Application of CE-ESI-MS in forensic toxicology: Identification of piperazine-derived designer drugs in Ecstasy tablets and of food colorants in illicit drugs

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Abstract

In this work CE-ESI-MS procedures were developed for the separation and identification of piperazine-derived designer drugs in Ecstasy tablets as well as in so-called "smart drugs" and of food colorants in illicit drugs for batch-to-batch comparison.

Piperazine derivatives, mainly the isomers of 1-chlorophenylpiperazine (especially the 1,3-isomer, *m*-CPP), 1-benzylpiperazine (BzP) and 1-[3-trifluoromethylphenyl]-piperazine (TFMPP), increasingly appear on the illicit drug market as active substances in Ecstasy tablets and "smart drugs". A CE-ESI-MS/MS procedure with a run buffer consisting of 100 mmol/L formic acid at pH 2.4 and 10% (v/v) 2-propanol in a 75 µm i.d. fused-silica capillary of 82 cm length was employed for the separation and identification of five piperazines (MS conditions: dry gas flow: 4 L/min at 250°C, nebulizer gas pressure: 4 psi, sheath liquid: 2-propanol/water (50/50, v/v) at 3 µL/min). Baseline separation was achieved within 13 minutes using a high voltage of +25 kV. A second tailor-made procedure for the baseline separation of the three positional isomers of 1-chlorophenylpiperazine (*o*-CPP, *m*-CPP and *p*-CPP) at +28 kV was developed by adding 10 mmol/L 2-hydroxypropyl-beta-cyclodextrin to the separation buffer.

The ratio of food colorants present in many Ecstasy tablets and heroin samples can point to links between different seizures. A CE-MS procedure was developed for the trace analysis of sulpho-group containing azo- and triarylmethane-type food colorants and applied to Ecstasy tablets as well as heroin samples. Extremely high selectivity was achieved by employing a low pH run buffer (200 mmol/L formic acid) in a counter-electro-osmotic separation mode (-25 kV) and with negative ion ESI-MS detection for the even at pH 2.2 negatively charged colorants.

1. Introduction

Capillary electrophoresis coupled to mass spectrometry with electrospray ionisation as a soft ionisation technique combines extremely efficient separation and sensitive detection. State-of-the-art ion-trap mass spectrometers with high-capacity traps successfully cope with the demands of minute sample amounts handled by CE and deliver structural information via fragmentation in auto-MS/MS and auto-MS^3 modes. Therefore, CE-ESI-MS/MS is a powerful technique for the separation of complex mixtures and court-proof identification of polar and thermolabile substances, especially in bio-analysis and forensic toxicology [1-3, 8, 12]. In this work two novel applications of illicit drug analysis by CE-ESI-MS^n are presented.

2. Experimental

2.1 Chemicals

1-Benzylpiperazine (BzP), hydrochloride salts of 1-[2-methoxyphenyl]-piperazine (*o*-MeOPP), 1-[2-chlorophenyl]-piperazine (*o*-CPP), 1-[3-chlorophenyl]-piperazine (*m*-CPP), 1-[4-chlorophenyl]-piperazine (*p*-CPP) and 1-[3-trifluoromethylphenyl]-piperazine (TFMPP) were purchased from Alfa Aesar (Karlsruhe, D) and N-[3,4-methylenedioxybenzyl]-piperazine (3,4-MDBzP) from Sigma-Aldrich Chemie (Steinheim, D). 2-Hydroxypropyl-beta-cyclodextrin, formic acid solution (1 mol/L in water, LC/MS-grade) and benzylamine were from Fluka (Taufkirchen, D). Water/2-propanol 50:50 (v/v) (LC/MS-grade) and 2-propanol (LC/MS-grade) were purchased from Riedel-de Haën (Seelze, D). The food colorant standard substances (E 102, E 104, E 110, E 151) were purchased from the Institute of Dyes and Organic Products, IBPO (Zgierz, Poland). The seized samples were provided by the Bundeskriminalamt Wiesbaden, Germany. Buffers and solutions were prepared in deionised water gained in-house from the water purifier system Milli-Q Synthesis A10 from Millipore (Schwalbach, D).

2.2 Instrumentation

The samples were introduced into a Beckman-Coulter P/ACE 5000 system by hydrodynamic injection with 34.5 mbar (0.5 psi) for 5 s. Separation was performed in a bare 75 µm i.d. (363 µm o.d.) fused silica capillary from Polymicro Technologies LLC (Phoenix, AZ, USA) with a length of 82 cm. For separation, the capillary inlet was put on a voltage of +25 kV / +28 kV for piperazine separation and of -25 kV for food colorant analysis, keeping the sprayer on ground potential. For CE system control the software Beckman P/ACE Station 1.2 was used. The CE unit was connected to an HCT plus ion-trap mass spectrometer (Bruker Daltonics, Bremen, D) via an Agilent coaxial sheath-liquid sprayer interface (Agilent Technologies, Palo Alto, CA, USA). Electrospray ionisation (ESI) was performed at 4500 V. The sheath liquid 2-propanol/water (50:50) was supplied a flow rate of 3 µL/min by a syringe pump (Cole-Parmer, Vernon Hill, IL, USA). Nebuliser gas pressure was set to 4 psi. Flow and temperature of dry gas (nitrogen) were 4.0 L/min and 250°C. For CE-MS analysis of piperazine derivatives ESI-MS spectra were obtained in the positive ion mode with a scan speed of 26000 *m/z* per second in the mass range 70-300 *m/z* with a target mass of 200 *m/z*. For CE-MS analysis of food colorants in illicit drugs ESI-MS spectra were obtained in the negative ion mode with a scan speed of 26000 *m/z* per second in the mass range 80-1000 *m/z* with a target mass of 400 *m/z*. Auto-MS^n experiments were performed by isolation and subsequent fragmentation. Postprocessing software DataAnalysis (version 3.2, Bruker Daltonics) was used for data processing.

3. Results and discussion

3.1 CE-ESI-MS/MS analysis of piperazine-derived designer drugs

1-Aryl-piperazine derivatives, mainly the isomers of 1-chlorophenylpiperazine (especially the 1,3-isomer, *m*-CPP), 1-benzylpiperazine (BzP) and 1-[3-trifluoromethylphenyl]-piperazine (TFMPP), increasingly appear on the illicit drug market as active substances in Ecstasy tablets and so-called "smart drugs". They roughly mimic the psychoactive effects of amphetamine, but are less potent [4, 5]. Until 2004 piperazine-derived designer drugs were predominantly sold in capsules via European internet websites as "synthetic stimulants", for example the 1-benzylpiperazine containing "A2" [4]. Occasionally also seizures of tablets containing BzP or mixtures of BzP and TFMPP have been reported. End of 2004 for the first time Ecstasy tablets with the active substance 1-[3-chlorophenyl]-piperazine (*m*-CPP) appeared on the illicit drug market in several European countries on a bigger scale beginning with so-called "multi-coloured" tablets, also known as "Arlequin" and "Jenaer Smarties". As a consequence, *m*-CPP was listed as a Schedule II substance in Germany´s Controlled Substance Act (Anlage II BtMG) in 2007 [9] and, thus, *m*-CPP´s legal status turned from designer drug to controlled substance.

Several analytical methods have been used for the analysis of 1-arylpiperazine derivatives, among them GC, GC-MS, HPLC-UV and CE-UV [4-7]. As the piperazine derivatives are polar, easily protonable organic bases with pK_a values of typically around 9, capillary electrophoresis utilising an acidic buffer is a highly suitable method for their separation and determination as reported by Bishop et al. for CE-DAD [7]. In this work, the flexibility and matrix tolerance of CE and the identification power of ESI-MS/MS were combined for the analysis of piperazine-derived drugs with special emphasis on the separation and identification of positional isomers.

3.1.1 CE-ESI-MS separation of five piperazine derivatives

A CE-ESI-MS procedure with a run buffer consisting of 100 mmol/L formic acid at pH 2.4 and 10% (v/v) 2-propanol in a 75 µm i.d. fused-silica capillary of 82 cm length was employed for the separation and identification of piperazine-derived designer drugs. The baseline separation of five forensically relevant piperazine-derived designer drugs with different substituents (BzP, 3,4-MDBzP, *o*-MeOPP, *m*-CPP and TFMPP) was achieved within 13 min using a high voltage of +25 kV. A reconstructed electropherogram (SIM mode) is shown in Figure 1.

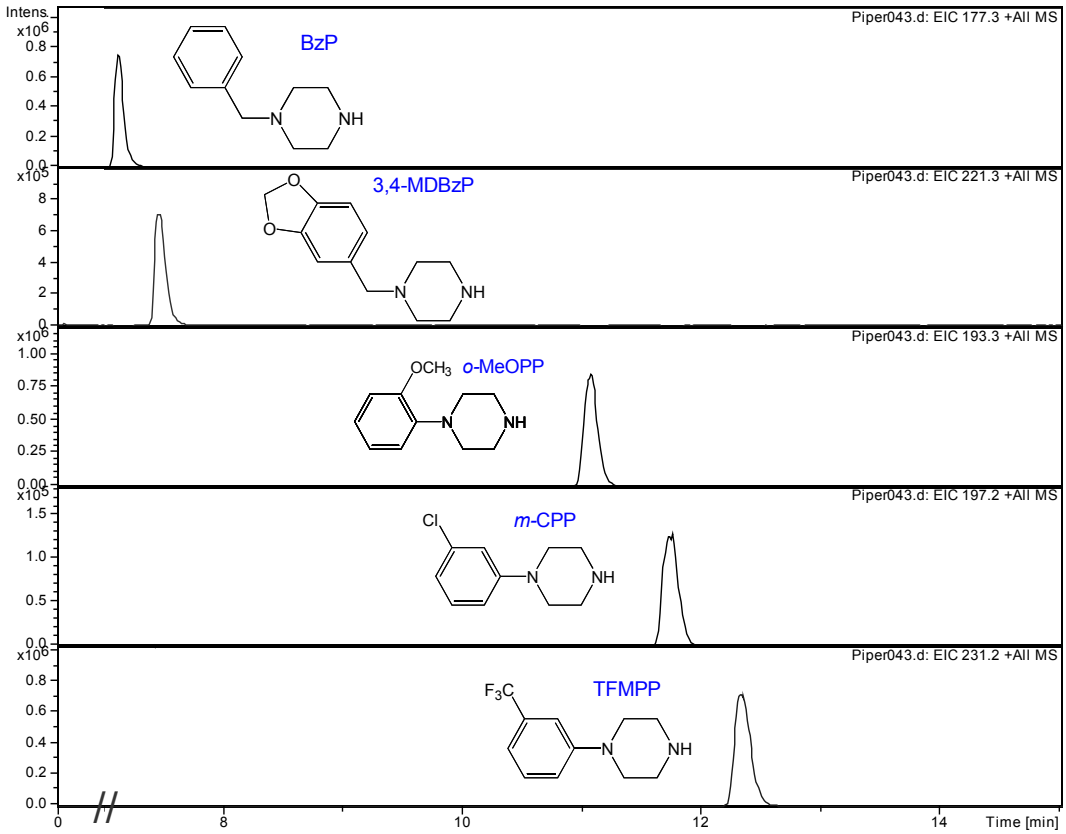

Fig. 1: CE-ESI-MS analysis of five piperazine-derived designer drugs (SIM mode, positive ion detection, for experimental parameters refer to the upper diagram of Fig. 3); $[M+H]^+$ molecular ion masses are in brackets: 1-benzylpiperazine, BzP (177.3 *m/z*), N-[3,4-methylenedioxybenzyl]-piperazine, 3,4-MDBzP (221.3 *m/z*), 1-[2-methoxy-phenyl]-piperazine, *o*-MeOPP (193.3 *m/z*), 1-[3-chlorophenyl]-piperazine, *m*-CPP (197.2 *m/z*), 1-[3-trifluoromethylphenyl]-piperazine, TFMPP (231.2 *m/z*).

3.1.2 CE-ESI-MS analysis of piperazine derivatives in a "smart drug" sample

The described CE-ESI-MS procedure was applied for the analysis of a so-called "smart drug" (synthetic stimulant with "legal" active compounds) named "X4 Ecstasy", available via the internet. In accordance with the label on the cardboard packaging (front-side: see Figure 2) the ingredients of the contained capsules should be BzP, TFMPP, an "amino acid blend" and Magnesium stearate. Interestingly, on the website of the internet supplier an additional ingredient called "piperazine blend" was listed and other internet suppliers also sell mixtures of TFMPP, *o*-MeOPP, *m*-CPP and *p*-CPP labelled as "X4".

For analysis, 2.4 mg of the powder contained in one of the "X4 Ecstasy" capsules were extracted with 1 mL of water/2-propanol (50:50 (v/v)). To 5 µL of the extract 5 µL of internal standard benzylamine (1 µg/mL) were added followed

by dilution with buffer to 400 µL. The resulting solution was directly injected into the separation capillary and submitted to CE-ESI-MS analysis. Figure 2 shows the extracted ion electropherograms (EIE) for the two piperazine-derived designer drugs present in the sample, BzP and TFMPP, further piperazine compounds were not detectable.

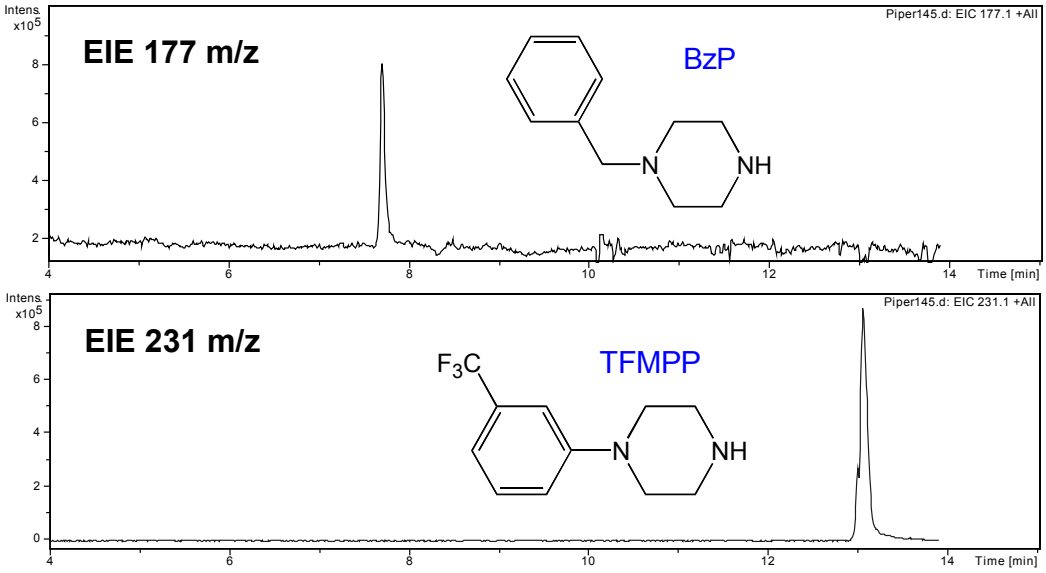

Figure 2:

CE-ESI-MS identification of piperazine-derived designer drugs in a "smart drug", sold via the internet (for experimental parameters refer to the upper diagram of Fig. 3).

The photograph on the right shows the front-side of the cardboard packaging and the capsules containing the drug mixture.

3.1.3 CE-ESI-MS baseline separation of positional isomers of piperazines

As a consequence of the new legal status of *m*-CPP in Germany as a controlled substance in combination with the still "legal" or designer drug status of its two positional isomers, *o*-CPP and *p*-CPP, there is a demand for analytical methods capable of separation and unambiguous identification of the three 1-chlorophenylpiperazines. One of the most distinct advantages of capillary electromigrative techniques is the fast-forward modification of selectivity by simply adding selectors to the separation buffer that more or less strongly interact with the analytes and, thus, influence their electrophoretic mobilities. The most important example in this respect is the use of cyclodextrins as buffer additives for chiral CE [8]. Chiral CE procedures are also suitable for positional isomers and have been reported for the separation of piperazine isomers by CE-DAD [7].

Employing cyclodextrins as additives in CE-ESI-MS procedures is not without problems as the non-volatile chiral selectors contribute to ion suppression in the electrospray process and cause contamination of the mass spectrometer [8]. Therefore, the chiral selector has to be carefully selected and its concentration should be kept as low as possible. In this work, a tailor-made procedure for the baseline separation of the three positional isomers of 1-chlorophenylpiperazine (*o*-CPP, *m*-CPP and *p*-CPP) at +28 kV was developed by adding 10 mmol/L 2-hydroxypropyl-beta-cyclodextrin to the buffer described in chapter 3.1.1.

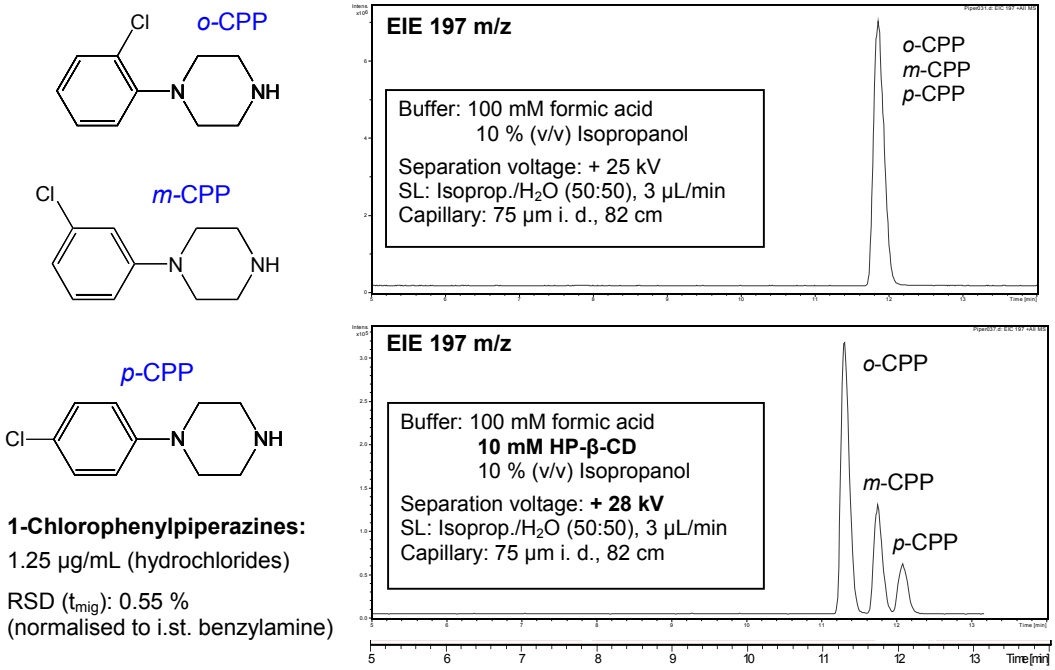

Fig. 3: Baseline-electroseparation of the three positional isomers of 1-chlorophenylpiperazine by CE-ESI-MS utilising the chiral selector 2-hydroxylpropyl-beta-cyclodextrin.

In Figure 3 the capillary electroseparation of an equimolar mixture of *o*-, *m*- and *p*-CPP by CE-ESI-MS with the acidic standard buffer (formic acid/2-propanol) in the upper diagram and with the chiral buffer system (containing HP-beta-CD) in the lower diagram is presented (reconstructed electropherograms, SIM mode). With the chiral buffer system baseline separation of the three positional isomers is achieved without extensive loss of sensitivity by ion suppression. Still, the signal-reducing effect of the chiral selector is responsible for the decrease in peak area from *o*-CPP to *p*-CPP: the elongated molecular structure of *p*-CPP leads to an especially strong interaction with the cyclodextrin ring. As a consequence, *p*-CPP exhibits the highest migration time (due to the interacting cyclodextrin´s co-migration with the slow EOF) and the highest extent of ion suppression, leading to a significant decrease in peak area.

3.1.4 CE-ESI-MS/MS discrimination of positional isomers of piperazines

The utilised ion-trap system is well suited for the discrimination of the three positional isomers of 1-chlorophenylpiperazine by recording multidimensional MS-spectra in the auto-MS^n mode in CE-ESI-MS analysis runs. The fragmentation patterns (positive ion mode) of the three investigated 1-chlorophenylpiperazine isomers are shown in Fig. 4. Although the fragmentation patterns differ (118-120, 180 *m/z*) a correlation with the migration time is recommended.

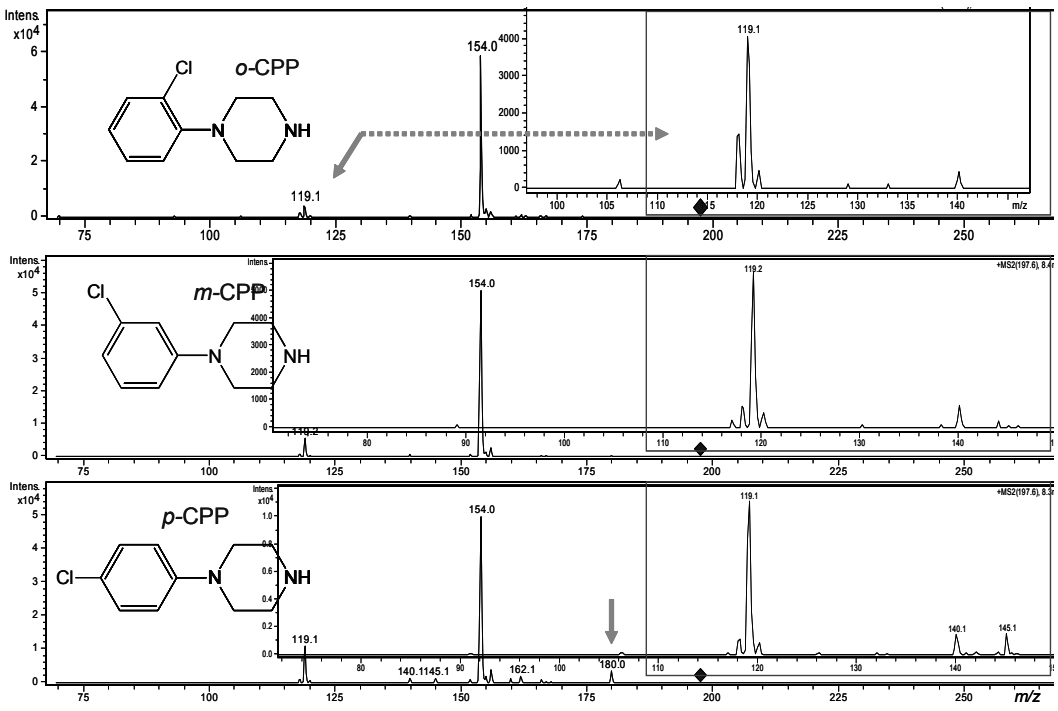

Fig. 4: Discrimination of the three positional isomers of 1-chlorophenylpiperazine by their ESI-MS^2-spectra (background subtracted, for experimental parameters refer to Fig. 3).

3.1.5 Identification of m-CPP in a seized "multi-coloured" Ecstasy tablet

The applicability of the developed CE-ESI-MS^n procedure with chiral cyclodextrin selector for the identification of the positional isomer of 1-chlorophenylpiperazine in a seized so-called "multi-coloured" Ecstasy tablet (diameter: 9 mm, weight: ~250 mg) was investigated. For CE-ESI-MS/MS analysis 2.5 mg of the powdered tablet were extracted with water by ultrasonication, diluted with run buffer and directly injected into the separation capillary. Identification of the 1,3-isomer (*m*-CPP) was achieved via an auto-MS/MS experiment and assignment of the migration time (Fig. 5). Consequently, the unambiguous discrimination between the controlled substance *m*-CPP and the designer drugs *o*-CPP and *p*-CPP in seized Ecstasy tablets is possible with the described chiral CE-ESI-MS/MS procedure.

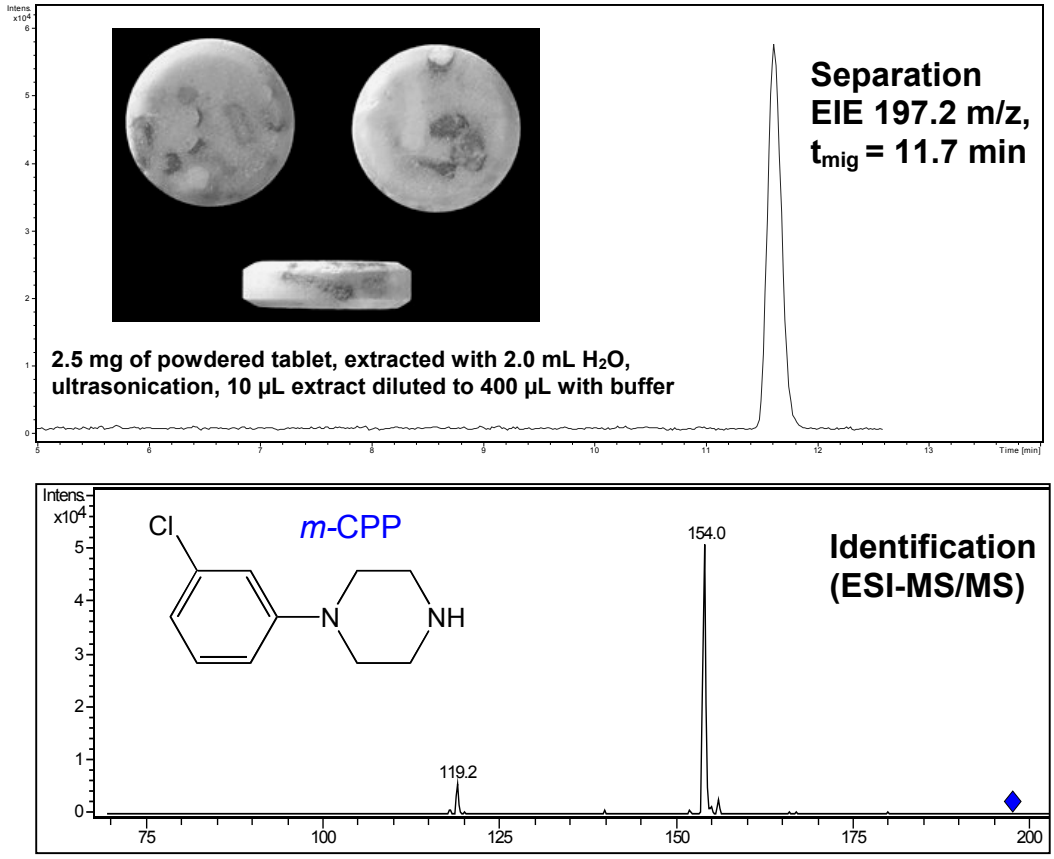

Fig. 5: Identification of 1-[3-chlorophenyl]-piperazine (*m*-CPP) as active substance in an aqueous extract of a seized "multi-coloured" Ecstasy tablet via CE-ESI-MS/MS (for experimental parameters refer to Fig. 3).

3.2 CE-ESI-MS analysis of food colorants in illicit drugs

In the last years, an increasing fraction of coloured Ecstasy tablets can be observed on the illicit drug market. Colour is a distinguishing feature and, in combination with other design features (especially the tablet logo), it contributes significantly to a brand identity and brand image and, accordingly, helps to improve the commercial success of an Ecstasy tablet. Furthermore, colouring of the tablets can mask unattractive shades of grey of the clandestinely manufactured drug substances as well as the brownish, often spotted discoloration because of the condensation of phenylethylamines and reducing sugars in the tablets (Maillard reaction). A mixture of food colorants is also frequently used to give diluents for heroin (often caffeine and paracetamol) a light brown colour to conceal the low diacetylmorphine content of cut-down (street-level) illicit heroin.

The identity and ratio of food colorants present in many Ecstasy tablets and heroin samples can point to links between different seizures on the diluent level and, thus, deliver valuable information for batch-to-batch comparison of drug samples. Furthermore, the distribution of the colorants in a tablet and the correlation between the colour and the concentrations of the main ingredients in heterogeneously coloured Ecstasy tablets can give important information about the tabletting process.

In the field of clandestine Ecstasy tablet production synthetic food colorants clearly are the predominating colouring agents. Other types of colorants like pigments (for example cadmium sulfide) are observed only in special cases. As most of the synthetic food colorants are either azo or triarylmethane components with at least one sulpho-group, they form single or multiple negatively charged species in aqueous solutions which can be easily separated by capillary electrophoresis. In the authors´ laboratory CE-DAD is applied for the analysis of food colorants in Ecstasy tablets with a modified version of a procedure described by Goldmann et al. [10]. Due to the limited sensitivity and selectivity of the optical detection scheme, using CE-DAD is difficult for analysis of food colorant traces and of positional isomers. Consequently, CE-ESI-MS with highly selective and sensitive MS detection in negative ion mode is an ideally suited analysis technique for the separation and unambiguous identification of synthetic food colorants even at trace levels. A co-electro-osmotic CE-ESI-MS procedure for anionic azo dyes with an alkaline ammonium acetate/acetonitrile buffer has been reported by Poiger et al. [11]. To increase the selectivity and to cope with problematic sample matrices, in this work a counter-electro-osmotic CE-ESI-MS procedure with a highly acidic run buffer (200 mmol/L formic acid at pH 2.2) was developed for the even at such low pH values negatively charged sulpho-group containing food colorants. At low pH the electro-osmotic flow (EOF) in bare fused silica capillaries is close to zero and, thus, the negatively charged analytes were detectable at the anode in short analysis times applying a high voltage of -25 kV to the capillary inlet. A comparable CE-ESI-MS separation

concept has been reported by Bringmann et al. [12] for the analysis of glucosinolates with sulpho moieties.

3.2.1 CE-ESI-MS procedure for the trace analysis of food colorants

A CE-ESI-MS procedure was developed for the trace analysis of sulpho-group containing azo- and triarylmethane-type food colorants in illicit drugs (Ecstasy tablets as well as heroin samples). Extremely high selectivity was achieved by employing a low pH run buffer (200 mmol/L formic acid) in a counter-electro-osmotic separation mode (-25 kV) and with negative ion ESI-MS detection for the even at pH 2.2 negatively charged colorants.

In Figure 6 the structures of the three food colorants tatrazine (E 102), sunset yellow (E 110) and brilliant black BN (E 151) present in cut-down heroin samples are depicted and Figure 7 shows the reconstructed electropherogram (SIM mode) for a mixture of the three named colorants which are separated within 9 min.

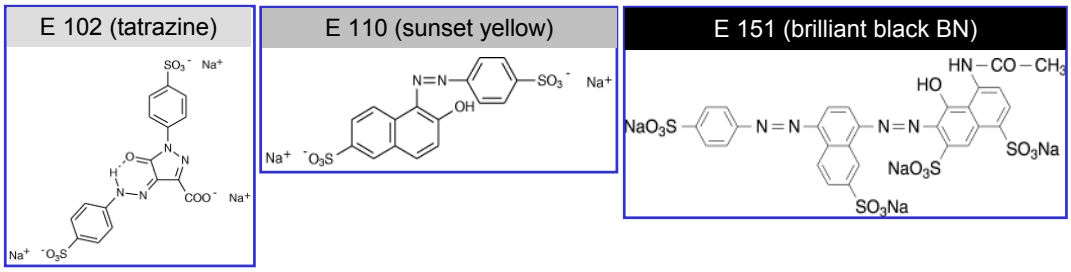

Fig. 6: Structures of the three food colorants E 102, E 110 and E 151 present in heroin diluents.

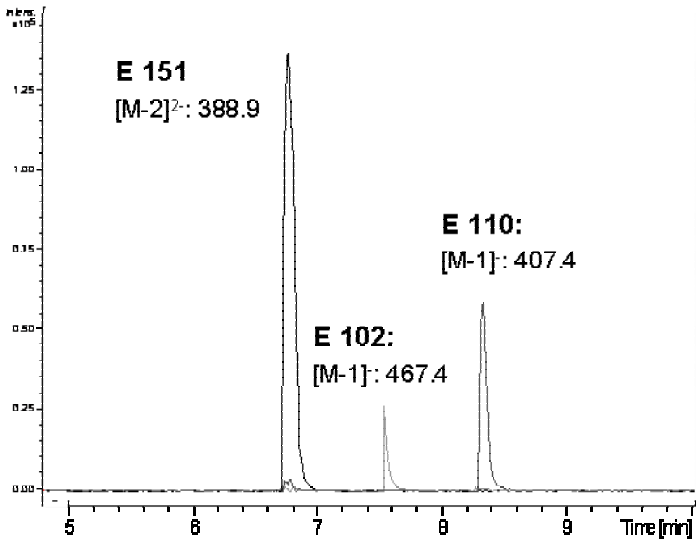

Fig. 7:
Extracted ion electrophero-grams for the separation of E 102, E 110 and E151 (c = 50 µmol/L in buffer).
Buffer:
200 mol/l formic acid at pH 2.2
Sheath liquid:
Isoprop./H_2O (50:50), 3 µL/min
Capillary: 75 µm i.d., 82 cm, HV: - 25 kV, injection 5 sec;
ESI-MS conditions:
Nebuliser: 4 psi, dry gas at 4 L/min, dry gas temp. 250 °, target mass: 400 m/z, mass scan 80-1000 m/z, negative ion detection mode.

Despite its higher molecular mass, E 151 shows the lowest migration time which is due to its higher charge-to-mass ratio because of the four sulpho-groups contained compared to only two sulpho-groups in E 102 and E 110. For E 102 and E 110 the mass traces of the $[M-1]^-$ molecular ions (E 102: 467.4 *m/z*, E 110: 407.4 *m/z*) delivered the best signal-to-noise ratio, in the case of E 151 the $[M-2]^{2-}$ molecular ion (388.9 *m/z*) was preferred (see Fig. 7).

In Figure 8 the corresponding ESI-MS spectra for the three colorants are shown. The presence of multiply charged molecular ions in the mass spectra facilitates the identification of the analytes. Additionally, the selectivity of the separation step is already exceptionally high, as only analytes which are negatively charged at pH 2.2 effectively migrate in the separation capillary. Therefore, in many cases the acquisition of multidimensional MS-spectra is not necessary for identification of the colorants.

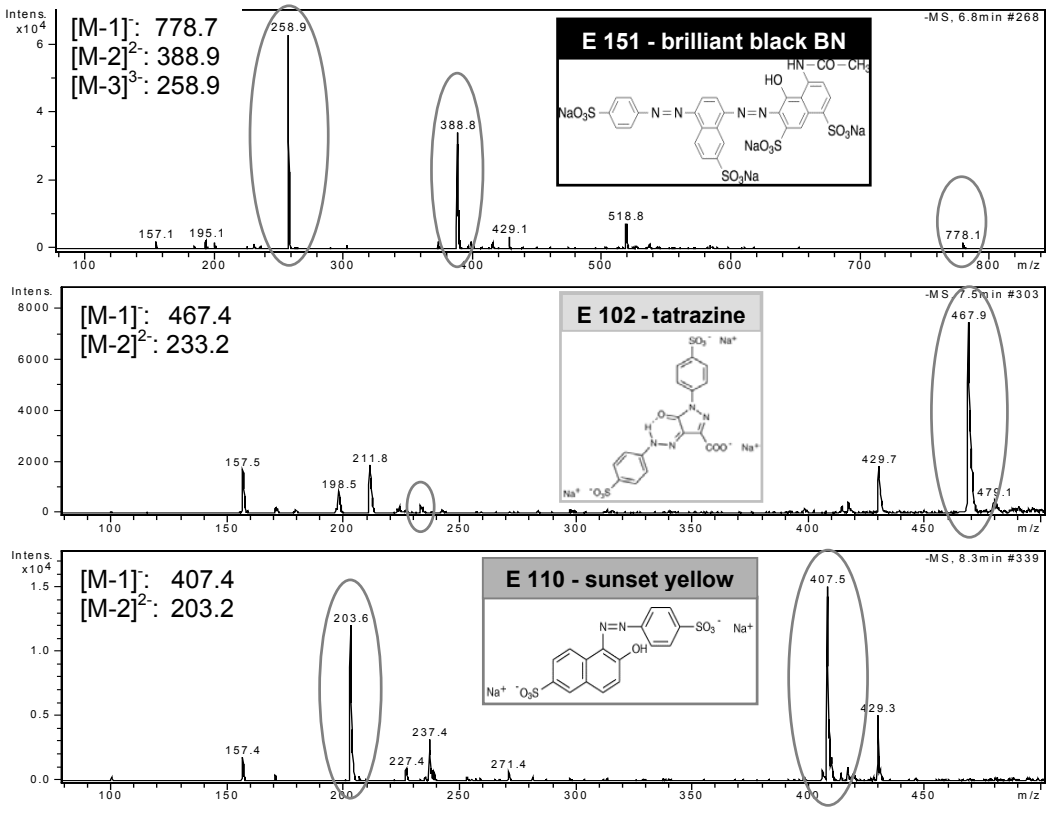

Fig. 8: ESI-MS spectra for E 102, E 110 and E 151 in negative ion detection mode (for experimental parameters refer to Fig. 7).

3.2.2 Batch-to-batch comparison of heroin samples via colorant analysis

The developed CE-ESI-MS procedure was applied for the batch-to-batch comparison of heroin samples based on the presence and the ratio of the three named food colorants. Although the food colorants are only present at trace levels the analysis with the counter-electro-osmotic CE-ESI-MS is possible without problems as the main compounds of heroin samples (active substances and cutting agents) do not disturb the electroseparation and the MS-detection. Neutral compounds like caffeine or paracetamol (and most organic acids at pH 2.2) are transported by the low residual electro-osmotic flow and organic bases (mono- and diacetylmorphine, opium alkaloids) form cations in the low pH buffer and are, thus, swept out of the capillary from the injection zone at the inlet side, when the negative high voltage is applied to the capillary at the beginning of the separation.

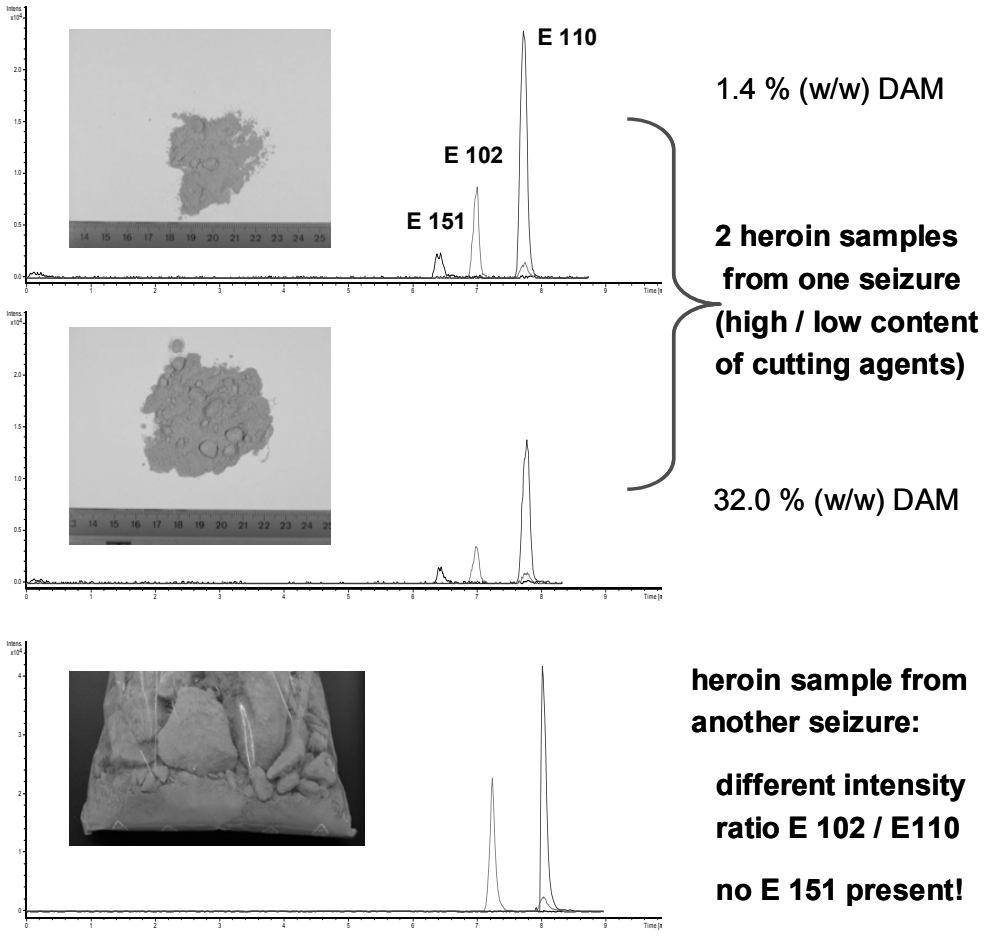

Fig. 9: Batch-to-batch comparison of seized heroin samples via CE-ESI-MS trace analysis of the food colorants E 102, E 110 and E 151 (for experimental parameters refer to Fig. 7).

For comparative analysis of three heroin samples 50 mg of each homogenised, powdered sample were extracted with 500 µL of run buffer, after centrifugation 100 µL of the extract were diluted with 200 µL buffer and directly injected into the separation capillary. Figure 9 shows the reconstructed electropherograms for three heroin samples analysed with the CE-ESI-MS procedure. The two samples on the top originate from the same seizure but are quite different with respect to their diacetylmorphine contents, although visually not distinguishable. In both samples all of the three food colorants are present with a similar relative intensity ratio, but in the first sample the concentration of the colorant mixture is higher corresponding with a much higher content of diluents (and lower DAM content) compared to the second sample. The third sample from another, not related seizure differs significantly from the other samples with the black colorant E 151 not being present and the intensity ratio of E 102/E 110 being higher.

3.2.3 CE-ESI-MS/MS identification of positional isomers in samples of E 104

Analysis of food colorants in Ecstasy tablets can be valuable with respect to drug profiling when the tablets contain mixtures of food colorants (e.g. yellow E 102 and blue E 131 in green tablets or E 122 and E 124 in red tablets), because the ratio of the colorants in mixtures can differ significantly. If only one colorant is present, the knowledge of its identity is less valuable as the number of synthetic food colorants that are permitted in the EC (and that clandestine producers of Ecstasy tablets have a straightforward access to) is quite limited. Yellow coloured Ecstasy tablets, for example, can be expected to contain either E 102 (tatrazine) or E 104 (quinoline yellow). But as the commercially available food colorants are typically not chemically pure there is the possibility to distinguish samples of the same colorant of different chemical manufacturers based on the presence and ratio of synthesis impurities or different positional isomers of the colorant substances. Thus, these differences, which are only accessible by analytical methods with high separation efficiency and high identification power, can be used to differentiate between samples coloured with the same type of food colorant.

In this work several samples containing the yellow colorant E 104 (pure colorants and a yellow coloured Ecstasy tablet) were analysed with CE-ESI-MS/MS. On the right side of Fig. 10 the EIE (of $[M-1]^-$ with 432.4 *m/z*) for an E 104 standard substance is shown. The two peaks with only slightly different migration times of the 432.4 *m/z* mass trace correspond to two positional isomers of E 104 (with different positions of the two sulpho-groups) which can also be discriminated by their MS/MS-spectra (see Fig. 10). The left side of Fig. 10 shows the EIE´s for the $[M-2]^{2-}$ molecular ion of E 104 (215.7 *m/z*) for two E 104 standards from different manufacturers and for an E 104 containing yellow Ecstasy tablet. It can be clearly seen that the intensity ratio for the two positional isomers of E 104 isomers is significantly different in the three samples.

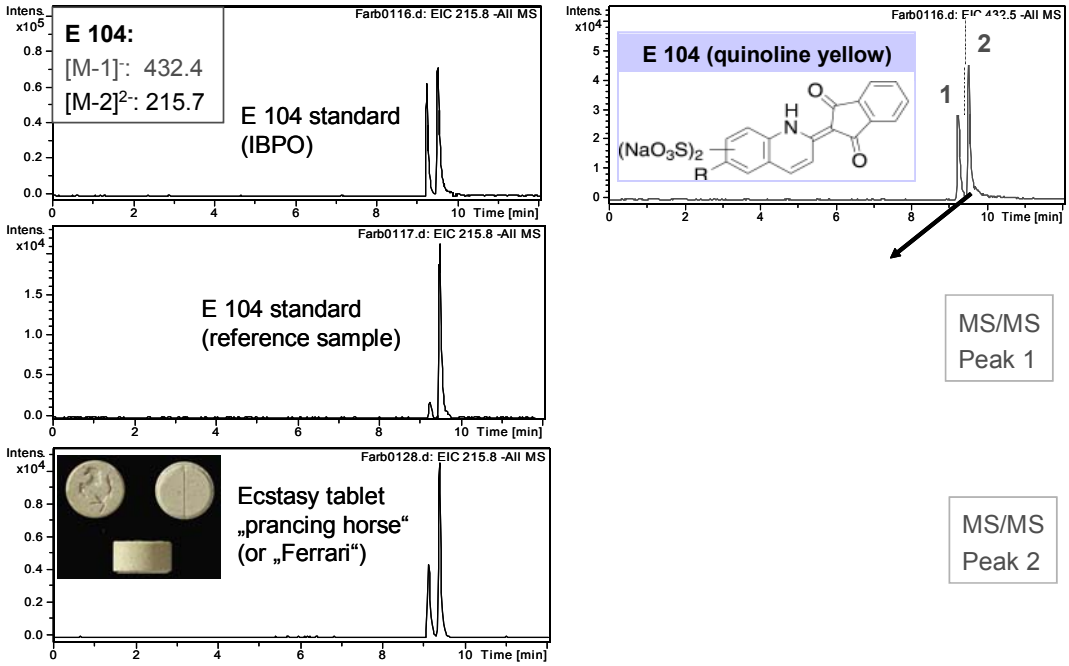

Fig. 10: CE-ESI-MS analysis of two positional isomers of quinoline yellow (E 104) in two different standard substances and in a yellow coloured Ecstasy tablet (extracted ion electropherograms for $[M-2]^{2-}$ / 215.7 m/z) on the left side. The discrimination of the two positional isomers of E 104 via the MS/MS spectra (precursor ion: $[M-1]^{-}$ / 432.4 m/z) is shown on the right side (for experimental parameters refer to Fig. 7).

4. Conclusion

In this work it was shown that the coupling of CE with ESI-MS is a powerful tool for forensic toxicological applications, demonstrated by the analysis of piperazine-derived designer drugs and trace analysis of food colorants in heroin samples and Ecstasy tablets. A CE-ESI-MS^n procedure with a cyclodextrin as chiral selector was optimised for the baseline separation and identification of positional isomers of piperazine-derived designer drugs. A highly selective counter-osmotic CE-ESI-MS procedure was developed for the separation and identification of positional isomers of the food colorant E 104 present in Ecstasy tablets and for trace analysis of food colorants in complex sample matrices.

The developed CE-ESI-MS^n procedures were successfully applied to the identification of piperazine derivatives and food colorants in seized “smart drugs”, Ecstasy tablets and heroin samples.

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