# Chemical Comprehensive Analysis of *Ephedra sinica* Extract, Lot No. RK-3-28-1-ES for the Botanical Safety Consortium

September 15, 2022

Submitted by:

Lori L. Smith and Kristin L. Aillon

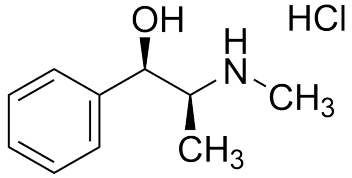
MRIGlobal
425 Dr Martin Luther King Jr Boulevard
Kansas City, MO 64110-2241

## Chemical Information: *Ephedra sinica* Extract

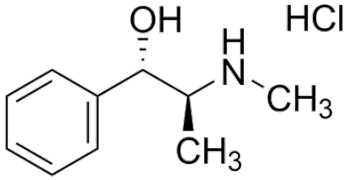
| | |
|---|---|
| CAS No.: 85940-38-1<br><br>DTXSID No.: DTXSID801018482 | Lot No.: RK-3-28-1-ES<br><br>Amount Received: 1 × ~ 153000 mg<br><br>Sample Receipt Date: 10/7/21<br><br>Appearance: Black Solid<br><br>Supplier: China Medical University<br>(Aerial parts, ground)<br>Post-Handling Supplier: University of Mississippi<br>(95% Ethanol extract)<br>Receipt Condition: Ambient, packaging intact<br>Shipping Containers: Clear-glass jar<br>Storage Condition at MRIGlobal: - 20°C under inert gas |

## Reference Standards

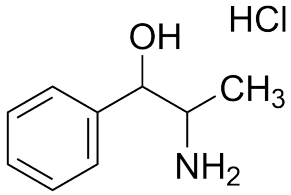
Name: Ephedrine hydrochloride (as (L)-Ephedrine)
CAS No.: 50-98-6
Supplier: USP (Rockville, MD)
Lot No.: R08630
Purity: 99.9% per C of A
Molecular Formula: $C_{10}H_{15}NO \cdot HCl$
Molecular Weight: 201.69
Structure:



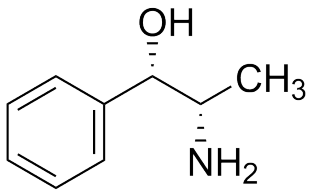

Name: Pseudoephedrine hydrochloride (as Pseudoephedrine)
CAS No.: 345-78-8
Supplier: USP (Rockville, MD)
Lot No.: R110F0
Purity: 100.0% per C of A
Molecular Formula: $C_{10}H_{15}NO \cdot HCl$
Molecular Weight: 201.69
Structure:



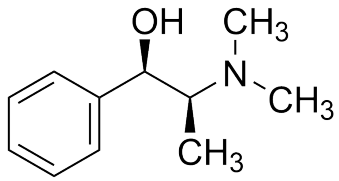

Name: DL-Norephedrine hydrochloride (as Norephedrine)
CAS No.: 154-41-6
Supplier: Sigma-Aldrich (St. Louis, MO)
Lot No.: 115K0718
Purity: >99% per C of A
Molecular Formula: $C_9H_{13}NO \cdot HCl$
Molecular Weight: 187.67
Structure:



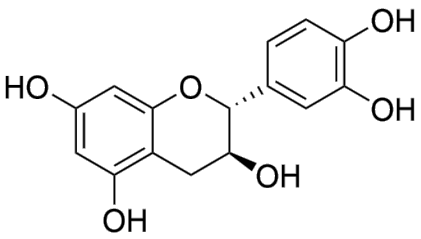
Name: Norpseudoephedrine
CAS No.: 492-39-7
Supplier: Cayman Chemical (Ann Arbor, MI)
Lot No.: 0511220
Purity: 99.4% per C of A
Molecular Formula: $C_9H_{13}NO$
Molecular Weight: 151.21
Structure:



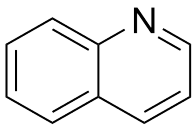

Name: N-Methylephedrine
CAS No.: 552-79-4
Supplier: Sigma-Aldrich (St. Louis, MO)
Lot No.: BCBL4243V
Purity: 99.6% per C of A
Molecular Formula: $C_{11}H_{17}NO$
Molecular Weight: 179.26
Structure:




Name: (D)-Catechin
CAS No.: 154-23-4
Supplier: USP (Rockville, MD)
Lot No.: R13070
Purity: 97% per C of A
Molecular Formula: $C_{15}H_{14}O_6$
Molecular Weight: 290.27
Structure:



| | |
|---|---|
| Name: | Quinoline |
| CAS No.: | 91-22-5 |
| Supplier: | Sigma-Aldrich (St. Louis, MO) |
| Lot No.: | BCBX3571 |
| Purity: | 98.7% per C of A |
| Molecular Formula: | $C_9H_7N$ |
| Molecular Weight: | 129.16 |
| Structure: | |



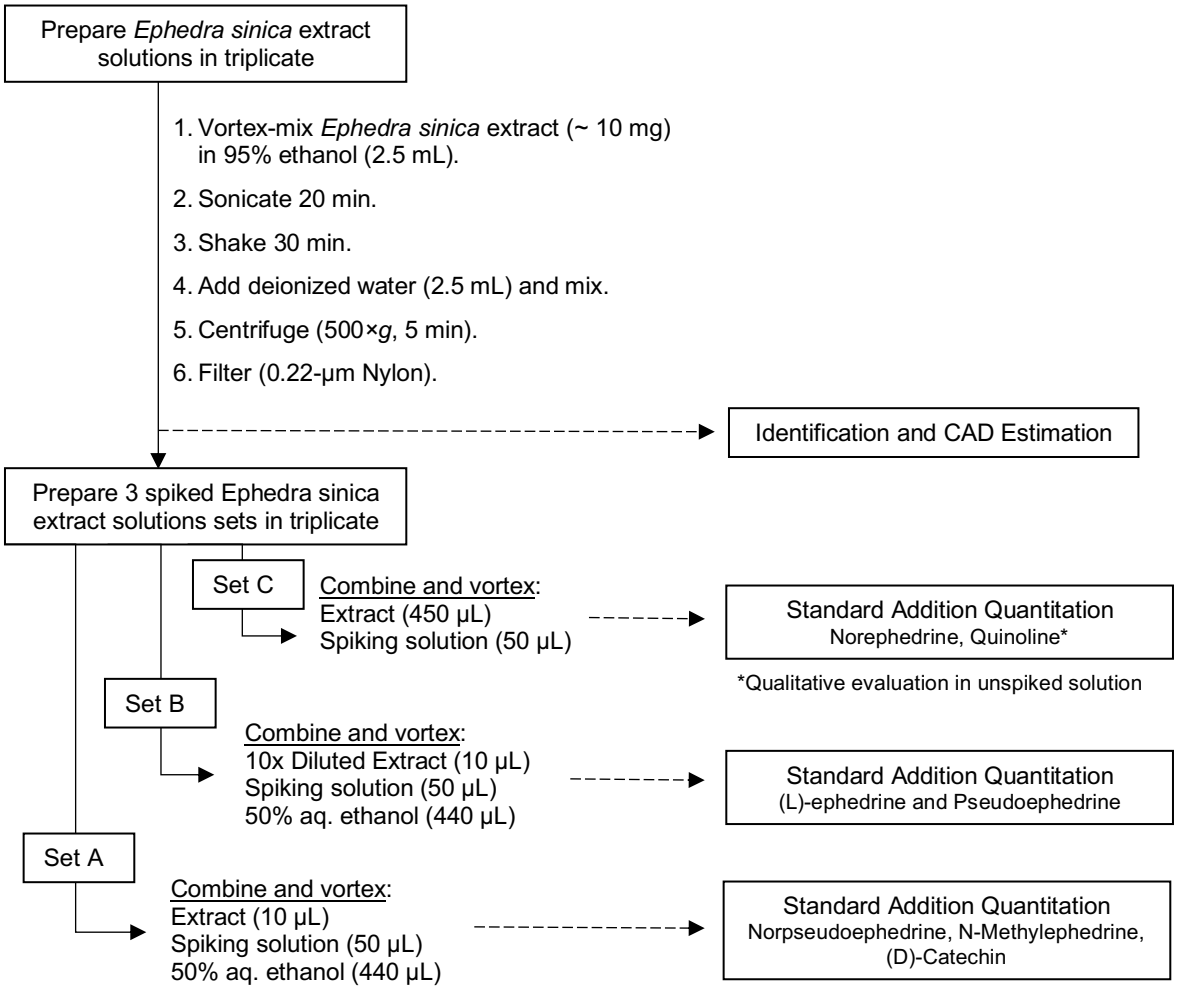




**Figure 1.** *Ephedra sinica* extract solution preparation flowchart.

## Table 1. UPLC-UV-CAD/HRMS System

| **Instrumentation:** | |
|---|---|
| **Liquid Chromatograph:** | Acquity UPLC with additional post-column inverse gradient Acquity UPLC pump (Waters Corporation; Milford, MA) |
| **Photodiode Array Detector (UV):** | Acquity PDA Detector (Waters Corporation; Milford, MA) |
| **Charged Aerosol Detector (CAD):** | Corona Veo RS (Thermo Fisher Scientific; Waltham, MA) |
| **High Resolution Mass Spectrometer (HRMS):** | Xevo G2-XS (Waters Corporation; Milford, MA) |

## Table 2. Identification and CAD Estimation - Acquisition Parameters

| **UPLC Parameters:** | |
|---|---|
| **Column:** | Avantor ACE Excel C18-PFP, 1.7 µm, 100x 2.1 mm (Advanced Chromatography Technologies, UK) |
| **Autosampler Temperature:** | 6°C |
| **Column Temperature:** | 30°C |
| **Injection Volume:** | 10 µL |
| **Flow Rate:** | 0.3 mL/min |
| **Mobile Phase A:** | 1% (v/v) Acetic Acid in Water |
| **Mobile Phase B:** | 1% (v/v) Acetic Acid in Acetonitrile |
| **Gradient Elution Profiles:** | Forward: 0% B held for 0.5 min; ramp to 10% B over 4 min; ramp to 55% B over 45 min; ramp to 100% B over 18 min and hold for 2.5 min; decreased to 0% B in 1 min and held for 4 min.<br><br>Inverse: 100% B held for 0.5 min; decrease to 90% B over 4 min; decrease to 45% B over 45 min; decrease to 0% B over 18 min and held for 2.5 min; ramped to 100% B in 1 min and held for 4 min. |
| **Run Time:** | 75 minutes |
| **UV (PDA) Parameters:** | |
| **2D Channel Wavelength:** | 254 nm |
| **3D Channel Wavelength Range:** | 190 to 400 nm, 1.2 nm resolution |
| **CAD Parameters:** | |
| **Sampling Rate:** | 10 points/sec |
| **Scale Factor:** | 1000 |
| **Evaporation Temperature:** | 35°C |
| **Scale/Power Function Value:** | 20 pA/1.0 |
| **Nitrogen** | 57.5 psi |
| **HRMS Parameters:** | |
| **Ionization/Acquisition Mode:** | ESI+; MSe sensitivity mode |
| **Acquisition Range:** | *m/z* 100 to *m/z* 1200 |
| **Scan Time:** | 0.3 s |
| **Lock Mass:** | Leucine encephalin<br>(m/z 278.1135, 556.2766; Scan for 0.200 s, interval: 45 s) |
| **Capillary Voltage:** | 2.5 kV |
| **Cone Voltage:** | 40 V |
| **Collision Energy:** | Low CE: 6 eV High CE: 20-30 eV |
| **Source Temperature:** | 110°C |
| **Desolvation Temperature:** | 250°C |
| **Cone Gas Flow:** | 50 L/h |
| **Desolvation Gas Flow:** | 600 L/h |
| **Data Acquisition System:** | UNIFI version 1.9.4 |

# Identification

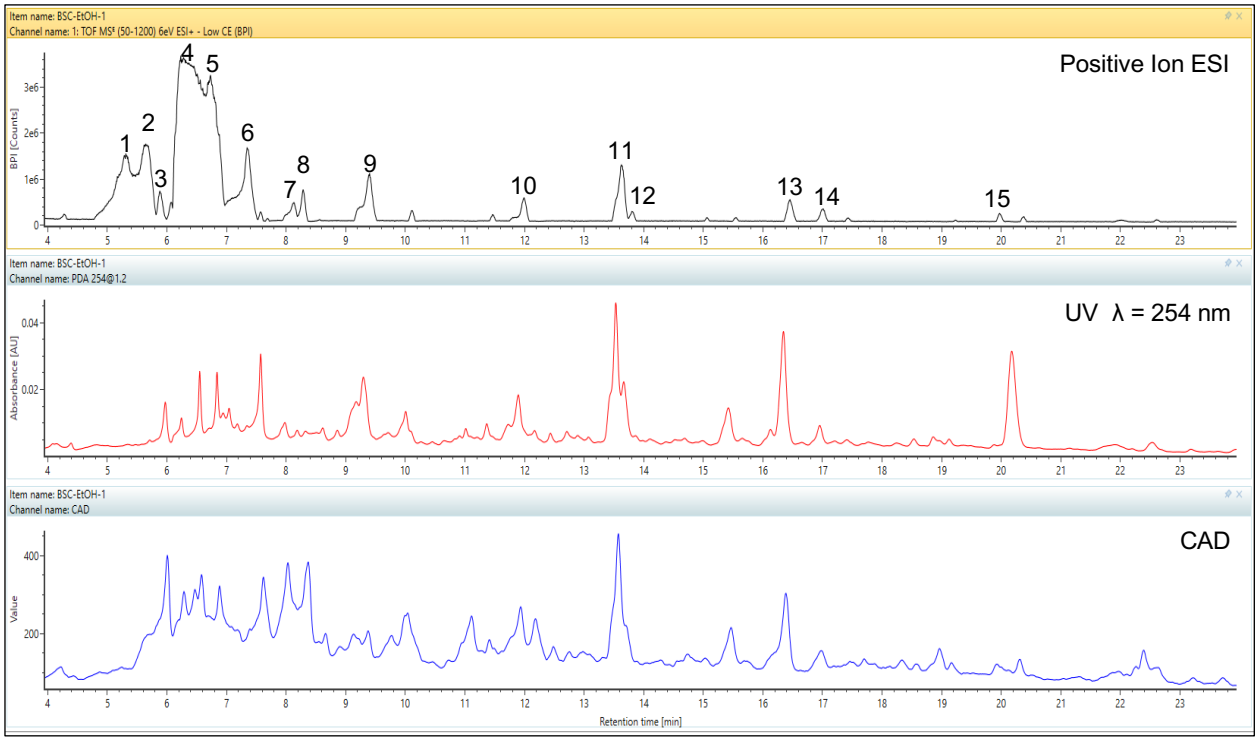
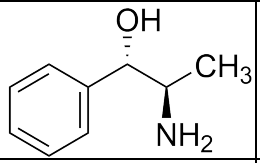
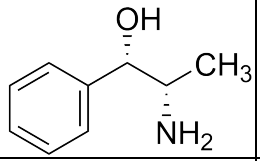
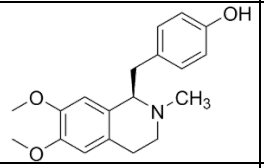
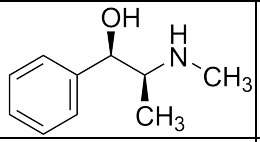
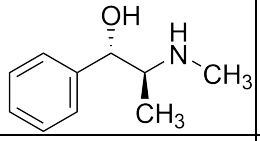
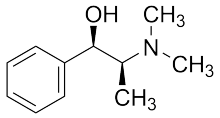
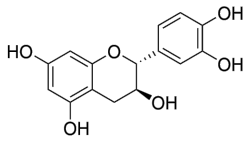
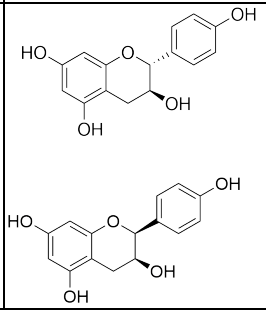
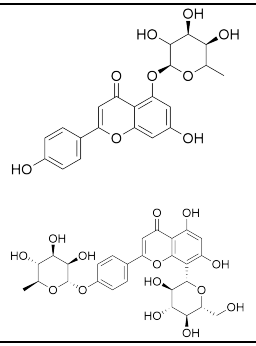
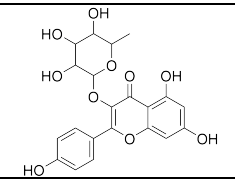
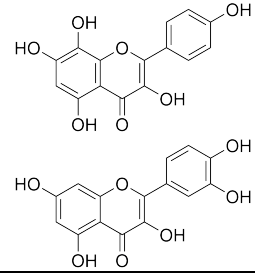



**Figure 2.** Partial chromatograms from the UPLC-UV-CAD/HRMS analysis of *Ephedra sinica* extract showing the majority of analyte peaks from the 75-min analysis. Peak numbers correlate with results presented in Table 3.

## Table 3. Identification Summary for *Ephedra sinica* Extract Components

| Peak Number | Retention Time (min) | Area (%)[a] | Observed Neutral Mass[b] (Da) | Mass Error (mDa) | Component ID | CAS | Molecular Formula | Structure | Confidence Level[c-h] | Additional Comments |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 5.31 | 10.29 | 151.0994 | -0.3 | Norephedrine | 14838-15-4 | $C_9H_{13}NO$ |  | 1 | Standard Addition Quantitation (see Table 12). |
| 2 | 5.65 | 9.38 | 151.0997 | 0.0 | Norpseudoephedrine | 492-39-7 | $C_9H_{13}NO$ |  | 1 | Standard Addition Quantitation (see Table 12). |
| 3 | 5.89 | 1.28 | 313.1536 | -14.2 | Armepavine | 524-20-9 | $C_{19}H_{23}NO_3$ |  | 3 | CAD Estimation (see Table 4). |
| 4 | 6.28 | 37.88 | 165.1155 | 0.1 | (L)-Ephedrine | 299-42-3 | $C_{10}H_{15}NO$ |  | 1 | Standard Addition Quantitation (see Table 12). |
| 5 | 6.73 | 16.21 | 165.1157 | 0.4 | Pseudoephedrine | 90-82-4 | $C_{10}H_{15}NO$ |  | 1 | Standard Addition Quantitation (see Table 12). |
| 6 | 7.35 | 8.19 | 179.1315 | 0.5 | N-methylephedrine | 552-79-4 | $C_{11}H_{17}NO$ |  | 1 | N-methylephedrine Standard Addition Quantitation (see Table 12).<br><br>Possible coelution with Pseudomethyl-ephedrine. |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 7[i] | 8.12 | 1.16 | 290.0798 | 0.8 | (D)-Catechin | 154-23-4 | $C_{15}H_{14}O_6$ |  | 1 | (D)-Catechin Standard Addition Quantitation (see Table 12).<br><br>Possible coelution with Epicatechin. |
| 8 | 8.28 | 1.28 | *m/z* 265.1553 | NA | Unknown | NA | NA | NA | 5 | Singly charged ion observed based on isotope distribution |
| 9 | 9.39 | 3.62 | *m/z* 220.0611 | 0.63 | Unknown | NA | $C_{11}H_9NO_4$ | NA | 4 | NA |
| 10A[j] | 11.89 | 1.33 | 274.0813 | -2.8 | Afzelechin and/or Epiafzelechin | 2545-00-8 and/or 36801-69-1 | $C_{15}H_{14}O_5$ |  | 3 | CAD Estimation (see Table 4); coelutes with Peak 10B. |
| 10B[j] | 11.99 | 1.33 | *m/z* 565.1589 | NA | Unknown | NA | NA | NA | 5 | Singly charged ion observed based on isotope distribution; coelutes with Peak 10A. |
| 11 | 13.63 | 3.70 | 416.1004 and 578.1644 | -10.3 and 0.8 | Apigenin-5-rhamnoside and Vitexin rhamnoside | Unassigned and 32426-34-9 | $C_{21}H_{20}O_9$ and $C_{27}H_{30}O_{14}$ |  | 3 | CAD Estimation (see Table 4).<br><br>Peak 11 has a leading shoulder indicating the presence of coeluting compounds. |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 12 | 13.81 | 0.35 | 432.1067 | 1.1 | Kaempferol-3-O-rhamnoside | Unassigned | $C_{21}H_{20}O_{10}$ |  | 3 | CAD Estimation (see Table 4). |
| 13 | 16.45 | 1.11 | 302.0442 | 1.6 | Herbacetin and/or Quercetin | 527-95-7 and/or 117-39-5 | $C_{15}H_{10}O_7$ |  | 3 | CAD Estimation (see Table 4). |
| 14 | 17.00 | 0.58 | *m/z* 679.5142 and *m/z* 340.2611 | NA | Unknown | NA | NA | NA | 5 | Singly and doubly protonated ions observed based on isotope distribution |
| 15 | 19.97 | 0.30 | *m/z* 905.6828 and *m/z* 453.3455 | NA | Unknown | NA | NA | NA | 5 | Singly and doubly protonated ions observed based on isotope distribution |
| 16[k] | 57.00 | 0.66 | 255.2558 | 35.2 | Heptadecylamine | 4200-95-7 | $C_{17}H_{37}N$ |  | 3 | No CAD response detected for quantitation estimate. |
| | Total Area (%) | 96.97 | | | | | | | | |

[a] Area (%) = (Peak Area / Total Peak Area) × 100.
[b] Reported observed mass-to-charge (*m/z)* ratio when peak identification was not definitive (Confidence Levels 4 and 5).
[c] Assignment based on published guidelines.[4]
[d] Level 1: Confirmed structure – Matched retention time, accurate mass, and fragmentation pattern to reference standard.
[e] Level 2: Proposed structure – Matched accurate mass and fragmentation pattern with library data or published literature; indicates single possible structure.
[f] Level 3: Tentative structure – Matched accurate mass and some fragments yet insufficient evidence to assign single structure (i.e., positional isomers).

[g] Level 4: Unequivocal molecular formula - Unambiguous match to molecular formula without fragmentation information.
[h] Level 5: Exact mass of interest – Accurate mass without sufficient evidence to assign molecular formula or structure.
[i] Low abundance (≤ 0.30%) peak eluting near (D)-Catechin identified as Epicatechin.
[j] Peak 10A/10B are unresolved chromatographic peaks with unique mass spectral profiles; Area % is reported as a total peak area.
[k] Peak 16 (Retention Time ~ 57.00 min) is not displayed in partial chromatogram presented in Figure 2.

## Estimation of Concentration Using CAD Response

Quantitation of *Ephedra sinica* extract components identified by accurate mass measurements and library searching (i.e., assigned Confidence Level 3; no components were assigned Confidence Level 2) was attempted using CAD responses as an estimate based on (D)-catechin response and concentration (i.e., single-point calibration). The mean CAD peak area ratio of the component to (D)-catechin was multiplied by the (D)-catechin concentration determined by standard addition quantitation (0.351% (w/w); see Table 8). CAD estimated results are summarized in Table 4.

**Table 4. CAD Estimated Results Summary for *Ephedra sinica* Extract**

| Compound | CAS | Determined Conc. Replicate 1 (% w/w) | Determined Conc. Replicate 2 (% w/w) | Determined Conc. Replicate 3 (% w/w) | Mean ± SD (RSD, %) |
|---|---|---|---|---|---|
| Armepavine | 524-20-9 | 0.321 | 0.270 | 0.332 | 0.308 ± 0.033 (10.7%) |
| Afzelechin/Epi-afzelechin[a] | 2545-00-8 and/or 36801-69-1 | 0.190 | 0.193 | 0.249 | 0.211 ± 0.033 (15.6%) |
| Apigenin-5-rhamnoside and Vitexin rhamnoside and Kaempferol-3-O-rhamnoside | Unassigned and 32426-34-9 and Unassigned | 0.360 | 0.355 | 0.383 | 0.366 ± 0.015 (4.1%) |
| Herbacetin/Quercetin | 527-95-7 and/or 117-39-5 | 0.222 | 0.222 | 0.243 | 0.229 ± 0.012 (5.2%) |

[a] Coeluting unknown identified by (+) ESI-HRMS may contribute to CAD response, artificially elevating the final result (see Additional Comments, Table 3).

## Table 5. Standard Addition Quantitation - Acquisition Parameters

| | |
|---|---|
| **UPLC Parameters:** | |
| **Column:** | Avantor ACE Excel C18-PFP, 1.7 µm, 100x 2.1 mm (Advanced Chromatography Technologies, UK) |
| **Autosampler Temperature:** | 6°C |
| **Column Temperature:** | 30°C |
| **Injection Volume:** | 1 µL |
| **Flow Rate:** | 0.3 mL/min |
| **Mobile Phase A:** | 1% (v/v) Acetic Acid in Water |
| **Mobile Phase B:** | 1% (v/v) Acetic Acid in Acetonitrile |
| **Gradient Elution Profiles:** | Forward: 0% B held for 0.5 min; ramp to 5% B over 4 min; ramp to 15% B over 9 min; decreased to 0% B in 0.5 min and held for 1 min.<br><br>Inverse: 100% B held for 0.5 min; decrease to 95% B over 4 min; decrease to 85% B over 9 min; ramped to 100% B in 0.5 min and held for 1 min. |
| **Run Time:** | 15 minutes |
| **Retention Time (min):** | Norephedrine: 7.00<br>Quinoline: 7.30<br>Norpseudoephedrine: 7.59<br>(L)-Ephedrine: 9.20<br>Pseudoephedrine: 9.85<br>N-Methylephedrine: 10.95<br>(D)-Catechin: 12.85 |
| **UV (PDA) Parameters:** | |
| **2D Channel Wavelength:** | 254 nm |
| **3D Channel Wavelength Range:** | 190 to 400 nm, 1.2 nm resolution |
| **CAD Parameters:** | |
| **Sampling Rate:** | 10 points/sec |
| **Scale Factor:** | 1000 |
| **Evaporation Temperature:** | 35°C |
| **Scale/Power Function Value:** | 20 pA/1.0 |
| **Nitrogen** | 57.5 psi |
| **HRMS Parameters:** | |
| **Ionization/Acquisition Mode:** | ESI+; MSe sensitivity mode |
| **Acquisition Range:** | *m/z* 100 to *m/z* 1200 |
| **Scan Time:** | 0.3 s |
| **Lock Mass:** | Leucine encephalin<br>(m/z 278.1135, 556.2766; Scan for 0.300 s, interval: 30 s) |
| **Capillary Voltage:** | 2.5 kV |
| **Cone Voltage:** | 40 V |
| **Collision Energy:** | Low CE: 6 eV High CE: 20-30 eV |
| **Source Temperature:** | 110°C |
| **Desolvation Temperature:** | 250°C |
| **Cone Gas Flow:** | 50 L/h |
| **Desolvation Gas Flow:** | 600 L/h |
| **Data Acquisition System:** | UNIFI version 1.9.4 |

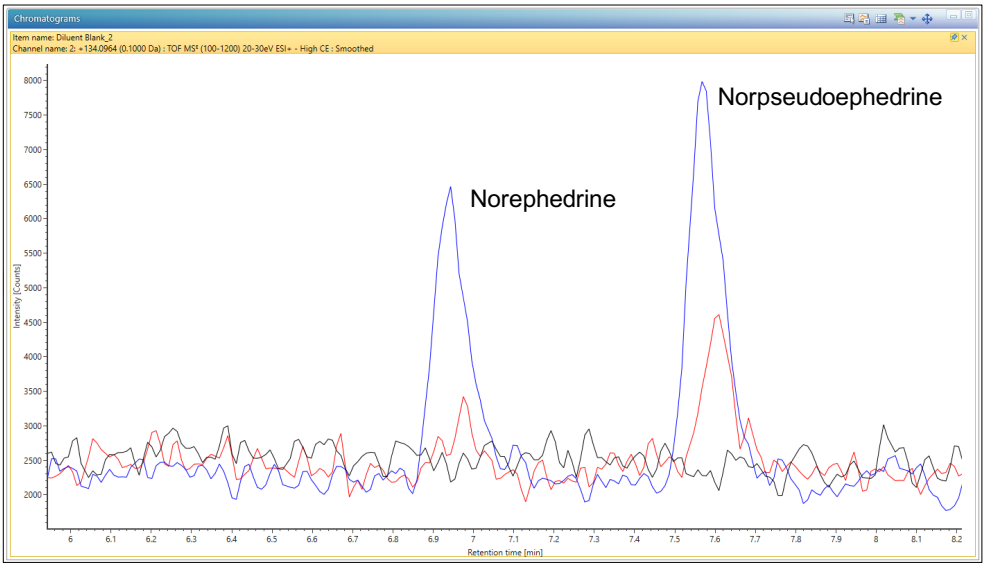
## Standard Addition Quantitation

Standard addition calibration solutions were prepared as presented in Figure 1. Individual stock standard solutions of ephedrine hydrochloride (as (L)-ephedrine), pseudoephedrine hydrochloride (as pseudoephedrine), DL-norephedrine hydrochloride (as norephedrine), norpseudoephedrine, N-methylephedrine, (D)-catechin, quinoline, and 1-phenylpropane-1,2-dione were prepared at ~ 1 mg/mL, diluted in 95% ethanol. Mixed component spiking solutions were prepared at three concentrations in 50% (v/v) aqueous ethanol (1.0, 2.5, and 5.0 µg/mL for each component) for quantitation by standard addition methodology.

The linear regression equation relating peak areas of the standard addition calibration solutions to their additional analyte concentration was determined for data without weighting. A 4-point calibration curve with triplicate standards at the lower (unspiked) and upper limits of the concentration range was used for quantitation. The concentration of each analyte in the test article solution was calculated by extrapolation of the linear regression equation to the x-axis, application of appropriate dilution factor(s), and unit conversion to express results as a weight-to-weight percentage.

Representative extracted ion chromatograms, standard addition calibration curves, and quantitation summary tables are presented in Figures 3 – 12 and Tables 6 – 12.

A.




B.

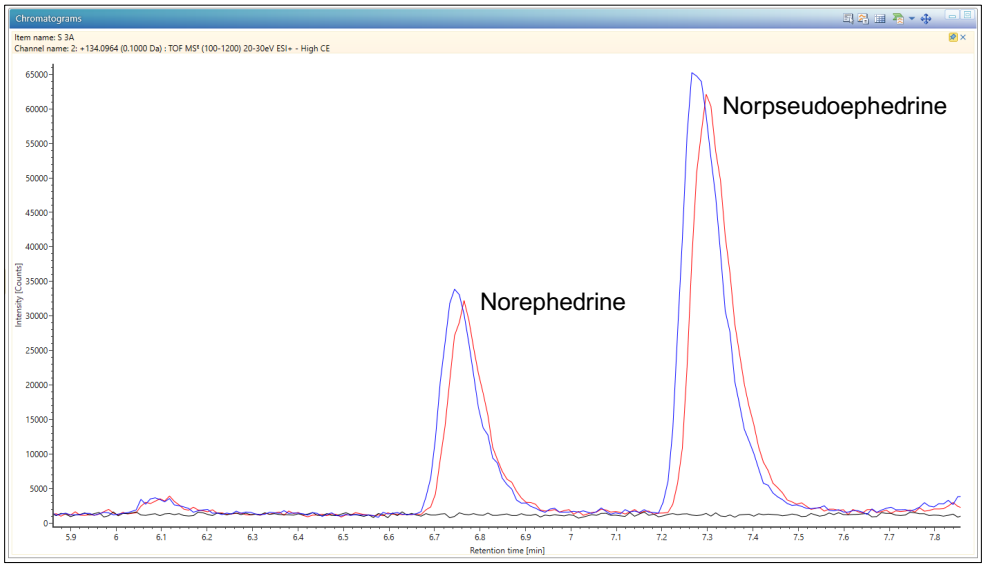



**Figure 3.** Extracted ion chromatograms (*m/z* 134.0964) for diluent blank (black), unspiked extract (A: Set A and B: Set C; red), and spiked extract (~ 250 ng/mL added; blue).

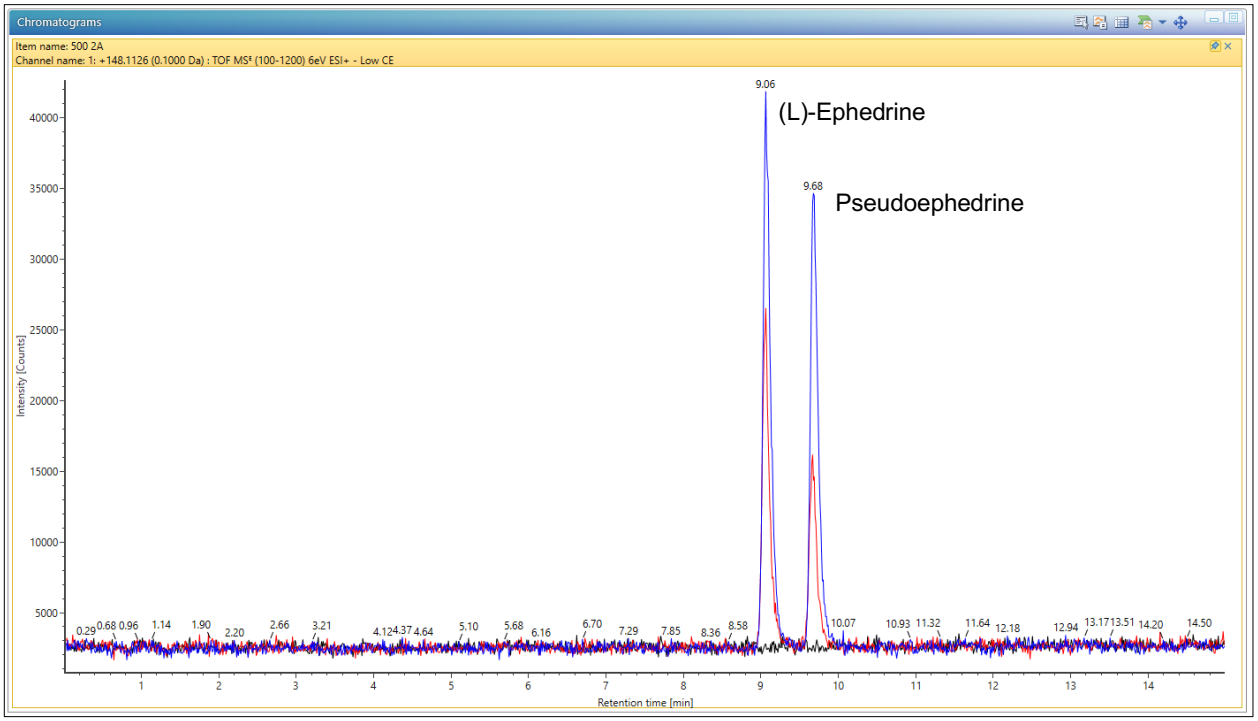



**Figure 4.** Extracted ion chromatograms (*m/z* 148.1126) for diluent blank (black), unspiked extract (Set B; red), and spiked extract (~ 100 ng/mL added; blue).

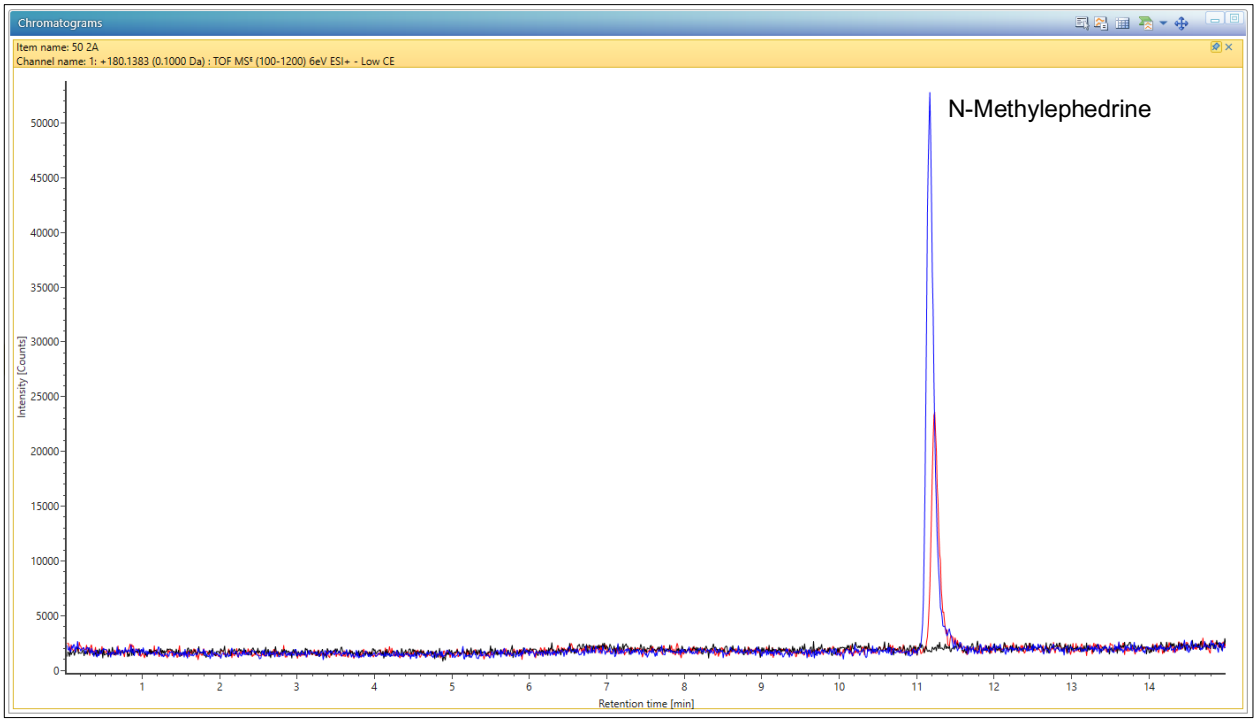



**Figure 5.** Extracted ion chromatograms (*m/z* 180.1383) for diluent blank (black), unspiked extract (Set A; red), and spiked extract (~ 100 ng/mL added; blue).

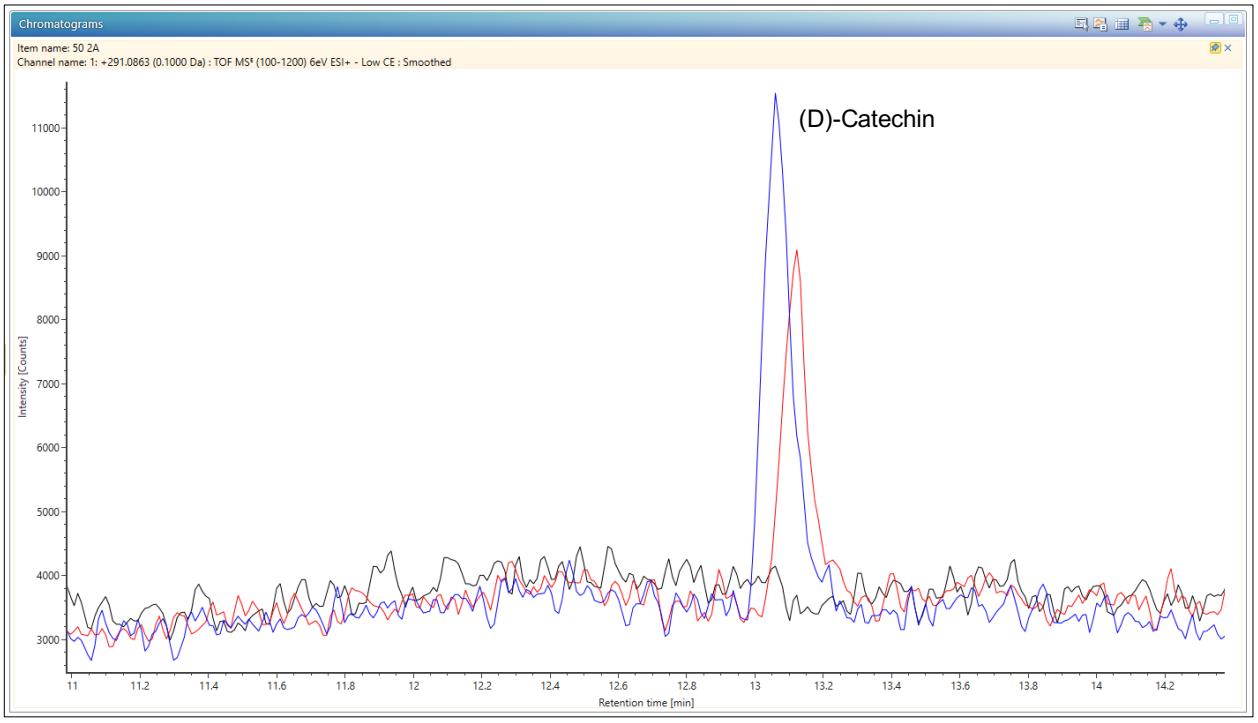



**Figure 6.** Extracted ion chromatograms (*m/z* 291.0863) for diluent blank (black), unspiked extract (Set A; red), and spiked extract (~ 100 ng/mL added; blue).

## Standard Addition Quantitation – Calibration

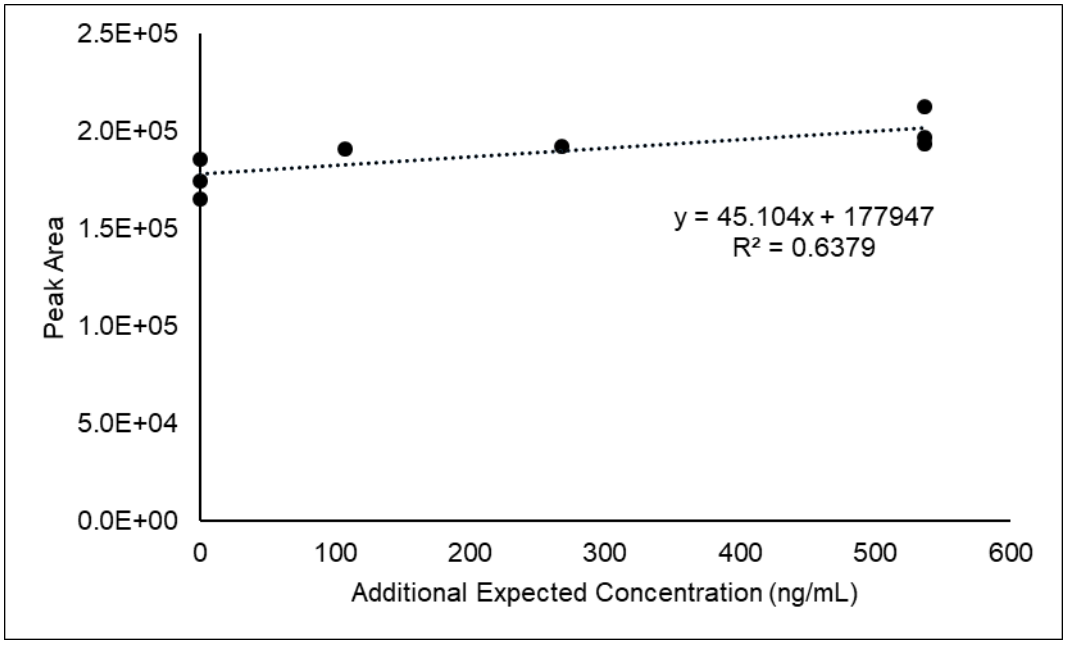



**Figure 7.** Representative standard addition calibration curve - Norephedrine

**Table 6.** Norephedrine Quantitative Results Summary for *Ephedra sinica* Extract

| Extract Replicate | Subsample (mg) | Slope | Y-Intercept | Correlation Coefficient ($R^2$) | X-Intercept[a] | Determined Concentration[b] (% w/w) |
|---|---|---|---|---|---|---|
| BSC-EtOH-1 | 11.058 | 45.104 | 177947 | 0.6379 | 3945.3 | 0.198 |
| BSC-EtOH-2 | 10.486 | 28.108 | 169511 | 0.5935 | 6030.8 | 0.320 |
| BSC-EtOH-3 | 10.276 | 32.949 | 155967 | 0.5393 | 4733.6 | 0.256 |
| | | | | | Mean ± SD (% RSD) | 0.258 ± 0.061 (23.6%) |

[a] X-Intercept = Absolute value of (Y-Intercept/Slope).

[b] Determined Conc. (% w/w) = X-Intercept × (500 µL/450 µL) × (5 mL/2.5 mL) × (2.5 mL/Subsample in mg) × (1000 mg/1 g) × (1 g/$10^9$ ng) × 100.

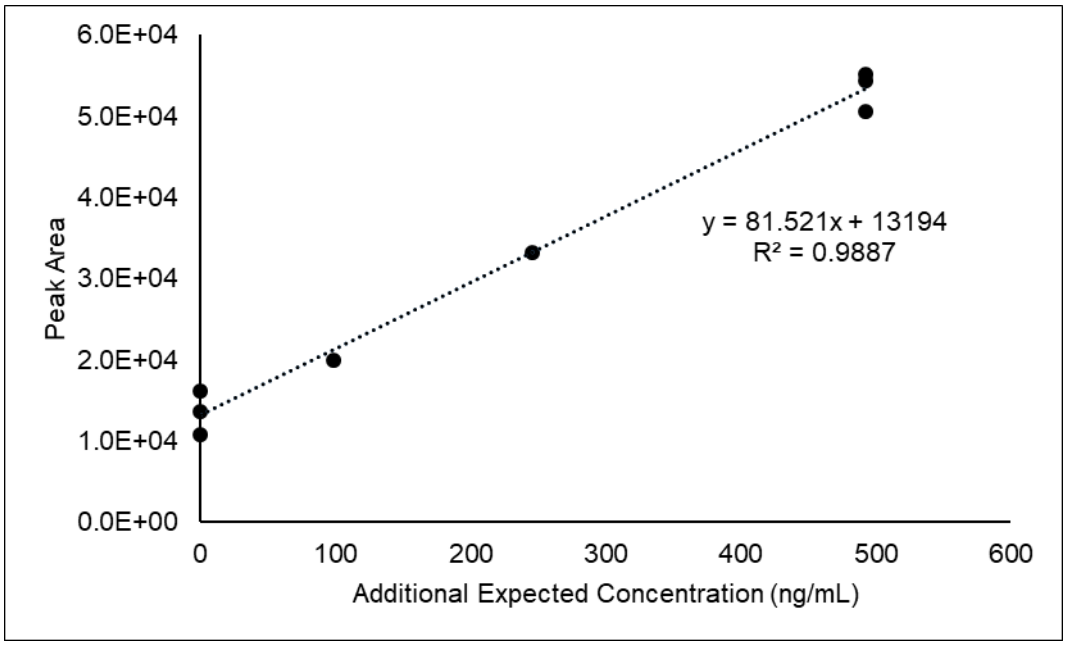



**Figure 8.** Representative standard addition calibration curve – Norpseudoephedrine

**Table 7.** Norpseudoephedrine Quantitative Results Summary for *Ephedra sinica* Extract

| Extract Replicate | Subsample (mg) | Slope | Y-Intercept | Correlation Coefficient ($R^2$) | X-Intercept[a] | Determined Concentration[b] (% w/w) |
|---|---|---|---|---|---|---|
| BSC-EtOH-1 | 11.058 | 81.521 | 13194 | 0.9887 | 161.85 | 0.366 |
| BSC-EtOH-2 | 10.486 | 77.741 | 12398 | 0.9795 | 159.47 | 0.380 |
| BSC-EtOH-3 | 10.276 | 73.963 | 12049 | 0.9773 | 162.91 | 0.396 |
| | | | | | Mean ± SD (% RSD) | 0.381 ± 0.015 (3.9%) |

[a] X-Intercept = Absolute value of (Y-Intercept/Slope).

[b] Determined Conc. (% w/w) = X-Intercept × (500 µL/10 µL) × (5 mL/2.5 mL) × (2.5 mL/Subsample in mg) × (1000 mg/1 g) × (1 g/$10^9$ ng) × 100.

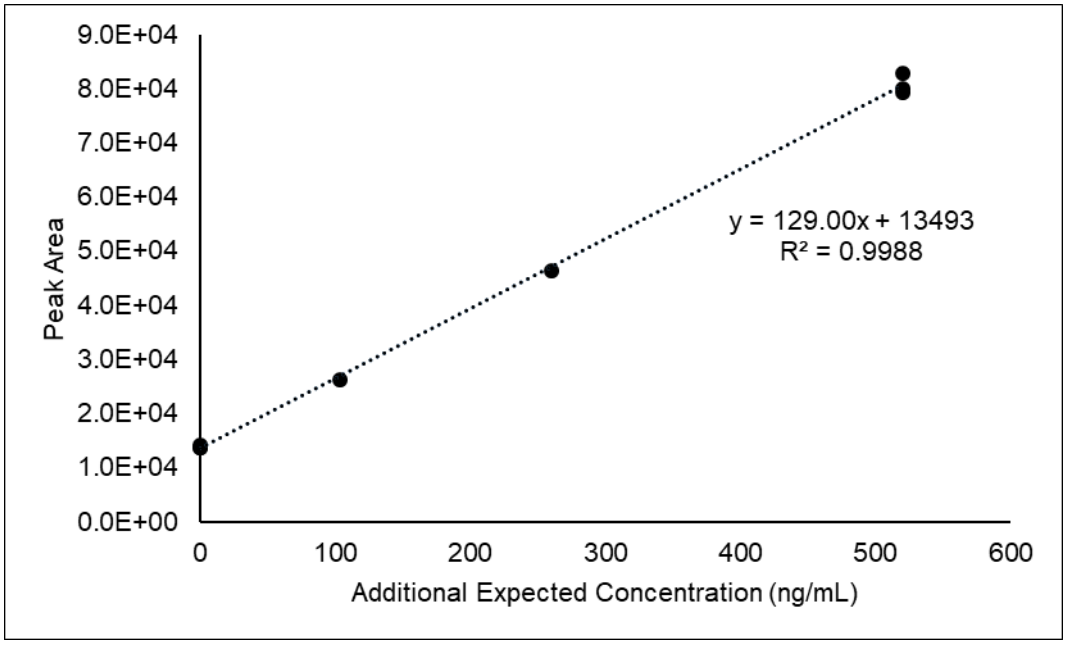



**Figure 9.** Representative standard addition calibration curve – (L)-Ephedrine

**Table 8.** (L)-Ephedrine Quantitative Results Summary for *Ephedra sinica* Extract

| Extract Replicate | Subsample (mg) | Slope | Y-Intercept | Correlation Coefficient ($R^2$) | X-Intercept[a] | Determined Concentration[b] (% w/w) |
|---|---|---|---|---|---|---|
| BSC-EtOH-1 | 11.058 | 129.00 | 13494 | 0.9988 | 104.60 | 2.36 |
| BSC-EtOH-2 | 10.486 | 111.14 | 12223 | 0.9939 | 109.97 | 2.62 |
| BSC-EtOH-3 | 10.276 | 99.776 | 11849 | 0.9956 | 118.76 | 2.89 |
| | | | | | Mean ± SD (% RSD) | 2.62 ± 0.27 (10.1%) |

[a] X-Intercept = Absolute value of (Y-Intercept/Slope).
[b] Determined Conc. (% w/w) = X-Intercept × (500 µL/50 µL) × (500 µL/10 µL) × (5 mL/2.5 mL) × (2.5 mL/Subsample in mg) × (1000 mg/1 g) × (1 g/$10^9$ ng) × 100.

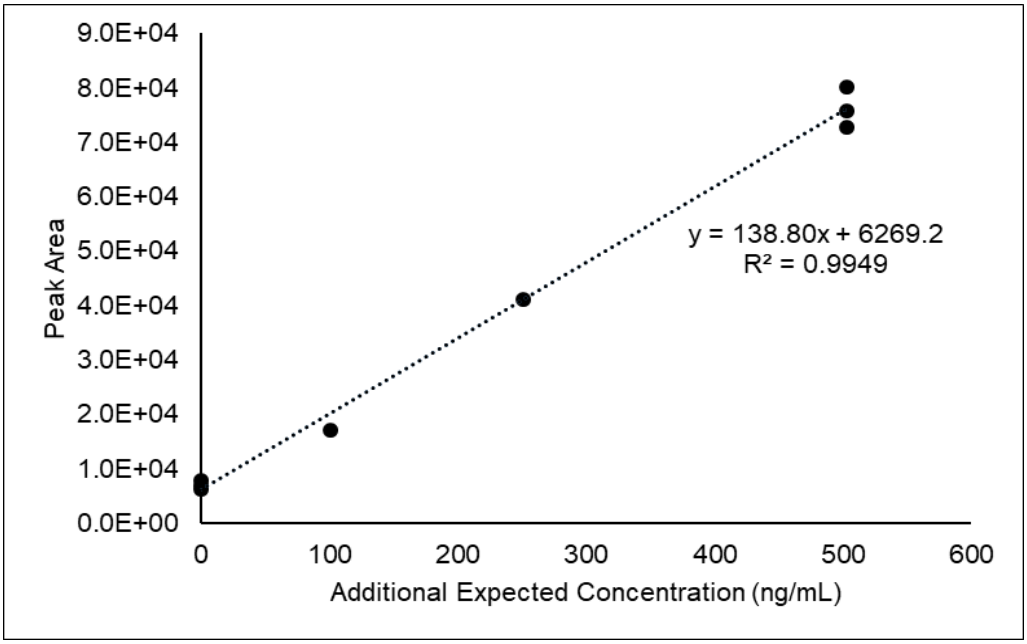



**Figure 10.** Representative standard addition calibration curve – Pseudoephedrine

**Table 9.** Pseudophedrine Quantitative Results Summary for *Ephedra sinica* Extract

| Extract Replicate | Subsample (mg) | Slope | Y-Intercept | Correlation Coefficient ($R^2$) | X-Intercept[a] | Determined Concentration[b] (% w/w) |
|---|---|---|---|---|---|---|
| BSC-EtOH-1 | 11.058 | 138.80 | 6269.2 | 0.9949 | 45.166 | 1.02 |
| BSC-EtOH-2 | 10.486 | 121.91 | 7804.4 | 0.9895 | 64.015 | 1.53 |
| BSC-EtOH-3 | 10.276 | 113.62 | 5776.4 | 0.9927 | 50.837 | 1.24 |
| | | | | | Mean ± SD (% RSD) | 1.26 ± 0.26 (20.2%) |

[a] X-Intercept = Absolute value of (Y-Intercept/Slope).

[b] Determined Conc. (% w/w) = X-Intercept × (500 µL/50 µL) × (500 µL/10 µL) × (5 mL/2.5 mL) × (2.5 mL/Subsample in mg) × (1000 mg/1 g) × (1 g/$10^9$ ng) × 100.

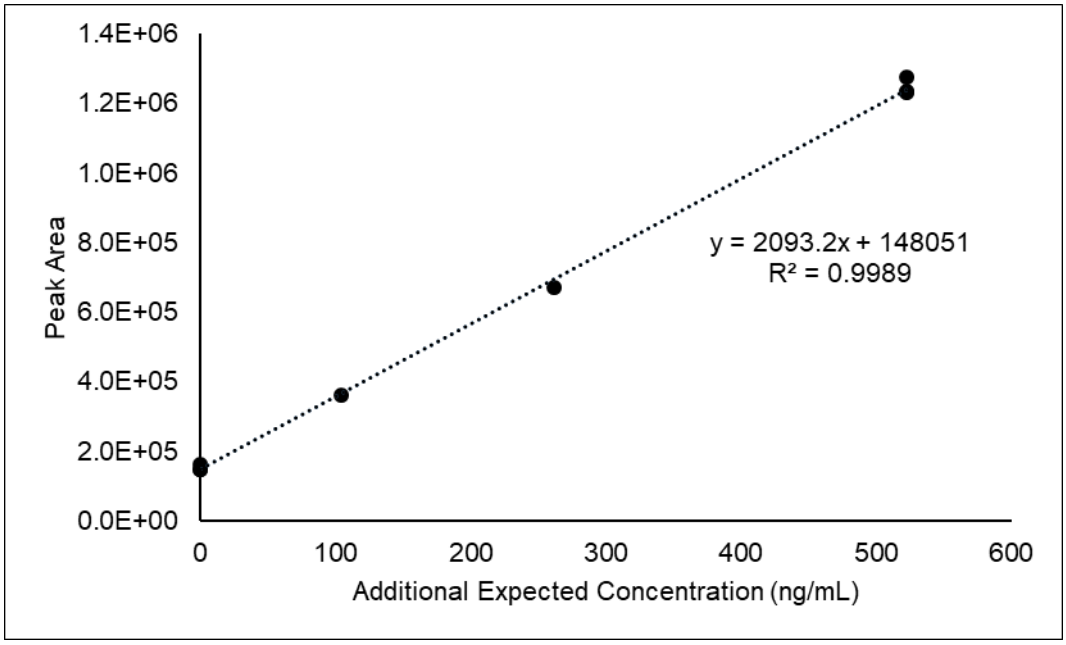



**Figure 11.** Representative standard addition calibration curve – N-Methylephedrine

**Table 10.** N-Methylephedrine Quantitative Results Summary for *Ephedra sinica* Extract

| Extract Replicate | Subsample (mg) | Slope | Y-Intercept | Correlation Coefficient ($R^2$) | X-Intercept[a] | Determined Concentration[b] (% w/w) |
|---|---|---|---|---|---|---|
| BSC-EtOH-1 | 11.058 | 2093.2 | 148051 | 0.9989 | 70.729 | 0.160 |
| BSC-EtOH-2 | 10.486 | 2080.9 | 130116 | 0.9922 | 62.528 | 0.149 |
| BSC-EtOH-3 | 10.276 | 1851.6 | 129970 | 0.9990 | 70.192 | 0.171 |
| | | | | | Mean ± SD (% RSD) | 0.160 ± 0.011 (6.9%) |

[a] X-Intercept = Absolute value of (Y-Intercept/Slope).
[b] Determined Conc. (% w/w) = X-Intercept × (500 µL/10 µL) × (5 mL/2.5 mL) × (2.5 mL/Subsample in mg) × (1000 mg/1 g) × (1 g/$10^9$ ng) × 100.

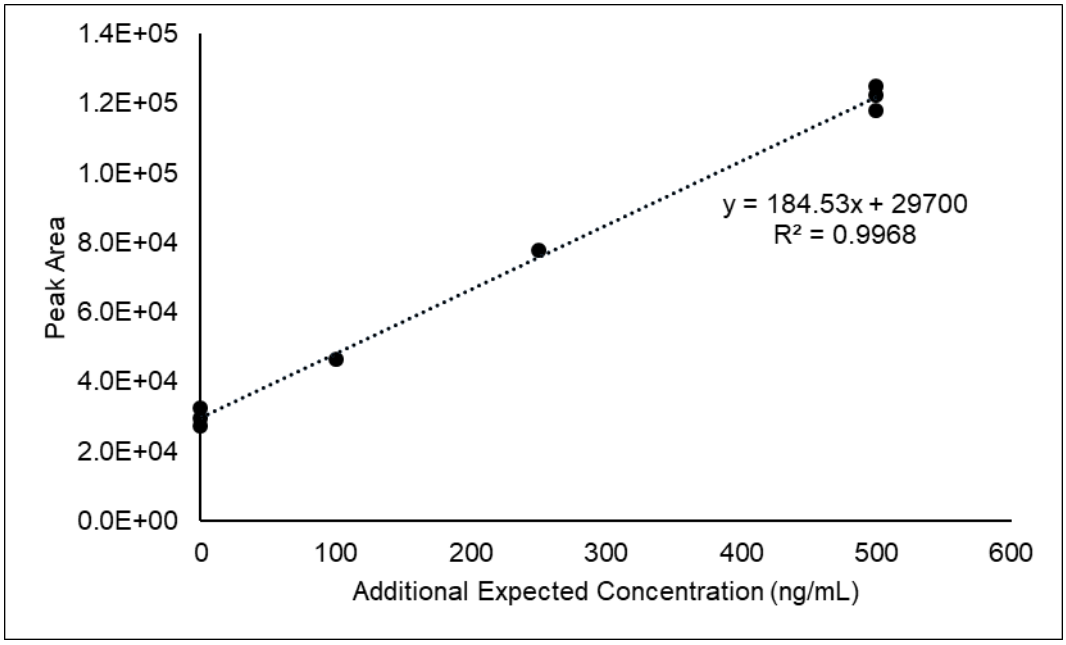



**Figure 12.** Representative standard addition calibration curve – (D)-Catechin

**Table 11.** (D)-Catechin Quantitative Results Summary for *Ephedra sinica* Extract

| Extract Replicate | Subsample (mg) | Slope | Y-Intercept | Correlation Coefficient ($R^2$) | X-Intercept[a] | Determined Concentration[b] (% w/w) |
|---|---|---|---|---|---|---|
| BSC-EtOH-1 | 11.058 | 184.53 | 29700 | 0.9968 | 160.95 | 0.364 |
| BSC-EtOH-2 | 10.486 | 182.49 | 23847 | 0.9828 | 130.67 | 0.312 |
| BSC-EtOH-3 | 10.276 | 157.51 | 24467 | 0.9960 | 155.34 | 0.378 |
| | | | | | Mean ± SD (% RSD) | 0.351 ± 0.035 (10.0%) |

[a] X-Intercept = Absolute value of (Y-Intercept/Slope).
[b] Determined Conc. (% w/w) = X-Intercept × (500 µL/10 µL) × (5 mL/2.5 mL) × (2.5 mL/Subsample in mg) × (1000 mg/1 g) × (1 g/$10^9$ ng) × 100.

**Table 12. Standard Addition Quantitative Results Summary for *Ephedra sinica* Extract**

| Reference Compound | CAS | Determined Conc. Replicate 1 (% w/w) | Determined Conc. Replicate 2 (% w/w) | Determined Conc. Replicate 3 (% w/w) | Mean ± SD (RSD, %) |
|---|---|---|---|---|---|
| Norephedrine | 14838-15-4 | 0.198 | 0.320 | 0.256 | 0.258 ± 0.061 (23.6%) |
| Quinoline | 91-22-5 | ND | ND | ND | ND |
| Norpseudoephedrine | 492-39-7 | 0.366 | 0.380 | 0.396 | 0.381 ± 0.015 (3.9%) |
| (L)-Ephedrine | 299-42-3 | 2.36 | 2.62 | 2.89 | 2.62 ± 0.27 (10.1%) |
| Pseudoephedrine | 90-82-4 | 1.02 | 1.53 | 1.24 | 1.26 ± 0.26 (20.2%) |
| N-Methylephedrine[a] | 552-79-4 | 0.160 | 0.149 | 0.171 | 0.160 ± 0.011 (6.9%) |
| (D)-Catechin | 154-23-4 | 0.364 | 0.312 | 0.378 | 0.351 ± 0.035 (10.0%) |

ND = No response detected above baseline noise in undiluted extract solutions.
[a] Possible coelution with pseudomethylephedrine.