

An ideal tool for multiple element analysis using on-line XRF technology, from low ppm to percentage levels

# **C-QUAND** On-line XRF analysis of multiple elements in liquid process streams

# Introduction

The C-QUAND is the latest Hobré on-line XRF analyzer. The C-QUAND is an ideal on-line solution for elemental analysis in liquids. The C-QUAND is capable of measuring multiple elements, from silicon (Z=14) to uranium (Z=92), and from ppm to percentage levels. It is a continuous, non-destructive, low-maintenance analyzer without the need for additional reagents. C-QUAND also offers considerable savings in analysis time and operational costs compared to alternative analytical techniques.

# Principle of operation

The C-QUAND analyzer is designed to measure multiple elements in liquid samples. Using Energy Dispersive X-ray Fluorescence (EDXRF) technology, a well-known element measurement principle, which measures the characteristic X-rays generated by the atoms in the sample.

#### X-Ray Fluorescence principle

The C-QUAND has a powerful 15 watt, 0-50 kV X-ray source, with a silver (Ag) anode. The X-rays knocks the inner electron out of the K or L orbit of the atom, leaving a void. The now unstable atom will fill the void with an electron from the outer orbits. The difference in energy is emitted as a photon with a distinct energy level, unique to the element. The fluorescence is collected by the silicon drift detector (SDD). The detector collects these events as counts, which are directly proportional to the concentration of the element of interest.



Figure 1. X-ray fluorescence principle



Figure 2. Measuring cell

## Measuring cell

The X-rays generated by the source are first filtered by one of six different optical filters. The X-rays then travel through a thin window before hitting the actual sample. The window material can be beryllium for sulfur measurement in hydrocarbons, or a polyimide such as Kapton® or Upilex® for the measurement of metals in aqueous solutions.

The measuring cell is constantly flushed with fresh samples. Importantly, this method of measurement is continuous throughout the entire measurement, not just at the beginning. The measuring cell also contains a solid reference metal, which is used for automatic gain correction.



# Applications

- Pipeline monitoring (total sulfur & metals measurement)
- Fuel Blending (total sulfur measurement)
- Sulfur and/or metals in all Refinery processes
- Salt, metals and sulfur in crude oil
- Sulphate in injection water
- Catalyst monitoring for PTA and PIA plants

# Total sulphur measurement

The XRF method is the most suitable method for total sulphur analysis in petroleum products. Other techniques based on injection techniques and conversion methods to  $SO_2$  and  $H_2S$  will not work when the final boiling point of the product exceeds 450 °C. Such methods also experience issues with FAME in biodiesel; when the sample is heated, esterification can occur and block the injection valves.

X-ray absorption techniques are not considered due to matrix effects and their poor sensitivity in the low ppm range.

# PTA en PIA plants

The oxidation of p-xylene to produce (Purified) Terephthalic Acid (PTA) is enhanced by Co-Mn acetate and tetrabromo-ethane catalyst addition. Continuous measurement of the Co-Mn acetate and tetrabromo-ethane catalyst in PTA production enables better control of the process thus enhancing yields, improving product quality and increasing throughput.

The process conditions are challenging, with high temperatures, high solid content, possible precipitation or sample freezing, and a corrosive medium. It is therefore critical for the success of the operation to use a well-designed sample conditioning system. Hobré Instruments has extensive experience with this application.

- Metals in mining and minerals
- Fe, Ni, Zn and Sn in metal plating
- Fe, Co, Ni, Cu, S, Cl, As, Mo & PGM in metal refining/ recycling
- Cu, Ni, Mo, V, W, Fe & Co in metal recycling
- Waste water monitoring
- Metal extraction

# Metals refining & recycling

Process streams from mining, metal refining, metal recycling, or metal plating contain a mixture of elements in various concentrations and chemical forms. This is where XRF fluorescence proves to be very valuable.

Most UV-vis methods need to break down all the organometallic complexes, then make sure the metal in the solution is in the same oxidation state, and add another complexing agent, before a color can be measured.

This is all very laborious and time consuming, and needs multiple steps with different chemical reagents – it is best avoided with an on-line analysis method.

XRF on the other hand, does not require chemicals, multiple steps or any sample preparation apart from filtering. XRF fluorescence is a direct measurement at an atomic level, therefore it doesn't matter in what form the element is presented, the analyzer will measure it.

# "The C-QUAND measures elements from sillicon (Z=14) to uranium (Z=92)"

# **Turnkey solutions**

The key to success of the on-line XRF analysis is the sample system. A well-designed sample system will ensure maximum measurement availability. Hobré Instruments has unique proprietary solutions for challenging environments, such as highly corrosive liquids and samples with high particle load. Our inhouse XRF laboratory would be happy to review your specific application.

# **Relevant industries**

- Oil & Gas
- Metals & refining
- Mining & minerals
- Petrochemicals
- Polymers Environemental
- Food & pharmaceuticals
- Drilling and wells
- Steel industry
- Waste water

# Easy low-maintenance operation

## Easy Operation with Routine Usage

Fast start-up with automatic stabilization and an optional validation system ensure that the system will operate with minimal attention from the operator. Specially-coated cell windows minimize window contamination, which allows longer periods of operation without maintenance. Process trends are directly showed on the HMI display.

#### Simple Calibration

C-QUAND

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X-RAYS

The C-QUAND gives a very linear response, over a wide range of concentrations. In this example, the sulfur analyzer was calibrated using a blank and a 1000 ppm standard to measure samples of 10, 20 and 100 ppm.



#### *Figure 3. Calibration graph*

In some cases, the application requires a threepoint calibration, which can be achieved simply by injecting the sample into the cell with a syringe and verifying the spectrum.

#### Cell Cleaning / Calibration Validation

In some systems, it may be necessary to clean the cell and revalidate the calibration. The C-QUAND can be supplied with an automatic system for just this function. This system automatically, on user-defined timing, flushes the cell and introduces a known standard. By measuring this standard, the C-QUAND can automatically adjust the calibration and continue to provide accurate results without operator intervention.

#### Analysis with Minimum Maintenance

The EDXRF analysis method is a direct measurement without the need for extensive sample preparation or conversion reactions. It does not require additional chemicals or buffers that need frequent replacement. The analyzer has no moving parts that need periodic maintenance or that are likely to block.

The sample cell can operate at a temperature up to 80°C so precipitation reactions in the analyzer can be avoided. Gain adjustment and normalization are routinely done by the analyzer itself. Flushing of the sample cell, and periodic validation can be fully automated.

Therefor the C-QUAND analyzer has very low operating costs, and requires minimum operator interference or maintenance. Together with a proper designed sample conditioning system, up-time of the on-line EDXRF analyzer is high comparted to other techniques.

# Benefits & Features

- On-line analyzer
- Highly sensitive silicon drift detector (SDD)
- Continuous non-destructive analysis
- Close to zero maintenance; only cleaning of the flow cell window
- Flow cell can be removed easily
  - Analyzer data available via 4 20 mA, MODBUS TCP/IP or MODBUS RS485
  - Analysis up to 15 elements possible with a single analyzer
  - Linear results from ppm to percentage levels
- Automatic drift correction

# Technical Specifications

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ANALYTICAL	
Measurement principle	Energy dispersive X-ray fluorescence
Element range	Si to U (Z=14 to 92)
Measurement range	From 0.5 ppm to % levels (application specific)
Simultaneous elements	up to 15 elements
Filter wheel	6 filters
Accuracy	Appropriate to application
Calibration	Less than 5 samples
CENTRAL PROCESSING AND CONTROLLING UNIT	
Industrial PC	Linux operating system, custom touch keyboard with 8" TFT color display
Analog outputs	2 or 4 x 4-20mA active
Communications	MODBUS via RS485 or TCP/IP
Ambient temperature	5 – 40°C
MEASUREMENT HEAD	
X-ray detector	
HV power supply	0-50kV
Source	
Stability	Automatic drift and back scatter peak correction; temperature and ambient pressure correction
Sample window	Kapton <sup>®</sup> , Upilex <sup>®</sup> , beryllium
UTILITY REQUIREMENTS	
Power supply	
Power consumption	100 VA (150 VA with heated sample cell)
SAMPLE CONDITIONS	
Sample flow rate	
	< 80°C
	Aunospheric urain
VISCOSILY	Less than 100 CSt at cell temperature (Heated sample cell available)
Contaminants	Sample to be liftered < to micron and free water removed from hydrocarbon streams
GENERAL	
Size	HxWxD = 1000 x 400 x 360 mm
Valve contr <u>ol</u>	Automatic stream switching, automatic cell flushing, automatic validation
	Liquid Sample Recovery Control done by analyser (optional)
Inputs	Flow and level alarms (optionally) read by analyser
Diagnostics	High level of diagnostics
Remote supervision/control	External control of analyser possible
Hazardous Area Clasification	Exp. (pending)

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HOBRÉ INSTRUMENTS

HOBRÉ IS A LEADER IN THE DESIGN, MANUFACTURING AND MAINTENANCE OF ONLINE ANALYZERS, SAMPLE SYSTEMS AND COMPLETE TURNKEY ANA-LYZER SYSTEMS. ESTABLISHED IN 1978, OUR COMPANY FOCU-SES ON PROVIDING SOLUTIONS FOR THE OIL AND GAS, (PETRO)-CHEMICAL, POWER GENERA-TION, STEEL AND FOOD AND PHARMA INDUSTRIES WORLD-WIDE.

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HOBRÉ INSTRUMENTS NETWERK 4 1446 WK PURMEREND THE NETHERLANDS TELEPHONE +31 299 420 871 TELEFAX +31 299 423 302

INFO@HOBRE.COM WWW.HOBRE.COM

