

SULFUR DIOXIDE IN SOLVENT NUMBER 3991

The AMETEK 682T-HP is designed for analysis of sulfur in highly viscous hydrocarbons. This system is ideal for processes where sample pressures are high and in situations where fouling of flowcell windows with paraffin or similar substances can occur.

The 682T-HP system is fast, sensitive and compact, providing continuous, reliable detection of sulfur at pressures up to 800 psi. It can operate either as a standalone analyzer or can be tied in to plant-wide automation systems to provide real-time strategic measurements.

Among the key features of the analyzer are a fully integrated densitometer, automatic density compensation, touchscreen controls, password-protected screens, and rapid update rates (as frequent as one per every five seconds).

Operation of the analyzer is regulated by an integrated processor, which monitors instrument parameters such as process temperature, density, and system diagnostics. It also provides a comprehensive suite of alarms and a standard platform for communicating to a plant-wide distributed control system (DCS). Due to its unique design and robust construction, the system has minimal, if any, sample conditioning and recovery requirements.

XRT ON-LINE HIGH-PRESSURE SYSTEM

In the cross-section diagram (Figure 1), the sample flows through a flowcell and is irradiated with a source of high-energy X-rays which are absorbed by the sulfur atoms. A detector on the opposite side measures the X-rays that pass through. That signal intensity is inversely proportional to the sulfur concentration.

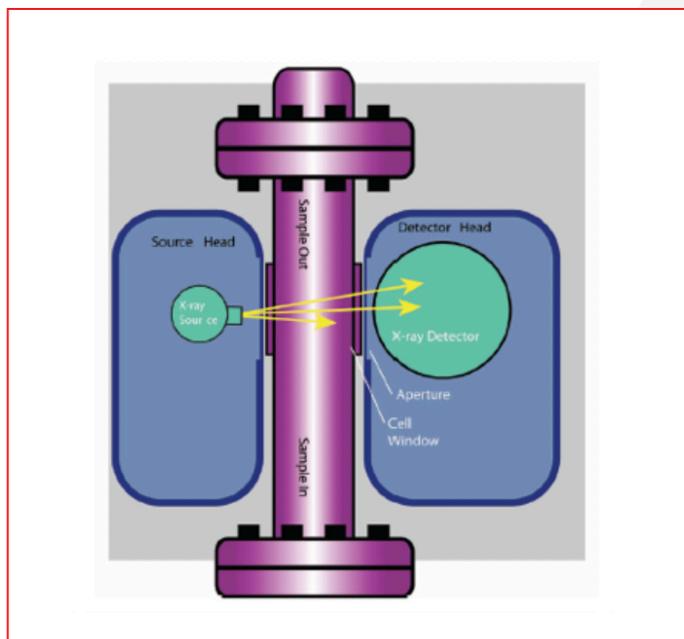


Figure 1. Flowcell cross section diagram

THE STUDY

Objective

Data collected during this study will demonstrate the calibration accuracy and instrument precision of the 682T-HP. This data is from the analysis of sulfur dioxide (SO₂) in solvent.

Background

The analysis of sulfur in petroleum products can be achieved using simple sample preparation and non-destructive analyses. The type of samples analyzed here are petroleum-based solvents, where the amount of SO₂ in the solvent needs to be determined.

Sample preparation

The sample was poured into a bucket marked with the appropriate volume for the analyzer and then poured into the sample inlet of the 682T-HP. The valves were closed and the analyzer results were logged for at least one hour. The sample was then emptied into a bucket and poured back into the original container.

682T-HP calibration results

Based on first pour results

Element: SO ₂		
Units: %	Std. Error of Estimate: 0.009	
Sample	Measured %	Given %
0.602 pour 1	0.6051	0.602
0.358 pour 1	0.3504	0.358
0.167 pour 1	0.1714	0.167

Figure 2. Measurement results

Precision demonstration

Element: SO ₂		Units: %		
Sample	Given	Mean	Std. Dev.	# Samples
.602 pour 1	0.602	0.6051	0.0133	69
.358 pour 1	0.358	0.3504	0.0079	60
.167 pour 1	0.167	0.1714	0.0077	58

Figure 3. The instrument performance shown here is based on 100 seconds per sample. Performance can be improved in several ways, such as increasing the accumulated detector counts or averaging results. Analyzing for a longer period or averaging results can increase detector counts and overall performance

Minimum detection limit

The Minimum Detection Limit (MDL) is defined as three times the standard deviation of analyzing the blank sample at least ten times in a static position. No blank was available for this study, but the 682T-HP has a MDL in similar petroleum-based products as indicated here.

Element	MDL
S	0.02%

Figure 4. Minimum detection limit

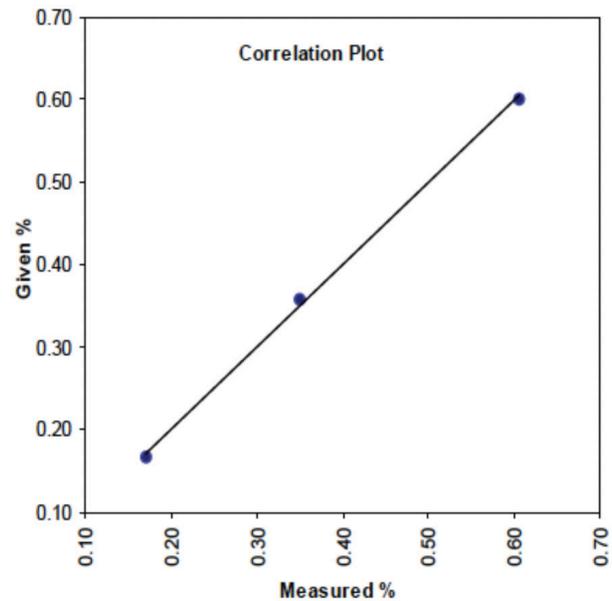


Figure 5. Correlation plot, showing best fit line

DISCUSSION

For these types of applications, a 682T-HP analyzer will provide excellent accuracy and stability. It should be noted that considerable SO₂ escaped from each sample during the pour process. Bubbles were evident in the bucket after pouring the sample and a sharp odor was noticeable until dissipated by ventilation. Subsequent measurements for each sample had consistently lower recorded SO₂ sample.

The nature of the samples may make calibration difficult as the SO₂ content appears to decrease rapidly when the sample container is open to the atmosphere. Despite this property, accurate calibrations seem possible if proper care is taken to minimize the sample exposure to atmosphere during the calibration process. The analysis of the process flow should not have this problem because it is expected to be a closed system.

An alternate method for calibration may be preferable if the calibration samples can be made with a non-volatile sulfur mixture in the same solvent matrix.

CONCLUSION

The data shows the 682T-HP analyzer can easily distinguish SO₂ content in solvent. Repeatability is excellent if the sample is not open to the atmosphere.

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