

# Calibrating the IRmadillo – What you need to know



## What is calibration & why do you need it?

The IRmadillo is an in-line process analyser that uses infrared light. It works by recording a spectrum of the process under analysis and then interpreting that spectrum in many different ways to provide useful information. The IRmadillo can run multiple calibrations at once to act as a concentration meter for a vast array of chemicals and process states. The IRmadillo can be calibrated to function in two different modes; quantitative, qualitative or both.

■ **Quantitative:** in this mode the IRmadillo is a concentration meter measuring the amount of a given chemical or group of chemicals (i.e. ethanol and sugar %wt in fermentation, or ppm of phosphorous in edible oil refining).

■ **Qualitative:** in this mode the IRmadillo measures process states or conditions (i.e. for processes that may involve scaling “is the probe fouled?”, or in a fermentation process “contaminated batch”).

In both cases the instrument needs to record a spectrum and then have relevant reference data as an input into calibration.

By combining the spectrum with the reference data we can train the instrument on what it needs to measure. Keit uses a combination of techniques to do this (PCA, PCR, PLS, LWR and SVR) and will choose the best technique for your process.

## How does the instrument get calibrated?

There are two stages in the calibration of an instrument:

■ **Chemical calibration:** this is where we map out the different chemicals and their concentration ranges that could occur in your process. This is to train the instrument on the different responses it would expect to see during use. See the following table for comparison of the four different ways this can be achieved.

■ **Process tuning:** this is where the instrument is allowed to run in your process for a period of time to record the specifics of that installation (i.e. temperature, pressure, air bubbles, etc.) and chemical nuances that are hard to control (i.e. reactive intermediates or trace chemicals that are not measured by off-line reference).

Once both chemical calibration and process tuning are complete then the instrument is calibrated. At this stage the output of the IRmadillo can be linked to your DCS, PLC or other control system and used as an analyser.

## Chemical calibration – what are the options?

There are four different ways to accelerate chemical calibration.

Calibration Type	Description & Requirements
<b>Starter</b>	<p><b>Description:</b> Keit uploads a pre-formed calibration on the instrument from legacy installs before the instrument ships</p> <p><b>Requirements:</b> Legacy installs – currently only offered for certain applications</p>
<b>Foundation</b>	<p><b>Description:</b> Keit performs a calibration at Keit or the customer site on the instrument before installing the instrument with samples provided by the customer and spiked with chemicals.</p> <p><b>Requirements:</b> Samples provided to Keit before the instrument is installed. Ideally samples are shipped to Keit in the UK and the foundation calibration is performed before shipping (which could result in a small delay in shipment).</p>
<b>Hybrid</b>	<p><b>Description:</b> Keit performs the calibration with spectra from multiple instruments with samples provided by the customer after the instrument has been installed. Performed at Keit.</p> <p><b>Requirements:</b> Samples provided to Keit. This removes the delay to shipment but the samples still need to be sent to Keit.</p>
<b>Process Perturbation</b>	<p><b>Description:</b> The process is deliberately changed and perturbed to cause chemical change on the instrument after installation – either through process change or spiking.</p> <p><b>Requirements:</b> You need to deliberately perturb your process to induce change – and be comfortable with what that means to your product.</p>

If none of the above options are suitable then accelerated chemical calibration won't be possible and calibration will have to naturally wait for these chemical changes to be observed. This is still perfectly valid but will take a longer period of time.

## What do I have to do so Keit can calibrate the instrument?

**The golden rule of calibration is that calibrations are only as good as the data used to build them.**

It is absolutely critical that Keit receives good quality reference data. Failure to provide this means calibration will not work, and will have to be started again. Please ensure;

- **That both the time at which the sample was taken and the time at which the sample was analysed are accurately recorded:** it's critical to note when a sample was taken from the process as this is what Keit typically uses to calibrate against. But some samples are not stable over time – they change. If this is the case you may need to change your sampling process to minimise the time these samples are waiting for analysis.
- **That you sample regularly enough to capture the whole process:** sampling once or twice throughout a batch might not be frequent enough. Keit needs to understand what the typical batch looks like and can then recommend how often to sample.
- **That the reference data are accurate and precise:** the most important thing is that the primary reference technique (i.e. HPLC, ICP, titration, chemical assays etc.) is performed accurately and precisely. We will be assuming this is 100% accurate and any mistakes here will carry through to the final instrument calibration.
- **That the installation is robust and stable:** the IRmadillo needs a purge of clean, dry air or nitrogen (Keit can supply an air handling unit that filters air from an existing compressed air generator). If the air provided to the instrument is not dry or stable, calibration will fail. Likewise, the instrument needs a good quality installation into the process with representative flow past the probe or calibration will also fail.

## How long does initial calibration take?

That is a slightly difficult question to answer. If the process is allowed to drift naturally then calibration could take months – especially for fairly stable continuous processes. Keit will also ask to look at your legacy reference data to better understand how long the process tuning is likely to need. Some helpful information to know in advance is:

- How often do you perform process maintenance and cleaning?
- How often does your process instigate automated changes such as filter back-washes or centrifuge discharges?
- How often do you change feedstock? Is it batch or continuous feed?

Keit can then propose a sensible time for the process tuning – typically this is up to one month but might be longer for some processes.

## How long will my calibration last?

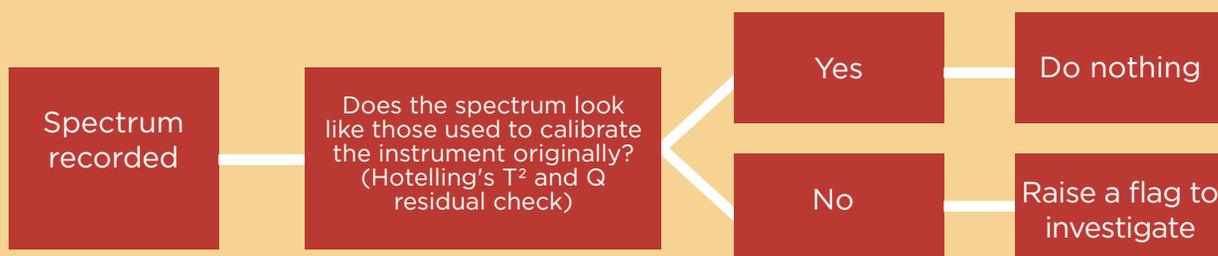
This depends on the process and feedstock. The IRmadillo has been shown to be very robust and stable and has very little instrument drift.

Calibrations are typically stable for many months at a time – if not years. When they do need adjustment this is often very minimal – because the IRmadillo is built using FTIR spectroscopy (instead of the more complicated NIR).

If you have an active Calibration Support Plan (CSP) then Keit can augment your existing calibration very quickly (typically within two weeks) with a small amount of samples and reference data to reflect the current state of your process and feedstock.

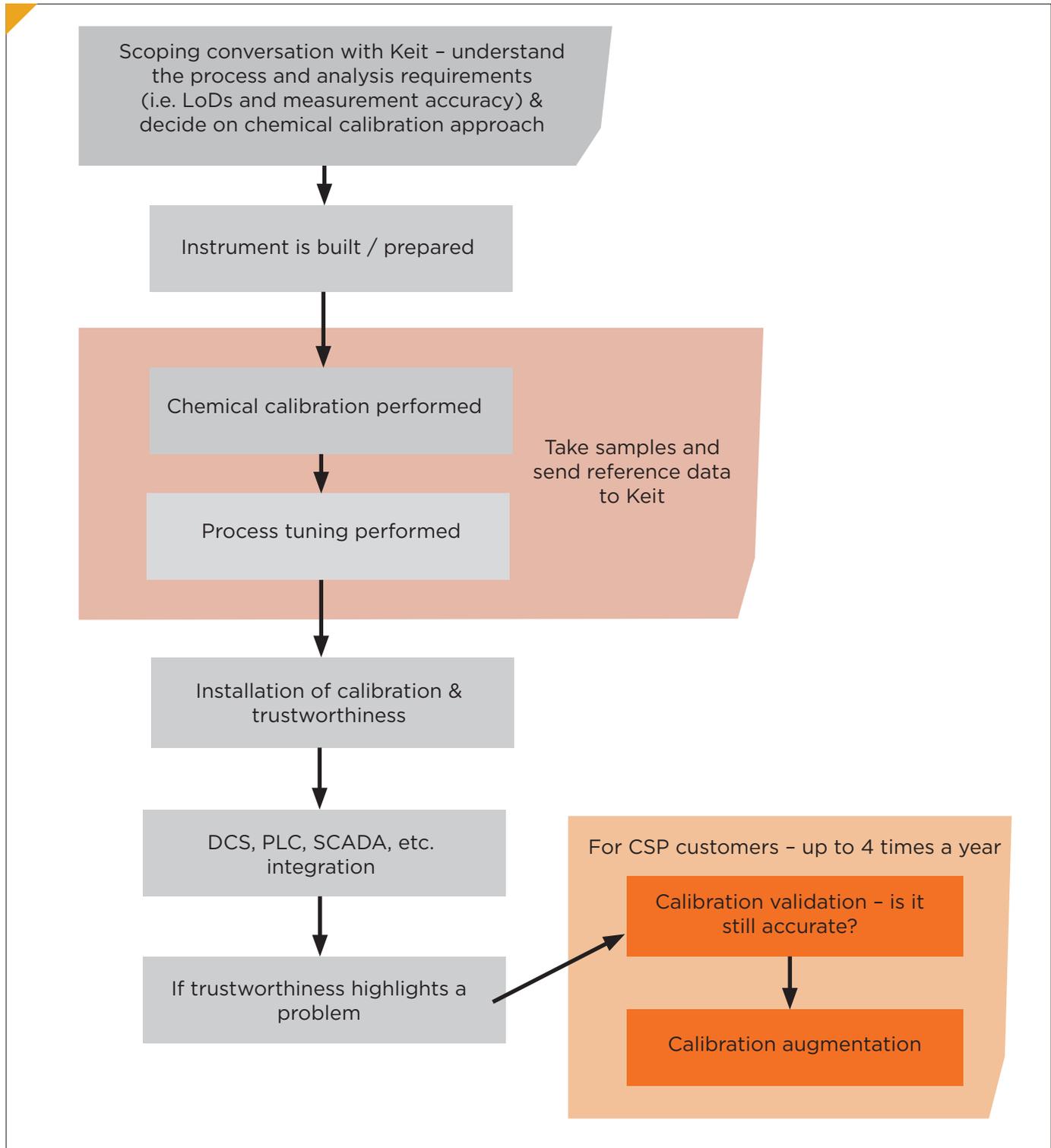
## What is “trustworthiness” and how does it work?

Keit is committed to ensuring customers can trust every measurement made using the IRmadillo. So we build “trustworthiness” models with every calibration we build. These only raise a flag when the IRmadillo believes something has drifted and the instrument needs attention. Normally this is because of purge failure or known process changes (such as cleaning). If there’s been a gradual process or instrument drift outside of calibration then trustworthiness will alert you to initiate a recalibration. A flow diagram of the trustworthiness is shown here:



## The Keit calibration process

Below is a simple flowchart highlighting how Keit performs an initial calibration on an instrument and its maintenance and up-keep over time.



## Checklist for calibration

Here is a checklist for things that you and Keit will need to have ready to properly calibrate an instrument:

### 1. Process needs

- I have a target calibration performance (a limit of detection or level of accuracy needed) for each chemical to be measured and a definition of process states for qualitative calibrations
- I have legacy reference data to send to Keit for analysis to understand how long process tuning is likely to take

### 2. Chemical calibration approach

- Keit has told me I can have a starter calibration  
OR
- I can send samples to Keit for a foundation or hybrid calibration  
OR
- I can do lab sampling at my site before installing the instrument on-line for a foundation calibration  
OR
- I can perturb my process to force chemical concentration change on-line  
OR
- I understand I may have to plan for a longer term calibration to capture all my chemical change

### 3. Reference data

- I have a reliable off-line reference technique for each chemical I want to measure (i.e. HPLC, GC, ICP, titration, etc.) and I know it is accurate and precise
- I have a process of taking samples that will accurately record the time the sample was taken from the process
- I know that my samples are stable after taking off-line  
OR
- I will perform my reference analysis fast enough to prevent degradation of the sample and will record the time the sample was analysed as well as the time the sample was collected

### 4. Installation requirements

- I will have dry, clean and reliable air for purging the instrument  
OR
- I have bought/rented an air dryer from Keit and will have it installed before calibration
- I know the instrument will be seeing a representative flow of the process and I will sample from close by to the installation point
- I have a method for Keit to have remote access to the instrument for downloading and uploading data to and from the IRmadillo
- I know my installation will not cause problems for the instrument (i.e. I'm not directly next to a very hot vessel, I'm not in an area with acidic or caustic spray on the IRmadillo enclosure, etc.)

### Interested in finding out more?

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