ASBC
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Tom Mancuso PerkinElmer Inc.

Introduction When brewing beer, brewers not only need to confirm the quality of the raw materials that are used, but also ensure that by-products of the fermentation process do not negatively impact the taste of the finished beer product.
Two vicinal diketone (VDK) compounds which can negatively impact the flavor of beer are 2,3-butanedione (diacetyl) and 2,3 pentanedione. The concentrations of VDK typically range from 1-50 ppb in lighter beers, but they can reach several hundred ppb or more in darker beers. Acetaldehyde is a fermentation spillover product, which has significant effects on the flavor of beer. Excellent quality beers have low levels of acetaldehyde under 3 to 8 ppm.
Historically, breweries use two separate GCs, or two separate analytical runs in the testing of beers - one for the analysis of VDKs, and another for the analysis of acetaldehyde.
In this poster, an optimized method for the simultaneous analysis of VDK and acetaldehyde in beer was established using a PerkinElmer Clarus® 690 GC ECD/FID with a TurboMatrix™ HS-40.

Experimental **Sample Preparation**

Samples were prepared by transferring the beer to a wide mouth beaker and sonicating briefly for full degassing. Another common method for degassing is to pour the beer through filter paper. After degassing, 5 mL of the beer sample was transferred to a headspace vial. The vial was sealed immediately, with the PTFE side of the septum facing toward the sample.
Instrumentation A PerkinElmer Clarus 690 GC with a TurboMatrix HS-40 were used to perform these experiments. The Clarus 690 GC was configured with an electron capture detector (ECD) and a flame ionization detector (FID), and utilized PerkinElmer Elite-5 and BAC-1 columns installed in the injector via a two-hole ferrule. Nitrogen was used as the carrier gas for this study. The HS and GC conditions required for the analysis are listed below.

Gas Chromatograph	Clarus 690 GC
GC Column	VDK = Elite 5 30M x 0.32mm x 1.0um AA = BAC 1 30M x 0.32mm x 1.8um
Headspace Connector	2-Hole Ferule
GC Injector	CAP with a 2 mm straight bore liner
Injector Temperature	225°C
Carrier Gas	Nitrogen
Carrier Gas Flow	3.0 ml/min (psi = 11.9)
Split Flow	5 ml/min
Oven Program Initial Temperature	45 °C
Hold Time	1.3 min.
Ramp 1	25 °C/min to 135 °C
Hold Time	0.0 min (4.9 min run time)
Sample Rate	3.125
Bunching Factor	1
Noise Threshold	VDK = 500; AA = 25
Area Threshold	VDK = 5000; AA = 100

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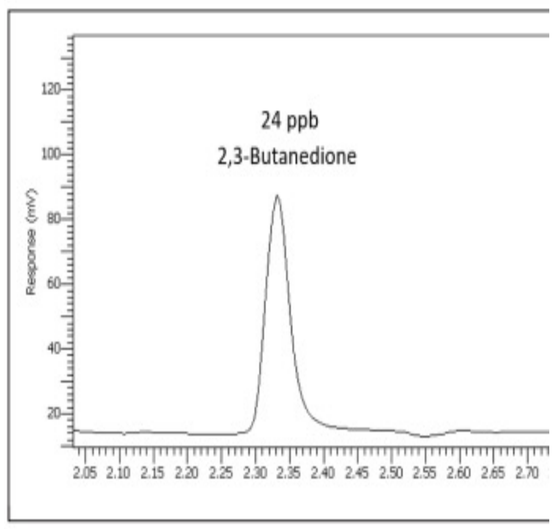
Noise Threshold	VDK = 500; AA = 25
Area Threshold	VDK = 5000; AA = 100
Detectors	**ECD & FID**
Temperature	ECD = 150 °C; FID = 250 °C
N2 Makeup	30 ml/min
Attn	ECD = -6; FID = -6



Calibration The calibration curves were prepared by dissolving 2,3-butanedione (diacetyl), 2,3-pentanedione and acetaldehyde in 5 mL of 5%Ethanol/95% water (Table 3 and 4). Each calibration standard was transferred to a separate headspace vial and capped with a PTFE/Silicone septum to ensure the lowest possible background levels.

Compound name	Level 1 (ppb)	Level 2 (ppb)	Level 3 (ppb)	Level 4 (ppb)
2,3-Butanedione (ppb)	9.6	24	48	120
2,3-Pentanedione (ppb)	5.57	13.9	27.8	69.6

Compound name	Level 1 (ppm)	Level 2 (ppm)	Level 3 (ppm)	Level 4 (ppm)
Acetaldehyde (ppm)	1	2	4	8

Results and Discussion The calibration curves were plotted as the peak area versus the amount of the analyte. The determination coefficients (r2) were better than 0.998, showing the reliability of the analysis in the range of 10 to 120 ppb for 2,3-butanedione, and 6 to 70 ppb for 2,3-pentanedione and 1 to 8 ppm for Acetaldehyde.
Calibration curves in Figures 1 and 2.
The PerkinElmer Elite BAC-1 column showed superior properties to separate acetaldehyde and ethanol.

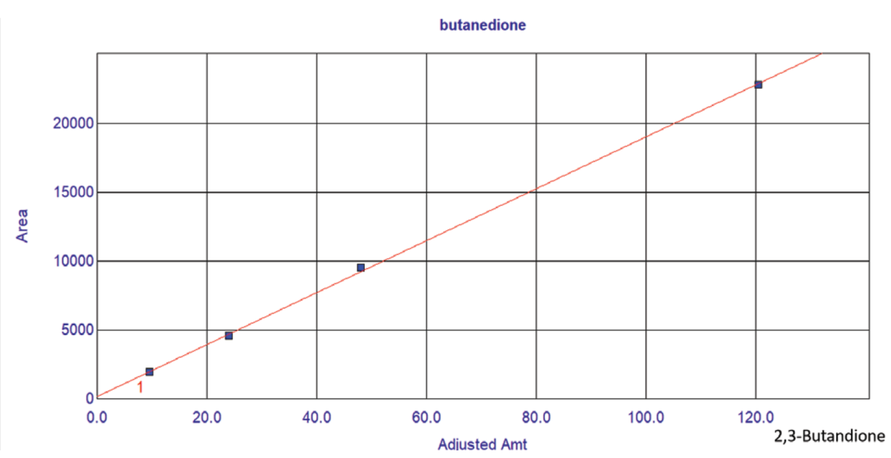


Figure 1 2,3-Butanedione and 2,3-Pentanedione Calibration Curves ECD

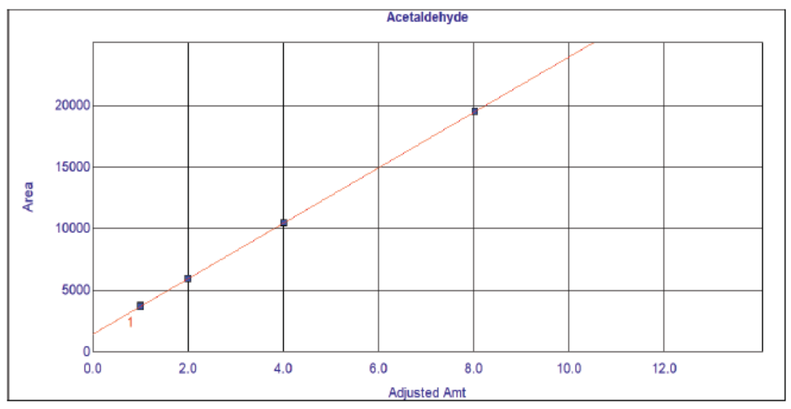


Figure 2 Acetaldehyde Calibration Curve FID

Conclusion and Acknowledgement

The results obtained in this experiment demonstrate that the Clarus® 690 GC with ECD/FID, used in conjunction with the TurboMatrix™ HS-40, offers a solution for the simultaneous determination of VDKs and acetaldehyde in beer, providing both performance and stability.

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