

SUPPORTING INFORMATION

Determination of Total Phenol and Six Polyphenolic Components in Polyphenol Extract of *Cinnamomi Cortex* by Quantitative Nuclear Magnetic Resonance Spectroscopy

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Determination of total sugar content by phenol sulfuric acid method

Glucose reference substance (25 mg) was accurately weighed and dissolved in a 100 mL brown volumetric flask. Accurately took 2 mL, 4 mL, 6 mL, 8 mL and 10 mL of the reference solution in a 25 mL brown volumetric flask respectively. Took 2 mL of glucose solution of different concentration into a brown reagent bottle, added 1.22 mL of phenol solution (6%) and 5 mL of concentrated sulfuric acid. Mixed them evenly. After 20 minutes, the absorbance was measured with ultraviolet spectrophotometer at 490 nm with reagent blank reference solution. The standard curve of absorbance to glucose concentration was regressed.

Accurately weighed cinnamom polyphenol extract sample in brown volumetric flask, added water to dissolve, ultrasonic mixing, cooled down, diluted it with water to the scale and shaken up. Accurately took 5 mL of the reference solution in a 25 mL brown volumetric flask. Took 2 mL of solution, added 1.22 mL of phenol solution (6%) and 5 mL of concentrated sulfuric acid, and mixed them evenly. The absorbance was measured at 490 nm with the reagent blank reference solution.

Table S1 Determination of Total Sugar in Cinnamon Polyphenol Extract Samples by Phenol Sulfuric Acid Method (n=3)

Sample	carbohydrate content(%)
Sample1	54.49
Sample2	24.32
Sample3	45.78

Table S2 Intra-day and intra-day precision of total phenol qNMR determination (n=6)

Sample No	Frequency	1	2	3	4	5	6	Average	RSD(%)
Sample 1	A _s /A _r	2.81	2.81	2.81	2.80	2.81	2.80	2.81	0.18
	A _s /A _r	2.80	2.80	2.80	2.81	2.81	2.81	2.80	0.29
Sample 2	A _s /A _r	9.26	9.30	9.26	9.29	9.26	9.30	9.28	0.22
	A _s /A _r	9.26	9.27	9.32	9.30	9.25	9.31	9.29	0.31
Sample 3	A _s /A _r	1.30	1.34	1.32	1.31	1.30	1.30	1.31	1.22
	A _s /A _r	1.27	1.30	1.31	1.23	1.30	1.28	1.28	2.28
Self-made sample	A _s /A _r	3.33	3.45	3.45	3.45	3.33	3.33	3.39	1.86
	A _s /A _r	3.45	3.33	3.45	3.33	3.33	3.23	3.35	2.51

Table S3 Linear relationship of total phenol qNMR determination

Zero intercept linear equation	R	Non-zero intercept linear equation	R	Theoretical slope value	Actual slope value	Deviation of slope value (%)
EC y=0.3567x	0.9999	y=0.3646x-0.0068	0.9999	0.3518	0.3567	1.40

Table S4 The recovery of total phenol qNMR determination (n=4)

	1	2	3	4	Mean(%)	RSD(%)
Sample weight (mg)	70.6	70.7	70.6	70.4		
Content(mg)	22.0	22.0	22.0	21.9		
Added(mg)	0.338	0.857	1.19	1.85		
Measured quantity(mg)	22.3	22.9	23.2	24.0		
Recovery(%)	98.5	97.3	102	104	100	3.09

Table S5 Intra-day and inter-day precision of qNMR determination of polyphenol components (n=6)

	Sample	Frequency	1	2	3	4	5	6	Average	RSD(%)	
Method 1 (DMSO-d ₆ method)	GA	A _s /A _r	1.30	1.30	1.32	1.30	1.30	1.30	1.30	1.30	0.54%
		A _s /A _r	1.32	1.30	1.32	1.32	1.30	1.30	1.31	0.72%	
	EC	A _s /A _r	0.88	0.87	0.88	0.87	0.87	0.87	0.87	0.87	0.72%
		A _s /A _r	0.88	0.87	0.87	0.88	0.87	0.88	0.88	0.88	0.79%
	ECG	A _s /A _r	0.13	0.13	0.13	0.13	0.13	0.13	0.13	0.13	0.54%
		A _s /A _r	0.13	0.13	0.13	0.13	0.13	0.13	0.13	0.13	0.72%
	EGCG	A _s /A _r	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.54%
		A _s /A _r	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.72%
	EGC	A _s /A _r	0.32	0.32	0.33	0.32	0.32	0.32	0.32	0.33	0.54%
		A _s /A _r	0.33	0.32	0.32	0.33	0.32	0.32	0.32	0.32	1.48%
	GCG	A _s /A _r	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.54%
		A _s /A _r	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.72%
Method 2 (D ₂ O method)	EC	A _s /A _r	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	1.08%
		A _s /A _r	0.05	0.05	0.05	0.05	-	-	-	-	1.19%
	EGC	A _s /A _r	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.78%
		A _s /A _r	0.02	0.02	0.02	0.02	-	-	-	-	0.79%
	GCG	A _s /A _r	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	1.04%
		A _s /A _r	0.01	0.01	0.01	0.01	-	-	-	-	1.14%

Table S6 Linear relationship of qNMR determination of polyphenol components

		Zero intercept linear equation	R	Non-zero intercept linear equation	R	Theoretical slope value	Actual slope value	Deviation(%)
Method 1 (DMSO-d ₆ method)	GA	y=1.2441x	0.9999	y=1.2465x-0.003	0.9998	1.2004	1.2441	3.64
	EC	y=0.3567x	0.9999	y=0.3646x-0.0068	0.9999	0.3518	0.3567	1.40
	EGCG	y=0.4449x	0.9998	y=0.4535x-0.008	0.9996	0.4455	0.4449	0.14
	ECG	y=0.2291x	0.9997	y=0.2222x+0.0022	0.9995	0.2308	0.2291	0.75
	EGC	y=0.6507x	0.9999	y=0.6598x-0.0044	0.9999	0.6668	0.6507	2.41
	GCG	y=0.4314x	0.9999	y=0.4200x+0.0077	0.9999	0.4455	0.4314	3.17
Method 2 (D ₂ O method)	EC	y=0.6872x	0.9999	y=0.6728x+0.0212	0.9999	0.7036	0.6872	2.32
	EGC	y=0.6666x	0.9999	y=0.6622x+0.0069	0.9997	0.6668	0.6666	0.03
	GCG	y=0.4377x	0.9996	y=0.4157x+0.0104	0.9999	0.4455	0.4377	1.76

Table S7 Recovery of qNMR determination DMSO-d₆ method of polyphenol components (n=4)

Sample	Frequency	1	2	3	4	Mean(%)	RSD(%)
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GCG	Sample weight (mg)	40.8	35.4	45.2	43.3		
	Content (mg)	0.610	0.530	0.680	0.650		
	Added (mg)	0.290	0.840	1.67	3.03		
	Measured quantity (mg)	0.910	1.37	2.35	3.66		
	Recovery (%)	101	99.8	100	99.2	100	0.71
ECG	Sample weight (mg)	41.6	39.8	34.9	45.2		
	Content (mg)	1.67	1.60	1.40	1.82		
	Added (mg)	1.88	1.94	2.95	3.41		
	Measured quantity (mg)	3.62	3.66	4.53	5.44		
	Recovery (%)	99.5	102	102	102	102	0.09
GA	Sample weight (mg)	10.0	10.2	9.70	8.73		
	Content (mg)	1.29	1.31	1.25	1.12		
	Added (mg)	0.560	0.750	0.880	1.05		
	Measured quantity (mg)	1.85	2.06	2.11	2.15		
	Recovery (%)	102	99.5	98.4	98.1	100	1.85
EGCG	Sample weight (mg)	40.1	40.8	38.8	34.9		
	Content (mg)	4.10	4.18	3.97	3.57		
	Added (mg)	1.46	2.29	4.18	6.52		
	Measured quantity (mg)	5.57	6.49	8.14	10.2		
	Recovery (%)	101	101	99.7	101	101	0.72
EC	Sample weight (mg)	38.8	40.8	40.1	34.9		
	Content (mg)	0.460	0.480	0.470	0.410		
	Added (mg)	0.350	0.510	0.560	1.05		
	Measured quantity (mg)	0.810	0.990	1.04	1.44		
	Recovery (%)	98.8	100	102	97.8	99.7	1.61
EGC	Sample weight (mg)	40.8	41.6	45.2	43.3		
	Content (mg)	0.510	0.520	0.570	0.550		
	Added (mg)	0.500	1.12	1.91	4.18		
	Measured quantity (mg)	1.02	1.67	2.51	4.74		
	Recovery (%)	101	103	102	100	102	0.89

Table S8 Recovery of qNMR determination D_2O method of polyphenol components ($n=4$)

Sample	Frequency	1	2	3	4	Mean(%)	RSD(%)
EGC	Sample weight (mg)	61.2	63.9	58.1	56.0		
	Content (mg)	0.140	0.140	0.130	0.130		
	Added (mg)	1.13	3.07	4.47	6.00		

GCG	Measured quantity (mg)	1.26	3.15	4.56	6.24	99.6	1.51
	Recovery (%)	99.5	98.2	99.0	102		
	Sample weight (mg)	57.3	58.2	62.9	64.9		
	Content (mg)	0.0500	0.0500	0.0600	0.0600		
	Added (mg)	2.13	3.33	4.00	5.66		
EC	Measured quantity (mg)	2.13	3.42	3.91	5.76	99.4	2.34
	Recovery (%)	97.7	101	96.4	101		
	Sample weight (mg)	57.3	58.2	62.9	64.9		
	Content (mg)	0.150	0.150	0.170	0.170		
	Added (mg)	0.530	1.82	3.68	4.36		
	Measured quantity (mg)	0.670	1.970	3.84	4.53	98.2	1.04
	Recovery (%)	97.5	99.6	97.3	98.3		

Table S9 HPLC Method Validation including precision, linearity, sensitivity and recovery

Compound	RSD%(n=6)	Linearity	Sensitivity		recovery rates(Mean±RSD)(n=3)
	intra-day	correlation coefficient	LOD (µg/mL)	LOQ(µg/mL)	Sample 1 (%)
GA	0.34	0.9999	0.07	0.24	95.67±1.32
EC	2.71	0.9999	0.40	1.30	95.32±2.31
ECG	0.26	0.9999	0.17	0.58	98.02±2.52
EGCG	0.22	0.9999	0.26	0.88	100.73±5.30
EGC	0.29	0.9997	0.26	0.88	99.95±0.11
GCG	1.48	0.9999	0.66	2.20	95.91±1.30