**Supporting Materials**

1. Calibration curve of DMAA standard in the GC method
2. Calibration curve of the internal standard (2-aminopentane) in the GC method
3. Response factor:

Concentration of internal standard

Concentration of analyte

×

Peak area of analyte

Peak area of internal standard

F=

Thus, the response factors in this study are calculated from the following equation:

Slope of analytes calibration curve

Slope of internal standard calibration curve

F=

Response factor of DMAA to internal standard = 1.02.

1. Calibration curve of DMAA in HPLC-Fl method

Mobile phase: 40%ACN + 60%H2O (containing 0.1%TFA). See details in the experimental section.

1. Validation of the HPLC-Fl quantification method

2 mg of DMAA standard was spiked into 5 g of Aura Cacia geranium oil (in which no DMAA was detected) and stirred for 10 min to make the mixture homogeneous. Take 200 mg of the mixture and derivatize it with 40 mg dansyl chloride following the procedure in the experimental section.

The product was diluted to a concentration in the linear range of the calibration curve and quantified by the HPLC-Fl method.

The experimental data was compared with the calculated concentration. The recoverage of DMAA is 93%.

1. LC-MS chromatograms of (A) dansyl-DMAA standard from ChromaDex and (B) dansylated Now Foods geranium oil. The diastereomeric ratio of the standard was ~1.42 (see Results and Discussion) and the diastereomeric ratio found in the geranium oil samples were ~0.80. Note that the retention order of the peaks in this LC-MS is the opposite of that for the GC separation shown in Figure 2. The ratios here are taken as the area of the more retained peak (second peak) divided by that of the first peak.

Mobile phase: 40%ACN + 60%H2O(containing 0.1%TFA). The mass spectra data were recorded in a positive mode of electrospray ionization for selected ion *m/z* 349. See details in the experimental section.



(A)



(B)