

## Using the Extrel<sup>®</sup> MAX300-IG<sup>™</sup>

# Process Mass Spectrometer to Monitor Vacuum Oxygen Decarburization

Extrel CMS  
Application Note  
Steel VOD  
IAV100B

### Abstract:

Vacuum Oxygen Decarburization (VOD) involves the use of an oxygen flow to remove carbon during the production of high quality steel. The stream of effluent gases that is generated by VOD contains the oxidized carbon leaving the molten metal, and process control models can use the composition of the off gas to ensure that production is occurring as efficiently as possible. Quantitative, real-time analysis of the vessel exhaust allows for tighter control than is possible with slower technologies, making stand-alone mass spectrometry particularly useful for monitoring VOD.

An Extrel MAX300-IG was used to demonstrate the measurement speed and repeatability of a quadrupole mass spectrometer in a decarburization application and to calculate  $\Delta C$ , a critical control parameter.

## Vacuum Oxygen Decarburization Process Modeling

VOD is a proven method for converting low quality, high carbon, scrap and iron into marketable, high quality steel. Oxygen is allowed to flow over the surface of the molten bath causing the oxidation of carbon which leaves the liquid as gaseous carbon monoxide and carbon dioxide. The process takes place in a closed vessel and the melt is stirred using a flow of nitrogen or, more commonly, argon. VOD is carried out under vacuum to prevent the additional oxidation of metallic constituents, like chromium, that are important in the final alloy.

The decarburization process is controlled via one of several models that indicate the proper setting of variables including oxygen flow rate, lance position, and stirring intensity. The benefits of efficient process control include: reduced consumption of oxygen and stir gas, better

chromium retention, shorter treatment time, and less wear on the converter and other equipment. Ensuring the proper carbon content at the end of decarburization eliminates the possibility of having to reblow the batch.

Many VOD modeling strategies rely on dynamic parameters obtained from online process monitoring to allow for adjustment to operating variables in response to changing conditions within the bath. The carbon composition of the gas mixture exiting the vessel is one of the most useful indicators of the amount of carbon remaining in the product, making fast, precise, gas analysis a critical contributor to efficient process monitoring and control.

The ability to measure hydrogen, nitrogen, oxygen, argon, carbon monoxide, and carbon dioxide across a wide range of concentrations is important for any gas analysis system used to monitor VOD, and the speed, flexibility, and dynamic range of a quadrupole mass spectrometer allows for a faster reaction to process upsets, and tighter, more efficient control than technologies with slower analysis cycles.

From the quantitative measurement of carbon dioxide and carbon monoxide in the vessel's effluent the manufacturer can identify critical carbon, the point at which the flow of oxygen should be reduced. These data are also used to determine the amount of carbon that has left the batch during a given analysis cycle. This value, known as  $\Delta C$ , reveals the rate of decarburization, and can be used to control the carbon content of the end product.  $\Delta C$  for each analysis cycle is calculated using the formula:

$$\Delta C = \int_{t-\Delta t}^t \frac{dC}{dt} dt \approx Q \times \Delta t \times (k_1 CO\% + k_2 CO_2\%)$$

Where  $k_1$  and  $k_2$  are mass conversion factors for carbon monoxide and carbon dioxide, respectively, and  $Q$  is total mass flow.

Flow measurements of the stir gas combined with the compound's concentration from the mass spectrometer indicate total gas flow leaving the system. As the mixture composition shifts over time, fast gas analysis, carried out with high precision, provides the most accurate value for  $\Delta C$ .

### The MAX300-IG

The MAX300-IG is a 7th generation process mass spectrometer capable of performing quantitative analysis on a wide variety of compounds at concentrations ranging from 100 % down to 10 ppb. The 19 mm quadrupole mass filter used by the system allows for increased sample throughput, generating high analytical repeatability and long term stability.



Sample gas is pumped to the mass spectrometer from the VOD vessel. As the vacuum increases with each stage of the process, automated control of the sample pressure within the MAX300's ionizer ensures that, as the pressure above the melt drops, there is no change to the instrument's sensitivity.

**Figure 1.**  
The MAX300-IG Quadrupole Mass Spectrometer for Industrial Gas Analysis and Process Control Automation

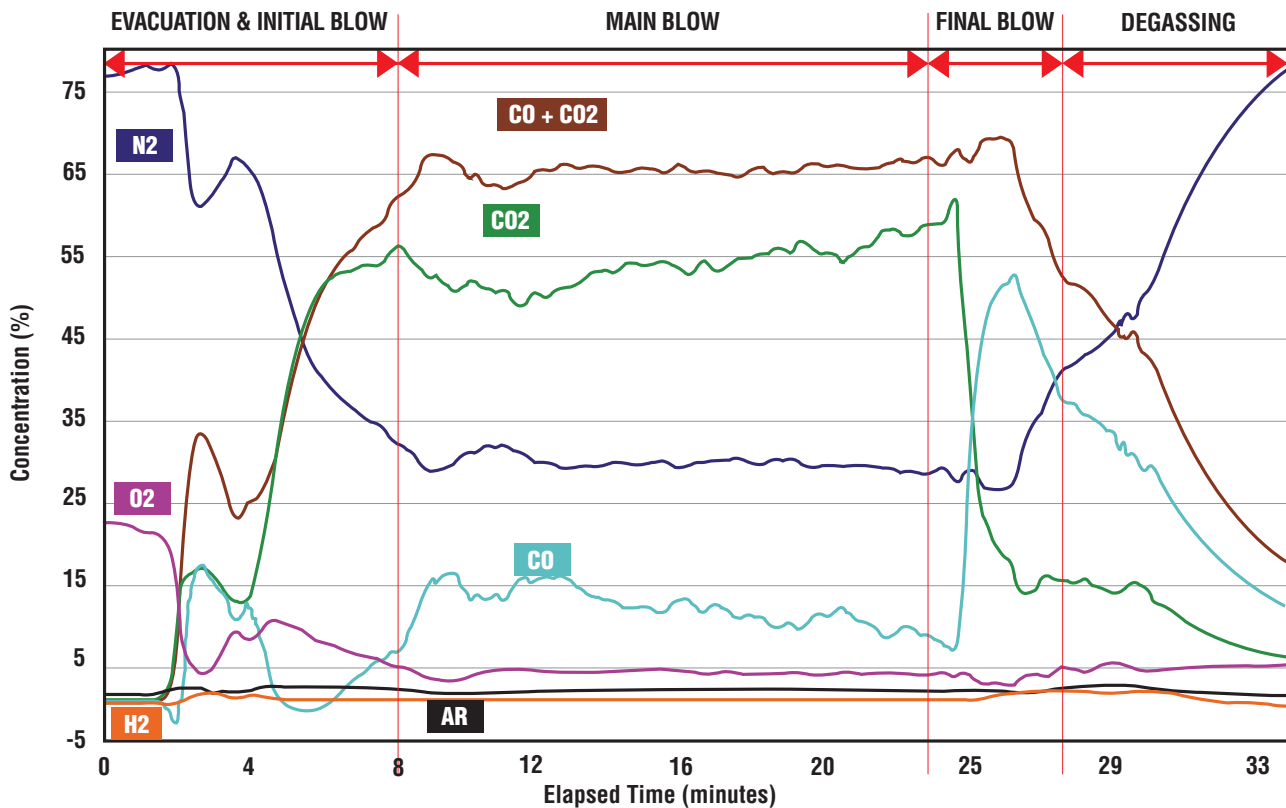
# Real-Time Analysis of VOD

An analysis method was created to monitor the components of the effluent stream leaving the VOD vessel (Fig. 2).

The Questor 5 software platform that runs the MAX300 performs its quantitative analysis at a rate of 400 msec per component. This means that the analysis cycle for the measurement of the eight compounds present in the VOD application is <3.2 seconds. At that speed the mass spectrometer was able to record the dynamic profile of the reactor exhaust during a VOD treatment (Fig. 3). The changes that occur provide important information about what stage the process is in and when the batch has reached critical carbon. From this information the control room can make real-time adjustments to oxygen flow, stirring, or lance position.

Fragment Matrix									
Add Chemicals	Sensitivity	Relative Abundance							
		m/z 2	m/z 4	m/z 12	m/z 14	m/z 18	m/z 32	m/z 40	m/z 44
<input checked="" type="checkbox"/> H2	.175	100							
<input checked="" type="checkbox"/> HE	.040		100						
<input checked="" type="checkbox"/> CO	1.000			2.69	0.79				
<input checked="" type="checkbox"/> N2	1.000				5				
<input checked="" type="checkbox"/> H2O	.800					100			
<input checked="" type="checkbox"/> O2	.980						100		
<input checked="" type="checkbox"/> AR	1.500							100	
<input checked="" type="checkbox"/> CO2	1.860			3.8					100
<b>Background intensity:</b>		.000000	.000000	.000000	.000000	.000000	.000000	.000000	.000000
<b>Detector:</b>		Farad	Farad	Farad	Farad	Farad	Farad	Farad	Farad
<b>Ion Repeat:</b>		1	1	1	1	1	1	1	1

**Figure 2.** The MAX300-IG VOD analysis method containing the list of analytes, detection masses, and calibration values. This image is a screen capture of the Questor 5 Software.



**Figure 3.** The composition profile of the off gas generated during a VOD treatment. 637 analysis cycles were recorded over the course of the 34 minute run. Shifting concentrations within the sample indicate the onset of each stage of the process:

- A. Evacuation and Initial Blow:** Nitrogen is displaced as the vessel is pumped down and oxygen is introduced.
- B. Main Blow:** The oxygen flow is maximized producing a steady out gassing of carbon dioxide and carbon monoxide.
- C. Final Blow:** The drop in the CO+CO2 trend indicates the onset of critical carbon, oxygen flow is reduced and vacuum is increased.
- D. Degassing:** The oxygen is stopped and the vessel reaches its ultimate vacuum, driving out dissolved gases like nitrogen, argon and hydrogen.

## VOD Data Precision

Next, the analysis of a stable mixture of VOD gases was carried out to determine the repeatability of the concentration values and the effect of instrument precision on calculated control parameters (Table 1).

The precision of the MAX300-IG data was shown to be quite good, with the repeatability of most components falling in the low ppm range. As the stability study was necessarily carried out using a gas blend instead of vessel off gas there is no mass flow data for the calculation of  $\Delta C$ . In the absence

of a flow controller measurement, however, the impact of the variability of the carbon dioxide and carbon monoxide measurement on the calculated control parameter can still be considered, with the data collected indicating a value for  $\Delta C/Q = 51.2266 \pm 0.0176$  seconds. The high precision analysis of carbon concentrations ensures a high precision for the  $\Delta C$  value.

**Table 1.**

Analysis data from the measurement of a certified gas blend designed to provide a stable version of a typical VOD effluent mixture. Each concentration represents the average of 5300 samples recorded over 4 hours and 42 minutes. Analytical precision is reported in both relative standard deviation (RSD) and standard deviation (STD).

Name	Concentration (%)	RSD (%)	STD (ppm)
Hydrogen	0.99	0.18	18
Helium	3.31	0.11	36
Carbon Monoxide	19.98	0.06	124
Nitrogen	35.40	0.02	70
Water	0.50	0.14	7
Oxygen	2.99	0.03	9
Argon	10.00	0.02	18
Carbon Dioxide	27.30	0.02	54

## Conclusion

Dynamic control models for VOD rely on fast, high precision, gas analysis data to maximize process efficiency. The MAX300-IG measured all of the components of the reactor off gas and provided each analysis cycle to the DCS in <3.2 seconds. The analyzer was able to track the full range of all components as concentrations shifted significantly

during the run, revealing the onset of each stage in real-time. In addition, an analysis of a stable mixture of VOD gases indicated that the precision of the measurement, and of the calculated parameter,  $\Delta C$ , is sufficiently high to ensure that the MAX300-IG functions as a valuable tool for VOD process control and efficiency.

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