

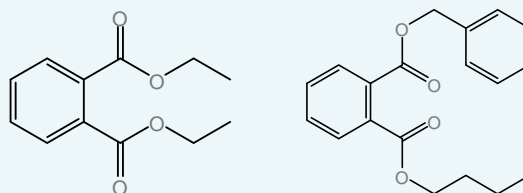


Analysis of Phthalate Esters in Sweet White Sake

Phthalate esters are a type of plasticizer added to plastics and rubbers for the purpose of providing ductility for further processing. The amount of addition is about 5 % – 50 %, and it is known that phthalate esters will leach out upon contact with many solvents. In recent years, phthalate esters have been suspected of endocrine disruption, and specification values have been placed on some components. For example, in June of 2012, the Chinese Health Ministry specified the limit for plasticizer residue as not more than DBP 0.3 mg/kg. However, DBP was detected in sweet white sake at a concentration exceeding the limit in November and newspapers, etc. reported the news. GC and GC-MS are used for the official analysis of phthalate esters. However, HPLC-UV was used here for analysis of 7 phthalate esters. DAD was used for the analysis of sweet white sake and the identification of detected peaks were also confirmed based on their spectra.

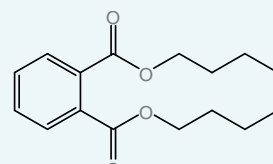
Component Names and Structural Formulas of Phthalate Esters

	Abbrev.	Component Name	MW
1	DEP	Diethyl phthalate	222.24
2	BBP	Butyl benzyl phthalate	312.36
3	DBP	Dibutyl phthalate	278.35
4	DPP	Dipentyl phthalate	306.44
5	DCHP	Dicyclohexyl phthalate	330.42
6	DHP	Dihexyl phthalate	334.46
7	DEHP	Bis(2-ethylhexyl)phthalate	390.56

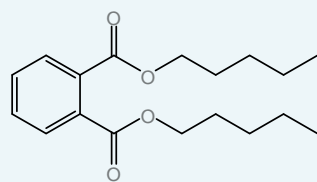


1 : DEP

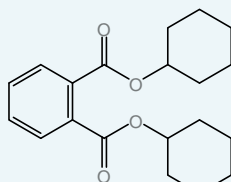
2 : BBP



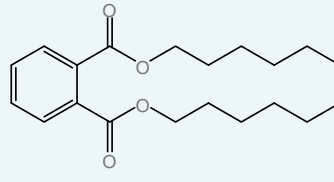
3 : DBP



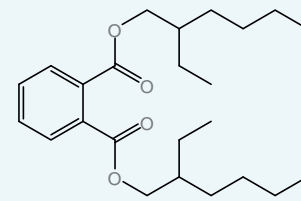
4 : DPP



5 : DCHP

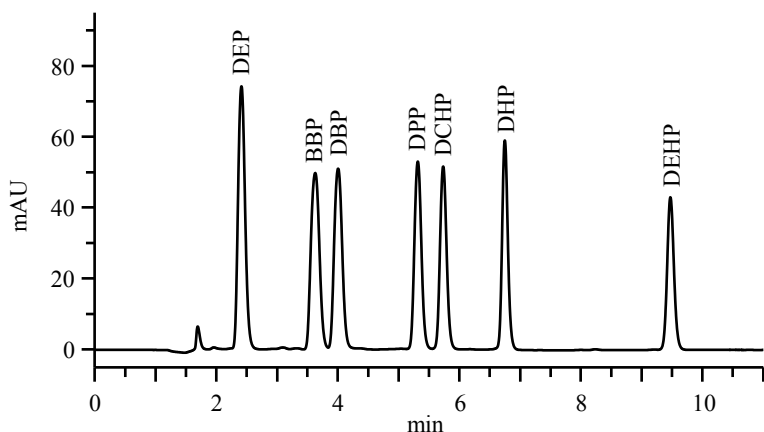


6 : DHP



7 : DEHP

Analysis of standard samples (by UV detector)



Chromatogram of standard samples
(Concentration of each : 5 µg/mL)

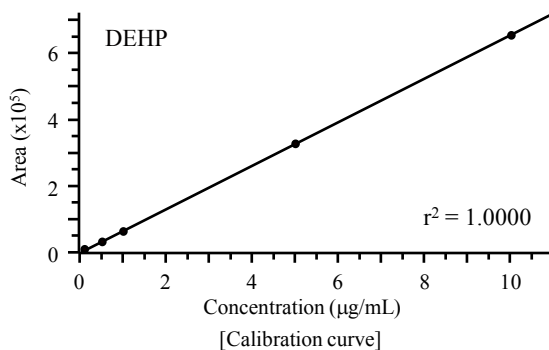
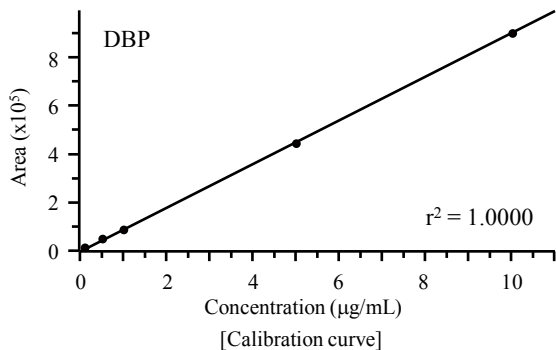
[Analytical conditions]

Column : HITACHI LaChrom C18 (5 µm)
4.6 mm I.D. × 150 mm
Eluent : (A) H₂O
(B) CH₃CN
(0 min) B 85 % → (3 min) B 95 %
→ (4 - 8 min) B 100 % →
(8.1 - 15 min) B 85 %
Flow rate : 1.0 mL/min
Column temp. : 30 °C
Detection : UV 224 nm
Injection vol. : 50 µL



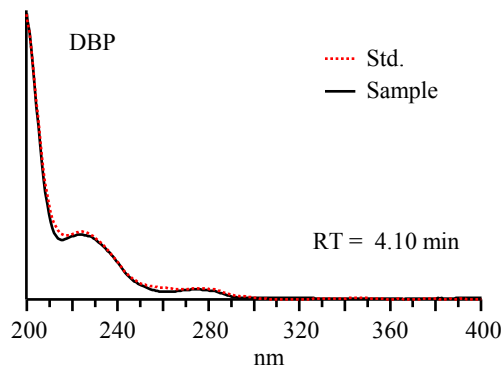
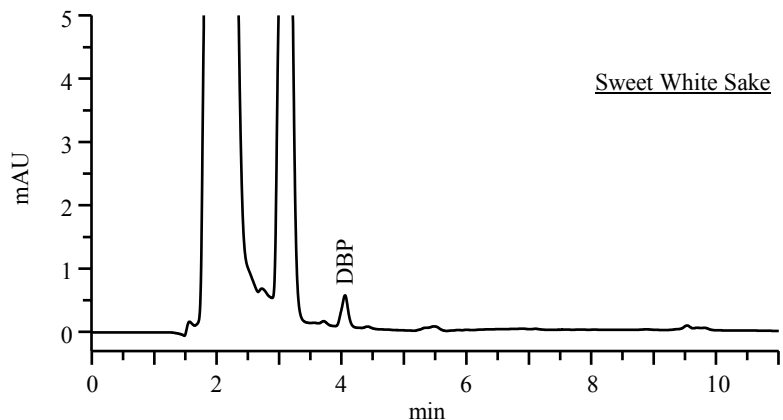
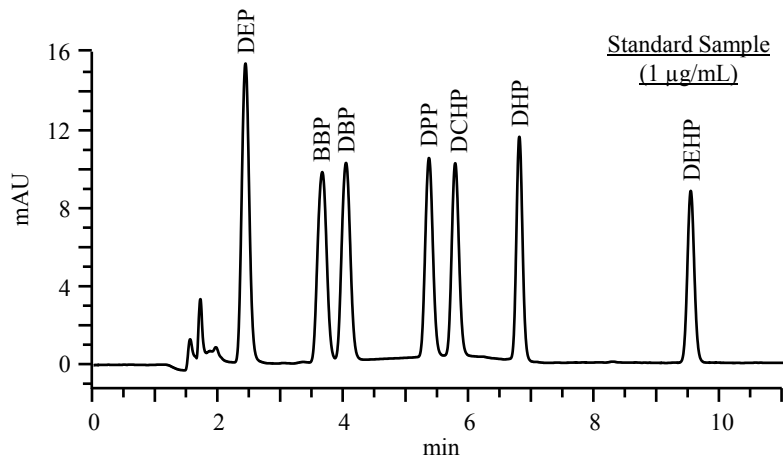
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Linearity



For each of the 7 components analyzed, good linearity with a correlation coefficient of 0.9999 – 1.0000 was obtained over the concentration range of 0.1 – 10 $\mu\text{g/mL}$.

Analysis of Phthalate Esters in Standard Samples and Sweet White Sake (by DAD)



[Analysis of Standard Samples and Sweet White Sake (injection of 50 μL each)]

A peak considered to be DBP was found in the sweet white sake analyzed. When its spectrum was compared with the spectrum of the DBP standard sample, the shapes were found to be equivalent. The ability of qualitative analysis can be improved by using DAD as seen above.

System configuration : Primaide 1110 Pump, 1210 Auto sampler, 1310 Column Oven, 1410 UV, 1430 DAD

NOTE : These data are an example of measurement; the individual values cannot be guaranteed.
The system is for research use only, and is not intended for any animal or human therapeutic or diagnostic use.