FIRST ● Newsletter

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Advancing Carbon and Sulfur Assessment: Combustion Analysis with Minimal Furnace Maintenance

By Dr. Eric S. Oxley, Product Manager GA, Bruker Elemental, Billerica, MA USA

Carbon and Sulfur Content… Why Care?

The elemental content of solid materials is well-known to have an influence on its physical properties. For example, carbon and sulfur, shown in powder form in Figure 1, can have a positive or negative influence. The trending nature of these influences is summarized in Table 1, showing in general that carbon has a positive influence and sulfur an adverse impact. For these reasons many production facilities of metals, minerals and other inorganic compounds place emphasis on assessing and controlling the carbon and sulfur content from raw material to finished product.

Figure 1. Carbon (left) and sulfur (right) can influence the properties of many inorganic materials, both positively and negatively.

These factors have allowed the instrumentation for measuring carbon and sulfur to become ubiquitous in the aforementioned industries, as well analytical testing laboratories and other sectors. Based on combustion of the sample material and infrared detection of the combusted products, the method is rapid, reliable and sensitive to even low concentrations of carbon and sulfur (e.g., ppm). With these figures of merit, it's certainly no surprise this technique has become universally accepted as "the standard" for carbon and sulfur assessment in metals.

While proven to be very efficient at converting solid material into its detectable gaseous constituents, combustion of material produces undesirable byproducts. Over time and after many sample analyses these byproducts (e.g., metal oxide dust) will accumulate in the combustion furnace area of the instrument if the dust is not filtered and eventually removed. Erratic results, or even component failure, can result if not addressed. Disassembly of the furnace and manual cleaning of components is often required, resulting in unfortunate instrument downtime and cumbersome regular maintenance procedures. If only the combustion-based instrumentation could incorporate methods to minimize, or even eliminate, this maintenance downtime…

Table 1. General influencing trends of carbon and sulfur on the physical properties ofmetals.

Physical Property	Carbon Influence	Sulfur Influence
Tensile Strength	Strongly increasing	Slightly reducing
Hardness	Strongly increasing	Slightly increasing
Strain	Slightly reducing	Strongly reducing
Stretching Limit	Strongly increasing	No effect
Notch Impact Strength	Slightly reducing	Strongly reducing
Long-term Strength	Strongly increasing	Strongly reducing
Thermal Conductivity	Slightly reducing	Strongly reducing
Electrical Conductivity	Slightly reducing	Strongly reducing
Wear Resistance	Strongly increasing	Slightly increasing
Cold Workability	Strongly increasing	Strongly reducing
Hot Formability	Slightly reducing	Strongly reducing
Cutting Quality	Strongly increasing	Slightly increasing
Corrosion Resistance	Slightly reducing	Slightly reducing

To circumvent these maintenance issues, Bruker has incorporated a new feature on their proven **G4 ICARUS CS HF** carbon and sulfur analyzer that provides a virtually maintenance-free furnace. This

G4 ICARUS, already providing features that reduce general maintenance requirements, now includes a new pneumatically-assisted cleaning assembly that can scrub and dispose of these interfering combustion byproducts. It's actually the degree of cleaning efficiency and subsequent disposal of these byproducts that separates it from other designs.

Advancing Carbon and Sulfur Assessment
So what is the operating principle of the **G4 ICARUS CS HF** (Figure 2) and, more importantly, how does this new furnace "autocleaning" feature work?

The **G4 ICARUS** converts the solid sample of interest into gaseous components which are measured by infrared detectors and processed into tangible carbon and sulfur concentrations. Separating itself from other commercially-available combustion analyzers, the **G4 ICARUS** has many unique and beneficial features.

*Figure 2. The Bruker **G4 ICARUS CS HF** carbon and sulfur analyzer with automatic cleaning.*

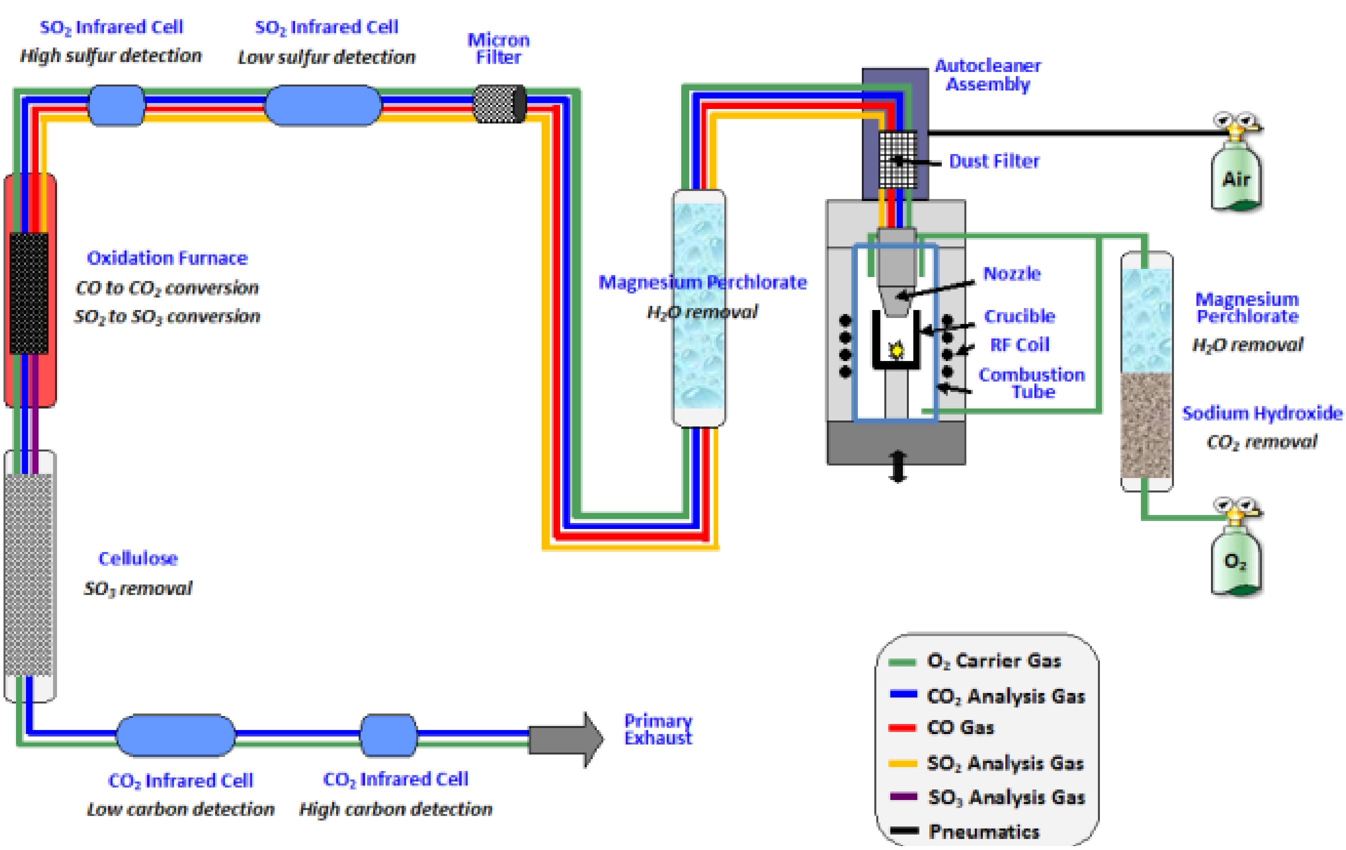

*Figure 3. Block diagram representation of the **G4 ICARUS CS HF** showing primary components and the analytical flow path.*

Combustion Process

The **G4 ICARUS** utilizes a high-frequency (HF) induction furnace, as represented in the block diagram of Figure 3, to rapidly combust solid samples. The sample, typically up to 1g depending on the application, can be analyzed directly with the **G4 ICARUS** regardless of configuration. Shapes such as powders, pins, chips, drillings, and many others are possible. The sample is placed in a ceramic crucible along with a material (i.e., accelerator) that will readily couple with the electromagnetic field produced by an RF coil that surrounds the crucible. The accelerator is necessary to ensure a good coupling with the electromagnetic field and to provide additional energy to the combustion process so that a fluid melt is achieved. Accelerators are chosen based on the sample application and include most commonly: tungsten, copper, iron, and tin. Though many applications are possible on the **G4 ICARUS**, Table 2 lists some common ones along with the corresponding accelerator recommendation.

*Table 2. A few of the many **G4 ICARUS** sample applications and their associated mass and accelerator recommendations.*

Material	Sample Mass (g)*	Accelerator**
Steel	0.500	1 scp W
Stainless Steel	0.500	1 scp W
Copper / alloys	0.250 – 0.500	1 scp W
Brass / bronze	0.250 – 0.500	1 scp W
Aluminum	Varies	1 scp W
Carbides (W/Si)	0.100 – 0.25	1scp W + 2scps Cu
FeSi	0.100 – 0.200	1scp W + Sn + 1scp Fe
FeCr	0.250 – 0.500	2 scps W
Slag	0.200	1scp W + Sn + 1scp Fe
Cement	0.100 – 0.150	1 scp W
Oxides / Sulfides	0.005 – 0.050	1scp W + Sn + 1scp Fe
Ceramics	Varies	1scp W + Sn + 1scp Fe

**Nominal sample mass. May be reduced if carbon and sulfur content is especially high.*

***1 scp W = ~1.5g tungsten; 1 scp Cu = ~0.8g copper; 1 scp Fe = ~1g iron chips; 1 scp Sn = ~0.4g tin*

By providing a high pressure, oxygen-rich (O_2) environment in the furnace, the sample material and accelerator combust reaching temperatures above 1500°C while liberated carbon and sulfur compounds are oxidized to form carbon dioxide (CO_2) and sulfur dioxide (SO_2), respectively. A small amount of carbon monoxide (CO) can also be produced, depending on the concentration of carbon in the sample. These liberated gaseous constituents are represented as colored lines in the block diagram of Figure 3.The critical components of the **G4 ICARUS**, including the furnace area, purifying reagents and infrared (IR) detectors, are also represented in this figure.

Furnace Viewing Port

One of the unique benefits provided with the **G4 ICARUS** is its incorporation of a viewing port on the front of the furnace. This handy addition, shown in Figure 4, allows the efficiency of the combustion process to be monitored in real-time. Even better, the integrity of the combustion tube can be monitored.

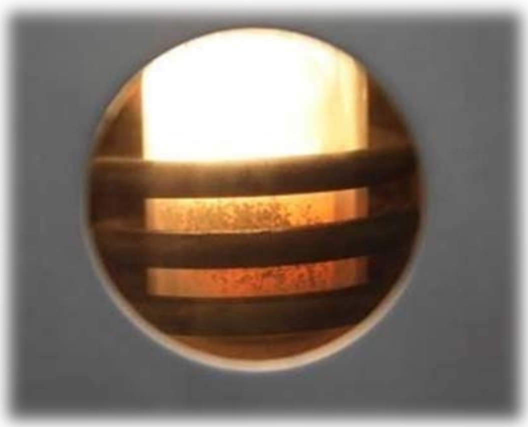

*Figure 4. Combustion viewing port found on the front of the **G4 ICARUS** analyzer.*

Extraction Nozzle

The liberated sample gases must be extracted from the furnace and transported downstream for eventual detection (Figure 3). The transport mechanism inherent with the Bruker **G4 ICARUS HF** analyzer provides many tangible benefits for its users. By utilizing the high pressure in the furnace and an "extraction" nozzle located directly above the crucible, the gaseous components can be efficiently extracted from the furnace with minimal dilution from the oxygen carrier gas. The components comprising the novel furnace design are shown in Figure 5. Note specifically the extraction nozzle and its slight immersion in the top

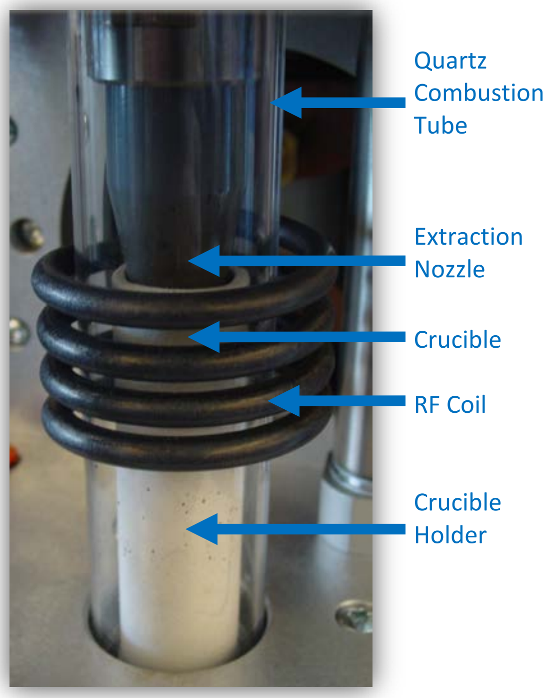

*Figure 5. Furnace of the **G4 ICARUS HF** analyzer featuring a patent-pending extraction design.*

of the ceramic crucible. Destructive byproducts that can splatter against a surrounding quartz combustion tube (Figure 5) with traditional furnace designs are instead extracted by the nozzle with this new, unique design. Users benefit from limited dust contamination with improved analytical results and ultimately, extended quartz tube lifetimes. The extraction nozzle also eliminates the need for a lance, which is a narrow oxygen-supplying orifice located above the crucible (i.e., in lieu of the large-diameter nozzle of the **G4 ICARUS**) that can become clogged with splatter from the combustion process. Further, the efficient extraction of the sample gases with the **G4 ICARUS** improves analysis times and measurement sensitivities compared to traditional, diffusion-dominated furnace purge designs.

Furnace Autocleaner

The chief, recent addition to the original design of the **G4 ICARUS**, as described in the introductory section, is a cleaning device (Figure 6) that efficiently removes byproducts from the combustion process. This time-saving device compromises a pneumatically driven piston with integrated components that effectively clean the furnace area, including a metallic dust filter (i.e., "Dust Filter" in Figure 3) and the aforementioned extraction nozzle. The completion of each analysis invokes a downward stroke of the piston that performs said cleaning.

One of the most impressive features is what happens to byproducts liberated by the cleaning cycle. Dust is wiped from the metallic filter and splattering particles are removed from the inner rim of the extraction nozzle by various blades on the plunger of the cleaning mechanism. Supported by a small pulse of oxygen flow the particles are efficiently transported into the spent crucible. So the byproducts are tossed when the crucible, with combusted sample material, is disposed. No longer required is abrasive cleaning of the quartz tube by brushes and a noisy, space-consuming vacuum cleaner required. The efficiency at which the byproducts are removed from the furnace in the **G4 ICARUS** allows the user to typically go hundreds of analyses before disassembly and manual cleaning of the furnace is required! And this automatic cleaning device is included as standard on all **G4 ICARUS** analyzers.

Gas Purification and Flow Control

The combusted gas stream exiting the furnace area, as represented in Figure 3, is purified by directing it through a drying reagent, magnesium perchlorate, to remove any moisture that may have been produced or released during combustion. Failure to remove this moisture could result in the formation of sulfuric acid through combination with the SO_2 that is present in the stream. This would not only

Figure 6. New furnace autocleaning feature found standard with the ***G4 ICARUS****.*

lead to sulfur recovery losses, but would also prove detrimental to the fittings and components within the analyzer.

Also located downstream of the furnace are pressure and flow regulating components (not represented in Figure 3). These ensure consistent combustion, transport and detection is achieved from one analysis to the next. The analyzer can also be checked for undesirable atmospheric leaks through incorporation of these components (i.e., leak check).

Infrared Detection

The purified gas stream, consisting of O_2, CO_2, SO_2 and possibly small amounts of CO, is now ready to be quantified. This quantification is achieved by using selective and stable non-dispersive infrared (NDIR) detectors found in the **G4 ICARUS.**

The gases are directed through an infrared detector that will respond exclusively to the amount of SO_2 in the stream. Upon exiting this detector the gas stream flows through a heated oxidation furnace filled with platinized silica ($PtSiO_2$). This furnace will catalytically oxidize CO to CO_2 and convert some of the previously-detected SO_2 to sulfur trioxide (SO_3). Because carbon content in the sample is assessed with a selective CO_2 IR detector, this CO to CO_2 oxidation process ensures a representative quantification of total carbon content with no losses as a result of CO. Passing the sample stream through a cellulose-filled trap will scrub SO_3 to protect downstream components. The gas stream, now comprising only O_2, CO_2, and possibly a small amount of non-oxidized SO_2, flows through a selective CO_2 cell to measure the carbon content before exiting through the exhaust.

The analysis time for a single analysis with the Bruker **G4 ICARUS CS HF** is nominally 40 seconds depending on the sample application, sample mass and carbon/sulfur concentration.

Representative Data

Now that the beneficial features of the **G4 ICARUS CS HF** have been highlighted, including the new autocleaner, what follows are examples of representative data collected on this analyzer for a range of sample applications. This data was extracted from Application Notes, which are comprehensive guidelines that Bruker provides to its users to ensure the best possible data is achieved with their Bruker analyzer(s). Of course, application development is also provided to customers during instrument installation and training.

*Table 2. Data collected on the **G4 ICARUS CS HF** by analyzing two different steel samples.*

Name	Mass (g)	Accelerator*	Carbon (%)	Sulfur (%)
Steel 1	0.8314	1 scp W	0.02709	0.00036
Steel 1	0.9050	1 scp W	0.02736	0.00046
Steel 1	0.8738	1 scp W	0.02750	0.00042
		Average:	**0.02732**	**0.00041**
		Std. Deviation:	**0.00021**	**0.00005**
		%RSD:	**0.76**	**12.18**
Steel 2	0.9884	1 scp W	0.02822	0.00051
Steel 2	0.8426	1 scp W	0.02820	0.00050
Steel 2	0.7666	1 scp W	0.02813	0.00047
		Average:	**0.02818**	**0.00049**
		Std. Deviation:	**0.00005**	**0.00002**
		%RSD:	**0.17**	**4.22**

**1 scp W = ~1.5g tungsten*

*Table 3. Data collected on the **G4 ICARUS CS HF** by analyzing two different slag-based samples.*

Name	Mass (g)	Accelerator*	Carbon (%)	Sulfur (%)
Slag 1	0.2018	1 scp W / 1 scp Fe	0.43137	0.26205
Slag 1	0.1172	1 scp W / 1 scp Fe	0.42270	0.26101
Slag 1	0.2018	1 scp W / 1 scp Fe	0.42784	0.27173
		Average:	**0.42730**	**0.26493**
		Std. Deviation:	**0.00436**	**0.00591**
		%RSD:	**1.02**	**2.23**
Slag 2	0.1998	1 scp W / 1 scp Fe	0.05509	0.28462
Slag 2	0.2054	1 scp W / 1 scp Fe	0.05509	0.28291
Slag 2	0.2024	1 scp W / 1 scp Fe	0.05650	0.28127
		Average:	**0.05556**	**0.28293**
		Std. Deviation:	**0.00081**	**0.00168**
		%RSD:	**1.47**	**0.59**

**1 scp W = ~1.5g tungsten; 1 scp Fe = ~1g iron chips*

*Table 4. Data collected on the **G4 ICARUS CS HF** from cast iron sample analyses.*

Name	Mass (g)	Accelerator*	Carbon (%)	Sulfur (%)
Cast Fe	0.2518	1 scp W / 1 scp Fe	1.312	0.0192
Cast Fe	0.2543	1 scp W / 1 scp Fe	1.312	0.0189
Cast Fe	0.2540	1 scp W / 1 scp Fe	1.304	0.0163
Cast Fe	0.2548	1 scp W / 1 scp Fe	1.315	0.0163
Cast Fe	0.2524	1 scp W / 1 scp Fe	1.314	0.0183
Cast Fe	0.2512	1 scp W / 1 scp Fe	1.296	0.0172
Cast Fe	0.2555	1 scp W / 1 scp Fe	1.301	0.0173
Cast Fe	0.2515	1 scp W / 1 scp Fe	1.304	0.0174
Cast Fe	0.2540	1 scp W / 1 scp Fe	1.301	0.0164
Cast Fe	0.2564	1 scp W / 1 scp Fe	1.305	0.0188
		Average:	**1.306**	**0.0176**
		Std. Deviation:	**0.0064**	**0.0011**
		%RSD:	**0.49**	**6.36**

**1 scp W = ~1.5g tungsten; 1 scp Fe = ~1g iron chips*

*Table 5. Data collected on the **G4 ICARUS CS HF** from silicon carbide sample analyses.*

Name	Mass (g)	Accelerator*	Carbon (%)	Sulfur (%)
SiC	0.02575	1 scp W / 1 scp Fe	29.39	0.0690
SiC	0.02762	1 scp W / 1 scp Fe	29.68	0.0651
SiC	0.02820	1 scp W / 1 scp Fe	29.46	0.0738
SiC	0.02532	1 scp W / 1 scp Fe	29.66	0.0664
SiC	0.02535	1 scp W / 1 scp Fe	29.52	0.0676
SiC	0.02514	1 scp W / 1 scp Fe	29.45	0.0772
SiC	0.02587	1 scp W / 1 scp Fe	29.61	0.0759
SiC	0.02597	1 scp W / 1 scp Fe	29.53	0.0758
SiC	0.02642	1 scp W / 1 scp Fe	29.58	0.0756
SiC	0.02700	1 scp W / 1 scp Fe	29.51	0.0706
		Average:	**29.54**	**0.0717**
		Std. Deviation:	**0.0941**	**0.0045**
		%RSD:	**0.32**	**6.26**

**1 scp W = ~1.5g tungsten; 1 scp Fe = ~1g iron chips*

Bruker Elemental
40 Manning Road
Billerica, MA 01821 USA
+1 (978) 663-3660
info@bruker-axs.com
www.bruker-axs.com