WASSON·ECE INSTRUMENTATION
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Eclipse Process Gas Chromatographs:
Maximizing efficiencies in monomer process control

Competition in the monomer market is increasingly fierce and the quality of the final product is critical to maintaining and increasing market share. Premium product requires sophisticated process monitoring and control at every stage of development. Eclipse process gas chromatographs couple MS, VUV, PDHID, TCD and FID detector combinations to characterize an extremely broad range of analytes from fixed gases to C20+. These sensitive analyzers can quickly detect ppm-ppb levels of impurities and catalyst poisons; ultimately improving product quality and saving time and money.

Capillary Chromatography in an Online Gas Chromatograph: Eclipse process gas chromatographs (PGCs) are the most sensitive and capable online analyzers available to the hydrocarbon processing industry. Our patented micro-convection ovens (MCOs, Fig. 1) enable the precise temperature controls required for capillary column chromatography. Low sensitivity analysis (ppm to ppb) is achieved by combining the precision of the MCOs with electronic pressure programming. Interfacing between the Eclipse PGC and the plant data communication systems allows for rapid responses to changing stream conditions.

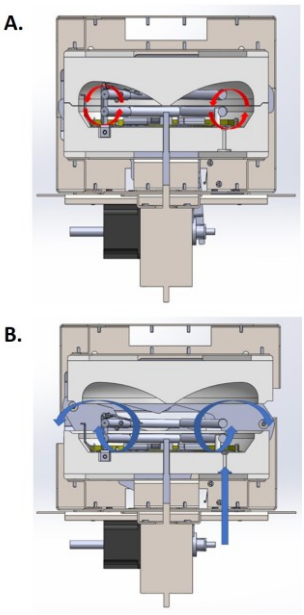

Figure 1. Eclipse MCO capillary column ovens enable rapid and precise temperature programming with convection heating (A) and cooling (B).

Impurities in ethylene and propylene product streams are costly in at least two respects. First, the purity of the monomer product, and the price which can be demanded for it, is directly impacted by trace impurities such as heavy hydrocarbons, sulfurs, arsine, phosphine or oxygenates. Additionally, the catalyst required to produce the monomer is poisoned by common by-products such as hydrogen sulfide, acid gases or methanol which reduce catalyst efficiency and shorten the life of the catalyst bed. Consequently, careful control of monomer process streams is required at multiple stages of product development.

Eclipse Monomer Analyzers: Measuring impurities in ethylene and propylene streams

The Eclipse Monomer Analyzer described here, designed for one of the largest O&G companies in North America, identifies C1-C6 hydrocarbons and common catalyst poisons (H2S, COS, methyl mercaptan, ethyl mercaptan, methanol, arsine and phosphine) in an ethylene matrix. To resolve and quantify such diverse analytes, the PGC is configured with 7 capillary columns housed in 2 temperature programmable MCOs and 1 isothermal oven. Two chromatography methods direct eluates to three multiplexed detectors (FID, PDHID and MSD) for unambiguous compound identification and quantification in just over 30 minutes.

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In the first method, 200 ppm hydrocarbons in an ethylene stream were resolved on a 50-meter column in MCO1 and speciated by FID (Fig.2A). The minimum detectable limit (MDL) was determined to be 2 ppm for this mixture.

Simultaneously, another 50-meter column in MCO2 resolved methanol, methyl mercaptan and ethyl mercaptan which were then detected by an Agilent 5977B mass spectrometer (Fig. 2B). The compounds were detected in select ion mode at MDLs of 50, 30 and 40 ppb, respectively.

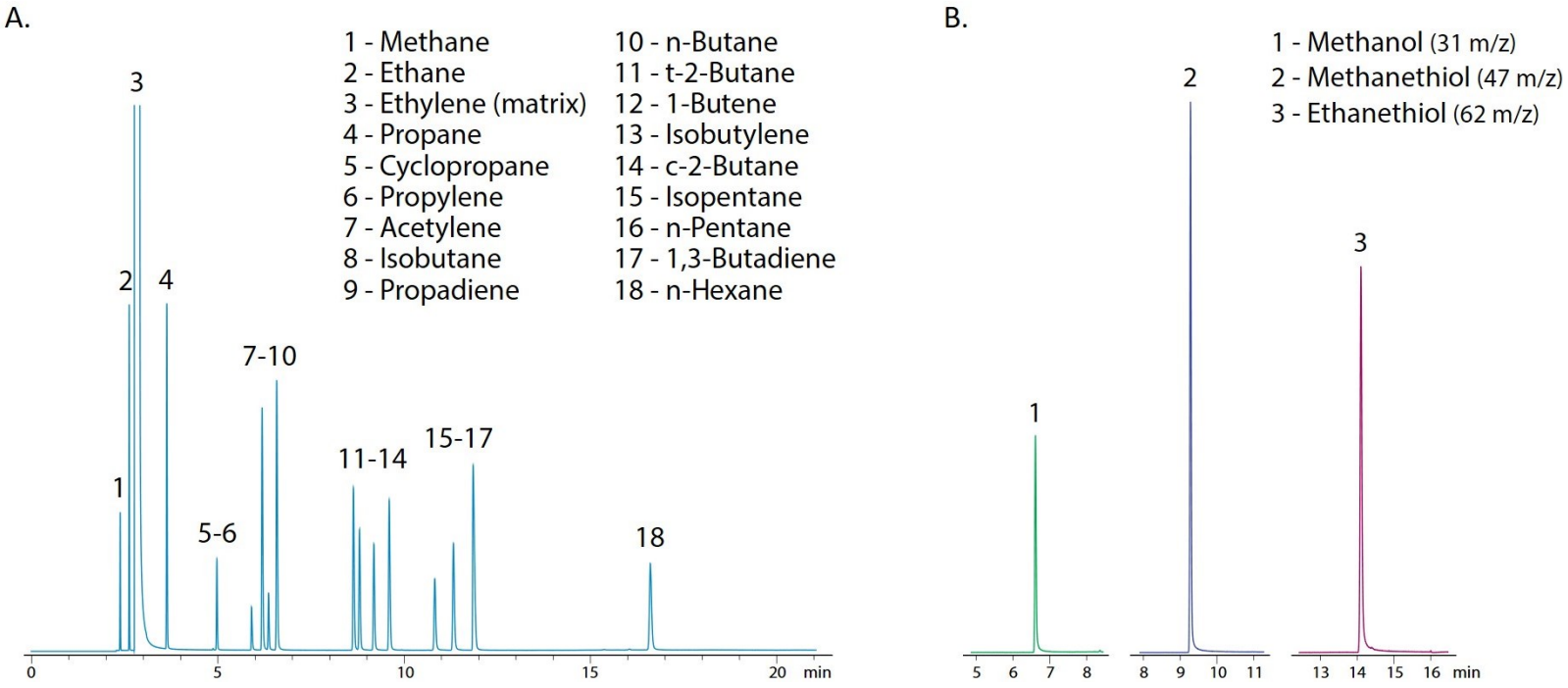

Figure 2. Method 1 analytes. FID chromatogram of C1-C6 hydrocarbons (A) and MSD select ion peaks for methanol, methyl mercaptan and ethyl mercaptan (B).

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The second method utilized 4 columns in the isothermal oven to separate hydrogen, oxygen/argon, nitrogen, carbon monoxide and carbon dioxide fixed gases (10ppm each). Eluates were detected by a Valco PDHID (Fig. 3A). The MDL for these analytes was 0.7 ppm.

The second method also used the columns in MCO2 and the MSD to detect H_2S and COS (10 ppm each) (Fig. 3B). The MDL for COS was 10 ppb while that of H_2S was 40 ppb. Arsine and phosphine were also examined using this approach (10 ppm each) (Fig. 3B) and found to have MDLs of 15 ppb and 30 ppb respectively.

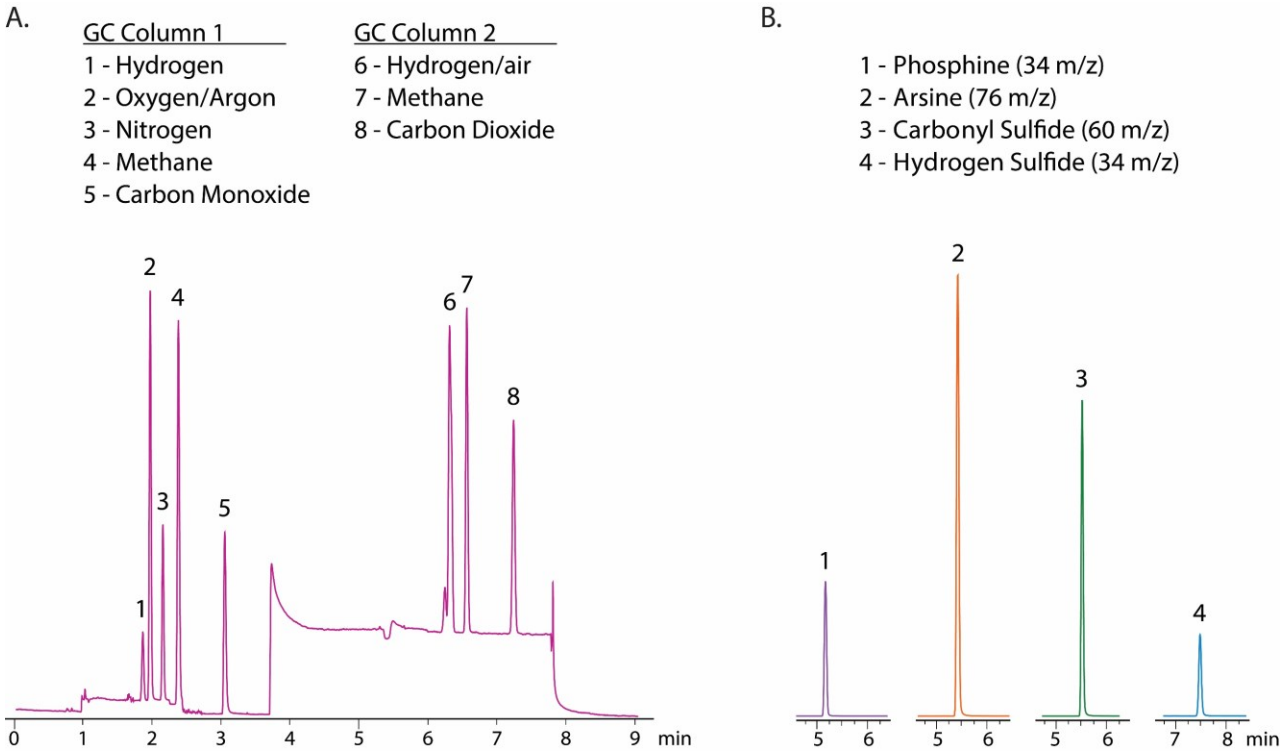

Figure 3. Method 2 analytes. PDHID chromatogram of common fixed gases (A), and MSD select ion peaks for phosphine, arsine, carbonyl sulfide and hydrogen sulfide (B).

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The Eclipse Monomer Analyzer provides online, lab-quality analysis of monomer streams in a fraction of the time required to complete these analyses by traditional methods.

Reproducibility studies, performed on an Eclipse Super RGA system, examined the precision of Eclipse temperature and electronic pressure controls. Figure 4 demonstrates highly reproducible retention times and peak areas, results rivaling those obtained on laboratory GCs and unparalleled in online PGCs.

A. Retention Times

	Methane	Pentane	Hexane	Heptane	Octane	Nonane	Decane	Undecane
Day 1	2.744	3.619	4.687	6.645	9.581	13.197	17.099	21.035
	2.775	3.653	4.723	6.683	9.613	13.225	17.118	21.038
	2.746	3.625	4.696	6.66	9.603	13.223	17.12	21.047
	2.747	3.626	4.697	6.663	9.604	13.223	17.118	21.049
	2.747	3.626	4.698	6.666	9.606	13.224	17.121	21.049
Day 2	2.747	3.625	4.694	6.659	9.598	13.213	17.108	21.036
	2.747	3.627	4.698	6.665	9.603	13.217	17.111	21.029
	2.745	3.624	4.694	6.662	9.601	13.215	17.106	21.033
Retention Time								
Avg.	2.750	3.628	4.698	6.663	9.601	13.217	17.113	21.040
StDev	0.010	0.010	0.011	0.010	0.009	0.009	0.008	0.008
RSD%	0.373	0.285	0.225	0.157	0.096	0.070	0.046	0.037

Data from 8 injections of Hydrocarbon blend over 2 days

B. Peak Areas

	Methane	
Peak Areas	83767	83886
	84037	84020
	83813	83891
	83992	83966
	83727	83806
	83880	83859
Peak Area		
Avg.	83887	
StDev	100.155	
RSD%	0.119	

Data from 12 injections of 5% Methane in Argon

Figure 4. Eclipse retention times and peak areas show excellent RSD %. **A.** Highly reproducible hydrocarbon retention times are a function of temperature, pressure and backpressure control. **B.** Reproducible methane peak areas are a function of split inlet performance and mass flow control.

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Wasson-ECE Instrumentation has 35 years of experience designing, building and applicating the most sophisticated GC systems in the field. With Eclipse, Wasson-ECE is leading the revolution in process gas chromatography. Eclipse analyzers are fully customizable to meet the needs of the most challenging processes (Fig. 5, Table 1) and are ready to transform your monomer processes.

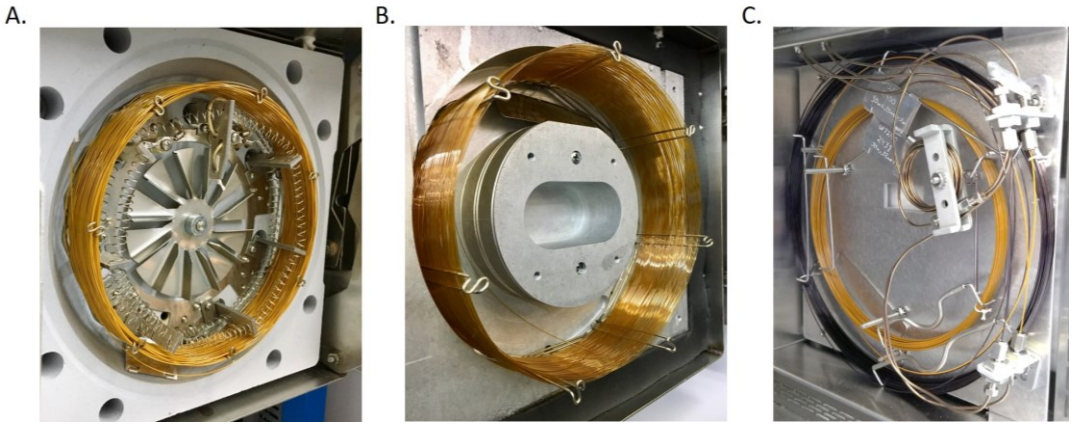

Figure 5. Eclipse capillary column MCOs deliver precisely controlled temperatures up to 225°C (A) and Peltier cooled to -20°C (B). Isothermal ovens are also available.

Table 1. Key features enabling lab-quality capillary column chromatography on a process GC.

Eclipse Technology
• Online MSD, VUV, PDHID, TCD and FID
• Capacity for up to 6 capillary columns
• Full electronic pressure programming
• Two programmable micro-convection ovens
• Two isothermal ovens
• Local 19" touchscreen interface
• MODBUS RTU, TCP and REST automation
• Wasson-ECE's new chromatography data system
• Sample systems with multiplexing for up to 16 sample streams
• Rated Class I, Division 2 and ATEX Zone 2 for hazardous locations

More information online:
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