

The use of Solid Phase Extraction (SPE) for detection Methylphenidate and Ritalinic Acid in small volume plasma samples

Viola Nguyen¹, Blessing Akitunde¹, Dhoone Menezes de Sousa¹,
Shyamalee Dassanayake ¹

¹Environmental Medicine & Climate Science , Icahn School of Medicine NY 10023, USA.

*Corresponding author. Email: viola.nguyen@mssm.edu

This methodology describes an extraction of Methylphenidate (MPH) and Ritalinic Acid (RA), from plasma using solid-phase extraction (SPE), followed by silylation reaction. In addition an ion chromatographic method was developed for the specific GC determination of MPH and RA. Treated plasma samples were passed through SPE cartridge with Hydrophilic-Lipophilic-Balanced (HLB) sorbent to retain and elute target analytes. Using N-Methyl-N-(trimethylsilyl)trifluoroacetamide (MSTFA) and N-Methyl-bis(trifluoroacetamide) (MBTFA) reagents, eluent was derivatized and the non-polar product was further analyzed using GC-MS. A calibration curve for MPH and RA was constructed in the range 2-250 ug/mL. The SPE resulted in higher extraction recovery (mean x %) with % R.S.D.s similar in both matrix and solvent x%, respectively).

1 Method and materials

Native standards for Ritalinic Acid hydrochloride (1.0 mg/mL in MeOH) and Methylphenidate hydrochloride (1.0 mg/mL in MeOH) were purchased from Sigma Aldrich (St. Louis MO, USA). Both labeled standards for (\pm)-threo-Methylphenidate- D_4 HCl 100ug in MeOH) and (\pm)-threo-Ritalinic acid- D_{10} HCl (100ug in MeOH) were obtained from Cerilliant (Round Rock, TX). BioChemed Services (Winchester, VA, USA) provided with bovine plasma. HPLC grade formic acid, ammonium hydroxide, methanol and distilled water were purchased from Fisher Chemical (Nazareth, PA). Alongside with solvents, derivatizer MBTFA [N-methyl-bis(trifluoroacetamide)] was also purchased through Fisher Chemical. MSTFA (N-methyl-n-trimethylsilyl-trifluoroacetamide) derivatizer was purchased from RESTEK (Bellefonte, PA). Solid-phase extraction columns (Oasis PRiME HLB 3 cc Vac Cartridge, 60 mg) were acquired from Waters Corp (Milford, MA).

1.1 Sample Preparation

1.2 Instrumentation

The detection of analytes was performed by GC–MS/MS with XXX . Mass Hunter QQQ software was used for the data acquisition and quantification (?)

2 Results and discussion

2.1 Linearity

2.2 Precision and accuracy

2.3 Recoveries

3 Discussion and conclusion

3.1 Reliability

3.2 Practicality

4 References

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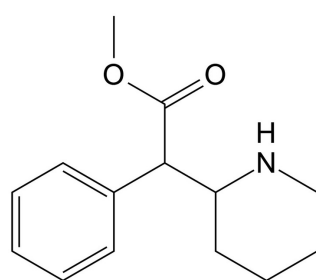
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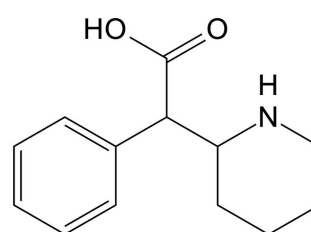
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methylphenidate



ritalinic acid

Figure 1: Chemical structures of methylphenidate (MPH) and its primary metabolite ritalinic acid (RA).

References and Notes

Acknowledgments

Funding:**Author contributions:****Competing interests:**

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Supplementary materials

Materials and Methods

Supplementary Text

Figs. S to S

Tables S to S

References (7-0)

Movie S1

Data S1

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First Author*[†], A. Scientist[†], Someone E. Else

*Corresponding author. Email: example@mail.com

[†]These authors contributed equally to this work.

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Materials and Methods

Supplementary Text

Figures S1 to S3

Tables S1 to S4

Captions for Movies S1 to S2

Captions for Data S1 to S2

Other Supplementary Materials for this manuscript:

Movies S1 to S2

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