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Journal of Chromatography A, 870 (2000) 53–68

JOURNAL OF CHROMATOGRAPHY A

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Profiling of impurities in illicit methamphetamine by high-performance liquid chromatography and capillary electrochromatography

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Abstract

High performance liquid chromatography (HPLC) with photodiode array (PDA) UV and fluorescence (FL) detection, and capillary electrochromatography (CEC) with laser-induced fluorescence (LIF) detection were investigated for the analysis of acidic extracts derived from illicit methamphetamine. These compounds include major impurities from the hydriodic acid/red phosphorous reduction method, i.e., 1,3-dimethyl-2-phenylnaphthalene and 1-benzyl-3-methylnaphthalene, and other trace-level, structurally related impurities. For certain of these solutes, HPLC with conventional FL detection gave at least a 60× increase in sensitivity over UV detection. In addition, other highly fluorescent impurities were detected in methamphetamine produced via four other synthetic routes. The use of a rapid scanning FL detector (with acquisition of ''on the fly'' excitation or emission) provided structural information and gave ''optimum'' excitation and emission detection wavelengths. CEC with LIF detection using UV laser excitation provided greatly improved chromatography over HPLC, with good detection limits in the low ng/ml range. Both methodologies provide good run-to-run repeatability, and have the capability to distinguish between samples.

Keywords: Forensic analysis; Methamphetamine

1. Introduction

Impurity profiling of illicit drugs such as methamphetamine is important for deriving strategic or tactical intelligence [1]. For ''natural'' drugs (e.g., cocaine or morphine), strategic intelligence involves the determination of geographical origin, whereas for synthetic drugs [e.g., methamphetamine or phencylidine (PCP)], strategic intelligence involves the determination of synthetic origin. Tactical intelligence involves the determination of whether two or more exhibits (whether of common geographical or synthetic origin) came from an identical source, i.e., same batch from the same laboratory. Virtually all illicitly produced drugs contain both organic and inorganic impurities. The analysis of the organic impurities, which are complex mixtures of solutes often present at trace levels, require techniques which offer a high degree of resolution, specificity and sensitivity.

Methamphetamine is usually synthesized using

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0021-9673/00/$ – see front matter
PII: S0021-9673(99)00849-3

either an achiral precursor [e.g., phenyl-2-propanone (P2P)] [2] or via a direct stereospecific reduction of one of its corresponding enantiomeric α-hydroxyphenethylamine precursors [3,4] (i.e., ephedrine or pseudoephedrine). In the USA, the currently most prevalent synthetic method is the hydriodic acid/red phosphorus (HI/Red P) reduction of ephedrine or pseudoephedrine [5]; however, there are four other synthetic routes also in common use, and numerous variants of all routes are observed. These latter routes include the so-called ''dry reduction'' [6] (HI/Red P with very little water), hypophosphorus acid reduction [7], and sodium/ammonia reduction [8] (all of ephedrine or pseudoephedrine), and reduction of phenyl-2-propanone by the mercury–aluminum amalgam reduction.

Impurity profiling of methamphetamine has been previously accomplished using capillary gas chromatography (GC) with either flame ionization detection (FID) [9–12], combined FID with nitrogen–phosphorus detection (NPD) [13] or mass spectrometry (MS) [9,14] on either basic or weakly acidic extracts. Although high-resolution separations are obtained using capillary GC, the analysis of solutes that are thermolabile, nonvolatile and highly polar by GC is problematic. In addition, while relatively sensitive, FID and NPD lack specificity.

High-performance liquid chromatography (HPLC) is also viable for the analysis of methamphetamine impurities, including solutes that can be difficult to analyze using GC. However, only moderate resolution separations are obtainable using HPLC. This limitation can be partially compensated for by using a highly sensitive and selective fluorescence (FL) detection mode. Capillary electrochromatography (CEC), another liquid phase technique, combines capillary electrophoresis (CE) separation efficiency with HPLC selectivity. The utility of CEC has been recently demonstrated for cannabinoid profiling [15] and forensic drug screening [16]. When coupled with a highly sensitive laser-induced fluorescence (LIF) detection method, this high-resolution technique is well suited for methamphetamine profiling. LIF has been previously used with CE for the profiling of illicit heroin [17] and cocaine [18].

In this paper, HPLC using photodiode array (PDA) UV and/or FL detection, and CEC with LIF detection are presented for impurity profiling of methamphetamine.

2. Experimental

2.1. Reagents and analytes

Tris(hydroxymethyl)aminomethane (Tris) and sodium dodecyl sulfate (SDS) were obtained from Sigma (St. Louis, MO, USA), and Life Technologies (Gaithersburg, MD, USA), respectively. HPLC-grade acetonitrile was purchased from Burdick and Jackson (Muskegon, MI, USA) and Aldrich (Milwaukee, WI, USA). Deionized water was obtained using a Millipore Milli-Q purification system (Bedford, MA, USA).

Standards of 1,3-dimethyl-2-phenylnaphthalene (NI 1) and 1-benzyl-3-methylnaphthalene (NI 2) were synthesized as previously reported [19] and their identities confirmed by a comparison of their proton nuclear magnetic resonance and/or mass spectra with published data [3,19]. A sample containing 38% (w/w) NI 1 and 52% (w/w) NI 2 plus related trace impurities (hereafter ''NI'') was synthesized from P2P and isolated from a crude mixture by alumina column; this was used for optimization studies.

Authentic HI/Red P-produced samples of methamphetamine were seized from clandestine laboratories. Samples representing dry reduction, Na/ammonia reduction, hypophosphorus acid reduction and P2P reduction methods for producing methamphetamine hydrochloride were synthesized in this laboratory via procedures typically utilized in illicit laboratories.

The HPLC mobile phases were internally mixed from solvent reservoirs containing acetonitrile and water. The CEC mobile phase was prepared by combining 30 parts of 5.0 m*M* Tris–5 m*M* SDS, pH 8.0, with 70 parts acetonitrile.

2.2. Analytical solutions

For HPLC and CEC analyses, appropriate aliquots of a methylene chloride stock solution of NI which contained 0.39 mg/ml NI 1 and 0.54 mg/ml NI 2 were evaporated to dryness and reconstituted in the mobile phase. This same solution was used for limit of detection and additional experiments.

For isocratic HPLC and CEC, acidic extracts derived from approximately 20 mg equivalents of methamphetamine samples were dissolved in 200 μl

and 100 μl of mobile phase, respectively, while for gradient HPLC analyses, acidic extracts obtained from approximately 50 mg equivalents of methamphetamine were used. The acidic extracts were prepared by dissolving an appropriate quantity of sample in 5.0 ml of sulfuric acid and extracting it three times with 3 ml of methylene chloride. The combined extracts were then dried over sodium sulfate and reconstituted with an appropriate amount of methylene chloride, aliquots of this reconstituted solution were used for all HPLC and CEC analyses.

2.3. Instrumentation

A Hewlett-Packard Model 1100 HPLC system (Waldbronn, Germany) equipped with a quaternary pumping system, a 1100 series PDA–UV detector, a Model 1046A FL detector, and a 1100 series rapid scanning FL detector, was used for all HPLC analyses. The Model 1046A FL detector only allows for single excitation and emission wavelengths at given times, and also only allows acquisition of spectral information in the stop flow mode. In contrast, the 1100 rapid scanning FL detector allows for either multiple excitation or emission wavelengths at any given time, and acquisition of spectral information in the continuous flow mode. A 11.0 cm×4.7 mm I.D. HPLC cartridge system (5 μm Partisil ODS 3) operated at ambient temperature was used (Whatman, Clifton, NJ, USA).

The CEC–LIF experiments were performed using a laboratory-built system. High voltage was provided by a 0–30 kV Bertran Associates Series 225 power supply (Hicksville, NY, USA). For safety purposes, this device was interlocked with the door of a plexiglass enclosure which contained the CEC apparatus. The buffer vials were not pressurized and the column was operated at ambient temperature with no temperature control. Electrokinetic injections were performed manually by placing the column in the sample vial and applying 2 kV for a timed interval. Laser excitation (mechanically chopped at 250 Hz) was provided by a Coherent intracavity-doubled argon ion laser (Santa Clara, CA, USA) operating at 257 nm. A cross-beam configuration was used, i.e., the emission collection axis was perpendicular to that of the excitation. The collected fluorescence was filtered both optically and spatially to remove residual excitation and was detected using a photomultiplier tube (PMT). The signal was processed using a lock-in amplifier in conjunction with the mechanical chopper, and data were stored and processed using EZChrome Elite chromatography software (San Ramon, CA, USA).

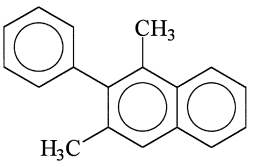
CEC columns were prepared in the laboratory as previously described [20]. For this study, the columns were packed with 1.5 μm nonporous ODS II particles obtained from Micra Scientific (Northbrook, IL, USA). The columns were 32 cm (of which 23.5 cm was packed)×75 μm I.D.×365 μm O.D. Detection windows (~2 mm long) were burned into the column walls, approximately 2 mm downstream of the outlet frit, using a Teledyne Kinetics resistively heated wire stripper (Solana Beach, CA, USA).

Columns were conditioned with degassed mobile phase using a Unimicro Technologies manual syringe pump (Pleasanton, CA, USA). After conditioning, a stable electroosmotic flow was established by slowly increasing the voltage across the column up to the running potential. The current was then monitored by measuring the voltage drop across a 10 kΩ resistor in series with the column.

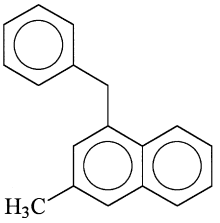
3. Results and discussion

3.1. Impurity profiling by HPLC

NI 1 and NI 2 are the two major impurities derived from acid catalyzed condensation of P2P, and are therefore formed as byproducts during the HI/Red P reduction of ephedrine or pseudoephedrine to methamphetamine [3,19,21] (P2P is formed by dehydration/hydrolysis of ephedrine or pseudoephedrine). As shown in Fig. 1, these compounds both contain the highly fluorescent naphthalene chromophore. GC analysis of a synthetic mixture of NI 1 and NI 2 (synthesized directly from P2P) also revealed the presence of numerous other unidentified, minor compounds. Since these were all derived from P2P, it is rational to also expect the same impurities in illicit methamphetamine. Therefore FL detection was investigated in tandem with reversed-phase HPLC for the analysis of hydrophobic methamphetamine impurities.



1,3-dimethyl-2-phenylnaphthalene (NI 1)



1-benzyl-3-methylnaphthalene (NI 2)

Fig. 1. Structures of the two major napthalene-based impurities derived from HI/Red P reduction method.

3.1.1. FL versus UV detection

In preliminary studies, stop flow analysis of the eluting peaks using FL detection (Model 1046A detector) gave excitation maxima of 229 nm and 228 nm and emission maxima of 343 nm and 340 nm, respectively, for NI 1 and NI 2. As shown in Fig. 2, similar spectral information was obtained using the rapid scanning FL detector.

For NI 1, the limit of detection via UV was 3.9 ng/ml ($S/N=3$, UV 228 nm), while for fluorescence a limit of detection of 165 pg/ml [$S/N=3$, excitation 229 nm and emission 340 nm (rapid scanning FL detector)] was obtained. For NI 2, the limit of detection via UV was 2.8 ng/ml ($S/N=3$, UV 228 nm), while for fluorescence a limit of detection of 460 pg/ml [$S/N=3$, excitation 229 nm and emission 340 nm (rapid scanning FL detector)] was obtained. For fluorescence, the data represents an apparent limit of detection; there were small carryover peaks present for NI 1 and NI 2 which represent the apparent noise level. The rapid scanning FL detector gave approximately a 2× lower limit of detection than the conventional FL detector.

The trace impurities in NI were also analyzed via HPLC with UV and FL detection. As previously noted, it was believed that since trace impurities were also derived from the same synthetic process which produces NI 1 and NI 2 in illicit samples (i.e., acid-catalyzed condensation of P2P), they would also be present in seized exhibits. As shown in Fig. 3, there is a significant enhancement of the FL signal over the UV signal, especially for those solutes eluting after NI 1. For several solutes, there is at least a 60× increase in S/N for FL over UV detection. The increased gain of FL over UV for the minor impurities versus NI 1 and NI 2 is probably due to the lack of carry-over peaks in the measured chromatographic region.

Spectral information obtained for the solutes separated in Fig. 3 strongly indicate that the highly fluorescent, trace-level impurities compounds are (as postulated) structurally similar to NI 1 and NI 2. As shown in Fig. 3A, little or no PDA–UV data is available for most of these solutes; however, contour plots obtained by the rapid scanning detector show that their excitation and emission spectra are similar NI 1 and NI 2 (see Figs. 4 and 5).

3.1.2. Sample analysis

The HPLC analyses of samples seized from three different illicit HI/Red P laboratories (see Fig. 6) gave similar but distinct fluorescent profiles. Although for the most part the same peaks are present (especially in the region after NI 1), there are differences in the absolute and relative intensities of the peaks. Excitation and emission contour plots for these three samples show that most peaks (especially in the region after NI 1) have excitation and emission spectra similar to NI 1 and NI 2. Again, due to the similarities in the fluorescence profiles of the synthetic mixture and the three samples (cf. Figs. 3 and 6), it is probable that most of the highly fluorescent trace impurities in methamphetamine are also naphthalene-containing compounds, and therefore originate from P2P.

Repeatability (RSD, $n=8$) of retention time (t_R), relative retention time (RRT), peak area (Area) and relative peak area (RArea) were examined for several peaks from the sample shown in Fig. 6C. As shown in Table 1, good precision was obtained for peaks 1–4 for t_R, RRT, Area and RArea, with RSDs of $\leq$0.46, $\leq$0.39, $\leq$1.83 and $\leq$2.69%, respectively. Improved precision was obtained for RRT versus t_R using peak 4 as a reference compound.

The analysis of two additional methamphetamine

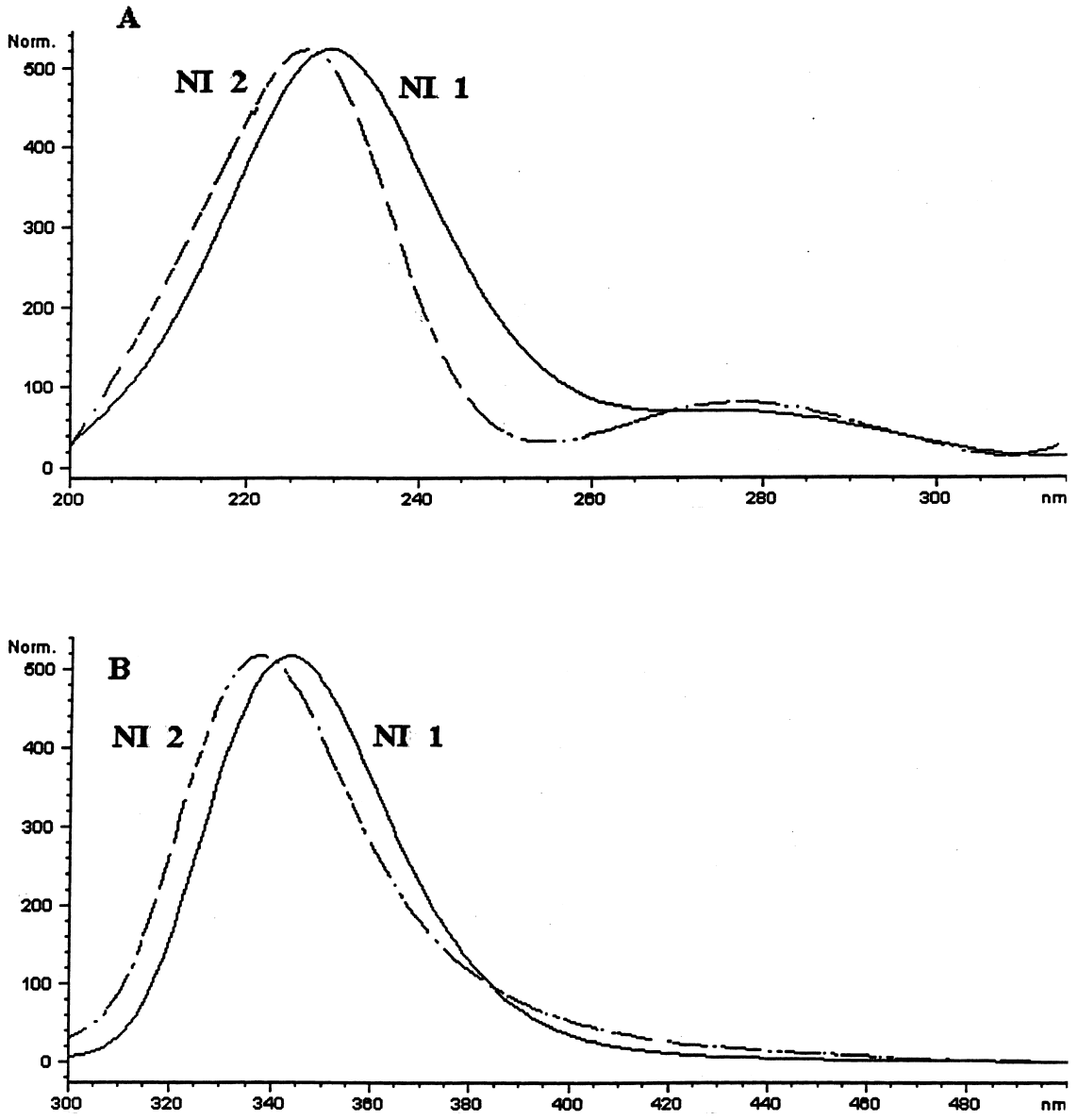


Fig. 2. (A) Excitation spectra and (B) emission spectra of NI 1 and NI 2. Conditions: acetonitrile–water (70:30) at a flow-rate of 1.5 ml/min. A Whatman 5 μm ODS 3 (11.0 cm×4.7 mm I.D.) column was used at ambient temperature with 25 μl injections.

samples synthesized by the dry reduction and the hypophosphorous acid methods, respectively, also gave peaked enriched chromatograms via FL detection. Using the same excitation and emission wavelengths used for the previous samples, fluorescence detection gave considerable increases in sensitivity over UV detection. However, the peak patterns for these latter samples (under the same fluorescence conditions) are considerably different from each other (cf. Figs. 7A and 8A), and are also different from the samples derived from the standard HI/Red P method (cf. Figs. 6, 7A and 8A), and clearly indicate a different class or classes of solutes versus those obtained from the HI/Red P synthesis;

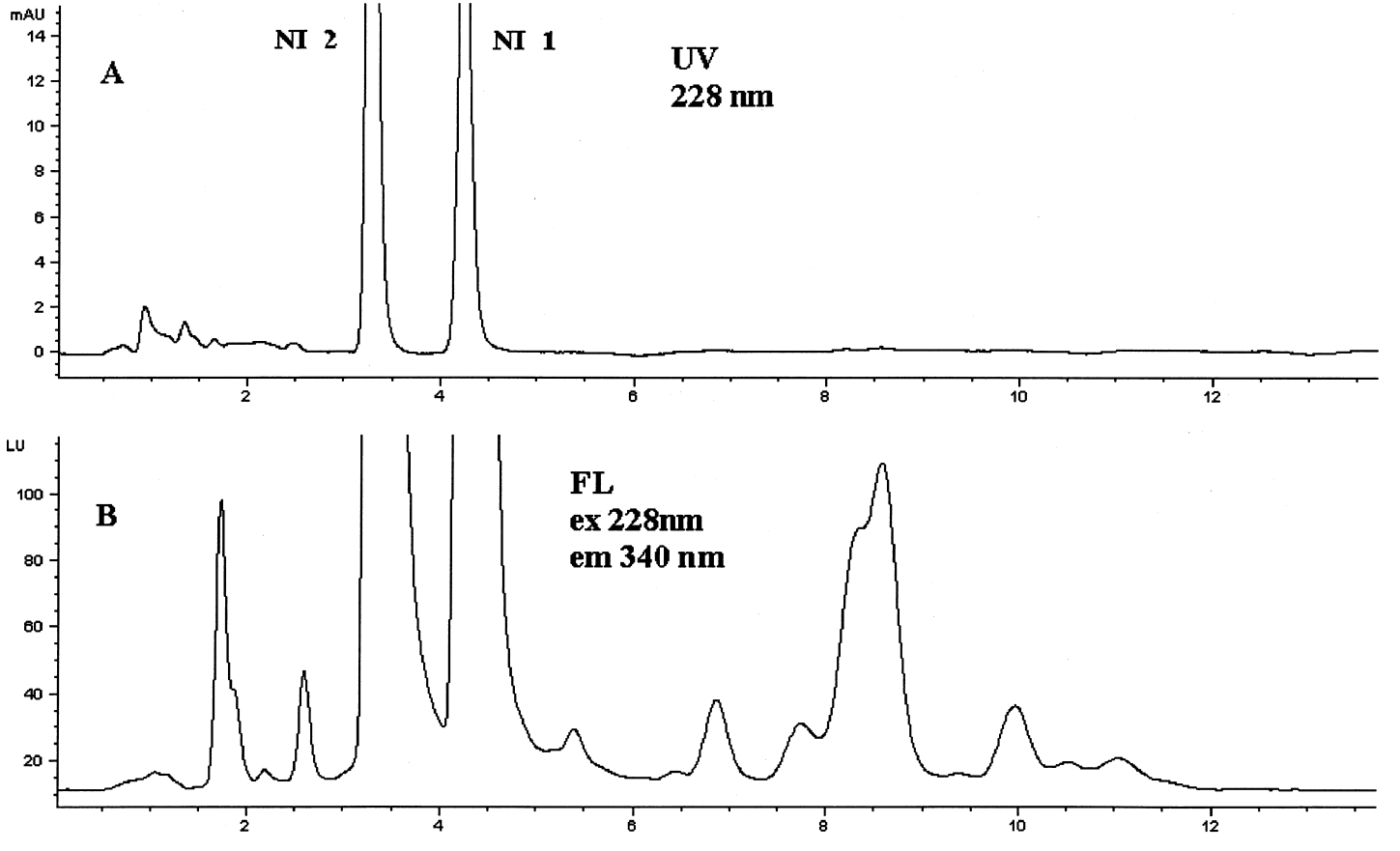


Fig. 3. HPLC with (A) UV detection and (B) FL detection of synthetic mixture containing high levels of NI 1 and NI 2 plus trace impurities. Other conditions identical to Fig. 2. *x*-Axis: Time in min. LU=luminescence units.

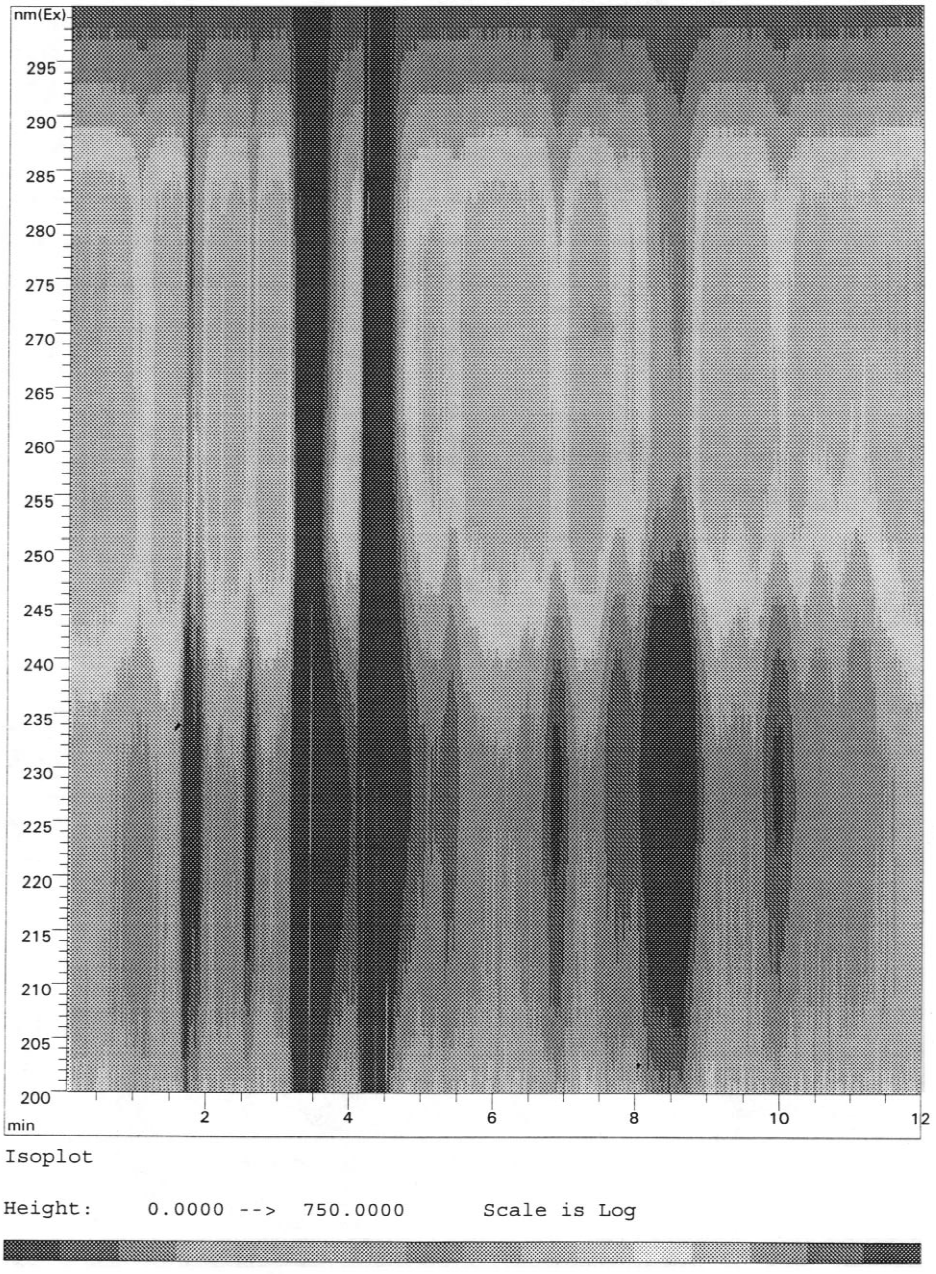


Fig. 4. Contour plot (isoplot) of excitation spectra of peaks contained in Fig. 3.

both have a preponderance of compounds with excitation maxima between 235 and 290 nm and emission maxima between 358 and 390 nm. For the sample derived from the hypophosphorus acid method, the peaks at 8.5 and 8.8 min may arise from the same compounds (i.e., at similar migration times and FL spectra) present in the three samples synthesized by the HI/Red P method.

The contour plots help in obtaining ''optimized'' fluorescence conditions. For the hypophosphorus

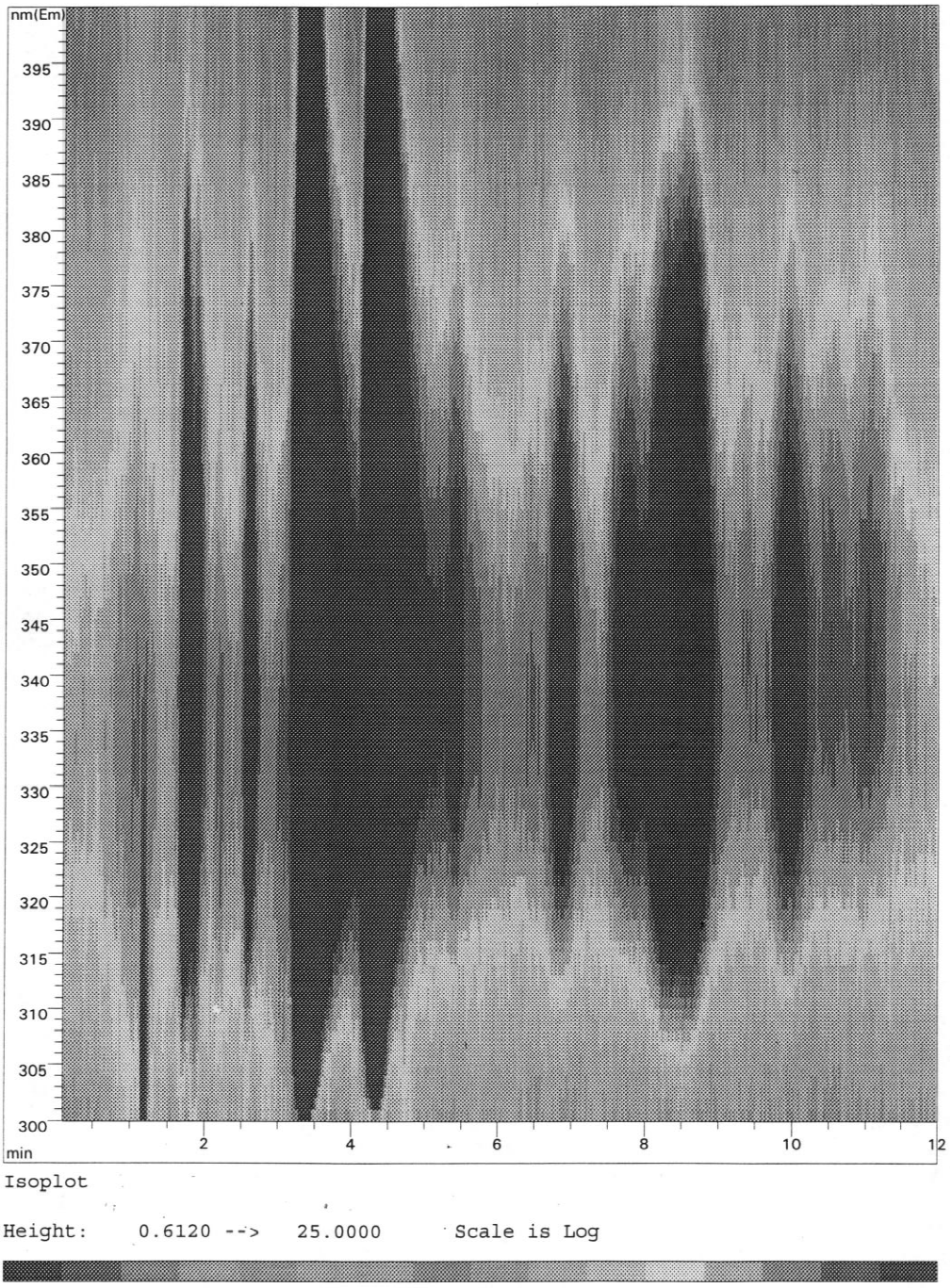


Fig. 5. Contour plot (isoplot) of emission spectra of peaks contained in Fig. 3.

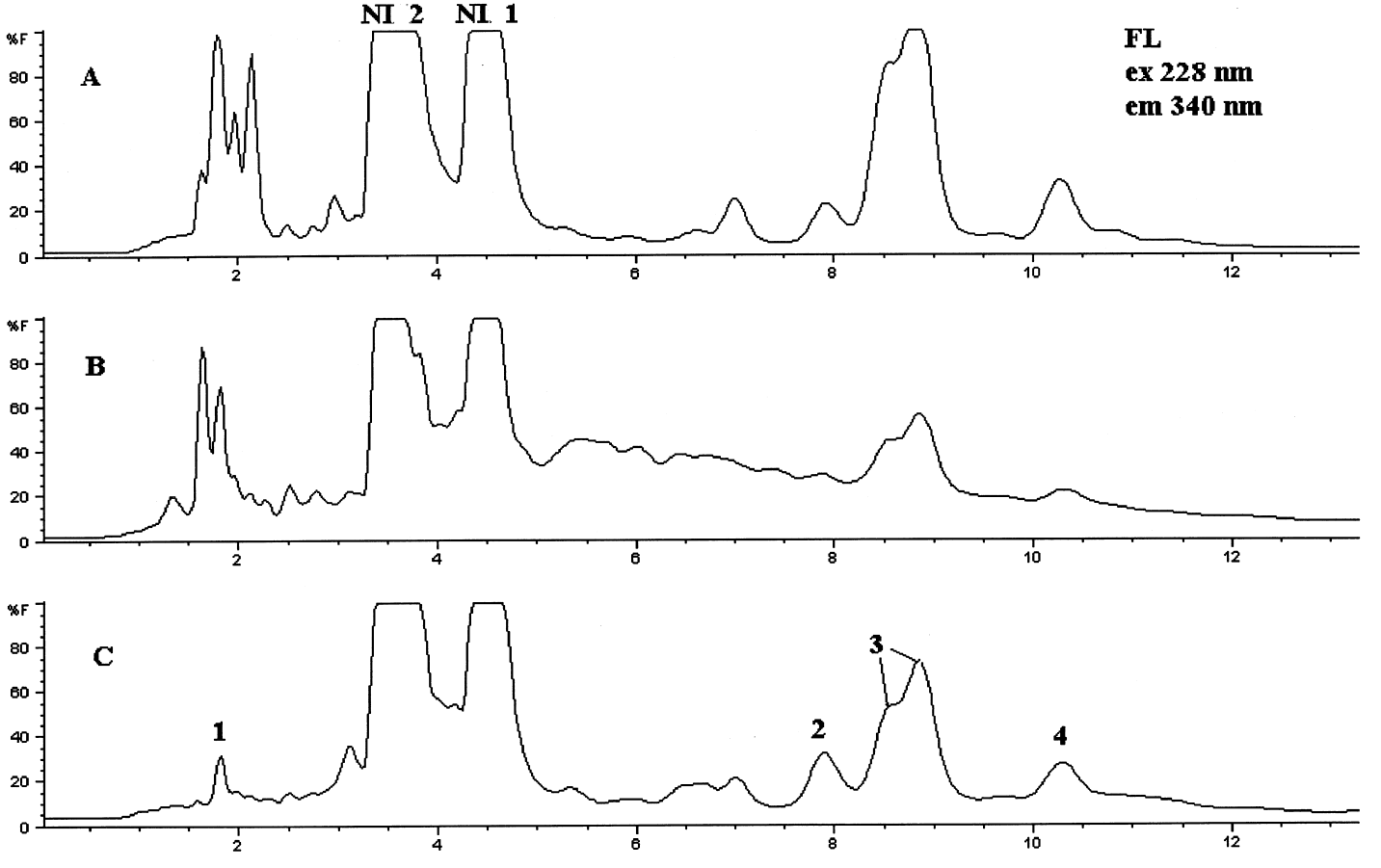


Fig. 6. HPLC with FL detection of three samples seized from different HI/Red P laboratories. Conditions identical to Fig. 2. Precision data for peaks 1–4 are shown in Table 1. *x*-Axis: Time in min. F=fluorescence.

Table 1
HPLC repeatability (RSD, $n=8$) of retention time (t_R), relative retention time (RRT), peak area (Area) and relative peak area (RArea) for selected peaks (see Experimental for conditions)

Peak	RSD (%)			
	t_R	RRT	Area	RArea
1	0.46	0.39	1.83	1.48
2	0.24	0.06	1.36	2.69
3	0.26	0.06	1.07	1.80
4	0.27		1.32	

acid sample, better overall signal-to-noise for the various peaks is obtained, with excitation at 228 nm and emission at 370 nm, versus excitation at 228 nm and emission at 340 nm (cf. Fig. 7A and B). In the case of the sample synthesized by the dry reduction method, changing both the excitation and/or emission wavelength(s) gives better signal-to-noise for certain peaks (cf. Fig. 8A–C). Again, the use of excitation and emission contour plots help to obtain ''optimized'' conditions. The rapid scanning FL detector was able to simultaneously acquire the chromatograms depicted in Fig. 8A and B. In fact, this detector can simultaneously acquire up to four corresponding excitation or emission wavelengths for a given excitation or emission wavelength. As was previously shown (and will be further illustrated), this feature can be very useful for impurity profiling.

Two methamphetamine exhibits synthesized by the Na/ammonia reduction method and a P2P synthetic route, respectively, were also analyzed using the ''optimum'' fluorescence conditions obtained from the contour plots (see Fig. 9A and B). Unlike the previous samples, peak enriched chromatograms were not obtained. For methamphetamine produced by the Na/ammonia reduction method, a major peak is obtained at 5.8 min (see Fig. 9A), while for the sample synthesized from P2P, three major peaks were obtained at 2.2, 3.0 and 3.6 min, respectively (see Fig. 9B). Again, FL detection for certain impurities offered significantly increased sensitivity over UV detection.

For most of the samples analyzed (see Figs. 6–9), there is a need to improve overall peak resolution. Lowering solvent strength, employing gradient elution, increasing column length, decreasing particle size, or combinations of most of the above are possible approaches. For samples derived from the HI/Red P process, lowering the solvent strength (by decreasing from 70 to 60% acetonitrile) improved resolution of early and mid-eluting compounds, but had a negligible effect on the resolution of the later-eluting solutes. For these same samples, gradient elution improved resolution, but again only for the early eluting solutes (see Fig. 10B). However, both of these approaches resulted in significant increases in run times (2.5–3.4×). In addition, a more concentrated extract and larger injection size was used for gradient analysis than for isocratic analysis. This allowed a peak enriched chromatogram using PDA–UV detection at 210 nm (e.g., see Fig. 10A). The UV contour plot revealed that the complex mixture had a wide diversity of spectral properties. A discussion of the UV spectra of these methamphetamine impurities is beyond the scope of this manuscript; however, Fig. 10A reveals the potential of PDA–UV detection for impurity profiling. Concentrated extracts of all three exhibits derived from the HI/Red P process had distinctly different gradient elution chromatographic profiles. For these same samples, there were no defining features of the PDA–UV contour plots (in contrast to the FL plots). The increased sensitivity and/or increased selectivity that FL detection offers over PDA–UV detection is illustrated in Fig. 10.

3.2. Impurity profiling by CEC

For CEC, greatly improved separations versus HPLC were obtained for both NI and a seized exhibit produced in an illicit HI/Red P laboratory (cf. Figs. 3B, 6C, 11A and B). This arose primarily because CEC allowed the use of a 23.5 cm long capillary packed with 1.5 μm reversed-phase packing material. Due to pressure limitations when using HPLC, increasing column length and/or decreasing particle size will only give a small increase in plate counts and therefore only marginal increases in resolution. In contrast, the lack of a pressure drop in CEC allows the use of smaller particles sizes and longer columns than are possible by HPLC. In combination with the higher efficiencies obtainable with electroosmotic flow versus hydrodynamic flow, this allows for much higher peak efficiencies using CEC. For the late eluting compounds, the plate count was approxi-

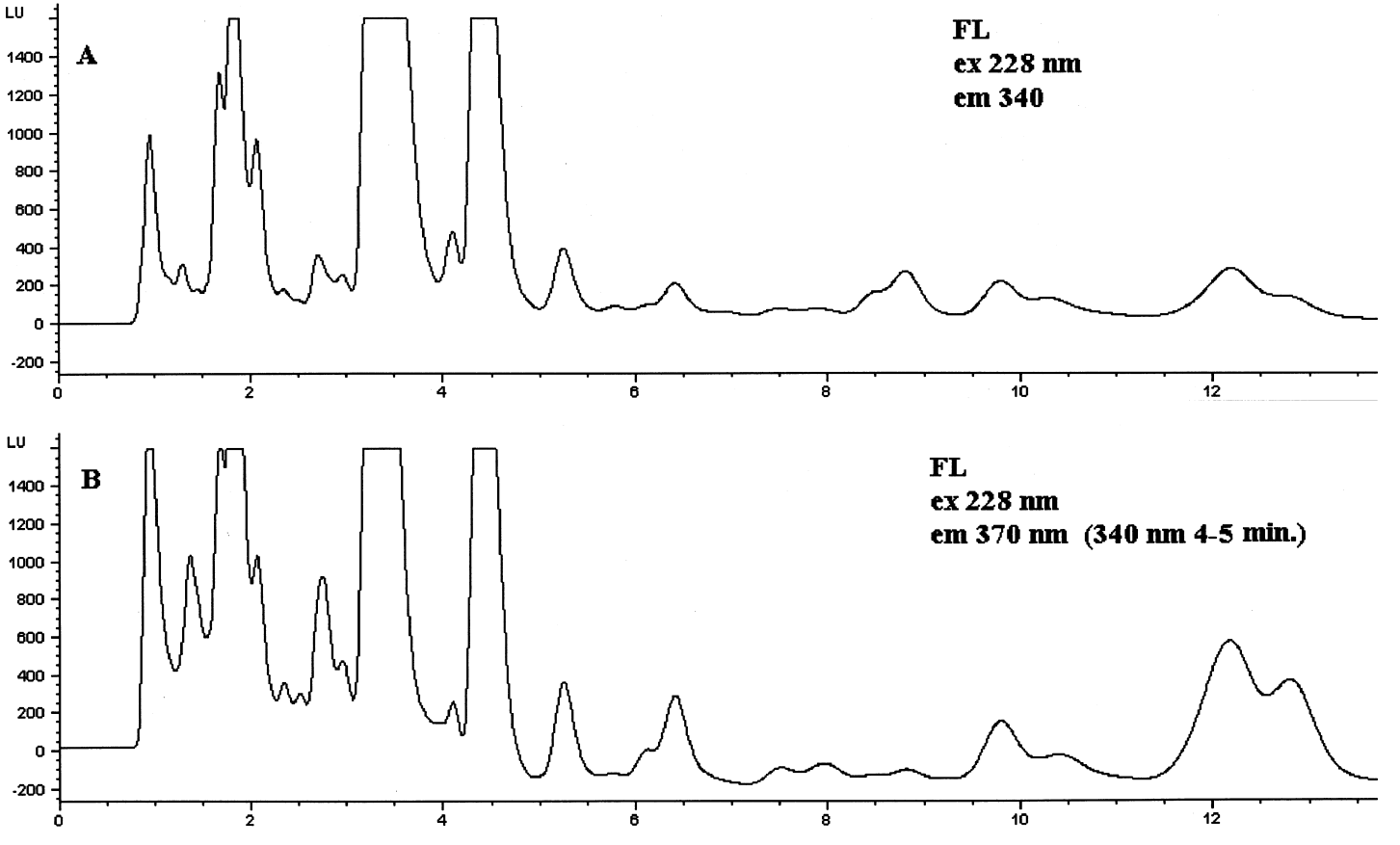


Fig. 7. HPLC with multiple FL detection (two separate runs) of sample synthesized by the hypophosphorus acid method. Other conditions identical to Fig. 2. *x*-Axis: Time in min.

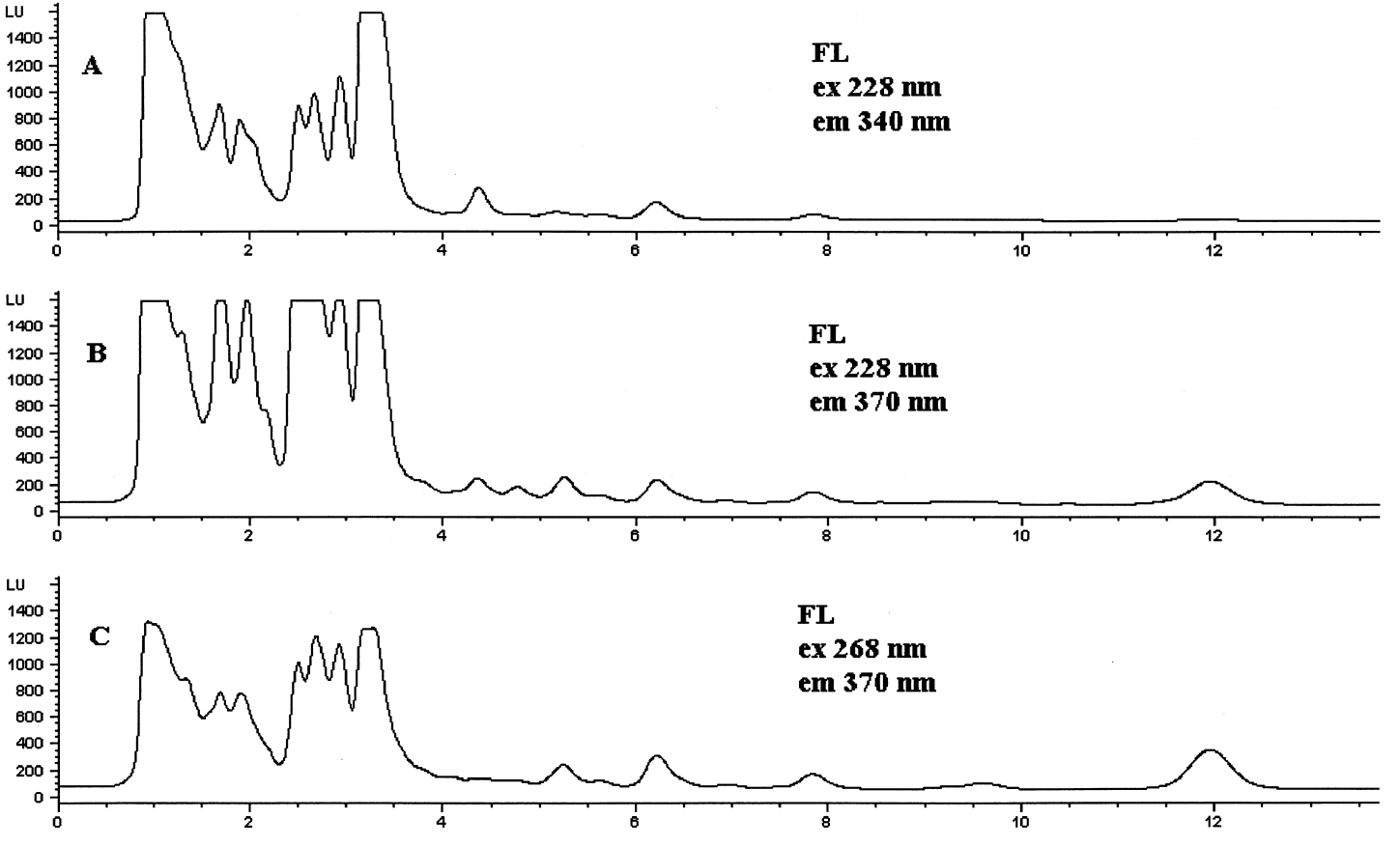


Fig. 8. HPLC with multiple FL detection (two separate runs) of sample synthesized by the dry reduction method. Other conditions identical to Fig. 2. *x*-Axis: Time in min.

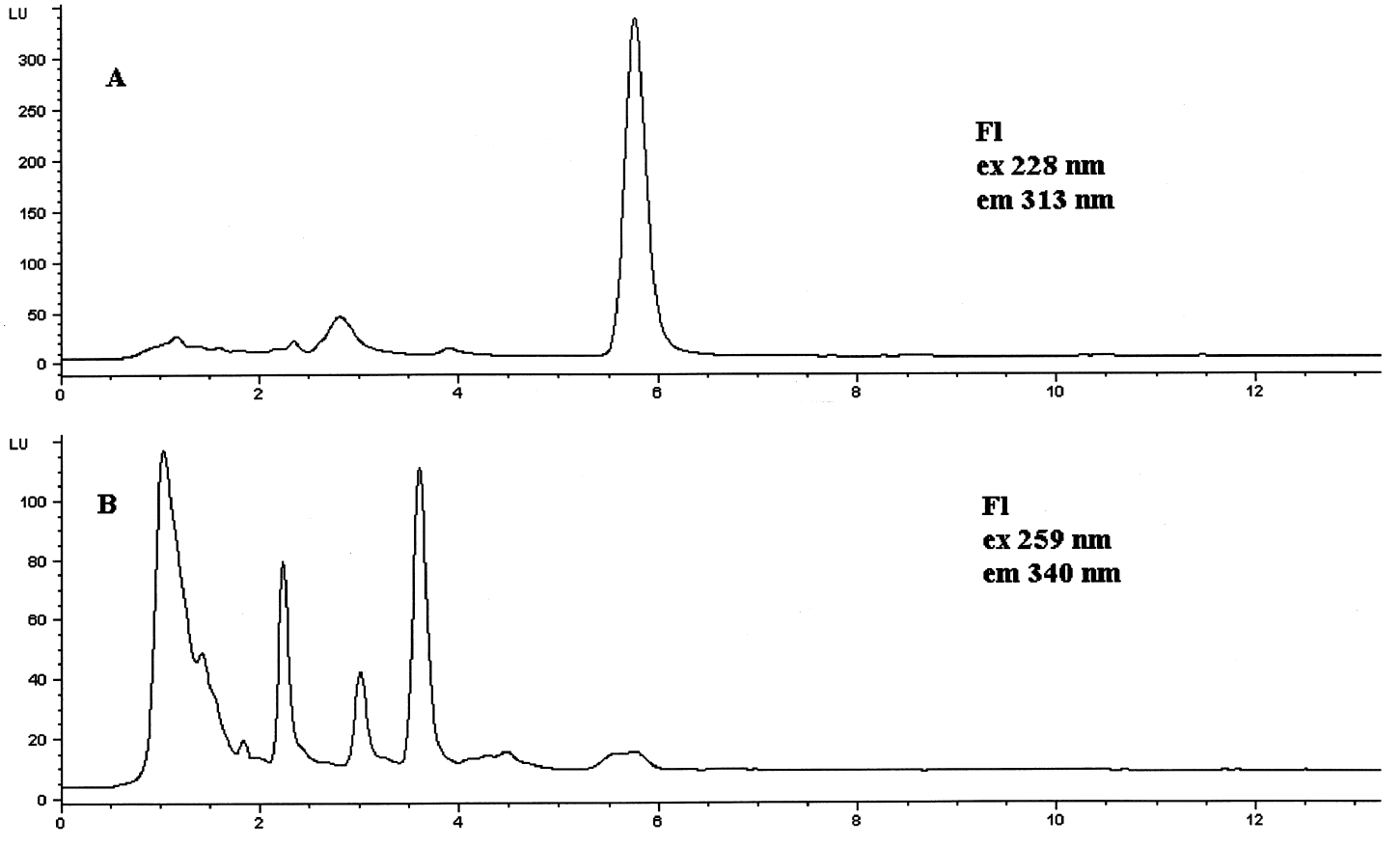


Fig. 9. HPLC with FL detection of samples synthesized by (A) Na/ammonia reduction method and (B) P2P method. Other conditions identical to Fig. 2. *x*-Axis: Time in min.

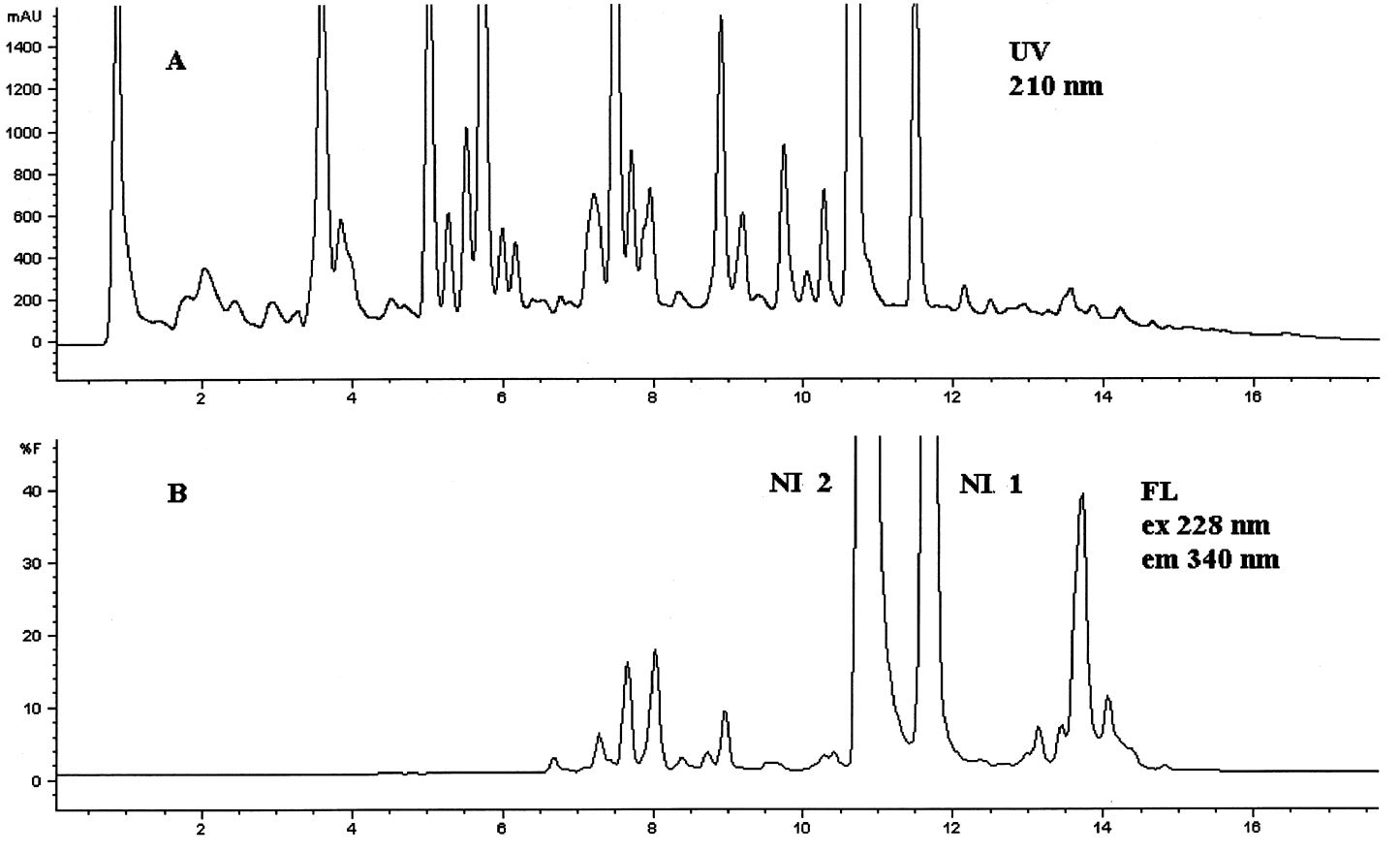


Fig. 10. HPLC gradient with (A) UV detection and (B) FL detection of sample seized from HI/Red P laboratory (same sample as Fig. 6A except 2.5× more concentrated). Initial conditions: acetonitrile–water (30:70) with 15 min linear ramp. Final conditions: pure acetonitrile with 5-min hold. A Whatman 5 μm ODS 3 (11.0 cm×4.7 mm I.D.) column was used at ambient temperature with a 100 μl injection and a flow-rate of 1.5 ml/min. *x*-Axis: Time in min.

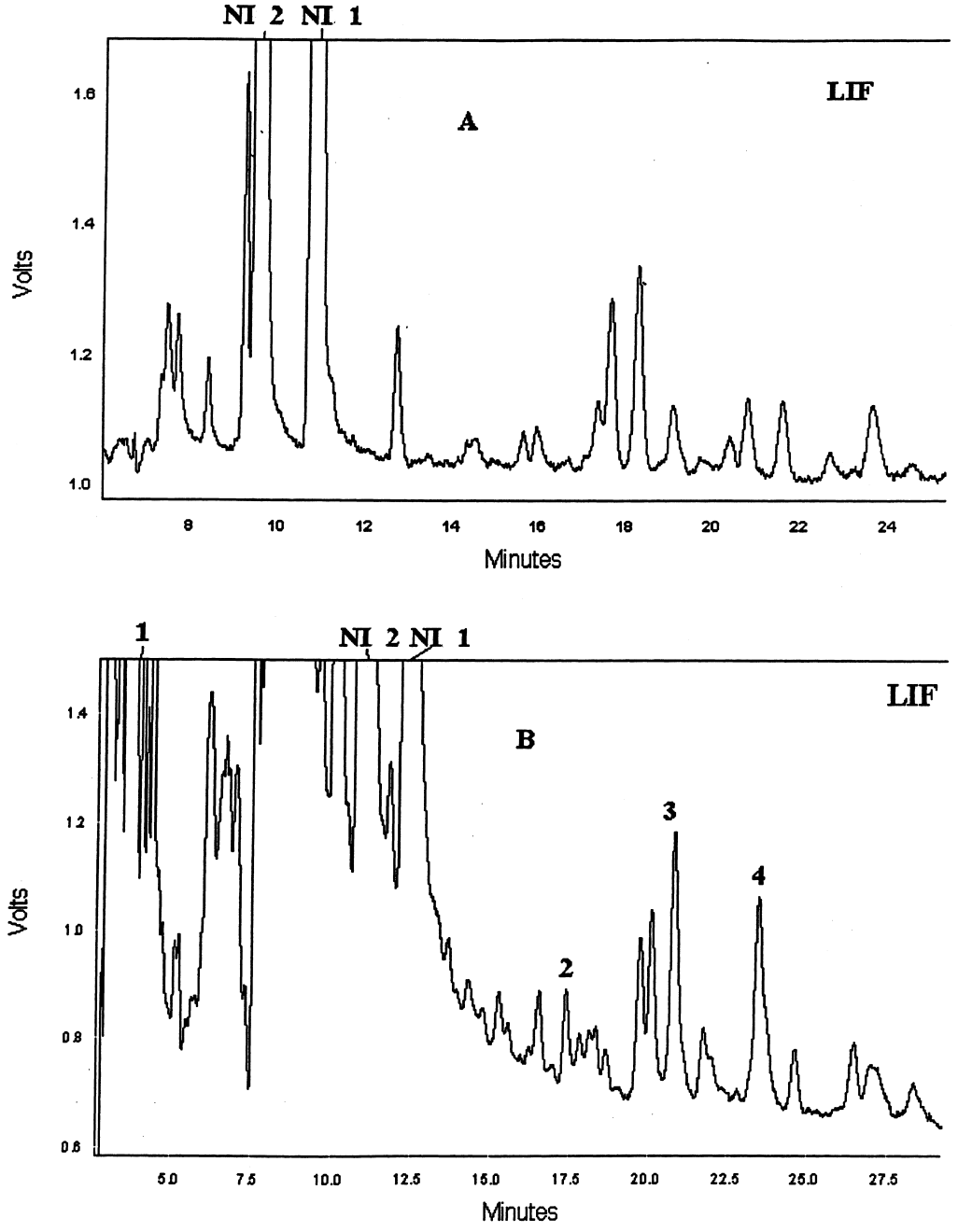


Fig. 11. CEC with LIF detection of (A) synthetic mixture containing high levels of NI 1 and NI 2 plus trace impurities and (B) sample seized from HI/Red P laboratory (same sample as Fig. 6C). Conditions: acetonitrile–5.0 m*M* Tris buffer with 5 m*M* SDS (pH 8.0) (70:30). A 75 μm capillary packed with 1.5 μm nonporous ODS II particles (total length of 32 cm, of which 23.5 cm were packed) was used at ambient temperature. Electrokinetic injections of 8.0 s at 2.0 kV were used. Precision data for peaks 1–4 are shown in Table 2.

mately 200 000/m (compared to approximately 40 000/m via HPLC). As expected, CEC analysis of three samples analyzed by HPLC (HI/Red P method) gave distinctly different FL profiles. The differentiation of these samples is facilitated by the significantly greater number of points of comparison which CEC generates.

In spite of excitation significantly removed from the maxima of the naphthalene-like impurities, LIF detection at 257 nm still gave good limits of detection: $S/N=3$ for NI 1 and NI 2 were 8.7 and 10.8 ng/ml, respectively.

Repeatability (RSD, $n=8$) of t_R, RRT, Area and RArea were examined for several peaks from the sample shown in Fig. 11B. As shown in Table 2, good precision values were obtained for peaks 1–4 for t_R and RRT, with RSDs of $\leq$0.66 and $\leq$0.38%, respectively. By using peak 4 as a reference, improved precision was obtained for RRT versus t_R. As also shown in Table 2, poor precision was obtained for Area (RSD$\geq$14.6%), however, satisfactory precision was obtained for RArea (RSD$\leq$4.63%). The use of a manual injection system probably contributes to the poor area precision; however, automated injection systems for CEC can also give poor results; therefore, the use of corrected areas with an appropriate reference compound is desirable.

SDS was added to the mobile phase in order to inhibit bubble formation [22]. However, despite operating at a relatively low voltage (10 kV) and using this mobile phase additive with a low concentration of a relatively nonconductive buffer (current 2 μA), there was still a persistent problem with bubble formation. The presence of bubbles, which lead to reduced flows or column failure, would be greatly reduced by pressurizing the inlet and outlet vials. In addition, such a system would also allow increased voltages and reductions in analysis time.

Table 2
CEC repeatability (RSD, $n=8$) of retention time (t_R), relative retention time (RRT), peak area (Area) and relative peak area (RArea) for selected peaks (see Experimental for conditions)

Peak	RSD (%)			
	t_R	RRT	Area	RArea
1	0.31	0.38	14.6	4.48
2	0.59	0.06	19.8	4.63
3	0.66	0.06	18.3	4.42
4	0.63		15.0	

4. Conclusions

The utility of HPLC and CEC for impurity profiling of illicit methamphetamine was demonstrated. For HPLC, FL offered significantly lower limits of detection versus UV. The use of contour plots obtained by a scanning FL detector was extremely useful for identification and analysis. CEC afforded greatly improved chromatography compared to HPLC with good detection limits using LIF.

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