

Supporting Information

Determination of Os and other Platinum Group Elements in Active Pharmaceutical Ingredients by ICP-MS

Vitoria Hagemann Cauduro^a, Alessandra Schneider Henn^{a,b}, Cezar Augusto Buzzi^a, Marcia Foster Mesko^c, Erico Marlon Moraes Flores^{a}*

⁹ *^a Departamento de Química, Universidade Federal de Santa Maria, 97105-900, Santa*
10 ^b Maria, RS, Brazil.

b Departamento de Química, Universidade Federal de Minas Gerais, 31270-901, Belo Horizonte, MG, Brazil.

*13 ^c Centro de Ciências Químicas, Farmacêuticas e de Alimentos, Universidade Federal de
14 Pelotas, 96160-000, Capão Do Leão, RS, Brazil.*

16 *Corresponding author: Erico Marlon Moraes Flores
17 Phone/Fax: +55 55 3220 9445.
18 E-mail: ericommf@gmail.com

21

22 **Table S1.** Studies published in the last decade on the determination of Os in pharmaceutical
23 products.

Sample preparation method	Determination Technique	Os Recovery (%)	Ref.
MIC using 400-700 mg of sample and 20% HNO ₃ as absorbing solution.	ICP-MS	> 200	Nam <i>et al.</i> , 2011 ¹
Dissolution of 250 mg of sample in 0.009 mmol L ⁻¹ KBrO ₃ in 1% HNO ₃ and 1% HCl.	ICP-MS	80-100	Van Hoecke <i>et al.</i> , 2012 ²
MAWD using 250 mg of sample and aqua regia as digestion solution.		600-700	
HPA digestion using 100 mg of sample and concentrated HNO ₃ as digestion solution.	ICP-MS	74-89	Venzago <i>et al.</i> , 2013 ³
Stabilizing solution composed of acetic acid (0.5%) containing thiourea (0.01 mol L ⁻¹) and ascorbic acid (0.1 g L ⁻¹).			
MAWD using 450 mg of sample and reverse aqua regia as digestion solution.	ICP-OES	162	Støving <i>et al.</i> , 2013 ⁴
MAWD using 100 mg of sample and concentrated HNO ₃ as digestion solution.	SF-ICP-MS e ICP-MS	102-128	Fischer <i>et al.</i> , 2014 ⁵
Stabilizing solution composed of thiourea (concentration not informed).			
MAWD-SRC using 500 mg of sample and reverse aqua regia as digestion solution.	ICP-MS	> 160	Muller <i>et al.</i> , 2015 ⁶
MAWD using 400 mg of sample and a mixture of HNO ₃ +HCl (1+1) as digestion solution.	ICP-MS	Poor	Wollein <i>et al.</i> , 2015 ⁷
Dissolution of 10 mg of sample in a solution composed of 5% HCl, 0.1% acetic acid, 0.076% thiourea and 0.01% ascorbic acid.	ICP-MS	94-97	Chahrour <i>et al.</i> , 2017 ⁸
MAWD using 200 mg of sample and a mixture of HNO ₃ (7 mL), H ₂ O ₂ (1 mL) and HCl (3 mL) as digestion solution.	USN-ICP-OES	233-281	Menoutis <i>et al.</i> , 2018 ⁹
MAWD-SRC using 500 mg of sample and 2 mol L ⁻¹ HNO ₃ as digestion solution.	ICP-OES and ICP-MS	< 60	Pinheiro <i>et al.</i> , 2019 ¹⁰
MAWD using 0.064 - 0.230 mg of sample and aqua regia as digestion solution.	ICP-MS	79-92	Gu <i>et al.</i> , 2021 ¹¹
Stabilizing solution composed of 0.01 mol L ⁻¹ thiourea.			
MAWD using 500 mg of sample and 6 mL of HNO ₃ +HCl 1+1 as digestion solution.	ICP-MS	96-103	This study
Stabilizing solution composed of 85 mmol L ⁻¹ acetic acid, 10 mmol L ⁻¹ thiourea and 0.6 mmol L ⁻¹ ascorbic acid.			

24 MIC: Microwave-induced combustion; MAWD: microwave-assisted wet digestion; HPA: high pressure ashing;
25 MAWD-SRC: MAWD with single reaction chamber.

26 **Table S2.** Operational conditions for PGEs determination by ICP-OES and ICP-MS.

Parameter	ICP-OES	ICP-MS
RF Power, W	1400	1300
Plasma gas flow rate, L min ⁻¹	15.0	15
Auxiliary gas flow rate, L min ⁻¹	0.2	1.2
Nebulizer gas flow rate, L min ⁻¹	0.7	1.02
Spray chamber	Cyclonic	Cyclonic
Nebulizer	Gencone	Concentric
Observation view	Axial	-
Sampler and skimmer cones	-	Pt
Element	Wavelength (nm)	Isotope (m/z)
Os	-	192
Ir	-	193
Pd	-	105
Pt	-	195
Rh	-	103
Ru	-	101
C	193.030	-
Y	371.029	-

27

28

29 **References**

- 30 1. K. H. Nam, R. Isensee, G. Infantino, K. Putyera, and X. Wang, *Spectroscopy*, 2011,
31 26. 36-41.
- 32 2. K. Van Hoecke, C. Catry, and F. Vanhaecke, *J. Anal. At. Spectrom.*, 2012, 27. 1909-
33 1919. <https://doi.org/10.1039/C2JA30128H>
- 34 3. C. Venzago, M. Popp, J. Kovac, and A. Kunkel, *J. Anal. At. Spectrom.*, 2013, 28.
35 1125-1129. <https://doi.org/10.1039/C3JA50040C>
- 36 4. C. Støving, H. Jensen, B. Gammelgaard, and S. Stürup, *J. Pharm. Biomed. Anal.* ,
37 2013, 84. 209-214. <https://doi.org/10.1016/j.jpba.2013.06.007>
- 38 5. L. Fischer, B. Zipfel, G. Koellensperger, J. Kovac, S. Bilz, A. Kunkel, C. Venzago,
39 and S. Hann, *J. Pharm. Biomed. Anal.* , 2014, 95. 121-129.
40 <https://doi.org/10.1016/j.jpba.2014.02.016>
- 41 6. A. L. Muller, J. S. Oliveira, P. A. Mello, E. I. Muller, and E. M. M. Flores, *Talanta*,
42 2015, 136. 161-169. <https://doi.org/10.1016/j.talanta.2014.12.023>
- 43 7. U. Wollein, B. Bauer, R. Habernegg, and N. Schramek, *Eur. J. Pharm. Sci.*, 2015,
44 77. 100-105. <https://doi.org/10.1016/j.ejps.2015.05.028>
- 45 8. O. Chahrour, J. Malone, M. Collins, V. Salmon, C. Greenan, A. Bombardier, Z. Ma,
46 and N. Dunwoody, *J. Pharm. Biomed. Anal.* , 2017, 145. 84-90.
47 <https://doi.org/10.1016/j.jpba.2017.06.045>

- 48 9. J. Menoutis, A. Parisi, and N. Verma, *J. Pharm. Biomed. Anal.* , 2018, 152. 12-16.
49 <https://doi.org/10.1016/j.jpba.2018.01.008>
- 50 10. F. C. Pinheiro, A. I. Barros, and J. A. Nóbrega, *Anal. Chim. Acta*, 2019, 1065. 1-11.
51 <https://doi.org/10.1016/j.aca.2019.03.016>
- 52 11. X. Gu, S. Zhu, L. Yan, L. Cheng, P. Zhu, and J. Zheng, *J. Anal. At. Spectrom.*, 2021,
53 36. 512-517. <https://doi.org/10.1039/D0JA00519C>
- 54