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Analytical characterization and pharmacological evaluation of the new psychoactive substance 4-fluoromethylphenidate (4F-MPH) and differentiation between (±)-threo- and (±)-erythro- diastereomers.

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\[
\begin{align*}
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\text{H} & \quad \text{O} \\
\text{N} & \quad \text{O} \\
\text{H} & \quad \text{O} \\
\end{align*}
\]

Methylphenidate

\[
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\text{H} & \quad \text{O} \\
\text{N} & \quad \text{O} \\
\text{H} & \quad \text{O} \\
\end{align*}
\]

4-Fluoromethylphenidate
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3. Preparation of 4F-MPH tablets for GC-MS/LC-MS analysis

For analysis of the 4F-MPH tablets by gas chromatography mass spectrometry (GC-MS), the tablet was crushed using a mortar and pestle and 10 mg was added to 1 mL of methanol. This solution was added to a Corning® Costar® Spin-X® centrifuge tube filter (cellulose acetate membrane, 0.45 µm) (Corning Inc, United States) and centrifuged at 2500 rpm for 3 minutes. Furthermore, 100 µL of this filtered solution was added to 900 µL methanol in a GC vial.

For analysis of the 4F-MPH tablets by liquid chromatography mass spectrometry (LC-MS), the tablet was crushed using a mortar and pestle and 10 mg was added to 1 mL of acetonitrile/water (1:1) with 0.1% formic acid. This solution was added to a Corning® Costar® Spin-X® centrifuge tube filter (cellulose acetate membrane, 0.45 µm) (Corning Inc, United States) and centrifuged at 2500 rpm for 3 minutes. Furthermore, 20 µL of this filtered solution was added to 980 µL acetonitrile/water (1:1) with 0.1% formic acid in a LC vial.
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(±)-threo-4F-MPH

(±)-threo-4F-MPH
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(±)-threo-4F-MPH raceme (HCl salt in DMSO)

(±)-erythro-4F-MPH raceme (HCl salt in DMSO)
4F-MPH vendor product identified as (±)-threo- and (±)-erythro-4F-MPH racemes (HCl salt in DMSO)

4F-MPH vendor product identified as (±)-threo-4F-MPH racemate (HCl salt in DMSO)
Heteronuclear Multiple Bond Correlation (HMBC)

(±)-threo-4F-MPH racemate (HCl salt in DMSO)

(±)-erythro-4F-MPH racemate (HCl salt in DMSO)
4F-MPH vendor product identified as \((\pm)-\text{threo}\) and \((\pm)-\text{erythro}\)-4F-MPH racemates (HCl salt in DMSO)

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Tablet containing (±)-threo-4F-MPH racemate
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Station  
Supervisor  
Analysis Name  
Acquisition Date  
Sample Description

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**SmartFormula Settings**

Low value of mSigma indicates good isotopic pattern match.
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Submitter
Supervisor
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Acquisition Date: 06/07/2016 15:10:46
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![Graph](image)

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**SmartFormula Settings**

Low value of mSigma indicates good isotopic pattern match
McLaughlin et al. Supporting Information Drug Testing and Analysis

Trinity College Dublin
Bruker Open Access LC-MS - Formula Identification Report

Sample-ID
Submitter
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Acquisition Date: 06/07/2016 15:14:03

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SmartFormula Settings
Low value of mSigma indicates good isotopic pattern match

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**SmartFormula Settings**

Low value of mSigma indicates good isotopic pattern match.