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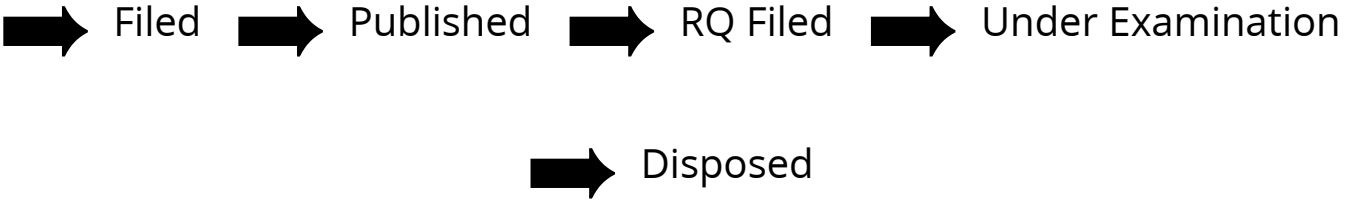
Application Details	
APPLICATION NUMBER	202121004204
APPLICATION TYPE	ORDINARY APPLICATION
DATE OF FILING	31/01/2021
APPLICANT NAME	1 . JOSHI SUMIT ASHOK 2 . Dr. SHUBHRAJIT Mantry 3 . Dr. GANESH Yogiraj Dama 4 . Dr. SHRIRAM Ramesh Pethakar
TITLE OF INVENTION	A HPLC METHOD FOR QUANTITATIVE DETERMINATION OF EMTRICITABINE IN A PHARMACEUTICAL DOSAGE FORM.
FIELD OF INVENTION	PHYSICS
E-MAIL (As Per Record)	vijay@patlex.in
ADDITIONAL-EMAIL (As Per Record)	vijay@patlex.in
E-MAIL (UPDATED Online)	
PRIORITY DATE	
REQUEST FOR EXAMINATION DATE	--
PUBLICATION DATE (U/S 11A)	12/02/2021

Application Status	
APPLICATION STATUS	Awaiting Request for Examination

			View Documents



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शासकीय जर्नल

OFFICIAL JOURNAL
OF
THE PATENT OFFICE

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पेटेंट कार्यालय का एक प्रकाशन
PUBLICATION OF THE PATENT OFFICE

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(54) Title of the invention : A HPLC METHOD FOR QUANTITATIVE DETERMINATION OF EMTRICITABINE IN A PHARMACEUTICAL DOSAGE FORM.

(51) International classification	:G01N0030020000, G01N0030340000, G01N0033180000, A23L0033180000, B01D0015160000
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1)JOSHI SUMIT ASHOK
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3)Dr. GANESH Yogiraj Dama
4)Dr. SHRIRAM Ramesh Pethakar

(57) Abstract :
The invention relates to a reverse phase isocratic Reverse phase HPLC method for the quantitation of drug Emtricitabine in a dosage form. The method comprising the steps of: combining an Emtricitabine sample with water to obtain a solution; injecting the said solution into a C18 column (250 x 4.5 mm i.d., 5µm,) fitted with guard column C18 (ODS;4x3.0 mm) at a temperature of 30°C; pumping of a mobile phase comprising a first eluent A and a second eluent B at a flow rate of: 0.8 ml/min for a run time of 10 minutes; and determining the amount of the Emtricitabine at a wavelength of 282nm with an UV detector; wherein the eluent A and the eluent B are present in an isocratic ratio of 15:85 (v/v) throughout the analysis

No. of Pages : 15 No. of Claims : 3

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मुद्रांक विक्री बाबतची नोंदवही अ. 6443/12 दि. 27/11/2020

मुद्रांक शुल्क रक्कम 500

मुद्रांक विकत घेणाऱ्याचे नाव

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मुद्रांक विकत घेणाऱ्याची सही

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FORM 26

THE PATENTS ACT, 1970

(39 of 1970)

&

THE PATENT RULES, 2003

FORM FOR AUTHORISATION OF A PATENT AGENT/OR ANY PERSON IN A MATTER OR PROCEEDING UNDER THE ACT

(See sections 127 and 132; rule 135)

Power of Attorney in the name of **Vijaykumar Shivpuje** of the address **Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra, India** in respect of the patent application filing and prosecution in India.

FORM 26

THE PATENTS ACT, 1970

(39 of 1970)

&

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FORM FOR AUTHORISATION OF A PATENT AGENT/OR

ANY PERSON IN A MATTER OR PROCEEDING UNDER THE ACT

(See sections 127 and 132; rule 135)

We

Name	Nationality	Adress
Dr. SUMIT Ashok Joshi	Indian	SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504.
Dr. SHUBHRAJIT Mantry	Indian	SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504.
Dr. GANESH Yogiraj Dama	Indian	SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504.
Dr. SHRIRAM Ramesh Pethakar	Indian	SSPMS Shramjivi College of Pharmacy, Omerga, Dist. Osmanabad, Maharashtra, India- 413606.

hereby authorize **Vijaykumar Shivpuje (IN-PA 1096)** of the address **Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra, India** to act on our behalf, as our Agent, in connection with Granted patents and pending applications or any future cases, their renewals and maintenance, objections, oppositions, rectifications, cancellations, assignments and other matters and proceedings relating thereto and to receive all notices, requisitions and communications until further notice.

We further authorize our said agents to appoint any person or persons on our behalf to do all what is necessary in the matters and proceedings.

I hereby revoke all previous authorization, if any made, in respect of, same matter or proceeding.

Dated this 30th day of Jan. 2021

Name	Signature
Dr. SUMIT Ashok Joshi	
Dr. SHUBHRAJIT Mantry	
Dr. GANESH Yogiraj Dama	
Dr. SHRIRAM Ramesh Pethakar	

To,

The Controller of Patents,

The Patent Office

At Mumbai.

FORM 1 THE PATENTS ACT, 1970 (39 of 1970) and THE PATENTS RULES, 2003 **APPLICATION FOR GRANT OF PATENT** **(See section 7, 54 & 135 and sub-rule (1) of rule 20)**	**(FOR OFFICE USE ONLY)**

	Application no:	
	Filing Date:	
	Amount of Fee Paid:	
	CBR No:	
	Signature:	
1. APPLICANT'S REFERENCE/ IDENTIFICATION NO. (AS ALLOTTED BY OFFICE)		

2. TYPE OF APPLICATION [Please tick (✓) at the appropriate category]

Ordinary (✓)		Convention ( )		PCT-NP ( )	
Divisional ( )	Patent of addition ( )	Divisional ( )	Patent of addition ( )	Divisional ( )	Patent of addition ( )

3 A. APPLICANT (S)

Name in full	**Nationality**	**Country of residence**	**Address of the applicant**	
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				Khamundi, Taluka Junnar
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			City	Pune
			State	Maharashtra
			Country	India
			Pin Code	410504
Dr. SHRIRAM Ramesh Pethakar	Indian	India	House No.	SSPMS Shramjivi College of Pharmacy, Omerga
			Street	Omerga
			City	Osmanabad
			State	Maharashtra
			Country	India
			Pin Code	413606

3 B. CATEGORY OF APPLICANT [Please tick (✓) at the appropriate category]

Natural person (✓)	Other than natural person		
	Small entity ( )	Startup ( )	Others ( )

4. INVENTORS [Please tick (✓) at the appropriate category]

Are all the inventor(s) same as the applicant(s) named above?	Yes (✓)	No ( )

If "NO", furnish the details of the inventor (s) **NA**

5. TITLE OF THE INVENTION

A HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

6. AUTHORISED REGISTERED PATENT AGENT (S)	IN/PA No.	1096
	Name	Vijaykumar Shivpuje
	Mobile No.	09768665354
7. ADDRESS FOR SERVICE OF APPLICANT IN INDIA	Name	Vijaykumar Shivpuje
	Postal address	Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra.

	Telephone No.	NA
	Mobile no.	09768665354
	Fax No.	NA
	E-mail ID	vijay@patlex.in

8. IN CASE OF APPLICATION CLAIMING PRIORITY OF APPLICATION FILED IN CONVENTION COUNTRY, PARTICULARS OF CONVENTION APPLICATION

Country	Application Number	Filing date	Name of the applicant	Title of the invention	IPC (as classified in the convention country)
N/A	N/A	N/A	N/A	N/A	N/A

9. IN CASE OF PCT NATIONAL PHASE APPLICATION, PARTICULARS OF INTERNATIONAL APPLICATION FILED UNDER PATENT CO-OPERATION TREATY (PCT)

International application number	International filing date
N/A	N/A

10. IN CASE OF DIVISIONAL APPLICATION, FILED UNDER SECTION 16, PARTICULARS OF ORIGINAL (FIRST) APPLICATION

Original (first) application No.	Date of filing of original (first) application
N/A	N/A

11. IN CASE OF PATENT OF ADDITION, FILED UNDER SECTION 54, PARTICULARS OF MAIN APPLICATION OR PATENT

Main application/patent No.	Date of filing of main application
N/A	N/A

12. DECLARATIONS

(i) Declaration by the inventor (s)

(In case the applicant is an assignee: the inventor(s) may sign herein below or the applicant may upload the assignment or enclose the assignment with this application for patent or send the assignment by post/electronic transmission duly authenticated within the prescribed period).

~~I~~/We, the above mentioned inventor(s) ~~is~~/are the true & first inventor(s) for this Invention and declare that the applicant(s) herein ~~is~~/are ~~my~~/our assignee or legal representative.

(a) Date: 31st January 2021.

(b) Signature(s):

(c) Name(s): **Dr. SUMIT Ashok Joshi** **Dr. SHUBHRAJIT Mantry**

Dr. GANESH Yogiraj Dama **Dr. SHRIRAM Ramesh Pethakar**

(ii) Declaration by the applicant(s) in the convention country N/A

(In case the applicant in India is different than the applicant in the convention country: the applicant in the convention country may sign herein below or applicant in India may upload the assignment from the applicant in the convention country or enclose the said assignment with this application for patent or send the assignment by post/electronic transmission duly authenticated within the prescribed period).

I/We, the applicant(s) in the convention country declare that the applicant(s) herein is/are my/our assignee or legal representative.

(a) Date:

(b) Signature(s):

(c) Name(s) of the signatory

(iii) Declaration by the applicant(s):

I/~~We~~, the applicant(s) hereby declare(s) that:-

- ~~I am~~/ We are in possession of the above-mentioned invention.
- The Complete/ ~~provisional~~ specification relating to the invention is filed with this application.
- ~~The invention as disclosed in the specification uses the biological material from India and the necessary permission from the competent authority shall be submitted by me/us before the grant of patent to me/us.~~
- There is no lawful ground of objection to the grant of the patent to me/us.
- ~~I am~~/ We are the true and first inventor(s).
- ~~I am~~/ We are the assignee or legal representative of true & first inventors.
- ~~The application or each of the applications, particulars of which are given in Paragraph-8 was the first application in convention country/countries in respect of my/our invention~~.
- ~~I/We claim the priority from the above mentioned application(s) filed in convention country/countries and state that no application for protection in respect of the invention had been made in a convention country before that date by me/us or by any person from which I/We derive the title.~~
- ~~My/Our application in India is based on international application under Patent Cooperation Treaty (PCT) as mentioned in Paragraph 9.~~
- ~~The application is divided out of my/our application particulars of which are given in Paragraph-10 and pray that this application may be treated as deemed to have been filed on _____ under section 16 of the Act.~~

○ ~~The said invention is an improvement in/or modification of the invention particulars of which are given in Paragraph-11.~~

13. FOLLOWING ARE THE ATTACHMENTS WITH THE APPLICATION

(a) Form 2

Item	Details	Fee	Remarks
Complete/ ~~provisional~~ specification)#	No. of pages (13)	1600	
No. of claim(s)	No, of claims (**3**) and no. of pages (**1**)		
Abstract	No. of pages (**1**)	0	
No. of drawing(s)	No. of drawings (0) and No. of pages (0)	N/A	N/A

In case of a complete specification, if the applicant desires to adopt the drawings filed with his provisional specification as the drawings or part of the drawings for the complete specification under rule 13 (4), the number of such pages filed with the provisional specification are required to be mentioned here.

(b) ~~Complete specification (in conformation with the international application)/ as amended before the International Preliminary Examination Authority (IPEA) as applicable (2 copies).~~

(c) ~~Sequence listing in electronic form~~

(d) ~~Drawings (in conformation with the international application)/ as amended before the International Preliminary Examination Authority (IPEA), as applicable (2 copies).~~

(e) ~~Priority document(s) or a request to retrieve the priority document(s) from DAS (Digital Access Service) if the applicant had already requested the office of first filing to make the priority document(s) available to DAS.~~

(f) ~~Translation of priority document/Specification/International Search Report/International Preliminary report on patentability.~~

(g) Statement and undertaking on Form 3

(h) Declaration of Inventorship on Form 5

(i) ~~Power of authority~~

(j)……

Total fee ☐ **1600** in online payment ~~cash~~/ ~~Banker's cheque/Bank Draft bearing No.…… Date…… on …… Bank.~~

~~I~~/We hereby declare that to the best of ~~my~~/our knowledge, information and belief the fact and matters slated herein are correct and ~~I~~/We request that a patent may be granted to me/us for the said invention.

Dated this 31st Day of January 2021

Signature:

Name: Digitally signed by,

Agent for the Applicant

Vijaykumar Shivpuje, IN/PA 1096,

Sri Kripa, Akshay Nagar,

Old Ausa Road, Latur, 413531,

Maharashtra, India.

To, The Controller of Patents

The Patent Office, at…**Mumbai**…

Note: -

* Repeat boxes in case of more than one entry.
* To be signed by the applicant(s) or by authorized registered patent agent otherwise where mentioned.
* Tick (✓)/ cross (x) whichever is applicable/ not applicable in paragraph-12.
* Name of the inventor and applicant should be given in full, family name in the beginning.
* Strike out the portion which is/are not applicable.
* For fee: See First Schedule;

FORM 2

THE PATENT ACT 1970

(39 of 1970)

&

The Patents Rules, 2003

COMPLETE SPECIFICATION

(See section 10 and rule13)

1. Title of the invention

A HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

2. Applicant(s)

Name	**Nationality**	**Address**
JOSHI SUMIT ASHOK	Indian	SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Pune, Maharashtra, India, 410504.
Dr. SHUBHRAJIT Mantry	Indian	SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Pune, Maharashtra, India, 410504.
Dr. GANESH Yogiraj Dama	Indian	SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Pune, Maharashtra, India, 410504.
Dr. SHRIRAM Ramesh Pethakar	Indian	SSPMS Shramjivi College of Pharmacy, Omerga, Osmanabad, Maharashtra, India, 413606.

3. Preamble to the description

The present disclosure relates to a HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form

4. DESCRIPTION (Description shall start from the next page.)

The following specification particularly describes the invention and the manner in which it is to be performed.

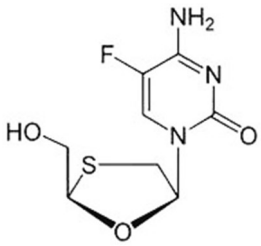
FIELD OF THE INVENTION

The present disclosure relates to an analytical method for quantitative determination of Emtricitabine in a pharmaceutical dosage form. More specifically, the present disclosure relates to a HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

BACKGROUND OF THE INVENTION

Pharmaceutical stability of a drug can be defined as the ability of a particular dosage form to maintain the physical, chemical, microbiological, therapeutic, and toxicological properties during transport, storage and usage by the patient. Chemical degradation of a drug may result in loss of its potency or an increase in toxicity. The stability of a drug in a dosage form is altered by dissolution, pulverization, or addition to other materials (excipients) or under the influence of various environmental factors such as humidity, temperature, and light. However, clinical use of a medicine is unacceptable if the amount of degradation is relatively high. Hence, there is a need to develop and validate simple and effective methods for testing stability or the quantitative determination of a drug in a dosage form that can serve as tool for quality control studies in pharmaceutical industries and drug testing laboratories.

Emtricitabine (EMC) is a synthetic deoxycitidine analogue and has been approved as an anti-retroviral agent in the treatment of HIV-type 1 infections. The chemical name of the EMC is 5-fluoro-1-[(2R,5S)-2 (hydroxymethyl)-1,3-oxathiolan-5-yl] cytosine or 4-amino-5-fluoro-1-[(2R, 5S)-2-(hydroxymthyl)-1, 3-oxathiolan-5-yl] pyrimidin-2-one. It is a nucleoside reverse transcriptase inhibitor that competitively inhibits the HIV reverse transcriptase enzyme and blocks the HIV replication.



Chemical structure of Emtricitabine

In the past, chromatography-based methods have been used for the determination of Emtricitabine in biological fluids along with other anti-retroviral drugs. Some of the HPLC

based methods that are known for quantitative estimation of Emtricitabine use gradient mode wherein the ratio of eluents of mobile phase vary during the course of analysis. There are few literatures mentioning HPLC based methods for quantitative determination of Emtricitabine in dosage forms (Der Pharma Chemica, 2010, 2(2): 281-285; Journal of Taibah university for science,2019, vol. 13, no. 1, 1137–1146; Asian Journal of Chemistry Vol. 21, No. 8 (2009), 5979-5983).

However, there is still a need to develop a much better (rapid, stable, cost effective and robust) high performance liquid chromatographic method for quantitative estimation of Emtricitabine in various dosage form.

The present invention provides a simple, precise and accurate HPLC method in isocratic mode for the estimation of emtricitabine in bulk drug samples and in pharmaceutical dosage form.

OBJECT OF THE INVENTION

The main object of the present invention is to provide an analytical method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

It is another object of the present invention to provide a HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

It is yet another object of the present invention to provide a HPLC method suitable for stability and quality control studies of Emtricitabine in a pharmaceutical dosage form.

It is yet another object of the present invention to provide a simple, precise, accurate, rapid and cost-effective method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

SUMMARY OF THE INVENTION

The present disclosure relates to a HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form, the method comprising the steps of:

-combining an Emtricitabine sample with water to obtain a solution;

-injecting the said solution into a C18 column (250 x 4.5 mm i.d., 5µm,) fitted with guard column C18 (ODS; 4x3.0 mm) at a temperature of 30°C;

-pumping of a mobile phase comprising a first eluent A and a second eluent B at a flow rate of: 0.8 ml/min for a run time of 10 minutes; and

-determining the amount of the Emtricitabine at a wavelength of 282nm with an UV detector;

wherein the eluent A and the eluent B are present in a isocratic ratio of 15:85 (v/v) throughout the analysis.

DETAILED DESCRIPTION

The present disclosure relates to an analytical method for quantitative determination of Emtricitabine in a pharmaceutical dosage form. More specifically, the present disclosure relates to a HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

1. Explanation of Terms

In order to provide a clear and consistent understanding of the terms used in the present specification, a number of definitions are provided below. Moreover, unless defined otherwise, all technical and scientific terms as used herein have the same meaning as commonly understood to one of ordinary skill in the art to which this invention pertains.

As used herein, the term "about" means that the numerical value is approximate and small variations would not significantly affect the practice of the disclosed embodiments. Where a numerical limitation is used, unless indicated otherwise by the context, "about" means the numerical value can vary by ±10% and remain within the scope of the disclosed embodiments.

The use of the terms "a" and "an" and "the" and similar referents in the context of describing the elements (especially in the context of the following claims) are to be construed to cover both the singular and the plural, unless otherwise indicated herein or clearly contradicted by context. The terms "comprising," "having," "including," and "containing" are to be construed as open-ended terms (i.e., meaning "including, but not limited to,") unless otherwise noted. Recitation of ranges of values herein are merely intended to serve as a shorthand method of referring individually to each separate value falling within the range, unless otherwise indicated herein, and each separate value is incorporated into the specification as if it were individually recited herein. All methods described herein can be performed in any suitable order unless otherwise indicated herein or otherwise clearly

contradicted by context. The use of any and all examples, or exemplary language (e.g., "such as") provided herein, is intended merely to better illuminate the embodiments and does not pose a limitation on the scope of the claims unless otherwise stated. No language in the specification should be construed as indicating any non-claimed element as essential to the practice of the invention.

The term "HPLC", as used herein, is used to refer to High-performance liquid chromatography, a technique in analytical chemistry used to separate, identify, and quantify drug in a dosage form.

The term "Pharmaceutical dosage Form ", as used herein, refers to a physical form of a dose of a chemical compound used as a drug or medication intended for administration or consumption.

The term "isocratic", as used herein, refers to maintenance of constant or same ratio of eluents of mobile phase throughout the analysis.

The term "Limit of Detection", as used herein, refers to the smallest amount or concentration of the analyte in the test sample that can be reliably distinguished from zero.

The term “Limit of Quantitation” as used herein, refers to the lowest concentration of the analyte that can be determined with an acceptable repeatability and trueness.

2. Embodiments

Embodiments of this invention are described herein.

The present disclosure relates to a HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form, the method comprising the steps of:

-combining an Emtricitabine sample with water to obtain a solution;

-injecting the said solution into a C18 column (250 x 4.5 mm i.d., 5µm,) fitted with guard column C18 (ODS; 4x3.0 mm) at a temperature of 30°C;

-pumping of a mobile phase comprising a first eluent A and a second eluent B at a flow rate of: 0.8 ml/min for a run time of 10 minutes; and

-determining the amount of the Emtricitabine at a wavelength of 282nm with an UV detector;

wherein the eluent A and the eluent B are present in an isocratic ratio of 15:85 (v/v) throughout the analysis.

In accordance with another embodiment, the eluent A is Acetonitrile and eluent B is water.

In accordance with an embodiment, the pharmaceutical dosage form is selected from the group consisting of capsule, tablet, pellet and pill.

The present invention is now illustrated by a non-limiting example.

3. Examples

The analysis was carried out by High Performance Liquid Chromatography as described below:

Equipment details:

Shimadzu Prominence – *i* series LC-2030c 3D plus
Software: Lab solutions Version 5.97 SP1
Column: Shim-pack GIST C18 (250 x 4.5 mm i.d., 5µm)

HPLC conditions

Mobile phase: Acetonitrile: Water 15:85 (v/v)
Flow rate: 0.8 ml/min
Temperature: 30 °C

Method of analysis

The chromatographic separation of the Emtricitabine was carried out using a Shim-pack GIST C18 column (250 x 4.5 mm i.d., 5µm,) fitted with guard column C18 (ODS;4x3.0 mm i.d., Phenomenex, USA). The mobile phase consists of a combination of acetonitrile and water in the proportions of 15:85 v/v, eluted at the flow rate of 0.8 mL/min. The UV detection was done at 282 nm, column temperature was maintained at 30 °C, and the injection volume of each sample was 10 µL. The total chromatographic run time was 10 min.

Method validation

The method was validated according to ICH guidelines in terms of precision, robustness, Limit of detection (LOD), Limit of Quantitation (LOQ) and accuracy.

System suitability

The system suitability test was carried out using a freshly prepared standard stock solution of Emtricitabine to check various parameters. System suitability results are as follows.

Table1

System suitability test parameters (n=6)

Compound	Retention time ($t_{R,}$ min)	RSD % of retention time	Peak area	RSD % of peak area	Theoretical plates (N)	Tailing factor (T)
Emtricitabine	5.39	0.08	144084	0.04	8986	1.21

n = number of replicates; RSD = relative standard deviation

Precision

Precision was studied to find out intra and inter day variations in the test methods of emtricitabine at 3 different concentration levels, three times same day and different day respectively. The %RSD was calculated which should be less than 2%.

Table 2

Intraday and interday precision of Emtricitabine

Analyte	Content (µg/mL)	Intraday (n = 6)		Inter day (n = 3)					
				Day 1		Day 2		Day 3	
		Found (µg/mL)	% RSD	Found (µg/mL)	% RSD	Found (µg/mL)	% RSD	Found (µg/mL)	% RSD
Emtricitabine	2.00	2.05	0.16	2.05	0.14	2.05	0.05	2.05	0.12
	6.00	6.20	0.07	6.20	0.06	6.17	0.07	6.17	0.02
	10.00	10.33	0.09	10.33	0.04	10.32	0.04	10.33	0.05

n = number of replicates; RSD = relative standard deviation

Accuracy

Accuracy was determined by recovery study of emtricitabine. A known amount of working standard

emtricitabine was added into pre-analysed sample and subjected to the proposed HPLC method.

Table 3

Recovery studies of Emtricitabine (n = 3).

Compound	Contents µg mL^{-1}	Quantity added (µg)	Theoretical amount (µg)	Recovered amount (µg)	Recovery (%)	% RSD
Emtricitabine	4.30	2.09	6.39	5.96	100.30	0.08
	4.30	4.29	8.59	7.84	100.13	0.01
	4.30	6.41	10.71	9.54	100.10	0.03

n = number of replicates; RSD = relative standard deviation

Robustness

This was done by small deliberate changes in the chromatographic conditions at 3 different levels -1, 0, +1 and retention time of the drug was noted. The factors selected were flow rate, column temperature and percentage of Acetonitrile in the mobile phase. The results were indicated that the selected factors remained unaffected by small variations of these parameters.

Table 4

Results of robustness study (n = 6).

Parameter	Modification	Emtricitabine				
		% RSD of peak area	% RSD of retention time	Theoretical plates (N)	Tailing factor (T)	Percentage recovery
Flow rate	0.7 mL/min	0.04	0.07	10183	1.19	114.00
	Optimized	0.07	0.28	9073	1.20	100.00
	0.9 mL/min	0.01	0.10	8288	1.20	89.02
Mobile phase composition	14% ACN	0.07	0.10	9342	1.19	100.34
	Optimized	0.07	0.28	9.73	1.20	100.00
	16 % ACN	0.06	0.11	8819	1.20	101.04
Column temperature	29 °C	0.02	0.02	9122	1.19	99.57
	Optimized	0.07	0.28	9073	1.20	100.00
	31 °C	0.10	0.09	9241	1.19	99.62

n = number of replicates; RSD = relative standard deviation

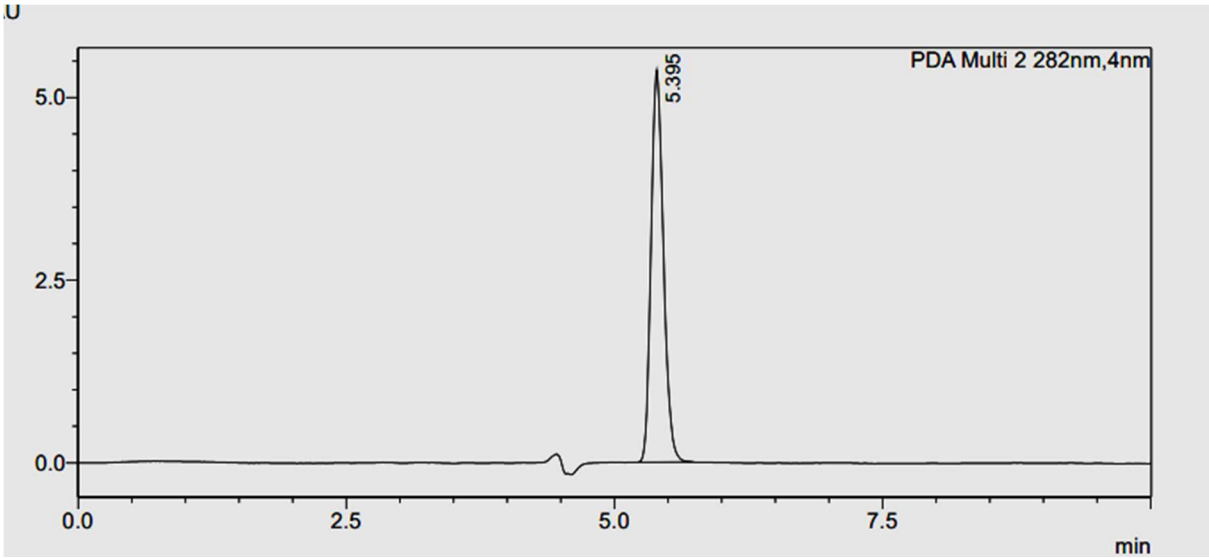
Limit of Detection and limit of Quantification

Limit of detection and limit of quantitation were calculated by the method which was based common approach which is to compare measured signals from samples with known low concentrations of analyte with those of blank samples, the minimum concentration at which the analyte can be reliably detected is established.

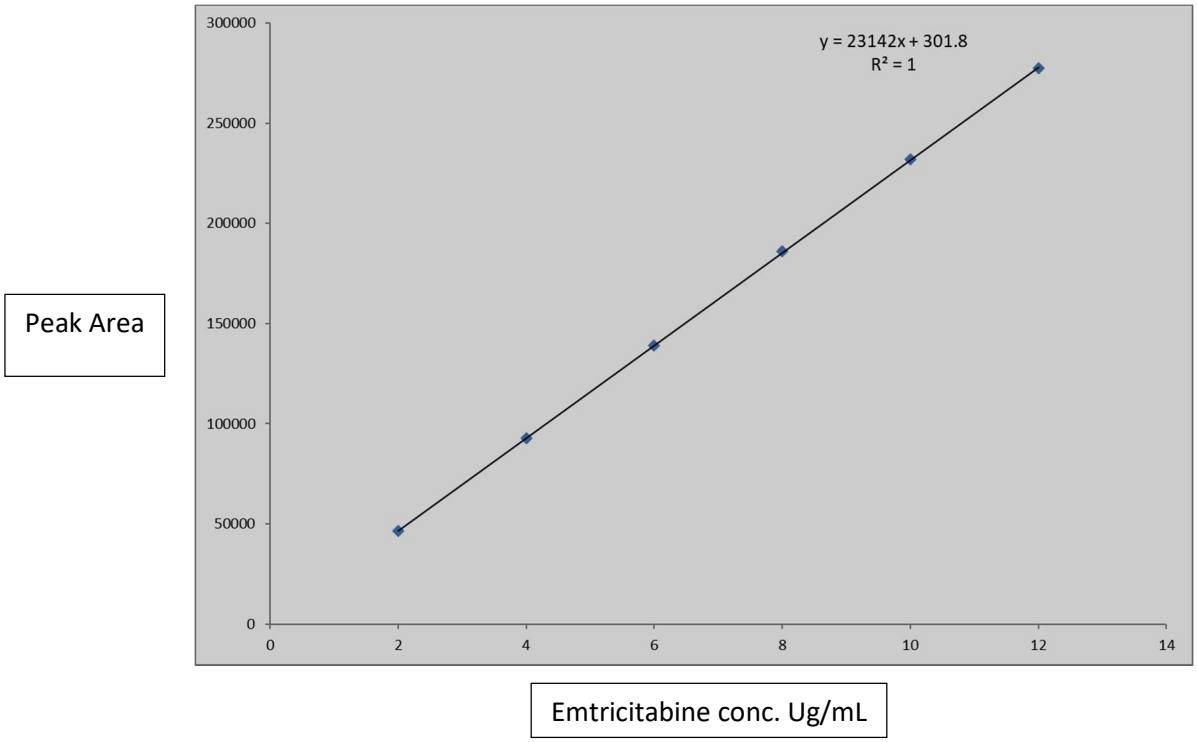
Limit of detection (LOD) and limit of quantitation (LOQ) were found to be 0.06904 ug/mL and 0.209212 ug/mL respectively

Analytical HPLC method was developed and validated for the determination of Emtricitabine in capsule dosage form. The developed method was found to be specific, accurate, precise and robust for its intended use.

Chromatogram of Emtricitabine standard



Calibration curve for linearity data



We claim:

1. A HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form, the method comprising the steps of:
-combining an Emtricitabine sample with water to obtain a solution;
-injecting the said solution into a C18 column (250 x 4.5 mm i.d., 5µm,) fitted with guard column C18 (ODS;4x3.0 mm) at a temperature of 30°C;
-pumping of a mobile phase comprising a first eluent A and a second eluent B at a flow rate of: 0.8 ml/min for a run time of 10 minutes; and
-determining the amount of the Emtricitabine at a wavelength of 282nm with an UV detector;
wherein the eluent A and the eluent B are present in an isocratic ratio of 15:85 (v/v) throughout the analysis.

2. The method as claimed in claim1, wherein the eluent A is Acetonitrile and eluent B is water.

3. The method as claimed in claim1, wherein the pharmaceutical dosage form is a tablet, capsule, pill or pellet.

Dated this 31st day of January 2021

Digitally signed by,
Agent for the Applicant
Vijaykumar Shivpuje, IN/PA 1096,
Sri Kripa, Akshay Nagar,
Old Ausa Road, Latur, 413531,
Maharashtra, India.

ABSTRACT

A HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form

The invention relates to a reverse phase isocratic Reverse phase HPLC method for the quantitation of drug Emtricitabine in a dosage form. The method comprising the steps of: combining an Emtricitabine sample with water to obtain a solution; injecting the said solution into a C18 column (250 x 4.5 mm i.d., 5µm,) fitted with guard column C18 (ODS;4x3.0 mm) at a temperature of 30°C; pumping of a mobile phase comprising a first eluent A and a second eluent B at a flow rate of: 0.8 ml/min for a run time of 10 minutes; and determining the amount of the Emtricitabine at a wavelength of 282nm with an UV detector; wherein the eluent A and the eluent B are present in an isocratic ratio of 15:85 (v/v) throughout the analysis

FORM 5

THE PATENTS ACT, 1970

(39 OF 1970) &

The Patents Rules, 2003

DECLARATION AS TO INVENTORSHIP

[See section 10 (6) and rule 13(6)]

1. NAME OF THE APPLICANT (S)	1. **Dr. SUMIT Ashok Joshi** 2. **Dr. SHUBHRAJIT Mantry** 3. **Dr. GANESH Yogiraj Dama** 4. **Dr. SHRIRAM Ramesh Pethakar**

hereby declare that the true and first inventor(s) of the invention disclosed in the complete specification filed in pursuance of ~~my~~/our application numbered _____ dated_____ ~~is~~/are **Dr. SUMIT Ashok Joshi, Dr. SHUBHRAJIT Mantry, Dr. GANESH Yogiraj Dama, and Dr. SHRIRAM Ramesh Pethakar.**

2. INVENTORS

INVENTOR (1)

(a) NAME: **Dr. SUMIT Ashok Joshi**

(B) NATIONALITY: Indian

(C) ADDRESS: SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504.

Dated this 31st January 2021

Signature: -

Name of the signatory: - **Dr. SUMIT Ashok Joshi**

INVENTOR (2)

(a) NAME: **Dr. SHUBHRAJIT Mantry**

(B) NATIONALITY: Indian

(C) ADDRESS: SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504.

Dated this 31st January 2021

Signature: -

Name of the signatory: - **Dr. SHUBHRAJIT Mantry**

INVENTOR (3)

(a) NAME: **Dr. GANESH Yogiraj Dama**

(B) NATIONALITY: Indian

(C) ADDRESS: SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504.

Dated this 31st January 2021

Signature: -

Name of the signatory: - **Dr. GANESH Yogiraj Dama**

INVENTOR (4)

(a) NAME: **Dr. SHRIRAM Ramesh Pethakar**

(B) NATIONALITY: Indian

(C) ADDRESS: SSPMS Shramjivi College of Pharmacy, Omerga, Dist. Osmanabad, Maharashtra, India- 413606.

Dated this 31st January 2021

Signature: -

Name of the signatory: - **Dr. SHRIRAM Ramesh Pethakar**

3. DECLARATION TO BE GIVEN WHEN THE APPLICATION IN INDIA IS FILED BY THE APPLICANT(S) IN THE CONVENTION COUNTRY: -

Not applicable.

4. STATEMENT (to be signed by the additional inventor(s) not mentioned in the application form)

~~I~~/We assent to the invention referred to in the above declaration, being included in the complete specification filed in pursuance of the stated application.

Dated this 31st January 2021.

Signature of the additional inventor(s) : -

Name: -

To, The Controller of Patent,

The Patent Office, at…**Mumbai**…

FORM 1 THE PATENTS ACT, 1970 (39 of 1970) and THE PATENTS RULES, 2003 **APPLICATION FOR GRANT OF PATENT** **(See section 7, 54 & 135 and sub-rule (1) of rule 20)**	**(FOR OFFICE USE ONLY)**

	Application no:	
	Filing Date:	
	Amount of Fee Paid:	
	CBR No:	
	Signature:	
1. APPLICANT'S REFERENCE/ IDENTIFICATION NO. (AS ALLOTTED BY OFFICE)		

2. TYPE OF APPLICATION [Please tick (✓) at the appropriate category]

Ordinary (✓)		Convention ( )		PCT-NP ( )	
Divisional ( )	Patent of addition ( )	Divisional ( )	Patent of addition ( )	Divisional ( )	Patent of addition ( )

3 A. APPLICANT (S)

Name in full	**Nationality**	**Country of residence**	**Address of the applicant**	
Dr. SUMIT Ashok Joshi	Indian	India	House No.	SGMSPM's Sharadchandra Pawar College of Pharmacy
			Street	At Dumbarwadi, Post Khamundi, Taluka Junnar
			City	Pune
			State	Maharashtra
			Country	India
			Pin Code	410504
Dr. SHUBHRAJIT Mantry	Indian	India	House No.	SGMSPM's Sharadchandra Pawar College of Pharmacy
			Street	At Dumbarwadi, Post

				Khamundi, Taluka Junnar
			City	Pune
			State	Maharashtra
			Country	India
			Pin Code	410504
Dr. GANESH Yogiraj Dama	Indian	India	House No.	SGMSPM's Sharadchandra Pawar College of Pharmacy
			Street	At Dumbarwadi, Post Khamundi, Taluka Junnar
			City	Pune
			State	Maharashtra
			Country	India
			Pin Code	410504
Dr. SHRIRAM Ramesh Pethakar	Indian	India	House No.	SSPMS Shramjivi College of Pharmacy, Omerga
			Street	Omerga
			City	Osmanabad
			State	Maharashtra
			Country	India
			Pin Code	413606

3 B. CATEGORY OF APPLICANT [Please tick (✓) at the appropriate category]

Natural person (✓)	Other than natural person		
	Small entity ( )	Startup ( )	Others ( )

4. INVENTORS [Please tick (✓) at the appropriate category]

Are all the inventor(s) same as the applicant(s) named above?	Yes (✓)	No ( )

If "NO", furnish the details of the inventor (s) **NA**

5. TITLE OF THE INVENTION

A HPLC method for quantitative determination of Emtricitabine in a pharmaceutical dosage form.

6. AUTHORISED REGISTERED PATENT AGENT (S)	IN/PA No.	1096
	Name	Vijaykumar Shivpuje
	Mobile No.	09768665354
7. ADDRESS FOR SERVICE OF APPLICANT IN INDIA	Name	Vijaykumar Shivpuje
	Postal address	Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra.

	Telephone No.	NA
	Mobile no.	09768665354
	Fax No.	NA
	E-mail ID	vijay@patlex.in

8. IN CASE OF APPLICATION CLAIMING PRIORITY OF APPLICATION FILED IN CONVENTION COUNTRY, PARTICULARS OF CONVENTION APPLICATION

Country	Application Number	Filing date	Name of the applicant	Title of the invention	IPC (as classified in the convention country)
N/A	N/A	N/A	N/A	N/A	N/A

9. IN CASE OF PCT NATIONAL PHASE APPLICATION, PARTICULARS OF INTERNATIONAL APPLICATION FILED UNDER PATENT CO-OPERATION TREATY (PCT)

International application number	International filing date
N/A	N/A

10. IN CASE OF DIVISIONAL APPLICATION, FILED UNDER SECTION 16, PARTICULARS OF ORIGINAL (FIRST) APPLICATION

Original (first) application No.	Date of filing of original (first) application
N/A	N/A

11. IN CASE OF PATENT OF ADDITION, FILED UNDER SECTION 54, PARTICULARS OF MAIN APPLICATION OR PATENT

Main application/patent No.	Date of filing of main application
N/A	N/A

12. DECLARATIONS

(i) Declaration by the inventor (s)

(In case the applicant is an assignee: the inventor(s) may sign herein below or the applicant may upload the assignment or enclose the assignment with this application for patent or send the assignment by post/electronic transmission duly authenticated within the prescribed period).

~~I~~/We, the above mentioned inventor(s) ~~is~~/are the true & first inventor(s) for this Invention and declare that the applicant(s) herein ~~is~~/are ~~my~~/our assignee or legal representative.

(a) Date: 31st January 2021.

(b) Signature(s):

(c) Name(s): **Dr. SUMIT Ashok Joshi** **Dr. SHUBHRAJIT Mantry**

Dr. GANESH Yogiraj Dama **Dr. SHRIRAM Ramesh Pethakar**

(ii) Declaration by the applicant(s) in the convention country N/A

(In case the applicant in India is different than the applicant in the convention country: the applicant in the convention country may sign herein below or applicant in India may upload the assignment from the

applicant in the convention country or enclose the said assignment with this application for patent or send the assignment by post/electronic transmission duly authenticated within the prescribed period).

I/We, the applicant(s) in the convention country declare that the applicant(s) herein is/are my/our assignee or legal representative.

(a) Date:

(b) Signature(s):

(c) Name(s) of the signatory

(iii) Declaration by the applicant(s):

I/~~We~~, the applicant(s) hereby declare(s) that:-

- ~~I am~~/ We are in possession of the above-mentioned invention.
- The Complete/ ~~provisional~~ specification relating to the invention is filed with this application.
- ~~The invention as disclosed in the specification uses the biological material from India and the necessary permission from the competent authority shall be submitted by me/us before the grant of patent to me/us.~~
- There is no lawful ground of objection to the grant of the patent to me/us.
- ~~I am~~/ We are the true and first inventor(s).
- ~~I am~~/ We are the assignee or legal representative of true & first inventors.
- ~~The application or each of the applications, particulars of which are given in Paragraph-8 was the first application in convention country/countries in respect of my/our invention~~.
- ~~I/We claim the priority from the above mentioned application(s) filed in convention country/countries and state that no application for protection in respect of the invention had been made in a convention country before that date by me/us or by any person from which I/We derive the title.~~
- ~~My/Our application in India is based on international application under Patent Cooperation Treaty (PCT) as mentioned in Paragraph-9.~~
- ~~The application is divided out of my/our application particulars of which are given in Paragraph-10 and pray that this application may be treated as deemed to have been filed on _____ under section 16 of the Act.~~
- ~~The said invention is an improvement in/or modification of the invention particulars of which are given in Paragraph-11.~~

13. FOLLOWING ARE THE ATTACHMENTS WITH THE APPLICATION

(a) Form 2

Item	Details	Fee	Remarks
Complete/ ~~provisional~~ specification)#	No. of pages (13)	1750	
No. of claim(s)	No, of claims (**3**) and no. of pages (**1**)		

Abstract	No. of pages (**1**)	0	
No. of drawing(s)	No. of drawings (0) and No. of pages (0)	N/A	N/A

In case of a complete specification, if the applicant desires to adopt the drawings filed with his provisional specification as the drawings or part of the drawings for the complete specification under rule 13 (4), the number of such pages filed with the provisional specification are required to be mentioned here.

(b) ~~Complete specification (in conformation with the international application)/ as amended before the International Preliminary Examination Authority (IPEA) as applicable (2 copies).~~

(c) ~~Sequence listing in electronic form~~

(d) ~~Drawings (in conformation with the international application)/ as amended before the International Preliminary Examination Authority (IPEA), as applicable (2 copies).~~

(e) ~~Priority document(s) or a request to retrieve the priority document(s) from DAS (Digital Access Service) if the applicant had already requested the office of first filing to make the priority document(s) available to DAS.~~

(f) ~~Translation of priority document/Specification/International Search Report/International Preliminary report on patentability.~~

(g) Statement and undertaking on Form 3

(h) Declaration of Inventorship on Form 5

(i) ~~Power of authority~~

(j)……

Total fee ☐ **1600** in online payment ~~cash~~/ ~~Banker's cheque/Bank Draft bearing No……. Date…… on …… Bank.~~

~~I~~/We hereby declare that to the best of ~~my~~/our knowledge, information and belief the fact and matters slated herein are correct and ~~I~~/We request that a patent may be granted to me/us for the said invention.

Dated this 31st Day of January 2021

Signature:

Name: Digitally signed by,

Agent for the Applicant

Vijaykumar Shivpuje, IN/PA 1096,

Sri Kripa, Akshay Nagar,

Old Ausa Road, Latur, 413531,

Maharashtra, India.

To, The Controller of Patents

The Patent Office, at…**Mumbai**…

Note: -

* Repeat boxes in case of more than one entry.
* To be signed by the applicant(s) or by authorized registered patent agent otherwise where mentioned.
* Tick (✓)/ cross (x) whichever is applicable/ not applicable in paragraph-12.
* Name of the inventor and applicant should be given in full, family name in the beginning.
* Strike out the portion which is/are not applicable.
* For fee: See First Schedule;

FORM 9

THE PATENTS ACT, 1970

(39 OF 1970)

&

The Patents Rules, 2003

REQUEST FOR PUBLICATION

[See section 11A (2); rule 24A]

---

1. Name, address and Nationality of the applicant (s)	~~I~~/ We 1. JOSHI SUMIT ASHOK 2. Dr. SHUBHRAJIT Mantry 3. Dr. GANESH Yogiraj Dama 4. Dr. SHRIRAM Ramesh Pethakar hereby request for early publication of ~~my~~/our application for patent No. _____ dated_____ under section 11A(2) of the act.

Dated this 31st day of January 2021.

2. To be signed by the applicant or his authorized registered patent agent. Name of the person who has signed	Digitally signed by, **Agent for the Applicant** Vijaykumar Shivpuje, IN/PA 1096, Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra, India. To, The Controller of Patents, The Patent Office, At **Mumbai.**

FORM 9

THE PATENTS ACT, 1970

(39 OF 1970)

&

The Patents Rules, 2003

REQUEST FOR PUBLICATION

[See section 11A (2); rule 24A]

---

1. Name, address and Nationality of the applicant (s)	~~I~~/ We 1. JOSHI SUMIT ASHOK 2. Dr. SHUBHRAJIT Mantry 3. Dr. GANESH Yogiraj Dama 4. Dr. SHRIRAM Ramesh Pethakar hereby request for early publication of ~~my~~/our application for patent No. _____ dated_____ under section 11A(2) of the act.

Dated this 31st day of January 2021.

2. To be signed by the applicant or his authorized registered patent agent. Name of the person who has signed	Digitally signed by, **Agent for the Applicant** Vijaykumar Shivpuje, IN/PA 1096, Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra, India. To, The Controller of Patents, The Patent Office, At **Mumbai.**

FORM 3 **THE PATENTS ACT, 1970** **(39 OF 1970)** **and** **THE PATENTS RULES, 2003** **STATEMENT AND UNDERTAKING UNDER SECTION 8** **[See section 8, rule 12]**	
1. Name of the applicant (s),	~~I~~/We 1. **Dr. SUMIT Ashok Joshi** SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504. 2. **Dr. SHUBHRAJIT Mantry** SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504. 3. **Dr. GANESH Yogiraj Dama** SGMSPM's Sharadchandra Pawar College of Pharmacy, At Dumbarwadi, Post Khamundi, Taluka Junnar, Dist Pune, Maharashtra, India- 410504. 4. **Dr. SHRIRAM Ramesh Pethakar** SSPMS Shramjivi College of Pharmacy, Omerga, Dist. Osmanabad, Maharashtra, India- 413606. hereby declare,
2. Name, address and nationality of the joint applicant	(i) that ~~I~~/We have not made any application for the same/substantially the same invention outside India. **Or** (ii) ~~that I/We who have made this application No.……dated ……alone/jointly with……made for the same/substantially same invention, application(s) for patent~~

		~~in the other countries, the particulars of which are given below:~~			
Name of the country	Date of application	Application No	Status of the application	Date of publication	Date of grant
N/A					
3. Name and address of the assignee		(iii) that the rights in the application(s) have been assigned to …… that I/~~We~~ undertake that upto the date of the grant of the patent by the Controller, I/~~We~~ would keep him informed in writing the details regarding corresponding applications for patents filed outside India within six months from the date of filing of such application. Dated this 31st January 2021			
4. To be signed by the applicant or his authorized patent agent		Signature			
5. Name of the natural person who has signed		Digitally signed by, **Agent for the Applicant** Vijaykumar Shivpuje, IN/PA 1096, Sri Kripa, Akshay Nagar, Old Ausa Road, Latur, 413531, Maharashtra, India.			
		To, The Controller of Patents, The Patent Office, at…**Mumbai**…			
Note: - Strike out whichever is not applicable					