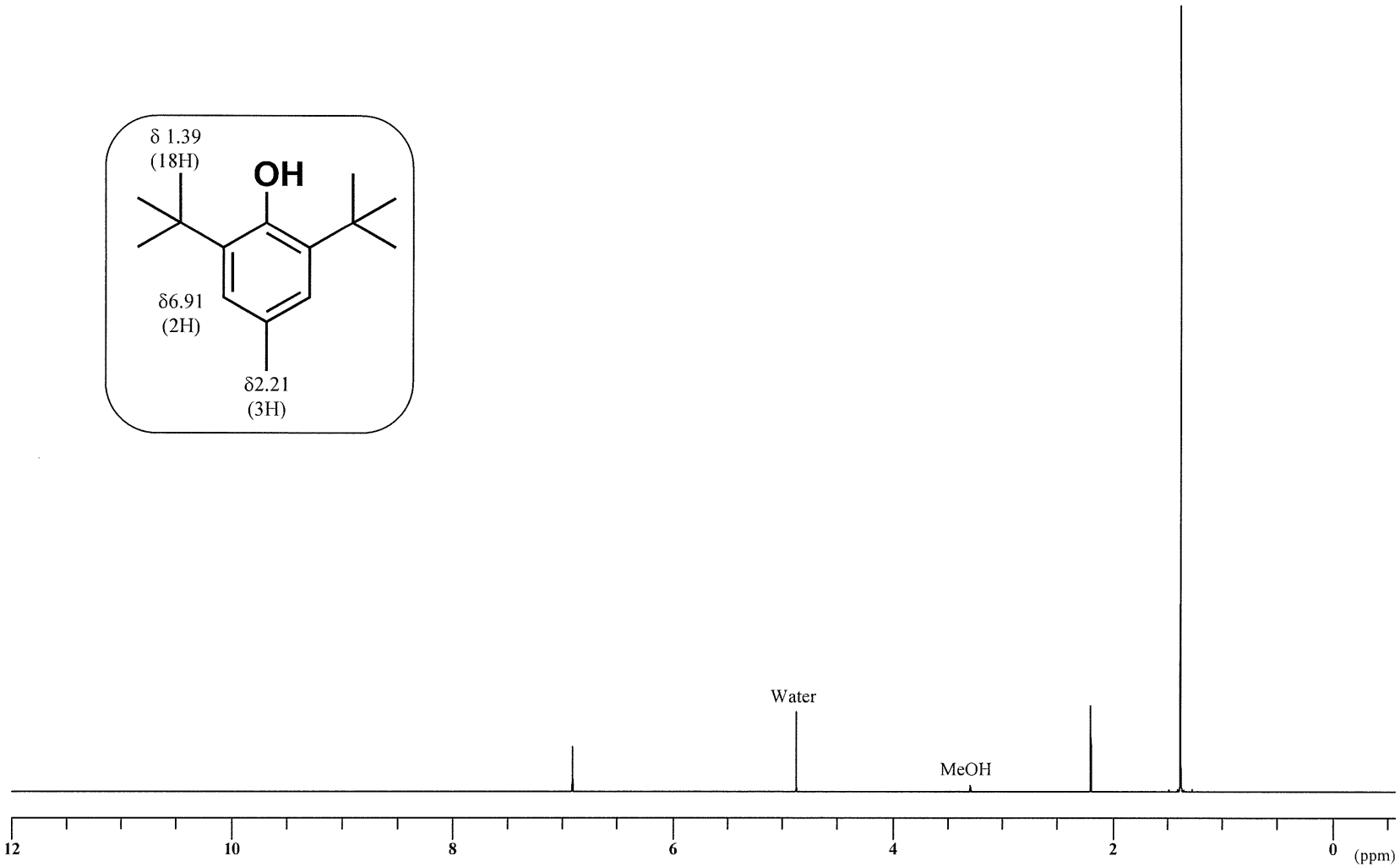

Fig.1 ^{1}H NMR spectrum of BHT in MeOH-d_4

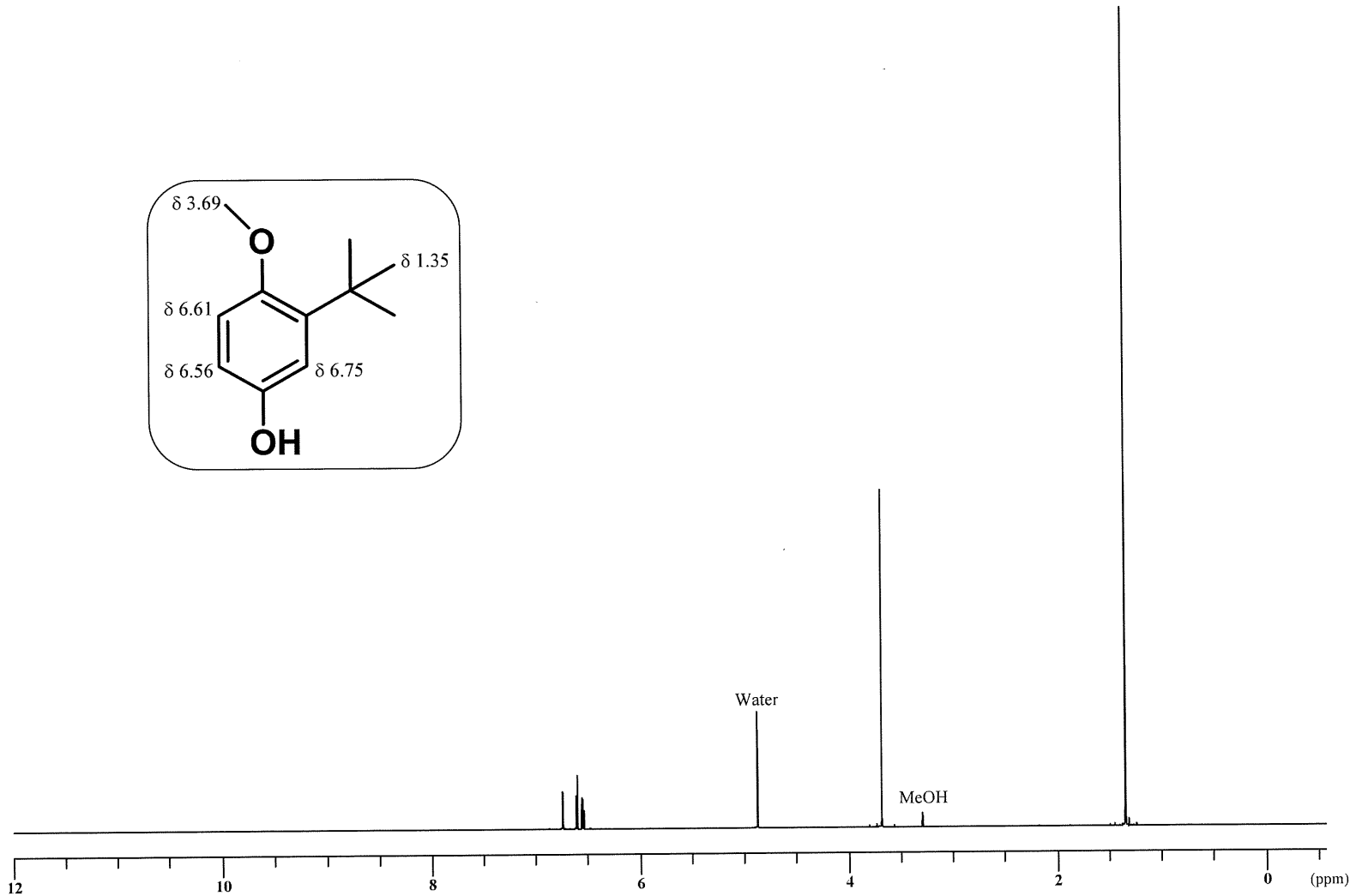

Fig.2 ^{1}H NMR spectrum of BHA in MeOH-d_4

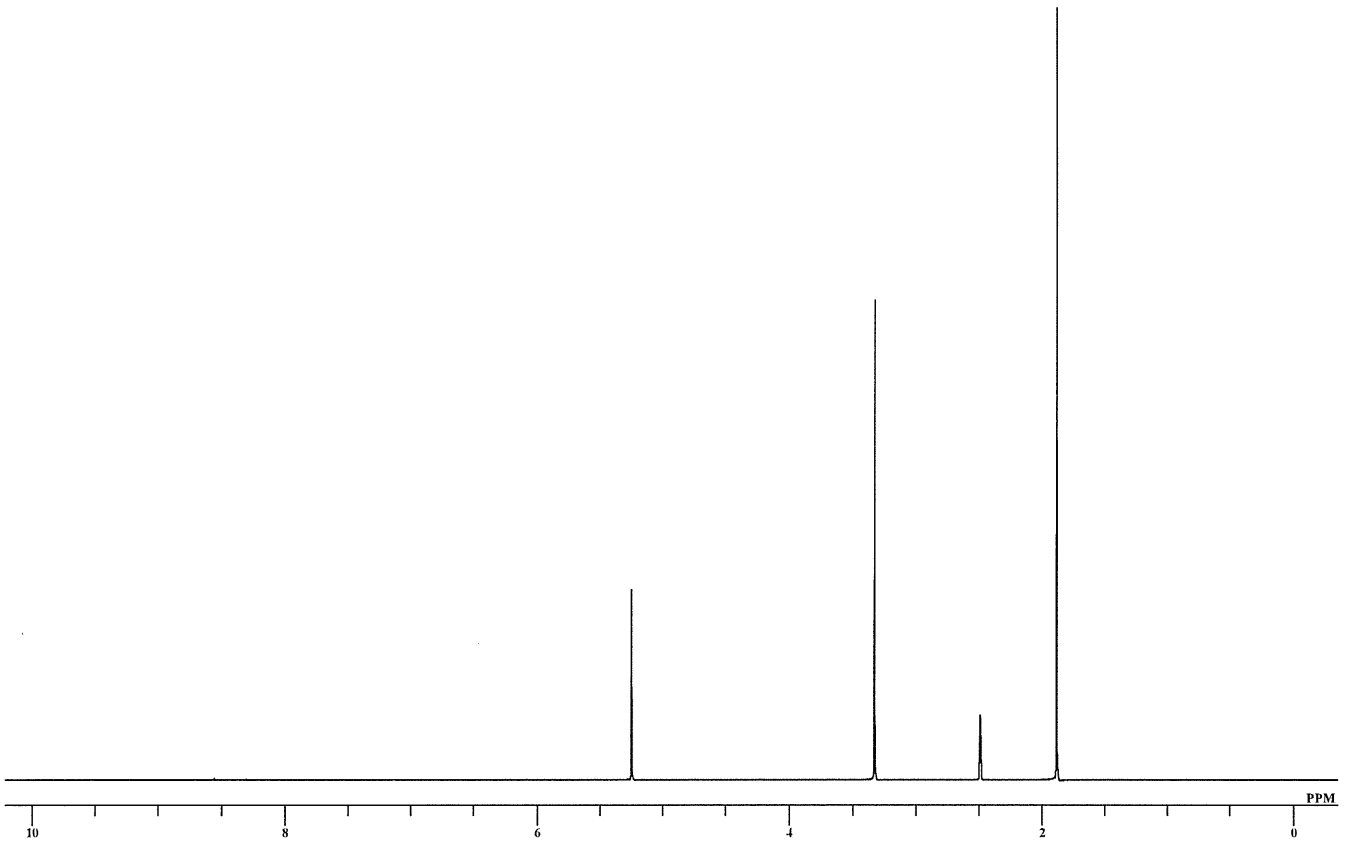

Fig.3 ^{1}H NMR spectrum of acesulfame potassium in DMSO-d_6

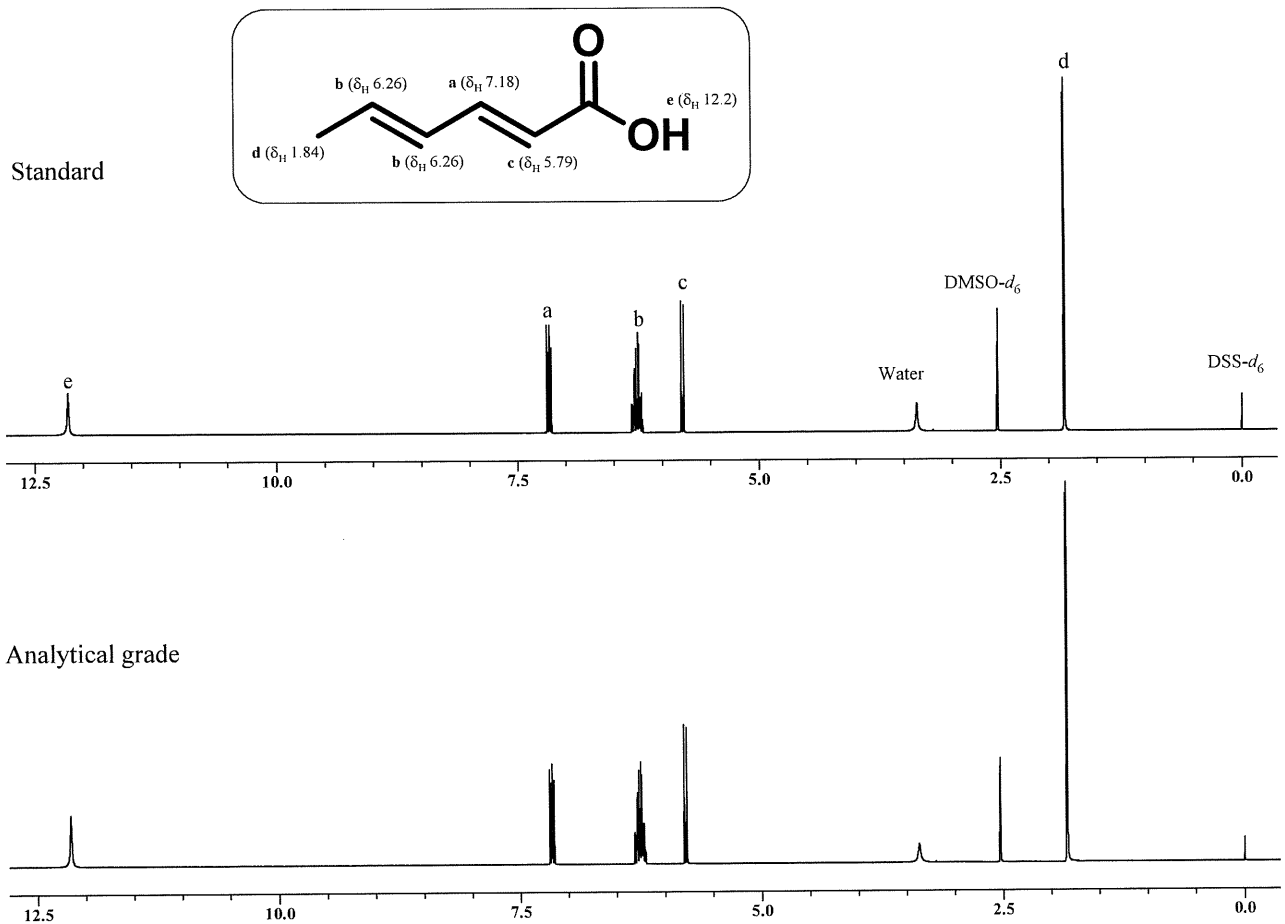

Fig.4 ^{1}H NMR spectra of sorbic acid standard and reagent (analytical grade) in DMSO-d_6 containing DSS-d_6

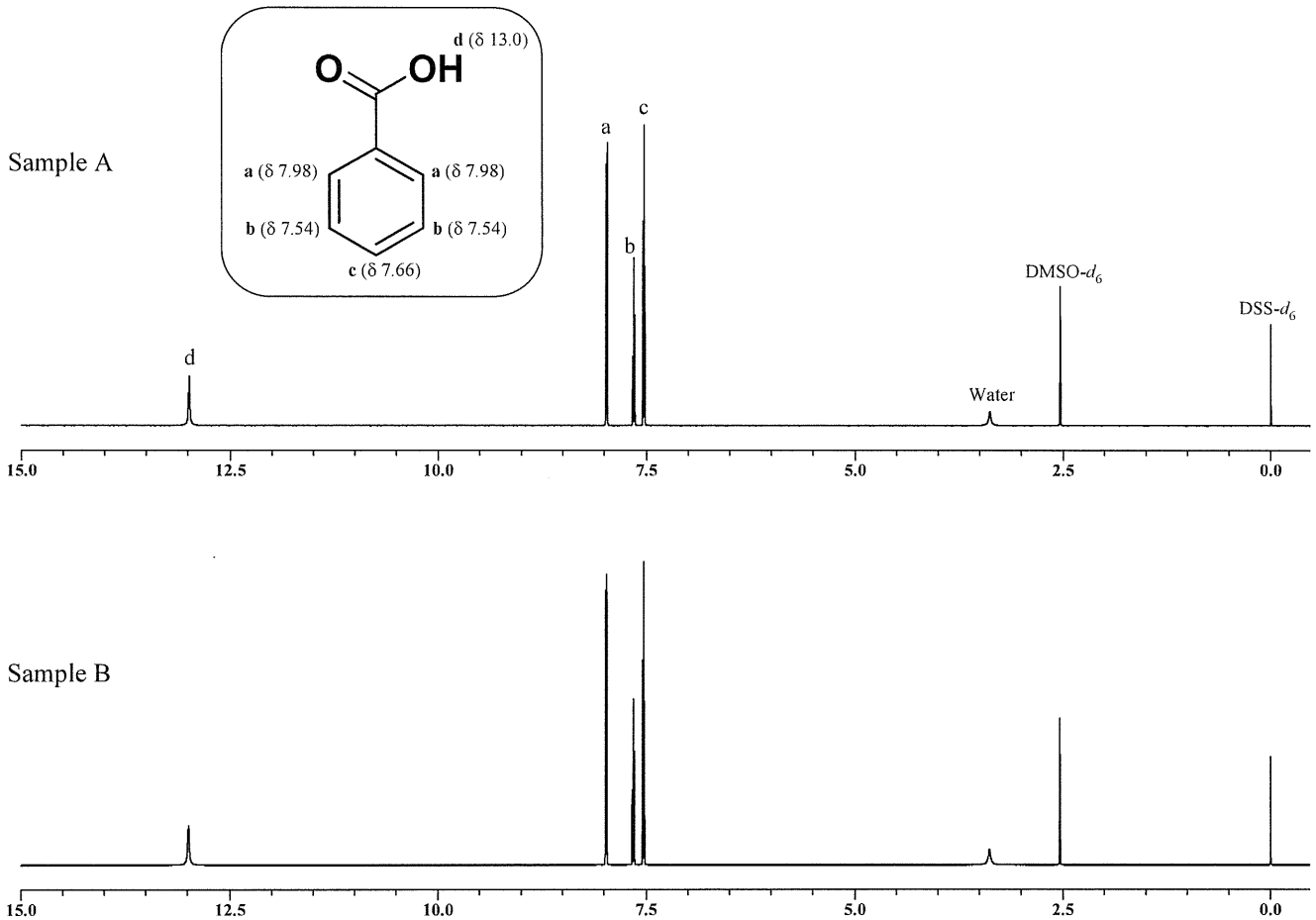

Fig.5 ^{1}H NMR spectra of benzoic acid reagents (analytical grade) in DMSO-d_6 containing DSS-d_6

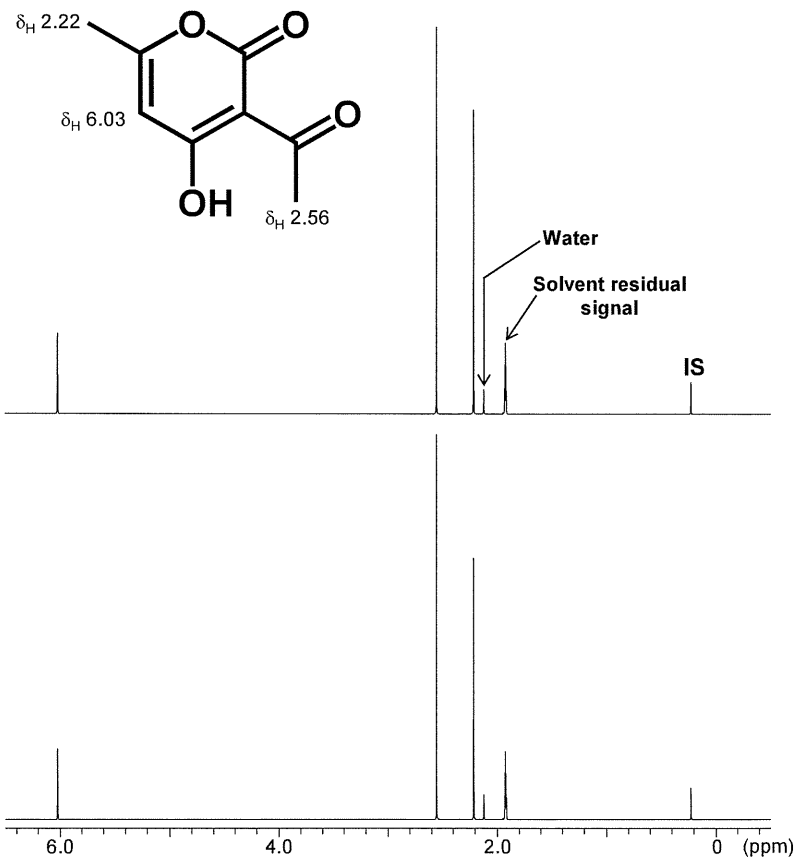

Fig.6 ^{1}H NMR spectra of dehydroacetic acid standard (top) and reagent (bottom) in acetnitrile-d_3 containing 1,4-BTMSB-d_4

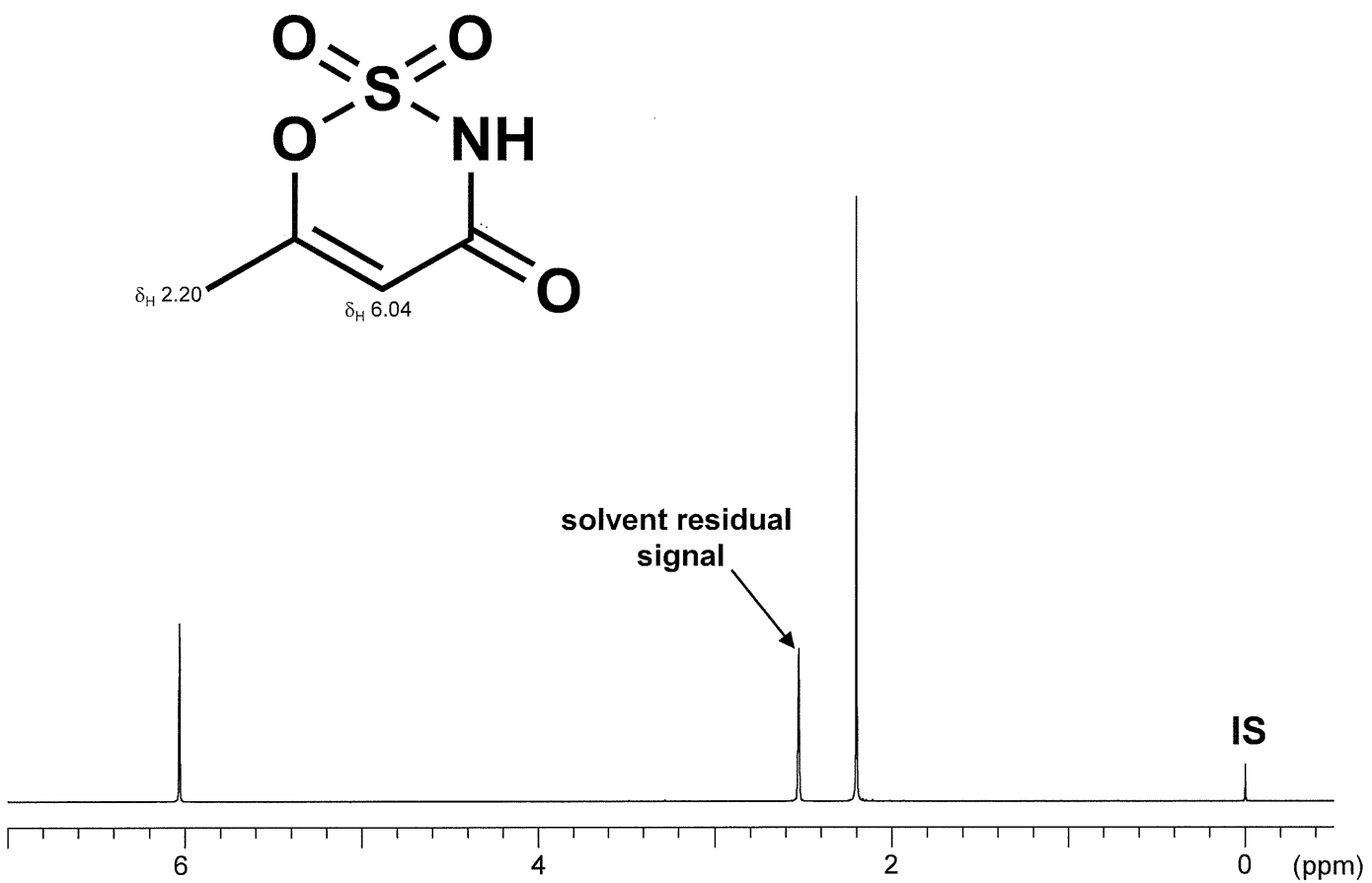

Fig.7 ^{1}H NMR spectum of acesulfame in DMSO-d_6 containing DSS-d_6

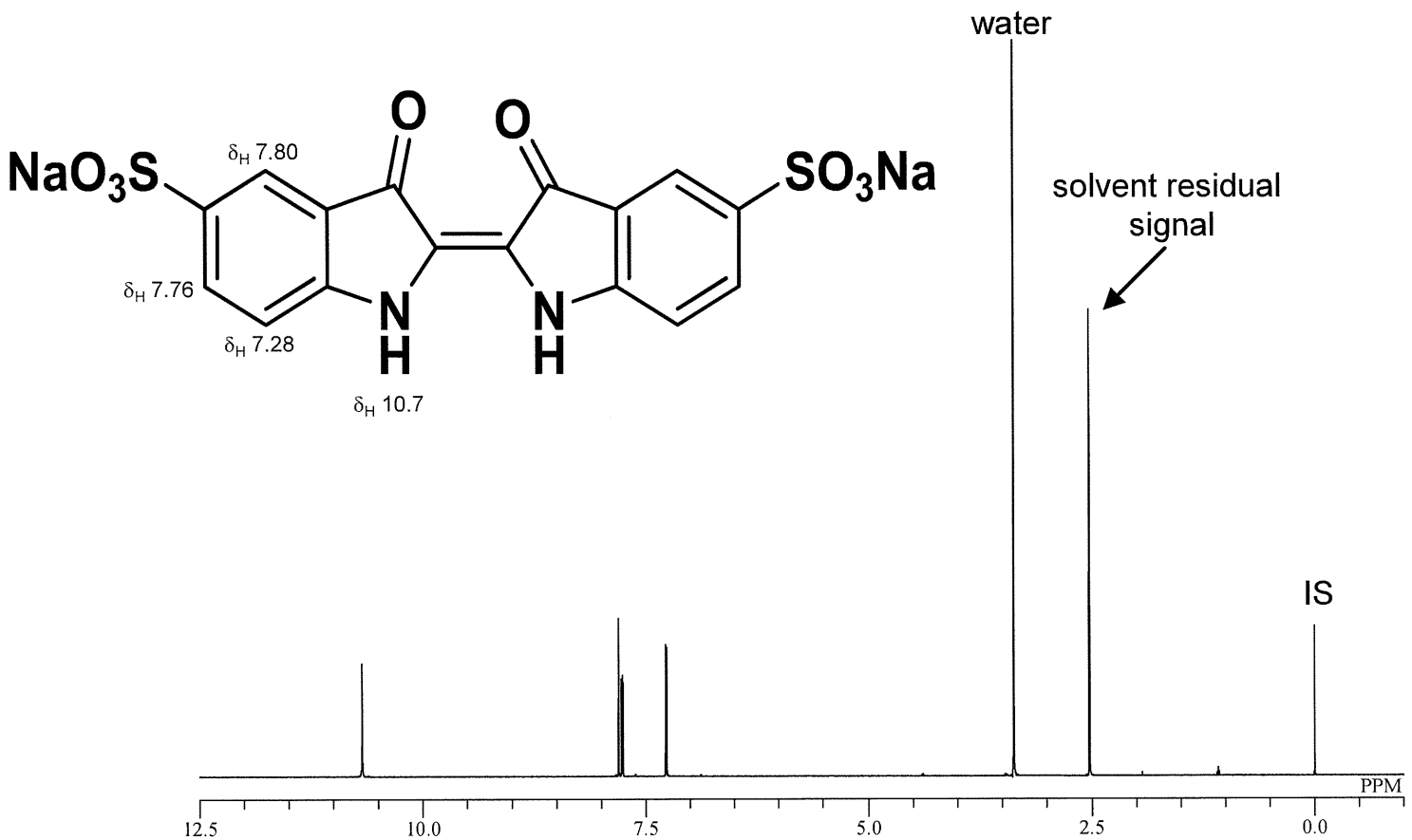

Fig.8 ^{1}H NMR spectrum of Food color B2 in DMSO-d_6 containing DSS-d_6

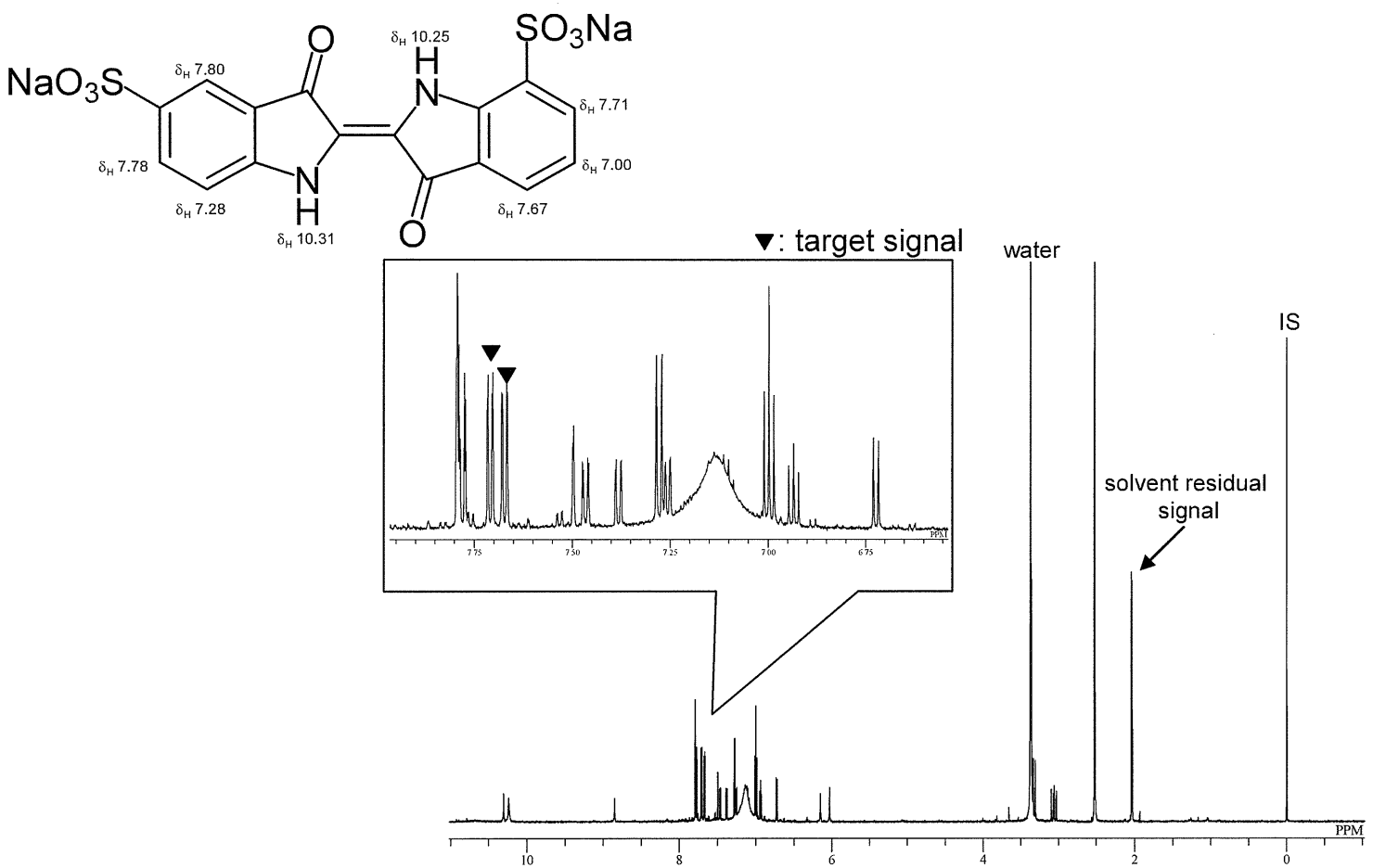

Fig.9 ^{1}H NMR spectrum of sodium salt of 2-(1,3-dihydro-3-oxo-7-sulfo-2H-indol-2-ylidene)-2,3-dihydro-3-oxo-1H-indole-5-sulfonic acid in DMSO-d_6 containing DSS-d_6

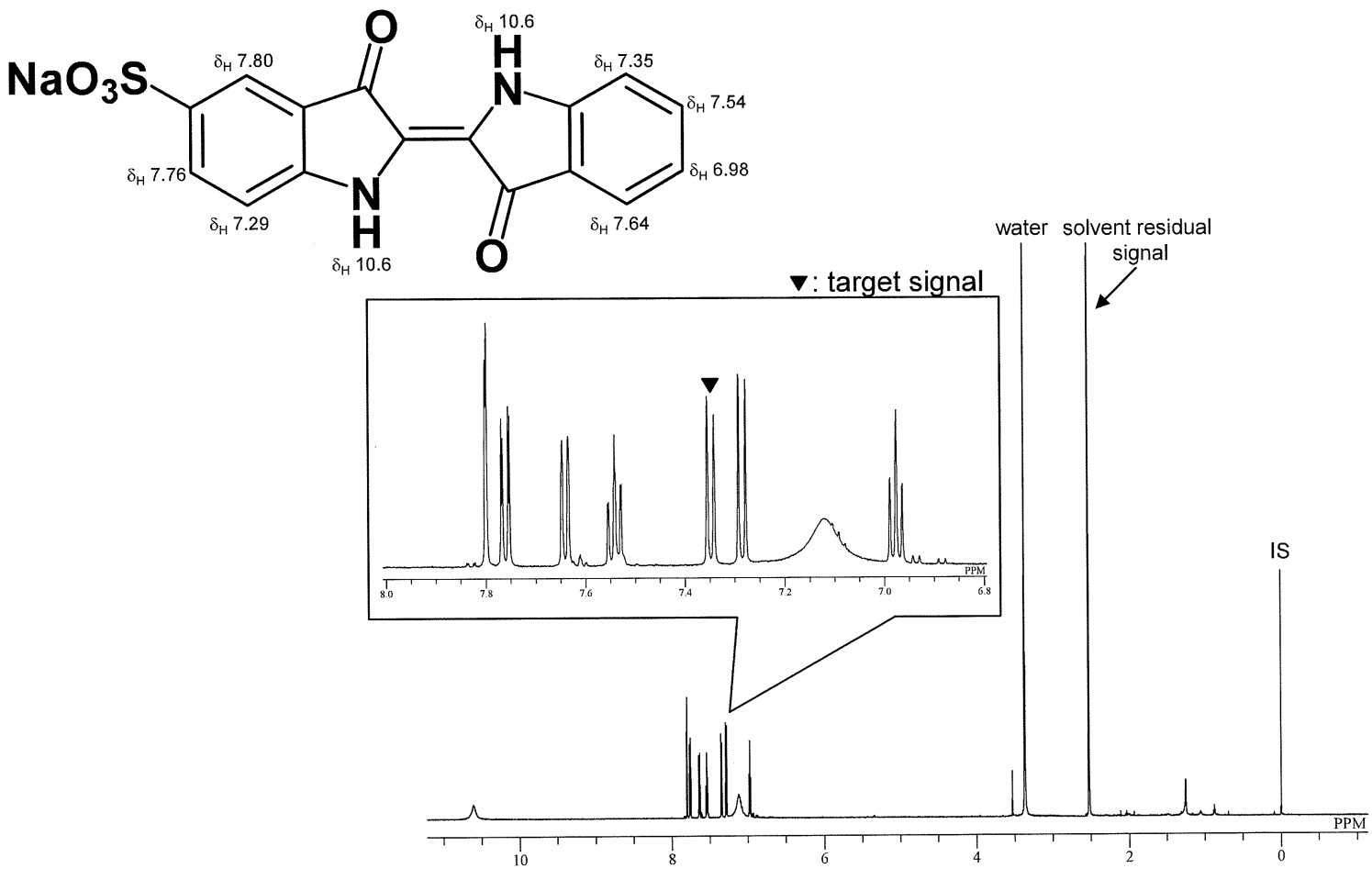

Fig.10 ^{1}H NMR spectrum of sodium salt of 2-(1,3-dihydro-3-oxo-2H-indol-2-ylidene)-2,3-dihydro-3-oxo-1H-indole-5-sulfonic acid in DMSO-d_6 containing DSS-d_6

Table 2 Purity of sorbic acid standard and reagent (analytical grade) determined by qNMR (n=3)

Signal (δ, ppm)	Number of proton	Standard		Reagent	
		Purity (%)	RSD (%)	Purity (%)	RSD (%)
1.84	3	99.2	0.3	99.0	0.3
5.79	1	99.3	0.3	99.0	0.3
6.26	2	99.7	0.3	99.4	0.2
7.18	1	99.0	0.3	98.8	0.2
12.2	1	92.1	0.6	91.8	0.7

Table 3 Purity of benzoic acid reagents (analytical grade) determined by qNMR (n=3)

Signal (δ, ppm)	Number of proton	Sample A		Sample B	
		Purity (%)	RSD (%)	Purity (%)	RSD (%)
7.53	2	99.6	0.1	99.7	0.0
7.65	1	99.4	0.3	99.4	0.1
7.98	2	99.7	0.2	99.8	0.1
13.0	1	92.5	1.4	92.3	1.0

Table 4 Purity of dehydroacetic acid standard and reagent (analytical grade) determined by qNMR (n=3)

Signal (δ, ppm)	Number of protons	Standard		Reagent	
		Purity (%)	RSD (%)	Purity (%)	RSD (%)
2.22	3	99.7	0.1	99.0	0.1
2.56	3	99.7	0.1	98.9	0.2
6.03	1	99.6	0.1	98.8	0.1

Table 5 Purity of acesulfame determined by qNMR (n=3)

Signal (δ, ppm)	Number of protons	Purity (%)	RSD (%)
2.20	3	97.8	0.1
6.04	1	97.7	0.1

Table 6 Purity of Food color B2 determined by qNMR (n=3)

Signal (δ, ppm)	Number of protons	Purity (%)	RSD (%)
7.28	2	91.1	0.3
7.76	2	91.0	0.3
7.80	2	91.5	0.1
10.7	2	89.3	0.8

Table 7 Purity of sodium salt of 2-(1,3-dihydro-3-oxo-7-sulfo-2H-indol-2-ylidene)-2,3-dihydro-3-oxo-1H-indole-5-sulfonic acid determined by qNMR (n=3)

Signal (δ, ppm)	Number of protons	Purity (%)	RSD (%)
7.67	1	40.1	0.5
7.71	1	40.1	0.5

Table 8 Purity of sodium salt of 2-(1,3-dihydro-3-oxo-2H-indol-2-ylidene)-2,3-dihydro-3-oxo-1H-indole-5-sulfonic acid determined by qNMR (n=3)

Signal (δ, ppm)	Number of protons	Purity (%)	RSD (%)
6.98	1	65.4	1.4
7.35	1	63.9	0.3
7.54	1	67.3	0.1

Table 9 Comparison of purities of sorbic acid (standard) determined by qNMR and neutralization titration methods

	Purity (%)
qNMR	99.2 ± 0.3
Neutralization titration	99.4 ± 0.1

qNMR: Values represent the mean ± standard deviation of purities obtained from three signals (δ_H 1.86, 5.79, and 7.18).

Titration: Values represent the mean ± standard deviation of three independent experiments.

Table 10 Comparison of purities of benzoic acid (reagent) determined by qNMR and neutralization titration methods

	Purity (%)
qNMR	99.6 ± 0.2
Neutralization titration	99.7 ± 0.1

qNMR: Values represent the mean ± standard deviation of purities obtained from three signals (δ_H 7.53, 7.65, and 7.98).

Titration: Values represent the mean ± standard deviation of three independent experiments.

Table 11 Comparison of purities of dehydroacetic acid (reagent) determined by qNMR and neutralization titration methods

	Purity (%)
qNMR	98.9 ± 0.1
Neutralization titration	98.7 ± 0.2

qNMR: Values represent the mean ± standard deviation of purities obtained from three signals (δ_H 2.22, 2.56, and 6.03).

Titration: Values represent the mean ± standard deviation of three independent experiments.

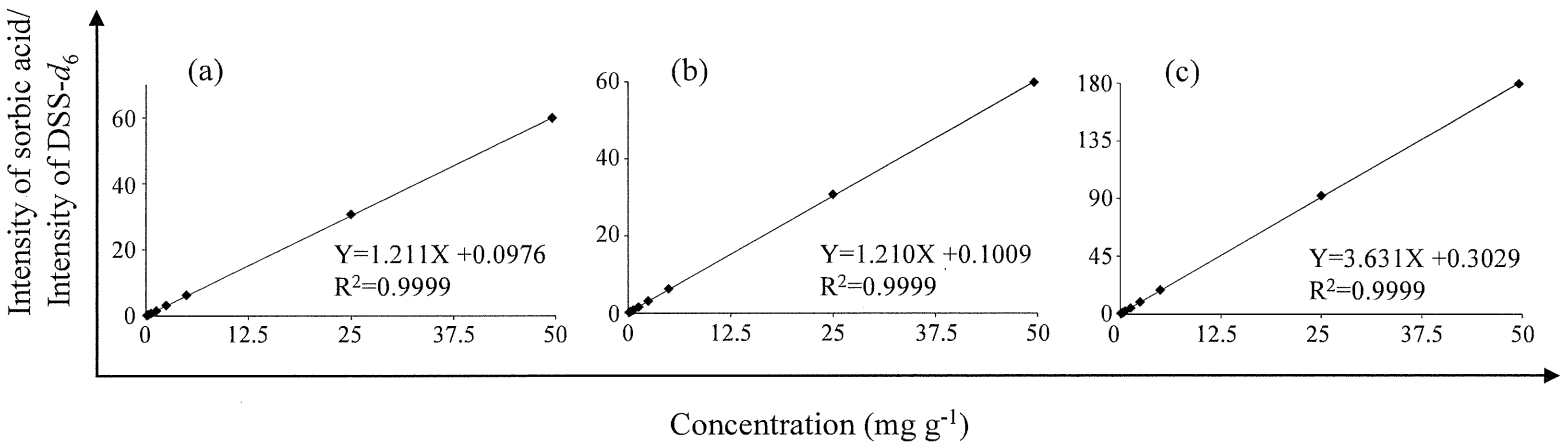

Fig. 11 Relationship between sorbic acid (SA) concentration and ratio of the integral of SA: DSS-d_6 signals.

(a) δ_H 7.18. (b) δ_H 5.79. (c) δ_H 1.84.

Table 12 Recoveries of sorbic acid from processed foods.

Sample	0.063 g kg^{-1} spiked (0.0063 g kg^{-1} spiked)*		0.13 g kg^{-1} spiked (0.013 g kg^{-1} spiked)*		Maximum usage level spiked		
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Level (g kg^{-1})	Recovery (%)	RSD (%)
Cheese	56.9	2.4	98.8	1.6	3.0	97.1	3.1
Fish paste	61.7	8.9	84.4	4.0	2.0	100.2	0.5
Sausage	61.2	4.5	81.1	4.3	2.0	89.1	2.1
Dried cuttlefish	60.1	5.8	99.7	0.6	1.5	94.5	0.8
Syrup	83.5	5.9	96.2	1.8	1.0	99.5	0.9
vegetable pickeled in soybean sauce	59.7	1.2	80.3	1.0	1.0	99.6	0.5
Jam	65.3	8.9	98.9	2.8	1.0	99.2	0.8
Soybean paste	75.0	0.2	90.5	6.7	1.0	92.8	2.3
Noodle soup	78.5	2.9	86.3	4.6	0.50	97.7	0.7
Ketchup	79.5	1.2	93.5	6.8	0.50	98.7	1.2
Beverage containing *Lactobacillus* species	71.2	3.4	86.4	2.0	0.050	93.3	3.0

* Beverage containing *Lactobacillus* species

Each recovery value represents the mean of three independent experiments on the same day.

RSD, intra-day relative standard deviation.

Fig. 12 ^{1}H NMR spectra (0–8 ppm) of each sample solution spiked with SA at the maximum usage level of each processed food (upper), at 0.13 g kg^{-1} (beverage containing *Lactobacillus* species, 0.013 g kg^{-1}) (middle), and blank (lower). (a) Cheese. (b) Fish paste. (c) Sausage. (d) Dried cuttlefish. (e) Syrup. (f) Vegetables pickled in soybean sauce. (g) Jam. (h) Soybean paste. (i) Noodle soup. (j) Ketchup. (k) Beverage containing *Lactobacillus* species. Signals marked with asterisks were used for quantification and the recoveries were calculated. IS, internal standard (DSS-d_6).

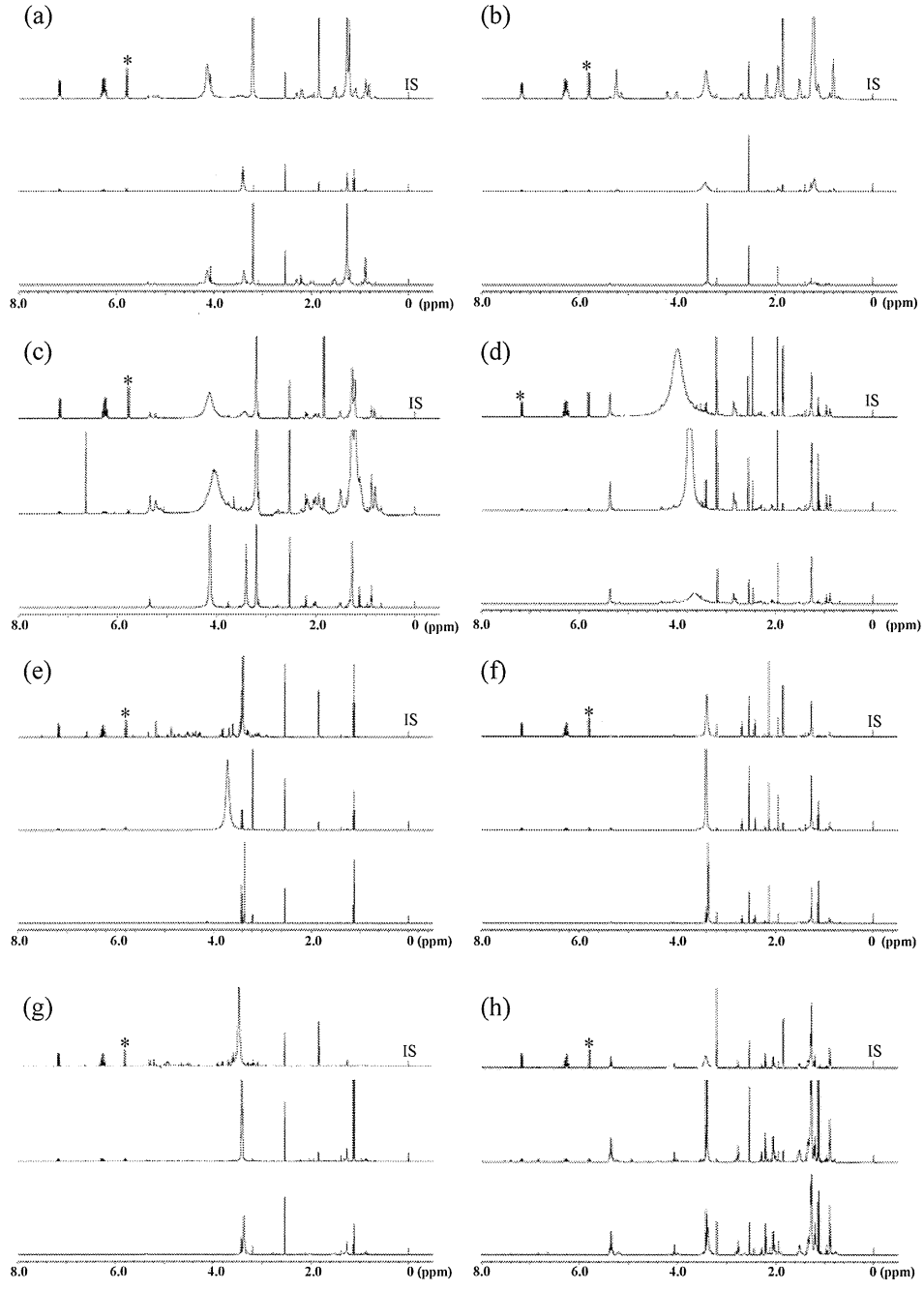

Fig. 12 continued

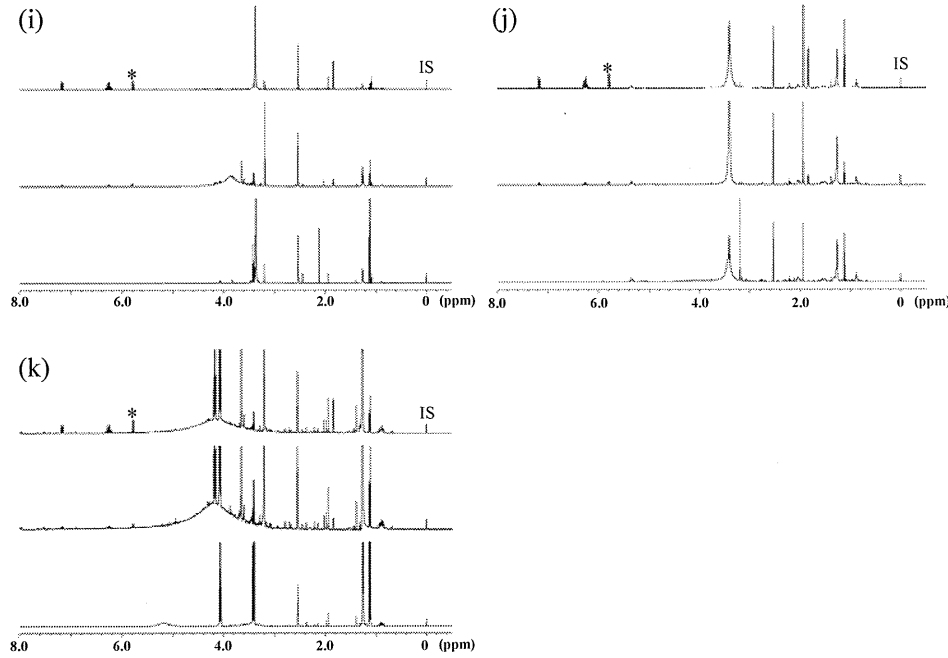

Table 13 Comparison of sorbic acid contents in commercial foods determined by two methods.

Sample	Proposed method (Solvent extraction/qHNMR)		Conventional method (Steam distillation/HPLC)	
	Content ($g\ kg^{-1}$)	RSD (%)	Content ($g\ kg^{-1}$)	RSD (%)
Cheese	0.25	5.5	0.27	4.3
Fish paste	1.46	2.9	1.42	3.1
Sausage	0.68	3.4	0.75	1.2
Dried cuttlefish	0.72	1.4	0.62	0.5
Syrup	0.66	1.6	0.66	0.9
Jam	0.62	2.8	0.59	2.8

Each value represents the mean of three independent experiments.

RSD, relative standard deviation.

Fig. 13 ^{1}H NMR spectra of each sample solution from commercially produced food with sorbic acid. (a) Cheese. (b) Fish paste. (c) Sausage. (d) Dried cuttlefish. (e) Syrup. (f) Jam. Signals marked with asterisks were used for quantification and the contents were calculated. IS, internal standard (DSS-d_6).