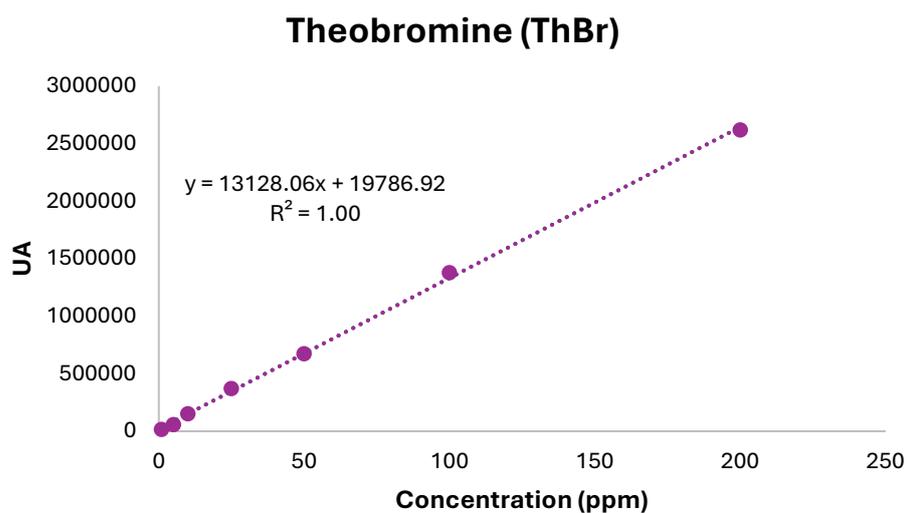
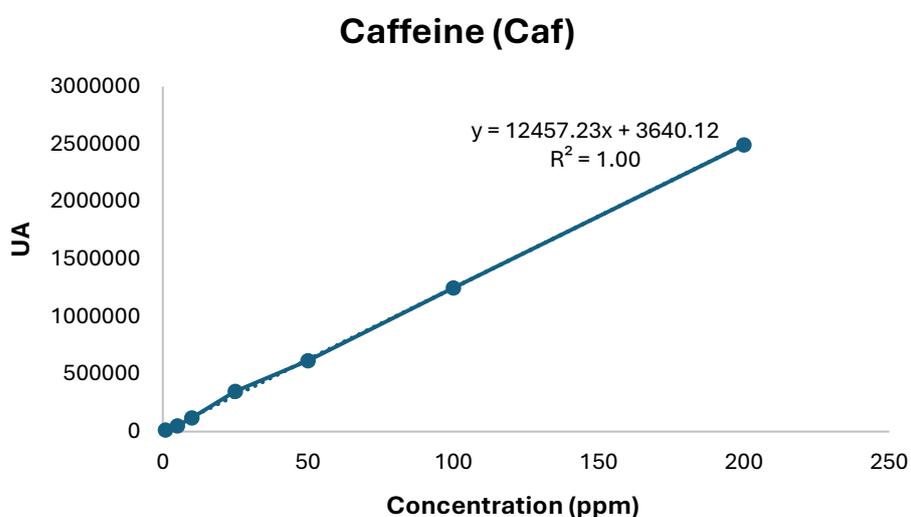


## Simulated Gastrointestinal Digestion of Cocoa Powder (INFOGEST): Methylxanthine Bioaccessibility, and Antioxidant Properties

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- **Calibration Curves of Standards**



- **Solvent Optimization Data (%Ethanol)**

<b>Code (%Eth)</b>	<b>Sample weight (g)</b>	<b>ThBr Area (UA)</b>	<b>Caf Area (UA)</b>	<b>ThBr Concentration (mg/g sample)</b>	<b>Caf Concentration (mg/g sample)</b>	<b>Methylxantines (mg/g sample)</b>
R0-0%	0.4988	1312315	85265	9.87	0.66	10.53
R1-0%	0.5064	1165557	74500	8.62	0.56	9.18
R0-20%	0.5127	1546690	121462	11.34	0.92	12.27
R1-20%	0.5109	1301786	101665	9.56	0.77	10.33
R0-40%	0.5055	1486333	112770	11.05	0.87	11.92
R1-40%	0.5036	1325516	103702	9.87	0.80	10.67
R0-60%	0.5059	1561923	120927	11.61	0.93	12.54
R1-60%	0.5076	1640019	125764	12.16	0.97	13.12
R0-80%	0.4992	1676157	137245	12.64	1.07	13.71
R1-80%	0.5123	1677913	137547	12.33	1.05	13.38
R0-100%	0.5053	1690810	133153	12.60	1.03	13.62
R1-100%	0.5062	1741534	134751	12.95	1.04	13.99

- **Temperature Optimization Data**

<b>Code (T°)</b>	<b>Sample weight (g)</b>	<b>ThBr Area (UA)</b>	<b>Caf Area (UA)</b>	<b>ThBr Concentration (mg/g sample)</b>	<b>Caf Concentration (mg/g sample)</b>	<b>Methylxantines (mg/g sample)</b>
R1-30	0.4993	3231428	206786	12.2491	0.8165	13.07
R2-30	0.5185	3372363	213770	12.3132	0.8133	13.13
R1-40	0.5064	3234882	176882	12.0904	0.6866	12.78
R2-40	0.5078	3354684	227162	12.5063	0.8834	13.39
R1-50	0.5142	3499450	222725	12.8868	0.8551	13.74
R2-50	0.5075	3481978	280910	12.9914	1.0964	14.09
R1-60	0.5135	3463867	219365	12.7724	0.8431	13.62
R2-60	0.5173	3609264	138397	13.2138	0.5228	13.74
R1-70	0.5045	3439513	221223	12.9083	0.8655	13.77
R2-70	0.5106	3452850	203218	12.8039	0.7844	13.59

- BBD-RSM Experiments Data**

Exp.	%EtOH	Temp. (°C)	Cycle (s <sup>-1</sup> )	Amplitude (%)	Sample Weight (g)	ThBr Area (UA)	Caf Area (UA)	ThBr Concentration (mg/g sample)	Caf Concentration (mg/g sample)	Methylxantines (mg/g sample)
1	80	50	0.6	35	0.4975	3255544	212537	12.39	0.84	13.23
2	60	50	0.6	50	0.5060	3359275	246350	12.57	0.96	13.53
3	80	50	0.6	35	0.5012	3361136	225783	12.70	0.89	13.59
4	80	70	0.6	50	0.5104	3387263	215416	12.56	0.83	13.40
5	80	70	0.6	20	0.5036	3292967	228980	12.38	0.90	13.28
6	100	30	0.6	35	0.5101	1878136	22717	6.94	0.08	7.01
7	80	30	0.6	50	0.5062	3320048	242241	12.42	0.95	13.36
8	80	50	0.9	20	0.5027	3272744	227671	12.32	0.89	13.22
9	60	50	0.9	35	0.5028	3398605	253631	12.80	1.00	13.79
10	60	70	0.6	35	0.5052	3388645	236336	12.70	0.92	13.62
11	80	30	0.9	35	0.5002	4879205	503878	18.50	2.01	13.19
12	60	50	0.6	20	0.5010	3360292	207012	12.70	0.81	13.51
13	80	30	0.3	35	0.5055	3298953	203590	12.35	0.79	13.15
14	60	30	0.6	35	0.4992	3397478	221906	12.89	0.88	13.76
15	80	70	0.3	35	0.4998	3307077	231272	12.53	0.91	13.44
16	100	50	0.9	35	0.5039	2168157	72685	8.12	0.27	8.39
17	80	70	0.9	35	0.5006	3263370	219287	12.34	0.86	13.20
18	100	70	0.6	35	0.5000	2050712	59971	7.74	0.23	7.96
19	60	50	0.3	35	0.5045	3448352	273105	12.94	1.07	14.01
20	80	50	0.3	20	0.5089	3374280	242419	12.55	0.94	13.49
21	100	50	0.3	35	0.5019	1979325	45885	7.43	0.17	7.60
22	100	50	0.6	50	0.5007	1990106	42654	7.49	0.16	7.65
23	100	50	0.6	20	0.5056	2035467	46437	7.59	0.17	7.76
24	80	30	0.6	20	0.5027	3280375	231070	12.35	0.91	13.26
25	80	50	0.9	50	0.5051	3288009	213339	12.32	0.83	13.15
26	80	50	0.6	35	0.5049	3351805	253765	12.57	0.99	13.56
27	80	50	0.3	50	0.4963	3260696	245741	12.44	0.98	13.41

- Time Optimization Data (minutes)

Code (t(min))	Sample Weight (g)	ThBr Area (UA)	Caf Area (UA)	ThBr Concentration (mg/g sample)	Caf Concentration (mg/g sample)	Methylxantines (mg/g sample)
R1-5	0.5087	3240914	222236	12.06	0.86	12.92
R2-5	0.5063	3238250	260104	12.11	1.02	13.12
R1-10	0.5021	3194443	269987	12.04	1.06	13.11
R2-10	0.5028	3244730	254901	12.21	1.00	13.22
R1-15	0.5045	3291719	254082	12.35	0.99	13.35
R2-15	0.4991	3310924	247454	12.56	0.98	13.54
R1-20	0.4989	3271762	243493	12.41	0.96	13.38
R2-20	0.5041	3311339	253547	12.43	0.99	13.43
R1-30	0.5023	3350626	253975	12.63	1.00	13.63
R2-30	0.5062	3363466	230035	12.58	0.90	13.48

- **Repeatability and Intermediate Precision Data**

Code	Sample Weight (g)	ThBr Area (UA)	Caf Area (UA)	ThBr Concentration (mg/g sample)	Caf Concentration (mg/g sample)	Methylxantines (mg/g sample)
Day 1- 1	0.502	3375080	277289	12.73	1.09	13.82
Day 1- 2	0.5064	3391383	254977	12.68	1.00	13.67
Day 1- 3	0.5078	3367079	237370	12.55	0.92	13.48
Day 1- 4	0.5043	3384531	279108	12.71	1.10	13.80
Day 1- 5	0.4975	3381619	252776	12.87	1.00	13.87
Day 1- 6	0.4986	3362145	256425	12.77	1.02	13.78
Day 1- 7	0.5021	3424592	265015	12.91	1.04	13.96
Day 1- 8	0.5071	3453555	239728	12.89	0.93	13.83
Day 1- 9	0.5031	3400646	269557	12.80	1.06	13.86
Day 2- 1	0.5056	3447514	262313	12.91	1.03	13.94
Day 2- 2	0.5017	5818546	582876	13.28	1.13	14.41
Day 2- 3	0.5077	3451445	265693	12.87	1.04	13.91
Day 2- 4	0.5072	3474447	281915	12.97	1.10	14.07
Day 2- 5	0.5032	3441309	257543	12.95	1.01	13.96
Day 2- 6	0.5085	3453852	284874	12.86	1.11	13.97
Day 2- 7	0.5023	3384128	257499	12.75	1.01	13.77
Day 2- 8	0.5014	3459740	282949	13.06	1.12	14.18
Day 2- 9	0.5074	3478157	280256	12.98	1.09	14.07
Day 3- 1	0.5071	3478496	259545	12.99	1.01	14.00
Day 3- 2	0.4984	3502080	241506	13.31	0.96	14.26
Day 3- 3	0.5041	3401086	266829	12.77	1.05	13.82
Day 3- 4	0.4969	3406378	272559	12.98	1.09	14.06
Day 3- 5	0.5076	3503599	268894	13.07	1.05	14.12
Day 3- 6	0.5068	3523267	276434	13.16	1.05	14.21
Day 3- 7	0.4987	3618395	288367	13.20	1.07	14.27
Day 3- 8	0.4993	3824643	270238	13.38	1.06	14.43
Day 3- 9	0.5039	3996752	277817	13.35	1.08	14.43

- **Real Samples Analysis Data**

Code	Sample weight (g)	ThBr Area (UA)	Caf Area (UA)	ThBr Concentration (mg/g sample)	Caf Concentration (mg/g sample)	Methylxantines (mg/g sample)
C-1 R1	0.5076	5243888	443574	19.60	1.74	21.34
C-1 R2	0.5059	5299261	446976	19.87	1.76	21.63
C-1 R3	0.505	5339978	438182	20.06	1.73	21.79
C-2 R1	0.5086	4418090	341231	16.47	1.33	17.80
C-2 R2	0.4976	4479216	374930	17.07	1.50	18.56
C-2 R3	0.5072	4503006	367748	16.83	1.44	18.27
C-3 R1	0.4976	3942905	334586	15.01	1.33	16.35
C-3 R2	0.4978	3965452	360521	15.09	1.44	16.53
C-3 R3	0.5029	3995229	361524	15.05	1.43	16.48
C-4 R1	0.5092	977497	74943	3.58	0.28	3.86
C-4 R2	0.5012	1727257	149270	6.49	0.58	7.07
C-4 R3	0.5032	955140	68502	3.54	0.26	3.80
C-5 R1	0.5062	1046964	74656	3.86	0.28	4.15
C-5 R2	0.4984	1064479	75329	3.99	0.29	4.28
C-5 R3	0.507	1056413	70773	3.89	0.27	4.16
C-6 R1	0.4976	1316107	88682	4.96	0.34	5.30
C-6 R2	0.5066	1290999	89544	4.78	0.34	5.12
C-6 R3	0.4985	1325061	82758	4.99	0.32	5.30

- **Extracts Post-Digestion Data**

	<b>Code</b>	<b>Sample weight (g)</b>	<b>ThBr Area (UA)</b>	<b>Caf Area (UA)</b>	<b>ThBr Concentration (mg/g sample)</b>	<b>Caf Concentration (mg/g sample)</b>	<b>Methylxantines (mg/g sample)</b>
Oral phase	C-1.1.	1.0166	6995593	782913	2.61	0.31	2.92
	C-1.2.	1.0790	7496872	927150	2.64	0.34	2.98
	C-2.1.	1.0800	7214420	852246	2.54	0.32	2.85
	C-2.2.	1.0275	7366458	880966	2.72	0.34	3.07
	C-4.1.	1.0563	4999056	336442	1.80	0.13	1.92
	C-4.2.	1.0018	5988344	493863	2.27	0.20	2.47
Gastric phase	C-1.1.	1.0166	7917717	1098717	5.33	0.78	6.10
	C-1.2.	1.0790	7064670	918177	4.48	0.61	5.09
	C-2.1.	1.0800	6760617	828742	4.28	0.55	4.83
	C-2.2.	1.0275	7851514	986670	5.23	0.69	5.92
	C-4.1.	1.0563	4657219	377934	3.01	0.26	3.27
	C-4.2.	1.0018	4522650	333522	3.08	0.24	3.32
Intestinal phase	C-1.1.	1.0166	8265946	781734	9.89	0.98	10.87
	C-1.2.	1.0790	7325251	693805	8.25	0.82	9.07
	C-2.1.	1.0800	8599404	893776	9.68	1.06	10.74
	C-2.2.	1.0275	7603200	728154	9.00	0.91	9.90
	C-4.1.	1.0563	2350161	169668	2.69	0.20	2.89
	C-4.2.	1.0018	2405486	120541	2.90	0.15	3.05

- **Digested solid analysis data**

<b>Code</b>	<b>Sample Weight (g)</b>	<b>ThBr Area (UA)</b>	<b>Caf Area (UA)</b>	<b>ThBr Concentration (mg/g sample)</b>	<b>Caf Concentration (mg/g sample)</b>	<b>Methylxantines (mg/g sample)</b>
C-1 R1.	0.5419	2107627	224288	7.34	0.82	8.15
C-1 R2	0.6001	2604033	268624	8.20	0.89	9.09
C-2 R1.	0.6217	3401902	168579	10.36	0.53	10.89
C-2 R2	0.6503	3054100	248516	8.89	0.76	9.64
C-4 R1	0.1738	127041	17239	1.18	0.16	1.33
C-4 R2	0.2333	169917	18136	1.23	0.12	1.35