

Proficiency Testing
Toxic Chemicals in Food

7th Annual Foodborne Pathogen
Analysis Conference

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Proficiency Test
LC-MS
February 2005

Interim FERN Chemistry method

Poison screen by LC-MS FCC T022(004)

Identification of colchicine only

Post results on eLEXNET/fax/or email within 3 days

LC-MS Method Overview

Purpose

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

Instrumentation

Thermo Finnigan LTQ Ion Trap Mass Spectrometer

Agilent 1100 Binary Capillary HPLC

LC-MS Equipment



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.

LC-MS Screen

- If a control is available for comparison an unlimited number of toxic chemicals may be detected in the screen

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Sample 1 cranberry-grape juice

Sample 2 cranberry-grape juice + 2 ug/ml colchicine

Sample 3 cranberry-grape juice

Sample 4 cranberry-grape juice + 0.1 ug/ml cochicine

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34 labs participated

33 labs reported results into Elexnet

32 labs met the reporting deadline

1 lab did not get correct results for samples 1,3,4

4 labs did not get correct results for sample 2

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ICP-MS

Analysis of an unknown

GC-MS Method Overview

Purpose

To evaluate a wide variety of foodstuffs for the presence of potentially toxic materials amenable to analysis by GC.

Instrumentation

Agilent 5973N 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 Screen

- If a control is available for comparison an unlimited number of toxic chemicals may be detected in the screen

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

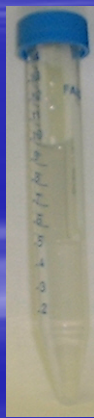
Wet Cyanotesmo paper with distilled water

Acidify sample with H_2SO_4

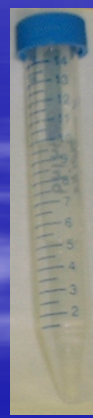
Suspend Cyanotesmo paper in vessel and cap tightly

Cyanide Screen Data Analysis

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



Negative



Positive

CT Method Analysis Time

LC-MS

Total analyst time = 0.5 – 1 hour per sample

Total instrument time = 1.5 – 2 hours per sample

ID of unknown time = ?

GC-MS

Total analyst time = 0.5 – 1 hour per sample

Total instrument time = 1 – 1.5 hours per sample

ID of unknown time = ?

15-20 samples/24 hrs.

Limited by instrumentation not personnel

CT Method Analysis Time

Cyanide Screen

Total analyst time = 0.25 – 0.5 hours per sample

Total test time = 2 hours per sample(s) – up to 20 samples

200 + samples / day

Other CT Methods

Inductively Coupled Plasma – Mass Spectrometry
(ICP-MS)

elemental analysis

ICP-MS time dependent on food matrix digestion

Other CT Methods

ELISA

Ricin

Bot toxin

Other CT Methods

Fourier Transform – Infrared Spectroscopy (FT-IR)

identification of unknown solids and liquids

Ion Chromatography (IC)

detection of inorganic anions

Methods under Development

Ricin

QTOF-MS

Fluoroacetate

IC

GC-MS

Azide

IC

GC-MS

Paraquat

LC-MS