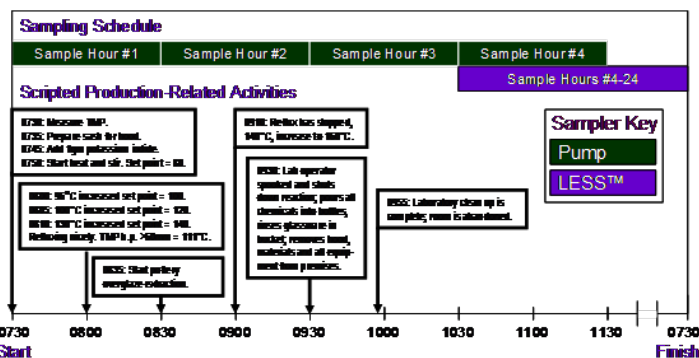


Chemical Signature Collection Demonstration with LESS™

Mock Scenario

An individual chemist is working on a synthetic scheme to make an illicit chemical, using a makeshift hood, glassware and equipment. After three hours, the individual shuts down the activity abruptly for fear of being arrested; he bottles up chemicals, boxes up equipment and trash, removes the hood, and leaves the premises. It is assumed that prior to beginning the scripted activities in this scenario, the individual made and purified trimethylphosphite (TMP) from phosphorus trichloride, methanol, and triethylamine and that he will react TMP with sodium iodide to make dimethyl methylphosphonate (DMMP), an intermediate end product. The activity timeline is shown below.



Testing

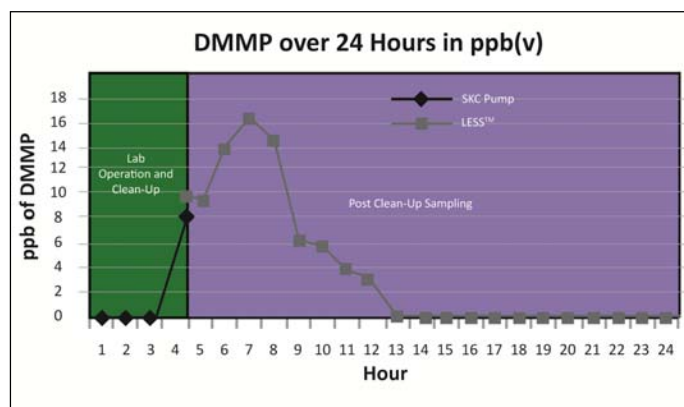
Samples were collected over a 24-hour period during and after a chemical synthesis simulating the illicit production of banned chemical. During and immediately following synthesis activities, samples were collected inside the clandestine laboratory using a traditional constant air flow pump. After the lab was cleaned and emptied of all equipment and materials (at the start of hour four), the LESS™ sampler was introduced, mimicking the arrival of law enforcement after the lab operator fled the scene. CAMSCO's glass ATD tubes (Carboxen 569) were used in both samplers. The pump-collected tubes were manually unsealed, attached to the pump's tubing, detached after each hour and resealed by a technician. The LESS™ sampler manifold was loaded with 23 tubes and programmed to automatically collect air samples on each of 21 tubes, from Hour 4 through Hour 24 and included two QC blanks.

Analytical

Sorbents were removed from the tubes and extracted with high purity acetone (2 mL). Sample extracts were analyzed on a Thermo Fisher Focus GC/Quantum Access Triple Quadrupole mass spectrometer (GC/MS) using a 30 meter 1701 column. One μL from each sample extract was injected into a Thermo FocusLiner™ inlet liner and operated in split mode with a 10:1 split. Instrument control and data collection were accomplished by Thermo Xcalibur x2.0.7. Samples were quantified based on a 5-point calibration curve, all with R2 values of greater than 0.99. Instrument cleanliness was verified by acetone blanks injected every five samples, and calibration and sensitivity were verified by calibration check and sensitivity check standards analyzed every 10 samples.

Results

DMMP, an intermediate end product, was demonstrated to be a likely indoor air signature for small-scale synthesis in homeland security scenarios. DMMP was found in air samples for 11 hours after clean-up of the make-shift laboratory, as shown in the graph below. The other reactants and material used were not detected.



Conclusions

The results show that the intermediate product was present for a considerable time in the room after the reaction. Employment of a more sensitive/analytical method (such as thermal desorption, GC/MS or a solvent extraction, DESI/MSn) may have resulted in DMMP detections past 11 hours.

LESS™ System Specifications

Manifold Sample Capacity: 28 Sorbent tubes (industry standard 89mm L x 6.4mm OD)

Independent Flow Paths: 2 (14 positions per flow path)

Flow Range Options per path:

- 0-30 mL/min
- 0-200 mL/min
- 0-1000 mL/min

Flow accuracy +/- 5% (based on flow sensor manufacturer specs)

Maximum concurrent sample tubes at any given time: 2

Operational Modes:

- Programmed cascade samples
- Duplicate samples
- Distributive pair samples (two samples concurrent at different flow rates).
Example: one sample at 50ml/min and the other at 200ml/min

Operational temperature range: 4°-50°C

Flow path wetted materials: Silco treated 6061 Aluminum, 316 Stainless Steel, and FKM

Isolation valve wetted materials: 316 stainless steel, PEEK, FKM, and epoxy

Flow Control: Mass Flow Meter utilizing Pulse Width Modulation pump speed control (x2)

Sample Inlet Filter: 30 Micron

Input Power: 85-220 VAC 50/60 Hz

System native power: 12 VDC

Power Consumption: Minimum 1 Watt, Maximum 10 Watt

Communication Options:

- RS-232
- RS-485
- Ethernet
- Fiber optic
- Modbus
- Cellular transceiver
- Short haul modem
- Telephone modem
- Satellite transceiver
- Spread spectrum radio

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