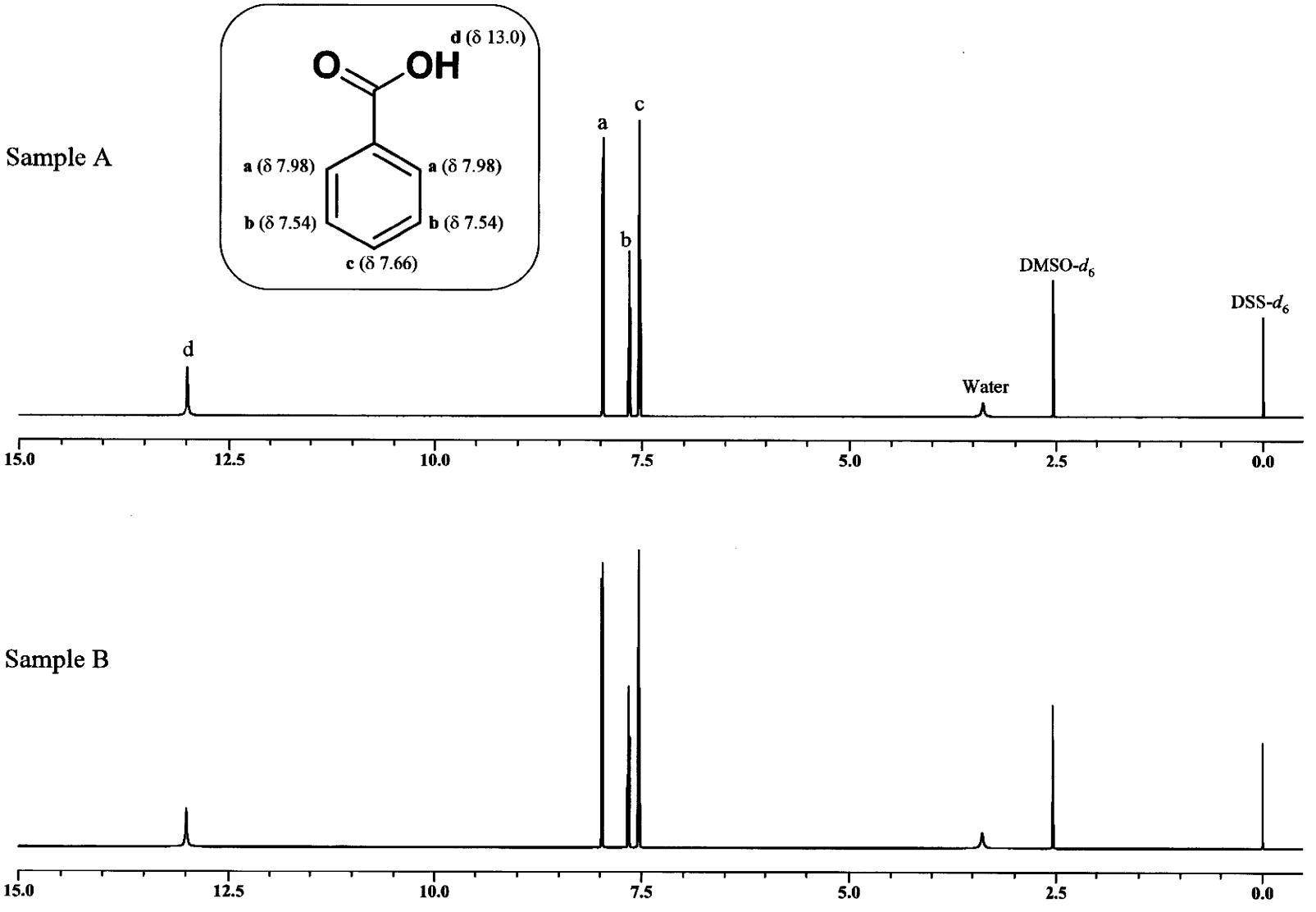

Fig.7 ^{1}H NMR spectra of analytical grade of benzoic acid (samples A and B) in DMSO-d_6 containing DSS-d_6

Table 3 Content of benzoic acid of samples A and B determined from qNMR method

	Sample A		Sample B	
Signal (ppm)	Content (%)	RSD (%)	Content (%)	RSD (%)
7.54	99.6	0.1	99.7	0.0
7.66	99.4	0.3	99.4	0.1
7.98	99.7	0.2	99.8	0.1
Mean	99.6	0.2	99.6	0.1

Table 4 Comparison of content of sorbic acid determined from neutralization titration method and qNMR method

	Content (%)	RSD (%)
Neutralization titration	99.4	0.1
qNMR	99.3	0.6

Table 5 Comparison of content of benzoic acid determined from neutralization titration method and qNMR method

	Content (%)	RSD (%)
Neutralization titration	99.7	0.1
qNMR	99.6	0.2

Table 6 Recoveries of sorbic acid from samples

Sample	Spiked level (g/kg)				Upper limit for usage (g/kg)
	0.13 (0.013)*		Upper limit		
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	
Cheese	98.8	1.6	97.1	3.1	3.0
Fish paste	84.4	4.0	100.2	0.5	2.0
Sausage	81.1	4.3	89.1	2.1	2.0
Dried cuttlefish	99.7	0.6	94.5	0.8	1.5
Syrup	96.2	1.8	99.5	0.9	1.0
Soybean sauce pickled vegetable	80.3	1.0	99.6	0.5	1.0
Jam	98.9	2.8	99.2	0.8	1.0
Soybean paste	90.5	6.7	92.8	2.3	1.0
Noodles soup	86.3	4.6	97.7	0.7	0.50
Ketchup	93.5	6.8	98.7	1.2	0.50
Lactic acid bacteria beverage	86.4	2.0	93.3	3.0	0.050

The recoveries were calculated from the signal of δ 7.18 ppm (dried cuttlefish and lactic acid bacteria beverage) and δ 5.79 ppm (the other samples)
* Lactic acid bacteria beverage

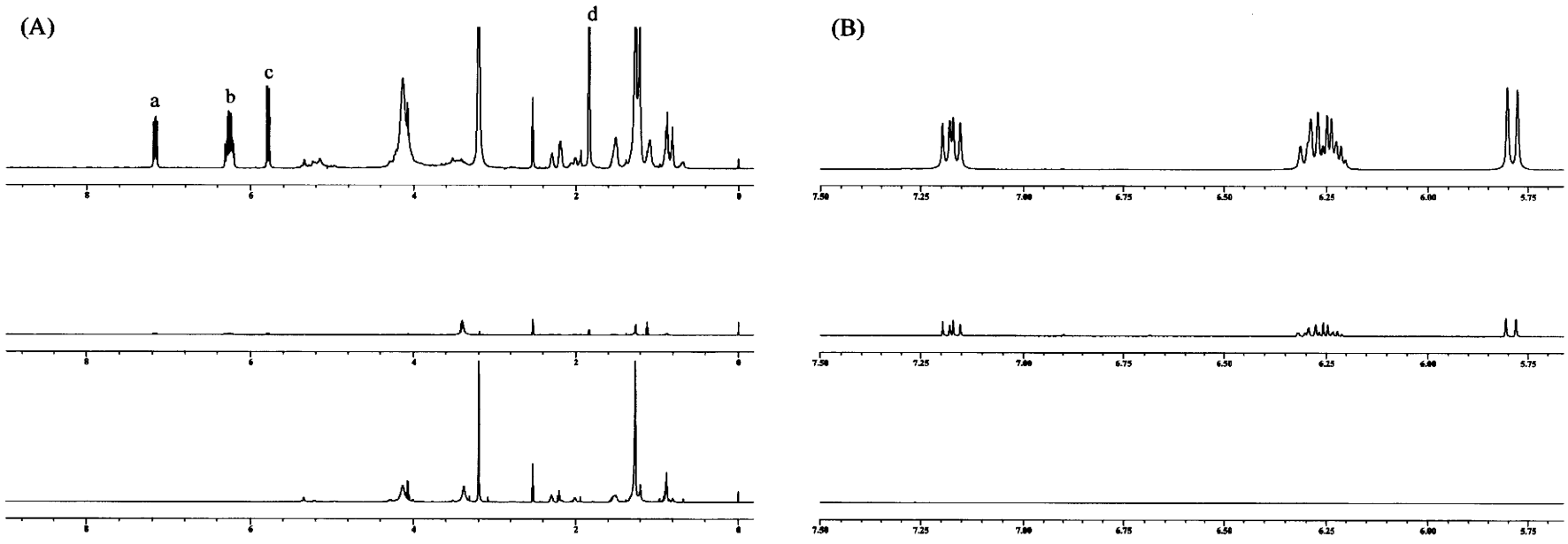

Fig.8 ^{1}H NMR spectra of sample solution from cheese with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 3.0 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

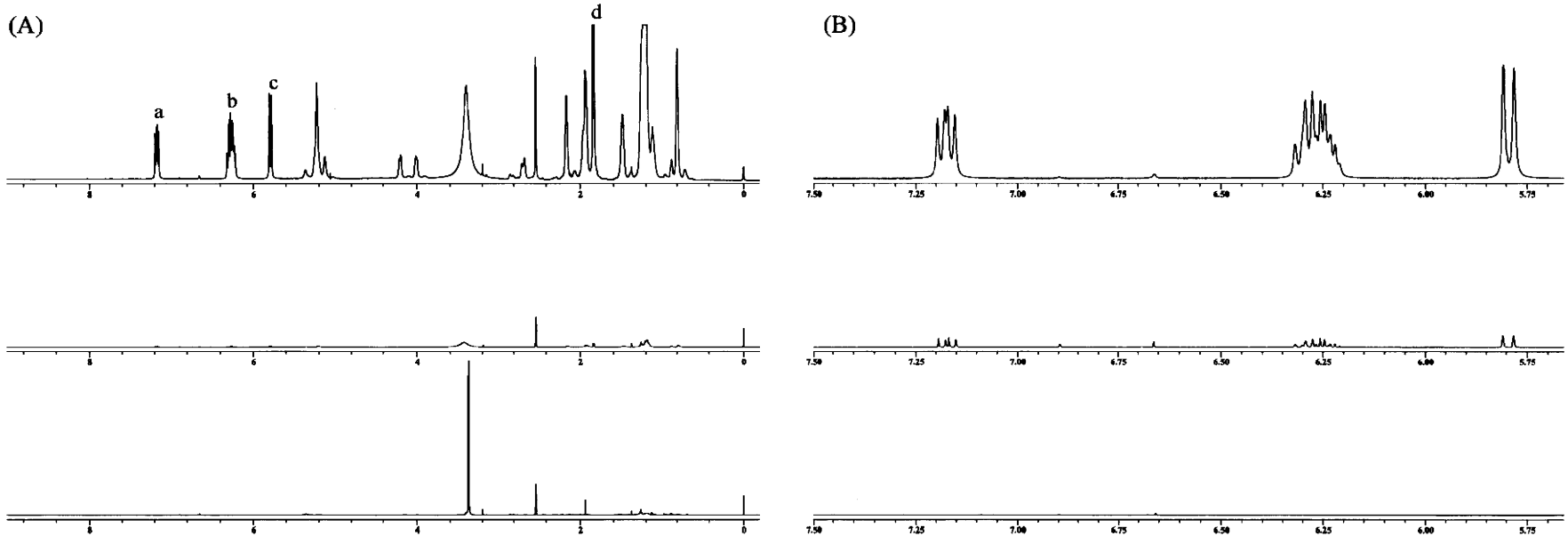

Fig.9 ^{1}H NMR spectra of sample solution from fish paste with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)
Spiked sample with sorbic acid at the concentrations of 2.0 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

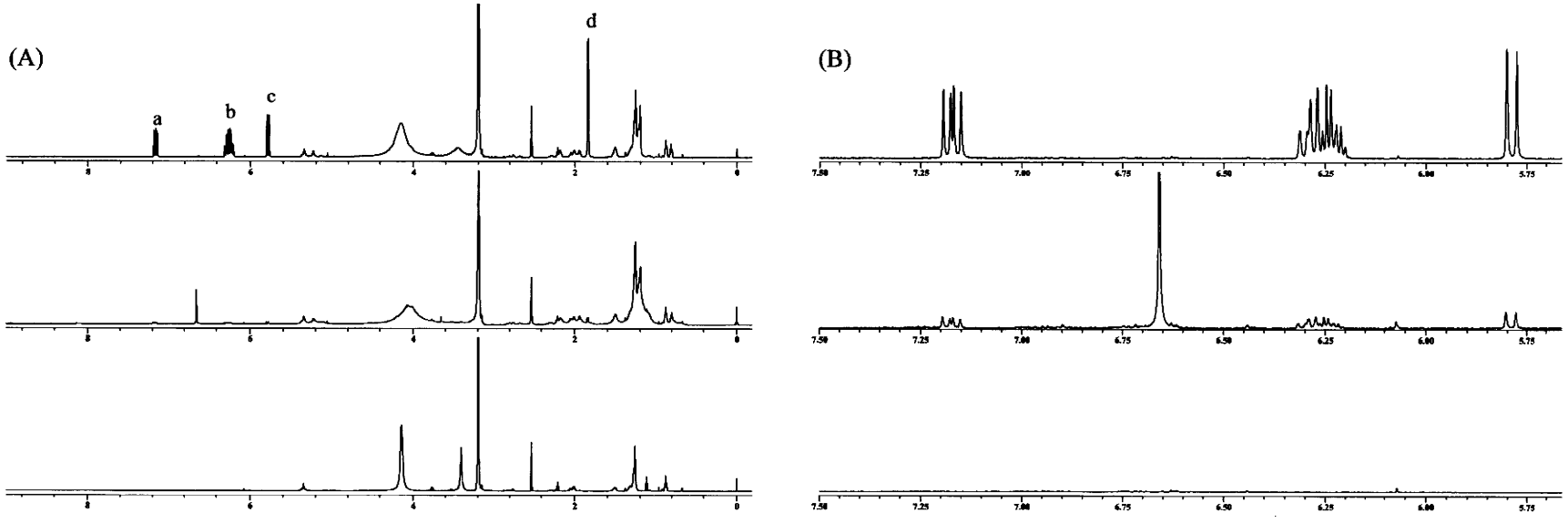

Fig. 10 ^{1}H NMR spectra of sample solution from sausage with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 2.0 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)

a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

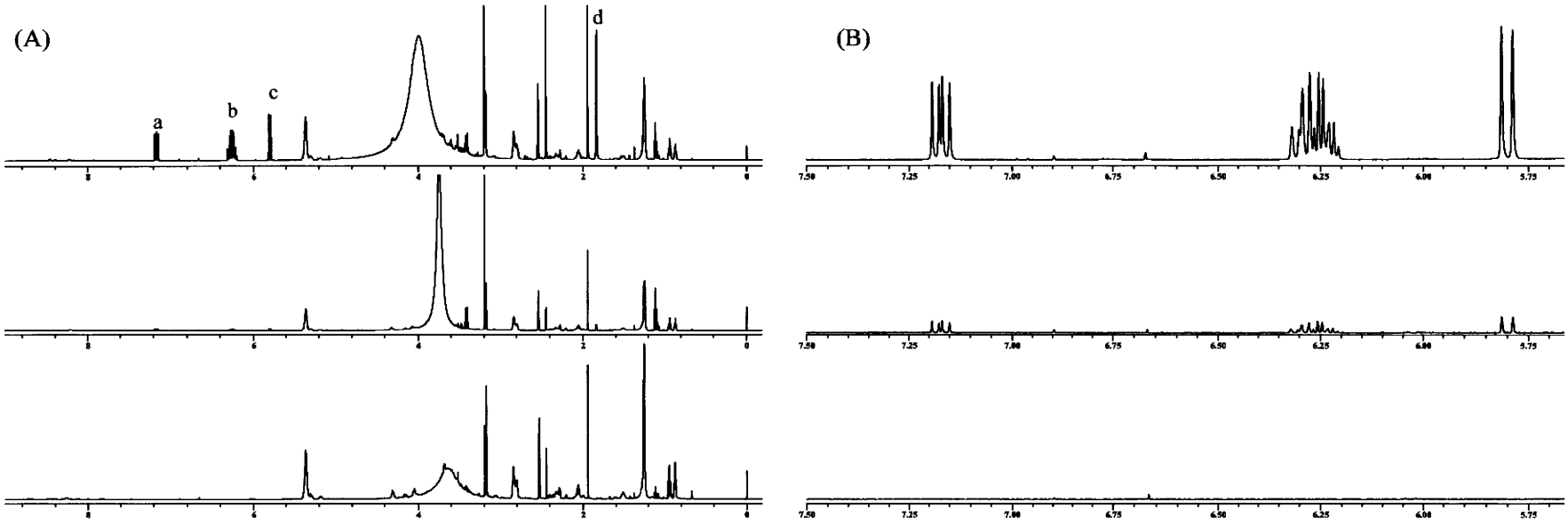

Fig.11 ^{1}H NMR spectra of sample solution from dried cuttlefish with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 1.5 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)

a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

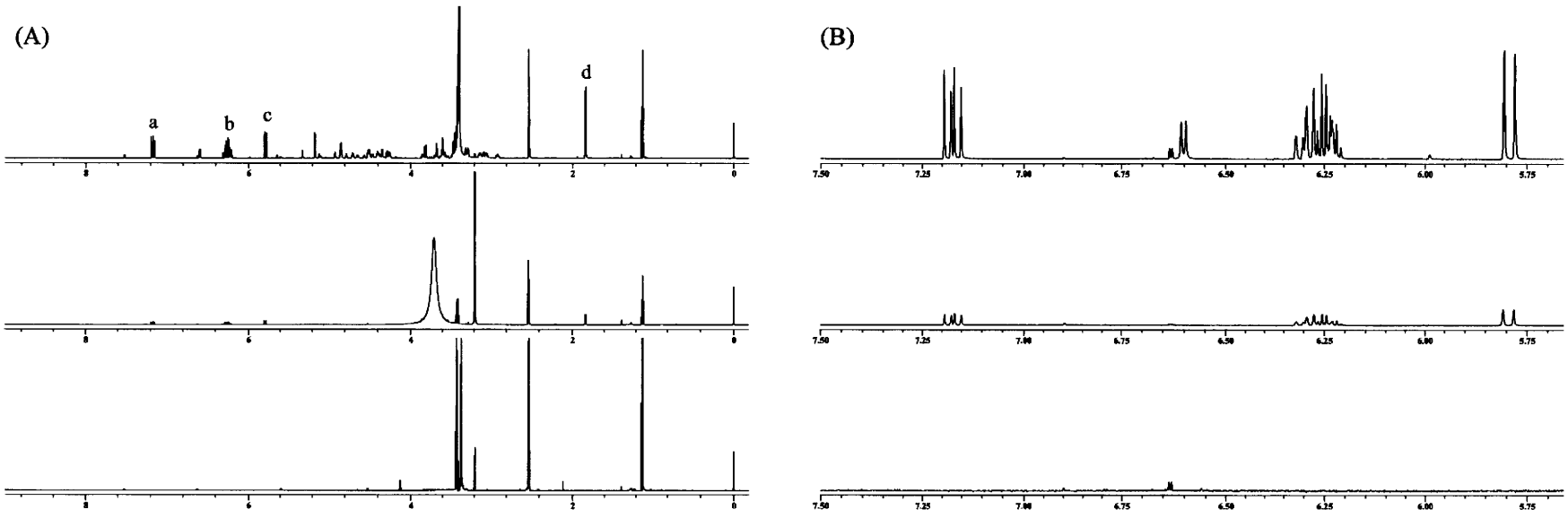

Fig.12 ^{1}H NMR spectra of sample solution from syrup with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)
Spiked sample with sorbic acid at the concentrations of 1.0 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

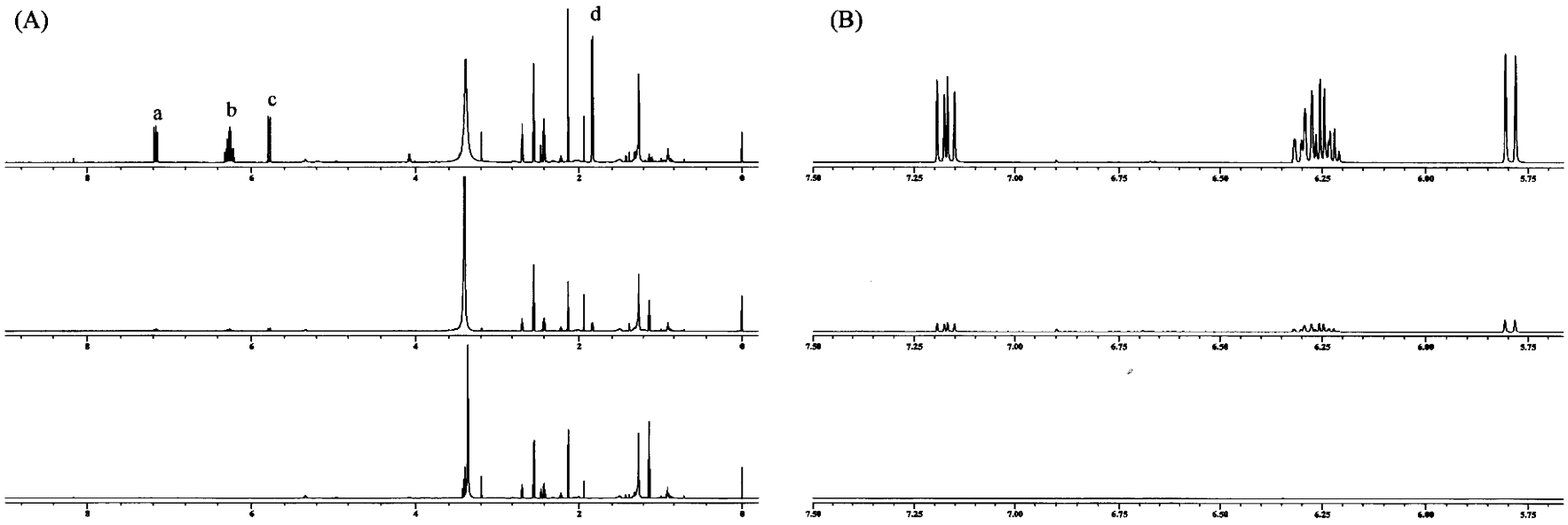

Fig.13 ^{1}H NMR spectra of sample solution from soybean sauce pickled vegetable with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 1.0 g/kg (upper) and 0.13 g/kg (middle); blank sample(lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

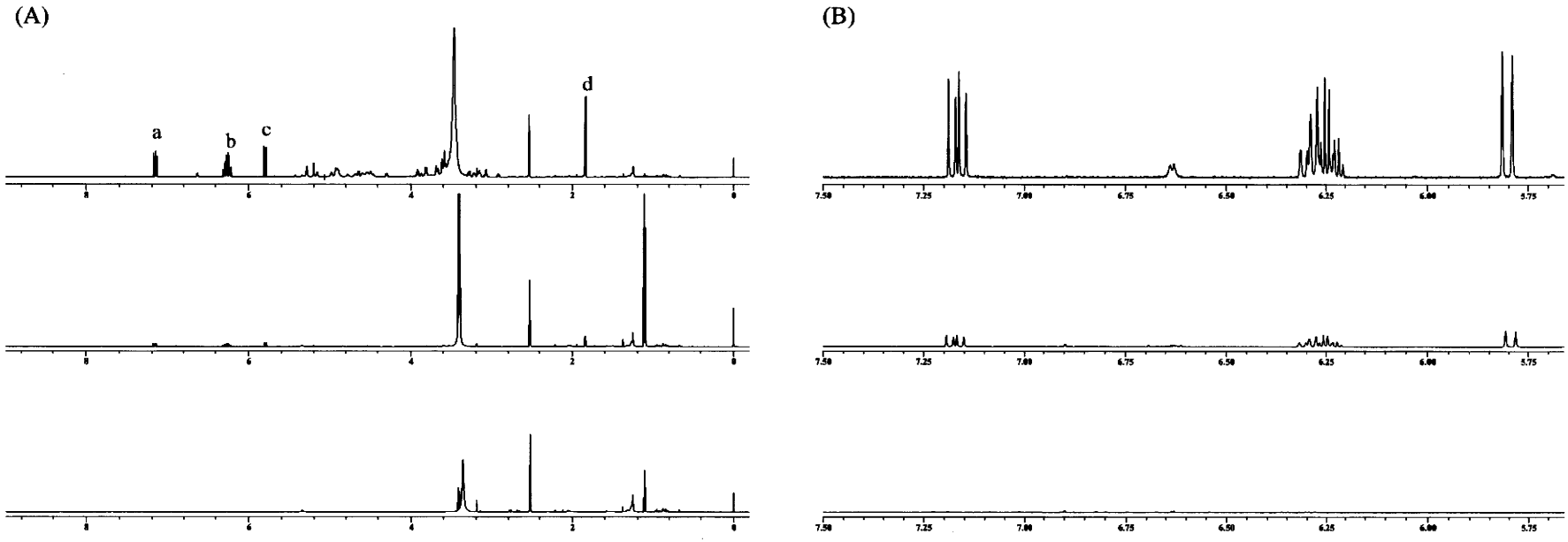

Fig.14 ^{1}H NMR spectra of sample solution from jam with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 1.0 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

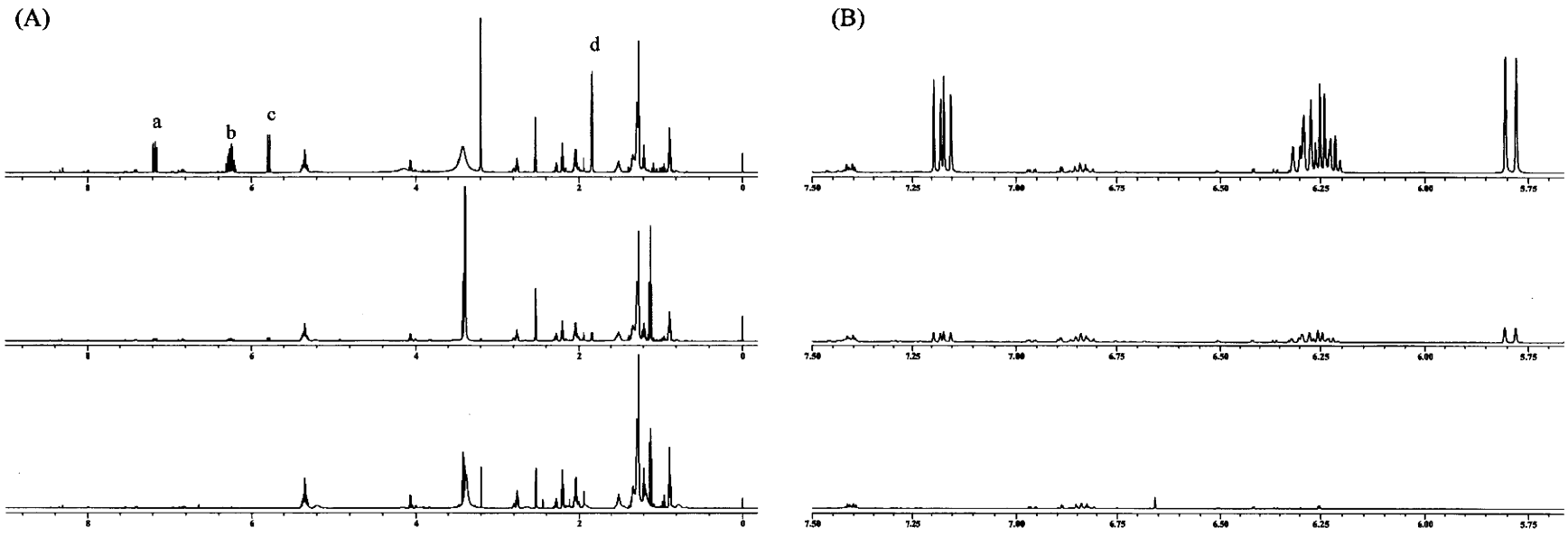

Fig.15 ^{1}H NMR spectra of sample solution from soybean paste with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 1.0 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)

a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

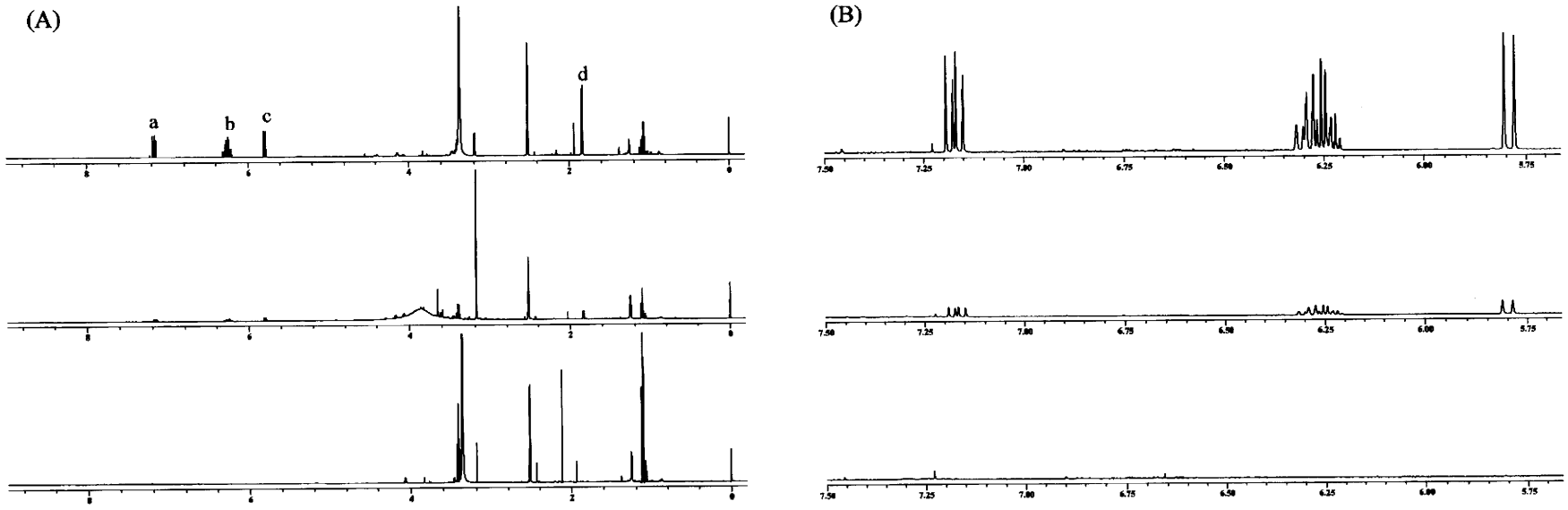

Fig.15 ^{1}H NMR spectra of sample solution from noodles soup with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 0.50 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

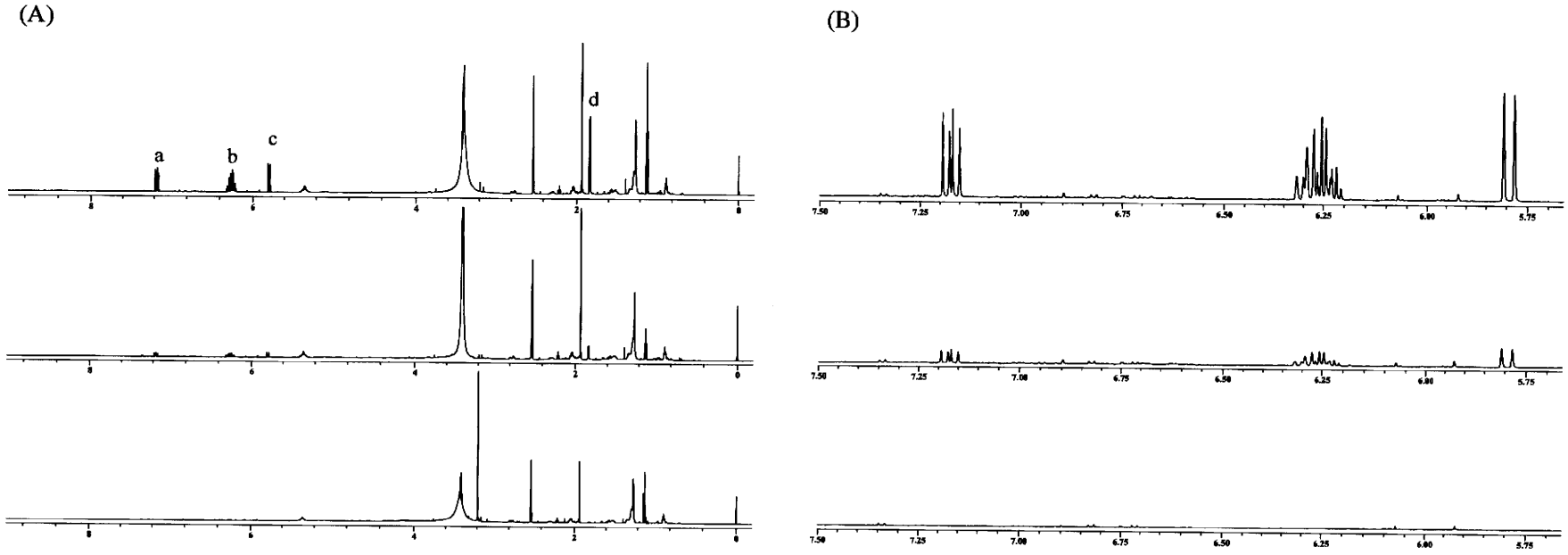

Fig.16 ^{1}H NMR spectra of sample solution from ketchup with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)
Spiked sample with sorbic acid at the concentrations of 0.50 g/kg (upper) and 0.13 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

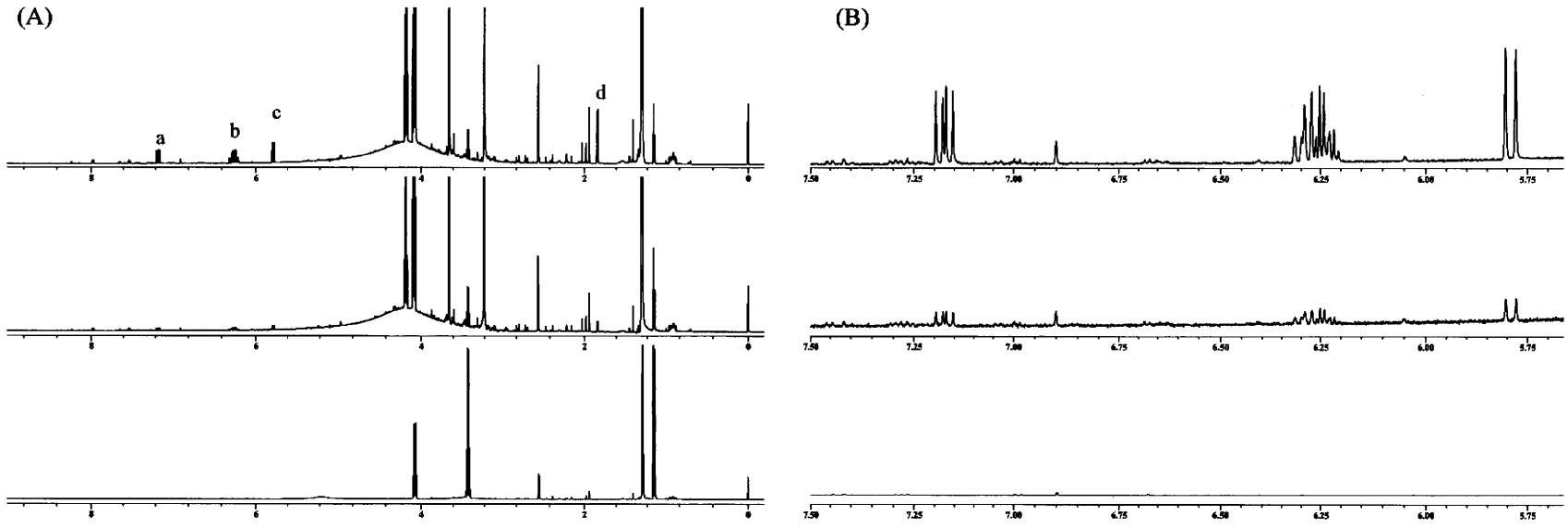

Fig.17 ^{1}H NMR spectra of sample solution from lactic acid bacteria beverage with or without spiked sorbic acid (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

Spiked sample with sorbic acid at the concentrations of 0.050 g/kg (upper) and 0.013 g/kg (middle); blank sample (lower)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

Table 7 Comparison of sorbic acid contents in commercial food determined by two methods

Sample	Developed method (Solvent extraction/qNMR)			Official method (Steam distillation/HPLC)	
	Target signal (δ, ppm)	Content (g/kg)	RSD (%)	Content (g/kg)	RSD (%)
Fish paste	5.79	1.46	2.9	1.42	3.1
Syrup	5.79	0.66	1.6	0.66	0.9
Jam	5.79	0.62	2.8	0.59	2.8
Dried cuttlefish	7.18	0.72	1.4	0.62	0.5
Sausage	5.79	0.68	3.4	0.75	1.2

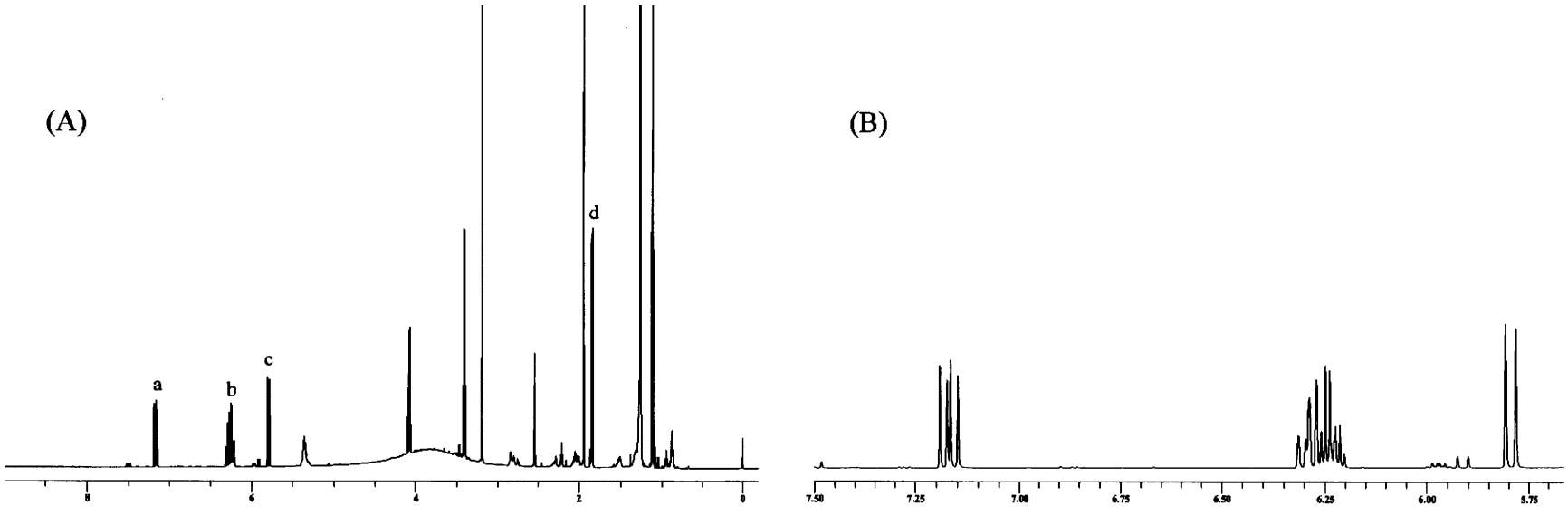

Fig.18 ^{1}H NMR spectrum of sample solution from fish paste (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

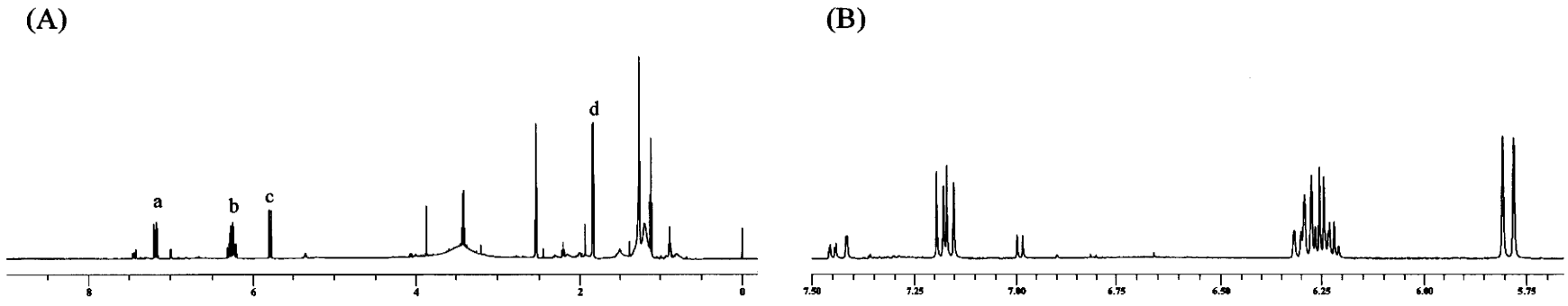

Fig.19 ^{1}H NMR spectrum of sample solution from syrup (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

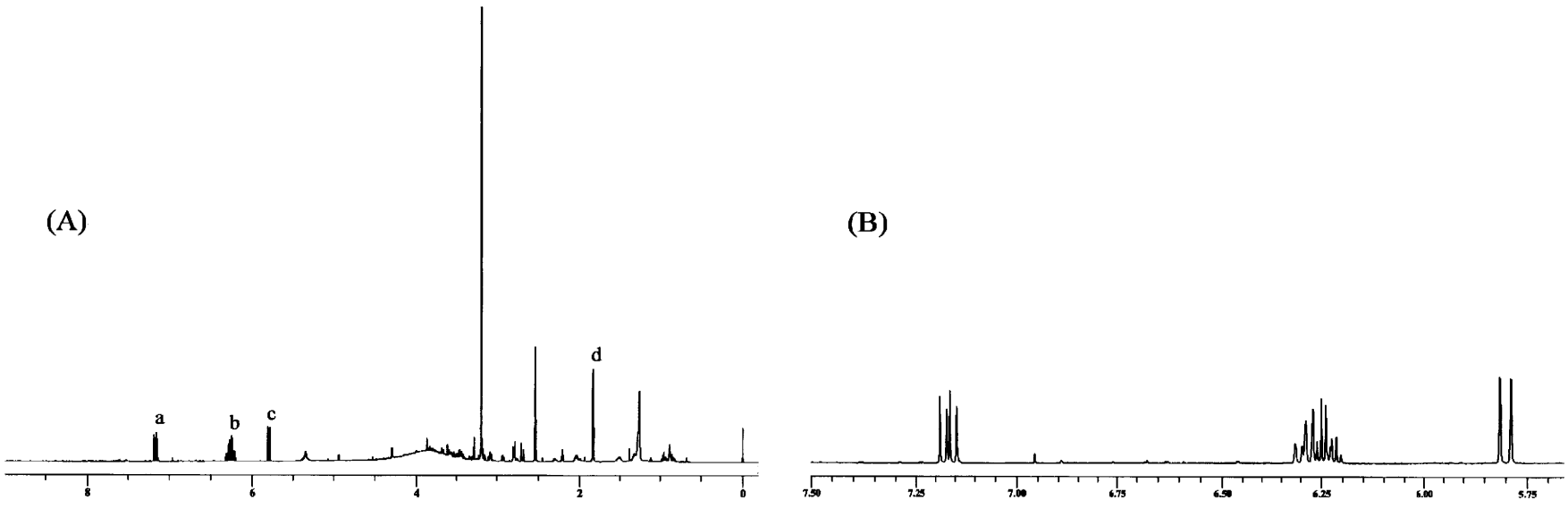

Fig.20 ^{1}H NMR spectrum of sample solution from jam (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)
a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)

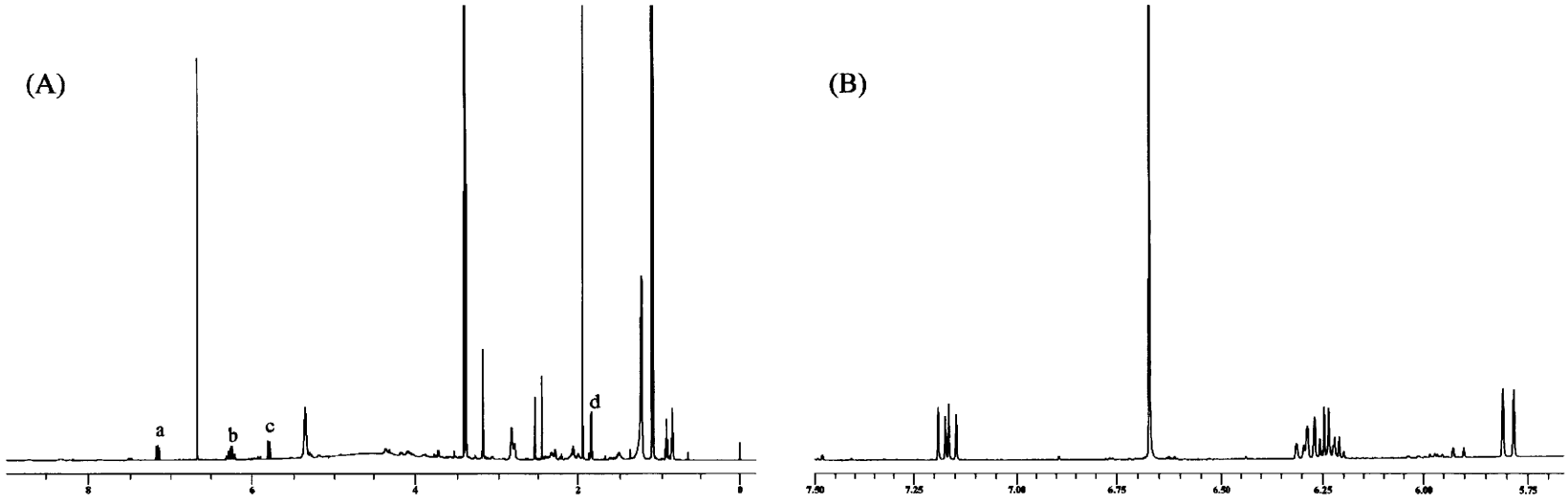

Fig.21 ^{1}H NMR spectrum of sample solution from dried cuttlefish (A) and enlarged view in the range of δ 5.6-7.5 ppm (B)

a: δ 7.18 ppm, b: δ 6.26 ppm, c: δ 5.79 ppm, d: δ 1.84 ppm (see Fig.1)