

Determination of 32 Low Molecular Mass Organic Acids in Biomass by Ion Chromatography Mass Spectrometry

Low Molecular Mass Organic Acids (LMMOAs) are degradation products during pretreatment processes for biofuel production that are known to inhibit microbial fermentation processes, and thus reduce the conversion efficiency of biomass to biofuels. To better understand and optimize the conversion process, an IC/MS method to quantitate LMMOA profiles during biofuel production was developed. The IC/MS method is particularly useful in separating and profiling close eluters using the power of high performance anion exchange chromatography coupled with multiple SIM scans to identify over 30 compounds in a single analytical run. LMMOAs were detected and reported with various concentrations ranging from 0.140 ppm to > 200 ppm.

CONDITIONS:

| | | |
|----------|--|---------------------------|
| System: | ICS-2000 | |
| Columns: | IonPac®AS11-HC analytical column with AG11-HC guard (2 mm) | |
| Eluent: | Hydroxide gradient: | |
| | Time /min | Concentration / mM |
| | -10 | 1.0 |
| | 0 | 1.0 |
| | 8 | 1.0 |
| | 18 | 15 |
| | 28 | 30 |
| | 38 | 60 |
| | 40 | 1.0 |

| | |
|----------------|---|
| Eluent Source: | EGC II KOH |
| Flow Rate: | 0.38 mL/min |
| Inj. Volume: | 25 µL (or mL, etc.) |
| Detection: | Suppressed Conductivity Mass Spectrometric Detection For Suppressed Conductivity Detection: Suppressor: ASRS-300 Anion Self-Regenerating Suppressor (2 mm) Regenerant: Deionized water Regenerant Flow Rate: 0.5 mL/min Mass Spectrometric Detection: Instrument: MSQ Plus Ionization Method: Electrospray Ionization (ESI) source Scan Mode: Negative Selected Ion Monitoring (SIM) Nebulizer Gas: Nitrogen at 80 psi Probe Temperature: 450 °C Needle Voltage: 3000 V |

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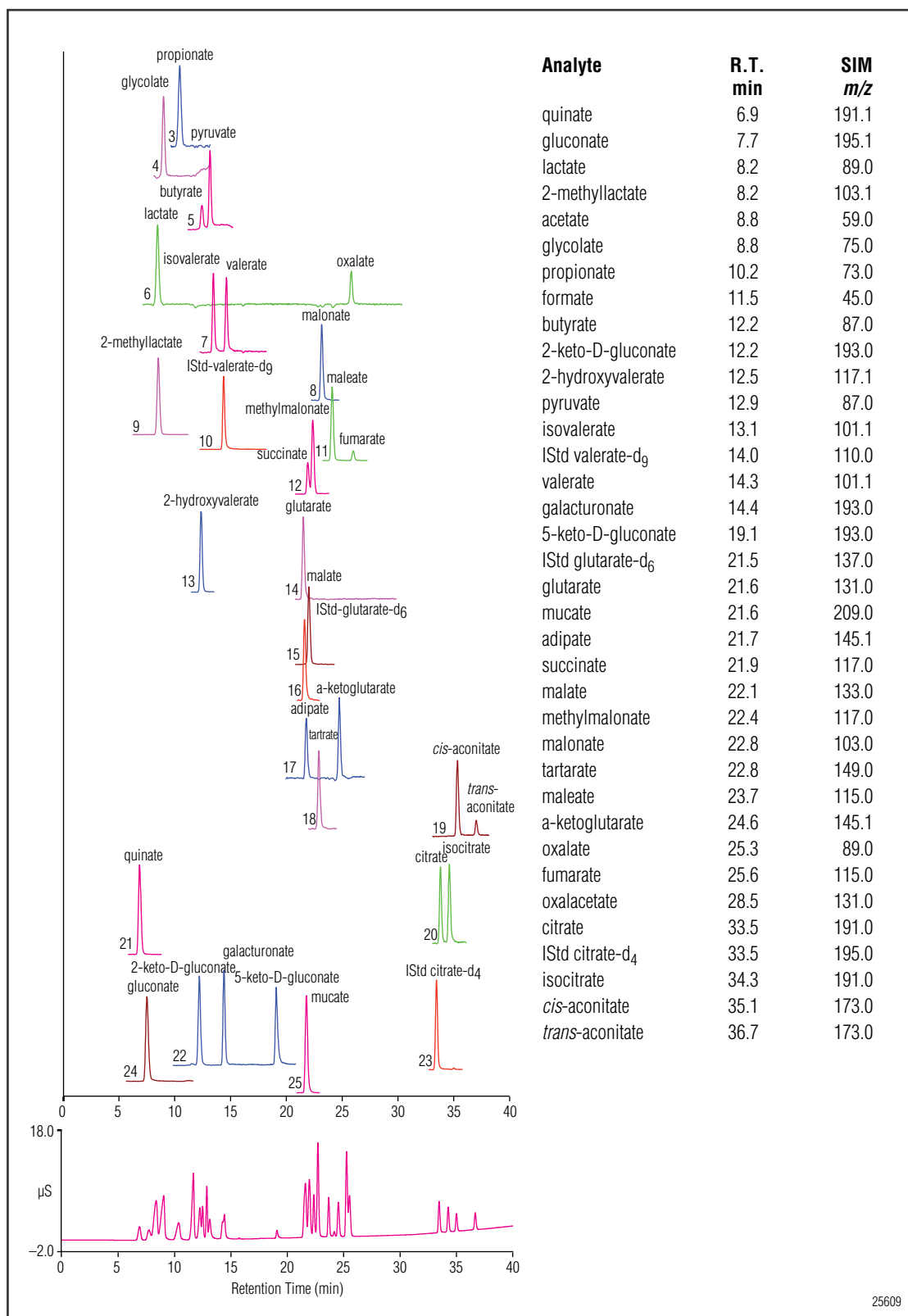


Figure 1. CD and MS SIM chromatograms of 32 small organic acids.

1. Each SIM chromatogram is normalized to the greatest peak in that channel.

2. 100 ppb for each internal standard.

3. MS detection: 50 ppb for each analyte except for cis- and trans-aconitate whose combined total concentration is 50 ppb

Conductivity detection: 5 ppm for each analyte, aconitantes have a total concentration of 5 ppm.

4. SIM channels are sorted by increasing m/z from top to bottom.

5. SIM scan numbers 1 and 2 (formate and acetate) are not shown due to weak response; scans 4 - 23 are identified and summarized in the table above.

CONCLUSION:

IC/MS is a robust and highly selective method for monitoring LMMOAs and other trace-level fermentation inhibitors present in complex matrices, such as fermentation broths. The power of IC/MS detection enables quantitation of more than 30 closely eluting compounds in real-world samples which are difficult to fully resolve chromatographically.

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