



<b>ATF-LS-E07</b> <b>Pyrodex System</b>	Published Online: <b>March 2018</b>
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### **I. Scope:**

Liquid chromatography is a separation technique based on selective interactions between a stationary phase and a mobile phase. This method is used to analyze intact samples and combustion products of Hodgdon Pyrodex<sup>®</sup> and Triple Seven to indicate the presence of dicyanodiamide (DCDA), benzoic acid and 3-nitrobenzoic acid.

Pyrodex<sup>®</sup>, a patented commercially available black powder substitute, contains (REDACTED).

The columns utilized have polystyrene divinylbenzene packings. The column separation mechanism is based on weak anion exchange. An UV detector is used to detect DCDA, benzoic acid and 3-nitrobenzoic acid eluting from the HPLC system.

### **II. References:**

Bender, E.C., 1989a, The Analysis of dicyanodiamide and sodium benzoate in Pyrodex<sup>®</sup> by HPLC, *Crime Laboratory Digest*, **16**, 78-83

Validation

See above mentioned reference.

### **III. Apparatus/Reagents:**

#### **Instrument Parameters:**

Either of the following columns can be used to achieve the separation:

- Brownlee Polypore H (PS-DVB H) 10 micron particle size, 10.0 cm long, 4.6 mm I.D.
- Phenomenex Rezex RFQ-Fast Fruit H+ (8%), 10.0 cm long, 7.8 mm I.D.

Injection Volume: 5 - 25 uL

As instrument parameters such as detection wavelength, flow rate, mobile phase, and column temperature may change depending on laboratory and sample conditions; refer to the parameter sheet for specific values.

#### **Standard Solutions and/or Reagents**

An authentic standard solution can be prepared by utilizing Pyrodex<sup>®</sup> or Triple Seven dissolved in water.

### **IV. Safety Precautions:**

Make sure appropriate eye protection is worn

### **V. Procedures:**

#### **Sampling and Sample Prep**

(REDACTED) can be extracted from Pyrodex<sup>®</sup> particles or post blast debris with water. The extract is filtered using a suitable filter of 0.45 micron pore size or less.

### **Analytical Procedures**

1. Allow instrument to equilibrate.
2. Run a blank to ensure the system is clean.
3. Run a standard.
4. Run blanks until the system is clean.
5. Run sample. Repeat step 4 and 5 until all samples are analyzed.
6. Run blanks until the system comes out clean.
7. Run a standard.

### **VI. Quality Assurance/Quality Control:**

#### **Possible sources of error:**

1. Temperature has a dramatic effect on retention time, column backpressure and selectivity.
2. Benzoates are a common environmental interference.
3. The retention times of the first and last standards should not shift more than 5% within a sequence.
4. Since (REDACTED) degrades in solution, the extract should be analyzed as soon as possible.

### **Glossary**

**DCDA-** dicyanodiamide (a.k.a. cyanoguanidine)

**HPLC-** high performance liquid chromatography

**LC-** liquid chromatography

**PPM-** parts per million

**PS-DVB H-** Polystyrene Divinylbenzene in the Hydrogen form

**UV-** Ultraviolet