"PHENETHYLAMINE" STREET SAMPLES ENCOUNTERED ON THE BELGIAN DRUG MARKET

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ABSTRACT: Phenethylamines remain very popular among recreational drug users for their stimulating and the mood-modifying properties. Street samples (n = 478; 260 powders, 197 tablets and 21 other dose forms confiscated by Belgian law enforcement authorities in the period 1991–1998) were analyzed by light pipe gas chromatography-Fourier transform infrared spectrometry (GC/FTIR). A computerized street sample database (Microsoft Access® 97) was developed. The database contains GC/FTIR analysis results of the street samples regarding phenethylamines and other interesting compounds such as by-products, impurities, filling agents and adulterants, in combination with the ballistic characterization of the dose forms (visual color, description of tablet logos including logo pictures and EDU logo numbers, dimensions, geometry, score type...).

KEY WORDS: Street samples; Phenethylamines; Gas chromatography-Fourier transform infrared spectrometry (GC/FTIR).

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INTRODUCTION

Phenethylamines, a group of structurally related compounds with amphetamine as basic molecule, remain very popular among recreational drug users for their stimulating and mood-modifying properties. Many of these phenethylamines have become controlled substances and are illegally synthesized in various clandestine laboratories throughout Europe and the United States. New, closely related derivatives are constantly developed in order to create new pharmacological properties, evade drug legislation or circumvent governmental control on precursors. Unambiguous identification of these phenethylamines in street samples is essential for the successful prosecution of designer drug cases in a court of law, but is also interesting from a pharmacological-toxicological point of view.

EXPERIMENTAL

Instrumentation and data acquisition

A Perkin-Elmer (Buckinghamshire, UK) Autosystem GC was interfaced with a light pipe GC/IR System 2000 and connected to a FTIR System 2000 with a mid-infrared source and a medium band liquid nitrogen-cooled mercury cadmium telluride (MCT) detector. A CIS-3 programmed temperature vaporization (PTV) injector (Gerstel, Brielle, the Netherlands) was used in the splitless mode. Temperature-programmed separations were carried out on a Hewlett-Packard (Palo Alto, CA, USA) Ultra-1 methylsilicone capillary column (25 m x 0.32 mm i.d., 0.52 μm film thickness). The carrier gas was helium at a flow rate of 1.8 ml/min. The gold-coated light pipe (12 cm x 1 mm i.d.) was heated at a constant temperature of 270°C. Real time spectra were obtained by addition of two scans, with a spectral resolution of 8 cm^{-1} and 32 background scans. The scan range was from 4000 to 580 cm^{-1}. Chromatograms were calculated by the Gram-Schmidt vector orthogonalization method. Gram-Schmidt reconstruction was performed using 10 basis vectors throughout the run. Baseline correction was performed on the reconstructed Gram-Schmidt (GS) chromatogram and low-noise vapor-phase FTIR spectra were generated after co-addition.

Vapor-phase FTIR spectral library

A unique vapor-phase FTIR spectral library was generated by injecting methanolic stock solutions (1.0 mg/ml) of commercial reference standards and in-house synthesized amphetamine analogues into the GC/FTIR system. The obtained reference vapor-phase FTIR spectra were stored in the computer-based library after normalization. The "phenethylamine" library contains up to now 159 vapor-phase FTIR spectra of amphetamine analogues and other toxicologically relevant drugs.

GC/FTIR analysis of "phenethylamine" street samples

Street samples (n = 478; 260 powders, 197 tablets and 21 other dose forms confiscated by Belgian law enforcement authorities in the period 1991–1998) were analyzed by gas chromatography-Fourier transform infrared spectrometry (GC/FTIR). Aliquots of the methanolic solutions of the exhibits were injected into the GC/FTIR system. The obtained vapor-phase FTIR spectra were submitted to a spectral search on the laboratory-made "phenethylamine" library. Quantitative GC/FTIR analyses were performed after derivatization with heptafluorobutyric anhydride [1].

RESULTS AND DISCUSSION

The hyphenated technique of capillary gas chromatography coupled with Fourier transform infrared spectrometry (GC/FTIR) was successfully applied to the analysis of

phenethylamines in confiscated dose forms. With light pipe GC/FTIR, unique vapor-phase FTIR spectra were generated. The obtained vapor-phase FTIR spectra were submitted to a spectral search on the laboratory-made "phenethylamine" vapor-phase FTIR spectral library allowing the unequivocal identification of different phenethylamines.

Several commonly encountered illicit amphetamines were identified in the street samples using this approach. Novel phenethylamines, such as α-phenethylamine (APEA), N-methyl-a-phenethylamine (MAPEA), N-methyl-1-(1,3-benzodioxol-5-yl)-2-butanamine (MBDB), N,N-dimethyl- 3,4-methylenedioxyamphetamine (MDMMA) and 4-bromo-2,5- dimethoxyphemethylamine (also known under the street names 2C-B or Nexus), were encountered in judicial samples, indicating some possible new trends on the Belgian drug market.

The chemical variability of the "phenethylamine" street samples confiscated by law enforcement authorities on the Belgian drug market in the period 1991–1998 is presented in Figures 2 and 3.

α-phenethylamine (APEA) was encountered in street powders en post mortem samples of two drug addicts in 1994 [2]. In 1997 N-methyl-α-phenethylamine (MAPEA) was identified in different street tablet on the Belgian drug market. The identification of α-phenethylamine and its N-methyl derivative supports the hypothesis that a new de-

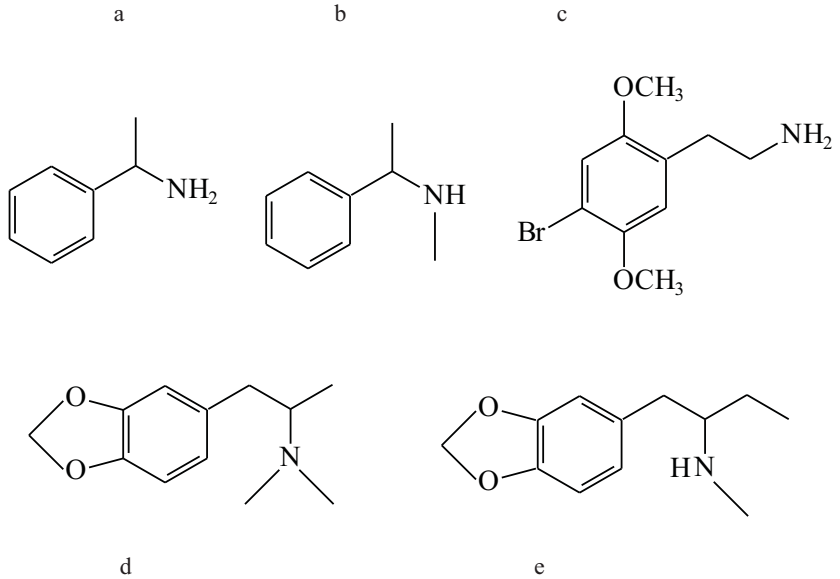

Fig. 1. Chemical structures of some novel phenethylamines: a) α-phenethylamine; b) N-methyl-α-phenethylamine; c) 4-bromo-2,5-dimethoxyphenethylamine; d) N,N-dimethyl-3,4-methylenedioxyamphetamine; e) N-methyl-1-(1,3-benzodioxol-5-yl)-2-butanamine.

Fig. 2. Chemical variability of 197 “phenethylamine” street tablets.

Fig. 3. Chemical variability of 260 “phenethylamine” street powders.

signer drug class, so-called 1-phenethylamines, is intentionally synthesized in European basement chemistry labs.

4-bromo-2,5-dimethoxyphenethylamine (2C-B) was encountered in very small street tablets (diameter ± 5 mm) within a commercial packaging and is still not controlled under Belgian drug legislation.

N-methyl-1-(1,3-benzodioxol-5-yl)-2-butanamine (MBDB) was identified as the first representative of the butanamines in different street tablets on the Belgian drug market and is a controlled substance since January 1999.

A computerized street sample database (Microsoft Access® 97) was developed (available on CD-ROM for forensic institutes and law enforcement authorities). The database contains GC/FTIR analysis results (in some cases also additional GC/MS results) of the street samples regarding phenethylamines and other interesting compounds such as by-products, impurities, filling agents and adulterants, in combination with the ballistic characterization of the dose forms. The street sample database is divided in a tablet database, a powder database and a database for other dose forms. Figure 4 represents the main record screen of a street tablet in the tablet database with general information (case

registration (identification) number, origin, stock contents, users' attributes, packaging) and ballistic characterization (tablet shape, tablet hardness, color, dimensions, weight). Other fields of the same exhibit can be shown by clicking on three different buttons that were created in the main screen. The "analysis" button reveals the GC/FTIR (and occasionally GC/MS) analysis results of the street tablet. Description of tablet logos including EDU logo numbers together with tablet scoring, score type and score width are shown after clicking the "logo/symbol/text-scoring" button (Figure 5). By clicking on the "pictures" button digital photographs (*.tif files) of the two tablet surfaces and the belly band of the street tablet are pictured (Figure 6).

The main record screen of each street powder in the powder database provides general information (case registration (identification) number, origin, stock contents, users' attributes, packaging, weight), and ballistic characterization (powder properties and color). Analysis results can be obtained after clicking on the "analysis" button.

The "other dose form" database contains the form (e.g. starch or gelatin capsule, solution or liquid, etc.) and the complete description of the dose form including some general information (origin, weight, stock contents). The "analysis" button on the main screen reveals the analysis results for each of the other dose forms.

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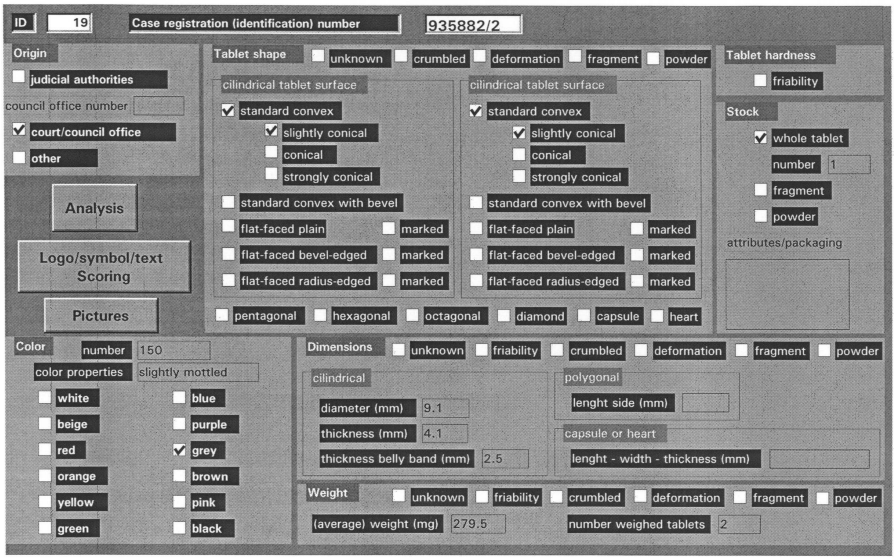

Fig. 4. Main record screen of a street tablet in the tablet database.

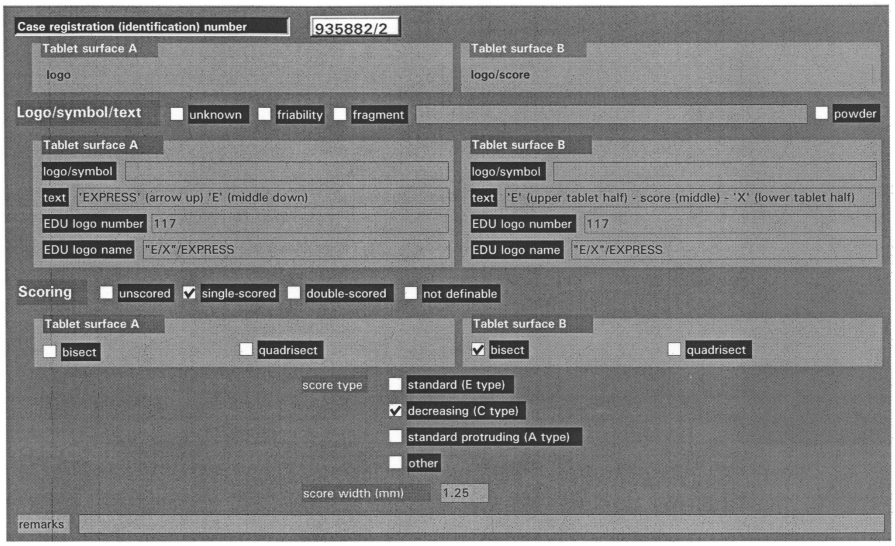

Fig. 5. "Logo/symbol/text-scoring" record screen of a street tablet in the tablet database.

Fig. 6. "Pictures" record screen of a street tablet in the tablet database.

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