UNTARGETED HILIC-MS-BASED METABOLOMICS APPROACH TO EVALUATE COFFEE ROASTING PROCESS: CONTRIBUTING TO AN INTEGRATED METABOLOMICS MULTIPLATFORM

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Figure captions.

Figure S1. Base peak chromatograms (BPC) obtained in positive ionization mode for green coffee (GCB) (A); light coffee (LRC) (B); medium coffee (MRC) (C); and dark coffee (DRC) (D) under optimal separation conditions. HILIC-MS conditions are summarized in section 3.3.

Figure S2. Hierarchical cluster analysis (HCA) in positive (A) and negative (B) ionization modes for the four groups of coffee samples (GCB, LRC, MRC, and DRC) submitted to different roasting process.

Figure S3. Diagrams of the trends observed for all the tentatively and unequivocally compounds both in negative and positive ionization mode along the coffee roasting process.

Figure S1.

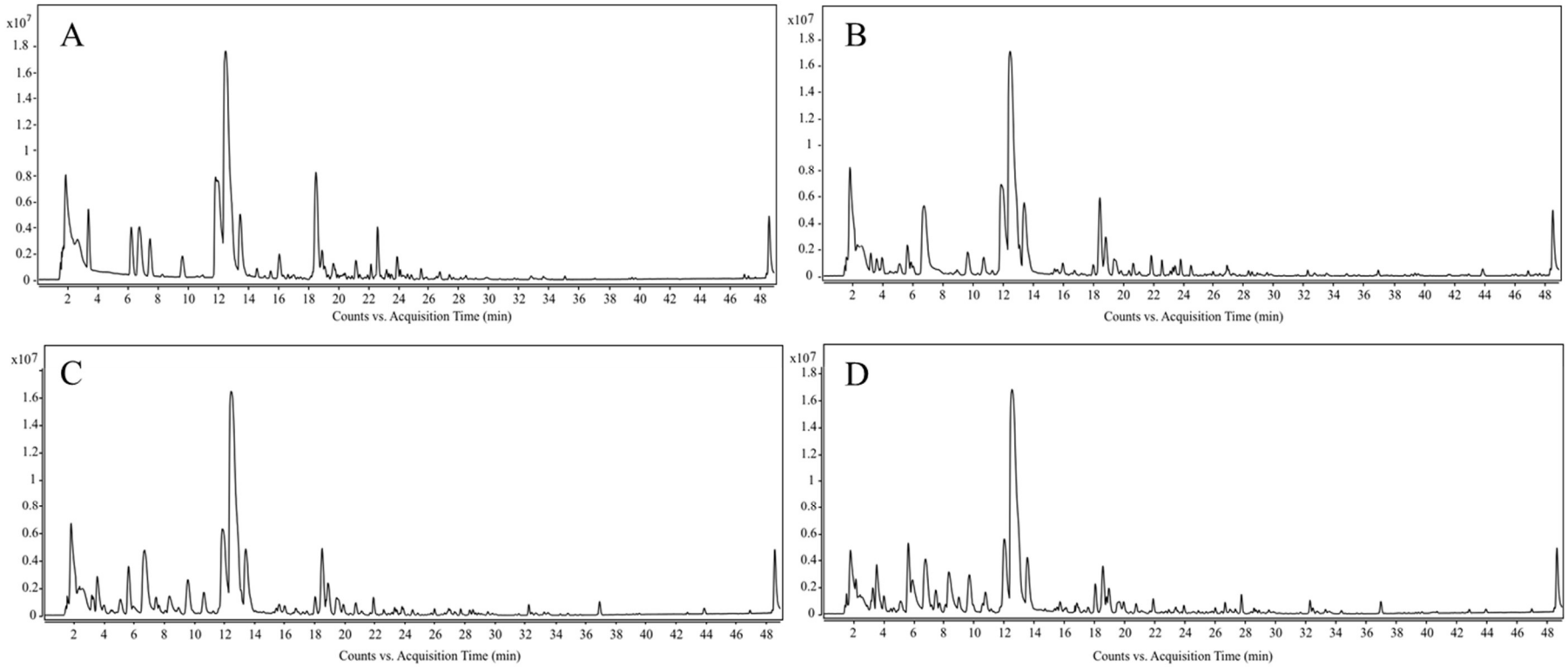


Figure S2-A

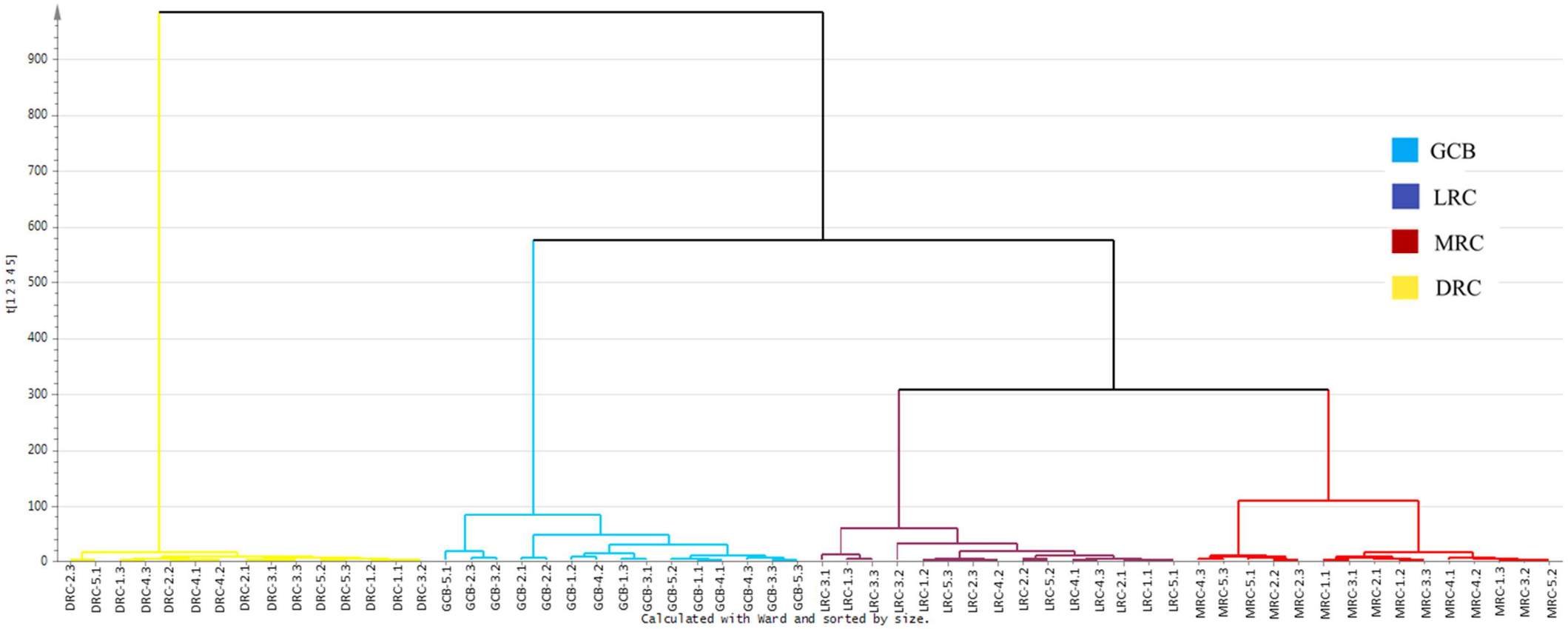

t[1 2 3 4 5]
900
800
700
600
500
400
300
200
100
0
GCB
LRC
MRC
DRC
DRC-2.3
DRC-5.1
DRC-1.3
DRC-4.3
DRC-2.2
DRC-4.1
DRC-4.2
DRC-2.1
DRC-3.1
DRC-3.3
DRC-5.2
DRC-5.3
DRC-1.2
DRC-1.1
DRC-3.2
GCB-5.1
GCB-2.3
GCB-3.2
GCB-2.1
GCB-2.2
GCB-1.2
GCB-4.2
GCB-1.3
GCB-3.1
GCB-5.2
GCB-1.1
GCB-4.1
GCB-4.3
GCB-3.3
GCB-5.3
LRC-3.1
LRC-1.3
LRC-3.3
LRC-3.2
LRC-1.2
LRC-5.3
LRC-2.3
LRC-4.2
LRC-2.2
LRC-5.2
LRC-4.1
LRC-4.3
LRC-2.1
LRC-1.1
LRC-5.1
MRC-4.3
MRC-5.3
MRC-5.1
MRC-2.2
MRC-2.3
MRC-1.1
MRC-3.1
MRC-2.1
MRC-1.2
MRC-3.3
MRC-4.1
MRC-4.2
MRC-1.3
MRC-3.2
MRC-5.2
Calculated with Ward and sorted by size.

Figure S2-B

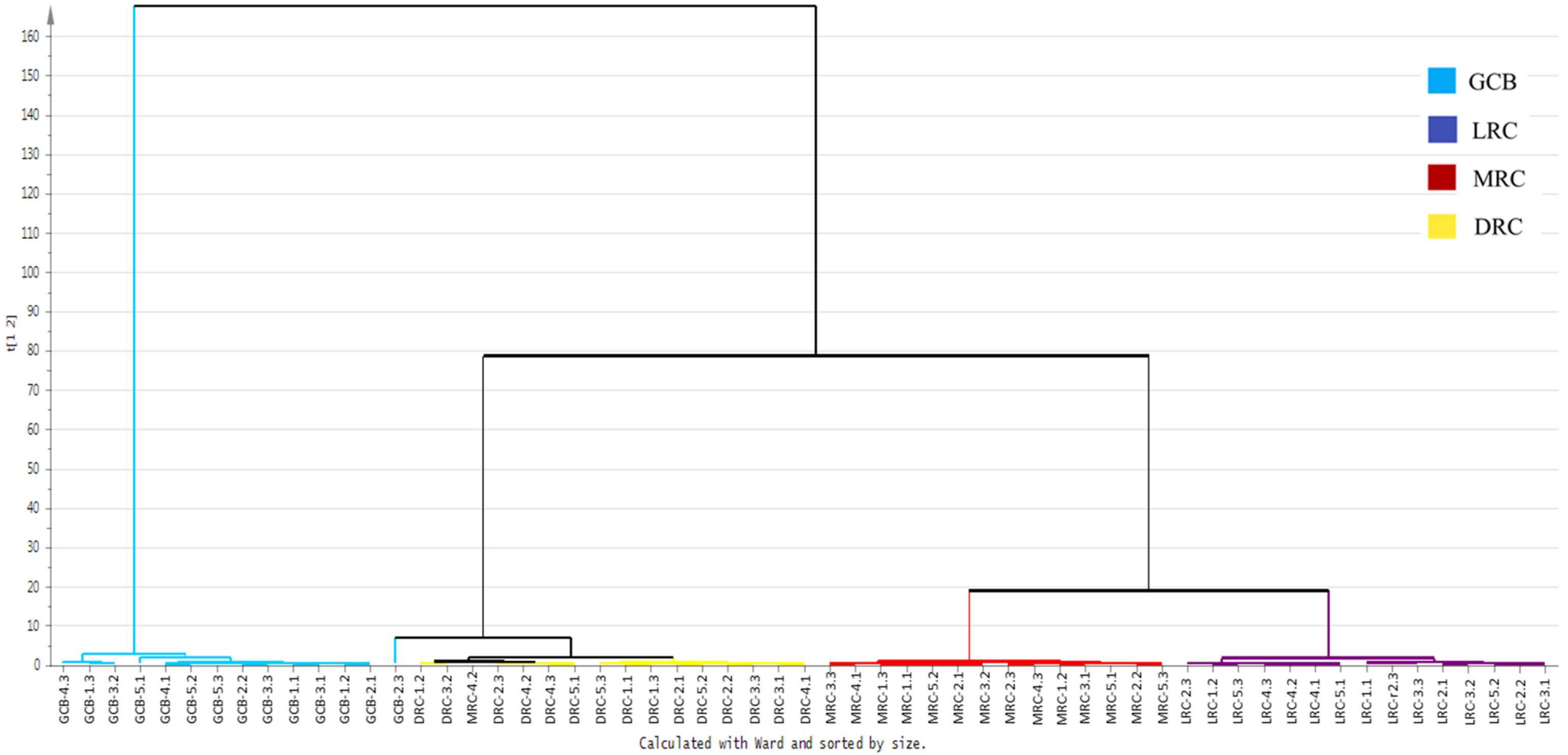
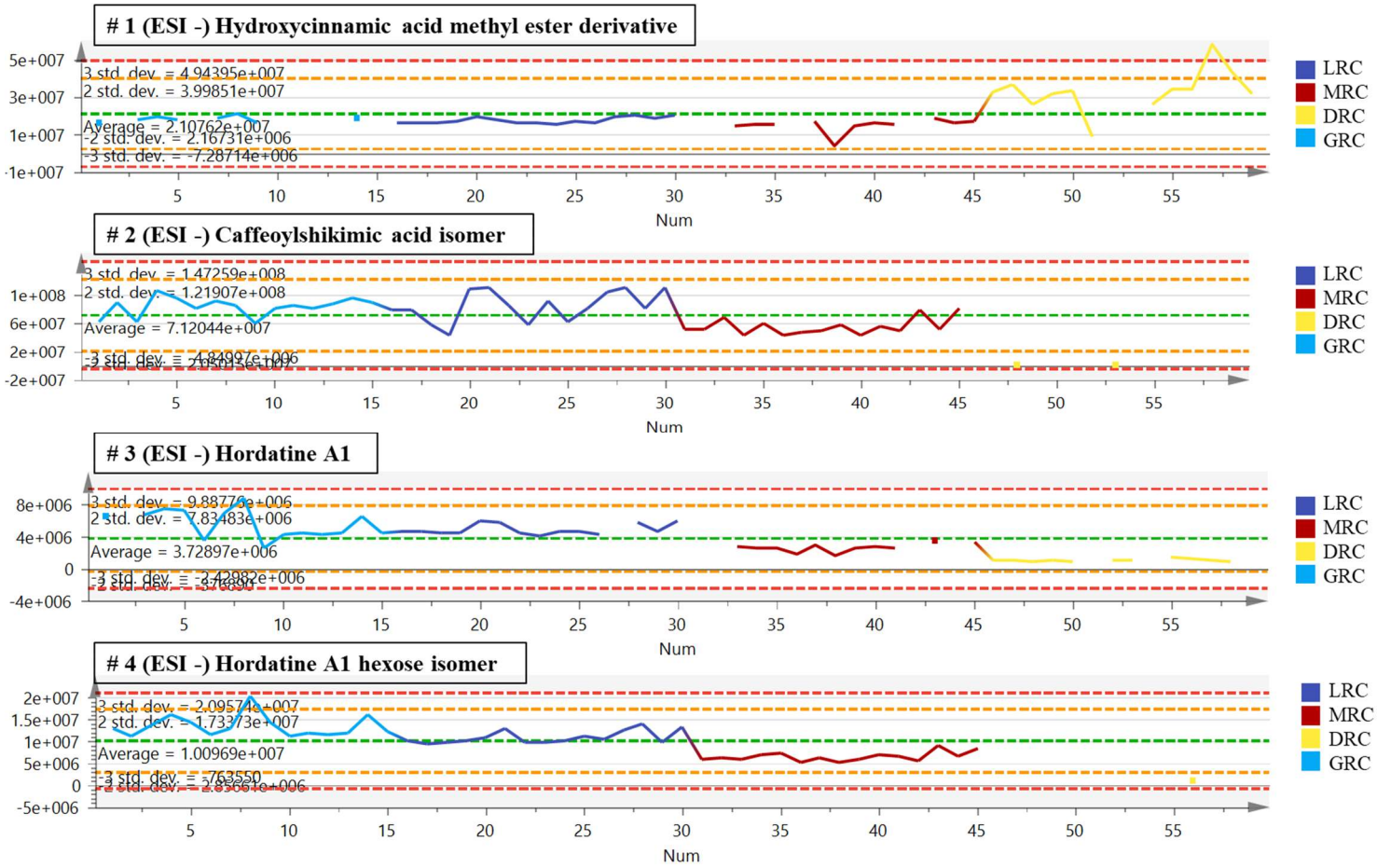
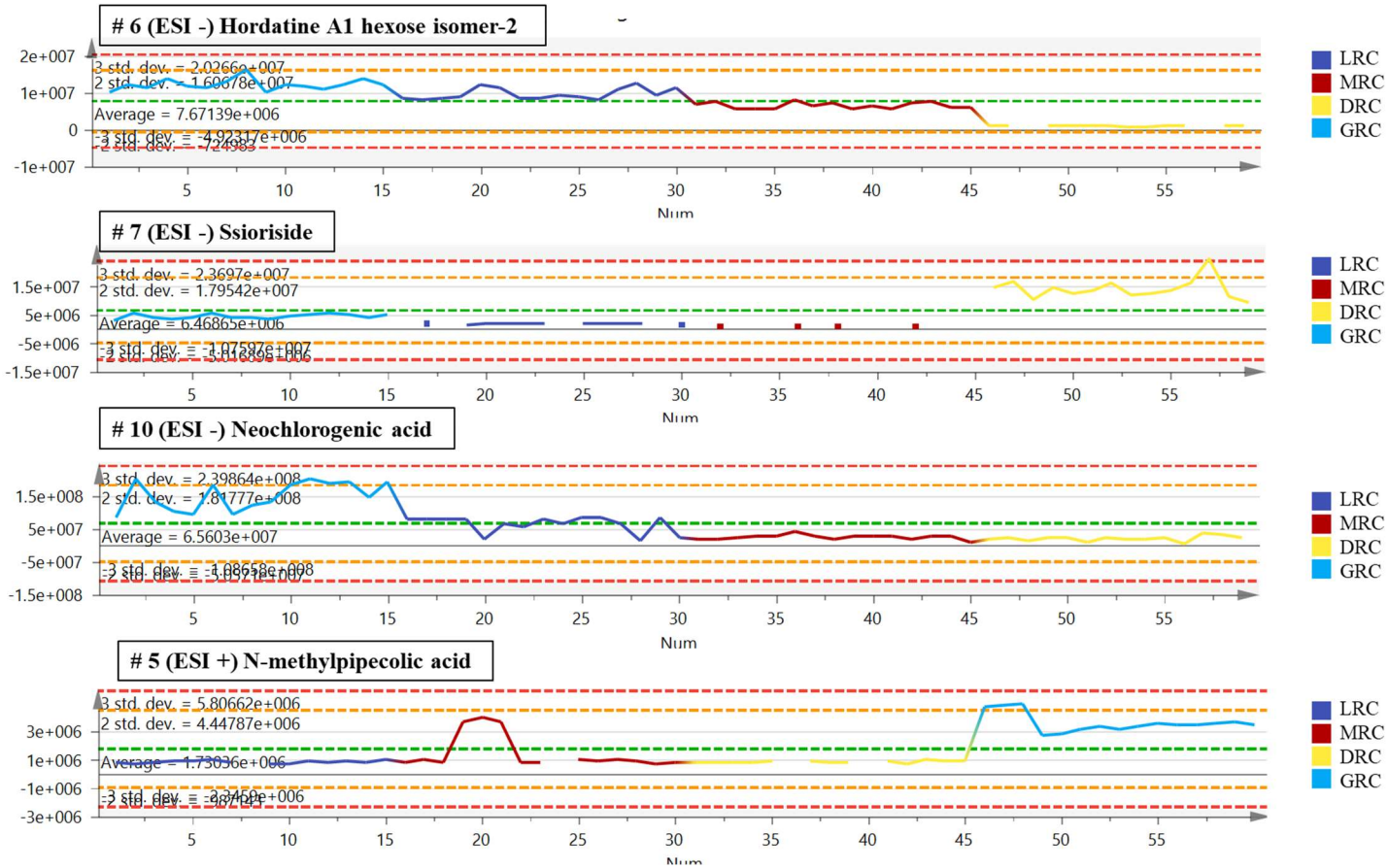
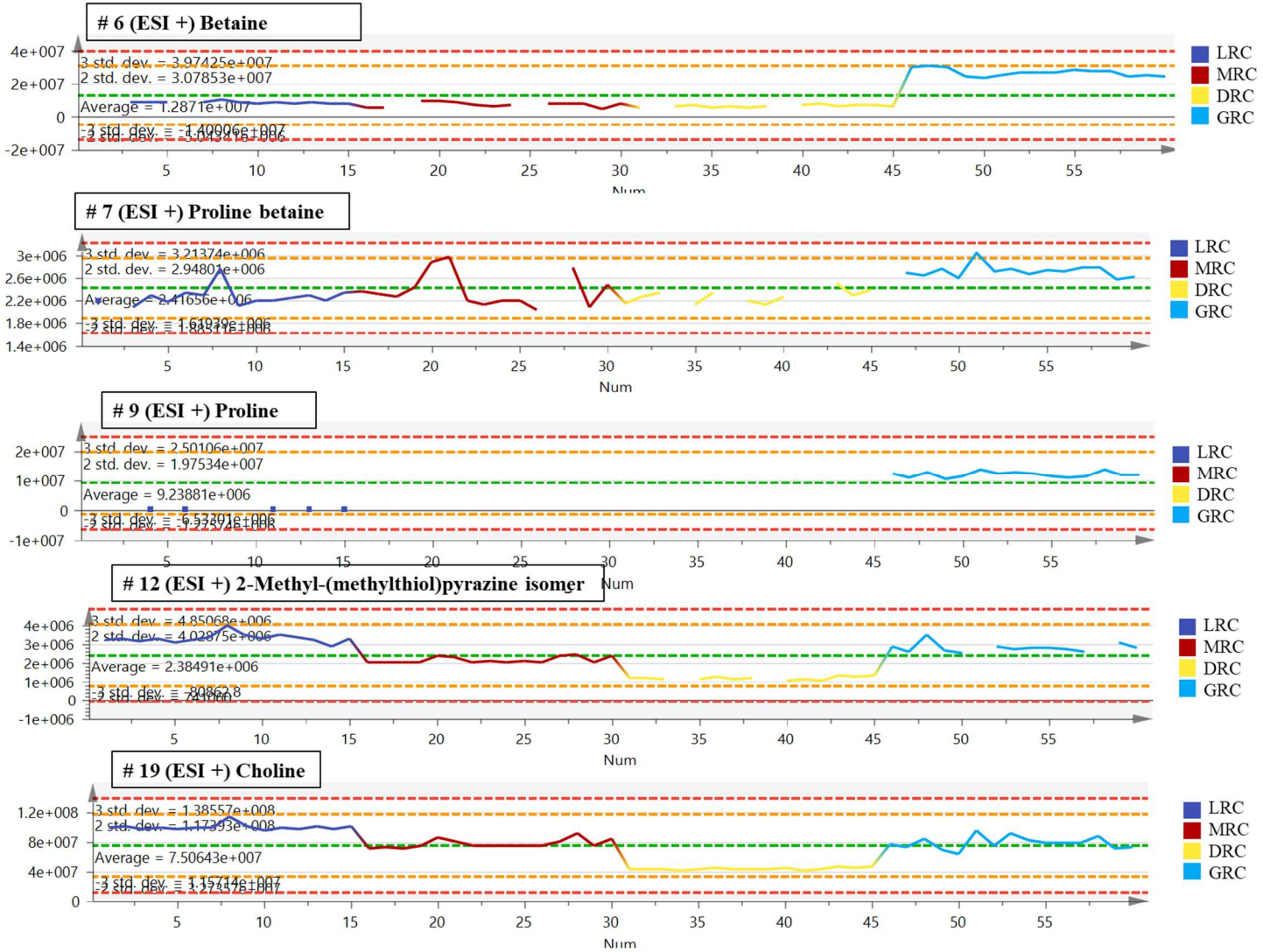

GCB
LRC
MRC
DRC
t[1 2]
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
GCB-4.3
GCB-1.3
GCB-3.2
GCB-5.1
GCB-4.1
GCB-5.2
GCB-5.3
GCB-2.2
GCB-3.3
GCB-1.1
GCB-3.1
GCB-1.2
GCB-2.1
GCB-2.3
DRC-1.2
DRC-3.2
MRC-4.2
DRC-2.3
DRC-4.2
DRC-4.3
DRC-5.1
DRC-5.3
DRC-1.1
DRC-1.3
DRC-2.1
DRC-5.2
DRC-2.2
DRC-3.3
DRC-3.1
DRC-4.1
MRC-3.3
MRC-4.1
MRC-1.3
MRC-1.1
MRC-5.2
MRC-2.1
MRC-3.2
MRC-2.3
MRC-4.3
MRC-1.2
MRC-3.1
MRC-5.1
MRC-2.2
MRC-5.3
LRC-2.3
LRC-1.2
LRC-5.3
LRC-4.3
LRC-4.2
LRC-4.1
LRC-5.1
LRC-1.1
LRC-r2.3
LRC-3.3
LRC-2.1
LRC-3.2
LRC-5.2
LRC-2.2
LRC-3.1
Calculated with Ward and sorted by size.

Figure S3.


1 (ESI -) Hydroxycinnamic acid methyl ester derivative
3 std. dev. = 4.94395e+007
2 std. dev. = 3.99851e+007
Average = 2.10762e+007
-2 std. dev. = 2.16731e+006
-3 std. dev. = -7.28714e+006
5e+007
3e+007
1e+007
-1e+007
5 10 15 20 25 30 35 40 45 50 55
Num
LRC
MRC
DRC
GRC
2 (ESI -) Caffeoylshikimic acid isomer
3 std. dev. = 1.47259e+008
2 std. dev. = 1.21907e+008
Average = 7.12044e+007
-3 std. dev. = -1.48997e+006
1e+008
6e+007
2e+007
-2e+007
5 10 15 20 25 30 35 40 45 50 55
Num
LRC
MRC
DRC
GRC
3 (ESI -) Hordatine A1
3 std. dev. = 9.88773e+006
2 std. dev. = 7.83483e+006
Average = 3.72897e+006
-3 std. dev. = -2.42982e+006
8e+006
4e+006
0
-4e+006
5 10 15 20 25 30 35 40 45 50 55
Num
LRC
MRC
DRC
GRC
4 (ESI -) Hordatine A1 hexose isomer
3 std. dev. = 2.09574e+007
2 std. dev. = 1.73373e+007
Average = 1.00969e+007
-3 std. dev. = -763550
2e+007
1.5e+007
1e+007
5e+006
0
-5e+006
5 10 15 20 25 30 35 40 45 50 55
Num
LRC
MRC
DRC
GRC


6 (ESI -) Hordatine A1 hexose isomer-2
3 std. dev. = 2.0266e+007
2 std. dev. = 1.60678e+007
Average = 7.67139e+006
-3 std. dev. = -4.92317e+006
-2 std. dev. = -7.24583e+006
2e+007
1e+007
0
-1e+007
5 10 15 20 25 30 35 40 45 50 55
Num
7 (ESI -) Ssioriside
3 std. dev. = 2.3697e+007
2 std. dev. = 1.79542e+007
Average = 6.46865e+006
-3 std. dev. = -1.07597e+007
-2 std. dev. = -5.01639e+006
1.5e+007
5e+006
-5e+006
-1.5e+007
5 10 15 20 25 30 35 40 45 50 55
Num
10 (ESI -) Neochlorogenic acid
3 std. dev. = 2.39864e+008
2 std. dev. = 1.81777e+008
Average = 6.5603e+007
-3 std. dev. = -1.08658e+008
-2 std. dev. = -1.86571e+008
1.5e+008
5e+007
-5e+007
-1.5e+008
5 10 15 20 25 30 35 40 45 50 55
Num
5 (ESI +) N-methylpipecolic acid
3 std. dev. = 5.80662e+006
2 std. dev. = 4.44787e+006
Average = 1.73836e+006
-3 std. dev. = -2.93459e+006
-2 std. dev. = -4.27083e+006
3e+006
1e+006
-1e+006
-3e+006
5 10 15 20 25 30 35 40 45 50 55
Num
LRC
MRC
DRC
GRC


6 (ESI +) Betaine
3 std. dev. = 3.97425e+007
2 std. dev. = 3.07853e+007
Average = 1.2871e+007
-3 std. dev. = -1.40996e+007
-2 std. dev. = -5.04541e+006
LRC
MRC
DRC
GRC
Num
7 (ESI +) Proline betaine
3 std. dev. = 3.21374e+006
2 std. dev. = 2.94801e+006
Average = 2.41656e+006
-3 std. dev. = 1.51939e+006
-2 std. dev. = 1.88511e+006
LRC
MRC
DRC
GRC
Num
9 (ESI +) Proline
3 std. dev. = 2.50106e+007
2 std. dev. = 1.97534e+007
Average = 9.23881e+006
-3 std. dev. = -6.52391e+006
-2 std. dev. = -3.92762e+006
LRC
MRC
DRC
GRC
Num
12 (ESI +) 2-Methyl-(methylthiol)pyrazine isomer
3 std. dev. = 4.85068e+006
2 std. dev. = 4.02875e+006
Average = 2.38491e+006
-3 std. dev. = -741068
-2 std. dev. = 808628
LRC
MRC
DRC
GRC
Num
19 (ESI +) Choline
3 std. dev. = 1.38557e+008
2 std. dev. = 1.17393e+008
Average = 7.50643e+007
-3 std. dev. = 1.15714e+007
-2 std. dev. = 3.27347e+007
LRC
MRC
DRC
GRC
Num