MK-PB-0008-AC Rev B

DCN 175

May, 2010

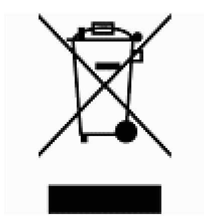
ReactIR™ Sampling Technology Guide

Gain a Clear View of Your Chemistry

METTLER TOLEDO

4 May 2010

Address comments to:
Mettler-Toledo AutoChem, Inc.
7075 Samuel Morse Drive
Columbia, MD 21046
Tel: + 1 866.333.6822
Fax: +1 410.910.8600
www.mt.com/ReactIR
For technical support, contact: AutoChemCustomerCare@mt.com



In conformance with the European Directive 2002/96/EC on Waste from Electrical and Electronic Equipment (WEEE), this device may not be disposed of in domestic waste. This also applies to countries outside the EU, per their specific requirements.

Please dispose of this product in accordance with local regulations at the collecting point specified for electrical and electronic equipment.

If you have any questions please contact the responsible authority or the distributor from which you purchased this device.

Should this device be passed onto other parties for private or professional use, the content of this regulation must also be related.

Thank you for your contribution to environmental protection.

Contents

Chapter 8 Alignment

Chapter 9 Care and Use

Chapter 10 Putting It All Together

1

Introduction

This guide describes the sampling technologies available for the METTLER TOLEDO ReactIR™ instruments that can include the iC10, 15, 45m, or MonARC system base unit (see Figure 1-6). Sampling technology is a key component of the ReactIR systems that enables robust interfacing to your chemistry for monitoring virtually any reaction. Once connected to the ReactIR system, the sampling technology directs the infrared source beam to the sensor for ATR (definition on page 14) sampling and then back to the detector. As a result, the process produces an infrared fingerprint of the reaction mixture in real time.

Probe Sampling Technology

Sampling technology where a probe monitors chemistry in a reactor consists of four parts—the sampling interface module (SIM), conduit, probe, and sensor—with the option to select distinct components. In the case of the DS Micro Flow Cell sampling technology, the chemistry flows to the sensor in the flow cell, so there is no probe or conduit. The sampling technology configuration that you choose depends on the chemistry that will be monitored *in situ* (definition on page 15).

Micro Flow Cell Sampling Technology

Sampling technology for flow chemistry in a laboratory includes the SIM and the sensor within a flow cell (see "DS Micro Flow Cell Sampling Technology" on page 12). Flow chemistry comes to the sensor through tubes that flow in and out of a flow cell.

Sampling Technology Examples

The upcoming images identify the sampling technology components in the following configurations:

- "Four Parts of Sampling Technology (Mirrored Conduit)" on page 8
- "DS Series Technology Sampling Technology" on page 9
- "DS Sampling Technology with Gas Cell" on page 11
- "DS Micro Flow Cell Sampling Technology" on page 12

Four Parts of Sampling Technology (Mirrored Conduit)

The image below features a K6 mirror conduit and a 16mm probe on a ReactIR iC10 base unit.

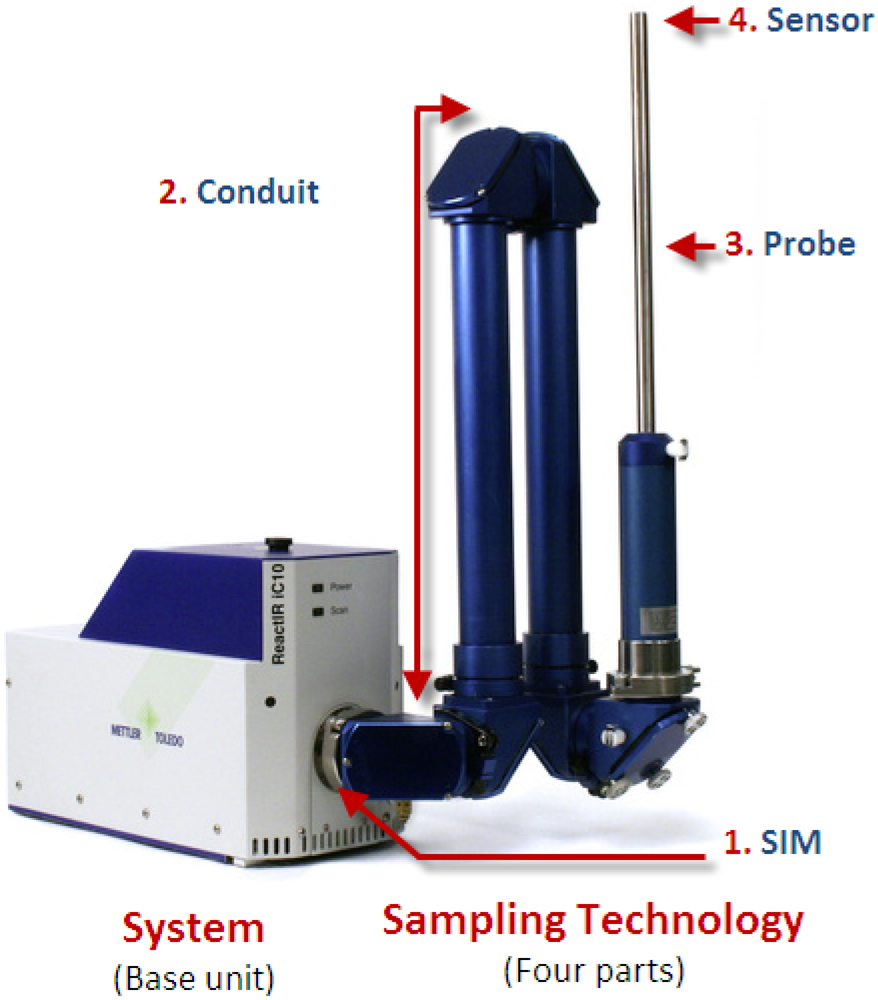


Figure 1-1 Sampling technology—(mirror conduit and probe on a ReactIR iC10)

In terms of the conduit and probe, a sampling technology can be integrated or non-integrated. Figure 1-1 shows an example of a non-integrated sampling technology where the conduit can separate from the probe. Figure 1-2 features the METTLER TOLEDO DS Series sampling technology with FiberConduit technology where the conduit and probe are integrated. All probes have integral sensors at time of purchase.

DS Series Technology Sampling Technology

The DS Series sampling technology offers single and multiple probe options when used with a ReactIR 45m base unit. Figure 1-2 shows both an integrated configuration (AgX Fiber) and a non-integrated (detachable Fiber-to-Sentinel) probe.

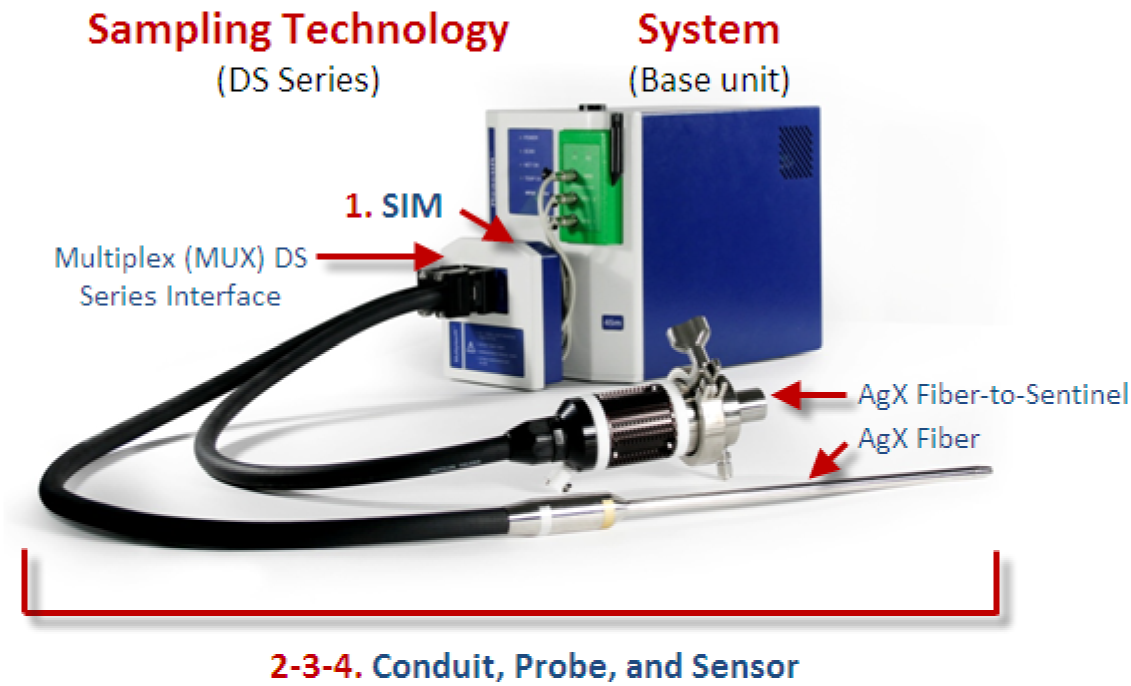


Figure 1-2 DS Series sampling technology—(shown on a ReactIR 45m)

The single DS Optical Interface Module, shown under "DS Series Optical Interface Modules" on page 22, applies to all the ReactIR base units.

Figure 1-3 is an example of the DS Series Technology on a ReactIR 15 base unit. ReactIR 15 base units use DS Series sampling technology, exclusively.

The following image shows the non-integrated (detachable Fiber-to Sentinel) configuration. Notice that the ReactIR 15 base unit has a DS SIM. No DS Series SIM adapter is required.

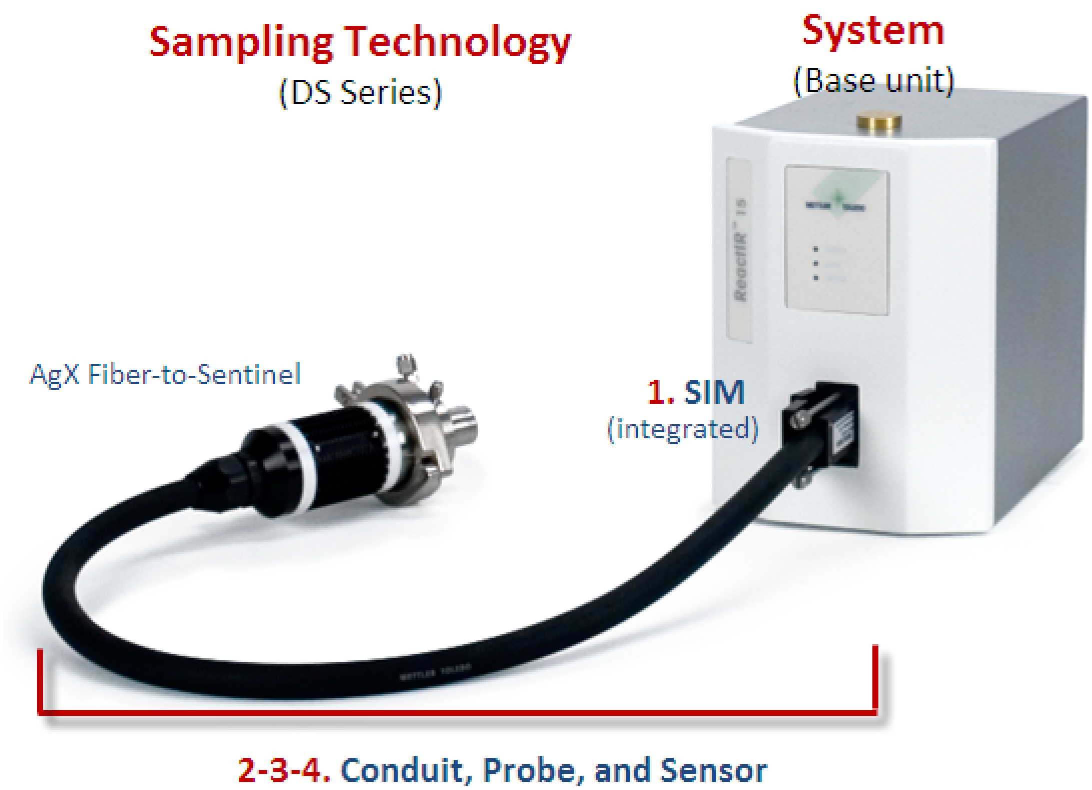


Figure 1-3 DS Series sampling technology—(shown on a ReactIR 15)

DS Sampling Technology with Gas Cell

In a sampling technology configuration with a gas cell, there are three components—SIM, FiberConduit, and Gas Cell (highlighted in yellow in Figure 1-4). A gas cell has no probe or sensor. The infrared beam passes through the reactive gas sample in a chamber in the gas cell and produces a single beam spectrum of the gas concentration.

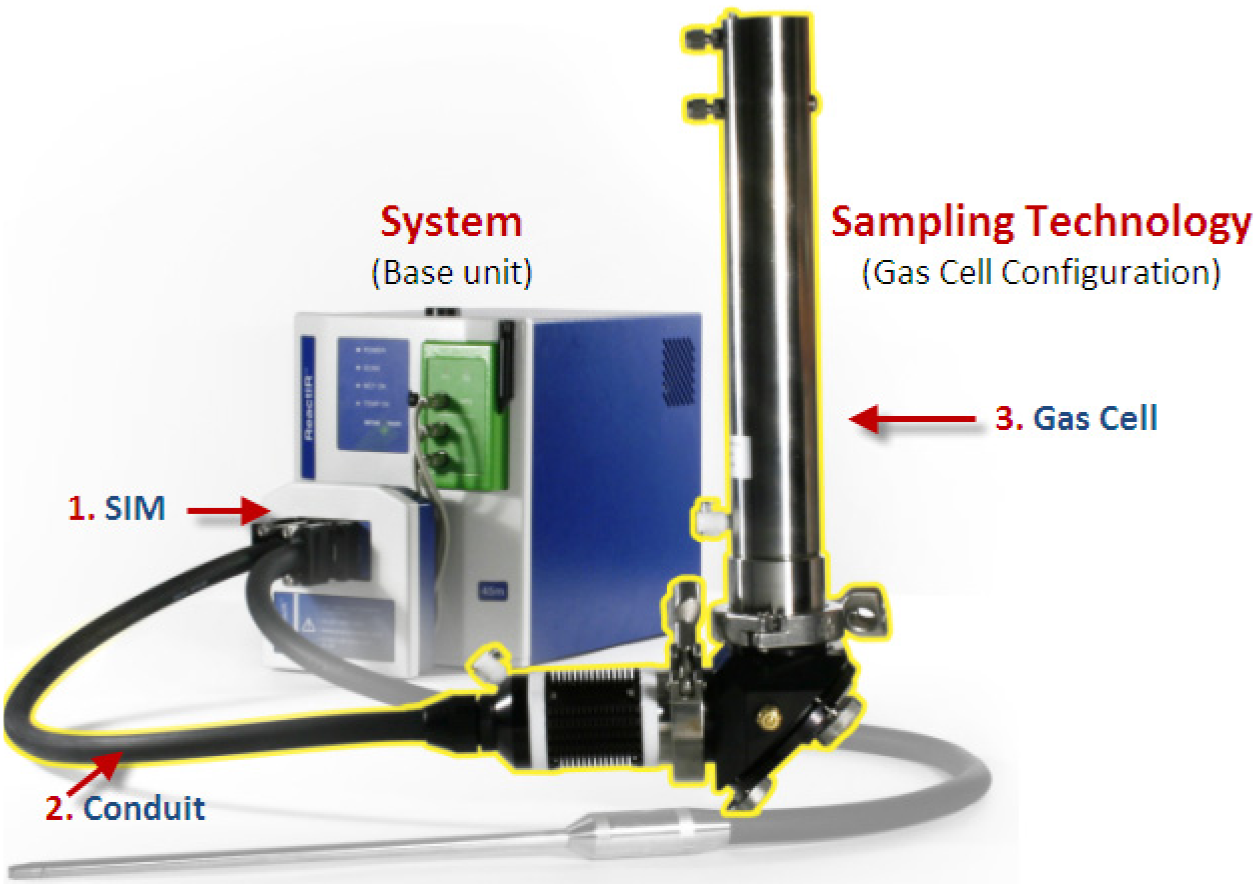


Figure 1-4 Sampling Technology with gas cell—(shown on ReactIR 45m with MultiplexIR)

The figure above features the MultiplexIR (MUX) configuration with multiple sampling technologies—the gas cell is highlighted. The second sampling technology, a DS Series FiberConduit, is grayed out.

DS Micro Flow Cell Sampling Technology

ReactIR sampling technologies include a flow cell option for monitoring flow chemistry in laboratory environments. With flow cells, there is no probe or conduit because the chemistry flows into the sensor and returns to the reactor as a seamless process.

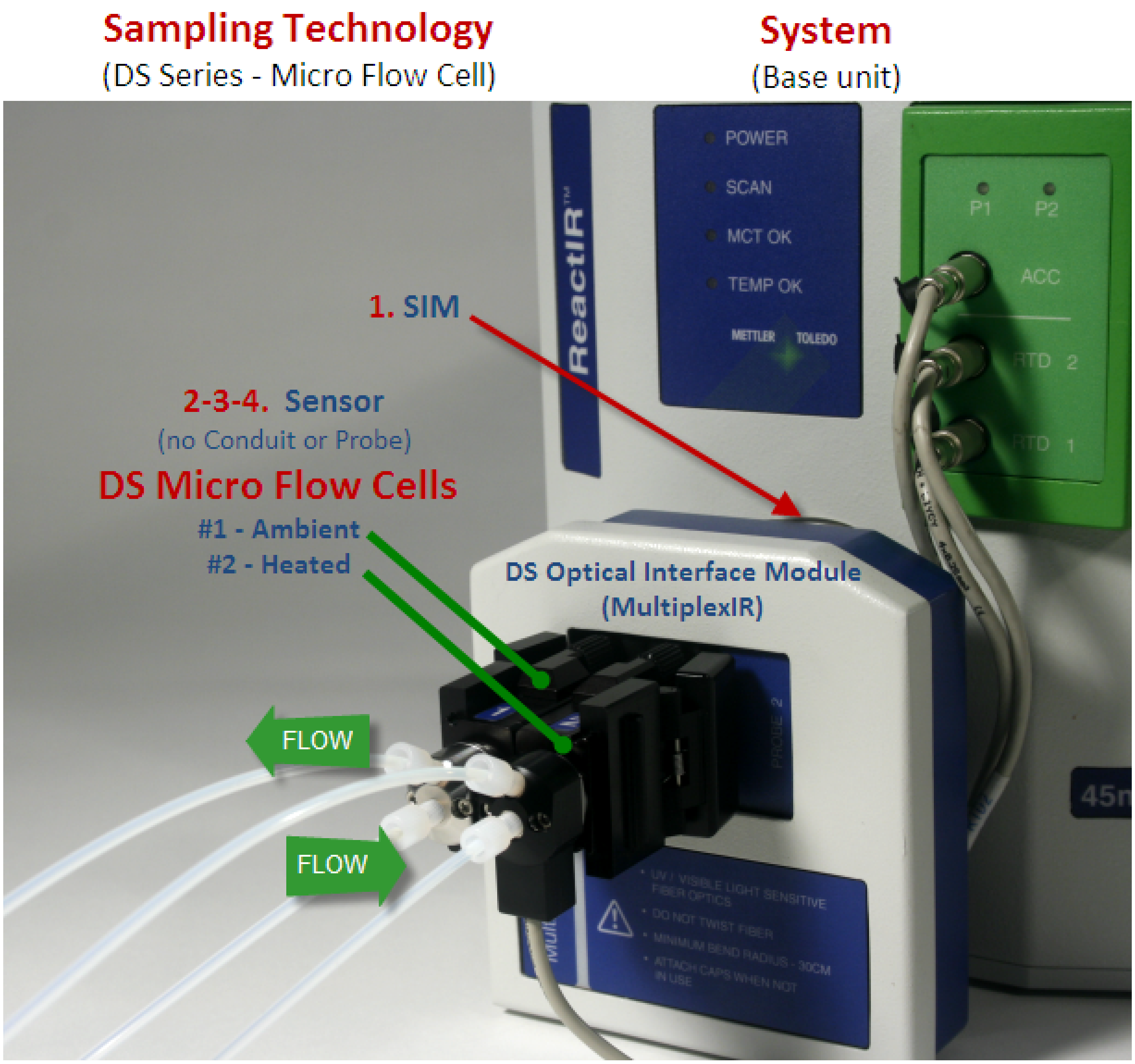


Figure 1-5 DS Micro Flow Cell sampling technology—(shown on a ReactIR 45m)

Figure 1-5 features two DS Micro Flow cells in a MultiplexIR (MUX) optical interface module. A single interface module configuration is also available. Flow cell sampling technology applies to ReactIR 45m and iC10, as well as to legacy ReactIR 4000 base units.

Definitions

Below are the key terms used to describe the sampling technologies available for infrared spectroscopy.

Table 1-1 Terminology

Term	Definition
Sampling technologies	Sampling technologies refer to the four-part configuration that inserts in your vessel and transfers reaction information to a ReactIR instrument base unit (see Figure 1-1 on page 8). The four parts include: (1) **Sample Interface Module** (SIM)—factory standard (2) **Conduit** (**Mirror** or **Fiber**) (3) **Probe** ** (4) **Sensor** ** ** In a gas cell configuration, the gas cell replaces the probe and sensor. Note: The DS Micro Flow cell sampling technology has no conduit or probe. Chemistry flows to the sensor in the flow cell. The sampling technology senses the infrared active molecules during chemical reactions that become an infrared fingerprint. Sampling technology options include the type of conduit, the length and diameter of the probe, and the composite material of the probe sensor. A standard Sampling Interface Module (SIM) is factory-supplied. Sampling technologies are sold separately.
Base unit	Base unit refers to the ReactIR instrument "box" containing the Fourier transform mid-infrared source and detector that interfaces to the sampling technology. A sampling technology configuration connects to the base unit. ReactIR base units described in this guide include: iC10, 15, 45m, and the MonARC.
Sample Interface Module (SIM)	A SIM is the interface on the instrument base unit where the conduit connects. The standard SIM is factory installed. The DS series sampling technology requires a special DS series Optical Interface Module that connects to the factory-standard SIM.

Table 1-1 Terminology (continued)

Term	Definition
Conduit	A conduit is a sampling technology option, sometimes referred to as the "transfer optics" or "transfer technology." A conduit contains the optics that transfer the infrared (IR) source light from the ReactIR to the probe in contact with chemistry, and then back to the detector. The conduit transfers the light by either mirrors or fiber optics, depending on customer requirements. **Mirror Conduits** Mirror conduits are identified as K4, K6, or K7, based on the number of mirrors in the path. **FiberConduit™** A FiberConduit uses AgX (silver halide) fibers to transfer the IR light.
Probe	A probe is the direct interface with your chemistry and includes an ATR sensor. The probe comes in different lengths and diameters.
Sensor	A sensor is a high refractive index material suitable for use in mid-infrared sampling by ATR. A variety of sensor and seal materials are available to match the chemical reaction requirements. **Sensor Composite** (Comp™) Diamond (DiComp™)—Extremely robust Silicon (SiComp™)—Sensitive to abrasion Cubic Zirconium (ZrComp™)—Robust **Seals** Gold Teflon
AgX	Silver Halide is the optical material used in FiberConduits to transfer infrared source light to the sensor and then back to the ReactIR detector.
ATR	Attenuated Total Reflectance (ATR) is an infrared sampling technique capable of measuring homogeneous and heterogeneous reaction mixtures with high reproducibility. All probes in this guide use this technique, with the exception of the gas cell configuration.
Sentinel™	A Sentinel is a type of composite probe with a standard size of 1-inch diameter and 1-inch insertion depth.
Flow Cell	A DS Micro Flow Cell is a type of sampling technology that enables inline monitoring of continuous flow chemistry as it moves past the sensor through input and output tubing.

Table 1-1 Terminology (continued)

Term	Definition
Gas Cell	A gas cell is a type of sampling technology that has no probe or sensor. Instead, it has a chamber with input and output ports for the analysis of reactive gases.
Single Beam	A single beam is the energy profile of the ReactIR system with or without a sample in contact with the sensor. Single beam can refer to a background or a sample. **Background**—Single beam without a sample **Sample**—Single beam with a chemical sample Single beam units are intensity versus wavenumber (cm^{-1}). See Appendix B, "Single Beam Samples" to see the background spectrum for individual sampling technologies.
Spectrum	A spectrum is the measure of mid-infrared absorbance versus wavenumber that results from ratioing a sample single beam to the background single beam. ReactIR automatically performs the ratio calculation during reaction monitoring. Spectrum units are absorbance versus wavenumber (cm^{-1}).
Contrast	Contrast is a test used to check the general functioning of a ReactIR instrument and sampling technology. A contrast test displays the single beam signal returned from the instrument.
in situ	The term *in situ* refers to analyzing chemistry in the reaction mixture, in real time, under actual reaction conditions.

ReactIR Base Units

Figure 1-6 shows the ReactIR DS Micro Flow Cell, iC10, 45m, 15, and MonARC base units capable of monitoring reactive chemistry during the discovery (research), development, and manufacturing phases of the chemical life cycle. The figure below shows a FiberConduit sampling technology on the iC10 and multiprobe FiberConduits on the 45m.

Figure 1-6 Base unit images

A Fourier transform infrared (FTIR) spectrometer in the base unit optically measures and monitors changes in chemical species as they react over a period of time.

Base Unit Overview

ReactIR iC 10—This base unit is similar to the ReactIR 15, but it supports mirror conduit as well as DS Series FiberConduit technologies.

ReactIR 15—This base unit has a built-in, single DS Optical Interface module SIM, so it can only be used with DS Series sampling technologies (mirrored conduits are not an option). It is factory-aligned and requires no rotational alignment. The base unit is sealed so there's no requirement for instrument purge. The ReactIR 15 include an integrated temperature monitor.

ReactIR 45m—This base unit is a full featured reaction analysis system for the most demanding applications. Designed to be flexible to use the full range of Comp probe and conduit technologies, the ReactIR 45m is the designed for the chemist or chemical engineer faced with the challenge of monitoring reactions across a wide range of temperature and pressure conditions and is ideal for kinetics and quantitative analysis. The 45m base unit supports MultiplexIR analysis that uses two sampling technologies.

MonARC—This base unit provides critical information about the identity and quantity of chemical reaction species present on an industrial scale inherent to kilo labs, pilot plants and production.

Base Unit Components

For details on a MonARC system, refer to the *MonARC Hardware Manual*. For details on a ReactIR iC10, 15, and 45m system, refer to the *ReactIR Specifications, Care, Use, and Safety Guide*. The ReactIR iC 10, 15, and 45m systems consist of the following:

- ReactIR iC10, 15, or 45m base unit (infrared spectrometer)
- 1.0-meter USB communications cable
- 24-hour cooling capacity, Mercury Cadmium Telluride (MCT) Detector

iC IR™ software works with the instrument and provides features to collect spectroscopic data and visualize, interpret, and report results.

Optional items:

- Stabilization Foot (for use with mirrored conduit to provide instrument stability)
- PC Workstation (with installed iC IR Software)

Sampling Technology Options

- 6.35mm diameter AgX (Silver Halide) FiberConduit
- 9.5mm diameter AgX (Silver Halide) FiberConduit
- DS Series Optical Interface Module
- DS Series MultiplexIR Module
- DS Micro Flow Cell
- 16mm diameter Probes

- Mirror-based optical conduit (4- or 6-mirror version)
- AgX Fiber-to-Sentinel
- 1-inch diameter Sentinel

How this Guide is Organized

Appendix A, "Sampling Technology Component Tables", starting on page 101, shows all the standard options for sampling technology configurations and the following chapters describe each option in detail, starting with the SIM:

- Chapter 2, "Sample Interface Modules (SIMs)" (page 21)
- Chapter 3, "Conduits" (page 25)
- Chapter 4, "Probes" (page 35)
- Chapter 5, "Sensors and Seals" (page 41)

The next chapter describes a special type of sampling technology for continuous flow chemistry where the probe is within the flow cell body.

- Chapter 6, "Connecting and Using DS Micro Flow Cells" (page 43)

The following chapters provide information on how to choose, connect, and align the sampling technologies, along with specifications on components:

- Chapter 7, "Connecting Conduit Sampling Technologies" (page 65)
- Chapter 8, "Alignment" (page 77)
- Chapter 9, "Care and Use" (page 95)
- Chapter 10, "Putting It All Together" (page 99)
- Appendix A, "Sampling Technology Component Tables" (page 101)
- Appendix B, "Single Beam Samples" (page 109)

Should you have questions that are not addressed in this document, please contact your local METTLER TOLEDO office or our Customer Care Department using the information under "Service and Technical Assistance" on page 19. Also, if you are viewing this document electronically, click any blue-colored link to go to the related information and instructions.

Repair Policy

METTLER TOLEDO warrants its products against defects in materials and workmanship for twelve months from the date of installation or fifteen months from the date of shipment. For details, please refer to the warranty provided with the instrument.

For assistance, please contact your Technical Application Consultant or send an email to **AutoChemCustomerCare@mt.com**.

It is recommended that you retain the original packing materials in the event you need to return the sampling technology. If factory service is required, contact Customer Care to request a Return Material Authorization (RMA) form.

Training Programs

Training for all ReactIR sampling technologies and software is available from the Columbia location of Mettler-Toledo AutoChem, Inc. Contact **AutoChemCustomerCare@mt.com**.

Service and Technical Assistance

METTLER TOLEDO has offices around the world. Contact the Mettler-Toledo AutoChem, Inc. headquarters in the USA for technical support or service. For additional information on ReactIR product lines and accessories, visit www.mt.com/reactir.

For specific application assistance at any time, contact a METTLER TOLEDO Technology and Applications Consultant (TAC) through the toll-free number below.

Table 1-2

Mettler-Toledo AutoChem, Inc. (Columbia, MD headquarters)	Tel: **+ 1.410.910.8500** Fax: **+1.410.910.8600** Email: **AutoChemCustomerCare@mt.com** Toll Free: **+1.866.333.6822**

1. SIM
2. Conduit
3. Probe
4. Sensor

2

Sample Interface Modules (SIMs)

This chapter describes the first component of a sampling technology—the Sample Interface Module (SIM). A SIM is the interface between the ReactIR base unit (instrument) and the rest of the sampling technology components.

Two types of SIMs exist for ReactIR base units:

- **Factory-standard SIM**—Uses a clamp to connect and disconnect a sampling technology from a base unit.
- **Factory-standard DS SIM**—Provides an integrated, single DS interface for connecting DS-Series sampling technologies.
- **DS Series Optical Interface Module**—DS-Series sampling technologies use a special adapter that connects to the factory-standard SIM

Factory-Standard SIM

The factory-standard SIM comes installed on the base unit of a ReactIR to begin connection of the sampling technology of your choice.

Figure 2-1 Factory-standard SIM (45m)

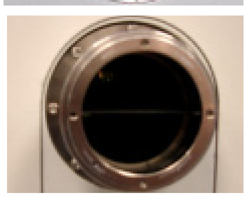


Figure 2-2 Factory-standard SIM (iC10)

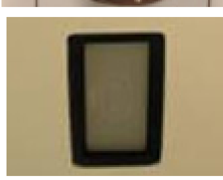


Figure 2-3 Factory-standard DS SIM (15)

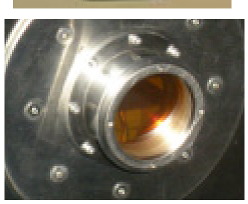


Figure 2-4 Factory-standard SIM (MonARC)

DS Series Optical Interface Modules

- The SIM adapter for DS Series sampling technologies is called an Optical Interface Module. It comes in two models—a single and a multiprobe..

Note: The Single Optical Interface Module is integrated in a ReactIR 15 base unit.

Single Optical Interface Module

The Single DS Series Optical Interface Module is available with or without an RTD (Resistive Temperature Detector) connection to monitor temperature of the reaction mixture in contact with the probe.

With RTD connection   Without RTD connection

Figure 2-5 Single DS Series Optical Interface Module (with and without RTD)

MultiplexIR (MUX) Optical Interface Module

The MultiplexIR DS Series Optical Interface Module (also called the "Mux") enables dual probe usage to monitor two independent reactions at the same time. This type of sampling technology is only available with the ReactIR 45m base unit with DS Series integrated FiberConduit and probe. All MUX Optical Interface Modules have two temperature monitoring connections, one for each probe.

Front View   Rear View

Figure 2-6 Multiplex SIM Adapter (front and rear)

Note: The third wire powers the motor that moves to Probe 1 or Probe 2 position.

SIMs and ReactIR Base Units

The following table shows the two types of SIMs, along with the features that are available with the DS series. Features include the ability to monitor temperature with one or two RTDs and the ability to use multiple probes.

Table 2-3 Sample Interface Module (SIM) and Base Unit Feature Availability

SIM Type	Sampling Points	RTD	ReactIR Base Unit
Factory-standard	**Single**		45m
			iC10
			MonARC
Factory-standard DS	**Single**	**standard**	15
DS Series	**Single**	optional	45m
	Multiple	**standard**	

See Chapter 7, "Connecting Conduit Sampling Technologies."

Factory SIM with DS Series Optical Interface Module

The following image shows a DS Series Optical Interface Module connected to the factory-standard SIM (for installation steps, see "DS Series Optical Interface Connection" on page 67):

Figure 2-7 DS Series Single Optical Interface Module installed on SIM

For your convenience, below is the part number information for ordering purposes.

- DS Series Optical Interface Module—for single probe (MTPN: 14440001)
- DS Series Multiplex Optical Interface Module—for multiple probes (MTPN: 14440000)
- DS Series Optical Interface Module—for single probe without RTD connection (MTPN: 14440002)

Mechanical Dividers

FiberConduit probes require a mechanical divider in the factory-installed SIM for optimum performance of ReactIR iC10, 45m, and MonARC systems. (Figure 7-11 on page 74 shows a mechanical divider in the SIM of a 45m base unit.)

If you do not have the mechanical divider in your base unit, please contact your local Account Manager, TAC, or our Customer Operations department (see contact information under "Service and Technical Assistance" on page 19) to order the necessary component.

For your convenience, below is the part number information for ordering purposes.

FiberConduit Mechanical Dividers:

- ReactIR iC10 SIM Divider (MTPN: 14170071)
- MonARC SIM Divider (MTPN: 14400070)
- ReactIR 4000 SIM Divider (MTPN: 14170072)

3

Conduits

This chapter includes the following sections for the two types of conduits:

"FiberConduit™" on page 25

"Mirror Conduits" on page 29

FiberConduit™

A FiberConduit consists of a flexible IR transmission fiber composed of silver chloride/silver bromide. The fiber is referred to as silver halide (AgX). The flexible design of the FiberConduit facilitates ease of use and easy integration with various reactors. The conduit sheath is constructed of a steel mesh in a PVC cover. FiberConduits require the use of a liquid nitrogen cooled MCT or Stirling Engine MCT detector for infrared measurements.

The FiberConduit is available in detachable or integrated probe styles that connect directly to the factory-standard SIM or to a DS Series Interface Module.

Table 3-4 FiberConduit Connections

Connection Type (to SIM)	Probe Type	Image
Bell-Style	Detachable—Bell-to-Bell (Sentinel)	
	Integrated—Bell-to-Integrated probe	

Table 3-4 FiberConduit Connections (continued)

Connection Type (to SIM)	Probe Type	Image
DS Series	Detachable—Fiber-to-Bell (Sentinel)	
	Integrated	

Detachable Probe—To be connected to a Sentinel probe prior to sampling a reaction mixture

Integrated Probe—Factory-ready to insert into a reaction mixture

The following image shows the two styles of FiberConduit with a DS Series Multiplex adapter on a ReactIR 45m:

Figure 3-8 FiberConduit—Detachable and integrated styles (DS Series Multiplex)

The image below shows an integrated style FiberConduit. The integrated probe and conduit attaches to the base unit with a bell-shaped connector that attaches to the factory-standard SIM.

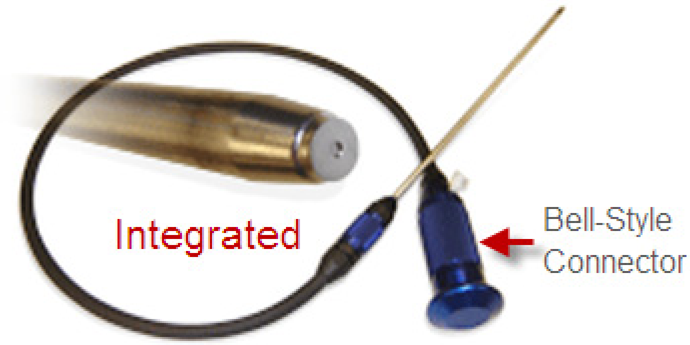


Figure 3-9 FiberConduit—Integrated style (shown with Bell-shaped connector)

The image below shows a detachable style FiberConduit-to-Sentinel. The Sentinel probe attaches and detaches from the FiberConduit via a bell-shaped adapter. The Fiber-to-Sentinel sampling technology attaches to the base unit via a DS Series Single adapter that connects to the factory-standard SIM

Figure 3-10 FiberConduit—Detachable style (DS Series FiberConduit)

Specifications

Table 3-5 FiberConduit Specifications by Type of Connection

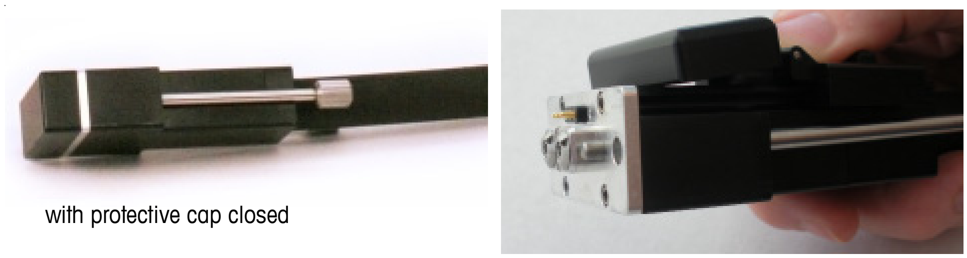
Specification	Type of Connection	
	Bell-Style	**DS Series**
Material of Construction	**Fiber**—Silver Halide, a flexible IR transmission fiber composed of silver chloride/silver bromide **Protective sheath**—Thermoplastic Rubber (TPR)	**Fiber**—Silver Halide, a flexible IR transmission fiber composed of silver chloride/silver bromide **Protective sheath**—Thermoplastic Rubber (TPR)
Operating Temperature (reaction mixture temperature)	-40° to 200°C based on probe diameter and type of conduit (see page 105)	-80° to 200°C based on probe diameter and type of conduit (see page 105)
Diameter	21mm (includes outer protective sheath)	21mm (includes outer protective sheath)
Length	1, 1.5, and 2 meter **Note**: FiberConduit length does not include the length of the probe. Refer to Sampling Technology Probe/ Conduit Availability on page 101 for a list standard conduit lengths available.	1.0m (DS Fiber-to-Sentinel only), 1.5m and 2 meter **Note**: FiberConduit length includes the probe length as the fiber continues through the probe to the sensor (see Figure 4-18).
Bend radius	30cm	30cm
Optical Range (with sensor)	Up to 2800 to 650 cm^{-1} (depending on length) Refer to "Conduits" on page 106 and Chapter 10, "Putting It All Together".	Up to 2800 to 650 cm^{-1} (depending on length) Refer to "Conduits" on page 106 and Chapter 10, "Putting It All Together".
Purge Utility	Dry air at a nominal force of 10psig (0.69barg) and flow rate of 10SCFH (4.71Lpm)	No purge required.

Reference the Chapter 4, "Probes" for specific information on the probe component of the sampling technology.Refer to the Appendix B, "Single Beam Samples" for examples of how the conduit length affects the single beam (background).

Handling a FiberConduit

The FiberConduit contains silver halide fibers that are flexible, but still require attention to proper handling to maintain useful performance over time. Take the following precautions to improve the longevity of the FiberConduit. As a point of reference, communication fibers are more flexible and robust than infrared transmitting fibers.

- Do not bend the FiberConduit beyond a 30cm radius.
- Replace protective cap on both ends of fiber when not in use—the internal AgX fibers are photosensitive to room and sunlight.
  NOTE: For the DS Series AgX fibers, the protective cap is integrated with the connector on the fiber assembly (see Figure 3-11).
- Hold the FiberConduit at both ends when transporting.
- Return FiberConduit to its original box or suitable container when not in use.
- If using the DS Series FiberConduit, return the Optical Interface Module to original box or suitable container when not in use (see "DS Series Optical Interface Modules" on page 22).



with protective cap closed

with protective cap open

Figure 3-11 DS Series connector on FiberConduit

For information about handling a ReactIR 45m or iC10 base unit, refer to "Handling a ReactIR Base Unit" on page 95.

Mirror Conduits

Mirror conduits use high precision optical mirrors to direct the infrared source beam to the sensor and then back to the ReactIR detector. Each point of articulation or knuckle houses a reflecting mirror, thus the naming convention of K4, K6, or K7. The number indicates the quantity of mirrors that also reflects the increase in conduit length with each additional knuckle.

The following image shows a K6 conduit paired with a 16mm probe and a K4 with a Sentinel:

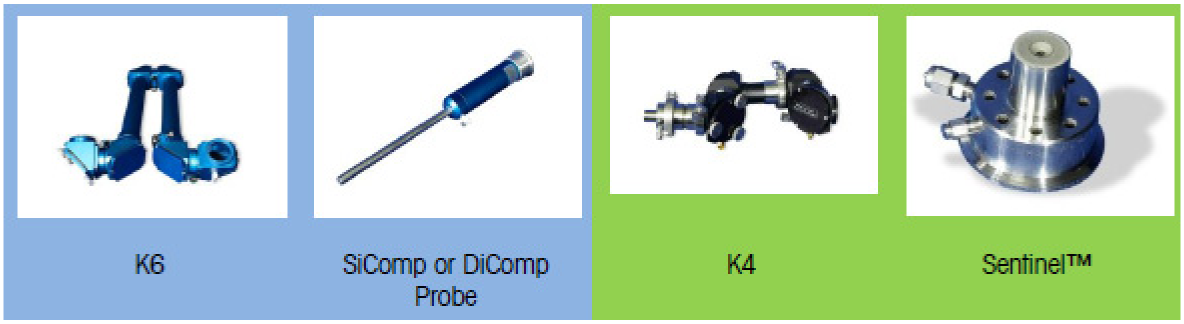


Figure 3-12 Mirror conduits and probes

Details about the K4 and K6 conduits appear on the upcoming pages.

K4 Conduit

The K4 conduit is an articulated arm that provides a path for the infrared beam to travel to a sampling device (for example, an ATR-based probe) and back to the detector. The K4 conduit consists of four mirrors and a short tube between mirrors #2 and #3. Mirror #4 is used for alignment.

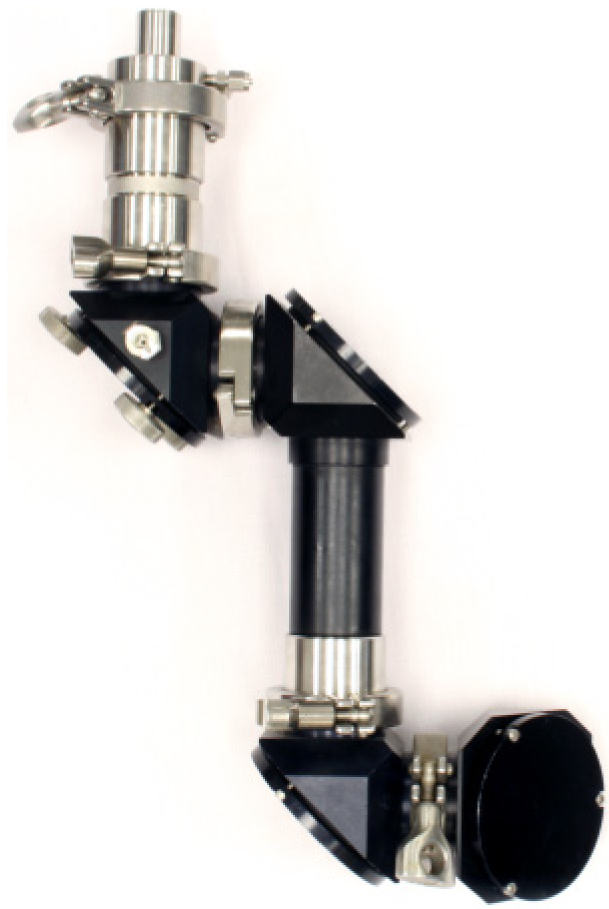


Figure 3-13 K4 Conduit (with Sentinel)

K6 Conduit

The K6 conduit is an articulated arm that provides a purged path for the infrared beam to travel to a remote sampling device (for example, ATR-based probe) and back to the detector. The K6 conduit consists of six mirrors and two tubes between mirrors #2, #3, #4, and #5. Mirror #6 is used for alignment.

Figure 3-14 K6 Conduit (with 16mm probe)

Specifications

Mirror conduits share the same specifications except for length, as noted below.

Table 3-6 Mirror Conduit Specifications

Specification	Details
Material of Construction	**Mirror**—Aluminum **Knuckle/Conduit**—Anodized aluminum
Temperature (ambient)	0° to 40°C For probe temperature specification, see Chapter 10, "Putting It All Together."
Length (extended)	K4—17 inches K6—43 inches K7—53 inches
Optical Range	4000 to 650 cm^{-1} Refer to "Conduits" on page 106 and Chapter 10, "Putting It All Together."
Purge Utility	Dry air at a nominal force of 10psig (0.69barg) and flow rate of 10SCFH (4.71Lpm)

4

Probes

In ReactIR probe-based sampling technology, a probe inserts directly into your vessel for in-situ monitoring of the reaction chemistry. (See Chapter 6, "Connecting and Using DS Micro Flow Cells" for flow chemistry sampling technology.)

The probe tip contains the sensor and seal specified for the type of chemistry to be analyzed. Probe selection is based on the size of the reaction vessel. The following images show examples of 6.35, 9.5, and 16mm probe diameters:

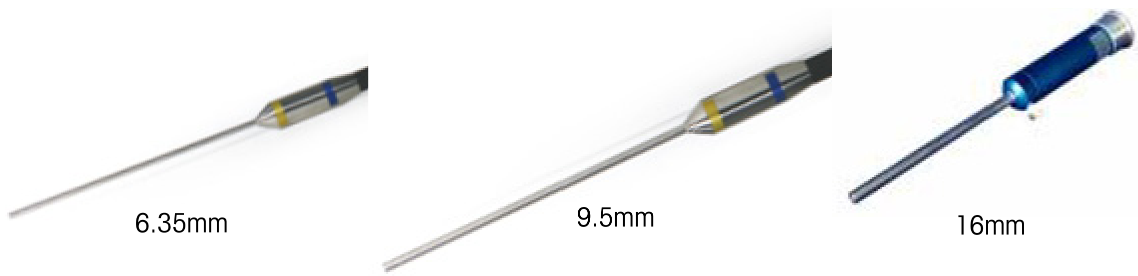


Figure 4-15 Probes—In three diameters

Figure 4-16 Probe—25mm (Sentinel)

This chapter has the following sections:

Composite Design

All standard ReactIR probes are, by design, a composite of infrared transmitting materials that creates the optical path for ATR sampling of the reaction mixture. Comp™ (composite) probes consist of a Hastelloy C-276 tube with a ZnSe focusing element, sensor, and seal at the tip—all of which make up the 'wetted' materials (see Figure 4-15 and Figure 4-17).

The composite probes were developed specifically for spectroscopic monitoring of chemical and biological reactions. The probes pack high sensitivity into a very small "hot spot" and can be inserted into vessels through a variety of standard and custom fittings. These probes can be used with a glassware adapter or with adapters specific to a particular vessel.The sensor composite is diamond (DiComp™) or silicon (SiComp™), or cubic zirconium (ZrComp™).

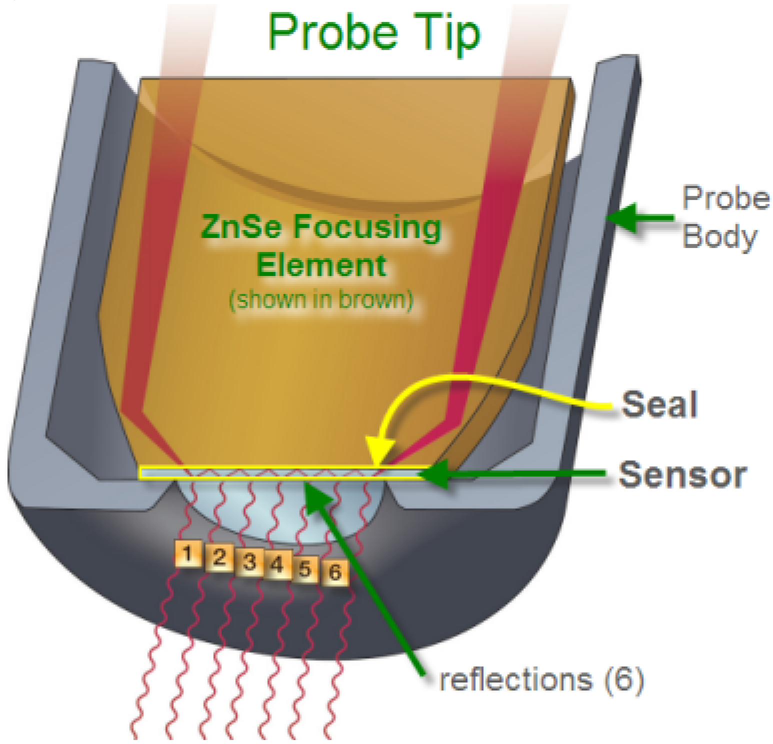


Figure 4-17 Diagram of probe tip

The seal between the metal housing and the sensor is available in Teflon and Gold, to ensure chemical compatibility. For general information, see "Sensors and Seals" on page 41. For details about sensor and seal properties, see "Sensors and Seals" on page 107.

Note: Comp™ (composite) probes have been used for more than a decade to study thousands of chemical reactions. Despite the probe's resistance to chemical attack, there are conditions that will damage the probe.

It is the customer's responsibility to verify, in advance, the chemical compatibility of the probe with the chemistry to be monitored. Test kits containing shavings of the probe body material, Diamond sensor, Silicon as an alternate to Diamond Sensor, and Gold sensor seal are available upon request.

Contact your Technical Applications Consultant for details or advice on chemical and physical compatibility of probes with your chemistry.

Comp Probe Specifications

Probe length is the distance from the tip of the sensor to the threaded end of a fitting or, in the case of integrated probes, to the nose piece. It is generally expressed as the "wetted" length. The actual probe length represents the overall length of the probe, including the threaded end or nose piece. (In Appendix A see the table entitled, "Probe Length—Insertion Depth" on page 103.)

Below is one example of the dimensional options.

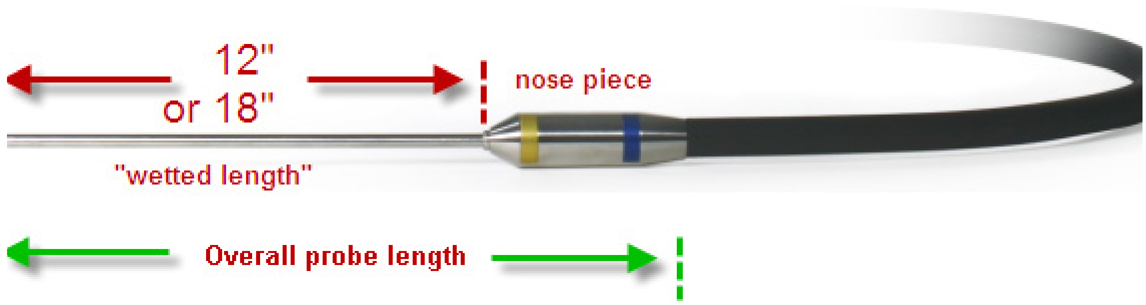


Figure 4-18 Length options (9.5mm DS probe)

Note:	The image above shows the color-coded identification on the nose piece of DS Series probes. Blue = DiComp Clear = SiComp Gold = Gold seal White = Teflon seal

Conduit length is described in Chapter 3, "Conduits".

For detailed probe specifications, see Appendix A:

- "Probe Length—Insertion Depth" on page 103
- "Probe—Temperature and Pressure" on page 105
- "Probe—Conduit Availability" on page 101

Sentinels

A Sentinel (Figure 4-16 on page 35) is a type of composite probe that is one inch (25mm) in diameter. Sentinels are available with a variety of options for interfacing to process reaction vessels including: plant reactors, autoclaves, and other specialty vessels, as well as integrating to a slip stream through special flow cell designs.

Probe-to-Reaction Vessel Compatibility

The following table shows the probe diameters that fit various size adapters in reaction vessels:

Table 4-7 Probe-to-Reaction Vessel Compatibility

Adapter Size	Probe Diameter	Volume
10/18	6.35mm	10mL–2L
14/10		
14/20		
19/22		
24/25		
24/40		
29/26		
24/40	9.5mm	50mL–2L
24/25	16mm	250mL–2L
24/29		
29/32		
29/42		
custom *	25mm (Sentinel)	> 2L

* Available with options for interfacing to process reaction vessels including:

- Plant reactors
- Autoclaves
- Slip Stream (through special flow cell designs)
- Specialty vessels

Number of IR Reflections by Probe/Micro Flow Cell

The following table shows the number of reflections (Figure 4-17) achieved based on probe diameter. Included is the DS Micro Flow Cell (Chapter 6). The 25mm Sentinel has a standard number of reflections, but additional reflection options are available as specials.

Table 4-8 Reflections per Probe Length (plus DS Micro Flow Cell)

	Probe Diameter				Micro Flow Cell
	6.3	9.5	16	25	
Reflections	6	6	6	9 *	7
Acetone Absorbance	0.2	0.2	0.2	0.3	0.28

* Additional number of reflections available by special order with Sentinel: 1, 3, 18, 30

Number of IR Reflections by Probe/Micro Flow Cell

5

Sensors and Seals

The selection of a sensor and seal for a sampling technology is based on the chemical compatibility requirements of the chemistry to be monitored. Refer to Appendix A on page 107 for sensor and seal properties.

- A sensor is a high refractive index material suitable for use in mid-infrared sampling by Attenuated Total Reflectance (ATR), defined on page 14. A variety of sensor and seal materials are available to match the chemical reaction requirements.
    - Diamond (DiComp)—Extremely robust
    - Silicon (SiComp)—Sensitive to abrasion
    - Cubic Zirconium (ZrComp)—Robust
- A seal surrounds the sensor to protect the probe. Two materials are commonly used:
    - Gold—Standard material
    - Teflon—Alternate, chemically inert material for use if gold can be attacked by the chemistry.

The following diagram shows the location and function of the sensor and seal at the probe tip:

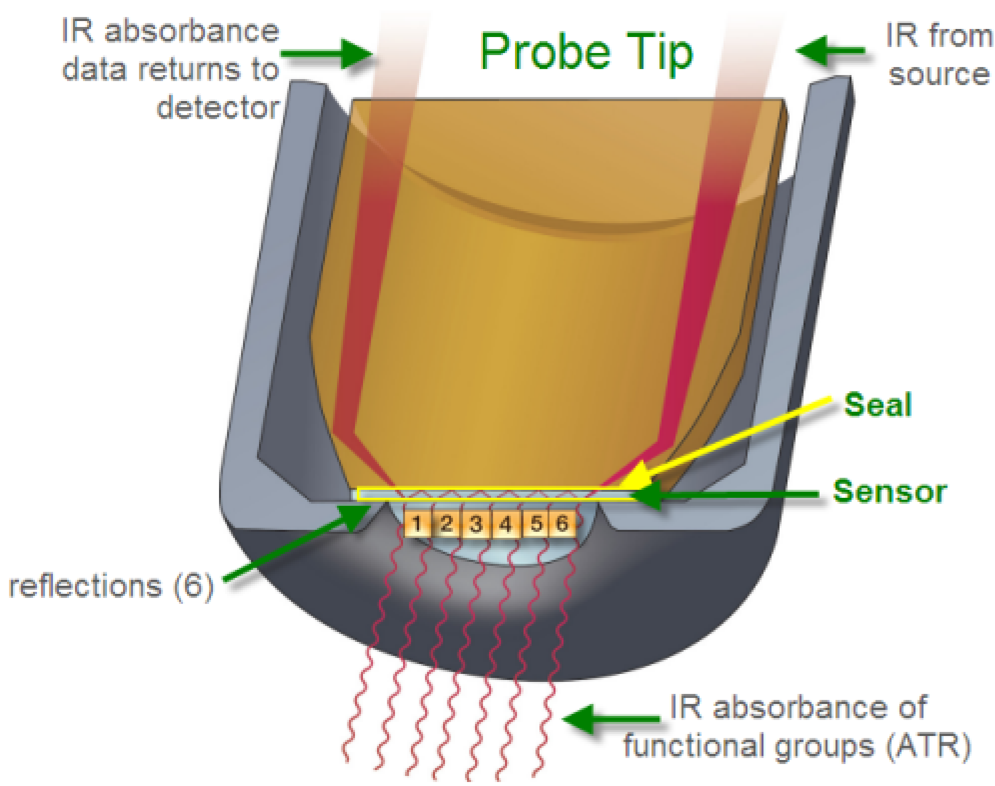


Figure 5-1 Sensor and seal at probe tip

Refer to "Number of IR Reflections by Probe/Micro Flow Cell" on page 38 for details on how the probe diameter affects the number of reflections.

Sensor Test Kits

METTLER TOLEDO has test kits available to check the chemistry for sensor, seal, and probe body compatibility. Use the contact information on page 19 to get test kit details.

6

Connecting and Using DS Micro Flow Cells

In the following sections, this chapter introduces the DS Micro Flow Cells and includes instructions for installing and using them to monitor flow chemistry:

Introduction

The DS Micro Flow Cell sampling technology is an inline analytical tool that enables processing of continuous flow chemistry without sampling. The flow cell consists of a body and head and is available in an ambient or heated model. A protective cover slides back to expose the optical connection to the ReactIR Optical Interface Module (sold separately). Flow tubing with Omnifit-style 1/4-28 fittings is user-supplied.

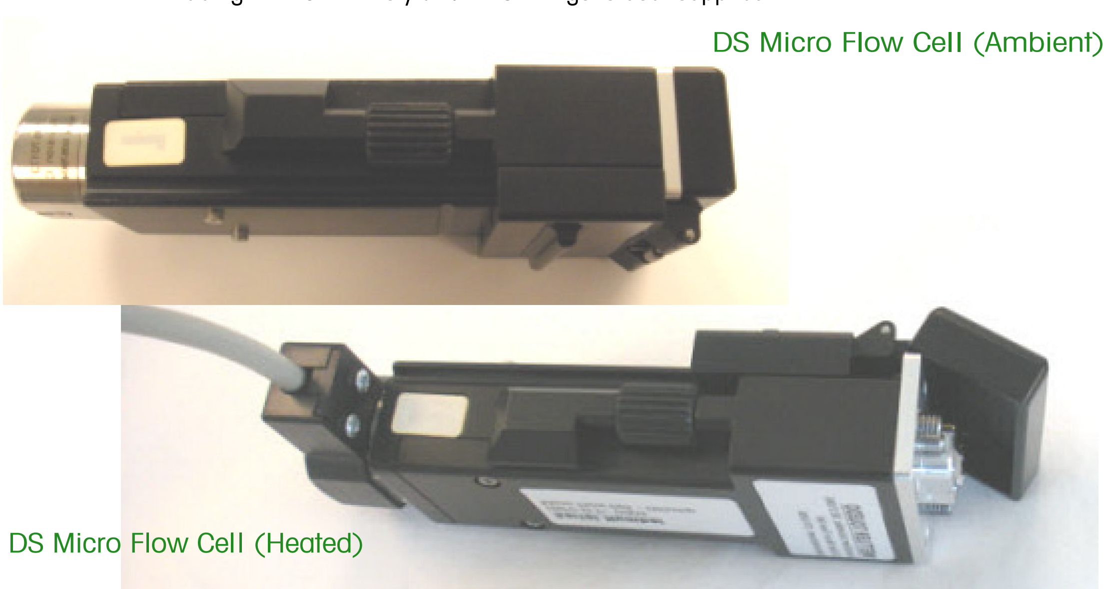


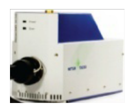
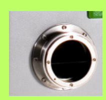
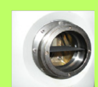
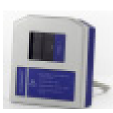
Figure 6-1 DS Micro Flow Cells—Ambient and heated models

The DS Micro Flow Cell connects to a ReactIR base unit through a single or dual-channel interface. A single Optical Interface Module connects to a ReactIR 4000, iC10, or 45m base unit. On a ReactIR 45m, the dual-channel interface (MultiplexIR Optical Interface Module) can monitor multi-stage flow chemistry with two flow cells or monitor two separate flow chemistries. Instructions for installing a DS Optical Interface Module and a DS Micro Flow Cell begin on page 53.

ReactIR Base Units and Flow Cell Options

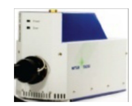
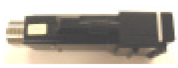
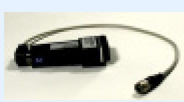
The following tables describe the DS Micro Flow Cell options that are available for specific ReactIR base units, starting with the types of optical interface modules for each base unit:

Table 6-1 DS Optical Interface Modules for ReactIR Base Units

			ReactIR Base Units		
Image	Component	Part Number	45m	iC10	4000
					
	Factory SIM				
	SIM Divider		included	14170071*	14170072*
Optical Interface Modules (sold separately)					
	**MultiplexIR (MUX)** Optical Interface Module	14440000	✓		
	**Single** Optical Interface Module with RTD	14440001	✓		
	**Single** Optical Interface Module without RTD	14440002		✓	✓

Below are the DS Micro Flow Cell options. Notice that each flow cell model include one head of a specified volume. You can order extra heads to interchange on the flow cell body.:

Table 6-2 DS Micro Flow Cell Options for ReactIR Base Units

			ReactIR Base Units			
Image	Component	Part Number	15	45m	iC10	4000
						
DS Micro Flow Cell Models						
	**DS Micro Flow Cell—Ambient** (Includes flow cell body and one head)		✓	✓	✓	✓
	Head volume: 50µL	14430683				
	10µL	14430685				
	**DS Micro Flow Cell—Heated** (Includes flow cell body and one head, plus heater controller and power supply not shown)		✓	✓	✓	✓
	Head volume: 50µL	14430682				
	10µL	14430684				
	Heater Controller	14430686				
Tubing and Omnifit-style 1/4-28 fittings are not provided.						

The following figure shows examples of single and multiple DS Micro Flow Cells installed in ReactIR base units:

Figure 6-2 DS Micro Flow Cell examples

Below is an enlarged image of two DS Micro Flow Cells with the direction of flow identified. The flow cell on the right is the model with heater.

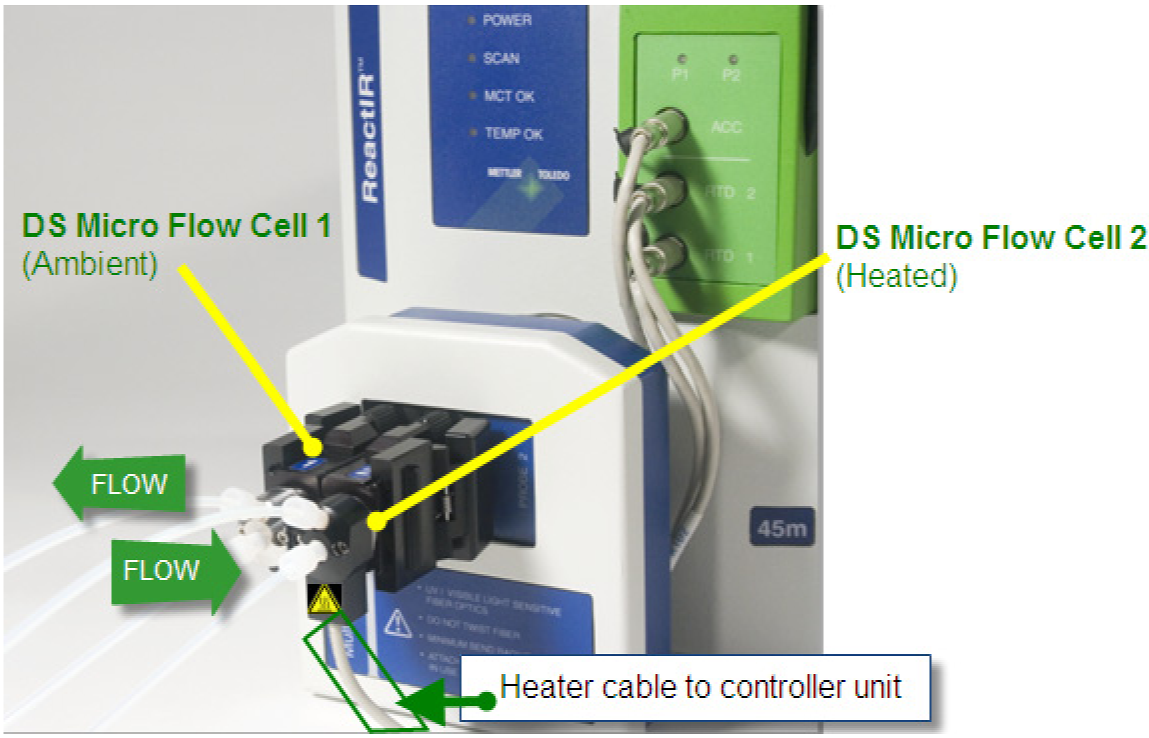


Figure 6-3 Two DS Micro Flow Cells on ReactIR 45m

Parts of a DS Micro Flow Cell

A DS Micro Flow Cell consists of the cell body and head. The head is available in an ambient or heated model of 10 or 50 microliter (µL) volume capacity (see Figure 6-5). In addition, a cover slides back to expose the optical connector that attaches to the ReactIR Optical Interface Module. Two thumb screws in the vertical plane secure the flow cell to the interface module.

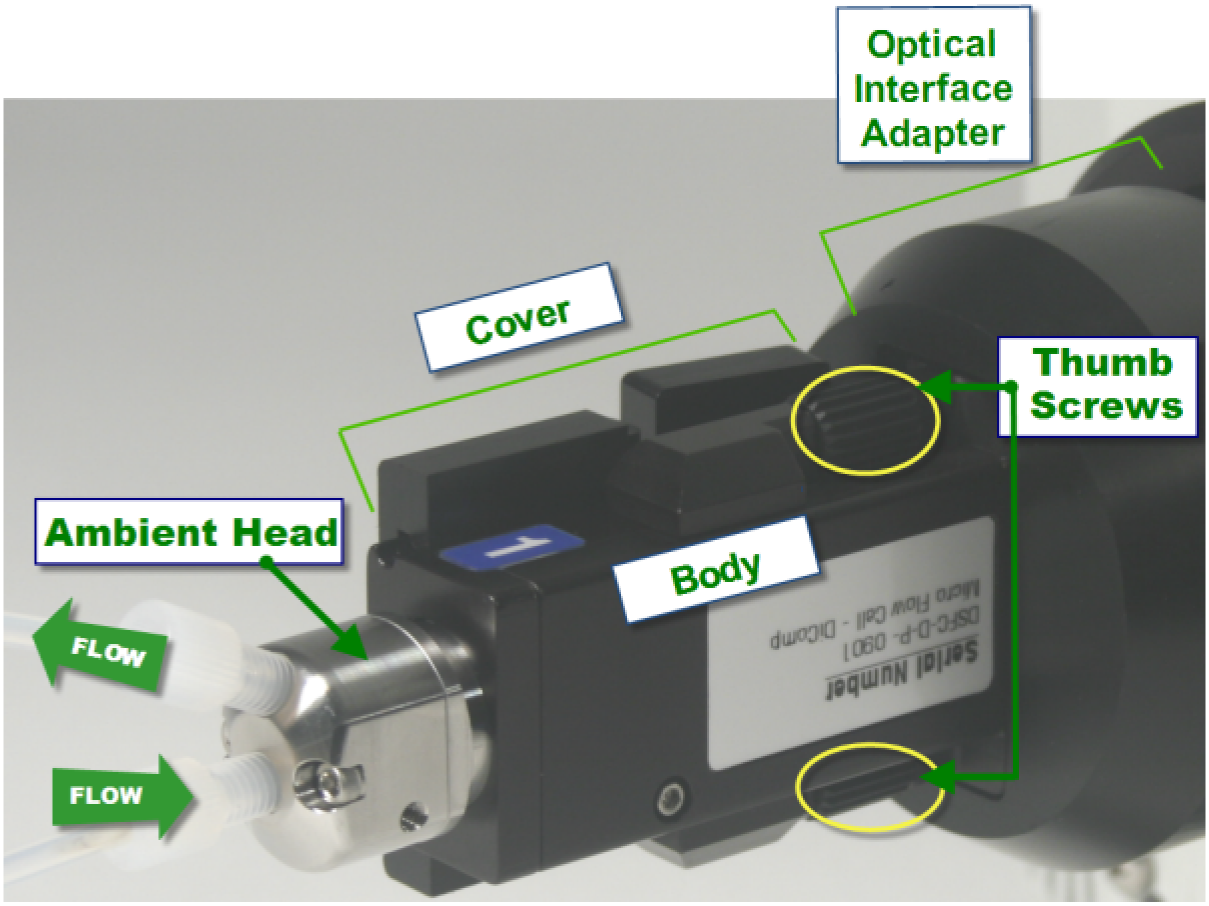


Figure 6-4 DS Micro Flow Cell (ambient)—Labeled

A DS Micro Flow Cell is available in 10 microliters (µL) or 50µL flow volume capacities.

Figure 6-5 Flow cell head internal volume—10µL (left) or 50µL (right)

Heated Model

A DS Micro Flow Cell heated model to maintain the temperature of the flow chemistry during the monitoring process. In a heated model, a sleeve surrounds the flow cell head and connects to a heater controller. Instructions for setting the temperature are under "Changing Temperature Setting" on page 60.

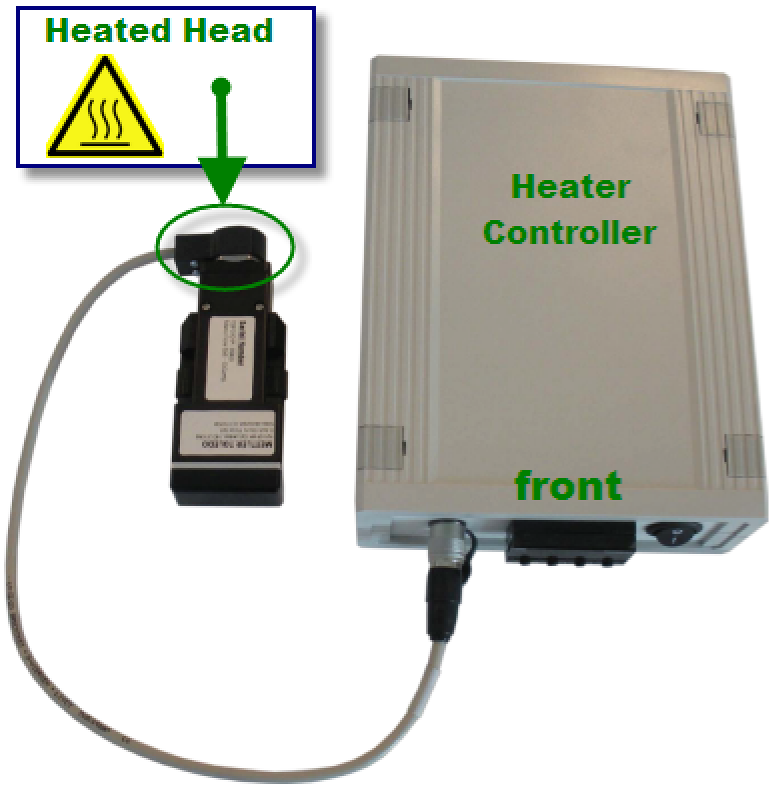


Figure 6-6 Heater connection to controller

Safety

Use care when touching a heated flow cell head that has been in use as it can reach temperatures up to 75°C (167° F).

WARNING—A warning label on the heated head reminds you that it can be hot, so handle with caution.

The heated head has a built-in thermal cut-off switch set to shut down at 90° C (194° F), however **do not exceed 75° C**.

The heater controller includes a power supply that connects to the back of the unit.

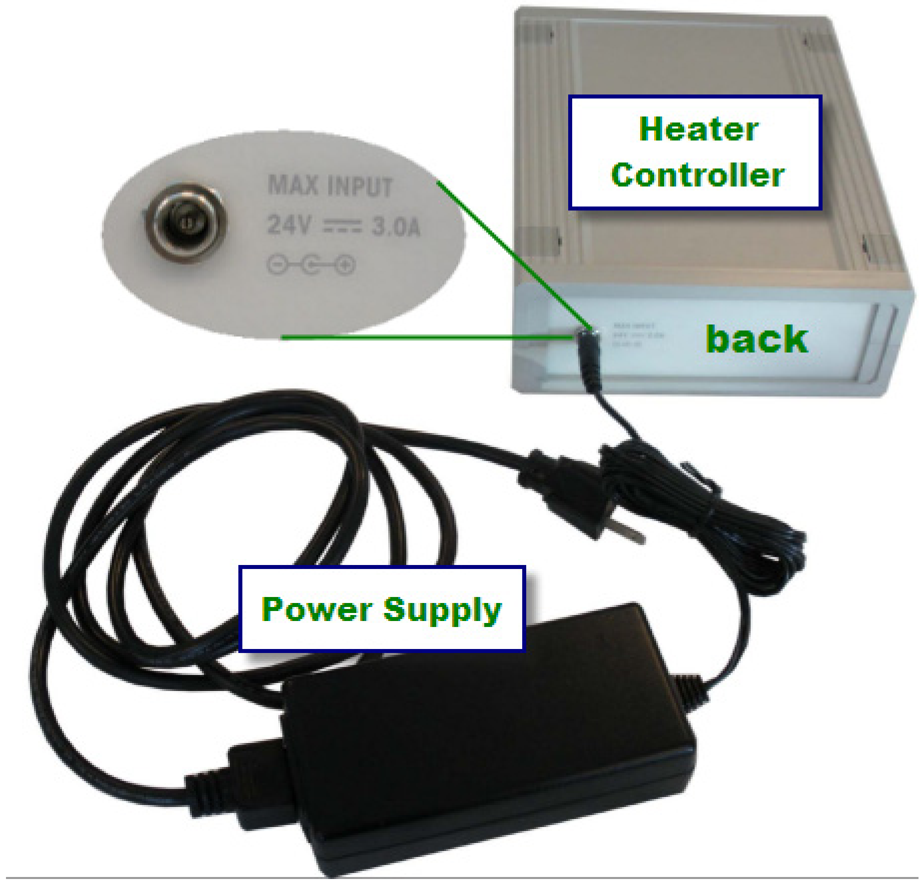


Figure 6-7 Heater controller power supply

DS Micro Flow Cell Specifications

This section provides specifications and dimensions for the two DS Micro Flow Cell models.

DS Micro Flow Cell (Ambient)

Table 6-3 Ambient DS Micro Flow Cell Details

Specification	Details
Name	DS Micro Flow Cell–DiComp–Ambient or DS Micro Flow Cell–SiComp–Ambient (includes flow cell body and one head of specified volume)
Description	DS Micro Flow Cell with Integrated DiComp Sensor or DS Micro Flow Cell with Integrated SiComp Sensor
Inlet and Outlet Ports	For use with 1/4-28 flat bottom (Omnifit style) fittings
Internal Volume	10µL (microliter) head **or** 50µL head (see Figure 6-5)
Pressure Rating	**DiComp**: Ambient to 30bar (435 psig) **SiComp**: Ambient to 7bar (102 psig)
Optical Window	4000 to 650 cm^{-1}
pH Range	**DiComp**: 1 to 14 **SiComp**: 1 to 10
Wetted Materials	C-22, diamond and gold
O-ring	Kalrez 4079, K#903 (o-ring size) 7.65 I.D. x 1.63mm dia. (0.301 I.D. x 0.064in. dia.)

DS Micro Flow Cell (Heated)

Table 6-4 Heated DS Micro Flow Cell Details

Specification	Details
Name	DS Micro Flow Cell–DiComp–Heated or DS Micro Flow Cell–SiComp–Heated (includes flow cell body, heated head of specified volume, and heater controller with power supply)
Description	DS Micro Flow Cell with Integrated DiComp Sensor or DS Micro Flow Cell with Integrated SiComp Sensor
Inlet and Outlet Ports	For use with 1/4-28 flat bottom (Omnifit style) fittings
Internal Volume	10µL (microliter) head **or** 50µL head (see Figure 6-5)
Temperature Range	Ambient to 60°C (140°F) (externally controlled, not software controlled)

Table 6-4 Heated DS Micro Flow Cell Details (continued)

Specification	Details
Pressure Rating	**DiComp**: Ambient to 30bar (435 psig) **SiComp**: Ambient to 7bar (102 psig)
Optical Window	4000 to 650 cm^{-1}
pH Range	**DiComp**: 1 to 14 **SiComp**: 1 to 10
Wetted Materials	C-22, diamond and gold
O-ring	Kalrez 4079, K#903 (o-ring size) 7.65 I.D. x 1.63mm dia. (0.301 I.D. x 0.064in. dia.)
Electrical power supply (Heater Controller)	24VDC, 3A

Dimensions

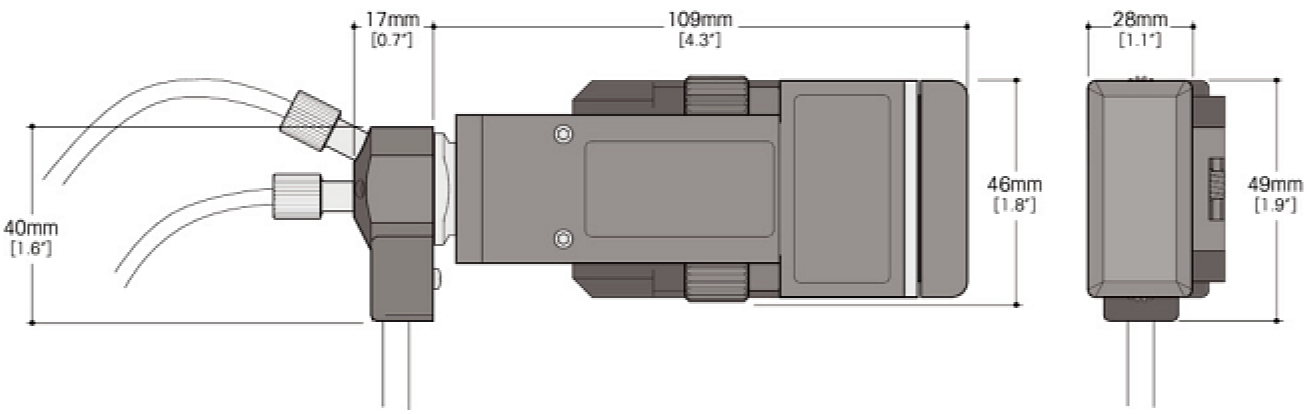


Figure 6-8 DS Micro Flow Cell—Dimensions

Connecting DS Micro Flow Cell to Optical Interface Module

A DS Micro Flow Cell connects to the Sample Interface Module (SIM) on a ReactIR 45m, iC10, or 4000 base unit through a DS Optical Interface Module. A Quick Reference sheet of connection instructions comes with the Optical Interface Module and a separate Quick Reference comes with the DS Micro Flow Cell.

Before You Begin

Some existing ReactIR systems have an older style retaining ring on the SIM that must be replaced with the newer model that includes a pin-hole guide (Figure 6-9).

Verify that the SIM on your system has a mechanical divider and order one, if necessary (Table 6-1 DS Optical Interface Modules for ReactIR Base Units on page 6-44 shows part numbers.).

Figure 6-9 SIM showing mechanical divider and pin-hole guide

Connecting DS Optical Interface Module to SIM

The following instructions demonstrate how to connect a DS Optical Interface Module to a SIM. The same instructions apply to ReactIR 45m, iC10, and 4000 base units.

Note: ReactIR 15 base units have a factory-standard DS SIM, so it is not necessary to install a DS Optical Interface Module. Simply directly connect the DS Micro Flow Cell as described "Connecting Micro Flow Cell to Optical Interface Module" on page 55.(The DS SIM is shown on page 21.)

1. If you need to replace an older style retaining ring with the new style that includes a pin-hole guide, use the 0.050" Allen key (provided) to remove the four screws that hold the retaining ring on the factory-standard SIM. Then, remove the ring.

   Note: Removal of the retaining ring from the factory-standard SIM only applies to existing ReactIR base units with the old style ring. New base units ship with a retaining ring that includes the pin-hole guide shown in Figure 6-9.

2. Replace the old retaining ring with the DS-Series retaining ring that includes a pin-hole guide. Pin hole must be in the seven o'clock (7:00) position.
3. Replace and tighten the four screws.
4. Remove the protective cap on the back of the DS Optical Interface Module and install the module on the SIM, as follows.
   a. Take special care to insert the pin into the pin-hole guide.

b. Notice that the front of an Optical Interface Module has one or two D-shaped labels that indicate the orientation for the sampling technology connection. The figure below shows the MultiplexIR and SINGLE Optical Interface Modules installed.

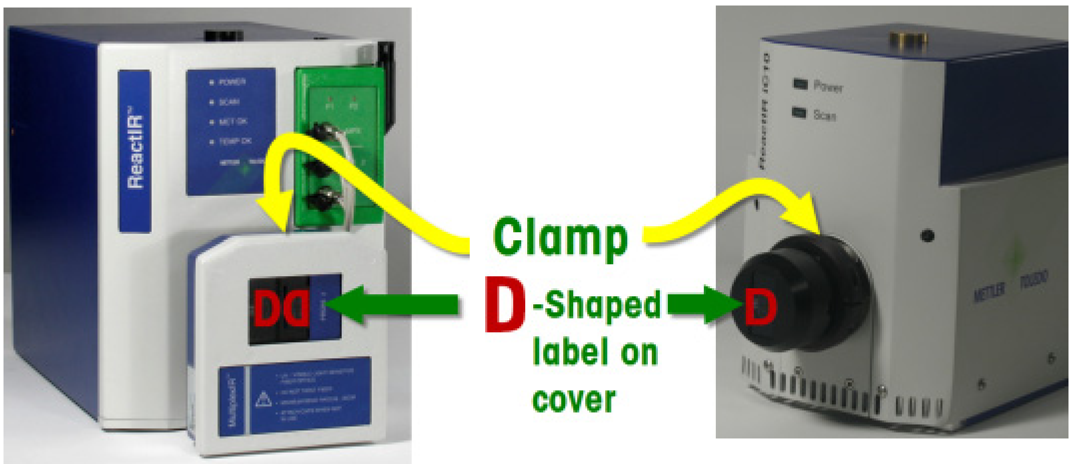


Figure 6-10 Multiplex (MUX) and Single Optical Interface Modules installed

5. Position and tighten the clamp (provided) around the SIM.

Connecting Micro Flow Cell to Optical Interface Module

1. On the DS Micro Flow Cell, use your thumb to slide the protective cap back and expose the optics connector.

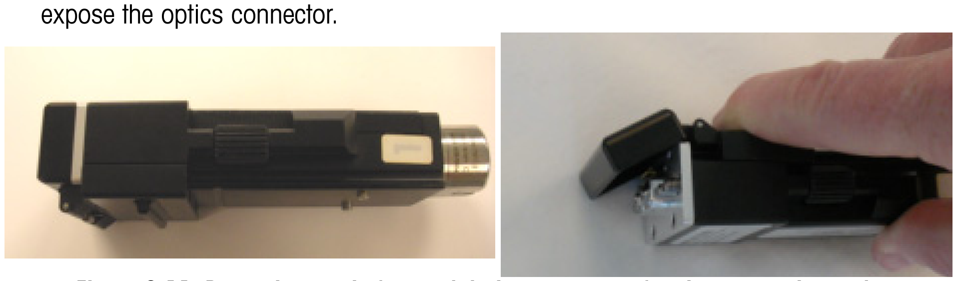


Figure 6-11 Protective cap before and during exposure of optics connection end

2. Insert the DS Micro Flow Cell optics connection end as follows, depending on the type of ReactIR base unit:
   - For ReactIR iC10, 45m, or 4000, insert the flow cell optics connection end into the Optical Interface Module.
   - For a ReactIR 15 base unit, insert the flow cell options connection directly into the DS SIM on the front of the unit.

   The cover on the interface module flips in as you guide the flow cell into position.

3. Secure the DS Micro Flow Cell with the two captive screws in the vertical plane. Figure 6-12 shows the thumb screw and identifies the Micro Flow Cell cover, body, and head.

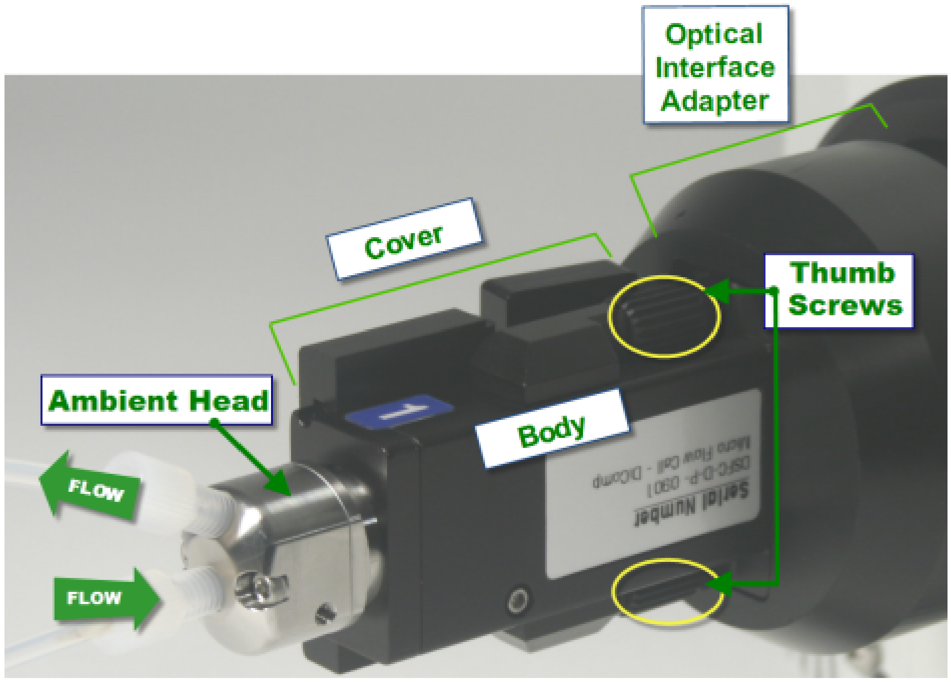


Figure 6-12 DS Micro Flow Cell (ambient) installed in a SINGLE Optical Interface Module

TIP: You can tighten both thumb screws with a single hand motion.

- Place your hand around the flow cell so your thumb is over the top screw and your fingers are below the bottom screw.
- Then, touch the top and bottom screws and roll both your thumb and fingers in a clockwise rotation.

4. If you have the heated flow cell model, connect the cord from the flow cell head to the front of the heater controller. (The receptacle is labeled "Flow Cell.")

Figure 6-13 DS Micro Flow Cell (heated) connected to heater controller

---

Note: About Two Types of Temperature Components

- Optical Interface Modules for the ReactIR 45m include single or multiple Resistive Temperature Detectors (RTDs) that are unrelated to the heated DS Micro Flow Cell. The heated model flow cell connects to an external heater controller as described on "Heated Model" on page 50.
- RTDs communicate with iC IR software to monitor the affects of temperature changes on reactive chemistry. The SINGLE Optical Interface Module includes one RTD that plugs in to the ReactIR base unit (RTD1). The MultiplexIR (MUX) Optical Interface Module has three cables—two for RTD 1, RTD 2, and a third for Accessory (ACC). The ACC cable controls the motor that switches from Probe 1 to Probe 2.

---

5. Following installation, connect your tubing to the flow cell head with the flow direction as shown under "Setting Flow Direction" on page 58.

Note: If it is necessary to reverse the flow cell head to achieve the proper flow direction, see "Reorienting a DS Micro Flow Cell Head (if needed)" on page 61 before connecting your tubing.

---

6. If you are installing a heated DS Micro Flow Cell:
   a. Connect the Heater Controller power supply to the back of the controller (see Figure 6-7 on page 51.) and plug in the power supply.
   b. Set the desired temperature (see "Changing Temperature Setting" on page 60).

Using DS Micro Flow Cells

Once a flow cell is connected to the Optical Interface Module of your ReactIR base unit, the next step is to connect your flow chemistry to the flow cell head in the prescribed flow direction (Figure 6-14). If you have the heated model, set the heat to the desired temperature as described on page 60, and monitor the chemical spectroscopy though iC IR software.

Setting Flow Direction

A specific direction of the flow to the DS Micro Flow Cell is required to ensure the chemistry comes in proper contact with the sensor. The following diagram shows the correct direction:

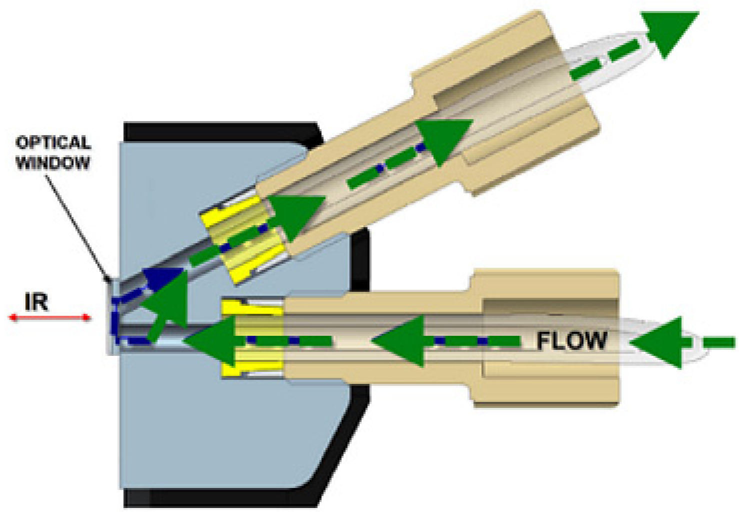


Figure 6-14 Flow direction

Below is an flow direction image of a heated DS Micro Flow Cell.

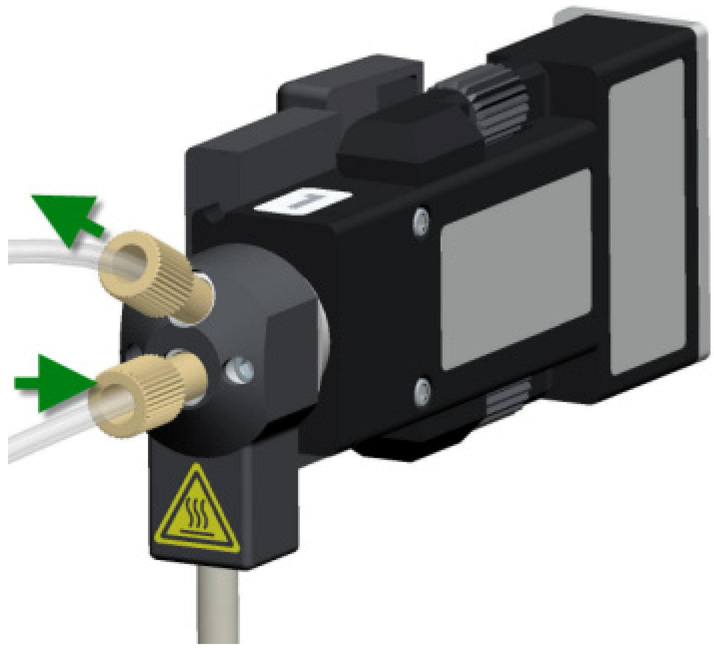


Figure 6-15 Flow direction (shown on heated model)

1. Insert the 1/4-28 Omnifit-style fitting of the input tube to the center of the DS Micro Flow Cell head as shown.

Figure 6-16 Input and output to flow cell head (ambient)

2. Insert the output fitting in the upper opening.
3. Check that each fitting is securely inserted in the openings.
4. If you have the heated model, proceed to set the temperature as described next.

Changing Temperature Setting

The temperature for a DS Micro Flow Cell heated head is factory set at a low temperature. Before using the heated model, set the temperature according to the flow chemistry being monitored. Once heated, the flow cell head can be extremely hot. See "Safety" on page 50 for the warning on the heated head temperature.

Use the buttons on the front of the temperature controller to set the temperature required for your flow chemistry, as follows:

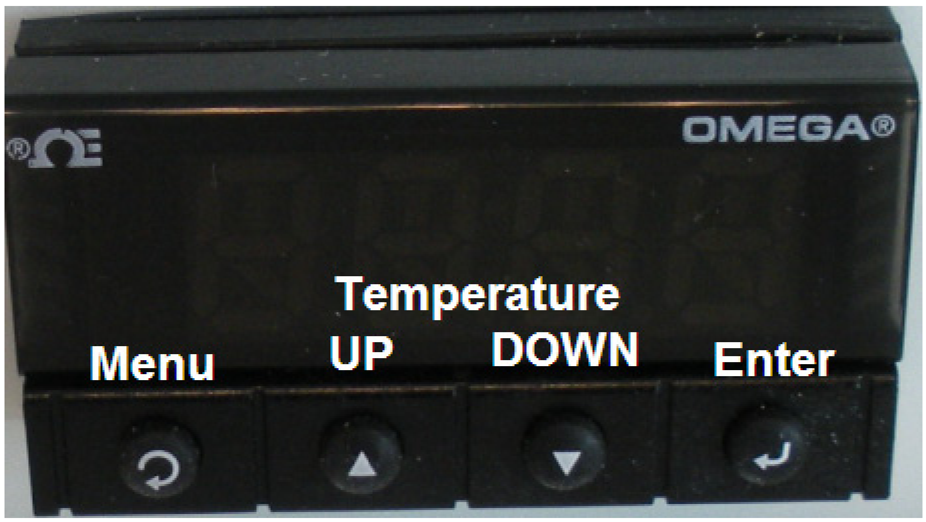
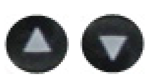


Figure 6-17 Temperature controller—setting buttons

1. Press the **Menu** button and cycle to the **SP1** menu option.

2. Press the **Enter** button to go into temperature setting mode.



3. Press the temperature **UP** and **DOWN** buttons to cycle to the required temperature.

4. Press **Enter** to save the temperature setting.

5. Press **Menu** again to return to the default menu setting.

Switching Head Volumes

A DS Micro Flow Cell is available with one of two volume options—10 or 50 microliters. If you want to switch one flow cell head for another, use the instructions under "Reversing a DS Micro Flow Cell Head for Proper Input Flow" on page 62 to loosen the two screws that hold the head to the body.

Reorienting a DS Micro Flow Cell Head (if needed)

Flow input to a DS Micro Flow Cell must be straight into the center of the flow cell head as shown in Figure 6-16 on page 59. Flow cell heads are symmetrically designed to allow 180-degree vertical reorientation in the flow cell body, if needed. Before attaching your tubing, verify proper orientation of the flow cell head.

For example, if you are installing two DS Micro Flow Cells in a ReactIR 45m, the flow cell body inserts one way into the D-shaped receptacle on the MUX interface. As a result, it may be necessary to reverse one of the flow cell heads to maintain the proper flow direction. Figure 6-18, below, shows this type of installation.

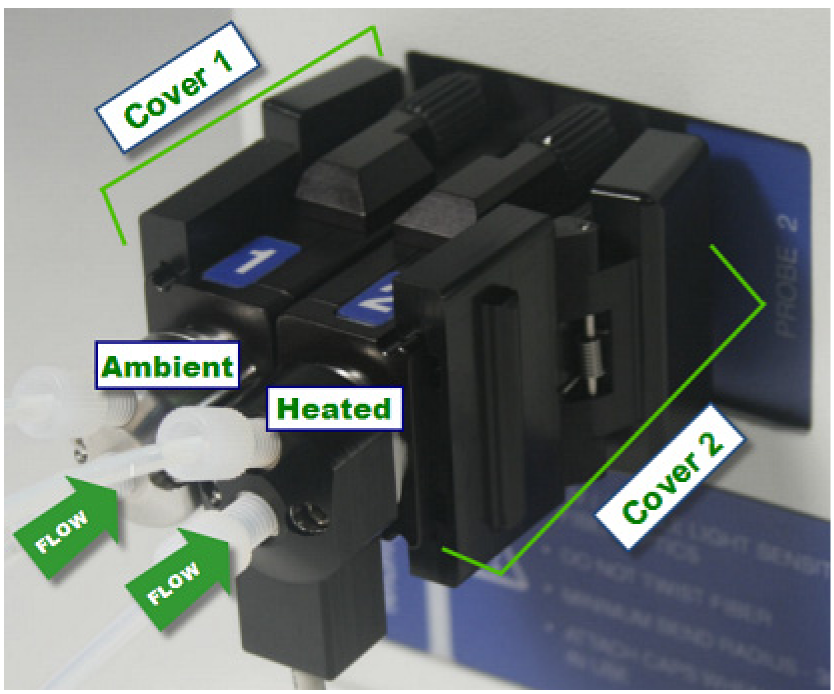


Figure 6-18 Two flow cells (ambient on left, heated on right) in proper orientation

A proper orientation has the following characteristics:

- Slide-back protective covers are on the outside of flow cell bodies. This applies to MultiplexIR (MUX) installations on ReactIR 45m base units.

- Input flow tube is at the center of the flow cell head with the output flow directed upward. (Figure 6-14 on page 58, Figure 6-16 on page 59, and Figure 6-18 on page 61 illustrate flow direction.)

Reversing a DS Micro Flow Cell Head for Proper Input Flow

If the flow direction requires correction, use the instructions below to reverse the flow cell head. Figure 6-20 features the ambient model, but the same instructions apply to a heated flow cell head.

WARNING—A warning label on the heated head reminds you that it can be hot, so verify that the heater controller is off before reorienting a heated flow cell head.

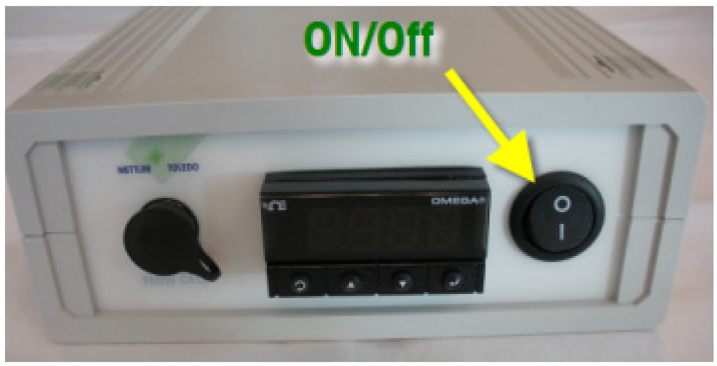


Figure 6-19 Heater controller front panel—Power

1. Using a 3/32 allen key (provided), loosen the two screws and disengage the flow cell head.

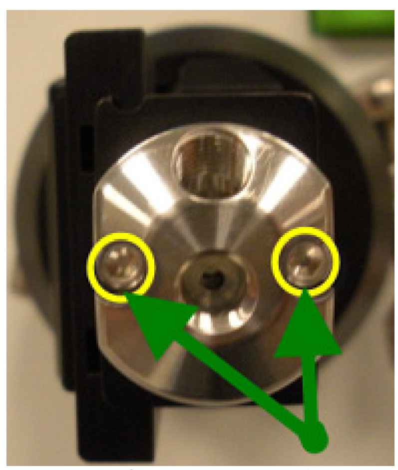


Figure 6-20 DS Micro Flow Cell head screws (shown on ambient model)

2. Rotate the head 180° and reinsert it into the flow cell body. Take care to use the alignment pin so the input flow tube aligns with the center of the flow cell head.
3. Tighten the screws.

Using iC IR software

iC IR software for the ReactIR base unit enables you to monitor the DS Micro Flow Cell data to see and analyze the rapid consumption of reagents and formation of product in real time. Unstable intermediates can be observed *in situ* to provide seamless and analysis of chemical reactions in progress. Refer to the online Help or the *iC IR User Guide* for instructions

Maintaining DS Micro Flow Cells

As part of a regular maintenance schedule, perform the following maintenance on the DS Flow Cells:

- Clean the points where the chemistry flows into the DS Micro Flow Cell head at the Omnifit-style fittings. Refer to Figure 6-16 on page 59 shows the flow direction on an ambient flow cell head.
- Replace the o-ring in the DS Micro Flow Cell head with the Kalrez o-ring specified under "DS Micro Flow Cell Specifications" on page 52

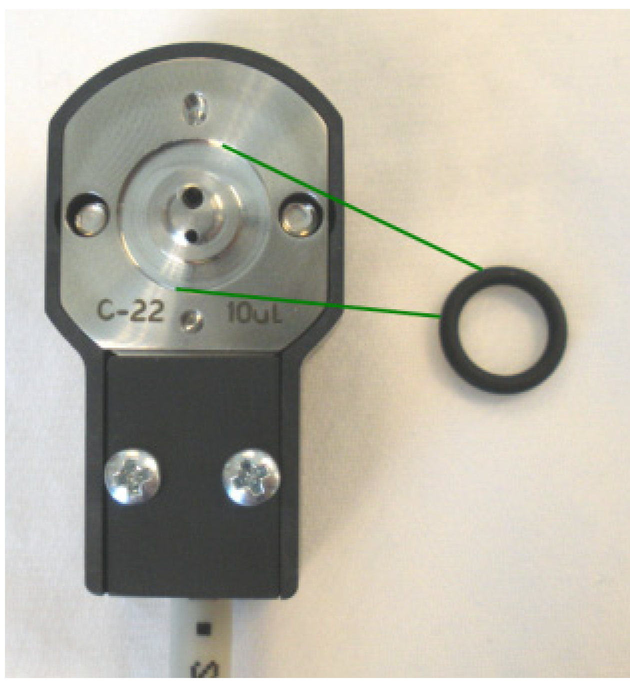


Figure 6-21 O-ring in flow cell head—10µL (heated)

7

Connecting Conduit Sampling Technologies

Once you have selected the sampling technology components that meet your reaction vessel and chemical compatibility requirements, this chapter describes how to connect the FiberConduit or Mirror conduit components to your ReactIR or MonARC base unit.

Note: See Chapter 6, "Connecting and Using DS Micro Flow Cells" for instructions on how to connect the flow cell sampling technology.

If you ordered a sampling technology along with your ReactIR or MonARC instrument, a METTLER TOLEDO Field Service Engineer sets up the connections. If you want to switch from one sampling technology to another, this chapter provides the procedures in the following sections:

The connection information starts with the Sample Interface Module (SIM) and moves through the other sampling technology components—the conduit and the probe (with sensor and seal).

Note: Integrated FiberConduits, including the DS Series, provide the conduit and probe as a single unit.

Connecting a Conduit to the SIM

Conduits connect to the Sample Interface Module (SIM) on the ReactIR base unit. The procedure varies based on the type of conduit—mirror or fiber—and whether the connection is directly to the factory-standard SIM (Bell-Style connection) or through a DS Series interface that connects to the SIM. Refer to Chapter 2, "Sample Interface Modules (SIMs)."

Mirror Conduits—Direct Connection

Mirror conduits (K4, K6 K7) connect directly to the SIM with a bell-shaped connector, and a clamp secures the connection. Below are instructions for a composite probe and a Sentinel (on a K4 mirror conduit).

Note: ReactIR 15 base units use DS sampling technologies only and do not accept mirror conduits.

Composite Probe (with a K6 conduit)

1. Place the Comp probe on the last mirror of the conduit and tighten the clamp.
2. Connect the first mirror of the conduit to the ReactIR SIM (located on the front of the 45m or iC10 base units and on the side of a MonARC—see Chapter 2).
3. Tighten the clamp.
4. Connect purge utility at knuckle 6 and at the probe body.

Sentinel (with a K4 conduit)

1. On the K4 conduit, place the Sentinel on the spacer connected to the last mirror that has the adjustment screws, and tighten the clamp.
2. Install a 25% energy composition screen in the SIM (see Figure 7-10).
3. Attach the K4 conduit to the SIM (located on the front of the 45m or iC10 base units and on the side of a MonARC—see Chapter 2).
4. Tighten the clamp.
5. Connect purge utility at knuckle 4.

Figure 7-1 Sentinel (shown on a ReactIR 45m)

FiberConduits—Bell-Style or with DS Optical Interface Module

FiberConduits connect to the ReactIR base unit either directly (with a bell-style adapter) or through a DS Series Optical Interface Module.

Note: ReactIR 15 base units include a factory-standard DS SIM that only accepts DS sampling technologies.

Bell-Style Connection

1. Attach the FiberConduit bell-style connector to the ReactIR SIM (round metal housing located on the front of the instrument—see page 21), and tighten the clamp.
2. For systems that require a purge, face the purge connector on the FiberConduit housing toward the horizontal or vertical side, based on the probe diameter, as follows:
   9.5 mm diameter—3 o'clock or 9 o'clock position (horizontal)
   6.35 mm diameter—6 o'clock or 12 o'clock position (vertical)
3. Connect the purge utility at bell-style connector.

DS Series Optical Interface Connection

A DS Series FiberConduit connects to ReactIR iC10, 45m, and MonARC base unit through a DS Optical Interface Module. The DS interface module comes in three models—one for single probe connections, one for single probe with RDT, and one for multiple probes (MultiplexIR or "MUX", see image on page 22). The single models are ideal for laboratory use as they fit under a hood.

Note: ReactIR 15 base units have a factory-standard integrated single DS optical interface module. The interface module is a DS SIM that is built in to the front of the unit (see page 21).

At the probe end, a DS Series has two configurations—an integrated composite probe or a bell-style connector (Figure 7-2). You can connect either a Sentinel or gas cell to the bell-style connector. (Figure 7-3 shows a gas cell configuration.) In either case, a purge utility is required at the bell-style connector (also shown in figures).

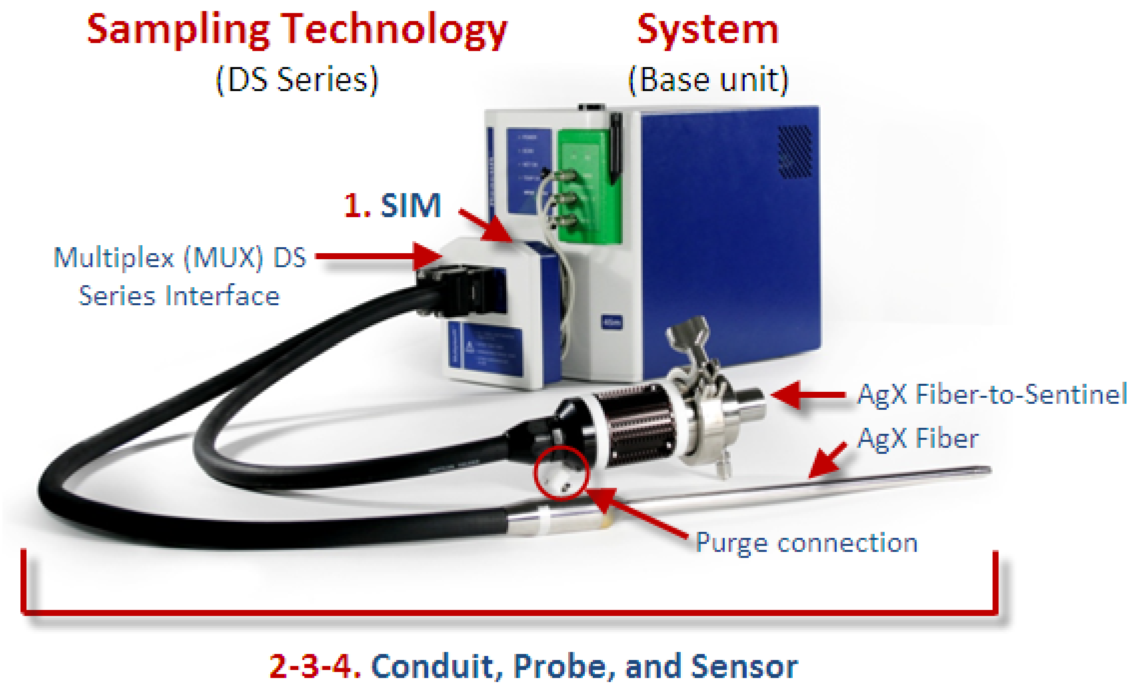


Figure 7-2 DS Fibers attached to a 45m through Multiplex interface

DS Series Fiber-to-Gas Cell

The following image highlights a gas cell sampling technology in a DS Series Multiplex (MUX) configuration:

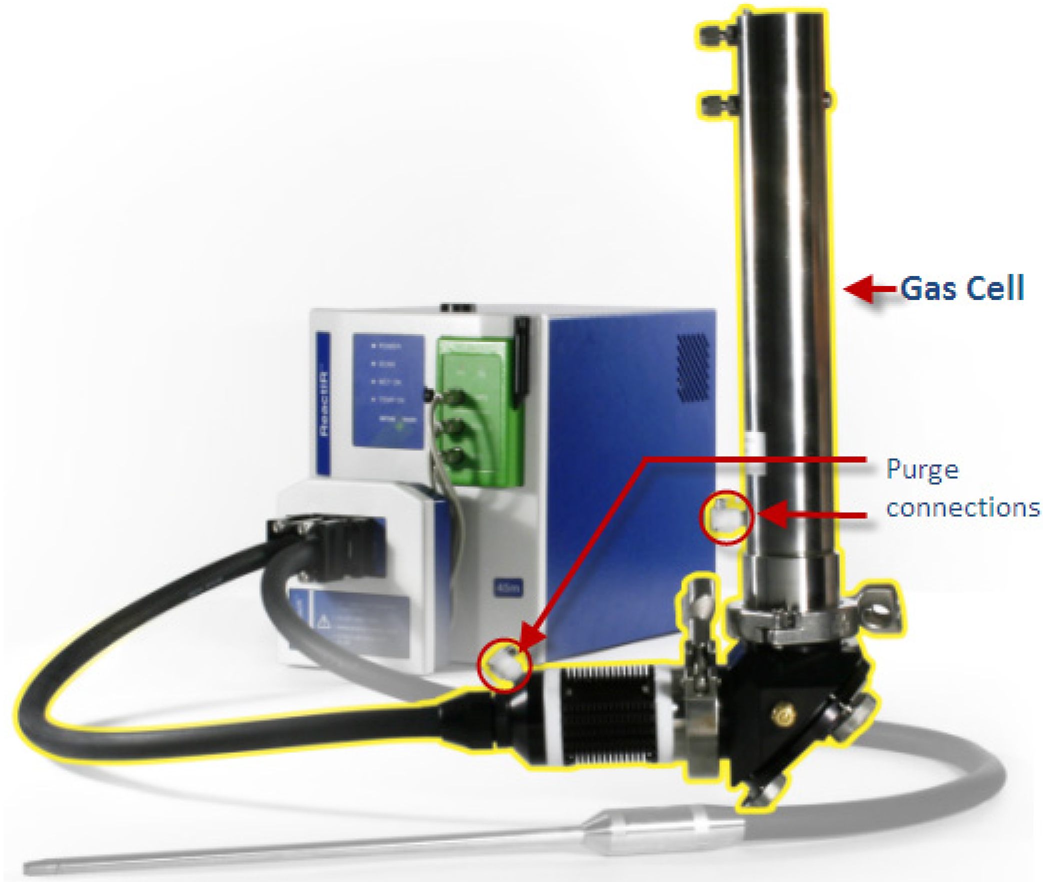


Figure 7-3 DS Fibers attached to a 45m with gas cell

How to Connect a DS Series Interface Module to the SIM

The procedure below features a DS Series Optical Interface Module—Single on a MonARC. The same instructions apply to the 45m and iC10 (or ReactIR 4000) base units. ReactIR 45m base units can use the MultiplexIR (MUX) interface module.

1. Remove the retaining ring from the factory-standard SIM by unscrewing the four screws shown in red circles in Figure 7-4.

Note: Removal of the retaining ring from the factory-standard SIM only applies to existing ReactIR instruments. New instruments come with a retaining ring that includes the pin-hole guide shown in Figure 7-4.

2. Replace the retaining ring with the DS retaining ring that includes a pin-hole guide.

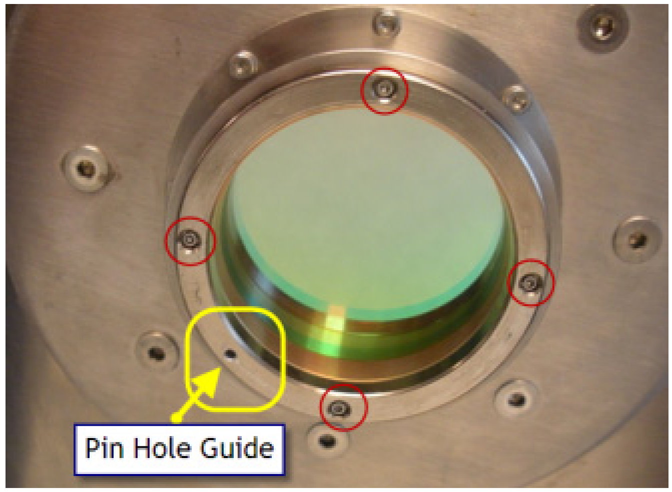


Figure 7-4   DS Series interface retaining ring with pin hole guide

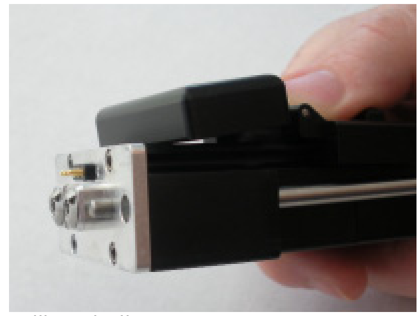
3. Install the DS Optical Interface Module onto the flange that surrounds the SIM.
   Take special care to insert the Optical Interface pin into the pin hole guide—not one of the screw holes.

Figure 7-5 DS Series optical interface on SIM flange

4. Tighten the clamp. Notice that the cap shows the orientation of the connection in the "D" position.

5. At the connector end of the DS Series FiberConduit, use your thumb to slide the protective cap back to expose the optics.

with protective cap closed



with protective cap open

Figure 7-6 DS Series connector on FiberConduit

6. Insert the connector into the Optical Interface Module. The protective cap on the adapter flips in as you push the FiberConduit connector into position.
7. Secure the DS Series AgX FiberConduit with the two captive screws in the vertical plane. (The second screw is not visible in the figure below.)

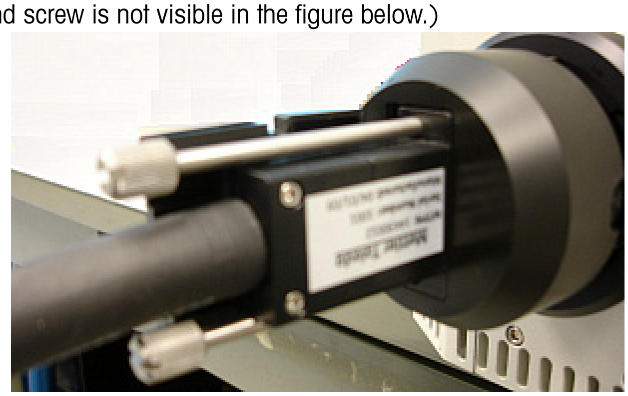


Figure 7-7 DS Series optical interface with fiber installed

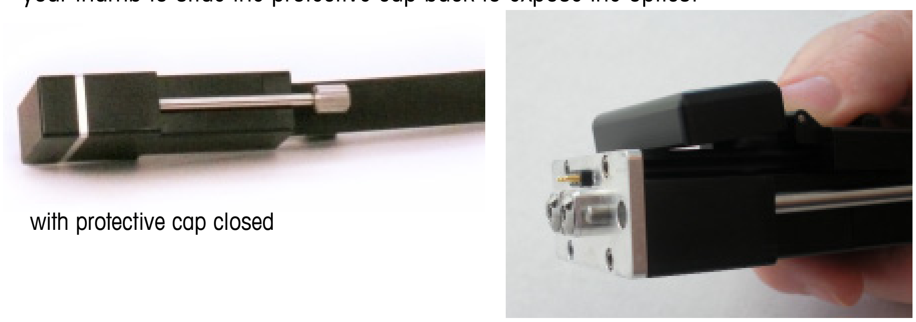
Note: No rotational alignment is necessary for integrated DS series probes because they are prealigned. However, a DS series Fiber-to-Sentinel configuration does require alignment of the sentinel (see page 82).

8. If applicable, connect purge utility to the probe end at the bell-style connector (see Figure 7-2 and Figure 7-3.

How to Connect DS Series Sampling Technologies to a DS SIM

The procedure below features a DS SIM that is in a ReactIR 15 base unit by design. Due to the embedded design of the SIM, a DS sampling technology connects directly to the base unit—there is no need to first connect a DS Optical Interface Module. As a result, you can connect a single DS sampling technology AgX FiberConduit probe or a DS Micro Flow Cell to the ReactIR 15 by the following steps:

1. At the connector end of the DS Series FiberConduit or DS Micro Flow Cell, use your thumb to slide the protective cap back to expose the optics.



with protective cap closed

with protective cap open

Figure 7-8 DS Series connector on FiberConduit

2. Insert the connector into the DS SIM at the front of the ReactIR 15 base unit. The protective cap on the DS SIM flips in as you push the FiberConduit connector into position.
3. Secure the DS Series AgX FiberConduit with the two captive screws in the vertical plane. (The second screw is not visible in the figure below.)

Figure 7-9 DS Series FiberConduit installed on a ReactIR 15

Switching Sampling Technologies

To change from one sampling technology to another using the bell-style connection to a standard Sample Interface Module (SIM), follow the steps in this section.

Note: Use a 25% energy compensation screen when the sampling technology is a Sentinel and K4 Conduit with MCT detector.

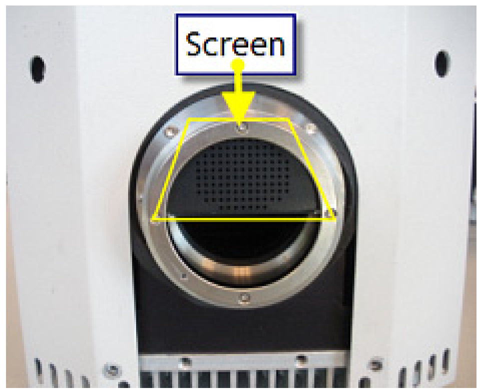


Figure 7-10 Energy compensation screen (in an iC10)

1. Disconnect sampling technology by loosening clamp that connects the conduit to the instrument.
2. If you are switching from one type a conduit to another, adjust the Gain potentiometer. Use a small flat head screwdriver and adjust the gain pot on the preamp either up or down until the peak height is between 20,000–25,000.

Table 7-1 Gain Pot Adjustments on Preamps

Switching FROM:	Switching TO:	Gain Pot Adjustment
K4 or K6	FiberConduit	**UP** (clockwise)
FiberConduit	K4 or K6	**DOWN** (counterclockwise)

Skip this step if you are switching from one type of FiberConduit to another or from one type of mirror conduit to another.
The upcoming figures show the location of the gain pot on each ReactIR base unit.

Adjusting Gain on a 45m

Figure 7-11 Gain pot adjustment on 45m

Adjusting Gain on an iC10

Below is the location on newer model iC10 base units.

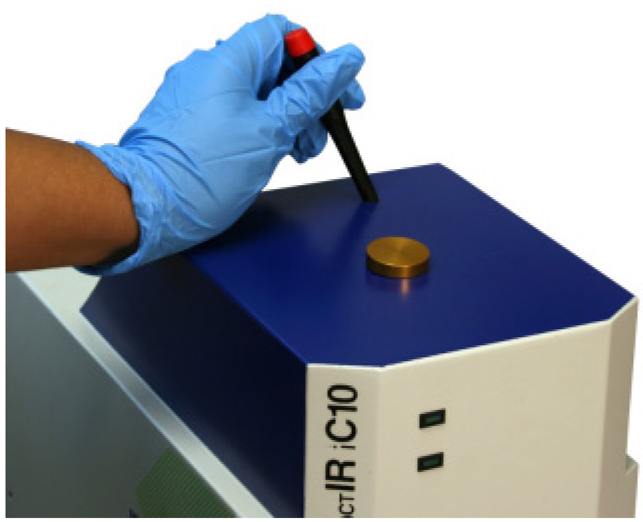


Figure 7-12 Gain pot adjustment on newer model iC10

Please note that on some older model ReactIR iC10s, it is necessary to remove the cover to the instrument to access the gain pot on the preamp.

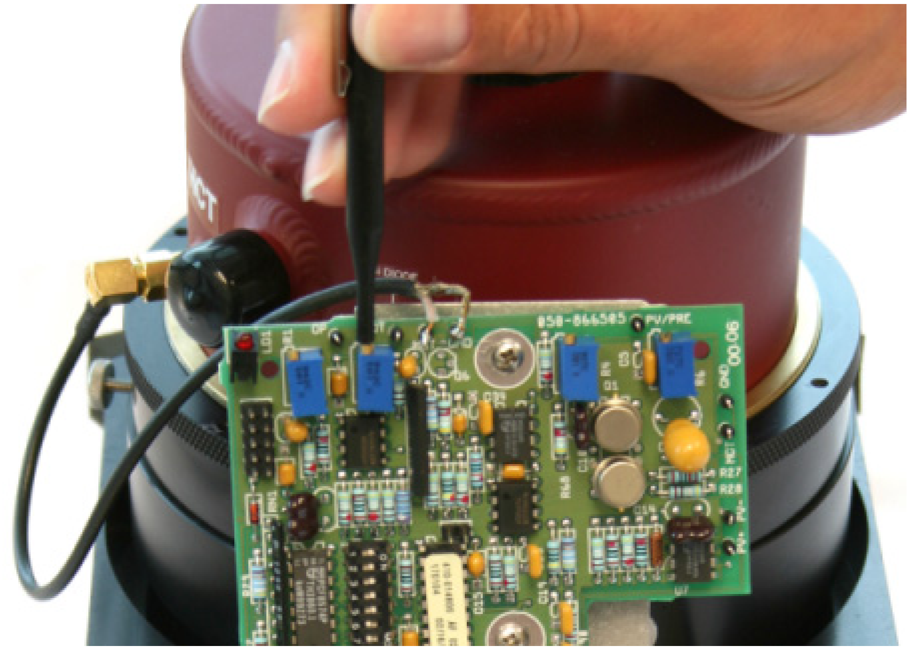


Figure 7-13 Gain pot adjustment on older model iC10 (with cover removed)

Adjusting Gain on a MonARC

Below is the location on the preamp board inside the MonARC enclosure.

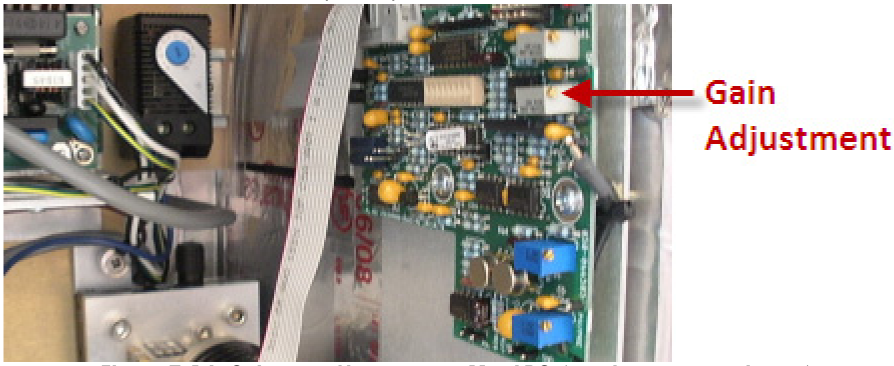


Figure 7-14 Gain pot adjustment on MonARC (requires open enclosure)

3. Reconnect new sampling technology to the SIM following the steps for the type of sampling technology being connected—Mirror or FiberConduit.

FiberConduit—Follow the direct-connect procedure or the procedure for a DS Series AgX fiber through a DS Interface Module. See "FiberConduits—Bell-Style or with DS Optical Interface Module" on page 67.

Mirror Conduit—See "Mirror Conduits—Direct Connection" on page 66.

8

Alignment

This chapter describes how to align the sampling technology for FiberConduit and Mirror conduits. It also includes instruction for aligning the detector in each ReactIR base unit.

Notes:
- DS Series FiberConduit probes are prealigned at the factory and require no rotational alignment, with one exception. DS Series FiberConduit-to-Sentinel configurations require alignment.
- iC IR software provides alignment features for ReactIR 45m and iC10 base units.
- The ReactIR 15 detector is factory aligned.
- MonARC embedded software (PIA) provides detector alignment features.

Alignment information and instructions are in the following sections:

Overview

ReactIR systems are factory aligned using the purchased sampling technology. When no sampling technology is ordered, a "gold standard" 6.35mm DiComp FiberConduit technology is used. As a result, it is expected that only minimal alignment will be required at installation and after periods of use. Alignment can be performed at two points in an instrument—at the sampling technology and in the detector that is inside the base unit.

Sampling Technology Alignment

ReactIR sampling technologies have either adjustable or fixed alignments. Adjustable sampling technologies can be aligned on site. Fixed sampling technologies are prealigned at the factory. DS Series integrated FiberConduit sampling technologies are fixed and therefore do not require alignment. The procedure varies depending on whether the sampling technology has a flexible FiberConduit or a mirror (articulated) conduit.

The upcoming table identifies the type of alignment based on the type of conduit for each sampling technology component.

Table 8-1 Sampling Technology Alignment

Component	Conduit Type	Type of Alignment
1. SIM		**FIXED**
2. Conduit	Mirror Conduit (K4 or K6)	**Mirror adjustment**
	FiberConduit—Bell-style	**Bell rotation**
	FiberConduit—DS Series	Not Applicable—Prealigned
3. Probe	Mirror Conduit (K4 or K6)	**Probe rotation**
	Fiber-to-Sentinel—Bell-style	**Sentinel rotation**
	Fiber-to-Sentinel—DS Series	**Sentinel rotation**
	FiberConduit/Integrated Probe	**FIXED probe**
4. Sensor		**FIXED**

Detector Alignment

ReactIR detector alignment is referred to as X-Y alignment. This chapter includes instructions on detector alignment for ReactIR iC10, 45m, and MonARC base units. MonARC detector alignment occurs through the embedded software (PIA) on the analyzer, and is also described in the *MonARC Software User Guide.*

The following table identifies the detector alignment procedure for each ReactIR base unit.

Table 8-2 Detector Alignment Procedure by Type of Base Unit

ReactIR Base Unit	Detector X-Y Alignment Procedure
MonARC	Performed at instrument touch screen (PIA embedded software)
iC10	Performed by adjusting screws on each side of base unit
45m	Performed through iC IR software (software-driven or auto-align)

Before You Begin

Before performing an alignment procedure, power up the system, check the current alignment, and make sure the probe is clean. (See "Cleaning the Probe" on page 95.)

Power Up ReactIR Base Unit

Ensure the system has been powered up for the minimum time required per base unit, as follows:

- **ReactIR 45m**—At least one (1) hour from a cold start. If the system has been powered up overnight or for more than one hour, proceed to start the iC IR software.

- **ReactIR iC10**—At least three (3) hours. If the system has been powered up overnight or for more than three hours, proceed to start the iC IR software.
- **MonARC**—At least three (3) hours. If the system has been powered up overnight or for more than three hours, proceed to the MonARC touch screen software.

Start the Software

iC IR software provides a wizard to guide you through the process to check alignment for the ReactIR 45m and iC10. The wizard includes a cleaning step. Ensure the software "Configure Instrument" procedure is set for the sampling technology being used.

MonARC PIA embedded software provides a detector alignment feature at the analyzer, through the PIA embedded software. The software automatically starts when you power up the MonARC base unit. Ensure the system settings and recipe for the sampling technology being used have been transferred to the instrument through the iC RCT software.

ReactIR Alignment Procedures

The following table identifies the type of alignment actions required for the ReactIR base units according to the type of sampling technology in use:

Table 8-3 ReactIR Sampling Technology Alignment Actions

	Sampling Technology	Alignment Actions			
		Rotate Conduit	Rotate Probe	Align Knuckles	Align Detector
A	**Mirror Conduit / Detachable Probe**	n/a	X	X	X
B	**FiberConduit/ Detachable Probe (Sentinel)**	X	X	n/a	X
C	**FiberConduit/ Integrated Probe**	X	n/a	n/a	X
D	**DS Series/ Fiber-to-Sentinel**	n/a	X	n/a	X
E	**DS Series/ Integrated Probe**	n/a	n/a	n/a	X

How to Align Your Sampling Technology

This section provides the steps to align sampling technology, based on the categories (A-E) listed in Table 8-3. Select your sampling technology and go to the designated page for step-by-step alignment instructions.

- "A. Mirror Conduit/Detachable Probe" on page 80
- "B. FiberConduit/Detachable Probe (Sentinel)" on page 81
- "C. FiberConduit/Integrated Probe" on page 82
- "D. DS Series/Fiber-to-Sentinel" on page 82
- "E. DS Series/Integrated Probe" on page 83

Instructions for detector alignment, based on the type of ReactIR base unit you have, begin on "How to Align Your Detector" on page 84.

Note: For your reference, Appendix B, "Single Beam Samples" shows sample single beams based on the type of sampling technology and the conduit length.

A. Mirror Conduit/Detachable Probe

The instructions below provide steps for aligning mirror (articulated) conduits on React IR base units. Before you begin, make sure the software is configured for the sampling technology in use.

1. Use the software to view the Peak Height and Contrast.

   iC IR software (ReactIR 45m and iC10)

   a. Click **Configure Instrument** on the Start Page (see Figure 8-9).
   b. Click **Next** through the wizard windows until the Align Probe window appears.
   c. Observe the Peak Height and Contrast.

   Embedded PIA software (MonARC)

   a. Press **Test** from the Select menu on the touch screen (see Figure 8-8).
   b. Press the **Align** tab along the left side of the main window.
   c. Press **Start**.

   During the alignment process, a background data file appears in the main window, and it is updated every second.

2. Rotate detachable probe (16mm or Sentinel) on the end of the conduit 180 degrees in each direction to maximize Contrast.
   Observe the resulting single beam that should closely match the shape of the single beam in Figure 8-1 for DiComp or SiComp.

3. Adjust knurled knobs on mirror conduit, one-at-a-time, while observing Peak Height and Contrast.

Adjust a quarter turn in one direction and then the other direction to determine whether Peak Height and Contrast increase. An increase in Peak Height without a change in Contrast is acceptable. Locate the adjustment point that yields maximum values.

4. Repeat the adjustment for the other knurled knobs (three total) with the same goal in mind.
5. If necessary, repeat the adjustment for the entire cycle until maximum Peak Height and Contrast are achieved.

Note: Small adjustments at each knob through several cycles will speed alignment and best maximize performance.

6. Proceed to "How to Align Your Detector" on page 84.

B. FiberConduit/Detachable Probe (Sentinel)

Below is the procedure for rotating the conduit at the bell-style end that connects to the base unit and the detachable probe (Sentinel) separately.

1. Attach the FiberConduit to the SIM on the front of the ReactIR 45m or iC10 base unit or on the side of the MonARC.
2. Use the software to view the Peak Height and Contrast.

   iC IR software (ReactIR 45m and iC10)

   a. Click **Configure Instrument** on the Start Page (see Figure 8-9).
   b. Click **Next** through the wizard windows until the Align Probe window appears.
   c. Observe the Peak Height and Contrast.

   Embedded PIA software (MonARC)

   a. Press **Test** from the Select menu on the touch screen (see Figure 8-8).
   b. Press the **Align** tab along the left side of the main window.
   c. Press **Start**.

      During the alignment process, a background data file appears in the main window, and it is updated every second.
3. Rotate the bell-style end until the purge connector is in the appropriate position based on probe diameter. Then, rotate further, if necessary, to maximize Contrast:
   - **9.5mm**—3:00 or 9:00 position
   - **6.35mm**—6:00 or 12:00 position
4. Slowly rotate the detachable probe (Sentinel) to maximize Contrast.

   Observe the resulting single beam that should closely match the shape of the single beam in Figure 8-1 for DiComp or SiComp.
5. Secure the optimal position with a clamp
6. Proceed to "How to Align Your Detector" on page 84.

C. FiberConduit/Integrated Probe

The procedures below apply to a ReactIR base unit that uses an integrated FiberConduit probe with a bell-style end that connects to the base unit.

Note: **DS Series** FiberConduit probes are prealigned and do **not** require rotational alignment except for the FiberConduit-to-Sentinel configuration (see "D. DS Series/Fiber-to-Sentinel" on page 82).

1. Attach the FiberConduit (6.35 or 9.5mm) to the SIM on the front of the ReactIR 45m or iC10 base unit or on the side of the MonARC.
2. Use the software to view the Peak Height and Contrast.

   iC IR software (ReactIR 45m and iC10)

   a. Click **Configure Instrument** on the Start Page (see Figure 8-9).

   b. Click **Next** through the wizard windows until the Align Probe window appears.

   c. Observe the Peak Height and Contrast.

   Embedded PIA software (MonARC)

   a. Press **Test** from the Select menu on the touch screen (see Figure 8-8).

   b. Press the **Align** tab along the left side of the main window.

   c. Press **Start**.

   During the alignment process, a background data file appears in the main window, and it is updated every second.
3. Rotate the bell-style end until the purge connector is in the appropriate position based on probe diameter. Then, rotate further, if necessary, to maximize Contrast:
   - **9.5mm**—3:00 or 9:00 position
   - **6.35mm**—6:00 or 12:00 position

   Observe the resulting single beam that should closely match the shape of the single beam in Figure 8-1 for DiComp or SiComp.
4. Proceed to "How to Align Your Detector" on page 84.

D. DS Series/Fiber-to-Sentinel

The following steps are only for the DS Series/Fiber-to-Sentinel sampling technology:

1. Attach the DS Series connector end to the base unit with the appropriate DS Series Optical Interface Module. ("DS Series Optical Interface Connection" on page 67)
2. Slowly rotate the detachable probe (Sentinel) to maximize Contrast.

   Observe the resulting single beam that should closely match the shape of the single beam in Figure 8-1 for DiComp or SiComp.
3. Secure the optimal position with a clamp
4. Proceed to "How to Align Your Detector" on page 84.

E. DS Series/Integrated Probe

1. Attach the DS Series connector end to the base unit with the appropriate DS Series Optical Interface Module.

- For iC10 and 45m:
  Refer to "DS Series Optical Interface Connection" on page 67 for information on the appropriate DS Optical Interface Module and "How to Connect a DS Series Interface Module to the SIM" on page 69 for instructions.
- For ReactIR 15:
  Refer to "How to Connect DS Series Sampling Technologies to a DS SIM" on page 72 for the ReactIR 15.

Note: DS Series FiberConduits are factory-aligned, so no rotational alignment is necessary for the sampling technology.

2. For ReactIR iC10 and 45m base units, proceed to "How to Align Your Detector" on page 84. No detector alignment is required for a ReactIR 15.

Single Beams for DiComp and SiComp

The following image shows the background spectrum for a DiComp and SiComp FiberConduit. The AgX fiber has an optical range from 2800 to 650 cm^{-1}, depending on fiber length. As the fiber length increases, the upper end cutoff reduces to lower wavenumbers.

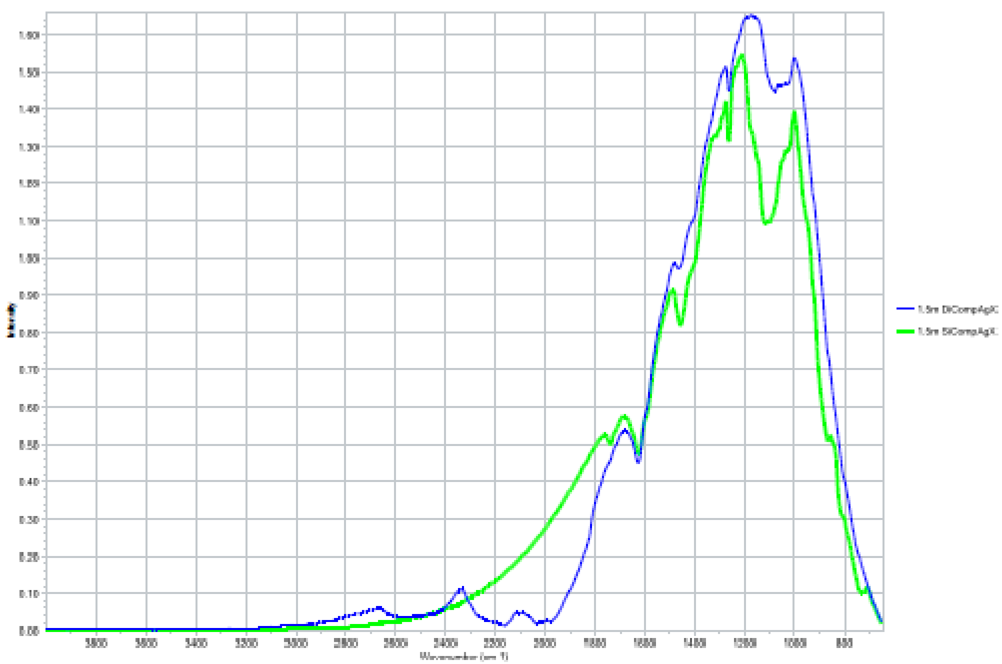


Figure 8-1 Single beam for DiComp (blue) and SiComp (green) 1.5m AgX FiberConduit

Refer to Appendix B, "Single Beam Samples" for additional examples.

How to Align Your Detector

Detector alignment affects the detector inside the ReactIR base unit. Alignment procedures vary by type of base unit. Instructions begin on the following pages:

- "Aligning a ReactIR 45m Detector" on page 86
- "Aligning a ReactIR iC10 Detector" on page 90
- "No Alignment Necessary for ReactIR 15 Detector" on page 90
- "Aligning a MonARC Detector" on page 90

Before You Begin

Prior to beginning any detector alignment, ensure the following preliminary conditions:

1. **MCT detector cooled**—If the ReactIR base unit configuration includes a mercuric cadmium telluride (MCT) detector, ensure the detector is cooled down and filled with liquid nitrogen before system power up and that the sampling technology components are mounted to the instrument

   Note: MonARC has an Stirling (SE MCT) detector—No liquid nitrogen is needed.

2. **Ensure the MCT detector is filled with liquid nitrogen**—Be sure the supply of liquid nitrogen is adequate following the period when the system was powered up and fill if needed.

Caution

Liquid nitrogen must be handled in accordance with applicable standards:

- Always wear a face shield, goggles, and insulating gloves when filling the base unit with liquid nitrogen.
- Use care in pouring liquid nitrogen into the ReactIR base unit. The sudden drop in temperature my fracture the funnel.
- Let the liquid nitrogen stabilize in the funnel and vent off. Do not overfill. Liquid nitrogen is explosive when not allowed to vent properly.

a. Fill the MCT cooling chamber with liquid nitrogen (N_2) until the chamber is full. The port is located on the top of the unit as shown in the photo below and described next.

Figure 8-2 Liquid nitrogen chamber

- **ReactIR 45m**—Remove the black plug from the top of the base unit and insert supplied funnel into the opening. Add several funnels full of liquid nitrogen.
- **ReactIR iC10 and 15**—Remove the gold cap from the top of the ReactIR base unit and insert supplied funnel into the opening.

b. Add several funnels full of liquid nitrogen. At some point, the liquid nitrogen may "blow back," creating a jet of nitrogen vapor and possibly liquid. After blowback subsides, add several more funnels full of liquid nitrogen.

c. Once the liquid nitrogen spills from the top of the detector, replace the black plug (45m) or gold cap (iC10 and 15).

Aligning a ReactIR 45m Detector

Detector alignment is performed through the iC IR software by a manual or automatic process. The Detector mirror motor on the ReactIR 45 can be adjusted manually, during a live experiment, by using directional buttons (**N**, **S**, **E**, **W**) buttons on the Advanced Tools panel of the Contrast and Align test or by an auto-align option.

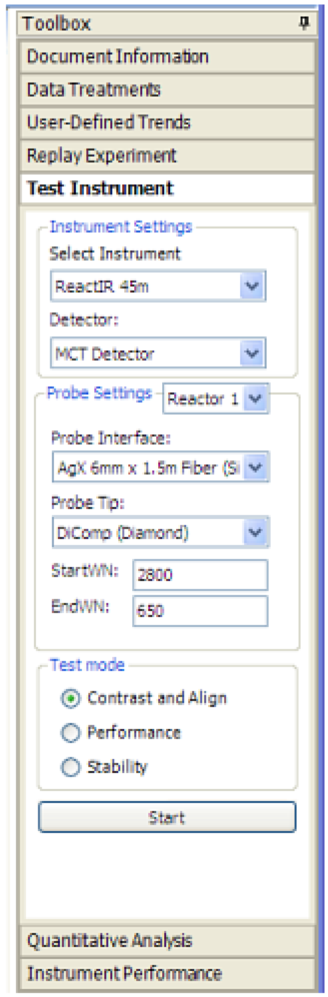


Figure 8-3 Contrast and Align test (Test Instrument task pane)

During a live experiment, access the Advanced tools panel through the Test Instrument task pane.

1. In the Test mode section, select Contrast and Align.
2. Click **Start**.
3. In the Test Instrument tab, double-click in the chart to display the Advanced Tools section.

4. Verify that the Motor Selection is set to Detector XY.
5. In the Alignment Option section, use the N, W, E, and S buttons to adjust the detector by the number of steps shown in the Steps text box.

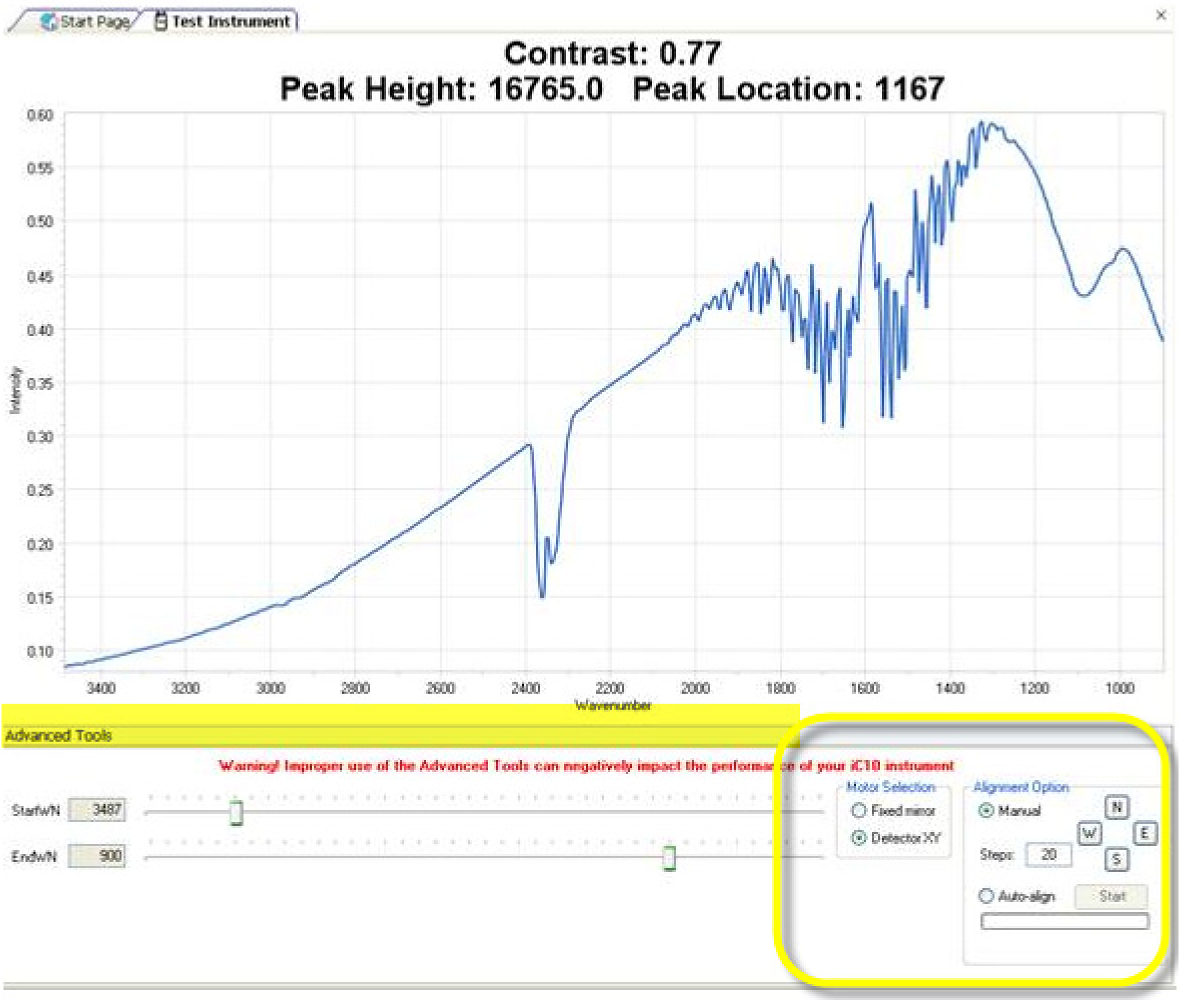


Figure 8-4 Test Instrument tab

Manual Adjustment Option

When manually aligning the instrument's detector, turn all adjustment points in concert with one another.

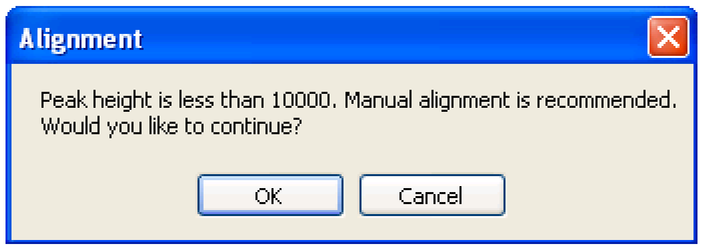
Note: Gain achieved in one step can affect gain achieved in other steps. If significant increases of signal and/or contrast are achieved during any step of the alignment procedure, it is important that the entire procedure be repeated to ensure maximum alignment.

Auto-Alignment Option

The Auto-align option is only used with the ReactIR 45m and it is only enabled if the Detector XY check box is checked in the Advanced Tools panel of the Contrast and Align test window.

1. In the Alignment Option section, select Auto-align.
2. Click **Start**.

Note: If the peak height is less than 10,000, a message prompts you to perform a manual alignment.



During the auto-align process, the progress bar displays a visual indication of the automatic alignment process. The system should find the best-aligned mirror position automatically within five minutes. You can abort the auto-align process at any time. If the process is aborted before completion, the iC IR software uses the best mirror position found up to the point the process ended.

Note: During the auto-align process, the iC IR does not allow user to select a different mirror or manually adjust the mirror positions.

Click the progress bar under the Auto-align check box to open the Auto-Align Information window that provides details about the alignment results.

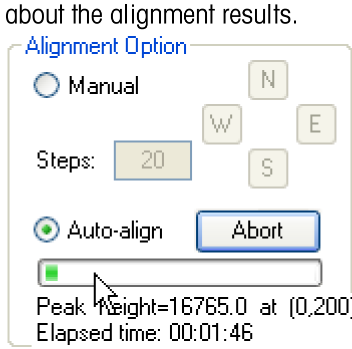


Figure 8-5 Auto-align option in progress

Figure 8-6 Auto-align information (ReactIR 45m)

Aligning a ReactIR iC10 Detector

A ReactIR iC10 has two alignment screws for the detector.

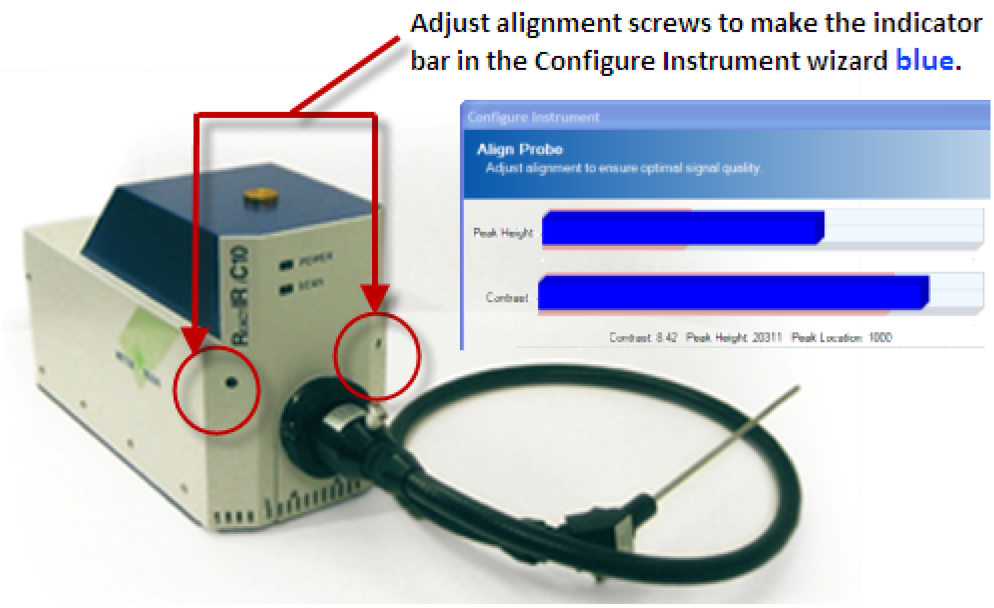


Figure 8-7 Fiber Alignment on an iC10

1. At the Start Page in iC IR, click **Configure Instrument** under Instrument Maintenance.
2. Select the instrument and click **Next** until the Align Probe window appears.
3. If the Peak Height or Contrast bars are red, adjust the alignment screws shown in Figure 8-7 until the bars turn blue.
   a. Insert a 1/8-inch ball driver into the adjustment screws on each side of the base unit.
   b. Rotate each adjustment screw gradually, forward and backward, to maximize both the Peak Height and the Contrast values.

No Alignment Necessary for ReactIR 15 Detector

The ReactIR 15 detector is factory aligned.

Aligning a MonARC Detector

1. Log on to the MonARC touch screen as a Manager and press the **Test** button from the Select accordion menu on the left side of the screen.
2. Press the Align tab along the left side of the main window.
3. Press the **Start** button along the bottom of the main window (The button name changes to Quit after you press Start).

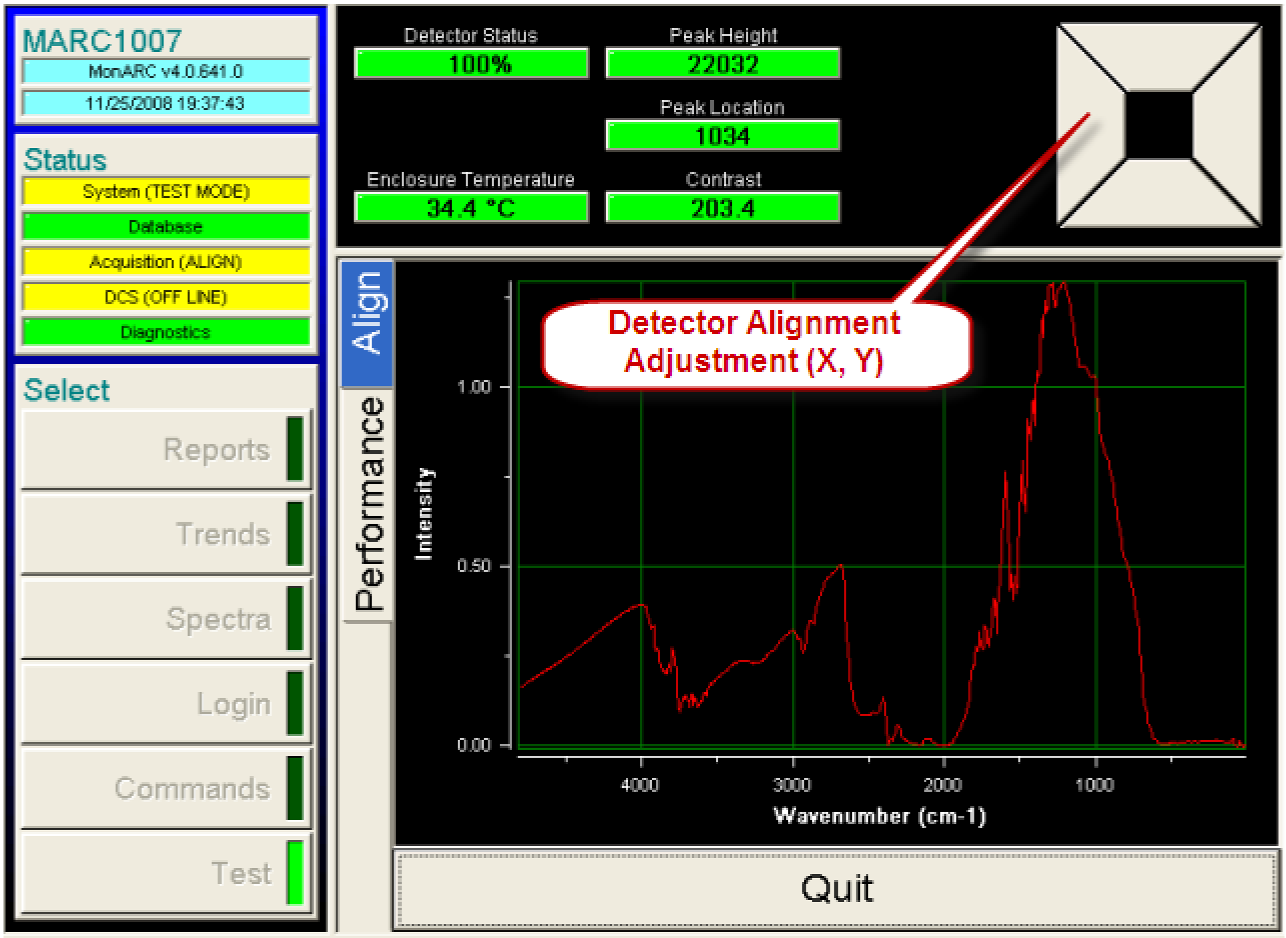


Figure 8-8 Alignment in MonARC (Align tab)

During the alignment process, a background data file appears in the main window, and it is updated every second.

4. Align the system detector by adjusting the X and Y buttons on the Align tab.
5. Press Quit when the parameters shown in the dashboard reach the targeted values.

Using Configure Instrument Feature in Alignment Procedure

This procedure applies to ReactIR 45m and iC10 base units. Use iC IR software to view the single beam during the alignment procedure.The procedure for using the software to align various sampling technologies and the detector is identical. Be sure the instrument configuration has been done (see Figure 8-9) to set the parameters to match the sampling technology being used.

Note: DS Series Integrated FiberConduits are prealigned at the factory and do not require alignment of the sampling technology (see page 82 for DS Series Fiber-to-Sentinel alignment procedures).

1. Before beginning the sampling technology or detector alignment, click **Configure Instrument** on iC IR Start page.

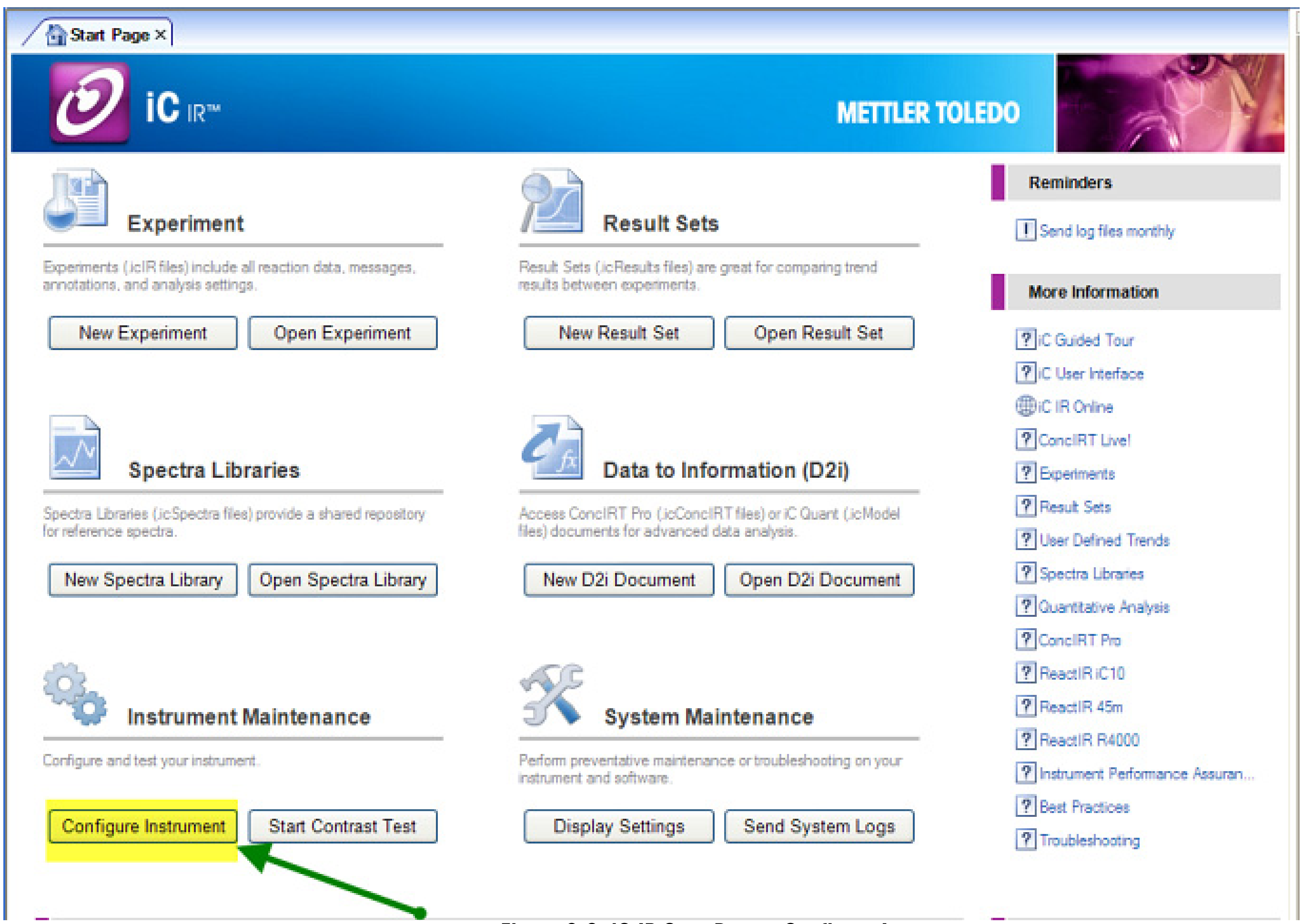


Figure 8-9 iC IR Start Page—Configure Instrument

2. Observe the single beam as you perform the alignment procedure.

Using the iC IR Software to Check and Align

The Align Probe wizard in iC IR software helps you physically align the probe (sampling technology) on ReactIR 45m, iC10, and 15 base units as you begin an experiment. When properly aligned, the probe readings of Peak Height and Contrast display as green bars:

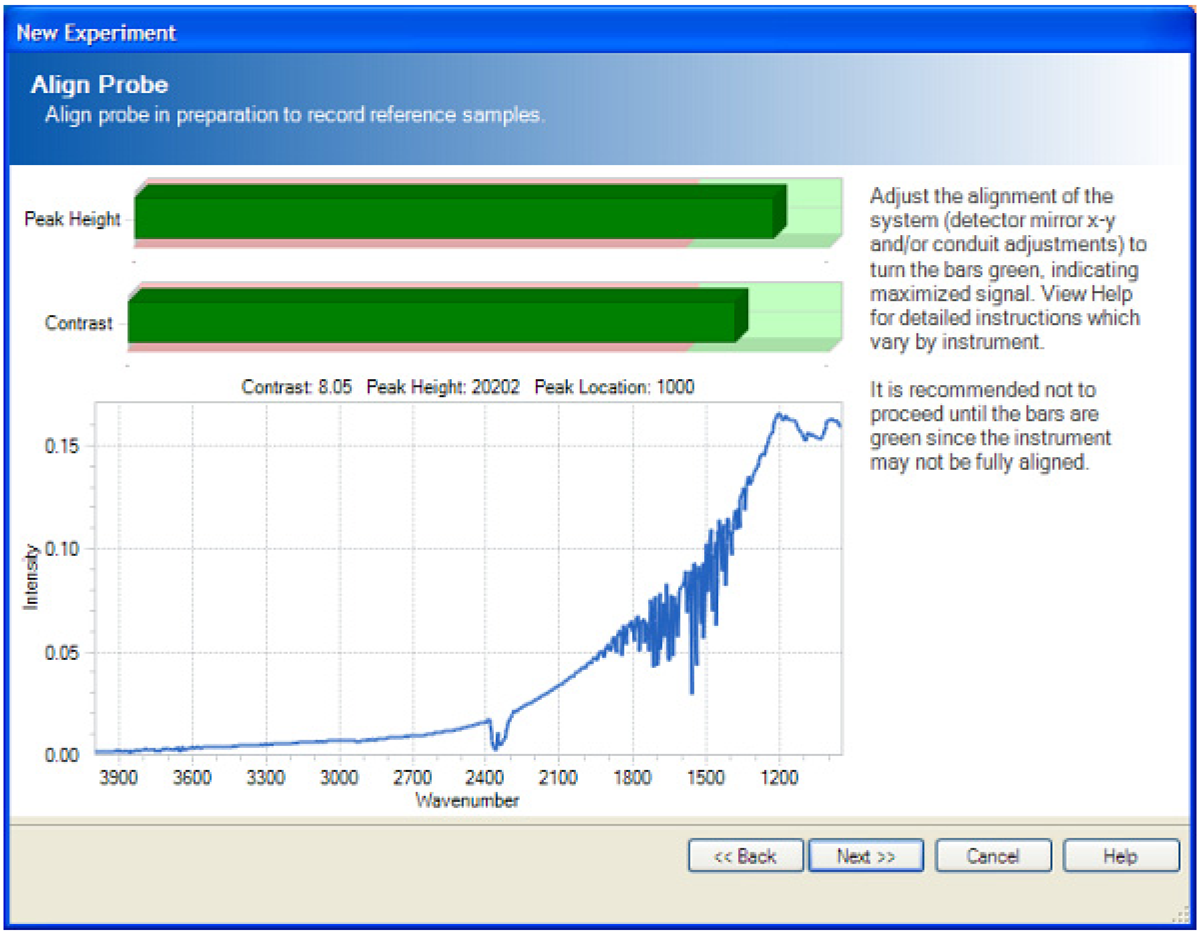


Figure 8-10 Probe alignment in iC IR software (New Experiment)

If the bars are not green, adjust the alignment of the detector (x-y adjustment and/or the sampling technology) to achieve the optimal Peak Height and Contrast.

Alignment procedures vary depending on the ReactIR base unit and whether the conduit is fiber or mirror. Follow the instructions for the specific sampling technology described under "How to Align Your Sampling Technology" on page 80. Alignment procedures are described under "How to Align Your Detector" on page 84) and in the online Help, and vary by ReactIR base unit.

Note: If you select **Configure Instrument** from Instrument Maintenance section on the Start Page, the "blue" bars indicate the alignment is in the acceptable range. (If you are using multiple probes on a ReactIR 45m, the alignment considers the separate Gain settings that can be applied to each probe in the Configuration—Probe acquisition settings). This takes into consideration the slight variation that can exist between the two probes.)

Figure 8-11 shows the alignment window in the Configure Instrument wizard (see "Using Configure Instrument Feature in Alignment Procedure" on page 92):

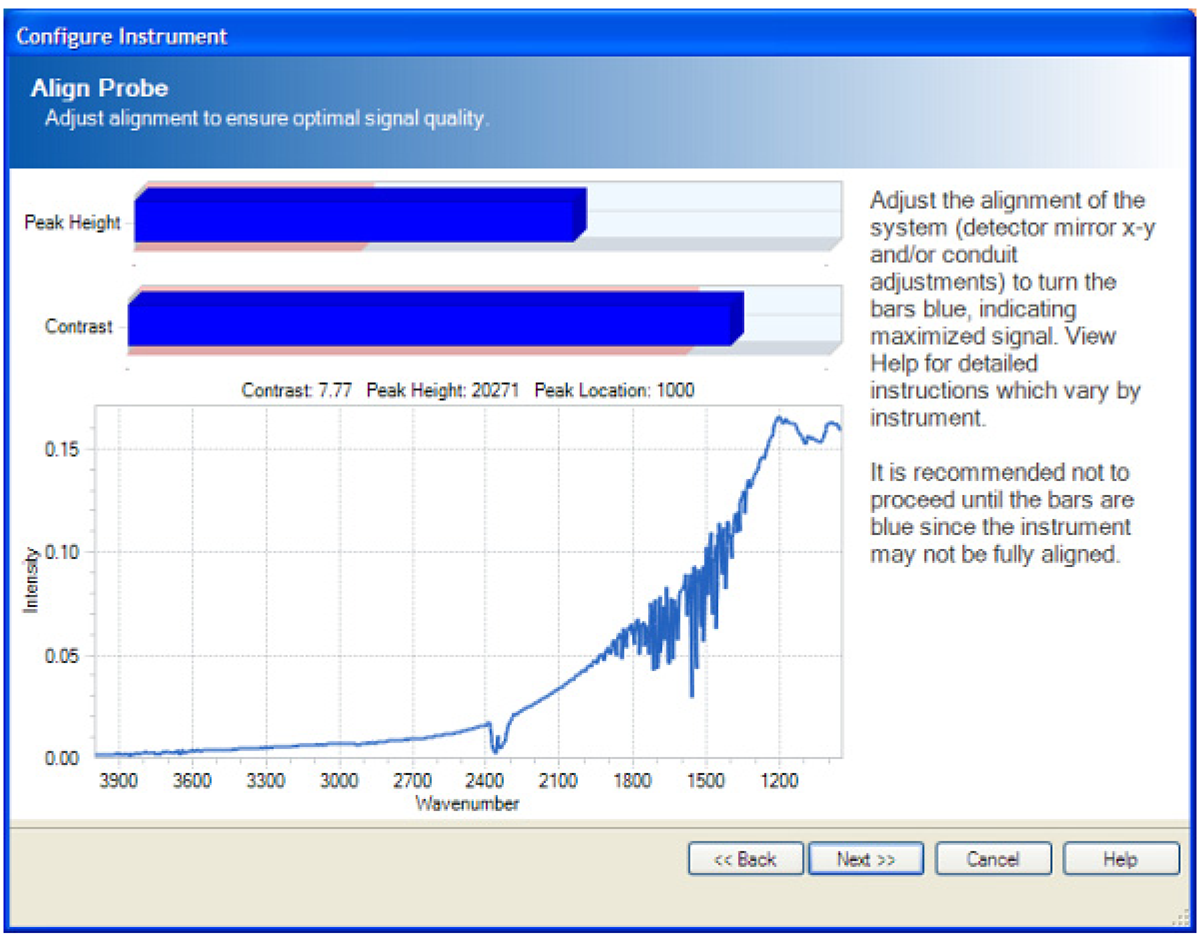


Figure 8-11 Probe alignment in iC IR software (Configure Instrument)

9

Care and Use

Ongoing care and use of your sampling technology involves alignment as described in Chapter 8, "Alignment." This chapter provides additional care and use information. (For DS Micro Flow Cells, please refer to Chapter 6, "Connecting and Using DS Micro Flow Cells".)

Handling a ReactIR Base Unit

These instructions apply only to the ReactIR 45m and iC 10 base units. For MonARC systems, refer to the Care and Maintenance chapter of the *MonARC Hardware Manual*.

Caution
The system can be awkward when handling, so follow safety guidelines.

- When moving the ReactIR™ iC10 or 45m system, it is highly recommended to use a cart or similar type device. The system can be hand-carried by using the integrated handle on the rear of the system along with the front sampling attachment port.
- DO NOT hand carry the system with the fiber optic or the mirror conduit attached to the system. Remove the conduit first before moving the unit.
- ONLY move the system with the conduit attached by using a cart or similar device. This will prevent any possible damage to the unit or personnel.
- ONLY lift the system using BOTH the handle in the rear and the sampling port on the front of the unit. Lifting the unit in other manners may cause damage to the system or personal injury should the cover unexpectedly become detached.

For information about the sampling technology, refer to "Handling a FiberConduit" on page 29.

Cleaning the Probe

Two methods exist for cleaning a probe—physical and optical.

Step 1—Physical Cleaning

The probe can be cleaned using the following procedure:

1. Rinse the sensor and probe body with the solvent from the previous reaction mixture or a solvent that will dissolve any residue.
2. Wipe the sensor with a lint-free towel to dry.

3. Visually inspect the sensor. If a residue is present repeat the above two steps until no residue is observed.

Step 2—Optical Cleaning

Follow the procedure below to determine the optical cleanliness of the sensor.

Note: Refer to the *iC IR User Guide* (software manual) for instructions on how to collect spectra. For a MonARC, refer to the *MonARC 4.x User Guide*.

1. Collect a Background spectrum (single beam).
2. Clean the sensor with the appropriate solvent.
3. Collect a sample spectrum (single beam) and observe that there are no peaks due to a residue from the reaction or from the solvent.

   Note: In the spectrum, any peaks that show negative absorbance indicate you have removed material (reaction components) from the sensor. Conversely, peaks that show positive absorbance indicate you added material on the sensor from the cleaning (for example unevaporated solvent or residue from impure solvent).

4. If peaks are present in the single beam spectrum, repeat the above steps until you observe no peaks.

   Note: Generally one or two cleaning cycles will produce a sample spectrum that shows a flat baseline across the spectral region, indicating no discernible reaction component peaks.

Warning:

- **For the AgX FiberConduit**, DO NOT try to clean the lenses that are located in the bell-shaped adapter (shown in Figure 3-9 on page 27). Attempting to clean these will result in surface scratches that will degrade the fiber performance.
- **For the DS Series AgX FiberConduit**, DO NOT try to clean the optics housed in the DS Series FiberConduit AND DO NOT try to clean any of the optical components housed inside the single or MultiplexIR (MUX) Optical Interface Modules. Attempts to clean these delicate optics will result in damage and lowered performance.

Attempts to clean lenses and optics **void the warranty** of the device.

Storing a Sampling Technology

When the experiment using the ReactIR sampling technology is complete, and will be changed to another configuration or stored for any reason, follow the steps below:

1. SLOWLY remove the probe from the reactor.

> **Note:** Take care not to strike the probe body or the fiber sheathing sharply on any hard surfaces. Shock due to striking the probe or sheathing may cause damage to the fiber

2. Physically clean the probe ("Step 1—Physical Cleaning" on page 95).
3. Place the provided covers on each end of the FiberConduit. The DS Series FiberConduit connector has an "integrated" cover to protect the optics.
4. Place the FiberConduit in the storage box (provided at the time it was delivered) when not in use, or place in a suitable location where the FiberConduit sampling technology is safe and secure.

10

Putting It All Together

This chapter combines the ReactIR sampling technology component information into a summarized table for ease of reference. Appendix A on page 101 provides tables of specifications on the individual sampling technology components. Appendix B on page 109 shows sample single beams from specific sampling technology configurations.

Sampling Technology Options for ReactIR Base Units

The following table describes the options for ReactIR iC10 and 45m base units:

Table 10-4 ReactIR 45m, iC10, and MonARC Sampling Technology Options

Probe Diameter		**6.35mm**		**9.5mm**		**16mm**	**25mm**	
		FiberConduit *	DS Series	FiberConduit *	DS Series	K6	K4, K7	FiberConduit, DS Series
Probe Length		305mm 457mm	203	305mm 457mm	305mm	178mm 299mm 362mm	29mm	29mm
Probe Sensor		Diamond	Diamond Silicon	Diamond Silicon	Diamond Silicon	Diamond, Silicon Cubic Zirconium	Diamond, Silicon Cubic Zirconium	Diamond, Silicon Cubic Zirconium
Fiber Length		1m 2m	1.5m	1.5m	1.5m 2m	N/A	N/A	1m 2m
Temperature		-40°C to 120°C	-80°C to 180°C	-80°C to 180°C	-80°C to 180°C	-80°C to 200°C	-80°C to 350°C ***	-80°C to 350°C ***
Pressure (Max)		34bar (500psi)	69bar (1000psi)	69bar (1000psi)	69bar (1000psi)	7bar (100psi)	310bar (4500psi) ***	310bar (4500psi) ***
Infrared Range ** (cm^{-1})	1m 1.5m	2800-650	2800-650	2800-650	2800-650	4000-650	4000-650	2800-650
	2m	1950-650	1950-650	1950-650	1950-650			1950-650

* Green columns indicate discontinued product.
** Infrared range differs based on fiber length.
***Check your Sentinel specifications for the applicable temperature and pressure.
NOTE: Diamond absorbs between 2300cm^{-1} to 1950cm^{-1}

Sampling Technology Options for ReactIR 15

ReactIR 15 base units use DS Sampling Technologies exclusively.

Table 10-5 ReactIR 15 Sampling Technology Options

Probe Diameter		6.35mm	9.5mm	25mm
		DS Series	DS Series	DS Series
Probe Length		203	305mm	29mm
Probe Sensor		Diamond Silicon	Diamond Silicon	Diamond, Silicon Cubic Zirconium
Fiber Length		1.5m	1.5m 2m	1m 2m
Temperature		-80°C to 180°C	-80°C to 180°C	-80°C to 350°C **
Pressure (Max)		69bar (1000psi)	69bar (1000psi)	310bar (4500psi) **
Infrared Range * (cm^{-1})	1m 1.5m	2800-650	2800-650	2800-650
	2m	1950-650	1950-650	1950-650

* Infrared range differs based on fiber length.

**Check your Sentinel specifications for the applicable temperature and pressure.

NOTE: Diamond absorbs between 2300cm^{-1} to 1950cm^{-1}

A

Sampling Technology Component Tables

This appendix describes the standard sampling technology configuration options in table format for ease of reference. Configurations other than those listed here are considered "specials."

- "Probe—Conduit Availability" on page 101
- "Probe Length—Insertion Depth" on page 103
- "Probe—Temperature and Pressure" on page 105
- "Conduits" on page 106
- "Sensors and Seals" on page 107

See Appendix B, "Single Beam Samples" to see the background spectrum for each sampling technology.

Probe—Conduit Availability

The table below features the probe diameter options available with specific types of conduits.

Table A-6 Sampling Technology Probe/Conduit Availability

Probe Diameter	Conduit	Conduit Length		Standard Availability *
		meters	inches	
6.35mm	K4			
	K6			
	K7			
	FiberConduit (bell-style)	1.0		X
		1.5		X
		2.0		X
	DS Series	1.5		X

Table A-6 Sampling Technology Probe/Conduit Availability (continued)

Probe Diameter	Conduit	Conduit Length		Standard Availability *
		meters	inches	
9.5mm	K4			
	K6			
	K7			
	FiberConduit (bell-style)	1.5		X
		2.0		X
	DS Series	1.5		X
		2.0		X
16mm	K4			
	K6		43	X
	K7			
	FiberConduit (bell-style)			
	DS Series			
25mm (Sentinel™)	K4		17	X
	K6			
	K7		53	X
	FiberConduit (bell-style)	1.0		X
		1.5		X
		2.0		X
	DS Series	1.0		X
		2.0		X

* Special orders available

Probe Length—Insertion Depth

Table A-7 shows the length options available for each probe diameter and conduit style.

Table A-7 Sampling Technology Probe Length

Probe Diameter	Conduit	Probe Length *		Insertion Depth * (Wetted Length)	
		inches	mm	inches	mm
6.35mm	FiberConduit (bell-style)	12	305	Same as Probe Length	
		18	457		
	DS Series	8	203		
9.5mm	FiberConduit (bell-style)	12	305		
		18	457		
	DS Series	12	305		
		18	457		
16mm	K6	10	254	7	178
		15	381	12	305
		17	432	14	362
25mm (Sentinel)	K4	Same as Insertion Length		1	25
	K7				
	FiberConduit (bell-style)				
	DS Series				

* Measurements are rounded for ease of reference.

FiberConduit Probe Lengths

Probe length for FiberConduit sampling technologies is the distance from the probe tip to the threaded end of an adapter fitting, or in the case of DS-Series probes, the nose piece.

Below is one example of the probe length options for a FiberConduit probe.

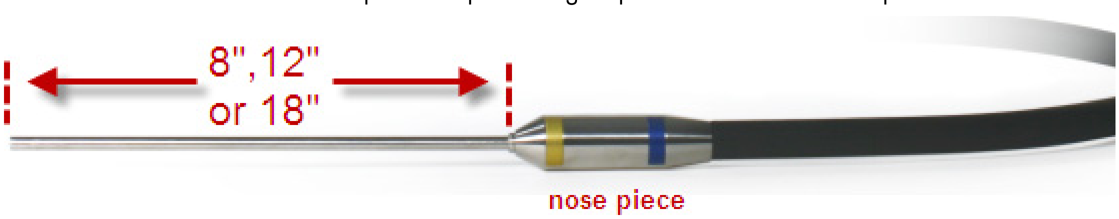


Figure A-12 Length options (9.5mm DS Series FiberConduit probe)

Mirror Conduit Probe Lengths

Probe length for Mirror conduit sampling technologies is expressed as the "wetted" length because allowance has been made for the portion of the probe reserved for your adapter fittings.

The example in Figure A-13 shows of the length options for a 16mm probe that is intended for use with a Mirror conduit.

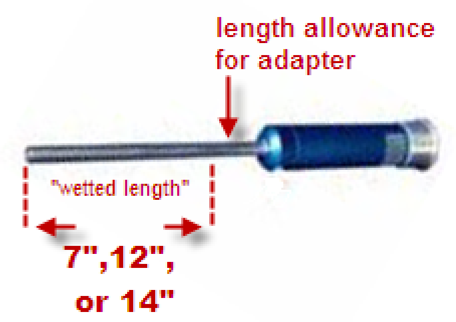


Figure A-13 Length options (16mm probe for Mirror conduit)

Probe—Temperature and Pressure

The following table provides details about the temperature, pressure, and PH properties of each probe. Probe versions for high pressure and high temperature, highlighted below, are available upon request.

Table A-8 Probe Temperature/Pressure Properties

Probe Diameter	Conduit	Temperature Range **	Pressure * (Vacuum/Upper Limit)			
			7 Bar/ 100 psi	34 Bar/ 500 psi	69 Bar/ 1000 psi	107 Bar/ 1500 psi
						High Pressure Model
6.35mm	FiberConduit	-40° to 120°C		X		
	DS Series	-80° to 180°C			X	
9.5mm	FiberConduit	-40° to 120°C		X		
	DS Series	-80° to 180°C			X	
16mm	K6	-80° to 200°C	X			X
25mm (Sentinel)	K4	-80° to 200°C	X			X
	K7		X			X
	FiberConduit		X			X
	DS Series		X			X

* Vacuum (10 Torr) to upper limit
Pressures apply to all sensors—DiComp, SiComp, and ZrComp.
Specials available up to 4500psi.
** Specials available up to 350°C

The sensor material affects the chemical properties (PH, hardness, and refractive index) of the probe as described in "Sensors and Seals" on page 107.

Conduits

The following table provides details about the variety of conduits available. An integrated probe refers to a conduit and probe that are together and do not require a clamp connection.

Table A-9 Conduit Properties

Conduits	Probe Connection	Length		Type of Transfer Optics	Optical Range
		Meters	Inches		cm^{-1}
K4	Detachable	0.4	17	mirror	**4000–650**
K6		1.1	43		
K7		1.3	53		
FiberConduit (bell-style)	Integrated	1.5	59	fiber	**2800–650**
		2.0	79		**1950–650**
	Detachable	1.0	39		**2800–650**
		1.5	59		**2800–650**
		2.0	79		**1950–650**
DS Series	Integrated (6.35 and 9.5)	1.5	59	fiber	**2800–650**
	(9.5 only)	2.0	79		**1950–650**
	Detachable	1.0	39		**2800–650**
		2.0	79		**1950–650**

Sensors and Seals

The following table provides details about the variety of sensors available, with the chemical properties and the optical range (spectral wavenumber–cm^{-1}).

Note: Probes are constructed of Alloy C276 as standard. Other materials are available by special order.

Table A-10 Sensor Properties

Type of Sensor	pH Range	Optical Range	Hardness (Knoop)	Refractive Index	Seal *
Diamond	1-14	**4000–2200 cm^{-1}** **1950–650 cm^{-1}**	Extremely robust (7000)	**2.4**	Gold
Silicon	1-10 **	**4000–650 cm^{-1}**	Sensitive to abrasion–caution when cleaning (1150)	**3.4**	Gold
Cubic Zirconium	1-14 **	**4000–1450 cm^{-1}**	Robust (1250)	**2.15**	Gold

* Teflon (PTFE) is available as a special order.
** Sensor chips are available for testing sensor/chemistry compatibility.

Refer to Appendix B, "Single Beam Samples" to see the background spectrum for each sampling technology.

B

Single Beam Samples

This appendix provides samples of typical single beam fingerprints generated by the sampling technologies in the ReactIR instruments—iC10, 15, 45m, and MonARC. Slight variations will occur. These samples can also be referred to as "background" spectra. Refer to the single beams to assist you in selecting the proper probe—sensor combination and in identifying the fingerprint of a proper alignment.

Systems—All

Probes—6.3, 9.5, 16, 25

Conduit—DS Series, FiberConduit, K6, K4, K7

Sensor—DiComp, SiComp, ZrComp

Notes: An asterisk (*) next to a conduit type indicates a discontinued sampling technology offering. For example, DS Series FiberConduits have replaced the standard FiberConduit sampling technologies.

Single beam samples are grouped by ReactIR instrument, as follows:

- "Single Beams—6.3mm Probes" on page 110
- "Single Beams—9.5mm Probes" on page 111
- "Single Beams—16mm Probes" on page 113
- "Single Beams—25mm Probes (Sentinel)" on page 114
- "Single Beams—DS Micro Flow Cells" on page 117

Single Beams—6.3mm Probes

Notice that the conduit length affects the single beam. For example, the samples below show a DiComp 2m and a SiComp 1.5mm probe and there is greater throughput (intensity) with a shorter conduit. Also notice that the sensor type affects the single beam. For example, compare the DiComp 1.5 with the SiComp 1.5 in the following table.

Table B-1 Single Beams Samples—6.3mm Probes

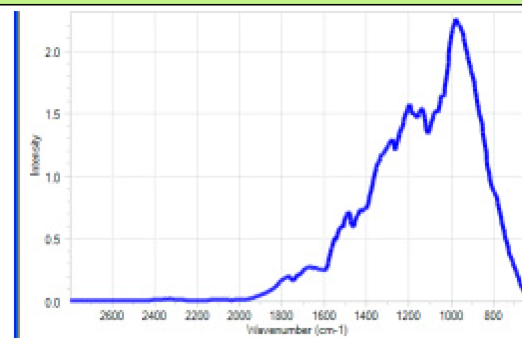
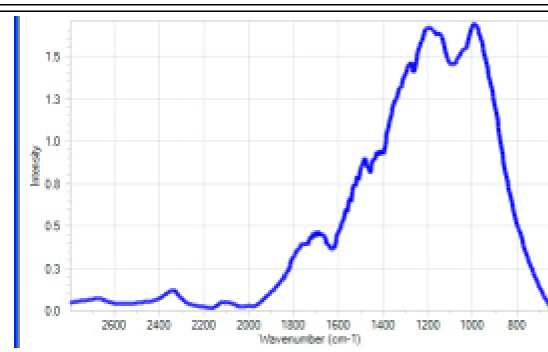
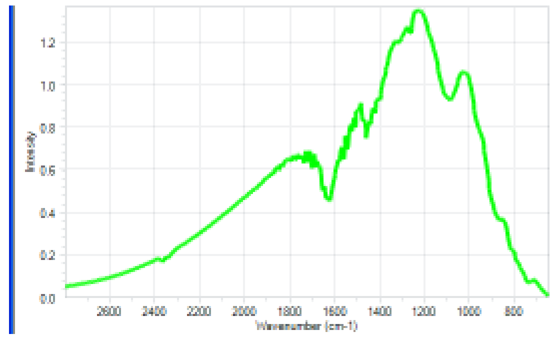
Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
			
FiberConduit *	DiComp	2	
	SiComp		
	ZrComp		
DS Series	DiComp	1.5	

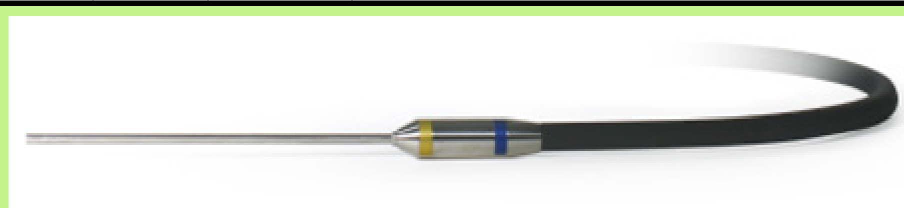
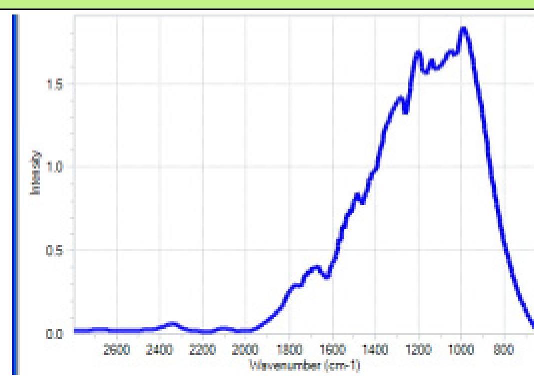
Table B-1 Single Beams Samples—6.3mm Probes (continued)

Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
	SiComp	1.5	
	ZrComp		

Single Beams—9.5mm Probes

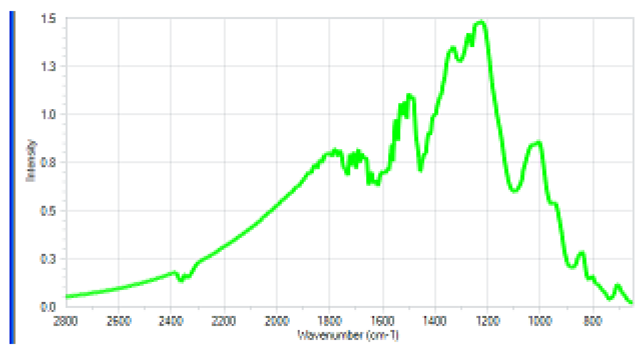
Notice that the conduit length affects the single beam. For example, the samples below show a DiComp 2m and a DiComp1.5m probe and there is greater throughput (intensity) with a shorter conduit. Also notice that the sensor type affects the single beam. For example, compare the DiComp 2m with the SiComp 1.5m in the following table:

Table B-2 Single Beams Samples—9.5mm Probes

Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
			
FiberConduit *	DiComp	2	

Single Beams—9.5mm Probes

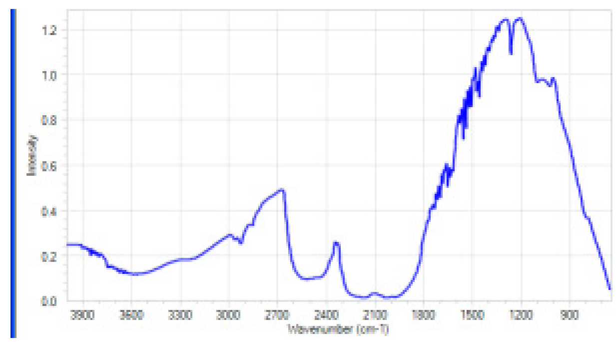
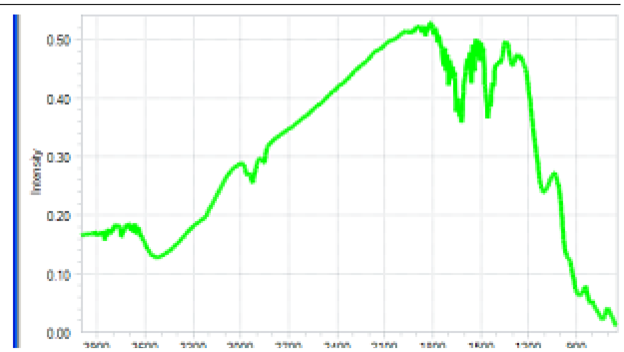
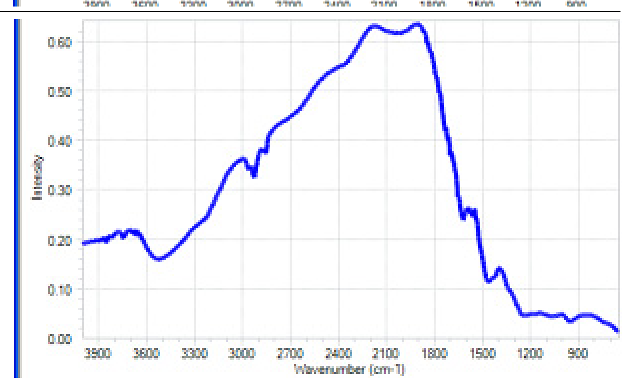
Table B-2 Single Beams Samples—9.5mm Probes

Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
	SiComp		
	ZrComp		
DS Series	DiComp	1.5	
	SiComp	1.5	
	ZrComp		

Single Beams—16mm Probes

Notice the single beam changes with different sensor types. Conduit length does not apply.

Table B-3 Single Beams Samples—16mm Probes

Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
			
K6	DiComp		
	SiComp		
	ZrComp		

Single Beams—25mm Probes (Sentinel)

Table B-4 Single Beams Samples—25mm Probes

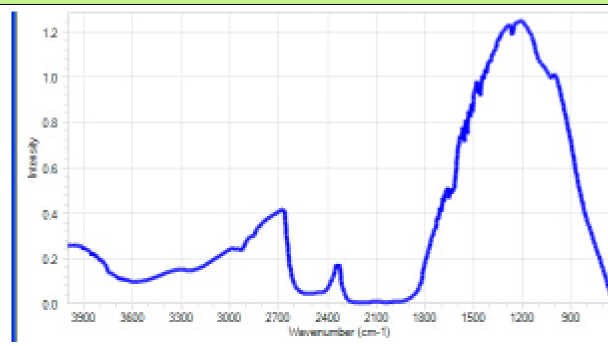
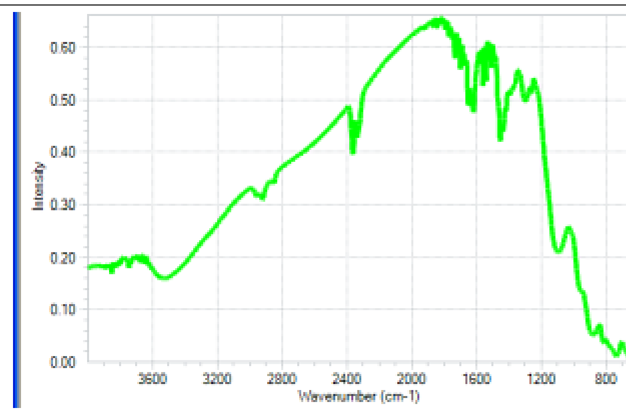
Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
			
K4	DiComp		
	SiComp		
	ZrComp		
K7	DiComp		
	SiComp		
	ZrComp		

Table B-4 Single Beams Samples—25mm Probes (continued)

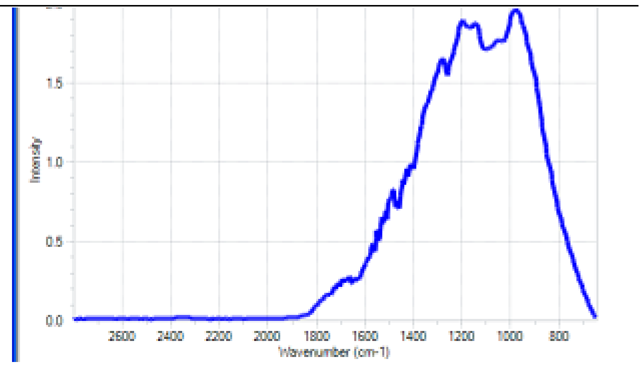
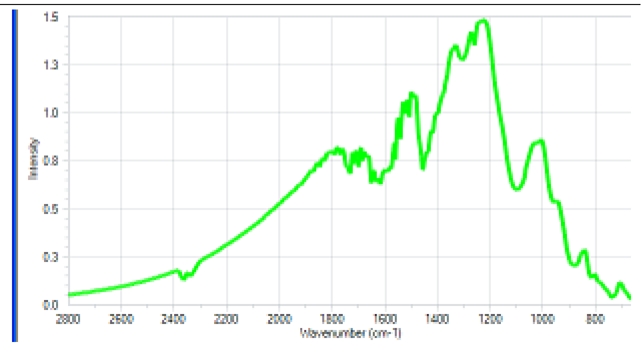
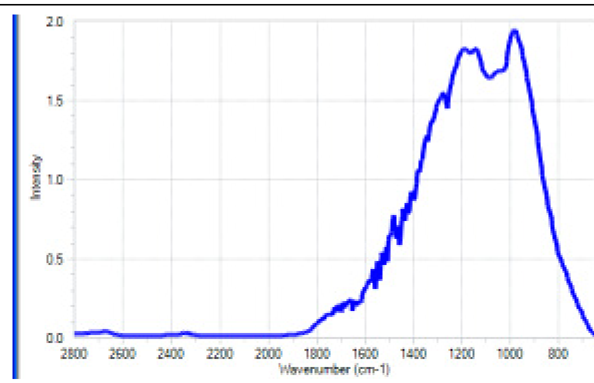
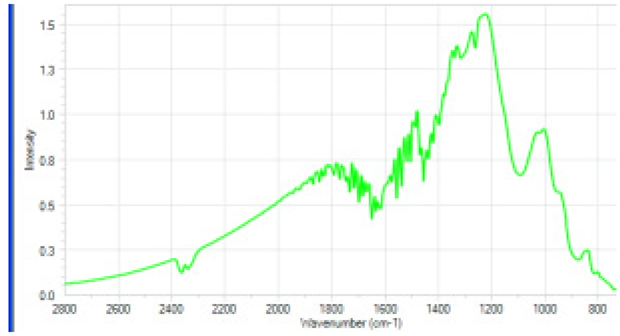
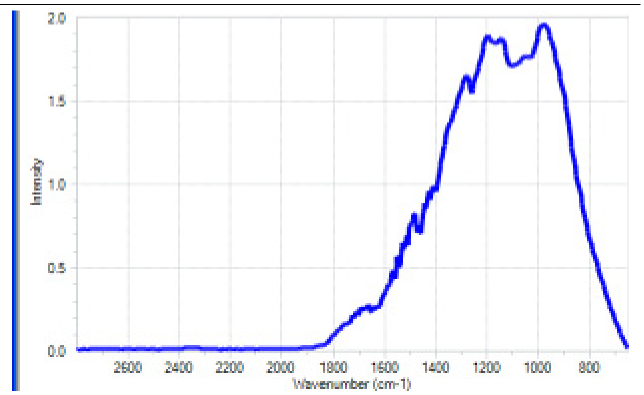
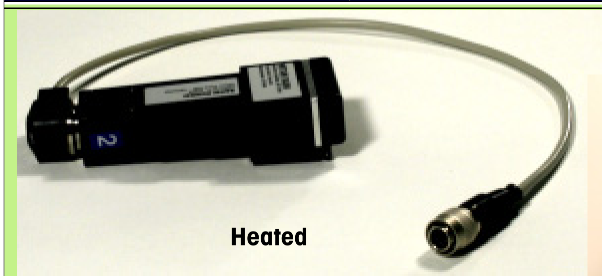
Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
FiberConduit *	DiComp	2	
	SiComp	2	
	ZrComp		
DS Series	DiComp	1	

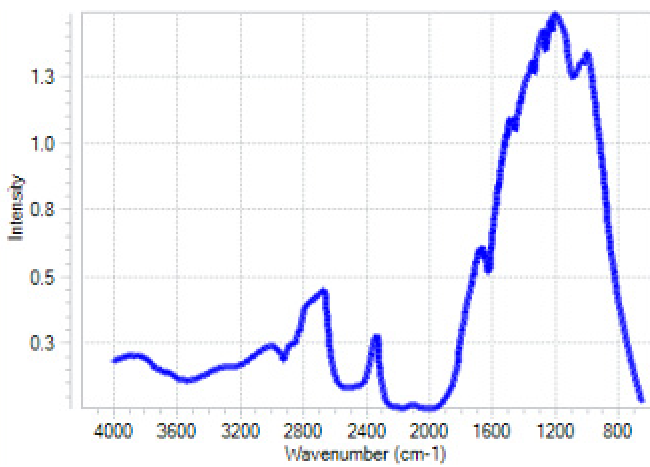
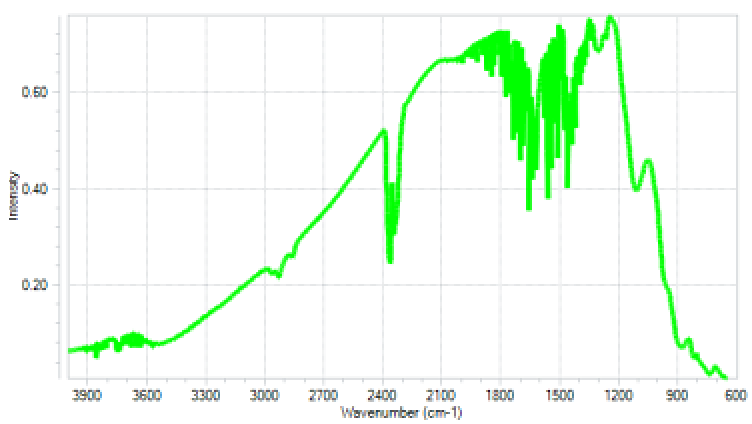
Table B-4 Single Beams Samples—25mm Probes (continued)

Conduit	Sensor	Conduit Length (m)	Single Beam (Background)
DS Series (continued)	SiComp	1	
	ZrComp		
	DiComp	2	
	SiComp	2	
	ZrComp		

Single Beams—DS Micro Flow Cells

Table B-5 Single Beams Samples—DS Micro Flow Cell

<table>
<tr><th>Sensor</th><th>Single Beam (Background)</th></tr>
<tr><td colspan="2">


</td></tr>
<tr><td>DiComp</td><td>
</td></tr>
<tr><td>SiComp</td><td>
</td></tr>
</table>

Index