DETERMINATION OF TBC LINEARITY AND CARRY-OVER IN 1,3-BUTADIENE WITH THE DVLS BUTADIENE ANALYZER

Polymerization-grade 1,3-Butadiene contains polymerization inhibitors to prevent spontaneous auto-polymerization during storage and transport. The prescribed inhibitor for 1,3-Butadiene is para-tert-butyl catechol (TBC). Current method ASTM D1157 determines TBC traces in 1,3-Butadiene with a detection limit of 50 mg/kg, requiring evaporation of large volumes of Butadiene.

Figure 1: The DVLS Butadiene Analyzer

Due to its chemical structure, TBC has low solubility in non-polar hydrocarbon streams and tends to adsorb onto active surfaces. To provide a safe and accurate alternative to D1157 Da Vinci Laboratory Solutions B.V. (DVLS) developed the DVLS Butadiene Analyzer containing a Liquefied Gas Injector (LGI). This Application Note reports on the tests of TBC linearity and carry-over in 1,3-Butadiene with the DVLS Butadiene Analyzer.

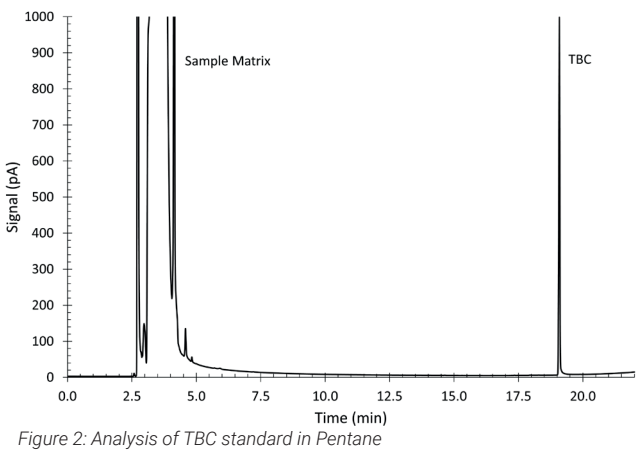

Figure 2: Analysis of TBC standard in Pentane

Experimental

The DVLS Butadiene Analyzer is configured on an Agilent 8890 GC equipped with a DVLS Liquefied Gas Injector (LGI) and DVLS Pressure Station. The LGI injects the sample directly from the gas cylinder into the DVLS Programmable Temperature Inlet (PTI), followed by a separation on a capillary column and detection using a Flame Ionization Detector (FID). This sampling approach eliminates the need for evaporation and prevents analyst exposure to 1,3-Butadiene.

Table 1: Dilution series of TBC

Standard	Concentration [mg/kg]
1	23.4
2	49.1
3	68.0
4	96.2
5	122
6	142

A stock solution containing 3.2% TBC in Methanol was prepared. Known amounts of this stock were diluted with Pentane in a copper strip cylinder as shown in Table 1. The standards shown in Table 1 were injected in triplicate. To check for carry-over, blank Pentane was injected after the standard 6 (containing 142 mg/kg) was injected. Finally, a sample was analyzed to determine the detection limit and repeatability.

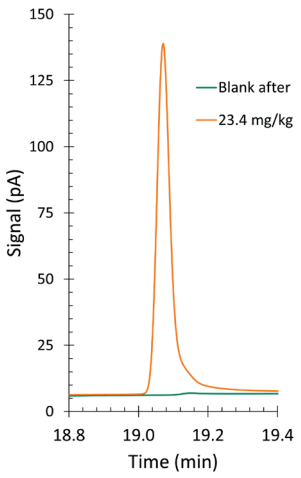

Figure 3: Analysis of TBC at 23.4 mg/kg concentration level in Pentane

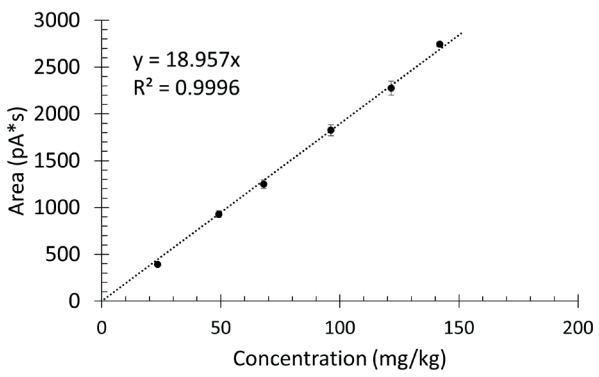

Figure 4: Regression of the TBC standards

Results

Figure 2 shows a chromatogram of a TBC standard in Pentane, Figure 3 displays the analysis of the TBC standard at 23.4 mg/kg concentration level along with the blank Pentane injected after standard 6 containing the highest concentration. The blank chromatogram seen in Figure 3 does not show a quantifiable trace of the TBC introduced during the previous analysis. This shows the system does not have any carry-over for this compound, even at high concentrations. Figure 4 shows a linear regression of the injected TBC concentrations plotted against the corresponding peak areas from Table 1.

The results of the linear regression in Figure 4 demonstrate that the TBC analysis shows excellent linearity over a broad concentration range.Table 2 lists the repeatability with a relative standard deviation (RSD) below 5% for TBC and a limit of detection (LOQ) below 1 mg/kg.

Table 2: Repeatability and LOQ results

Concentration [mg/kg]	Height [pA]	RSD [%]	r [mg/kg]	LOQ [mg/kg]
28.47	42.90	1.4	1.1	0.13

Conclusion

The DVLS Butadiene Analyzer is a GC system equipped with a Liquefied Gas Injector, designed for determining inhibitors such as TBC. Tests on TBC linearity and carry-over in Pentane showed a consistent linear response across the tested concentration range, with no carry-over effects. Detection limits were below 1 mg/kg, and relative standard deviations remained under 5%. These results indicate that the DVLS Butadiene Analyzer is a safer and more reliable alternative to the ASTM D1157 method, offering improved performance in terms of sensitivity and reproducibility.

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