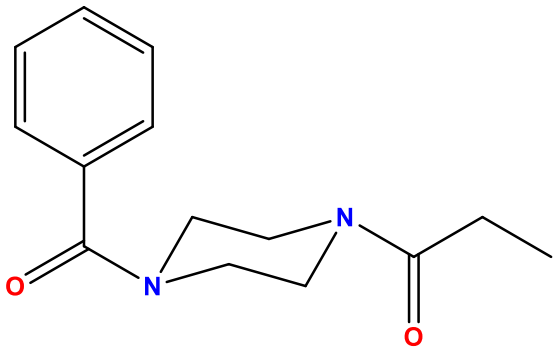
SUNIFIRAM

Analytical Report

The purpose of this document is to demonstrate the identity and high purity of **SUNIFIRAM**. For this purpose, the usual techniques and laboratory equipment in organic chemistry were implemented. The obtained experimental data can be contrasted with the online reports of other suppliers (see references).

IDENTIFICATION DATA

Structure

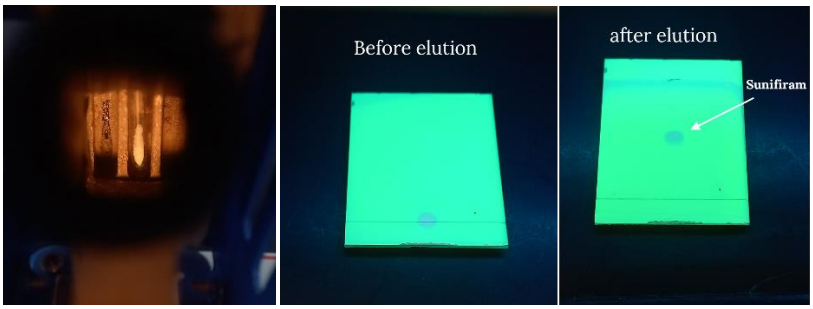


IUPAC name	1-(4-benzoylpiperazin-1-yl)propan-1-one
Common name	SUNIFIRAM (DM-235)
Chemical formula	$C_{14}H_{18}N_2O_2$
Molecular weight	246.31
CAS number	314728-85-3
Appearance	Off-white powder

LABORATORY TECHNIQUES/EQUIPMENT	
Melting point	Mel-Temp Apparatus – Electrothermal
TLC	Aluminum TLC plate, silica gel coated with fluorescent indicator F254. Ethyl Acetate as elution solvent
ATR-IR	Nicolet iS5 FTIR Spectrometer
^{1}H & ^{13}C NMR (400 MHz)	Bruker Avance III. $CDCl_3$
ESI-MS	Agilent 6410B triple quadrupole LC/MS-ESI

Melting point & TLC

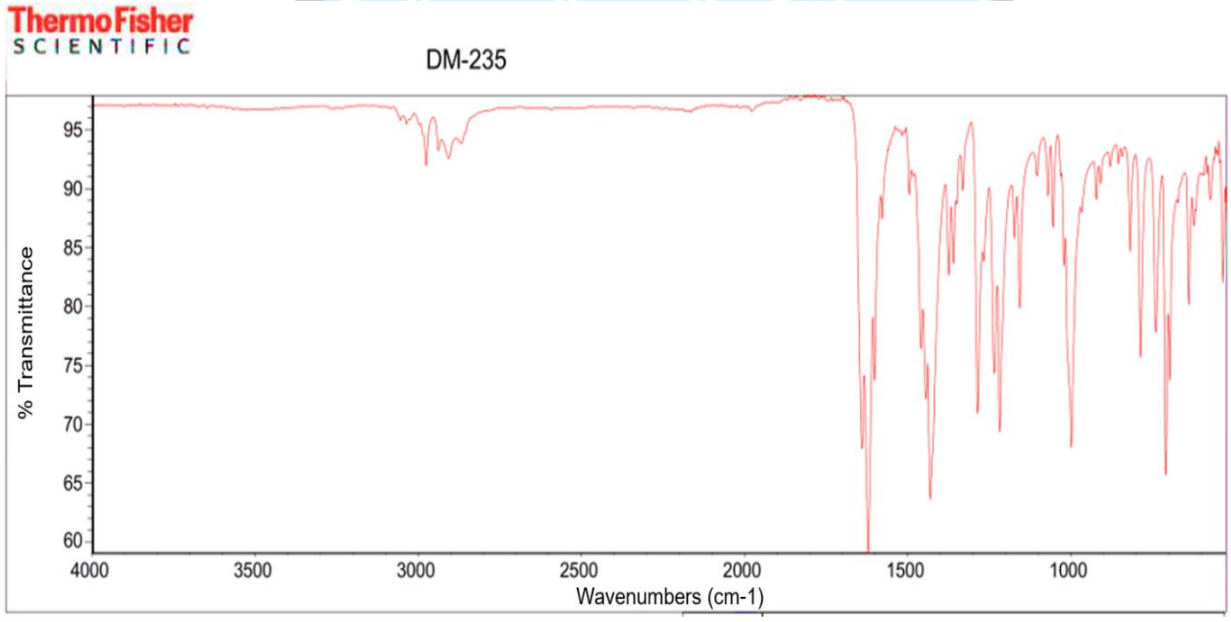
The white solid melts at 92 °C. The reported melting point by newmind [1] and LGC [2] are 93-97 °C and 95 °C, respectively. The TLC shown a defined single spot, indicative of a single component.



Melting point and TLC of sunifiram

ATR-IR

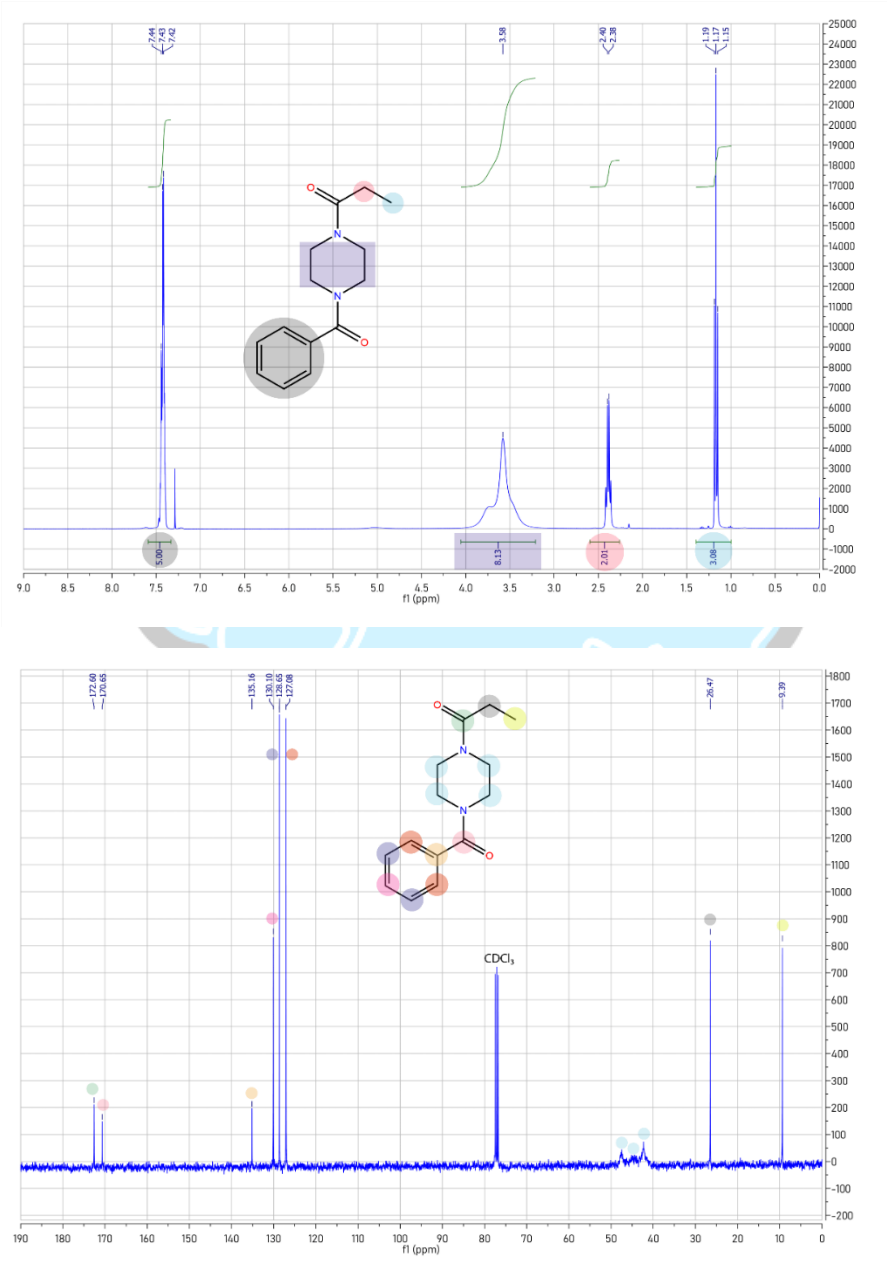
The experimental spectrum is in accordance or even identical with the reported spectrum by the analytical report of LGC [2], NFL [3] and Science.bio [4]. The spectrum displays only sunifiram signals, indicative of the identity and purity of sunifiram.



ATR-IR spectrum of sunifiram

^{1}H & ^{13}C NMR (400 MHz)

The integration values and shifts match with the chemical structure of sunifiram and correspond with the reported spectrum of LGC [2] and NFL [3]. Broadening and lack of multiplicity around 3.58 ppm are due to slow rotations about the N-COPh bonds [5]. For clarity hydrogen and carbon assignations in colours are shown. The absence of other signals in the spectra is a qualitative indicator of the purity of sunifiram.

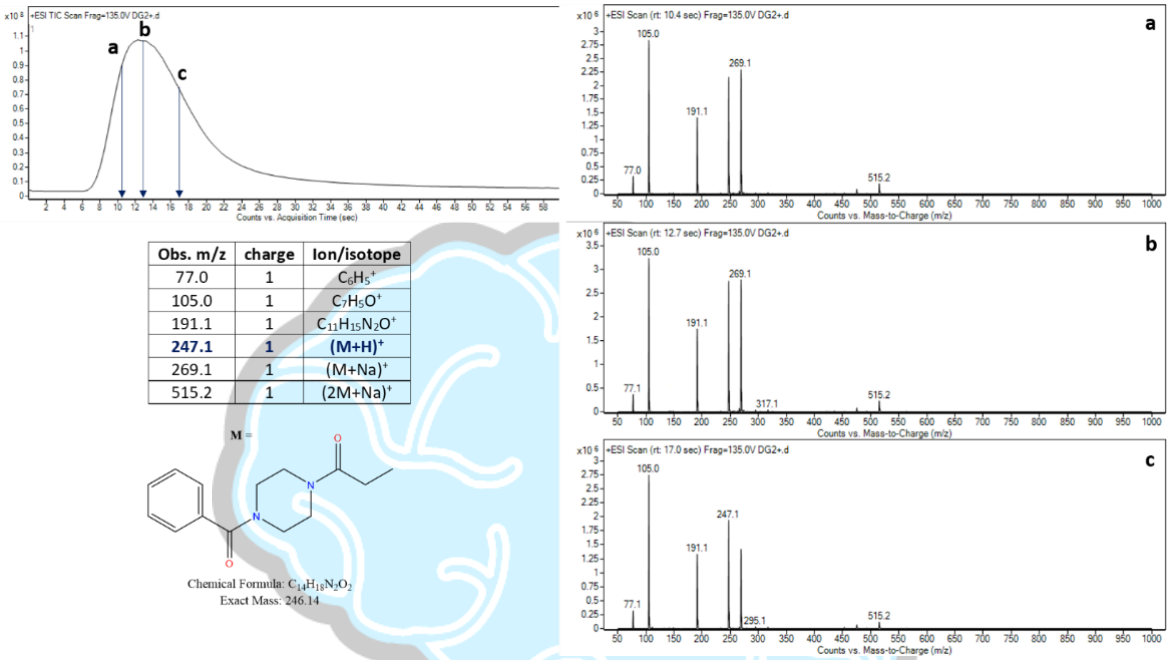


^{1}H and ^{13}C NMR spectra of sunifiram

SUNIFIRAM

ESI-MS

The $(M+H)^+$ ion (m/z = 247.1) correspond with sunifiram (M = 246.1). The proposed fragmentation mechanism of sunifiram using the ESI fragmentation rules for amides correspond with the obtained fragments in the spectra [6] . Due to the use of distilled water (not deionized) the $(M+Na)^+$ and $(2M+Na)^+$ adducts are also present [3]. As the sample was not separated trough liquid chromatography and the mass spectra were almost identical at three different times means that the sample it´s only high purity sunifiram.

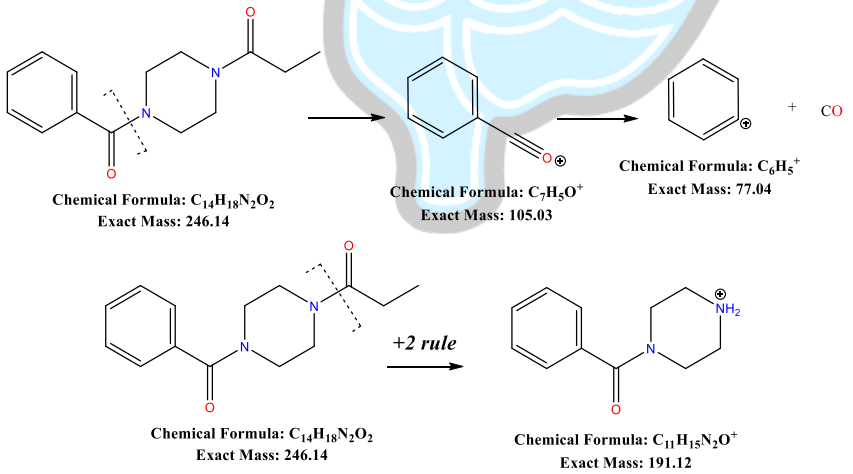


Obs. m/z	charge	Ion/isotope
77.0	1	$C_6H_5^+$
105.0	1	$C_7H_5O^+$
191.1	1	$C_{11}H_{15}N_2O^+$
247.1	**1**	**$(M+H)^+$**
269.1	1	$(M+Na)^+$
515.2	1	$(2M+Na)^+$

M =

Chemical Formula: $C_{14}H_{18}N_2O_2$
Exact Mass: 246.14

ESI-MS spectra of sunifiram.



Fragmentation mechanism of sunifiram using the ESI fragmentation rules for amides.

REFERENCES

[1] Certificate of analysis sunifiram (newmind):

https://d2jfq6df77491c.cloudfront.net/wysiwyg/lab_analysis/sunifiram.20161116.product.article.pdf

[2] Certificate of analysis sunifiram (LGC):

https://assets.lgcstandards.com/sys-master%2Fpdfs%2Fh21%2Fhe2%2F10170203570206%2FCOA_LGCFOR3274.00_ST-WB-CERT-2643847-1-1-1.PDF

[3] Analytical report of sunifiram, NFL (National Forensic Laboratory):

https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=&cad=rja&uact=8&ved=2ahUKEwj5pbHNrrfsAhVQHqwKHXSMBmoQFjAAegQIAxAC&url=https%3A%2F%2Fwww.policija.si%2Fapps%2Fnfl_response_web%2F0_Analytical_Reports_final%2FSunifiram-ID-1537-16-rpt_120816.pdf&usg=AOvVaw08-i3qqyJWPVBU9GVMSs1b

[4] ATR-IR of sunifiram (Science.bio):
https://www.dropbox.com/sh/1hzyqxu5m6bylk1/AAA2Sfx2BvN85uUuwhaDFA-ia/Sunifiram%20%28200312%29%20FT-IR%20HPLC%2005-18-2020.pdf?dl=0

[5] Petride, H., Drăghici, C., Florea, C. *et al.* RuO_4-mediated oxidation of N-benzylated tertiary amines. 3. Behaviour of 1,4-dibenzylpiperazine and its oxygenated derivatives. *cent.eur.j.chem.* **4**, 674–694 (2006). https://doi.org/10.2478/s11532-006-0039-8

[6] Avi Weissberg and Shai Dagan. Interpretation of ESI(+)-MS-MS spectra—Towards the identification of "unknowns". *International Journal of Mass Spectrometry*. 299 (2011) 158–168.https://doi.org/10.1016/j.ijms.2010.10.024.