

LC-MS Sample Preparation

Sample

Acetonitrile extraction / dilution

Shake / vortex / sonicate

Centrifuge

Filter

Analyze

LC-MS Data Analysis

30 analytes – alkaloids, drugs, natural toxins

Evaluate total-ion chromatograms (TICs) and extracted-ion chromatograms (EICs).

Evaluate full scan MS and dependent scan MS/MS data.

Compare to FCC built mass spectral library.

GC-MS Method Overview

Purpose

To screen a wide variety of matrices for the presence of poisons and toxins

Instrumentation

Agilent 5973/75N MSD equipped with electron-impact ionization source and option for positive or negative chemical ionization

GC-MS Equipment



GC-MS Sample Preparation

Sample

1 M glycine buffer (pH 10) and separately
1 M glycine buffer (pH 3)

Acetonitrile extraction add NaCl to saturate aqueous layer

Centrifuge

Analyze

GC-MS Data Analysis

8 analytes – alkaloids, drugs, pesticides

Evaluate total-ion chromatograms (TICs) and extracted-ion chromatograms.

Evaluate mass spectral data.

Compare to Wiley Registry of Mass Spectral Data, 7th Edition.

GC-MS Data Analysis

National Institute of Standards and Technology

Automated Mass Spectral Deconvolution & Identification System (AMDIS)

Currently 4000 hazardous chemicals

Cyanide Screen

Purpose

To provide a qualitative method for the detection of cyanide (CN⁻).

Equipment

Cyanotesmo Test Paper

Cyanide Antidote Packages

Cyanide Screen Sample Preparation

Sample

Check pH

Wet Cyanotesmo paper with distilled water

Acidify sample with H₂SO₄

Suspend Cyanotesmo paper in vessel and cap tightly

Cyanide Screen Data Analysis

Check color of test strips periodically and note any color change.



Negative



Positive

Cyanide Screen

Purpose

To provide a qualitative method for the detection of cyanide (CN⁻).

Chloramine-T Pyridine- Barbituric acid colorimetric method modification of EPA 335.2

Equipment

Visible spectrometer

Ricin

Primary screening techniques:

Lateral flow

ELISA

Confirmatory Technique

Separation from matrix/trypsin digestion/LC-MS

Botulinum Toxin

Primary screening techniques

ELISA

Confirmatory Techniques

Mice

FERN Training Chemistry

ICP-MS August 7-10

GC-MS August 15-17

GC-MS proposed FY07 Virginia

Proficiency Tests LC-MS February 2005

Interim FERN Chemistry method

Poison screen by LC-MS FCC T022(004)

Identification of colchicine only

30 of 34 labs got correct results

Proficiency Tests LC-MS September 2005

Interim FERN Chemistry method

Poison screen by LC-MS FCC T022(004)

Identification of Strychnine and Eserine

26 of 28 labs got correct results

Proficiency Tests GC-MS September 2005

Interim FERN Chemistry method

Poison screen by GC-MS FCC T021(003)

Identification of Strychnine

27 of 28 labs got correct results

Proficiency Tests ICP-MS September 2005

Interim FERN Chemistry method

Toxic element screen by ICP-MS FCC TO26 (001)

Identification and quantitation of arsenic and thallium in apple juice

Solution 1.....3.6 ug/ml As

Solution 2.....1.5 ug/ml As + 1.8ug/ml Tl

Proficiency Tests
ICP-MS
September 2005

21 of 21 labs correctly identified arsenic in both samples
21 of 21 labs correctly identified thallium
20 of 21 labs reported a correct result for both samples
Sample 1 As (3.6 ug/ml) rsd 13%
Sample 2 As (1.5 ug/ml) rsd 14%
Sample 2 Tl (1.8 ug/ml) rsd 5%

Proficiency Tests
Ricin
Feb/May 2005

Interim FERN Chemistry method
Ricin screen by lateral flow device LFD 21
Ricin screen by ELISA 16
Feb. Quantitative results a problem
May Qualitative results

- FERN Method Submission
- FERN Guidance for Validation of Chemistry Methods
- Contact the FERN National Program Office

- **New Mexico State University's Physical Science Laboratory**
 - LC-MS, GC-MS, ICP-MS procedures to detect toxic chemicals in foods

- IDEAS???
- GAPS???