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Investigations on a “Black Market’s Drug - Carphedon“
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INVESTIGATION ON A "BLACK MARKET'S DRUG - CARPHEDONE"

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INTRODUCTION
During the past years "the black market" supports to the athletes drug with name "Carphedone". We are not found appropriate literature data on this drug. The investigation was carried out by the drug "Carphedone" bought from "the black market" and the active substance was isolated by HPLC. The chemical, spectroscopic (UV, IR, MS, $^1$H and $^{13}$C NMR) and chromatographic (CG, GPC and HPLC) properties of the substance were studied. The urine samples of volunteer after administration of one tablet "Carphedone" were analysed too.

EXPERIMENTAL

Figure 1. Used methods and techniques for isolation and identification of the substance from tablet

$^1$H, $^{13}$C (DEPT, CH90)

2D NMR techniques
a) NOESY
b) $^1$H,$^1$H Homonuclear shift correlatia (COSY)
c) $^1$H, $^{13}$C Hetero-nuclear correlatia
d) $^1$H, $^{13}$C Hetero-nuclear multiple bond correlation (HMBC)

HPLC - Waters instrument, column-µ Bondapak C 18 with 19mm ID x25 cm, 10 μm, flow rate - 2 ml/min; eluent H₂O/ AcN= 40:60; λ=220 nm; Fractions are collected from 6.2 to 6.8 min

Grind and dissolve in H₂O/ CH₃OH
Filter 0.4μm pore size

HPLC Fractions

Dry substance

Dry substance

IR

GPC

GC/MS

different
derivatisation

NMR
Figure 4. IR spectrum of carphedone.

Figure 3. GPC chromatogram of carphedone (HP 5890 II).

Figure 2. GC/NPD chromatogram of carphedone (HP 5890 II) with deuterated internal standard: (1) 10 ppm TMS, (2) 10 ppm tetradecane.
Figure 5. 250 MHz $^1$H NMR spectrum of Carphedone in CDCl$_3$

Figure 6. 250 MHz proton correlated 2D NMR spectrum (COSY) of Carphedone in CDCl$_3$.

Crosspeaks
- H-3/ H-4
- H-4/ H-5

Figure 7. 250 MHz $^{13}$C NMR spectrum of Carphedone in CDCl$_3$

Figure 8. 250 MHz $^1$H-$^{13}$C 2D NMR spectrum of Carphedone

Crosspeaks
- 2.769 and 2.677/ 38.3
- 3.696/ 37.4
- 3.696/ 141.7
- 3.590 and 3.902/ 55.5
- 4.021/ 46.6
2-oxo-4-phenyl-1-pyrrolidinomethanone.

This formula is very similar to the well-known neurotoxic drug Phencyclidine (2-PCP). The obtained results of spectroscopic and chromatographic analyses confirmed the new formula of Carphedone.

Chemical Structure of Carphedone

(HMBE) of Carphedone

All NMR spectra were recorded by "Bruker DRX 250" in CDCl₃.

Figure 10. 250 MHz H-13C multiple bond correlated spectrum of Carphedone.

Figure 9. 250 MHz NOESY spectrum of Carphedone.
Figure 11. EI mass spectra and fragmentation of Carphedone (A) and Piracetam (B)
Dimethylated (2) derivatives are formed from both substances.

Figure 12: EI mass spectra and fragmentation of methylated with CH₃, Carphedone and Phlacem. Under condition of methylation mono-(1) and di- (2)
Figure 13. EI mass spectra of silylated with N-Methyl-N-trimethylsilyl-trifluoroacetamide (MSTFA) Piracetam (A) and Carphedone (B).

ANALYSES OF URINE SAMPLES AFTER ADMINISTRATION OF CARPHEDONE

The urine samples are collected up to 16 hours after administration of 1 tablet Carphedone from health volunteer. The samples were analysed by GC/NPD and GC/MSD after carrying out a procedure for free stimulants (extraction at pH=14). Unchanged Carphedone and its Metabolite was detected and identified by GC/MSD in analysed urine samples after application of Carphedone.

Analytical parameters:

<table>
<thead>
<tr>
<th>Analysis</th>
<th>GC analyses</th>
<th>GC/MSD analyses</th>
</tr>
</thead>
<tbody>
<tr>
<td>Analytical instrument</td>
<td>GC/NPD HP 5890 II</td>
<td>HP GC 5890/ MSD 5970</td>
</tr>
<tr>
<td>Carrier gas:</td>
<td>Nitrogen (1 ml/min flow)</td>
<td>Helium (1 ml/min flow)</td>
</tr>
<tr>
<td>Split ratio:</td>
<td>1:15</td>
<td>1:10</td>
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<td>Analytical column:</td>
<td>HP Ultra-2 16m, 0.25 mm i.D.,</td>
<td>HP - 5, 20 m, 0.25 mm i.D.</td>
</tr>
<tr>
<td></td>
<td>0.20 μm film thickness</td>
<td>0.20 μm film thickness</td>
</tr>
<tr>
<td>Temperature program:</td>
<td>70°C, 20°C/min till 300°C</td>
<td>120°C, 15°C/min till 300°C</td>
</tr>
<tr>
<td>Injection:</td>
<td>2 μl</td>
<td>1 μl</td>
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</tbody>
</table>
Figure 15. GC chromatogram of urine sample after administration of Carphedone: nicotine - 1, caffeine - 2, Metabolite of Carphedone - 3.

Figure 16. Excretion curves of Carphedone (●) and Metabolite (○).

Figure 14. EI mass spectra of Metabolite of Carphedone: un derivatized (A) and after methylation (B).

Concentration, µg/ml urine

Time, h

0 0.4 0.8 1.2 1.6 2 2.4

0 4 8 12 16

Mw = 161