Analysis of Oxalic Acid in Food with Amide Column

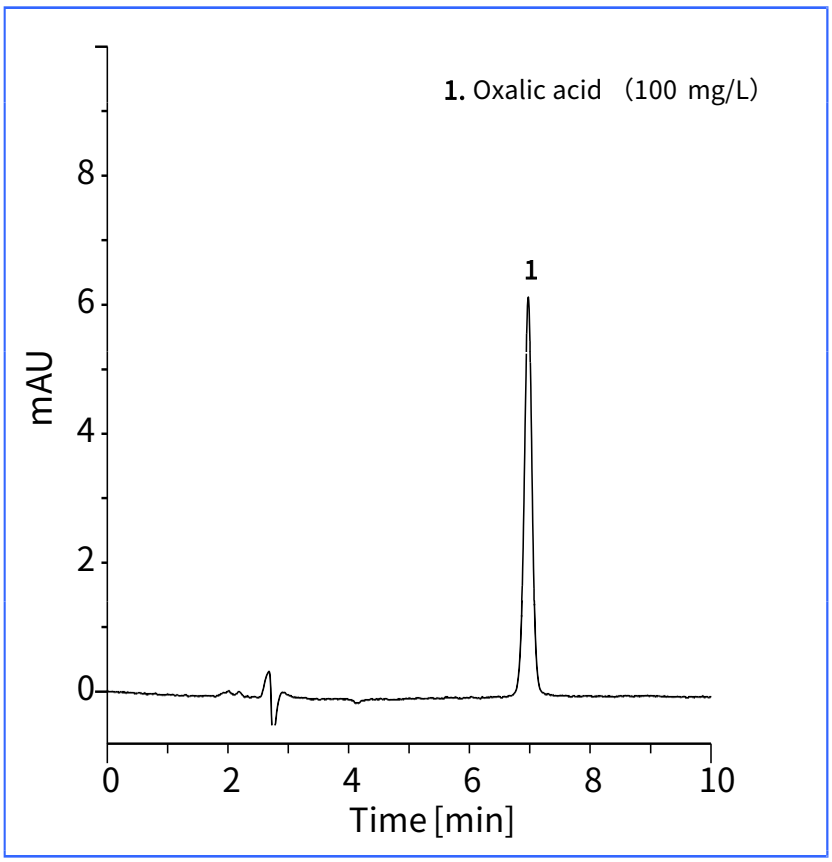
Oxalic acid is found in various foods, some of which contains around 1% oxalic acid. ODS column is often used as a separation column for analysis of oxalic acid. However, it is difficult to separate oxalic acid from interfering peaks because oxalic acid is highly hydrophilic and poorly retained on ODS columns.

Inertsil Amide, in which porous silica gel having a chemically bonded carbamoyl group is packed, was used in this note. In contrast to ODS columns, oxalic acid was well retained on Inertsil Amide, and its concentration in pickled ginger was determined.

Porous silica gel having a chemically bonded carbamoyl group is added as a packing material for chromatography in Japanese Phramacopeia 16, which went into effect in April, 2011. (K. Kanno)

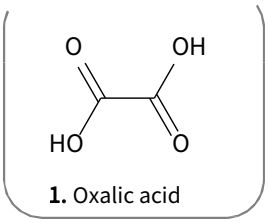
A Chromatogram Obtained from Standard Solution

HPLC conditions



Column : Inertsl Amide (5 µm, 4.6 mm I.D. x 250 mm)
Eluent : A) CH_3CN
B) 30 mM Na_2HPO_4 (pH 6.8, H_3PO_4)
A/B = 65/35, v/v
Flow rate : 1.0mL/min
Col. Temp. : 50 ˚C
Detected : UV 220 nm
Inj. Vol. : 5µL

Chemical Structure



1. Oxalic acid

Structures are created using Chemistry 4-D Draw which is provided by ChemInnovation Software, Inc.

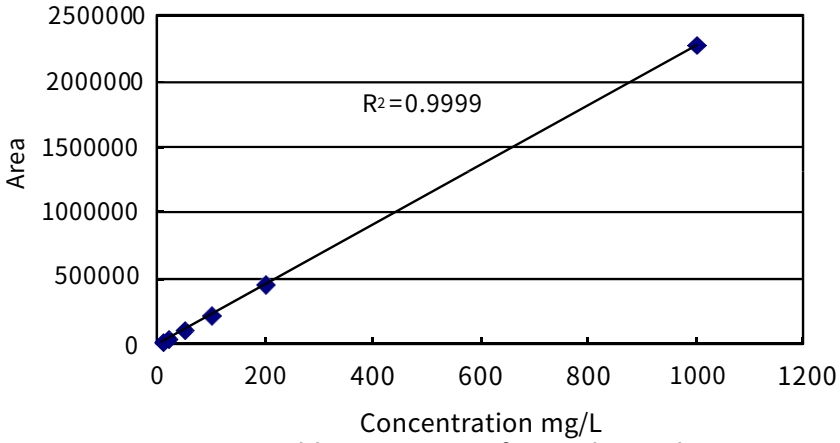
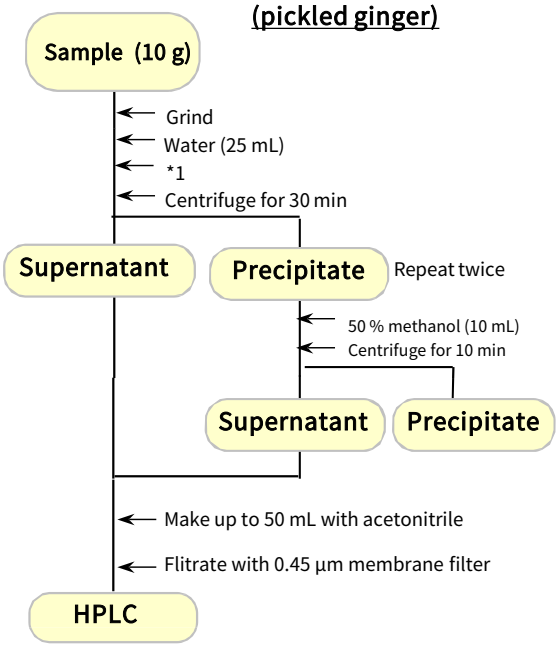


Figure 1. Calibration curve for oxalic acid

Table 1. Peak area for consecutive injection (100 mg/L)

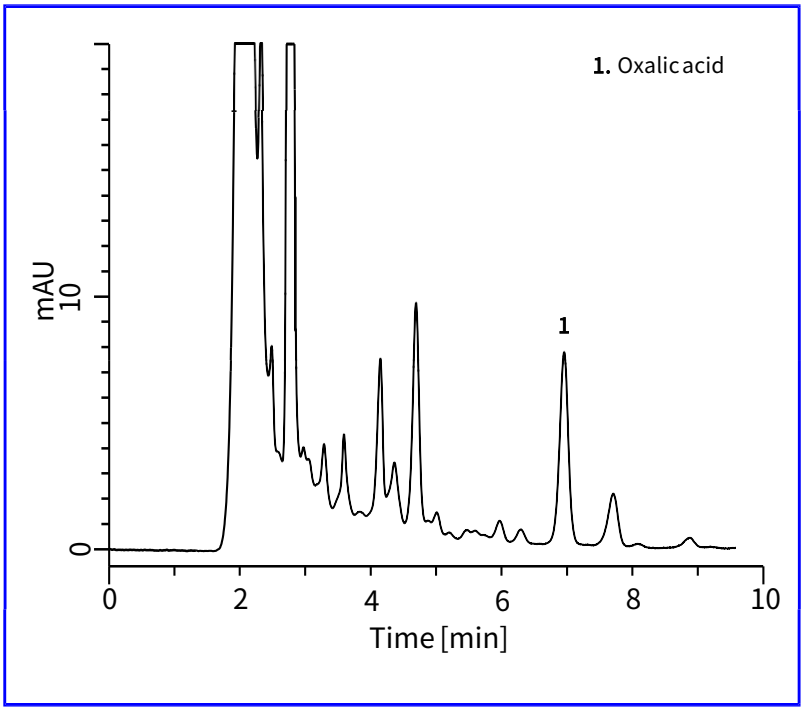
	Peak area
1st injection	228967
2nd injection	228243
3rd injection	228072
4th injection	227364
5th injection	227627

An example of sample pretreatment (pickled ginger)



*1 In case sample contains ascorbic acid, recovery of oxalic acid may be lowered. Ascorbic acid should be removed prior to the pretreatment, for example by ascorbate oxidase, when needed. (Reference: Japanese standard methods of analysis in food safety regulation)

A Chromatogram obtained from ginger sample



A know-how for using amide column ①

Amide column is usually used in HILIC mode. Salts soluble not only in water but also in organic solvent are recommended for the mobile phase because organic solvent content is quite high in HILIC mode.

＜Recommended salts and their concentration＞
Ammonium acetate or ammonium formate ～10 mM

These salts, however, are not unsuitable when analytes have to be detected with low-wavelength UV absorbance. In such a case, phosphate, sodium, and pottasium salts should be used as shown in this note. But, it is necessary to take care not to precipitate because their solubility in acetonitrile is low compared with ammonium acetate and ammonium formate.

HPLC column: Inertsil Amide (5 µm, 4.6 mm I.D.×250mm)
Cat. No. 5020-07836

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