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NITRIC ACID PLANT CEM'S

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MCERTS
European EN14181 QAL 3 Compliant
US EPA 40 CFR Part 60&75 Compliant
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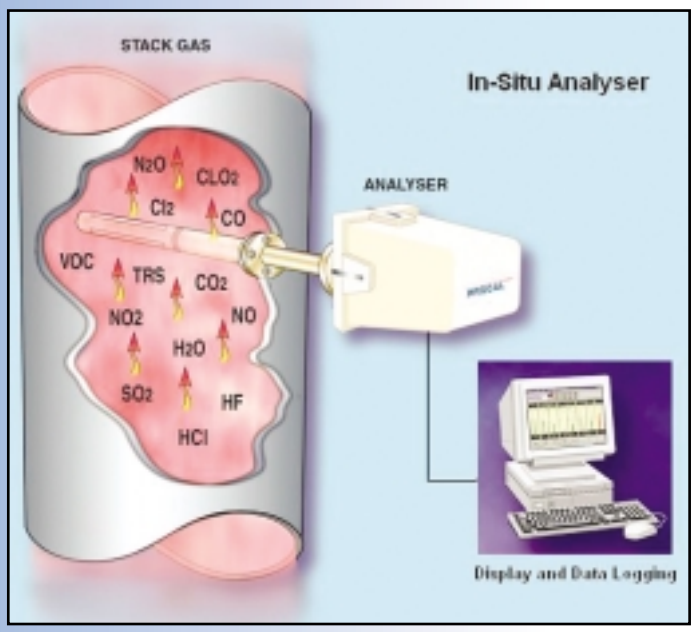
Introduction

Continuous Emission and Process Monitoring in Nitric Acid Plants using In-Situ Analysers



Procal Analytics Ltd have been supplying continuous monitoring systems into the Nitric Acid industry for over 20 years. The instruments are designed to withstand the harsh environment with minimum maintenance requirements. The analysers are capable of monitoring up to six gas species simultaneously, displaying, data logging and transmitting the concentrations.

The unique design was the result of work carried out on the ICI Kuhlmann processes between 1987-90, ICI were concerned over the reliability and hence running costs of their CEM and process extractive systems. The problem was associated with the maintenance of the sample systems, problems occurred due to corrosion of key components and blockages. As tighter emission regulations where introduced the reliability of the CEM became more critical, process instruments used as part of a plant control had to achieve a high availability.

To overcome the problems with extractive systems across stack analysers were installed, initial trials proved the benefit of analysers which measured in the process there by avoiding the need for sample handling systems. The main drawback was identified as limited zero and calibration capabilities; this might have been acceptable in process applications but on regulatory CEM systems where the capability of challenging the system with both zero and span gases is desirable if not mandatory.

The reason for the requirement to both zero and span is to remove the drift inherent in all analytical instruments and demonstrate to the authorities that the analyser is in calibration for that period.

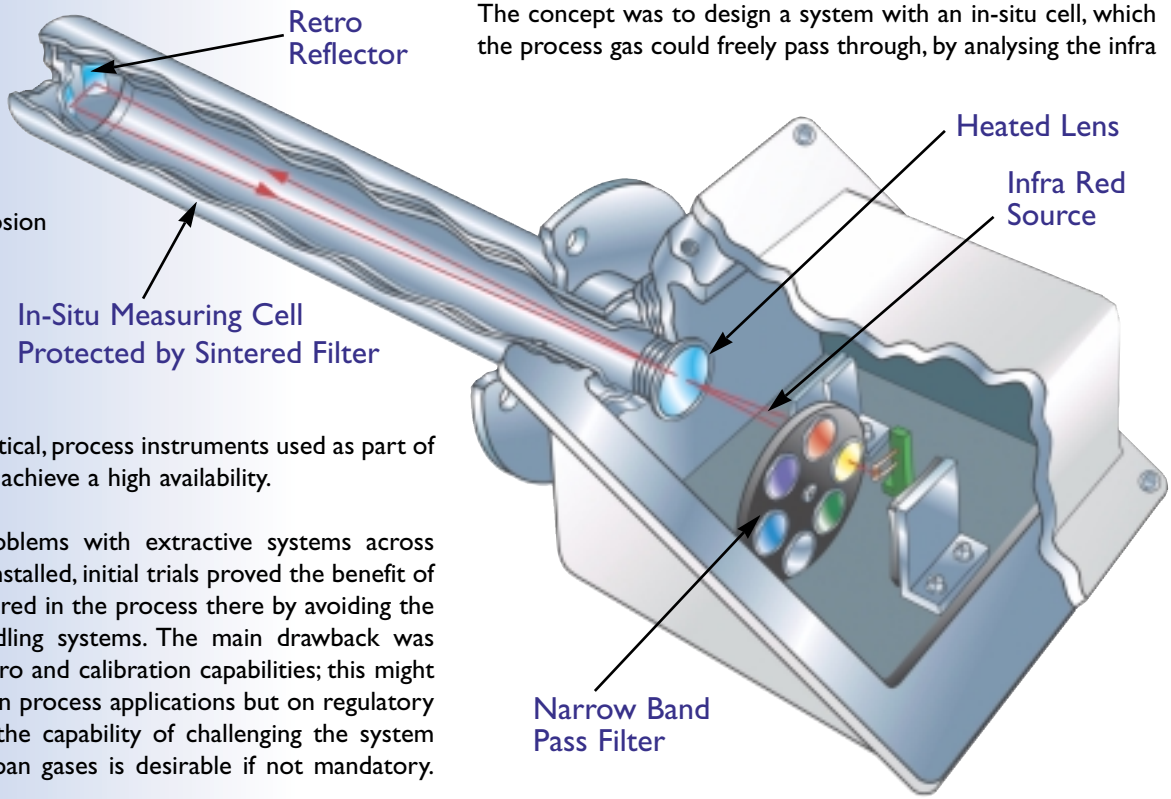
The Requirement

Clearly the ideal solution was to develop an instrument with the capability of measuring on stack, negating the need for the extractive system, with the capability of periodically checking both zero and span. A further consideration was the number of gas species required to be monitored, initially the measurement consisted of NO and NO2 however after consideration it was decided that there would be interest in other gases such as N2O, NH3 and CH4. It would be undesirable if not impracticable to mount an instrument on the stack for each gas species to be monitored therefore there was a requirement for a "multi gas" or multi component" analyser.

In addition parts of the process are at an elevated pressure and therefore the need for the analyser to withstand a high process pressure was a requirement. The levels of NOx experienced were relatively high whereas this made the analytical measurement easier it introduced the potential of corrosion. It was already determined the "availability" of the measurement had to be high therefore any process "wetted" component within the analyser had to be designed to withstand the harsh environment.

The Solution

The concept was to design a system with an in-situ cell, which the process gas could freely pass through, by analysing the infra

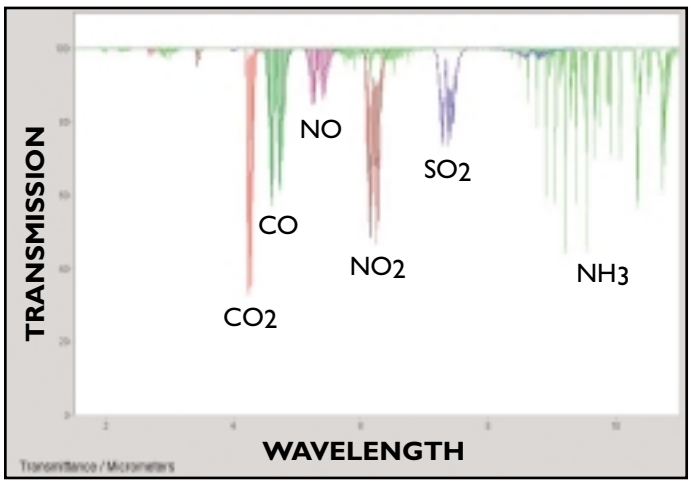


red absorption at specific wavelengths the concentration of the gas could be determined. To protect the optical surfaces from contamination by particulates it was decided to enclose the in-situ cell with a sintered stainless steel filter. With the measuring "cell" of the analyser in the process it had to be manufactured from corrosive resistant materials. This was relatively simple for the mechanical structure as it could be manufactured from stainless steel or even more exotic materials such as hastelloy or inconel. The optical components presented more of a challenge, the lens or window was manufactured from calcium fluoride (CaF2), the retro reflector from glass with an aluminium reflecting surface which was protected by a acid resistant overcoat, The secondary benefit of enclosing the measurement cell with the sintered filter was the ability to introduced compressed gas to force out the process gas and enable both zero and calibration of the analyser.

Infra red Technique

The system comprises of an infrared source, which generates infra red radiation from 2 to 12µm and a series of narrow band filters are used to select specific wavelengths.

The wavelengths are chosen are dependent on the gas species to be monitored, each gas having a separate absorption characteristic. By comparing the energy of each wavelength

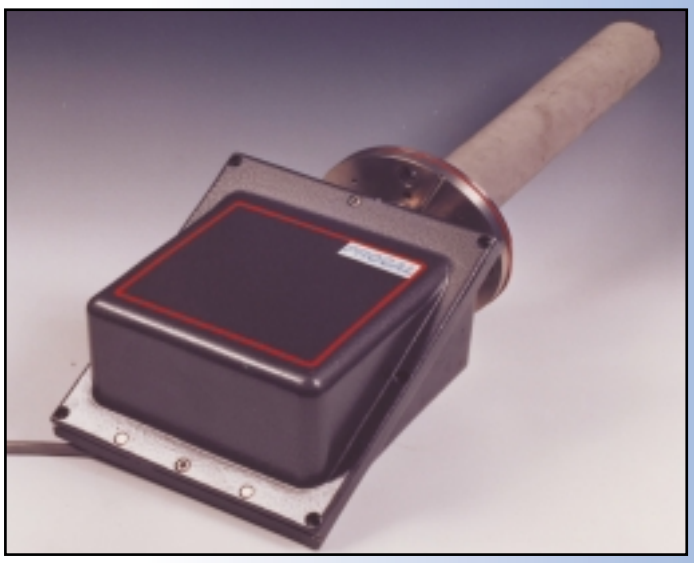


after passing through the in-situ cell the concentration of the gas can be calculated. Other factors which will affect the absorption such as path length, temperature and pressure are either fixed or measured, and automatic compensation applied. In addition due to the wide absorption characteristics of both water vapour and carbon dioxide (CO2) these gases are also monitored and a compensated for, this also gives the ability to report on a wet or dry bases dependant on the requirements of the EPA.

Early Analysers

The first analysers to be installed on Nitric Acid Plants were installed at ICI's Wilton and Severnside Plants. These were calibrated to monitor NO and NO2 Installed 1989

Range	Concentration	Gas Species
1	0 - 1,700ppm	NO
2	0 - 300ppm	NO2
3	0 - 2,000ppm	NOx

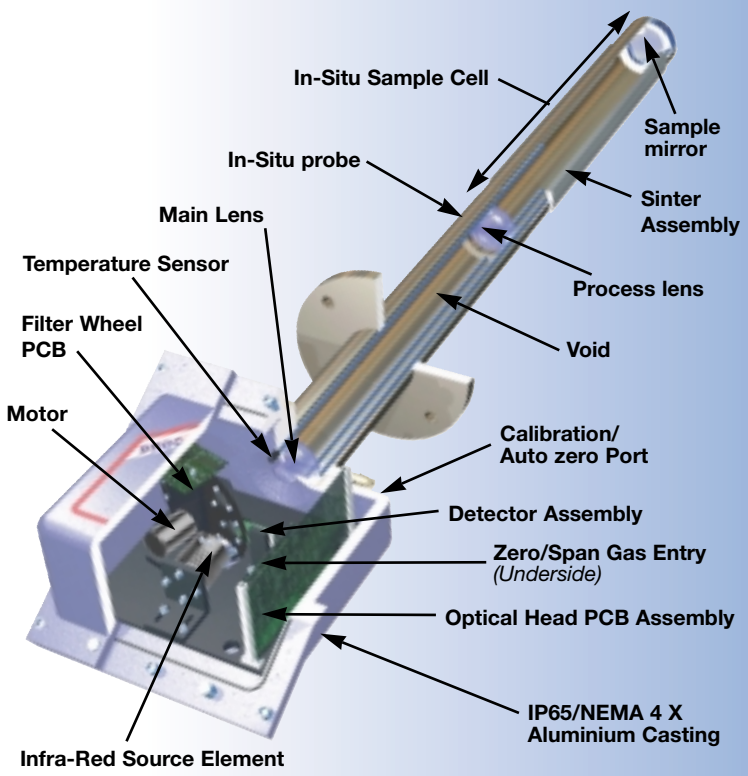


Subsequently ICI installed a further six units in 1999 to monitor higher levels, Several other analysers where installed on site in various countries to monitor NOx and NH3.

Range	Concentration	Gas Species
1	0 - 1,500ppm	NO
2	0 - 1,000ppm	NO2
3	0 - 3,000ppm	NOx

Current Configuration Introduced 1992

Whereas the analysers performed within specification it was clear that there where several improvements which could enhance the in-situ technique. The main one was associated with the analyser lens arrangement, to avoid acidic

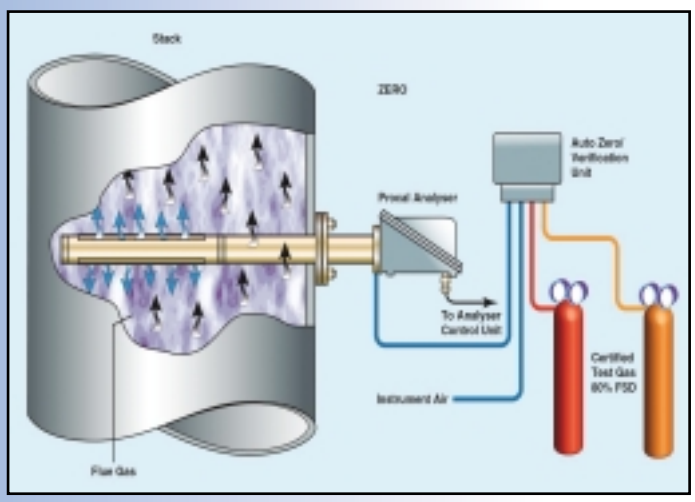


condensation forming on and damaging the lens assembly it was heated. If the heater failed or the ambient temperature was low condensation would form. This was overcome by extending the

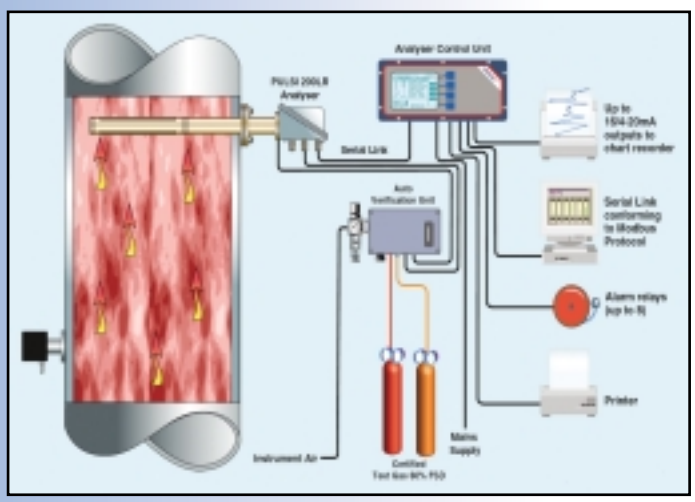
probe and mounting a second lens further out in the process which would be heated by the process. In applications where the process temperature was low and near the dew point a robust probe heater could be incorporated. In the event that the heater fails the analyser detects that the measuring cell is close to the dewpoint of the process gas and automatically applies compressed air to protect the probe.
In addition to continuously monitoring the sample temperature to compensate for pressure effects and elevated pressure applications a continuous pressure measurement and correction was introduced.
The number of gas species measured was extended from four to six including methane CH4 and CO.

Auto Zero and Calibration

To comply with regulations such as US EPA the analyser has the capability to auto zero / calibrate. This is achieved by introducing zero and span gas into the in-situ measurement cell. This excludes the process gas enabling a true zero and span to be measured and if necessary adjusted.



Display, Data Logging and Transmission



The calculated concentrations can be displayed on a PC or weatherproof industrial PC. Up to four analysers can be connected to the control system with inputs for other instruments such as opacity, velocity and oxygen. The data can be transmitted to the DCS in analogue or modbus formats.

Example Installations

Dyno Nobel (formerly Nutrite), Ontario, Canada
Four Units installed 1996

Range	Concentration	Gas Species
1	0 - 500ppm	NO
2	0 - 500ppm	NO2
3	0 - 8,000ppm	CH4
4	0 - 15%	CO2

Orica, Queensland, Australia
First Installed 2004Originally analysers were supplied for emission reporting,

Range	Concentration	Gas Species
1	0 - 500ppm	NO
2	0 - 2,500ppm	NO
3	0 - 300ppm	NO2
4	0 - 1,500ppm	NO2
5	0 - 500ppm	N2O

however systems are now also used for process efficiency, including NOx catalytic reduction unit control.

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