Supplemental data

Including:

Supplementary Table 1 The demographic details of the participants selected for the study.

Supplementary Table 2. Food frequency exposure ratings used to classify volunteers into different levels (low, medium, high) of habitual exposure to dietary citrus foods.

Supplementary Table 3. Discrimination of habitual dietary citrus exposure by negative ionisation mode Flow Infusion Electrospray-Ionisation Mass Spectrometry (FIE-MS) fingerprint analysis of overnight (PRE) and fasting urine in free-living volunteers.

Supplementary Table 4. Top signals explanatory of habitual dietary citrus exposure level following analysis of 'PRE' and 'Fasting' urines from two studies by positive ion mode Flow Infusion Electrospray-ionisation Mass Spectrometry.

Supplementary Table 5. Top signals explanatory of habitual dietary citrus exposure level following analysis of 'PRE' and 'Fasting' urines from two studies by negative ion mode Flow Infusion Electrospray-ionisation Mass Spectrometry.

Supplementary Table 6 Welch's-*t*-test results for the positive ion mode signals *m/z* 144, 166 and 182 in fasting and PRE urine for High vs Low, High vs Medium and Low vs Medium habitual citrus fruit consumer classes.

Supplementary Table 7 AUC (area under the ROC (receiver operating characteristic) curve), the sensitivity (true-positive rate) and the specificity (false-positive rate) results for the positive ion mode *m/z* 144, 166 and 182 in fasting and PRE urine for High vs Low habitual citrus fruit consumer classes.

Supplementary Table 1 The demographic details of the participants selected for the study.

Study	Identification	Sex	Age (y)	BMI (kg/m^2)*	Citrus category
1	204	Male	30	22.7	High
1	214	Female	59	25.2	High
1	216	Male	21	25.1	High
1	219	Female	59	20.5	High
1	222	Male	74	24.7	High
1	213	Female	62	27.8	Low
1	218	Male	59	26.5	Low
1	220	Male	48	26	Low
1	223	Male	27	21.6	Low
1	215	Male	20	25.3	Medium
1	217	Male	23	20.6	Medium
1	221	Male	48	27.3	Medium
2	302	Male	69	27.7	High
2	303	Female	70	24.2	High
2	304	Male	29	22.7	High
2	331	Male	64	26.9	High
2	332	Male	35	25	High
2	333	Male	54	24.8	High
2	312	Male	26	21.2	Low
2	318	Male	59	28.1	Low
2	320	Male	48	26.5	Low
2	330	Female	66	27.5	Low
2	334	Male	64	24.3	Low

* measured body mass index, BMI

Supplementary Table 2. Food frequency exposure ratings used to classify volunteers into different levels (low, medium, high) of habitual exposure to dietary citrus foods.

*Frequency	Rating (FFQ Score)
Never	0.000
1-3 per month	0.066
Once a week	0.140
2-4 per week	0.430
5-6 per week	0.780
Once a day	1.000
2-3 per day	2.500
4-5 per day	4.600
6+ per day	6.000

The citrus exposure food classes included grapefruit (one fruit equals one serving, oranges (including oranges, satsumas, mandarins, tangerines, clementines) and fruit juice (a glass of pure fruit juice (100%) equals one serving).

Supplementary Table 3. Discrimination of habitual dietary citrus exposure by negative ionisation mode Flow Infusion Electrospray-Ionisation Mass Spectrometry (FIE-MS) fingerprint analysis of overnight (PRE) and fasting urine in free-living volunteers.

Mass range	Discrimination of habitual dietary citrus exposure level					
	PRE Urine			Fasting Urine		
	Tw value	AUC	Margin	Tw value	AUC	Margin
m/z 15-110	0.73	0.49	-0.01	0.72	0.61	0.06
m/z 100-220	2.01	0.84	0.15	1.26	0.75	0.11
m/z 210-510	1.49	0.79	0.10	1.72	0.80	0.11
m/z 500-1200	1.21	0.52	0.00	1.36	0.71	0.03

Principal Component-Linear Discriminant Analysis (PC-LDA) and Random Forest (RF) classification of data acquired by FIE-MS (negative ion mode) analysis of pre-test day evening/night urine samples 'PRE' and fasting urine, after a 12h (minimum) fast, from 12 individuals. For PC-LDA, 'dietary citrus exposure' from Table 1 was the class structure applied consisting of 'High', 'Medium' and 'Low' citrus consumers. Pair-wise RF comparisons were made between 'High' and 'Low' citrus consumers; Tw, Discriminant Function 1 Eigenvalue; Margin, RF classification margin; ACC, classification accuracy.

Supplementary Table 4. Top signals explanatory of habitual dietary citrus exposure level following analysis of 'PRE' and 'Fasting' urines from two studies by positive ion mode Flow Infusion Electrospray-ionisation Mass Spectrometry.

Rank	Study 1 PRE	Study 1 Fasting	Study 2 PRE	Study2 Fasting
1	182	182	201	182
2	166	144	182	169
3	144	145	181	201
4	201	166	104	202
5	175	135	169	183
6	145	183	143	104
7	169	152	109	166
8	125	148	139	152
9	183	165	115	213
10	104	167	166	177
11	167	153	194	181
12	188	104	206	167
13	206	197	203	189
14	148	201	178	143
15	153	208	184	207
16	165	161	110	203
17	160	155	174	168
18	186	175	152	198
19	113	151	198	197
20	134	181	168	184
21	152	134	183	145
22	191	219	208	180
23	108	198	196	121
24	135	202	105	185
25	143	193	177	210
26	161	188	121	113
27	155	101	151	174
28	163	206	207	127
29	198	105	127	208
30	218	129	156	194
31	190	141	136	110
32	219	149	112	136
33	215	160	147	154
34	106	184	189	141
35	162	150	114	146

Top explanatory signals ranked using a combination of three methods; Random Forest Importance scores (RF), area under the ROC (receiver operating characteristic) curve (AUC), Welch and false discovery rate (FDR) adjusted P-values of 'PRE' urine, pre-test day over-night void and fasting' urine (after a 12h (minimum) fast) fingerprints (*m/z* 210-510) between Pair-wise comparisons of 'High' and 'Low' citrus consumers; (A), Study 1, 9 individuals, 18 urine samples; (B), Study 2, 11 individuals, 66 urine samples.

Supplementary Table 5. Top signals explanatory of habitual dietary citrus exposure level following analysis of 'PRE' and 'Fasting' urines from two studies by negative ion mode Flow Infusion Electrospray-ionisation Mass Spectrometry.

Rank	Study 1 PRE	Study 1 Fasting	Study 2 PRE	Study 2 Fasting
1	211	321	358	350
2	314	345	280	320
3	282	252	457	258
4	337	235	495	507
5	457	502	350	243
6	363	397	226	345
7	323	259	394	376
8	401	365	376	226
9	501	455	338	319
10	331	507	303	318
11	479	362	406	351
12	299	501	243	462
13	397	278	345	225
14	414	442	346	327
15	301	256	390	289
16	376	331	401	338
17	371	499	410	415
18	476	294	435	239
19	452	299	225	397
20	241	290	397	298
21	345	427	419	396
22	306	480	299	250
23	412	389	314	403
24	281	297	367	217
25	321	426	357	435
26	508	236	306	234
27	324	395	501	369
28	470	312	509	416
29	373	355	302	509
30	347	304	451	373
31	217	366	360	233
32	284	309	337	410
33	294	336	336	457
34	435	471	250	477
35	223	419	438	456

Top explanatory signals ranked using a combination of three methods; Random Forest Importance scores (RF), area under the ROC (receiver operating characteristic) curve (AUC), Welch and false discovery rate (FDR) adjusted P-values of 'PRE' urine, pre-test day over-night void and fasting' urine (after a 12h (minimum) fast) fingerprints (*m/z* 210-510) between Pair-wise comparisons of 'High' and 'Low' citrus consumers; (A), Study 1, 9 individuals, 18 urine samples; (B), Study 2, 11 individuals, 66 urine samples.

Supplementary Table 6 Welch's-*t*-test results for the positive ion mode signals *m/z* 144, 166 and 182 in fasting and PRE urine for High vs Low, High vs Medium and Low vs Medium habitual citrus fruit consumer classes.

	Fasting urine					
	High vs Low		High vs Medium		Low vs Medium	
m/z	*P*-value	FDR adjusted *P*-value	*P*-value	FDR adjusted *P*-value	*P*-value	FDR adjusted *P*-value
144	0.000	0.018	0.455	0.916	0.134	0.500
166	0.001	0.029	0.234	0.755	0.198	0.618
182	0.000	0.001	0.279	0.788	0.215	0.618

	PRE urine					
	High vs Low		High vs Medium		Low vs Medium	
m/z	*P*-value	FDR adjusted *P*-value	*P*-value	FDR adjusted *P*-value	*P*-value	FDR adjusted *P*-value
144	0.001	0.026	0.075	0.433	0.142	0.391
166	0.000	0.018	0.083	0.445	0.080	0.360
182	0.000	0.018	0.333	0.686	0.105	0.360

Study 1 data is displayed here where n=12; *P*-values calculated by Welch's *t*-test; FDR, false discovery rate; 'PRE', pre-test day over-night urine voids; 'Fasting', spot urine sample after a 12h (minimum) fast.

Supplementary Table 7 AUC (area under the ROC (receiver operating characteristic) curve), the sensitivity (true-positive rate) and the specificity (false-positive rate) results for the positive ion mode *m/z* 144, 166 and 182 in fasting and PRE urine for High vs Low habitual citrus fruit consumer classes.

Urine	*m/z*	AUC	Sensitivity	Specificity
PRE	144	97.7	92.2	74.2
	166	97.5	84.9	86.7
	182	98.8	87.4	94.1
Fasting	144	97.6	89.2	79.6
	166	93.9	80.8	79.7
	182	99.1	88.7	89.0

Study 1 data is displayed here where n=9; randomised re-sampling using 100 bootstraps was used; 'PRE', pre-test day over-night urine voids; 'Fasting', spot urine sample after a 12h (minimum) fast.