

■ Analysis Example of Maleic Acid in Starch

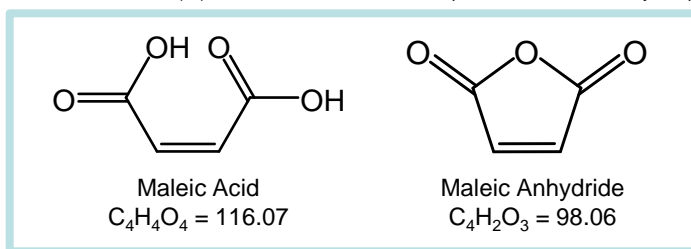
AS/LC-028

Maleic anhydride is used for a broad range of applications as a raw material for fine chemical synthesis and leads to the manufacturing of unsaturated polyester resins, organic acids, and resin improving agents. In May of 2013, maleic acid was detected from products containing starch such as tapioca, paste for oden, and meat balls in Taiwan. As a result of the investigation, the cause was found to be the use of unauthorized starch processed with maleic anhydride. The addition of this type of starch makes food products palatable. However, its use in foods is prohibited in Taiwan as a large intake will lead to reduced renal functions.

Also in Japan, maleic acid and maleic anhydride are not authorized by the Minister of Health, Labour, and Welfare and thus, its use as an additive is considered to be an infringement of the Food Sanitation Act. Based on this background, the notification concerning the test method for maleic acid and maleic anhydride was issued by the MHLW (\*1).

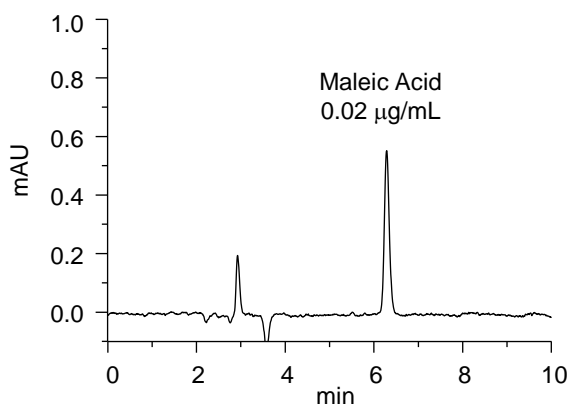
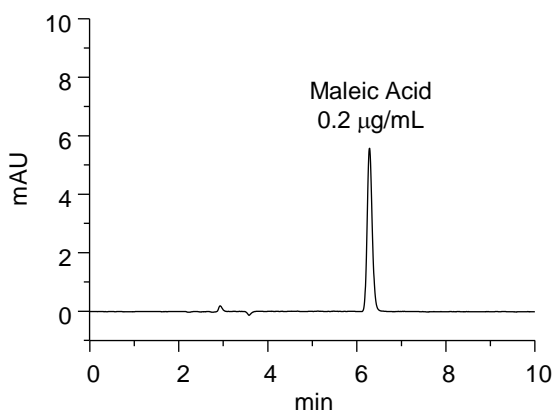
This time, maleic acid was analyzed under the analytical conditions of the test method. The preparation method for the actual sample is also introduced here.

(\*1) Notification No. 0621-2 of Department of Food Safety, Inspection and Safety Division (June 21, 2013)



[Structural Formulas of Maleic Acid and Maleic Anhydride]

■ Analysis Example of Maleic Acid Standard Sample



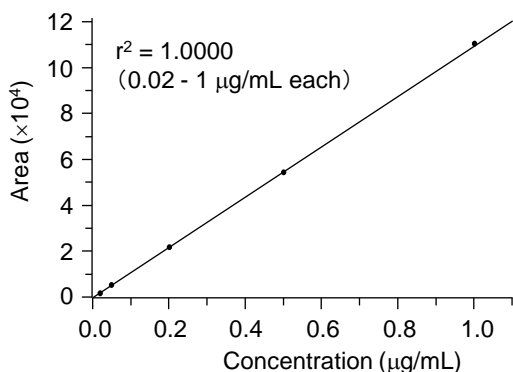
[Analysis Example of Maleic Acid Standard Sample]

<Analytical Conditions>

Column : LaChrom II C18 (5 µm) 4.6 mm I.D. × 250 mm  
 Eluents : (A) 0.1 % phosphoric acid (B) Methanol  
           A / B = 98 / 2 (v/v)  
 Flow rate : 1.0 mL/min  
 Column temperature : 40°C  
 Detection wavelength : UV 214 nm  
 Injection vol. : 20 µL

(Note) Maleic anhydride is hydrolyzed in the preparation and quantitatively analyzed as maleic acid. Thus, only maleic acid standard sample is analyzed.

■ Linearity and Reproducibility for Maleic Acid Standard Sample (n=6)

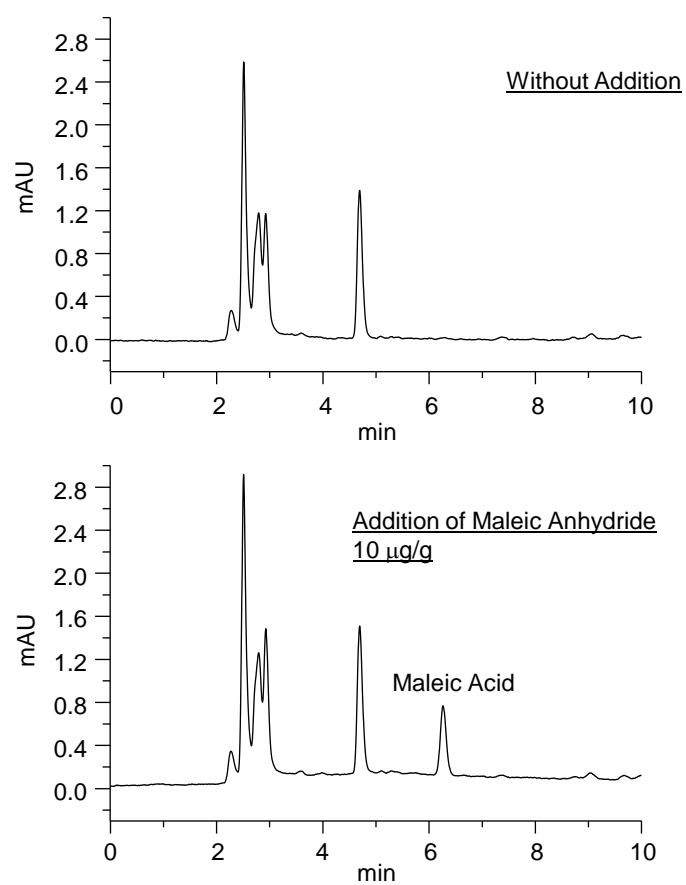


	Maleic Acid 0.2 µg/mL		Maleic Acid 0.02 µg/mL	
	Retention time	Area	Retention time	Area
Mean	6.301 min	21907	6.297 min	2229
%RSD	0.05	0.31	0.06	1.47

The detection limit of this test method is 10 mg/kg which is converted to be 0.02 µg/mL of maleic acid.

The above results indicate that the sensitivity, linearity, and reproducibility were all good for the maleic acid standard sample.

■ Analysis Example of Maleic Acid in Corn Starch



<Preparation Method for Corn Starch>

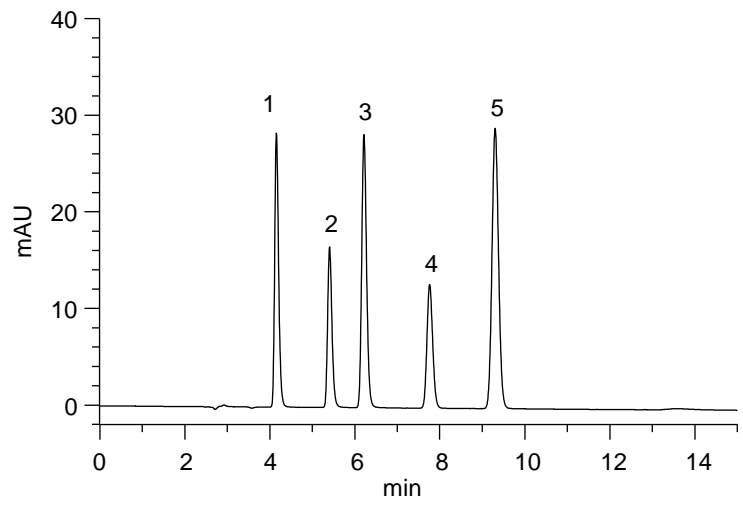
- Sample Approx. 1 g
  - ← Add 10 µg/g of maleic anhydride
  - ← 25 mL of 50 vol % methanol solution
- Shake 30 min
  - ← 20 mL of potassium hydroxide test solution
- Mix
- Let it stand 2 hr
  - ← Approx. 3 mL of hydrochloric acid test solution
  - ← Make up to 50 mL with purified water
- Let it stand a few min
- Supernatant 0.1 mL
  - ← Make up to 1 mL with purified water
- Filtration 0.2 µm PVDF filter
- Analytical sample (20 µL)

[Analysis Example of Corn Starch]

Maleic acid was not detected from the corn starch prepared by the specified procedure. When the corn starch to which maleic anhydride was added at 10 µg/g (near the detection limit of the test method) was analyzed, the maleic acid peak was well separated from interfering peaks.

■ Reference: Confirmation for Separation of Organic Acids

This method states that when changing the conditions or column to other than those specified, the separation of maleic acid from organic acids such as fumaric acid, acetic acid, maleic acid, and succinic acid should be confirmed. As a result of the confirmation, it was found that maleic acid was well separated from those organic acids.



Component name	Concentration (mg/L)
1. Malic acid	100
2. Acetic acid	100
3. Maleic acid	1
4. Succinic acid	100
5. Fumaric acid	1

Main system configuration: Chromaster 5110 pump, 5210 autosampler, 5310 column oven, 5410 UV detector

NOTE: These data are an example of measurement; the individual values cannot be guaranteed.