Savitribai Phule Pune University

(Formerly University of Pune)

Two Year Post-Graduate Program in Chemistry

(Faculty of Science & Technology)

Choice Based Credit System Syllabus (2019 Pattern)
of

M.Sc. (Chemistry) Part-II

Physical Chemistry, Inorganic Chemistry, Organic Chemistry
Drug Chemistry and Analytical Chemistry

for

Colleges Affiliated to Savitribai Phule Pune University

Implemented from Academic Year
2020-2021

Title of the Course: M.Sc. (Chemistry) (Part-II)

1. Structure of the Course:

Basic structure/pattern (Framework) of the proposed postgraduate syllabus for the two years integrated course leading to M.Sc. (Chemistry) in the colleges affiliated to Savitribai Phule Pune University. The general structure for the M. Sc-II year Chemistry (all specializations)is as follows:

Semester - III			
Sr. No.	**Paper No**	**Description**	**Credit**
1	CCTP-7	Core Compulsory Theory Paper	4
2	CCTP-8	Core Compulsory Theory Paper	4
3	CCTP-9	Core Compulsory Theory Paper	4
4	CBOP-3	Choice Based Optional Paper - Theory	4
5	CCPP-3	Core Compulsory Practical Paper	4
Semester-IV			
6	CCTP-10	Core Compulsory Theory Paper	4
7	CCTP-11	Core Compulsory Theory Paper	4
8	CBOP-4	Choice Based Optional Paper - Theory	4
9	CBOP-5	Choice Based Optional Paper – Practical/ Project	4
10	CCPP-4	Core Compulsory Practical Paper	4

Choice of the optional papers: All colleges are encouraged to give the choice of optional papers to the students and conduct the separate classes if 40% or more students opt a different course than 60% or less students.

The specializations are:

1. **Physical Chemistry**
2. **Inorganic Chemistry**
3. **Organic Chemistry**
4. **Drug Chemistry**
5. **Analytical Chemistry**
6. **Biochemistry**

2. Teaching Hours

a) Theory – Each credit of theory is equivalent to 12 teaching hours + 3 tutorial hours. For 1 credit of theory there will be 1 L of 1 hour per week. Thus, 1 theory course will have total 15 weeks of teaching and it will be distributed as of 48 h for teaching and 12 h for tutorials and internal evaluation. In case of theory paper consisting of sections, each section is of 2 credits and time allotted will be 24 h teaching and 6 h for tutorials and internal evaluation.

b) Practical – Each credit of practical is equivalent to 24 teaching hours + 6 tutorial hours. For 1 credit of practical there will 2 L of 1 h per week. Thus, 1 practical course will have total 15 weeks of teaching and it will be distributed as of 96 h for performing practical and 24 h for tutorials and internal evaluation. i) Each experiment will be allotted 4 h time (one practical session) and for 1 course two sessions of 4 h per week should be allotted or ii) In case practical course is extended for one year, then total 30 weeks (15 week per sem.) and 4 h

(one practical session) per week should be allotted to one practical course. ***There shall not be more than 10 students in one batch of practical.***

3. Examination: Each theory and practical course carry 100 marks equivalent to 4 credits. Each course will be evaluated with Continuous Assessment (CA) and University Assessment (UA) mechanism. Continuous assessment shall be of 30 marks (30%) while university Evaluation shall be of 70 marks (70%). To pass the course, a student has to secure 40% mark in continuous assessment as well as university assessment i.e. 12 marks in continuous assessment and 28 marks in university assessment.

For Continuous assessment teacher must select variety of procedures for examination such as: i) Written test / Mid Semester test (not more than one for each course), ii) Term paper, iii) Viva-Voce, Project / survey / field visits iv) Tutorials v) Group discussion vi) Journal / Lecture / Library notes vii) Seminar presentation, viii) Short quiz ix) assignment x) research project by individual student or group of student xi) An open book test, etc.

Each practical course will be extended over the year and practical examination will be conducted at the end of academic year.

3. M.Sc. (II) Organic Chemistry

Course Structure

Sr. No.	Paper No. & Course Code	Course Nam e	Credits
		Semester - III	
1	**CCTP-7** CHO-350	Organic Reaction Mechanism and Biogenesis	4
2	**CCTP-8** CHO-351	Structure Determination of Organic Compounds by Spectroscopic Methods	4
3	**CCTP-9** CHO-352	Stereochemistry and Asymmetric Synthesis of Organic Compounds.	4
4	**CBOP-3** CHO-353 Theory	CHO-353-A) Protection - De-protection, Chiron approach and Carbohydrate Chemistry	4
		Or	
		CHO-353B) Designing Organic Syntheses and Heterocyclic Chemistry	4
5	**CCPP-3** CHO-354	**Practical I**: Solvent Free Organic Synthesis	4
		Semester – IV	
6	**CCTP-10** CHO-450	Chemistry of Natural Products	4
7	**CCTP-11** CHO-451	Organometallic Reagents in Organic Synthesis	4
8	**CBOP-4** CHO-452 **Theory**	CHO-452 A) Medicinal Chemistry CHO-452 B) Applied Organic Chemistry	4 4
9	**CBOP-5** CHO-453 Practical	**Practical III: Select any two Sections**	4
		Section-I: Ternary Mixture Separation	2
		Section-I: Carbohydrates Synthesis and Isolation of Natural Products	2
		Section-I: Project / Industrial Training/ Internships/ Summer Project	2
10	**CCPP-4** CHO-454	**Practical II**: Convergent and Divergent Organic Syntheses.	4

PRINCIPAL
K.K.H.A. Arts, Comm. & S.P.H. College, Chandwad Dist. Nashik.

ESTD - 1928

PROJECT REPORT

ON

"Synthesis and characterisation of of 1'-(2-amino-6-chloropyrimidin-4-yl)-6-chlorospiro[chromane-2,4'-piperidin]-4-one

by

Mr. Bachhav Yogesh Shivaji

(M. Sc. II Organic Chemistry)

Under the guidance

Dr.Valmik S. Aware

(Assistant Professor)

Department of Chemistry

KKHA Arts, SMGL Commerce & SPHJ Science College, Neminagar

Chandwad (Nashik)-423101

Affiliated to University of Pune, **Maharashtra (India).**

Year 2020-2021

ESTD - 1928

KKHA Arts, SMGL Commerce & SPHJ Science College, Neminagar

Chandwad (Nashik) - 423101

DEPARTMENT OF CHEMISTRY

CERTIFICATE

This is to certify that Mr. Yogesh Shivaji Bachhav of M.Sc. II (Organic Chemistry) class has satisfactorily completed his project work on topic of **"Synthesis and characterisation of 1'-(2-amino-6-chloropyrimidin-4-yl)-6-chlorospiro[chromane-2,4'-piperidin]-4-one ."**

As per the rule laid down by University of Pune, in practical fulfilment ofcourse CH-453 during academic year 2020-2021.

Dr. A.M.Patil	**Prof.V. S.Aware**	
(HOD)	**(Project Guide)**	**(External Examiner)**

HEAD
Department of Chemistry
SNJB's K.K.H.A.Arts, S.M.G.L.Commerce
& S.P.H.J.Science College,
Chandwad-423 101 Dist- Nashik

ACKNOWLEDGEMENT

First I would like to express my sincere gratitude to **Dr. V.S. Aware** Sir for his guidance and suggestions. I feel this thesis is an expression of the hard work carried out under his guidance. I must thankful to him for giving me his valuable time and guidance in my practical work which will be helpful to explore new horizon in my future career in research .

The name of our principal **Dr.G.H.Jain** needs a very special attention in the entire process of my research carrier. He not only extended the administrative and infrastructural support for this work but motivated me in achieving the targets with sincerity and honesty.He has always encouraged me with his words and actions, remarks and reactions.

I would like to thank, **Dr.A.M.Patil** Head Dept. of Chemistry for giving the opportunity to do this work. Also I am thankful to **Prof. S.P. Khairnar, Dr.R.S. Sancheti, Prof. M.A. Todarwal madam for** their direct or indirect motivation. I would also like to thank you to all teaching staff in chemistry department.

Thank you to members of the Department of Chemistry who made me feels a part ofthe "chemistry" family. My special thanks to my entire lab mates and especially to **Mr. Audumbar Bidgar** and all other non-teaching staff for their co-operation during my experimental work in laboratory.

I invariably feel short of words to express my sincere thanks to my parents whose efforts have brought me to this stage, who always inspired me and provided constant support me in my decisions and whose blessings made the journey worth the effort.

BACHHAV

(Mr. BACHHAV YOGESH SHIVAJI)

Abbreviations:

BOC:	t-Butyloxycarbonyl
DCM	Dichloro methane
TFA	Trifluro acetic acid
RT	Room temperature
h	hours
TLC	Thin layer Chromatography
$CDCl_3$	Deuterated Chloroform
DMSO	Dimethyl sulphoxide
ACC	Acetyl-CoA carboxylase
GPCR	G-Protein Coupled Receptor
(SCD)-1	stearoyl-CoA desaturase-1
HDAC	Histone deacetylase
DOR	δ-Opioid receptor
11β-HSD1	11β-hydroxysteroid dehydrogenase type 1

GENERAL REMARKS

1) The ^{1}H NMR spectra were recorded on a Brucker AvanceDPX (500 MHz) instruments using DMSO-*d*6 and $CDCl_3$ solvent. Chemical shifts are expressed in δ (ppm) units downfield to internal standard TMS. The ^{1}H data is expressed using standard notations such as chemical shift, splitting pattern for assignment.

2) Melting Points were determined using a Staurt Melting Point Apparatus, Mod. SMP-10 in open capillary tubes and measured in ^{o}C.

3) All reactions were monitored by Thin Layer Chromatography on 0.2 mm silica gel F-254 (Merck) plates using UV light (254 and 366 nm) for detection.

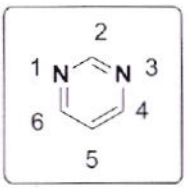
4) All chemicals and reagents were purchased from S. D. Fine, Merck, Across, Aldrich,Fluka, were purified and dried according to the procedures given in literature.

Content

Synthesis and characterisation of 1'-(2-amino-6-chloropyrimidin-4-yl)-6-chlorospiro[chromane-2,4'-piperidin]-4-one

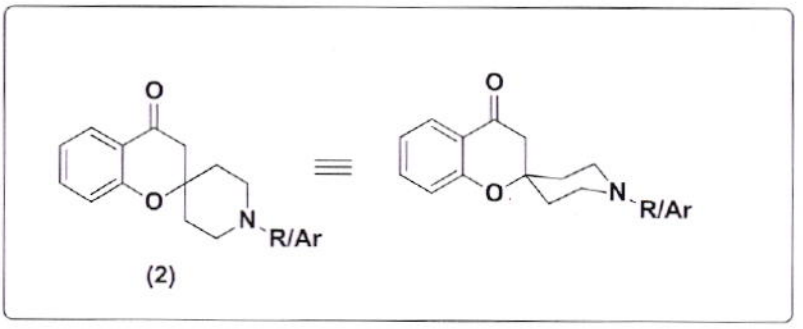
1. Introduction:

Pyrimidine (**1**) is six membered heterocyclic compounds with two nitrogen's at 1 & 3 position in the ring. It undergoes aromatic Nucleophilic substitution reaction if proper substituent (leaving group) is their at appropriate position especially at C_2, C_4 and C_6 carbons. Such kind of nucleophilic substitution reactions are very commonly used in designing and synthesis of biologically active compounds as well as already used many marketed drugs. Pyrimidine is known for their diverse biological activities and reported for to use as Anticancer agents, Anti-HIV agents, Anaesthetic agents, Antibacterial agents, Cardiotonic/Bronchodialtors and many more biological activities.[1]



(1)

Pyrimidine can be used with many other moieties in order to enhance biological activity and to generate novel scaffold. The 6,6-spiro ring system like spirochromane (**2**) is an interesting moiety due to their anti-arythmic, anti-cancer, anti-tubercular, anti-microbials, histamine-3 antagonists, acetyl-CoA carboxylase (ACC) inhibitors, stearoyl-CoA desaturase-1 (SCD)-1 inhibitors, histone deacetylase (HDAC) inhibitors, anti-malarial and δ opioid receptor (DOR) agonists activity. [2]



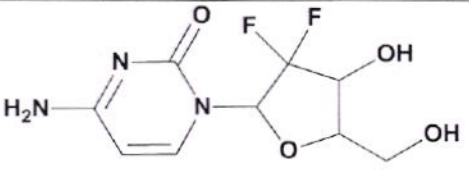
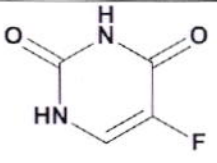
(2)

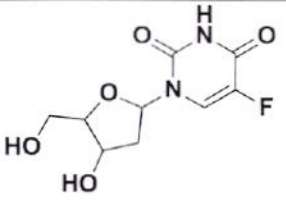
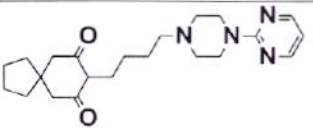
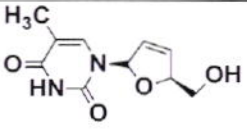
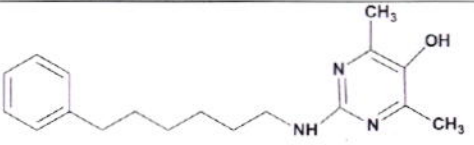
Due to presence of piperidine ring in spirochromane it can generate non-flat molecules with three dimensional structure. Such three dimensionality reported to interact more with biological target than flat molecules and enhance biological activities and physiochemical properties.[3]

Based on this, it will be interesting to club the spirochromane (6,6-system) and properly substituted pyrimidine to generate novel and biological active molecules.

2. Study of Literature:

After scanning plethora of literature it is reported that pyrimidine is medicinally important and present in several marketed drugs as given below.[4]

Sr. No	Structure	Name of drug	Use
1		Gemcitabine **(3)**	Anti-Cancer
2		5-Fluorouracil **(4)**	Anti-Cancer
3		Thonzylamine **(5)**	Anti-histaminic

4		Floxuridine (**6**)	Anti-Cancer
5		Buspirone (**7**)	Anti-psycotic
6		Stavudine (**8**)	Anti-HIV
7		Enazadrem (**9**)	Anti-Psoriatic

Freeman-Cook and their co-workers from Pfizer reported the synthesis of azole based spirochromane **(10)** as Acetyl-CoA Carboxylase (ACC) inhibitors with great inhibitory activity.[5] In another study Uto and his co-workers reported oxadiazole and pyridazine based novel series of spirochromane **(11)** based highly potentstearoyl-CoA desaturase (SCD)-1 inhibitors.[6] Bourdonnec and his colleagues reported the synthesis of phenyl substituted spirochromane (**12**) as delta (δ) opioid receptor inhibitors (DOR) with good oral bioavailability.[7] In another study Lepifre et al. reported the synthesis of novel spirochromane (**13**) as a 11β-hydroxysteroid dehydrogenase type 1 (11β-HSD1) inhibitors.[8] Ashok and his co-workers reported furon based spirochromane (**14**) for anti-inflammatory and anti-oxidant property.[9] Abdellatif and co-worker designed and synthesised novel spirochromane (**15**) and evaluated as cytotoxic agent against three human cancer cell lines; human ovarian cancer (A2780), human breast carcinoma (MCF-7), and human colorectal adenocarcinoma (HT-29) using

MTT assay.[10] Takeru et.al synthesised naphthalene containing spirochromane **(16)** as ACC inhibitors.[11]

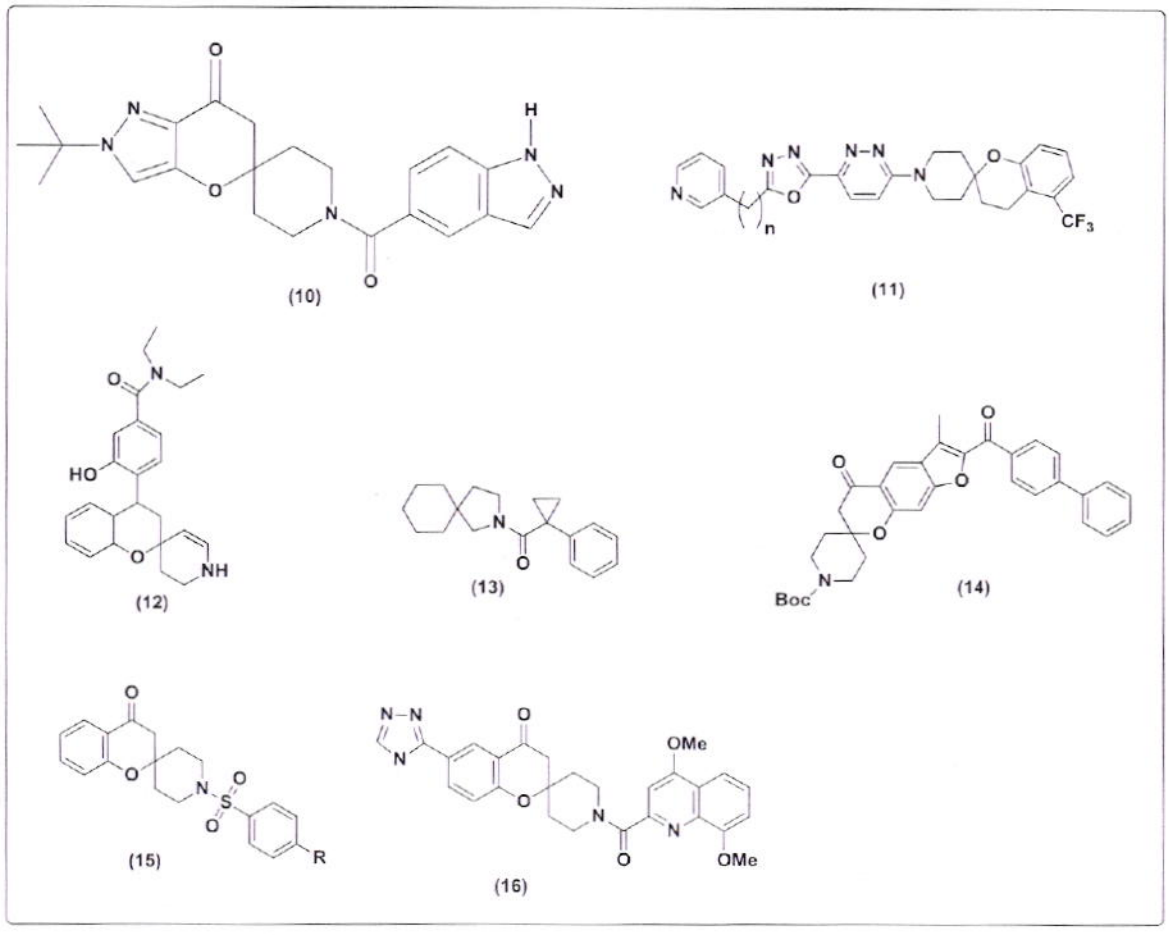


Fig: 2 Biologically active spirochromane

3. Present work:

Based on the previous literature search of pyrimidine and spirochromane (6, 6 system) is biologically active compounds. Thus, we want to do synthesis of novel molecules in which pyrimidine and spirochromane is present in the same target molecule. (**Fig: 1**)

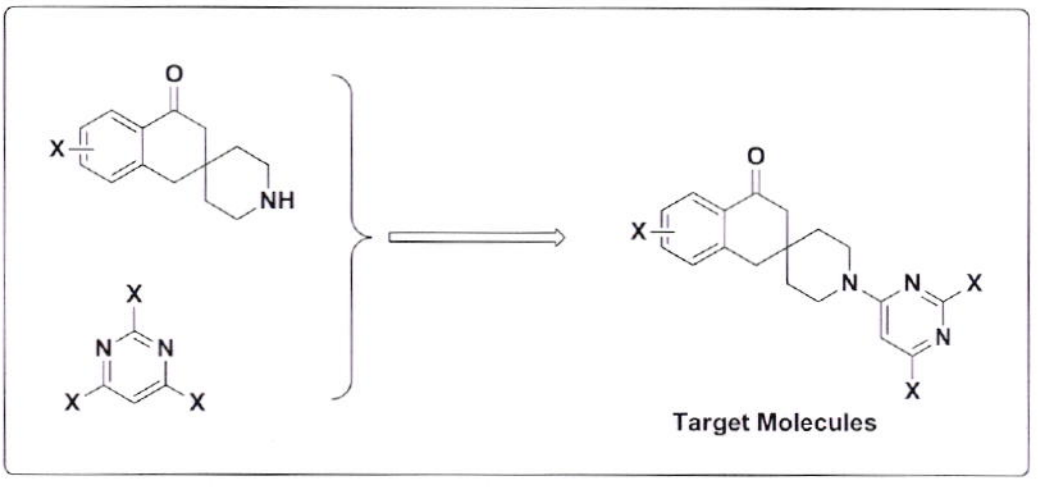
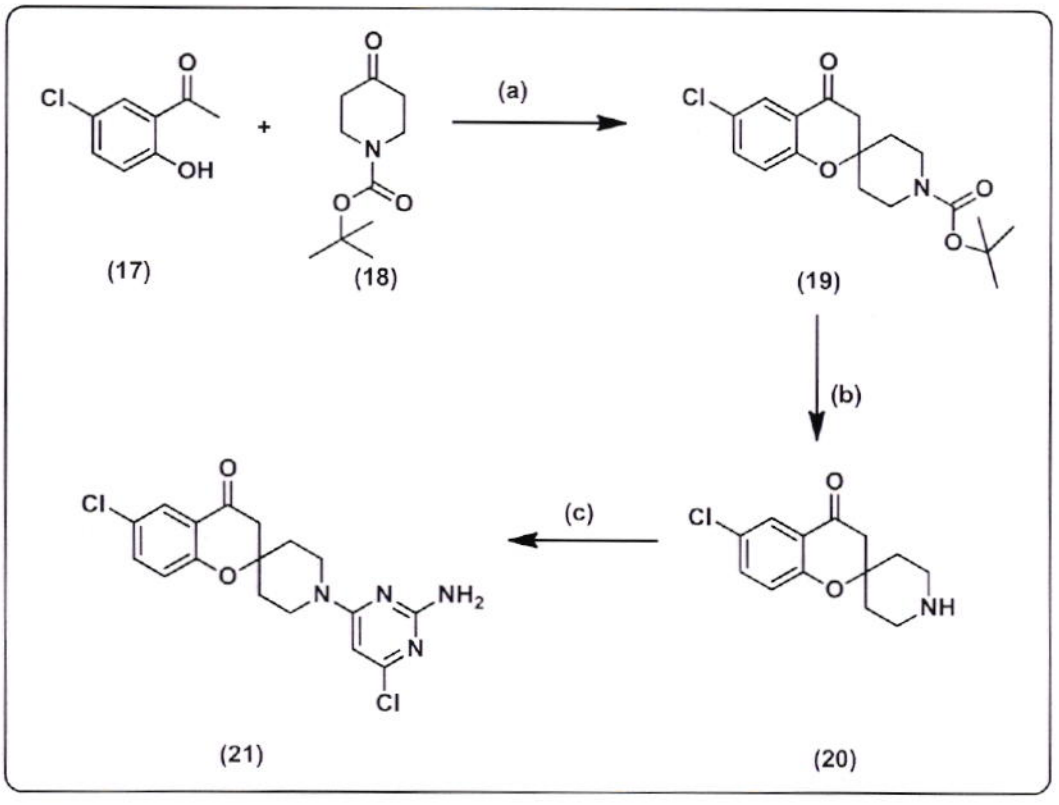


Fig:1 Designing of pyrimidine containing spirochromane

4. Experimental Section

In order to synthesize such novel molecules we have treated 2-hydroxy acetophenone (**17**) with N-Boc Piperidone (**18**) to afford N-Boc spirochromane (**19**). The deprotection of compound **19** on treatment with TFA provided spirochromane amine (**20**). The amine 20 on treatment with chlorosubstituted pyrimidine undergoes nucleophilic substitution reaction to afford novel pyrimidine containing spirochromane (**21**). (**Scheme-1**)

Scheme-1:

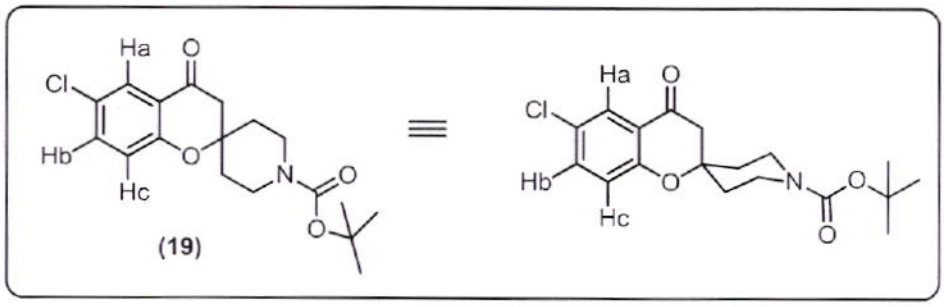


Reagents and conditions: (**a**) Pyrrolidine, Methanol, 65° C, 3h (**b**) DCM, TFA, RT, 2h (**c**) 2-amino, 4,6-dichloro pyrimidine, 1,4-dioxane,NEt_3, 3h , reflux

Synthesis of tert-butyl 6-chloro-4-oxospiro[chromane-2,4'-piperidine]-1'-carboxylate (**19**)

In RBF 2-Hydrox acetophenone **17** (1 g, 0.005 mol) and N-BOC piperidone **18** (1.28 g, 0.006 mol) was dissolved in Methanol (25 mL) was refluxed in presence of pyrrolidine catalyst at 65° C for 3 h. Then cooled reaction mixture and evaporated excess methanol on rotary evaporator. Then diluted with water (100 ml) and extracted with Ethyl acetate (3x 20 mL) and dried over Sodium sulphate. Then evaporated organic layer and purified using

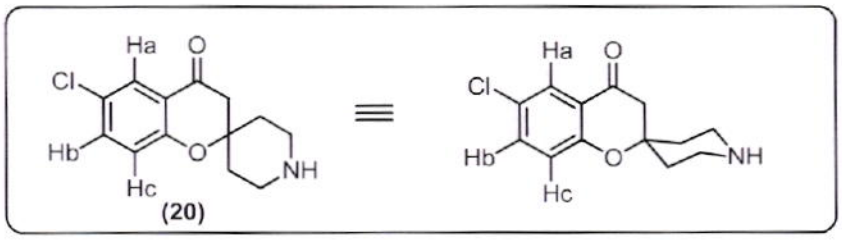
silica gel column chromatography. (2 % EtoAc+ n-Hexane) to afford off white solid of intermediate **19**. TLC System: 30 % EtOAc+ n-Hexane.



Off White Solid: Yield: 94 %; **^{1}H-NMR** ($CDCl_3$, 500 MHz, TMS) : δ 1.46 (s, 9,t-Bu-*H*) , 1.58-1.64 (m, 2H, -CH_2), 1.99 (m, 2H, -CH_2), 2.71 (s, 2H, -CH_2-CO-), 3.17-3.22 (m, 2H, N-CH_2) , 3.87 (bs, 2H, N-CH_2), 6.95 (d, 1H, *J*=8.8Hz, Ar-*Hc*) , 7.42 (dd, 1H, *J*=2.7 & 8.8 Hz, Ar-*Hb*), 7.81 (d, 1H, *J*=2.7Hz, Ar-*Ha)*

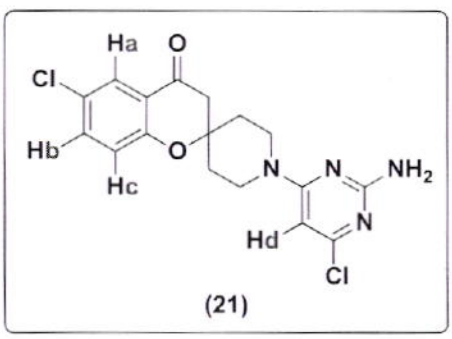
*Synthesis of 6-chlorospiro[chromane-2,4'-piperidin]-4-one (**20**)*

In RBF N-Boc Oxaspirochromane **19** (1.6 g, 0.004 mol) was dissolved in dichloromethane (DCM) and was added TFA (10 ml) and was heated over a period of 2h at 50°C . Then removed excess DCM on rotary evaporator and added aq. 10 % $NaHCO_3$ till pH = 9. Then extraction with Ethyl acetate (3 x 10 mL) was completed and dried organic layer sodium sulphate. The organic layers were combined and evaporated over rotary evaporator. The crude mixture was purified by using silica gel column chromatography (20 % Pet ether+EtOAc) to afford yellow solid of intermediate **20**.



Yellow Solid: Yield: 90 %; **^{1}H-NMR** (DMSO-d_6, 500 MHz, TMS): δ 1.91-1.87 (m, 2H, -CH_2) , 2.04-2.07 (m, 2H, -CH_2) , 2.50 (s, 2H, -CO-CH_2), 3.05-3.15 (m, 4H, -N-CH_2) , 7.18 (d, 1H, *J*=8.5 Hz, Ar-*Hc*), 7.65-7.67 (m, 2H, Ar-*Ha & Hb)*

*Synthesis of 1'-(2-amino-6-chloropyrimidin-4-yl)-6-chlorospiro[chromane-2,4'-piperidin]-4-one (**21**)*

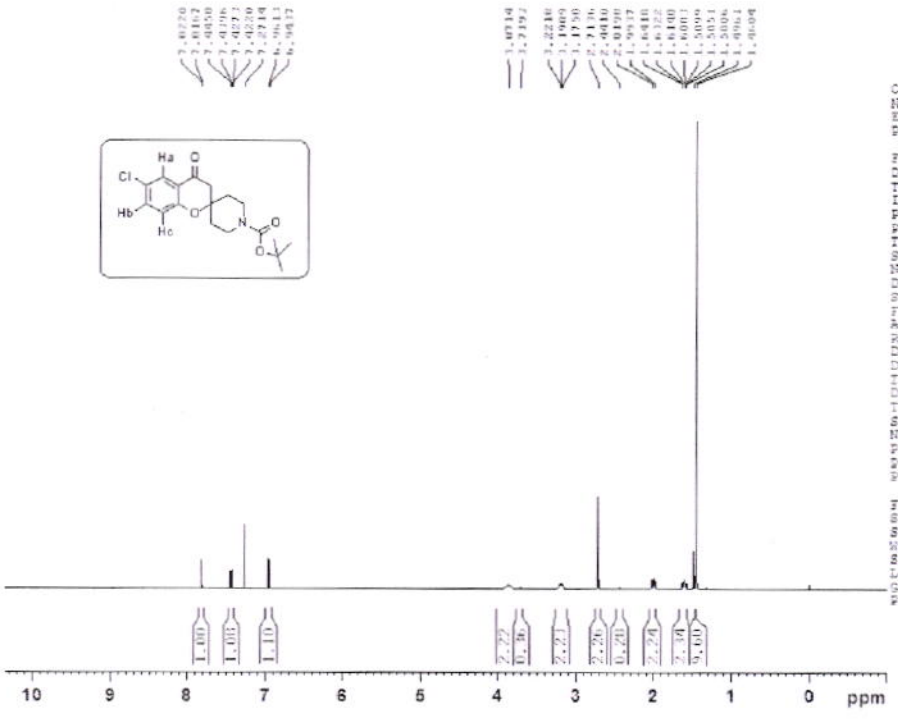


(21)

In RBF compound **20** (0.2 g ,0.0008 mol) and triethyl amine (0.39 ml,0.0031 mol) and 2,amine 4,6-dichloro pyrimidine (0.168 g ,0.0010) was dissolved in 1,4-dioxane (20 ml) and refluxed at 60° C for 3 h. Then was cooled reaction mixture and evaporated excess 1,2-dioxane on rotary evaporator. The crude mixture was treated with water (10 ml) and dried it on vaccum pump.Then washed with n-Hexane and dried further on rotary evaporator to afford off white solid of compound **21.** TLC system: 30% ethyl acetate + n-hexane

Off white solid: Yield: 90 %; **^{1}H-NMR** (DMSO-d_6, 500 MHz, TMS): δ 1.67-1.70 (m, 2H, -CH_2), 1.90-192 (m, 2H, -CH_2), 2.88 (s, 2H, -CO-CH_2), 3.24-3.34 (m, 4H, -N-CH_2) , 6.13 (s, 1H, -Pyrimidine-*H*), 6.48 (bs, 2H, -NH_2), 7.17(d (d, 1H, *J*=10 Hz, Ar-*Hc*), 7.64 (dd,1H, *J*=10 Hz & 5Hz, Ar-*Hb*), 7.65(*J*=10 Hz, Ar-*Ha*).

5. Spectral Analysis :

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BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.

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P1               10.00 usec
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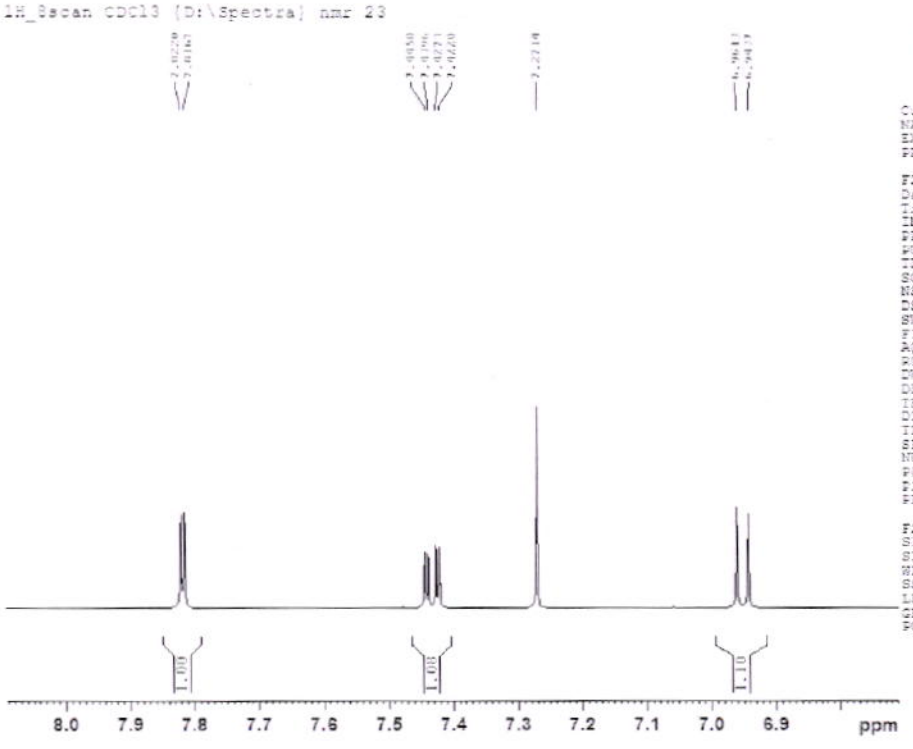


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BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.

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NAME          Feb03-2021
EXPNO              230
PROCNO               1

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Time              16.13 h
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PROBHD  Z119470_0333
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TD              65536
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DS                   0
SWH          14705.883 Hz
FIDRES        0.448788 Hz
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RG              98.7854
DW              34.000 usec
DE               6.79 usec
TE              303.1 K
D1           1.00000000 sec
TD0                  1
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NUC1                1H
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P1               10.00 usec
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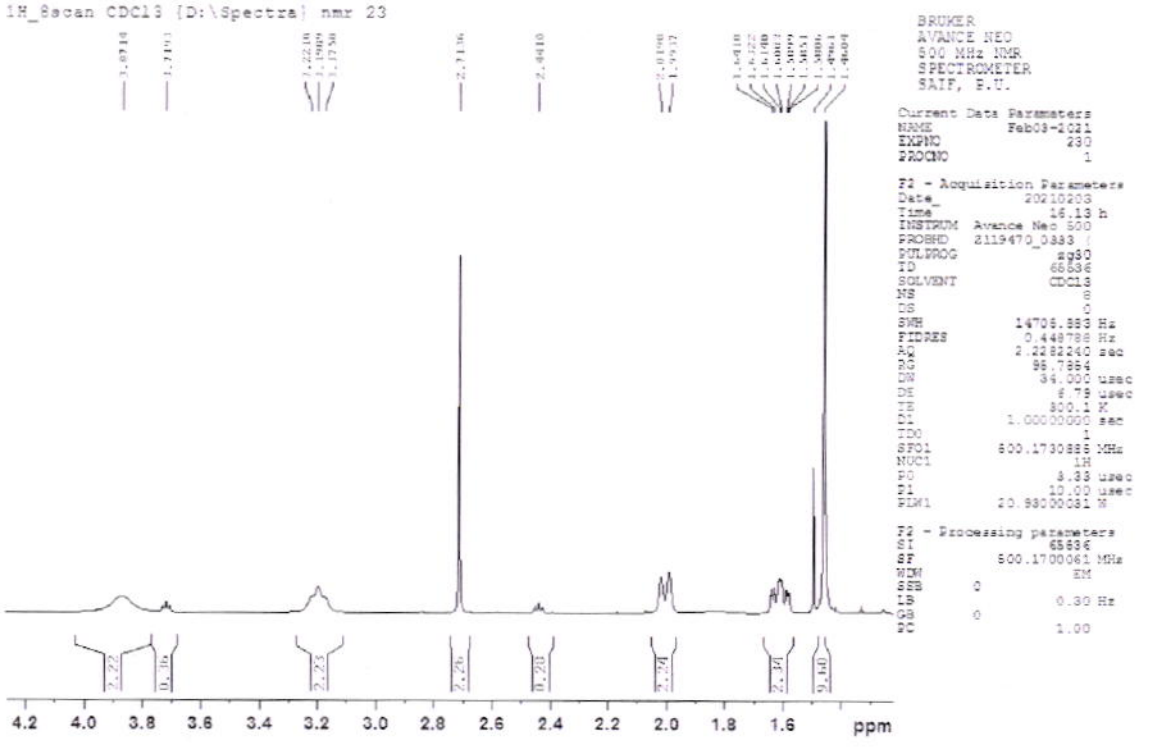
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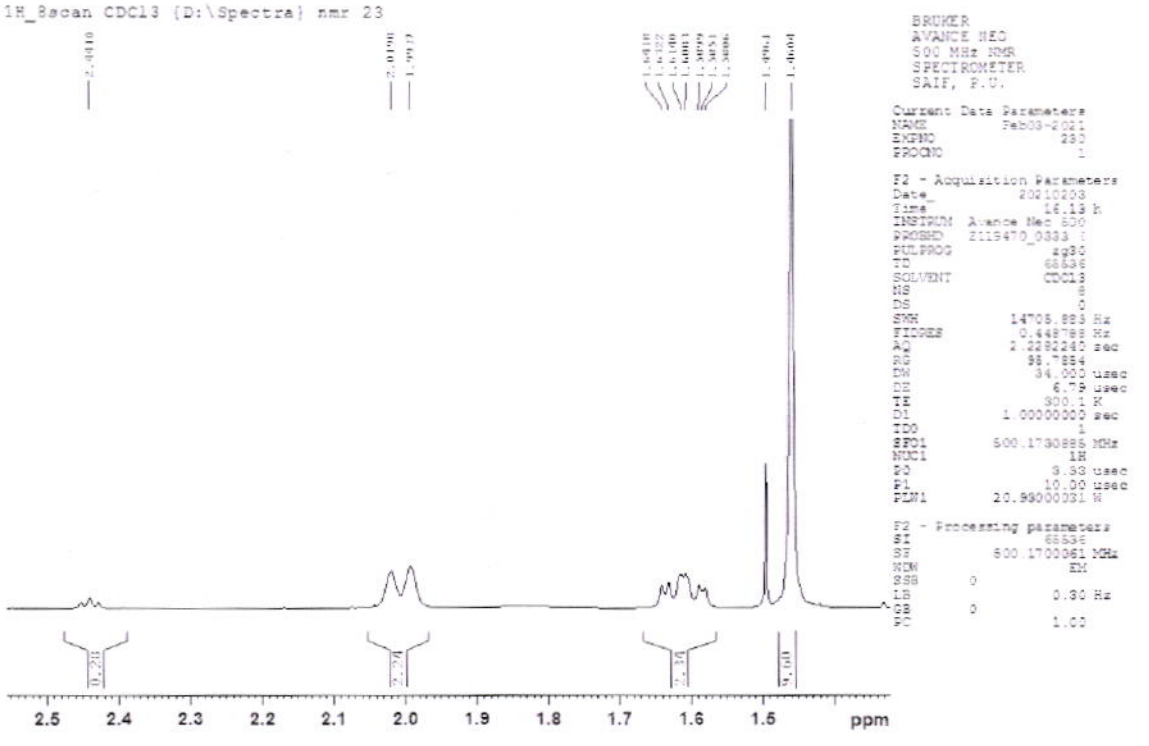
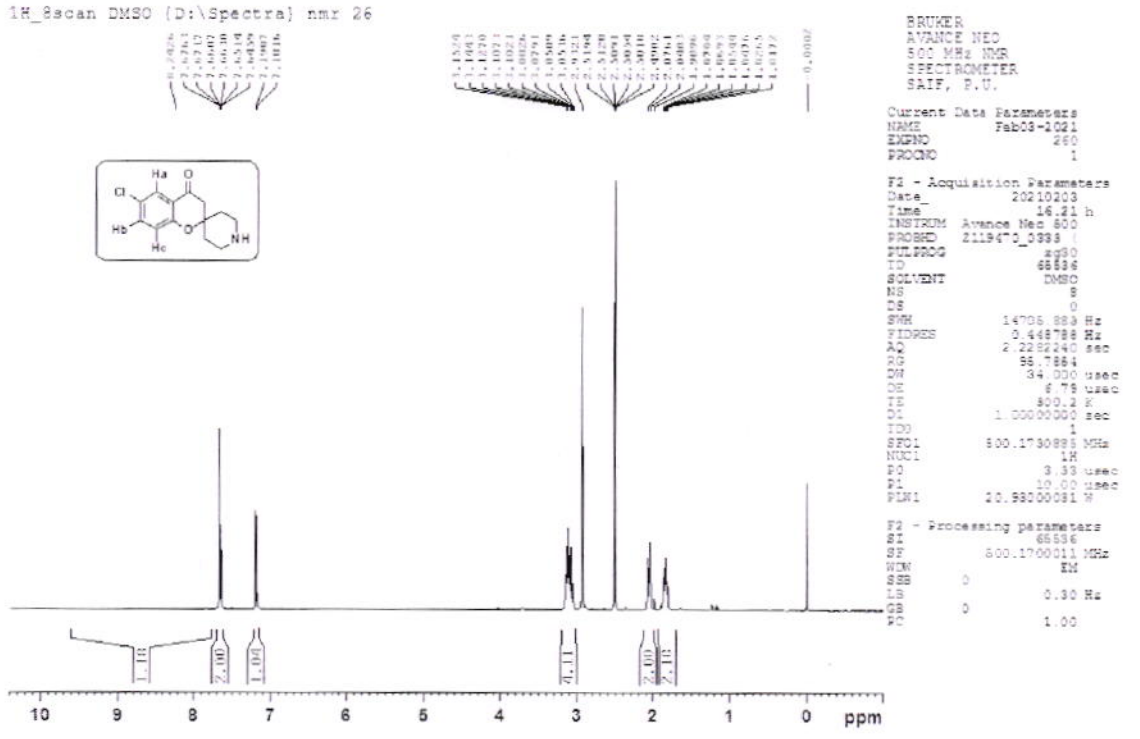
SFO1 600.1730885 MHz
NUC1 1H
P0 8.33 usec
P1 10.00 usec
PLW1 20.99000031 W
F2 - Processing parameters
SI 65536
SF 600.1700061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


1H_8scan CDCl3 {D:\Spectra} nmr 23
BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.
Current Data Parameters
NAME Feb03-2021
EXPNO 230
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210203
Time 16.13 h
INSTRUM Avance Neo 600
PROBHD 2119470_0383 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 14705.883 Hz
FIDRES 0.448786 Hz
AQ 2.2282240 sec
RG 98.7854
DW 34.000 usec
DE 6.79 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SFO1 600.1730885 MHz
NUC1 1H
P0 8.33 usec
P1 10.00 usec
PLW1 20.99000031 W
F2 - Processing parameters
SI 65536
SF 600.1700061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

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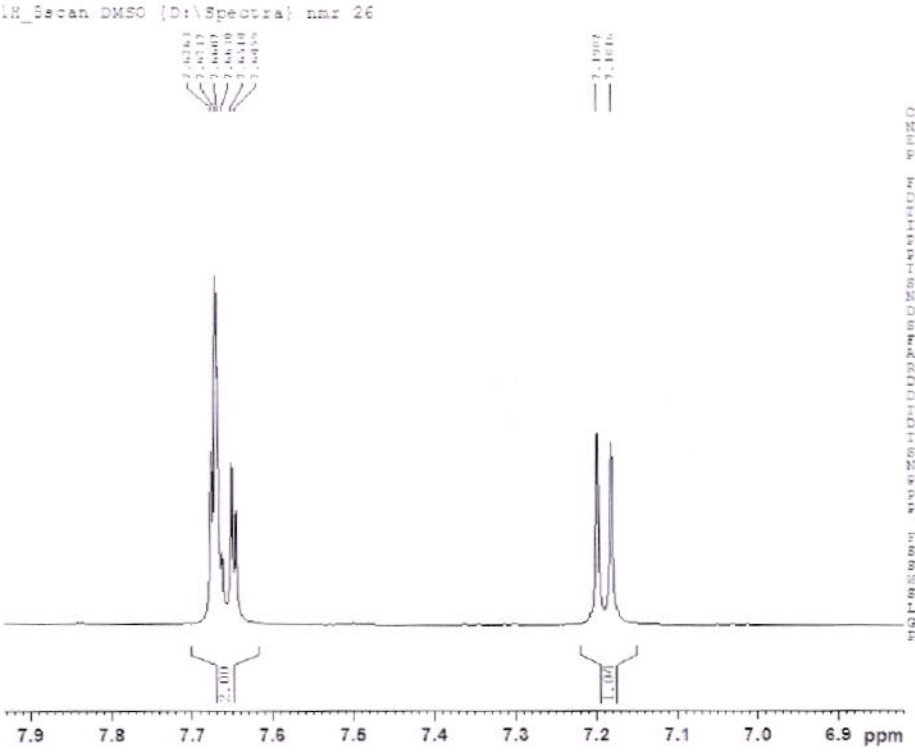
BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.

Current Data Parameters
NAME          Feb03-2021
EXPNO              260
PROCNO               1

F2 - Acquisition Parameters
Date_        20210203
Time_          16.21 h
INSTRUM  Avance Neo 600
PROBHD  2119470_0333 (
PULPROG          zg30
TD              65536
SOLVENT          DMSO
NS                   8
DS                   0
SWH          14705.883 Hz
FIDRES        0.448788 Hz
AQ           2.2282240 sec
RG             95.7864
DW               34.000 usec
DE                6.79 usec
TE              300.2 K
D1           1.00000000 sec
TD0                  1
SFO1       600.1730895 MHz
NUC1               1H
P0                3.33 usec
P1               10.00 usec
PLW1       20.93000031 W

F2 - Processing parameters
SI              65536
SF         600.1700011 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

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BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.

Current Data Parameters
NAME          Feb03-2021
EXPNO              260
PROCNO               1

F2 - Acquisition Parameters
Date_        20210203
Time_          16.21 h
INSTRUM  Avance Neo 600
PROBHD  2119470_0333 (
PULPROG          zg30
TD              65536
SOLVENT          DMSO
NS                   8
DS                   0
SWH          14705.883 Hz
FIDRES        0.448788 Hz
AQ           2.2282240 sec
RG             95.7864
DW               34.000 usec
DE                6.79 usec
TE              300.2 K
D1           1.00000000 sec
TD0                  1
SFO1       600.1730895 MHz
NUC1               1H
P0                3.33 usec
P1               10.00 usec
PLW1       20.93000031 W

F2 - Processing parameters
SI              65536
SF         600.1700011 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```
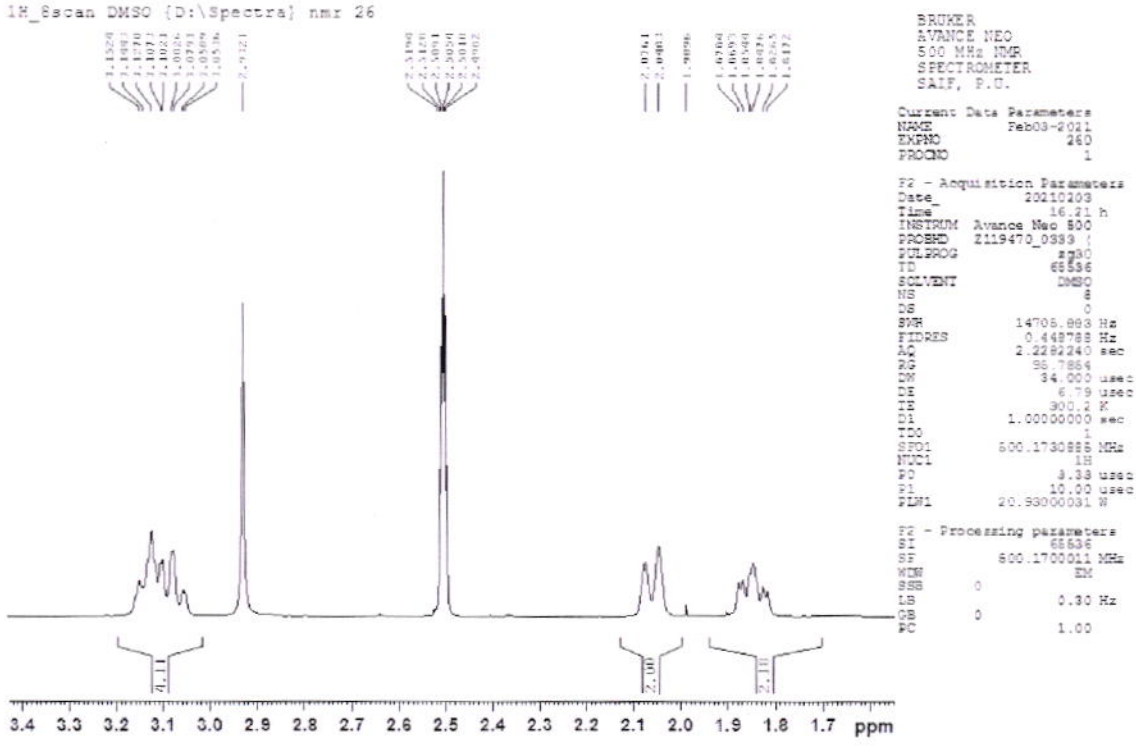
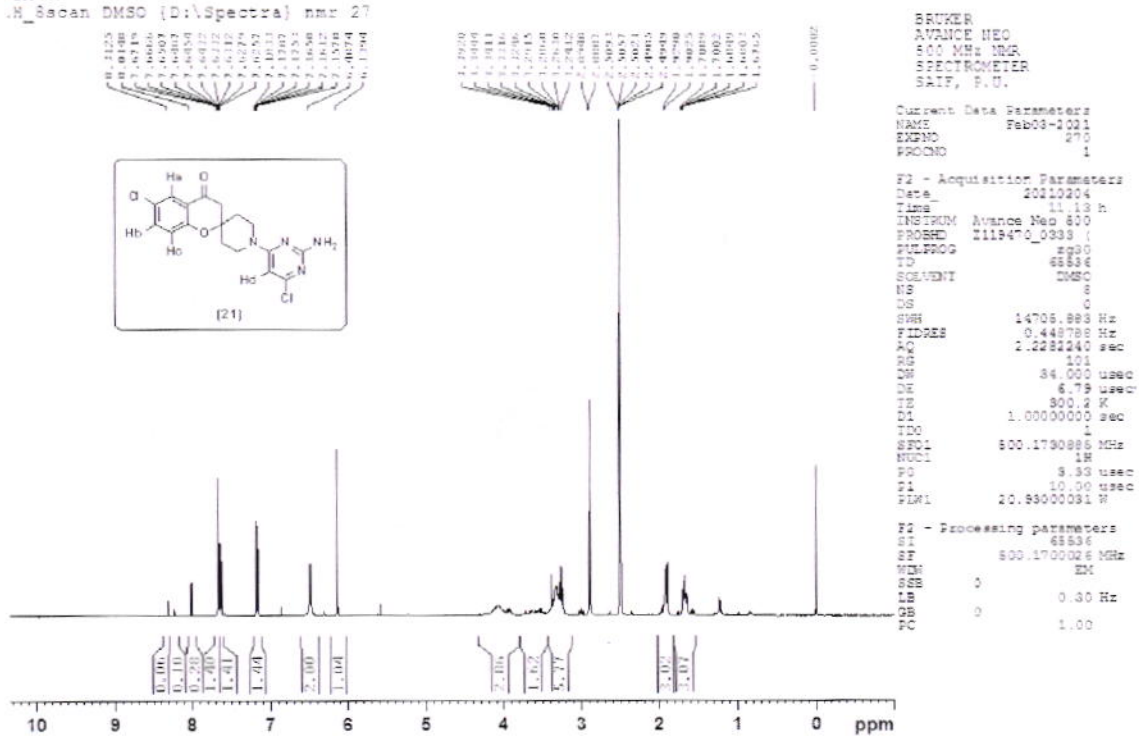
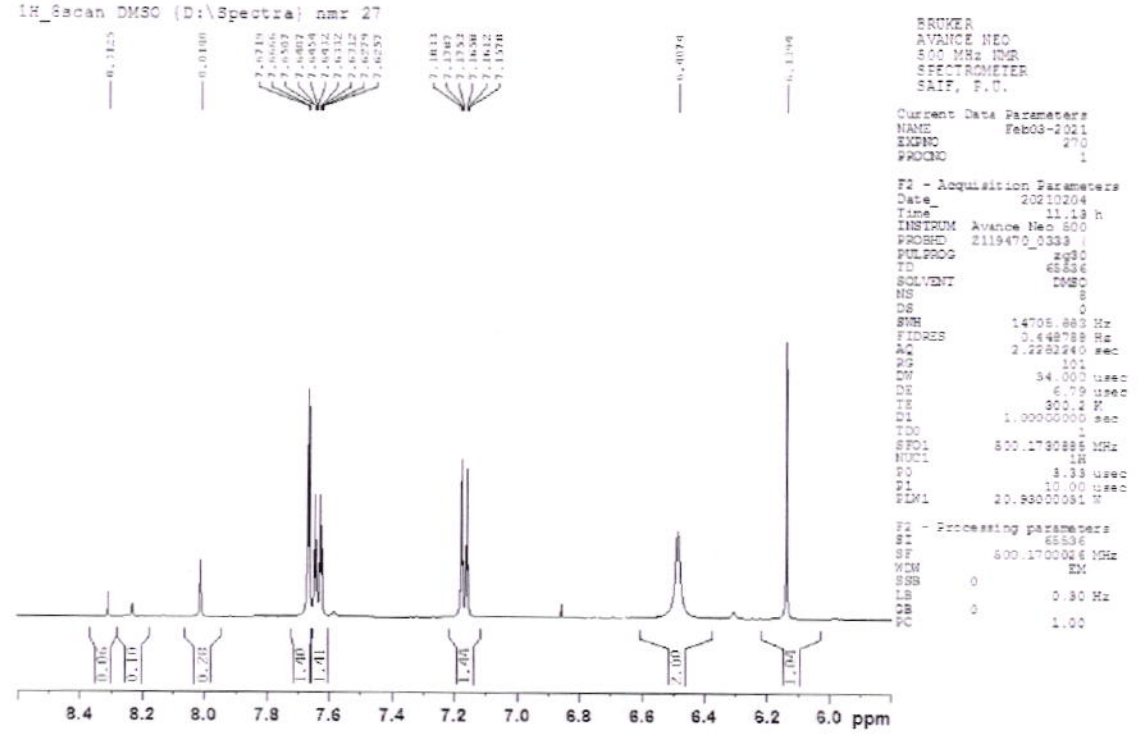
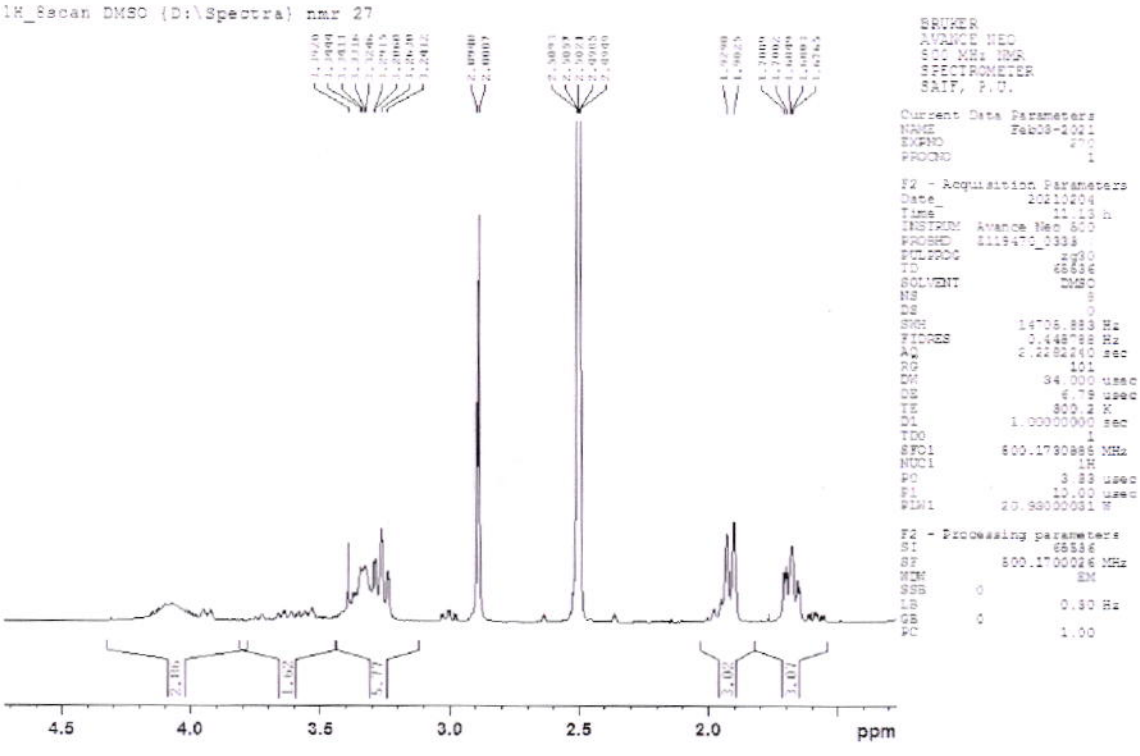


1H_8scan DMSO (D:\Spectra) nmr 26
BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.
Current Data Parameters
NAME Feb03-2021
EXPNO 260
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210203
Time 16.21 h
INSTRUM Avance Neo 600
PROBHD Z119470_0333 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 14705.883 Hz
FIDRES 0.448788 Hz
AQ 2.2282240 sec
RG 96.7864
DW 34.000 usec
DE 6.79 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 600.1730885 MHz
NUC1 1H
P0 3.38 usec
P1 10.00 usec
PLW1 20.93000031 W
F2 - Processing parameters
SI 65536
SF 600.1700011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.11
2.00
2.10
3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 ppm


1H_8scan DMSO (D:\Spectra) nmr 27
BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.
Current Data Parameters
NAME Feb03-2021
EXPNO 270
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210204
Time 11.13 h
INSTRUM Avance Neo 600
PROBHD Z119470_0333 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 14705.883 Hz
FIDRES 0.448788 Hz
AQ 2.2282240 sec
RG 101
DW 34.000 usec
DE 6.79 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 600.1730885 MHz
NUC1 1H
P0 3.38 usec
P1 10.00 usec
PLW1 20.93000031 W
F2 - Processing parameters
SI 65536
SF 600.1700026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ha
Hb
Hc
Hd
[21]
0.96
0.98
1.40
1.41
1.44
2.00
1.04
2.84
1.82
5.77
3.07
5.07
10 9 8 7 6 5 4 3 2 1 0 ppm


1H_8scan DMSO (D:\Spectra) nmr 27
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
6.0 ppm
BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.
Current Data Parameters
NAME Feb03-2021
EXPNO 270
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210204
Time 11.13 h
INSTRUM Avance Neo 600
PROBHD Z119470_0333 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 14705.883 Hz
FIDRES 0.448788 Hz
AQ 2.2282240 sec
RG 101
DW 34.000 usec
DE 6.79 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 600.1790885 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 20.93000031 W
F2 - Processing parameters
SI 65536
SF 600.1700026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


1H_8scan DMSO (D:\Spectra) nmr 27
4.5
4.0
3.5
3.0
2.5
2.0
ppm
BRUKER
AVANCE NEO
600 MHz NMR
SPECTROMETER
SAIF, P.U.
Current Data Parameters
NAME Feb03-2021
EXPNO 270
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210204
Time 11.13 h
INSTRUM Avance Neo 600
PROBHD Z119470_0333
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 14705.883 Hz
FIDRES 0.448788 Hz
AQ 2.2282240 sec
RG 101
DW 34.000 usec
DE 6.79 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 600.1790885 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 20.93000031 W
F2 - Processing parameters
SI 65536
SF 600.1700026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

6. Conclusion:

The multistep synthesis of pyrimidine containing spirochromane molecule is achieved by synthesising the required intermediate. Only single novel molecule (**21**) is synthesized and characterised by ^{1}H-NMR.

7. References:

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