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DBUH+I_3 complex an efficient catalyst for the synthesis of 2-phenyl benzimidazole and benzothiazole derivatives

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Abstract: Herein, we have reported the facile synthesis of various benzimidazole / benzothiazole by using DBU-Iodine-Iodide as a green and simple catalyst. The R_2NH+I_3 complexes have been formed by reacting an aqueous mixture of ammonium iodide and molecular iodine with the aqueous solution of amine. The structure of R_2NH+I_3 complexes has confirmed by spectroscopic techniques. The prepared amine-iodine complexes have screened as a catalyst in the synthesis of benzimidazole / benzothiazoles. Among the screened catalyst DBUH+I_3 complex has been found as an efficient catalyst. The synthesis of benzimidazoles and benzothiazoles has been achieved with the reaction of *o*-phenylene diamine /*o*- amino thiophenol and various substituted aryl aldehyde using DBUH+I_3 as a catalyst. The present protocol has offered some advantages over other reported protocols such as the mild reaction condition, commercially available precursors, inexpensive catalyst, short reaction time, the broad scope of the substrate, high yield, simple isolation of the product, and environmentally benign method.

Keywords: Amine-iodine complexes; Benzimidazole; Benzothiazole; Oxidative cyclization; organocatalysis

INTRODUCTION

Benzimidazoles and benzothiazoles are valuable heterocyclic scaffolds due to their many applications in diverse fields such as agrochemicals, veterinary, and pharmaceuticals.[1-3] They are potent privileged bicyclic aromatic nuclei in organic and medicinal chemistry. They showed diverse biological activity.[4-7] Benzimidazole and benzothiazole has found as the core structural skeleton in a variety of drug molecules specifically pantoprazole, riluzole, clemizole, bendamustine, thiabendazole, telmisartan, benzitramide, omeprazole, Hoechst

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33342, pimobendan, Mibefradil, Dovitinib EGFR-3, sulfathiazole, ritonavir, abafungin, tiazofurin, and benazolin. This class of heterocyclic compound displays valuable properties like photochromic, biochemical luminescence, and solvatochromic properties.[8-9] These heterocyclic molecules have significant biological activity and great pharmaceutical potential, to attract more attention of synthetic chemists. These heterocyclic molecules have significant biological activity and great pharmaceutical potential, to attract more attention from synthetic chemists (**Figure 1**).

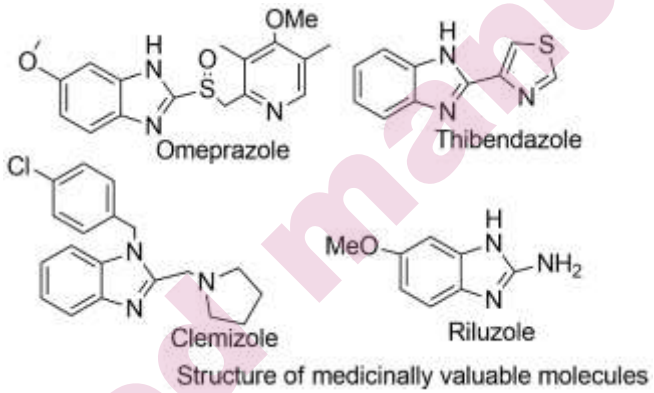

Figure 1 : Benzimidazoles ring containg drug molecules

The robust method for synthesis of these molecules involves the treatment of *o*-phenylenediamine[10] and 2-amino thiophenol[11] with carbonyl compounds, such as aldehyde using Bronsted or Lewis acid catalyst[12] and carboxylic acids[13] or their derivative (nitrile, amide, ester, acid chloride)[14] at elevated temperature. Another approach involves metal-catalyzed direct alkylation of these molecules via C-H activation followed by carbon-carbon bond formation.[15] Synthesis of these molecules was achieved by microwave,[16] ultrasonic wave,[17] ionic liquid,[18] ionic liquid gel,[19] nanomaterial,[20] DMF,[21] and under oxidative condition using various oxidative and catalytic reagents cited in the reference.[22-23] The certain green synthesis of benzimidazole was accomplished by homogeneous catalysis such as use of triflate erbium catalyst,[24] use of active deep eutectic solvent[25] and montmorillonite K 10 heterogenous green catalyst.[26] Generally, nearly all methods of benzimidazole synthesis have worked for benzothiazole.[27] The reported methods have limitations such as harsh reaction conditions, poor yield, high temperature, hazardous and carcinogenic solvent, expensive catalyst, side reaction, slow reaction rate, toxic reagents or tedious workup procedure, and difficulty to isolate the product from the reaction mixture. Consequently, a search for better catalyst, environmentally benign methodology has continued for the economy and

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operational simplicity. Our developed amine-iodine complex catalytic procedure is overcoming these problems.

Iodine catalysis has been known for more than 100 years. It has remarkably catalyzed various types of reactions.[28-29] The drawback of molecular iodine catalyzed synthesis of 2-substituted benzimidazole and benzothiazole is the sublimation of molecular iodine and moisture sensitivity, we have overcome these problems in amine-iodine-iodide complex organocatalyst.

We have synthesized the new R_2NH+I_3 complexes using amine, ammonium iodide, and molecular iodine.[30] The R_2NH+I_3 complexes were characterized spectroscopic technique and confirmed.[31] These catalysts were air-stable, and iodine never sublimates or deliquescent. Amine-iodine complex has catalyzes the synthesis of 2-aryl benzimidazole and benzothiazole, offers several advantages namely short reaction time, easy workup procedure, and environmentally benign protocol. Amine-iodine complexes are organocatalysts that have an indispensable part of synthetic green chemistry because of their stability, less expensive, less toxic, and easily applicable to a wide range of substrates. Herein, we reported amine-iodine complexes catalyzed condensation and cyclization of a wide variety of aryl aldehyde with *o*-phenylenediamine and *o*-amino thiophenol, respectively. Here, we described the synthesis of new amine-iodine complexes (1a-e) and their synthetic application.

EXPERIMENTAL

The Commercially available chemical reagents and solvents were used and their purity was ensured before use. Solvents that were entirely dry and free of impurities were used. Reaction of the progress was checked on Merck TLC Silica gel 60 F254 plates using UV lamp (365 nm and 254 nm) and iodine chamber. The melting point was determined using open capillary method. The recorded melting points were uncorrected. PerkinElmer FTIR spectrometer was used to record IR spectra. Bruker Avance III HD NMR 500 MHz spectrometer is used to obtained ^{1}HNMR and ^{13}C NMR spectra in DMSO d6 and $CDCl_3$. HRMS analysis was obtained on a Bruker Impact II UHR-TOF mass spectrometer system.

Preparation of DBU-Iodine complexes

2.665 g of Ammonium iodide (18.352 mmol, 2.8 eq.) has added to 5.2 mL water (2 volumes) to got a clear solution in a 250 mL beaker, followed by the addition of 1.667 g of iodine (6.568 mmol, 1 eq). This solution was added dropwise to a stirred solution of 1g DBU (6.568 mmol, 1 eq) in 8 mL water (8 Volume) in a 250 mL round bottom flask. The solid product has formed during addition, stirred the mixture for 15 minutes, and filtered off the solid product. The product has been washed with cold water and dried under a vacuum to provide the desired complexes. After drying the complex, the yield has been reported.

Typical Process for the synthesis of benzimidazole / benzothiazole from o-phenylenediamine/thiophenol and aldehyde.

A mixture of *o*-phenylenediamine/*o*-amino thiophenol (1 mmol) and aryl aldehydes (1 mmol) has dissolved in 2 mL ethanol in a 25 mL round bottom flask. The catalyst DBUH+I_3 complex (1a) (15 mol%) had added to the reaction mixture, and the reaction mixture was stirred

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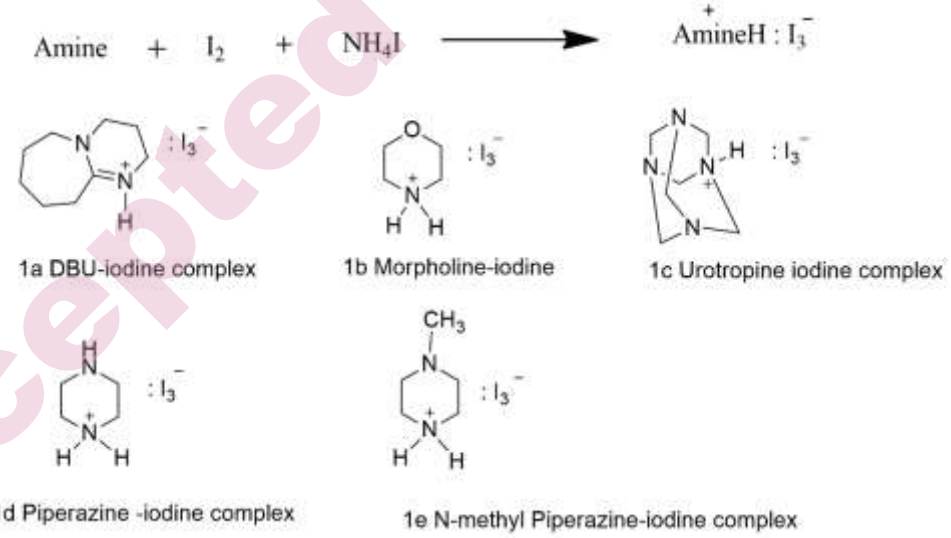
for 30 min. The progress of the reaction was monitored by (hexane: ethyl acetate) TLC. After completion of the reaction, the solvent has evaporated under a vacuum. The crude reaction mixture was quenched with 20 % sodium thiosulfate solution. The product was isolated by extracting with ethyl acetate. The organic layer was dried over sodium sulfate and purified by column chromatography. The structure of the compound had confirmed by the spectroscopic techniques and matched with the reported.

RESULTS AND DISCUSSION

We have prepared a series of R_2NH_2 + I_3 complexes (**1a-1e**) with minor modification in the reported procedure[27-28] by replacing potassium iodide with ammonium iodide. This change has led to a drastic change in the structure and composition of catalysts. In the previous reported procedure by Livia et.al.[72] has formed a precipitate of the complex with composition R_2NH: I_2: KI. In the present work, we have got a composition as R_2NH_2+ I_3 (**Scheme 1**). Amine must contain two heteroatoms in the cyclic system for precipitation and stability of the complex. The amine like pyrrolidine, piperidine, and amino acid viz proline did not form solid complexes by the same procedure as a result of a single nitrogen atom in the cyclic structure.



Scheme 1 Synthesis of Amine-H-I_3 complex and structure of respective complex

The various amine-iodine-iodide complexes have prepared using easily available amine, ammonium iodide, and molecular iodine. The molecular iodine was dissolved in the aqueous solution of ammonium iodide then added to an aqueous solution of amine dropwise, amine-iodine-iodide complex precipitate of respective amine obtained (**Table 1**). The product was washed with excess water till filtrate free from ammonia confirmed by moist turmeric paper.

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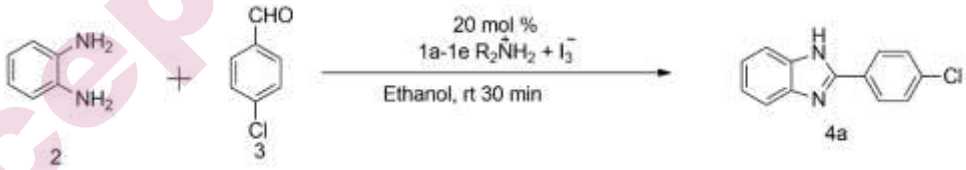
Table 1 : Synthesis of R_2NH_2+ I_3 complexes[a]

No.	Complex	Color	% Yield[b]
1	DBUH+I_3 complex	Greenish Yellow	92
2	MorpholineH+I_3 complex	Orange Yellow	62
3	UrotropineH+I_3 complex	Brown Yellow	58
4	PiperazineH+I_3 complexes	Dark Brown Yellow	73
5	N-methyl piperazineH+I_3 complexes	Pinkish Yellow	66s

[a]:Amine (6.568, 1 equivalent), Iodine (6.568 mmol, 1 equivalent) and Ammonium iodide (18.352 mmol, 2.5 equivalent) in 2mL water [b]: Isolated yield after purification

The structure of synthesized amine-iodine complexes (**1a-1e**) has confirmed by spectroscopic techniques such as UV, IR, HRMS, EDS, ^{1}HNMR, and ^{13}CNMR. These new homogenous catalysts have screened for the synthesis of 2-aryl benzimidazole. We have chosen ethanol as a solvent for screening catalytic activity of the amine-iodine complex catalyst because freely soluble in ethanol and partly soluble in various other organic solvents.

Initially, our studies have being with the screening of prepared amine iodine complexes (**1a-1e**) for synthesis of benzimidazole, via condensation and cyclization reaction of commercially available *o*-phenylenediamines with *p*-choro benzaldehyde (**Scheme 2**). The DBUH-I_3 complex has given high yield of 2-(4-chlorophenyl)-*1H*-benzimidazole and the results are given in **Table 2**.



Scheme 2 Model reaction for screening of R_2NH_2+ I_3 complex for synthesis of benzimidazole

Table 2 : Screening of R_2NH_2+ I_3 complex catalyst in the synthesis of 2-(4-chlorophenyl)-1H-benzimidazole(**4a**)[a]

Sr. No.	Complex	% Yield[b]
1	DBUH+I_3 complex	91
2	MorpholineH+I_3complex	74
3	UrotropineH+I_3 complex	85
4	PiperazineH+I_3 complexes	80
5	N-methyl piperazineH+I_3 complexes	78
6	Iodine	70
7	Without catalyst	Trace

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[a]Reaction condition: *o*-phenylenediamine (1 mmol), *p*-chlorobenzaldehyde (1 mmol), $R_2NH_2+I_3$ complex (**1a**-**1e**) (20 mol %) in ethanol (2 mL) at room temperature for 30 minutes; [b] isolated yield after purification

Next, we have decided to optimize the amount of DBUH+I_3 complex with the same reaction condition. The amount of DBUH-I_3 was optimized by increasing the amount from 5 mol % to 20 mol % for 1 mmol scale reaction. When the reaction has performed in the absence of the catalyst, the product has formed in a very trace amount (**Table 3, entry 1**). The yield has increased with the mol % of amine-iodine complex (**Table 3, entry 2-5**). Nevertheless, there was no increase in the yield when the amount of R_2NH_2+ I_3 catalyst loading has increased from 15 % mol to 20 % mol. From **Table 3**, it has observed, the 15 mol% of DBUH-I_3 complex was sufficient to achieve excellent yield.

Table 3: Optimizing the amount of DBUH+I_3 complex in synthesis of 2-(4-chlorophenyl)-1H-benzimidazole (4a)[a]

Entry	Catalyst quantity in mol %	% Yield[b]
1	Without catalyst	Trace
2	5	65
3	10	80
4	15	91
5	20	91

[a]Reaction condition: *o*-phenylenediamine (1 mmol), *p*-chlorobenzaldehyde (1 mmol), DBUH+I_3 complex (**1a**) (mol %) in ethanol (2 mL) at room temperature for 30 minutes; [b] isolated yield after purification.

We have studied the effect of various solvents on product yield (**Table: 4 entry 1-9**). Among the screened solvent, ethanol, toluene, and chloroform have given excellent yield, and ethanol has found the best solvent for the reaction as a high amount of product has obtained. Second, fortunately the choice of ethanol also falls on the fact that it is less toxic and more eco-sustainable solvent than chloroform and toluene. Hence, we have selected the solvent for the synthesis of benzimidazole. The solvent DMF, DMSO, and acetonitrile offered a moderate product yield.

Table 4: Effect of solvent in synthesis of 2-(4-chlorophenyl)-1H-benzimidazole (4a) using DBUH-I_3 complex catalyst[a]

Entry	Name of solvent	% Yield[b]
1	Ethanol	91
2	Toluene	86
3	Dimethyl formamide	58
4	Dimethyl sulphoxide	66
5	Chloroform	80

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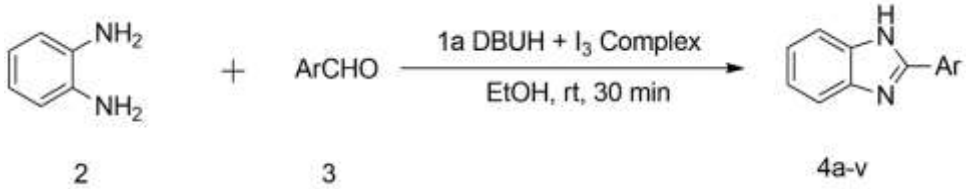
6	Acetic acid	50
7	Acetonitrile	61
8	Tetrahydrofuran	31
9	Water	25

[a]Reaction condition: *o*-phenylenediamine (1 mmol), *p*-chlorobenzaldehyde (1 mmol), DBUH+I_3 complex (**1a**) (mol %) in ethanol (2mL) at room temperature for 30 minutes; [b] isolated yield after purification

With the investigated optimum reaction condition, we have synthesized various substituted benzimidazole **(Scheme 3**). The 2-aryl substituted benzimidazole have been synthesized from o-phenylene diamine (1mmol) with several substituted aryl aldehyde (1 mmol) via condensation and cyclization reaction in the presence of DBUH+I_3 complex (15 mol%) at room temperature in ethanol (**Table 5**). It was found that various substituted aryl aldehyde containing electron-donating groups (*p*- halogen and methoxy, (**Table 5 entry 1, 4, 5, 16**) and electron-withdrawing group (nitro, **Table 5 entry 2, 6, 14**) were formed the product with good yield, under optimized condition. The heterocyclic aromatic aldehyde (**Table 5, entry 10a, 13a**) gave a comparatively lower yield under the same condition. Hydroxy benzaldehyde (**Table 5, entry 11, 12**) has afforded an unexpectedly low yield, which may be due to solubility in water. The aryl aldehyde bearing electron-withdrawing at ortho/para nitro group (**Table 5, entry 13, 15**) has afforded product in poor yield. The *o*-substituted aryl aldehyde (**Table 5, entry 3, 12, 15**) has afforded a low yield due to steric hindrance in cyclization.

Thus, the R_2NH_2+I_3 complex was catalyzing the synthesis of 2-aryl substituted benzimidazole using a diverse range of aryl aldehydes and *o*-phenylenediamine. All synthesized benzimidazole derivatives were characterized by ^{1}H NMR, ^{13}C NMR and compared physical constant with standard data. The ^{1}H NMR displays a characteristic nitrogen-bearing proton chemical shift value 12.5-13.5 δ reflected in each derivative whereas, the ^{13}C NMR show a typical chemical shift value 150 δ for carbon located between two nitrogens.



Scheme 3 DBU-Iodine-Iodide catalyzed synthesis of substituted benzimidazole

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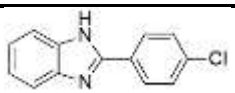
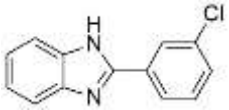
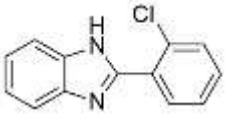
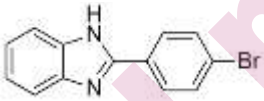
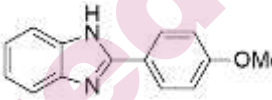
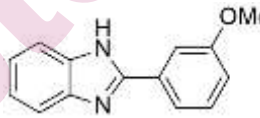
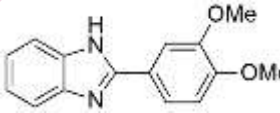
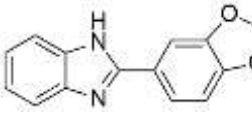
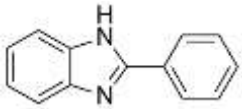
Table 5 : Synthesis of 2-aryl substituted benzimidazole[a]

Entry	Product (4)	% Yield[b]	M. P. °C	Literature M. P. °C
1	 **4a**	91[c]	290-293	290-292[29]
2	 **4b**	86[c]	228-230	227-229[20]
3	 **4c**	73[e]	232-234	231-233[29]
4	 **4d**	78[d]	286-290	292-293[29]
5	 **4e**	76[d]	223-225	222-223[30]
6	 **4f**	81[c]	202-205	200-202[29]
7	 **4g**	64[d]	225-227	223-226[31]
8	 **4h**	72[d]	238-240	239-241[30]
9	 **4i**	80[c]	243-245	242-244[20]

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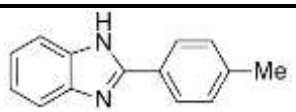
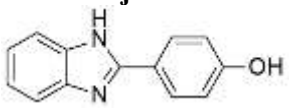
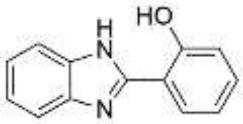
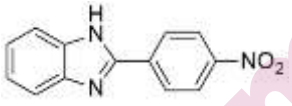
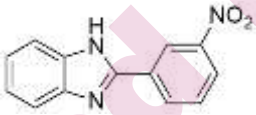
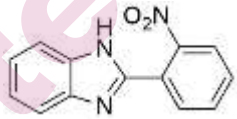
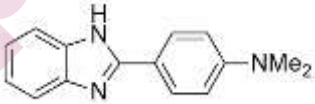
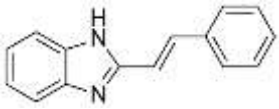
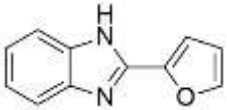
10	 **4j**	65[d]	216-219	214-216[20]
11	 **4k**	80[c]	252-254	254-255[29]
12	 **4l**	44[c]	204-206	205-206[31]
13	 **4m**	41[d]	301-303	300[18]
14	 **4n**	72[c]	196-198	199[18]
15	 **4o**	38[c]	229-231	230[18]
16	 **4p**	72[d]	280-283	277-279[29]
17	 **4q**	51[f]	270-273	164-166[20]
18	 **4r**	68[d]	226-228	221-223[20]

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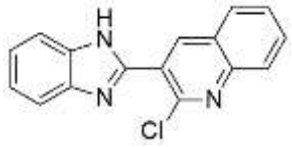
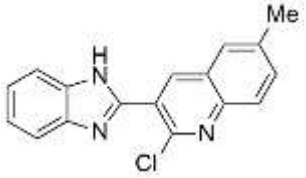
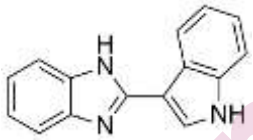
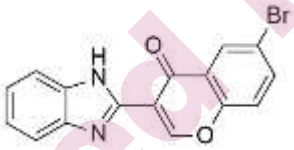
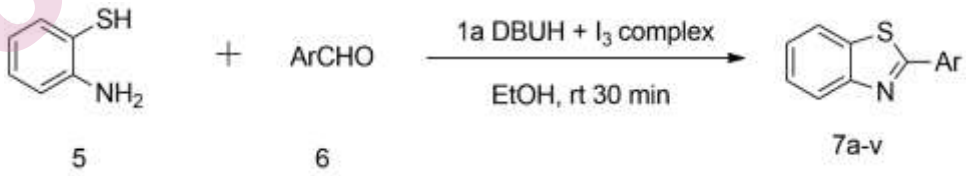
19	 **4s**	63[d]	219-222	202[32]
20	 **4t**	73[d]	221-224	220[33]
21	 **4u**	72[f]	220-223	226-227[23]
22	 **4v**	76[e]	269-271	---

[a]Reaction condition: *o*-phenylenediamine (1 mmol), substituted arylaldehyde (1 mmol), DBUH+I_3 complex (**1a**) (15 mol%), EtOH 2 ml, 30 min. at rt; [b]Isolated yield after purification; [c] product was purified by recrystallization in ethanol; [d]product was purified by column chromatography mobile phase hexane: ethyl acetate; [e]product was purified by recrystallization in chloroform; [f]product was purified by column chromatography mobile phase chloroform.



Scheme 4 DBUH+I_3 catalysed synthesis of benzothiazole derivatives.

The synthesis of 2-substituted aryl benzothiazole derivative (**Scheme 4**) has achieved from 2-amino thiophenol and diversity of aryl aldehydes in the presence of DBUH+I_3 complex (**1a**). The aromatic aldehyde bearing electron-donating group [*p*- halogen, methoxy, hydroxyl, amino, (**Table 6** entry 1, 4, 5, 8, 11, 12, 13, and 17)] and electron-withdrawing group (*m*- halogen, methoxy, nitro group,

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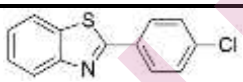
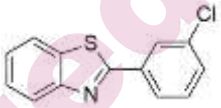
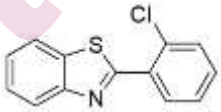
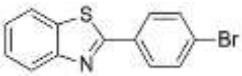
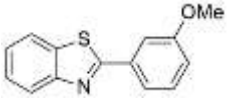
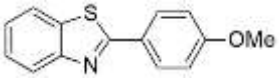
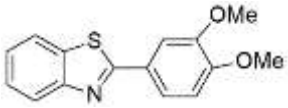
Table 6 entry 2, 6, 14, 15, 16) provided a good yield of the product under same optimized process. Also, this reaction works well with the heterocyclic aromatic aldehyde to form a product (**7**) in moderate yield (**Table 6, entry 19, 20, 21, 22**). The *o*-substituted benzaldehyde has afforded a poor yield of the product because of a steric hindrance (**Table 6 entry 3**). The unexpectedly *o*-nitro benzaldehyde has afforded a product in the higher yield owing to the high polarity of aldehyde **(Table 6 entry 16**). Overall, the amine-iodine complex has remarkably catalyzed the synthesis of 2-substituted aryl benzothiazole derivatives. The structure of all synthesized compounds has confirmed by NMR spectroscopic data and compared physical constant with standard data. The ^{13}C NMR spectra of benzothiazole have shown a characteristic value of chemical shift 168 δ for carbon between two heteroatoms sulfur and nitrogen.

Table 6 : Synthesis of 2-aryl substituted benzothiazole[a]

Entry	Product (7)	% Yield[b]	M. P. °C	Literature M. P. °C
1	 **7a**	84[d]	115-117	111-112[31]
2	 **7b**	72[c]	94-95	93-94[31]
3	 **7c**	58[d]	80-82	83-84[31]
4	 **7d**	80[c]	127-129	129-131[33]
5	 **7e**	74[c]	120-121	120-122[34]
6	 **7f**	64[d]	99-102	98-100[35]
7	 **7g**	61[f]	229-231	230-232[36]

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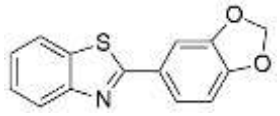
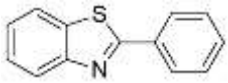
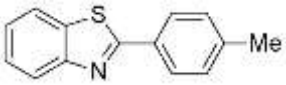
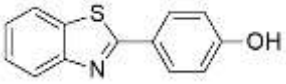
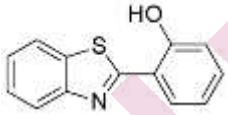
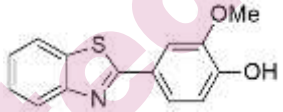
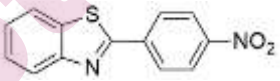
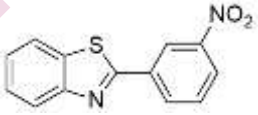
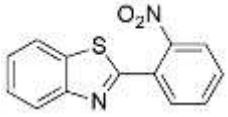
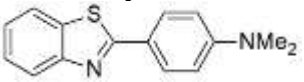
8	 **7h**	83[d]	130-132	128-130[37]
9	 **7i**	91[d]	112-113	109-110[33]
10	 **7j**	62[e]	85-86	87-88[38]
11	 **7k**	79[c]	227-229	225-227[39]
12	 **7l**	86[c]	131-132	124-126[39]
13	 **7m**	83[c]	160-162	161-163[39]
14	 **7n**	82[e]	320-322	228-230[39]
15	 **7o**	78[e]	190-193	185-187[36]
16	 **7p**	80[e]	195-197	191-193[40]
17	 **7q**	92[c]	161-163	160-162[39]

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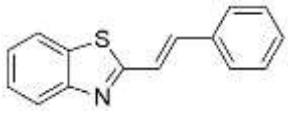
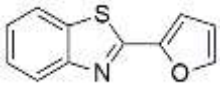
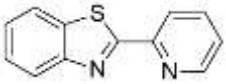
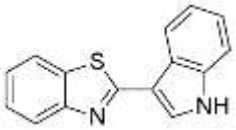
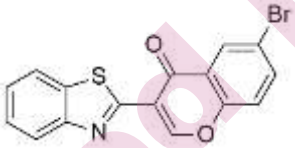
18	 **7r**	65[d]	107-110	110-112[39]
19	 **7s**	67[d]	103-104	101-102[35]
20	 **7t**	73[c]	132-134	130-132[40]
21	 **7u**	72[e]	146-148	144-147[40]
22	 **7v**	78[c]	254-256	New Compound

[a]Reaction condition: *o*-amino thiophenol (1 mmol), substituted arylaldehyde (1 mmol), DBUH+I_3 complex (**1a**) (15 mol%), Ethanol 2 mL, 30 min. at rt; [b]Isolated yield after purification; [c] product was purified by recrystallization in ethanol; [d]product was purified by column chromatography mobile phase hexane: ethyl acetate; [e]product was purified by recrystallization in chloroform; [f]product was purified by column chromatography mobile phase chloroform.

Further the scope of reaction has extended with the aliphatic aldehydes like crotonaldehyde, propionaldehyde, acetaldehyde, and formaldehyde with *o*-phenylenediamine and *o*-amino thiophenol. The reaction has not proceeded with aliphatic aldehydes and has not afforded the desired product.

Although the exact mechanism is not clear, a proposed mechanism for the formation of benzimidazole and benzothiazole is shown in **Scheme 5**. In first step the aldehyde (**A**) oxygen was protonted by abstraction of proton from DBUH + I_3 complex and form compound (**C**) and liberates DBU + I_3 complex. Simultaneously liberated DBU + I_3 complex, I^- abstract the hydrogen from amines (**B**) to form compound (**D**) and liberates DBU + I_2 complex. In next step (**C**) and (**D**) reacted to form intermediate (**E**). The intermediate (**E**) on reaction DBU + I_2 complex, DBU abstract the proton of XH to form X^- and Iodine coordinate with I_2 undergo

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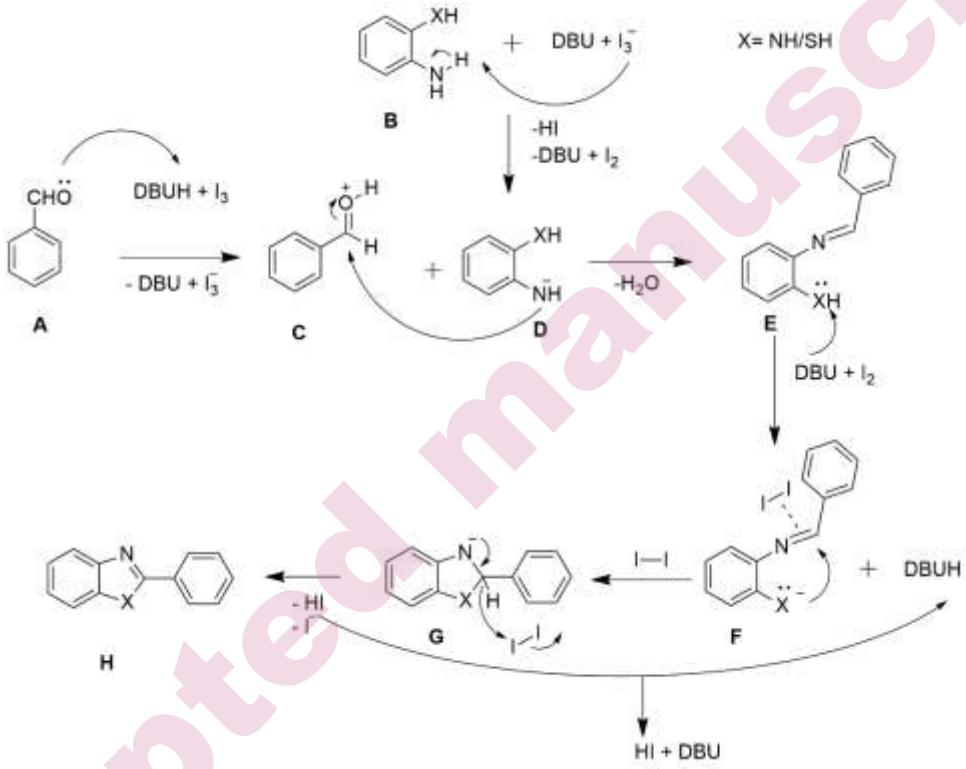
cyclization to form intermediate G which undergo oxidative elimination to form C-N double bond to formed final product (**H**).



Scheme 5 Tentative mechanism of DBUH-I_3 catalyzed synthesis of benzimidazole and benzothiazole

CONCLUSION

In the present work, we have prepared the new R_2NH_2+I_3 complexes and studied their catalytic activity in the preparation of 2-aryl substituted benzimidazole and benzothiazole derivatives. Among the screened Amine-Iodine catalysis, DBUH+I_3 has found an efficient catalyst for the preparation of 2-aryl substituted benzimidazole and benzothiazole. We have believed that the present method is more convenient, efficient, greener, simple, and environmentally benign than most reported methods in the Literature. The present method has not afforded the benzimidazole and benzothiazole derivatives with aliphatic aldehydes.

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SUPPLEMENTARY MATERIAL

Additional data are available electronically at the pages of journal website: https://www.shd-pub.org.rs/index.php/JSCS/article/view/11893 , or from the corresponding author on request.

ИЗВОД

КОМПЛЕКС DBUH+I3 КАО ЕФИКАСАН КАТАЛИЗАТОР ЗА СИНТЕЗУ ДЕРИВАТА 2-ФЕНИЛБЕНЗИМИДАЗОЛА И БЕНЗОТИАЗОЛА

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У овом раду је описана једноставна синтеза различитих бензимидазола/бензотиазола, употребом DBU-јод-јодида као једноставног и еколошки прихватљивог катализатора. Настаје комплекс R_2NH+I_3 у реакцији смеше амонијум јодида, молекулског јода и амонијака у води. Структура комплекса R_2NH+I_3 потврђена је спектроскопским техникама. Каталитичке особине добијеног амин-јодидног комплекса су испитане у реакцији синтезе бензимидазола/бензотиазола. Од испитаних катализатора DBUH+I3 комплекс се показао као ефикасан. Синтеза бензимидазола и бензотиазола је постигнута у реакцијама *о*-фенилендиамина /*о*- аминотиофенола са различитим супституисаним арил-алдехидима користећи DBUH+I3 комплекс као катализатор. У односу на друге, приказани протокол има неколико предности, као што су благи реакциони услови, комерцијално доступни прекурсори, катализатор који није скуп, кратко реакционо време, ширк опсег супстрата, висок принос, једноставан поступак изоловања производа, и поступак који није штетан за животну средину.

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J. Serb. Chem. Soc. **00(0)**S1-S212 (2023) *Supplementary material*

SUPPLEMENTARY MATERIAL TO

DBUH+I_3 complex an efficient catalyst for the synthesis of 2-phenyl benzimidazole and benzothiazole derivatives

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General:

All local brand chemicals were purchase checked their purity by TLC and purified. The melting point were determined in open capillary and are uncorrected. For analysis technique following instruments were used.Solvents that were entirely dry and free of impurities were used. Reaction of the progress was checked on Merck TLC Silica gel 60 F254 plates using UV lamp (365 nm and 254 nm) and iodine chamber.

Sr. No.	Analysis Type	Instrument
1	HRMS	Brucker Impact HD
2	UV-visible Spectrum	shimadzuCorp, Model UV-2600
3	IR Spectrum	shimadzu Corp, FTIR-shimay, Model IR affinity
4	FESM	FEI Nova NanoSEM 450
5	EDS	Brucker XFlash 6130
6	TGA-DTA	shimadzu Corp
7	NMR (^{1}H &^{13}C)	500MHz &125MHZ Brucker

Synthesis of amine-iodine complexes

Ammonium iodide (2.8 eq.) was added to water (2 volume) has obtained clear solution in 250 mL beaker and then added iodine (1 eq). This mixture of the solution was added dropwise to a stirred solution of amine (1 eq) in water (8 Volume) in 250 mL round bottom flask. The solid product has formed during addition, stirred mixture for 15 minutes and filter off the solid product. The product was washed with cold water and dried under vacuum to provide the desired complexes and the yield of the complex was reported.

Typical Process for the synthesis of benzimidazole / benzothiazole from o-phenylenediamine/thiophenol and aldehyde.

A mixture of *o*-phenylenediamine/*o*-amino thiophenol (1 mmol) and arylaldehyde (1 mmol) was dissolved in 2 mL ethanol in 25 mL round bottom flask. The catalyst (**1a**) (15mol%) was added and the reaction mixture was stirred for 30 min. The progress of the reaction was monitored by (hexane: ethyl acetate) TLC. The TLC clearly have showed the disappearance of thestarting material. After completion of the reaction, the solvent was evaporated under vacuum. The crude solid product was extracted in ethyl acetate after the addition of 20 % sodium thiosulphate solution. The organic layer was dried over sodium sulfate and purified by column chromatography. The structure of the compound was confirmed by the spectroscopic techniques and match with the reported.

1a. DBU-Iodine complex (Table 1, Entry 1, 1a): Greenish Yellow solid M. P. 87^{0}C.

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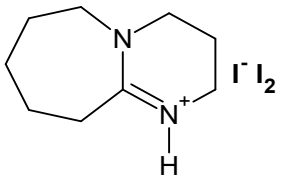
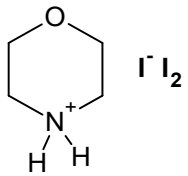


M. F. = $C_9H_{17}N_2\ I^-\ I_2$ Mol. Wt. = 533.79

HRMS: Positive ion polarity: 153.138 (cal. 153.242).
Negative ion polarity: 126.904 (cal. 126.904), 380.712 (cal. 380.713).
UV-visible Spectrum(nm): 210, 307,364 (λ_{max} =**364nm**).
IR Spectrum(cm^{-1}): 530, 601, 633, 1203, 1319, 1440, 1574, 1638, 3133, 3267.
SEM: Clumpy and agrummerated morphology.
Field Emission Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (FESEM - EDS):

Element	At. Number	Wt. %	At. %
Iodine	53	78.97	26.69
Carbon	6	17.57	62.74
Nitrogen	7	3.45	10.57
		100	100

TGA: DBU-iodine complex was stable up to 200°C after that gradual weight loss start up to 380°C then fast weight loss observed and stop 410°C. After 410°Cslow weight loss starts and end by complete vanishing of complex at 500°C.
DTA: Endotherm was observed at 110°Cand exotherm at 410°C. Both peaks are very sharp.
Strong exotherm and sharp weight loss was located in graph at 410°C.
^{1}HNMR:(500 MHz,DMSO-d_6):δ9.47 (s, 1H), 3.55 (t, 2H *J*=3.55 Hz), 3.48 (t, 2H *J*=3.48 Hz), 3.24-3.26 (m, 2H), 2.63 (t, 2H *J*=2.64 Hz), 1.92(q, *J*=1.94Hz) 1.54-1.72 (m, 6H);**^{13}CNMR:(125 MHz,DMSO-d_6) δ**:165.88, 53.89, 48.38, 38.10, 32.22, 28.70, 26.38, 23.78, 19.34.
1b. Morpholine-Iodine complex (Table 1, Entry 2, 1b): Orange Yellow solid M. P. 78^{0}C.



M. F. = $C_4H_9NO\ I^-\ I_2$ Mol. Wt. = 467.73

HRMS: Positive ion polarity: 88.075 (cal. 88.126).
Negative ion polarity: 126.905 (cal. 126.904), 380.713 (cal. 380.713).
UV-visible Spectrum(nm): 210, 360, 365, 366. (λ_{max} = **360nm**).

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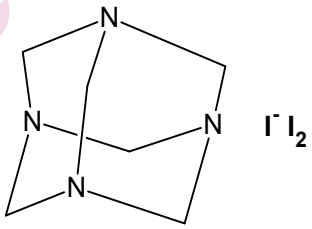
IR Spectrum (cm^{-1}): 585, 626, 817, 859, 1006, 1033, 1083, 1159, 1243, 1295, 1357, 1438, 2858, 3183.
SEM: Clumpy and agrummerated morphology.
Field Emission Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (FESEM - EDS):

Element	At. Number	Wt. %	At. %
Iodine	53	66.21	16.45
Carbon	6	23.94	62.86
Oxygen	8	5.36	10.57
Nitrogen	7	4.49	10.11
		100	100

TGA: The morpholine-iodine complex was stable up to 150°C then underwent fast weight loss till 300°Cthen gradual weight loss observed end at 500°Cby complete disappearing complex.
DTA: It displaysharp endotherm at 150°C and very broad exotherm peak at 480°C.
^{1}HNMR: (500 MHz, DMSO-d_6):δ 3.66-3.77 (m, 4H), 3.35-3.41(m, 4H);**^{13}CNMR:(125 MHz, DMSO-d_6)δ**:44.09, 45.24, 64.46, 65.55.
1c. Urotropine-Iodine complex (Table 1, Entry 3, 1c): Brown Yellow solid M. P. 130^{0}C.



M. F. = $C_6H_{13}N_4$ I^- I_2 Mol. Wt. = 521.76

HRMS: Positive ion polarity: 141.113 (cal. 141.192).
Negative ion polarity: 126.905 (cal. 126.904).
UV-visible Spectrum(nm): 308, 113, 324, 369 (λ_{max} = **369nm**).
IR Spectrum (cm^{-1}): 523, 656, 705, 734, 819, 901, 991, 1028, 1230, 1250, 1381, 1455.
SEM: Clumpy and agrummerated morphology.
Field Emission Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (FESEM - EDS):

Element	At. Number	Wt. %	At. %
Iodine	53	74.50	22.60
Carbon	6	16.03	51.37
Nitrogen	7	09.47	26.03
		100	100

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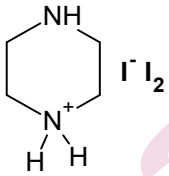
TGA: The complex was very stable up to 255°C after that sharp decrease in weight continue till temperature 340°C.
DTA: It show sharp three band at temperature 145°C, 255°Cand 450°C.
^{1}HNMR:(500 MHz, DMSO-d_6):δ4.73(s, 12H);**^{13}CNMR:(125 MHz, DMSO-d_6)δ**:73.85.

1d. Piperazine-Iodine complex (Table 1, Entry 4, 1d): Dark Brown Yellow Solid M. P. 346^{0}C.



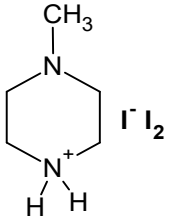
M. F. = $C_4H_{11}N_2$ I^- I_2 Mol. Wt. = 467.74

HRMS: Positive ion polarity: 87.091(cal. 87.142).
Negative ion polarity: 126.905 (cal. 126.904).
UV-visible Spectrum(nm): 210, 306, 319, 361, 368 (**λ_{max} = 368nm**).
IR Spectrum (cm^{-1}): 636, 860, 988, 1084, 1242, 1358, 1400, 1436, 3180.
SEM: Clumpy and agrummerated morphology.
Field Emission Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (FESEM - EDS):

Element	At. Number	Wt. %	At. %
Iodine	53	89.50	45.67
Carbon	6	07.55	40.73
Nitrogen	7	02.94	13.60
		100	100

TGA: The complex show stability till temperature 115°Cafter sharp and slow weight loss continue up to 325°C.
DTA: This graph indicates one sharp exothermic band at 325°C.
^{1}HNMR: (500 MHz, DMSO-d_6): δ8.48(s, 2H), 3.81 (s, 1H), 3.22(t, 1H, *J*=3.22 Hz), 3.07 (t, 4H, *J*= 3.08Hz), 2.99 (s, 1H), 2.61-2.64 δ(q, 1H);**^{13}CNMR: (125 MHz, DMSO-d_6) δ**:47.85, 46.74, 44.28, 43.54.

1e. N-Methyl-Piperazine-Iodine complex (Table 1, Entry 5, 1e): Pinkish Yellow solid M. P. 178^{0}C.



M. F. = $C_5H_{13}N_2$ I^- I_2 Mol. Wt. = 481.75

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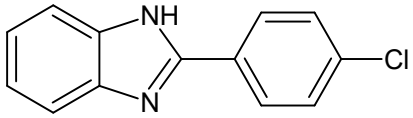
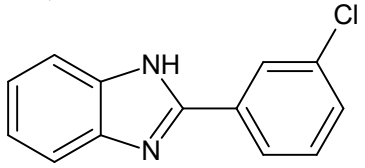
HRMS: Positive ion polarity: 101.107 (cal. 101.168).
Negative ion polarity: 126.905 (cal. 126.904).
UV-visible Spectrum(nm): 210, 306, 317, 365 (λ_{max} = **364nm**).
IR Spectrum (cm^{-1}): 573, 847, 893, 960, 990, 1100, 1365, 1438, 1553, 1651, 2436, 2707.
SEM: Clumpy and agrummerated morphology.
Field Emission Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (FESEM - EDS):

Element	At. Number	Wt. %	At. %
Iodine	53	76.30	23.96
Carbon	6	18.18	60.32
Nitrogen	7	05.52	15.71
		100	100

TGA: The complex was stable till 200°C above this temperature gradual weight loss till 320°C.
DTA: One sharp exothermic band observed at 320°C.
^{1}HNMR:(500 MHz, DMSO-d_6): δ8.46 (s, 2H), 2.96-3.05(m, 4H), 2.61-2.63(m, 4H), 2.35(s, 3H);**^{13}CNMR:(125 MHz, DMSO-d_6) δ**:51.47, 45.40, 43.02.
CHARACTERISATION DATA OF 2-SUBSTITUTED PHENYL BENZIMIDAZOLE.
1. 2-(4-chlorophenyl)-*1H*-benzimidazole (Table 5, Entry 1, 4a): Yellow solid M. P. 290-293°C (290-292°C)[1]



^{1}HNMR: (500 MHz, DMSO-d_6): **δ** 12.98 (s, 1H), 8.17-8.20 (m, 2H), 7.73 (d, 1H, *J*=7.73 Hz), 7.64-7.68 (m, 1H), 7.63 (t, 1H, *J*= 7.62 Hz), 7.61 δ (d, 1H *J*= 7.60 Hz), 7.20-7.36 (m, 2H); **^{13}CNMR: (125 MHz, DMSO-d_6)δ**: 150.61, 144.20, 135.48, 134.95, 129.54, 129.27, 128.60, 123.24, 122.31, 119.43, 111.88.
2. 2-(3-chlorophenyl)-1*H*-benzimidazole (Table 5, Entry 2, 4b): Brown solid M. P. 228-230°C (227-229°C)[2]



^{1}HNMR: (500 MHz, DMSO-d_6): **δ** 13.04 (s, 1H), 8.23 (t, 1H *J*= 8.22 Hz), 8.17 (t, 1H *J*= 8.17 Hz), 8.15 (t, 1H, *J*= 8.13 Hz) 7.57-7.66 (m, 1H), 7.55 (t, 1H, *J*= 7.54 Hz), 7.30 (q, 1H), 7.20 – 7.27 δ (m, 2H); **^{13}CNMR: (125 MHz, DMSO-**

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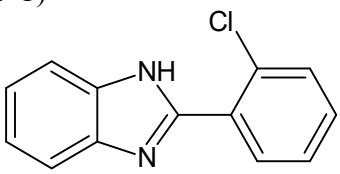
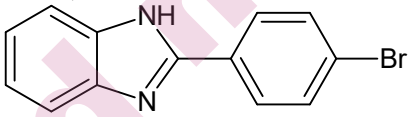
***d*$_6$)δ**: 150.19, 144.11, 135.45, 134.23, 132.67, 131.42, 130.01, 126.48, 125.48, 123.43, 122.41, 119.56, 111.98.

3. 2-(2-chlorophenyl)-*1H*-benzimidazole (Table 5, Entry 3, 4c):Yellow solid M. P. 232-234°C (231-233^{0}C)[1]

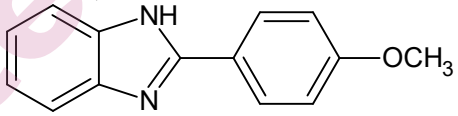


^{1}HNMR: (500 MHz, DMSO-*d*$_6$): δ 12.73 δ (s, 1H), 7.90-7.91 (m, 1H), 7.66 (d, 1H, *J*= 7.65 Hz), 7.65 (d, 2H, *J*=7.65 Hz), 7.50-7.56 (m, 2H), 7.22-7.26 (m, 2H); **^{13}CNMR: (125 MHz, DMSO-*d*$_6$)δ** :149.55, 132.56, 132,09, 131.68, 130.82, 130.43, 127.91, 122.72, 120.07.

4. 2-(4-bromophenyl)-*1H*-benzimidazole (Table 5, Entry 4, 4d):Yellow solid M. P. 286-290°C (292-293°C)[1]

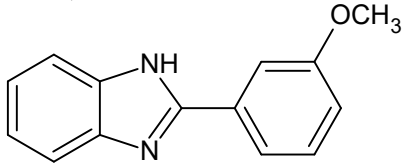


5. 2-(4-Methoxyphenyl)-*1H*-benzimidazole (Table 5, Entry 5, 4e):White solid M. P. 223-225°C (222-223°C)[3]



^{1}HNMR: (500 MHz, DMSO-*d*$_6$): δ 12.73 (s, 1H), 8.10 - 8.12 (m, 2H), 7.61 (d, 1H, *J*= 7.60 Hz), 7.48 (d, 1H, *J*=7.48 Hz), 7.17 (t, 2H, *J*= 7.16 Hz) 7.13 (d 1H *J*=7.13 Hz) 7.11 (d, 1H *J*=7.10 Hz) 3.84 (s, 3H); **^{13}CNMR: (125 MHz, DMSO-*d*$_6$)δ**: 161.05, 151.79, 144.34, 135.43, 128.45, 123.15, 122.53, 121.90, 118.95, 114.83, 111.49.

6. 2-(3-methoxyphenyl)-1*H*-benzimidazole (Table 5, Entry 6, 4f):Yellow solid M. P. 202-205°C (200-202°C)[1]



7. 2-(3,4-dimethoxyphenyl)-*1H*-benzimidazole (Table 5, Entry 7, 4g): White solid M. P. 225-227°C (223-226°C)[4]

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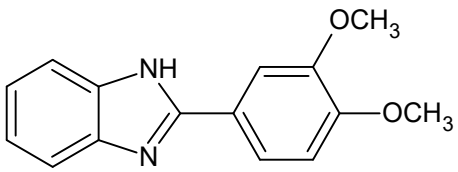
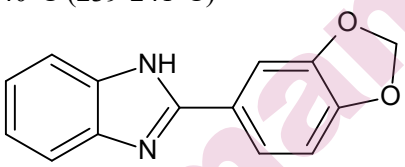
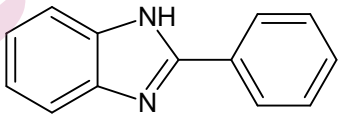


8. 2-(2H-1,3-benzodioxol-5-yl)-*1H*-benzimidazole (Table 5, Entry 8, 4h):Yellow solid 238-240°C (239-241°C)[3]



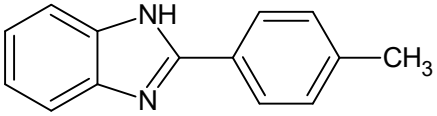
^{1}HNMR: (500 MHz, DMSO-d_6): δ 12.80 (s, 1H), 7.67 (q, 1H, *J*= 7.66 Hz), 7.45-7.47 (m, 1H), 7.27 (d, 1H, *J*= 7.26 Hz), 7.21-7.23 (m, 3H), 6.59(d, 1H, *J*= 6.58 Hz), 5.96 (s, 2H); **^{13}CNMR: (125 MHz, DMSO-d_6)δ**: 153.50, 148.07, 147.04, 143.02, 136.25, 124.22, 123.00, 122.60, 119.85, 119.55, 111.53, 108.91, 107.22, 101.58.

9. 2-phenyl-*1H*-benzimidazole (Table 5, Entry 9, 4i): Brown solid M. P. 243-245°C (242-244°C)[2]

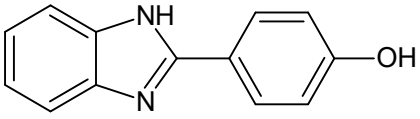


^{1}HNMR: (500 MHz, DMSO-d_6): δ 12.91 (s, 1H), 8.19 (t, 2H *J*=8.18 Hz), 7.67 (d, 1H, *J*=7.67 Hz), 7.53-7.57 (m, 3H), 7.50 (t, 1 H *J*=7.48 Hz) 7.18-7.24 (m, 2H); **^{13}CNMR: (125 MHz, DMSO-d_6)δ**: 151.68, 144.28, 135.47, 130.64, 130.30, 129.41, 129.25, 127.09, 126.90, 122.99, 122.13, 119.34, 111.78.

10. 2-(4-methylphenyl)-*1H*-benzimidazole (Table 5, Entry 10, 4j):Brown Solid M. P. 216-219°C (214-216°C)[2]



11. 4-(*1H*-benzimidazole-2-yl) phenol (Table 5, Entry 11, 4k): White solid M. P. 252-254°C (254-255°C)[1]



^{1}HNMR: (500 MHz, DMSO-d_6): δ 15.33 (s, 1H), 10.87 (s, 1H), 8.25 (d, 2H *J*= 8.24 Hz) 7.77-7.81 (m, 2H), 7.51-7.54 (m, 2H), 7.09-7.11 (d, 2H, *J*=7.09 Hz);

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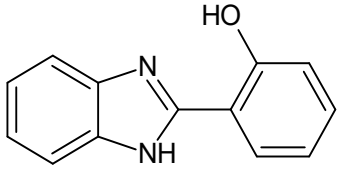
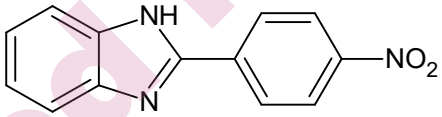
^{13}CNMR: (125 MHz, DMSO-d_6)δ: 162.86, 149.60, 132.12, 130.74, 125.97, 116.98, 114.04, 113.78.

12. 2-(*1H*-benzimidazole-2yl) phenol (Table 5, Entry 12, 4l):Brown solid 204-206°C (205-206°C)[5]

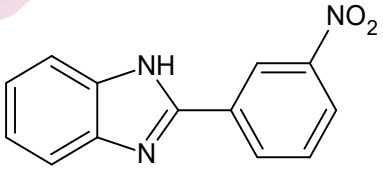


^{1}HNMR: (500 MHz, DMSO-d_6): δ 13.29 (s, 1H), 13.09 (s, 1H), 8.06 (d, 1H, *J*=8.05 Hz), 7.94-7.97 (m, 2H), 7.90 (d, 1H, *J*=7.89Hz), 6.61-7.64 (m, 2H), 7.48-7.51(m, 1H), 7.38-7.41(m, 1H); **^{13}CNMR: (125 MHz, DMSO-d_6)δ**: 156.85, 152.58, 142.33, 131.85, 128.83, 127.16, 123.10, 122.99, 119.50, 116.88, 115.50, 111.29.

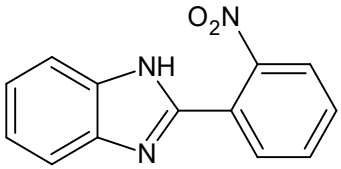
13. 2-(4-nitrophenyl)-*1H*-benzimidazole (Table 5, Entry 13 4m):Yellow solid M. P. 301-303°C (300°C)[6]



14. 2-(3-nitrophenyl)-*1H*-benzimidazole (Table 5, Entry 14, 4n):Yellow solid M. P. 196-198°C (199°C)[6]



15. 2-(2-nitrophenyl)-*1H*-benzimidazole (Table 5, Entry 15, 4o):Yellow solid M. P. 229-231°C (230°C)[6]



^{1}HNMR: (500 MHz, DMSO-d_6): δ 13.06 (s, 1H), 8.03 (dd, 1H, *J*=8.02 Hz), 7.98 δ (dd, 1H, *J*=7.97 Hz) 7.85-7.88 (m, 1H), 7.74-7.77 (m, 1H) 7.69 (d, 1H, *J*=7.65 Hz), 7.57 (d, 1H *J*=7.56 Hz) 7.20-7.29 (m, 2H); 13**CNMR: (125 MHz, DMSO-d_6)δ**:149.42, 147.76, 144.05, 135.07, 133.12, 131.38, 124.77, 124.67, 123.56, 122.36, 119.71, 112.14.

16. 4-(*1H*-benzimidazole-2-yl)-N, N-dimethylaniline (Table 5, Entry 16,4p):Yellow solid M. P. 280-283°C (277-279°C)[1]

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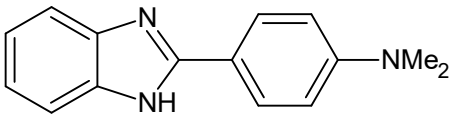
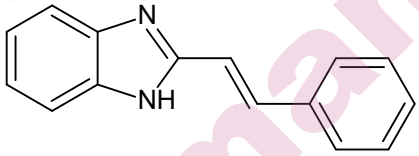
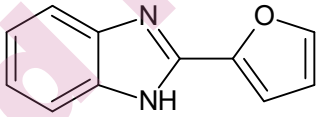


^{1}HNMR: (500 MHz, $CDCl_3$): 12.51 (s, 1H), 7.61-7.64 (m, 1H), 7.23-7.26 (m, 1H), 7.15-7.20 (m, 1H), 7.01 (d, 1H, *J*=7.01 Hz), 6.67-6.74 (m, 2H), 3.00 (m, 3H), 2.92 (m, 3H); **^{13}CNMR: (125 MHz, $CDCl_3$)δ**: 155.04, 149.98, 143.30, 136.38, 130.31, 126.94, 124.34, 122.16, 119.26, 117.39, 112.81, 111.81, 110.40, 40.57, 40.23.

17. 2-[(*E*)-2-phenylethenyl]-*1H*-benzimidazole (Table 5, Entry 17 4q):Yellow solid M. P. 270-273°C (164-166°C)[2]

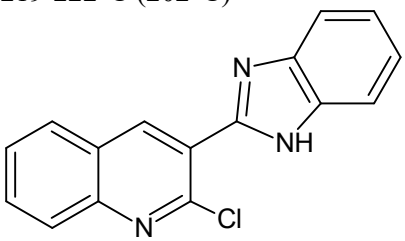


18. 2-(furan-2-yl)-*1H*-benzimidazole (Table 5, Entry 18, 4r):Brown solid M. P. 226-228°C(221-223°C)[2]



^{1}HNMR: (500 MHz, DMSO-d_6): δ 12.95(m, 1H), 7.96(dd, *J*=1.71&0.90Hz, 1H), 7.57(d, *J*=7.11Hz, 1H), 7.51 (d, *J*=7.15Hz, 1H) 7.17-7.22 (m, 3H), 6.72(dd, *J*=3.4 Hz &0.95 Hz, 1H); **^{13}CNMR: (125 MHz, DMSO-d_6) δ**:147.12, 143.37, 135.00, 134.44, 129.02, 128.68, 127.15, 123.01, 121.90, 117.81, 112.59.

19. 3-(*1H*-benzimidazole-2-yl)2-chloroquinoline (Table 5, Entry 19, 4s):Yellow solid M. P. 219-222°C (202°C)[7]



^{1}HNMR: (500 MHz, DMSO-d_6): δ 10.41 (s, 1H), 9.34 (s, 1H), 8.08 (d, 1H, *J*=8.06 Hz), 8.06 (d, 1H, *J*=8.05Hz), 7.98-8.00 (m, 1H), 7.81-7.85 (m, 1H), 7.35-7.60 (m, 2H); **^{13}CNMR: (125 MHz, DMSO-d_6) δ**: 147.57, 147.02, 145.58, 143.07, 141.48, 133.96, 131.98, 128.41, 128.30, 128.09, 127.05, 124,12, 123.22, 122,74, 119.86, 111.29.

20. 3-(*1H*-benzimidazol-2-yl)-2-chloro-6-methylquinoline (Table 5, Entry 20, 4t):White solid M. P. 221-224°C (220°C)[7]

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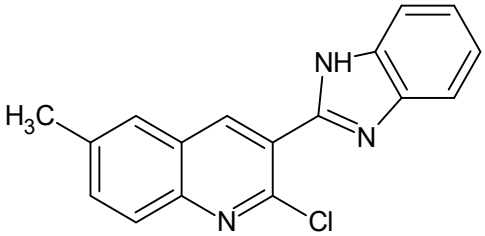
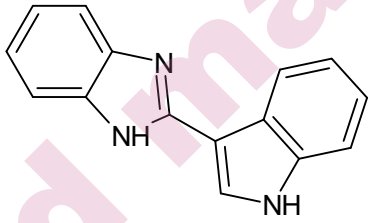


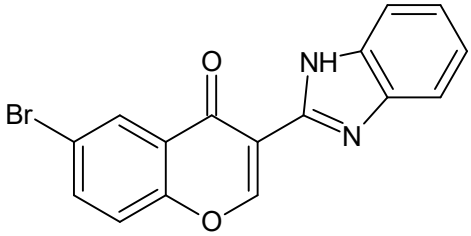
^{1}HNMR: (500 MHz, DMSO-*d*$_6$): δ 12.94 (s, 1H), 8.87 (s, 1H), 8.59 (s, 1H), 7.75-7.78 (m, 2H), 7.61-7.74 (m, 2H), 7.28-7.59 (m, 2H), 2.63 (s, 3H); **^{13}CNMR: (125 MHz, DMSO-*d*$_6$) δ**: 148.31, 146.73, 145.95, 141.20, 138.32, 134.71, 127.93, 127.60, 126.83, 124.87, 123.53, 122.41, 119.66, 112.32, 21.64.

21. 2-(1*H*-indol-2-yl)-*1H*-benzimidazole (Table 5, Entry 21, 4u):Black solid 220-223°C (226-227°C)[17]



^{1}HNMR:(500 MHz DMSO-*d*$_6$): **δ** 12.59 (s, 1H), 11.66 (s, 1H), 8.49 (t, 1H, *J*= 8.48 Hz), 8.14 (d, 1H, *J*=8.13 Hz), 7.49-7.55 (m, 2H), 7.54-7.55 (m, 2H), 7.49-7.50 (q, 1H), 7.19-7.21 (m, 1H), 7.13-7.16 (m, 1H); **^{13}CNMR:**(125 MHz DMSO-*d*$_6$) δ: 149.84, 136.96, 126.77, 125.54, 122.73, 121.78, 120.78, 112.41, 106.74.

22. 3-(*1H*-benzimidazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one (Table 5, Entry 22, 4v): Yellow solid M. P. 269-271°C.



^{1}HNMR: (500 MHz, DMSO-*d*$_6$):δ 12.65 (s, 1H), 8.32 (s, 1H), 9.41 (s, 1H), 8.32 (d, 1H, *J*= 8.31 Hz), 8.06-8.09 (m, 1H), 7.67-7.70 (m, 1H), 7.62-7.66 (m, 1H), 7.18-7.22 (m, 2H); **^{13}CNMR: (125 MHz, DMSO-*d*$_6$) δ**: 174.11, 158.91, 155.05, 145.27, 142.70, 137.89, 134.93, 127.82, 125.57, 122.70, 122.40, 121.96, 119.22, 118.73, 115.03, 112.97.

HRMS: [MF: $C_{16}H_{10}O_2N_2Br$(M+H)]: 342.99 (Calculated: 342.16)

CHARACTERISATION DATA OF 2-SUBSTITUTED PHENYL BENZOTHIAZOLE.

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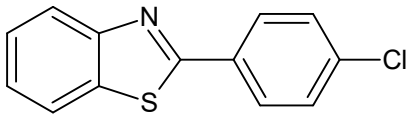
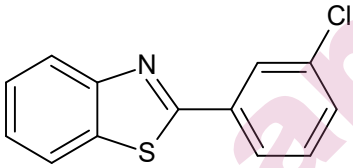
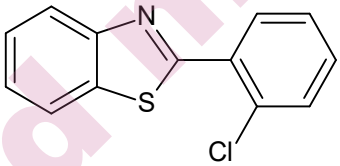
1. 2-(4-chlorophenyl)-1,3-benzothiazole (Table 6, Entry 1, 7a): White solid M. P. 115-117°C (111-112°C)[8]



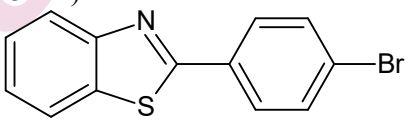
2. 2-(3-chlorophenyl)-1,3-benzothiazole (Table 6, Entry 2. 7b): White solid M. P. 94-95°C (93-94°C)[8]



3. 2-(2-chlorophenyl)-1,3-benzothiazole (Table 6, Entry 3, 7c): White solid M. P. 80-82°C (83-84°C)[8]

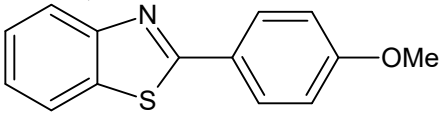


4. 2-(4-bromophenyl)-1,3-benzothiazole (Table 6, Entry 4, 7d): White Solid M. P. 127-129°C (129-131°C)[9]



^{1}HNMR: (500 MHz, $CDCl_3$): **δ** 8.06 (d, 1H, *J*=8.05 Hz), 7.94-7.91 (m, 2H), 7.90(d, 1H, *J*= 7.89 Hz), 7.61-7.64(m, 2H), 7.48-7.51 (m, 1H), 7.38-7.41 (m, 1H); **^{13}CNMR: (125 MHz, $CDCl_3$)δ**: 166.70, 154.06, 135.03, 132.54, 132.23, 128.90, 126.51, 125.45, 125.42, 123.31, 121.67.

5. 2-(4-methoxyphenyl)-1,3-benzothiazole (Table 6, Entry 5, 7e): White Solid M. P. 120-121°C (120-122°C)[9]



^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.02-8.04 (m, 3H), 7.86 (d, 1H, *J*=7.86 Hz), 7.44-7.48 (m, 1H), 7.33-7.36 (m, 1H), 6.98-7.01 (m, 2H), 3.87 (s, 3H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 167.86, 161.91, 154.22, 134.85, 129.10, 126.43, 126.19, 124.78, 122.81, 121.50, 114.36, 55.46.

6. 2-(3-methoxyphenyl)-1,3-benzothiazole (Table 6, Entry 6, 7f): Yellow solid M. P. 99-102°C (98-100°C)[10]

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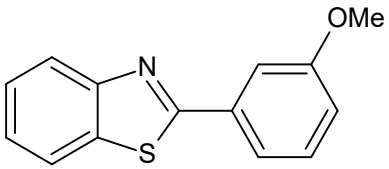
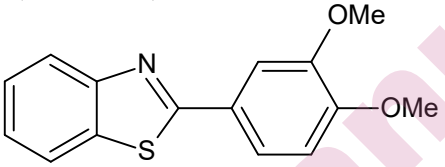
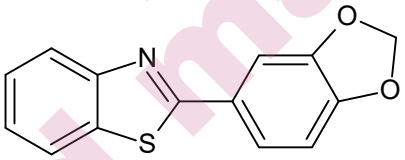


7. 2-(3,4-dimethoxyphenyl)-1,3-benzothiazole (Table 6, Entry 7, 7g): Brown solid M. P. 229-231°C (230-232°C)[11]

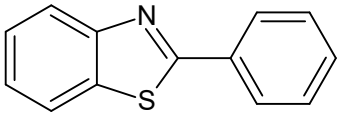


8. 2-(2*H*-1,3-benzodioxol-5-yl)-1,3-benzothiazole (Table 6, Entry 8, 7h):Yellow solid M. P. 130-132°C (128-130°C)[12]

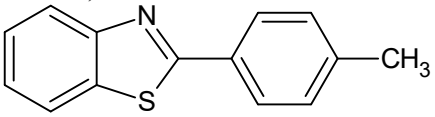


^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.00 (d, 1H, *J*=7.99 Hz), 7.81 (d, 1H, *J*=7.81 Hz), 7.57 (d, 1H, *J*=7.56 Hz), 7.42-7.45 (m, 1H), 7.30-7.35 (m, 1H), 6.85 (d, 1H, *J*=6.84 Hz), 5.99 (s, 2H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 167.49, 154.01, 150.01, 148.29, 134.80, 127.94, 126.20, 124.89, 122.86, 122.66, 122.43, 108.56, 107.43, 101.67.

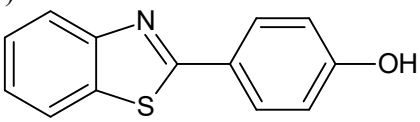
9. 2-phenyl-1,3 benzothiazole (Table 6, Entry 9, 7i): White solid M. P. 112-113°C (109-110°C)[8]



10. 2-(4-methylphenyl)-1, 3-benzothiazole (Table 6, Entry, 10, 7j): Yellow solid M. P. 85-86°C (87-88°C)[13]



11. 4-(1,3-benzothiazol-2-yl) phenol (Table 6, Entry 11, 7k): White solid M. P. 227-229°C (225-227°C)[14]



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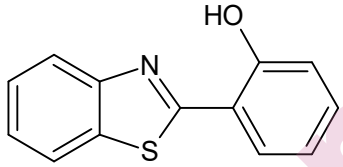
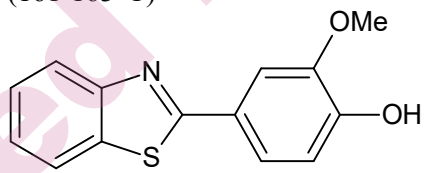
^{1}HNMR: (500 MHz DMSO-d_6): δ 10.24 (s, 1H), 8.09 (d, 1H, *J*=8.07), 8.07 (d, 1H, *J*=8.06), 7.93-7.00 (m, 2H), 7.49-7.52 (m, 1H), 7.39-7.42 (m, 1H), 6.96 (t, 2H, *J*=6.95); **^{13}CNMR: (125 MHz DMSO-d_6) δ**: 167.92, 160.99, 154.19, 134.57, 129.74, 129.51, 129.27, 126.89, 125.36, 124.50, 122.76, 122.58, 116.55.

12. 2-(1,3-benzothiazol-2-yl) phenol (Table 6, Entry 12, 7l): White solid M. P. 131-132°C (124-126°C)[14]



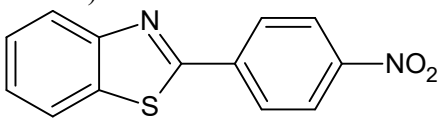
^{1}HNMR: (500 MHz, $CDCl_3$): δ 12.50 (s, 1H), 7.97 (d, 1H, *J*=7.96 Hz), 7.87 (d, 1H, *J*=7.87 Hz), 7.66-7.68 (m, 1H), 7.47-7.50 (m, 1H), 7.35-7.70 (m, 2H), 7.09-7.10 (m, 1H), 6.92-6.96 (m, 1H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 169.35, 157.92, 151.81, 132.73, 132.56, 128.39, 128.14, 126.66, 125.52, 122.16, 121.49, 119.53, 117.85.

13. 4-(1,3-benzothiazol-2-yl)-2-methoxyphenol (Table 6, Entry 13, 7m): White solid M. P. 160-162°C (161-163°C)[14]



^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.03 (d, 1H, *J*= 8.01 Hz), 7.86 (q, 1H), 7.71 (d, 1H, *J*= 7.70 Hz), 7.54 (q, 1H), 7.45-7.48 (m, 1H), 7.33-7.37 (m, 1H), 7.00 (q, 1H), 6.10 (s, 1H) 4.00(s, 3H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 168.15, 154.04, 148.52, 146.95, 134.81, 126.22, 126.17, 124.84, 122.72, 121.94, 121.51, 114.71, 109.24, 56.17.

14. 2-(4-nitrophenyl)-1,3-benzothiazole (Table 6, Entry14, 7n): Brown solid M. P. 320-322°C (228-230°C)[14]



^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.92 (s, 1H), 8.40 (d, 1H, *J*=8.40 Hz), 8.31 (d, 1H, *J*=8.30 Hz), 8.11 (d, 1H, *J*=8.10 Hz), 7.94 (d, 1H, *J*=7.93 Hz), 7.67 (t, 1H, *J*= 7.68 Hz), 7.56 (t, 1H, *J*=7.56 Hz), 7.45 (t, 1H, *J*=7.45 Hz); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 164.89, 153.93, 148.74, 135.17, 133.01, 130.12, 126.85, 126.05, 125.19, 123.75, 122.69, 122.32, 121.85.

15. 2-(3-nitrophenyl)-1,3-benzothiazole (Table 6, Entry 15, 7o): Yellow solid M. P. 190-193°C (185-187°C)[15]

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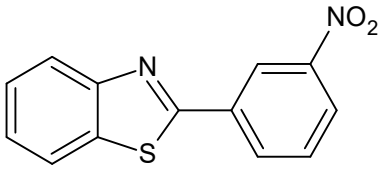
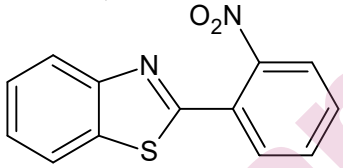
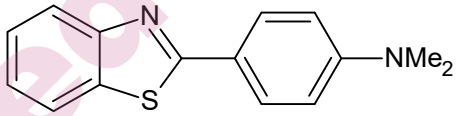


16. 2-(2-nitrophenyl)-1,3-benzothiazole (Table 6, Entry 16, 7p): Orange brown solid M. P. 195-197°C (191-193°C)[15]

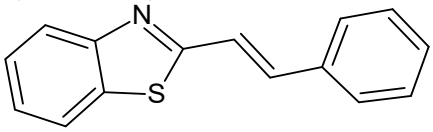


^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.08 (d, 1H *J*=8.07 Hz), 7.88-7.94 (m, 2H) 7.79 (q, 1H), 7.67-7.70 (m, 1H), 7.61-7.64 (m, 1H), 7.51-7.54 (m, 1H), 7.43-7.46 (m, 1H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 162.40, 153.51, 148.91, 135.79, 132.39, 131.81, 130.93, 128.10, 126.59, 125.87, 124.61, 123.94, 121.58.

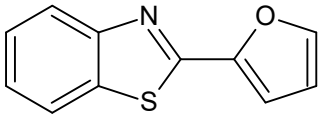
17. 4-(1,3-benzothiazol-2-yl)-*N*, *N*-dimethylaniline (Table 6, Entry 17, 7q):White solid 161-163°C (160-162°C)[14]



18. 2-[(*E*)-2-phenylethenyl]-1,3-benzothiazole (Table 6, Entry 18, 7r):White solid M. P. 107-110°C (110-112°C)[14]



19. 2-(furan-2-yl)-1,3-benzothiazole (Table 6, Entry 19, 7s): White solid M. P. 103-104°C (101-102°C)[10]



^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.04 (d, 1H, *J*=8.04 Hz), 7.88 (d, 1H, *J*=7.88 Hz), 7.60 (d, 1H, *J*=7.59 Hz), 7.47-7.50 (m, 1H), 7.36-7.39 (m, 1H), 7.18 (d, 1H, *J*=7.18 Hz), 6.59-6.60 (m, 1H); **^{13}CNMR: (125 MHz, $CDCl_3$)δ**:157.56, 153.74, 148.73, 144.70, 134.26, 126.48, 125.19, 123.11, 121.57, 112.53, 111.43.

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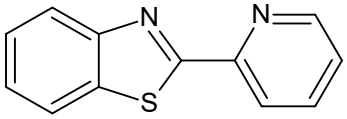
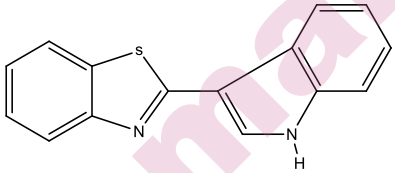
20. 2-(pyridin-2-yl)-1,3-benzothiazole (Table 6, Entry 20, 7t): Brown solid 132-134°C (130-132°C)[16]



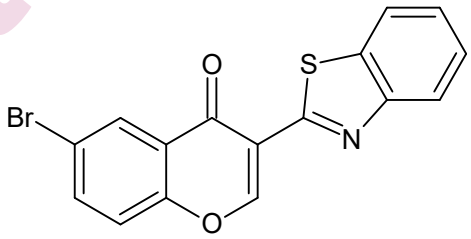
^{1}HNMR: (500 MHz, $CDCl_3$): δ 8.67-8.68 (m, 1H), 8.36 (d, 1H, *J*=8.35 Hz), 8.08 (d, 1H, *J*=8.08 Hz), 7.94 (d, 1H, *J*=7.94 Hz), 7.80-7.84 (m, 1H), 7.47-7.51 (m, 1H), 7.34-7.42 (m, 2H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 169.35, 154.25, 151.36, 149.63, 136.99, 136.09, 126.26, 125.63, 125.25, 123.55, 122.00, 120.73.

21. 2-(1*H*-indol-2-yl)-1,3-benzothiazole (Table 6, Entry 21, 7u):Brown solid M. P. 146-148°C (144-147°C)[16]



^{1}HNMR:(500 MHz, $CDCl_3$):δ 8.82 (s, 1H), 8.44 (d, 1H, *J*=8.43 Hz), 8.03 (d, 1H, *J*=8.03 Hz), 7.93 (d, 1H, *J*=7.92 Hz), 7.88 (d, 1H, *J*=7.87 Hz), 7.46 δ (t, 1H, *J*=7.46 Hz), 7.43 (t, 1H, *J*=7.41 Hz) 7.35-7.28 (m, 3H); **^{13}CNMR: (125 MHz, $CDCl_3$) δ**: 163.00, 153.730, 136.46, 133.84, 126.34, 126.07, 124.92, 124.23, 123.44, 122.11, 121.83, 121.30, 121.05, 112.46, 111.67.

22: 3-(1,3-benzothiazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one. (Table 6, Entry 22, 7v): Yellow solid M. P. 254-256 °C.



^{1}HNMR:(500 MHz, $CDCl_3$): **δ** 9.28 (s, 1H), 8.50 (d, 1H, *J*=8.49 Hz), 7.98-8.04 (m, 2H), 7.83 (q, 1H), 7.46-7.53 (m, 2H), 7.41 (t, 1H, *J*=7.40 Hz); **^{13}CNMR:(125 MHz, $CDCl_3$) δ:** 173.58, 158.03, 156.55, 154.69, 151.63, 137.33, 136.08, 128.98, 126.32, 125.17, 124.97, 122.57, 121.68, 120.34, 119.75, 118.52.

HRMS: [MF: $C_{16}H_9O_2NSBr(M+H)$]: 359.95 (Calculated: 359.21).

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HRMS OF AMINE-IODINE COMPLEXES

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Analysis Info

Acquisition Date 6/30/2021 6:11:10 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\DBU-I2_DMSO_GA2_01_2744.d

Method dlc_ms50-1200mz_10min_0.120ml flow_95b.m

Operator CIF

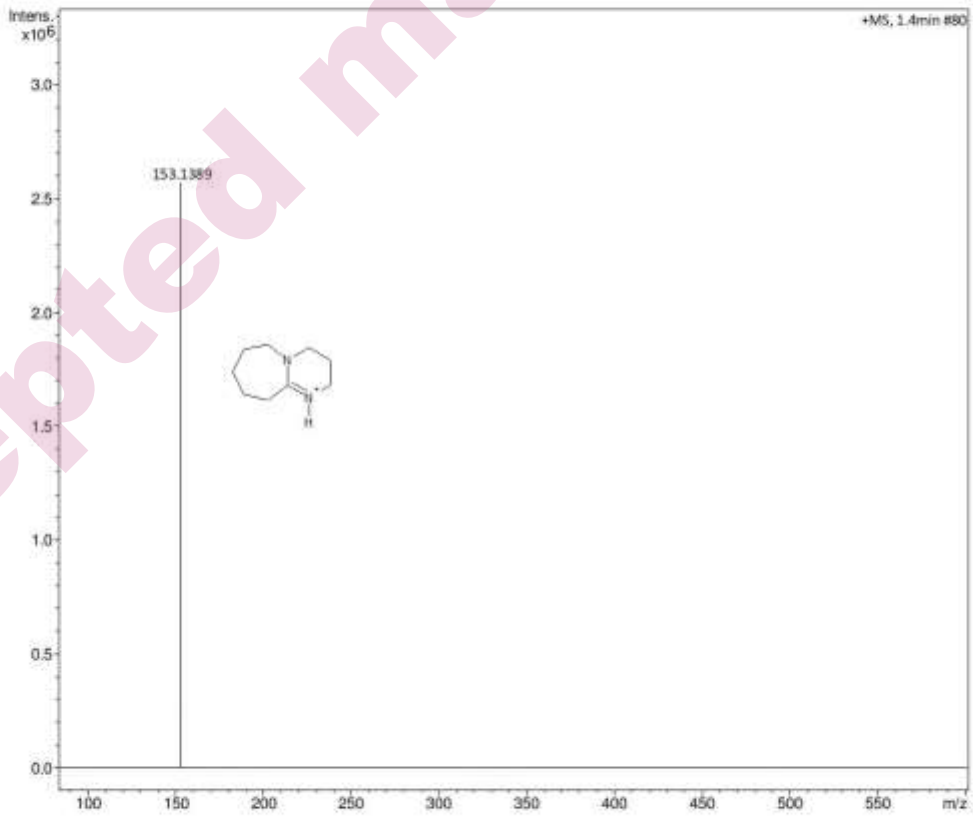
Sample Name DBU-I2_DMSO

Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



DBU-I2_DMSO_GA2_01_2744.d

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Fig: HRMS DBU-iodine Complex

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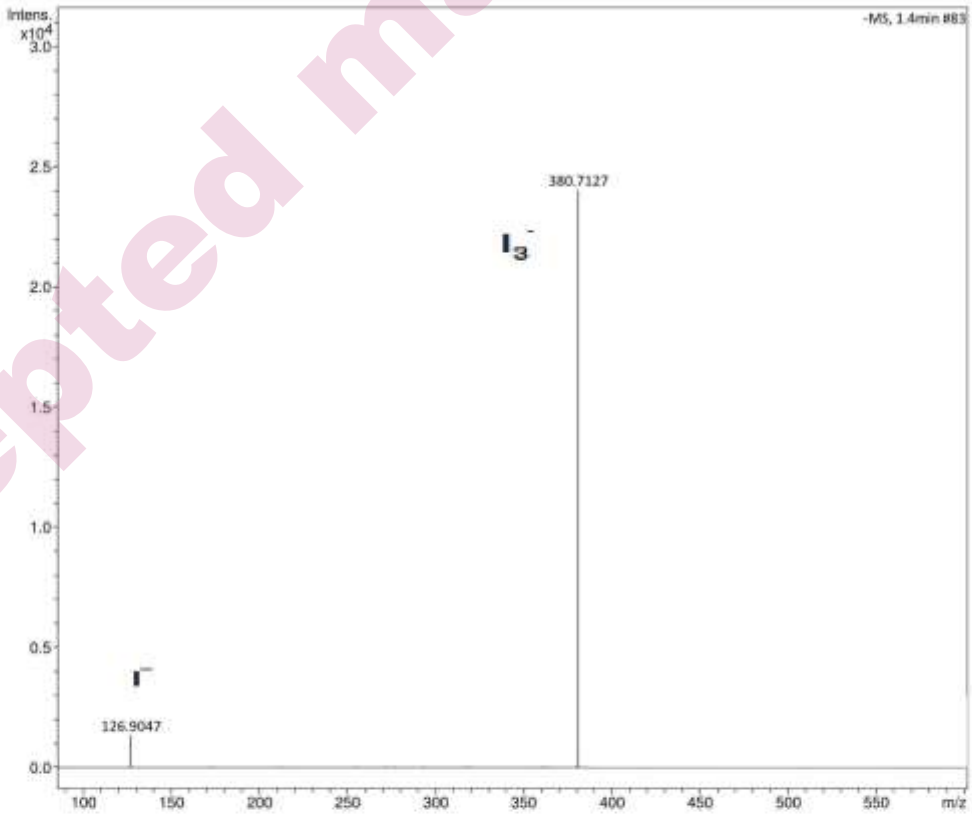
Analysis Info

Analysis Name	D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\DBU-I2_DMSO_neg_GA2_01_2759.d	Acquisition Date	7/1/2021 1:22:23 PM
Method	dlc_ms50-1200mz_12min_0.120ml flow_95b.m	Operator	CIF
Sample Name	DBU-I2_DMSO_neg	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



DBU-I2_DMSO_neg_GA2_01_2759.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 4:59:39 PM by: CIF Page 1 of 1

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Fig: HRMS DBU-iodine complex.

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Analysis Info

Acquisition Date 6/30/2021 6:21:51 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\Morph-I2_DMSO_GA3_01_2745.d

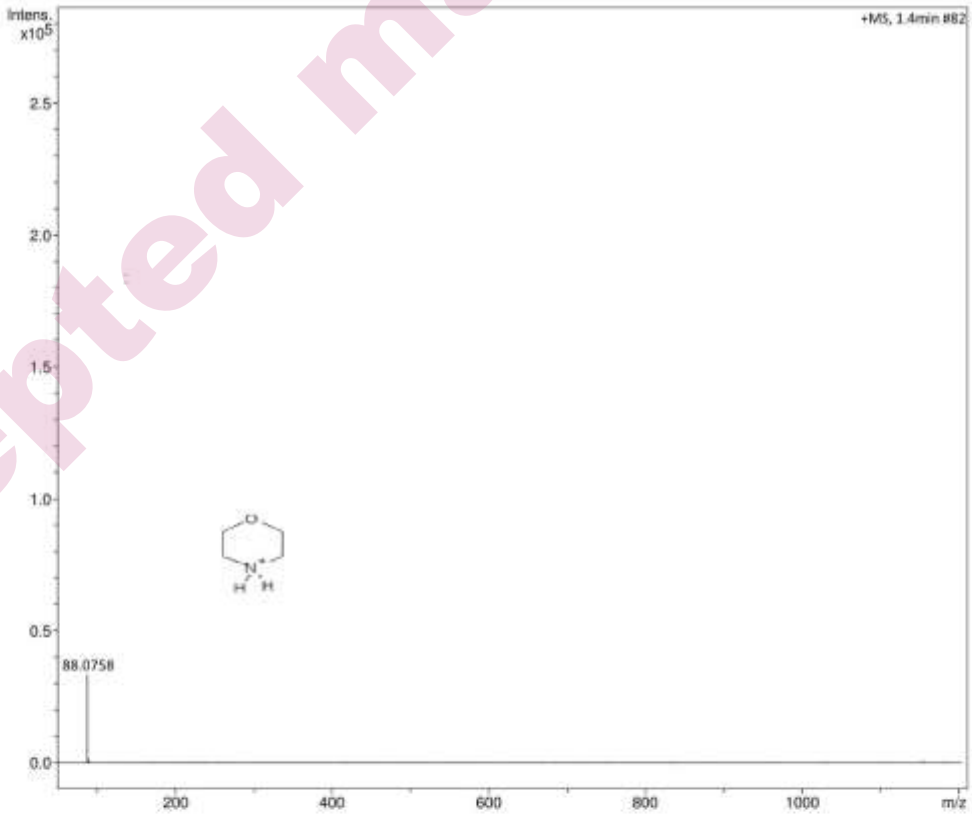
Method dlc_ms50-1200mz_10min_0.120ml flow_95b.m Operator CIF

Sample Name Morph-I2_DMSO Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Morph-I2_DMSO_GA3_01_2745.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 4:46:48 PM by: CIF Page 1 of 1

Fig: HRMS Morpholine-iodine complex

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Analysis Info

Acquisition Date 7/1/2021 1:35:04 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\Morph-I2_DMSO_neg_GA3_01_2760.d

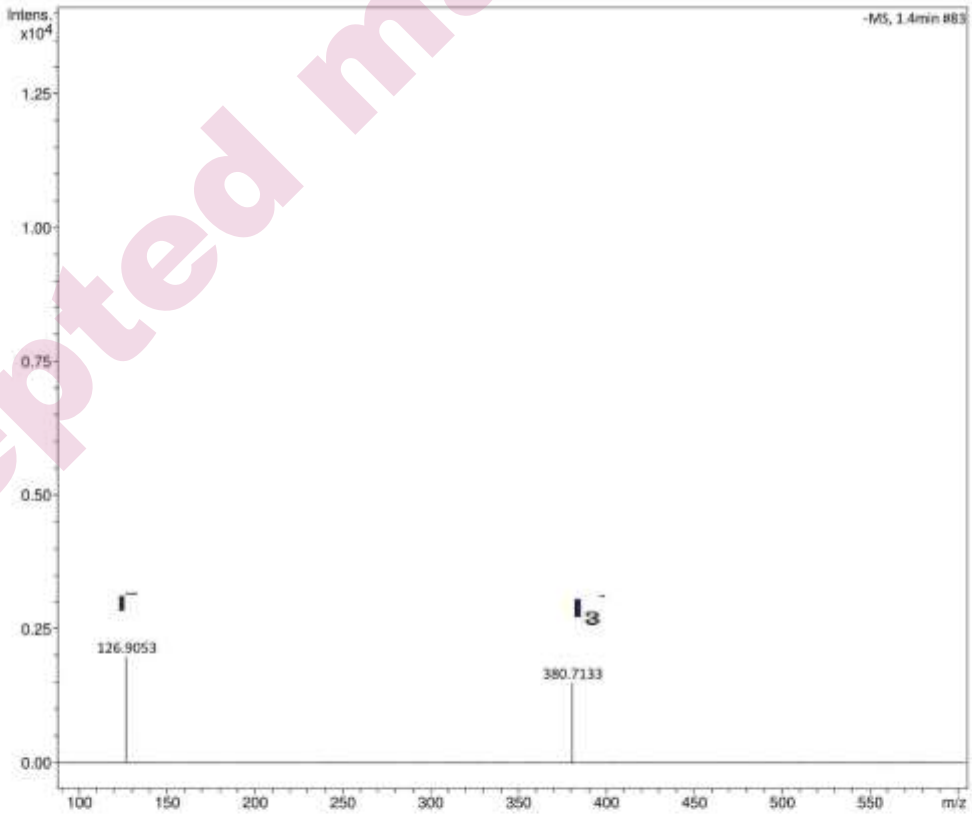
Method dlc_ms50-1200mz_12min_0.120ml flow_95b.m Operator CIF

Sample Name Morph-I2_DMSO_neg Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Morph-I2_DMSO_neg_GA3_01_2760.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 5:02:34 PM by: CIF Page 1 of 1

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Fig: HRMS Morpholine-iodine Complex

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Analysis Info

Acquisition Date 6/30/2021 6:32:30 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\Urotro.I2_GA4_01_2746.d

Method dlc_ms50-1200mz_10min_0.120ml flow_95b.m

Operator CIF

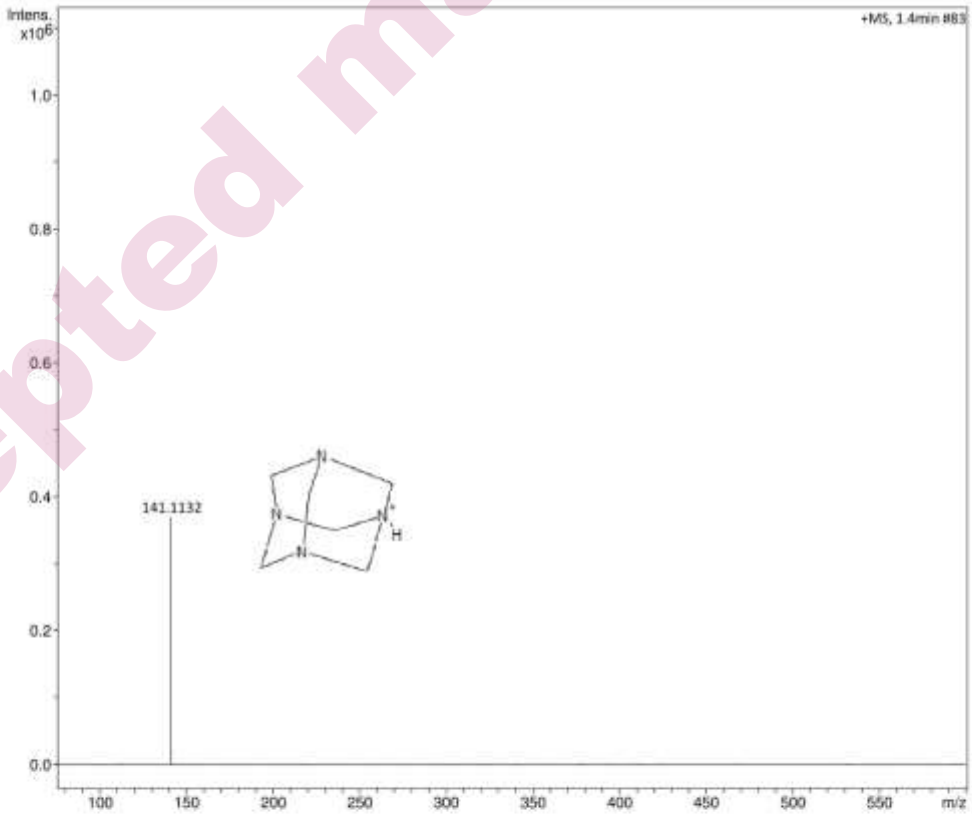
Sample Name Urotro.I2

Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Urotro.I2_GA4_01_2746.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 4:57:12 PM by: CIF Page 1 of 1

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Fig :HRMS Urotropine-iodine complex

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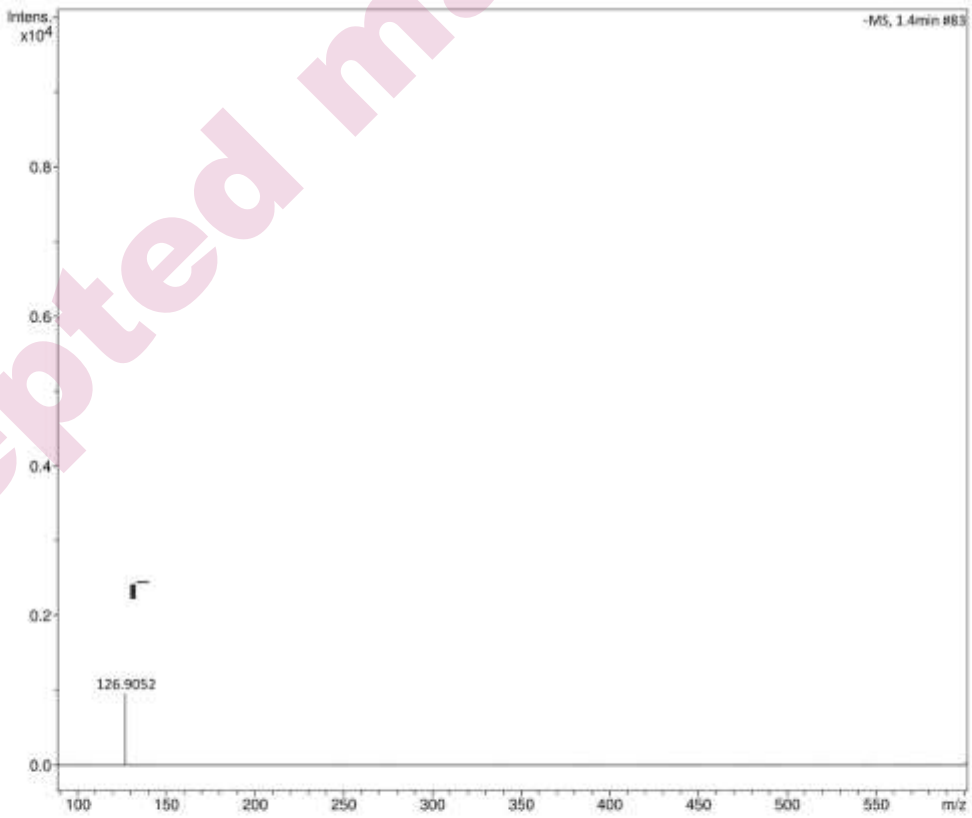
Analysis Info

Analysis Name	D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\Urotro.I2_neg_GA4_01_2761.d	Acquisition Date	7/1/2021 1:47:46 PM
Method	dlc_ms50-1200mz_12min_0.120ml flow_95b.m	Operator	CIF
Sample Name	Urotro.I2_neg	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Urotro.I2_neg_GA4_01_2761.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 5:16:09 PM by: CIF Page 1 of 1

Fig: HRMS Urotropine-iodine Complex

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Analysis Info

Acquisition Date 6/30/2021 6:43:10 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\Piper.I2_GA5_01_2747.d

Method dlc_ms50-1200mz_10min_0.120ml flow_95b.m

Operator CIF

Sample Name Piper.I2

Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C

Piper.I2_GA5_01_2747.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 4:55:04 PM by: CIF Page 1 of 1

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Fig: HRMS Piperazine-iodine Complex

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Analysis Info

Acquisition Date 7/1/2021 2:00:27 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\Piper.l2_neg_GA5_01_2762.d

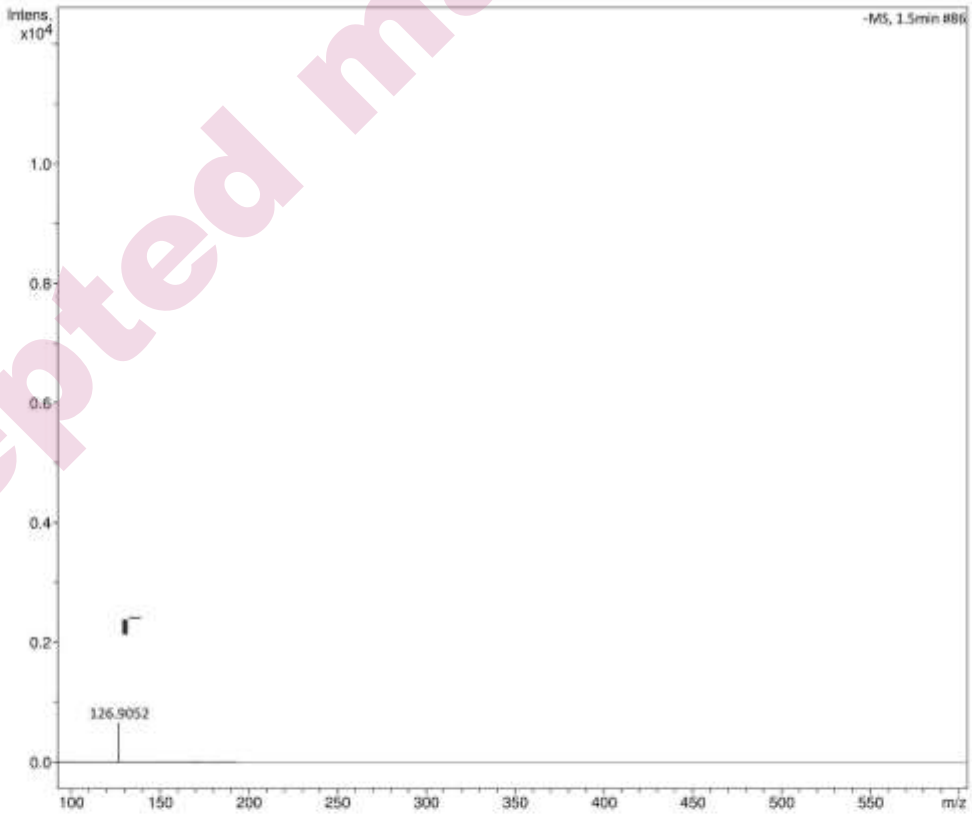
Method dlc_ms50-1200mz_12min_0.120ml flow_95b.m Operator CIF

Sample Name Piper.l2_neg Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Piper.l2_neg_GA5_01_2762.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 5:14:02 PM by: CIF Page 1 of 1

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Fig: HRMS Piperazine-iodine Complex

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Analysis Info

Acquisition Date 6/30/2021 6:00:30 PM

Analysis Name D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\N-Me_Pip.I2_GA1_01_2743.d

Method dlc_ms50-1200mz_10min_0.120ml flow_95b.m Operator CIF

Sample Name N-Me_Pip.I2 Instrument impact HD 1819696.00184

Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
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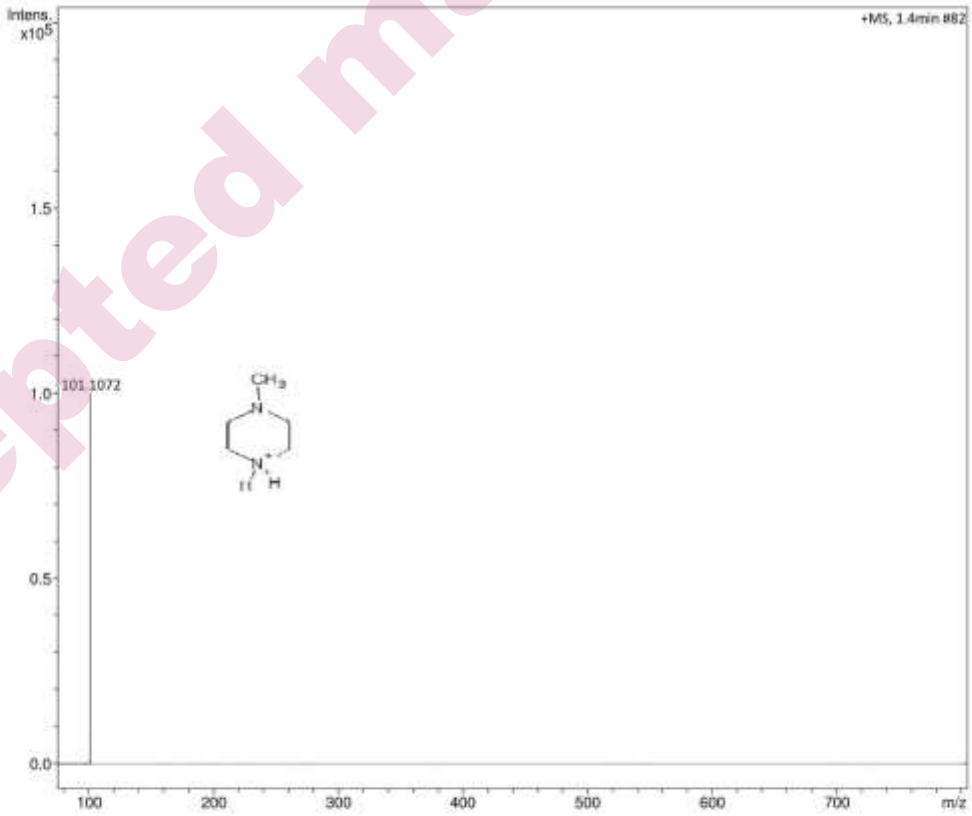

N-Me_Pip.I2_GA1_01_2743.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 4:50:37 PM by: CIF Page 1 of 1

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Fig: HRMS N-methyl Piperazine-iodine complex

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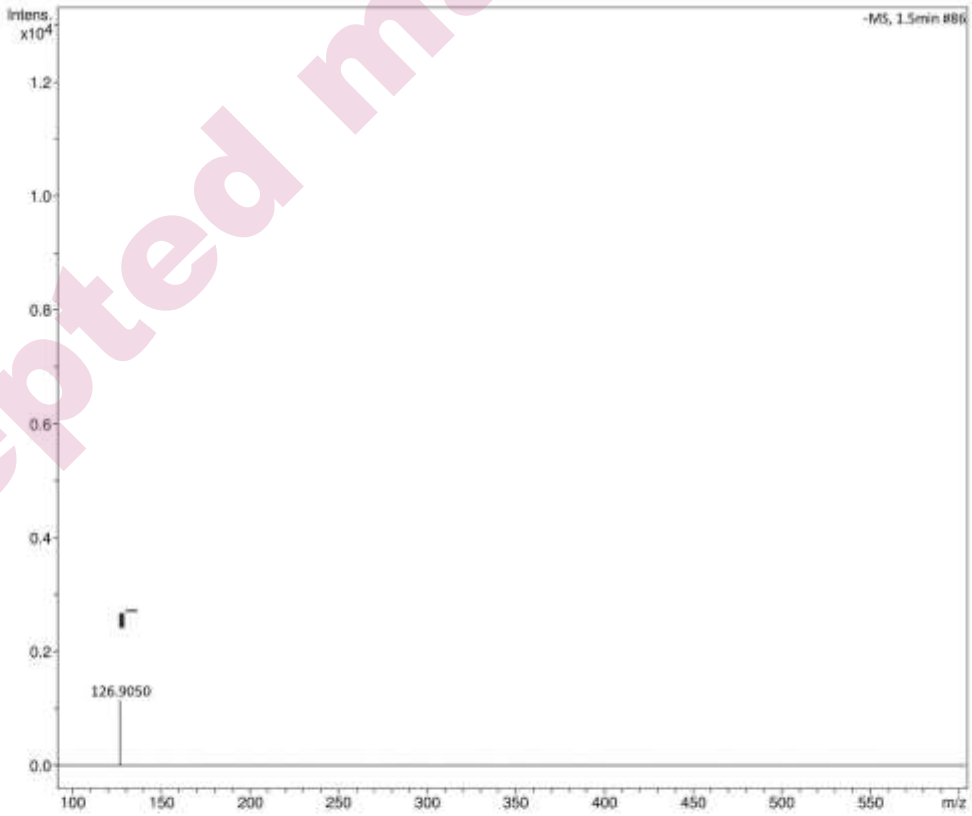
Analysis Info

Analysis Name	D:\Data\2021\JUNE 2021\SPPU COLLEGE\B. G. GHOLAP COLLEGE, SANGVI\DR. PRAMOD KULKARNI\RAMESH GAWADE\N-Me_Pip.l2_neg_GA1_01_2758.d	Acquisition Date	7/1/2021 1:09:43 PM
Method	dlc_ms50-1200mz_12min_0.120ml flow_95b.m	Operator	CIF
Sample Name	N-Me_Pip.l2_neg	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



N-Me_Pip.l2_neg_GA1_01_2758.d

Bruker Compass DataAnalysis 4.2 printed: 7/3/2021 5:11:20 PM by: CIF Page 1 of 1

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Fig: HRMS N-methyl Piperazine-iodine complex

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Graph: 1 (1a) DBU-iodine complex UV-visible spectrum.

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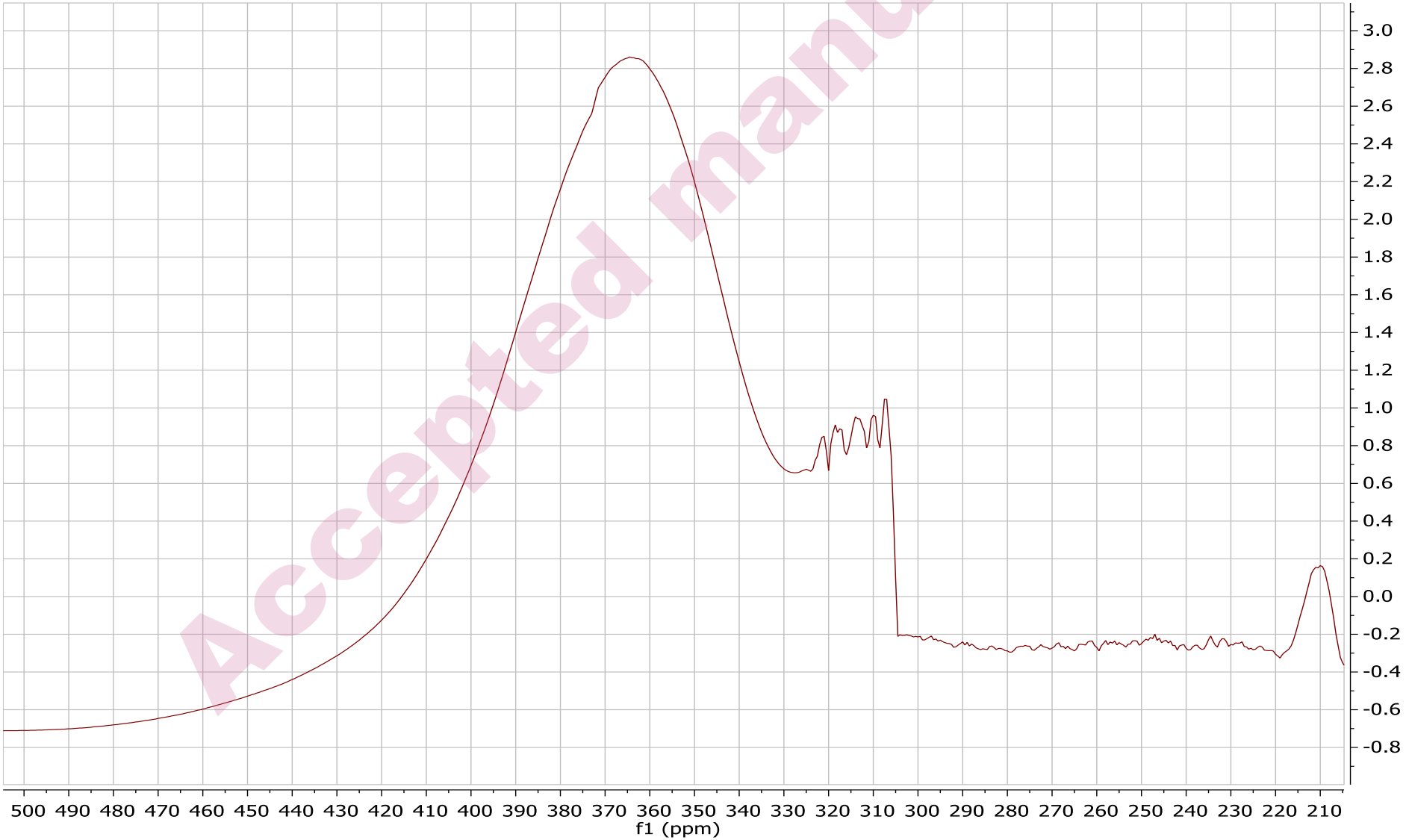

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Graph:2 (1b) Morpholine-iodine complex UV-visible spectrum.

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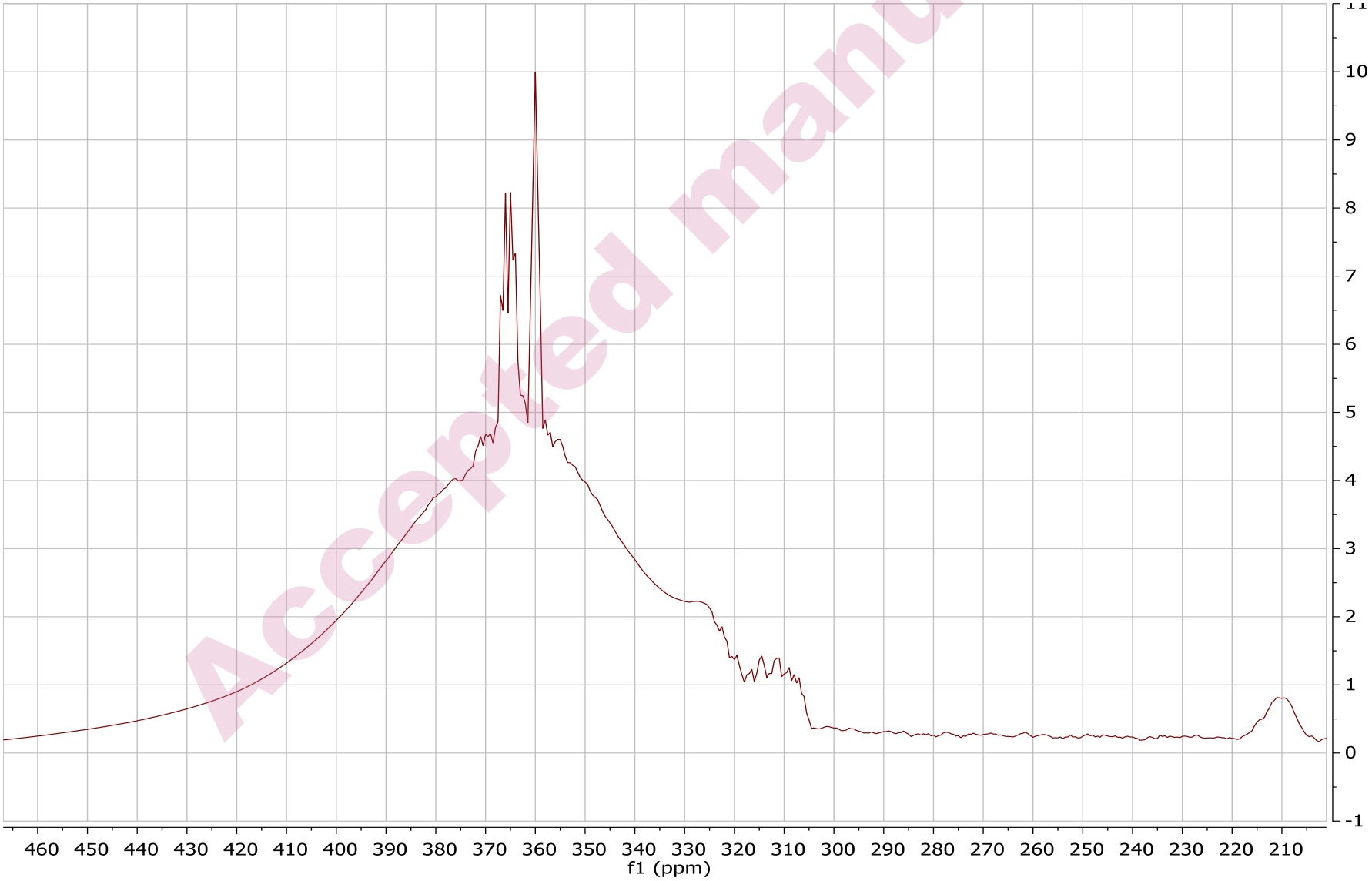
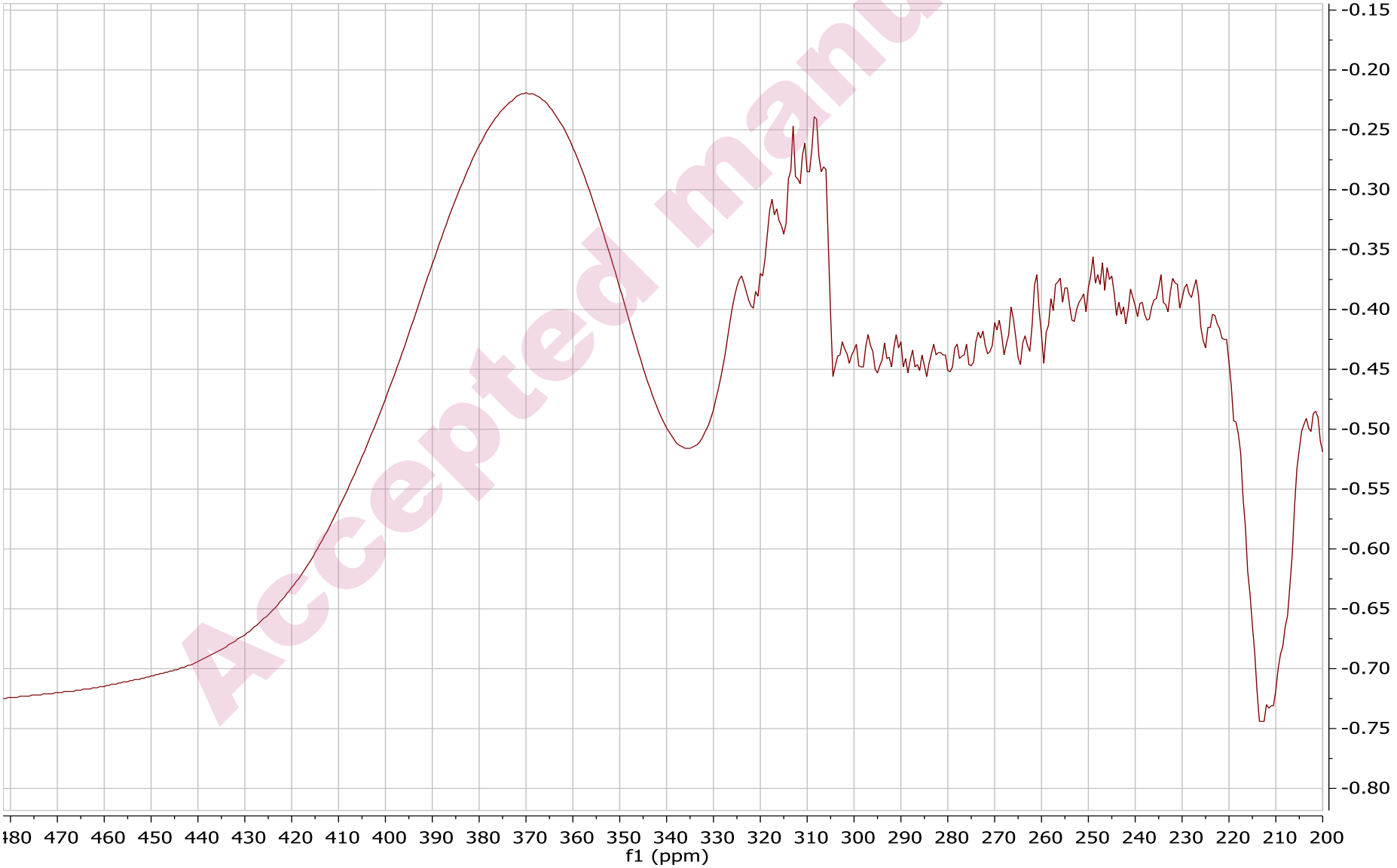


Accepted manuscript

Graph: 3. (1c) Urotropine-iodine complex UV-visible spectrum.

Accepted manuscript



Accepted manuscript

Graph: 4 (1d) Piperazine-iodine complex UV-visible spectrum.

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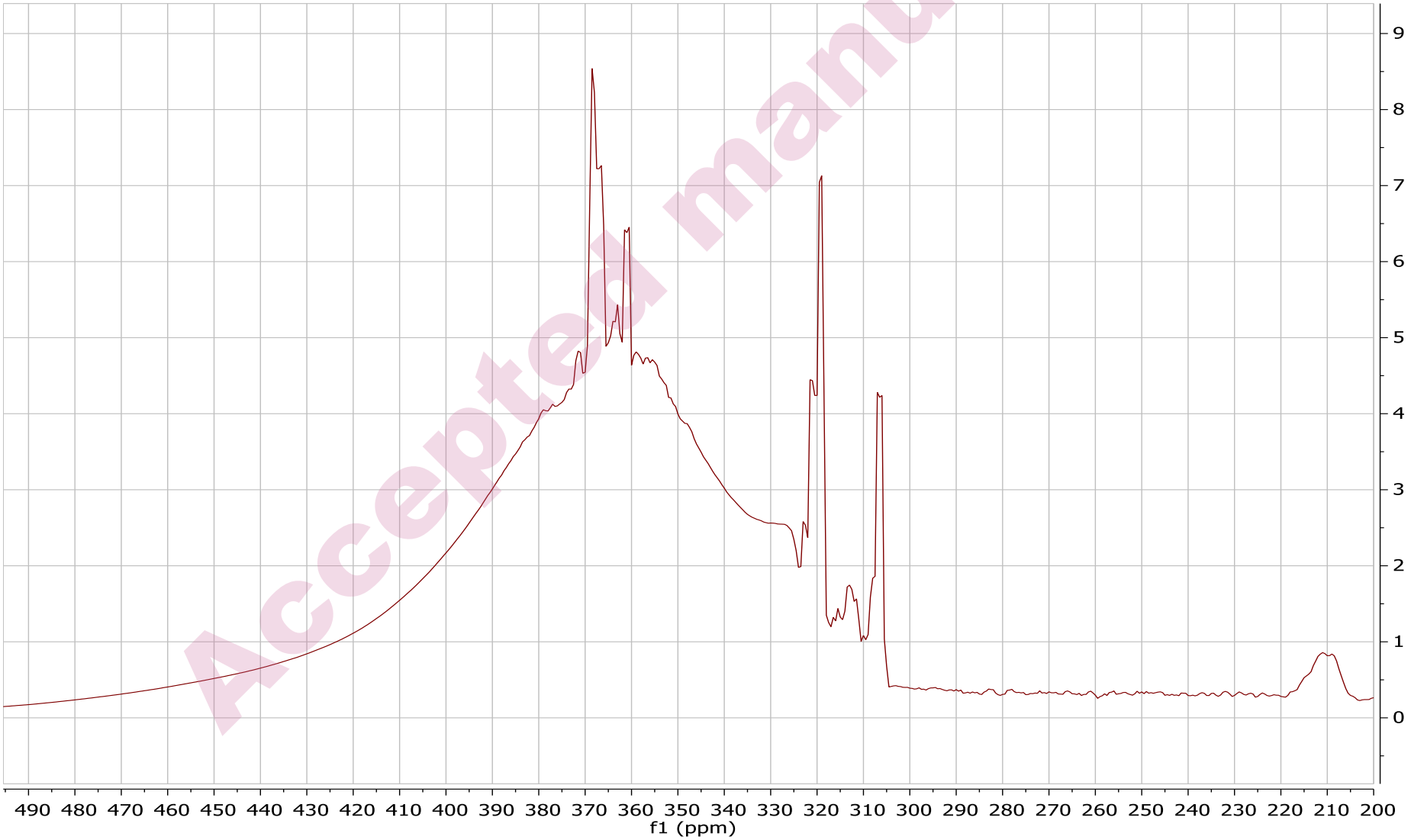

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Graph: 5 (1e) N-methyl-Piperazine-iodine complex UV-visible spectrum.

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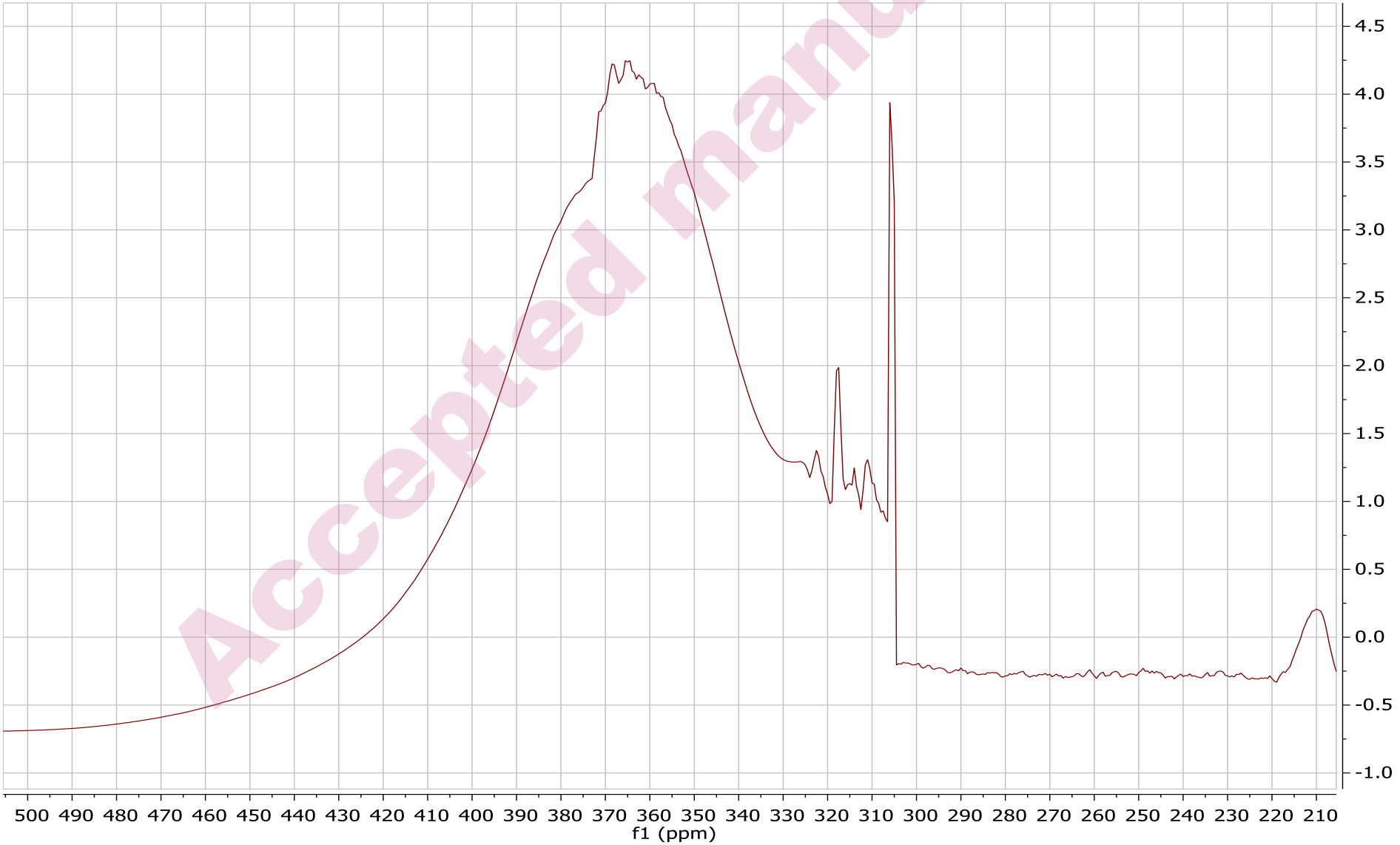

Accepted manuscript

Graph: 1 (1a) DBU-iodine complex IR spectrum.

Accepted manuscript

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Graph:2 (1b) Morpholine-iodine complex IR spectrum.

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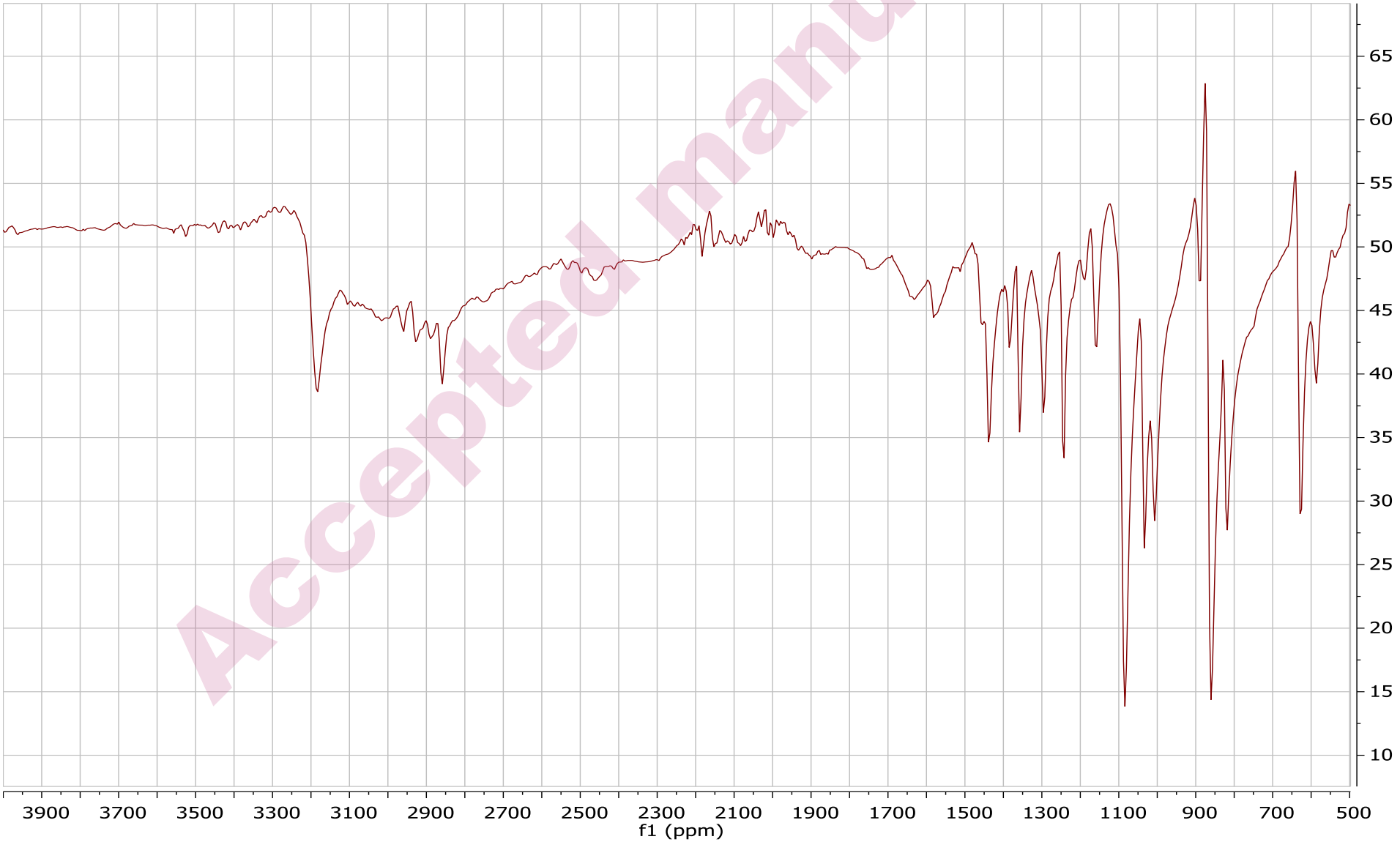
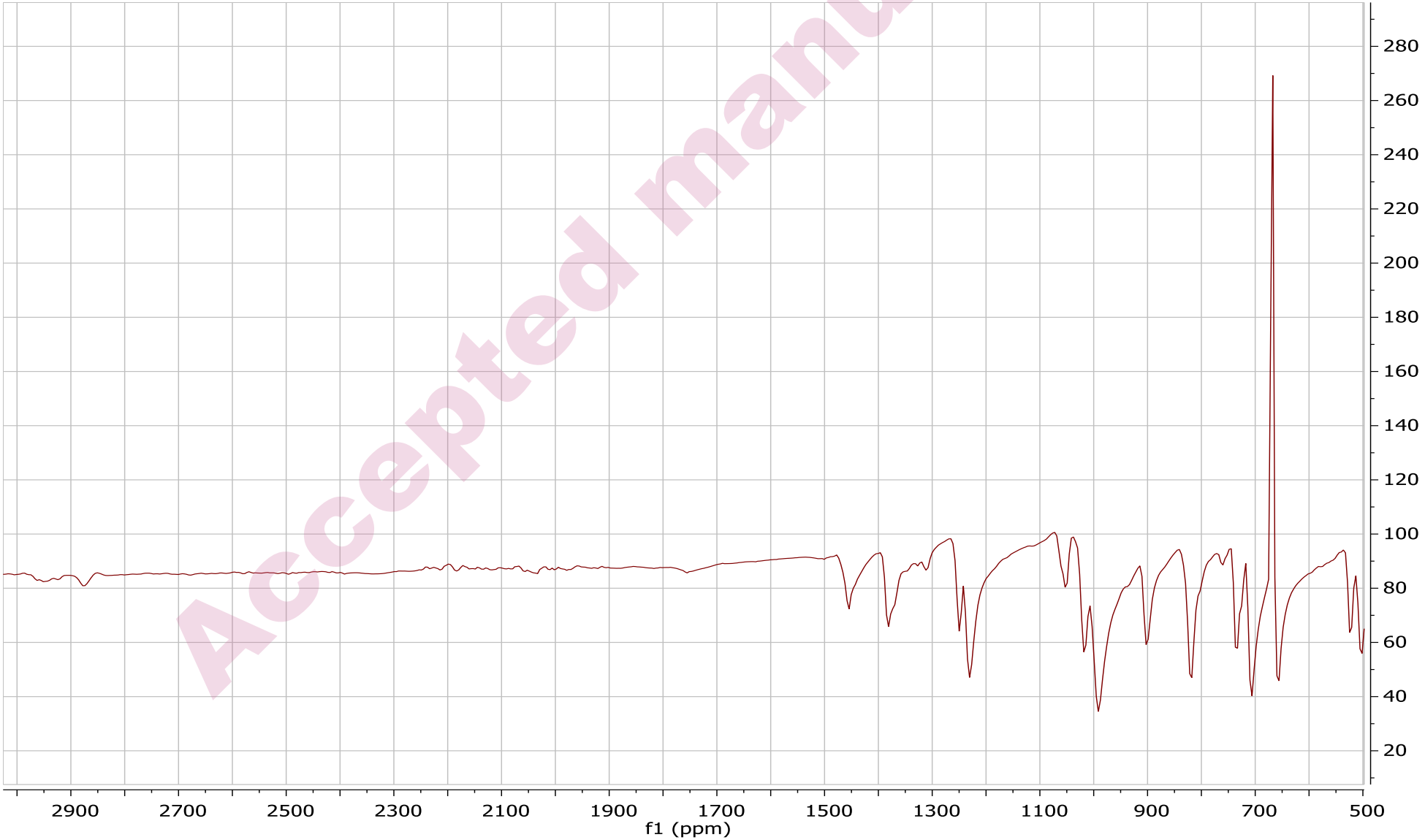


Accepted manuscript

Accepted manuscript

Graph: 3. (1c) Urotropine-iodine complex IR spectrum.

Accepted manuscript

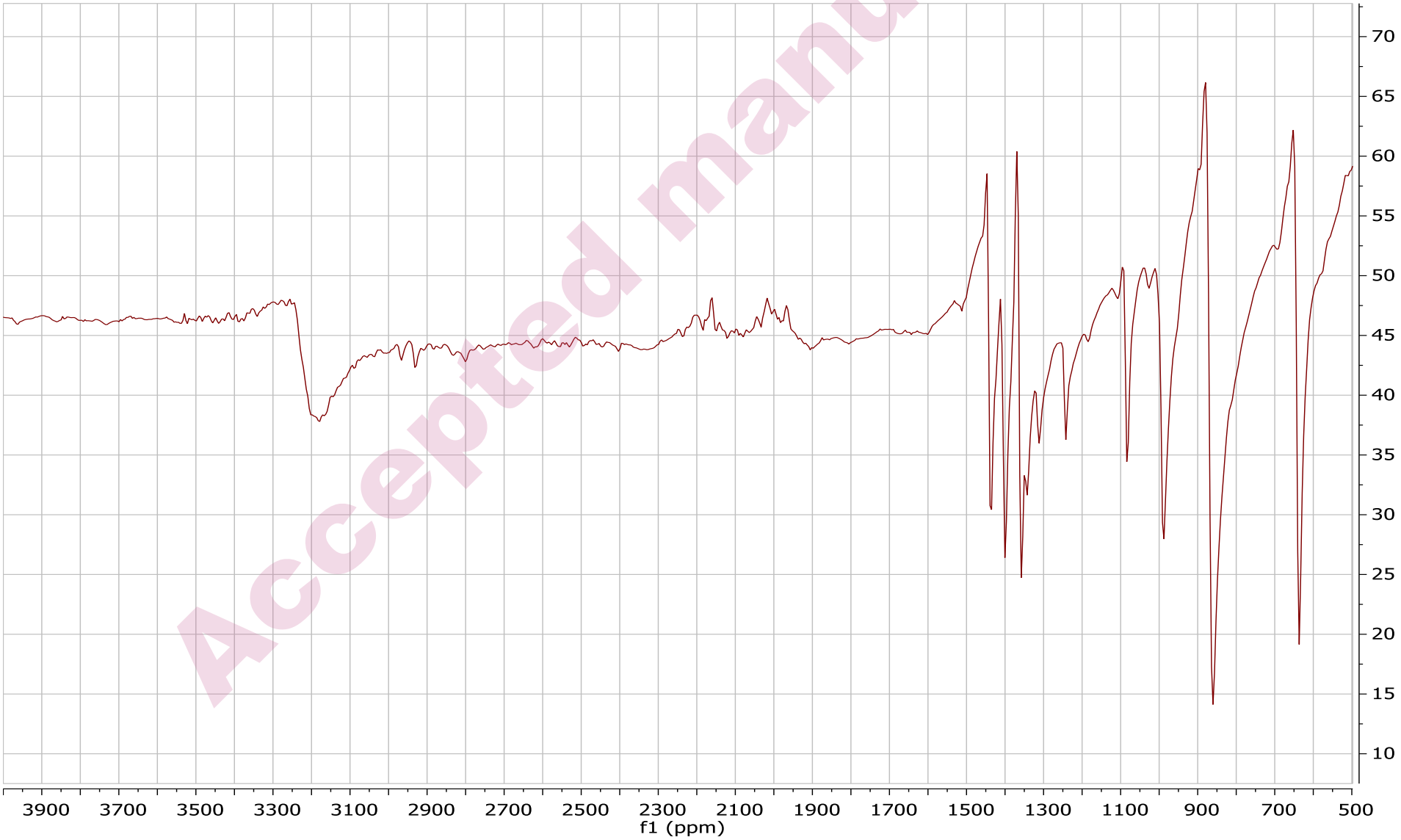


Accepted manuscript

Accepted manuscript

Graph: 4 (1d) Piperazine-iodine complex IR spectrum.

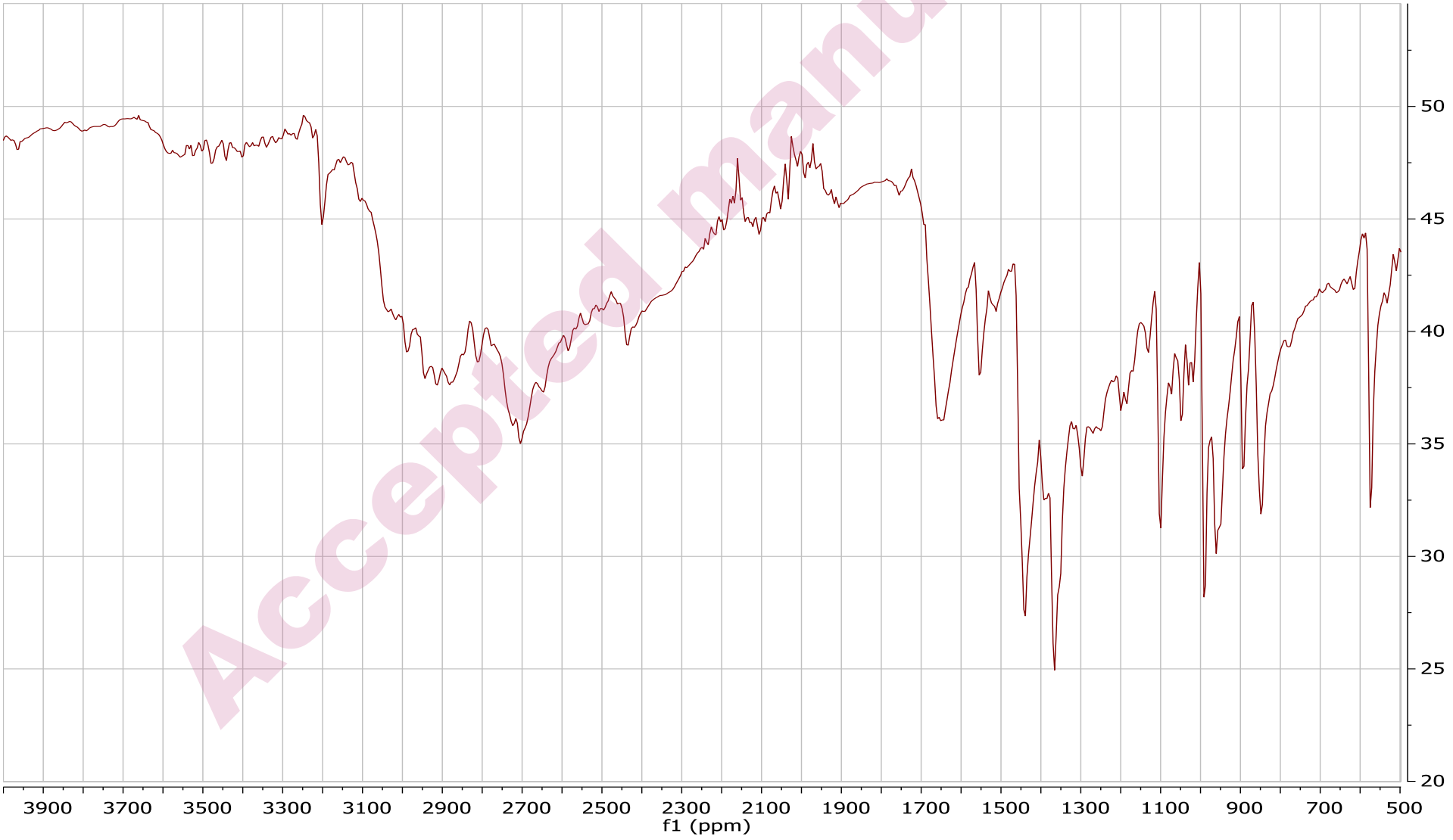
Accepted manuscript



Accepted manuscript

Graph: 5 (1e) N-methyl-Piperazine-iodine complex IR spectrum.

Accepted manuscript



Accepted manuscript

Accepted manuscript

Figure: 1 (1a) SEM of DBU-iodine complex indicate Morphology.

Figure: 2 (1b) SEM of Morpholine-iodine complex indicate Morphology.

Accepted manuscript

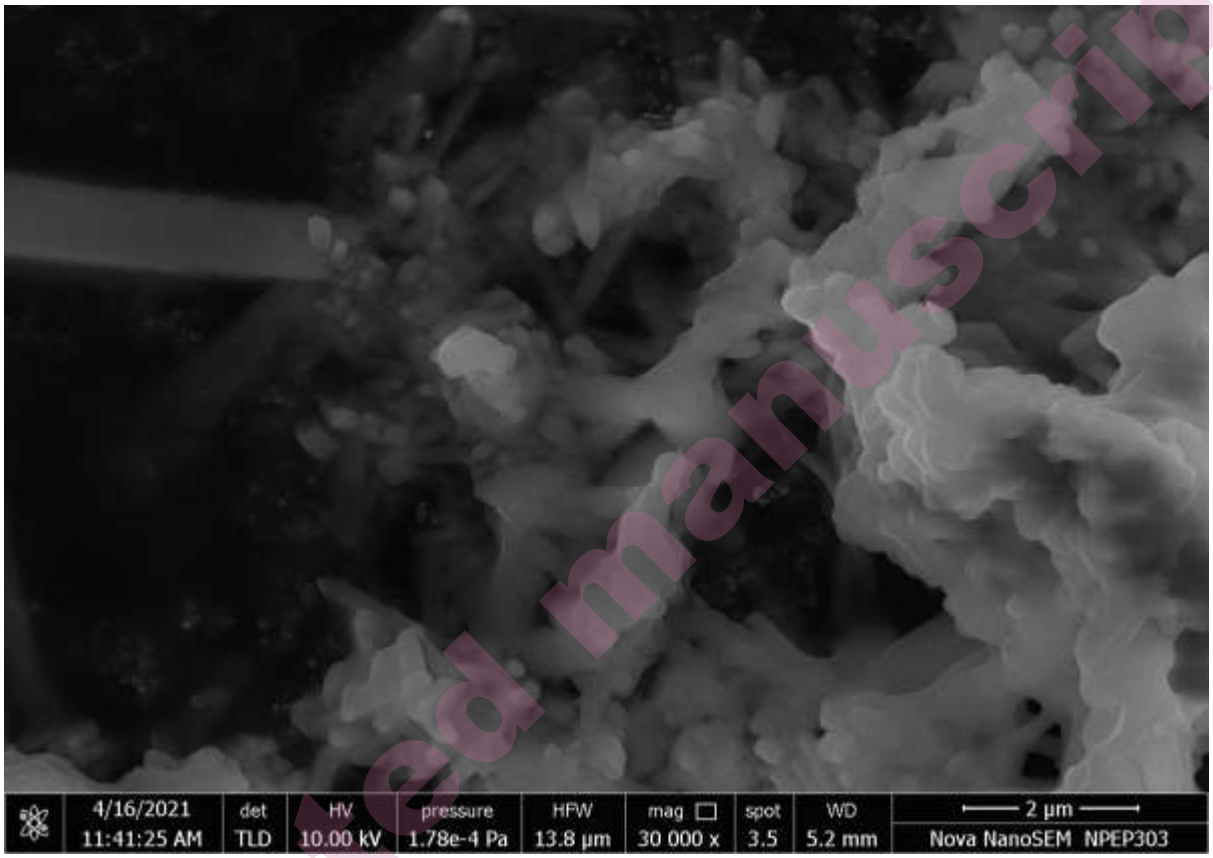


Figure: 3 (1c) SEM of Urotropine-iodine complex indicate Morphology.

Accepted manuscript

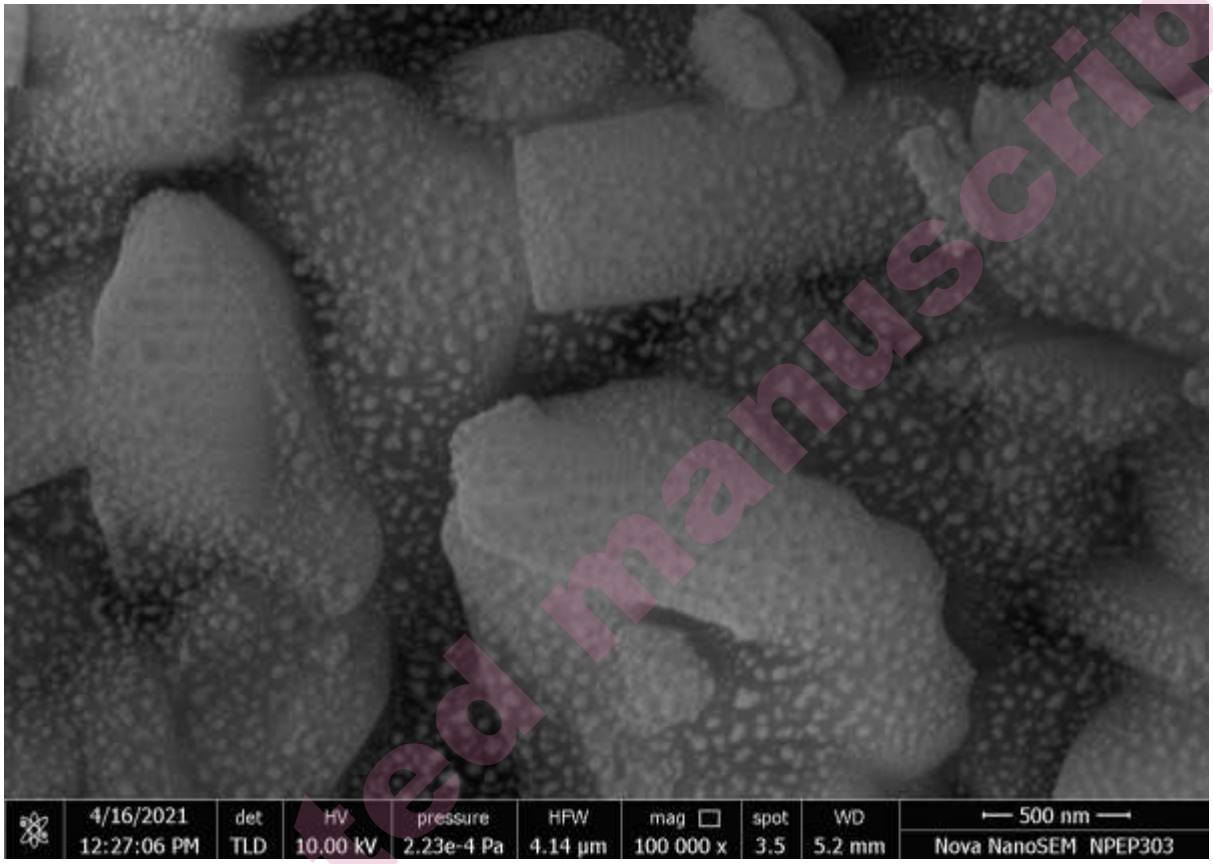


Figure: 4 (1d) SEM of Piperazine-iodine complex indicate Morphology.

Accepted manuscript

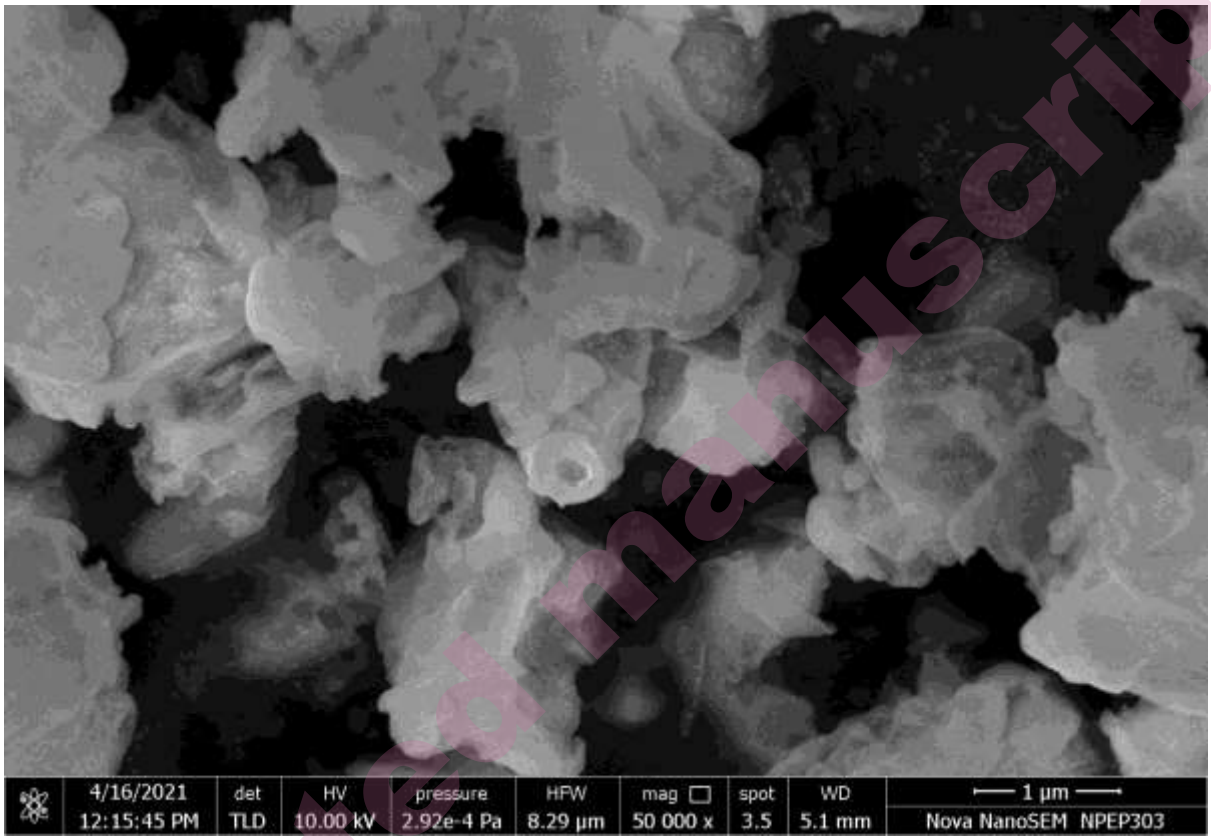
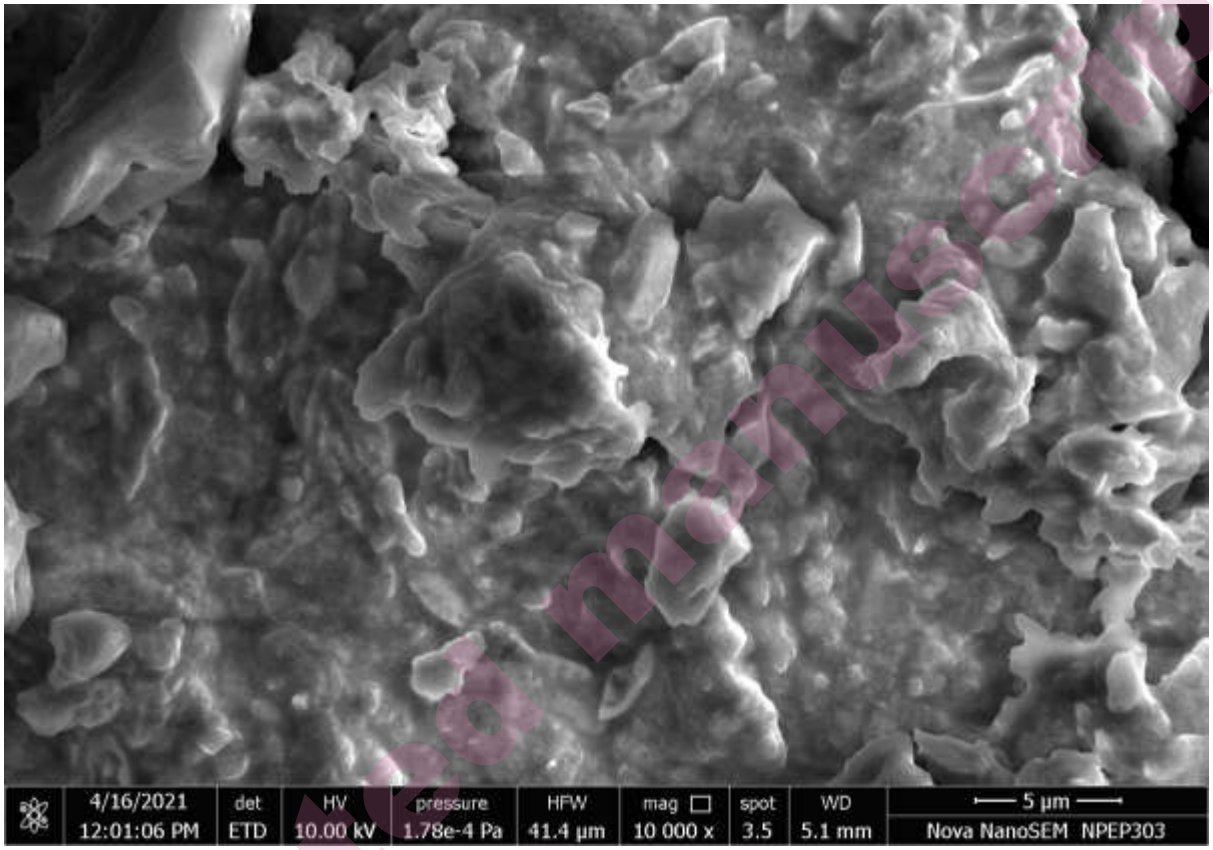


Figure: 5 (1e) SEM of N-Methyl-piperazine-iodine complex indicate Morphology.

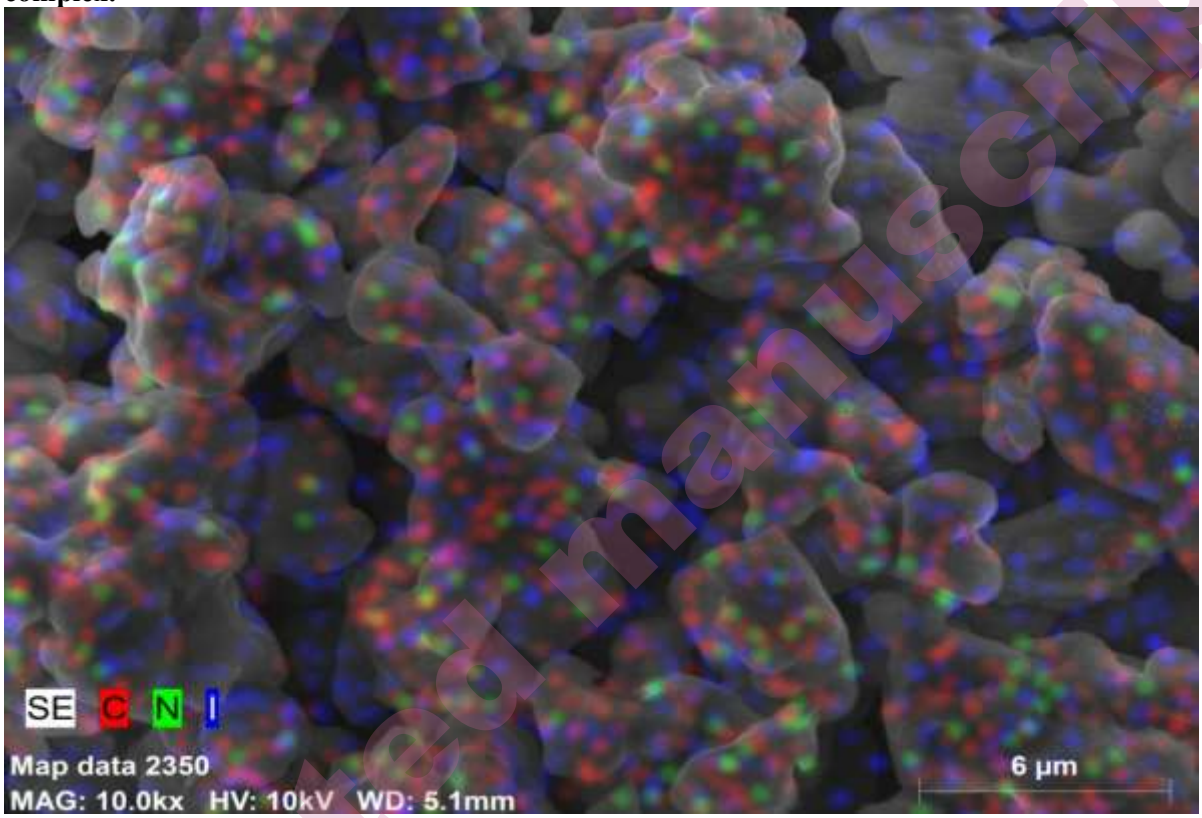
Accepted manuscript



Accepted manuscript

Field Emission Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (*FESEM* - EDS): Figure: 1 (1a) DBU-iodine

complex.



Accepted manuscript

Graph: 1 (1a) DBU-iodine complex.

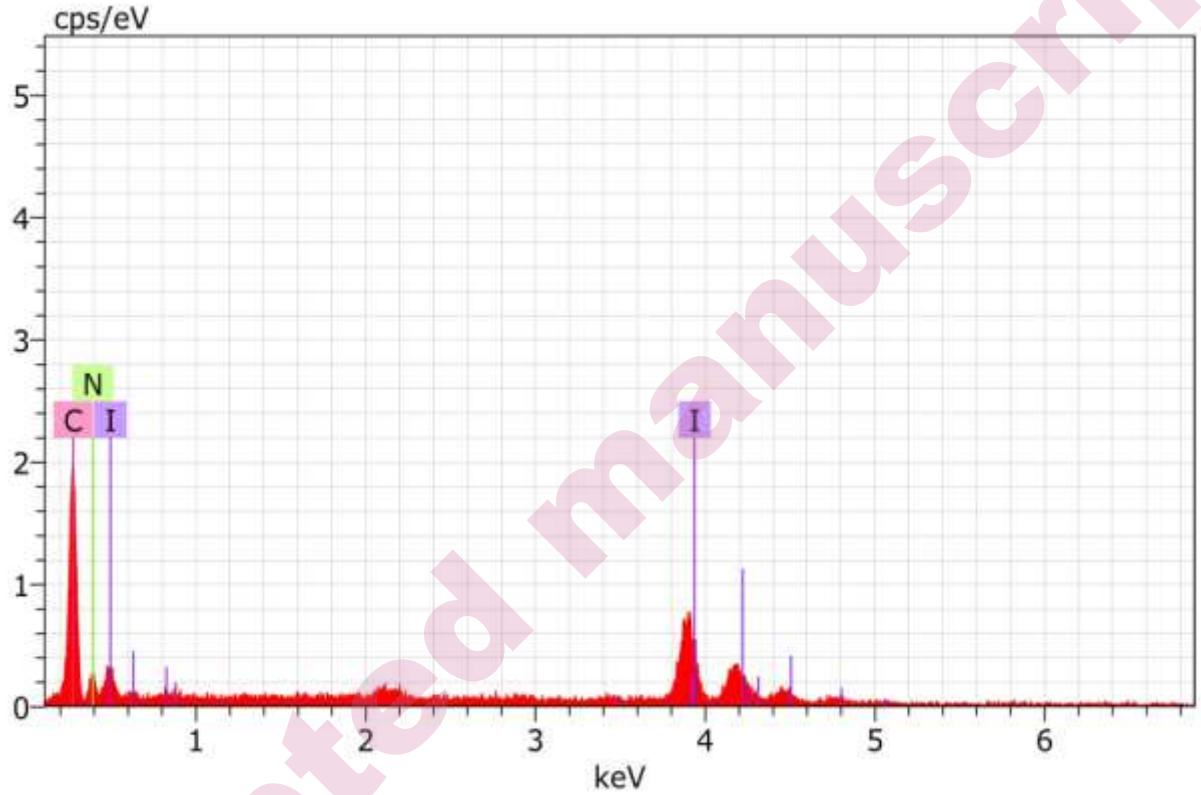
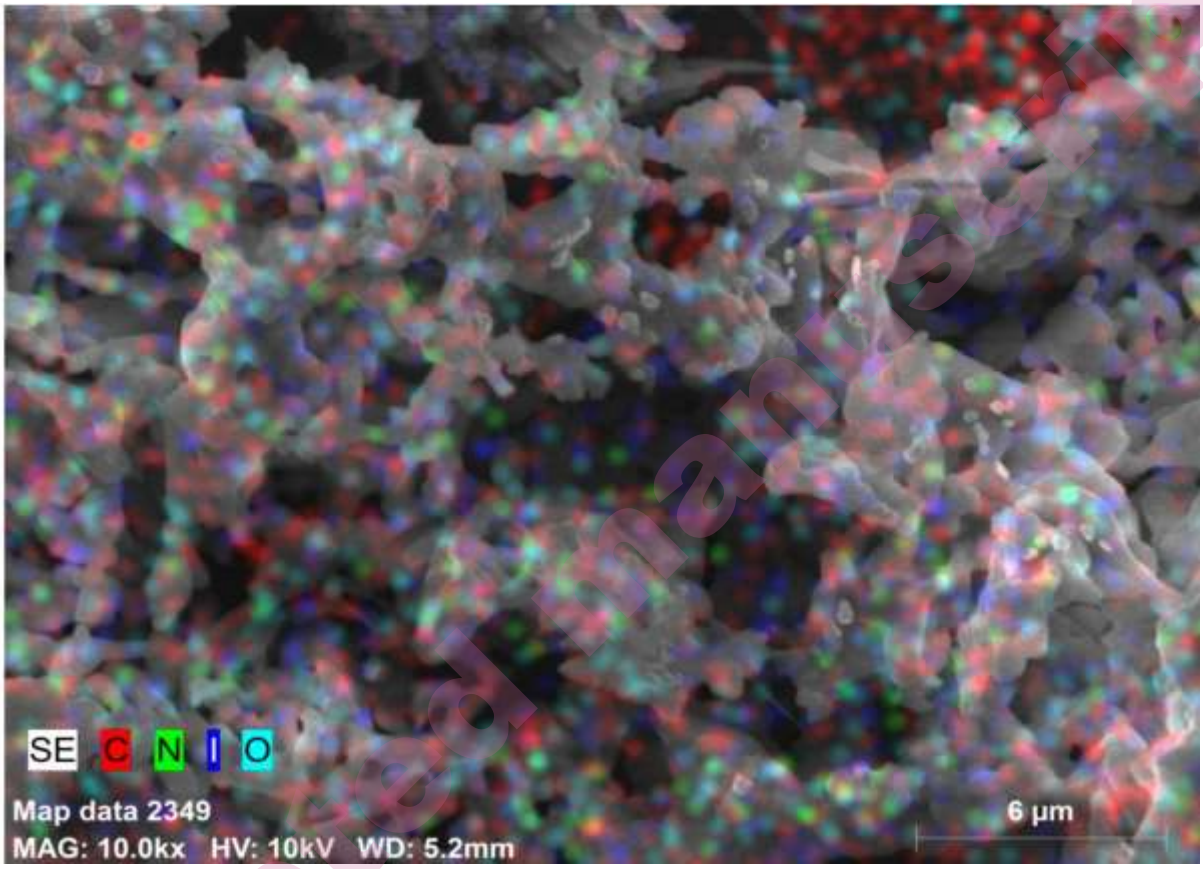


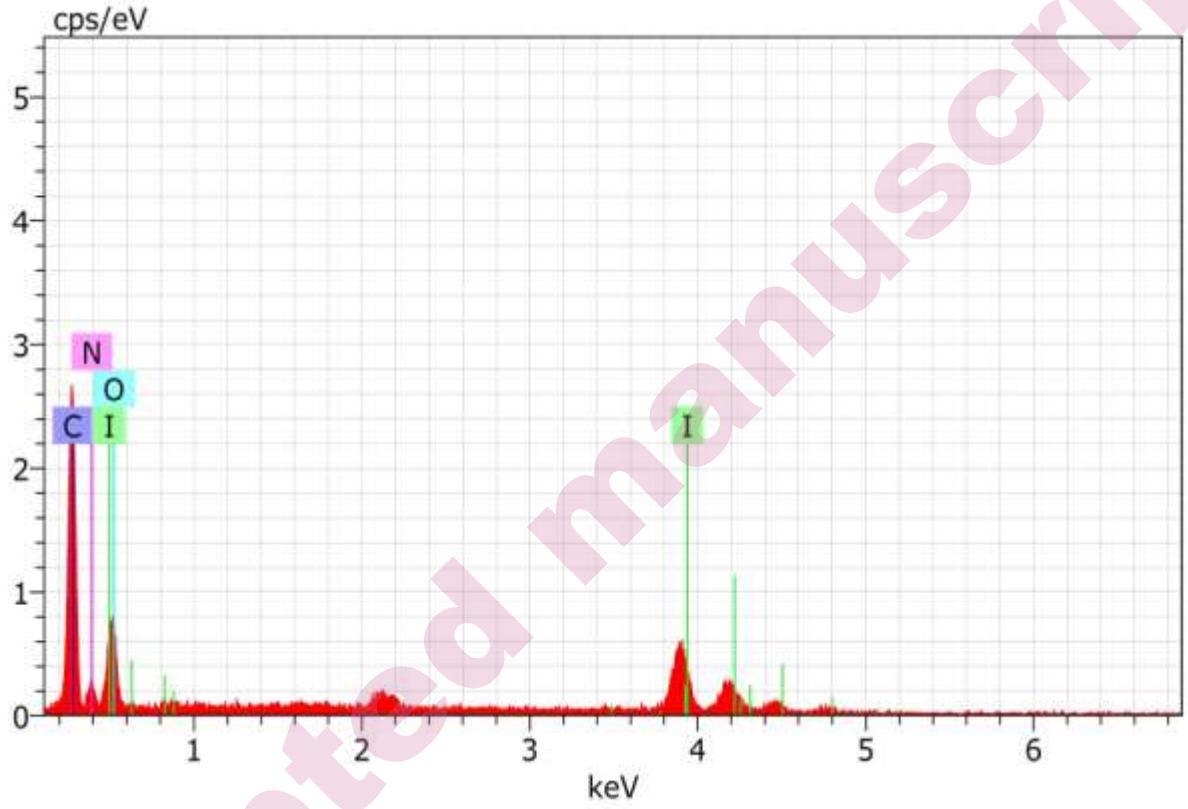
Figure: 2 (1b) Morpholine-iodine complex.

Accepted manuscript



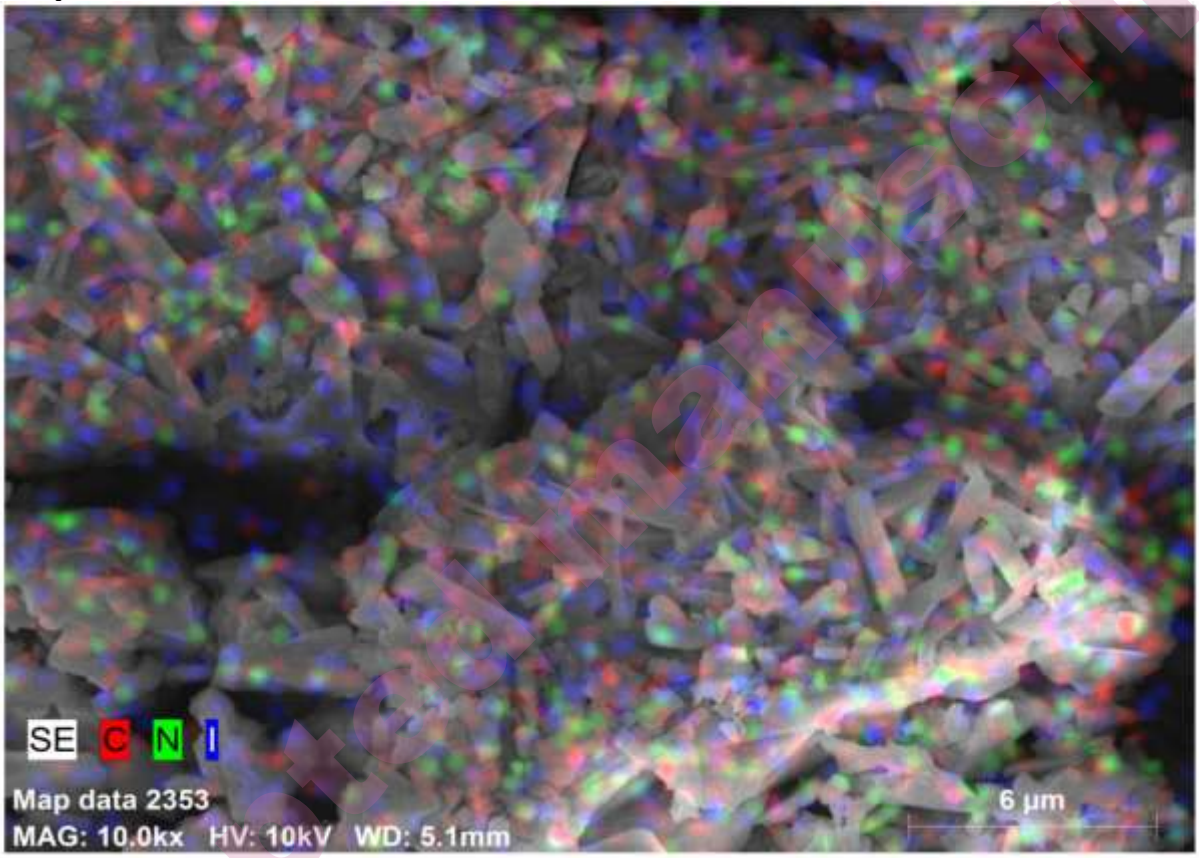
Accepted manuscript

Graph: 2 (1b) morpholine-iodine complexe.



Accepted manuscript

Figure: 3 (1c) Urotropine Iodine Complex.



Graph: 3 (1c) Urotropine Iodine Complex.

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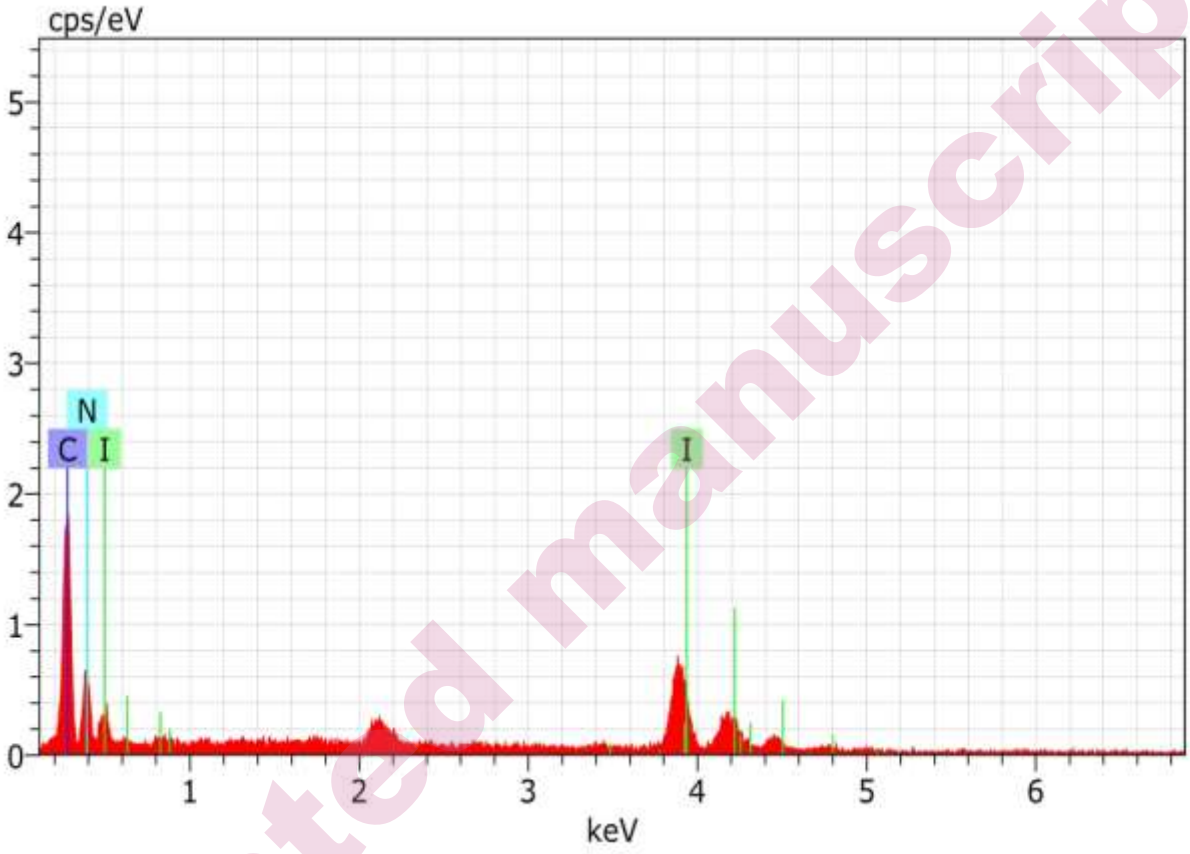
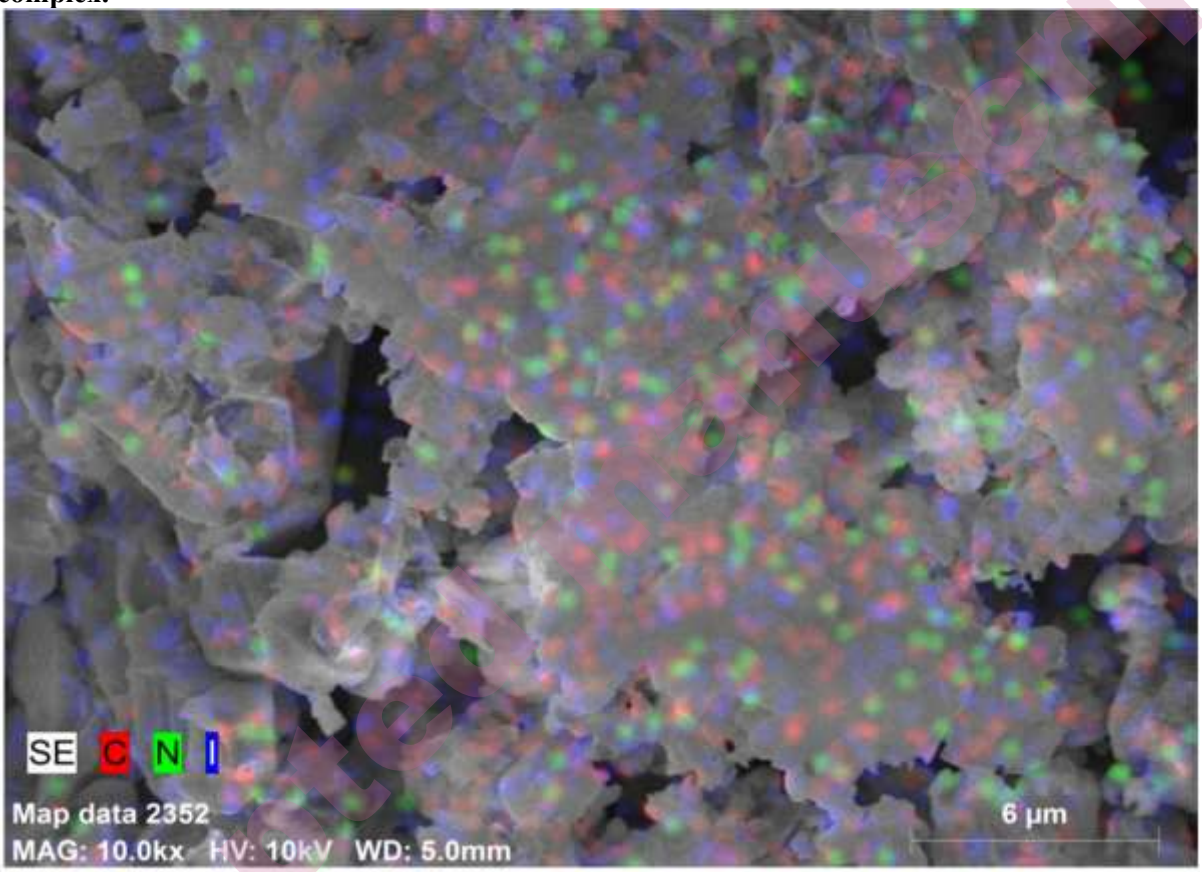


Accepted manuscript

Figure: 4 (1d) Piperazine-iodine complex.



Accepted Manuscript

Graph: 4 (1d) Piperazine-iodine complex.

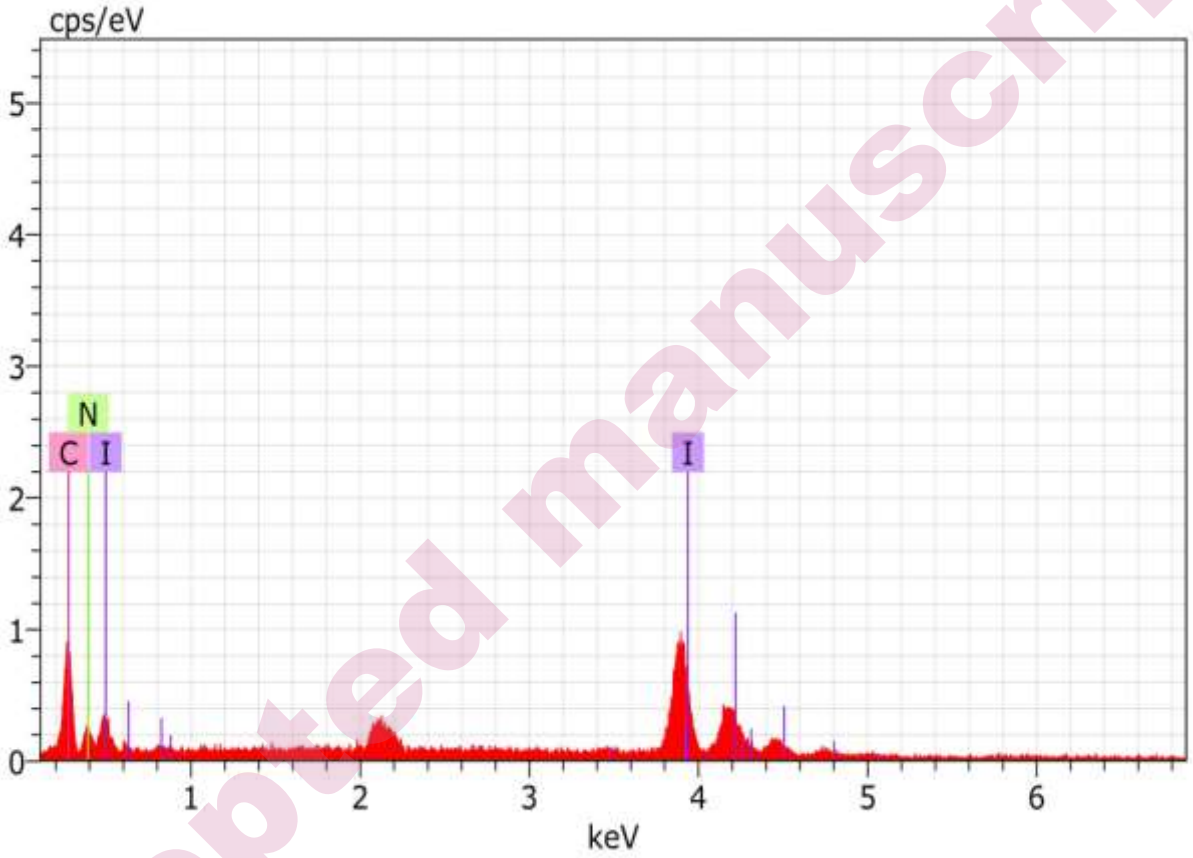
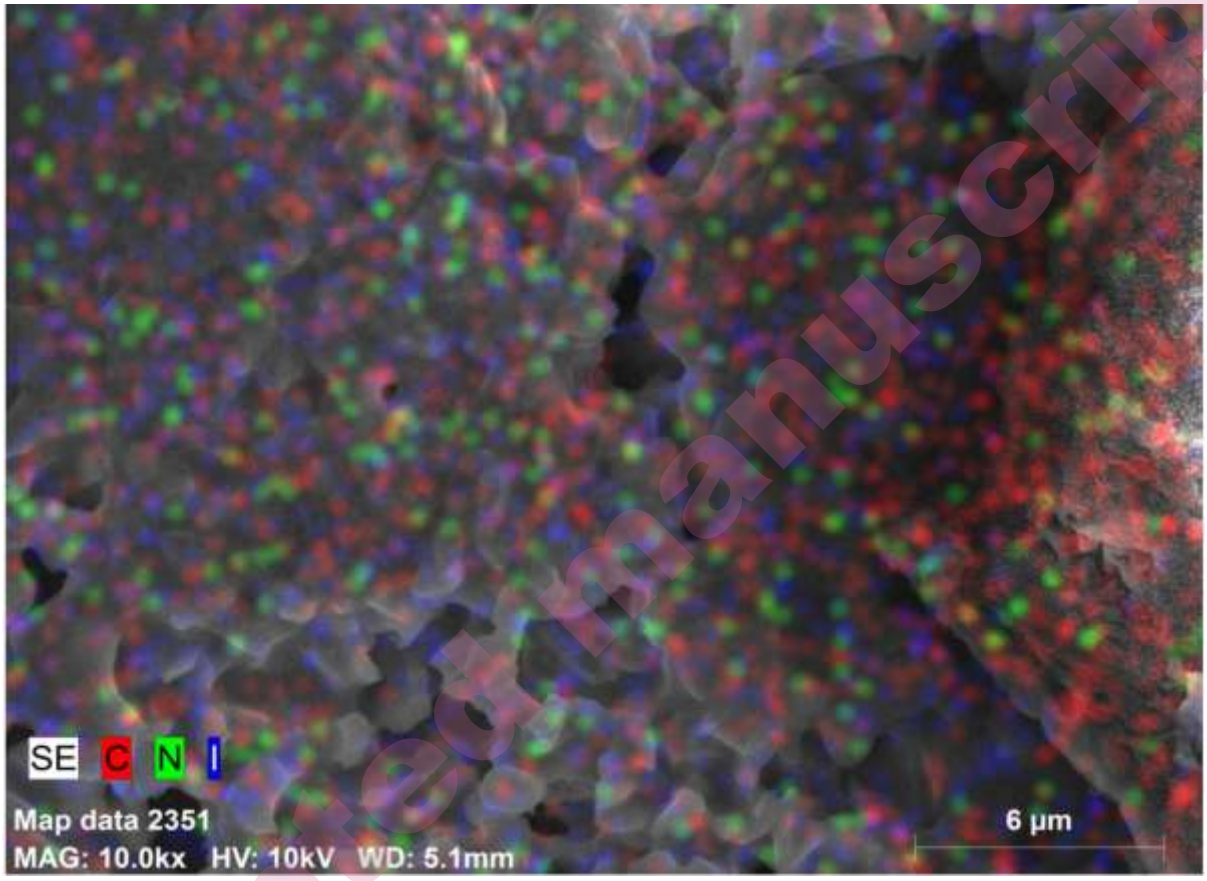


Figure: 5 (1e) N-methyl-piperazine-iodine complex.

Accepted manuscript



Graph: 5 (1e) N-methyl-piperazine-iodine complexe.

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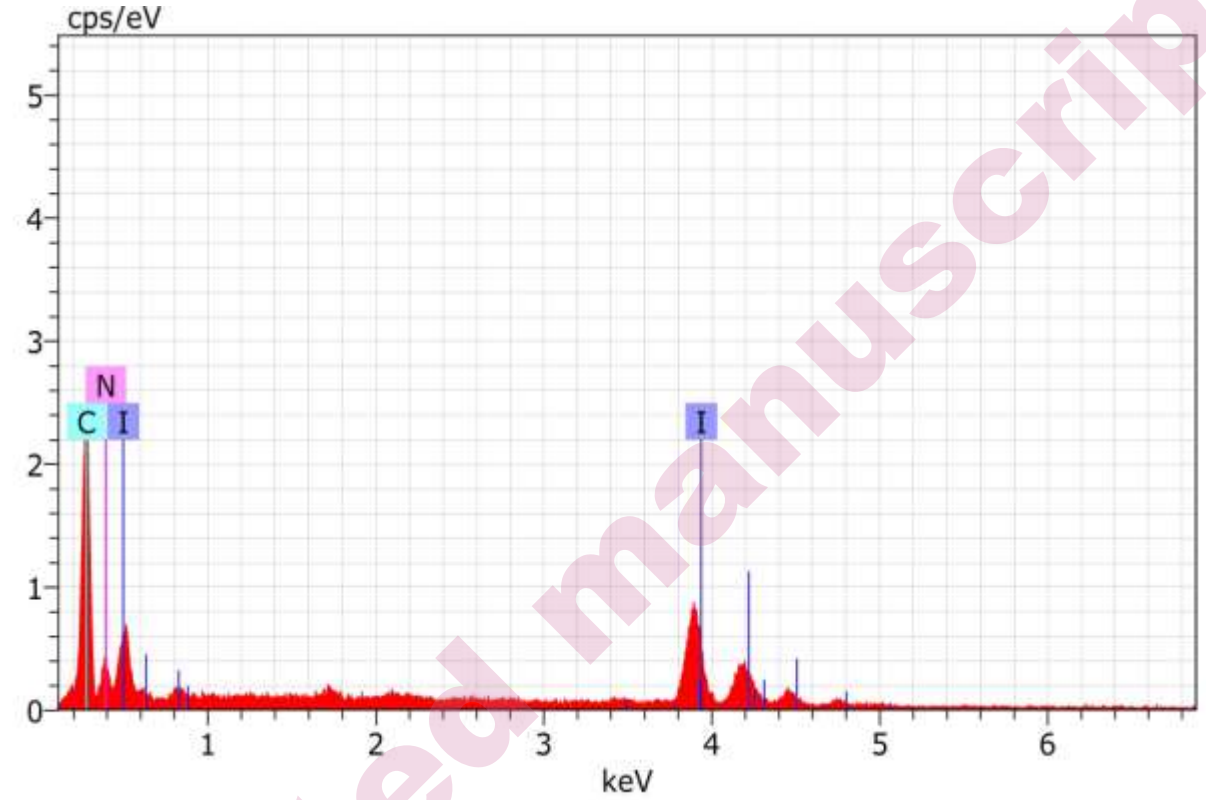
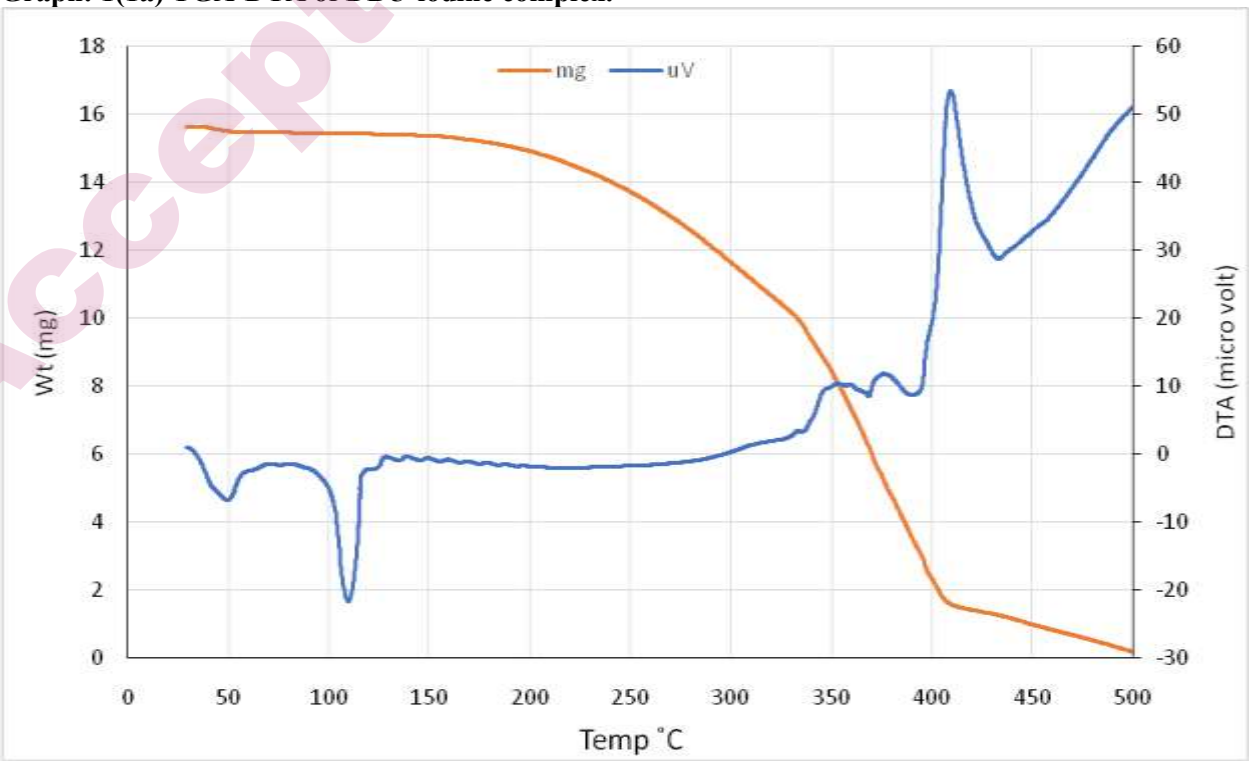


Graph: 1(1a) TGA-DTA of DBU-iodine complex.



Graph: 2(1b) TGA-DTA of Morpholine-iodine complex.

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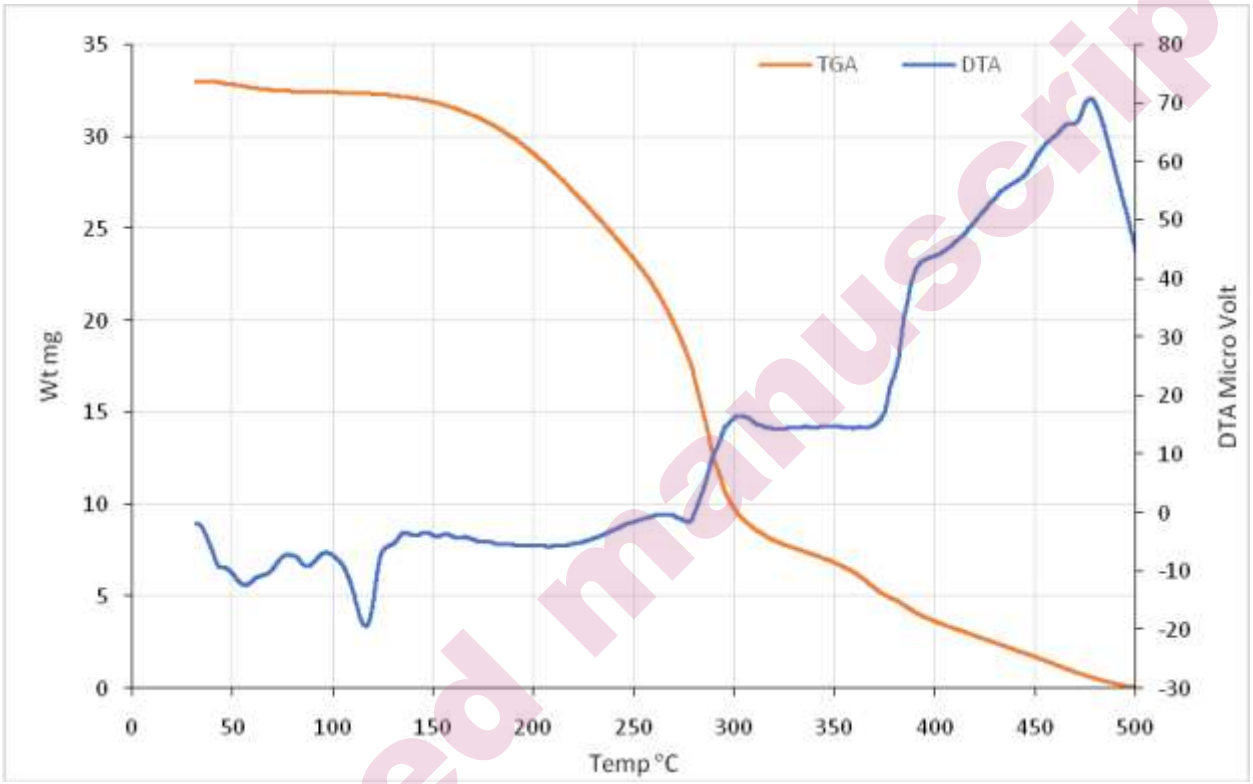

Graph: 3(1c) TGA-DTA of Urotropine-iodine complex.

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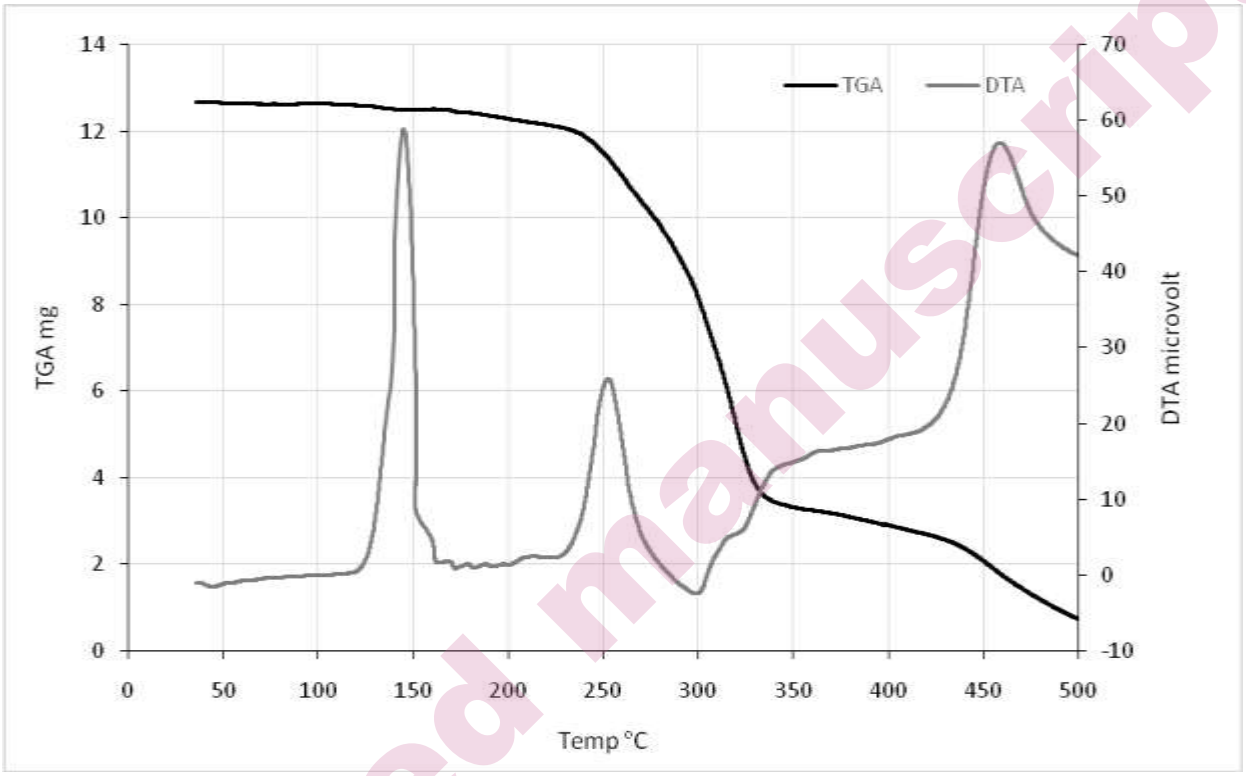
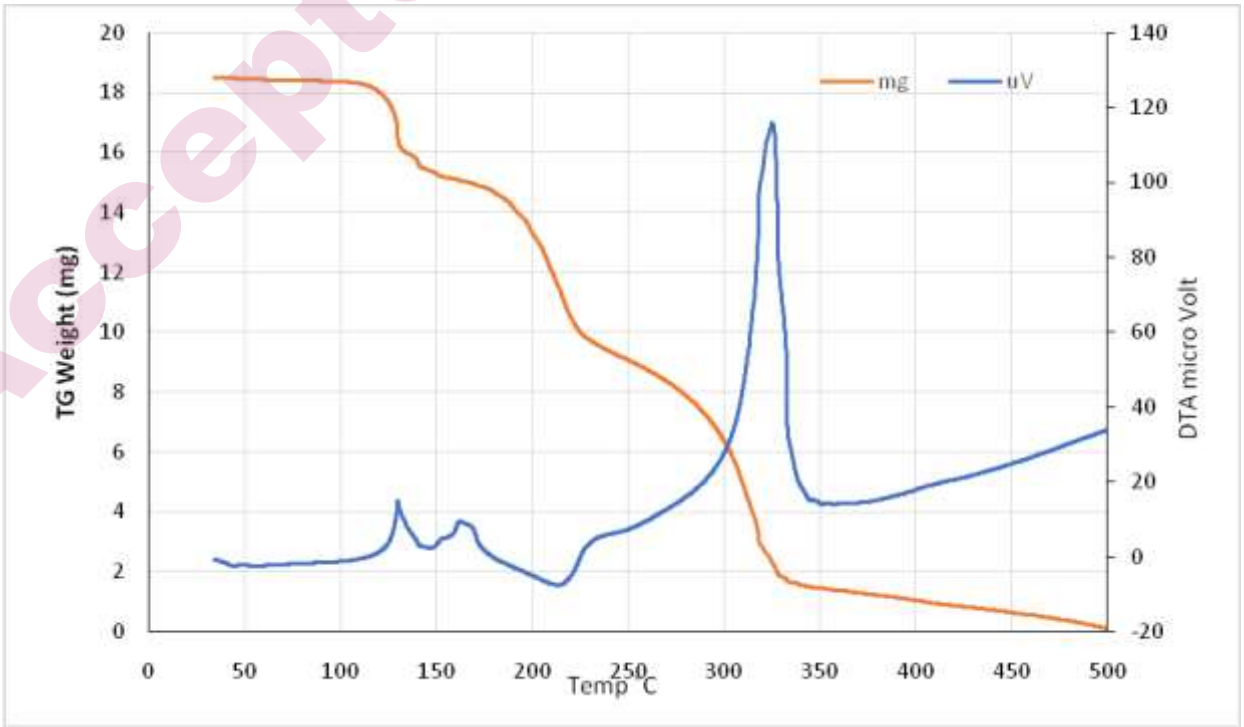
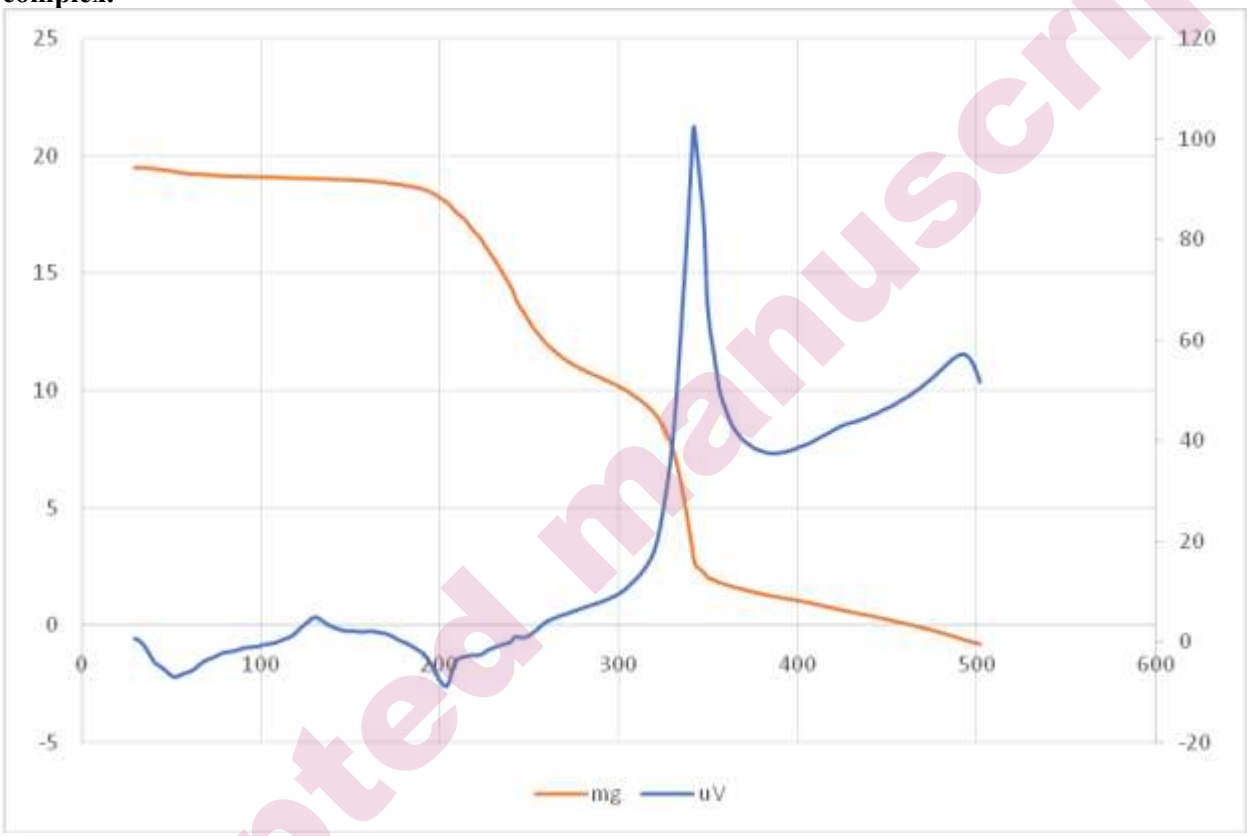


Graph: 4(1d) TGA-DTA of Piperazine-iodine complex.

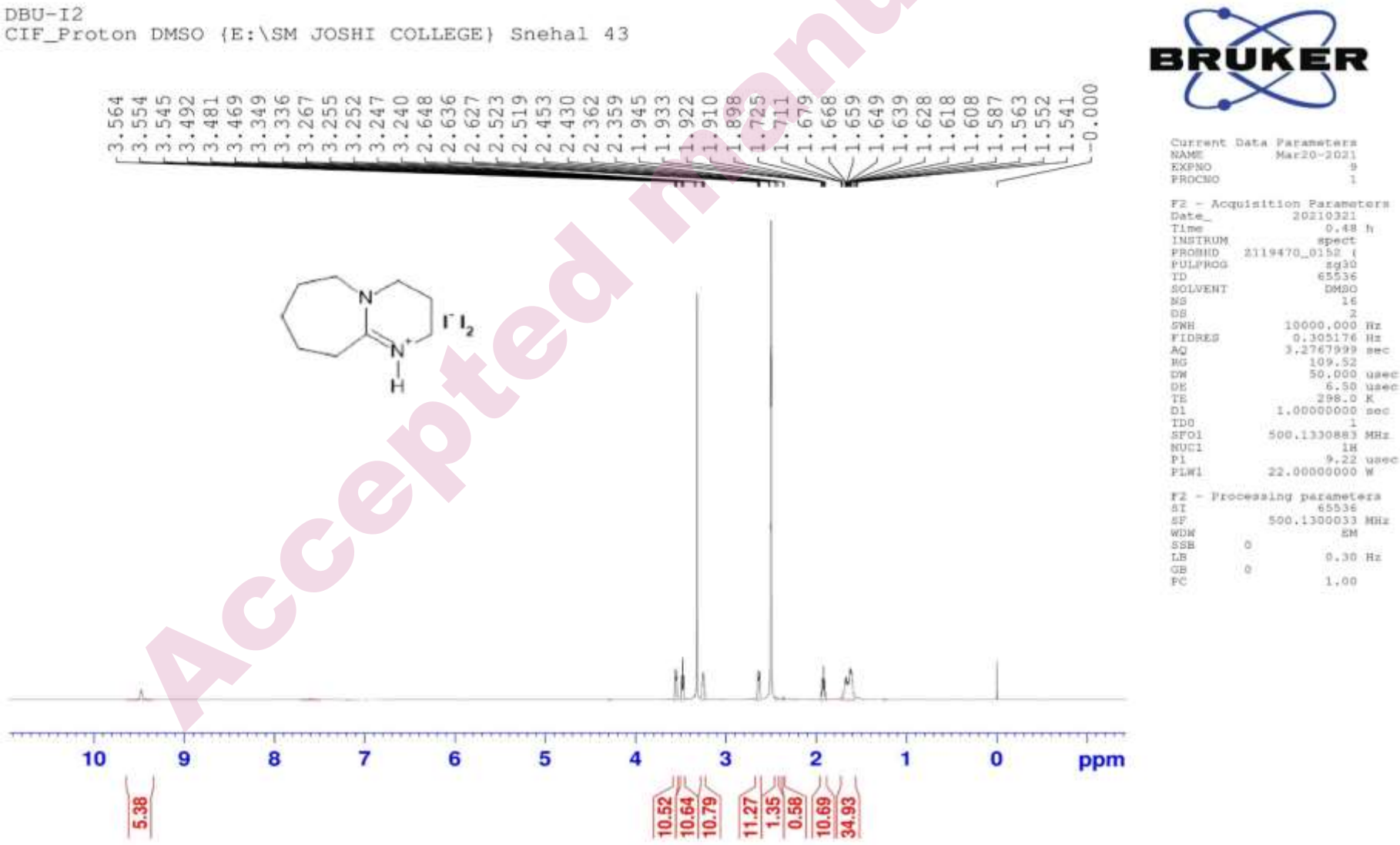


Accepted manuscript

Graph: 5(1e) TGA-DTA of N-methyl-piperazine-iodine complex.



Accepted manuscript



BRUKER

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DE 6.50 usec
TE 298.0 K
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TD0 1
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NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

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WDW EM
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Accepted manuscript

Fig: ^{1}H-NMR DBUH-I_3 complex (Table 1, Entry 1, 1a)

Accepted manuscript

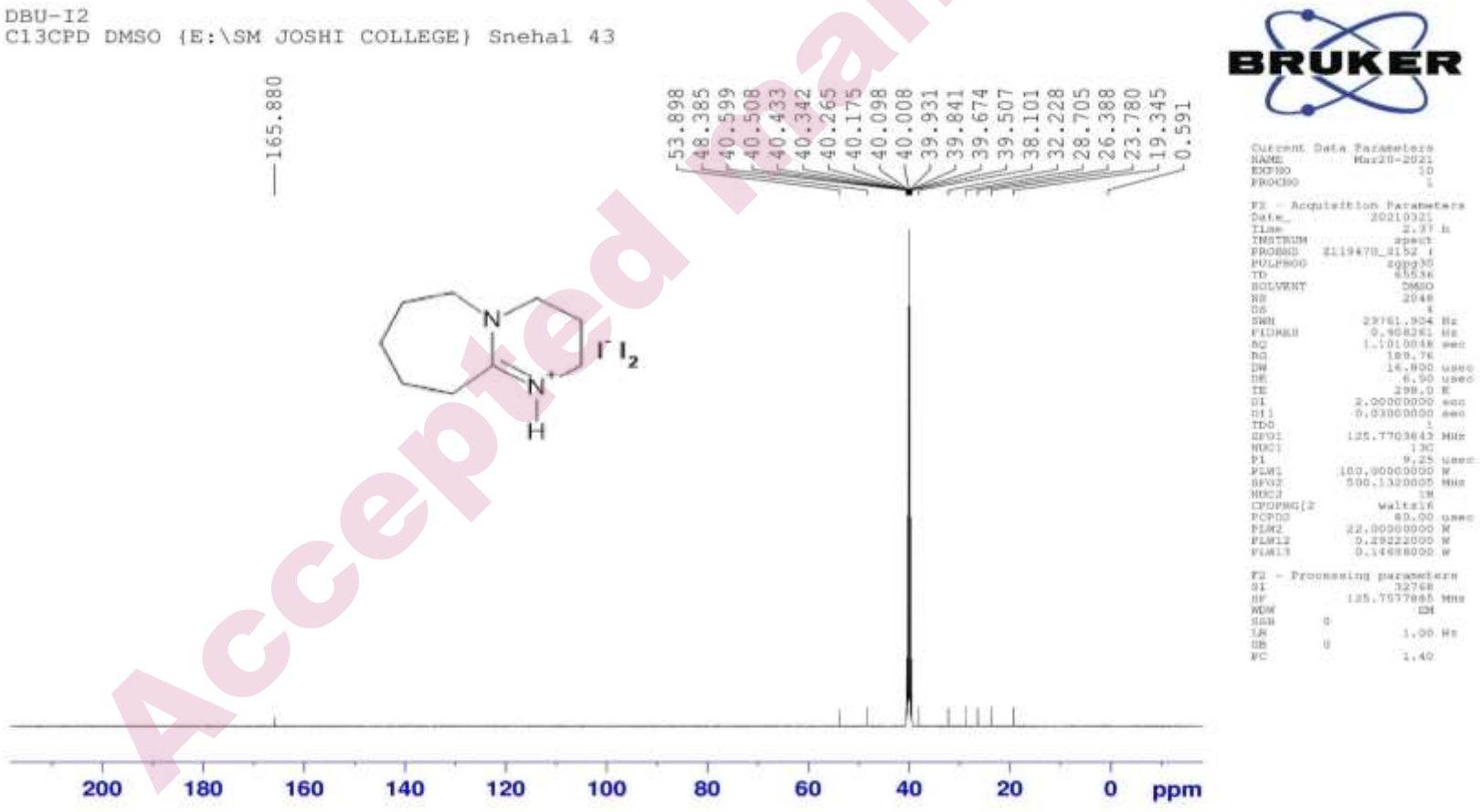

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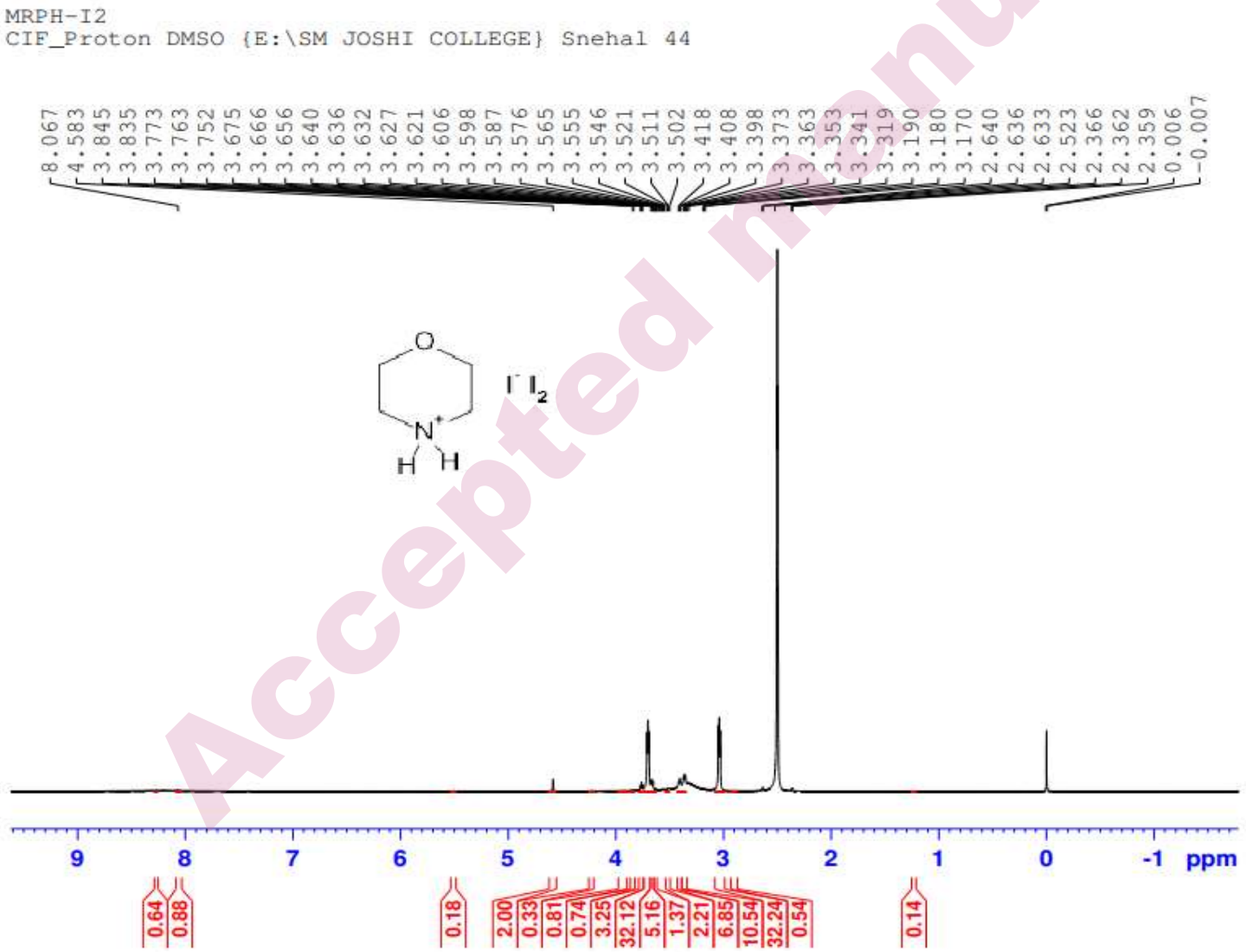
Fig: ^{13}C-NMR DBUH-I_3 complex (Table 1, Entry 1, 1a)

Accepted manuscript



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PLW1 22.00000000 W

F2 - Processing parameters
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WDW EM
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Accepted manuscript

Fig: ^{1}H-NMR Morpholine-Iodine complex (Table 1, Entry 2, 1b)

Accepted manuscript

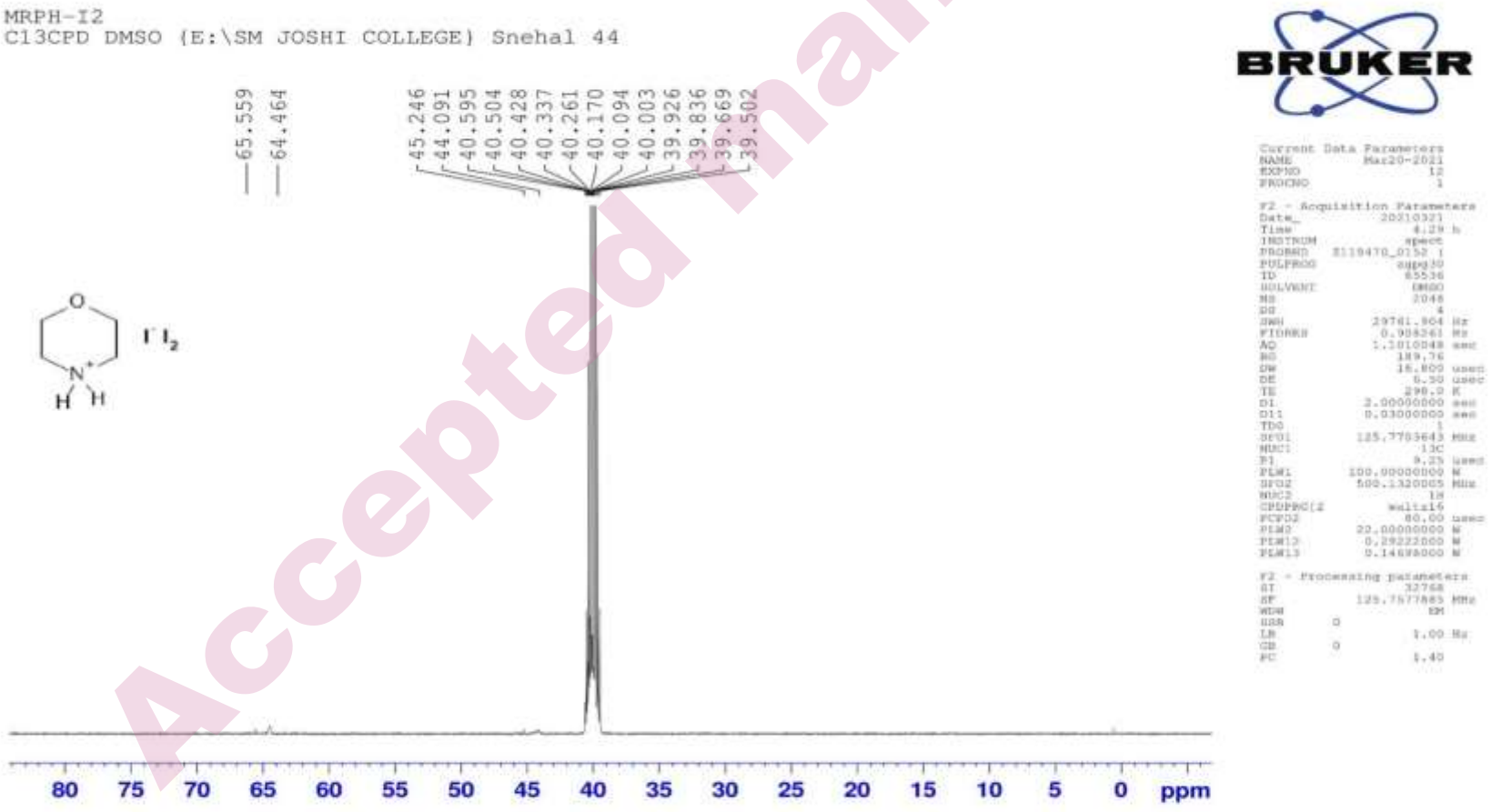

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Fig: ^{13}C-NMR Morpholine-Iodine complex (Table 1, Entry 2, 1b)

Accepted manuscript

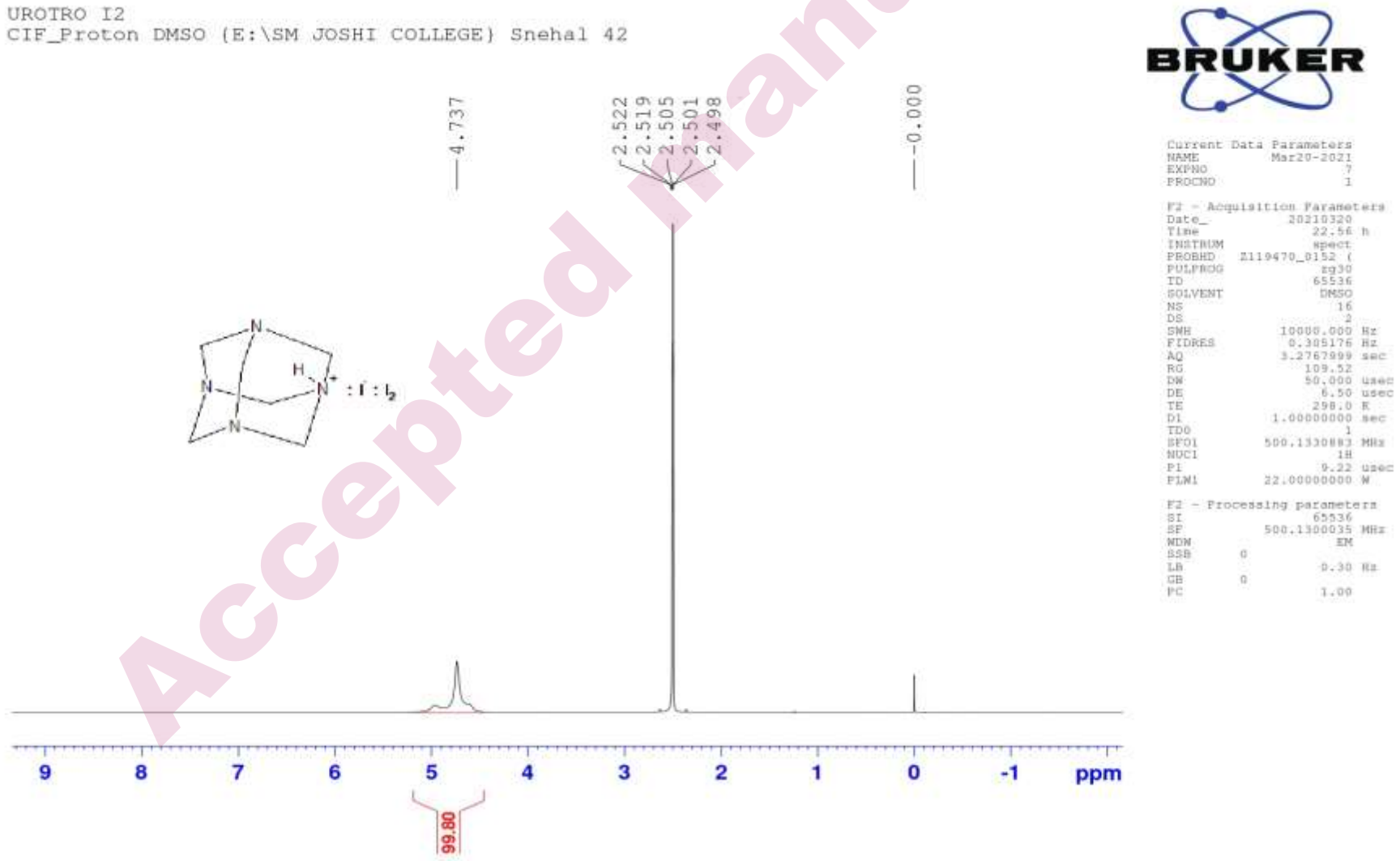


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Fig: ^{1}H-NMR Urotropine-Iodine complex (Table 1, Entry 3, 1c)

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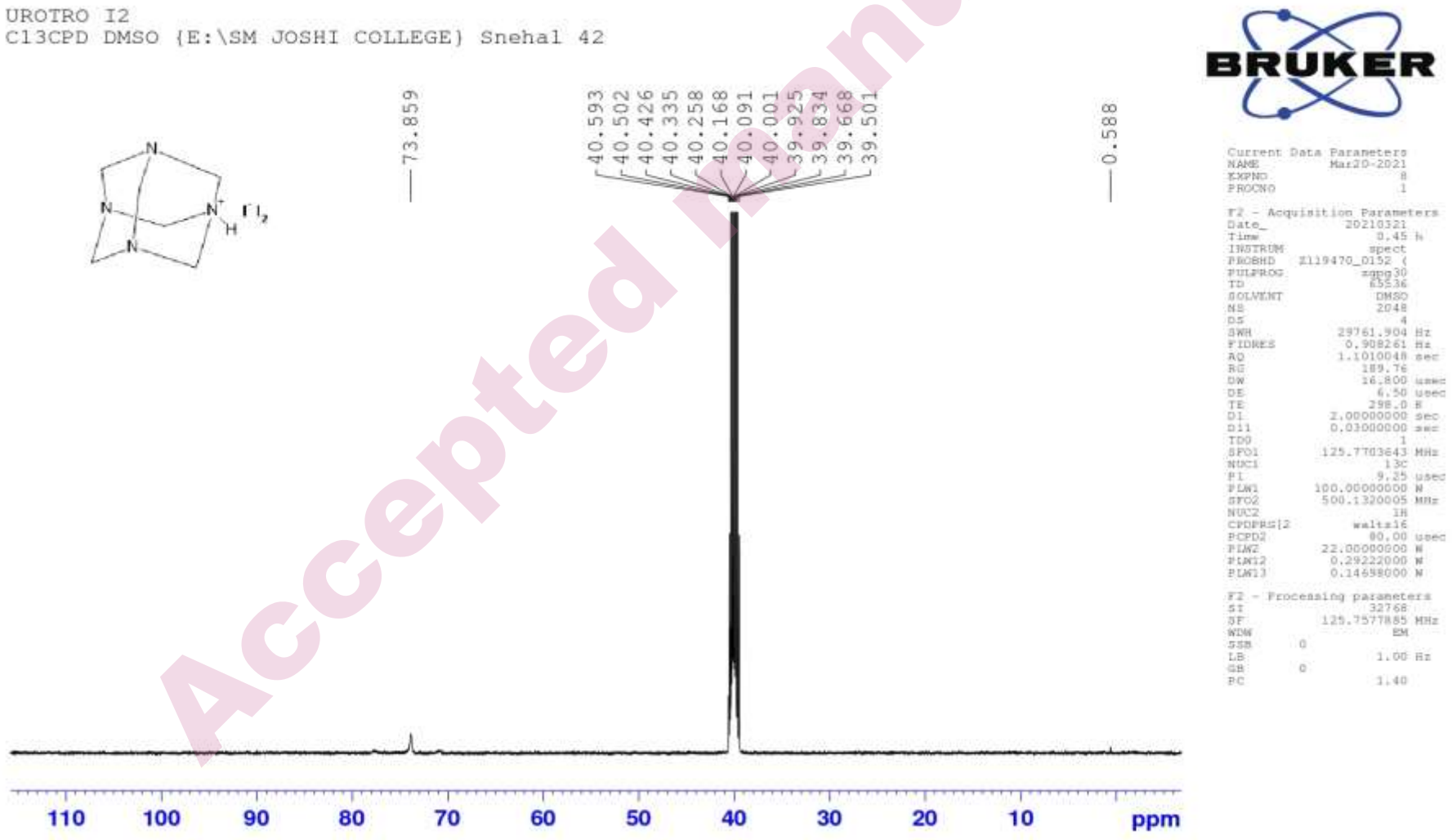

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Fig: ^{13}C-NMR Urotropine-Iodine complex (Table 1, Entry 3, 1c)

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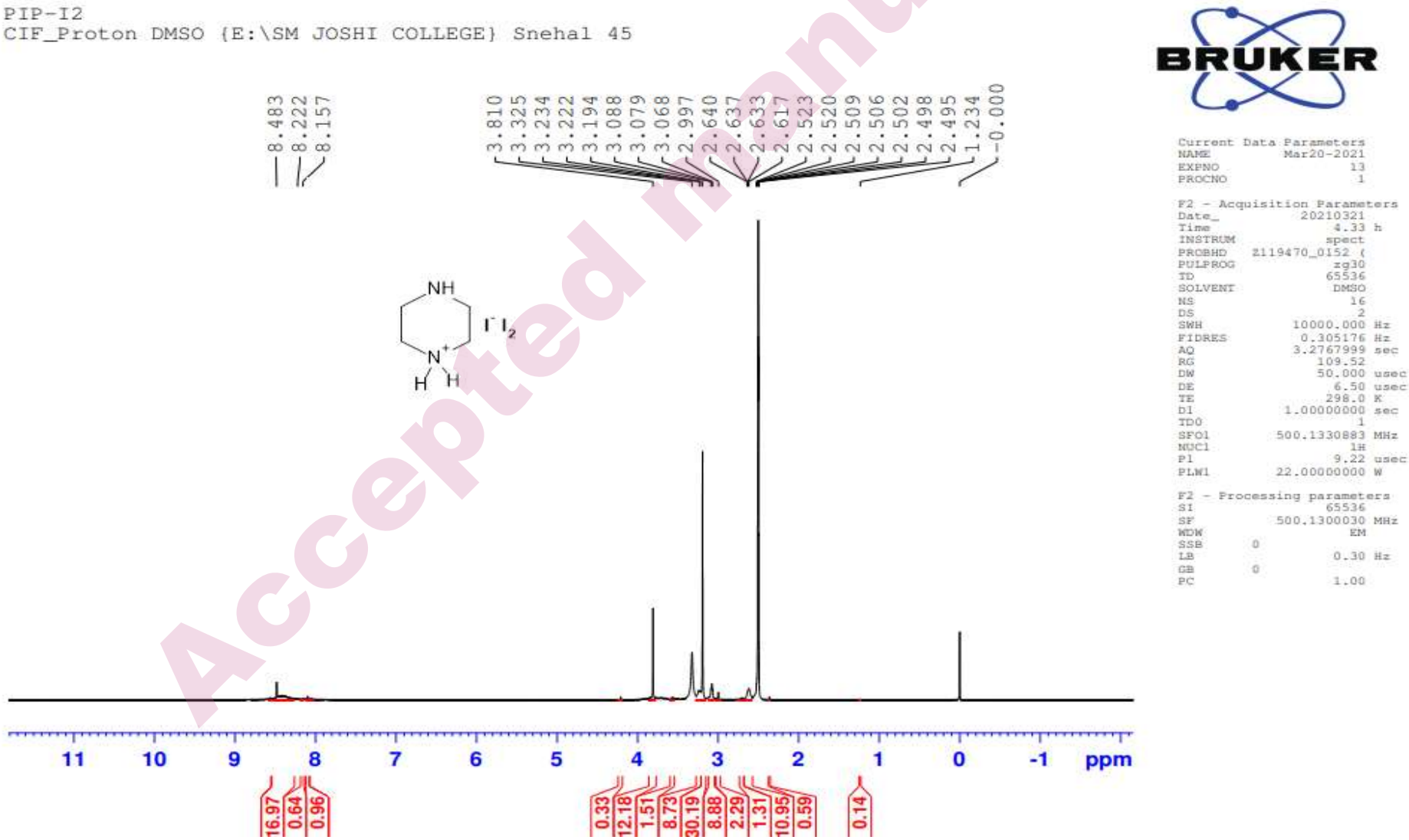

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F2 - Processing parameters
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WDW                           EM
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Fig: ^{1}H-NMRPiperazine-Iodine complex (Table 1, Entry 4, 1d)

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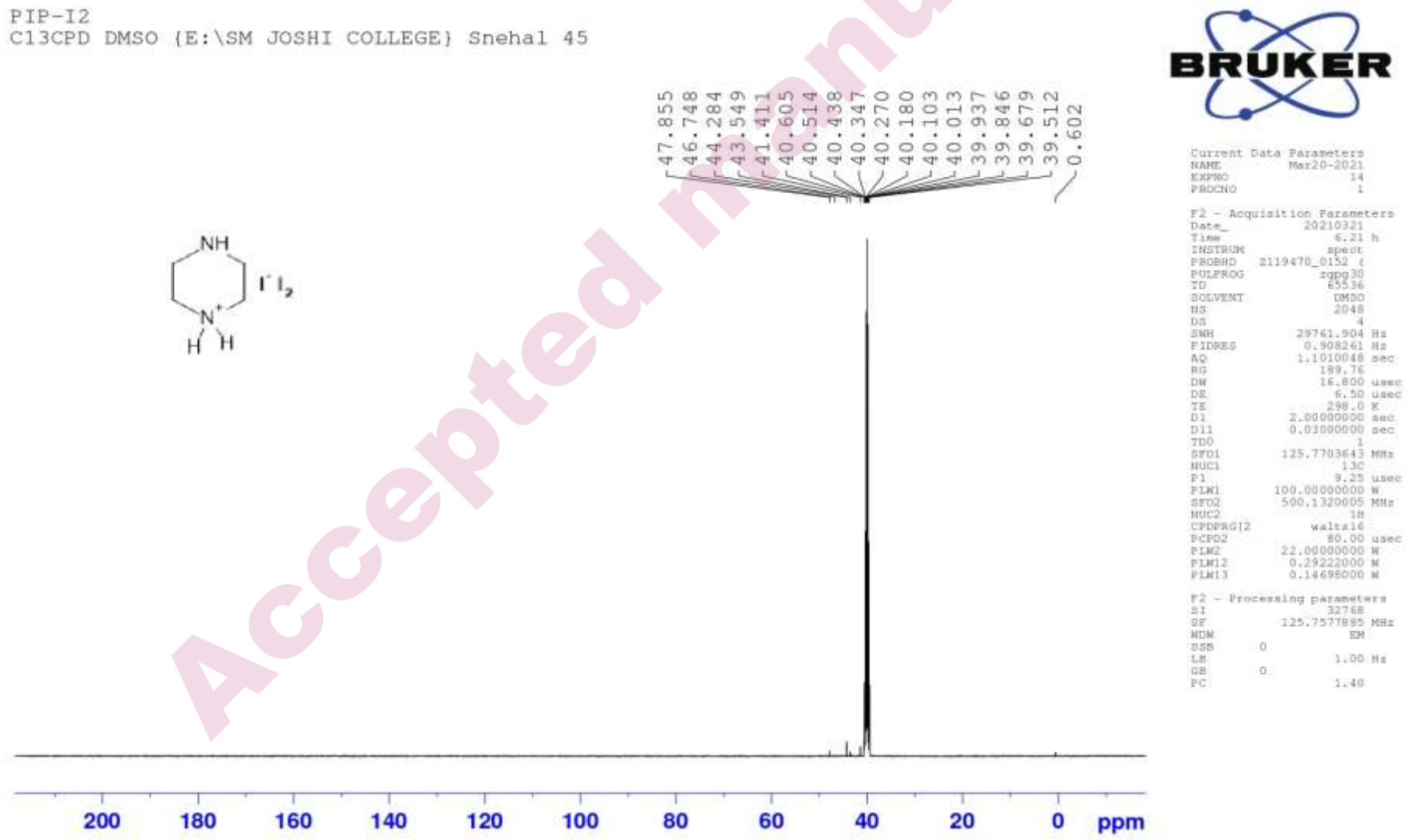

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PC 1.40

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Fig: ^{13}C-NMRPiperazine-Iodine complex (Table 1, Entry 4, 1d)

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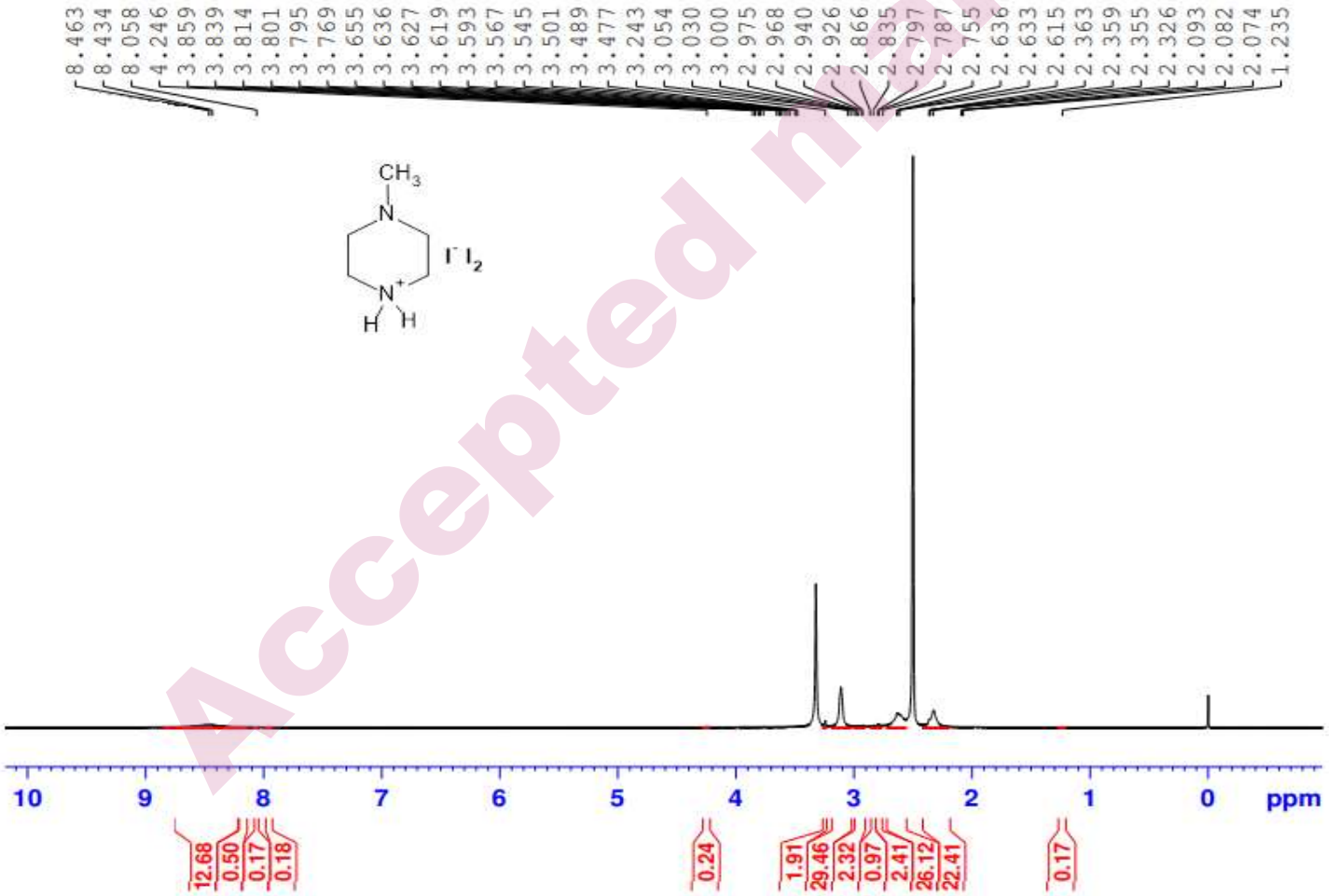
N METHYL PIP-I2
CIF_Proton DMSO {E:\SM JOSHI COLLEGE} Snehal 46



BRUKER

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PLW1 22.00000000 W

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PC 1.00

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Fig: ^{1}H-NMR N-Methyl-Piperazine-Iodine complex (Table 1, Entry 5, 1e)

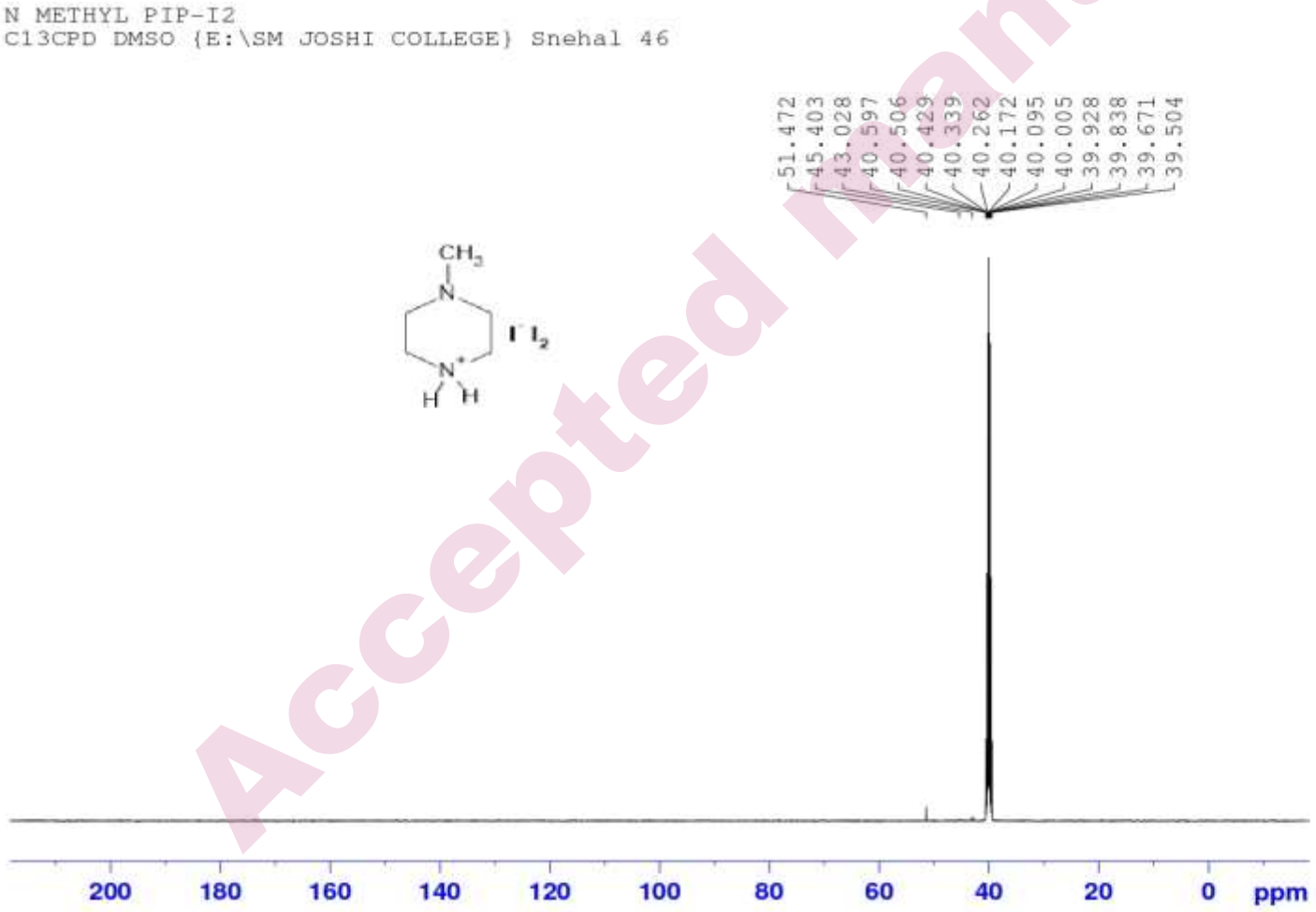

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Fig: ^{13}C-NMR N-Methyl-Piperazine-

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Iodine complex (Table 1, Entry 5, 1e)

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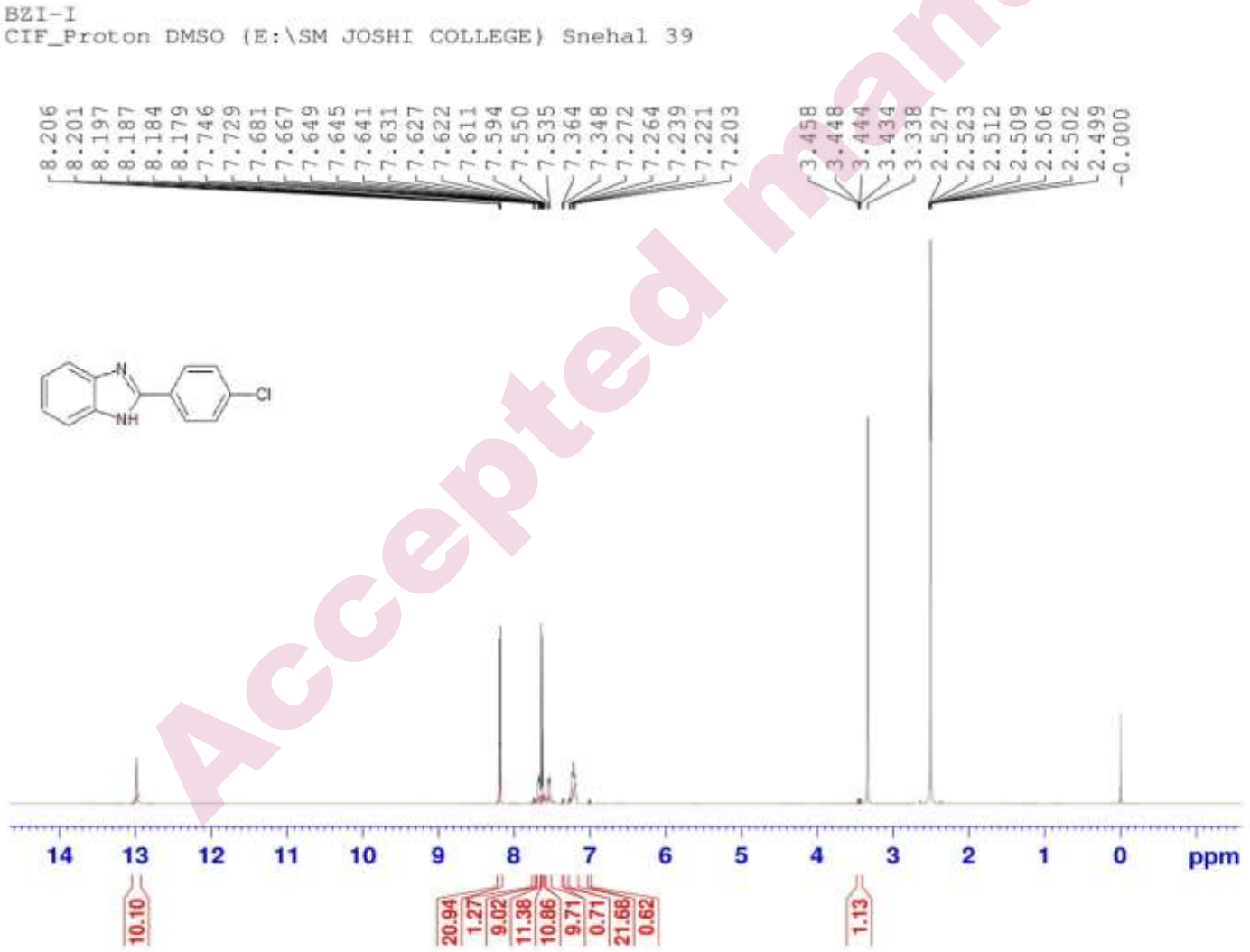

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PLW1 22.00000000 W

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Fig. ^{1}H-NMR of 2-(4-chlorophenyl)-*1H*-benzimidazole (Table 5, Entry 1, 4a)

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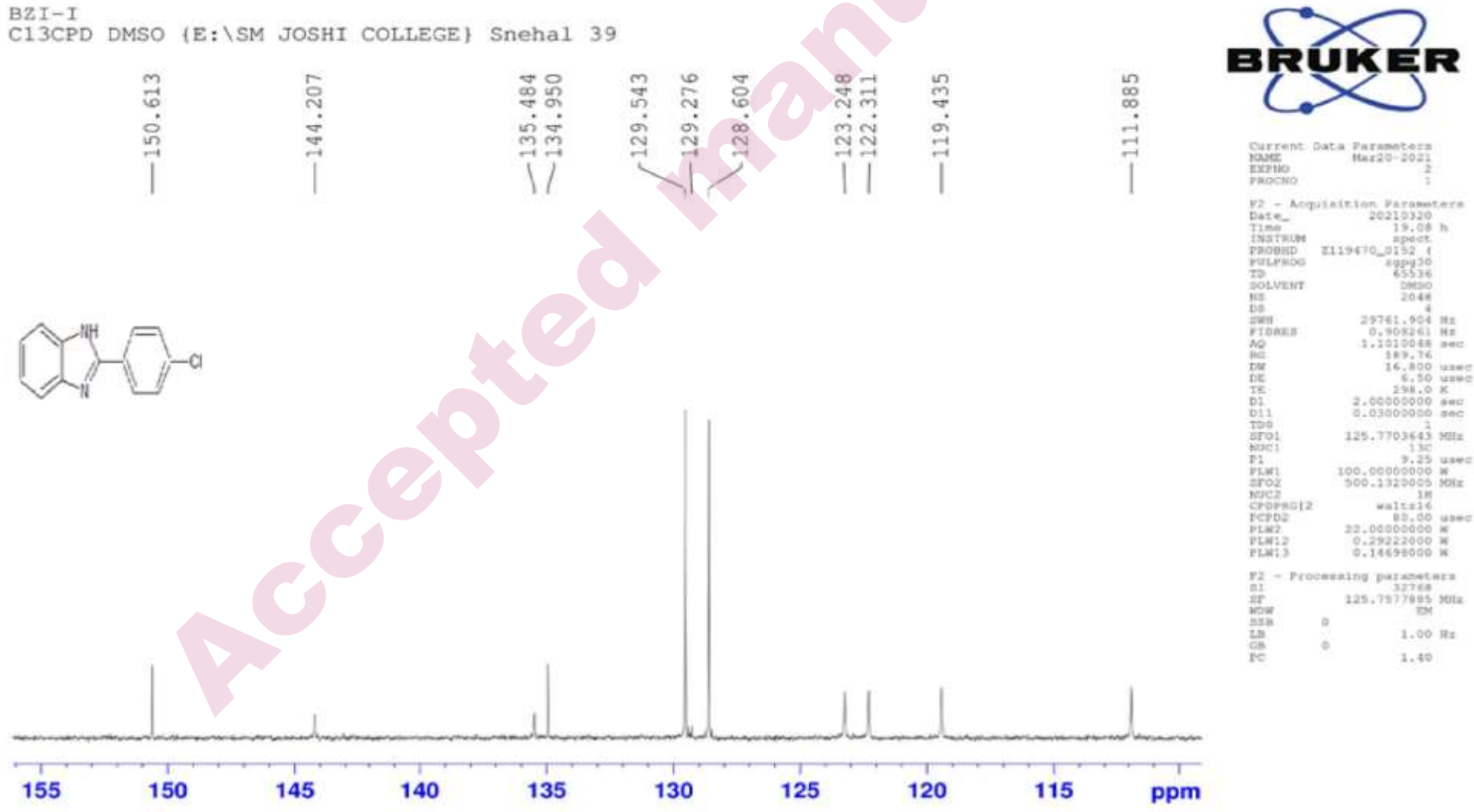

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RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577895 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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Fig. ^{13}C-NMR of 2-(4-chlorophenyl)-*1H*-benzimidazole (Table 5, Entry 1, 4a)

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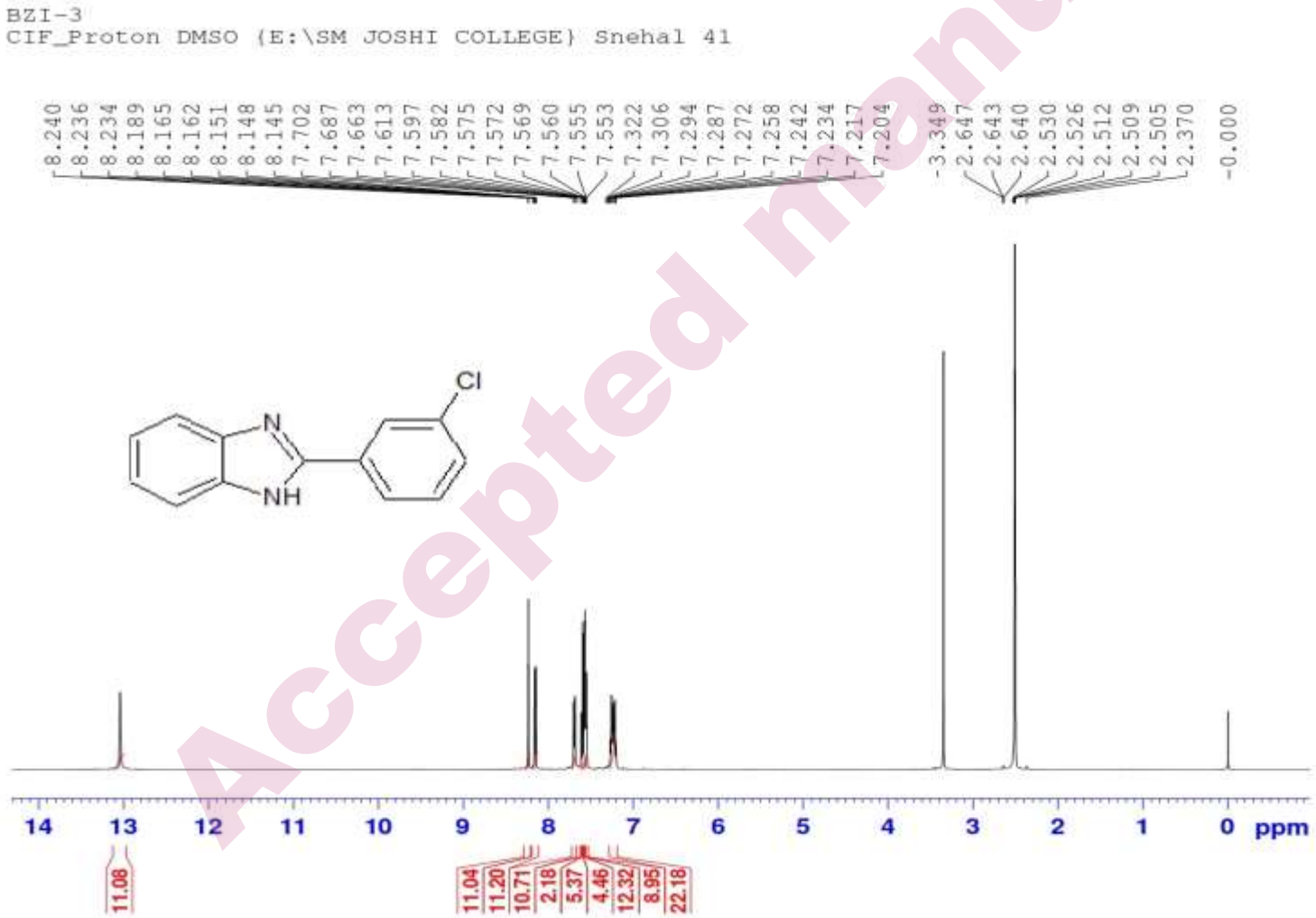

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Current Data Parameters
NAME Mar20-2021
EXPNO 5
PROCNO 1

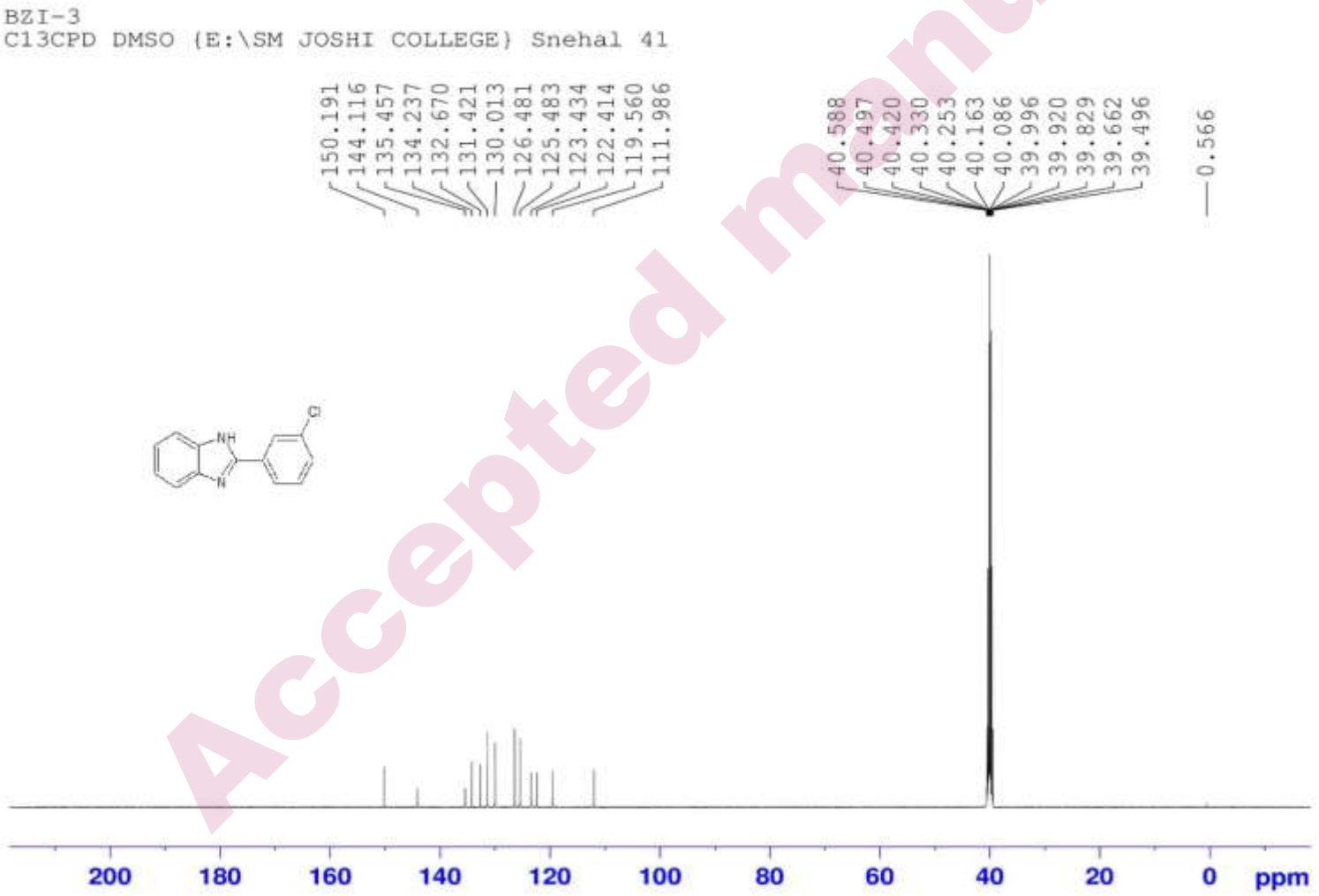
F2 - Acquisition Parameters
Date_ 20210320
Time 21.04 h
INSTRUM spect
PROBHD 2119470_0152 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1299997 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig:^{1}H-NMR2-(3-chlorophenyl)-1*H*-benzimidazole (Table 5, Entry 2, 4b)

Accepted manuscript



Current Data Parameters
NAME Mar20-2021
EXPNO 6
PROCNO 1

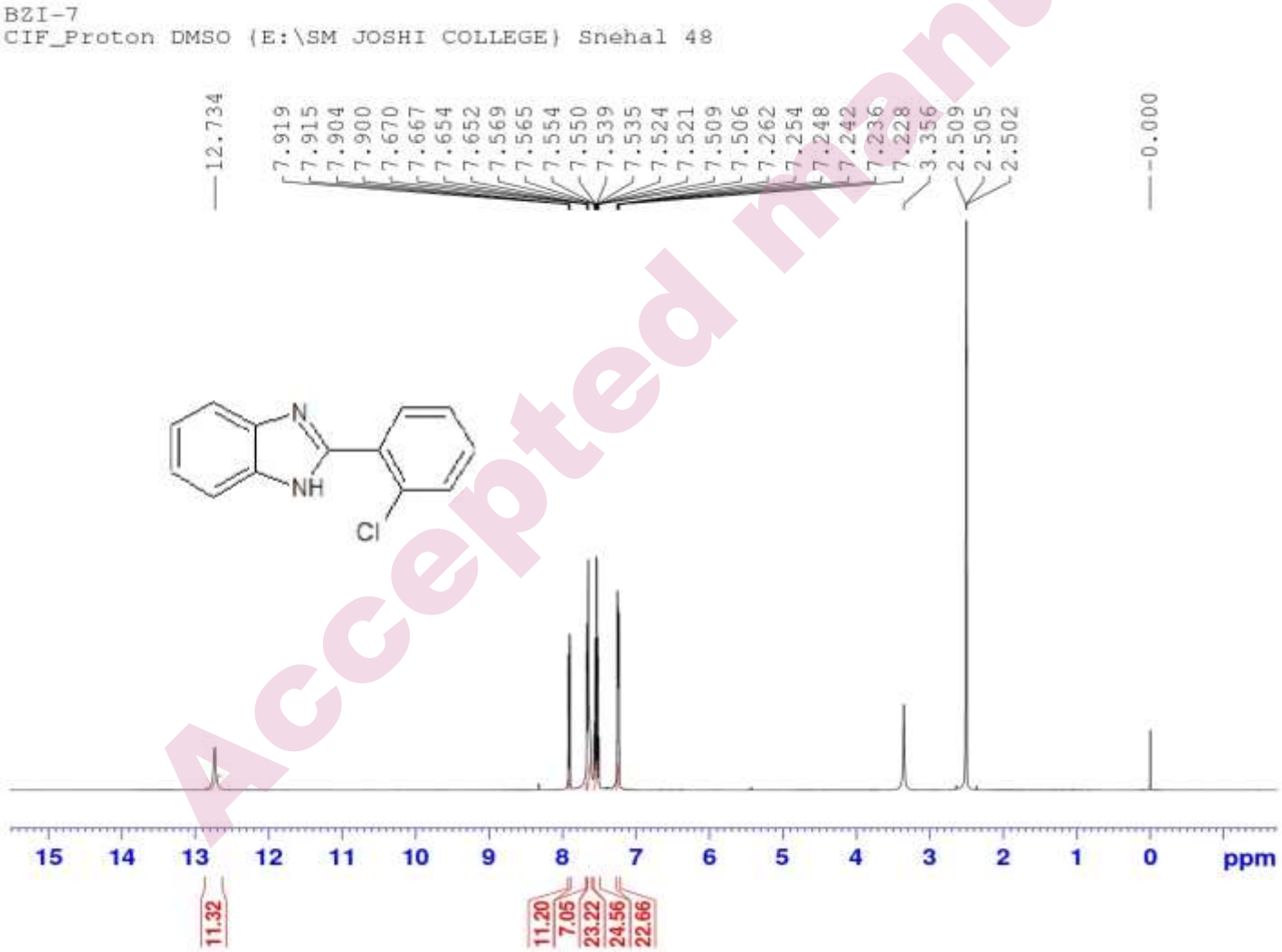
F2 - Acquisition Parameters
Date_ 20210320
Time 22.53 h
INSTRUM spect
PROBHD Z119478_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(3-chlorophenyl)-1*H*-benzimidazole (Table 5, Entry 2, 4b)

Accepted manuscript



Current Data Parameters
NAME Apr07-2021
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_	20210407	
Time	19.48	h
INSTRUM	spect	
PROBHD	I119470_0152 (	
PULPROG	zg30	
TD	65536	
SOLVENT	DMSO	
NS	64	
DS	2	
SWH	10000.000	Hz
FIDRES	0.305176	Hz
AQ	3.2767999	sec
RG	109.52	
DW	50.000	usec
DE	6.50	usec
TE	295.0	K
D1	1.00000000	sec
TD0	1	
SFO1	500.1330883	MHz
NUC1	1H	
P1	9.22	usec
PLW1	22.00000000	W

F2 - Processing parameters

SI	65536	
SF	500.1300012	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

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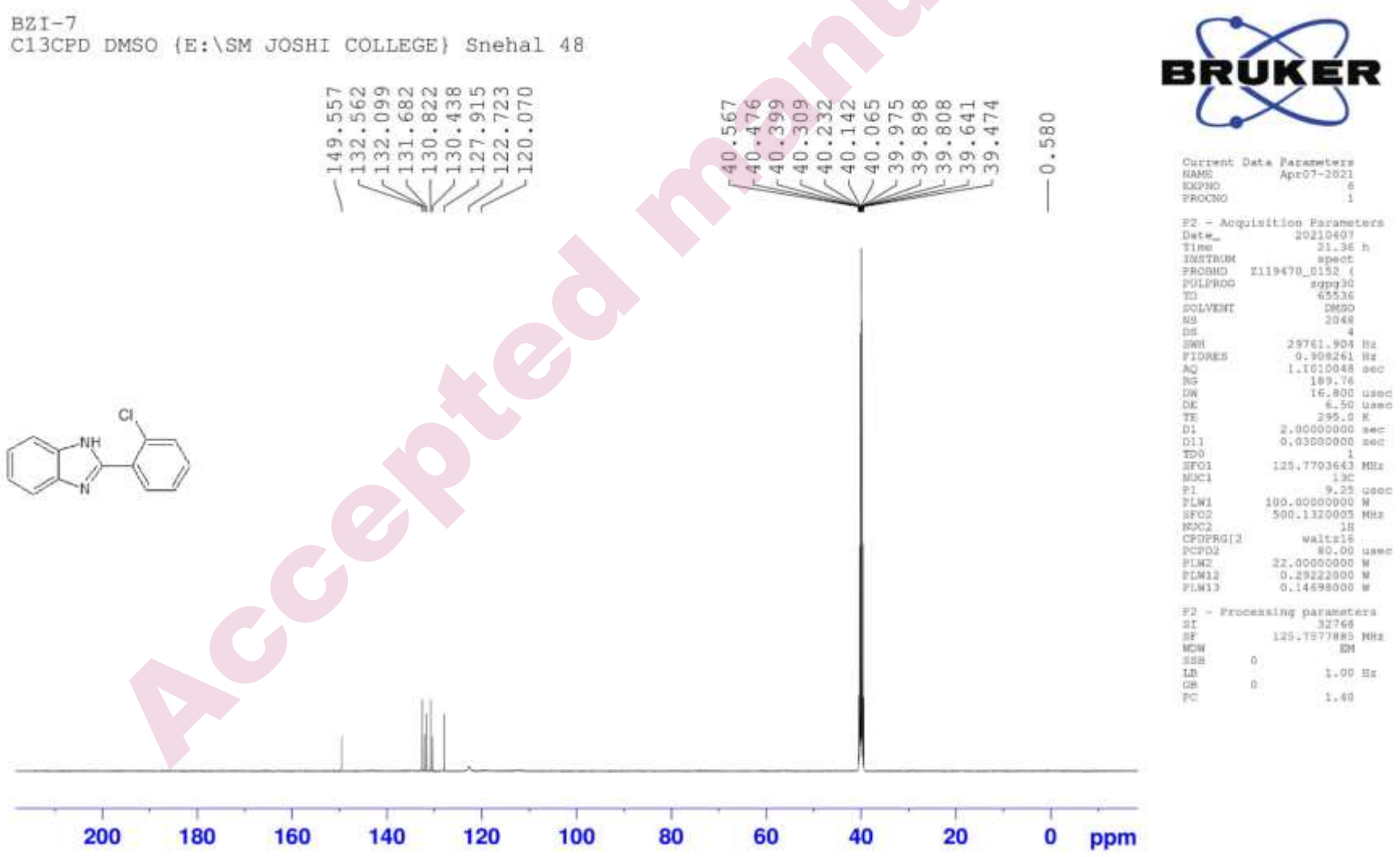
Fig: ^{1}H-NMR 2-(2-chlorophenyl)-*1H*-benzimidazole (Table 5, Entry 3, 4c)

Accepted manuscript



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Current Data Parameters
NAME                  Apr07-2021
EXPNO                          6
PROCNO                         1

F2 - Acquisition Parameters
Date_               20210407
Time                      21.36 h
INSTRUM                   spect
PROBHD     Z119470_0152 (
PULPROG                 zgpg30
TD                       65536
SOLVENT                   DMSO
NS                        2048
DS                           4
SWH                   29761.904 Hz
FIDRES                 0.908261 Hz
AQ                    1.1010048 sec
RG                       189.76
DW                       16.800 usec
DE                        6.50 usec
TE                       295.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1
SFO1                125.7703643 MHz
NUC1                        13C
P1                        9.25 usec
PLW1                100.00000000 W
SFO2                500.1320005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2                 22.00000000 W
PLW12                 0.29222000 W
PLW13                 0.14698000 W

F2 - Processing parameters
SI                        32768
SF                  125.7577885 MHz
WDW                          EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
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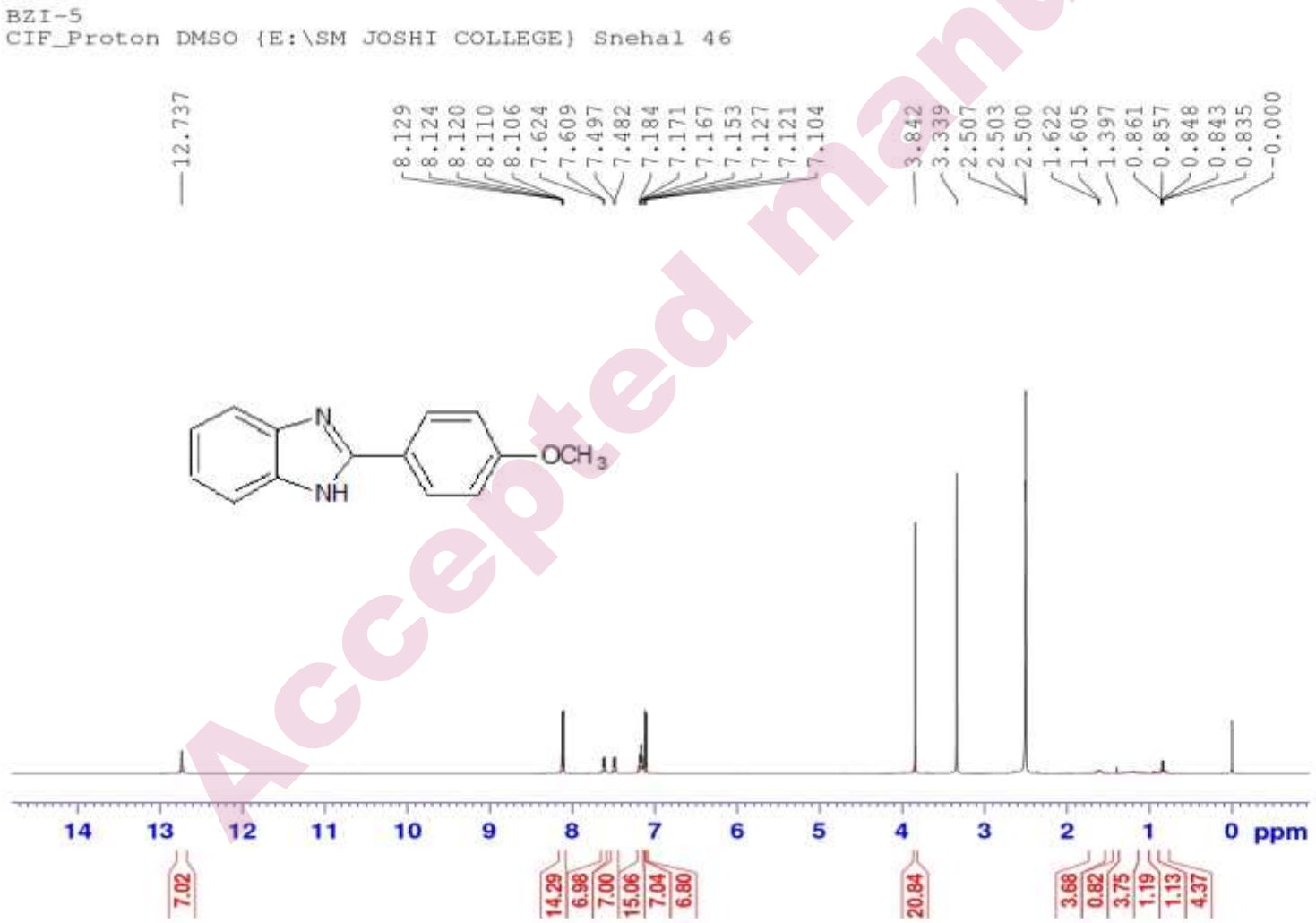
Accepted manuscript

Fig: ^{13}C-NMR 2-(2-chlorophenyl)-*1H*-benzimidazole (Table 5, Entry 3, 4c)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Apr07-2021
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210407
Time 15.55 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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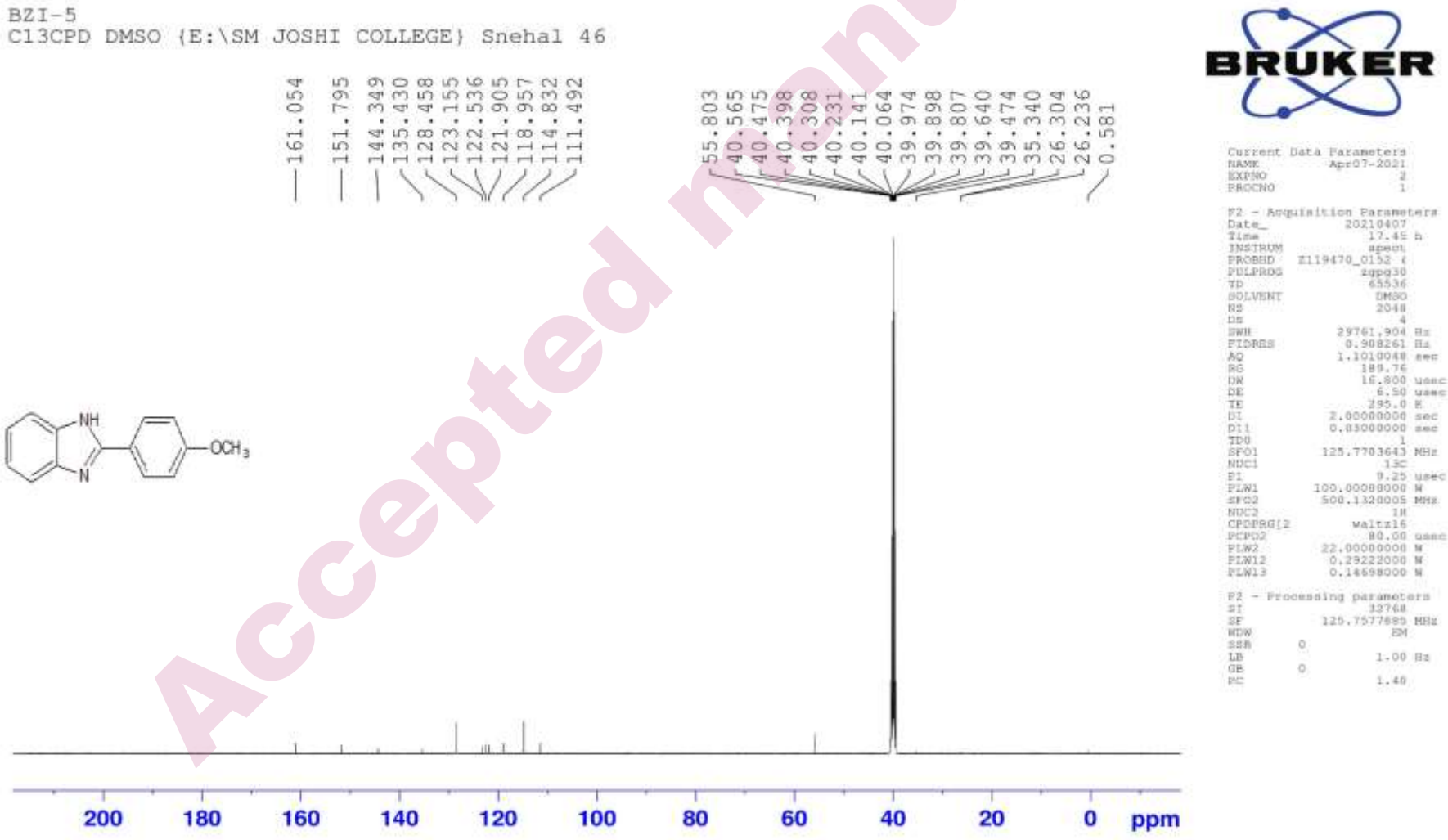
Fig: ^{1}H-NMR 2-(4-Methoxyphenyl)-*1H*-benzimidazole (Table 5, Entry 5, 4e)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Apr07-2021
EXPNO 2
PROCNO 1

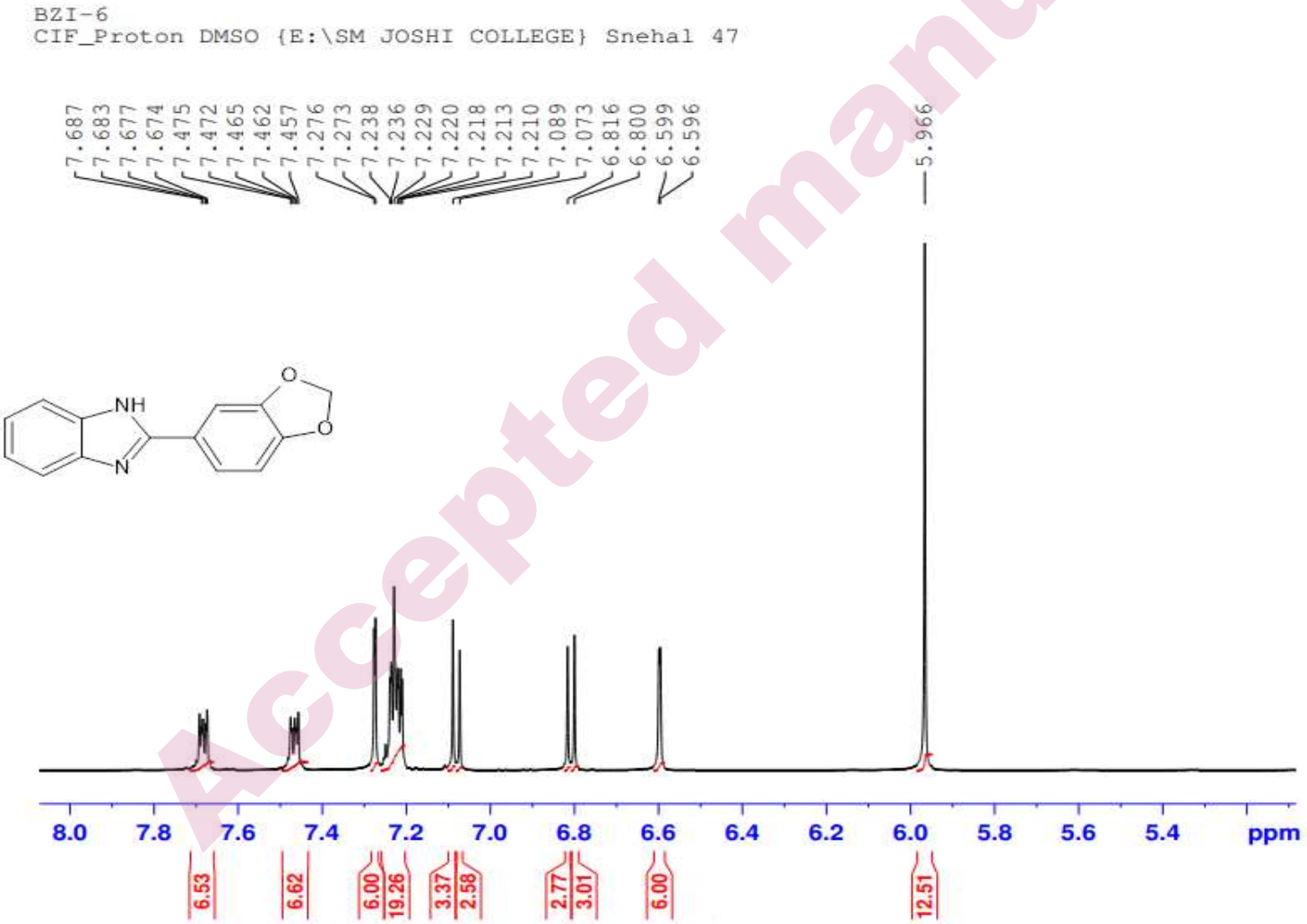
F2 - Acquisition Parameters
Date_ 20210407
Time 17.45 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577895 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(4-Methoxyphenyl)-*1H*-benzimidazole (Table 5, Entry 5, 4e)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Apr07-2021
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20210407
Time                       17.52 h
INSTRUM                    spect
PROBHD     Z119470_0152 (
PULPROG                     zg30
TD                         65536
SOLVENT                     DMSO
NS                            64
DS                             2
SWH                    10000.000 Hz
FIDRES                     0.305176 Hz
AQ                     3.2767999 sec
RG                         109.52
DW                         50.000 usec
DE                          6.50 usec
TE                         295.0 K
D1                    1.00000000 sec
TD0                            1
SFO1                 500.1330883 MHz
NUC1                          1H
P1                          9.22 usec
PLW1                 22.00000000 W

F2 - Processing parameters
SI                         65536
SF                   500.1300025 MHz
WDW                           EM
SSB                 0
LB                         0.30 Hz
GB                  0
PC                         1.00
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Fig: ^{1}H-NMR 2-(2H-1,3-benzodioxol-5-yl)-*1H*-benzimidazole (Table 5, Entry 8, 4h)

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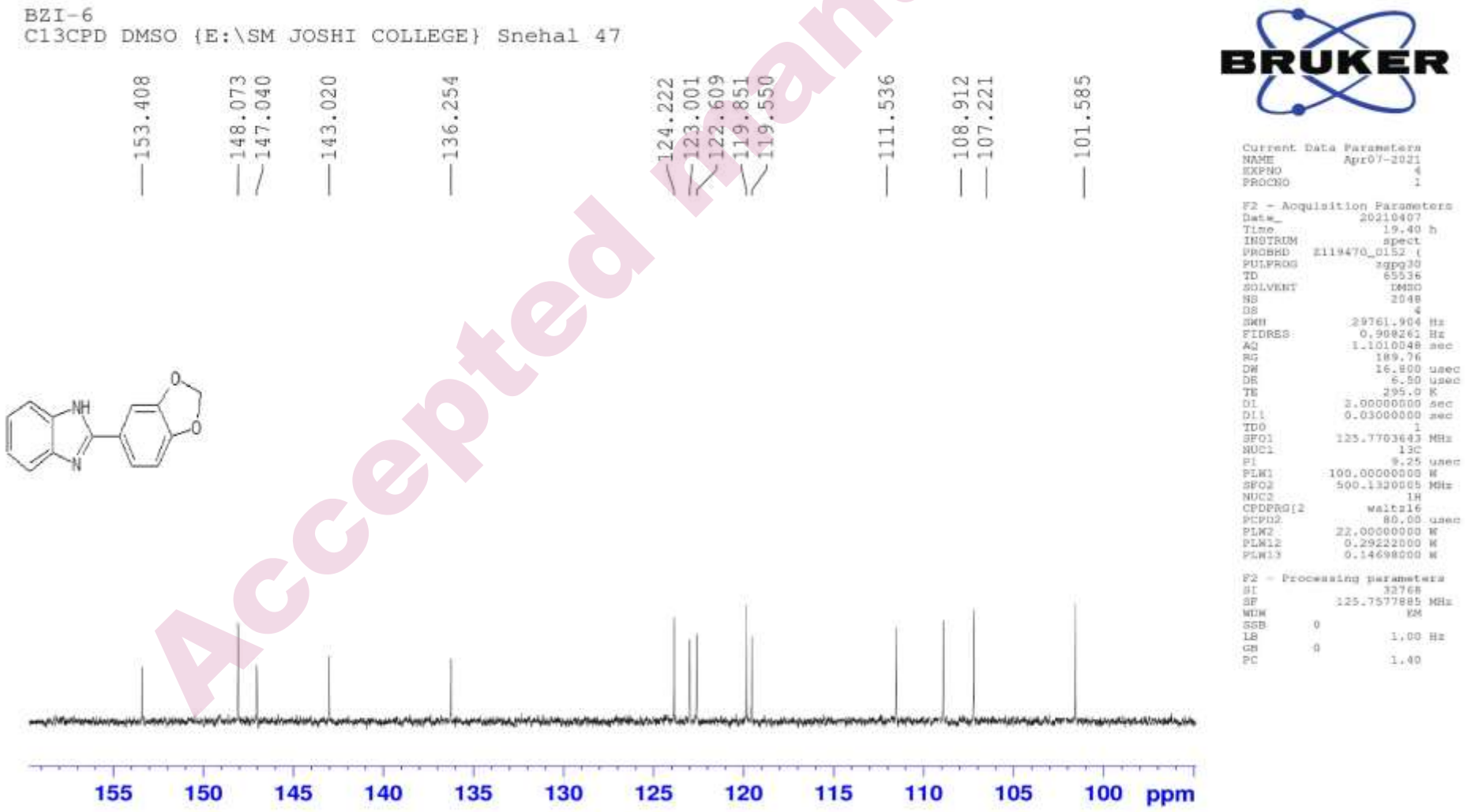


BRUKER

Current Data Parameters
NAME Apr07-2021
EXPNO 4
PROCNO 1

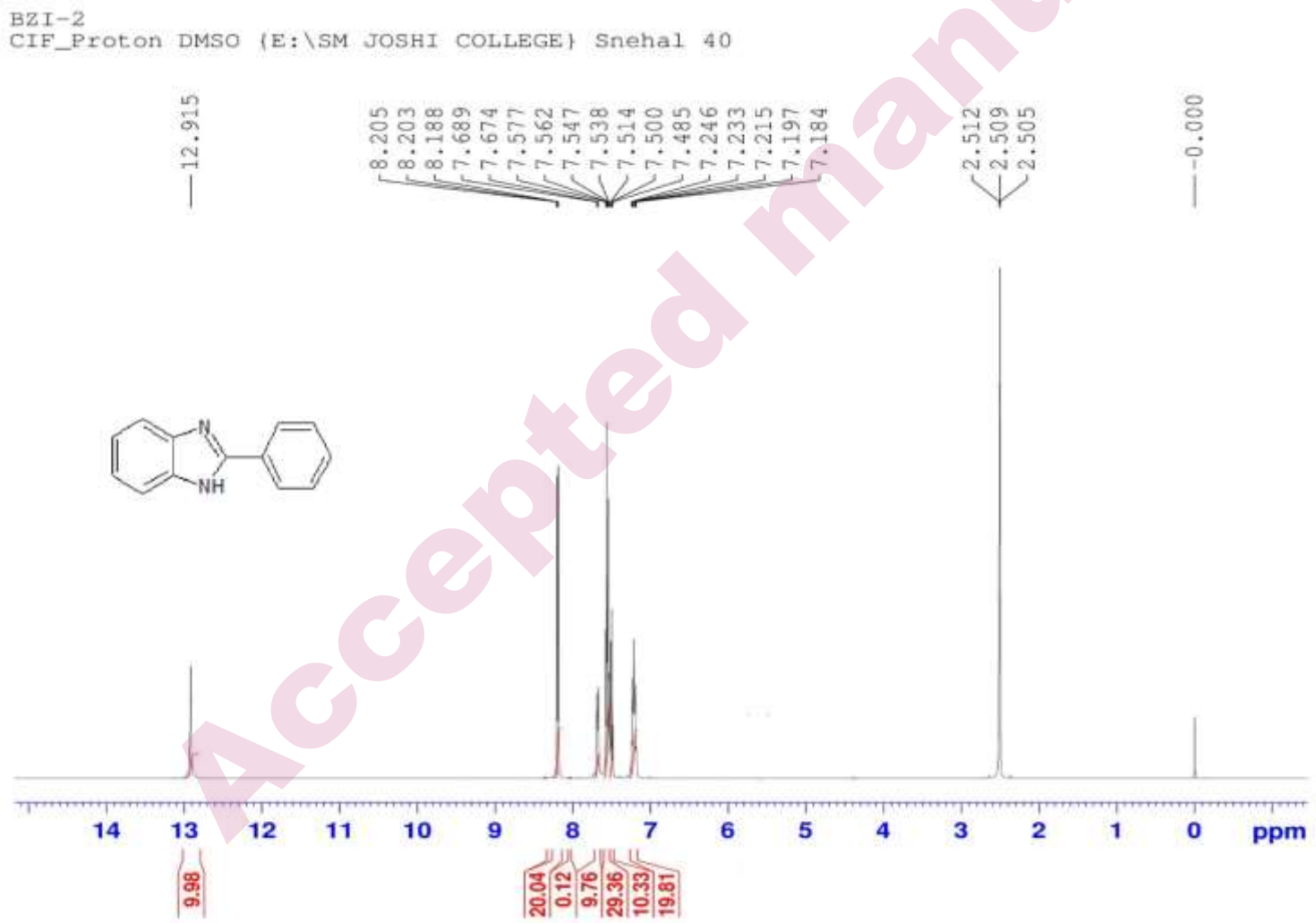
F2 - Acquisition Parameters
Date_ 20210407
Time 19.40 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(2H-1,3-benzodioxol-5-yl)-*1H*-benzimidazole (Table 5, Entry 8, 4h)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Mar20-2021
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_               20210320
Time                      19.12 h
INSTRUM                   spect
PROBHD     5119470_0152 (
PULPROG                   zg30
TD                       65536
SOLVENT                   DMSO
NS                          16
DS                           2
SWH                  10000.000 Hz
FIDRES                0.305176 Hz
AQ                    3.2767999 sec
RG                       109.52
DW                       50.000 usec
DE                        6.50 usec
TE                      298.0 K
D1                    1.00000000 sec
TD0                          1
SFO1                500.1330883 MHz
NUC1                        1H
P1                        9.22 usec
PLW1                22.00000000 W

F2 - Processing parameters
SI                       65536
SF                  500.1299997 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
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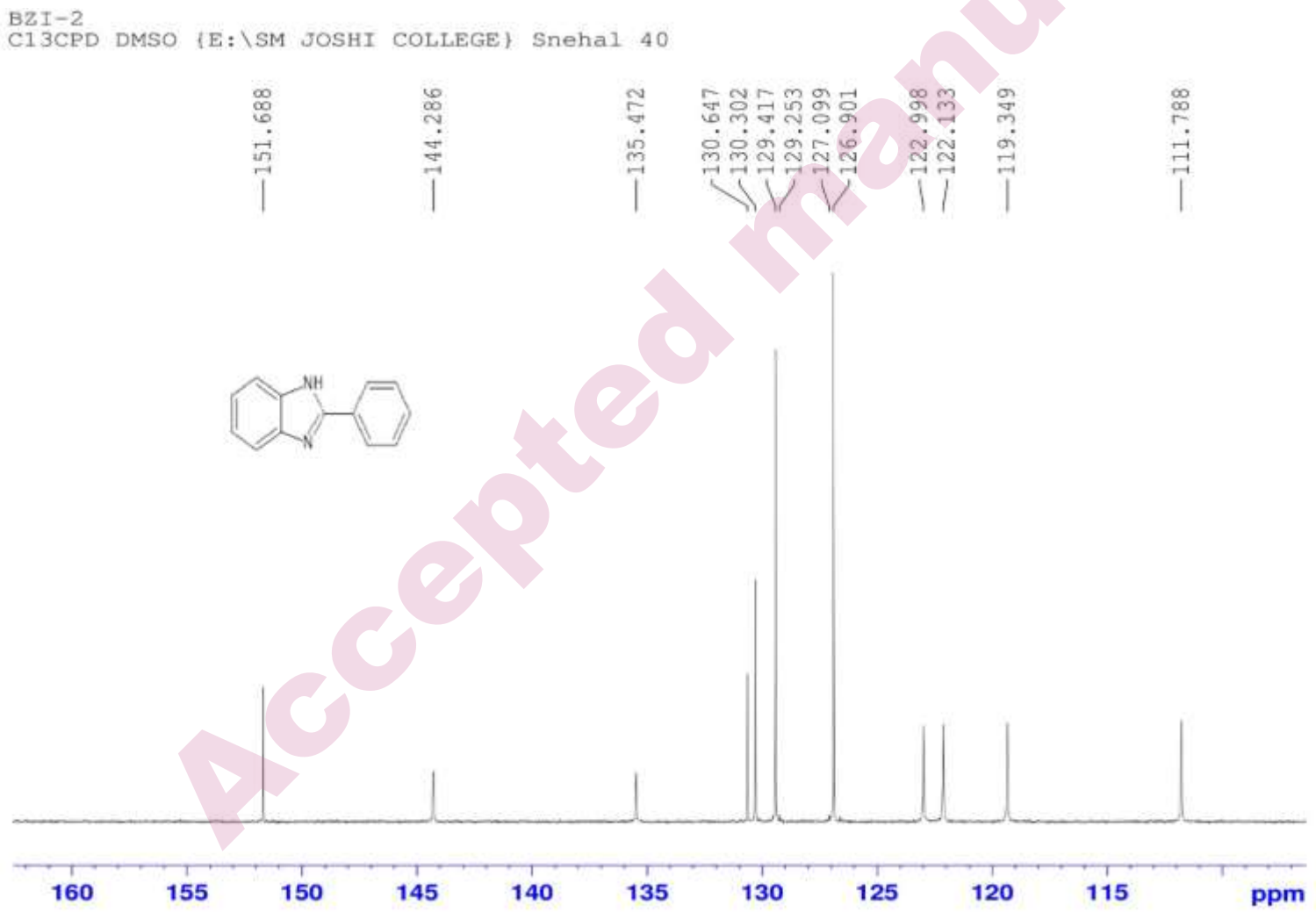
Accepted manuscript

Fig: ^{1}H-NMR2-phenyl-*1H*-benzimidazole (Table 5, Entry 9, 4i)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Mar20-2021
EXPNO 4
PROCNO 1

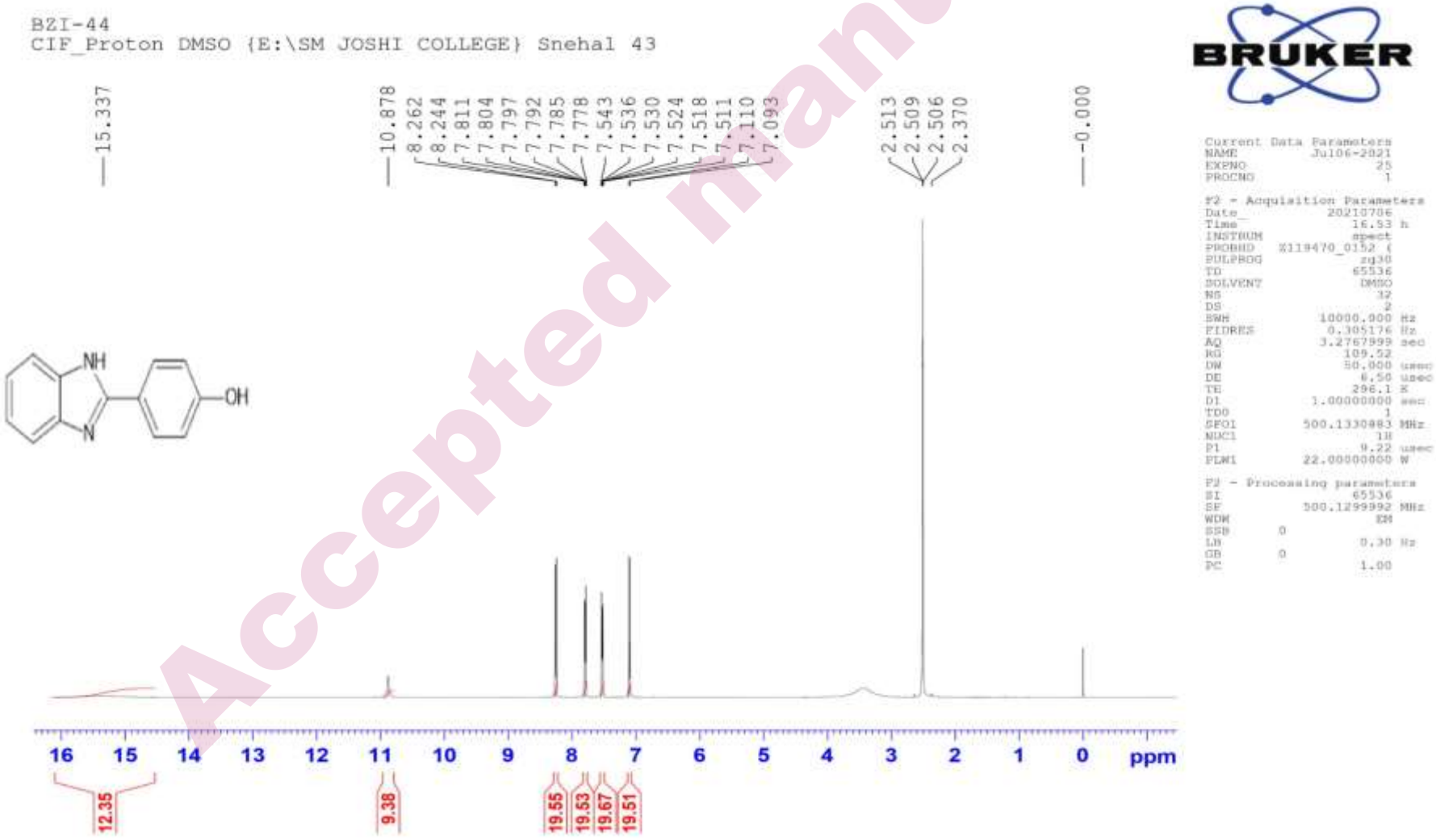
F2 - Acquisition Parameters
Date_ 20210320
Time 21.00 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14598000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR2-phenyl-*1H*-benzimidazole (Table 5, Entry 9, 4i)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Ju106-2021
EXPNO 25
PROCNO 1

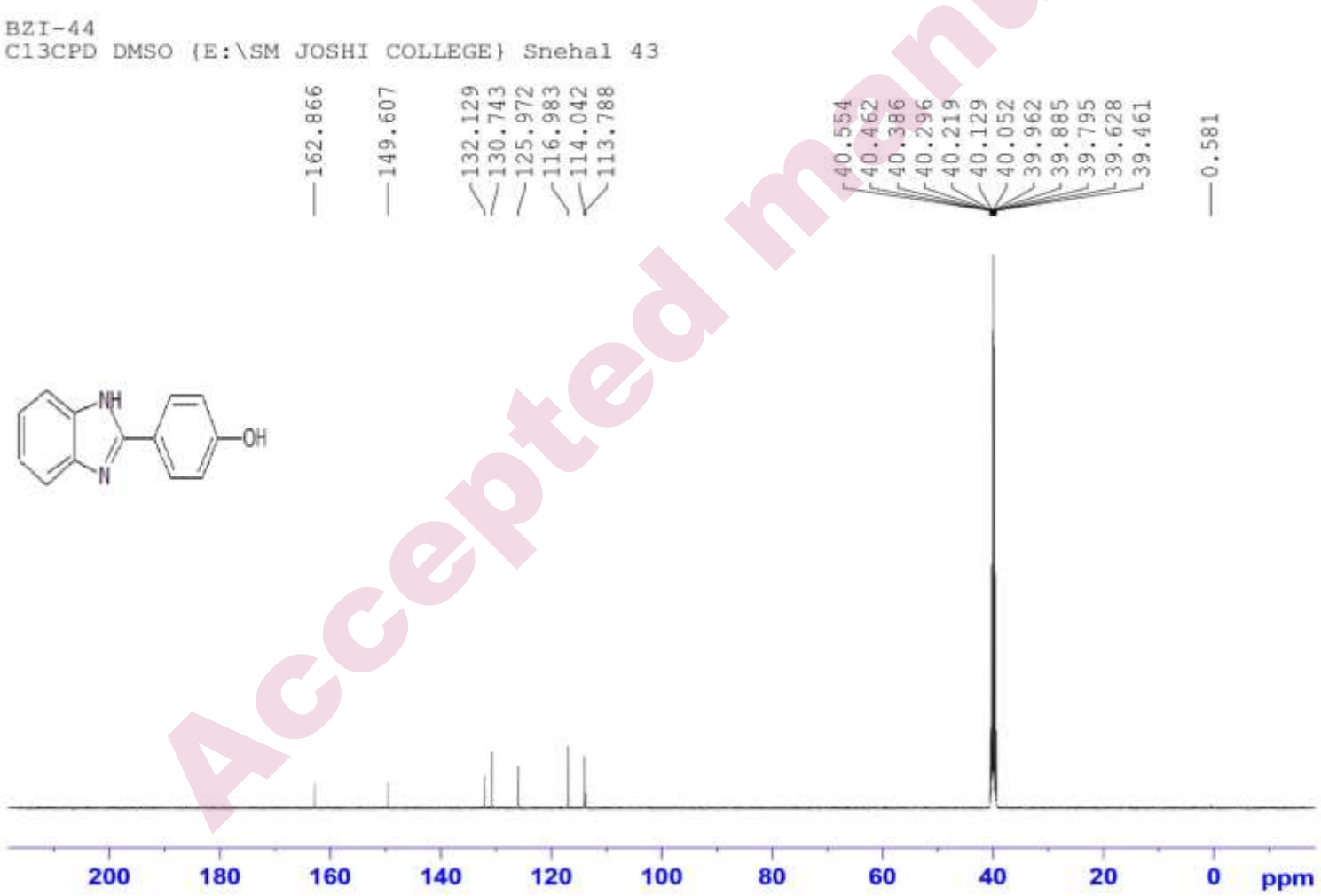
F2 - Acquisition Parameters
Date_ 20210706
Time 16.53 h
INSTRUM spect
PROBHD X119470_0152 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1299992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 4-(*1H*-benzimidazole-2-yl) phenol (Table 5, Entry 11, 4k)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 26
PROCNO 1

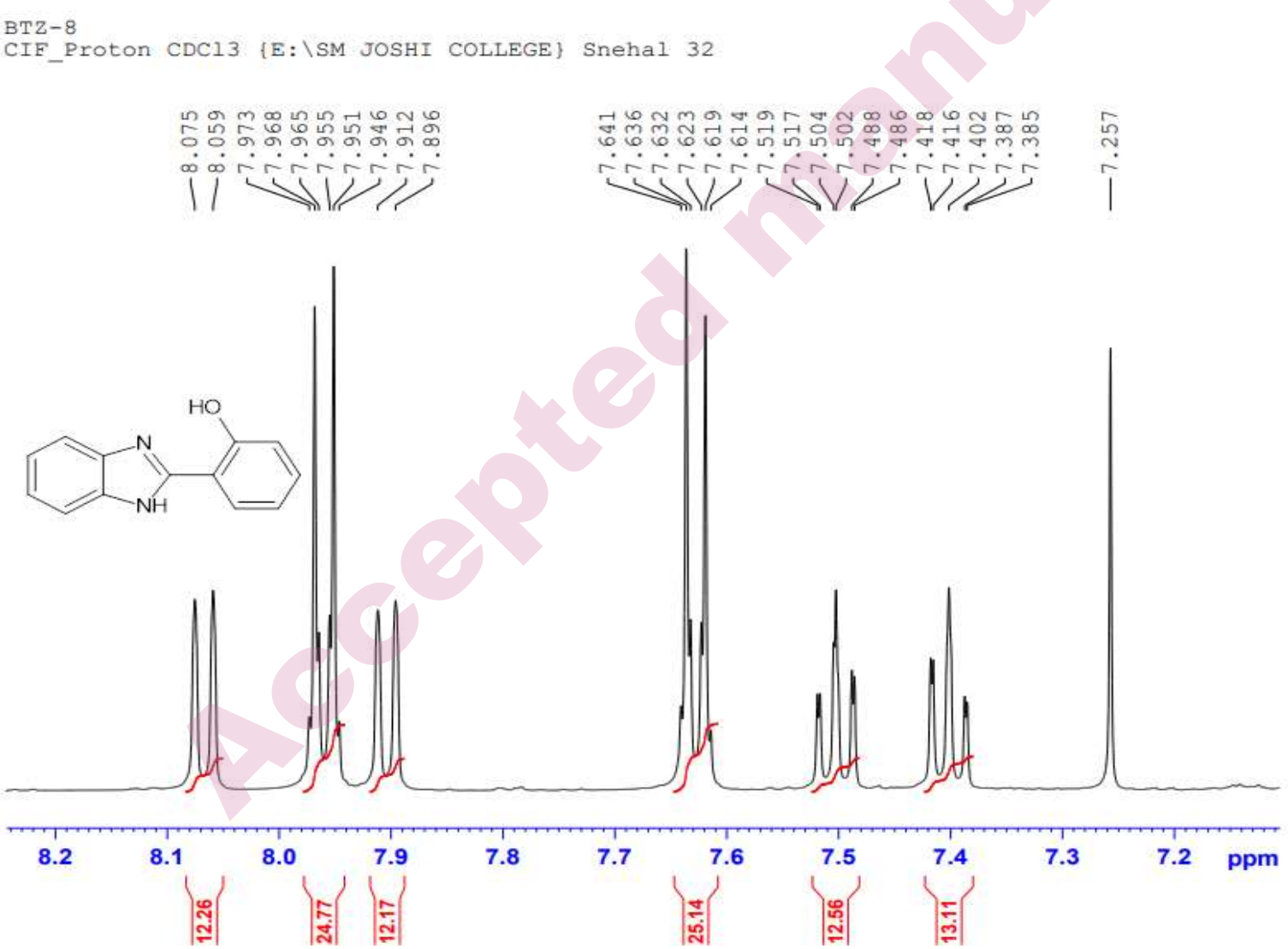
F2 - Acquisition Parameters
Date_ 20210707
Time 6.02 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 4-(*1H*-benzimidazole-2-yl) phenol (Table 5, Entry 11, 4k)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jul06-2021
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20210706
Time                       15.20 h
INSTRUM                    spect
PROBHD     Z119470_0152 (
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           32
DS                            2
SWH                   10000.000 Hz
FIDRES                0.305176 Hz
AQ                    3.2767999 sec
RG                       109.52
DW                       50.000 usec
DE                        6.50 usec
TE                       298.0 K
D1                  1.00000000 sec
TD0                           1
SFO1                500.1330883 MHz
NUC1                        1H
P1                        9.22 usec
PLW1               22.00000000 W

F2 - Processing parameters
SI                        65536
SF                  500.1300136 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
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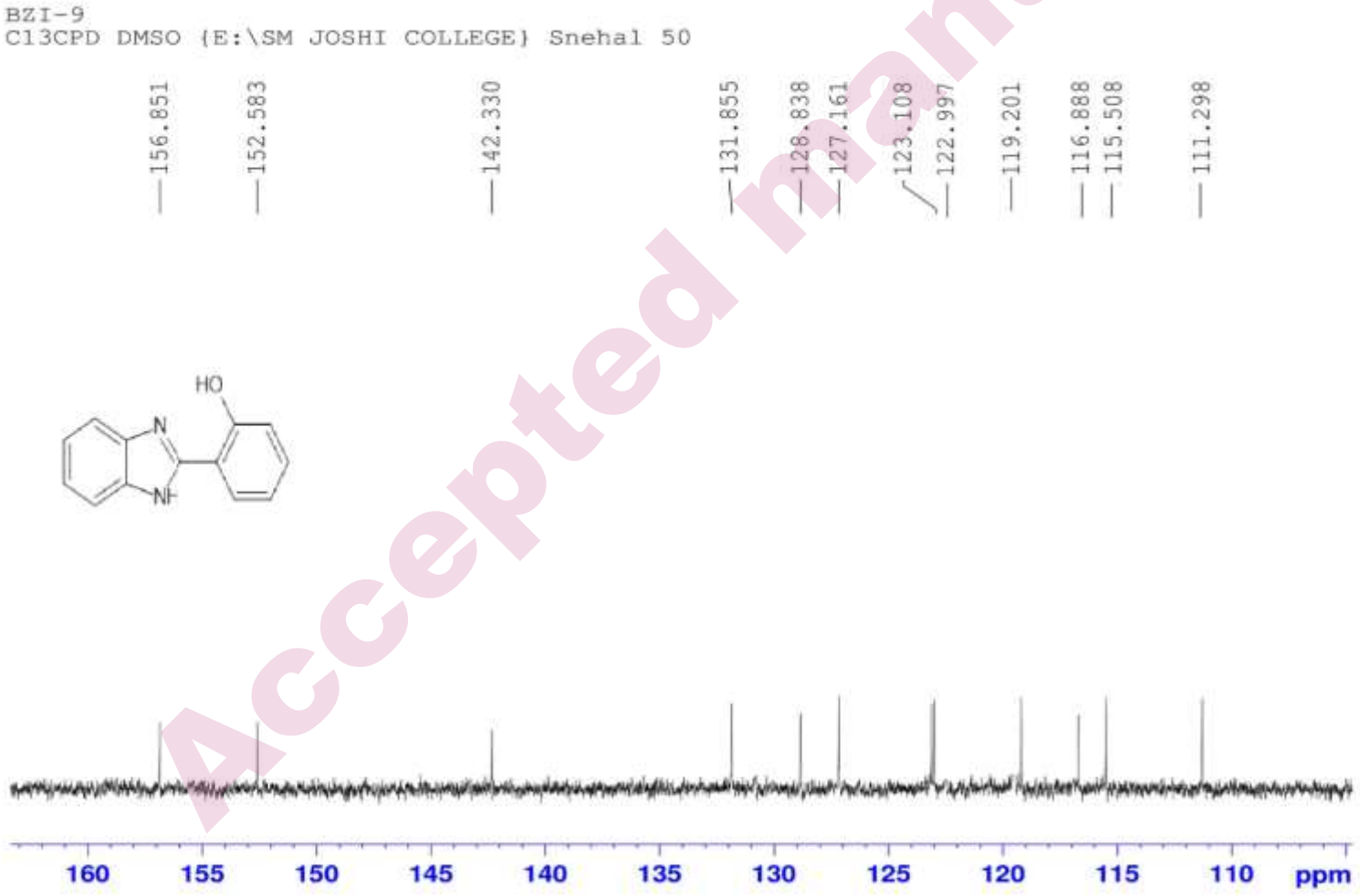
Accepted manuscript

Fig: ^{1}H-NMR 2-(*1H*-benzimidazole-2yl) phenol (Table 5, Entry 12, 4l)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Apr07-2021
EXPNO                         10
PROCNO                         1

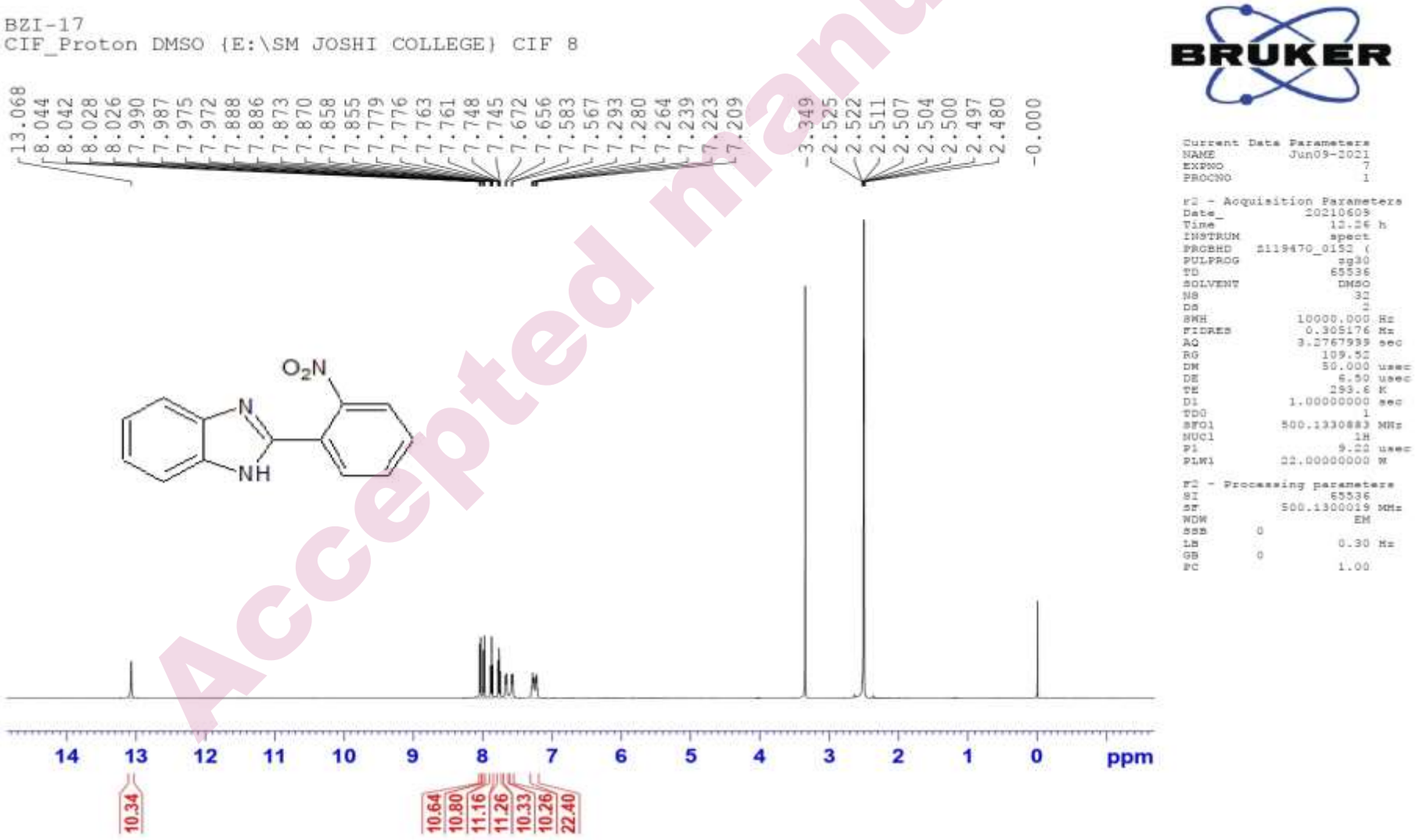
F2 - Acquisition Parameters
Date_               20210408
Time                      1.28 h
INSTRUM                   spect
PROBHD     5119470_0152 (
PULPROG                 zgpg30
TD                       65536
SOLVENT                  DMSO
NS                       2048
DS                          4
SWH                 29761.904 Hz
FIDRES                0.908261 Hz
AQ                    1.1010048 sec
RG                       189.76
DW                       16.800 usec
DE                        5.50 usec
TE                       295.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1
SFO1                125.7703643 MHz
NUC1                        13C
P1                         9.25 usec
PLW1                100.00000000 W
SFO2                500.1320005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2                 22.00000000 W
PLW12                 0.29222000 W
PLW13                 0.16698000 W

F2 - Processing parameters
SI                        32768
SF                  125.7577885 MHz
WDW                            EM
SSB                 0
LB                            1.00 Hz
GB                  0
PC                            1.40
```

Accepted manuscript

Fig: ^{13}C-NMR 2-(*1H*-benzimidazole-2yl) phenol (Table 5, Entry 12, 4l)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jun09-2021
EXPNO                          7
PROCNO                         1

r2 - Acquisition Parameters
Date_                   20210609
Time                       12.26 h
INSTRUM                    spect
PROBHD    5119470_0152 (
PULPROG                    zg30
TD                        65536
SOLVENT                    DMSO
NS                           32
DS                            2
SWH                   10000.000 Hz
FIDRES                    0.305176 Hz
AQ                        3.2767999 sec
RG                          109.52
DW                          50.000 usec
DE                           6.50 usec
TE                          293.6 K
D1                    1.00000000 sec
TD0                            1
SFO1                500.1330883 MHz
NUC1                         1H
P1                           9.20 usec
PLW1                 22.00000000 W

F2 - Processing parameters
SI                        65536
SF                  500.1300019 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
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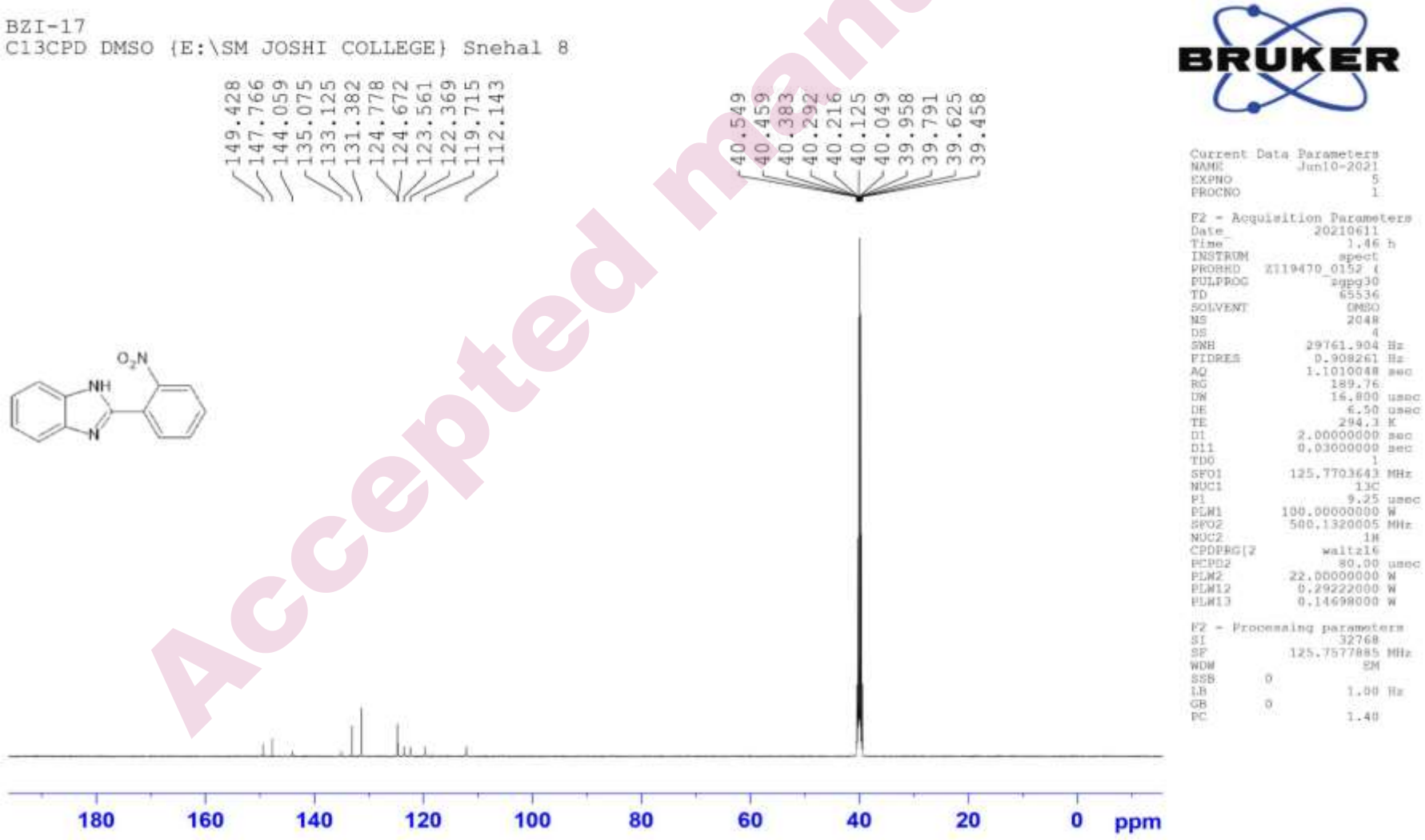
Accepted manuscript

Fig: ^{1}H-NMR 2-(2-nitrophenyl)-*1H*-benzimidazole (Table 5, Entry 15, 4o)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jun10-2021
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210611
Time 1.46 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(2-nitrophenyl)-*1H*-benzimidazole (Table 5, Entry 15, 4o)

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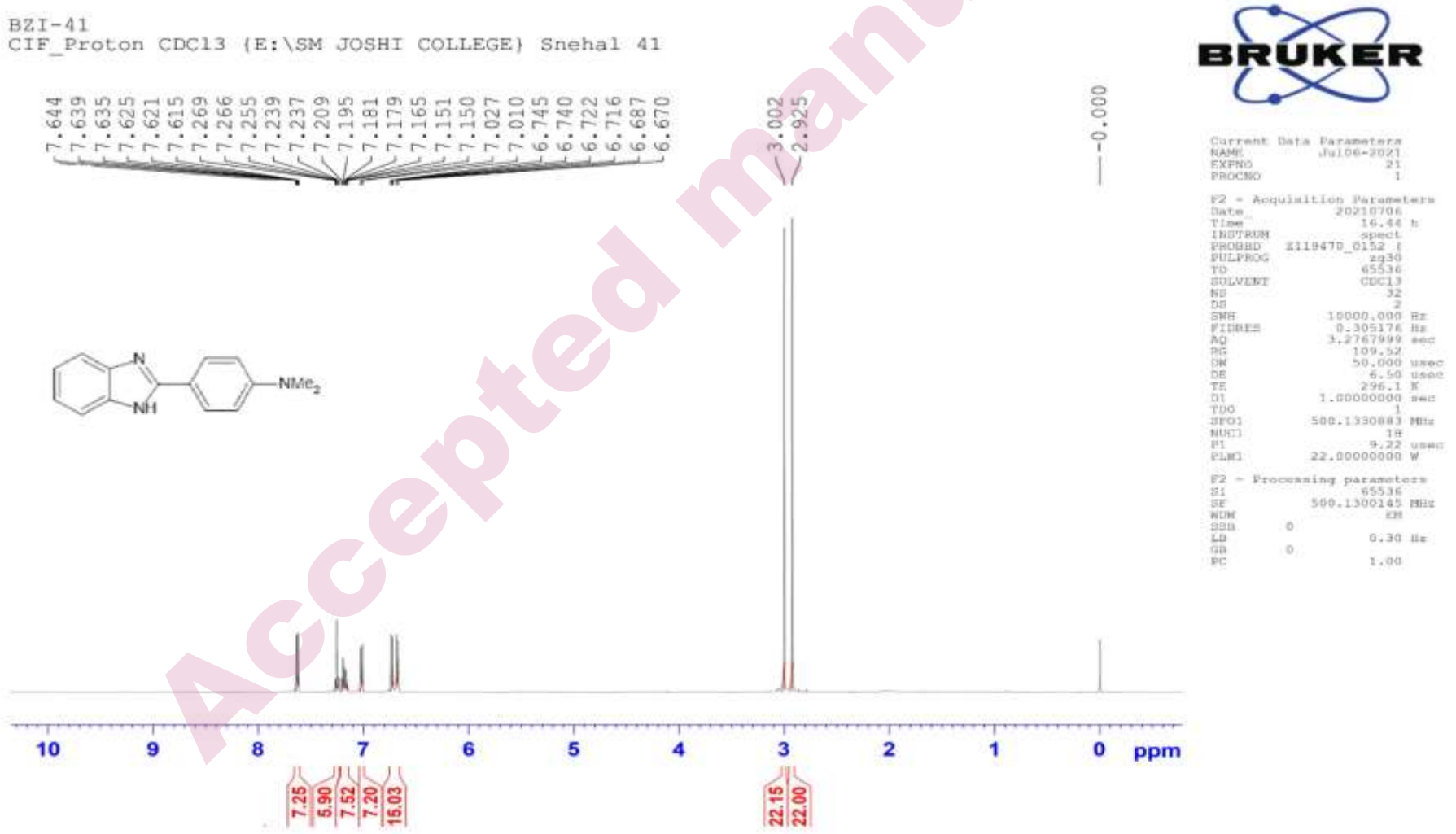

BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
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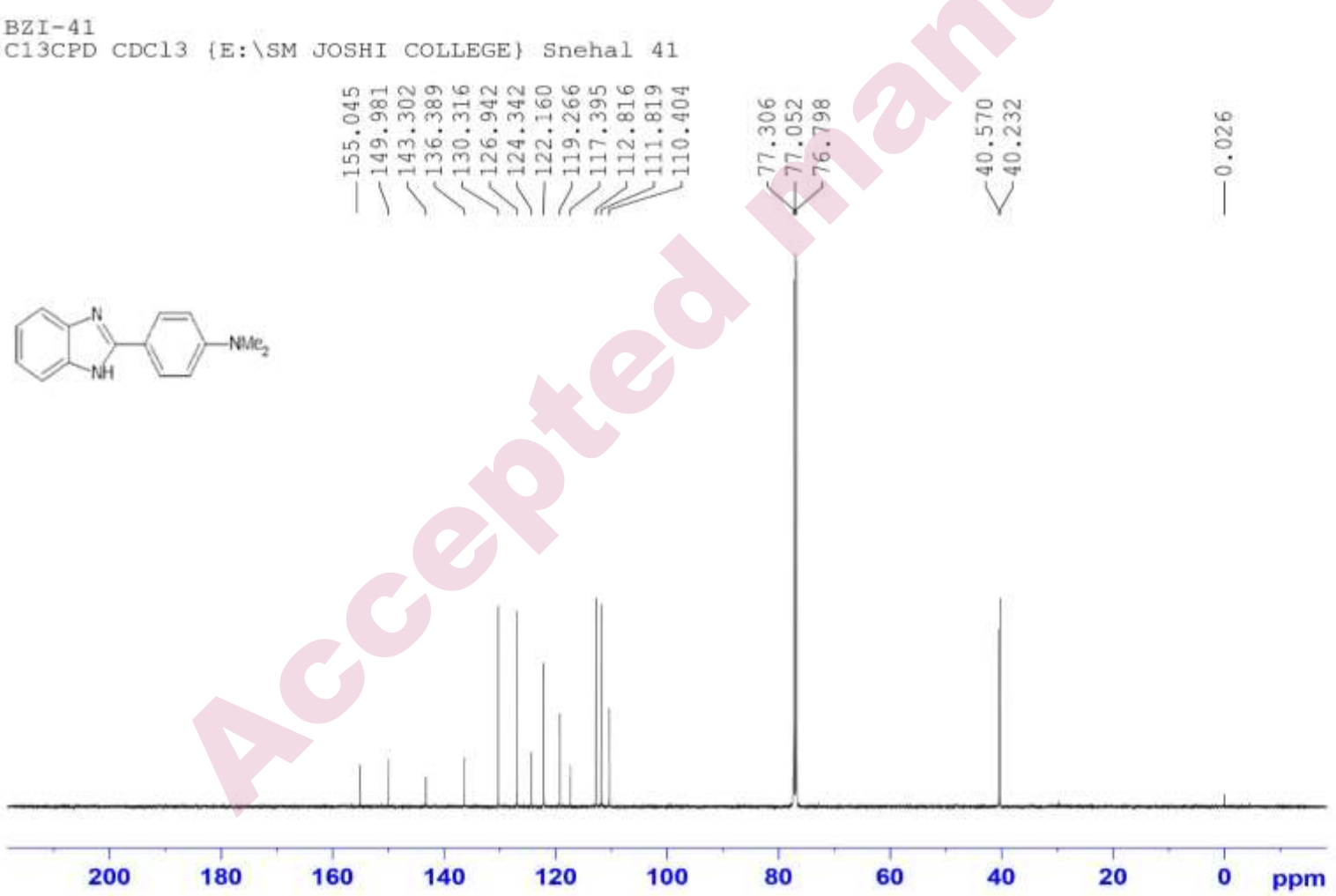
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300145 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 4-(*1H*-benzimidazole-2-yl)-N, N-dimethylaniline (Table 5, Entry 16,4p)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210707
Time 4.09 h
INSTRUM spect
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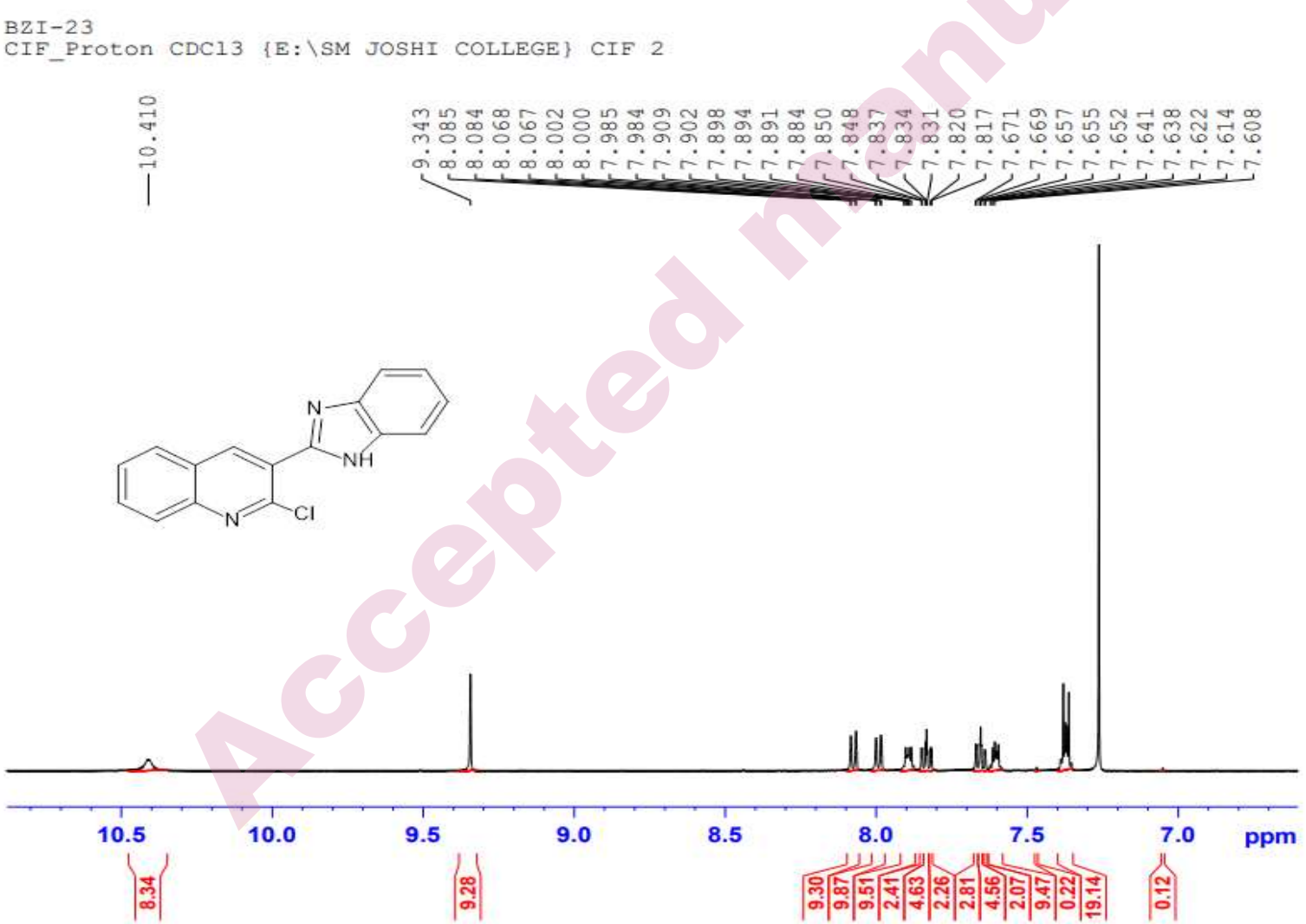
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 4-(*1H*-benzimidazole-2-yl)-N, N-dimethylaniline (Table 5, Entry 16,4p)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jun09-2021
EXPNO                          1
PROCNO                         1

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Time                       11.57 h
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PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            32
DS                             2
SWH                    10000.000 Hz
FIDRES                     0.305176 Hz
AQ                     3.2767999 sec
RG                        189.76
DW                         50.000 usec
DE                          6.50 usec
TE                         293.3 K
D1                    1.00000000 sec
TD0                            1
SFO1                 500.1330883 MHz
NUC1                          1H
P1                          9.22 usec
PLW1                22.00000000 W

F2 - Processing parameters
SI                         65536
SF                   500.1300108 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
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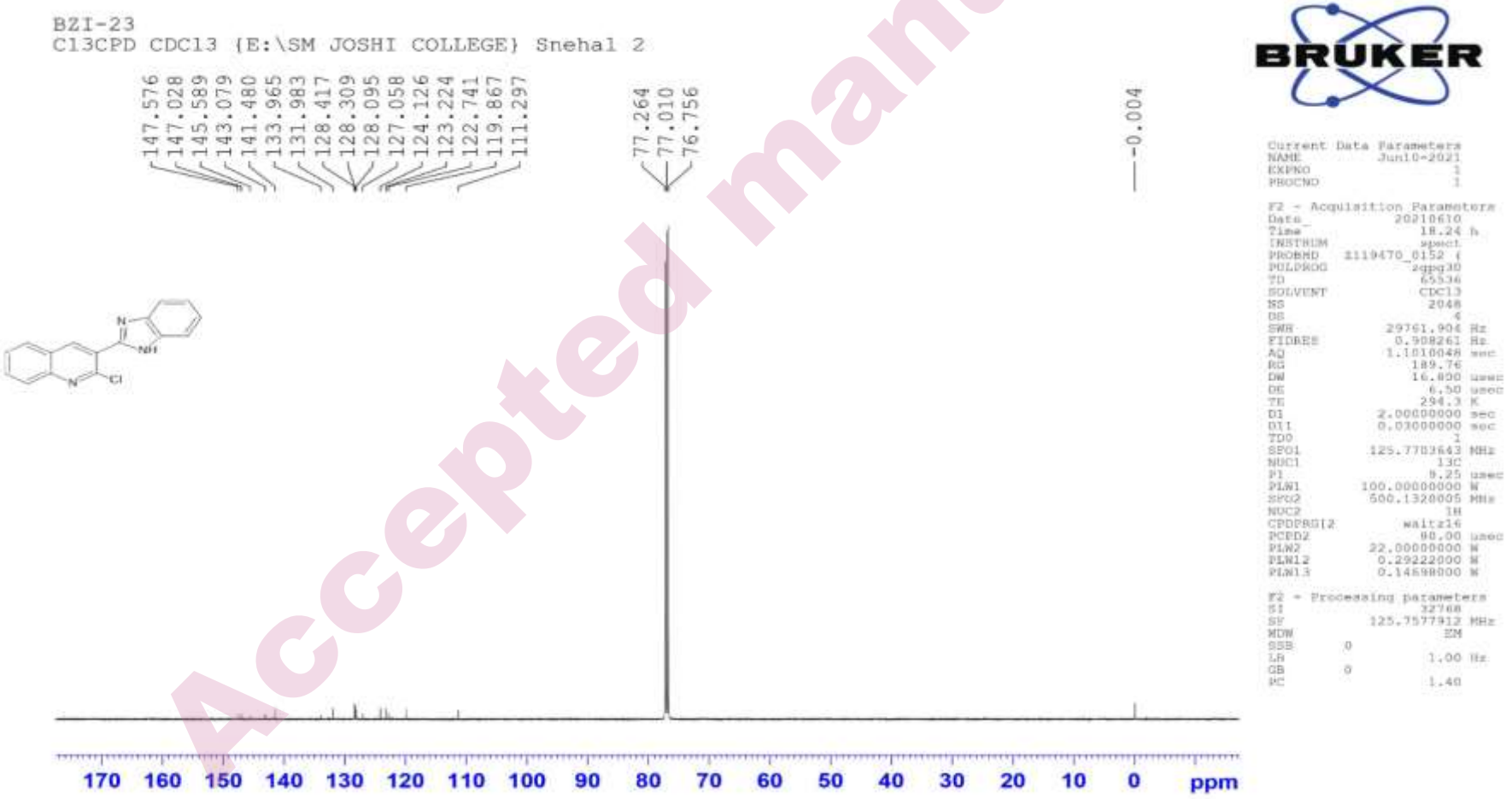
Accepted manuscript

Fig: ^{1}H-NMR 3-(*1H*-benzimidazole-2-yl)2-chloroquinoline (Table 5, Entry 19, 4s)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jun10-2021
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_               20210610
Time                   18.24 h
INSTRUM                spect
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PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       2048
DS                          4
SWH                 29761.904 Hz
FIDRES              0.908261 Hz
AQ                  1.1010048 sec
RG                     189.76
DW                     16.800 usec
DE                      6.50 usec
TE                     294.3 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1
SFO1             125.7703643 MHz
NUC1                      13C
P1                       9.25 usec
PLW1            100.00000000 W
SFO2             500.1320005 MHz
NUC2                      1H
CPDPRG[2                waltz16
PCPD2                   80.00 usec
PLW2             22.00000000 W
PLW12             0.29222000 W
PLW13             0.14698000 W

F2 - Processing parameters
SI                       32768
SF               125.7577912 MHz
WDW                         EM
SSB                  0
LB                      1.00 Hz
GB                   0
PC                      1.40
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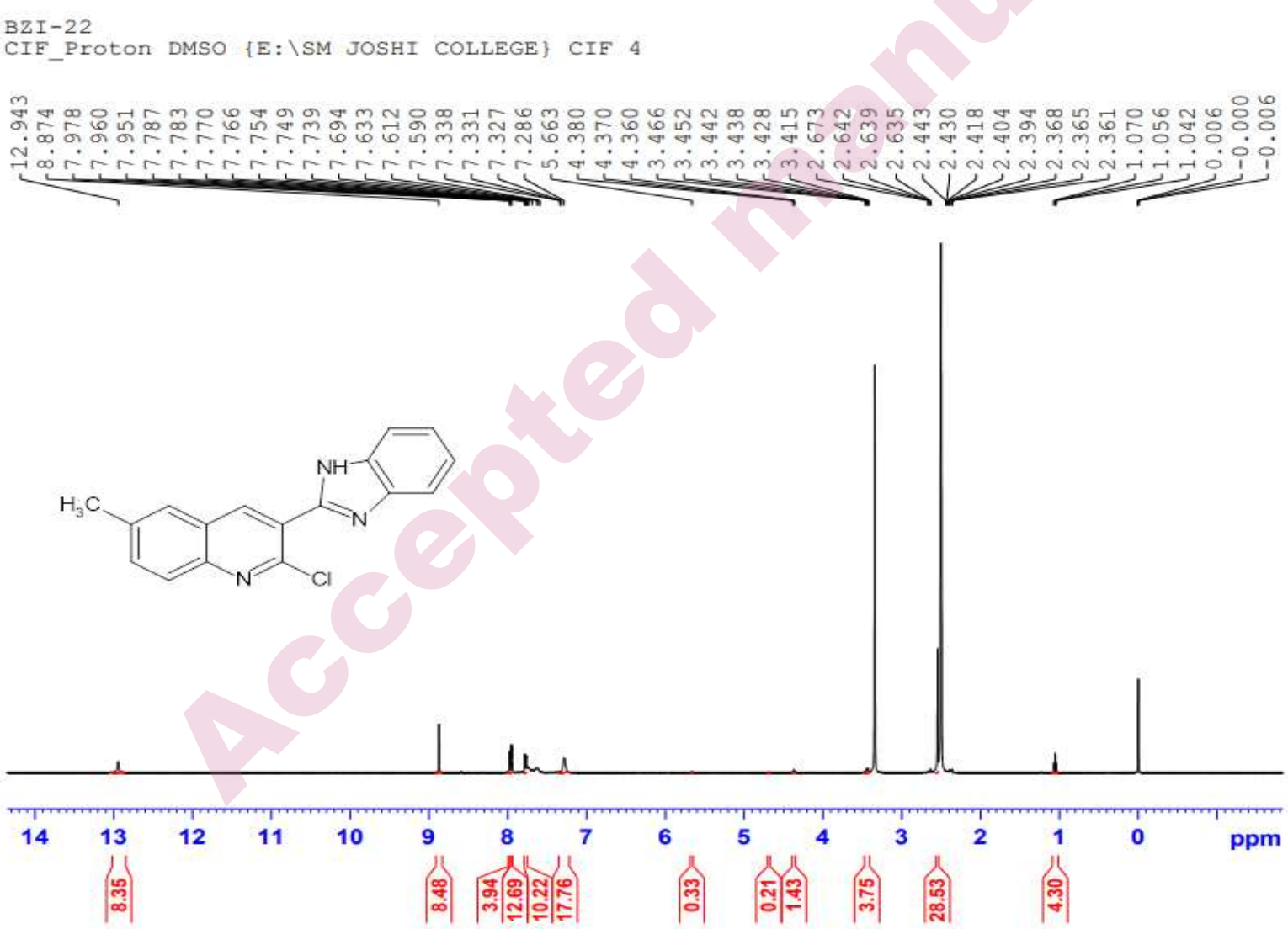
Accepted manuscript

Fig: ^{13}C-NMR 3-(*1H*-benzimidazole-2-yl)2-chloroquinoline (Table 5, Entry 19, 4s)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jun09-2021
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
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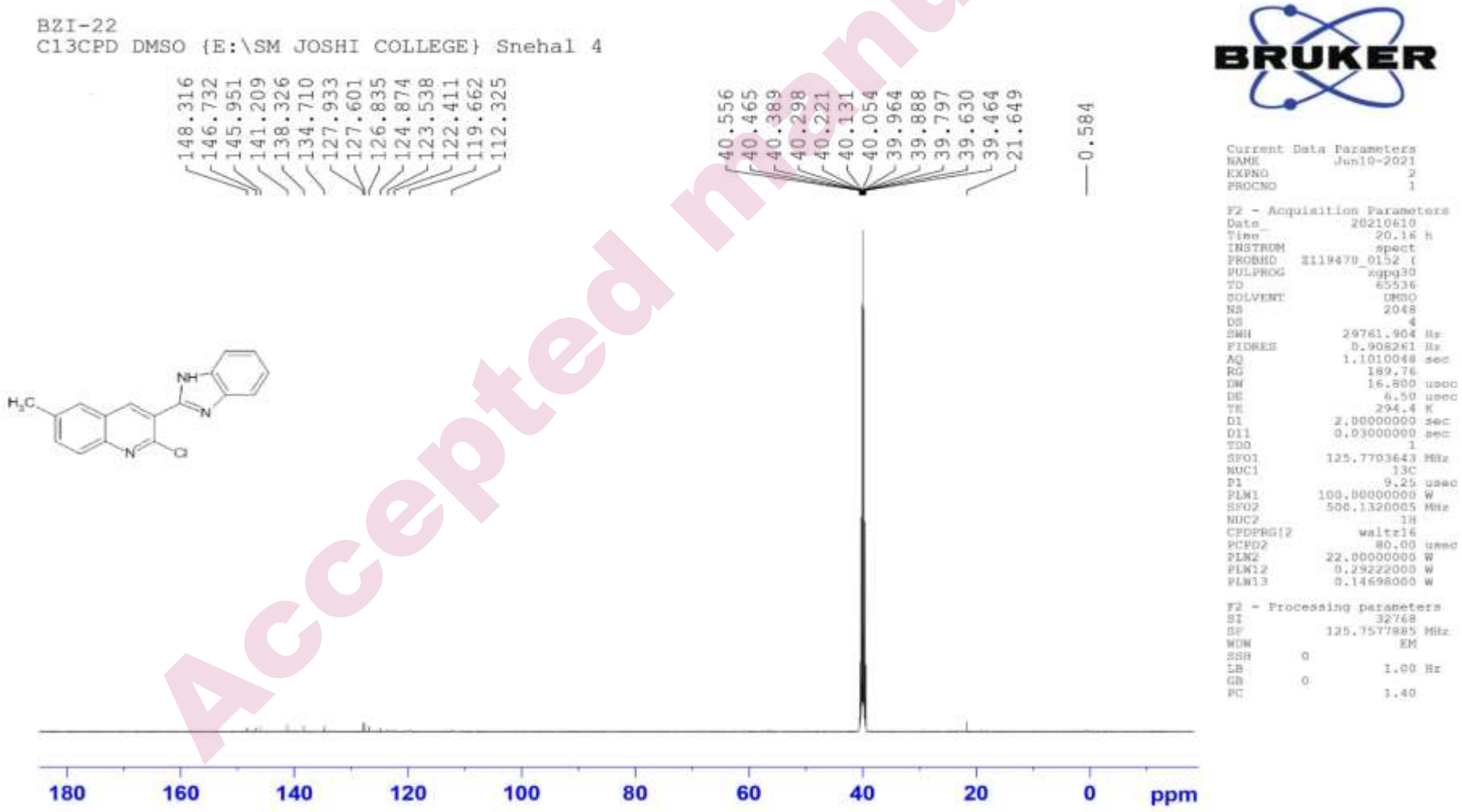
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300019 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 3-(*1H*-benzimidazol-2-yl)-2-chloro-6-methylquinoline (Table 5, Entry 20, 4t)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jun10-2021
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210610
Time 20.16 h
INSTRUM spect
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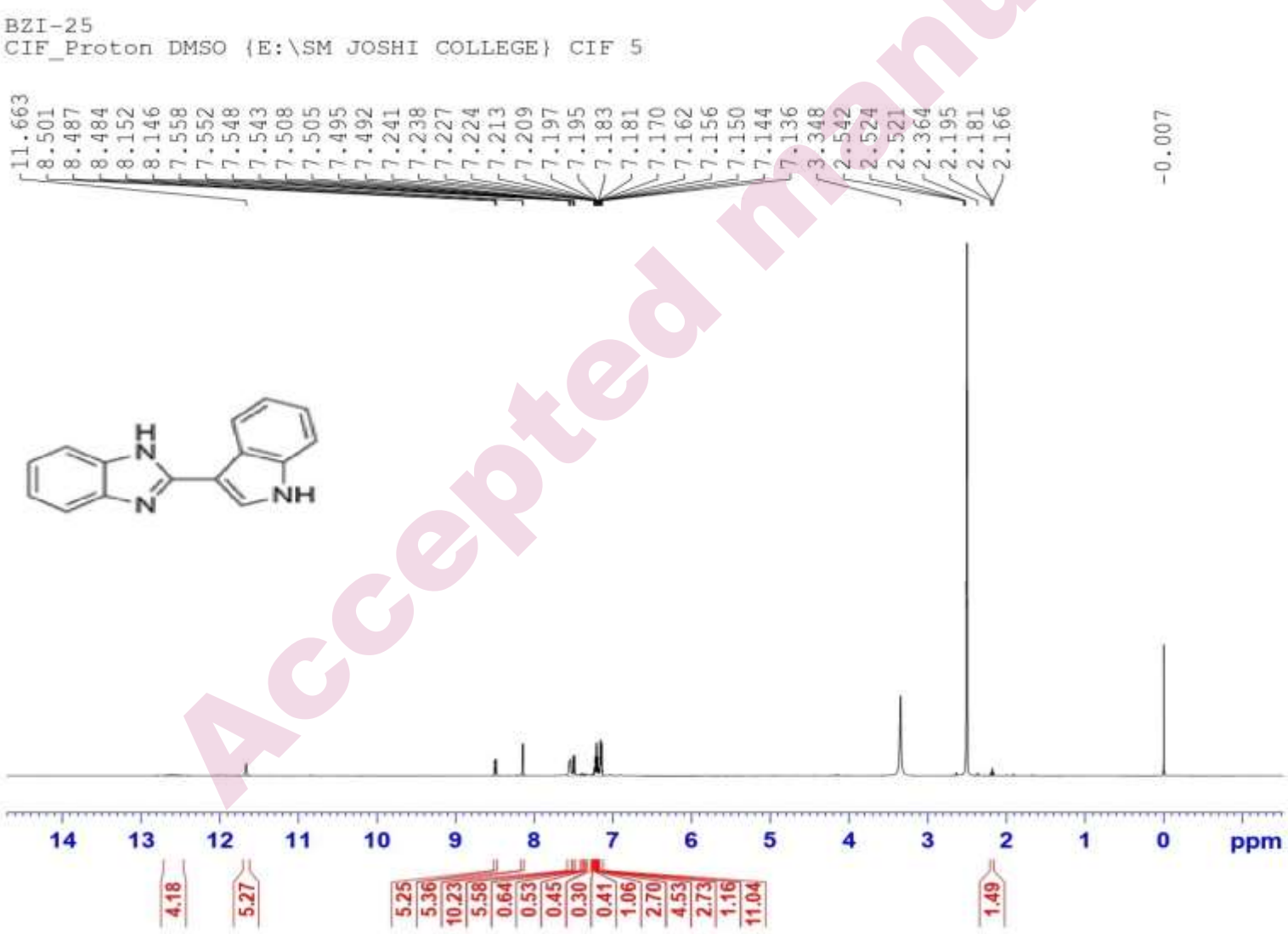
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 3-(*1H*-benzimidazol-2-yl)-2-chloro-6-methylquinoline (Table 5, Entry 20, 4t)

Accepted manuscript



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EXPNO                          4
PROCNO                         1

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PULPROG                   zg30
TD                       65536
SOLVENT                   DMSO
NS                          32
DS                           2
SWH                 10000.000 Hz
FIDRES                0.305176 Hz
AQ                    3.2767999 sec
RG                       109.52
DW                       50.000 usec
DE                         6.50 usec
TE                       293.4 K
D1                  1.00000000 sec
TD0                          1
SFO1                500.1330883 MHz
NUC1                        1H
P1                         9.22 usec
PLW1               22.00000000 W

F2 - Processing parameters
SI                       65536
SF                  500.1300024 MHz
WDW                         EM
SSB                  0
LB                         0.30 Hz
GB                   0
PC                         1.00
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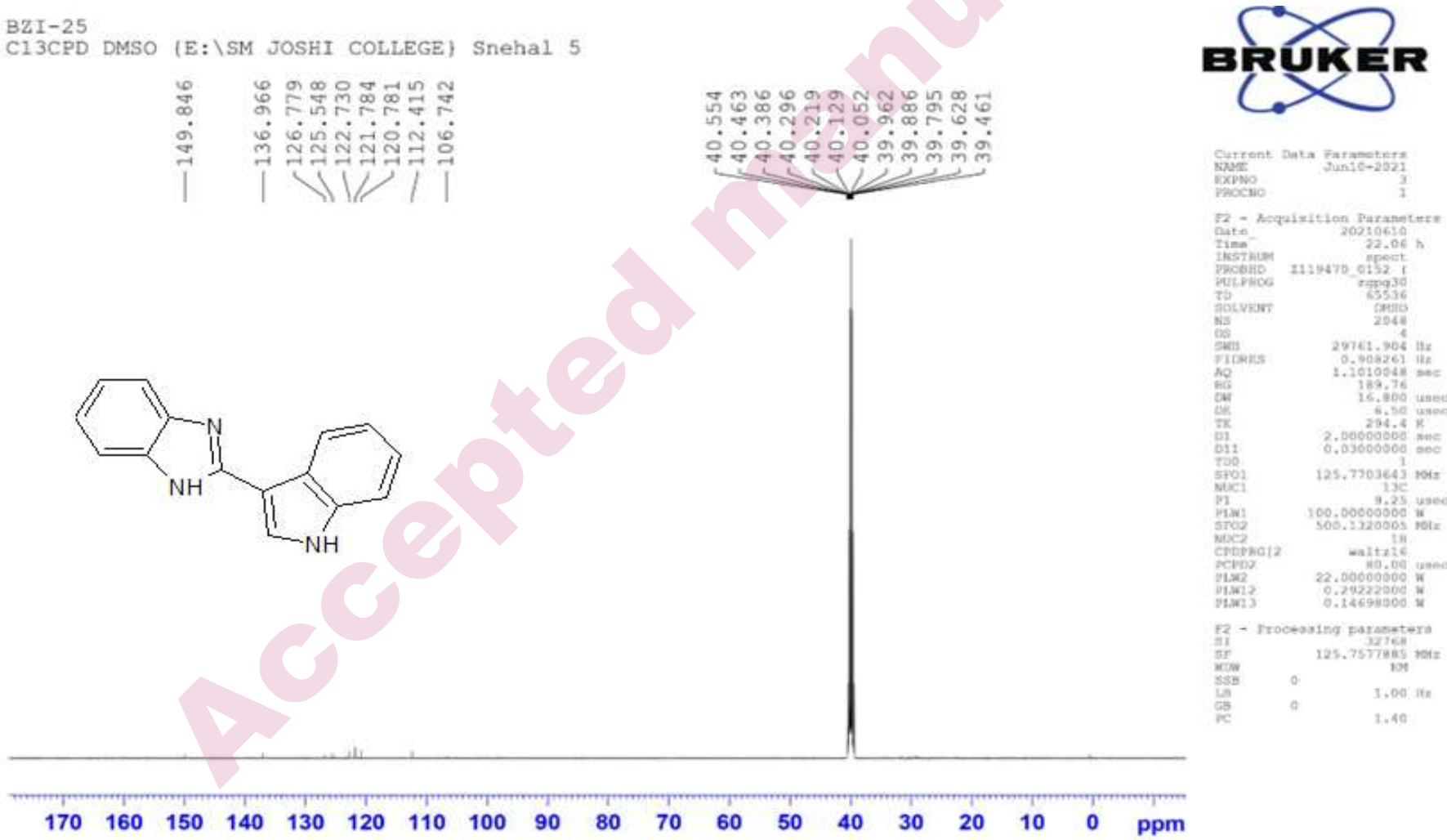
Fig: ^{1}H-NMR 2-(1*H*-indol-3-yl)-*1H*-benzimidazole (Table 5, Entry 21, 4u)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jun10-2021
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date 20210610
Time 22.06 h
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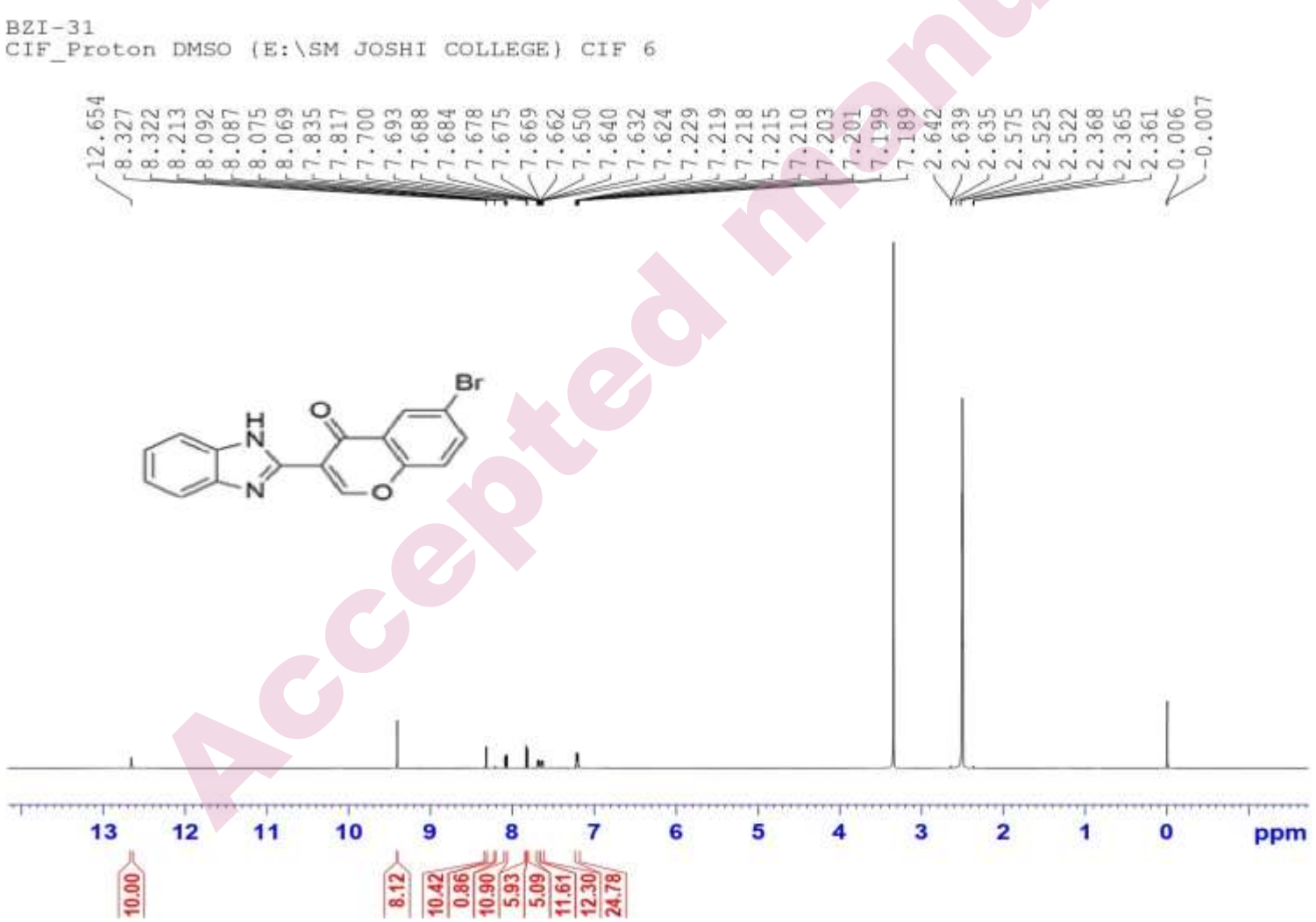
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(1*H*-indol-3-yl)-*1H*-benzimidazole (Table 5, Entry 21, 4u)

Accepted manuscript



BRUKER

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EXPNO                          5
PROCNO                         1

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Time                    12.16 h
INSTRUM                  spect
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PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                         32
DS                          2
SWH                10000.000 Hz
FIDRES                 0.305176 Hz
AQ                 3.2767359 sec
RG                       109.52
DW                     50.000 usec
DE                       6.50 usec
TE                      293.5 K
D1                 1.00000000 sec
TD0                         1
SFO1             500.1330883 MHz
NUC1                       1H
P1                       9.22 usec
PLW1              22.00000000 W

F2 - Processing parameters
SI                      65536
SF               500.1300019 MHz
WDW                        EM
SSB                 0
LB                       0.30 Hz
GB                  0
PC                       1.00
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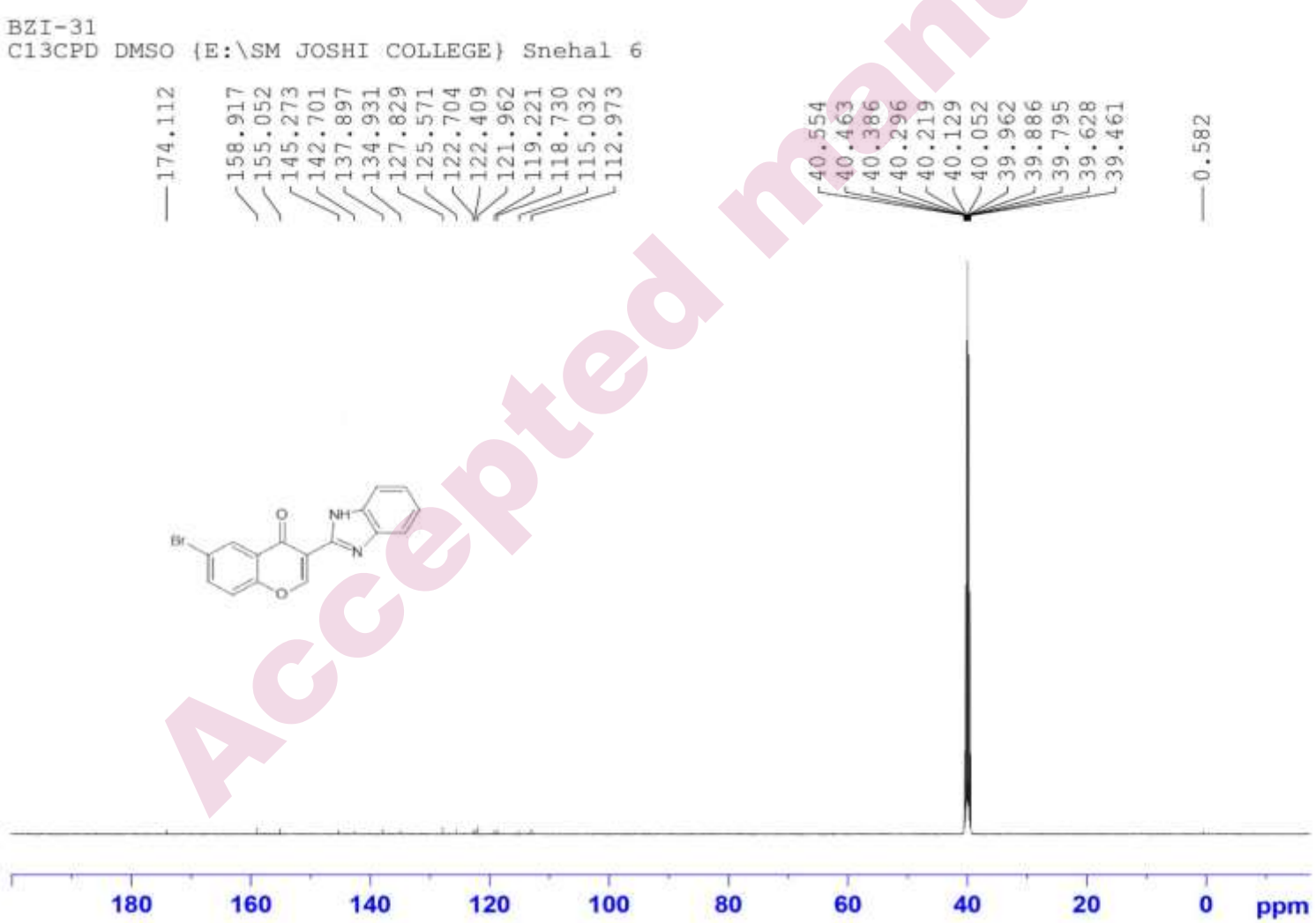
Fig: ^{1}H-NMR 3-(*1H*-benzimidazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one (Table 5, Entry 22, 4v)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jun10-2021
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210610
Time 23.56 h
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 3-(*1H*-benzimidazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one (Table 5, Entry 22, 4v)

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Fig: 3-(1*H*-benzimidazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one (Table 5, Entry 22, 4v)

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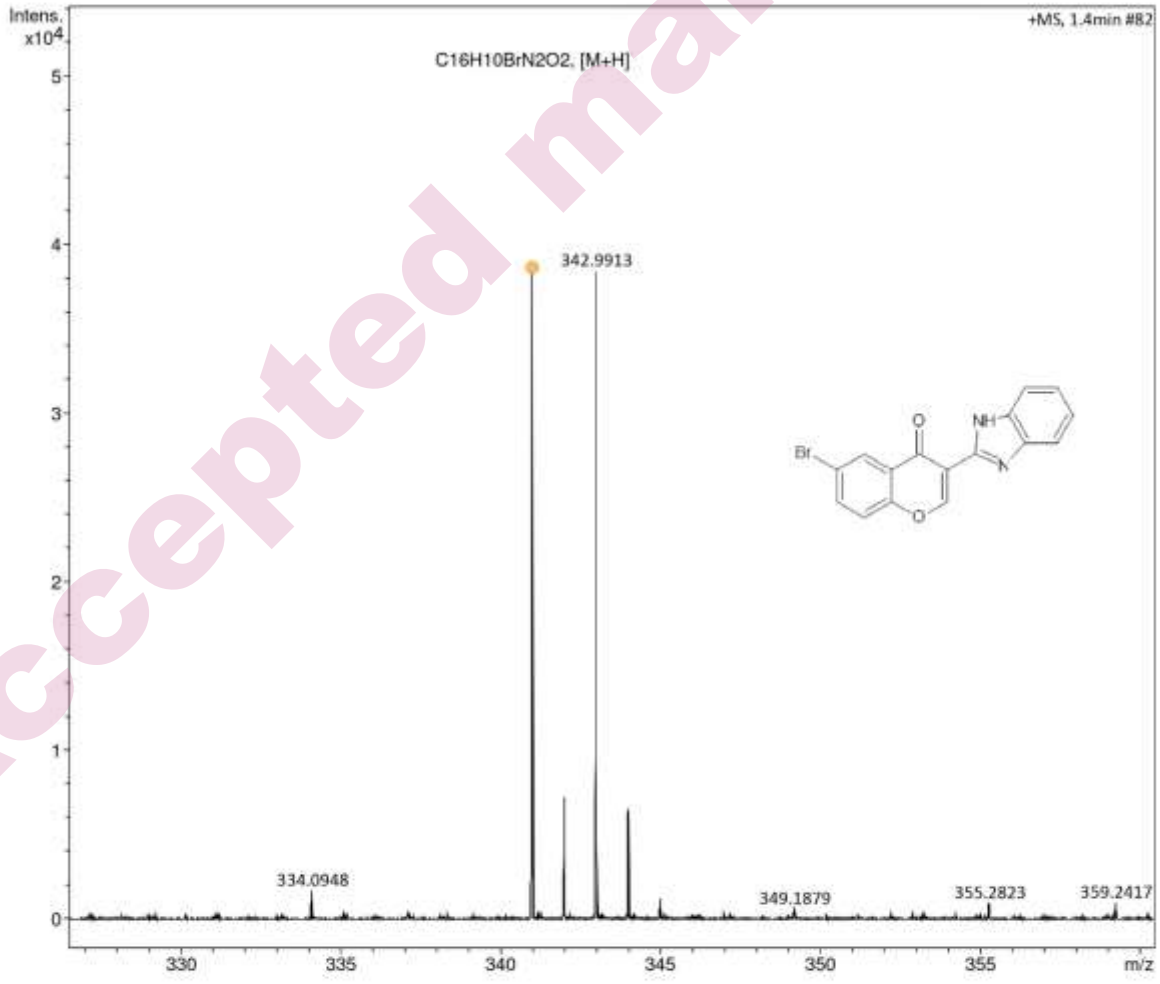
Analysis Info

Analysis Name	D:\Data\2022\JAN\SPPU COLLEGE\BABURAO GHOLAP COLLEGE, SANGVI\RAMESH GAWADE\BZT-31_GB1_01_3794.d	Acquisition Date	1/21/2022 2:32:50 PM
Method	dlc_ms50-1200mz_2500v_12min_0.120ml flow_95b.m	Operator	CIF
Sample Name	BZT-31	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	2500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule	Adduct
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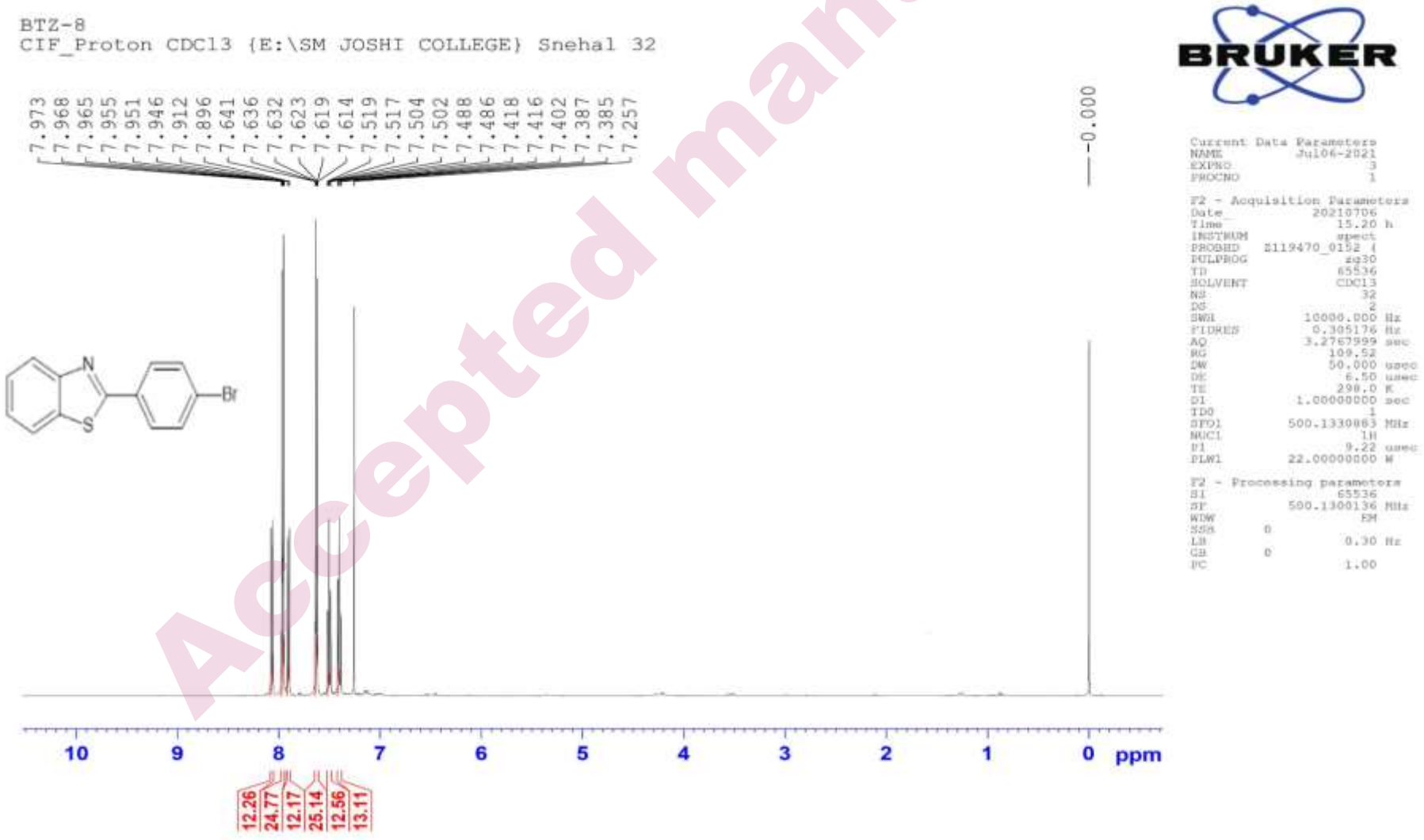
	1	C16H10BrN2O2	100.00	340.992016	-0.7	-2.1	8.3	12.5	even	ok	M+H



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
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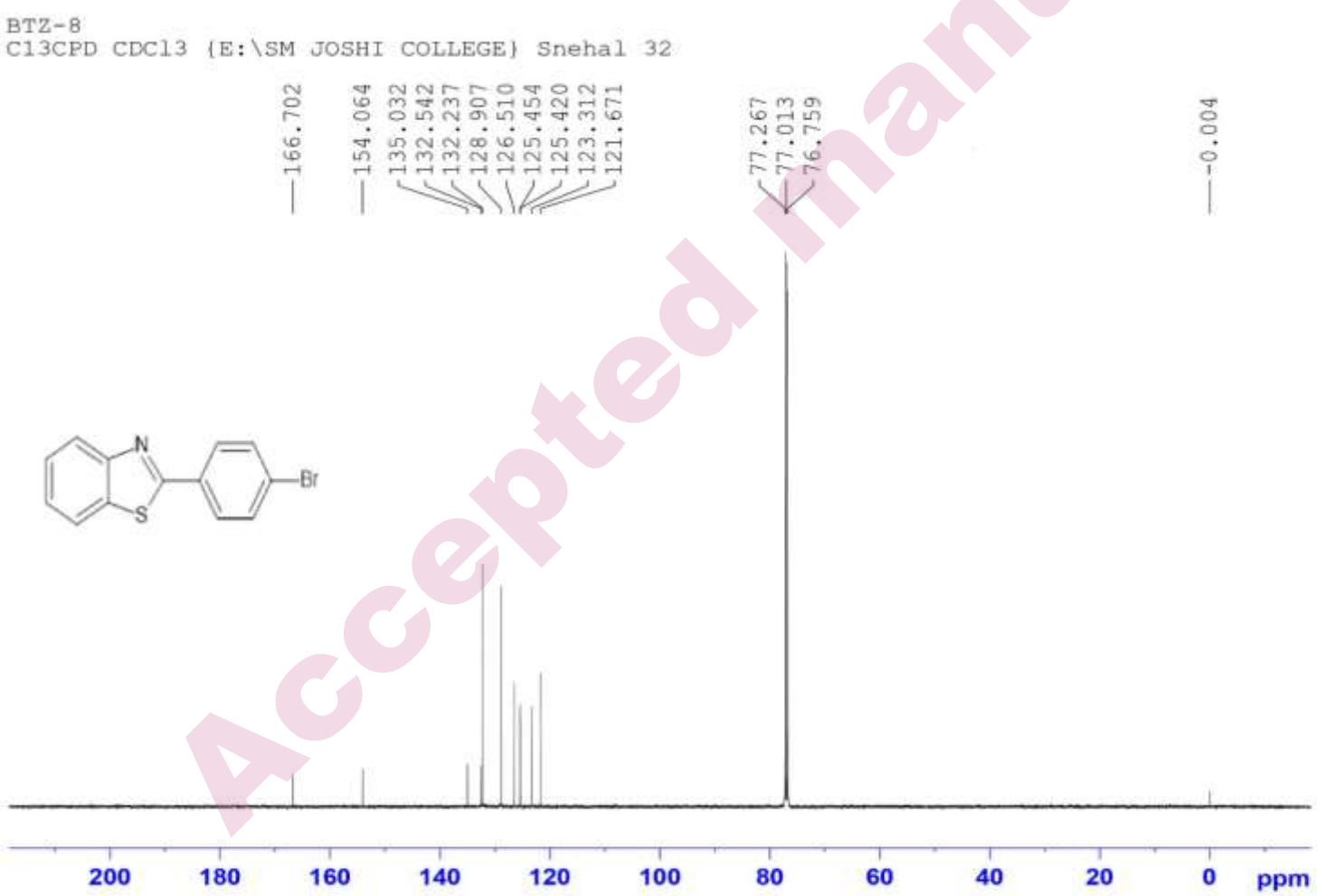
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300136 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 2-(4-bromophenyl)-1,3-benzothiazole (Table 6, Entry 4, 7d)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Ju106-2021
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date 20210706
Time 19.35 h
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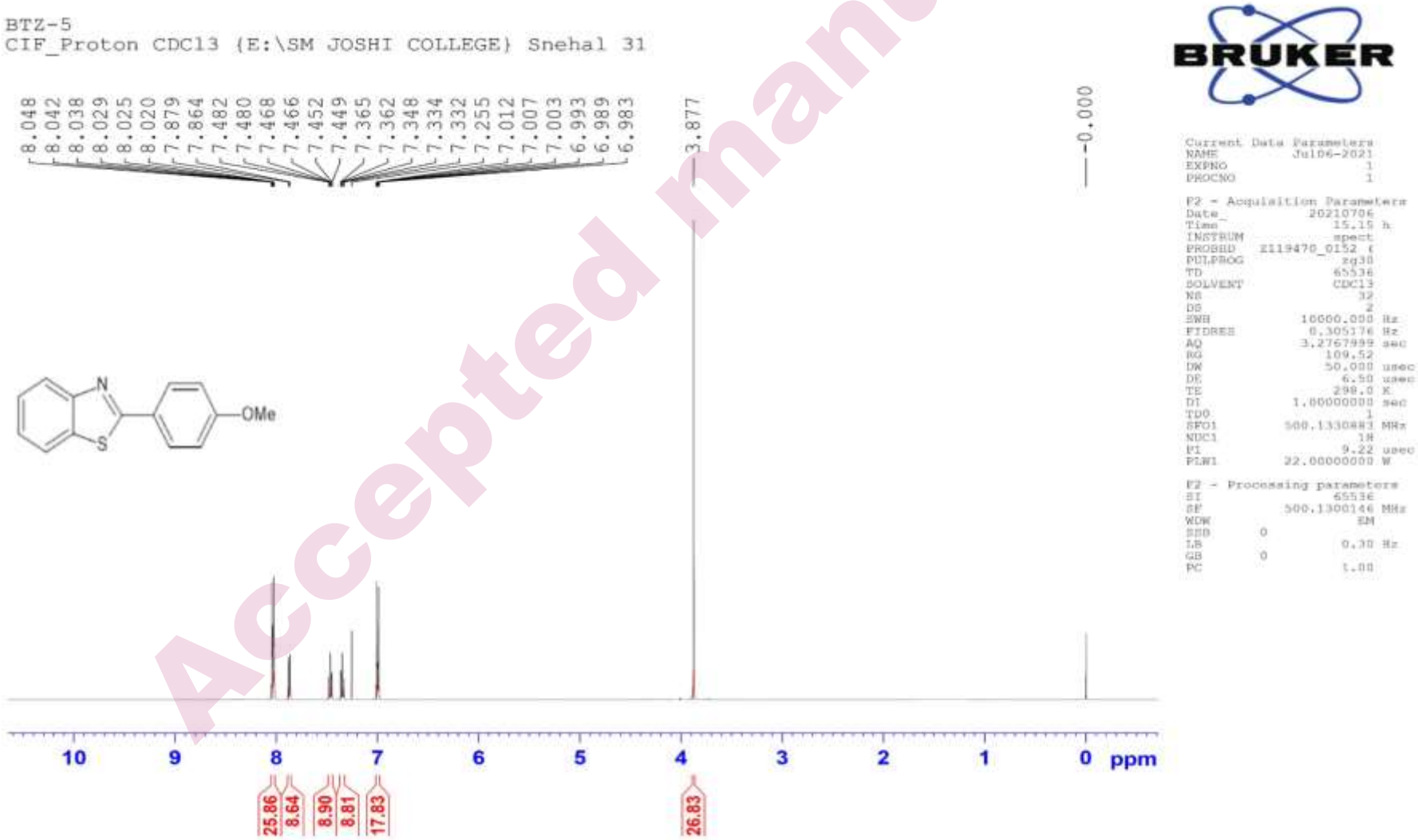
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577912 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(4-bromophenyl)-1,3-benzothiazole (Table 6, Entry 4, 7d)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 15.15 h
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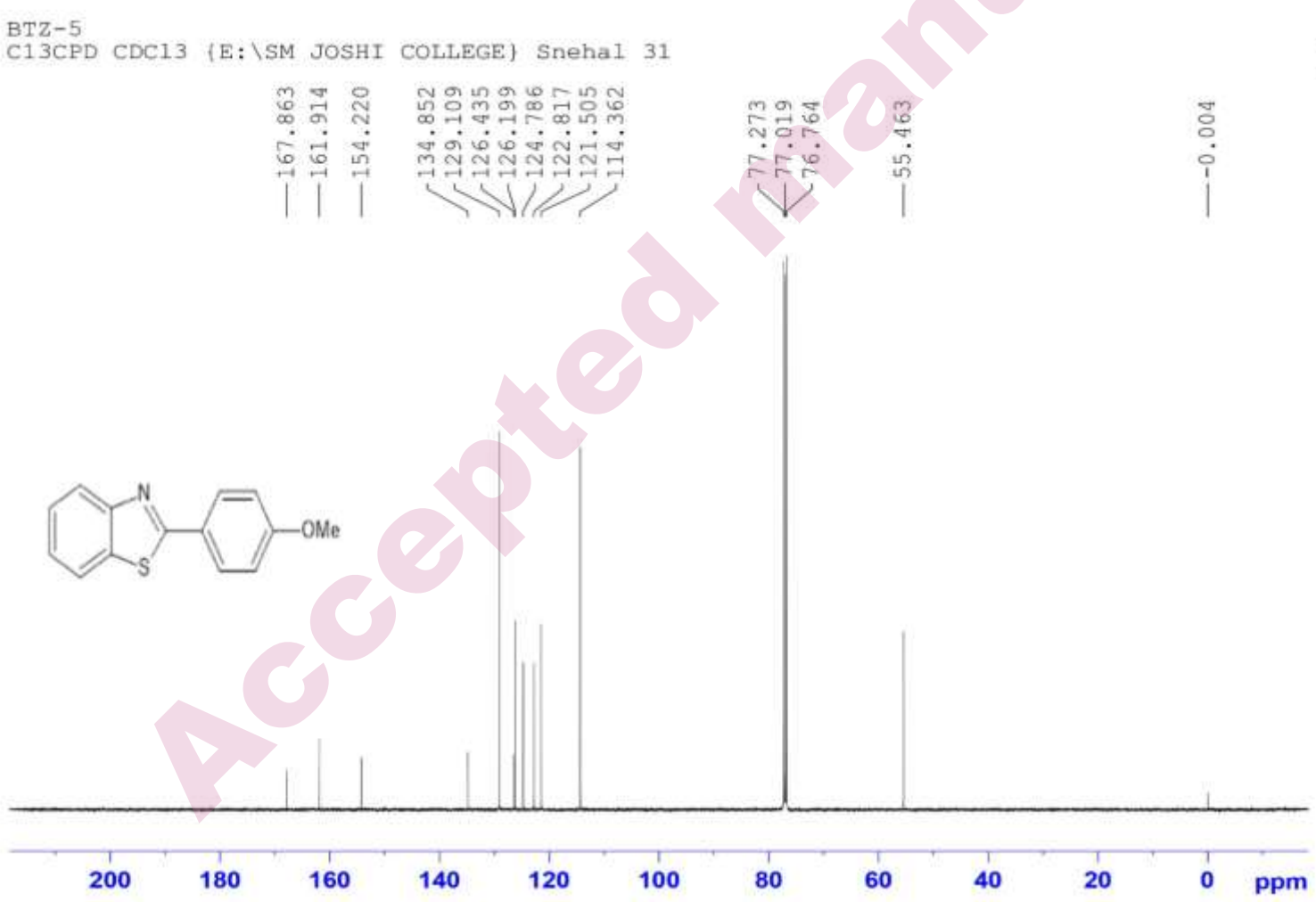
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330483 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 2-(4-methoxyphenyl)-1,3-benzothiazole (Table 6, Entry 5, 7e)

Accepted manuscript



BRUKER

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EXPNO                          2
PROCNO                         1

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PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 29761.904 Hz
FIDRES              0.909261 Hz
AQ                  1.1010048 sec
RG                     189.76
DW                     16.800 usec
DE                      6.50 usec
TE                     296.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1
SFO1               125.7703643 MHz
NUC1                       13C
P1                        9.25 usec
PLW1               100.00000000 W
SFO2               500.1320005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                22.00000000 W
PLW12               0.29222000 W
PLW13               0.14698000 W

F2 - Processing parameters
SI                         32768
SF                 125.7577912 MHz
WDW                           EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
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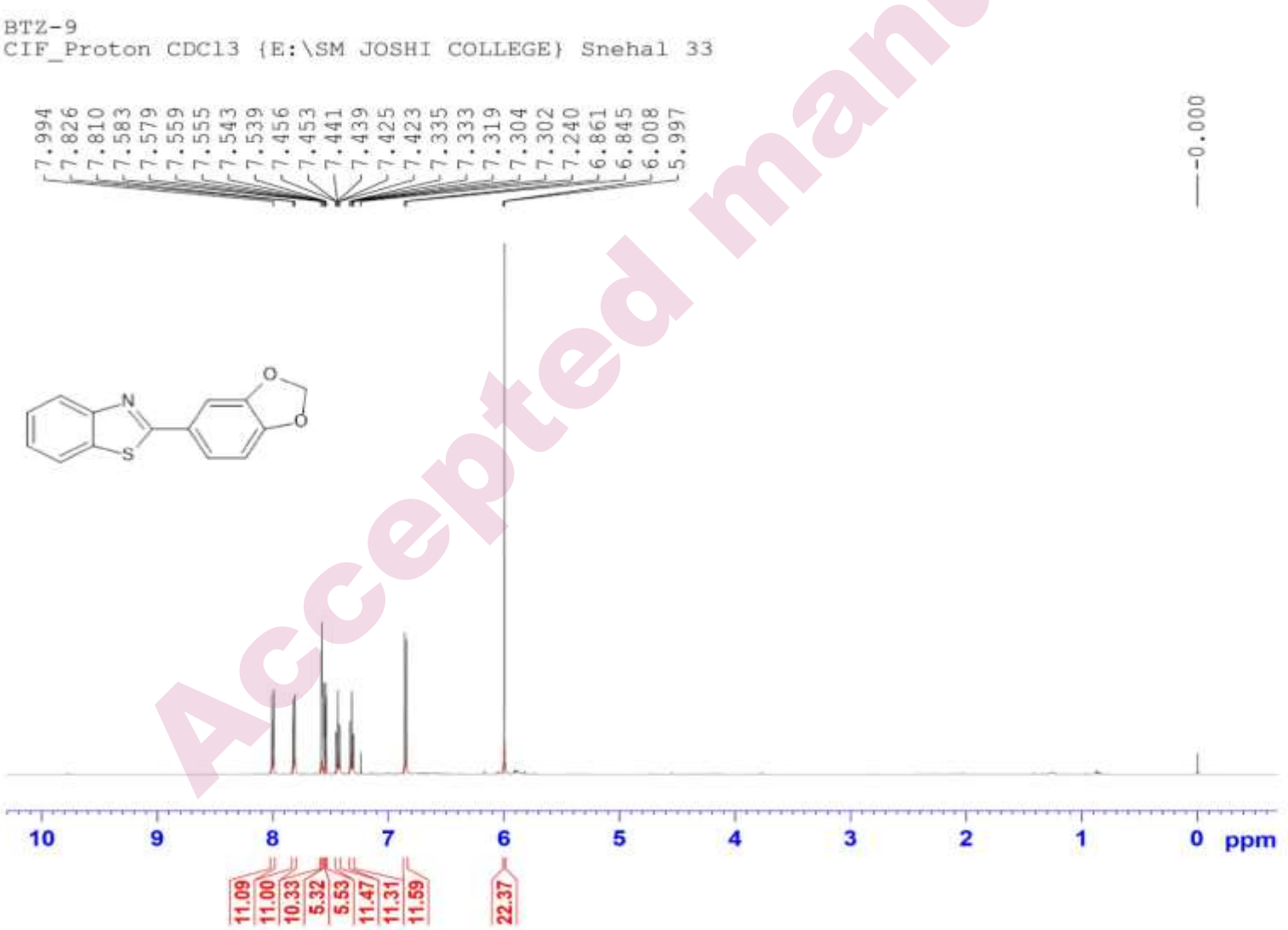
Accepted manuscript

Fig: ^{13}C-NMR 2-(4-methoxyphenyl)-1,3-benzothiazole (Table 6, Entry 5, 7e)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 38.45
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300218 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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Fig: ^{1}H-NMR 2-(2*H*-1,3-benzodioxol-5-yl)-1,3-benzothiazole (Table 6, Entry 8, 7h)

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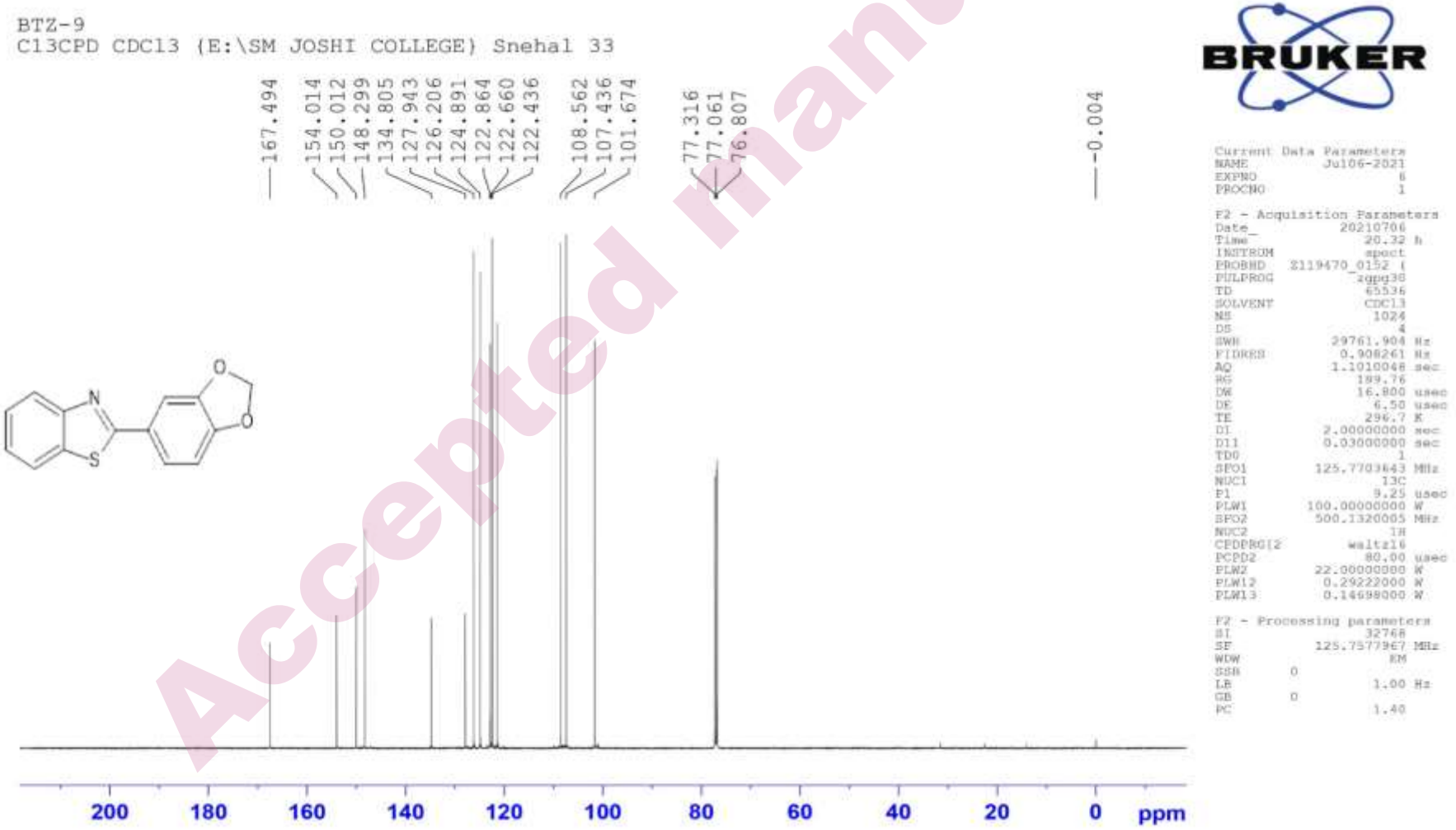

Accepted manuscript

Fig: ^{13}C-NMR 2-(2*H*-1,3-benzodioxol-5-yl)-1,3-benzothiazole (Table 6, Entry 8, 7h)

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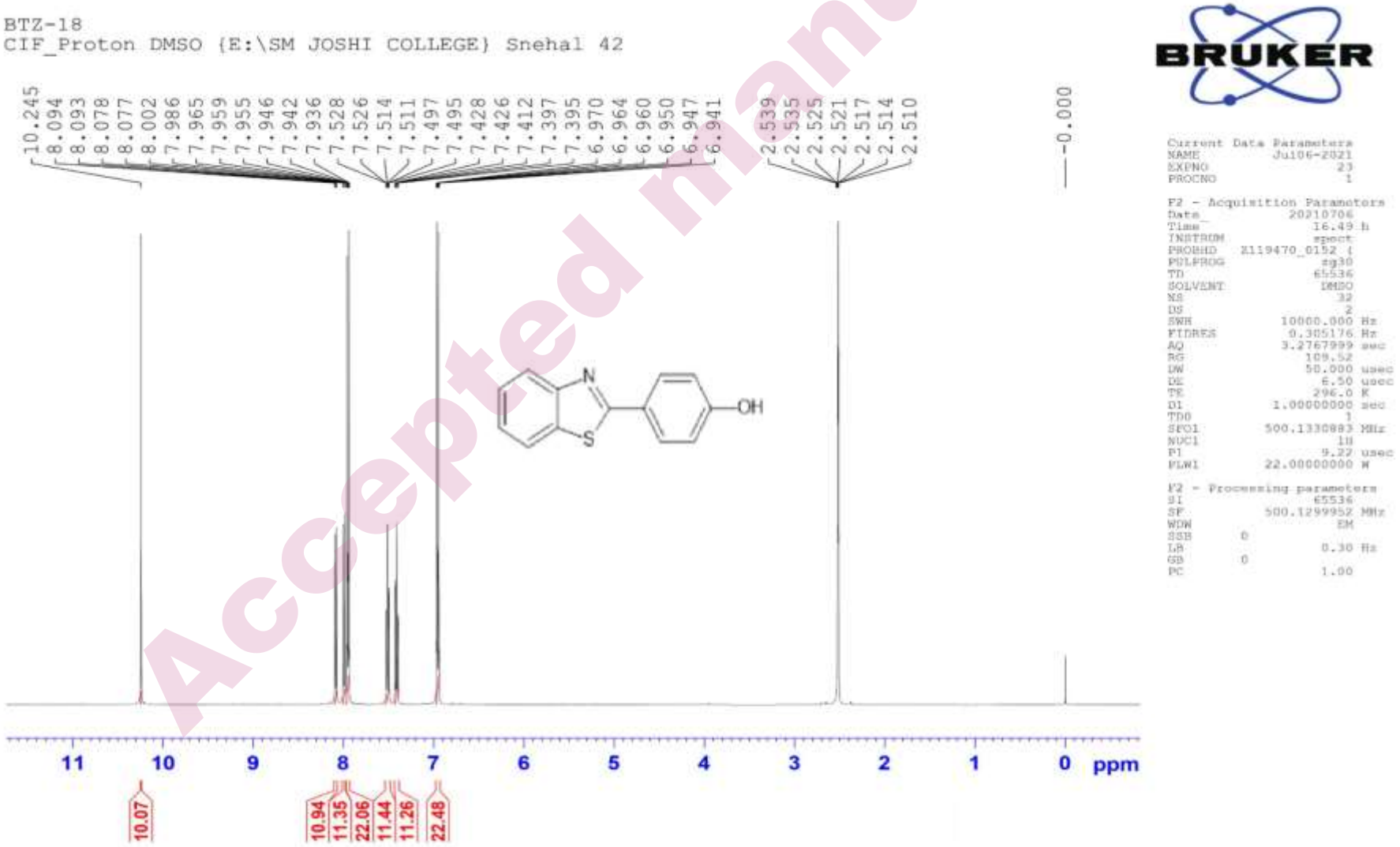


BRUKER

Current Data Parameters
NAME Ju106-2021
EXPNO 23
PROCNO 1

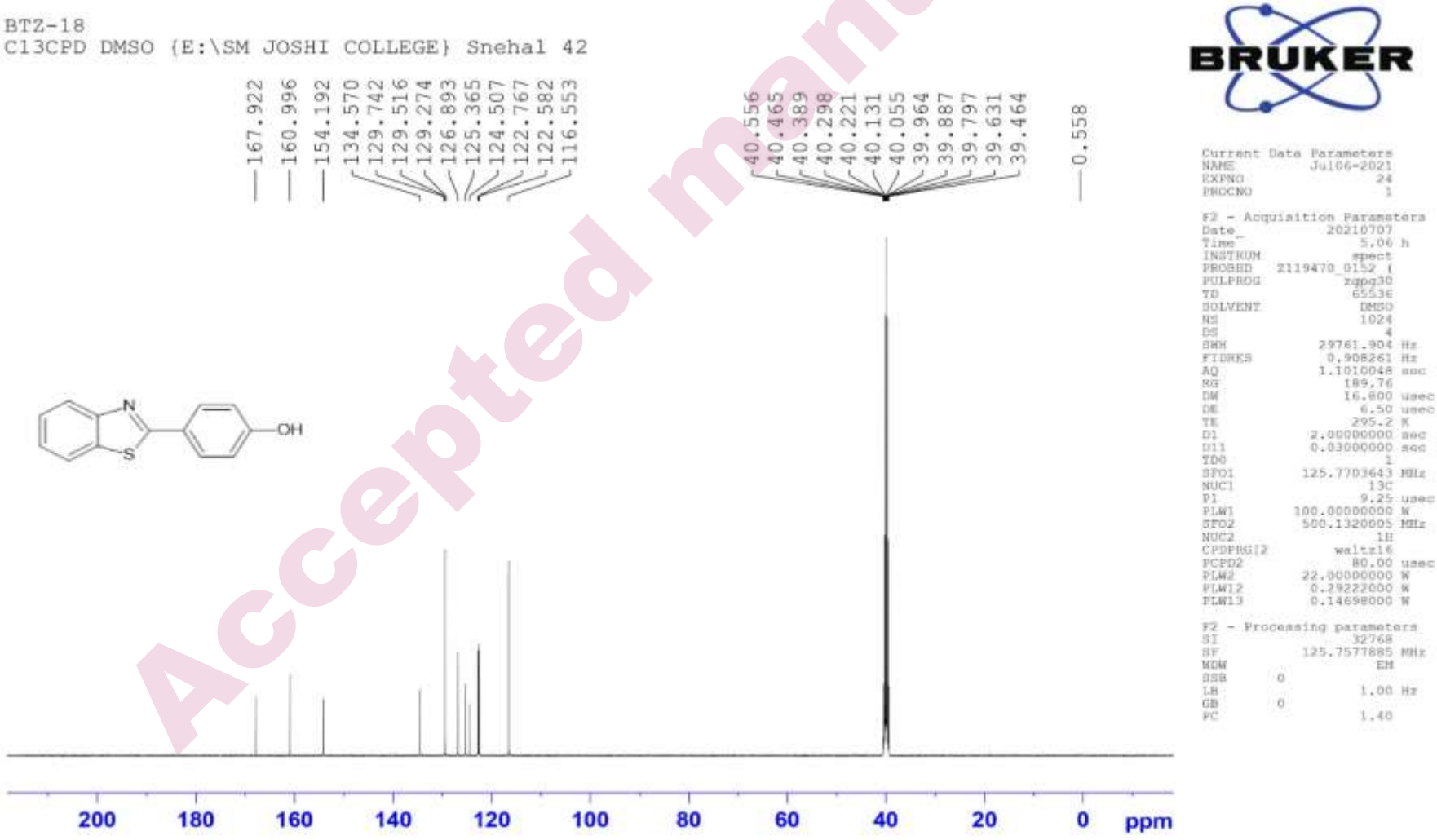
F2 - Acquisition Parameters
Date_ 20210706
Time 16.49 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1299952 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 4-(1,3-benzothiazol-2-yl) phenol (Table 6, Entry 11, 7k)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 24
PROCNO 1

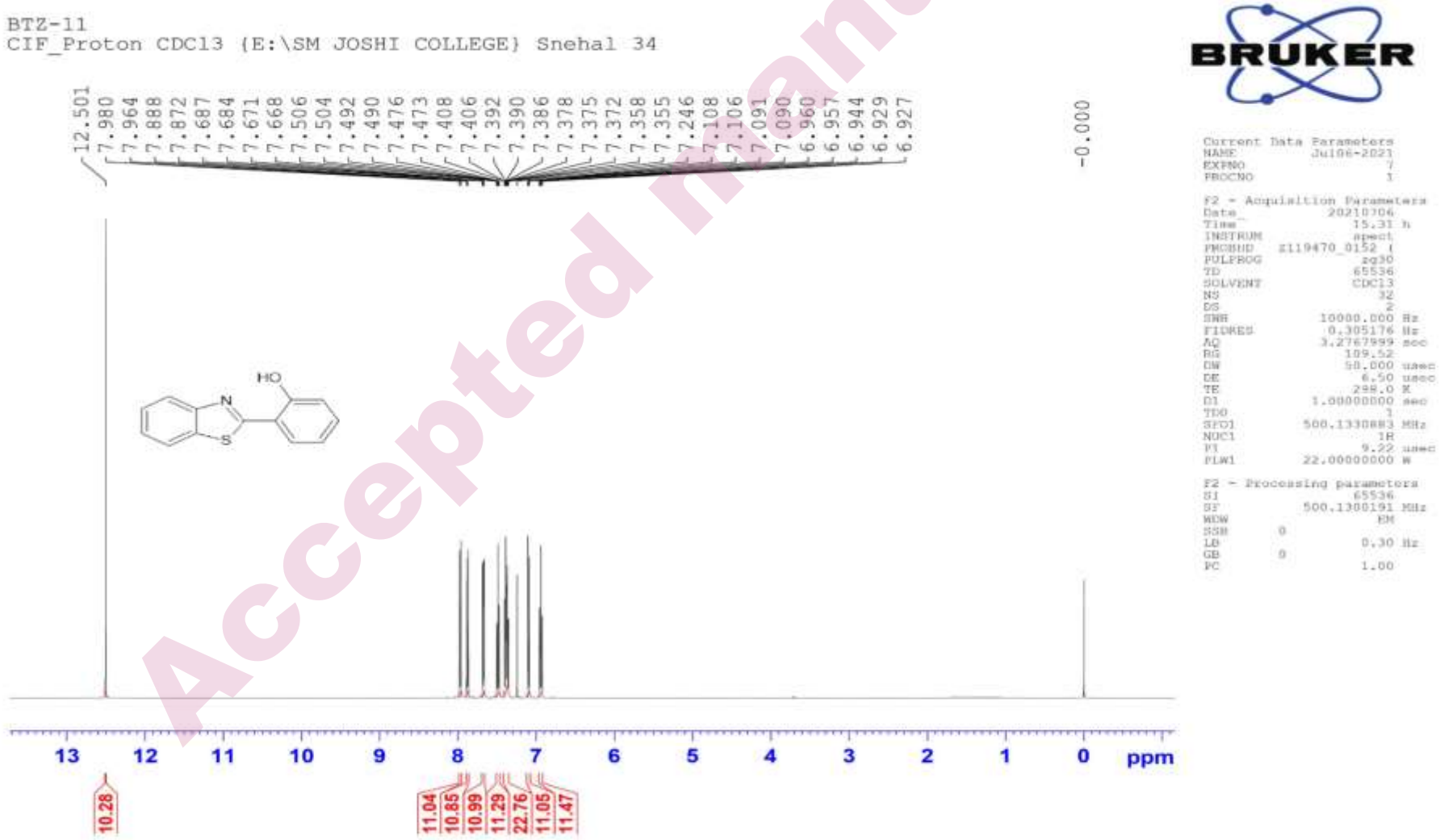
F2 - Acquisition Parameters
Date_ 20210707
Time 5.06 h
INSTRUM spect
PROBHD 2119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 4-(1,3-benzothiazol-2-yl) phenol (Table 6, Entry 11, 7k)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 7
PROCNO 1

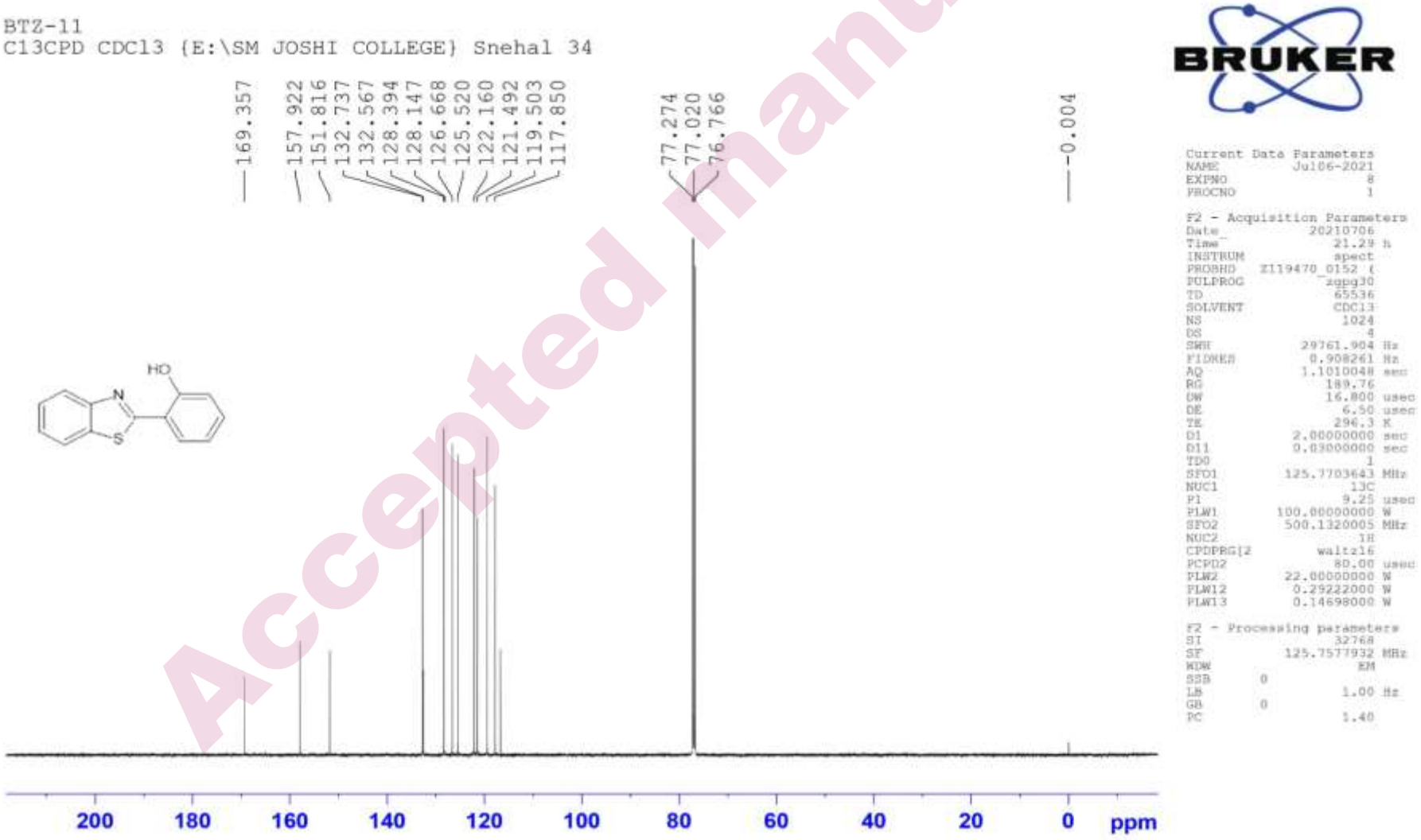
F2 - Acquisition Parameters
Date_ 20210706
Time 15.31 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300191 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 2-(1,3-benzothiazol-2-yl) phenol (Table 6, Entry 12, 7l)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 8
PROCNO 1

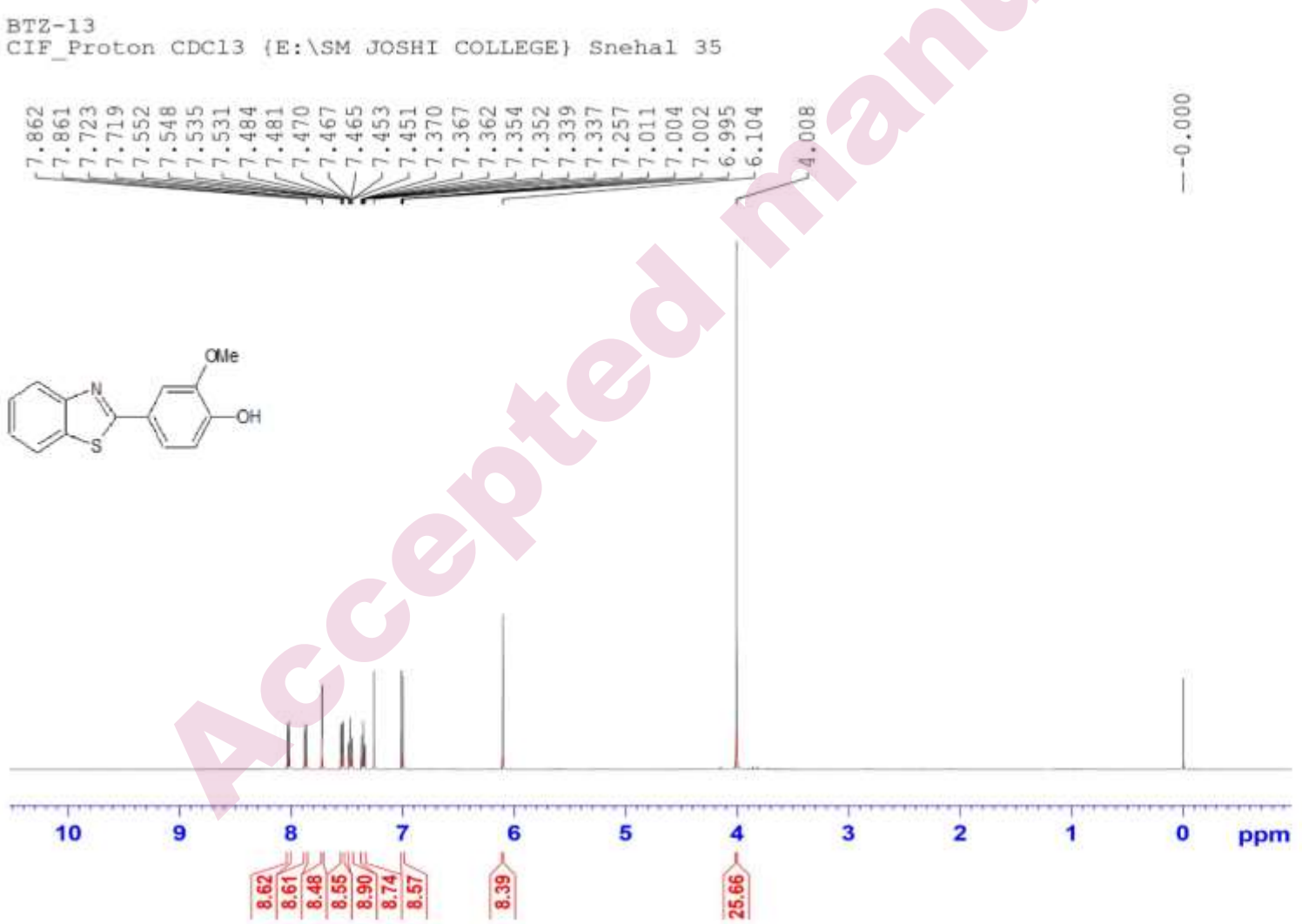
F2 - Acquisition Parameters
Date_ 20210706
Time 21.29 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577932 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{1}H-NMR 2-(1,3-benzothiazol-2-yl) phenol (Table 6, Entry 12, 7l)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 9
PROCNO 1

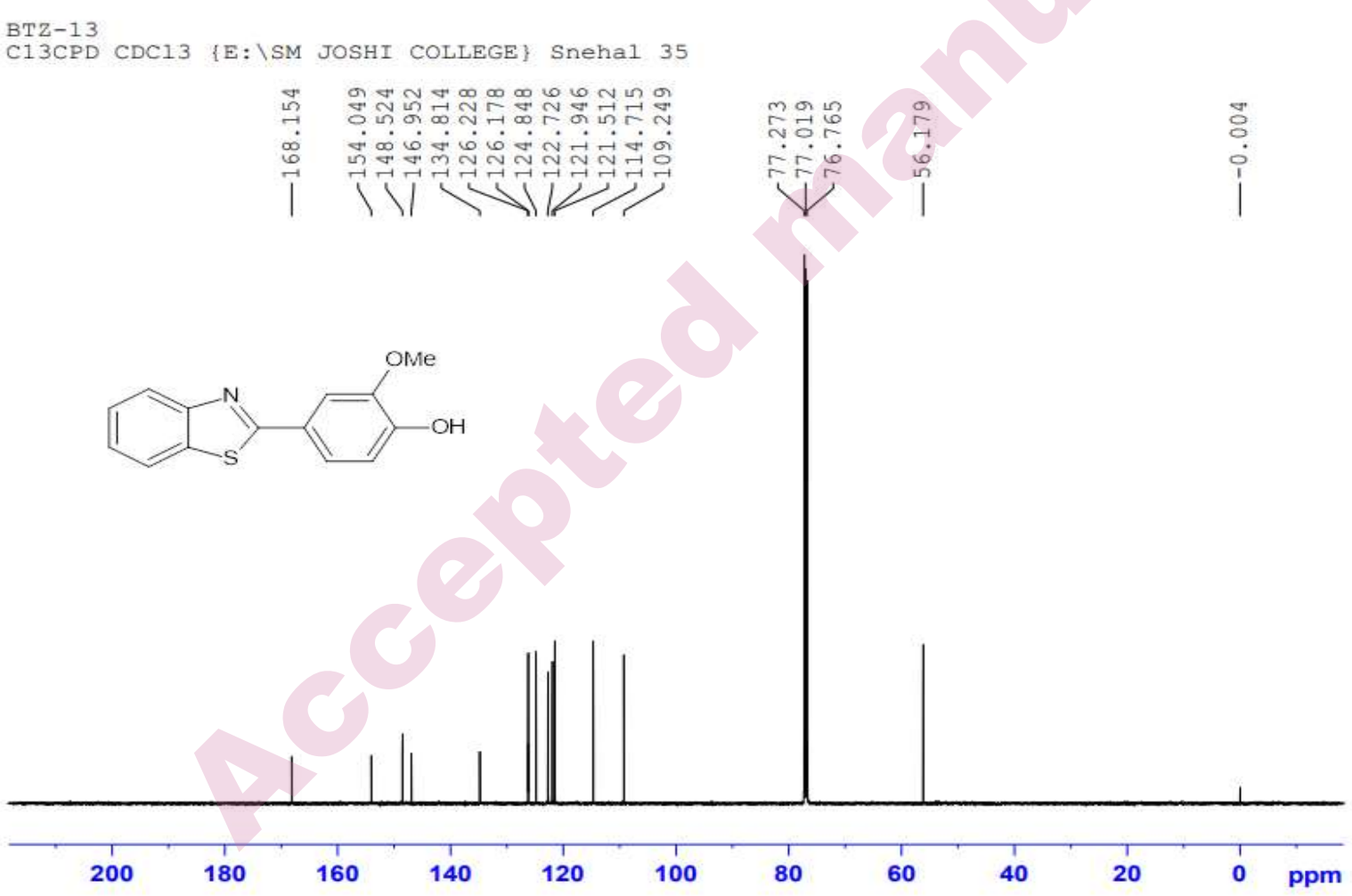
F2 - Acquisition Parameters
Date_ 20210706
Time 15.36 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 4-(1,3-benzothiazol-2-yl)-2-methoxyphenol (Table 6, Entry 13, 7m)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 10
PROCNO 1

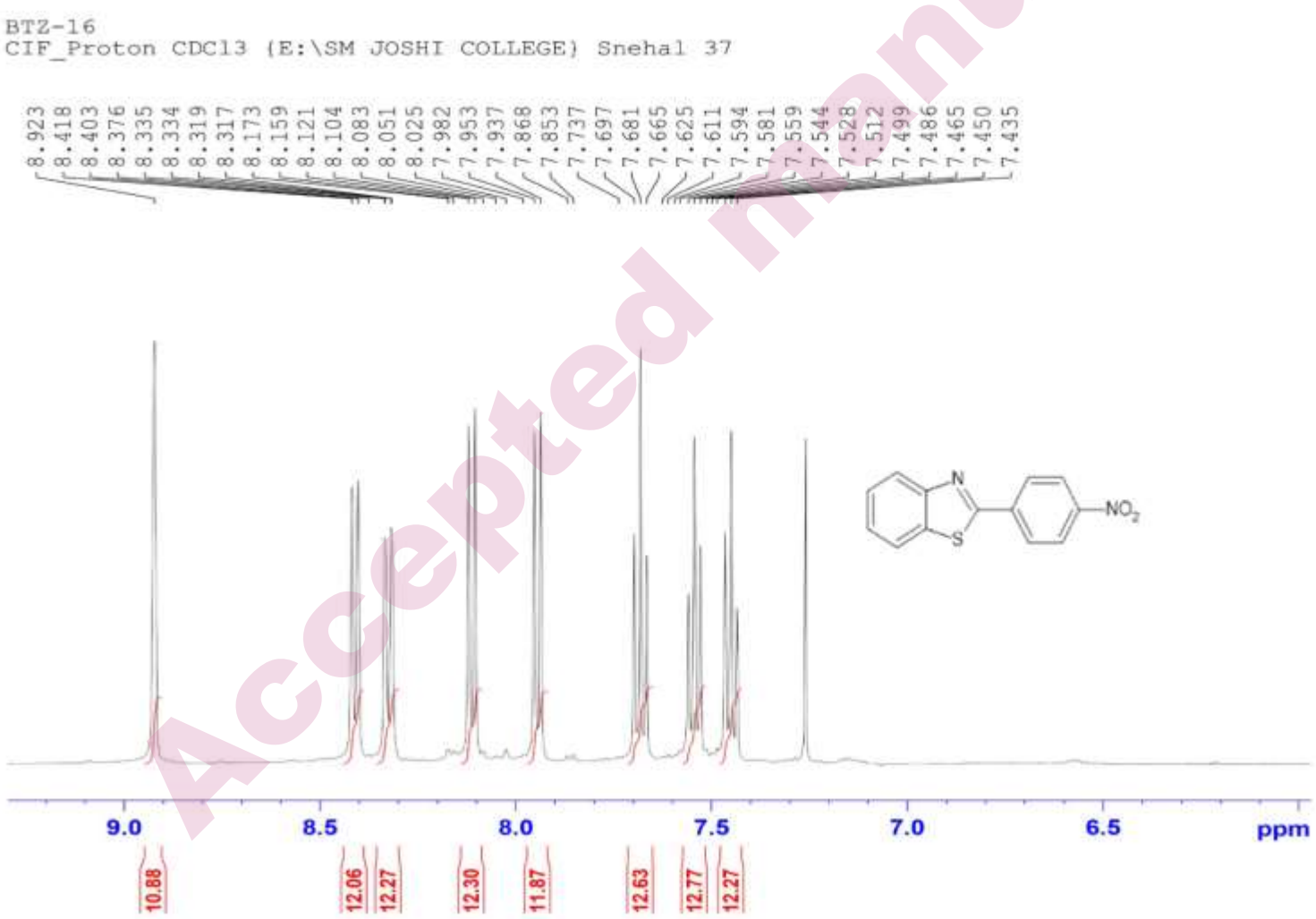
F2 - Acquisition Parameters
Date_ 20210706
Time 22.26 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577913 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 4-(1,3-benzothiazol-2-yl)-2-methoxyphenol (Table 6, Entry 13, 7m)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jul06-2021
EXPNO                         13
PROCNO                         1

F2 - Acquisition Parameters
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Time                      15.48 h
INSTRUM                   spect
PROBHD     X119470_0152 (
PULPROG                   zg30
TD                       65536
SOLVENT                   CDCl3
NS                           32
DS                            2
SWH                   10000.000 Hz
FIDRES                0.305176 Hz
AQ                    3.2767999 sec
RG                       109.52
DW                       50.000 usec
DE                         6.50 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                           1
SFO1                500.1330883 MHz
NUC1                        1H
P1                         8.22 usec
PLW1                 22.00000000 W

F2 - Processing parameters
SI                        65536
SF                  500.1300119 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
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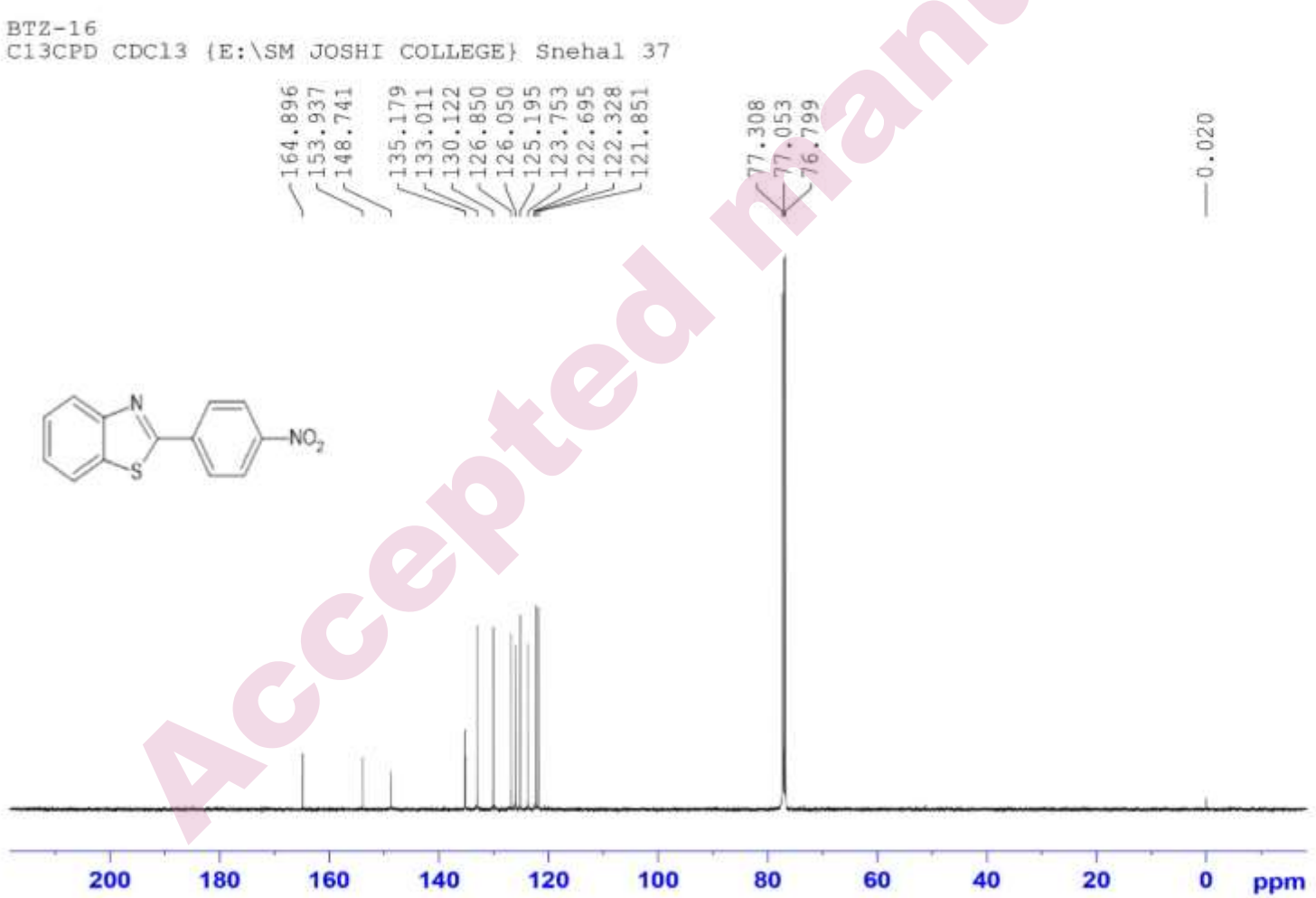
Accepted manuscript

Fig: ^{1}H-NMR 2-(4-nitrophenyl)-1,3-benzothiazole (Table 6, Entry14, 7n)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date 20210707
Time 0.21 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(4-nitrophenyl)-1,3-benzothiazole (Table 6, Entry14, 7n)

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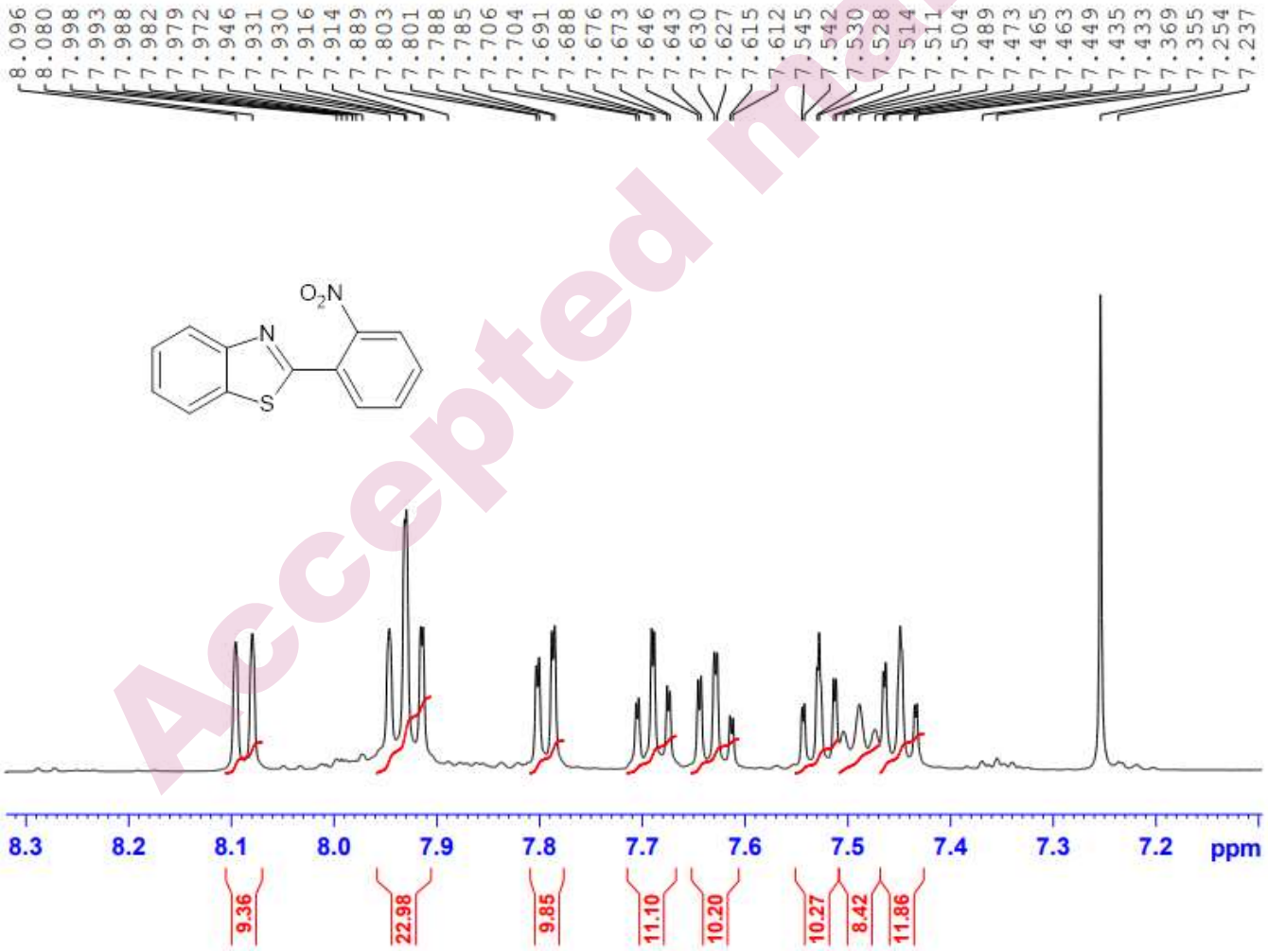
BTZ-17
CIF_Proton CDCl3 {E:\SM JOSHI COLLEGE} Snehal 38



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210706
Time 15.54 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 2-(2-nitrophenyl)-1,3-benzothiazole (Table 6, Entry 16, 7p)

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Current Data Parameters
NAME Jul06-2021
EXPNO 16
PROCNO 1

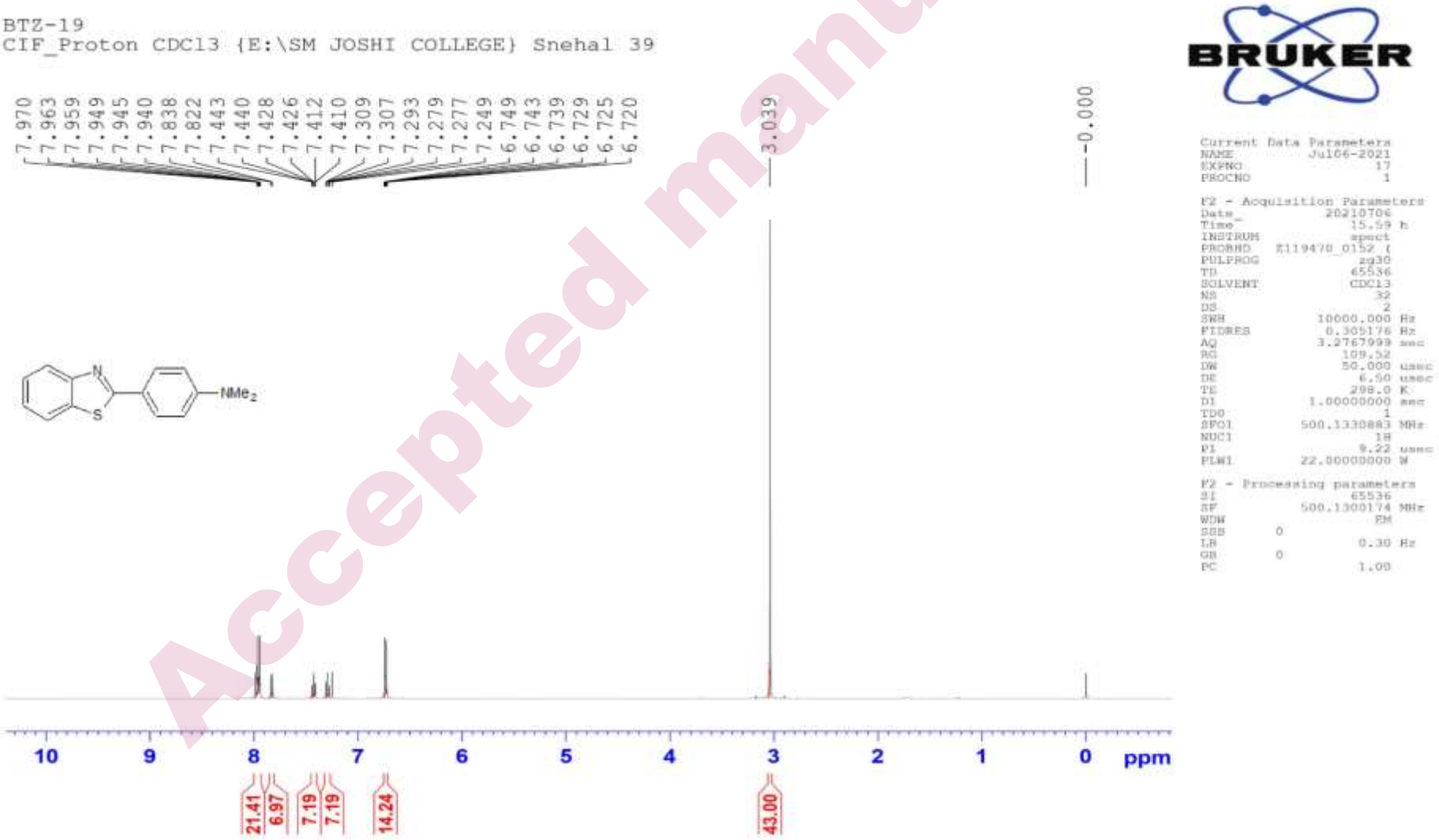
F2 - Acquisition Parameters
Date_ 20210707
Time 1.18 h
INSTRUM spect
PROBHD 2119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577921 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(2-nitrophenyl)-1,3-benzothiazole (Table 6, Entry 16, 7p)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jul06-2021
EXPNO                         17
PROCNO                         1

F2 - Acquisition Parameters
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Time                      15.59 h
INSTRUM                   spect
PROBHD     Z119470_0152 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          32
DS                           2
SWH                  10000.000 Hz
FIDRES                0.305176 Hz
AQ                    3.2767999 sec
RG                       109.52
DW                       50.000 usec
DE                         6.50 usec
TE                        298.0 K
D1                    1.00000000 sec
TD0                          1
SFO1                500.1330883 MHz
NUC1                        1H
P1                         9.22 usec
PLW1                22.00000000 W

F2 - Processing parameters
SI                       65536
SF                  500.1300174 MHz
WDW                         EM
SSB                  0
LB                         0.30 Hz
GB                   0
PC                         1.00
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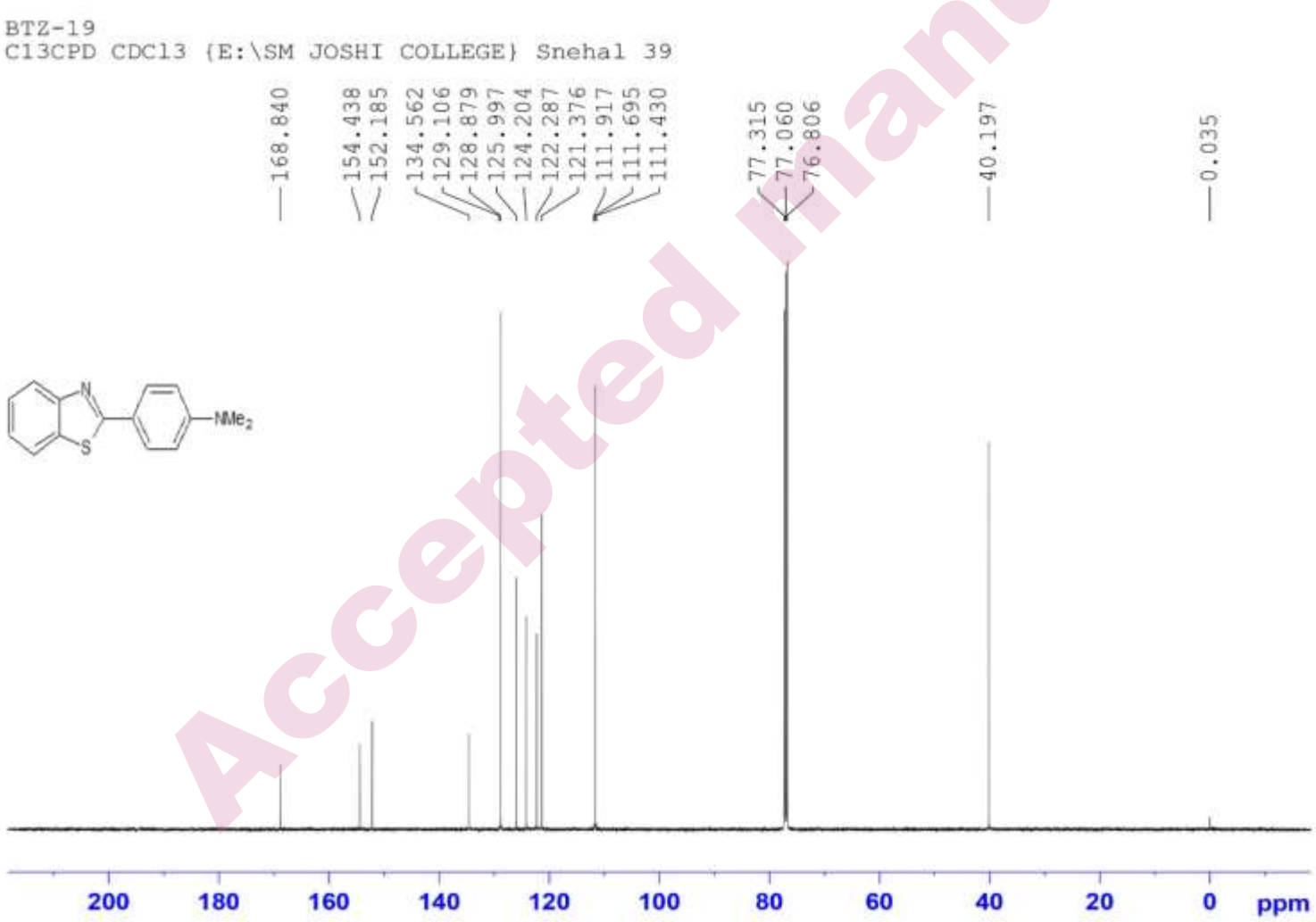
Fig: ^{1}H-NMR 4-(1,3-benzothiazol-2-yl)-*N*, *N*-dimethylaniline (Table 6, Entry 17, 7q)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jul06-2021
EXPNO                         18
PROCNO                         1

F2 - Acquisition Parameters
Date_               20210707
Time                      2.15 h
INSTRUM                   spect
PROBHD    Z119470_0152 (
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  29761.904 Hz
FIDRES               0.908261 Hz
AQ                   1.1010048 sec
RG                      189.76
DW                      16.800 usec
DE                        6.50 usec
TE                       295.4 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1
SFO1               125.7703643 MHz
NUC1                        13C
P1                        9.25 usec
PLW1               100.00000000 W
SFO2               500.1320005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2               22.00000000 W
PLW12               0.29222000 W
PLW13               0.14698000 W

F2 - Processing parameters
SI                       32768
SF                 125.7577885 MHz
WDW                         EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
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Accepted manuscript

Fig: ^{13}C-NMR 4-(1,3-benzothiazol-2-yl)-*N*, *N*-dimethylaniline (Table 6, Entry 17, 7q):

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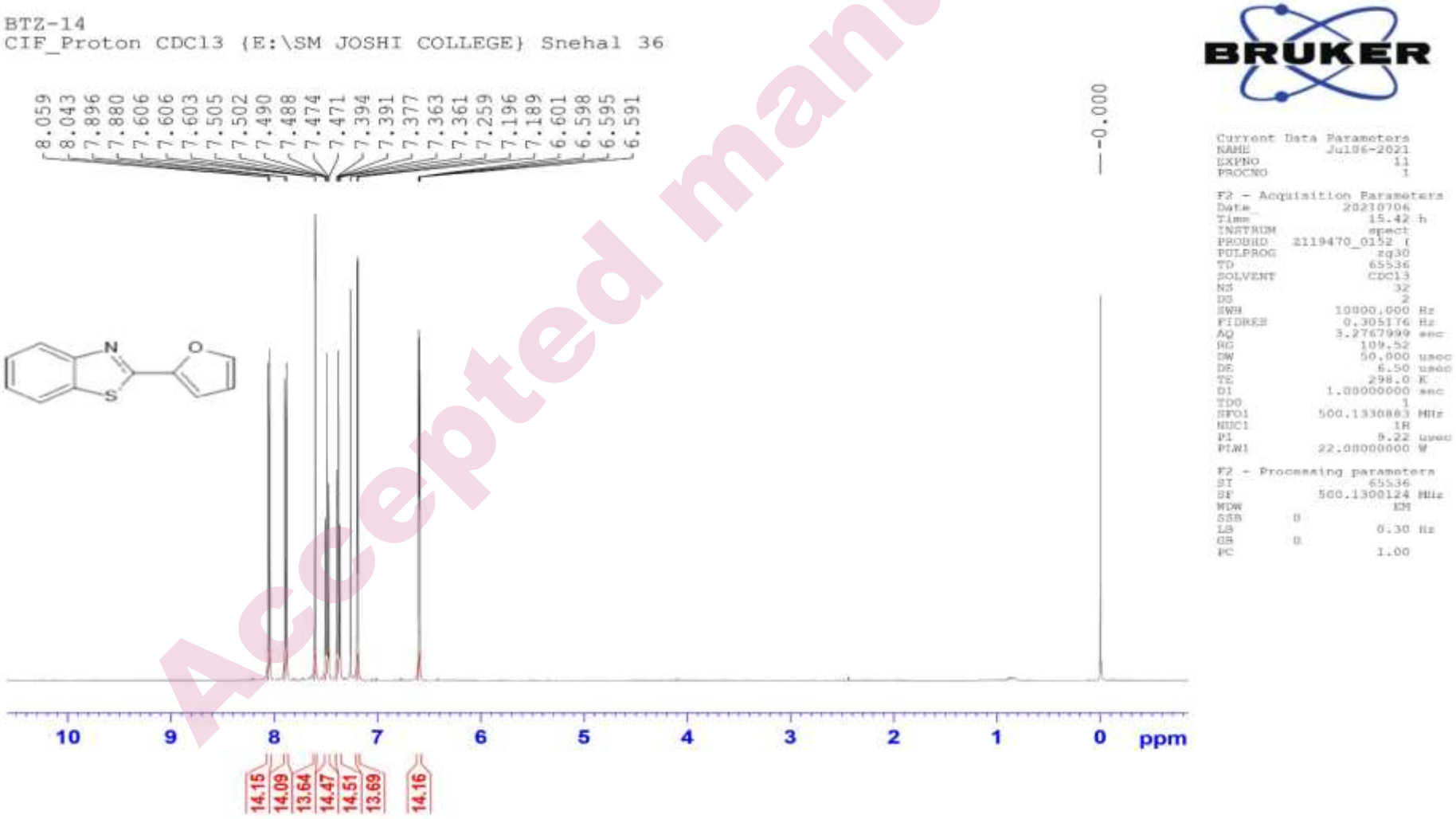

BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210706
Time 15.42 h
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 109.52
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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Fig: ^{1}H-NMR2-(furan-2-yl)-1,3-benzothiazole (Table 6, Entry 19, 7s)

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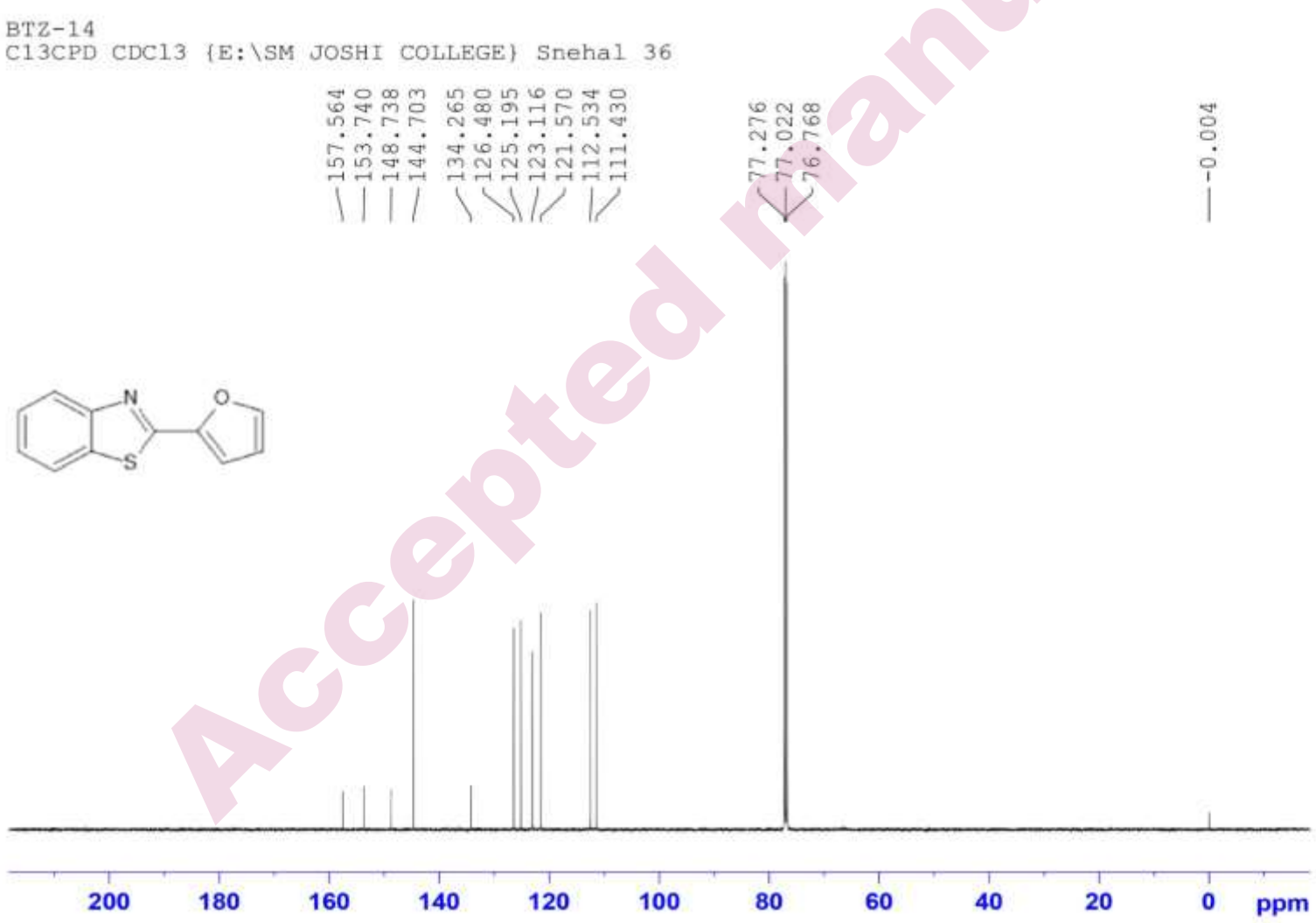

BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210706
Time 23.23 h
INSTRUM spect
PROBHD Z119470_0152 I
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577912 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{1}H-NMR2-(furan-2-yl)-1,3-benzothiazole (Table 6, Entry 19, 7s)

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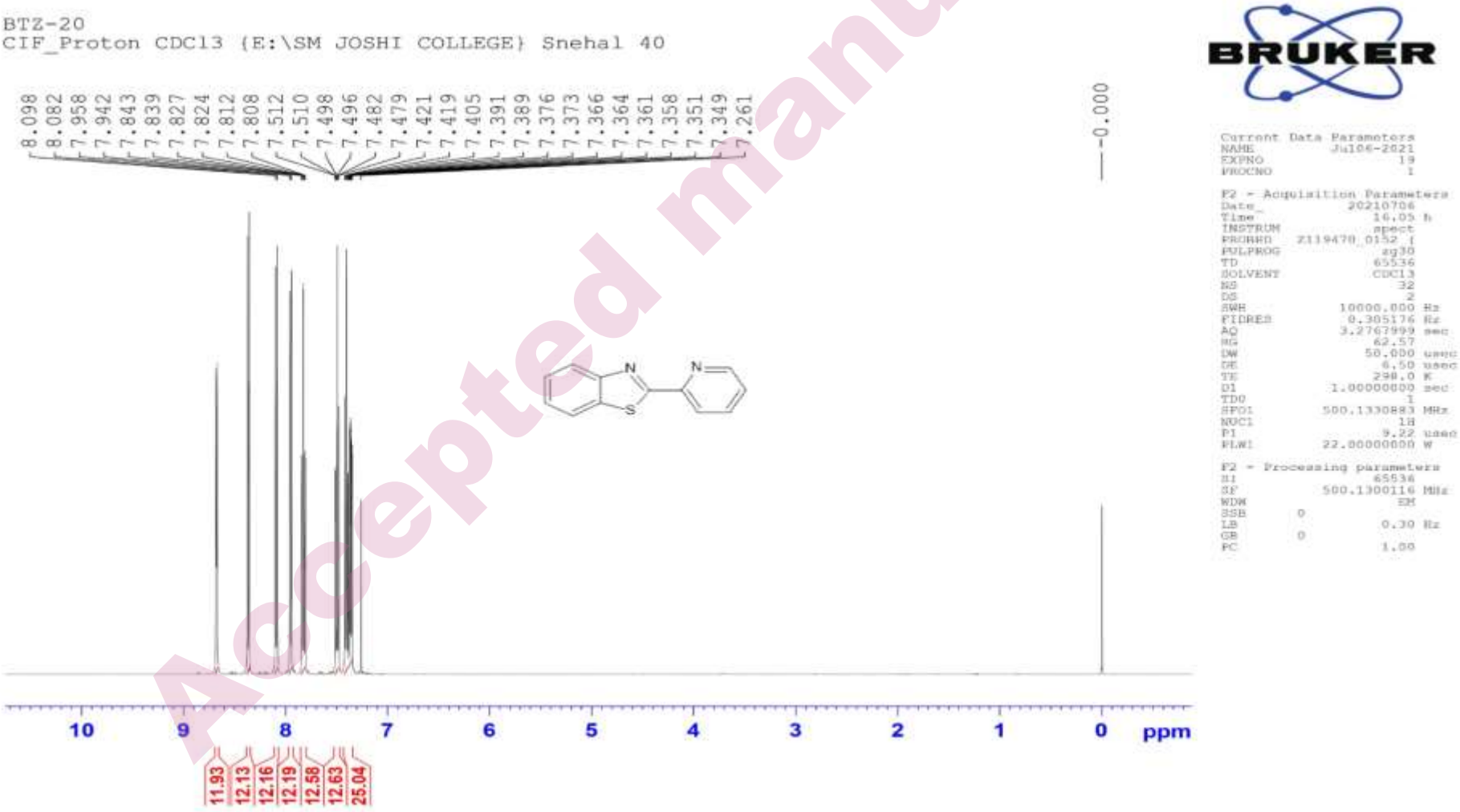


BRUKER

Current Data Parameters
NAME Ju106-2021
EXPNO 19
PROCNO 1

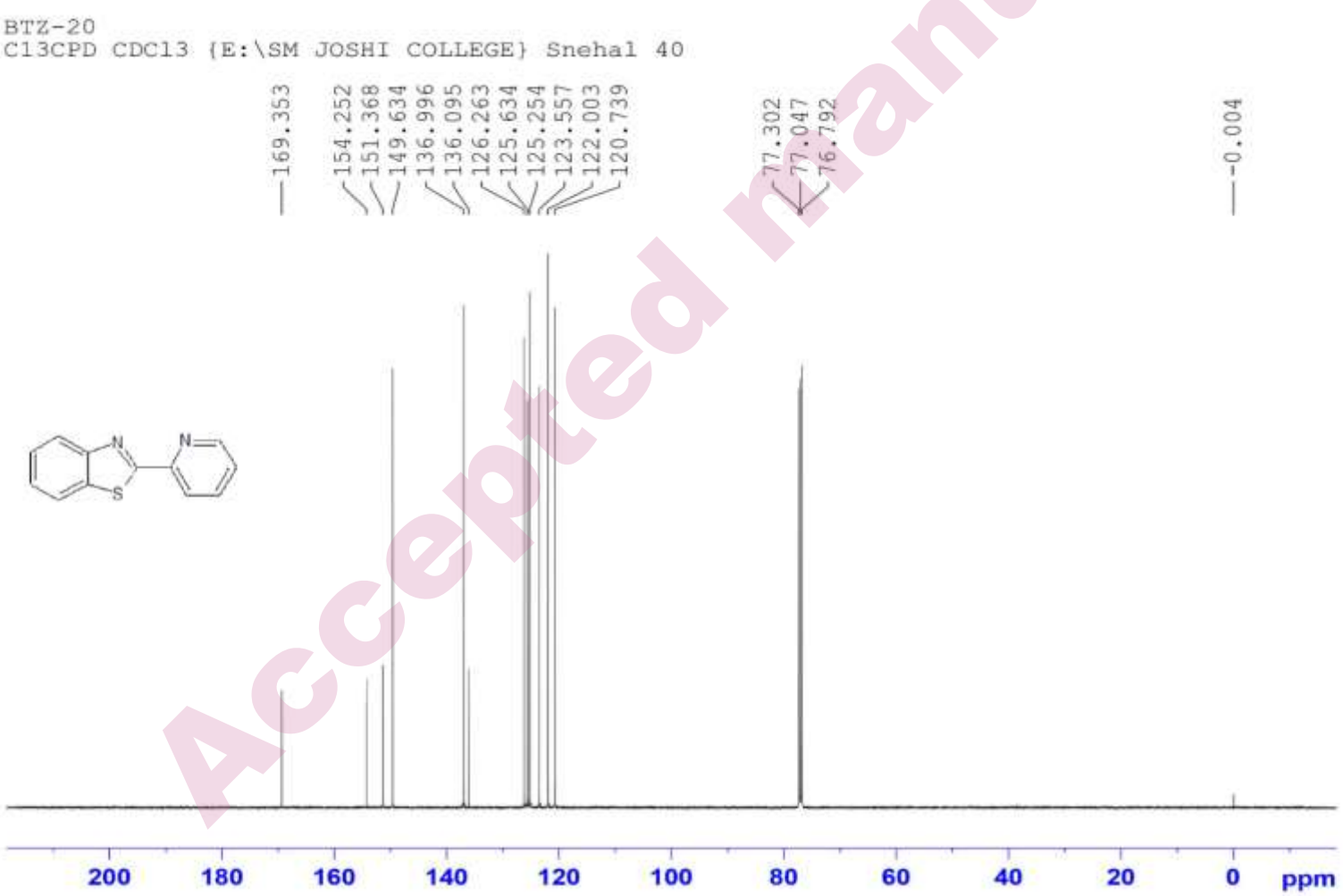
F2 - Acquisition Parameters
Date_ 20210706
Time 16.05 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 62.57
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Accepted manuscript

Fig: ^{1}H-NMR 2-(pyridin-2-yl)-1,3-benzothiazole (Table 6, Entry 20, 7t)

Accepted manuscript



BRUKER

Current Data Parameters
NAME Jul06-2021
EXPNO 20
PROCNO 1

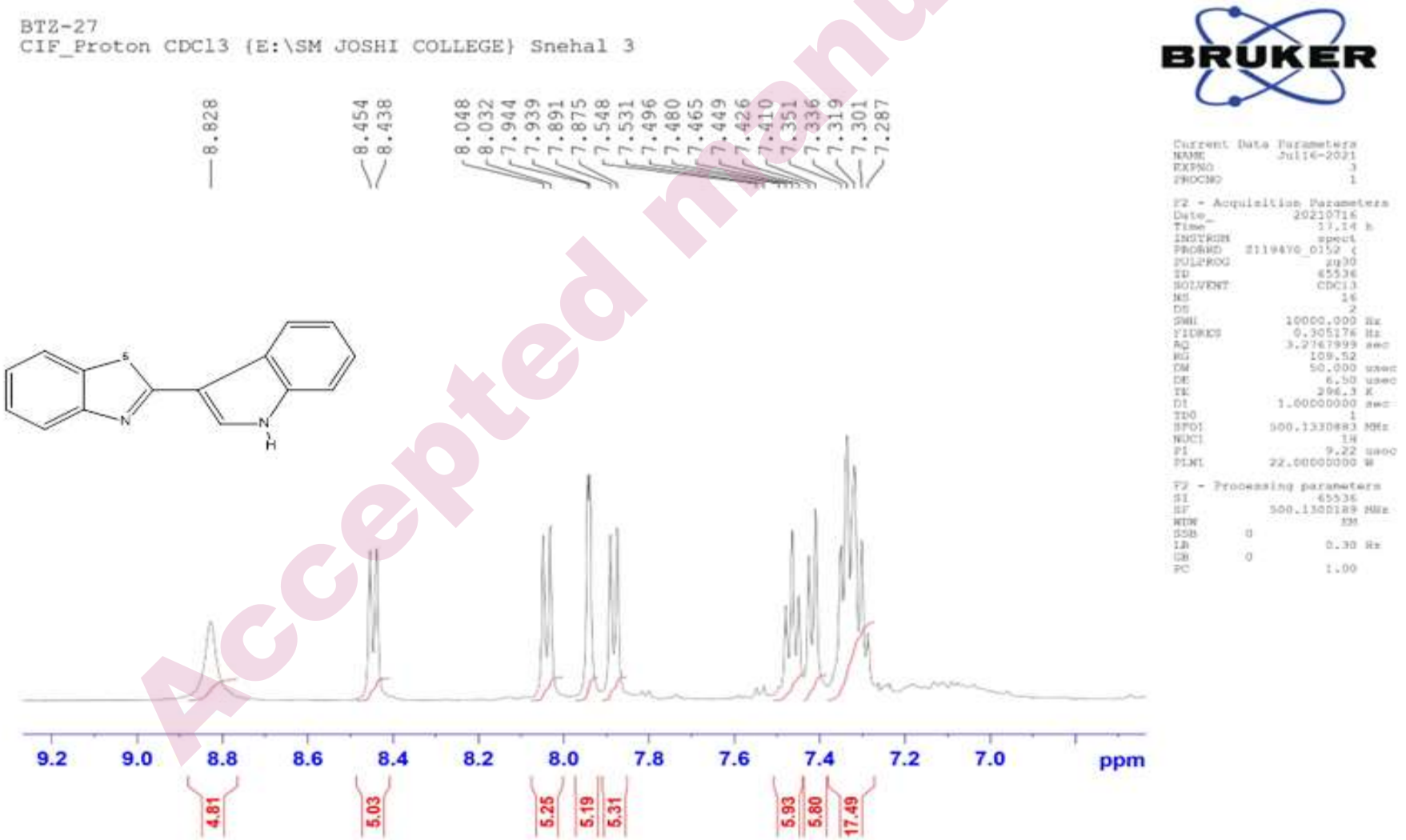
F2 - Acquisition Parameters
Date_ 20210707
Time 3.12 h
INSTRUM spect
PROBHD Z119470_0152 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577939 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Accepted manuscript

Fig: ^{13}C-NMR 2-(pyridin-2-yl)-1,3-benzothiazole (Table 6, Entry 20, 7t)

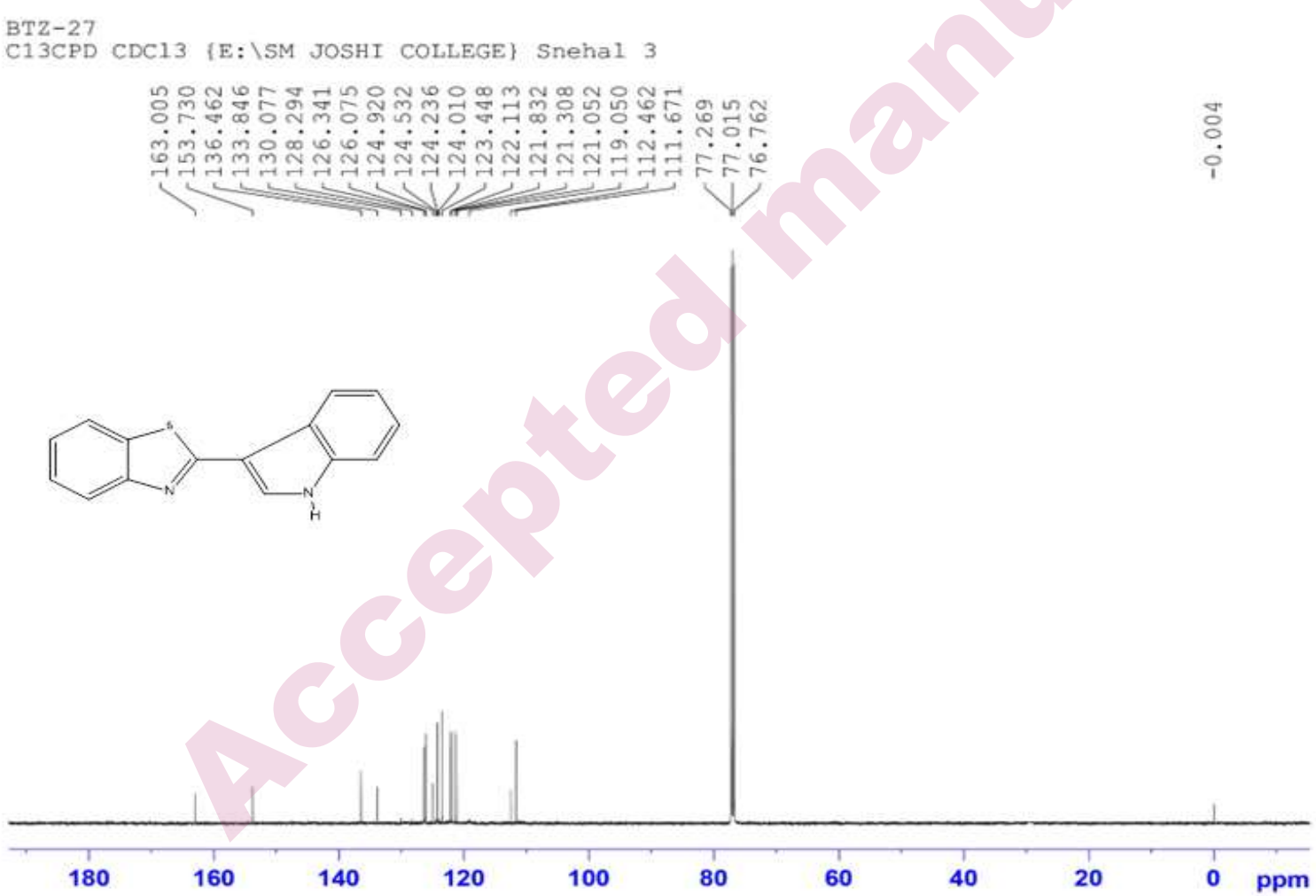
Accepted manuscript



Accepted manuscript

Fig: ^{1}H-NMR 2-(1*H*-indol-3-yl)-1,3-benzothiazole (Table 6, Entry 21, 7u)

Accepted manuscript



BRUKER

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Current Data Parameters
NAME                  Jul16-2021
EXPNO                          8
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20210717
Time                       10.08 h
INSTRUM                     spect
PROBHD    Z119470_0152 (
PULPROG                  zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                           1024
DS                              4
SWH                     29761.904 Hz
FIDRES                  0.908261 Hz
AQ                      1.1010048 sec
RG                         189.76
DW                         16.800 usec
DE                          6.50 usec
TE                         296.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1
SFO1                125.7703643 MHz
NUC1                         13C
P1                          9.25 usec
PLW1                100.00000000 W
SFO2                500.1320005 MHz
NUC2                         1H
CPDPRG[2                waltz16
PCPD2                      80.00 usec
PLW2                 22.00000000 W
PLW12                 0.29222000 W
PLW13                 0.14698000 W

F2 - Processing parameters
SI                          32768
SF                  125.7577930 MHz
WDW                            EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
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Fig: ^{13}C-NMR 2-(1*H*-indol-3-yl)-1,3-benzothiazole (Table 6, Entry 21, 7u)

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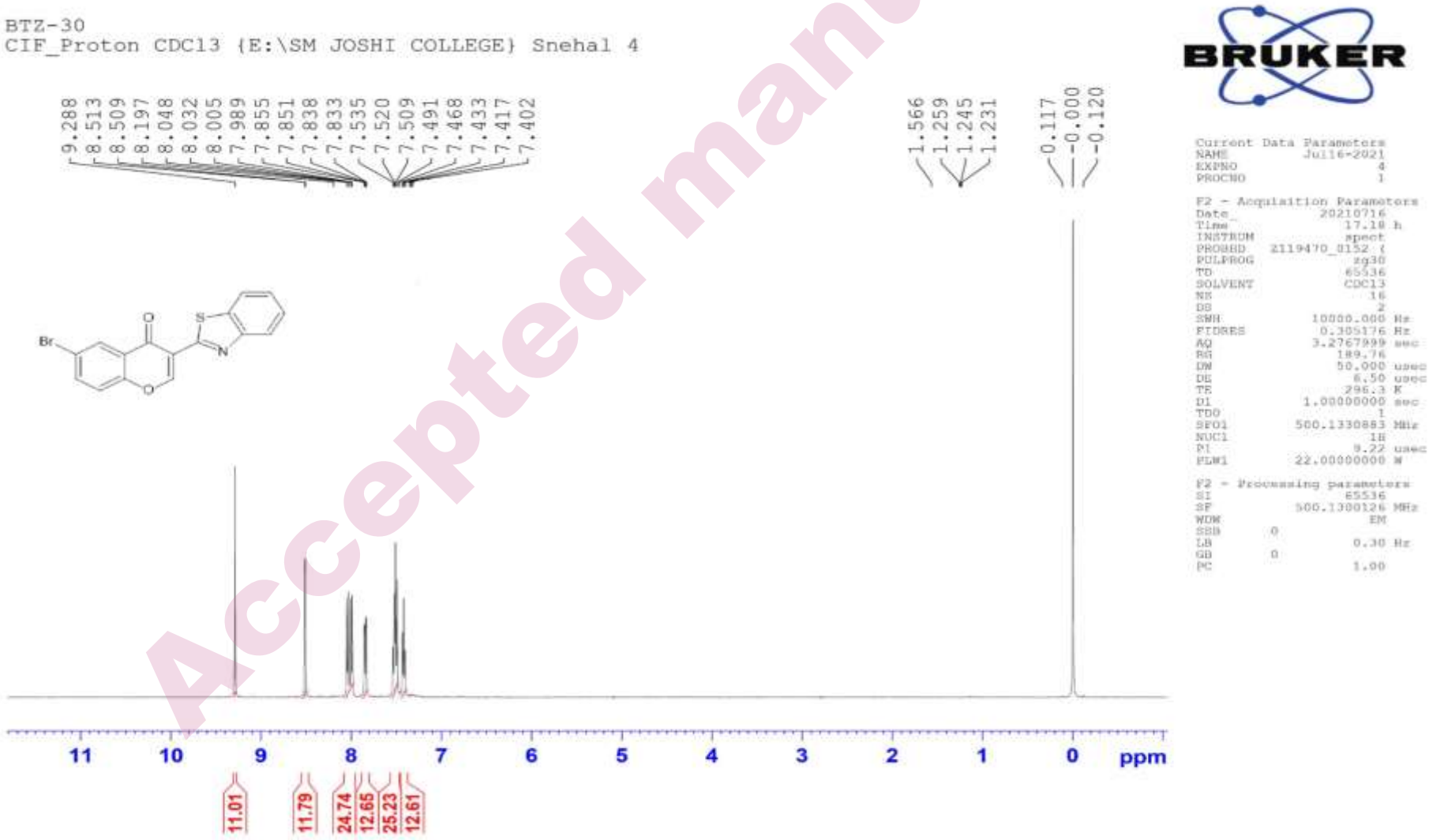

BRUKER

Current Data Parameters
NAME Jul16-2021
EXPNO 4
PROCNO 1

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TD 65536
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DW 50.000 usec
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TE 296.3 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P1 9.22 usec
PLW1 22.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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Fig: ^{1}H-NMR 3-(1,3-benzothiazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one. (Table 6, Entry 22, 7v)

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BRUKER

Current Data Parameters
NAME Julie-2021
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210716
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INSTRUM spect
PROBHD Z119470_0152 (
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SOLVENT CDCl3
NS 1024
DS 4
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FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 189.76
DW 16.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7783643 MHz
NUC1 13C
P1 9.25 usec
PLW1 100.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
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PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.29222000 W
PLW13 0.14698000 W

F2 - Processing parameters
SI 32768
SF 125.7577903 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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Fig: ^{13}C-NMR 3-(1,3-benzothiazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one. (Table 6, Entry 22, 7v)

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Fig: 3-(1,3-benzothiazol-2-yl)-6-bromo-4*H*-1-benzopyran-4-one. (Table 6, Entry 22, 7v)

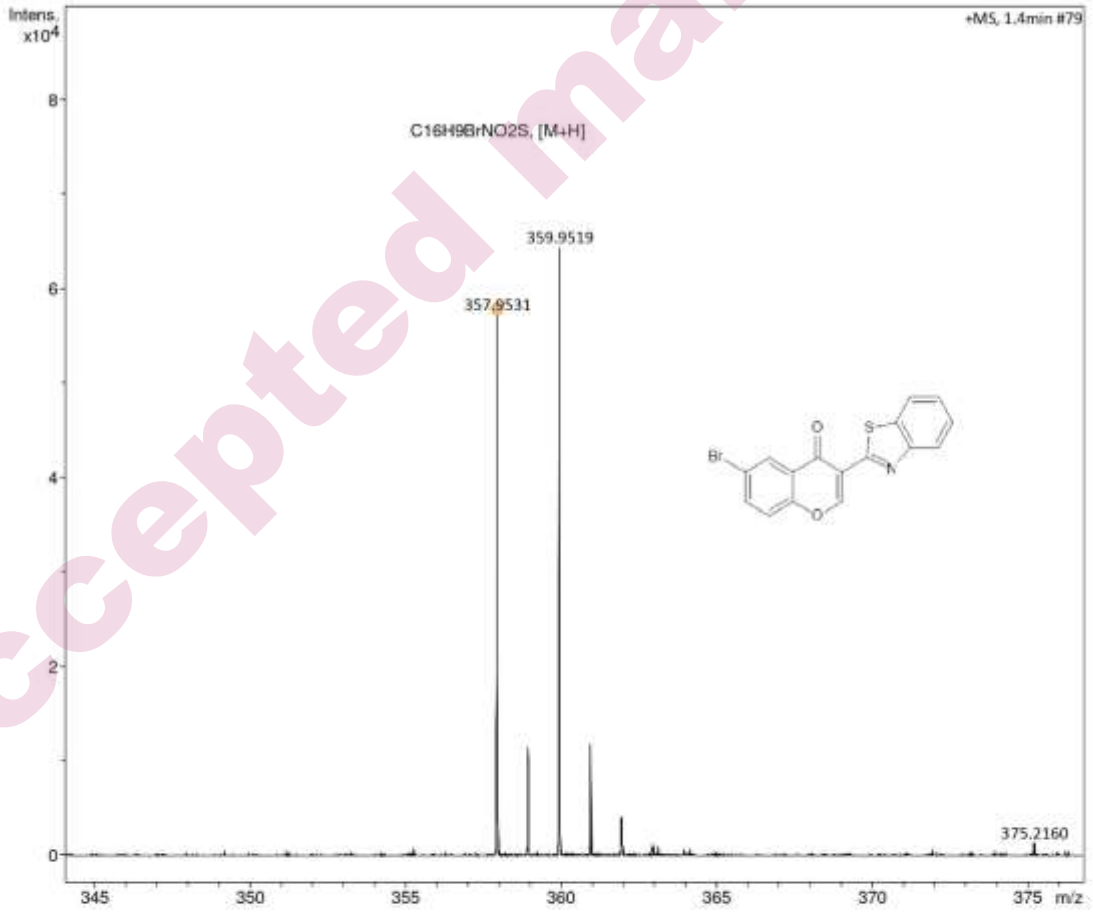
Savitribai Phule Pune University - Central Instrumentation Facility

Analysis Info

Analysis Name	D:\Data\2022\JAN\SPPU COLLEGE\BABURAO GHOLAP COLLEGE, SANGVI\RAMESH GAWADE\BTZ-30_GB2_01_3809.d	Acquisition Date	1/22/2022 3:07:50 PM
Method	dlc_ms50-1200mz_2500v_12min_0.120ml flow_95b.m	Operator	CIF
Sample Name	BTZ-30	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.7 Bar
Focus	Active	Set Capillary	2500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	1200 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule	Adduct
357.953121	1	C16H9BrNO2S	100.00	357.953188	0.1	0.2	33.4	12.5	even	ok	M+H
	1	C16H9BrNO2S	100.00	357.953188	0.1	0.2	33.4	12.5	even	ok	M+H

BTZ-30_GB2_01_3809.d

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