APN1041

The IRmadillo in the Distillation Process

Key Words

- Fuel ethanol
- Distillation
- Whole stillage
- Beer bottoms
- Distillation overheads

Abstract

Measuring the performance of the fuel ethanol distillation columns in real time allows the opportunity for constant process optimisation - thereby minimising energy consumption and maximising ethanol recovery whilst ensuring product quality targets are met. In separate studies, the IRmadillo spectrometer has proved able to measure the relevant concentrations with levels of accuracy that allow true optimisation of the process.

Introduction

The purification of ethanol following fermentation is normally achieved through distillation, followed by molecular sieves. Distillation consumes a significant amount of energy and needs to be closely managed to maximise product recovery, minimise energy consumption and achieve desired product purity. Operation of the columns needs to be optimised to minimise ethanol losses in the whole stillage (column bottoms) stream and at the same time minimising impurities such as acetal, methanol and longer chain alcohols (fusels) in the ethanol product. Distillation columns can be challenging for plant operators to control, due to the 'multivariable' nature of their behaviour and relatively slow process dynamics. Optimum performance is only achievable with continuous measurement of the whole stillage and the column overhead compositions.

Spectrometers allow continuous and detailed measurement of chemical concentrations in real time. The majority of process spectrometers are based on near infrared light, which is fundamentally less informative than mid infrared light. Conventional mid infrared spectrometers (which often use a Fourier transform and so are referred to as "FTIR spectrometers") have sensitive moving parts and fragile fibre probes – making them wholly unsuitable for production environments such as ethanol refineries.

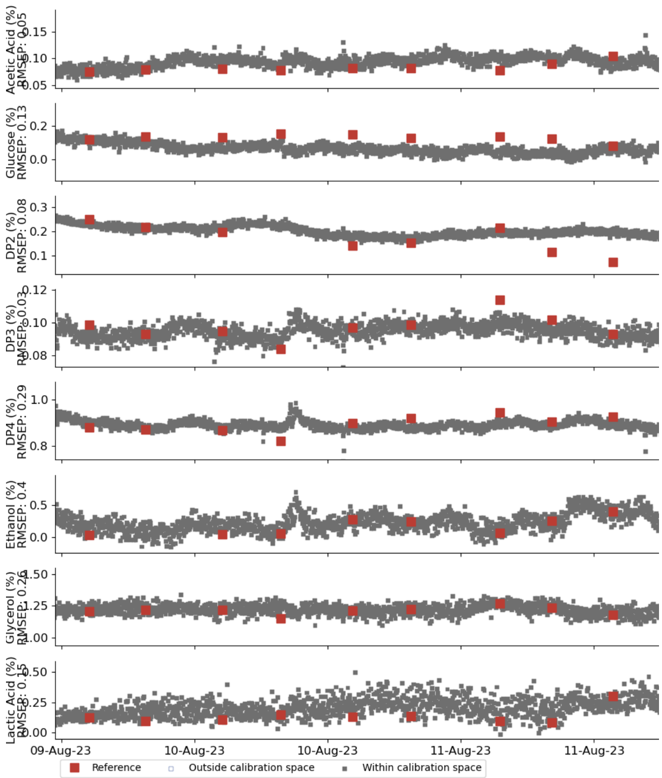

Figure 1: Measurements of species of interest at the bottom of the distillation column. Grey points are the output of the IRmadillo spectrometer. The red points are the measured samples from offline HPLC

The IRmadillo is a process analyser built using FTIR spectroscopy, but with static optics, removing the need for moving mirrors or fibre probes, and dramatically improving stability, reliability, and ruggedness.

Example use case - Whole Stillage

This application note presents data from an ethanol refinery in Iowa, measuring the concentration of ethanol and other components of the whole stillage, and also presents further lab data to demonstrate the ability of the IRmadillo to measure the concentration of acetal, methanol and pentanol in the column overheads. By measuring at both these locations at the top and bottom of the distillation column, the operators would be able to manage the column to maximise product quality, while minimising wastage and optimising power consumption.

The IRmadillo was installed in the beer column bottom, in order to measure the composition of the whole stillage. Models were built to measure Acetic Acid, Glucose, DP2, DP3 and DP4, Ethanol, Glycerol and Lactic Acid. The plant was able to observe in real time, the concentrations of those species of interest at levels that allowed tighter control of the distillation column.

Chemical	Range	Accuracy
Ethanol (%)	0-0.5	0.099
DP4 (%)	0.8-1.0	0.034
DP3 (%)	0.10-0.22	0.019
DP2 (%)	0.12-0.28	0.027
DP1 (%)	0.1-0.35	0.046
Lactic acid (%)	0.14-0.26	0.028
Glycerol (%)	1.05-1.4	0.055
Acetic acid (%)	0.04-0.14	0.016

Table 1: Measurement accuracy for species of interest in distillation

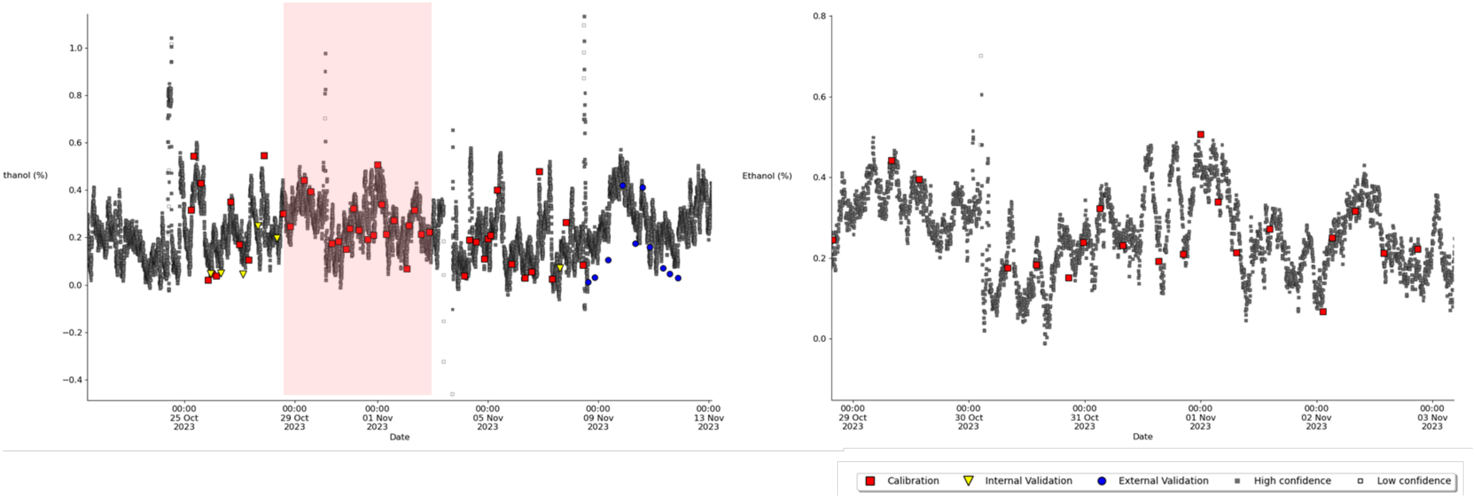

Figure 2: Tracking ethanol concentrations in the beer bottoms can be used to increase steam or direct the process liquid back to the beer column for reprocessing, maximizing ethanol yields.

Example use case – Distillation Overheads

Distillation separates chemicals using the property that different chemicals have different boiling points. The weakness of this approach is that chemicals with similar boiling points will tend to appear together in the finished distillate. This is the case with Acetal (BP 103C), Methanol (BP 65C) and Propanol (BP 97C), which are sufficiently similar to Ethanol, which has a boiling point of 78C. If the distillation column is operated incorrectly, such as at the wrong 'cut point', the ethanol may be contaminated with out-of-specification levels of contaminants.

Chemical	Accuracy
Acetal (ppm)	28
Methanol (ppm)	12
Propanol (ppm)	33

Table 2: Measurement accuracy for species of interest in distillation overheads

Real-time measurement of these components make it easier to avoid this situation. Calibration models were therefore built on laboratory samples of ethanol with contaminant levels of Acetal, Methanol and Propanol. The IRmadillo was shown to be able to measure all three contaminants at low concetration levels, making it ideal for ethanol product quality control.

Conclusions

The IRmadillo is able to measure, in real time, key components of interest in whole stillage and ethanol overheads, enabling optimisation of the distillation column for minimising ethanol wastage in the stillage. The measurements are well within the useable range and can be used to understand and control the process.

Keep in Mind

The IRmadillo can be calibrated to measure a large range of chemicals at the same time. This work shows the use for DP4, DP3, DP2, DP1, lactic and acetic acids, glycerol and ethanol measurement. We also measure acetal, methanol and propanol levels, but the IRmadillo can also be calibrated to measure applications in ethanol propagation, FAN, PAN and fusels in fermentation – all at the same time, in real-time.

Contact us

+44 (0) 1235 431260

(Sales) enquiries@keit.co.uk

(Product Support) support@keit.co.uk

www.keit.co.uk

KEIT INDUSTRIAL ANALYTICS

Measure | Trust | Improve

4 Zephyr Building, Eighth Street, Harwell Campus, Didcot, Oxfordshire, OX11 0RL, United Kingdom