

**A highly sensitive LC-MS/MS method for determination and
quantification of a recently identified N-nitrosamine impurity in the
sitagliptin phosphate monohydrate active pharmaceutical ingredient**

Zhen Wang,^{*a} Shujun Hu,^b Xiaoying Wu,^b Zuwei He,^b Chunlong Ke,^b and Miaomiao Hu^b

^a*Department of Chemistry, Yuquan Campus, Zhejiang University, Hangzhou 310058, China. E-mail: 896698921@qq.com*

^b*Chemical Process Research and Development, Zhejiang Tianyu Pharmaceutical Co., Ltd. Taizhou, 318020, China*

Supporting information

Chromatogram and MS/MS spectra of NTTP at different collision energies. MS source parameters. Data obtained in method repeatability study.

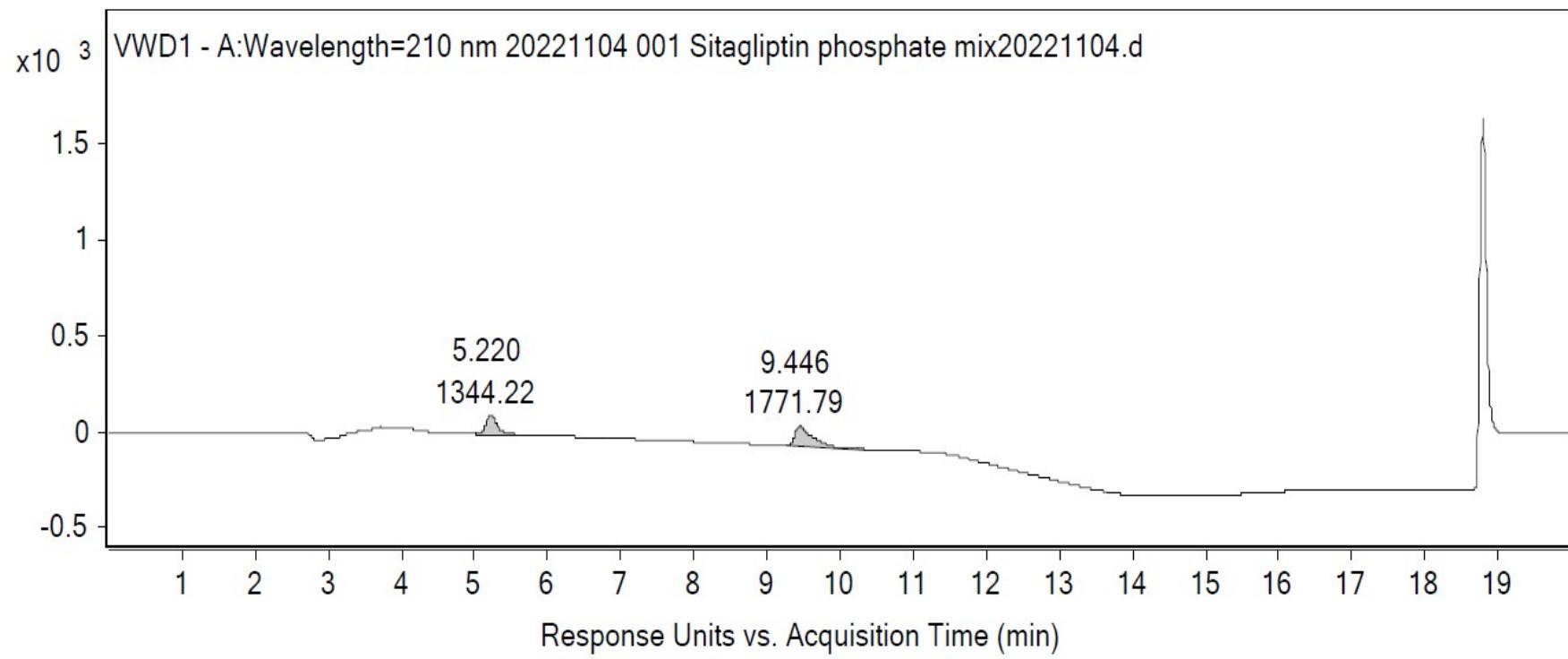


Figure S1. Chromatogram of sitagliptin phosphate monohydrate and NTTP on ZORBAX SB-C18 column

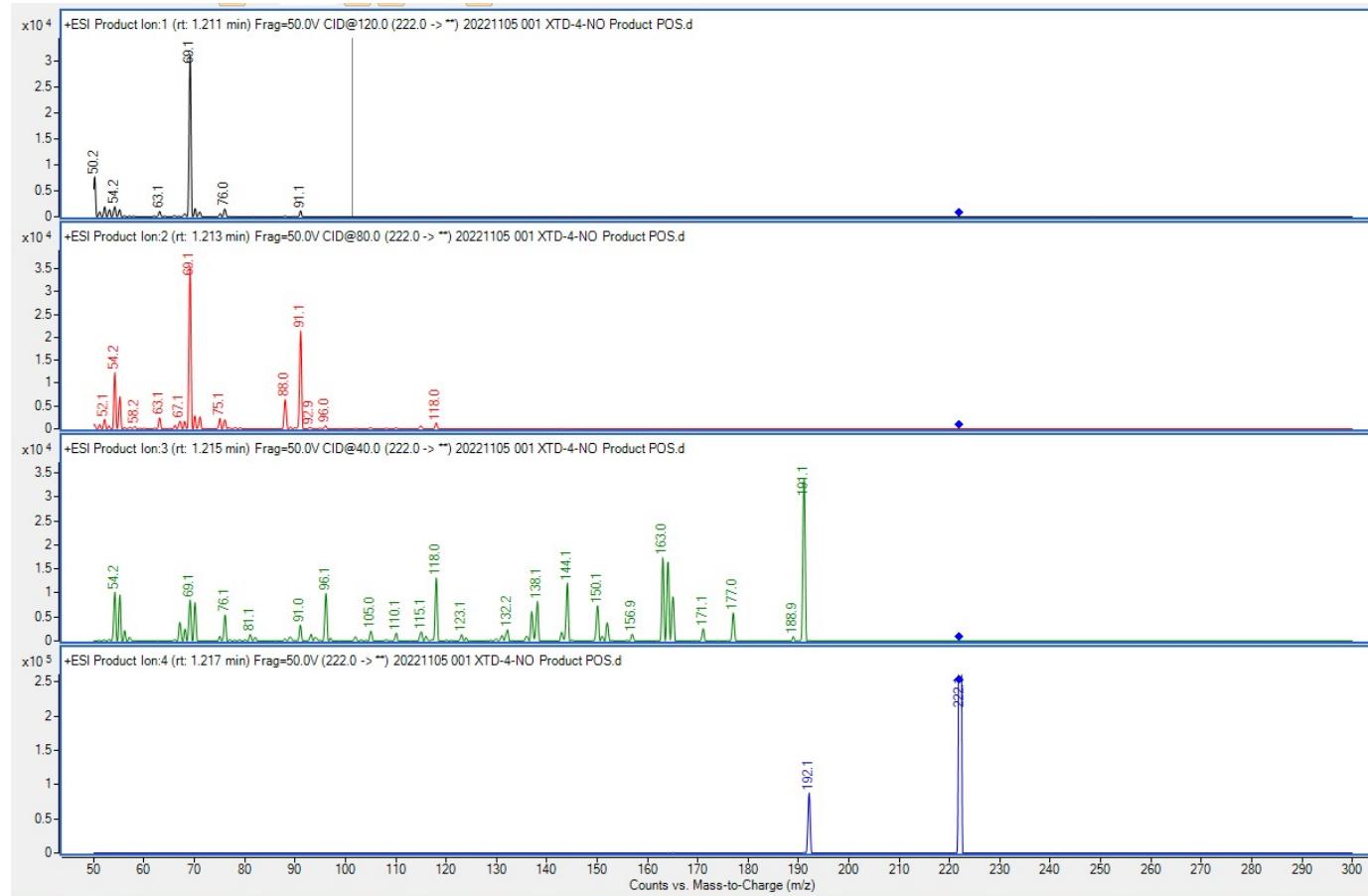


Figure S2. MS/MS spectra of NTTP at different collision energies (0, 40, 80 and 120 V)

Table S1. MS source parameters

Parametar	Value (+)
Gas temperature (°C)	300
Gas flow (l/min)	8
Nebulizer (psi)	45
Sheath Gas Heater (°C)	300
Sheath gas flow	11
Capillary (V)	3500
V Charging	500

Table S2. Data obtained in method repeatability study.

	Injection #	NTTP
Area (at 0.098 ppm)	1	661
	2	666
	3	620
	RSD %	3.89
Area (at 0.370 ppm)	1	2263
	2	2266
	3	2254
	RSD %	0.28
Area (at 0.925 ppm)	1	5067
	2	5057
	3	5036
	RSD %	0.31