



## Analysis of stimulant drugs in the wastewater of five Nordic capitals

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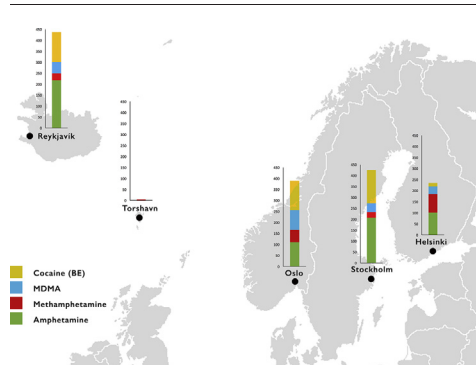
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### HIGHLIGHTS

- First Nordic comparison based on wastewater analysis of stimulant drugs in five capital cities
- Analytical performance was ensured by inter-laboratory comparison studies.
- Results revealed high use of amphetamines but not cocaine, compared with other European cities.
- Recreational use of cocaine and MDMA indicated by higher levels during weekends.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Wastewater-based epidemiology is an efficient way to assess illicit drug use, complementing currently used methods retrieved from different data sources. The aim of this study is to compare stimulant drug use in five Nordic capital cities that include for the first time wastewater samples from Torshavn in the Faroe Islands. Currently there are no published reports that compare stimulant drug use in these Nordic capitals. All wastewater samples were analyzed using solid phase extraction and ultra-high performance liquid chromatography coupled to tandem mass spectrometry. The results were compared with data published by the European Monitoring Centre for Drugs and Drug Addiction based on illicit drugs in wastewater from over 50 European cities. Confirming previous reports, the results showed high amphetamine loads compared with other European countries. Very little apparent abuse of stimulant drugs was detected in Torshavn. Methamphetamine loads were the highest from Helsinki of the Nordic countries, indicating substantial fluctuations in the availability of the drug compared with previous studies. Methamphetamine loads from Oslo confirmed that the use continues to be high. Estimated cocaine use was found to be in the lower range compared with other cities in the southern and western part of Europe. Ecstasy and cocaine showed clear variations between weekdays and weekends, indicating recreational use. This study further demonstrates geographical trends in the stimulant drug market in five Nordic capitals, which enables a better comparison with other areas of the continent.

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## 1. Introduction

Increasing use and higher potency of drugs of abuse poses a continuing threat to human health (European Monitoring Centre for Drugs and Drug Addiction, 2016b). Illicit drug use is known to have negative effects on crime rates and can cause serious public health issues such as higher risk of premature death and transmission of infectious diseases. These effects can lead to high economic and social costs (United Nations Office on Drugs and Crime, 2016). Monitoring drug use is crucial in order to fully understand the problem and develop efficient countermeasures (European Monitoring Centre for Drugs and Drug Addiction, 2016b).

Drug use and availability is known to show both temporal and geographical variations (Kankaanpää et al., 2014; Ort et al., 2014; Thomas et al., 2012). For example, amphetamines have a higher prevalence of use in northern and eastern Europe, as opposed to cocaine in western and southern Europe (European Monitoring Centre for Drugs and Drug Addiction, 2016b). National population surveys, information on drug seizures and clinical data have traditionally been used to monitor drug consumption. According to national reports based on population surveys that have been commissioned and compiled by the European Monitoring Centre for Drugs and Drug Addiction (EMCDDA), cannabis is the most commonly used illicit drug followed by amphetamines and cocaine in Denmark, Finland, Norway and Sweden (European Monitoring Centre for Drugs and Drug Addiction, 2017a, 2017c, 2017d, 2017e). Similar patterns in use have been reported for Iceland and the Faroe Islands, according to the European School Survey Project on Alcohol and Other Drugs (ESPAD) (European Monitoring Centre for Drugs and Drug Addiction, 2016a). However, these survey methods have shown significant limitations such as reporter bias, low response rates and struggle to provide information on rapidly changing trends in the drug market. Therefore, other measures are needed to complement these methods to ensure a more comprehensive assessment (Castiglioni et al., 2014).

Wastewater-based epidemiology (WBE) is an approach where urinary excretion products are quantified to assess illicit drug use of a large population (Zuccato et al., 2005). WBE can give reliable results that may quickly reveal short and long-term trends in the scale of drug use. This approach can therefore provide a valuable source of complementary data to support more traditional epidemiological methods (Ort et al., 2014; Terzic et al., 2010; Thomas et al., 2012; van Nuijs et al., 2011; Zuccato et al., 2011). The EMCDDA has published the findings of the European inter-disciplinary network “Sewage analysis CORE group – Europe” (SCORE) (SCORE, 2017). This network brought scientists from relevant disciplines together to support research and innovation in Europe based on the analysis of biomarkers in wastewater. The main aim was to ensure that novel technologies were transferred to full-scale applications in order for authorities to be able to utilize the information gathered. Furthermore, the goal was to establish protocols for wastewater analysis by coordinating international studies with inter-laboratory comparisons. This European-wide network has organized measurements of illicit drugs in wastewater each year since 2011 in over 50 cities (Ort et al., 2014; SCORE, 2017; Thomas et al., 2012).

WBE studies have been performed in most Nordic countries to complement other methods (Bramness et al., 2015; Kankaanpää et al., 2014; Ort et al., 2014; Östman et al., 2014; Thomas et al., 2012). Results have shown that amphetamines have been more dominant than cocaine on the stimulant drug market in Northern Europe (Bramness et al., 2015; Kankaanpää et al., 2014; Ort et al., 2014; Östman et al., 2014; SCORE, 2017; Thomas et al., 2012). Amphetamine is excreted unchanged in urine (30–74%) and is also an urinary metabolite of methamphetamine (4–7%) (Baselt, 2014). It is therefore important to assess the use of these two chemicals in parallel (Ort et al., 2014). WBE studies have demonstrated that methamphetamine use is prominent in central Europe and is reportedly produced in Lithuania or the Czech Republic before being exported to Scandinavian countries (Griffiths et al., 2008;

Mackul'ak et al., 2014; Ort et al., 2014). Amphetamine use has generally been higher than methamphetamine use in the Nordic countries with the exception of Denmark where the use of both drugs is low compared with cocaine (Baz-Lomba et al., 2016; SCORE, 2017). However, in recent years Norway and Finland have also showed high methamphetamine loads with continued high loads of amphetamine in parallel (Bramness et al., 2015; Ort et al., 2014; SCORE, 2017). Trends in 3,4-methylenedioxymethamphetamine (MDMA) use, based on interview surveys, have indicated that the drug is becoming more common in Europe among young people (European Monitoring Centre for Drugs and Drug Addiction, 2016b). Most recent reports based on WBE show that the largest MDMA loads are measured in northern European countries such as the Netherlands, Belgium, Norway and Denmark (Baz-Lomba et al., 2016; SCORE, 2017). WBE studies have demonstrated that cocaine use is most prominent in western European countries such as Belgium, the United Kingdom, Switzerland, Spain and the Netherlands (Baker et al., 2014; Baz-Lomba et al., 2016; Been et al., 2016; SCORE, 2017).

For the first time, this study aims to compare and discuss trends in stimulant drug use based on WBE in the capital cities of Norway, Iceland, Finland, Sweden and the Faroe Islands. Never before have illicit drugs in wastewater been analyzed from Torshavn in the Faroe Islands. Chosen for this study were the stimulant illicit drugs amphetamine, methamphetamine, MDMA and cocaine. Due to analytical difficulties it was not possible to include cannabis in this study (Causanilles et al., 2017). By using similar sample preparation methods, instrumental analysis and data processing, it was possible to achieve harmonized results that can be compared with reports from other European countries in the SCORE network (SCORE, 2017). The analytical performance of methodologies was also evaluated by external quality control cycles, which were performed by different laboratories (van Nuijs et al., 2018).

## 2. Materials and methods

### 2.1. Chemicals, reagents and materials

The following materials were used for the preparation and analysis of samples from Oslo, Reykjavik, Stockholm and Torshavn: reference standards for eight illicit drugs and/or major metabolites were amphetamine, methamphetamine, MDMA, cocaine, benzoylecgonine (BE), and cocaethylene. Reference standards were dissolved in methanol (MeOH) or acetonitrile (ACN) at concentrations of 1 mg/mL or 100 µg/mL. Corresponding isotope-labeled internal standards (ILIS) used were amphetamine- $d_8$ , methamphetamine- $d_{11}$ , MDMA- $d_5$ , cocaine- $d_3$ , BE- $d_3$  and cocaethylene- $d_3$  dissolved in MeOH or ACN at concentrations of 100 µg/mL. All reference standards and ILIS were purchased from Cerilliant (Round Rock, TX, USA). Standard stock solutions for reference standards were prepared at concentrations of 100 µg/mL in either MeOH or ACN. Mixed working solutions were prepared for the reference standards and the ILIS at concentrations of 1.0 µg/mL in MeOH. All standard and working solutions were stored at  $-20\text{ }^{\circ}\text{C}$ . HPLC-grade MeOH was from Rathburn Chemicals Ltd. (Walkerburn, SCT, UK) and HPLC-grade ACN was from VWR Chemicals (Oslo, NOR). Ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) solution  $\geq 25\%$  in water was from Fluka - Sigma-Aldrich (Oslo, NOR) and formic acid (FA) 98–100% (p.a.) was from Merck - Millipore (Billerica, MA, USA). Oasis HLB µElution plates (30 µm) were purchased from Waters (Milford, MA, USA).

The following materials were used for the preparation and analysis of samples from Helsinki: reference standards purchased from Sigma-Aldrich (St. Louis, MO, USA) were amphetamine sulphate, cocaine hydrochloride and MDMA hydrochloride. A reference standard donated by the UN Narcotics Laboratory (Vienna, Austria) was methamphetamine hydrochloride. Reference standards and ILIS purchased from Cerilliant (Round Rock, TX, USA) were BE, amphetamine- $d_6$ , cocaine- $d_3$ , MDMA- $d_5$ , methamphetamine- $d_{14}$  and BE- $d_3$  dissolved in MeOH or ACN at concentrations of 1 mg/mL or 100 µg/mL. Carbon 13-labeled

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