Da Vinci
LABORATORY SOLUTIONS

DVLS Butadiene Analyzer

Composition & Impurities Analysis in Butadiene, Crude C4 & Raffinate

The DVLS Butadiene Analyzer

1,3-Butadiene is formed as a by-product from steam cracking and is used as Monomer in the production of synthetic rubbers, nylon and plastics. As Butadiene contains approx. 100 ppm inhibitors and also forms Butadiene Dimer also known as 4- Vinylcyclohexene (4-VCH or VCH) it is essential to control all product specifications. Da Vinci Laboratory Solutions (DVLS) introduces the DVLS Butadiene Analyzer – an All-In-One Analyzer that determines the complete product specifications in a single run: the hydrocarbon composition & the impurities such as Dimer, inhibitors and other impurities.

Application Range

The DVLS All-In-One Butadiene Analyzer complies with the following analysis test methods:

Test Method	Analysis
ASTM D2593	Hydrocarbon composition and impurities in 1,3 Butadiene
ASTM D4424	Composition of Crude C4 and Raffinates
ASTM D2426	Dimer and Styrene Content of 1,3 Butadiene concentrates
DVLS Method	TBC Inhibitor, BHT Inhibitor, ACN, DEHA, DMF, NMP, Toluene, impurities, Residue
BASF Method Part I-2	Gas Chromatographic Analysis of Hydrocarbons
BASF Method Part I-3	Gas Chromatographic Analysis of VCH (Vinyl-Cyclohexene) in Gaseous C4-HCs
BASF Method Part I-7	Determination of 1-methyl-2–Pyrrolidone in C4 fraction

Dual Channel Configuration

The DVLS **All-In-One** Butadiene Analyzer is based upon the Agilent 8890 Gas Chromatograph (GC) with built-in intelligence and electronic pneumatic control (EPC) configured with:

- Capillary columns;
- Flame Ionization Detectors;
- Split/splitless (SSL) Inlet and Cool on-Column (COC) Inlet;
- Liquid Sampling Valve (LSV) and Gas Sampling Valve (GSV) for the sampling of liquefied gases;
- Liquefied Gas Injector (LGI) for the direct injection of liquefied gases;
- Pressure Station for a representative sample introduction.

Butadiene Dimer Results (ASTM D2426)

Calibration Matrix: i-Butane

Calibration Standard Density: 0.5629

RT [min]	Name	Area	Density	RRF	Amount (mg/kg)
10.871	Dimer	173216	0.8263	0.02000	3171
	Sum	173216			3171

Inhibitor Results (ASTM D1157 Modified)

Calibration Matrix: Ethanol

Calibration Standard Density: 0.7967

RT [min]	Name	Area	Density	RRF	Amount (mg/kg)
22.087	TBC	1540	1.06	0.03970	79
	Sum	1540			79

Simultaneous Analysis

The Agilent Gas Chromatograph consists of two channels; each channel is configured with its own sampling technique and detector.
The dual channel configuration enables a simultaneous analysis within 30 minutes of:

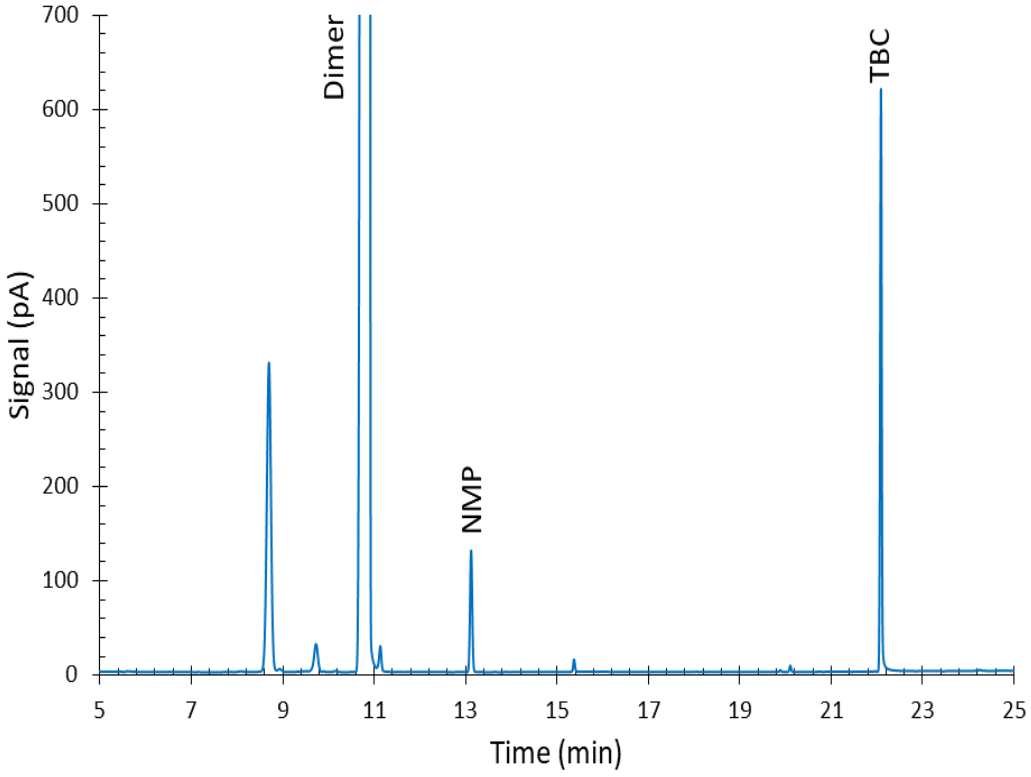
1. Hydrocarbon composition using the Liquid Sampling Valve and Dimer, inhibitors & impurities using the Liquefied Gas Injector;
2. Hydrocarbon traces < 1ppm using the Gas Sampling Valve and Dimer, inhibitors & impurities using the Liquefied Gas Injector.

The front channel is configured with a Liquid Sampling Valve and/or a Gas Sampling Valve for the analysis of:

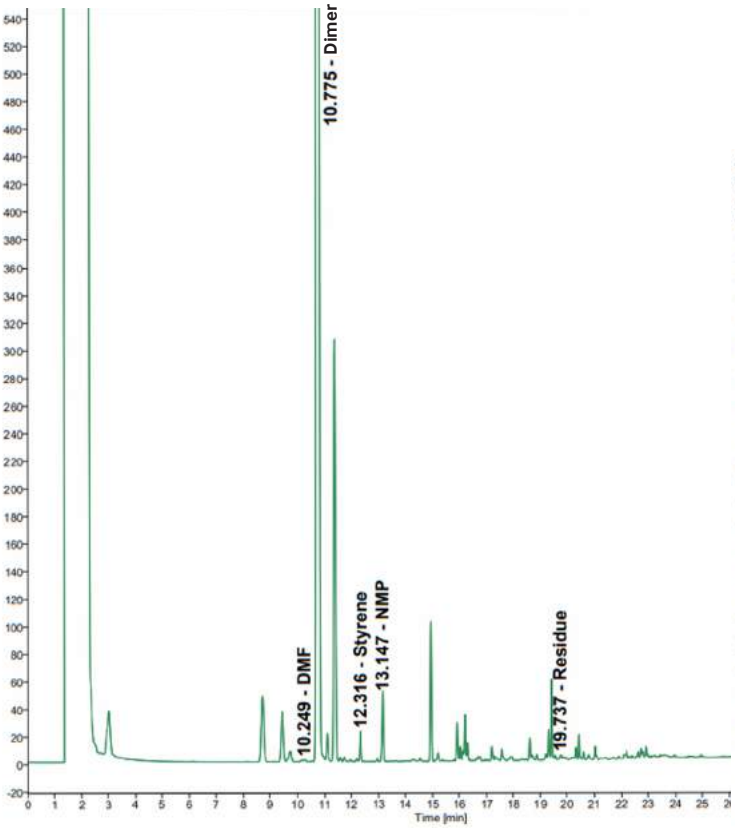
- Hydrocarbon impurities at ppm level;
- Butylene/Crude C4/1,3-Butadiene/Crude Butadiene/Raffinate composition.

The back channel is configured with the Liquefied Gas Injector for the analysis of:

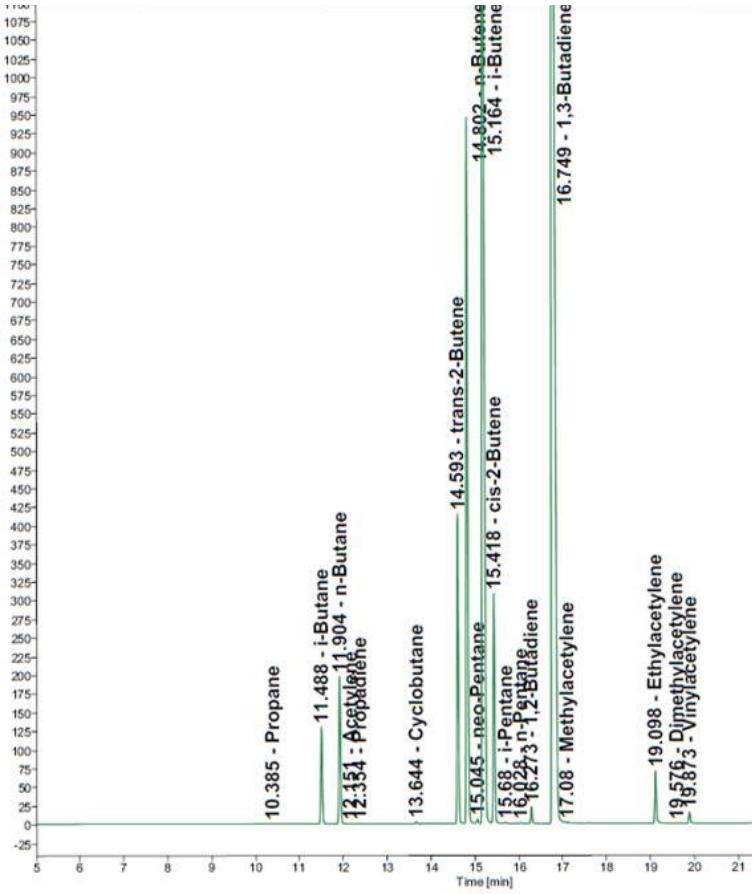
- Dimer and Styrene content;
- TBC and EC3071 Inhibitors;
- Residual solvents such as NMP, DMF, Toluene and Acetonitril.



Analysis of 1,3-Butadiene containing Dimer & TBC



Analysis of Crude C4 containing Dimer



ASTM D4424 Analysis of the composition of Crude C4

Butadiene Dimer Results (ASTM D2426)

Calibration Standard Density: 0.5629

RT [min]	Name	Area	Density	RRF	Amount (mg/kg)
7.396	Dimer	25737	0.8263	0.02000	678.0
8.637	Styrene	2	0.9087	0.02120	0.1
	Sum	25739			678.1

Inhibitor Results (ASTM D1157 Modified)

Calibration Standard Density: 0.5629

RT [min]	Name	Area	Density	RRF	Amount (mg/kg)
17.949	BHT	3	1.048	0.01970	0.1
18.091	TBC	3716	1.06	0.03700	232.3
	Sum	3720			232.4

Impurities Results (DVLS Method)

Calibration Standard Density: 0.7965

RT [min]	Name	Area	Density	RRF	Amount (mg/kg)
6.222	Toluene	90	0.8743	6.22900	784.5
11.829	NMP	5	1.0367	0.06380	0.5
	Sum	95			785.0

Residue Results (ASTM D1025 / D7756 Modified)

Calibration Standard Density: 0.6311

Rt Start	Rt End	Name	Area	RRF	Amount (mg/kg)
6.607	20.822	Residue	103	0.0200	2.1

Total Impurities: 1698 mg/kg

1,3-Butadiene Content: 99.51 % Weight (corrected for impurities)

Report of the Analysis of Impurities & Inhibitors in 1,3-Butadiene

The DVLS Butadiene Configuration

Performance Specifications

Compound	LOD (Wt ppm)
C1-C5 Hydrocarbons	< 0.5
BHT	< 0.2
DEHA	
DIMER	
DMF	
NMP	
Residue	
Styrene	
TBC	
Toluene	

The limit of detection of the Butadiene Analyzer

Safe Injection of Liquefied Gases

The Liquefied Gas Injector (LGI) is a dedicated GC solution for the direct injection of heavier components in light matrices such as C3 - C4 streams. The safe direct injection of the LGI requires only a small amount of the Butadiene sample.
The LGI is used to achieve sub ppm level detection limits for the analysis of traces and complies with ASTM D2426. It can be used as an alternative to:

- ASTM D1157 to determine TBC inhibitor;
- ASTM D1025 for the analysis of Residue C10 - C24 oily residue).

Repeatable Sample Introduction

To allow a safe and repeatable sample introduction of a (liquefied) gas Da Vinci Laboratory Solutions developed the DVLS Pressure Station. The Pressure Station introduces a (liquefied) gas sample into a chromatographic system and keeps the pressure at a constant level.

The sample cylinder is mounted onto the Pressure Station using quick connectors. For liquefied gas samples the Pressure Station adds high pressure Nitrogen to the sample cylinder, which controls the outlet pressure and flow. The sight glass enables a visual check of the liquid phase of the sample. The sample is introduced as a liquid using an external injection device such as a Liquefied Gas Injector or a Liquid Sampling Valve.

The Pressure Station is configured with a vaporizer to avoid accumulation of the waste injection. The waste sample is vented to a central waste system to ensure laboratory safety.

The DVLS Pressure Station

Key Benefits

- ☑ All-In-One Analyzer determines complete product specifications in a single run;
- ☑ Solution complies with ASTM D2593, D4424, D2426, DVLS & BASF methods;
- ☑ Analysis of Dimer, TBC and hydrocarbon composition within 30 minutes;
- ☑ Customized DVLS method for detecting various inhibitors and impurities at sub ppm level;
- ☑ Alternative to ASTM D1157 and ASTM D1025;
- ☑ Dual channel configuration allows simultaneous analysis of the composition and impurities;
- ☑ Innovative Liquefied Gas Injector and Pressure Station ensure lab safety.

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