

TECHNICAL NOTE

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Capillary Electrophoretic Analysis of Phosphorus Species in Clandestine Methamphetamine Laboratory Samples

ABSTRACT: The clandestine manufacture of methamphetamine is a spreading epidemic. Manufacturing methods are constantly changing, necessitating the implementation of new analytical tools to identify materials from these labs. Characterization of phosphate, phosphite, and hypophosphite ions is necessary to distinguish the various phosphorus–iodine manufacturing methods that are popular methods for reducing pseudoephedrine to methamphetamine. This work describes a capillary electrophoresis method to separate acetate, azide, bromide, carbonate, chlorate, chloride, chromate, fluoride, hypophosphite, iodide, nitrate, nitrite, perchlorate, phosphate, phosphite, sulfate, sulfite, and thiocyanate. The CELixerOA™ 8.2 dynamic coating system was modified by lowering the capillary temperature to 15°C and using an acid flush between runs to remove adsorbed materials. This allows detection of ions down to between 10 and 30 parts per million with percent relative standard deviations of normalized migration times under 0.1%. This method is a valuable tool for the characterization of phosphate, phosphite, and hypophosphite in routine analysis of clandestine methamphetamine manufacturing evidence and has a broader application in other areas of forensic analysis.

KEYWORDS: forensic science, methamphetamine, manufacture, capillary electrophoresis, phosphorus, dynamically coated capillaries, hypophosphorous acid, phosphate, phosphite, hypophosphite

Methamphetamine use in the United States has reached epidemic levels over the past decade. Clandestine methamphetamine laboratories present hazards to the health of individuals, communities, and the environment. The two most common methods of methamphetamine manufacture in the United States are the reduction of pseudoephedrine with lithium and anhydrous ammonia (1) or with red phosphorus and iodine or hydriodic acid (2). The latter method, and its variations, is the focus of this work.

Variations on illicit manufacturing methods develop as regulations and enforcement efforts address the availability of starting materials (Doug Snyder, US Drug Enforcement Administration, personal communication, 2001) (3). For example, other phosphorus-containing reducing agents, such as hypophosphorous acid and phosphorous acid, have been substituted for red phosphorus. These variations can present analytical challenges in determining the illicit manufacturing method. This is important because identifying the method of manufacture can assist in the investigation and prosecution of these cases by determining the chemicals used and by providing more accurate production capacity estimates.

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Funding Sources: This project was supported by Grant Number 2003-LT-BX-K004 awarded by the National Institute of Justice, Office of Justice Programs, US Department of Justice. Points of view in this document are those of the authors and do not necessarily represent the official position or policies of the US Department of Justice.

Received 29 Jan. 2005; and in revised form 9 July 2005; accepted 19 July 2005; published 26 Dec. 2005.

The analysis of organic species currently plays an important role in distinguishing common clandestine methamphetamine manufacturing methods. Variations on the phosphorus–iodine reduction method using different phosphorus reducing agents can produce the same organic by-products (Peter Vallely, personal communication, Queensland Health Scientific Services Australia, 2004). This presumably occurs because the organic precursor is subject to the same strong hydriodic acid reductive conditions in each variation. In this case, therefore, the analysis of inorganic species can often help determine which phosphorus-containing reducing agent was used.

A variety of elemental analysis techniques, including X-ray fluorescence (XRF), scanning electron microscopy-energy dispersion X-ray (SEM-EDX), and inductively coupled plasma-mass spectrometry (ICP-MS), may be used to identify inorganic materials, e.g., phosphorus (4), but cannot distinguish phosphate, phosphite, hypophosphite, and elemental phosphorus without coupled separation techniques or species-specific sample preparations. Some species-specific sample preparation techniques and analysis methods have been developed for the identification of specific inorganic species, including the sublimation of red phosphorus with subsequent analysis by gas chromatography-mass spectrometry (5,6). Several wet chemical methods are also available for the characterization of inorganic species (6–9), but the majority of these techniques do not meet the needs of this study: that is, to characterize phosphate, phosphite, and hypophosphite in complex mixtures at low concentrations.

A variety of liquid-chromatographic techniques have been used in the separation and analysis of anion species, including capillary electrophoresis (CE) and ion chromatography (IC) (10–13). Some of the demonstrated non-DNA forensic applications of CE include the analysis of solid dose drug samples, pre- and postblast explo-

sives, and toxicological samples (14–19). Lurie's demonstration of the application of CE ion analysis in understanding the origin of illicit heroin samples is of particular relevance (12). That study demonstrated the application of CE analysis to low levels of cations and anions in heroin samples for the purpose of identifying unusual salt forms of the drug and for comparison with other illicit samples for source determination.

CE provides rapid analysis, low part per million detection limits, needs very little sample preparation, and generates only small amounts of waste. Sample preparation is often as simple as diluting the material to an appropriate concentration in CE grade water or buffer. Commonly available UV–Vis diode array detectors can be used for both UV active and inactive compounds if an absorbing background electrolyte is included in the run buffer (20).

Any CE method used for analysis of routine clandestine laboratory evidence must provide reproducible migration times, good-quality peak shape, and separation of the anionic species of interest within a reasonable time frame. The characterization of phosphorus–iodine manufacturing methods requires the resolution of the three phosphorus oxyacids (hypophosphite, phosphite, and phosphate) and the halogens, in particular iodide. The identification of nitrate, phosphate, and sulfate may be useful in determining which type of ammonium salt was used in the clandestine generation of ammonia (21).

The current commercial methods for CE anion analysis evaluated did not adequately meet these requirements. Janssens et al. (22) developed a series of methods with improved electroosmotic flow (EOF) and migration time stability using dynamic coating reagents. The use of dynamically coated capillaries has been demonstrated for the forensic analysis of controlled substances (23,24). For the method used in this study, a bare fused-silica capillary is coated first with a polycation (initiator), followed by a background electrolyte containing a UV absorber (accelerator). This article outlines the development of a new CE method, by modifying this commercially available dynamic coating system, for the examination of anionic phosphorus species in samples from clandestine drug manufacturing laboratories. In this procedure, adsorbed material is removed and the coating is reapplied before each run. The described modifications of the manufacturer's recommended method, including changes in temperature, column length, and column conditioning, meet the above requirements for the analysis of clandestine laboratory evidence. In addition, the developed method is suitable as an inorganic anion analysis method for other forensic applications, including explosives analysis, other methamphetamine manufacturing methods, or simply identifying unknown inorganic species.

Materials and Methods

Chemicals

Reagent grade chemicals were purchased from Sigma-Aldrich Chemical Company (St. Louis, MO), JT Baker (Phillipsburg, NJ), EM Science (Gibbstown, NJ), and VWR (Westchester, PA). CE grade water was purchased from Agilent Technologies (Chicago, IL). A multi-anion standard solution of 100 parts per million (ppm) of acetate, azide, bromide, carbonate, chlorate, chloride, fluoride, hypophosphite, iodide, nitrate, nitrite, perchlorate, phosphate, phosphite, sulfate, and thiocyanate was prepared from their sodium or potassium salts or the free acid diluted in CE grade water. An internal standard solution of 100 ppm of bromide (1.3 mM), fluoride (5.3 mM), and acetate (1.7 mM) was prepared from their sodium or potassium salts diluted in CE grade water.

The CELixirOA™ 8.2 Dynamic Coating Buffer Kit was purchased from MicroSolv Technology Corporation (Long Branch, NJ). This kit includes CELixirOA™ Reagent A (the polycation initiator) and CELixirOA™ Reagent B (the background electrolyte accelerator, with UV absorber), at pH 8.2.

Instrumentation

All CE experiments were conducted using an Agilent Technologies Capillary Electrophoresis System (Waldbronn, Germany), equipped with a diode array UV detector. In this experiment, absorbance was monitored at 233 nm; however, absorbance data were collected between 190 and 600 nm.

Separations were carried out on fused-silica capillaries with an inside diameter (ID) of 50 µm. A 10 m spool of 50 µm ID fused-silica capillary was purchased from Polymicro Technologies (Phoenix, AZ) and cut to the desired length. The polyamide capillary coating was burned from the ends of the capillary and the detection window using a butane flame.

Sample Preparation

The hypophosphorous acid-iodide methamphetamine manufacturing experiment was conducted using previously described conditions (25). Samples of the highly acidic reaction mixture were taken periodically throughout the reaction and were prepared for CE analysis by diluting one drop of reaction mixture into 1 mL of CE grade water, followed by dilution of one drop of this solution into 0.4 mL of internal standards solution, described above.

Run Conditions

Optimized run conditions for the CELixirOA™ 8.2 method are detailed in Table 1. In optimizing the CELixirOA™ 8.2 method, temperature was varied between 15 and 30°C in 2.5°C intervals, column lengths of 64.5, 80.5, 101, and 112.5 cm were examined, and run voltage was evaluated at 30 and 20 kV, negative polarity. A variety of column conditioning approaches were investigated, including flushing the column with water, sodium hydroxide, hydrochloric acid, hydrobromic acid, and/or acetic acid.

Results and Discussion

Use of the modified CELixirOA™ 8.2 dynamic coating system (outlined in Table 1) allows the resolution of species of interest in the forensic evaluation of clandestine laboratory and explosive evidence, including acetate, azide, bromide, carbonate, chlorate, chloride, fluoride, hypophosphite, iodide, nitrate, nitrite, perchlorate, phosphate, phosphite, sulfate, and thiocyanate ions (Fig. 1). While resolved at 100 ppm, several of the species, in particular chloride and iodide, migrate close enough that higher concentrations of one ion could obscure the detection of neighboring ions. The distinction of chloride and iodide is enhanced by their different UV characteristics at the monitoring wavelength.

In addition to the anions shown in Fig. 1, migration of chromate, cyanide, dichromate, permanganate, and sulfite was evaluated. Dichromate converts to chromate at pH 8.2, with chromate exhibiting a sharp, resolved peak migrating shortly after sulfate. Cyanide and carbonate comigrate under these conditions. Sulfite is apparently moderately unstable under the run conditions and gives two sharp peaks, one for sulfate and a second peak (presumably sulfite) that migrates between fluoride and phosphite. Permanganate converts to manganese dioxide at pH 8.2 and is not observed.

TABLE 1—Recommended instrument conditions.

Position*	Buffer	Purpose
<i>Vial summary</i>		
Vial 1	0.1 N HCl or 0.1 N HBr	Preconditioning
Vial 2	CE grade water	Preconditioning
Vial 3	CE grade water	Preconditioning
Vial 4	CElixerOA 8.2 Solution A—Initiator	Preconditioning
Vial 5	CElixerOA 8.2 Solution B—Accelerator	Preconditioning
Vial 6	CElixerOA 8.2 Solution B—Accelerator	Run buffer
Vial 7	CElixerOA 8.2 Solution B—Accelerator	Run buffer
Vial 8	CE grade water	Flush waste
Vial 9	CE grade water	Stacking injection
Inlet (Vial #)	Outlet (Vial #)	Time (sec) [†]
<i>Column preconditioning flushes</i>		
HCl or HBr (1)	Waste (8)	30
Water (2)	Waste (8)	30
Water (3)	Waste (8)	30
Initiator (4)	Waste (8)	90
Accelerator (5)	Waste (8)	90
Initiator (4)	Waste (8)	90
Accelerator (5)	Waste (8)	120
<i>Instrument Conditions</i>		
Cassette temperature	15°C	
Capillary	50 μM inside diameter fused silica capillary, 80.5 cm actual length, 72 cm effective length [‡]	
Stacking injection	50 mbar, 2 sec, sample vial to run buffer (7) 10 mbar, 2 sec, water (9) to run buffer (7)	
Run buffers	Inlet vial (6), outlet vial (7)	
Run voltage	30 kV, negative polarity	
Run time	11 min (approximately 22 min including preconditioning)	
Detection	Monitor at 233 nm, 20 nm bandwidth, no reference [§]	

*Vial positions are included for subsequent reference, although the required buffers could be placed in any location so long as the method is adjusted accordingly.

[†]The instrument's standard flush pressure is used and will displace the volume of the capillary from the injection vial to the detector in approximately 90 sec.

[‡]This column dimension is commercially available or can be cut from a spool of capillary at a reduced cost.

[§]Other wavelengths may be useful for the detection of absorbing species, so it is recommended that the entire spectrum be saved.

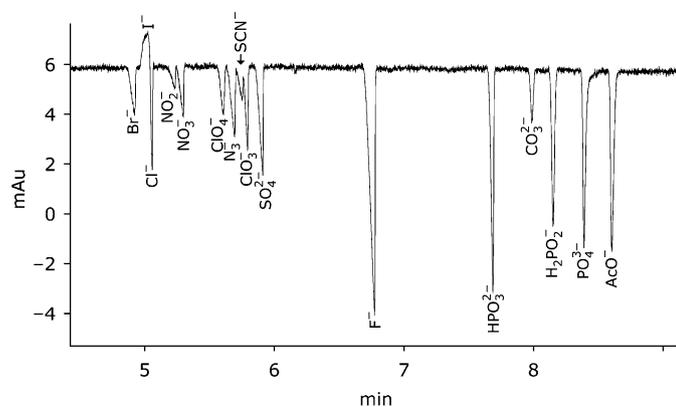


FIG. 1—Separation of a standard mixture of anions at 100 parts per million using the conditions outlined in Table 1. The capillary electropherogram (monitored at 233 nm) between 4.5 and 9 min is displayed.

All CE runs were carried out with UV detection at 233 nm, an absorbance maximum of the buffer's background electrolyte. At this wavelength all ions, except iodide, are observed as negative peaks resulting from displacement of the buffer's absorbing spe-

cies. Iodide is observed as a positive peak owing to its strong UV absorbance at this wavelength. While 233 nm is the optimal wavelength for indirect UV detection with this system, the selection of alternate monitoring wavelengths can be used to optimize the detection of specific species of interest.

Temperature, Column Length, and Run Voltage

Temperature, column length, and run voltage were the first parameters evaluated in optimizing the CElixerOA™ 8.2 method. For these experiments the column was conditioned with polycation initiator for 120 sec followed by the accelerator for 240 sec before each run. Lowering the temperature from 25 to 15°C improved the resolution of the halogen species, in particular iodide and chloride. Initial experiments on the effect of column length showed that increasing the column length from 64.5 to 80.5 cm improved separation of closely migrating species in the region between iodide and sulfate, in particular azide and thiocyanate. No further improvement was noted upon increasing column length to 101 or 112.5 cm. Subsequent experiments suggest that a 64.5 cm column may be sufficient for some applications provided the outlined reconditioning procedures are used. Phosphate, phosphite, and hypophosphite were all sufficiently separated on each of the column lengths evaluated. Reducing run voltage from 30 to 20 kV did not improve resolution of the species of interest.

Phosphate Peak Shape Effects

Using only flushes with the polycation initiator and the accelerator between runs, phosphate peak shape showed significant broadening and tailing as the number of injections on the column increased, with noticeable deterioration after five injections and significant deterioration after 15 injections (Fig. 2). Deterioration in the peak shape or resolution of other anion species was not

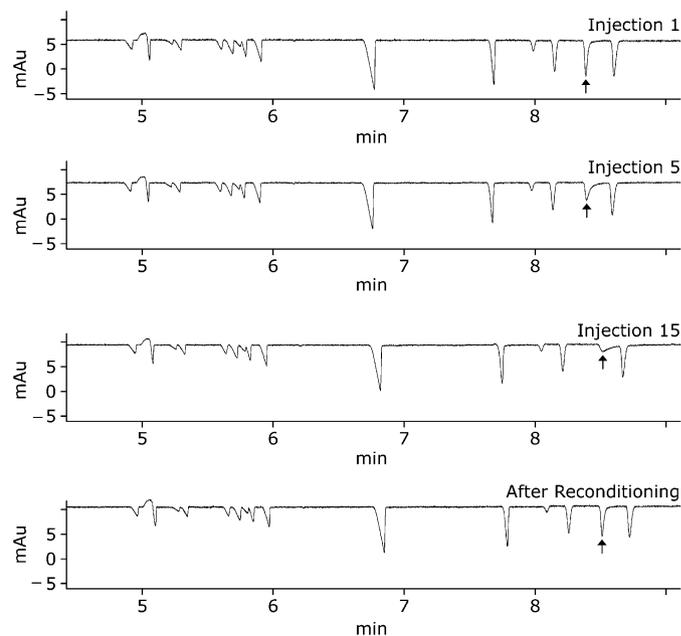


FIG. 2—If the recommended preconditioning steps are not included between each injection, gradual deterioration of phosphate peak (↑) shape is observed. The capillary electropherograms (monitored at 233 nm) between 4.5 and 9 min for the 1st, 5th, and 15th injections from a series of sequential injections of the standard 100 parts per million anion mixture are shown. Peak shape was recovered by reconditioning the column by flushing with 0.1 N hydrochloric acid (180 sec), water (once for 30 sec and once for 180 sec using separate vials to reduce possible carryover of chloride ions), the polycation initiator (120 sec), and the accelerator (240 sec).

observed with repeated injections. It is hypothesized that this deterioration was the result of the progressive adsorption of the triply charged phosphate ion to cationic sites.

Phosphate peak shape was not improved by the use of fresh buffer, flushing the column with either water or hydroxide (recommended by the manufacturer), or by removing the first 5 cm of the column. However, peak shape was recovered by conditioning the column using flushes of 0.1 N hydrochloric acid (180 sec), water (once for 30 sec and once for 180 sec using separate vials to reduce possible carryover of chloride ions), the polycation initiator (120 sec), and the accelerator (240 sec). 0.1 N hydrobromic acid or 1 N acetic acid can be substituted for the hydrochloric acid. The acid flush is necessary to remove the cationic polymer from the column surface (Bill Ciccone—MicroSolv Technology, personal communication, June 2004), which is not accomplished by flushing with hydroxide, water, or buffer.

These data support the hypothesis that phosphate adsorption to cationic sites is responsible for the deteriorating peak shape. It further suggests that this effect is not localized in the injection end of the capillary, since removal of a portion of the injection end of the capillary did not recover the phosphate peak shape.

Recommended Conditioning

The conditioning steps outlined above can be run periodically to address deteriorating peak shape or included in the preconditioning sequence run before each injection as part of the instrumental method. When included in the run's preconditioning, the flushes used for conditioning the column can be shortened to the times listed in Table 1 without adversely affecting the observed peak shapes and resolution over as many as 30 injections. It is necessary to include two sequential sets of initiator and accelerator flushes prior to each run to achieve reproducible migration times.

Table 1 provides a summary of the recommended instrument conditions including this sequence of flushes as preconditioning steps. This same conditioning sequence is recommended for preparing new capillaries. The hydroxide flushes often used in other methods to activate the fused silica surface prior to use are not recommended.

Limit of Detection

The approximate limits of detection were evaluated by serial dilution of an anion standard mix using the run conditions in Table 1. Using a standard 2 sec injection, ions showed limits of detection (LOD) between 10 and 30 ppm, with LOD being specified as peak height three times greater than signal to noise. Increasing the injection time from 2 to 20 sec increased the LOD by an order of magnitude, as expected. However, longer injection times should not be used on more concentrated samples as these result in peak overloading and loss of resolution.

Migration Time Reproducibility

The percent relative standard deviations (%RSD) of absolute migration times are less than 0.2% over 20 subsequent injections using the method summarized in Table 1 (using the 0.1 N hydrochloric acid flush). The %RSD of absolute migration times increased to 0.5% when evaluated over 30 subsequent injections, because of the gradual lengthening of migration times resulting from buffer depletion. Acetate, bromide, and fluoride were evaluated to determine if the use of one or more as internal standards improved the relative migration time %RSDs for the analytes of interest. Normalization of migration times to fluoride alone was sufficient to drop the %RSD for 30 subsequent injections to under 0.1%. This addresses the

criteria for stable migration times and ease of interpretation outlined above for the analysis of clandestine laboratory evidence.

Limitations

A potential consequence of the recommended prerinse procedure is the observation of persistent chloride when hydrochloric acid is used. If chloride is the anion of interest, 1.0 N acetic acid or 0.1 N hydrobromic acid may be used in place of the 0.1 N hydrochloric acid. Replacing the hydrochloric acid flush with hydrobromic acid resulted in detectable residual bromide ions, but no residual chloride. Replacing the hydrochloric acid flush with acetic acid resulted in the observation of residual acetate ion, and also a slow deterioration of phosphate peak shape, with tailing evident after 20 runs. This gradual deterioration was not observed with either hydrochloric or hydrobromic acid, where lower pHs are likely necessary to completely remove the polycation initiator.

Applications

Red phosphorus and hypophosphorous acid are two of the reducing agents used in the clandestine manufacture of methamphetamine. Each of these species is sequentially oxidized to phosphate in the process of forming hydriodic acid, which performs the reduction of ephedrine or pseudoephedrine to methamphetamine (25–27). Figure 3 outlines the oxidation steps required for conversion of these species to phosphate. As each method follows a separate oxidation pathway, the species observed in samples may help determine which manufacturing method was used in a particular case.

Hypophosphite, phosphite, phosphate, and iodide ions show sufficient solubility and stability for observation using the described CE method. To illustrate the utility of this method in the

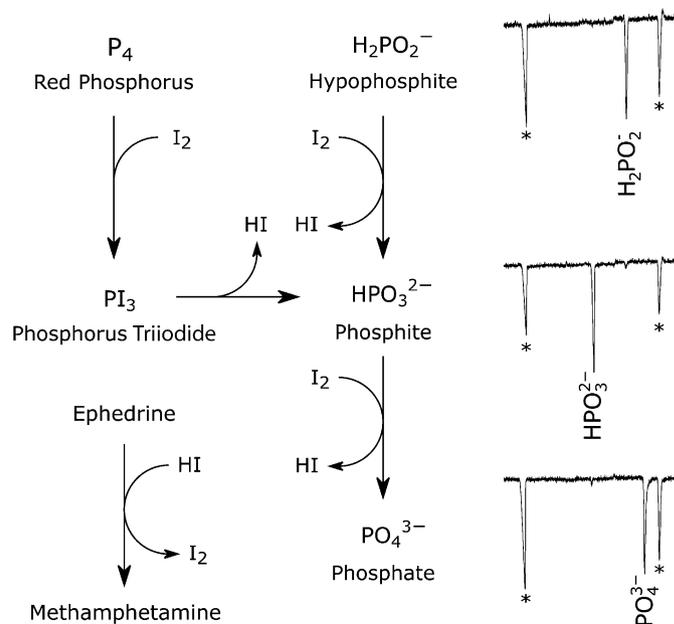


FIG. 3—Red phosphorus and hypophosphite are oxidized to phosphite and then to phosphate in the process of generating hydriodic acid from iodine. In the left-hand side, oxidation reactions for phosphorus species often used in the manufacture of methamphetamine are shown. In the right-hand side, the portions of the capillary electropherograms (monitored at 233 nm) between 6.5 and 9 min for samples taken from a typical hypophosphorous acid cook during the reaction process are shown. This region includes the fluoride and acetate internal standards (*) and each of the three phosphorous anions, but does not include the iodide peak observed in the bottom two samples.

analysis of samples from the clandestine manufacture of methamphetamine, actual samples were taken from a typical hypophosphorous acid-iodine reaction periodically throughout the reaction process, and analyzed using the described method. The initial sample (hypophosphorous acid only) showed only hypophosphite as expected. After all reagents were added and the reaction progressed, the sequential oxidation of hypophosphite to phosphite to phosphate was observed. Figure 3 shows three samples from this experiment that illustrate this conversion. At intermediate time points, more than one phosphorous species is often observed, although all three phosphorous oxyacids were not observed together. Further work will investigate the forensic significance of these ions individually, or as mixtures, in case samples.

This anion method also has other forensic applications. Nitrate, phosphate, and sulfate are common counter ions in ammonium-based fertilizers that are currently being used in the clandestine laboratories to generate liquefied ammonia. In addition, many of the separated anions have application to the evaluation of explosive materials and postblast residues (17,28,29).

Conclusions

The described CE anion analysis method, using a modified CELixerOA™ 8.2 protocol, provides a 22 min (including conditioning) method for the separation of acetate, azide, bromide, carbonate, chlorate, chloride, chromate, fluoride, hypophosphite, iodide, nitrate, nitrite, perchlorate, phosphate, phosphite, sulfate, sulfite, and thiocyanate. This method provides reproducible migration times and good peak shape for all of the referenced species.

These attributes make it suitable for the routine forensic analysis of clandestine laboratory evidence, where inorganic characterization of phosphate, phosphite, and hypophosphite may assist the analyst in distinguishing the various methamphetamine manufacturing methods using phosphorus or one of its oxyacids as reducing agents. The demonstrated separation may have other applications in forensic science, including the analysis of other methamphetamine manufacturing methods, or simply identifying unknown inorganic species.

Acknowledgments

Ira Lurie of the US Drug Enforcement Administration—Special Testing and Research Laboratory (Dulles, VA) provided technical assistance on this project. Robert Heegel, a summer intern, assisted in early work on the column reconditioning process while working on a methamphetamine manufacturing project. Martin McDermot of the Washington State Patrol Crime Laboratory (Seattle, WA) assisted in the review of this manuscript. This project was supported by Grant Number 2003-LT-BX-K004 awarded by the National Institute of Justice, Office of Justice Programs, US Department of Justice. Points of view in this document are those of the authors and do not necessarily represent the official position or policies of the US Department of Justice.

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