SHIMADZU
Excellence in Science

Application Data Sheet

No.95

GC-MS
Gas Chromatograph Mass Spectrometer

GC-MS Analysis of Catalytic Degradation Products of Lignin Model Compounds in Biomass Research

When converting biomass resources into energy or raw chemical materials, it is essential to improve the conversion yield and analyze the reaction process. This sort of analysis requires identifying volatile components; therefore, GC-MS systems, with their excellent qualitative capabilities, are useful.
This report presents an example of degrading a lignin model compound with a solid acid catalyst in an aqueous solution and directly analyzing the resulting solution using GC-MS without pretreatment.
Lignin accounts for 20 % to 30 % of woody biomass. However, it is only weakly reactive because of its strong macromolecular structure, and therefore difficult to degrade. Since anisole, phenol, guaiacol, and other lignin degradation products are useful materials, though, research is progressing into degradation methods that provide a high conversion yield. The analysis of the catalytic degradation reaction presented in this report revealed that guaiacol is mainly produced from lignin model compounds.
In addition, utilizing the GCMS-QP2010 Ultra, which features a differential vacuum system, enabled the acquisition of favorable results even when directly injecting the sample aqueous solution into the GC-MS.

Sample

Fig. 1 shows the lignin model compound (guaiacylglycerol-β-guaiacyl ether) used in this experiment.

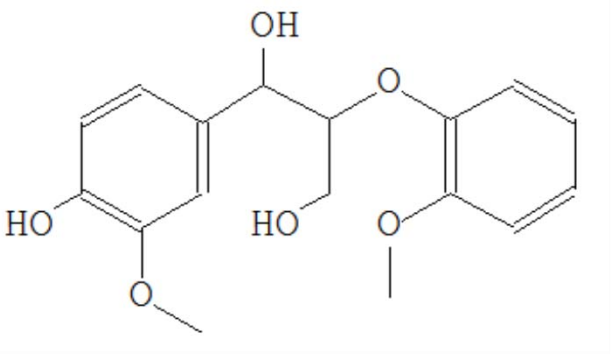

Fig. 1: Guaiacylglycerol-β-Guaiacyl Ether
($C_{17}H_{20}O_6$; Molecular Weight: 320; CAS No.: 7382-59-4)

Analysis Conditions

Table 1: Analysis Conditions

GC-MS:	GCMS-QP2010 Ultra
Column:	Stabilwax (30 m long, 0.25 mm I.D., df = 0.25 μm)
Glass Insert:	Split insert with wool (P/N: 225-20803-01)

[GC]	
Injection Unit Temp.:	230 °C
Column Oven Temp.:	50 °C (5 min) → (10 °C/min) → 250 °C (10 min)
Injection Mode:	Split (split ratio 10)
Carrier Gas Control:	Linear velocity (50.0 cm/sec)
Injection Volume:	0.5 μL

[MS]	
Interface Temp.:	250 °C
Ion Source Temp.:	230 °C
Measurement Mode:	Scan
Scan Event Time:	0.30 sec
Scan Mass Range:	*m/z* 15 to 550
Scan Speed:	2,000 u/sec

Analysis Results

Figs. 2 and 3 show the results obtained by degrading the lignin model compound guaiacylglycerol-β-guaiacyl ether with a solid acid catalyst in an aqueous solution, and then injecting the resulting solution directly into the GC-MS to measure it without pretreatment. As shown in Fig. 2, guaiacol (o-guaiacol), Peak 1, was mainly produced from the lignin model compound. Note that the actual lignin model compound used in this experiment was not detected by GC-MS due to degradation on vaporization at the injection port.

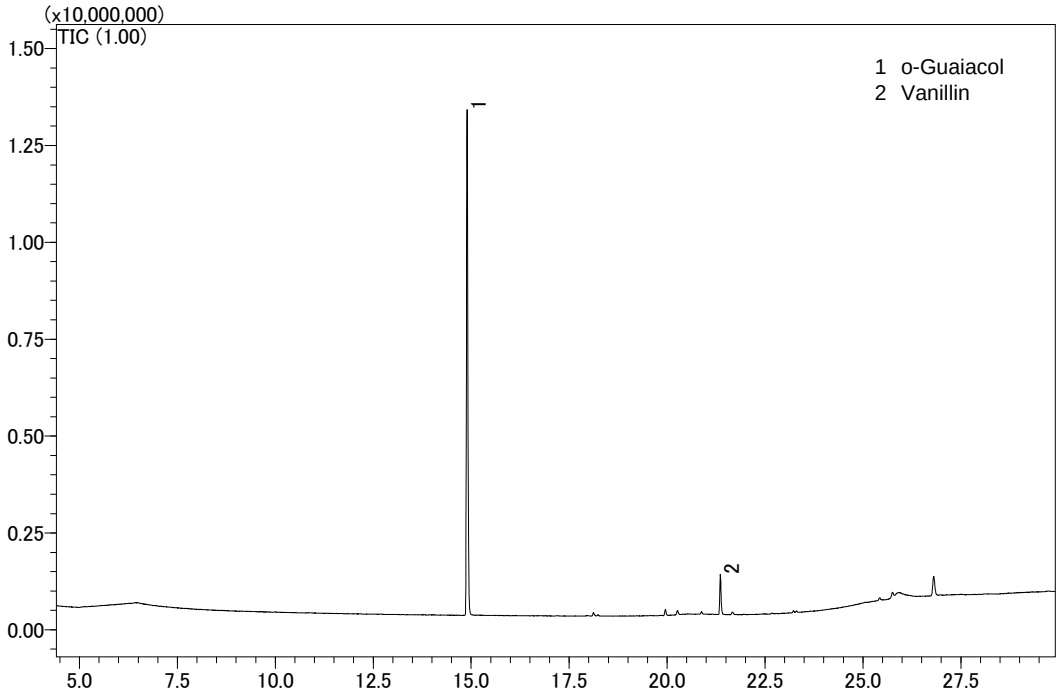

Fig. 2 Total Ion Current Chromatogram for the Degradation Solution

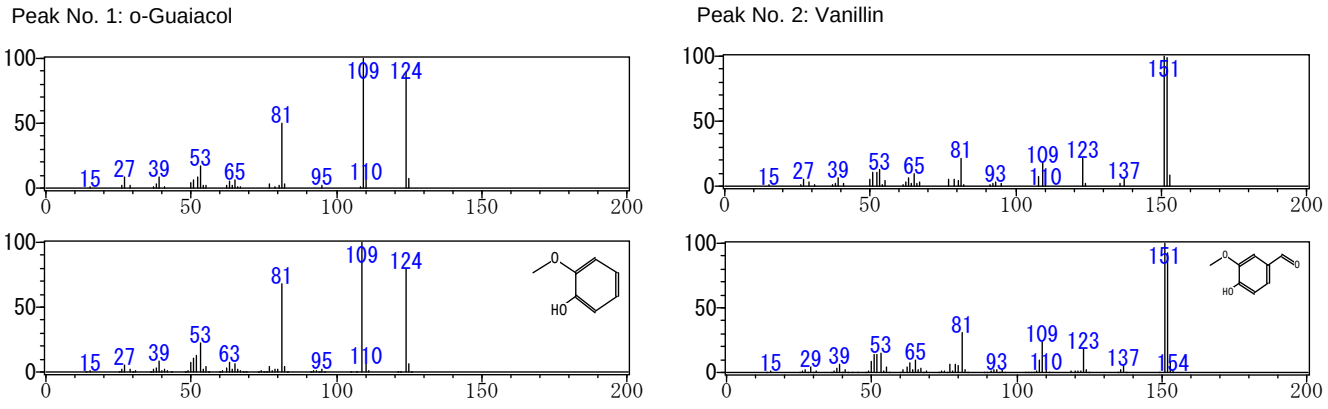

Fig. 3: Mass Spectra (Top: Mass Spectra for Peaks 1 and 2; Bottom: Library Search Results)

Note: Professor Michikazu Hara of the Materials & Structures Laboratory at the Tokyo Institute of Technology provided the sample.

First Edition: December, 2013

Shimadzu Corporation
www.shimadzu.com/an/

For Research Use Only. Not for use in diagnostic procedures.
The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu.
The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.
© Shimadzu Corporation, 2013