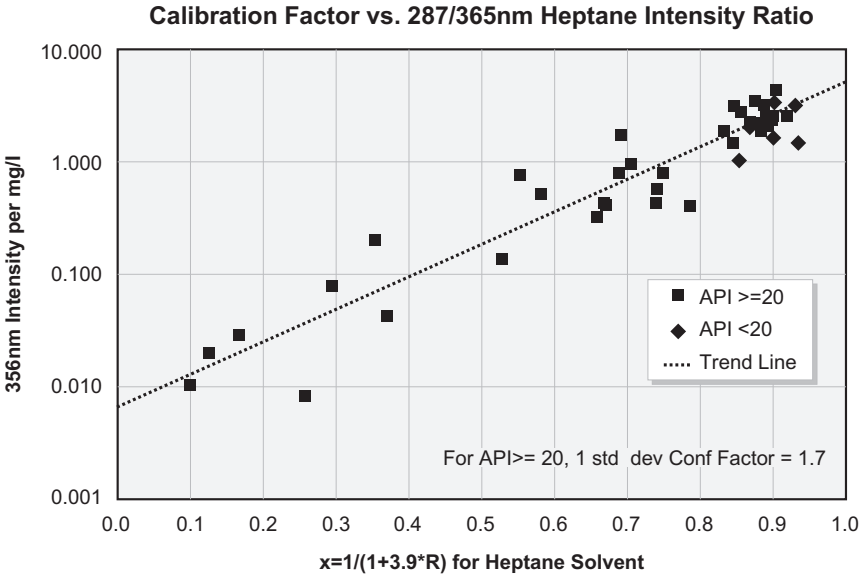


Figure 13
Intrinsic Oil Fluorescence in Heptane Solvent

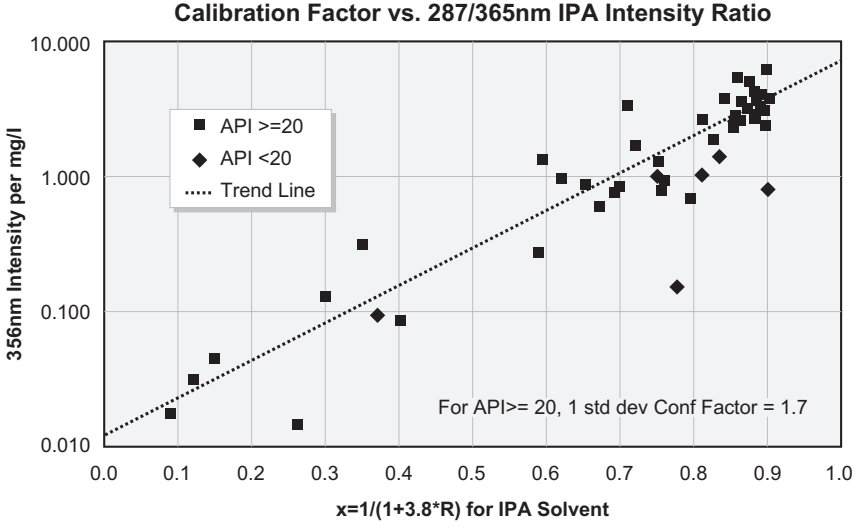


Figure 14
Intrinsic Oil Fluorescence in Isopropyl Alcohol

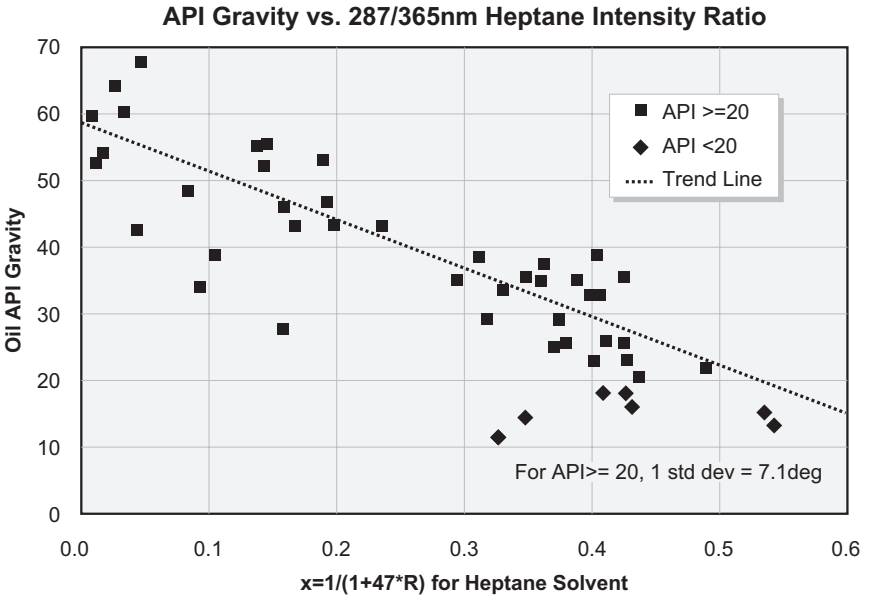


Figure 15
API Gravity vs. Fluorescence in Heptane Solvent

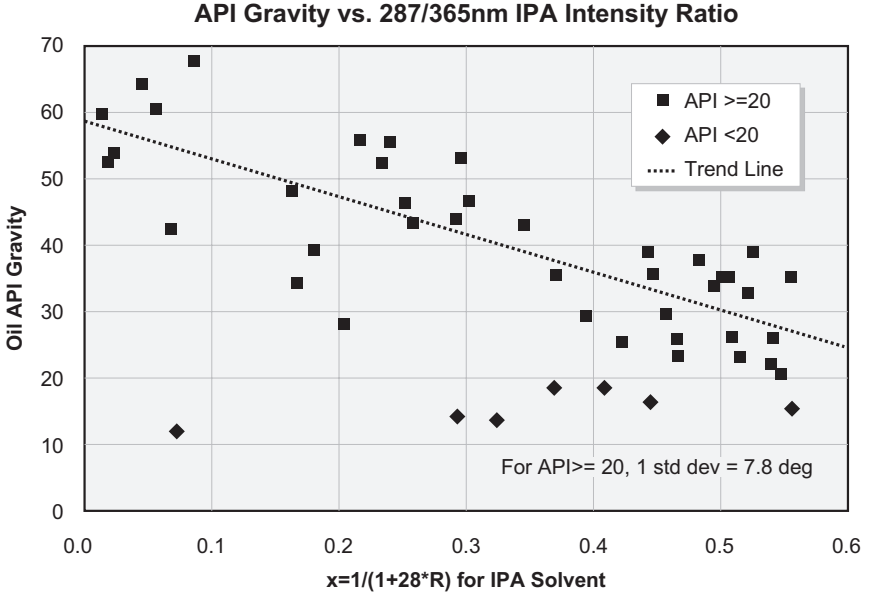


Figure 16
API Gravity vs. Fluorescence in Isopropyl Alcohol Solvent

Overview of the QFT2™ Process

You can apply QFT2™ analysis to any sample for which the extractable whole oil content is of interest. The principal rig-site application is analysis of drill cuttings, but other possible sample types include cores, archived cuttings, and environmental soil and water samples.

Obtaining and Preparing Samples

Regardless of the sample type, the first requirement is obtaining a representative sample. For most samples, you should spin dry without washing to minimize loss of oil.

QFT2™ requires a small sample quantity—usually only 0.5 g solids. For well consolidated formations, some grinding may be required to prepare the sample. When using PDC bits or when the formation is unconsolidated, sample grinding is not required. For field applications, measure a fixed volume of sample using a porcelain spoon or another device that measures 0.5 g of sample.

If you use the fixed volume sample method, make sure you use the same spoon (or other measuring device) to measure all samples for that particular well.

For laboratory applications, you should weigh samples using a 0.01 g resolution balance.

Extracting Oil and Measuring Fluorescence

To extract oil from the sample, place the measured sample in a small test tube and add 5 milliliters (ml) of heptane for dry samples or IPA for wet samples. Vigorously agitate the test tube for 30 seconds at ambient temperature. After allowing the solids to settle, filter the solution using a syringe filter. Next, measure the fluorescence of the extract relative to the QFT2™ standard on Filters A and B using a calibrated Turner Designs 10-AU instrument.

Capturing Your Results

You will capture all readings using the QFT2™ Data Logger Program. You will also use this program to capture calibration data and to convert sample readings to estimated Wt % Oil and API gravity. These results are accumulated in a data file, which you can then export to other programs as required for analysis and presentation of the data.

Correcting for UV-Absorption

The QFT2™ Data Logger Program also estimates and applies UV-absorption loss corrections, which can be significant for heavy oils. When the corrections are excessive, the program advises you to dilute and rerun the extract. In extreme cases, fluorescence can actually decrease with increasing concentration due to absorption losses, as shown in Figure 17. This situation is corrected by diluting the sample.

IMPORTANT

You should always dilute and rerun a sample when it has a distinct yellow coloration because the QFT2™ Data Logger Program cannot detect this condition.

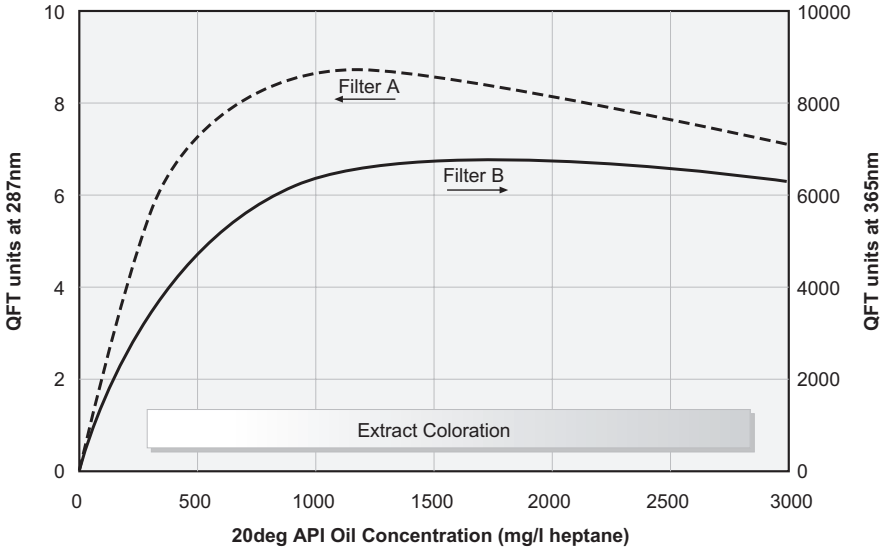


Figure 17
Absorption Loss Effects Causing Non-Linear QFT2™ Readings

Selecting an Extraction Method

Heptane is the preferred extraction solvent because it provides more reliable results for heavy oils. As a practical matter, however, using IPA permits more timely analysis and avoids filling the logging unit with samples being dried prior to extraction. When you use IPA, you should confirm results for important samples using heptane. For light oils, both solvents work equally well. When very light oils are of interest, use wet IPA extraction to avoid loss of volatile aromatics during drying.

Figure 18 shows QFT2™ estimated Wt % Oil and API gravity for the same sample extracted wet and dry using both heptane and IPA. The very low results for heptane extraction of the wet sample clearly shows the need for

sample drying when heptane is used. Given the accuracy of the QFT2™ estimates, results for the remaining three extraction methods agree well within expectations.

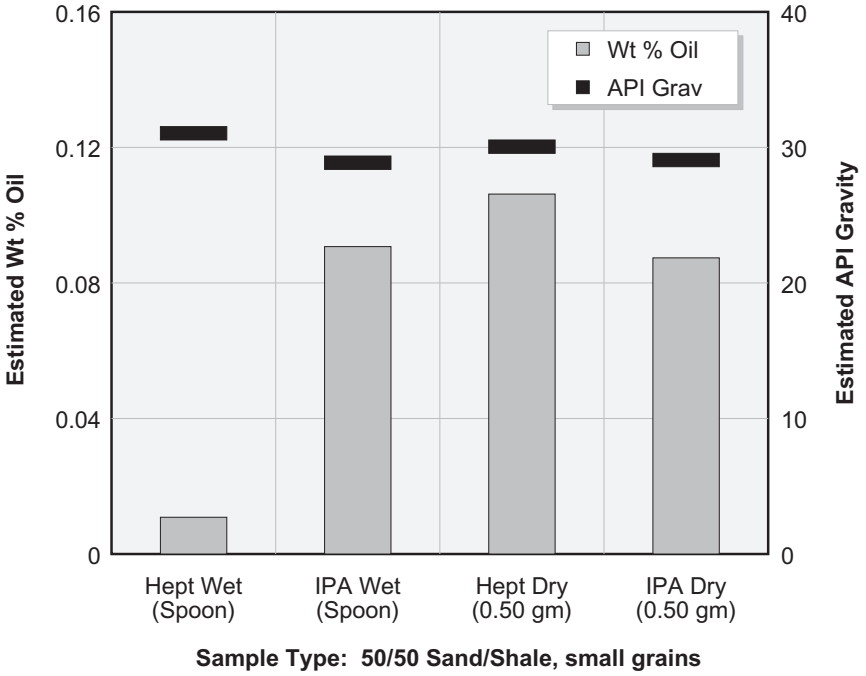


Figure 18
QFT2™ Results for Wet and Dry Extraction Methods

Effect of Water Content

The effect of water content on QFT2™ results for the IPA extraction method is shown in Figure 19. In this experiment a quantity of a thick gumbo-consistency sample was air dried. Then various amounts of water were back-added and mixed with 0.50 g of solids which were then extracted with 10 ml of IPA. The estimated oil content is seen to increase somewhat over the dry sample, perhaps due to clay expansion effects, but the change is minor. Water content variation produced no significant change in estimated oil gravity.

Specifying Detection Limits

Specifying detection limits for QFT2™ is difficult because two emission readings (both dependent on the oil type) are involved. If you have taken care in establishing accurate solvent blank readings, you can easily achieve a detection level of 0.2 QFT2™ units on Filter A. This value corresponds to a Wt % Oil value of less than 0.01% for all but the lightest oils. Using IPA provides somewhat more sensitivity because emission intensities are typically higher by a factor of about 1.8 than when using heptane.

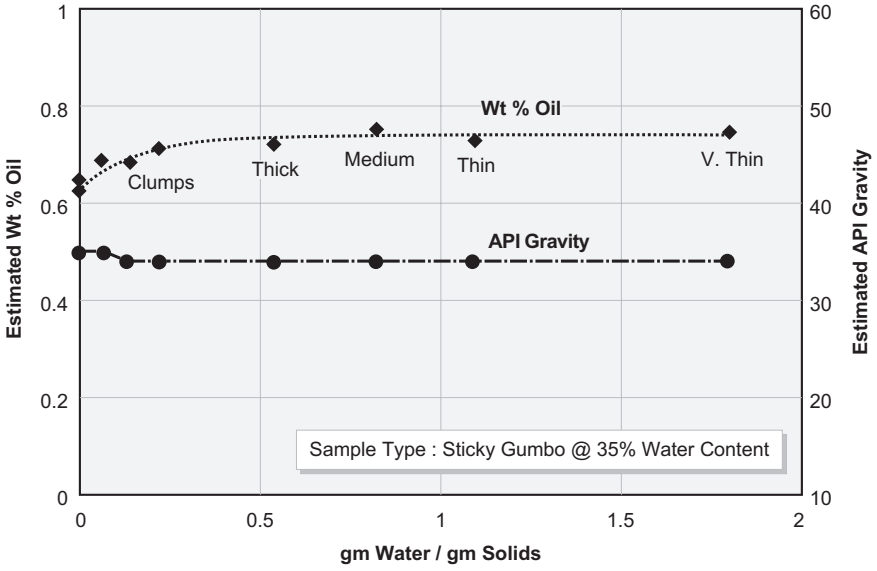


Figure 19
Effect of Water Content on IPA Extraction Results

Reproducibility of API Gravity and Weight Percent Oil Estimates

The reproducibility (not accuracy) of the API gravity estimate is inversely proportional to the Filter A reading. With reasonable intensity at Filter A (2.0 QFT™ units), the API gravity estimate varies by no more than ±2° for duplicate samples. The reproducibility of the Wt % Oil estimate when readings are well above the detection limit depends both on sample homogeneity and the care taken in measuring sample quantities. For homogeneous samples, reproducibilities of ±10% relative are typical when using the spoon and ±5% relative when using a 0.01 g resolution balance.

■■■■■

Chapter 3

Working with QFT2™ Data

The value of QFT2™ results relates directly to the quality of the samples you capture and the interpretation of the data you generate. To gain the best results, you should understand how your data is influenced by well conditions and operational factors. This knowledge will help you control the quality of the data at the well site and in the laboratory. Once you have captured the data, it is important to know how to correctly interpret and present it for optimum results. The following sections will help you in working effectively with QFT2™ data:

- **Factors Affecting the Data**
- **Interpreting Weight Percent Oil Values**
- **Presenting Data on Well Logs**

Factors Affecting the Data

To effectively use the QFT2™ system, you must be aware of the many factors that can affect your reported results. Many of these factors, in particular formation flushing and cuttings quality, are outside your control. You can, however, take steps to ensure the samples are as representative as possible of the drilled intervals. For example, make sure the mud log contains sufficient annotation to aid follow-up interpretation. In particular, you should note mud weights, bit types, and cuttings from coring operations. In most cases, you will obtain the best samples by simply spin-drying representative cuttings with minimal grinding. In some cases, you can remove by hand obvious contaminants such as cement or caving shale.

It is not possible to provide hard and fast rules covering all situations, but the goal of sample preparation is to retain to the maximum degree possible any formation oil present in the cuttings while avoiding extraneous sources of fluorescence.

The ideal QFT2™ sample would consist of uncontaminated cuttings from a well defined depth interval which still contained all in-place hydrocarbons. Real world samples, unfortunately, are subjected to numerous processes which affect QFT2™ oil extraction and measurement. You should become familiar with the following factors which can affect your QFT2™ data.

Formation Flushing

One of the most serious impediments to relating QFT2™ oil concentration estimates to actual formation oil content is flushing ahead of the bit due to overbalanced drilling and bit hydraulics. In extreme cases, detected hydrocarbons may actually decrease when a porous and permeable pay zone is entered (for example when the lithology changes from oily shale to loosely packed sand). When you interpret QFT2™ readings, you should always take into consideration formation type (i.e., unconsolidated sands, shale, etc.) and pressure balance.

In general, coarse, well consolidated cuttings should be only lightly ground to minimize extraction of oil from impermeable rocks of no commercial interest. When available, wireline logs having deep and shallow resistivity may aid in determining when flushing is important.

Cuttings Quality

Drill cuttings which disintegrate because of mechanical weakness or bit action will lose their hydrocarbons to the mud. This effect can be pronounced for uncemented sands and when PDC bits are employed (generating “rock flour”). To retain as much oil as possible in the cuttings, you should not wash QFT2™ samples before spin drying. For the same reason, samples collected for possible laboratory QFT2™ analysis should be handled as little as possible (wet, unwashed samples are usually preferred).

Borehole Washout

Collected cuttings normally contain variable amounts of rock from up-hole intervals, due either to sloughing formation or to the mechanical action of the drill string (stabilizers in particular). In some cases it will be clear that certain rock types are extraneous (for instance when caving shale chips are present), and you can avoid these by hand selection. Most often, however, you should analyze the entire collected sample and note on the log when hole enlargement may be contributing significantly to the QFT2™ readings.

IMPORTANT

Unrepresentative Samples

Make sure the QFT2™ sample is representative of the entire interval rather than just the ending depth.

The QFT2™ log can have no better depth resolution than the collection depth interval. As a practical matter, usually a 30 ft (or 10 m) sample interval is used due to the labor and time involved in running QFT2™.

When drilling zones of interest, run QFT2™ at more frequent intervals when possible (such as every 10 ft) to improve log resolution. You should also catch samples during strong gas shows as an aid in interpreting both the gas and QFT2™ readings.

Mud System Contaminants and Additives

At the start of a job, you should visit the drill floor and mud engineer's area to determine what types of organic compounds may end up in the mud system. If possible, obtain samples and run QFT2™ analyses to determine what, if any, effect these compounds may have. In general, whenever you see a QFT2™ increase without an accompanying gas increase, consider the possibility of fluorescence from the mud system. You can monitor the suction pit mud for background fluorescence by analyzing 0.5 ml mud samples using QFT2™ (IPA solvent if wet, heptane if dried).

The most common mud contaminant, particularly after trips, is pipe dope. The presence of pipe dope is usually signaled by an isolated spike in the QFT2™ readings with no corresponding gas peak. The estimated API gravity for the increase will be high (about 50). Figure 20 shows an example of how QFT2™ responds to various levels of pipe dope. (This particular sample was obtained from an offshore well. In general, pipe dopes vary in their specific formulation and fluorescence.)

IMPORTANT

Alert the rig floor crew that pipe dope in the mud system can result in false QFT2™ shows. Pipe dope contamination is to some extent an avoidable problem.

Asphaltic additives, such as "Soltex™" or "Black Magic™", also give false shows; in this case low apparent API gravity. IPA extracts will give considerably less response than heptane extracts due to limited solubility of

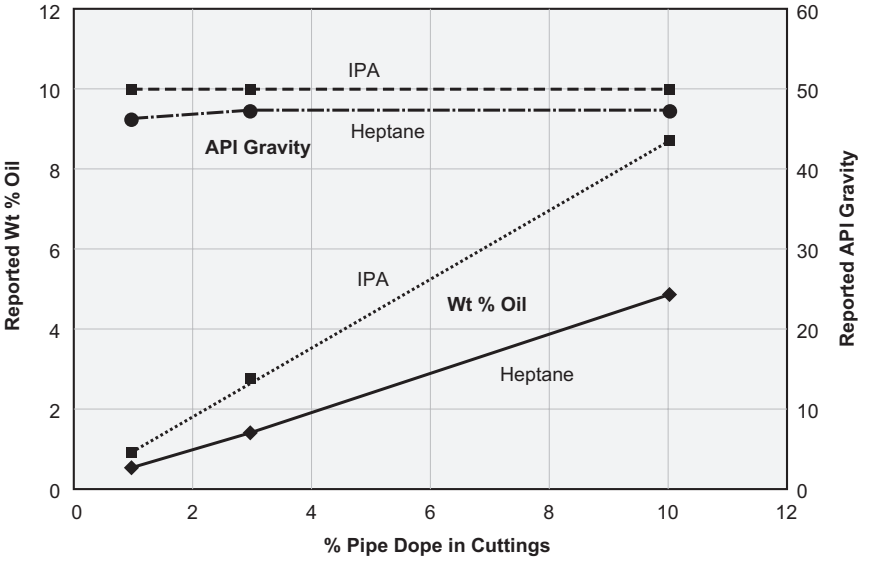


Figure 20
Effect of Pipe Dope Contamination on QFT2™ Results

asphaltics in IPA, as shown in Figure 21. In some cases, you can see asphaltic additives under a microscope, and you can clean up the sample by hand.

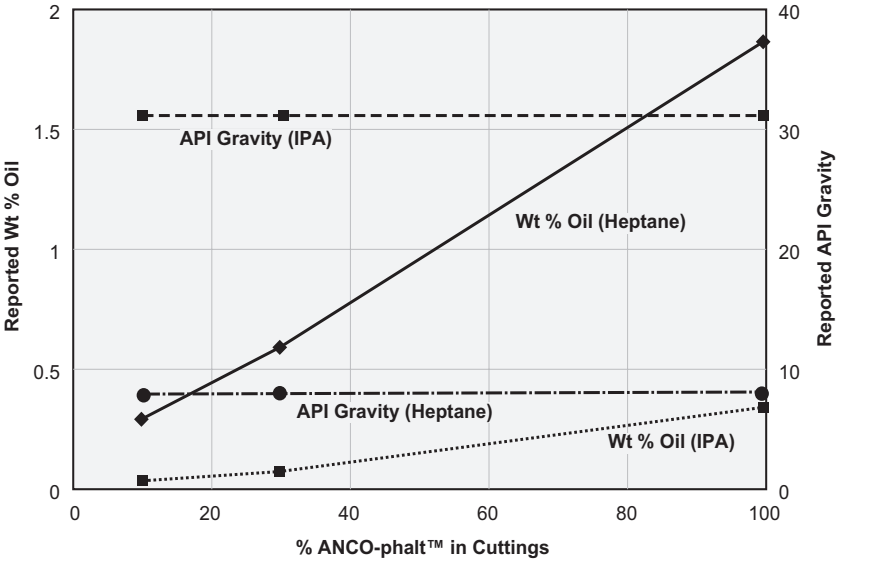


Figure 21
Effect of Asphaltic Additive on QFT2™ Results

IMPORTANT

If at all possible, avoid adding fluorescing hydrocarbons such as diesel or crude oil to the mud. These additives will create major problems for QFT2™, as shown in Figure 22.

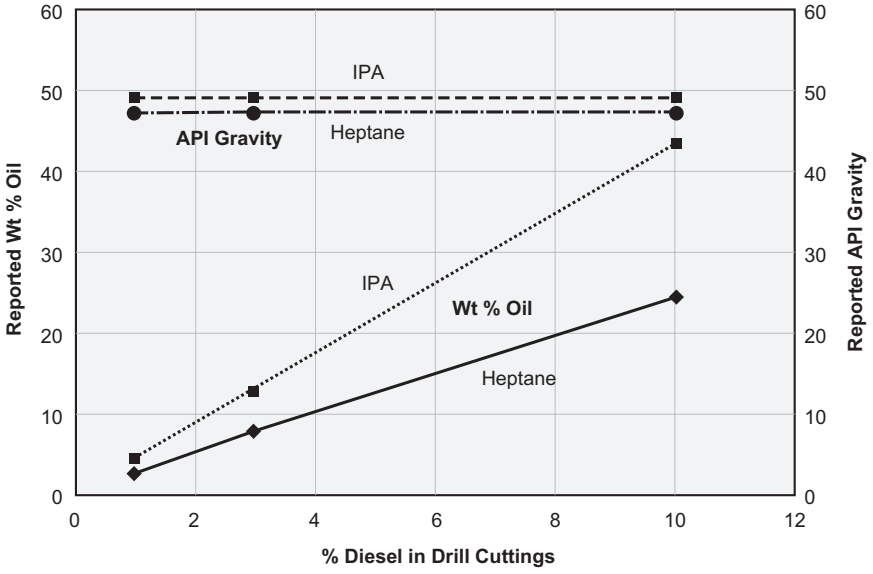


Figure 22
Effect of Diesel Addition on QFT2™ Results

Coal Beds and Non-Producible Asphalt

Non-producible formation hydrocarbons contain soluble aromatics and may give false QFT2™ shows. The QFT2™ response for coal is variable in both estimated oil quantity and API gravity depending on the coal maturity. Formation asphalt gives much the same QFT2™ response as asphaltic mud additives (low estimated API gravity, greater heptane solubility). You can usually detect these sources of QFT2™ interference by visually examining the sample. Detection of coal is often accompanied by dry gas (predominantly methane), whereas formation asphalt may have little or no associated gas.

The QFT2™ Data Logger Program does not recognize specific formation types; it gives API gravity based on fluorescence readings. The Wt % Oil for 100 mg/ml coal in heptane (0.5 g sample in 5 ml solvent) is no greater than typical background fluorescence values, as shown in Table 3.

Loss of Volatile Hydrocarbons

Though most fluorescing aromatics are of relatively low volatility, excessive drying time and temperature will cause loss of the lighter components. This is particularly true for condensates, in which the majority of the aromatics

Table 3
QFT2™ Response for Coals

Table 3. QFT2™ Response for Coals

Coal Type	Wt % Oil	API Gravity
Peat	0.020	45.0
Bituminous	0.010	0.0
Lignite	0.024	22.9
Anthracite	0.004	35.2

are small single-ring compounds. To minimize losses, use wet IPA extraction because the drying step is avoided altogether. When using heptane solvent, use air drying rather than heat lamp or oven drying.

You can use QFT2™ for after-the-fact analysis of dry samples which have been stored many years. Be aware however, that many light components have been lost and the API gravity estimates will likely be low for light oil zones.

Oil Base Mud

Using oil base mud severely limits the ability of QFT2™ to unambiguously detect formation hydrocarbons. Even though modern synthetic base muds have low fluorescence when newly prepared, the base oil acts much like the QFT2™ extraction solvent. As cuttings are circulated out (a process which may take several hours) any formation oil present tends to be extracted into the mud, particularly for permeable cuttings of commercial interest. Because the fluorescing components are of generally low volatility, they remain in the mud system and are recirculated thus causing a gradual rise in QFT2™ background fluorescence. Once the mud becomes contaminated, distinguishing the source of QFT2™ readings becomes problematic. Apparent QFT2™ shows are often caused by increases in sample porosity or mud retention rather than by actual formation hydrocarbons.

Interpreting Weight Percent Oil Values

In light of the many factors that can affect your data (see *Factors Affecting the Data* earlier in this chapter), you should become familiar with the following general guidelines for interpreting Wt % Oil values.

In the absence of contaminants, the Wt % Oil values will usually be within a factor of two of the actual cuttings oil content. How well this value represents the in-place oil content depends mainly on the degree of formation flushing and how well the cuttings retained oil as they were circulated out. In general, the estimated Wt % Oil will most often be a lower bound to actual in-place oil.

During overbalanced drilling, you can expect flushing in permeable zones. A high Wt % Oil estimate in overbalanced intervals may thus indicate a tight formation rather than producible hydrocarbons. In this case, you should make visual permeability ratings with particular care to help determine the significance of the high QFT2™ reading.

To convert Wt % Oil to the more commonly used Volume % Oil (oil porosity), multiply by the rock bulk density, and divide by the oil density. Calculate the oil density from API gravity using the following equation:

$$\text{Oil Density} = \frac{141.5}{(131.5 + \text{API Gravity})}$$

where:

Oil Density = g/cc

Because the Wt % Oil estimate is subject to considerable uncertainty, using 2.5 g/cc for the rock density will suffice for most situations. For example, if QFT2™ gave a Wt % Oil estimate of 1.8% and an API gravity estimate of 31, then you would estimate the corresponding oil porosity as follows:

$$\text{Oil Density} = \frac{141.5}{(131.5 + 31)} = 0.88\ \text{g/cc}$$

$$\text{Oil Porosity} = \frac{(1.8\%)(2.5)}{(0.88)} = 5.1\%$$

To get an even quicker (but rougher) estimate, ignore oil density variations altogether and simply use a combined multiplier of 3 (giving a result of 1.8% x 3 = 5.4% for the previous example). Given the joint uncertainties in the Wt % Oil estimate and in the fraction of retained oil, this simple "Wt % Oil times 3" calculation for estimating Volume % Oil is usually all that is warranted.

IMPORTANT

In general, total gas and estimated Wt % Oil should track to some extent. When comparing the gas reading and the QFT2™ reading, remember that QFT2™ measures the hydrocarbons that are *retained* in the cuttings, whereas the gas detector measures hydrocarbons that have been *released* to the mud. Ideally the liquid hydrocarbons (measured by QFT2™) would remain with the solids while the gas phase hydrocarbons (measured by gas detection) would be evolved. In practice, however, this gas/liquid phase partitioning will be imperfect; the major problem is loss of oil from the cuttings. The gas reading and the QFT2™ reading will, however, have similar responses to formation flushing.

Unless the cuttings oil retention is extremely poor, a gas increase with little QFT2™ increase is characteristic of high GOR shows. For this case, you would expect a high estimated API gravity. Conversely, heavy oil is indicated when a QFT2™ increase is accompanied by only a small gas increase. The extreme cases of no QFT2™ change during a gas show or the opposite almost always signals an equipment or analysis problem. For example, pipe dope contamination will cause a QFT2™ increase and a high API gravity estimate with no accompanying gas. If the API gravity estimate is not high, then a gas detection problem is likely.

The API gravity estimate has a standard deviation of 8 for oils of at least 20^{o} API. Heavy oils present special problems for wet sample IPA extracts and may give very high estimates. These oils, however, have yellow extracts when present in concentrations of commercial interest. In this case, you should use the dry sample heptane extraction method to obtain more reliable results.

Even though the API gravity estimate is subject to uncertainty, trends in oil character will usually be indicated correctly. In particular, changes in estimated API gravity can signal reservoir horizons and gas/oil or oil/water contacts.

Presenting Data on Well Logs

QFT2™ analysis provides two numbers: **Estimated Wt % Oil** based on total cuttings weight and **Estimated API Gravity**. When presenting and discussing these values, always include the qualifier “estimated”, because QFT2™ does not provide the same reliability as laboratory determinations of these quantities. QFT2™ provides a considerable advance over other rig-site fluorescence techniques such as use of the UV-box or single wavelength fluorometers. Results are given in numeric terms immediately meaningful to petroleum engineers rather than as subjective ratings or relative fluorescence units.

You should plot the estimated Wt % Oil and API gravity on the log as histograms in a single track. The Wt % Oil histogram should fill towards 0% or as the heavier line so that it is visually dominant. Present the Wt % Oil scale so that you can plot most of the values without rescaling. Normally a scale of 10^{o} to 60^{o} API gravity will effectively present the API gravity results when 10 across-track grids are used.

An example QFT2™ log track is shown in Figure 23. For zones having very low QFT2™ readings, the gravity estimate may be erratic because of the weakness of Filter A fluorescence. Since these zones are not likely to be of commercial interest, suppressing the gravity estimate may result in a more readable log. When possible, place the QFT2™ track adjacent to the lithology and total gas tracks because these are all closely related surface measurements.

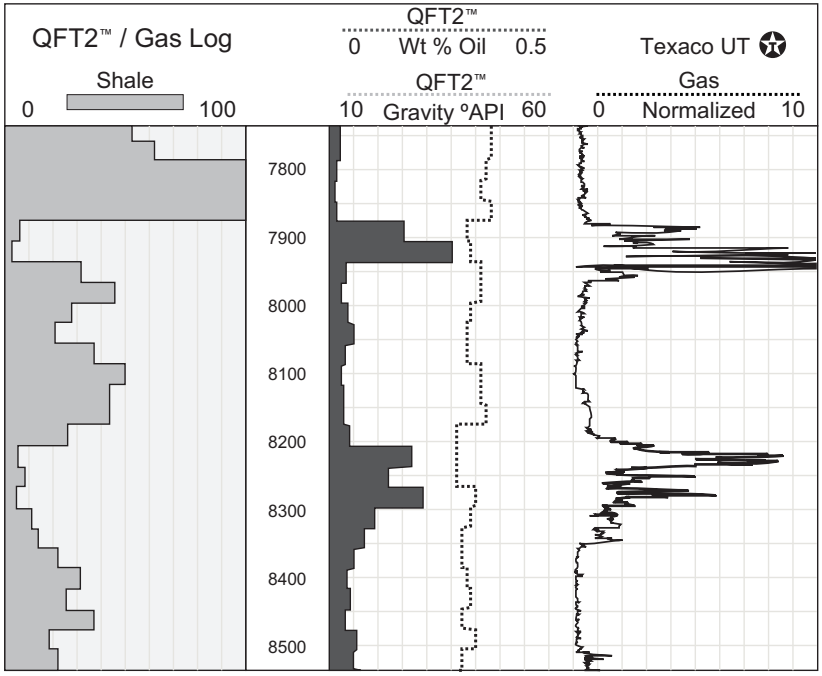


Figure 23
Recommended QFT2™ Log Track Presentation

Chapter 4

Getting Started

This chapter provides the information you will need to set up and prepare the QFT2™ system for field operation. To achieve the best results with this system, follow the instructions in each section below:

- ➢ **Setting Up the Equipment**
- ➢ **Using the Fluorometer**
- ➢ **Preparing to Acquire Sample Data**

Setting Up the Equipment

The QFT2™ technique requires two work areas—a sample preparation area containing solvent and extraction related items and an analysis area containing the 10-AU Fluorometer and a PC running the QFT2™ Data Logger Program. The following guidelines will help you in setting up equipment for each of these areas. For detailed information on QFT2™ equipment and supplies, see Chapter 7.

Sample Preparation Area

Detailed layout of the sample preparation area will depend on the amount of space available. Items in this area include:

- One or four liter solvent bottle equipped with dispenser pump
- Teflon® solvent wash bottle for rinsing reusable equipment
- Polypropylene flask with funnel for collecting waste solvent
- Large sealed metal can for long term waste solvent holding
- Spin dryer for removing excess water from samples
- Mortar and pestle (or hammer) for grinding samples
- Mechanical test tube mixer to aid in dispersing samples
- Test tube rack for solids settling and extract filtration
- Disposable supplies—test tubes, filters, paper towels, etc.

CAUTION

Make sure the area has good ventilation and does not block access to fire extinguishing equipment. If the area is not at the end of the logging unit, make sure fire extinguishers are available at either side. The solvents used for QFT2™, n-heptane and isopropyl alcohol (IPA), are volatile and quite flammable. Tightly seal solvent containers. Clean up any spills promptly to avoid accumulation of fumes. For further information on safety and health precautions for using these solvents, see the Material Safety Data Sheets (MSDS) in Chapter 8.

Analytic Equipment Area

The analysis area, which contains the 10-AU Fluorometer and data acquisition PC, requires a minimum of about 3 ft (1 meter) of bench space if you use a notebook PC placed on top of the fluorometer. You should place the polypropylene flask nearby on the floor to collect the waste from the fluorometer flow cell. Make sure the flask has a tightly fitting stopper through which the flow cell drain line passes to avoid large spills should the flask overturn. Check the flask at frequent intervals to ensure that it does not overflow.

Before using the equipment, review the Turner Designs 10-AU Manual and the QFT2™ Data Logger Program HELP FILE. When the program is running, you can display the HELP FILE on the PC screen by using the F1 key. But you may find it more convenient to refer to the HELP FILE in Chapter 6.

The 10-AU Fluorometer has its own internal microprocessor. The 10-AU screen and keypad are used for display and operator control functions. The HOME screen showing a bar graph of current fluorescent intensity will normally be displayed. To access control features, press the ENT key for the MAIN MENU display. All 10-AU variables and screens are identified by the sequence of keys required to access them from the MAIN MENU. For example, you access screen #2.11 (Run Blank) by keying '2' at the MAIN MENU followed by '1' and then another '1' at subsequent screens. Section 2 in the Turner Designs Manual contains a figure showing most of the 10-AU screens and variables.

For installation and general features of the QFT2™ Data Logger Program, see the HELP FILE, Appendix A (Chapter 6). Once you have positioned the Fluorometer and PC, follow the procedure in HELP FILE, Section 1 to set up the communications link between the 10-AU and the QFT2™ Data Logger Program.

IMPORTANT

If you have a new 10-AU or have just installed the Dual Filter Retrofit to an older instrument, you must also make the optimization and sensitivity adjustments to the flow cell (detailed in the HELP FILE, Appendix B) before you calibrate the instrument and run samples.

Using the Fluorometer

The Turner Designs 10-AU Dual Filter Fluorometer is a rugged field instrument specifically designed for QFT2™ applications. Unlike earlier models, it has two emission filters. You select these filters using the A and B position switch on the front of the sample compartment. The A and B filters provide measures of the single-ring and three-ring aromatic fluorescence of extracted oils. A suite of oils of widely differing compositions was used to develop equations for estimating oil content and API gravity from the two readings. For more information on QFT2™ calculations, see the HELP FILE, Section 5.

CAUTION

Before you begin to operate your Turner Designs 10-AU-015 Fluorometer, read and follow the following important safety instructions:

- Disconnect the power supply. When opening the instrument for any reason, disconnect from the power source to avoid electrical shock.
- Protect your eyes form UV radiation. Wear UV-blocking goggles when you operate the system with the cover removed.
- Use caution when working with the Clear Quartz Lamp. The lamp can break if handled improperly. Wear goggles, gloves, and protective clothing during the entire replacement procedure.
- Do not touch the quartz window. If you accidentally touch the front surface of the lamp, remove the fingerprints with a clean cloth.

Running Samples

To introduce fluid samples to the fluorometer, use the flow cell assembly, which consists of a clamp-on syringe barrel holder and valve connected via Teflon® lines to a vertical 1 mm ID quartz tube. The tube is mounted at the left of the sample compartment in a removable black plastic cylinder having entry and exit UV light slits. You must always remove this cylinder to access the compartment interior to replace the UV lamp or to perform other maintenance, because the cylinder prevents the compartment front face from being removed. For the procedure to optimize quartz tube positioning, see the HELP FILE, Appendix B.

When running samples, pour a minimum of four milliliters (ml) of sample into the flow cell syringe barrel with the valve lever vertical (closed). Turning the lever horizontal allows fluid to drain into the flow cell. The first two ml of sample are used to purge the quartz tube of rinse solvent and bubbles. An

additional one ml is used while taking readings; the final one ml may be required for sample dilutions. When dilutions are needed, use a one ml pipette to recover undiluted sample from the syringe barrel.

Rinsing the Flow Cell

Immediately after using the flow cell assembly, you should always rinse it using the solvent wash bottle to prevent residue build-up in the quartz tube (see *Preparing to Acquire Sample Data*, later in this chapter). Normally, you should leave the flow cell lines filled with solvent. If you allow the lines to drain, refill them with a few ml of solvent before running samples. This step may require using a plunger in the syringe barrel to initiate flow.

Adjusting Sensitivity

UV excitation is provided by a mercury (Hg) lamp in the right side of the front compartment. The lamp output is sampled by the 10-AU electronics via an internal fiber optic tube to compensate for variations in lamp intensity. When you set up the fluorometer, you may need to set the overall electronic gain of the fluorometer by adjusting the "Sensitivity Adjustment Knob" (actually a large slotted screw head) on the right side of the keypad near the power switch. This knob controls the lamp intensity fed into the fiber optic tube. For this adjustment procedure, see the HELP FILE, Appendix B.

Emission Filters

Between the mercury lamp and the quartz sample tube is a filter which isolates the desired excitation radiation. This light then passes through a narrow slit in the tube holder and irradiates the sample fluid. The light path between the filter and tube holder is shielded to minimize background radiation in the sample compartment. A similar slit in the tube holder is located at 90^{o} to the excitation slit towards the fluorometer back. This slit directs fluorescent radiation to the switchable emission filters. The filtered emission is then measured by a photomultiplier tube in the rear of the fluorometer.

Using Range Settings

To increase its dynamic range, the 10-AU has 3 range settings, LOW (x100), MED (x10) and HIGH (x1), each differing by a photomultiplier gain of 10. Except when calibrating, you should always operate the 10-AU in its AUTO range mode so that the 10-AU can automatically switch to the best range setting. When the 10-AU switches ranges, you may need to wait up to a minute for the new gain setting to stabilize.

Preparing to Acquire Sample Data

Before you begin the process of obtaining sample readings with the fluorometer, you should first follow the general instructions in this section.

1. Prepare cuttings and core extracts using the procedures in *Preparing Samples* in Chapter 5.

2. Go to the CONFIGURATION screen of the QFT2™ Data Logger Program to specify a data file name and other job specific variables. For detailed procedures, see the HELP FILE, Section 2.

3. Calibrate the 10-AU by using the QFT2™ standard solution to obtain a reading of 500 for filter B. This solution contains the single ring aromatic p-xylene for Filter A emission and the three ring aromatic phenanthrene for Filter B emission.

   For calibration procedures, see the HELP FILE, Section 3.

   The standard used for QFT2™ calibration consists of 200 mg/l of a p-xylene/phenanthrene mixture dissolved in n-heptane. The mixture has 50 parts by weight of p-xylene (for 287 nm fluorescence) to one part of phenanthrene (365 nm). For proper results, make sure both are of the highest available purity (99.5+ %). The standard will normally be supplied in a 100 ml dark glass bottle. Keep this bottle tightly sealed between uses to avoid loss of the heptane solvent. A complete system calibration (see *Calibration and Solvent Blank Readings* in Chapter 6) requires about 10 ml of standard. Never dilute the standard to "stretch" usage because UV absorption causes a non-linear response. For more information on the QFT2™ standard, see Chapter 7.

4. Use the QFT2™ Data Logger Program to capture sample readings for both filters for the standard and for solvent blanks.

IMPORTANT

Oil fluorescence at Filter A is usually quite weak, sometimes less than 1/100 of the Filter B reading. Thus, it is critical that you make accurate blank readings. (For step-by-step calibration procedures, see the HELP FILE, Section 3.)

5. Rinse the standard solution thoroughly from the flow cell assembly using the solvent wash bottle. Rinse with heptane, IPA, distilled water, IPA, and finally again with heptane. You must alternate the heptane and distilled water

rinses with IPA rinses because of the immiscibility of heptane and water.

You can usually tell that the flow cell needs cleaned when you see an increase in solvent blank readings. This is especially true when the samples contain a lot of salt.

6. To use the QFT2™ Data Logger Program to acquire sample readings, see the HELP FILE, Section 4.

7. Once you have captured the sample readings, go to the CALCULATE/ UPDATE Data Logger screen (see the HELP FILE, Section 5) to calculate estimated oil quantities and API gravity. This screen also shows estimated UV absorption corrections and notes when sample dilution is required.

- If dilution is not required, instruct the program to update the data file with the sample results.

8. To back-up the file or to use it for off-line data analysis, you can copy the file to a diskette while the program is running. For information on data file copying and file management, see the HELP FILE, Section 6.

■■■■■

Chapter 5

Preparing Samples For Analysis

After you have set up your work areas and analysis equipment (see Chapter 4), the next, and most critical step in the QFT2™ process is capturing and preparing representative formation samples for analysis. The following sections will help you to understand the analysis process, the supplies you will need, and the step-by-step procedures for collecting and preparing your samples for analysis:

- **Overview of the Sampling Process**
- **Equipment and Supplies**
- **Preparing Samples**

Overview of the Sampling Process

To ensure that your samples generate meaningful QFT2™ data, you should understand the underlying basis for the sampling procedures detailed later in this chapter. This section provides useful background information and general guidelines to assist you in collecting your samples and selecting an oil extraction method.

Collecting Samples

The first step in QFT2™ analysis is to obtain a formation sample representative of the depth interval specified by your client. You should follow the proper procedures for collecting and labeling samples that are representative of the formation drilled.

To minimize loss of moveable oil, make sure your samples are subjected to minimal handling. In most cases, you should spin-dry a portion of the collected sample without washing it with water. The amount of sample actually used for analysis is quite small (0.5 g dry weight), but make sure you obtain a representative portion free of obvious contaminants. You need not grind samples to a fine powder before extraction because, in general, only moveable

oil is of interest. Make sure, however, that particle size is small enough so that you can take a representative, uniformly mixed sample for analysis.

Selecting an Oil Extraction Method

To extract oil from QFT2™ samples for analysis, you must select either the dry sample extraction method (using n-heptane) or the wet sample extraction method (using isopropyl alcohol—IPA). The equations used for QFT2™ data reduction differ depending on which solvent is used, both to allow for solvent dependent fluorescence quenching effects and to correct for the differing solubilities of whole oils in heptane and IPA. Thus, you must inform the QFT2™ Data Logger Program which solvent (heptane or IPA) you used for extraction.

Heptane and IPA solvents must be of spectral grade purity because trace contaminants can contribute significant background fluorescence. Typically, solvents are purchased in liter or larger size bottles. Make sure the bottle is fitted with a calibrated dispensing pump. The advantages and limitations of both extraction methods are described below.

Dry Sample Extraction Using Heptane

Heptane is the preferred extraction solvent for QFT2™ analysis because of the lower solubility of oils in IPA. Using liquid chromatography, oils can be separated into four solubility fractions: saturates, aromatics, resins and asphaltenes. The asphaltenes are soluble in neither heptane nor IPA, and the resins are only partly soluble in IPA, as shown in Table 4. The QFT2™ data reduction equations attempt to correct for these effects. Oils are highly variable; however, and in the case of heavy oils, the corrections may perform poorly.

Table 4
Oil Solubility Fractions

Table 4. Oil Solubility Fractions

Oil Fraction	UV Fluorescent	Heptane Soluble	IPA Soluble
Saturates	No	Yes	Yes
Aromatics	Yes	Yes	Yes
Resins	Yes	Yes	Low
Asphaltenes	Yes	No	No

The general procedure for using the dry sample extraction method is as follows: After collecting and drying the samples, extract a measured weight of sample with a measured volume of solvent. The standard QFT2™ extraction, as originally developed, uses 5.0 ml of n-heptane to extract 0.50 g of air dried, lightly ground solids. If you do not have a 0.01 g resolution balance (preferred), use a porcelain spoon that holds approximately 0.5 g of solids. To avoid loss of volatile oil components, dry the samples at low temperatures. The drying step is essential because of the immiscibility of heptane and water. To speed the drying process, avoid using a large excess of sample and place the drying dish in a well ventilated, warm area. Using heptane for extractions has two practical limitations. First, the required low drying temperature delays analysis of samples. Second, logging units lack the space needed to dry the samples. During periods of rapid drilling many samples in various stages of dryness can accumulate.

Make sure heptane is always available in a Teflon® (not plastic) squeeze bottle for rinsing out the 10-AU flow cell. Heptane aids in removing residues that are only marginally soluble in IPA. It also flows through the cell more rapidly than IPA (because of its lower viscosity).

Wet Sample Extraction Using IPA

To avoid the need for drying, the isopropyl alcohol (IPA) wet sample extraction method was developed. In this process, you remove as much water as possible from the solids (normally by spin-drying), and then you use the porcelain spoon to obtain a standard volume of sample.

Experience has shown that, over a wide range of sample consistency, the actual solids content of well drained wet samples varies only over the fairly narrow range of 60-70%. Depending on the sample consistency, you may need to grind the sample to obtain a representative sample.

At the rig site, using IPA often is more practical than using heptane because you can extract wet samples (due to the miscibility of IPA with both oil and water). For rapid well site analysis and to avoid loss of volatiles, analyze the samples in IPA. Confirm API gravity estimates and Wt % Oil on important samples (zones of interest and shows) using the heptane extraction method. If you see a decrease in estimated API gravity, regard the heptane results as the more reliable ones.

The general procedure for the wet sample extraction method involves dispersing the contents of the spoon in IPA (normally 5 ml) and then following the same procedure used for heptane extraction. Keep in mind, you must

inform the QFT2™ Data Logger Program which solvent (heptane or IPA) you used because the equations used for data reduction are solvent dependent.

IMPORTANT

Wet samples can range in consistency from sticky muds to easily handled grains. Make sure fine grain samples are well dispersed in the solvent for complete oil extraction. Dispersing gumbo-like samples in IPA may require a long period of vigorous agitation, which is difficult when shaking samples by hand. You may need to use a mechanical agitation device, such as a "Vortex" test tube mixer. In addition, gumbo dispersions may settle so slowly that excessive time is required to obtain a clear, easily filtered supernatant. In these cases, you should use 10 ml rather than 5 ml of IPA.

Even if you use the IPA method, make sure heptane is always available in a Teflon® (not plastic) squeeze bottle for rinsing out the 10-AU flow cell. Heptane aids in removing residues that are only marginally soluble in IPA. It also flows through the cell more rapidly than IPA (because of its lower viscosity).

IMPORTANT

Never dispense IPA from a squeeze bottle (even if it is Teflon®) because trace chemicals (probably plasticizers) with very high UV absorbance are extracted. Use only glass containers for storing and dispensing IPA. If heptane is not available, you can use IPA for flow cell rinsing. However, slightly more sample will be required to obtain a steady reading. (The lower viscosity of the heptane rinse allows the sample to displace it much more easily than IPA.) You may store IPA for rinsing in a squeeze bottle. However, you should always take IPA used for blank readings from the solvent dispenser used for sample extractions.

Calculating the Dilution Factor for Solvent Extracts

When you prepare solvent extracts using standard quantities of 0.5 g (or one level spoonful) per 5 ml solvent, the "Dilution Factor" of the solvent extract is defined as 1.00. To calculate initial dilution factors for non-standard extracts, use the following equation:

$$\text{Dilution Factor} = \frac{(0.1)(\text{ml solvent})}{(\text{g sample})}$$

For example, if 10 ml of solvent is used for 0.5 g of sample because of poor solids settling, then the initial dilution factor is calculated as shown below:

$$\text{Dilution Factor} = \frac{(0.1)(10)}{(0.5)} = 2.00$$

If 5 ml of solvent is used to extract 1 g (or 2 spoonfuls) of sample to obtain doubled sensitivity, then the initial dilution factor is calculated as shown below:

$$\text{Dilution Factor} = \frac{(0.1)(5)}{(1)} = 0.50$$

If the extract is high in oil content, the QFT2™ program may inform you that sample dilution is recommended or required. This situation occurs when the calculated UV absorption corrections are excessive. Usually a 10 to 1 dilution will suffice to bring the extract concentration within range. To obtain a 10 to 1 dilution, use a pipet to obtain 0.50 ml of the extract, and then dilute the extract with 4.5 ml of the solvent. The new dilution factor for this case would be 10 times the old or initial dilution factor.

In general, you can calculate the new dilution factor using the following equation:

$$\text{New Dilution Factor} = \frac{(\text{Old Factor})(\text{New Total ml})}{(\text{ml Diluted})}$$

For example, if the old dilution factor is 2.00 and 0.20 ml of the extract is diluted with 4.8 ml of solvent, then the new dilution factor is calculated as shown below:

$$\text{New Dilution Factor} = \frac{(2)(0.2 + 4.8)}{(0.2)} = 50$$

As another example, because the solvent dispenser is set to deliver 5 ml, you may find it easier to make the usual 10 to 1 dilution by diluting 0.56 ml of extract with 5 ml of solvent, as shown below:

$$\text{Dilution Factor} = \frac{(5.00 + 0.56)}{(0.56)} = 10$$

As a final example, assume that the QFT2™ program indicates that dilution is required, but that you can only pipet 0.43 ml of extract from the 10-AU flow cell syringe. To obtain a 10 to 1 dilution, you should add 3.9 ml (9 x 0.43 = 3.9) of solvent because:

$$\text{Dilution Factor} = \frac{\left((9)(0.43)+0.43\right)}{(0.43)} = 10$$

When the initial extract has a distinct oil coloration, you should dilute the extract regardless of the QFT2™ program messages. The equations used to correct for UV absorption losses are only approximate. Under unusual circumstances, the readings may actually decrease at high oil concentration because of very high losses.

Equipment and Supplies

Table 5 and Table 6 show the equipment and supplies you should have on hand to prepare and analyze QFT2™ samples. For more detailed information and sources for equipment and supplies, see Chapter 7.

Table 5
Equipment for Preparing and Analyzing Samples

Table 5. Equipment for Preparing and Analyzing Samples

Item	Description
• Spin Dryer	For removing excess water from sample
• Mortar and Pestle (or hammer)	For grinding samples
• Porcelain Spoon	For measuring wet and dry samples
• Weighing Balance (Optional)	For measuring dry samples
• Marking Pen	For identifying dishes and test tubes
• Narrow Spatula	For transferring samples and mixing samples in test tubes
• Solvent Dispenser	5 ml hand pump fitted to solvent bottle
• Mechanical Mixer	For dispersing samples (Vortex or equivalent type)
• Test Tube Rack	For sample settling and filtration
• 10 ml Glass Syringe	For sample filtration (Leurlock)
• 5 ml Glass Syringe	For 10-AU flow cell (Leurlock)
• Teflon® Wash Bottle	For rinsing solvent off reusable items
• Pipet Hand Pump	For pipetting operations
• Flask with Funnel	For waste solvent (Polypropylene)

For more information, see Chapter 7.

Table 6
Supply Cost Per Sample

Table 6. Supply Cost Per Sample

Item	Description	Approximate Cost per Sample ($)*
• Spectral Grade n-Heptane	Dry extracts and equipment rinsing	0.25 Assumes 10–15 ml average usage
• Spectral Grade IPA	Wet extracts and equipment rinsing	0.25 Assumes 10–15 ml average usage
• QFT2™ Standard	10-AU calibration	
• Aluminum Drying Dishes	50 ml, 57 mm ID for dry extracts	0.12
• PTFE Syringe Filters	0.45 micron pore, non-sterile 13 mm diameter for heptane extract, 25 mm for IPA	0.90**
• 16 x 125 mm Test Tubes	Large (15 ml) test tubes for sample dispersions and dilutions	0.05
• 12 x 75 mm Test Tubes	Small (5 ml) test tubes for filtered extracts	0.03
• Seriological Glass Pipets	1 ml, 0.01 ml graduations for sample dilutions	0.15
		$1.50 Total

* Actual costs depend on supplier and quantities purchased. For more information, see Chapter 7.

** Note that the PTFE filter is the major cost item. Because of the high cost of spectral grade solvents, rinsing and reusing glassware other than syringes usually is not cost effective.

Preparing Samples

If you are new to preparing QFT2™ samples and you have not yet read *Preparing to Acquire Sample Data* in Chapter 4, or *Overview of the Sampling Process*, (earlier in this chapter), you will find it useful to read these sections before proceeding. Once you are familiar with this background information, follow the step-by-step instructions in this section to collect and prepare your samples for analysis.

Collecting Samples

1. Collect cuttings from the shaker or sluice box that are representative of the QFT2™ logging interval, as shown in Figure 24.

- In most cases, do not screen or wash the QFT2™ sample.
- Ensure the cuttings samples are accurately correlated to the depths from which they came.

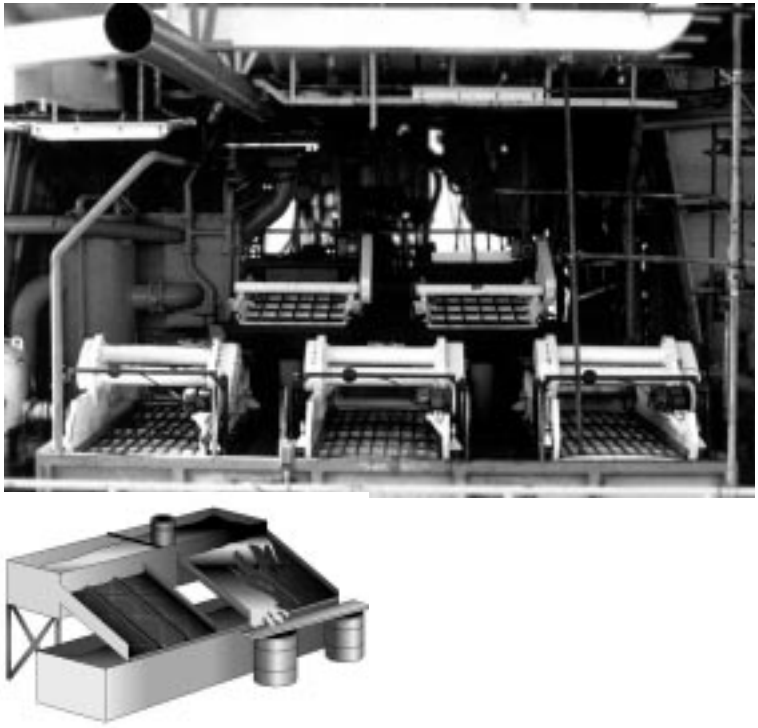


Figure 24
Collect cuttings from the shaker or sluice box that are representative of the QFT2™ logging interval

2. Spin dry a representative portion of the cuttings by doing the following:

a. Take a small representative portion of the sample and remove obvious contaminants such as LCM, cement chunks, and steel shavings. Place the sample in the screen basket, as shown in Figure 25a.

Figure 25a
Place sample in the screen basket

b. Place the screen basket in the drum of the spin dryer, as shown in Figure 25b.

Figure 25b
Place the screen basket in the drum of the spin dryer

c. Apply upward pressure on the drum to activate the switch. Continue until excess moisture is removed, as shown in Figure 25c.

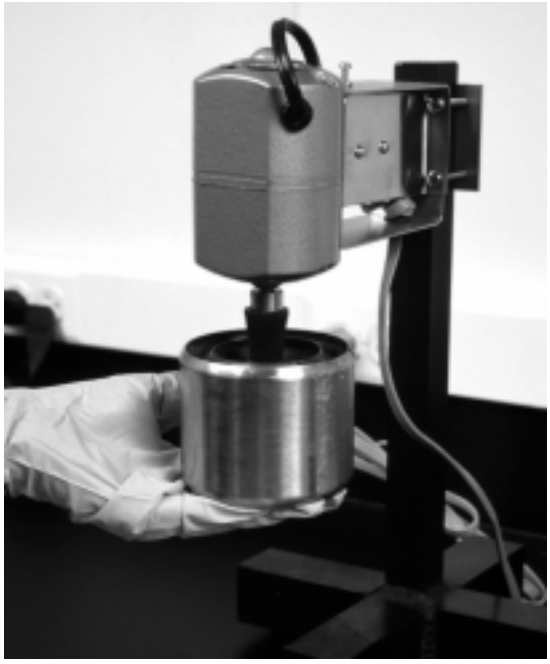


Figure 25c
Apply upward pressure on the drum to activate the switch

3. Select an oil extraction method, and proceed to either ***Dry Sample Extraction Using Heptane*** or ***Wet Sample Extraction Using IPA***.

To extract oil from QFT2™ samples for analysis, you must select either the dry sample extraction method (using n-heptane) or the wet sample extraction method (using isopropyl alcohol—IPA).

Wet sample extraction using IPA often is more practical at the rig site because you can extract wet samples (due to the miscibility of IPA with both oil and water). For rapid well site analysis and to avoid loss of volatiles, analyze the samples in IPA. Confirm API gravity estimates and Wt % Oil on important samples (zones of interest and shows) using the heptane extraction method. If you see a decrease in estimated API gravity, regard the heptane results as the more reliable ones.

Dry Sample Extraction Using Heptane

1. Transfer about 2.5 grams or three porcelain spoonfuls of sample to an aluminum weighing dish, spread the sample out to maximize air contact, as shown in Figure 26. Place the dish in a warm, well ventilated area until dry.

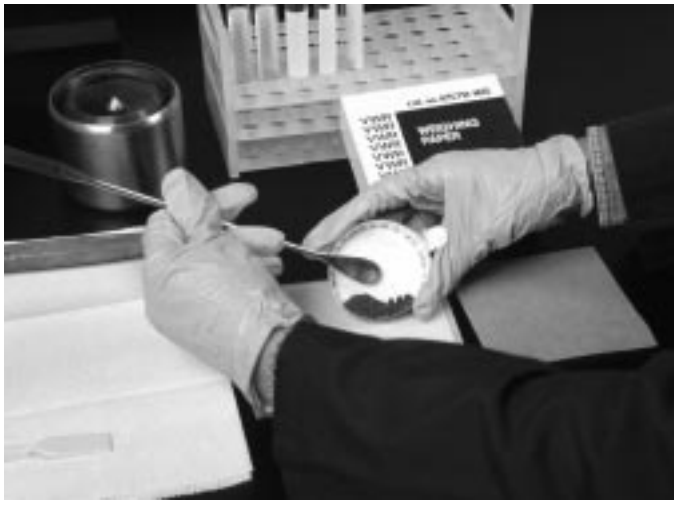


Figure 26
Transfer 2.5 grams or three porcelain spoonfuls of sample to an aluminum weighing dish

IMPORTANT

Never heat the sample using a heat lamp or oven. Heating drives off the volatile hydrocarbons, which can be significant for light oils and condensates.

2. Lightly crush or break up the dried sample, as shown in Figure 27 and Figure 28.

Figure 27
Lightly crush or break up the dried sample

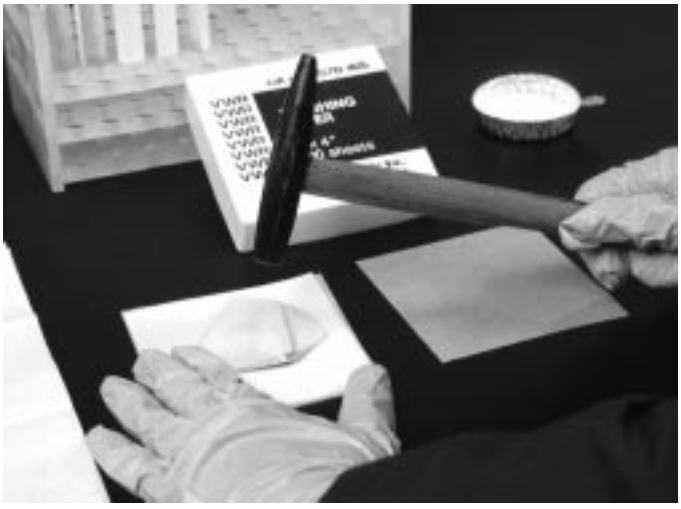


Figure 28
Lightly crush or break up the dried sample

3. Transfer 0.50 g or one level spoonful to a 16 x 125 mm test tube, as shown in Figure 29.

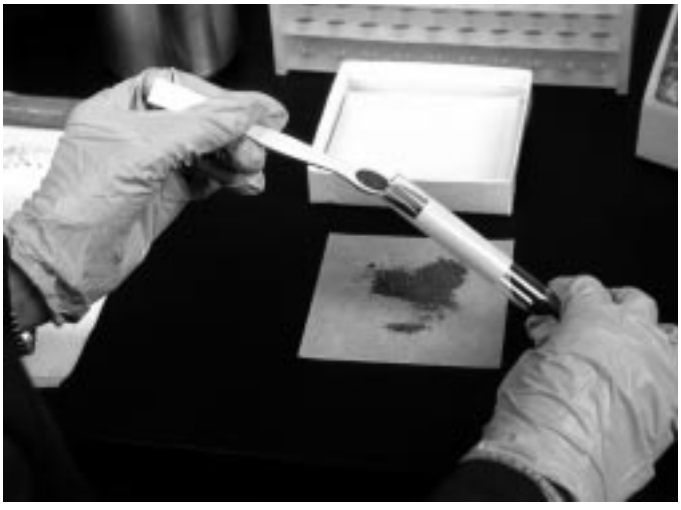


Figure 29
Transfer 0.50 g or one level spoonful to a 16 x 125 mm test tube

4. Add 5 milliliters (ml) of heptane using the solvent dispenser pump, as shown in Figure 30.

Figure 30
Add 5 milliliters (ml) of heptane using the solvent dispenser pump

5. Vigorously agitate the test tube for 30 seconds to extract the oil. A Vortex mixer or equivalent is recommended, as shown in Figure 31.

6. Proceed to *Filtering the Extract*.

Figure 31
Vigorously agitate the test tube for 30 seconds to extract the oil

Wet Sample Extraction Using IPA

1. If required, lightly grind the sample to aid solvent contact and to ensure sample uniformity.

2. Fill the porcelain spoon with sample and use the straight edge of a spatula to level the contents, as shown in Figure 32. Clean the exterior of the spoon with a laboratory wipe.

Figure 32
Fill the porcelain spoon with sample and use the straight edge of a spatula to level the contents

3. Place the spoon in a large (16 x 125 mm) test tube and add 5 milliliters (ml) of IPA by using the solvent dispenser pump, as shown in Figure 33.
   - If required, swirl the spoon to empty it of sample.
   - If swirling fails, use a small spatula to scrape the sample out of the spoon.
   - If the sample is sticky and difficult to disperse, add an additional 5 ml of IPA and use a small spatula to aid mixing as described in step 5.

Figure 33
Place the spoon in a large test tube and add 5 milliliters (ml) of IPA

4. Note the extract Dilution Factor to be entered in the QFT2™ program.
   - If you used 5 ml of solvent, enter a Dilution Factor of 1.00.
   - If you used 10 ml of solvent, enter a Dilution Factor of 2.00.

5. Vigorously agitate the test tube for a minimum of 30 seconds until the sample is completely dispersed, as shown in Figure 34.
   - Sticky samples may require using a Vortex mixer or equivalent.

     In this case, place a small spatula in the test tube and hold it firmly against the tube side using the edge of your finger while you are mixing. The spatula will both aid in dispersion and prevent the solvent from climbing to the tube top. Continue mixing until no mud clumps are visible.

Figure 34
Vigorously agitate the test tube for 30 seconds until the sample is completely dispersed

CAUTION

Using the Vortex mixer with a loose spatula in the test tube will likely break the test tube causing a solvent spill. Always hold the spatula firmly against the tube side with the edge of your finger and increase the mixing speed gradually.

6. Proceed to ***Filtering the Extract***.

Filtering the Extract

1. Place the test tube in the test tube rack and allow the solids time to settle.

- If the extract used only 5 ml of solvent and you fail to obtain at least 4 ml of supernatant within several minutes, mix in an additional 5 ml of solvent (see step 6) and allow it to settle once again.

2. Install the appropriate 0.45 micron PTFE filter on a clean, plungerless 10 cc glass syringe, as shown in Figure 35.

- For IPA, use a 25 mm filter
- For heptane, use a 13 mm filter

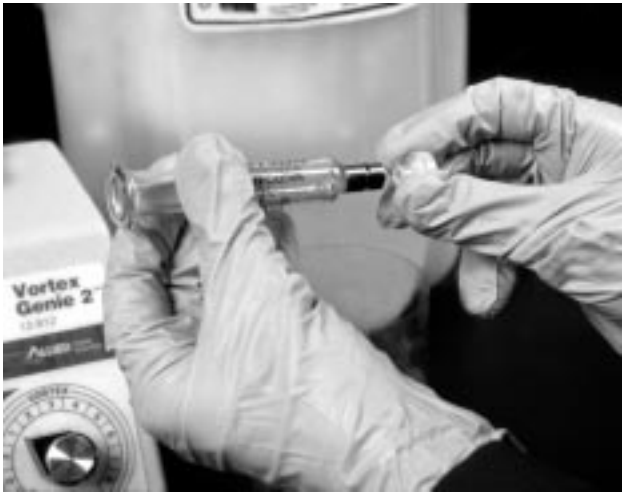


Figure 35
Install the appropriate 0.45 micron PTFE filter on a clean, plungerless 10 cc glass syringe

3. Place a 12 x 75 mm test tube in the test tube rack.

4. Insert the filter tip into the test tube, and pour the extract supernatant into the vertical syringe barrel, as shown in Figure 36. Try to avoid introducing particles because these may jam the syringe.

Figure 36
Insert the filter tip into the test tube, and pour the extract supernatant into the vertical syringe barrel

5. Insert the syringe plunger, and force the extract through the filter into the 12 x 75 mm test tube until the tube is nearly full (5 ml) or the syringe is empty, as shown in Figure 37. Four ml of filtrate is required for analysis.

Figure 37
Insert the syringe plunger, and force the extract through the filter into the 12 x 75 mm test tube

6. Note the extract Dilution Factor to be entered in the QFT2™ program.
   - If you used 5 ml of solvent, enter a Dilution Factor of 1.00.
   - If you used 10 ml of solvent, enter a Dilution Factor of 2.00.

7. Remove and discard the filter, as shown in Figure 38.

Figure 38
Remove and discard the filter

8. Rinse the syringe barrel and plunger with a few ml of solvent so they will be ready for the next sample, as shown in Figure 39.

- After processing ten samples, thoroughly clean syringes with hot, soapy water to remove fines, which can cause the syringe barrel to stick.
- Rinse the water-wet syringe barrel and plunger with IPA and air-dry them.

Figure 39
Rinse the syringe barrel and plunger with a few ml of solvent

Diluting the Samples

1. If you are running a series of samples, and your experience with the previous ones indicates that dilution will be required (extract color comparison, etc.), or if the QFT2™ Data Logger Program requires a dilution, do the following, as shown in Figure 40 and Figure 41.

a. Pipet 0.50 ml of the filtrate into a large (16 x 125) mm test tube.

b. Dilute with 4.5 ml of solvent and mix thoroughly.

Alternatively, you can use 0.56 ml of filtrate diluted with 5.0 ml of solvent.

Figure 40
Pipet 0.50 ml of the filtrate into a large test tube

Figure 41
Dilute with 4.5 ml of solvent and mix thoroughly

2. Note the extract Dilution Factor to be entered in the QFT2™ program.
   - The dilution factor to be entered is 10 times the extract dilution factor. For more information on calculating the dilution factor, see the HELP FILE, Section 4.

Analyzing the Samples

1. Your samples are now ready for analysis, as shown in Figure 42.
   - If you have not yet read *Preparing to Acquire Sample Data* in Chapter 4, you may find it useful to do so now. For the complete analysis procedures, see the HELP FILE, Section 4 and Section 5.

Figure 42
Your samples are now ready for analysis

2. If the QFT2™ Data Logger Program informs you that a dilution is required, do the following:
   - Remove 0.5 ml of the sample extract remaining in the flow cell assembly using a 1.0 ml pipet.
   - Dilute with 4.5 ml of solvent and mix thoroughly.
   - Calculate the new dilution factor using the equations in *Calculating the Dilution Factor for Solvent Extracts* earlier in this chapter.
   - Enter the new Dilution Factor into the QFT2™ Data Logger Program.
   - Thoroughly rinse the flow cell assembly with solvent.
   - Run the diluted sample.

■■■■■

Chapter 6

HELP FILE for the QFT2™ Data Logger Program

This chapter contains the same information as the on-screen HELP FILE in the QFT2™ Data Logger Program. The HELP FILE contains the following sections:

- **Fluorometer Configuration and COMM Link Setup**
- **The "System CONFIGURATION Inputs" Screen**
- **Calibration and Solvent Blank Readings**
- **The "Acquire SAMPLE Readings" Screen**
- **The "CALCULATE / File UPDATE" Screen**
- **Data File Copying and File Management**
- **Appendixes A–E**

Section 1

Fluorometer Configuration and COMM Link Setup

QFT2™ requires use of the Turner Designs Model 10-AU Dual Filter Mud Logging Fluorometer. The filter wavelengths must be 287 nm (Filter A) and 365 nm (Filter B). If you have a new 10-AU or have just installed a dual filter retrofit to an older 10-AU, the mechanical adjustments detailed in APPENDIX B should be performed. Operators unfamiliar with the 10-AU should review Section 2 (Operating Information) of the Turner Designs Manual for instructions on 10-AU keypad and screen usage.

By means of the 10-AU keypad and screen, enter the following:

Serial Data Baud Rate (#1.51):	**4800**
Interval (#1.52):	**1 second**
Subtract Blank (#2.12):	**NO**
Time Constant (#2.52):	**1 second**
Concentration Range Control (#2.43):	**AUTO**

Connect the serial data cable from the 10-AU to the PC (either COM1 or COM2 if both are installed) and confirm that data is received once per second.

Occasionally the UV lamp may misfire when the 10-AU is turned on. If the signal appears to be noisier than usual (as indicated by the PC screen plot), try cycling the power to the 10-AU.

NOTE that the 10-AU does not display on its HOME screen numeric values in excess of 999 (">999" will be shown). Internally however the 10-AU software can handle and transmit values well over 10,000. For some QFT2™ samples, readings of several thousand may be obtained for the 365 nm filter. These values will not be displayed on the HOME page but will be correctly transmitted (as shown by the PC screen "COMM Link:" item).

Section 2
The "System CONFIGURATION Inputs" Screen

Data File: EMMALEE3 **[.QF2 DOS ext]** **LIST FILES**

Well/Proj: Acme Oil Emma Lee #3 OCS-1560231

Analysts: PetroSearch Logging—KKS, SAH

QFT™ Solvent: Heptane **Instr. Corr. Factors**

Signal Avg: 20 **seconds** **287 nm:** 1.034 **[1.0]**

T/D: 08:15:34 15-May-98 **365nm:** 0.975 **[1.0]**

The CONFIGURATION PC screen contains job and data acquisition control operator inputs. When your updates are complete, key ESC to exit the screen. Then key "Y" at the following "Use Changes?" prompt to update the program configuration file and begin use of the entries.

Data File: Before acquiring data, the operator must specify a DOS file name for storing the results. You should use a descriptive name (up to 8 characters), such as the well name, so that the file is readily identifiable. Not all characters are allowed for DOS file names; the program will BEEP if you attempt to use an illegal one. Normally the same file should be used for the duration of the job. A DOS file extension of ".QF2" is always used for data files. Section 6 provides further file management details.

LIST FILES: This entry permits you to scan and optionally select from a list of the 25 most recently updated files. (Any files in excess of five indicates a need to move inactive files out of the QFT2™ program directory by means of the usual DOS commands so that they will not be "underfoot".) Once a file has been selected, the F2 key may be used to view it in case confirmation of the contents is needed.

Well/Proj: Specify the well or project identifier to be associated with the data file. This information is written to the file header and is also rewritten should you update it.

Analysts: Normally should contain the service company name and the names or initials of the loggers. This information is written to the file header and is also rewritten should you update it.

QFT Solvent: Specify the solvent, either heptane or IPA, to be used for sample extractions. This entry can be modified on a sample by sample basis by means of the "Acquire SAMPLE Readings" screen when you are using both solvents.

Signal Avg: Specify the averaging period to be used for the acquired fluorescence signal. A value of at least 20 seconds is recommended since the 287 nm readings can be quite weak (only slightly above the solvent blank) and subject to noise fluctuations. Averaging by the 10-AU itself should be disabled (set 10-AU item #2.52 to 1 second). An extremely noisy signal may indicate the need to replace the 10-AU UV lamp (also try cycling the 10-AU power to restart the lamp).

T/D: System Time/Date—All acquired values are tagged by the time and date shown at the screen upper right. Use this entry to change the PC time/date if it is incorrect. The Data Logger program is year 2000 compliant up to year 2080.

Instr. Corr. Factors for 287 and 365 nm: Sample intensities for both filters are divided by the Calibration Standard intensities before use in calculations. While this procedure removes most instrumental and filter efficiency effects, minor instrument to instrument differences remain because of variations in the spectral response of the filters and the 10-AU fluorometer. The instrument correction factors are instrument/filter set specific and will normally be determined by use of the procedure given in APPENDIX C before the 10-AU is shipped to the rig. When this is the case, the values will be noted by means of a label affixed to the 10-AU case. The allowed range for both factors is 0.5 to 2 but normally both will be close to 1. If the factors are not known, use values of 1 for both.

Section 3
Calibration and Solvent Blank Readings

QFT2™ calibration is a two stage procedure. First, by use of the QFT2™ standard solution, the 10-AU is calibrated on a stand-alone basis to give a reading of 500 units for the 365 nm filter. This procedure is identical to that documented for the older QFT™ technique with the exception that the standard value is specified as 500 rather than 800 units.

Once the 10-AU is calibrated, the QFT2™ program is used to capture 287 and 365 nm readings for heptane and/or IPA, and for the standard. Since the program directly uses the standard reading for data analysis, it is not critical that the 10-AU maintain precise calibration provided new blank and standard readings are obtained periodically. In practice, the 10-AU has proved to be quite stable. Particular care should be exercised when capturing the 287 nm solvent blank readings since most oils fluoresce only weakly at this wavelength.

3A. 10-AU Fluorometer Calibration

This section of the HELP file assumes the operator is familiar with the 10-AU keypad operation for selecting 10-AU screen numbers and items (designated by the codes #n.nn—for example item #2.43 is accessed by keying ‘2’ at the 10-AU MAIN menu followed by ‘4’ and then ‘3’ at following screens.)

1. Set the standard concentration to **500** (#2.2).

2. Set the concentration range control to **MANUAL** (#2.43).

3. Set the concentration range to **MED** (#2.42).

4. Select the **365 nm** filter (Position B).

5. Thoroughly rinse the flow cell with solvent (use heptane if available) until a steady blank reading is obtained. Acquire the blank reading by selecting #2.11 Run Blank. The Blank% value shown (screen #2.11) should be close to 0 and UP/DOWN adjustments should not be required. Press the "0" key while in screen #2.11 so that the 10-AU captures the blank value. Wait until you receive a "FINISHED" message (15 secs) before exiting the screen.

6. Drain the flow cell until the solvent just enters the syringe tip. (Avoid introducing air into the line or incorrect readings will be obtained.) Pour about 2 ml of standard into the syringe and again drain. Repeat. Then add 5 ml of the standard. Let 2 ml flow rapidly through the cell to expel any bubbles and to displace the solvent. (If IPA was used, add further 1/4 ml increments until a steady reading is obtained.) The PC screen plot can be used to follow the 10-AU readings; press F3 as required to rescale the plot. FIRMLY CAP the standard bottle to avoid solvent loss.

7. Acquire the standard reading by selecting 10-AU item #2.3 Run Standard. DO NOT ADJUST the span from its default of 48%. When a steady reading is seen press the "*" key so that the 10-AU captures the value. Wait until you receive a "FINISHED" message (15 secs) before exiting the screen.

   If the "Cal std val" item at the bottom of screen #2.0 is not in the range 125-160, the procedures detailed in APPENDIX B should be performed and the calibration then repeated.

8. Return the concentration range control to AUTO (#2.43) and confirm that blank subtraction is disabled (#2.12).

9. Confirm that the QFT2™ program is receiving a value close to 500 QFT™ units. Switch the filter to 287 nm (A) and check that the reading drops to about 30-65 (precise value depends on the relative efficiencies of the two filters and the 10-AU spectral response).

Leave the standard solution in the flow cell. At the PC keyboard, select the "CALIBRATION & BLANK Readings" screen in order to capture the standard readings.

3B. The "CALIBRATION & BLANK Readings" Screen

BLANK / Calibr STANDARD Readings ESC=Done

Heptane	**287 nm:**	0.71	08:45	28-Oct-97
	365 nm:	3.1	08:46	28-Oct-97
IPA	**287 nm**	0.75	08:48	28-Oct-97
	365 nm:	3.6	08:49	28-Oct-97
Standard	**287 nm:**	53.78	09:01	28-Oct-97
	365 nm:	497.2	09:02	28-Oct-97

Key R to READ -or- Key M for MANUAL update

After the 10-AU is calibrated to 500 units at 365 nm using the QFT2™ standard, this screen is used to capture solvent blank and standard readings. These values are used for calculation purposes and are saved in the program's configuration file so that they can be retrieved whenever the program is restarted.

Any changes made to the values on this screen are temporary until you key ESC and then confirm that you wish to save the changes. If you inadvertently update the wrong item and do not have a manual record of its previous value, key ESC and then tell the program to abandon any changes.

STANDARD Readings

Confirm that the 10-AU HOME screen shows "(AUTO)" and not "(MAN)". If not, change the 10-AU range control (#2.43) to AUTO.

If you have not just completed 10-AU calibration (described above), thoroughly rinse the flow cell with solvent (heptane if available) followed by a two 2 ml rinses of standard solution. Then add 4 ml of standard to the syringe. Allow 2 ml to rapidly flow through the cell to clear any bubbles. (If you are continuing from the 10-AU calibration, allow a further 1/4 ml of standard to flow.) FIRMLY CAP the standard bottle to avoid solvent loss.

Select the 287 nm filter (A) and wait for a steady reading (press the F3 key as required to rescale the screen plot). Cursor down to the "Standard 287 nm" item and press "R" to capture the reading. The program will automatically advance to the "365 nm" item. Select the 365 nm filter (B), let another 1/4 ml of standard flow, again wait for a steady reading (press F3), and then key "R" to capture it.

Key ESC and then "Y" at the "Use Changes?" prompt to update the program's configuration file. You need not recalibrate the 10-AU every time you take new standard readings. It is advisable to check the standard periodically and, if required, to capture new readings.

BLANK Readings (Same procedure for both heptane and IPA.) Confirm that the 10-AU HOME screen shows "(AUTO)" and not "(MAN)". If not, change the 10-AU range control (#2.43) to AUTO.

Wash down the syringe, including its top area, with solvent allowing the wash solution to drain just to the syringe tip. Repeat several times if the standard or a highly fluorescing sample was previously run. Rinse a clean test tube with the solvent to be run and dispense a convenient volume into the tube. Select the 365 nm filter (B) and continue to rinse the flow cell an ml at a time until a steady value is obtained. Use the F3 key to rescale the screen plot as needed.

Now select the 287 nm filter (A), wait for a steady reading, and allow another ml to flow to confirm that the flow cell is clean. Due to the weakness of oil fluorescence at 287 nm, IT IS CRITICAL that correct solvent blank readings be made. By means of the PC cursor, position to the heptane or IPA 287 nm entry and press "R". After capturing the reading, the program will automatically advance to the 365 nm entry. Select the 365 nm filter (B), allow a further 1/4 ml to flow, wait for a steady reading, and then press "R" again.

Key ESC and then "Y" at the "Use Changes?" prompt to update the program's configuration file. If required, repeat the "BLANK Readings" procedure for the other solvent (heptane or IPA).

If you begin use of a new bottle of solvent, you should confirm that the blank values are still valid. If not, capture new readings. When QFT2™ calculations are made (Section 5), the standard readings will be corrected using the heptane blank readings if either is found to be non-zero. Otherwise, the IPA blank readings are used for standard correction (i.e.; if heptane is not available, be sure that the heptane Blank entries are both zero–key "M" to manually update these items.)

Accumulation of residue in the flow cell is usually signaled by an increase in blank readings. A cleaning procedure which often works is to thoroughly rinse the flow cell with heptane, IPA, water, IPA and finally heptane once again. Note that IPA rinses must interleave the heptane and water rinses due to the immiscibility of heptane and water.

MANUAL DATA Entry

When you exit the calibration screen and confirm that you wish to use any changes, the QFT2™ program saves the screen information in its configuration file. It is nonetheless recommended that you write down the displayed values just in case the file becomes for any reason unreadable. To allow for this contingency, the program permits manual updates of screen items if you key "M" rather than "R".

Section 4

The "Acquire SAMPLE Readings" Screen

Dilution Factor: 1.00 **Solvent**: Hept

Sample Descrip: 8010 to 8040 ft DC

Readings

287 nm Sample:	17.86	14:07	01-Nov-97
Hept:	0.71	08:45	28-Oct-97
365 nm Sample:	1609.0	14:08	01-Nov-97
Hept:	3.1	08:46	28-Oct-97

Select Item, Key SPACE to UPDATE ESC=Done

This screen is accessed to enter sample information and to capture sample readings. Once updates are complete, key ESC and then "Y" at the "Use Changes?" prompt to exit. Next access the CALCULATE screen (Section 5) in order to obtain oil concentration and gravity estimates from the readings. The information on the "Acquire SAMPLE Readings" screen is saved in the program's configuration file so that the screen will reflect the last sample run when the program is restarted.

Dilution Factor: (normally 1.00): The standard QFT™ extraction (DF=1) uses 5.0 ml of solvent to extract 0.50 g of solids. If dilution is required (CALCULATE screen message), dilute 0.5 ml of the sample extract with 4.5 ml of solvent and specify a dilution factor of 10 (= new volume/old volume). If further dilution is required, another 10/1 dilution would result in a total factor of 100.

If a non-standard extraction is made, the Dilution Factor should be calculated from the equation DF = (ml actual/g actual) x 0.1. For example, if 1 g of solids is extracted with 5 ml solvent (to obtain doubled sensitivity) then the dilution factor is 5/1 x 0.1 = 0.50.

Solvent: Key "H" or "I" to select heptane or IPA. The equations used for QFT2™ calculations differ depending on the solvent so be sure this entry is correct (see APPENDIX D).

Sample Descrip: Enter description to appear in the data file line for this sample. If you will be importing the file into a spread sheet program, you should use a fixed position for the sample depth so that the program can consistently identify the numeric field. For example, 30 ft interval drill cuttings collected at 8040 ft might be described by "8010 to 8040 ft DC". On the next sample, edit the depths leaving the non-numeric characters unchanged. Note that the "Add File COMMENT" screen can also be used to add sample information.

Capturing SAMPLE Readings

Confirm that the 10-AU HOME screen shows "AUTO)" and not "(MAN)". If not, change the 10-AU range control (#2.43) to AUTO.

287 nm Sample: Switch to the 287 nm filter (A). If required, drain the previous sample from the flow cell until the level just enters the syringe tip. Using the heptane squeeze bottle, rinse the syringe interior with a few ml of heptane and again drain the cell. Add the sample extract (4 to 5 ml) to the syringe and allow about 2 ml to drain rapidly to clear any bubbles in the line. When a steady reading is obtained (press F3 to rescale the screen plot as needed), add a further 1/4 ml for confirmation. Then key SPACE or "R" to capture the value. (Note: if the cell was rinsed with IPA instead of heptane, you will likely have to add another 1/2 ml of sample in 1/4 ml increments to obtain a constant reading.) The program will automatically advance to the "365 nm Sample" item.

365 nm Sample: Switch to the 365 nm filter (B). If the 10-AU auto-ranges, usually from LOW to MED range, allow it to stabilize. (An auto-range to the HIGH scale may require up to a minute.) Then allow a further 1/4 ml of sample to flow and wait for a steady reading (press F3 to rescale the plot as needed). Key SPACE or "R" to capture it.

Switch back to 287 nm filter to minimize the time required for 10-AU range changing on the next sample. Check that all sample information is correct and then key ESC to exit the "Acquire SAMPLE Readings" screen. Key "Y" at the "Use Changes?" prompt.

Immediately access the CALCULATE screen next (see Section 5). If you receive a "Dilution Recommended" or "Dilution is REQUIRED" message (at screen right), pipette 0.5 ml of the extract remaining in the syringe into a test tube and dilute with 4.5 ml of solvent. Repeat the above "Acquire SAMPLE Reading" procedure specifying a Dilution Factor of 10 times the previous one. Be sure the undiluted sample is thoroughly rinsed from the syringe.

If the extract has a distinct oil coloration, dilution is recommended regardless of the CALCULATE screen message. The equations used to correct for UV absorption losses are only approximate. Under unusual circumstances, the readings may actually decrease at high oil concentration due to very high losses.

Section 5

The "CALCULATE / File UPDATE" Screen

QFT2 Calculations ----------

Sample: 8010 to 8040 ft DC — **Calculated Results:** [5]

Dilution Fctr: 1.00 **in** Hept — **Abs Corr @287 nm** **1.35**

	287 nm	**365 nm**	**Abs Corr @365 nm**	**1.22**
Sample Reading	17.86	1609.0	**Oil Conc mg/l**	**611.5**
Hept Blank.	0.71	3.1	Sample Dilution	-OK-
IPA Blank.	0.75	3.6	**Est'd Wt % Oil**	**0.612**
Calibr Reading	53.78	497.2	**Est'd API Grav**	**46**

--- F5=Update File ---------- F6=Recalculate ---------- ESC=Done ---

When you access the "CALCULATE / File UPDATE" screen, the information initially shown on the left is always the same as the information last entered on the "Acquire SAMPLE Readings" and "CALIBRATION & BLANK Readings" screens. The screen right displays the QFT2™ calculation results. In particular, the bottom two items on the right show QFT2™ estimates for oil saturation and API gravity. Immediately above you will normally see the message "Sample Dilution -OK-". When this is the case, simply press F5 to add the sample information to the data file. Once this is done, you can key F2 to view the updated file.

If instead, you receive a BEEP plus the message "Dilution Recommended" or "Dilution is REQUIRED", you should dilute the sample as described in Section 4 ("Acquire SAMPLE Readings") and reacquire the sample values.

When the extract has a distinct oil coloration but you do not receive a dilution warning, it is recommended that you add the calculation to the file (press F5) but also dilute and rerun the sample. If the results for the diluted sample differ significantly and the readings are clearly above background, use the diluted sample calculations.

If you will not be making another measurement immediately, rinse the 10-AU syringe body and flow cell with heptane (or IPA, if heptane is not available) to minimize oil residue accumulation.

Hept Blank: If you have left both heptane readings zero on the "CALIBRATION & BLANK Readings" screen (heptane not available at job site), the program will duplicate the IPA blank readings here. The "Calibr Reading" values are always corrected using the heptane blank since the standard is prepared as a heptane solution. Normally the solvent blanks are similar and the standard readings large so that the error introduced by using the IPA readings is small.

Abs Corr @287 nm: Shows the factor applied to the net 287 nm sample reading to correct for UV absorption losses.

If the factor is between 1.50 and 2.50, you will receive a "Dilution Recommended" message. Unless you are pressed for time or the sample is judged to be unimportant, you should dilute and rerun the sample (especially if the value is 2 or greater.) Keying F5 WILL result in a file update.

If the factor exceeds 2.50, you will see a "Dilution is REQUIRED" message and no final results will be displayed. Keying F5 WILL NOT result in a file update.

Abs Corr @365 nm: Shows the factor applied to the net 365 nm sample reading to correct for UV absorption losses. This value is always less than the 287 nm correction factor. Both the 287 and 365 nm factors are estimated from the 287/365 intensity ratio and the oil concentration. Since these are themselves dependent on the absorption factors, an iterative calculation is required. (The number of iterations needed to reach a stable result is shown in brackets at the top right.)

Oil Conc mg/l: Shows the estimated oil concentration of the flow cell solution (not adjusted for dilutions, if any). The oil calibration constant for fluorescence at 365 nm is calculated from the 287/365 intensity ratio. This constant is then applied to the 365 reading to estimate the oil concentration.

Est'd Wt % Oil: Formation oil content calculated from the estimated oil concentration and the specified Dilution Factor. The standard deviation for this calculation based on QFT2™ analysis of 41 oils ranging from 20° to 70° API corresponds to a factor of 1.7 uncertainty in actual oil content. Analysis of seven oils having gravities between 10° and 20° showed somewhat lesser accuracy for heptane solutions but, due to limited solubility, erratic results for IPA.

Est'd API Grav: Oil gravity calculated from the 287/365 intensity ratio. This result is insensitive oil concentration to the extent that the absorption loss estimates are correct. The standard deviation for this calculation is ±8° API for oils in the range 20° to 70°. Oils in the range of 10° to 20° give more variable, generally high estimates. Heavy oils can give very high results for IPA extracts due to limited oil solubility.

The program permits you to manually update all the items on the screen left to allow for the case of customized corrections. Updates other than to the sample description will result in a blanking of the calculated results. When your updates are complete, key F6 to obtain a recalculation using the revised values. These may be saved to the data file by keying F5 as described above. You should also include a file comment to explain the reason for the manual changes.

Manual changes to the "CALCULATE" screen are not saved in the program's configuration file. The next time you access the screen, the input items will revert to the current system values.

Section 6
Data File Copying and File Management

The "COPY File" item on the program main screen can be used to copy the currently selected data file (set via CONFIGURATION screen) to either drive A: or B:. The data file is a plain text (ASCII) file which can be edited using any text editor such as the MS-DOS EDIT or Norton NE programs. Provided care has been taken to provide a fixed depth position in the "Sample Descrip:" input, the data file can also be easily imported into spreadsheet programs such as Lotus 123 or Microsoft EXCEL. Data files are always given an MS-DOS file extension of ".QF2".

Even if you are not importing the data, it is strongly recommended that the current data file be copied to diskette at regular intervals simply for back-up purposes.

The QFT2™ program itself provides no file management and only accesses files in the currently active directory. The "LIST FILES' item on the CONFIGURATION screen will show up to the latest 25 data files but any number over five indicates the need for a cleanup of inactive .QF2 files. At job end (or startup) you should purge the QFT2™ program directory of inactive data files both to avoid confusion (and possible selection of an incorrect file) and to prevent access to information considered sensitive by the previous client.

Appendix A

Program Requirements, Installation and Operation

The QFT2™ Data Logger program requires the following:

- **PC with 386 CPU minimum (math chip not used)**
- **640 x 480 Graphics (VGA - color recommended)**
- **Either COM1 or COM2 serial data port**
- **At least 256K memory**
- **Hard Drive with 1 MB of free space**
- **MS-DOS 3.0 or later operating system**

Because of space limitations in the logging unit, it is recommended that a dedicated laptop PC be used. The above requirements are quite modest compared to "state-of-the-art" PC's needed to run Windows. A low cost laptop of adequate performance should be readily available (particularly on the used-PC market). Note that a mouse is not used. If the PC is equipped with a serial mouse, the mouse port (normally COM1) can be used for serial communications.

The QFT2™ program has been extensively tested in a Real-Mode MS-DOS environment. It will probably run in the "Full-Screen" mode under Windows provided a serial port not used by the mouse is available. If you receive "COMM Link:" error messages and you have verified that the 10-AU is properly configured, you should restart your computer in the MS-DOS mode and retry. Note that extended memory managers (such as MS-DOS EMM386.SYS) and disk caching software (such as MS-DOS SMARTDRV) may also cause COMM link problems if they operate the computer in the "Virtual 8086" mode or inhibit CPU interrupts for extended periods of time. Neither extended memory nor disk caching is required by the program.

Manual Program Installation and Program Files

Installation of the QFT2™ Data Logger program involves the copying of only two files, **QFT2.EXE** (executable file) and **QFT2.TXT** (this help file). Once the program begins running it creates the configuration file **QFT2.CFG** if one does not already exist. If you are transferring the program to another computer or directory and wish to retain current configuration (calibration) data, the QFT2.CFG file should also be copied. The program also creates a backup file called QFT2BKUP.CFG which need not be copied.

To start the program, use the MS-DOS "CD" command to change to the directory containing the program, and then key "QFT2™". If the computer is dedicated to QFT2™, these commands can be added as the final lines in the computer's AUTOEXEC.BAT file.

An example installation which assumes the program files are currently on the diskette in drive A:, and that the program directory is to be located on drive C: and is to be named "QFT":

C:\> **MD\QFT**	If required, create target directory.
C:\> **CD\QFT**	Change to it.
C:\QFT> **COPY A:QFT2.***	Copy QFT2.EXE and QFT2.TXT (plus QFT2.CFG if it is on the diskette).
C:\QFT> **QFT2**	Start QFT2.EXE running–all data files will reside in the \QFT directory.

If you wish to use the same computer with more than one 10-AU, then a separate installation should be made for each fluorometer so that each can have its own configuration file and set of data files. For example, if you have two 10-AU units (call them "A" and "B"), create the directories \QFT_A and \QFT_B (for example) and copy the same program files to both.

The QFT2.TXT file is a straight text file and may be edited using the MS-DOS EDIT program in case you wish to add any on-line help or operator instructions. The file display routine assumes a maximum line length of 76 characters.

Summary of Program Operation and Features

MAIN Screen: When the QFT2™ Data Logger program first starts running the screen top lists the following selectable items:

- **System CONFIGURATION Inputs**
- **CALIBRATION / BLANK Readings**
- **Acquire SAMPLE Readings**
- **Add File COMMENT**
- **COPY File**
- **CALCULATE / File UPDATE**
- **Set PLOT High/Low Scales**

To select an item, use the cursor arrow keys to highlight the desired one and then key SPACE or ENTER. Except for the plot scale, selection causes a "pop-up" screen to appear which contains operator inputs controlling various features of program operation (as described in previous sections).

To exit any of the "pop-up" screens, key ESC. If you have modified any variables saved in the program's configuration file, you will always be asked if you wish to use the changes–"Use Changes?". Keying ENTER or "Y" causes the changes to be used; keying "N" causes the changes to be discarded; and keying ESC returns access to the active "pop-up" screen.

When starting a new job, first update the CONFIGURATION screen items as required. Then check and/or update the CALIBRATION/BLANK screen items. Once these are correct, you analyze samples by alternating between the SAMPLE and CALCULATE/UPDATE screens. File comments should be used to add any information which might be required to interpret the sample readings (such as core bit runs). The data file can be copied to diskette at any time for backup purposes or to transfer it to another PC for further processing. The data file is a plain text (ASCII) file.

EDITING of screen items: Use the cursor arrow keys to highlight the desired item. Key a SPACE or the desired first character to begin editing. The field will change to bright characters on a dark background. By default new characters will overwrite the old ones but you can switch between overwrite and insert modes by pressing the INSERT key. If you are editing a numeric field, you can view the minimum allowed value by keying PAGE UP and the maximum by keying PAGE DOWN.

When your entry is complete, key ENTER. To cancel changes, key ESC. To restart changes, hold down the CTRL key and press "U" (undo). To erase all characters to the right of the cursor, hold down the CTRL key and press "D" (delete).

To activate a non-edit item (such as the "LIST FILES" entry on the CONFIGURATION screen), highlight it using the cursor keys and key SPACE or ENTER.

When you are not updating an edit item or viewing a file, the function keys along the top of the keyboard have the following actions:

F1 Display this help file. Use cursor keys to scroll.

F2 Display active data file. Use cursor keys to scroll.

F3 Rescale plot to show most recent fluorescence values.

F4 Rescale plot to show all buffered values (500 seconds).

F5 Write CALCULATE screen results to data file.

F6 Recalculate CALCULATE screen results using revised inputs.

F9 PAUSE or RESUME signal averaging and plotting.

F10 Exit QFT2™ program. The program will ask for confirmation.

Test Mode Program Operation

For the purpose of training and software demonstration, the program can be configured to generate "Test Mode" data. In this mode, the 10-AU (if connected) is ignored, and a dummy signal can be controlled by use of the keyboard (whenever you are not actively editing a screen item or viewing a file).

Hold down CTRL Key, Press	——— Action ———
F12	Enter and Exit Test Mode
Up Arrow	Increase value by 0.5
Down Arrow	Decrease by 0.5
PAGE UP	Increase by 5.0
PAGE DOWN	Decrease by 5.0

The test value has a random component added in order to generate a more realistic signal.

Appendix B

Optimization of 10-AU Fluorometer Performance

The 10-AU fluorometer requires two mechanical adjustments to optimize its performance:

1. Positioning of the flow cell holder for maximum intensity, and then

2. Setting the Sensitivity Adjustment Knob for maximum dynamic range.

These adjustments should be made for new fluorometers, old fluorometers when upgraded with the dual filter retrofit kit, and whenever the UV lamp is changed. Both adjustments require use of the QFT2™ standard. Both adjustments will result in the loss of any current 10-AU calibration so that they must be followed by a full calibration as described in Section 3.

Flow Cell Holder Positioning

The flow cell consists of a 1 mm inner diameter quartz tube mounted in a black plastic cylindrical holder machined with excitation and emission beam slits. Note that the holder for QFT2™ has wider slits than those used for the older single point QFT™ method (0.8 vs. 0.5 mm) because of the smaller emission filter size used by QFT2™.

Remove the two small cover plates immediately to the right of the filter selection knob and loosen the holder set screws. Replace the cover plates to avoid light leakage (need not be fully tightened).

The holder cylinder consists of three sections. The junctures of the top and bottom sections with the middle one should be just visible at the top and bottom of the 10-AU sample compartment exterior. If required, vertically position the holder until the top juncture is flush with the compartment top (not extremely critical). The O-rings used at either end of the middle section are quite tight—considerable effort may by required to move the holder.

Select the 365 nm filter (B) and fill the flow cell with QFT2™ standard. Carefully rotate the holder by small steps until a maximum reading is obtained—this adjustment is sensitive to small changes. If the reported value goes off scale, turn the sensitivity knob counter-clockwise as described in the

next section until a numeric reading is obtained. The exact value is not important at this stage. When done remove the set screw covers, retighten the set screws (do not over-tighten or the adjustment will change), and reinstall the covers.

Sensitivity Adjustment Knob

The overall electronic gain of the 10-AU is controlled by its Sensitivity Adjustment Knob. The signal/noise ratio is maximized by setting the gain such that a full scale photomultiplier signal (3 volts) is obtained for the maximum fluorescence intensity of interest (10,000 units for 365 nm filter). Since the QFT2™ standard provides 500 units of fluorescence, the sensitivity knob is adjusted to give a photomultiplier signal of 150 mv for the standard.

By means of the 10-AU keypad set the concentration range control to MANUAL (#2.43) and the concentration range to MED (#2.42). Access the second Diagnostic screen (#3.2 - key 3 at the MAIN MENU followed by an ENT). Confirm that the bottom item “Span level%” has its 10-AU default value of 48. (If not, use #2.6 “Reset calibration defaults”.) The second from the top item should be “PM signal output”. This is the value to be adjusted.

Rinse and fill the flow cell with QFT2™ standard and select the 365 nm filter (B). If “PM signal output” is 125-160 no adjustment is needed.

Otherwise, loosen the set screw at the front left of the keypad. By means of a coin or large screwdriver, rotate the slotted round top “knob” below the power light on the right side of the keypad casing until a reading close to 150 is obtained. Rotating the knob clockwise will increase the reading. The 10-AU response is slow. Wait after each adjustment for a steady PM signal. When done, retighten the set screw at the keypad left.

After recalibration of the 10-AU (Section 3.A), the “Cal std val” item at the middle of screen #3.2 (and at the bottom of screen #2.0) should be close to 150. You can monitor changes in the 10-AU gain due to lamp and electronics drift by checking this item after each calibration.

Appendix C

Determination of Instrument Correction Factors

Even after normalizing sample intensities by use of the calibration standard intensities, minor instrument specific differences are seen due to spectral response variations in the 287 and 365 nm filters and the 10-AU fluorometer. By use of a standard oil, currently supplied by Texaco, and the usual QFT2™ calibration standard, instrument correction factors may be determined. The oil standard consists of a heptane solution of a medium gravity oil. The bottle will have noted on its label oil to QFT2™ standard intensity ratio values for both 287 and 365 nm. The operator measures actual intensity ratios for his 10-AU and then divides them into the values noted on the oil bottle to calculate the instrument correction factors.

Example
Assume the oil standard is marked with the following values:

Oil / QFT2™ Standard	=	0.1536 at 287 nm	Example
	=	0.858 at 365 nm	Only

The operator makes the following readings taking special care to thoroughly rinse the flow cell between samples:

	287 nm	365 nm
Heptane Blank	0.28	2.4
QFT2™ Standard	50.13	470.9
Standard Oil	7.24	406.1

Obsd Oil/Stnd = (7.24-0.28) / (50.13-0.28) = 0.1396 at 287 nm

= (406.1-2.4) / (470.9-2.4) = 0.862 at 365 nm

Instr. Corr. Factor = 0.1536 / 0.1396 = **1.100** at 287 nm

= 0.858 / 0.862 = **0.995** at 365 nm

Due to the importance of this determination, it is highly recommended that it be performed in triplicate and the results averaged. This will guard against erroneous readings and also provide a better value for the weak, relatively noisy 287 nm oil signal. The results should be clearly noted by means of a permanent, easily visible label affixed to the case of the 10-AU instrument. If the computer to be used for QFT2™ data acquisition is available, the results should also be entered into the program's configuration file by means of the CONFIGURATION screen (Section 2).

Appendix D
Handling and Use of Solvents and Standards

QFT2™ involves use of n-heptane and, optionally, isopropyl alcohol (IPA) as oil extraction solvents. These must be of SPECTRAL GRADE purity since trace contaminants can contribute significant background fluorescence. Typically solvents will be purchased in liter or larger size bottles. When a bottle is in use, it is fitted a calibrated dispensing pump.

By means of liquid chromatography, whole oils are separated into four solubility fractions: Saturates, Aromatics, Resins, and Asphaltenes. All but the saturates contribute to oil fluorescence. The asphaltenes are insoluble in both heptane and IPA, while the resins are only poorly soluble in IPA. For this reason, heptane is the preferred extraction solvent when dry samples are examined. As a practical matter, IPA is often preferred at rig-site since wet samples can be extracted due to the miscibility of IPA with both oil and water. The equations used for QFT2™ data reduction differ according to which solvent is used, both to allow for solvent dependent fluorescence quenching effects and to correct for the differing solubilities of whole oils in heptane and IPA.

Regardless of which solvent is used for extraction, heptane should always be available in a Teflon® (NOT PLASTIC) squeeze bottle for use in rinsing out the 10-AU flow cell. Heptane aids in removing residues which are only marginally soluble in IPA, and also, due to its lower viscosity, flows through the cell more rapidly. Heptane blank readings are also needed during calibrating since the QFT2™ standard is prepared as a heptane solution.

IPA should NEVER BE DISPENSED from a squeeze bottle, even if Teflon, since trace chemicals (probably plasticizers) having very high UV absorbance are extracted. Only glass containers should be used for storing and dispensing IPA. If, for any reason, heptane is not available, IPA can be used for flow cell rinsing but somewhat more sample will be required to obtain a steady readings. (The lower viscosity of the heptane rinse allows it to be much more easily displaced by the following sample.) Rinse IPA may be stored in a squeeze bottle IPA used for blank readings, however, should always be taken from the solvent dispenser used for sample extractions.

The standard used for QFT2™ calibration consists of 200 mg/l of a p-xylene/phenanthrene mixture dissolved in n-heptane. The mixture has 50 parts by weight of p-xylene (for 287 nm fluorescence) to one part of

phenanthrene (365 nm). Both must be of the highest available purity (99.5+ %) for proper results. The standard will normally be supplied in a 100 ml dark glass bottle. This bottle must be kept **tightly sealed** between use to avoid loss of the heptane solvent. A complete system calibration, described in Section 3, requires about 10 ml of standard. The standard should never be diluted to "stretch" usage since UV absorption causes a non-linear response.

Appendix E

QFT2™ Licensing

The Quantitative Fluorescence Technique (QFT™) is a patented and licensed process developed by the Surface Logging Group of Texaco Upstream Technology. Licensing information is available from:

Texaco Development Corporation
Texaco Upstream Technology
QFT™ Licensing
3901 Briarpark Drive
Houston TX 77042 USA
713.954.6000

QFT2™ is an improvement on the older QFT™ process. To upgrade to QFT2™, current QFT™ users will need to purchase a 10-AU Fluorometer Dual Filter Retrofit kit from Turner Designs. In addition, QFT2™ manuals and PC software should be obtained from Texaco Upstream Technology. Licensing requirements and terms remain unchanged–current QFT™ licensees have full rights to practice QFT2™ and no distinction between QFT™ and QFT2™ need be made when reporting usage to Texaco.

■■■■■

Chapter 7

Selecting Equipment and Supplies

This chapter provides a detailed listing of the equipment and supplies you will need to set up and operate the QFT2™ system in the field. It also gives reference numbers and sources for each item. Brand names, suppliers, and part numbers are for illustrative purposes only, with the exception of the QFT2™ Data Logger Program and the Turner Designs 10-AU-CE Mud Logging Fluorometer. No endorsement is implied. This chapter contains the following sections:

- **Fluorometer**
- **Personal Computer**
- **Sample Preparation and Analysis Supplies**
- **Sources for Supplies**
- **Vendor Information**
- **Preparing the QFT2™ Standard**
- **Estimating Solvent Use**

Fluorometer

The primary equipment required for the Texaco QFT2™ system is the Turner Designs 10-AU-015-CE Mud Logging Fluorometer or the 10-AU-015 Mud Logging Fluorometer with Retrofit Kit, the accompanying Fluorometer User's Manual, and the QFT2™ Data Logger Program (inside the back cover of this guide).

The Turner Designs 10-AU Fluorometer is rugged, dust and water-resistant (to reduce corrosion of critical components), and tolerant of marginal power sources (115-230 VAC, 50-60 hz). The expanded dynamic range allows for the full range of oil concentrations normally encountered in drill cuttings without the need for excessive dilutions. A flow cell eliminates the need for expensive quartz test tubes and enables a more rapid sample analysis. A state-of-the-art microprocessor monitors numerous internal functions that alert the

user to malfunctions, and an internal diagnostics screen assists in troubleshooting. The integrity of the new design eliminates the need for frequent calibration, usually requiring one initial calibration and one or two calibration checks per well.

To contact Turner Designs, see *Vendor Information*, later in this chapter. Tables 7 through 10 show information on the 10-AU-015 Fluorometer and associated spare parts, accessories, and retrofit kits.

Table 7
Turner Designs 10-AU-015-CE Fluorometer

Table 7. Turner Designs 10-AU-015-CE Fluorometer

Part No.	Price ($)	Description
10-AU-015-CE	9,125.	System includes a Turner Designs Field Fluorometer with a Power and Signal cable, indoor case, 1 mm Continuous-Flow Cuvette System for mud logging, filters, lamp, user's manual, optical kit, fluid handling spares kit, and spare fuses. Please order 046-0150 and 10-AU-064 for 230 VAC. No additional instrumentation is required for the QFT2™ system, but disposables specified by Texaco must be acquired separately. If the instrument will be shipped to various locations, we recommend the purchase of a Transport Case, P/N 10-AU-060.

Table 8
Spare Parts for the 10-AU-015-CE Fluorometer

Table 8. Spare Parts for the 10-AU-015-CE Fluorometer

Part No.	Price ($)	Description
10-AU-165	100.	Fluid Handling Spares Kit
10-AU-155	100.	Syringe (for sample injection)
10-AU-147	896.	QFT2™ Filter Kit (includes excitation and emission filters)
10-AU-145	340.	Injector Assembly
10-AU-140	505.	1 mm Continuous-Flow Cuvette System Assembly
10-AU-064	15.	Fuses, DC, 4 AMP International, 5 ea.
10-046	55.	Clear Quartz Lamp
10-038R	395.	Excitation Filter, 254 nm
10-032	65.	1 N.D. Filter
10-023	45.	Desiccant Replacement Kit, 10/bottle
046-0150	15.	Line Cord, 230 VAC
034-0044	252.	Emission Filter, 365 nm
034-0038	252.	Emission Filter, 287 nm

Table 9
Accessories for the 10-AU-015-CE Fluorometer

Table 9. Accessories for the 10-AU-015-CE Fluorometer

Part No.	Price ($)	Description
10-AU-060	500.	Instrument Transport Case
10-AU-012L	280.	12 VDC Power and Signal Cable w/ Terminal Lugs
10-AU-012C	280.	12 VDC Power and Signal Cable w/ Clips
WARR-10	195.	Extended Warranty Program– One Year (**U.S. Customers only**)
WARR-102	350.	Extended Warranty Program– Two Years (**U.S. Customers only**)

Table 10
QFT2™ Retrofit Kits

Table 10. QFT2™ Retrofit Kits

Part No.	Price ($)	Description
10-AU-151	2,355.	QFT2™ Retrofit for Non-CE Instruments
10-AU-149	2,470.	QFT2™ Retrofit for CE Instruments

Personal Computer

To work with the QFT2™ Data Logger Program, you will need the minimum PC equipment and software shown in Table 11.

Table 11
PC System Minimum Requirements

Table 11. PC System Minimum Requirements

Description
• Dedicated laptop PC (recommended for space limitations)
• PC with 386 CPU minimum (math chip not used)
• 640 x 480 Graphics (VGA–color recommended)
• Either COM1 or COM2 serial data port
• At least 256K memory
• Hard Drive with 1MB of free space
• MS-DOS 3.0 or later operating system

Sample Preparation and Analysis Supplies

In addition to the fluorometer and a personal computer, you will also need several laboratory supplies to prepare samples and to analyze them using either the dry sample analysis method or the wet sample analysis method. These basic supplies are shown in Tables 12 through 14 and Figures 43 through 45. For a more detailed listing of the solvents, syringe filters, and other supplies as well as the sources for them, see *Sources for Supplies*, later in this chapter.

Table 12
Sample Preparation Supplies

Table 12. Sample Preparation Supplies

Number in Figure 43	Description
1	Spin Dry Sample Dryer
2	Aluminum Weigh Dish
3	Whatman Filter Paper
4	Weighing Paper
5	Permanent Marker
6	Porcelain Spatula
7	Metal Spatula
8, 9	Hammer or Mortar and Pestle

Figure 43
Sample Preparation Supplies

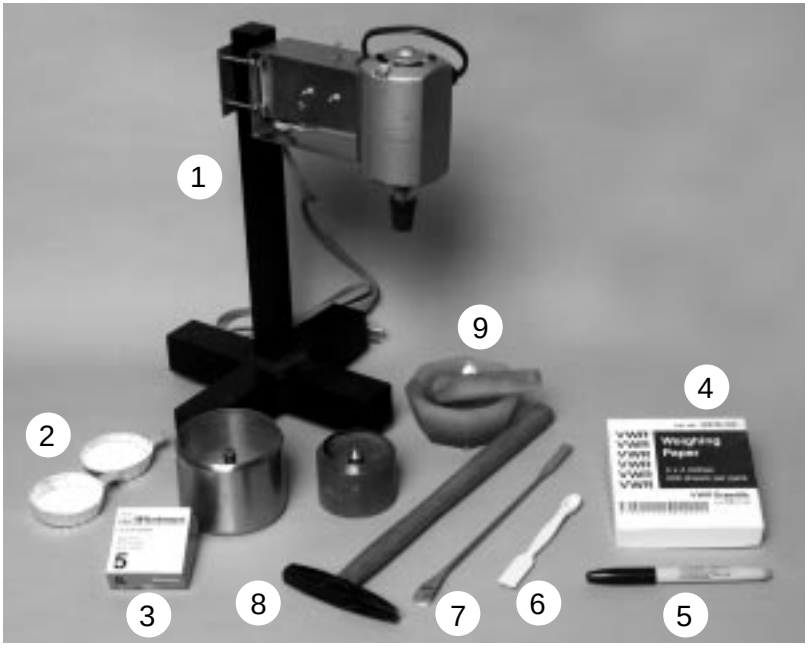


Table 13. Supplies for Dry Sample Analysis

Number in Figure 44	Description
1	Spectral Grade Heptane
2	PTFE Filters (13 mm, 0.45 µm)
3	Teflon® Wash Bottle
4	Safety Bottle Carrier
5	Universal Repipet II
6	Vortex Mixer
7	Green Pipet Pump and Disposable Pipet (5 ml x 1/10)–optional
8	Blue Pipet Pump and Disposable Pipet (1 ml x 1/100)
9	Luer-Lok Glass Syringe (10 cc x 1/10 cc)
10	Polypropylene Filtering Flask and Funnel
11	Test Tube Rack
12	Test Tubes – Large (16 x 125 mm), Small (12 x 75 mm)
13	Safety Glasses
14	Nitrile Gloves
15	QFT2™ Standard
16	Permanent Marker

Table 13
Supplies for Dry Sample Analysis

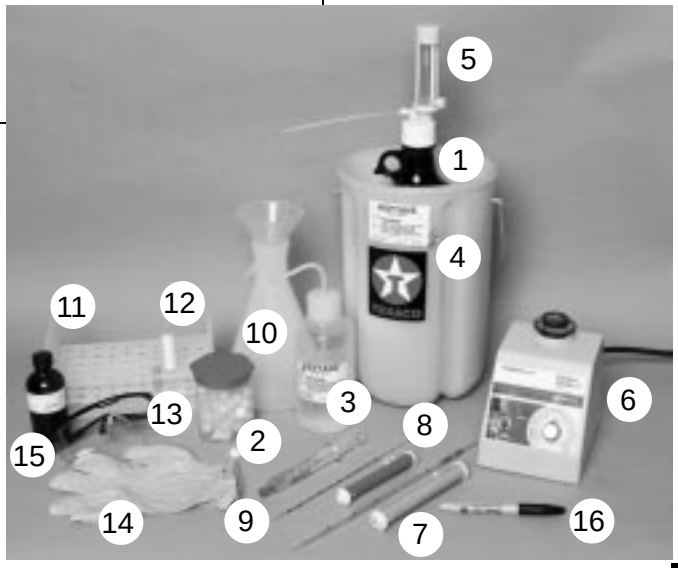


Figure 44
Supplies for Dry Sample Analysis

Table 14. Supplies for Wet Sample Analysis

Number in Figure 45	Description
1	Spectral Grade Isopropyl Alcohol
2	PTFE Filters (25 mm, 0.45 µm)
3	Teflon® Wash Bottle
4	Safety Bottle Carrier
5	Universal Repipet II
6	Vortex Mixer
7	Green Pipet Pump and Disposable Pipet (5 ml x 1/10)–optional
8	Blue Pipet Pump and Disposable Pipet (1 ml x 1/100)
9	Luer-Lok Glass Syringe (10 cc x 1/10 cc)
10	Polypropylene Filtering Flask and Funnel
11	Test Tube Rack
12	Test Tubes – Large (16 x 125 mm), Small (12 x 75 mm)
13	Safety Glasses
14	Nitrile Gloves
15	QFT2™ Standard
16	Permanent Marker

Table 14
Supplies for Wet Sample Analysis

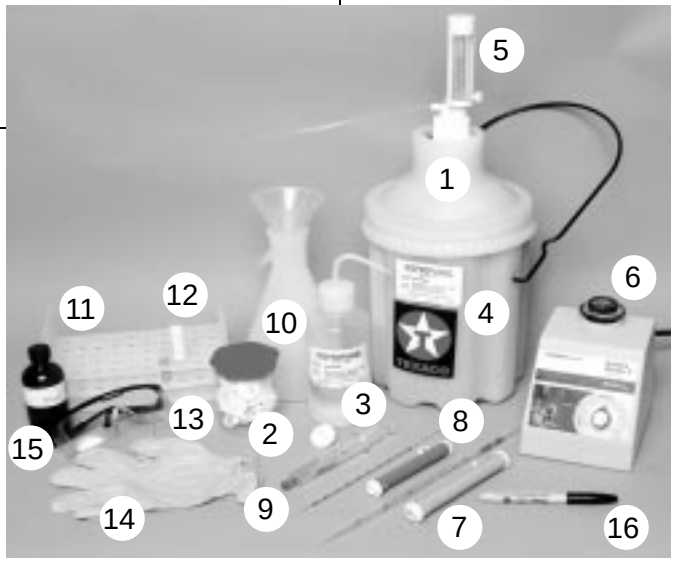


Figure 45
Supplies for Wet Sample Analysis

Sources for Supplies

This section provides a more detailed listing of and sources for the supplies you will need for preparing and analyzing samples. Before beginning your analysis, make sure you have the following two types of supplies ready:

- **General Supplies**
- **Solvents and Syringe Filters**

General Supplies

The terms and abbreviations used throughout this section are shown in Table 15.

Table 15
Terms and Abbreviations

Table 15. Terms and Abbreviations

Term	Abbreviation
Inside Diameter	ID
Outside Diameter	OD
package	pkg
case	cs
cubic centimeters	cc
pieces	pcs

Regardless of the type of analysis you perform, you will need to have on hand the general supplies described in Tables 16 through 18. These tables provide a detailed description, the VWR Catalog Number (unless specified otherwise), and suggested quantities. For prices and availability, contact the vendors listed in *Vendor Information*, later in this chapter.

IMPORTANT

Brand names, suppliers, and part numbers are for illustrative purposes only (with the exception of the QFT2™ Data Logger Program and the Turner Designs 10-AU-CE Mud Logging Fluorometer). No endorsement of any product or vendor is implied.

Table 16
General Supplies for QFT2™ Analysis

Table 16. General Supplies for QFT2™ Analysis

Description	VWR Catalog Number	Quantity
Polypropylene Filtering Funnels (Nalge 4250), 4/pkg I.D. Top 104 mm, Stem Length 99 mm	30253-129	1 pkg
Seriological Pipets Borosilicate glass, Disposable, 1000 pcs./cs (4 units of 250) (1 ml x 1/100) Kimble 72120-11100	53283-770	1 cs
Seriological Pipets Borosilicate glass, Disposable, 500 pcs./cs (5 units of 100) (5 ml x 1/10) Kimble 72120-5110	53283-774	1 cs
Pipet Pump Size 2 ml, Color Blue, Bel-Art Products F-37897	53502-222	1 ea
Pipet Pump Size 10 ml, Color Green, Bel-Art Products F-37898	53502-233	1 ea
Universal Repipet II Dispenser, Cap. 5 ml Lab Industries, Inc. Cat. No. 9005-U	53523-732	2 ea
Test Tubes (16 x 125 mm) Dispens-A-Pak Disposable Culture Tubes 1000/cs (4 dispenser pks)	15404-438	2 cs
Test Tube Rack Polypropylene, Bel-Art No. 18856 For Tubes 13-16 mm, No. of Places: 50	60916-349	2 ea

Table 17
General Supplies for QFT2™ Analysis

Table 17. General Supplies for QFT2™ Analysis

Description	VWR Catalog Number	Quantity
Lab Markers Black, Permanent, Waterproof, 10/pkg	52877-150	1 pkg
Weighing Paper Size 4 x 4 in. 500 sheets/pkg	12578-165	3 pkg
Spatula, Porcelain Length: 123 mm, Coors USA No. 60478	57925-046	2 ea
Mortar, Porcelain (or hammer) Cap. 400 ml, Dia. 130 mm, Coors USA No. 60322	50420-289	1 ea
Pestle, Porcelain (or hammer) Coors USA No. 60323	50420-482	1 ea
Syringe, Glass Multifit, Luer-Lok, 5 cc in 1/5 cc Alltech No. BD2131	Becton-Dickinson No. 2131	2 ea
Safety Bottle Carrier*** 4 L/1 gal., Nalgene No. 6501-4000	56609-222	1 ea
Step-On Waste Can (15 in. H x 13 in. Dia., 18 qt. Liner) Rubbermaid No. 6142	56610-094	1 ea
Filtering Flask with Tubulation Polypropylene, Cap. 1000 ml Nalgene No. 4101	29417-047	1 ea
Chemical Storage Can Safety, Type 1, Eagle, Cap. 2 gal. (7.6 liters)	56609-528	1 ea
Neoprene Stopper Size 8	59589-278	1 pkg
100 % Nitrile Gloves Disposable, Ambidextrous, Large, 100/pkg Manufactured by Best Inc. (No. 6005 PFL)	32891-811	1 pkg

* The safety bottle carrier is used to hold the 4 liter bottle of solvent. A safety bottle carrier for the 1 liter size bottle is not available.

Table 18
General Supplies for QFT2™ Analysis

Table 18. General Supplies for QFT2™ Analysis

Description	VWR Catalog Number	Quantity
Safety Glasses	33002-023	2 ea
Micro-Spoon Stainless Steel, Length: 9 in.	57952-253	1 ea
Genie 2 Mixer 115 V, 60 Hz-178; 220 V, 60 Hz-189	58815-178	1 ea
Disposable Aluminum Weighing Dishes (pack of 144)	25433-008	5 pkg
Filter Papers Whatman No. 5, Dia. 5.5 cm, (100/pkg)	28462-025	10 pkg
Wide Mouth Wash Bottle Teflon® PFA (Norton A1069739), 250 ml	16651-881	1 ea
Teflon® Tubing 12 in. long Teflon® inlet tube with locking sleeve, Labindustries No. 12-W	53526-810	1 ea
OR 24 in. long Teflon® inlet tube with locking sleeve, Labindustries No. 24-W	53526-815	1 ea
Standard for QFT2™	Supelco No. 86-8091	2 ea
Spin Dry Sample Dryer	U.S. GeoSupply No. 8015	1 ea
Teflon® Tubing (0.062 in. ID x 5/8 in. OD x 10 ft)	Upchurch Scientific No. 1523	1 ea

Solvents and Syringe Filters

In addition to the general supplies, you will need to select the solvent and syringe filters to perform wet sample or dry sample analysis on drill cuttings, cores, sidewall cores, or other samples (see *Selecting an Oil Extraction Method* in Chapter 5). This section provides detailed information on these items. Table 19 shows the general types of solvents and syringe filters required for each analysis method.

Table 19
Solvents and Syringe Filter Types for Wet Sample and Dry Sample Analysis

Table 19. Solvents and Syringe Filter Types for Wet Sample and Dry Sample Analysis

Item	Wet Sample Analysis	Dry Sample Analysis
Solvent	Isopropyl Alcohol (IPA)	n-Heptane
Filter Type	Syringe Only	Syringe, Uniprep, Autovial
Filter Specifications	Diameter: 25 mm Pore Size: 0.45 µm PTFE	Diameter: 13 mm Pore Size: 0.45 µm PTFE

If you are using the wet sample analysis method, see Table 20 for detailed information on solvent and syringe filter options.

Table 20
Solvents and Syringe Filter Options for Wet Sample Analysis

Table 20. Solvents and Syringe Filter Options for Wet Sample Analysis

Description	VWR Catalog Number	Quantity
2-Propanol (IPA)* **EM Science OmniSolv** 6 x 1 L/cs 4 x 4 L/cs OmniSolv 99.5% min., Glass Distilled, Suitable for Spectrophotometry, Chromatography, and Residue Analysis, Analysis on Label, UV Cut-off: 210 nm	EM-PX1834-6 EM-PX1834-1	4 cs 2 cs
Non-Sterile Syringe PTFE Filters Dia. 25 mm, Pore Size: 0.45 µm Acrodisc 24 CR PTFE Syringe Filter, 200/pkg Gelman Product No. 4219	28143-924	5 pkg
OR		
Whatman 25 mm PTFE Filters	Whatman No. 6785-2504	
OR		
Nalgene 25 mm PTFE Syringe Filter, 300/pkg, Nalgene No. 199-2045	28195-880	4 pkg
OR		
Nucleopore 25 mm PTFE	Nalgene No. 130667	
OR		
Alltech 25 mm Non-Sterile Syringe Filters, Dia. 25 mm, Pore Size: 0.45 µm	Alltech No. 2090	10 pkg

* The 4 liter size bottle of IPA is used for domestic operations. Regulations regarding shipment of flammable solvents to foreign countries by air require the 1 liter size bottles of IPA.

If you are using the dry sample analysis method, see Table 21 for detailed information on solvent and syringe filter options.

Table 21
Solvents and Syringe Filter Options for Dry Sample Analysis

Table 21. Solvents and Syringe Filter Options for Dry Sample Analysis

Description	VWR Catalog Number	Quantity
Heptane* 6 x 1 L/cs 4 x 4 L/cs OmniSolv 99.5% min., Glass Distilled, Suitable for Spectrophotometry, Chromatography, and Residue Analysis, Analysis on Label, UV Cut-off: 194 nm	EM-HX0078-6 EM-HX0078-1	4 cs 2 cs
UniPrep Syringeless Filter Pore Size: 0.45 µm PTFE, 50/box	28297-192	20 box
OR		
Acrodisc 13 CR PTFE Syringe Filter 100/pk, Dia. 13 mm, Pore Size: 0.45 µm, Gelman Product No. 4422	28143-981	10 pkg
OR		
Alltech 13 mm Non-Sterile Syringe Filters 100/pk, Dia. 13 mm, Pore Size: 0.45 µm, (You can substitute these filters for the Acrodiscs.)	Alltech No. 2165	10 pkg

* The 4 liter size bottle of heptane is used for domestic operations. Regulations regarding shipment of flammable solvents to foreign countries by air require the 1 liter size bottles of heptane.

Vendor Information

To contact vendors regarding the availability and prices for the equipment and supplies described in this chapter, see Table 22 for vendors in the United States and Table 23 for an international vendor.

Table 22
United States Vendors

Table 22. United States Vendors

Alltech Associates, Inc.
2051 Waukegan Road
Deerfield, IL 60015
800.255.8324
847.948.1078 Fax
www.alltechweb.com

EM Science
P.O. Box 70
480 Democrat Road
Gibbstown, NJ 08027
609.423.6300
www.emscience.com

International Datalink, Inc.
7702 FM 1960 E., Suite 226
Humble, TX 77346
281.852.8880
281.852.8882 Fax
plotrex1@flash.net

Supelco, Inc.
Supelco Park
Bellefonte, PA 16823-0048
800.247.6628
800.447.3044 Fax (US only)
www.sigma-aldrich.com

Turner Designs
845 W. Maude Avenue
Sunnyvale, CA 94086
408.749.0994
408.749.0998 Fax
www.turnerdesigns.com

Upchurch Scientific
619 West Oak Street
Oak Harbor, WA 98277
800.426.0191
800.359.3460 Fax
www.upchurch.com

U.S. GeoSupply, Inc.
Wellsite Geological Supplies
P.O. Box 40217
Grand Junction, CO 81504
970.434.3708 Tel/Fax
www.usgeosupply.com

VWR Scientific
P.O. Box 5025
Sugar Land, TX 77487
800.932.5000
770.232.9881 Fax
www.vwrsp.com

Table 23
International Vendor

Table 23. International Vendor

Norlab Instruments, Ltd.
Site 9, Kirkhill Place
Kirkhill Industrial Estates
Dyce, Aberdeen, AB2 OES. UK
(0224)724849
From US: 011-44-224-724849

Preparing the QFT2™ Standard

It is recommended that you purchase the QFT2™ standard through Supelco, Inc. (see *Vendor Information* for contact information) or through another analytical laboratory.

Should you prefer to prepare the QFT2™ standard; however, this section provides the information you will need to prepare 200 mg/l (+/-0.5%) of a Standard Concentrate in Heptane solvent, where the concentrate is 50 parts p-Xylene to 1 part Phenanthrene by weight (+/-0.25%).

Laboratory Supplies

The laboratory supplies you will need to prepare the Standard are shown in Table 24.

Table 24
Supplies for Preparing the QFT2™ Standard

Table 24. Supplies for Preparing the QFT2™ Standard

Description
Heptane (Spectral Grade or HPLC Grade, 5 liter)
p-Xylene (99.5%+)
Phenanthrene (99.5%+)
Bottle with Teflon® lined cap, 20 cc
Glass Funnel
Volumetric Flask, 5000 cc or 5 liter
Ground glass of PTFE volumetric stopper
Analytical Balance (Mettler AE 163 or equivalent)
Spatula (stainless steel)
Glass transfer pipets
Small rubber bulb
Pipet, 1 ml disposable
Pipet pump

Note: p-xylene and phenanthrene should be of highest available purity (99.5%+). In particular, the phenanthrene must be substantially free of anthracene. Aldrich Chemical (Cat. No. 26087-8, CAS No. 85-01-8) has been found to be satisfactory.

The Preparation Procedure

Preparing the QFT2™ Standard is a two-step process. First, you prepare a 50:1 by weight p-xylene/phenanthrene mixture in amounts sufficient for many lots of QFT2™ standard. And second, as new lots are required, you dilute the concentrate by weight to give 200 mg/l of concentrate in heptane. You should provide the QFT2™ Standard in 100 ml dark glass bottles fitted with tightly sealing Teflon® caps.

To prepare the Standard, do the following:

A. Prepare Standard Concentrate from p-Xylene/Phenanthrene.

1. Make sure the equipment you use is thoroughly clean and dry. Using disposable materials will eliminate contamination.

2. Using an analytical balance that is level and in good adjustment, place the 20 cc bottle on the balance and tare or zero the balance.

3. Using a small spatula, accurately weigh 0.2000 grams of phenanthrene into the bottle.

4. Multiply the exact weight by 50 and add this weight of p-xylene (density=0.861 g/cc). If a slight excess of xylene is added, allow it to volatilize until the desired weight is obtained (+/- 0.25%).

5. Tightly cap the concentrate, add any desired labels, and then record the total weight so that any losses due to long-term leakage can be detected.

B. QFT2™ Standard from Concentrate/Heptane (5 liter = 50 bottle example)

Supplies Needed

- Clean 5 liter glass container
- 5 liters of Spectral Grade Heptane
- Xylene/Phenanthrene Standard Concentrate

1. Weigh the concentrate bottle to confirm that no leakage has occurred.

2. Using a glass transfer pipet, weigh 0.100 grams of concentrate into a small bottle or flask.

3. Multiply the exact weight by 34080 (= 0.6816 g/ml x 5000/0.1) to obtain the final solution weight in grams.

4. Weigh the empty 5 liter container (must be clean of even trace amounts of fluorescing contaminants).

5. Rinse the weighed concentrate into the container using heptane and then add sufficient heptane to obtain the final desired weight.

6. Carefully seal the concentrate bottle and record its new weight.

7. The 5 liters of QFT2™ standard is used to prepare fifty 100 ml bottles of standard in its final form. Clearly mark the bottles "QFT2™ Standard" and indicate the lot number and date prepared.

You may scale this preparation up or down. If you prepare smaller lots, using a two-stage dilution process may provide more accuracy.

Storing and Packing the Standard

1. Store the standard in the light-sensitive glass volumetric flask or glass bottle to avoid exposure to any light. Use Teflon® or glass for the stopper on the storage and packing containers to prevent any leaching action from the solvent.

CAUTION **Never store the Standard in plastic containers.**

2. Keep in a dark, cool place. Remember to agitate the contents before removing your aliquot.

3. You may prolong the shelf life of the Standard by refrigerating it. If stored in the refrigerator, remove the day's aliquot several hours before using and allow this to stabilize or equilibrate to room temperature. Return the stock solution to the refrigerator.

Estimating Solvent Use

To estimate the amount of solvent you will need on a particular job when using the Turner Designs Model 10-AU Dual Filter Fluorometer, use the following guidelines:

1. Estimate the number of samples you will take per one thousand feet of interval you plan to sample.

For every 1000 feet (304.8 m):

- Sampling at 10 foot (3.048 m) intervals = 100 samples
- Sampling at 30 foot (9.14 m) intervals = 33 samples

2. Estimate the amount of solvent you will need based on the number of samples you estimated in step 1.

Average solvent use per sample is 10-15 ml of solvent, as shown in Table 25. Thus, when sampling at 10 foot (3.048 m) intervals, you will need 1.0 to 1.5 liters of solvent per 1000 feet (304.8 m). When sampling at 30 foot (9.14 m) intervals, you will need 0.33 to 0.5 liters per 1000 feet (304.8 m).

Table 25
Average Solvent Required per Sample

Table 25. Average Solvent Required per Sample

Solvent Required (ml)	**Solvent Use**
5	Initial extraction
5–10	Rinsing syringe and possible dilutions
10–15	Total volume per sample

Table 26 shows the solvent required (in liters) by total well depth for sampling frequencies of 10 ft (3m) and 30 ft (9 m).

Table 26
Solvent Required for Various Sampling Intervals

Table 26. Solvent Required for Various Sampling Intervals

Depth (TD) (Feet)	Depth (TD) (Meters)	Sample Frequency: 10 ft (3 m) Interval Solvent Required (Liters)	Sample Frequency: 30 ft (9 m) Interval Solvent Required (Liters)
500	150	0.5–0.75	0.2–0.25
1,000	300	1.0–1.5	0.3–0.5
2,000	600	2.0–3.0	0.7–1.0
3,000	900	3.0–4.5	1.0–1.5
4,000	1,200	4.0–6.0	1.3–2.0
5,000	1,500	5.0–7.5	1.7–2.5
6,000	1,800	6.0–9.0	2.0–3.0
7,000	2,100	7.0–10.5	2.3–3.5
8,000	2,400	8.0–12.0	2.6–4.0
9,000	2,700	9.0–13.5	3.0–4.5
10,000	3,000	10.0–15.0	3.3–5.0

Chapter 8

Material Safety Data Sheets

This chapter contains Material Safety Data Sheets (MSDS) for the QFT2™ standard, for heptane, and for isopropyl alcohol (IPA). These data sheets were provided by suppliers of these chemicals. Each section of the MSDS is shown in an individual table. This chapter contains the following sections:

- **Units and Nomenclature**
- **MSDS for the QFT2™ Standard**
- **MSDS for Heptane**
- **MSDS for Isopropyl Alcohol (IPA)**

Units and Nomenclature

The MSDS units used in this chapter are defined in Table 27, and the MSDS nomenclature is defined in Table 28.

Table 27
Units Used in MSDS

Table 27. MSDS Units

Quantity	Units
Cubic Centimeters	cc
Milligram per Cubic Meter	mg/m^3
Milligram per Kilogram	mg/kg
Millimeters of Mercury	mm HG
Parts Per Million	ppm

Table 28
Nomenclature For MSDS

Table 28. Nomenclature For MSDS

Term	Description	Term	Description
CAS No.	Chemical Abstracts Service Number	**NIOSH**	National Institute of Occupational Safety and Health
CERCLA	Community Environmental Response Compensation and Liability Act	**NTP**	National Toxicology Program
CL	Ceiling Limit	**OSHA**	Occupational Safety and Health Administration
CNS	Central Nervous System	**PEL**	Permissible Exposure Limit
IARC	International Agency for Research on Cancer	**RTECS**	Registry of Toxic Effects of Chemical Substances
LC50	Median Lethal Concentration	**SARA**	Superfund Amendment and Reauthorization Act
LD50	Median Lethal Dose	**STEL**	Short-Term Exposure Limit
LDLO	Lowest Lethal Dose Tested	**TLV**	Threshold Limit Value
LEL	Lower Explosive Limit	**TSD**	Treatment, Storage and Disposal
MSHA	Mining Safety and Health Administration	**TWA**	Time-Weighted Average
NFPA	National Fire Protection Association	**UEL**	Upper Explosive Limit

MSDS for the QFT2™ Standard

The MSDS for the QFT2™ standard was provided by a manufacturer of the QFT2™ standard, Supelco Inc. of Bellefonte, Pennsylvania. This MSDS was last revised by Supelco on February 12, 1998. To contact Supelco, see Table 29.

Supelco, Inc. Disclaimer While the information and recommendations set forth herein are believed to be accurate as of the date hereof (2.12.98), Supelco, Inc. makes no warranty with respect thereto and disclaims all liability from reliance thereon.

Section 1: General Information

General information on the QFT2™ standard and its manufacturer is shown in Table 29.

Table 29
QFT2™ Standard: General Information

Table 29. Section 1 – General Information

	Description
Catalog Number	868091 (Reorder Product by this number)
Product Name	QFT-2 Standard, 1 x 100 ml
Data Sheet Number	868091
Synonym	Analytical Standard in n-Heptane
Manufacturer	Supelco, Inc.
Phone Number	814.359.3441
Address	Supelco Park, Bellefonte, PA 16823-0048

Section 2: Hazardous Ingredients of Mixtures

A description of the hazardous ingredients contained in the QFT2™ standard are shown in Table 30.

Table 30
QFT2™ Standard:
Hazardous Ingredients of Mixtures

Table 30. Section 2 – Hazardous Ingredients of Mixtures

Chemical Name	Common Name	%	CAS No.	Formula	PEL	TLV	LD50 Value (mg/kg)	Conditions
Heptane	n-Heptane	99–100	142-82-5	CH3(CH2)5CH3	400 ppm	400 ppm	222	Intraperitoneal Mouse
Phenanthrene	Phenanthrene	0.0019	85-01-8	C14H10	N/A	0.2 mg/m3	700	Oral Mouse 4
Benzene, 1, 4-Dimethyl-	P-Xylene	0.0980	106-42-3	CH3C6H4CH3	100 ppm	100 ppm	5,000	Oral Rat 6

Footnotes: 4 Classified by IARC as a Class 3 carcinogen.
6 Subject to the reporting requirements of SARA Title III, Section 313.

Section 3: Physical Data

Physical data for the QFT2™ standard are shown in Table 31.

Table 31
QFT2™ Standard: Physical Data

Table 31. Section 3 – Physical Data

Physical Property	Description
Boiling Point	98˚ C
Melting Point	-91˚ C
Vapor Pressure	40 mm
Vapor Density	3.5 (Air=1)
Specific Gravity	0.690 g/ml (20˚ C, Water = 1)
Percent Volatile by Volume	100
Water Solubility	N/A
Evaporation Rate	4.3 (Butyl Acetate = 1)
Appearance	Clear, colorless liquid
Odor	Gasoline odor

Section 4: Fire and Explosion Hazard Data

Fire and explosion hazard data for the QFT2™ standard are shown in Table 32.

Table 32
QFT2™ Standard: Fire and Explosion Hazard Data

Table 32. Section 4 – Fire and Explosion Hazard Data

	Description
Flash Point	30˚ F
Flammable Limits	LEL 1.1, UEL 6.7
Extinguishing Media	CO_2, Foam, Dry Chemical Water may be ineffective.
Special Fire Fighting Procedures	Wear self-contained breathing apparatus when fighting a chemical fire.
Unusual Fire and Explosion Hazards	Vapors form explosive mixtures with air. Containers may explode under fire conditions.

Section 5: Health Hazard Data

Health hazard data for the QFT2™ standard are shown in Table 33.

Table 33
QFT2™ Standard: Health Hazard Data

Table 33. Section 5 – Health Hazard Data

Physical Property	Description
LD50 Value	222 mg/kg
Conditions	Intraperitoneal Mouse
TLV	400 ppm
PEL	400 ppm
Emergency and First Aid Procedures	
Eyes	Flush eyes with water for 15 minutes.
Skin	Remove contaminated clothing.
Inhalation	Immediately move to fresh air. Give oxygen if breathing is labored. If breathing stops, give artificial respiration.
Ingestion	Never give anything by mouth to an unconscious person. Never try to make an unconscious person vomit. Do not induce vomiting. Give large amounts of water. Give large amounts of milk.
Effects of Overexposure	
	May irritate eyes and/or skin. Animal teratogen, possible reproductive hazard. Dermatitis. Irritates nose and throat. Pulmonary edema. Headache. Nausea. Dizziness. Gastro-intestinal disturbances. Depresses central nervous system. Liver damage. Kidney damage. Reported mutagen. Carcinogenicity–indefinite in animals. Aspiration causes severe lung irritation–coughing excitement followed by depression.

Section 6: Reactivity Data

Reactivity data for the QFT2™ standard are shown in Table 34.

Table 34
QFT2™ Standard: Reactivity Data

Table 34. Section 6 – Reactivity Data

	Description
Stability	Stable
Conditions to Avoid	N/A
Incompatibility	Oxidizing agents, Fluorine, Chlorine, and Bromine
Hazardous Decomposition Products	N/A
Hazardous Polymerization	Will not occur.

Section 7: Procedures for Spills and Leaks

Procedures for spills and leaks when working with the QFT2™ standard are shown in Table 35.

Table 35
QFT2™ Standard: Procedures for Spills and Leaks

Table 35. Section 7 – Procedures for Spills and Leaks

Event	Action to Take
Material Release or Spill	Take up with absorbent material. Ventilate area. Eliminate all ignition sources.
Water Disposal Method	Comply with all applicable federal, state, or local regulations.

Section 8: Special Protection Information

Information on special protection required when working with the QFT2™ standard is shown in Table 36.

Table 36
QFT2™ Standard: Special Protection Information

Table 36. Section 8 – Special Protection Information

Item	Description
Respiratory Protection (specific type)	Wear self-contained breathing apparatus.
Protective Gloves	Wear neoprene gloves.
Eye Protection	Wear protective glasses.
Ventilation	Use only in well ventilated area.
Special	N/A
Other Protective Equipment	N/A

Section 9: Special Precautions

Special precautions required when working with the QFT2™ standard are shown in Table 37.

Table 37
QFT2™ Standard: Special Precautions

Table 37. Section 9 – Special Precautions

Activity	Precautions
Storage and Handling	Refrigerate in sealed container.
	Keep away from heat.
	Keep away from ignition sources.
Other Precautions	Avoid eye or skin contact.
	Avoid breathing vapors.

MSDS for Heptane

The Material Safety Data Sheet (MSDS) for heptane was provided by EM Science (A division of EM Industries) of Gibbstown, New Jersey. This MSDS was last revised by EM Science on November 20, 1991. To contact EM Science for more information, see Table 38.

Table 38
QFT2™ Standard: Chemical Product and Company Identification

Table 38. Section 1 – Chemical Product and Company Identification

	Description
Data Sheet Number	HX0074
Catalog Numbers	HX0074, HX0076, HX0078, HX0079, HX0080, HX0080P, HX0082, 9686
Product Name	Heptane
Synonyms	n-Heptane
Chemical Family	Aliphatic Hydrocarbon
Formula	CH3(CH2)5CH3
Molecular Weight	100.21
Manufacturer	EM Science (A division of EM Industries)
Information Phone Number	609.423.6300
Chemtrec Emergency Phone Number	800.424.9300
Address	P.O. Box 70 480 Democrat Road Gibbstown, NJ 08027

EM Science Disclaimer

The statements contained herein are offered for informational purposes only and are based upon technical data that EM Science believes to be accurate. It is intended for use only by persons having the necessary technical skill and at their own discretion and risk. Since conditions and manner of use are outside our control, we make no warranty, express or implied, of merchantability, fitness or otherwise.

Section 1: Chemical Product and Company Identification

Chemical product and company identification information for heptane is shown in Table 38.

Section 2: Composition / Information on Ingredients

The composition and information on ingredients for heptane are shown in Table 39.

Table 39
Heptane: Composition/Information on Ingredients

Table 39. Section 2 – Composition / Information on Ingredients		
Component	**CAS #**	**Appr. %**
Heptane	142-82-5	100%

Section 3: Hazards Identification

Hazards identification information for heptane is shown in Table 40.

Table 40
Heptane:
Hazards Identification

Table 40. Section 3 – Hazards Identification

	Description
Emergency Overview	Flammable liquid and vapor. Harmful if inhaled or swallowed. Irritating to skin, eyes, and mucous membranes. May cause damage to lungs and central nervous system.
Appearance	Colorless liquid; characteristic odor.
Potential Health Effects (Acute and Chronic)	**Symptoms of Exposure:** Irritating on contact with skin, eyes, or mucous membranes. Vapor irritating to eyes and respiratory passages. Severely irritating and damaging to lungs and central nervous systems. Can cause vertigo, incoordination; narcotic at high concentrations. **Medical Conditions Aggravated by Exposure:** Respiratory and CNS conditions. **Routes of Entry:** Inhalation, ingestion. **Carcinogenicity:** The material is not listed (IARC, NTP, OSHA) as a cancer causing agent.

Section 4: First Aid Measures

First aid measures for heptane are shown in Table 41.

Table 41
Heptane:
First Aid Measures

Table 41. Section 4 – First Aid Measures

Condition	Emergency First Aid
Overexposure	Get medical assistance for all cases of overexposure.
Skin Exposure	Wash thoroughly with soap and water.
Eyes Exposure	Immediately flush thoroughly with water for at least 15 minutes.
Inhalation	Remove to fresh air. Give artificial respiration if breathing has stopped.
Ingestion	Call a physician immediately. Only induce vomiting at the instructions of a physician. Never give anything by mouth to an unconscious person.

Section 5: Fire Fighting Measures

Fire fighting measures for heptane are shown in Table 42.

Table 42
Heptane:
Fire Fighting Measures

Table 42. Section 5 – Fire Fighting Measures

	Description
Flash Point	25˚ F (cc)
Flammable Limits LEL (%)	1.05
Flammable Limits UEL (%)	6.70
Extinguishing Media	CO_2, Dry Chemical, Foam. Use water spray to cool exposed containers and disperse vapors.
Fire Fighting Procedures	Wear self-contained breathing apparatus.
Fire and Explosion Hazards	Dangerous fire and explosion hazard. Vapor can travel distances to ignition source and flash back.

Section 6: Accidental Release Measures

Procedures for responding to accidental releases of heptane are shown in Table 43.

Table 43
Heptane: Accidental Release Measures

Table 43. Section 6 – Accidental Release Measures

Spill Response

- Evacuate the area of all unnecessary personnel.
- Wear suitable protective equipment listed under *Exposure Controls / Personal Protection* (Section 8).
- Eliminate any ignition sources until the area is determined to be free from explosion or fire hazards.
- Contain the release and eliminate its source, if this can be done without risk.
- Take up and containerize for proper disposal as described under *Disposal Considerations* (Section 12).
- Comply with Federal, State, and Local regulations on reporting releases. Refer to regulatory information for reportable quantity and other regulatory data.
- EM Science recommends Spill-X absorbent agents for various types of spills. For this product, the *Solvent Spill Treatment Kit* (Order No. SX0863) is recommended.

  Additional information on Spill-X products can be obtained through the EM Science technical service department at 609.354.9200.

Section 7: Handling and Storage

Procedures for handling and storing heptane are shown in Table 44.

Table 44
Heptane:
Handling and Storage

Table 44. Section 7 – Handling and Storage
• Keep container tightly closed. • Store in a cool, well-ventilated area away from strong oxidizers. • Do not breathe vapor. • Do not get in eyes, on skin, or on clothing. • Electrically ground all equipment when handling this product.

Section 8: Exposure Controls / Personal Protection

Exposure controls and personal protection for heptane are shown in Table 45a.

Table 45a
Heptane: Exposure Controls/ Personal Protection

Table 45a. Section 8 – Exposure Controls / Personal Protection

Engineering Controls and Personal Protective Equipment

- Ventilation, Respiratory Protection, Protective Clothing, and Eye Protection.
- Material must be handled or transferred in an approved fume hood or with equivalent ventilation.
- Protective gloves should be worn to prevent skin contact (Nitrile or equivalent).
- Safety glasses with side shields must be worn at all times.

Respiratory Protection

- If workplace exposure limit(s) of product or any component is exceeded (see TLV/PEL), a NIOSH / MSHA approved air supplied respirator is advised in absence of proper environmental control. OSHA regulations also permit other NIOSH / MSHA respirators (negative pressure type) under specified conditions (see your safety equipment supplier). Engineering and/or administrative controls should be implemented to reduce exposure.

Work / Hygienic Practices

- Wash thoroughly after handling.
- Do not take internally.
- Eye wash and safety equipment should be readily available.

OSHA (PEL) exposure guidelines for heptane are shown in Table 45b.

Table 45b
Heptane:
OSHA (PEL) Exposure Guidelines

Table 45b. Section 8 – OSHA (PEL) Exposure Guidelines for Heptane

	TWA		STEL		CL		
Component	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	Skin
Heptane	400	1600	500	2000			

ACGIH (TLV) exposure guidelines for heptane are shown in Table 45c.

Table 45c
Heptane:
ACGIH (TLV) Exposure Guidelines

Table 45c. Section 8 – ACGIH (TLV) Exposure Guidelines for Heptane

	TWA		STEL		CL		
Component	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	Skin
Heptane	400	1640	500	2050			

Section 9: Physical and Chemical Properties

Physical and chemical properties for heptane are shown in Table 46.

Table 46
Heptane:
Physical and Chemical Properties

Table 46. Section 9 – Physical and Chemical Properties

Property	Description
Boiling Point	98˚ C (760 mm HG)
Melting Point	-90.5˚ C
Specific Gravity	0.684 (20˚ C, Water = 1)
Vapor Pressure	40 mm HG (20˚ C)
Percent Volatile by Volume	100 %
Vapor Density	3.5 (Air=1)
Evaporation Rate	4.3 (Butyl Acetate = 1)
Solubility in Water	Insoluble
Appearance	Colorless liquid; characteristic odor.

Section 10: Stability and Reactivity

Stability and reactivity information for heptane is shown in Table 47.

Table 47
Heptane:
Stability and Reactivity

Table 47. Section 10 – Stability and Reactivity

	Description
Stability	Yes
Hazardous Polymerization	Does not occur.
Hazardous Decomposition	COX
Conditions to Avoid	Heat; vapor contact with ignition sources.
Materials to Avoid	Oxidizers, Phosphorus, and Chlorine

Section 11: Toxicological Information

Toxicological information for heptane is shown in Table 48.

Table 48
Heptane:
Toxicological Information

Table 48. Section 11 – Toxicological Information

	Description
Toxicity Data	1HL–HMN TCLO: 1,000 PPM/6M
Toxicological Findings	None. Cited in Registry of Toxic Effects of Chemical Substances (RTECS).

Section 12: Disposal Considerations

Disposal considerations for heptane are shown in Table 49.

Table 49
Heptane:
Disposal Considerations

Table 49. Section 12 – Disposal Considerations

	Description
EPA Waste Numbers	D001
Treatment	Incineration, fuels blending or recycle. Contact your local permitted waste disposal site (TSD) for permissible treatment sites. Always contact a permitted waste disposer (TSD) to assure compliance with all current local, state, and federal regulations.

Section 13: Transport Information

Transport information for heptane is shown in Table 50.

Table 50
Heptane:
Transport Information

Table 50. Section 13 – Transport Information

	Description
DOT Proper Shipping Name	Heptane
DOT ID Number	UN1206

Section 14: Regulatory Information

Regulatory information for heptane is shown in Table 51.

Table 51
Heptane:
Regulatory Information

Table 51. Section 14 – Regulatory Information

Component	SARA EHS (302)	SARA EHS TPQ (LBS)	CERCLA RQ (LBS)	OSHA Floor List	SARA 313	DEMINIMIS For SARA 313
Heptane				Y		

Section 15: Other Information

Other information for heptane is shown in Table 52.

Table 52
Heptane:
Other Information

Table 52. Section 15 – Other Information

	Description
NFPA Hazard Ratings	Health: 1 Flammability: 3 Reactivity: 0
Comments	None

MSDS for Isopropyl Alcohol (IPA)

The Material Safety Data Sheet (MSDS) for isopropyl alcohol (IPA) was provided by EM Science (A division of EM Industries) of Gibbstown, New Jersey. This MSDS was last revised by EM Science on October 11, 1993. To contact EM Science for more information, see Table 53.

Table 53
Isopropyl Alcohol: Chemical Product and Company Identification

Table 53. Section 1 – Chemical Product and Company Identification

	Description
Data Sheet Number	PX1830
Catalog Numbers	9634, PX1830, PX1834, PX1834PM, PX1835, PX1835P, PX1835S, PX1835T, PX1836, PX1837, PX1838, PX1838P
Product Name	Iso-Propyl Alcohol
Trade Name	Isopropanol, 2-Propanol
Chemical Family	Aliphatic Alcohol
Formula	CH3CHOHCH3
Molecular Weight	60.10
Manufacturer	EM Science (A division of EM Industries)
Information Phone Number	609.423.6300
Chemtrec Emergency Phone Number	800.424.9300
Address	P.O. Box 70 480 Democrat Road Gibbstown, NJ 08027

EM Science Disclaimer

The statements contained herein are offered for informational purposes only and are based upon technical data that EM Science believes to be accurate. It is intended for use only by persons having the necessary technical skill and at their own discretion and risk. Since conditions and manner of use are outside our control, we make no warranty, express or implied, of merchantability, fitness or otherwise.

Section 1: Chemical Product and Company Identification

Chemical product and company identification information for isopropyl alcohol (IPA) is shown in Table 53.

Section 2: Composition / Information on Ingredients

The composition and information on ingredients for isopropyl alcohol are shown in Table 54.

Table 54
Isopropyl Alcohol: Composition/Information on Ingredients

Table 54. Section 2 – Composition / Information on Ingredients		
Component	**CAS #**	**Appr. %**
Isopropyl Alcohol	67-63-0	100%

Section 3: Hazards Identification

Hazards identification information for isopropyl alcohol is shown in Table 55.

Table 55
Isopropyl Alcohol: Hazards Identification

Table 55. Section 3 – Hazards Identification

	Description
Emergency Overview	Flammable liquid and vapor. May cause eye injury. Harmful if inhaled or swallowed.
Appearance	Clear, colorless liquid; characteristic odor.
Potential Health Effects (Acute and Chronic)	**Effects of Ingestion or Inhalation:** Nausea, intoxication, central nervous system depression, headache, decreased blood rate, coma. High vapor concentrations cause irritation of eyes and respiratory system. Contact may cause eye injury, skin irritation. **Medical Conditions Aggravated by Exposure:** Respiratory and skin conditions. **Routes of Entry:** Inhalation, ingestion, or skin contact. **Carcinogenicity:** The material is not listed (IARC, NTP, OSHA) as a cancer causing agent.

Section 4: First Aid Measures

First aid measures for isopropyl alcohol are shown in Table 56.

Table 56
Isopropyl Alcohol: First Aid Measures

Table 56. Section 4 – First Aid Measures

Condition	Emergency First Aid
Overexposure	Get medical assistance for all cases of overexposure.
Skin Exposure	Wash thoroughly with soap and water.
Eyes Exposure	Immediately flush thoroughly with water for at least 15 minutes.
Inhalation	Remove to fresh air. Give artificial respiration if breathing has stopped.
Ingestion	If conscious, drink water and induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person.

Section 5: Fire Fighting Measures

Fire fighting measures for isopropyl alcohol are shown in Table 57.

Table 57
Isopropyl Alcohol: Fire Fighting Measures

Table 57. Section 5 – Fire Fighting Measures

	Description
Flash Point	53˚ F (TCC)
Flammable Limits LEL (%)	2
Flammable Limits UEL (%)	12
Extinguishing Media	CO_2; Dry Chemical; "Alcohol" Foam; Water spray to cool fire-exposed containers and disperse vapors.
Fire Fighting Procedures	Wear self-contained breathing apparatus.
Fire and Explosion Hazards	Dangerous fire and explosion hazard. Vapor can travel distances to ignition source and flash back. Hot organic chemical vapors or mists are susceptible to sudden spontaneous combustion when mixed with air. Ignition may occur at temperatures below published autoignition or ignition temperatures. Ignition temperatures decrease with increasing vapor volume and vapor/air contact time and are influenced by pressure changes. Ignition may occur at typical elevated temperature process conditions, especially in process operating under vacuum if subjected to sudden ingress of air, or outside process equipment operating under elevated pressure if sudden escape of vapors or mists to the atmosphere occurs.

Section 6: Accidental Release Measures

Procedures for responding to accidental releases of isopropyl alcohol are shown in Table 58.

Table 58
Isopropyl Alcohol: Accidental Release Measures

Table 58. Section 6 – Accidental Release Measures
Spill Response • Evacuate the area of all unnecessary personnel. • Wear suitable protective equipment listed under *Exposure Controls / Personal Protection* (Section 8). • Eliminate any ignition sources until the area is determined to be free from explosion or fire hazards. • Contain the release and eliminate its source, if this can be done without risk. • Take up and containerize for proper disposal as described under *Disposal Considerations* (Section 12). • Comply with Federal, State, and Local regulations on reporting releases. Refer to regulatory information for reportable quantity and other regulatory data. • EM Science recommends Spill-X absorbent agents for various types of spills. For this product, the *Solvent Spill Treatment Kit* (Order No. SX0863) is recommended. Additional information on Spill-X products can be obtained through the EM Science technical service department at 609.354.9200.

Section 7: Handling and Storage

Procedures for handling and storing isopropyl alcohol are shown in Table 59.

Table 59
Isopropyl Alcohol: Handling and Storage

Table 59. Section 7 – Handling and Storage
• Keep container closed. • Store in a cool area away from ignition sources and oxidizers.
• Do not breathe vapor. • Do not get in eyes. • Avoid prolonged or repeated skin contact. • Electrically ground all equipment when handling this product.

Section 8: Exposure Controls / Personal Protection

Exposure controls and personal protection for isopropyl alcohol are shown in Table 60a.

Table 60a
Isopropyl Alcohol: Exposure Controls/ Personal Protection

Table 60a. Section 8 – Exposure Controls / Personal Protection

Engineering Controls and Personal Protective Equipment

- Ventilation, Respiratory Protection, Protective Clothing, and Eye Protection.
- Protective gloves (Butyl rubber, PVC, or equivalent) should be worn to prevent skin contact.
- Safety glasses with side shields must be worn at all times.

Respiratory Protection

- If workplace exposure limit(s) of product or any component is exceeded (see TLV/PEL), a NIOSH / MSHA approved air supplied respirator is advised in absence of proper environmental control. OSHA regulations also permit other NIOSH / MSHA respirators (negative pressure type) under specified conditions (see your safety equipment supplier). Engineering and/or administrative controls should be implemented to reduce exposure.
- Material must be handled or transferred in an approved fume hood or with adequate ventilation.

Work / Hygienic Practices

- Wash thoroughly after handling.
- Do not take internally.
- Eye wash and safety equipment should be readily available.

OSHA (PEL) exposure guidelines for isopropyl alcohol are shown in Table 60b.

Table 60b
Isopropyl Alcohol: OSHA (PEL) Exposure Guidelines

Table 60b. Section 8 – OSHA (PEL) Exposure Guidelines for Isopropyl Alcohol

	TWA		STEL		CL		
Component	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	Skin
Isopropyl Alcohol	400	980	500	1225			

ACGIH (TLV) exposure guidelines for isopropyl alcohol are shown in Table 60c.

Table 60c
Isopropyl Alcohol: ACGIH (TLV) Exposure Guidelines

Table 60c. Section 8 – ACGIH (TLV) Exposure Guidelines for Isopropyl Alcohol

	TWA		STEL		CL		
Component	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	(PPM)	(Mg/M^3)	Skin
Isopropyl Alcohol	400	983	500	1230			

Section 9: Physical and Chemical Properties

Physical and chemical properties for isopropyl alcohol are shown in Table 61.

Table 61
Isopropyl Alcohol: Physical and Chemical Properties

Table 61. Section 9 – Physical and Chemical Properties

Property	Description
Boiling Point	82.3˚ C (760 mm HG)
Melting Point	-88.5˚ C
Specific Gravity	0.7854 (Water = 1)
Vapor Pressure	33 mm HG (20˚ C)
Percent Volatile by Volume	100 %
Vapor Density	2.07 (Air=1)
Evaporation Rate	2.88 (Butyl Acetate=1)
Solubility in Water	Miscible
Appearance	Clear, colorless liquid; characteristic odor.

Section 10: Stability and Reactivity

Stability and reactivity information for isopropyl alcohol is shown in Table 62.

Table 62
Isopropyl Alcohol: Stability and Reactivity

Table 62. Section 10 – Stability and Reactivity

	Description
Stability	Yes
Hazardous Polymerization	Does not occur.
Hazardous Decomposition	COX
Conditions to Avoid	Heat; contact with ignition source.
Materials to Avoid	Acids; Oxidizers, Halogens; Halogen Compounds; and Aldehydes.

Section 11: Toxicological Information

Toxicological information for isopropyl alcohol is shown in Table 63.

Table 63
Isopropyl Alcohol: Toxicological Information

Table 63. Section 11 – Toxicological Information

	Description
Toxicity Data	ORL–HMN LDLO: 3,570 mg/kg ORL–RAT LD50: 5,045 mg/kg IHL–RAT LC50: 16,000 PPM/8H
Toxicological Findings	Tests on laboratory animals indicate material may produce adverse mutagenic and reproductive effects. Cited in Registry of Toxic Effects of Substances (RTECS).

Section 12: Disposal Considerations

Disposal considerations for isopropyl alcohol are shown in Table 64.

Table 64
Isopropyl Alcohol: Disposal Considerations

Table 64. Section 12 – Disposal Considerations

	Description
EPA Waste Numbers	D001
Treatment	Incineration, fuels blending or recycle. Contact your local permitted waste disposal site (TSD) for permissible treatment sites. Always contact a permitted waste disposer (TSD) to assure compliance with all current local, state, and federal regulations.

Section 13: Transport Information

Transport information for isopropyl alcohol is shown in Table 65.

Table 65
Isopropyl Alcohol: Transport Information

Table 65. Section 13 – Transport Information

	Description
DOT Proper Shipping Name	Isopropanol (For all catalog numbers except PX1837).
DOT ID Number	UN1219

Section 14: Regulatory Information

Regulatory information for isopropyl alcohol is shown in Table 66.

Table 66
Isopropyl Alcohol: Regulatory Information

Table 66. Section 14 – Regulatory Information

Component	SARA EHS (302)	SARA EHS TPQ (LBS)	CERCLA RQ (LBS)	OSHA Floor List	SARA 313	DEMINIMIS For SARA 313
Isopropyl Alcohol				Y	Y	0.1

Section 15: Other Information

Other information for isopropyl alcohol is shown in Table 67.

Table 67
Isopropyl Alcohol: Other Information

Table 67. Section 15 – Other Information

	Description
NFPA Hazard Ratings	Health: 1 Flammability: 3 Reactivity: 0
Comments	None

■■■■■

Chapter 9

Fluorometer Quick Start Operating Instructions

This chapter contains the Turner Designs Quick Start Operating Instructions (*version 1.1, October 1998*) for the Model 10-AU-005-CE fluorometer.

IMPORTANT

QFT2™ analysis requires a Turner Designs Model 10-AU-005 Fluorometer. For specific instructions on setting up and calibrating this instrument for use with QFT2™, see Chapter 6.

1. INTRODUCTION

These instructions are designed to provide an overview of the functions of your 10-AU-005-CE Fluorometer. They include basic information to help you get started, and should be adequate for straightforward measurements involving rhodamine WT or fluorescein (follow the procedures for rhodamine in these instructions) or extracted chlorophyll. For Short or Long Wavelength Oil, refer to the user's manual.

The procedures and functions in these "Quick Start" pages are discussed in detail in the *10-AU-005-CE Fluorometer User's Manual*. If you run into any difficulty while following these instructions, please refer to your user's manual. The user's manual also contains information about optical filters, principles of fluorescence, data collection, sample system, and much more. Consult the table of contents or the index of the field or laboratory user's manual for the location of pertinent information.

To operate your fluorometer:

Step 1. Set the basic operating level. (You will only need to do this once, before operating your instrument for the first time.) See pages 143-144.

Step 2. Calibrate the instrument. See pages 145-148.

Step 3. Read samples. See pages 149-150.

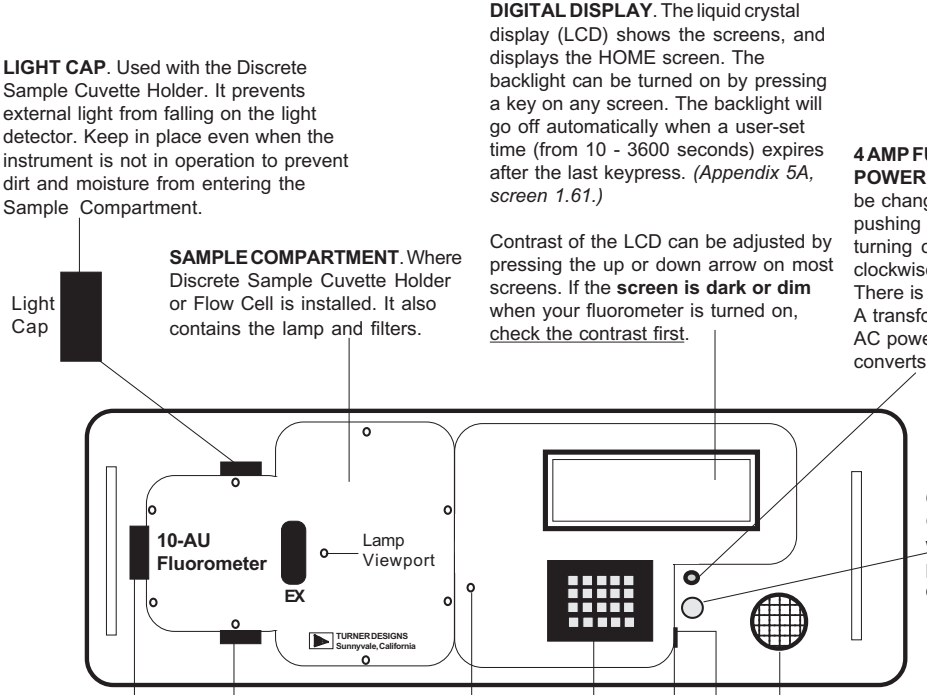
2. INSTRUMENT CONTROLS & INDICATORS

LIGHT CAP. Used with the Discrete Sample Cuvette Holder. It prevents external light from falling on the light detector. Keep in place even when the instrument is not in operation to prevent dirt and moisture from entering the Sample Compartment.

DIGITAL DISPLAY. The liquid crystal display (LCD) shows the screens, and displays the HOME screen. The backlight can be turned on by pressing a key on any screen. The backlight will go off automatically when a user-set time (from 10 - 3600 seconds) expires after the last keypress. *(Appendix 5A, screen 1.61.)*

Contrast of the LCD can be adjusted by pressing the up or down arrow on most screens. If the **screen is dark or dim** when your fluorometer is turned on, check the contrast first.

4 AMP FUSE (DC POWER). Fuse can be changed by pushing in and turning counter-clockwise. NOTE: There is no AC fuse. A transformer on the AC power cable converts AC to DC.



SENSITIVITY ADJUSTMENT KNOB. This recessed black screw sets the basic operating level (sensitivity) of the instrument, and is locked into position by the Sensitivity Adjustment Lock. The level must be set BEFORE calibrating for the first time. Thereafter, lock it down and DO NOT adjust unless you want to change the operating level significantly, or unless you change to a different optical filter kit. After the initial adjustment of this knob, sensitivity will be set during calibration using the keypad. You MUST recalibrate your instrument if the knob is adjusted. *See p. 143 or Appendix 6B of the user's manual.*

3. DIGITAL DISPLAY AND SCREENS OVERVIEW

(See the Screens Flow Chart, next page.)

1. **SCREENS**. Built into the fluorometer are a series of computerized screens, which are called up using the keypad and shown on the digital display. For easy identification, the **active screens** have a number in the **lower or upper right-hand corner**. Instructions and changes are entered on the keypad.

2. **DIGITAL DISPLAY CONTRAST**. The contrast of the LCD can be adjusted on any screen, except screen 2.11 (Run Blank) and screen 2.3 (Run Standard), by pressing ↑ or ↓.

   NOTE: If the screen is dark or dim when your fluorometer is turned on, check the contrast first. It is possible that the contrast has been decreased so much that the screen is too dark to view.

3. **HOME Screen**. When the instrument is turned on, the HOME screen is displayed (except when accessing or viewing other screens). Samples are read on the HOME screen. See page 150.

   If an alarm is activated when the instrument is operating, the words “ALARM ON!” will flash in the upper right hand portion of the HOME screen. Press <ESC> to see the alarm screen.

   You can usually **go to the HOME screen by pressing <HOME>**, except while in a help screen. **You must first exit the help screen** by pressing <ESC>.

4. **MAIN MENU**. From the HOME screen, you can access the Main Menu by pressing <ENT>. From the Main Menu, press a number to access:
   1. Operational parameters (not reviewed in these instructions; *refer to Appendix 5A of the user's manual*);
   2. Calibration (see pages145-148);
   3. Diagnostic information (see pages 151-152);
   4. Clock (to set, press <4> and follow instructions on the screen).

   NOTE: If the security ID is enabled, the fluorometer will ask for an ID entry before access to screens other than HOME is allowed.

5. **HELP SCREENS**. The HOME screen, the Main Menu, and the calibration screens have help screens, called up by pressing <?>, which list the commands and instructions for the screens.

   NOTE: <u>You MUST exit the help screen by pressing <ESC>, before keying other commands</u>. The system will ignore commands given unless the help screen is exited first.

6. **WARNING SCREENS**. There are warning screens throughout, which will alert you to invalid entries.

   NVRAM WARNING SCREEN. After several years, a warning screen will be displayed when the instrument is turned on: "WARNING! NEW NVRAM, <1> to set default." This message indicates that the NVRAM internal battery is low. The instrument will still operate, but every time it is turned off, it will lose instrument settings and calibration settings. See Section 4 of the user's manual for details.

7. **ALARM SCREEN**. An alarm screen indicates what alarm(s) has been triggered and diagnostic screens provide information on various internal fluorometer functions.

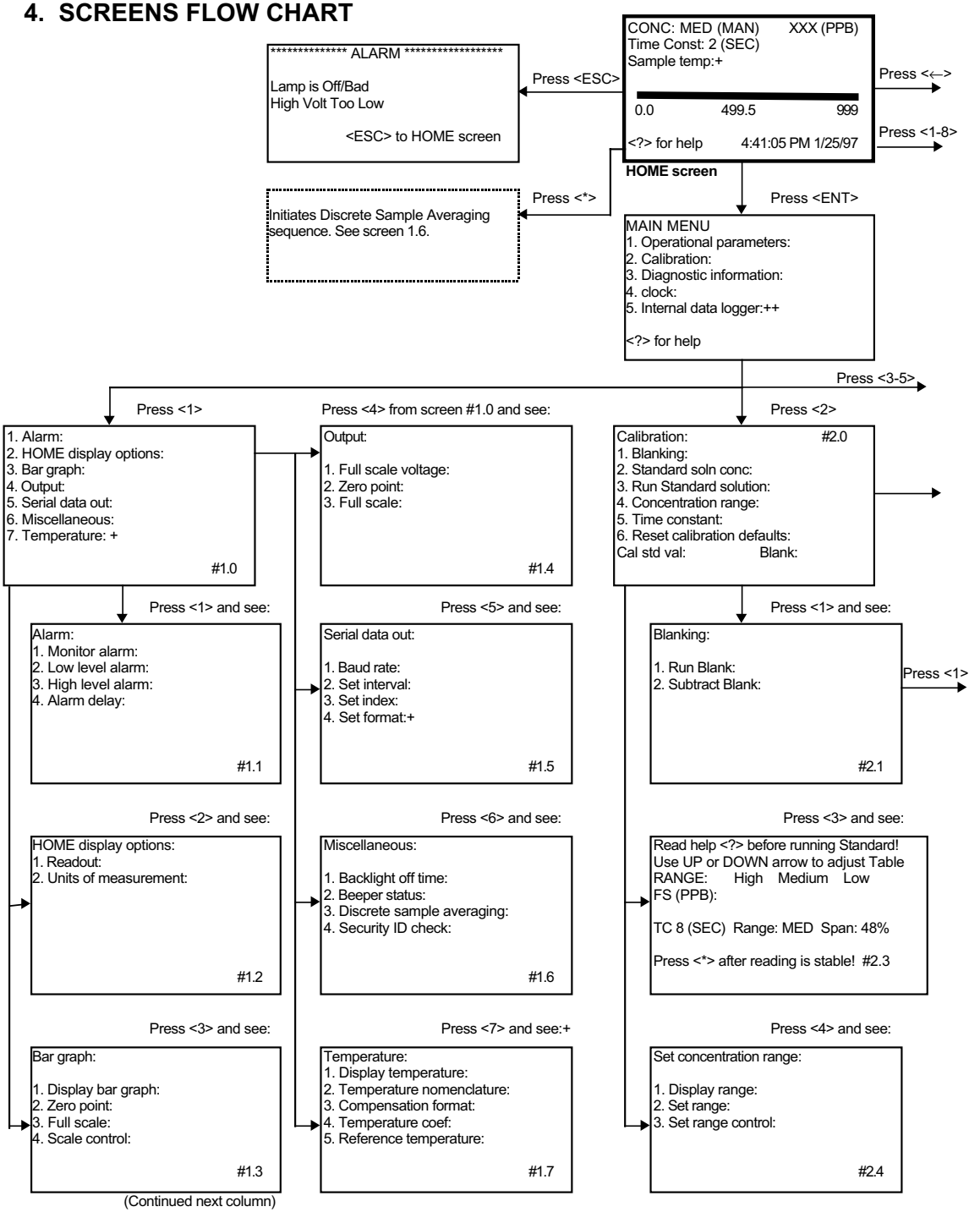
8. **MOVING THROUGH SCREENS**. The screens provide instructions on how to move back and forth between them; in most cases you use <ENT>. Pressing the number for a menu item accesses the screen for that function.

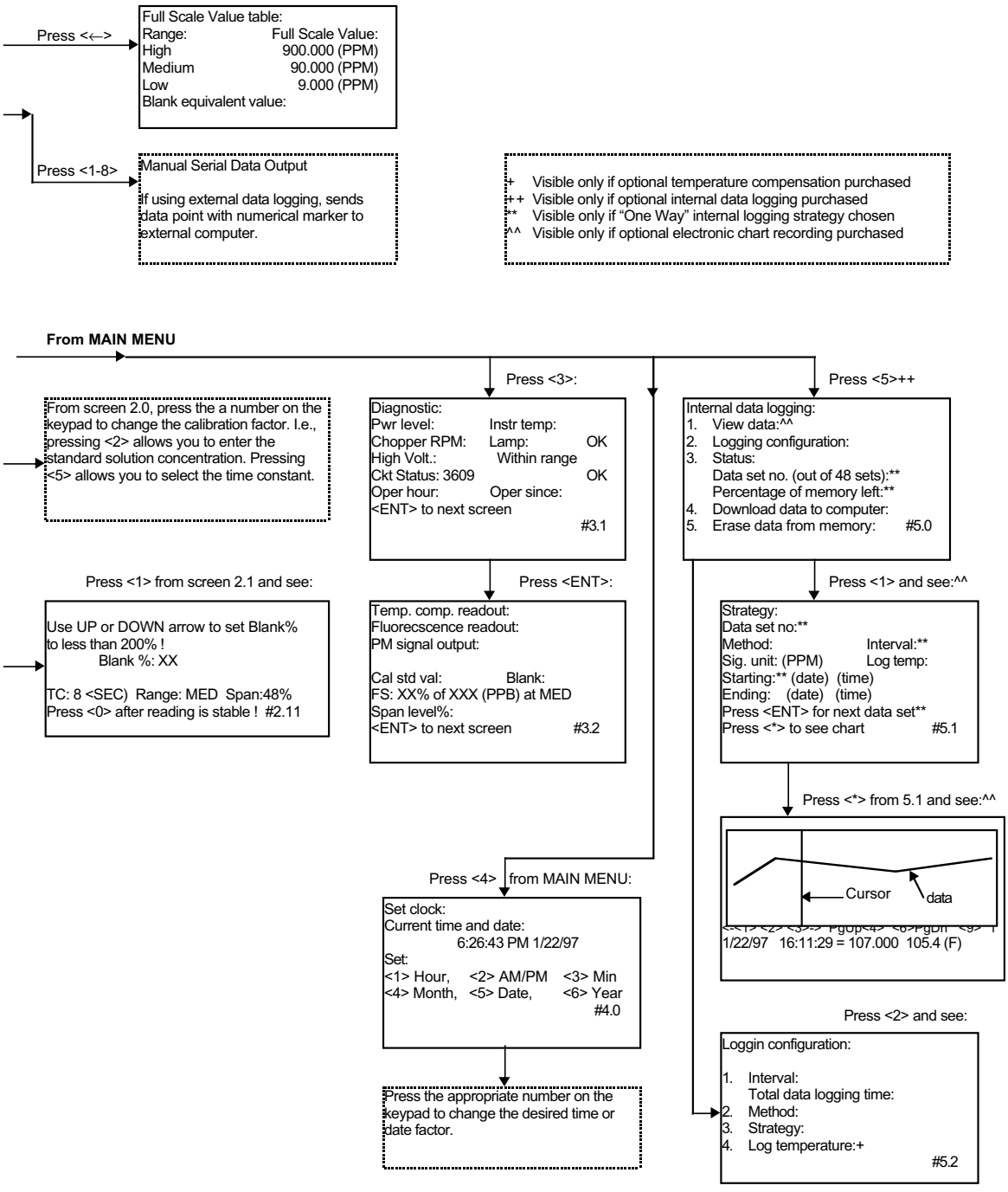
   You can escape to the previous screen by pressing <ESC>.

9. **CORRECTING ERRORS**. The **left arrow** can be used to **correct typing** errors when data are being entered or changed. It acts as a backspace or delete key.

10. **RESPONSE DELAY**. Under certain conditions (i.e., when sensitivity is adjusted, or instrument settings are changed, etc.), the **digital display will not react immediately** to the change, but will respond after a delay of about 10 seconds.

4. SCREENS FLOW CHART




Press <←>
Full Scale Value table:
Range: Full Scale Value:
High 900.000 (PPM)
Medium 90.000 (PPM)
Low 9.000 (PPM)
Blank equivalent value:
Press <1-8>
Manual Serial Data Output
If using external data logging, sends data point with numerical marker to external computer.
+ Visible only if optional temperature compensation purchased
++ Visible only if optional internal data logging purchased
** Visible only if "One Way" internal logging strategy chosen
^^ Visible only if optional electronic chart recording purchased
From MAIN MENU
From screen 2.0, press the a number on the keypad to change the calibration factor. I.e., pressing <2> allows you to enter the standard solution concentration. Pressing <5> allows you to select the time constant.
Press <3>:
Diagnostic:
Pwr level: Instr temp:
Chopper RPM: Lamp: OK
High Volt.: Within range
Ckt Status: 3609 OK
Oper hour: Oper since:
<ENT> to next screen
#3.1
Press <5>++
Internal data logging:
1. View data:^^
2. Logging configuration:
3. Status:
Data set no. (out of 48 sets):**
Percentage of memory left:**
4. Download data to computer:
5. Erase data from memory: #5.0
Press <1> from screen 2.1 and see:
Use UP or DOWN arrow to set Blank% to less than 200% !
Blank %: XX
TC: 8 <SEC) Range: MED Span:48%
Press <0> after reading is stable ! #2.11
Press <ENT>:
Temp. comp. readout:
Fluorecscence readout:
PM signal output:
Cal std val: Blank:
FS: XX% of XXX (PPB) at MED
Span level%:
<ENT> to next screen #3.2
Press <1> and see:^^
Strategy:
Data set no:**
Method: Interval:**
Sig. unit: (PPM) Log temp:
Starting:** (date) (time)
Ending: (date) (time)
Press <ENT> for next data set**
Press <*> to see chart #5.1
Press <*> from 5.1 and see:^^
Cursor
data
1/22/97 16:11:29 = 107.000 105.4 (F)
Press <4> from MAIN MENU:
Set clock:
Current time and date:
6:26:43 PM 1/22/97
Set:
<1> Hour, <2> AM/PM <3> Min
<4> Month, <5> Date, <6> Year
#4.0
Press the appropriate number on the keypad to change the desired time or date factor.
Press <2> and see:
Loggin configuration:
1. Interval:
Total data logging time:
2. Method:
3. Strategy:
4. Log temperature:+
#5.2

5. SETTING THE BASIC OPERATING LEVEL

The basic operating level of the Model 10-AU must be set for your application before calibrating the instrument for the first time. Thereafter, it is NOT necessary to adjust the basic operating level unless you change applications or cuvette sizes. *(See Appendix 6B of the user's manual.)*

The basic operating level is set on Screen 3.2 using the Sensitivity Adjustment Knob. (For Screen 3.2 definitions, see page 152.) Please note that the Model 10-AU is quite forgiving in terms of operating level and should give satisfactory results even at a low operating level. The goal when setting the basic operating level is a mid-range level of sensitivity for a typical sample concentration that will provide optimum results over a broad range of concentrations.

The procedure below will work well for most dye tracer studies using Rhodamine WT. It will provide satisfactory readings for extracted chlorophyll using 13 mm cuvettes. *(For in vivo chlorophyll or mapping, refer to Appendix 6B of the user's manual.)* If you plan to read very low or very high concentrations of dye or chlorophyll, or run into difficulty while following these instructions, refer to the user's manual, Appendix 6B.

NOTE: When using the discrete sample cuvette holder, take care not to spill liquids inside the fluorometer sample chamber. If you do have a spill, turn off the fluorometer immediately and consult the Sample System appendix of your user's manual.

1. Plug in the instrument. Turn the power ON. Allow it to warm up for 10 minutes.

2. Prepare one of the following standards (or obtain pre-made standards from Turner Designs):

   a. 100 ppb (20 ppb active ingredient) Rhodamine WT for studies using the 25 mm cuvette or continuous-flow cell;

   b. 150 - 200 ppb (µg/L) chlorophyll in 90% acetone for chlorophyll studies (extracted) using the 13 mm round cuvette.

3. Using the keypad of the Model 10-AU:
   a. Access screen 2.43 and set the concentration range control to MAN. (From screen 2.0, press <4>, then <3>. Press <ENT> to toggle to MAN.)

   b. On screen 2.42, for Rhodamine WT, set the instrument to the MED concentration range. For chlorophyll, set the range to HIGH. (From screen 2.0, press <4>, then <2>. Press <ENT> to toggle to MED or HIGH.)

   c. On screen 2.6, reset the calibration values to their default position. I.e., Blank will be zero; Cal std val will be 50.000; Standard soln conc will be 15.000; Span will be 48, etc. (From screen 2.0, press <6>, then <9> five times.)

   d. On screen 2.52, set the time constant to 2 seconds. (From screen 2.0, press <5>, then <2>, and <ENT> to toggle to 2 seconds.)

4. Unlock the Sensitivity Adjustment Lock with the Allen wrench provided.

5. Fill the clean flow cell or a cuvette with the standard prepared in step 2.

6. Access screen 3.2 by pressing <3> from the Main Menu, then <ENT>. Screen 3.2:

```
Fluorescence readout: XXXX.XXX
PM signal output: XXXX.XXX

Cal std val: XXX.XX   Blank: XX.XXX
FS: XX% of XXX.XXX (PPB) at MED/HIGH
Span level%: 48
<ENT> to next screen              #3.2
```

   Adjust the Sensitivity Adjustment Knob until the FS reads as recommended:

   For Rhodamine WT: 70-90% of 90.000 at MED.
   For chlorophyll: 30-50% of 900.000 at HIGH.

   It is **NOT** necessary to be exact; readings can be fine-tuned during calibration using the Span arrows, ↑ and ↓.

   Turning the Sensitivity Knob clockwise increases FS% (sensitivity); turning it counterclockwise decreases FS%.

   The Sensitivity Adjustment Knob is **very** responsive. Thus, turn it very slightly.Then pause until the reading reaches equilibrium. Repeat until desired FS% is obtained.

When you have reached the maximum or minimum, the FS% will not change, even though you turn the control.

7. When the desired reading is obtained, **LOCK** the Sensitivity Adjustment Lock by turning it clockwise with the Allen wrench. The basic operating level is now set, and you will use the Span and concentration ranges during calibration to set sensitivity for reading samples.

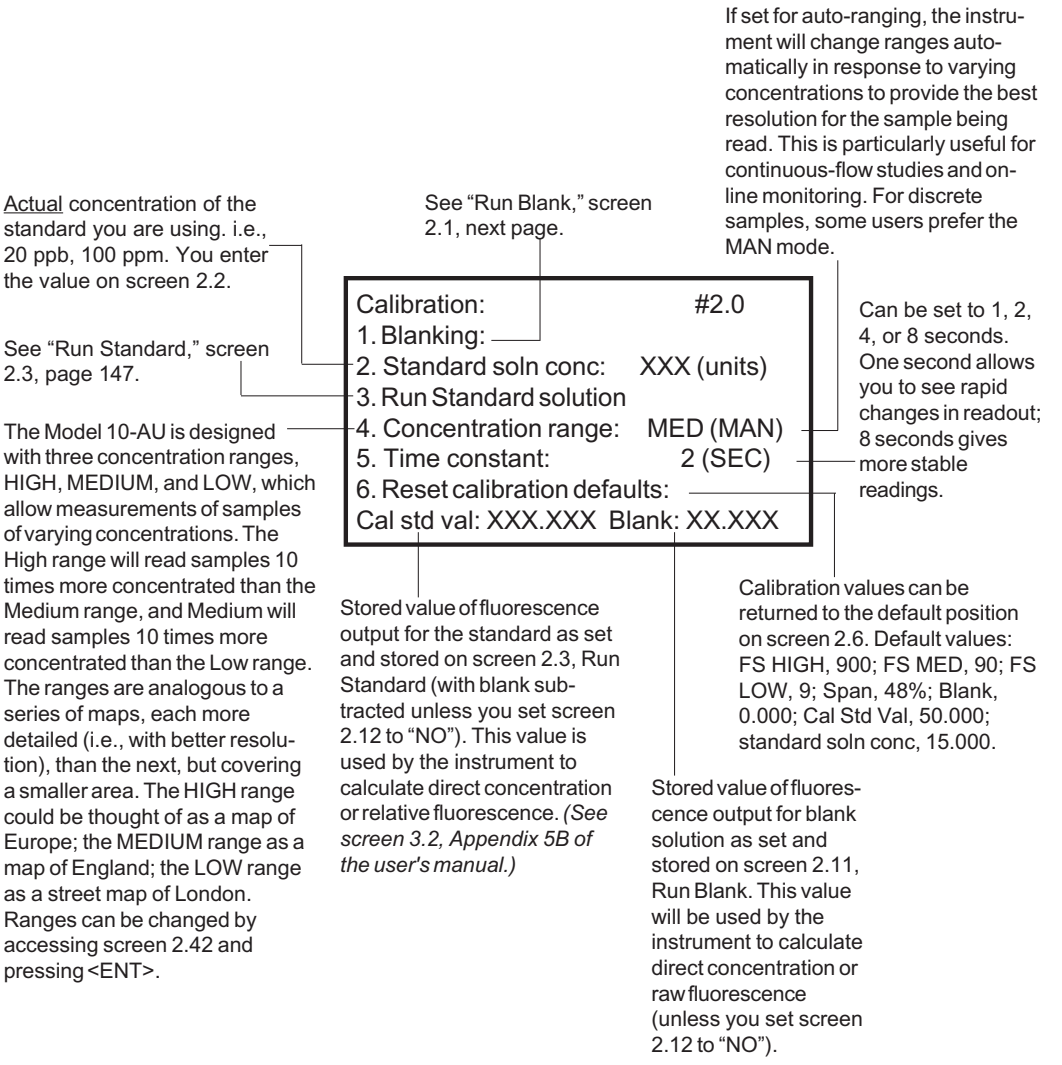
8. Press <ESC> to get out of screen 3.2.

9. Now, calibrate your Model 10-AU.

6. CALIBRATION

Calibration is begun from Screen 2.0. Menu items on Screen 2.0 are defined below, followed by separate pages defining Screen 2.11 "Run Blank" and Screen 2.3, "Run Standard". A step-by-step calibration procedure can be found on page 148. Access Screen 2.0, by pressing <2> from the Main Menu.

A. The Calibration Menu: Screen 2.0



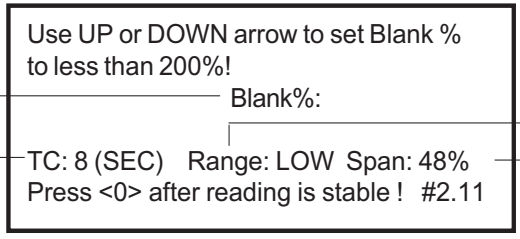
B. Run Blank: Screen 2.11

BLANKING. Blank is run on screen 2.11. Access screen 2.11 by pressing <1> from screen 2.0, then <1> again. Before measuring sample, a blank should always be collected. A blank is a sample of the water or solution you will work with, taken before any of the substance to be measured has been added. This fluid should be the matrix for your standard and will be used to set the instrument to read zero.

SUBTRACT BLANK. You can decide whether or not you would like blank to be subtracted by accessing screen 2.12. A "NO" setting means only that blank will not be subtracted from either the raw fluorescence or the concentration readout; it does not affect the current blank value as set during the last calibration.

The maximum blanking capability is 200% of full scale (300%), i.e., about 67% of the available signal can be used for blank, leaving 33% for samples (more than sufficient given the fluorometer's sensitivity). Once blanking is accomplished on one range, it is set for all ranges. The Model 10-AU automatically compensates for changes in concentration ranges. It is not necessary to adjust the Blank% to any particular number, as long as it is less than 200%. A low percentage number is preferred. (NOTE: The instrument will NOT prevent you from exceeding 200%.)

Tells you what range the instrument is currently operating in, i.e., HIGH, MED, or LOW. (Ideally, Blanking in LOW is recommended.)



TIME CONSTANT (TC). Note that during calibration, when Span is being adjusted and ↑ or ↓ is pressed, the TC will automatically cycle through the TC values, beginning with 1 second to give you the fastest response time, then moving to 2, 4, and finally 8 (most stable and accurate). During calibration, if ↑ or ↓ is not pressed, the TC remains at 8.

Equivalent to a fine adjustment of sensitivity. Span is adjusted in a continuous manner by pressing ↑ or ↓. Press <↑> to increase Span and <↓> to decrease it. When minimum sensitivity is reached, the words "<MIN SEN>" will appear just above the Span; "<MAX SEN>" will appear above the Span when maximum sensitivity is reached.

C. Run Standard: Screen 2.3

Normally, your standard will be a known concentration of the material that you plan to quantitate. The standard is run on screen 2.3, and consists of adjusting the Span using the <↑> and <↓> arrows until the readings on the Full Scale (FS) table are satisfactory for your study.

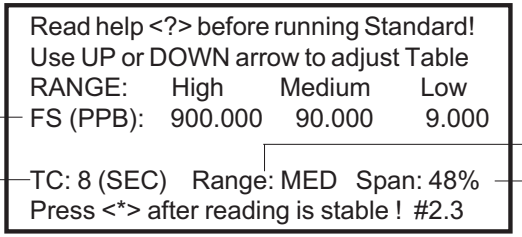
For single-point calibrations, and where a known concentration is needed, choose a standard with a concentration approximately 80% of the highest concentration you are reading, but still well within the linear range for your substance. *(See Appendix 6A, Linearity, of the user's manual.)* You can use a less concentrated standard, but a higher concentration will provide greater accuracy.

Full scale table (FS). The table on screen 2.3 indicates full scale (FS)--the maximum concentration or raw fluorescence data that can be read on each of the three ranges at the current Span level. Keep in mind that the numbers are full scale values, and it is not necessary or likely that the FS values will match the concentration of your standard.

BLK > FS means that blank is higher than full scale on that range. You will not be able to use that range for sample readings. This is acceptable if blank is high and you want to read high concentrations, if the FS on the higher ranges is acceptable.

OVER means that the signal from the standard exceeds the sensitivity level of the instrument for that range. Reduce Span or go to a higher range. (See next page.)

>9999 means the reading exceeds the maximum number of digits allowed. See the Routine Operation section of these instructions.



Tells you what range the instrument is currently operating in, i.e., HIGH, MED, or LOW.

TIME CONSTANT (TC). Note that during calibration, when Span is being adjusted and ↑ or ↓ is pressed, the TC will automatically cycle through the TC values, beginning with 1 second to give you the fastest response time, then moving to 2, 4, and finally 8 (most stable and accurate). During calibration, if ↑ or ↓ is not pressed, the TC remains at 8.

Equivalent to a fine adjustment of sensitivity. Span, is adjusted in a continuous manner by pressing ↑ or ↓. Press <↑> to increase Span and <↓> to decrease it. **NOTE:** FS **increases** as Span **decreases**, and vice versa. When minimum sensitivity is reached, the words "<MIN SEN>" will appear just above the Span; "<MAX SEN>" will appear above the Span when maximum sensitivity is reached.

D. Calibration Procedure

When calibrating, you are telling the instrument what zero is (Run Blank) and what a known concentration is (Run Standard); and you are adjusting the sensitivity using the keypad Span function so that you can read the concentrations of samples you desire. Once calibrated with a standard of known concentration, the Model 10-AU will give you direct concentration readout of samples. (NOTE: EPA Chlorophyll Method 445.0 requires before and after acidification calculations.)

Refer to your user's manual, Section 3 and Appendix 6B, if you are not using a known standard, or if you are attempting to read samples at very low concentrations.

NOTE: Changes in sample and ambient temperature, air bubbles in the sample, and linear range of the substance can significantly affect your results. *(See Appendix 6A of the user's manual.)*

BEFORE YOU BEGIN, if you have not already done so, set the Basic Operating Level, page 143. Have ready a blank and a standard of known concentration. For the best results over a broad range of concentrations, choose a standard concentration approximately 80% of the maximum concentration (within the linear range) you want to read.

Range for Rhodamine WT (25 mm round cuvette): low parts per trillion to 500 ppb (100 ppb of 20% active ingredient).

Range for chlorophyll *a* (extracted; 13 mm round cuvette): 0.05 ppb (µg/L) to 250 ppb (µg/L).

1. Plug in the instrument. Turn the power ON. Allow it to warm up for 10 minutes.

2. Using the keypad, set the instrument parameters as follows:

   a. Access screen 2.43 and set the concentration range control to AUTO.
   b. On screen 2.52, set the time constant to 2 seconds.

3. From screen 2.0, press <2> to access screen 2.2 (standard solution value). Enter the actual concentration for your standard. i.e., 100 ppb for the Rhodamine WT or 20 µg/L, etc., for chlorophyll.

4. **RUN BLANK**.

   a. From 2.0, press <1> to access screen 2.1, and make sure item 2 says "YES". If not, change to YES on screen 2.12. Press <HOME> to return to the HOME screen.
   b. **While on HOME screen**, insert your blank and replace the light cap if running discrete samples. **WAIT** about 10 seconds while the instrument determines the correct range.
   c. Then, access screen 2.11 from HOME by pressing <ENT>, <2>, <1>, and <1>.
   d. When the reading is stable, if Blank% is less than 200%, press <0>. If not, reduce the Span by pressing ↓ on the keypad until Blank% is less than 200%. Wait for the reading to stabilize before pressing <0>. **NOTE:** Pressing <ESC> on screen 2.11 before pressing <0>, will **abort the blank run** and retain the current settings for blank.

5. **RUN STANDARD**

   a. **Press <HOME>. While on the HOME screen**, insert your standard and replace the light cap if running discrete samples. **WAIT** about 10 seconds while the instrument finds the correct range.
   b. Then, access screen 2.3 from HOME by pressing <ENT>, <2>, and <3>. **NOTE**: Pressing <ESC> on screen 2.3 before pressing <*>, will **abort the standard run** and retain the current settings.
   c. When readings are stable, press <*>.

      NOTE: If reading a particularly low or high concentration standard, you can adjust the Span using ↑ and ↓, which increases or decreases sensitivity. Adjusting the Span can provide more or less sensitivity (range) for reading samples without having to reset the basic operating level. FS **increases** as Span **decreases**, and vice versa. Always **WAIT** about 10 seconds for readings to stabilize after pressing ↑ or ↓ before pressing <*>. Refer to the user's manual, Section 3, for details.

6. Press <HOME> to return to the HOME screen, and you are ready to begin reading samples.

7. ROUTINE OPERATION

Once the instrument is calibrated, insert a cuvette containing your sample into the Sample Compartment, or start sample flowing through the flow cell, and read the concentration or raw (relative) fluorescence data on the HOME screen. Refer to the HOME screen figure (right) for definitions of items displayed.

1. **AUTO-RANGING**. Decide whether you want to operate in the AUTO or MAN ranging mode. In the AUTO mode, the instrument will choose the range that gives the best resolution. Ordindarily, for continuous flow studies, set to AUTO. For discrete samples, the manual mode is preferred. To activate auto-ranging, access screen 2.43, and set to AUTO.

2. **MANUAL OPERATION**. If you choose to operate in the manual mode, you will have to change ranges yourself when concentrations are too high or too low for the range currently active. If you want the best possible resolution for your reading, especially on low concentrations, read your sample in the lowest range in which you can obtain an on-scale reading.

   OVER. In the manual mode, if the readout flashes “OVER,” it means the concentration reads higher than full scale for the current range. If, for example, the Model 10-AU is in MED and the reading is “OVER,” access screen 2.42 and change to the HIGH range. Return to the HOME screen to view the readout.

   If the reading is OVER on the High range, then the concentration exceeds the upper limits of detectability of the Model 10-AU as currently calibrated. You might try diluting the sample 1:1 until you obtain an on-scale reading to get some idea of the concentration. Or, you can reduce the sensitivity of the instrument by recalibrating and reducing the Span level. Or, consider changing the reference filter or changing to a smaller cuvette size. *(See Appendix 6A of the user's manual.)*

   DISPLAY FLASHES >999. If the reading exceeds 999, the maximum allowable for the HOME screen, the display will flash “>999”. Check the calibration table by pressing the left arrow. If the full scale reading for the current range is less than 9999.999, you can try reading your samples on screen 3.2. *(See Appendix 5B, screen 3.2, of the user's manual.)*

   Note: You can view and save sample readings to 7-significant digits by exporting the serial data output to a computer.

   Or, access screen 2.2 and reduce the standard solution concentration by a factor of 10. i.e., if it was 500, set it to 50, or 5, if necessary to make the full scale reading less than 999. If you do this, make note of the factor, as all of your samples will have to be multiplied by this factor to determine the actual concentration or relative fluorescence.

   MINUS(-). If the readout on the HOME screen has a minus sign in front of it, it means that the sample is less concentrated than blank. This is more likely where you have calibrated with high blank.

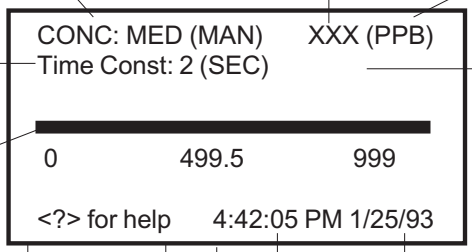
   In cases of high blank, where the calibration table full scale value for a range reads “BLK > FS,” which means than blank is higher than full scale on that range, you will not be able to use that range for sample readings.

3. **DISCRETE SAMPLE AVERAGING**. Decide whether to use the Discrete Sample Averaging capability. If so, access screen 1.63 and set the pre-delay period (default is 15 seconds) and the averaging period. This is a useful feature for ensuring consistent readings on discrete samples, especially with temperature-sensitive samples.

4. **DATA LOGGING**. Before reading samples, consider your data collection options. *(Details can be found in Appendix 11 of the user's manual.)*

5. **SENSITIVITY SETTING RETRIEVAL**. For long term studies, or where you would like to repeat a study with precision, it is possible to return to a previous instrument sensitivity setting. In order to retrieve a setting, you must make a note of three parameters (high voltage, range, Span%) **before** your study is complete. *(Seee Appendix 6C of the user's manual.)*

8. THE HOME SCREEN



Sample reading (with user-settable units), accurate after proper calibration. The screen can display a maximum of 3 digits, rounded off from the readings on screen 3.2 (which can support 7 digits (XXXX.XXX) for each range.) A reading greater than 999 will flash ">999". *(See Appendix 5B, screen 3.2, of the user's manual.)*

From screen 1.22, you may select a unit designation for your readout. You may also elect to display raw fluorescence data or direct concentration (accurate after proper calibration. If RAW is chosen, "(RAW)" will appear as the unit designation. *(Appendix 5A, screen 1.21, of the user's manual.)*

Concentration range (CONC) and whether you are operating in the AUTO or MANUAL mode. (Set range on screen 2.42; AUTO/MAN on screen 2.43.)

Time constant (see definition under screens 2.11 and 2.3)

At the option of the user, HOME may also display an analog bar graph (access screen 1.3 to change zero and full scale points, or set to auto-scale). *(See Appendix 5A, screen 1.3, of the user's manual.)*

If an alarm is activated when the instrument is operating, the words "ALARM ON!" will flash here, and the audio alarm will beep unless you have turned off the beeper. Press <ESC> to view any active alarms. *(See Section 4 of the user's manual.)*

Time

Date

Press <*>

Pressing <*> initiates the Discrete Sample Averaging sequence, which averages the readings over a user-settable period, and freezes the digital display for 10 seconds so you can note the reading. Thus, each sample can be read after the same amount of time has passed; and the averaging and freezing of the display minimizes both the inconvenience and potential error when readings fluctuate. When <*> is pressed, the words "Delay," then "Ave," and finally "DONE" will appear just above the units of measurement during the sequence. *(See Appendix 5A, screen 1.63, of the user's manual.)*

Press ← (left arrow)

Call up the Full Scale Value Table indicating the maximum concentration (or raw fluorescence signal) that can be read on each range under the current calibration.

Press <ENT>*

MAIN MENU
1. Operational parameters:
2. Calibration:
3. Diagnostic information:
4. Clock:

<?> for help

* If the security ID is enabled, you will be prompted for ID entry before you are allowed access to the MAIN MENU. *(See Appendix 5A, screen 1.64, of the user's manual.)* It is the policy of Turner Designs to provide ID access information by phone if a user requests it.

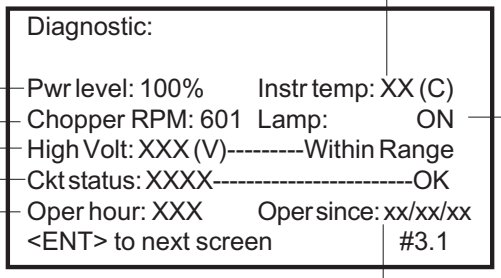
9. DIAGNOSTICS: SCREEN 3.1

Screen 3.1 contains information about the status of internal fluorometer functions and readings. If these values are normal, it is highly probable that the instrument is functioning correctly. *(See Table 2 in Section 4 of the user's manual for defaults and ranges for internal fluorometer functions.)*

Indicates the power level in percentage. Confirms **AC power** is at 115 V when reading is 100%. If your instrument is operating on **230VAC**, as long as the power level is greater than 10%, the instrument will function properly. For **DC power**, 10.2 V is 0% and 12 V is 100%. We have found that a 12-volt battery indicates approximately 70%; this is more than adequate. The **low power level alarm** will be triggered if the power level falls below 10%.

INSTRUMENT TEMPERATURE indicates the internal temperature of the fluorometer. If temperature rises above 90°C or falls below -20°C (based on internal sensor, accuracy: +/-5°C), it will trigger an alarm.

Indicates the speed in rpm of the chopper motor (which spins an optical device). It is regulated to 600 rpm, +/- 1% by internal software and hardware. If it falls below 500, an alarm is triggered.



LAMP reports whether the lamp is OFF/BAD or ON.

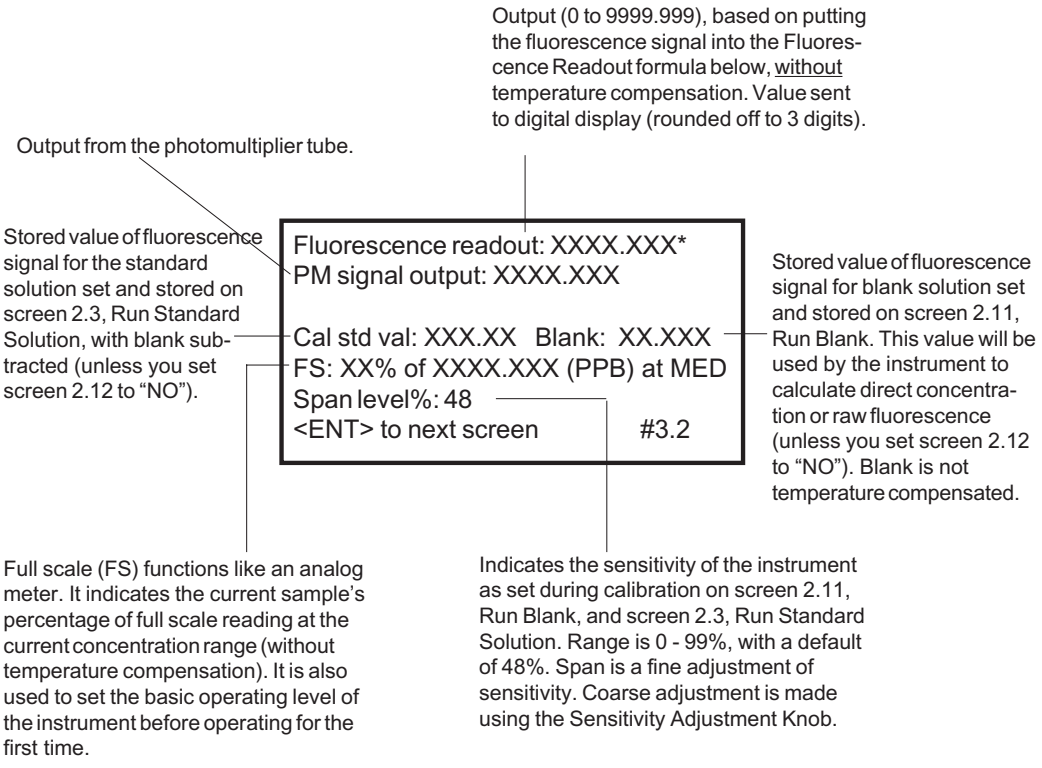
OPERATION HOUR shows how many hours the instrument has been operated.

OPERATION SINCE shows the earliest date at which the instrument has been operated continuously for one hour.

CIRCUIT STATUS reports a numerical value and whether it is OK or failure.The value depends on the range the fluorometer is in. In HIGH, is should be about 36; in MED about 360; in LOW about 3600.

HIGH VOLTAGE reports a numerical value and whether that value is Within Range, or Too High or Too Low. High voltage is one indication of the current operating level of the fluorometer. The number will vary: the higher the number the lower the concentration range (and typically, the greater the "noise" of the readings). High voltage will be lowest when the instrument is in the HIGH concentration range, intermediate when in the MED range, and highest in the LOW concentration range. High voltage can be increased or decreased using the Sensitivity Adjustment Knob. Once you have set the basic operating level and LOCKED the Sensitivity Knob Lock, high voltage should remain relatively stable for each of the ranges. It is a good indicator of the stability of the instrument; thus, you might want to record the high voltage level whenever you read a series of samples.

10. DIAGNOSTICS: SCREEN 3.2



Fluorescence readout formula:

$$\frac{\text{Standard Soln Conc.} \times \text{Sample Output}}{\text{Cal std val}}$$

Fluorescence signal at a given range for current sample (range depends on the sensitivity), with blank subtracted (unless you set screen 2.12 to "NO").

Actual concentration of standard solution as entered on screen 2.2 during calibration.

Stored value of fluorescence signal for the standard solution (set and stored on screen 2.3, Run Standard Solution) with blank subtracted (unless you set screen 2.12 to "NO").

* *For details of screen 3.2, including information on "Temp. comp. readout," see Appendix 5B, screen 3.2.*

Index

A

B

C

D

E

F

G

H

I

L

M

N

O

P

Q

R

S

T

U

V

W

QFT2™ Technical Papers

Surface Techniques to Measure Oil Concentration While Drilling

DeLaune, P.L., Texaco Exploration and Production Technology Department

SPWLA 33rd Annual Logging Symposium

Oklahoma City, Oklahoma USA

June 14 – 17, 1992

Abstract

Fluorescence has been used as a means of detecting oil while drilling since first introduced in the 1930s. Still in use today, this visual technique has long been recognized to be highly subjective and inconsistent, making it unreliable as a means for detecting oil. The major limitation is the fact that many crude oils fluoresce outside the visible region and therefore go undetected using visual techniques.

In the mid 1980s, Texaco began research and development to improve the value of surface data available during drilling operations. One of the first results of this effort was a technique for more accurately determining the amount of oil in drill cuttings and cores using a quantitative fluorescence measurement technique. This technique uses a portable fluorometer to measure the fluorescence of the crude oil that has been extracted from the sample. The intensity of the fluorescence is proportional to the amount of oil. These results are plotted as a function of depth to yield an Oil Concentration Profile of the well. By calibrating with a sample of the crude, a more quantitative calculation of the amount of oil in the pore spaces can be made, resulting in a calibrated OIL LOG.

This technique has been proven on more than 100 wells around the world. It can be used at the wellsite while drilling or in the laboratory after the well has been completed. It can effectively be used on fresh drill cuttings or cores, or samples from wells previously drilled. It has been used successfully on wells 20+ years old.

This paper discusses the technology behind this new technique. It also addresses the equipment, process, and procedure for performing this analysis at the wellsite and in the laboratory. Case histories are presented, including results and problems with using the technique on wells while drilling, after drilling, and on older wells for re-evaluation/correlation applications.

To obtain a copy of this technical paper, connect with the Society of Professional Well Analysts.

QFT2™ Technical Papers

SPE 56802
Enhanced Wellsite Technique for Oil Detection and Characterization

DeLaune, P.L., Spilker, K.K., Texaco Upstream Technology and Wright, A.C., Consultant

SPE Annual Technical Conference and Exhibition

Houston, Texas USA

October 3 – 6, 1999

Abstract

Fluorescence has been used as a means of detecting oil while drilling since first introduced in the 1930s. The visual technique is recognized to be subjective, making it unreliable for consistent detection of oil in formation samples. The Quantitative Fluorescence Technique (QFT™) introduced in the early 1990s uses a portable fluorometer to measure the fluorescence of crude oil extracted from a formation sample. The intensity of the fluorescence is proportional to the amount of oil in the sample. QFT™ was a substantial improvement over the visual technique for detection of oil in drill cuttings or cores, but still provided only a relative measure of oil in the sample.

An enhanced technique called QFT2™ has been developed that provides improved estimates of oil quantity and character from measurements made on drill cuttings or core samples. QFT2™ involves a two-point fluorescence measurement that yields estimates of both quantity (Weight % Oil) and oil type (API gravity). While these estimates are not a substitute for laboratory analysis, the speed, accuracy and ease of application make QFT2™ a practical tool for the determination of oil concentration and API gravity at the wellsite. QFT2™ can be used effectively on fresh drill cuttings or cores, or older samples from wells previously drilled. Results can be combined with wire-line hydrocarbon porosity data to estimate volume percent oil in the formation, and resultant data can be further evaluated to estimate oil mobility.

QFT2™ was developed by Texaco and finalized in a joint development project with ENI-Agip Division. The project evaluated a world-wide suite of oils to determine relationships between surface measurements and in-situ conditions.