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ORGANIC ACIDS IN WINE

ANALYTES

In elution order: Citric, tartaric, malic, succinic, lactic, fumaric, acetic.

INTRODUCTION

Determining the levels of organic acids is an important part of fermentation monitoring during winemaking. The balance of the acids directly influences the flavor and color of the wine, and must also be carefully controlled to assure proper fermentation and to prevent spoilage.

The concentration of these organic acids are most important to the winery. In order of importance, they are:

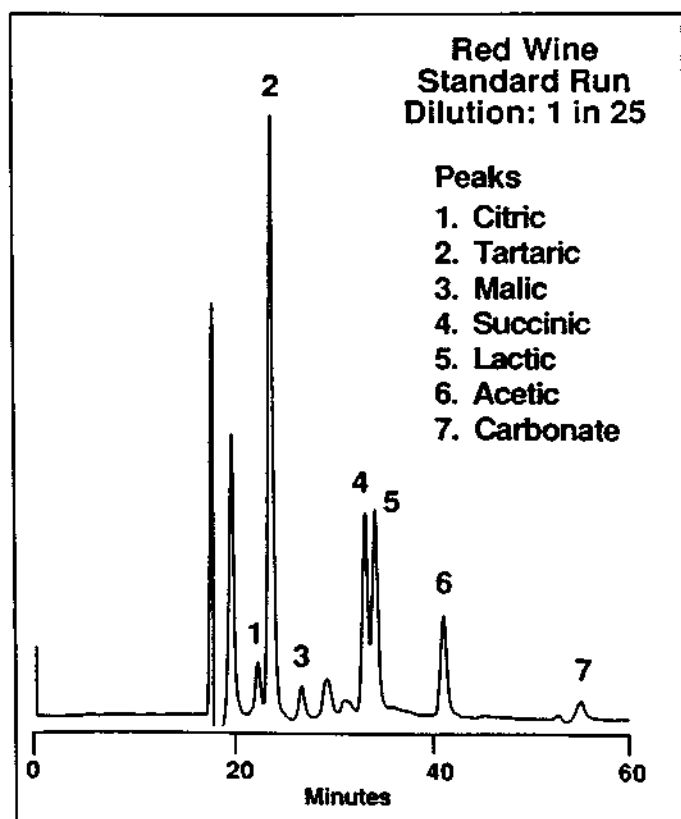
- Malic Acid – determined to measure the progress of malo-lactic fermentation.
- Acetic Acid – monitored to prevent spoilage; concentration must be determined for BATF regulations.
- Citric Acid – added to adjust acidity and to chelate metals; concentration must be determined for export limits.
- Tartaric Acid – added to lower the pH and adjust acidity; concentration is useful for deacidification and might be applied to cold stability testing.
- Fumaric Acid – added to prevent malo-lactic fermentation and adjust acidity (additions should be checked as fumaric is difficult to dissolve).

DISCUSSION OF METHOD

Organic acids in wine are determined by ion exclusion chromatography with chemical eluant suppression and conductivity detection. Since these organic acids do not contain UV chromophores, conductivity detection is far more sensitive than UV detection. Also, conductivity detection is more selective as there is no large phenolic front with red wines.

Included in the method are two sets of conditions. The Standard Run separates all the organic acids in 45 minutes. This run can be used for both routine monitoring and for enology research. The Fast Run elutes the acids in 15 minutes; however, lactic and malic acids are not resolved. (During both runs, carbonate elutes well after the last organic acid (acetic). It is not necessary to wait for carbonate to elute before the next injection is made as it will elute before the first peak in the second injection.)

After continued use, metals or other cations may build up on the column producing a decrease in resolution. The column can be regenerated by pumping 1 N HCl through the column for one hour, followed by a DI water rinse.



CONDITIONS

Standard Run

Separator Column: Two HPICE-AS1
Columns in Series

Column Temperature: 40° C

Sample Loop Volume: 50 μ L

Eluant: 2 mM Octanesulfonic
Acid in 2% 2-propanol

Eluant Flow Rate: 0.5 mL/min.

Suppressor: Anion MicroMembrane
(AMMS-ICE)

Regenerant: 5 mM TBAOH

Regenerant Flow Rate: 2 to 3 mL/min.

Conductivity Detector Range: 30 μ S

Expected Background Conductivity: 80 to 90 μ S

Fast Run

same as standard run except the following:

Separator Column: One HPICE-AS1

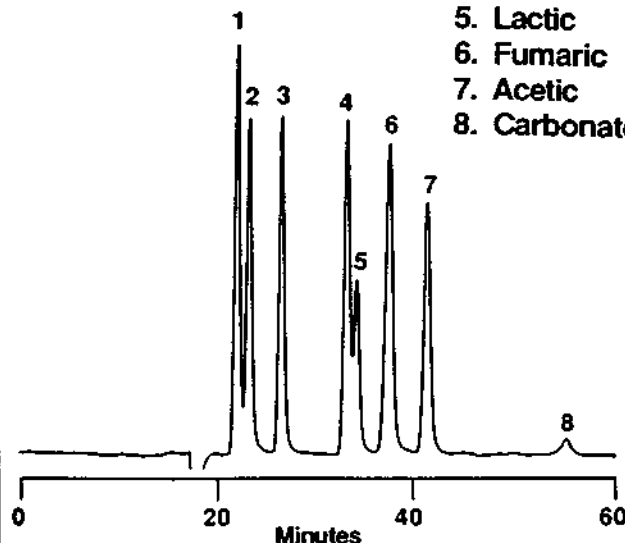
Column Temperature: 30° C

Eluant Flow Rate: 0.8 mL/min.

Organic Acid Standard Standard Run 50 ppm each

Peaks

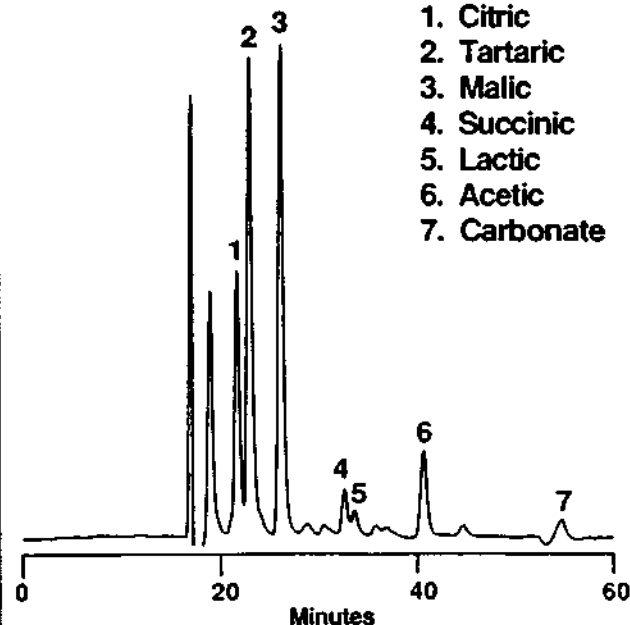
1. Citric
2. Tartaric
3. Malic
4. Succinic
5. Lactic
6. Fumaric
7. Acetic
8. Carbonate



White Wine Standard Run Dilution: 1 in 25

Peaks

1. Citric
2. Tartaric
3. Malic
4. Succinic
5. Lactic
6. Acetic
7. Carbonate



SOLUTIONS AND REAGENTS

Eluant: 2 mM Octanesulfonic
Acid in 2% 2-propanol.

Dilute 20.0 mL of Dionex 0.10 M octanesulfonic acid MPIC-CR2 Reagent (P/N 35362) and 20 mL of high purity 2-propanol per liter of 18 M ohm DI water.

Regenerant: 5 mM TBAOH

Dilute 10 mL of 55% TBAOH (tetrabutylammonium hydroxide) per 4 liters of 18 M ohm DI water.

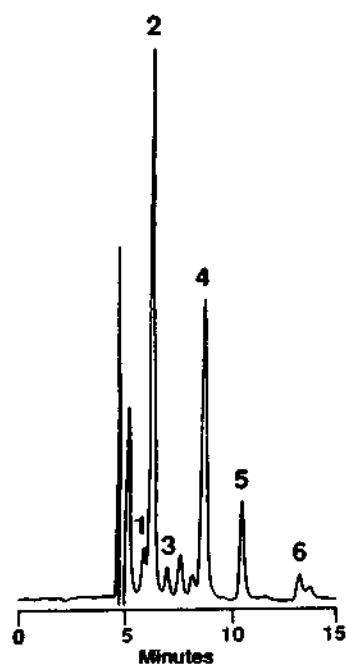
Sample Preparation:

Filter wine through an 0.45 micron membrane and pass through a C18 pre-sep cartridge. The pre-sep cartridge should be activated with 5 mL methanol followed with 5 mL DI water. Dilute wine 1 in 33 or 1 in 25 with DI water.

RECOMMENDED EQUIPMENT

Any Dionex Ion Chromatograph equipped with a Conductivity Detector: QIC, 2000i, 2000i/SP, or 4000i with Column Heater.

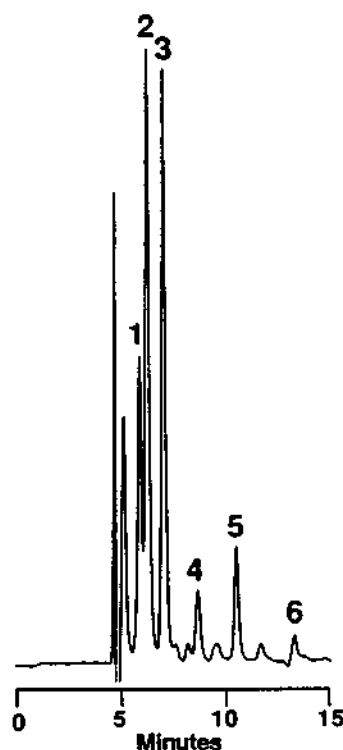
**Red Wine
Fast Run
Dilution: 1 in 33.3**



Peaks

1. Citric
2. Tartaric
3. Malic
4. Lactic & Succinic
5. Acetic
6. Carbonate

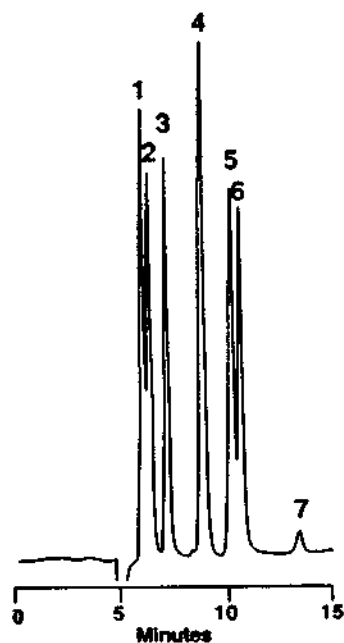
**White Wine
Fast Run
Dilution: 1 in 25**



Peaks

1. Citric
2. Tartaric
3. Malic
4. Succinic & Lactic
5. Acetic
6. Carbonate

**Organic Acid Standard
Fast Run
50 ppm each**



Peaks

1. Citric
2. Tartaric
3. Malic
4. Succinic & Lactic
5. Fumaric
6. Acetic
7. Carbonate