

Application Note

Introduction

Numerous compounds contribute to changes in beer flavor as it becomes stale. One of these compounds, (E)-2-nonenal, has been investigated as a major source of the papery/cardboard flavor that develops in aged beer¹.

This application note demonstrates the Teledyne Tekmar Atomx using the soil method to adequately detect this compound in beer. A GC/MS system using Selected Ion Monitoring (SIM) mode detects and quantifies (E)-2-nonenal levels in 5 beer samples.

The conditions listed, are one set of numerous possible conditions that accomplishes the separation and detection of these compounds. This does not preclude a scientific laboratory from operating this analysis with alternate conditions utilizing the Atomx soil method.



Method Conditions

Trap 1A (Tenax)

| Table I Atomx Modified Soil Method Parameters. | | | |
|--|-----------|-----------------------|------------|
| Purge Variable | Value | Purge Variable | Value |
| Valve Oven Temp | 200 °C | Condensate Purge Temp | 70 °C |
| Transfer Line Temp | 210 °C | Dry Purge Time | 0.50 min |
| Sample Vail Temp | 50 °C | Dry Purge Flow | 50 mL/min |
| Soil Valve Temp | 120 °C | Dry Purge Temp | 70 °C |
| Standby Flow | 25 mL/min | Desorb Variable | Value |
| Purge Ready Temp | 70 °C | Sweep Needle Time | 0.50 min |
| Condensate Ready Temp | 70 °C | Desorb Preheat Temp | 220 °C |
| Purge Mix Speed | Fast | Desorb Temp | 225 °C |
| Purge Time | 10.00 min | Bake Variable | Value |
| Purge Flow | 25 mL/min | Bake Time | 8.00 min |
| Purge Temp | 70 °C | Bake Flow | 200 mL/min |
| | | Bake Temp | 230 °C |

| Highlights |
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| The Teledyne Tekmar Atomx automated VOC sample prep system provides modern laboratories with a versatile VOC system to test multiple matrices. |
| It adequately detects (E)-2-nonenal in beer samples at mid ppt levels using the soil mode. |
| Salt added to the sample improves the detection limits for this compound. |

| Table II GC/MS System Settings | |
|--------------------------------|--|
| GC Settings | |
| Column | Rtx®-VMS, 20 m x 0.18 mm ID x 1 m, 0.9 mL/min Constant Flow, Helium |
| Inlet/Transfer Line | 230 °C, Split Ratio 60:1, Transfer Line 230 °C |
| Oven Program | 95 °C, 3.5 °C/min to 145 °C, 15 °C/min to 240 °C, 3 min final hold, 21.7 min Run |
| MS Settings | |
| Scan | 35.0 m/z to 200 m/z, Gain Factor 1, ATune, 1.4 min Solvent Delay |
| SIM | 55 m/z, 70 m/z, 83 m/z, 96 m/z, 111 m/z, Dwell Time 100 |
| Temperatures | Source 230 °C, Quad 150 °C |

Standards and Samples

Standard: (E)-2-Nonenal, Aldrich, 255653, 0.27 to 5.4 ppb in 5% (v/v) ethanol solution

Samples: 5 Lots of a Pale Lager Beer, 10 mL degassed sample with 6 g NaCl

Results

Figure 1 Comparison of the SIM Total Ion Chromatogram of a Beer Sample (in Triplicate, Lower) to its Spiked Sample (Upper)

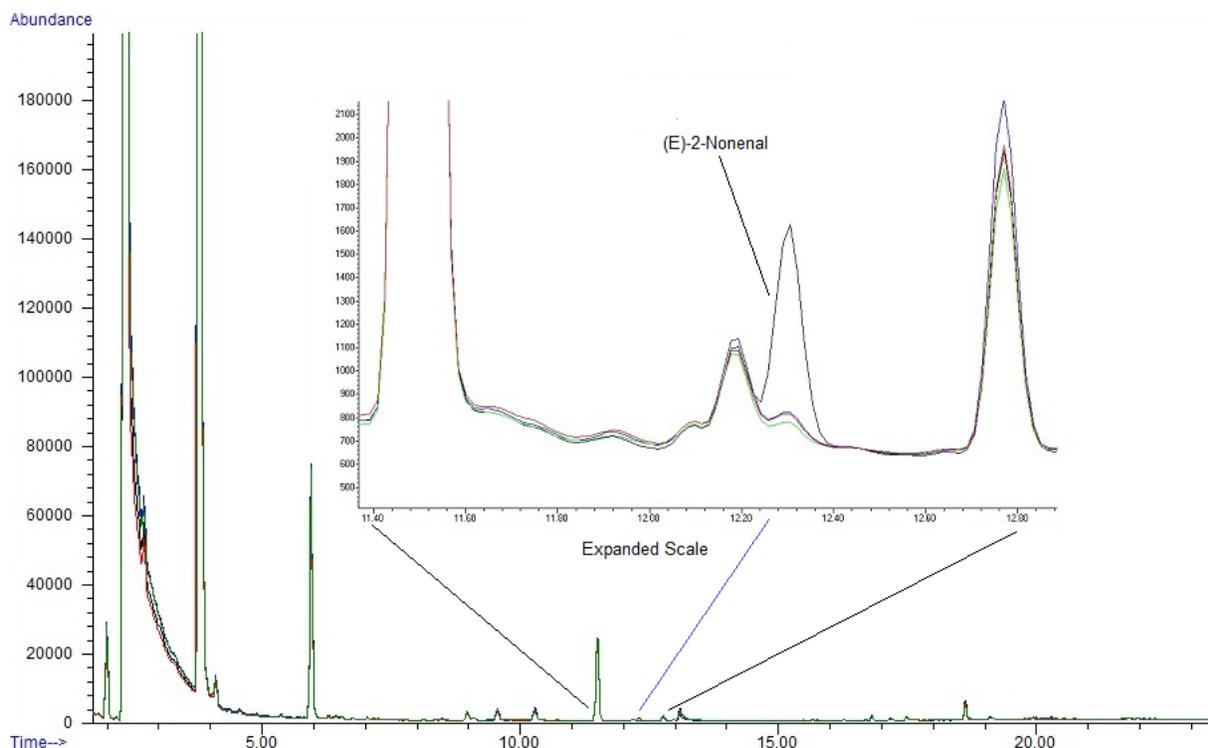


Table III Concentration of (E)-2-Nonenal in Five Lots of a Commercial Pale Lager

| Sample Lot | ppb |
|------------|-------|
| A | 0.180 |
| B | 0.463 |
| C | 0.261 |
| D | 0.184 |
| E | 0.082 |

References

1 – Scherer, R.; Wagner, R.; Kowalski, C.H.; Godoy, H. T. (E)-2-Nonenal Determination in Brazilian Beers using Headspace Solid-Phase Microextraction and Gas Chromatographic Coupled Mass spectrometry (HS-SPME-GC-MS) Cinc. Tecnol Aliment., Campinas, 30(Supl.1): 161-165, Maio 2010 [Online]
http://www.scielo.br/scielo.php?script=sci_arttext&pid=S0101-20612010000500024