KEIT INDUSTRIAL ANALYTICS

IRmadillo™

DOC1049R IRmadilloNA User Manual - Hardware

Model Number: ASM0627-09-N-Cx-O-Gx-D3x

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This document should be read in conjunction with DOC1048R IRmadilloNA User Manual – Safety.

1.1. Purging the IRmadilloNA

The IRmadilloNA spectrometer is designed to be continuously purged. This eliminates any interference in operation caused by changing levels of water vapour and carbon dioxide (CO_2) within the instrument, both of which interact strongly with infrared (IR) light.

The IRmadilloNA is fitted with purge inlets on the probe and spectrometer body. The Inlets are shipped with blanking plugs that need to be removed before use. There are breathers on both the probe and spectrometer body.

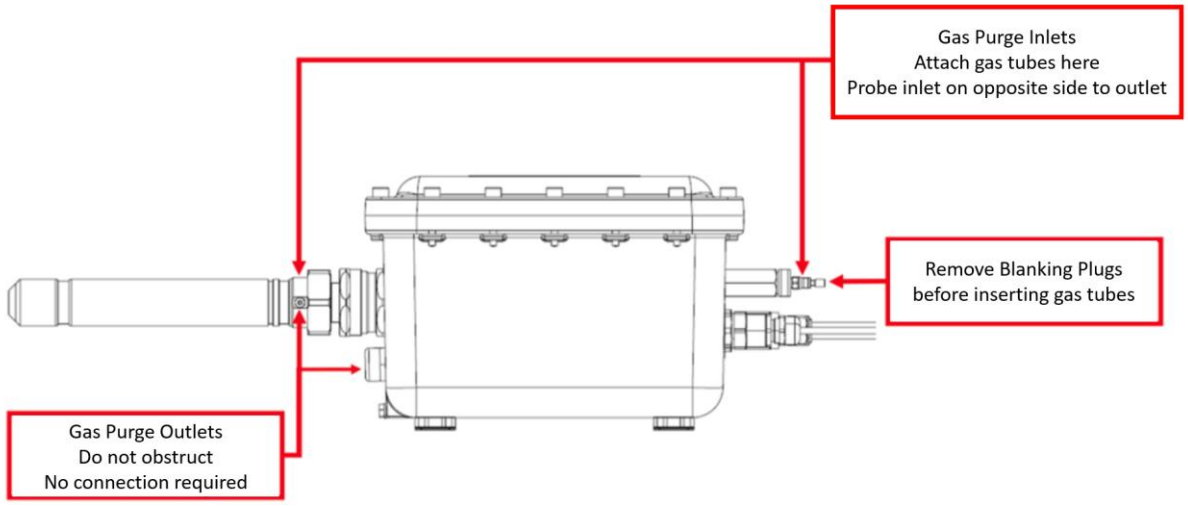

Figure 1: Purge inlet and outlet locations on ASM0627-09

The ideal purge gas is dry nitrogen. Alternatively, dry air from a purge gas generator can be used. The output from this purge gas generator should have a minimum quality according to ISO8573-1:2010 of Class 1:2:1 (dust:water:oil) or better. Keit can recommend suitable generators that achieve this outlet air quality; contact support@keit.co.uk. Purge gas generators may include an internal compressor or use factory-supplied compressed air with a minimum quality of Class 4:4:4 (dust:water:oil).

⚠ **NOTE** REMARQUE	**All FTIR spectrometers are very sensitive to water vapour and carbon dioxide. Even in well-controlled environments, e.g., laboratories, environmental changes can be large enough to cause significant changes in the spectrum. We STRONGLY recommend purging the instrument at all times.** Tous les spectromètres FTIR sont très sensibles à la vapeur d'eau et au dioxyde de carbone. Même dans des environnements contrôlés (des laboratoires, par exemple), les changements dans l'environnement peuvent être suffisants pour causer des variations de spectre significatives. Il est VIVEMENT recommandé de toujours purger l'instrument.

To purge the instrument:

- Remove the blanking plugs from the gas purge inlets on the instrument probe and body; these are push fit connectors.
- Connect either a 4mm or 5/32” diameter stainless steel or PTFE hose to the purge inlet ports.
- Ensure the gas tubes are connected to a suitable gas supply via a flow meter (we recommend 1 adjustable flow meter per instrument)
- Adjust the total flow to 0.5 - 2 litres per minute (The pressure drop through the unit is <1 barg).
- Do not turn off the purge during normal operation; this will impact the instrument’s performance.
- If the purge tubing is disconnected, the blanking plugs must be fitted to the inlet connections on the probe and body to maintain the IP rating and exclude dust.

The IRmadilloNA requires a stabilisation period of >12 hours once powered on and purging has started. This is to allow the internal components to reach a stable temperature and complete purging. Once fully purged the instrument is very robust against changes in ambient conditions, provided the purge gas supply is maintained.

1.2. Avoiding fouling

The diamond window at the tip of the probe must be in contact with process fluid to give a measurement. Even a thin layer of scaling or fouling will prevent the IRmadilloNA from being able to ‘see’ the process fluid.

To minimise the risk of fouling of the diamond window, we recommend taking the following steps during installation:

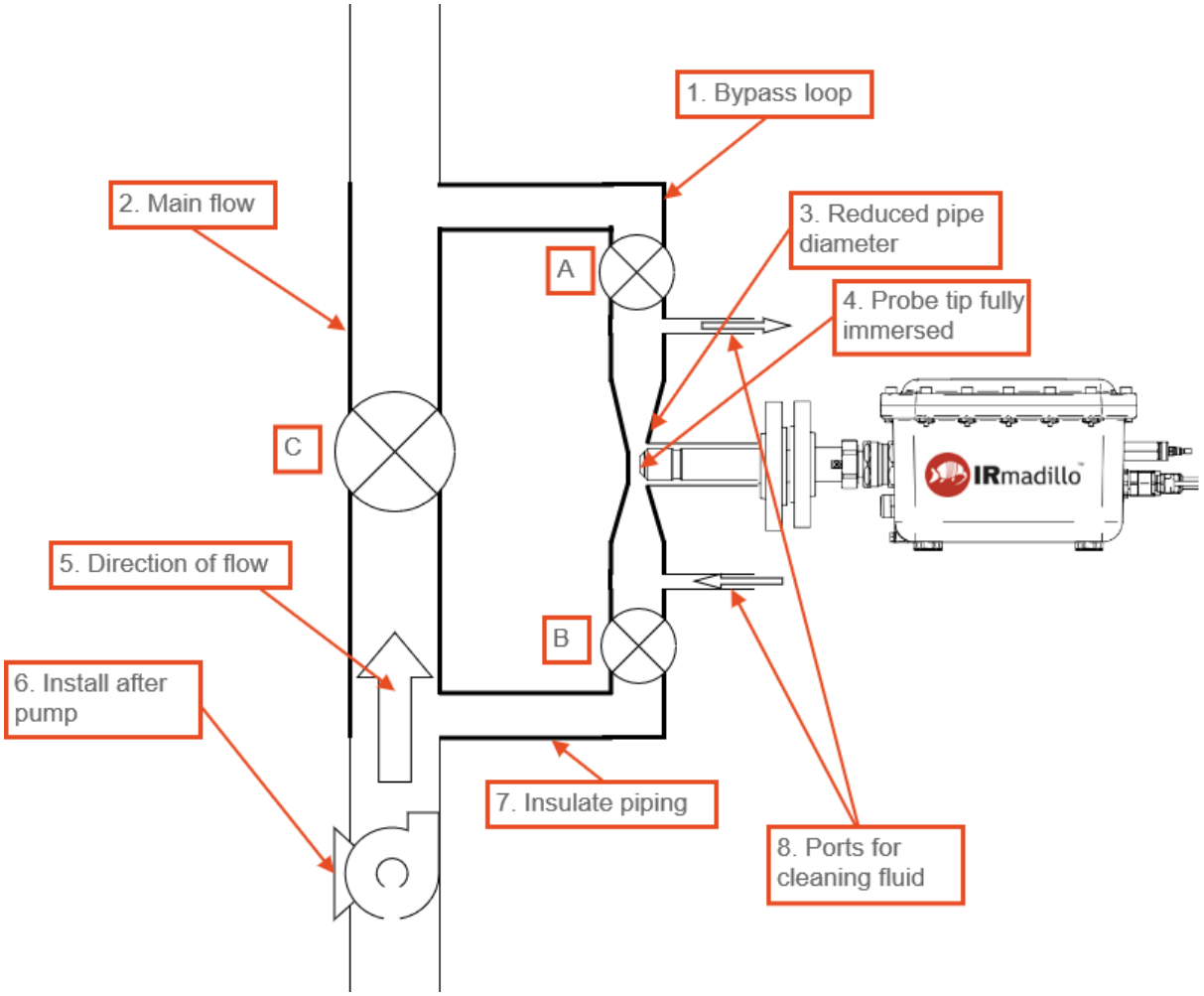

Figure 2: Installing the IRmadilloNA in a fouling environment.

1. Particularly for continuous processes, install the IRmadilloNA in a bypass loop which can be isolated using valves A and B. This makes it easy to retract the IRmadilloNA for manual cleaning if required without the need to interrupt the process flow.
2. Direct the flow through the IRmadilloNA's bypass loop by blocking or restricting the 'main' flow unless the bypass loop is to be isolated (valve C). Alternatively, this may be achieved by mounting in a pump bypass loop (offtake from pump discharge and return to pump suction).
3. Maximise the flow velocity past the IRmadilloNA by installing in a reduced-diameter section of the pipe. The minimum flow velocity should be 1.5 m/s (5 ft/s) unless there is a risk of abrasion. If so contact Keit to discuss installation.
4. Install the IRmadilloNA so that the probe tip is fully in the flow. We recommend putting the probe tip as close to the centre of the pipe as possible.
5. The direction of flow should be vertically upwards. This both minimises the risk of air bubbles becoming trapped around the probe tip and reduces the risk of settling of any sediment that is present.
6. To increase the pressure, install the IRmadilloNA after a pump. Higher pressure minimises the risk of sedimentation and bubbles.
7. Where possible, install the IRmadilloNA at a location where the process conditions are less prone to fouling. Depending on your process chemistry, this may be achieved by choosing a high temperature location and using heat tracing and insulation to avoid localised cooling. Higher process temperatures often maximise the solubility of potential fouling substances.

8. Fit ports either side of the IRmadilloNA's install point to allow clean-in-place or flushing with a suitable cleaning fluid.

1.3. Checking for warnings and checking the probe is clean

When you have connected to the powered-on IRmadilloNA the KeitSpec application should load automatically (within 3 minutes). It is important to confirm that the system is working without errors: information on IRmadilloNA warnings and errors can be found in *DOC0893 KeitSpec Software User Manual*, section 1, *Getting Started*.

Once the system is set up without errors, the probe tip must be cleaned, and a background spectrum must be taken before insertion into the vessel or pipe. If the probe is guaranteed clean, the factory background scan shipped with the instrument – and saved to the desktop of the controller – can be used. Cleanliness of the probe tip is important to performance; if you suspect fouling or scale build-up, perform a clean appropriate to your environment.

1.3.1. Cleaning instructions

Rinse the ATR element with a suitable solvent. The choice of solvent will depend on your process. You may fully immerse the ATR in solvent if needed. The following are suitable:

- Water
- Acetone
- Alcohols
- Surfactant solutions
- Alkanes (hexane, cyclohexane etc…)
- Ethers

Once exposed to a suitable solvent, wipe the ATR with a clean cloth. The metal tube of the probe can also be cleaned by wiping with a cloth.

For hard-to-remove fouling (such as biofilms or dried-on chemicals) Keit recommends an oxidative acid clean. First, clean the ATR using the methods outlined above, then perform the following: Souriau in-line plug

- If you have purchased one, place the PTFE sample cell on the end of the probe and fill with ~4ml of 1 M nitric acid solution. If you have not purchased one then please contact support@keit.co.uk for further information.
- Leave to soak for 1 hr
- Carefully remove the acid using a pipette and fill the sample cell with water
- Remove the water using a pipette
- Remove the sample cell and rinse the ATR again with water
- Carefully wipe the ATR with a cloth

It may be necessary to repeat the cleaning steps again if the contaminant has only been partially removed.

In some cases, it may be necessary to perform an alkali clean on the probe. First clean the ATR using the methods outline above, then perform the following:

- Place the PTFE sample cell on the end of the probe and fill with ~4ml of 1 M sodium hydroxide solution
- Leave to soak for 1 hr
- Carefully remove the alkali using a pipette and fill the sample cell with water
- Remove the water using a pipette
- Remove the sample cell and rinse the ATR again with water

- Carefully wipe the ATR with a cloth

It may be necessary to repeat the cleaning steps again if the contaminant has only been partially removed.

1.3.2. Recognising if the probe is clean

When looking to see if the instrument is clean, the primary area of interest within the spectrum is between 800 and 1800 cm^{-1}. This section of the absorbance plot should maintain a relatively flat line with no consistently repeating peaks. Cleaning Mode can be used to see the effects of the cleaning process in real time – see *DOC0893 KeitSpec Software User Manual,* Section 2, *The Spectrometer Tab*.

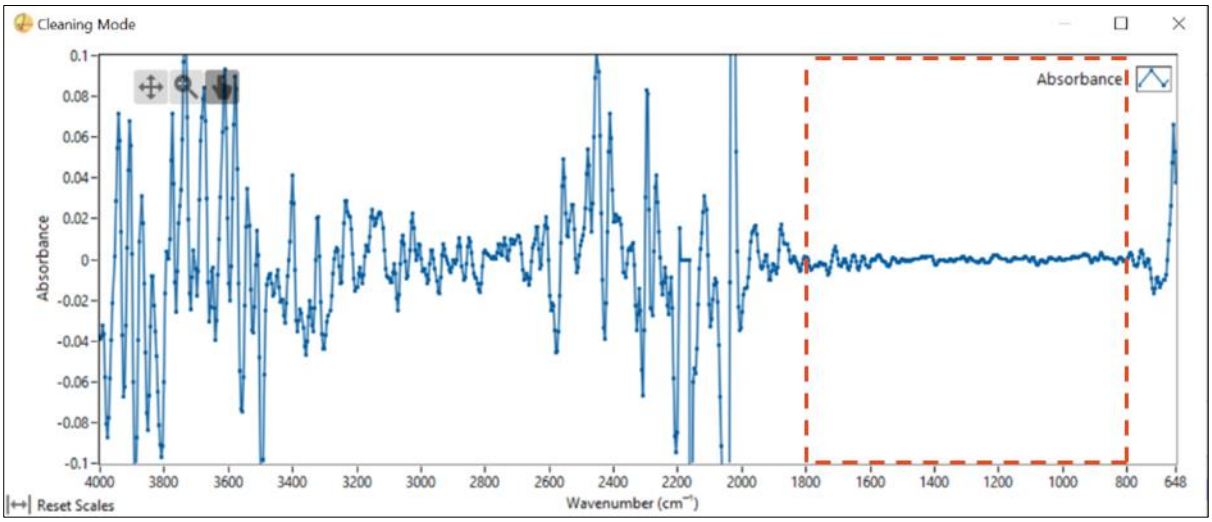

Figure 3: A clean probe showing no significant peaks within the 800 to 1800 cm^{-1} region.

Several sharp peaks in the region 800-1800 cm^{-1} indicate that the probe has not been cleaned properly.

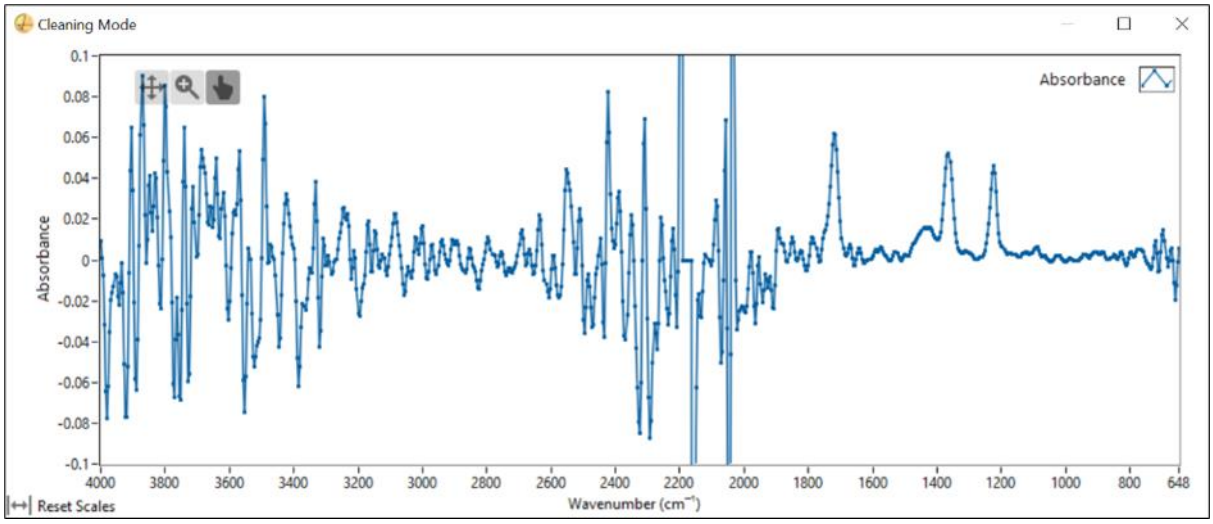

Figure 4: Sharp spikes within the region indicating an unclean probe

A peak at 1400 cm^{-1} indicates that there is some water on the probe. This could indicate poor purge quality.

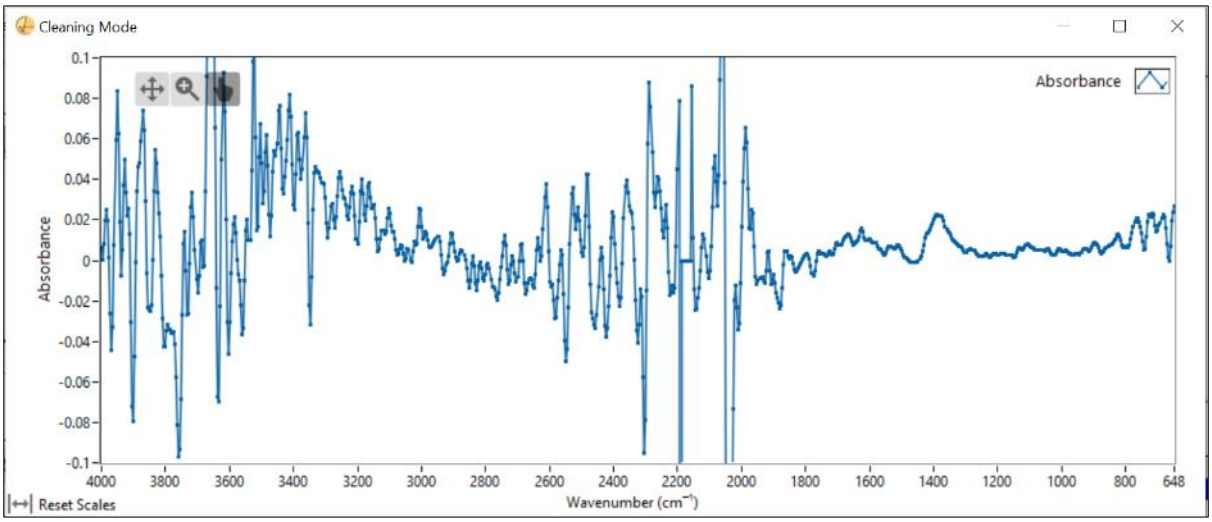

Figure 5: Peak indicating the water in the spectrum

A negative peak indicates that although the probe is clean now, it was not clean when the background was taken. Another background must be taken before any spectra can be measured.

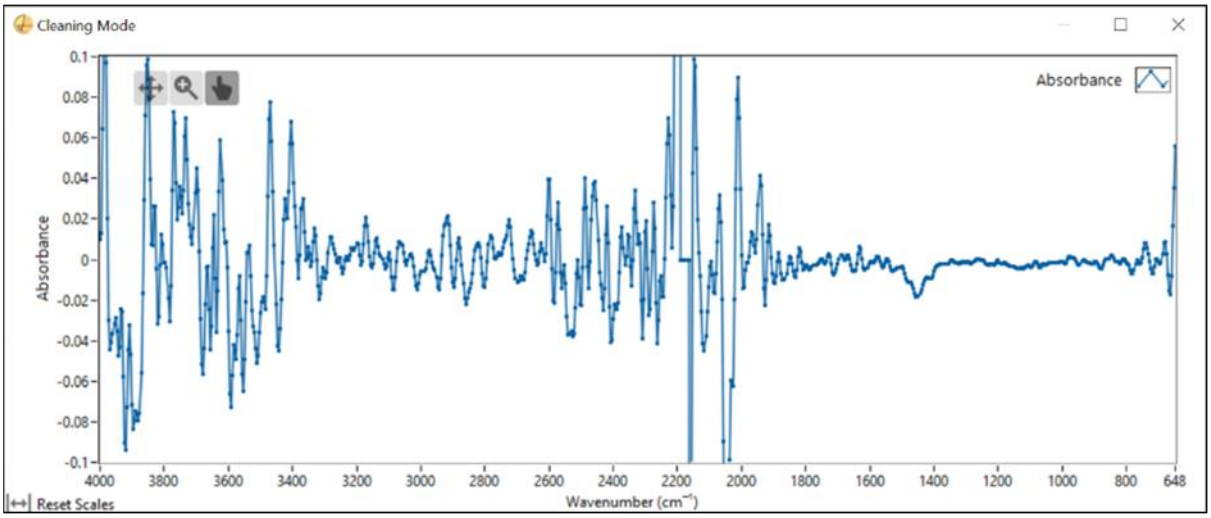

Figure 6: Negative peaks indicating the background was taken when the probe was unclean or not fully purged.

1.4. Taking a background

FTIR requires the collection of a background spectrum (also called a reference or baseline scan) before any absorption scans can be performed. This must be performed before the probe is inserted into the reaction/process of interest. The process for collecting a reference scan can be found in *DOC0893 KeitSpec Software User Manual,* Section 1, *Getting Started*.

2. MAINTENANCE

Probe accessories may contain O-rings, which we recommend replacing annually or, where worn or damaged, more frequently. See Section 4 Accessories for more information on accessories.

2.1. Spectrum health

The spectrum can be used as a good indication of the health of the instrument.

2.1.1. Use at high temperature

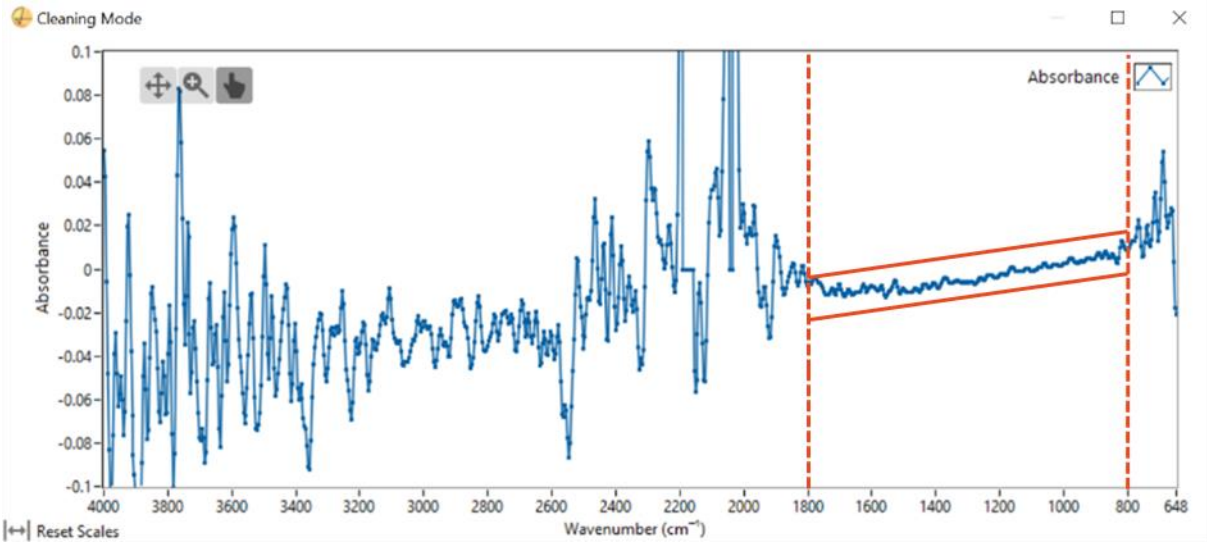

Figure 7: Probe is heated causing the spectrum to tilt

In Figure 7, the spectrum is tilted, with positive absorbance at lower wavenumbers and negative absorbance at high wavenumbers and the spectrum is not flat in the region 800-1800cm^{-1}. This indicates that the probe tip is at a higher temperature now than it was when the background was taken. This may cause chemometric models to give incorrect results unless the model uses an appropriate pre-treatment to remove the effect. Any model built by Keit will have this taken into account.

2.1.2. Emitter ageing

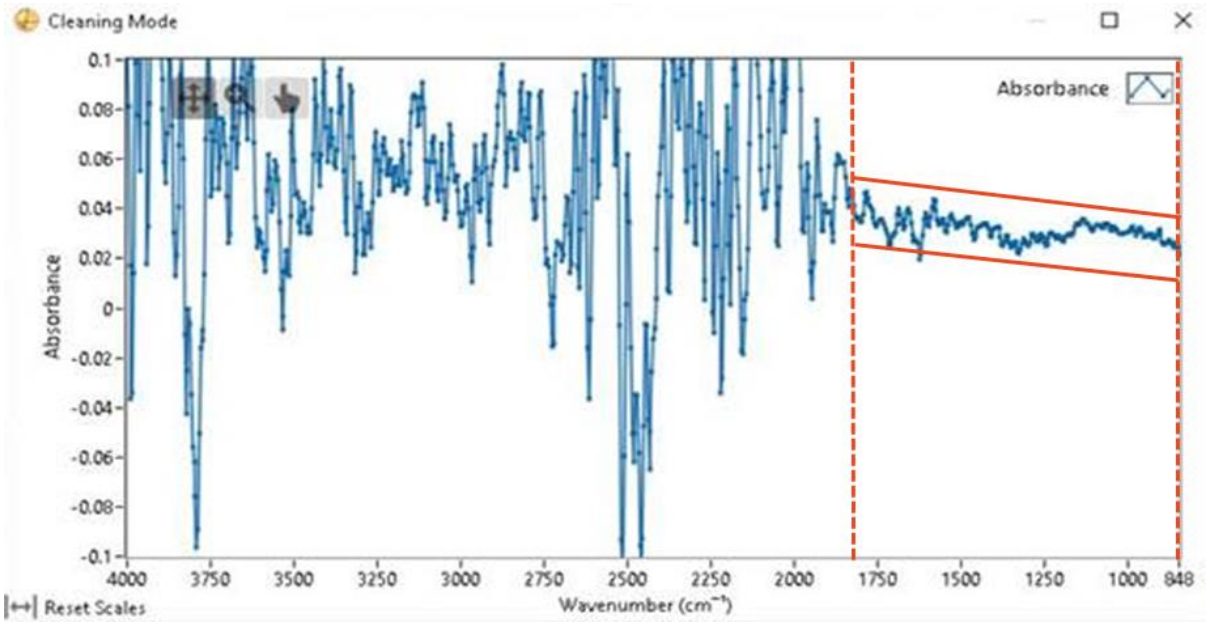

Figure 8: Spectrum is tilted showing signs of emitter ageing

The infrared emitter in the probe is expected to age over its lifetime. The results of this can be seen in Figure 8: the spectrum is tilted; the absorbance is positive across the whole spectrum; and the spectrum is not flat in the region 800-1800 cm^{-1} (contrast with Figure 3, above). If evidence of emitter ageing is seen, it may be required to take a new background scan to remove this effect – contact Keit for advice.

2.2. Remote health check

Keit can perform an annual examination of your system's health data remotely to determine if any servicing needs to be carried out. This includes review of internal diagnostics for warnings and errors, component performance for signs of premature ageing, and evaluation of background reference spectra history for signs of operator error or progressive damage.

Remote health checks are offered annually as a part of our MSP.

2.3. Cleaning the fibre optic data cable connectors

Once installed and in normal operation there should be no need to clean the fibre optic connectors. If the instrument needs to be moved so the connectors are disconnected, it is recommended that the connectors are cleaned before being re-connected.

The fibre connectors are cleaned with a cleaning pen as shown below.

Figure 9: Fibre-cleaning pen

⚠ **NOTE** REMARQUE	**Keep the dust caps on connectors and fibres when not in use. Never leave an exposed fibre connector.** Maintenez les bouchons antipoussière installés sur les connecteurs lorsqu'ils ne sont pas utilisés. Ne laissez jamais un connecteur exposé.
⚠ **NOTE** REMARQUE	**Protect the fibre from stress and tight bends (< 30mm bend radius not allowed!). Tight bends cause a stress at the glass surface and increase the probability of fracture.** Protégez la fibre des contraintes et des courbures serrées (rayon de courbure < 30 mm interdit !). Les courbures serrées provoquent une contrainte à la surface du verre et augmentent la probabilité de fracture.

2.3.1. Cleaning the Souriau fibre-optic connectors

Disconnect the connector plug from the back of the controller by gently turning the locknut anti-clockwise as shown below. Do not force it. Forcibly turning any other part of the assembly may break the connector.

Figure 10: Disconnecting Souriau connectors

To clean the female socket, remove the covers from the tip of the cleaning pen and press it into both female ports until it clicks.

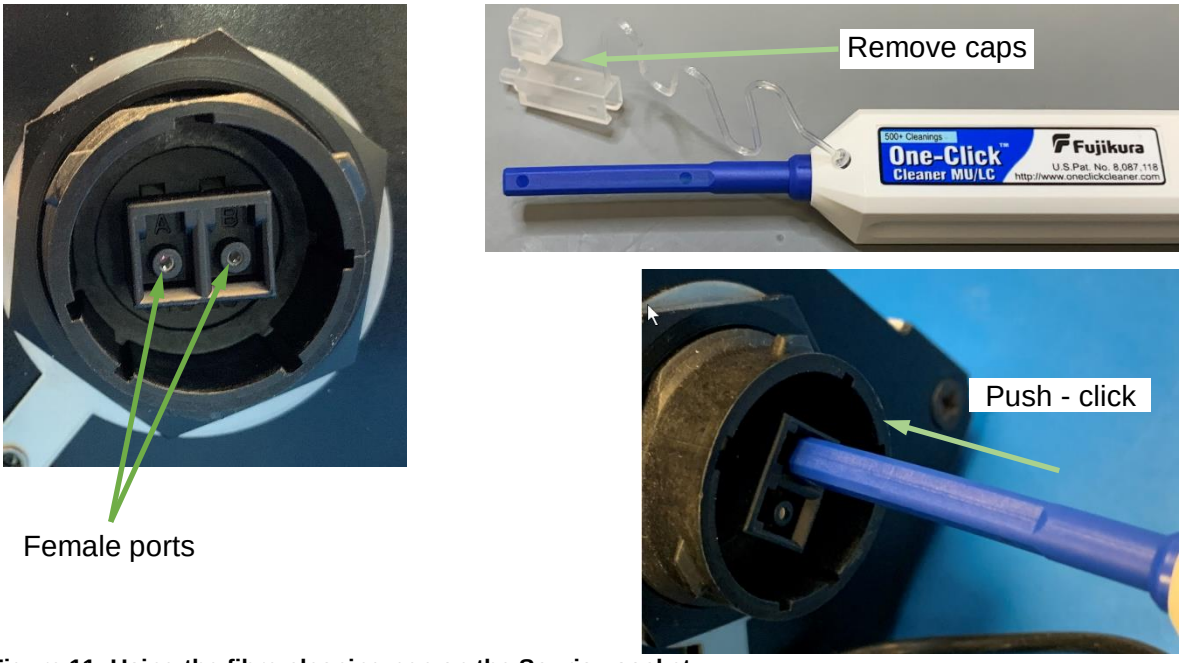

Figure 11: Using the fibre-cleaning pen on the Souriau socket

Cleaning the male plug is similar. The transparent cap remains fitted to the cleaning pen. The pen fits over each of the two male fibre ferrules and is aligned by the transparent cover. Push and click once on each ferrule.

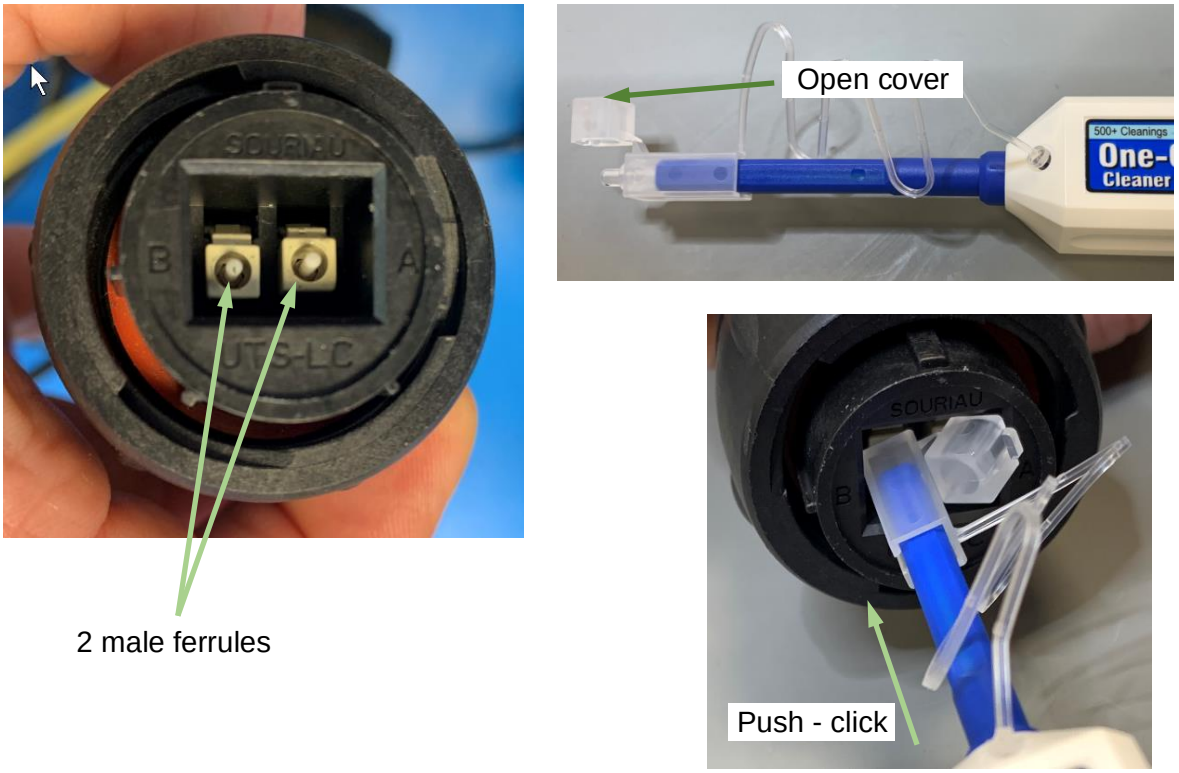

Figure 12: Using the fibre-cleaning pen to the Souriau plug

Once both halves of the connector have been cleaned then they can be reconnected. Align the keyways and turn the locknut clockwise until a distinct click is heard.

Figure 13: Reconnecting the Souriau connectors

3. COMPONENTS

3.1. Storage conditions

When installed with purge and power, the IRmadilloNA can thermally control its internal temperatures up to the limit specified on the product label. However, if the system is to be stored before its use Keit recommends an air-conditioned environment. The environment must be non-condensing, with a relative humidity below 95%. The IRmadilloNA or storage container should also be out of direct sunlight.

⚠ **WARNING** AVERTISSEMENT	**Storing the IRmadilloNA unpowered and unpurged in unsuitable conditions can cause damage to the instrument. If in doubt, please consult with Keit** Le stockage du spectromètre IRmadilloNA non alimenté et non purgé dans des conditions inadéquates peut endommager l'instrument. En cas de doute, contactez Keit.

In contrast, the controller must be both stored and operated in a safe, non-hazardous environment. Keit recommends an air-conditioned environment, which is non-condensing and has a relative humidity below 90%. See DOC1048R User Manual - Safety for more information.

⚠ **WARNING** AVERTISSEMENT	**Storing or operating the IRmadilloNA's controller in unsuitable conditions can cause damage. If in doubt, please consult with Keit** Le stockage ou l'utilisation du contrôleur IRmadilloNA dans des conditions inappropriées peuvent provoquer des dommages. En cas de doute, veuillez consulter Keit

4. ACCESSORIES

4.1. Sample cell - ASM1398

⚠	**WARNING** AVERTISSEMENT	**Not certified for hazardous environments** Non certifié pour les environnements dangereux

The sample cell is a ~5ml vessel attached at the end of the probe for performing manual sample analysis or used as part of a probe cleaning procedure. It is fixed in place using a tri clamp that locates over the rear of the sample cell and two semi-circular collars that fit around the probe. Ensure that the C-pieces make good contact with the probe and that the pins are seated inside their corresponding holes then use the clamp to fit the cell to the probe, clamping against the C-pieces. The clamp does not need to be tightened with significant force, we recommend to tighten the thumbscrew until resistance is felt + 1 turn. For volatile substances it is recommended to keep the stopper/cap in place during use.

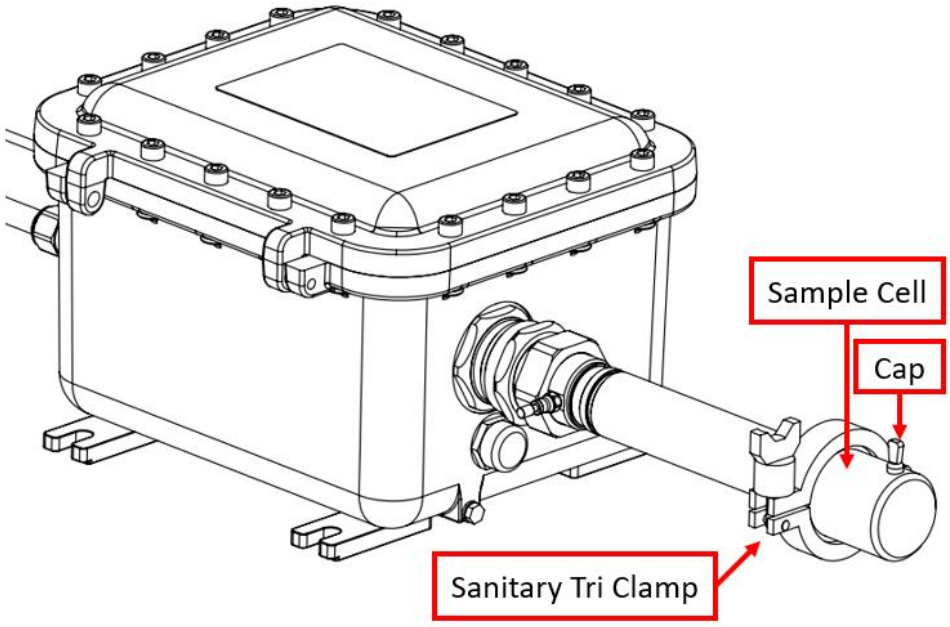

Figure 14: Sample cell fitted to IRmadilloNA probe

4.1.1. Maintenance

Inspect the O-ring regularly and replace if it has visually degraded, whether through swelling or other damage; use plastic forceps to remove the O-ring. This is particularly important if the chemistry that you are analysing is known to cause swelling of the O-ring elastomer. In any case, replace O-rings annually. For O-ring specifications please see document “Flow cells and sample cell for ASM0627-xx-x-Cx-O-Gx-D3x (Non-ATEX Certified).pdf”. Also inspect the clamp and C-pieces for any signs of degradation, cracks, or damage. If the clamp and/or the C-pieces shows signs of significant wear, do not re-fit them and contact support@keit.co.uk for advice.

4.1.2. Cleaning

The sample cell may be cleaned using standard laboratory cleaning practices, as appropriate for your reaction. Also ensure the ATR element has been thoroughly cleaned and dried before use of the sample cell.

4.2. Flow cells

⚠ **WARNING** AVERTISSEMENT	**Not certified for hazardous environments** Non certifié pour les environnements dangereux
⚠ **WARNING** AVERTISSEMENT	**Test for leaks after installation with non-hazardous chemistry before resuming normal operations.** Testez les fuites après l'installation avec des produits chimiques non dangereux avant de reprendre les opérations normales

The flow cell assemblies contain a pressure vessel and have been designed to withstand the analyte conditions that are specified on your product label. For detailed technical drawings, contact Keit.

4.2.1. High flow cell - ASM1392

The high flow cell can be secured to the end of the probe for continuous analysis of your process. The flow cell is fitted with two ¾" NPT ports on opposing sides, to accommodate a high flow rate offshoot pipe from the main process, passing the liquid in front of the ATR. This is an alternative to installing the IRmadilloNA into a main pipeline.

It is fixed in place using a tri clamp that locates over the rear of the flow cell and two semi-circular collars that fit around the probe. Ensure that the C-pieces make good contact with the probe and that the pins are seated inside their corresponding holes then use the clamp to fit the cell to the probe, clamping against the C-pieces. The clamp does not need to be tightened with significant force, we recommend tightening the thumbscrew until resistance is felt + 1 turn. The flow cell can be rotated through 360° to accommodate the user's installation. Please ensure that the flow through the cell is turbulent.

Maintenance and inspection method: Visually check that all O-rings inside the cell have not perished. They should show no signs of swelling, tearing, or degradation. For O-ring specifications please see document "Flow cells and sample cell for ASM0627-xx-x-Cx-O-Gx-D3x (Non-ATEX Certified).pdf". Also inspect the clamp and C-pieces for any signs of degradation, cracks, or damage. If the clamp and/or the C-pieces show signs of significant wear, do not re-fit them. Contact support@keit.co.uk for advice.

Do not use impact (e.g. a hammer) to separate components if the probe becomes stuck in the flow cell.

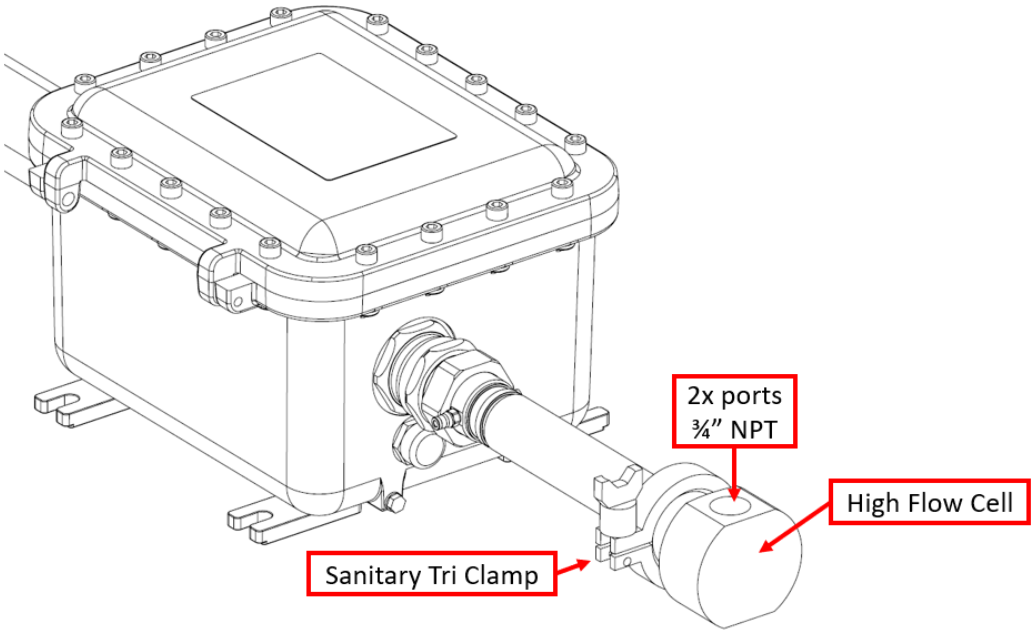

Figure 15: High flow cell fitted to IRmadilloNA probe

Table 1 – Specifications and safety limits (high flow cell)

Parameter	High Flow Cell
Pressure	Refer to high flow cell marking
Analyte Temperature	Refer to high flow cell marking
Body Material	Corrosion-resistant metal
O-ring Seal	FFKM (Kalrez)
Port Size	3/4" NPT
Cell Internal Volume	~27 ml
Intended Use	Only for use with ASM0627-09-N-Cx-O-Gx-D3x
Fluid Group	1
Minimum Inspection Frequency	Annual

4.2.2. Low flow cell - ASM1222

The low flow cell that can be secured to the end of the probe for continuous analysis of your process. The flow cell is equipped with two 1/16" NPT fittings to attach pipelines too, or it can be assembled with two ¼" Swagelok nuts attached to a short section of pipe on opposing sides. It is designed to force liquid past the ATR and is primarily used for lab work where flow rate and volume are low.

It is fixed in place using a tri clamp that locates over the rear of the flow cell and two semi-circular collars 'C-pieces' that fit around the probe. Ensure that the C-pieces make good contact with the probe and that the pins are seated inside their corresponding holes. Slide the

flow cell over the probe so the O-ring is in contact with the end of the probe. Then use the clamp to secure the C-pieces and cell. The clamp does not need to be tightened with significant force, we recommend tightening the thumbscrew until resistance is felt + 1 turn. The flow cell can be rotated through 360° to accommodate the user's installation. The cell should be checked for leaks prior to running hazardous chemistry through it.

Maintenance and inspection method: Visually check that all O-rings inside the cell have not perished. They should show no signs of swelling, tearing, or degradation. For O-ring specifications please see document "Flow cells and sample cell for ASM0627-xx-x-Cx-O-Gx-D3x (Non-ATEX Certified).pdf". Also inspect the clamp and C-pieces for any signs of degradation, cracks, or damage. If the clamp and/or the C-pieces shows signs of significant wear, do not re-fit them. Contact support@keit.co.uk for advice.

Do not use impact (e.g. a hammer) to separate components if the probe becomes stuck in the flow cell.

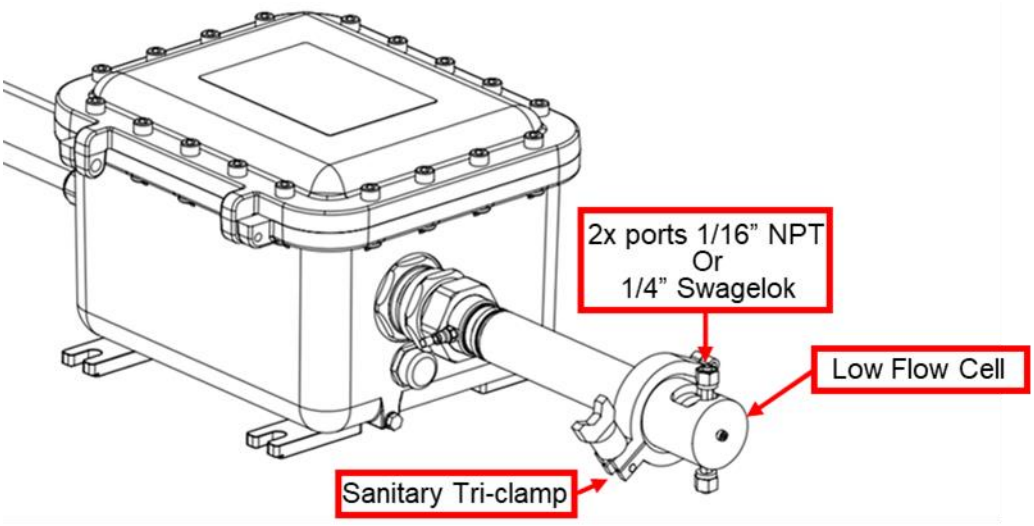

Figure 16**: Low flow cell fitted to IRmadilloNA probe**

Table 2 - Specifications and safety limits (low flow cell)

Parameter	Low Flow Cell
Pressure	Refer to low flow cell marking
Analyte Temperature	Refer to low flow cell marking
Body Material	Corrosion-resistant metal
O-ring Seal	FFKM (Kalrez)
Port Size	1/16" NPT or 1/4" Swagelok
Cell Internal Volume	~0.8 ml
Intended Use	Only for use with ASM0627-09-N-Cx-O-Gx-D3x

Fluid Group	1
Minimum Inspection Frequency	Annual

4.3. Inclined Stand – ASM0916

WARNING AVERTISSEMENT	**Not certified for hazardous environments** Non certifié pour les environnements dangereux

The inclined stand is designed for using the spectrometer as a bench top instrument for offline sampling. Angled at 35°, the probe can be positioned into beakers or other vessels for ease of use. It can also be configured for two heights to give added flexibility in how it is used.

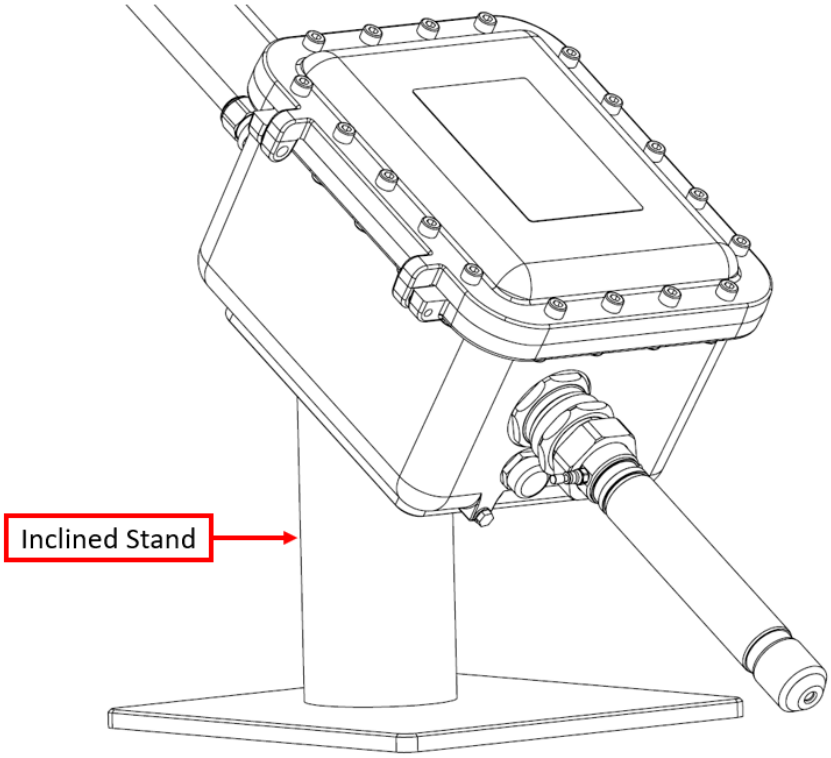

Figure 17: IRmadilloNA on an inclined stand

4.3.1. Safety

WARNING AVERTISSEMENT	**The spectrometer and inclined stand assembly weighs ~24kg. Use caution when handling to avoid injury.** L'ensemble spectromètre et support incliné pèse environ 24 kg. Soyez prudent lors de la manipulation pour éviter les blessures.

4.3.2. Installation

Assemble the inclined stand to the desired height; two positions are possible. For technical drawings, contact Keit at support@keit.co.uk.

To install the inclined stand to the instrument, carefully roll the spectrometer over such that the mounting holes are pointed upward, the instrument should be disconnected from power coms and purge to avoid damage to the cables or twisting.

Position the inclined stand over the mounting points such that the mounting plate lines up with the instrument. The spectrometer is located with 4 screws through the corners of the stand into the mounting holes on the spectrometer.

Return the instrument to the correct orientation and connect the purge, the power cable, and communications cable/s.