

SYNTHESIS AND CHARACTERIZATION OF SEMICARBOZONE

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Science in Chemistry

by

Abhinav Koushik v v (39030001)

Roopan Raj .B (39030026)

Sharmila .D (39030033)

SYNTHESIS AND CHARACTERIZATION OF SEMICARBOZONE



**DEPARTMENT OF CHEMISTRY
SCHOOL OF SCIENCE AND HUMANITIES**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

April – 2022



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BONAFIDE CERTIFICATE**

This is to certify that this Project Report is the bonafide work of **ABHINAV KOUSHIK (39030001) ROOPAN RAJ B (39030026) SHARMILA.D (39030033)** who carried out the project entitled "**SYNTHESIS AND CHARACTERIZATION OF SEMICARBAZONE**" under our supervision from November 2021 to April 2022.

Internal Guide

Dr. J. KARTHIKEYAN, M.Sc., Ph.D.,

Head of the Department

Dr. J. KARTHIKEYAN, M.Sc., Ph.D.

Submitted for Viva voce Examination held on _____

Internal Examiner

External Examiner

DECLARATION

We **ABHINAV KOUSHIK (39030039) ROOPAN RAJ B (39030026), SHARMILA .D (39030033)** hereby declare that the Project Report entitled **“SYNTHESIS AND CHARACTERIZATION OF SEMICARBAZONE”** done by me under the guidance of, **Dr. J. KARTHIKEYAN M.sc.,Ph.D.**, Head of the Department, department of Chemistry at **Sathyabama Institute of Science and Technology**, Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai - 600119 is submitted in partial fulfillment of the requirements for the award of Master of Science degree in Chemistry.

DATE:

PLACE: CHENNAI

**V.V. ABHINAV KOUSHIK
ROOPAN RAJ
SHARMILA.D**

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V.V.ABHINAV KOUSHIK

ROOPAN RAJ B

SHARMILA.D

ABSTRACT

A new semicarbazone derivative, phenyl semicarbazone is synthesized by using N,N,-bis (2- chloroethyl)aminobenzaldehyde). Semicarbazones are an important compounds obtained by condensation of semicarbazides with suitable aldehyde or ketones. Synthesis of new semicarbazones always plays a new role in many fields like chemical, agricultural and industries. The functional groups of the synthesized compound was identified by using FTIR,UV,NMR.

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LIST OF ABBREVIATION

FTIR	Fourier Transform Infrared Spectroscopy
NMR	Nuclear Magnetic Resonance Spectroscopy
UV	Ultra Violet spectroscopy

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CHAPTER 1

INTRODUCTION

1.1 SEMICARBAZONE

Thiosemicarbazones and semicarbazones are significant classes of mixtures that direction with numerous metal particles on account of the presence of a few contributor molecules like nitrogen, oxygen and sulfur deeply. The arrangement and portrayal of differently subbed thiosemicarbazones/semicarbazones have been investigated as soon as the 1950s and coordination of these mixtures with metal particles were widely concentrated in the 1960s.

Although these early revelations were achievements in the improvement of thiosemicarbazone/semicarbazone compounds, the absence of primary proof, for instance X-beam single precious stone design, may raise questions about the engineering of a portion of the metal edifices. Lobana et.al have accumulated a point by point survey on the holding and primary qualities of thiosemicarbazones specifically, and announced the improvement of more up to date procedures for planning fresher subsidiaries. In the new years consideration has focussed on the natural utilizations of these mixtures. In such manner, it has been viewed that some thiosemicarbazones have intense anticancer action.

Blend of different oximes, semicarbazones, phenyl hydrazones and their buildings with different progress metals are accounted for in the literature and viewed as dynamic as antibacterial, antitubercular, antilepral, antiviral, antimalarial and dynamic against specific sorts of tumours. Thinking about the significance of such progress metal buildings also, in continuation of our prior work on change metal edifices with oxime, semicarbazone and phenylhydrazone subsidiaries, in the current paper, combination, portrayal and antibacterial movement of Co(II), Ni(II) and Cu(II) buildings with 4-chlorobenzaldehyde oxime, 4-methylbenzaldehyde oxime, 4-nitrobenzaldehyde oxime, 4-chlorobenzaldehyde semicarbazone, 4-methylbenzaldehyde semicarbazone, 4-nitrobenzaldehyde semicarbazone, 4-chlorobenzaldehyde phenylhydrazone, 4-methylbenzaldehyde phenylhydrazone and

4-nitrobenzaldehyde phenylhydrazone are accounted for, what's more, contrast in antibacterial movement between the free ligands and buildings were considered without any zeroing in on the construction of the buildings.



Fig:1.1:Semicarbazone

The utilization of iron chelators containing thiosemicarbazones has likewise been widely contemplated as has the anticancer movement of novel thiosemicarbazones created through the blend of retro sections. Some thiosemicarbazones have been found to have antibacterial and antifungal action and the pharmacological profile of thiosemicarbazones appended to a heterocyclic center has been accounted for by Singhal et al. The natural profile displayed by these thiosemicarbazone and semicarbazone subordinates has prompted work in which these mixtures have been complexed with positron discharging isotopes (for example ^{64}Cu , ^{68}Ga) for use as imaging specialists. The radio-metal buildings (generally strikingly ^{64}Cu) of these ligands have been examined for their applications as sub-atomic imaging specialists. Pascu et al have integrated a progression of ^{68}Ga buildings of bis-thiosemicarbazones to assess their imaging potential.

Strangely, they report that the response of GaCl_3 with these ligands brings about the development of a square based pyramidal mono-chloride metal complex, inciting us to propose the capability of trading this chloride with different ligands, especially different incandescent lamp. The usage of iron chelators

containing thiosemicarbazones has moreover been broadly considered as has the anticancer development of novel thiosemicarbazones made through the mix of retro segments. Some thiosemicarbazones have been found to have antibacterial and antifungal activity and the pharmacological profile of thiosemicarbazones attached to a heterocyclic community has been represented by Singhal et al.

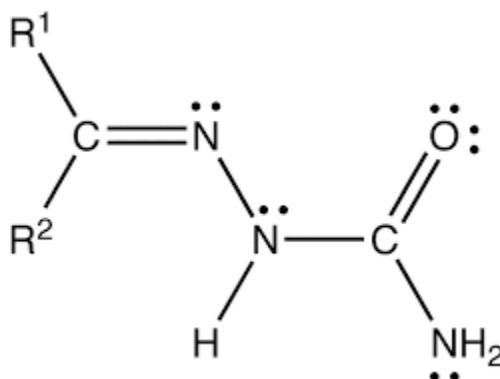


Fig:1.2:Structure of Semicarbazone

The regular profile showed by these thiosemicarbazone and semicarbazone subordinates has provoked work in which these combinations have been complexed with positron releasing isotopes (for instance ^{64}Cu , ^{68}Ga) for use as imaging subject matter experts. The radio-metal structures (for the most part strikingly ^{64}Cu) of these ligands have been inspected for their applications as sub-nuclear imaging subject matter experts. Pascu et al have coordinated a movement of ^{68}Ga structures of bis-thiosemicarbazones to evaluate their imaging potential. Unusually, they report that the reaction of GaCl_3 with these ligands achieves the advancement of a square based pyramidal mono-chloride metal complex, affecting us to propose the ability of exchanging this chloride with various ligands, particularly unique radiant light.

1.2 SYNTHESIS OF SEMICARBAZONE

1.2.1 Synthesis of ThioSemicarbazones and Semicarbazone

Refluxing aldehyde or ketone with a thiosemicarbazide creates thiosemicarbazones. For aldehydes, the response is normally finished in under 3 h and no acidic corrosive is required. For ketones, the response is normally run short-term with 1% acidic corrosive. Yields are for the most part more noteworthy than 90% besides with a couple of explicit ketones, for example, the 2-subbed aryl ketones. Combination of semicarbazones is done at room temperature. A sodium acetic acid derivation arrangement of semicarbazide hydrochloride salt is added to the ethanol arrangement of aldehyde or ketone. The item normally precipitates out with great yield.

1.2.2 Synthesis of the Cyclized Pyrazoline Analogues:

The Mannich response of different ketones with formaldehyde and dimethylamine hydrochloride creates the Mannich base precursor.¹⁶ The response is touchy to both how much hydrochloric corrosive and how much dissolvable present. The response works best when a base measure of ethanol and 2 μL of corrosive/mmol ketone is applied. The methyl aryl ketones gave significant returns above 80%, while the yields for other alkyl aryl ketones in the Mannich response were lower in the scope of 30-60%. In any case, the item blend contained just unreacted beginning material and the Mannich item. The unreacted beginning material was recuperated for full use. Buildup and cyclization of the antecedent with the thiosemicarbazide creates the cyclized pyrazoline analogs with yields somewhere in the range of 20% and half without optimization. The cyclization additionally works in the subbed thiosemicarbazide yet with a lower yield.

1.3 APPLICATIONS OF SEMICARBAZONE

Semicarbazone plays an essential role in agriculture, pharmaceutical and industrial chemistry and they are used as catalysts in various biological systems, polymers and dyes, besides some uses antifertility and enzymatic agents.

Lipophilicity, which controls the rate of entry in to the cell is modified by coordination. Co-ordination may lead to significant reduction of drug -resistance. Semicarbazones also used as spectrophotometric agents as well for the analysis of metal ions and are frequently used in the qualitative organic analysis of carbonyl compounds.

1.3.1 Anti-Fungal And Anti-Bacterial Activity:

Microbial resistance towards drug creates a very serious problem since last 3 decdes because of the development of resistance many drug are now useless which were very effective (coher 1992:Cunha 1998) The tozic effect produced by these anti-biotic is also reducing their significance. So for the need for a anti-microbial is always being there.

1.3.2 Anti- Cancer Activity:

Semicarbazones are also used is the treatment of cancer against EAC(Enrilich Ascites Carcinoma) The EAC cells are experimental tunormodels used worldwide in cancer research. Semicarbazones are crystalline solids. Useful for the identification of the parent aldehydes/ketones by melting point analysis. Semicarbazone in medicinal and pharmacy ,Application in synthesis and chemical analysis , Application in Co-ordination chemistry.

CHAPTER 2

LITERATURE SURVEY

1. TITLE:Synthesis and reducing power assay of methyl semicarbazone derivatives

AUTHOR:Manmohan Singhal, Arindam Paul P, Hemendra P. Singh

JOURNAL AND YEAR:Journal of Saudi Chemical Society. 2011

INFERENCE:In the present study we have designed a new pharmacophore 'Chalconesemicarbazone' by pharmacophore hybridization approach of drug design. A series of novel chalconesemicarbazones was synthesized and evaluated for their antioxidant activity by reducing power assay. Most of the compounds were found to be potent antioxidants. Free radicals play an important role in various pathological and xenotoxic effects so antioxidant may have protective role in these pathological conditions. Based on the results of reducing power assay 1-[1-(2,4-dihydroxyphenyl)-3-(2-hydroxyphenyl)allylidene]-4-(4-methylphenyl)semicarbazide (compound 18) and 1-[1-(2,5-dihydroxyphenyl)-3-(6-hydroxyphenyl)allylidene]-4-(4-methylphenyl)semicarbazide (compound 21) were the most active lead compounds. It was found that methoxy and hydroxyl substituted chalconesemicarbazones exhibited potent reducing power and unsubstituted compound showed less reducing potential.

2. TITLE:Synthesis and Structure-Activity Relationship Study of Potent TrypanocidalThioSemicarbazone Inhibitors of the Trypanosomal Cysteine Protease Cruzain

AUTHOR:Xiaohui Du, Chun Guo,Elizabeth Hansell, Patricia S. Doyle,Conor R. Caffrey,Tod P. Holler,James H. McKerrow,and Fred E. Cohen

JOURNAL AND YEAR:October3, 2001

INFERENCE:A novel series of potent thiosemicarbazone small-molecule inhibitors of the Trypanosoma cruzi cysteine protease cruzain have been identified. Some of these inhibitors have been shown to be trypanocidal. We initially discovered that

3'-bromopropiophenone thiosemicarbazone (1i) inhibited cruzain and could cure mammalian cell cultures infected with *T. cruzi*. 3'-Bromopropiophenone thiosemicarbazone showed no toxicity for mammalian cells at concentrations that were trypanocidal. Following this lead, more than 100 compounds were designed and synthesized. A specific structure-activity relationship (SAR) was established, and many potent analogues with IC₅₀ values in the low nanomolar range were identified. Eight additional analogues were trypanocidal in a cell culture assay, and this indicates that aryl thiosemicarbazone is a productive scaffold for killing the parasites. Kinetic studies show that these are time-dependent inhibitors. Molecular modeling studies of the enzyme-inhibitor complex have led to a proposed mechanism of interaction as well as insight into the SAR of the thiosemicarbazone series. The nonpeptide nature of this series, small size, and extremely low cost of production suggest this is a promising direction for the development of new antitrypanosome chemotherapy.

3. TITLE:Synthesis and Antibacterial Activity of Some Transition Metal Complexes of Oxime, Semicarbazone and Phenylhydrazone

AUTHOR:Majed M. Hania

JOURNAL AND YEAR:13 May 2009

INFERENCE:Co, Ni and Cu complexes have been prepared by reacting metal chloride with 4-chlorobenzaldehyde oxime, 4-methylbenzaldehyde oxime, 4-nitrobenzaldehyde oxime, 4-chlorobenzaldehyde semicarbazone, 4-methylbenzaldehyde semicarbazone, 4-nitrobenzaldehyde semicarbazone, 4-chloro benzaldehyde phenylhydrazone, 4-methylbenzaldehyde phenyl hydrazone and 4-nitrobenzaldehyde phenylhydrazone and their antibacterial activity have been studied and compared with their ligands against *E. coli* which gave significant results of activity.

4. TITLE:Synthesis, anticonvulsant and toxicity screening of newer pyrimidine semicarbazone derivatives

AUTHOR: Ozair Alam, Pooja Mullick, S.P. Verma, Sadaf J. Gilani, Suroor A. Khan, Nadeem Siddiqui, Waquar Ahsan

JOURNAL AND YEAR: European Journal of Medicinal Chemistry

INFERENCE: A number of N-(4,6-substituted diphenylpyrimidin-2-yl) semicarbazones (4aet) were synthesized and tested for their anticonvulsant activity against the two seizure models, maximal electroshock seizure (MES) and subcutaneous pentylenetetrazole (scPTZ). All the synthesized compounds possessed the four essential pharmacophoric elements for good anticonvulsant activity. Most of the compounds displayed good anticonvulsant activity with lesser neurotoxicity. To assess the unwanted effects of the compounds on liver, estimation of enzymes and proteins was carried out.

CHAPTER 3

AIM AND SCOPE

- The aim of the project is to synthesis and characterization of the semicarbazone.
- Basically semicarbazone are used in various field mainly in chemical and biological field. Synthesis of new semicarbazone always play a new role in both fields like industrial ,agricultural and medicinal.
- Semicarbazone are crystalline solids, useful for the identification parent aldehyde/ketones by melting point analysis.
- In today's decades the semicarbazones are used in treatment because they have anti-viral, antimicrobial , anti-cancer activities, usually mediated through binding copper or iron in cells.
- In future the semicarbazones will be one of the part in medicinal field for acuring cancer.

CHAPTER 4

MATERIALS AND METHODS

4.1. MATERIALS:

The materials used for this experiment are, beaker, test tube, glass rod, watch glass, filter paper, phenyl semicarbazide, N.N. bis (2-chloroethyl) amino benzaldehyde, HCl and ethanol.

4.2 METHOD:

4.2.1 Synthesis Of Semicarbazone:

0.15g of phenylsemicarbazone (0.01mole) and 0.24g of N.N bis (2-chloroethyl) amino benzaldehyde were transferred to separate 100 ml beaker. Add (5-10ml) of ethanol to both the beakers and stir it until they completely dissolved. Now transfer both solution to the 250ml RB flask. The mixture was refluxed for 1-2 hrs and the solid product formed was separated by filtration, washed several times with 50% ethanol and then dried.

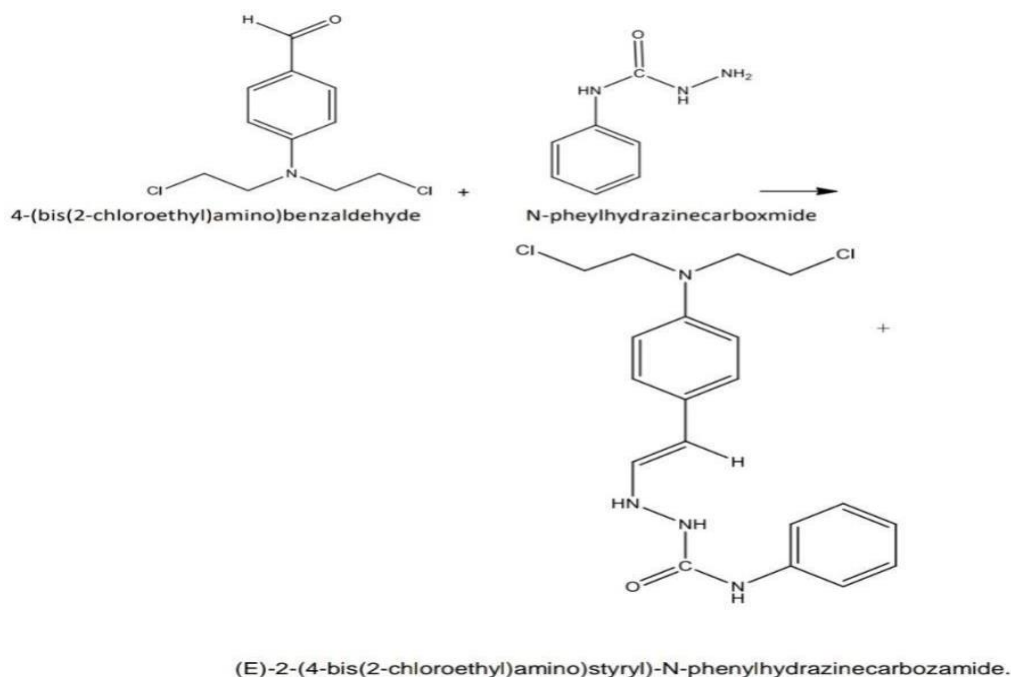


Fig:1.2: Synthesis of Phenyl Semicarbazone

4.3 CHARACTERIZATION TECHNIQUES

4.3.1. Fourier Transform Infrared Spectroscopy (FTIR)

Infrared spectroscopy takes advantage of the way that atoms retain frequencies that are normal for their design. These retentions happen at thunderous frequencies, for example the recurrence of the retained radiation matches the vibrational recurrence.

The energies are impacted by the state of the sub-atomic potential energy surfaces, the majority of the iotas, and the related vibronic coupling. Specifically, in the Born-Oppenheimer and symphonious approximations, for example when the sub-atomic Hamiltonian relating to the electronic ground state can be approximated by a symphonious oscillator in the neighborhood of the harmony sub-atomic math, the full frequencies are related with the typical modes comparing to the sub-atomic electronic ground state potential energy surface.

The thunderous frequencies are additionally connected with the strength of the bond and the mass of the particles at one or the flip side of it. In this way, the recurrence of the vibrations is related with a specific typical method of movement and a specific bond type.

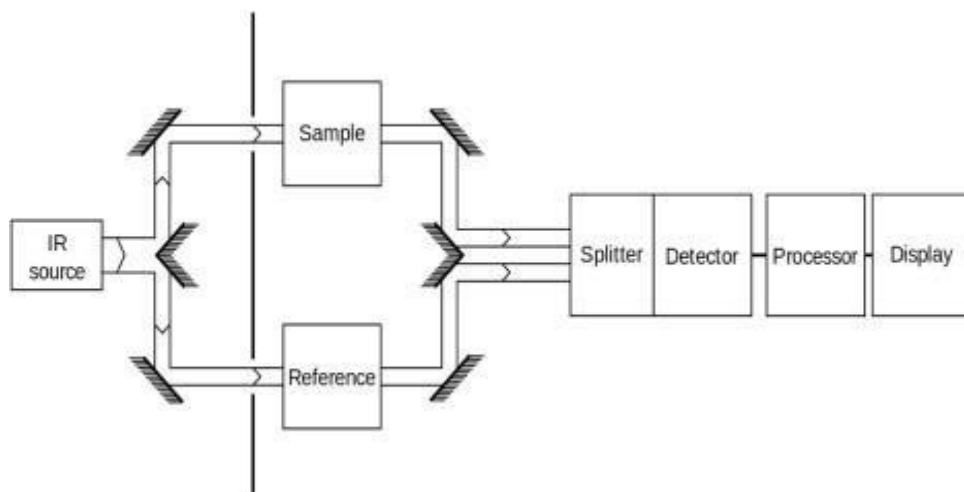


Fig.4.1. FTIR two beam absorption spectrometer

In order for a vibrational mode in a sample to be "IR active", it must be associated with changes in the dipole moment. A permanent dipole is not necessary, as the rule requires only a change in dipole moment. Six of these vibrations involve only the CH₂ portion: symmetric and anti-symmetric stretching, scissoring, rocking, and twisting.



Fig:4.2: FTIR Instrument

4.3.2 Nuclear Magnetic Resonance (NMR)

When white light is given to the surface of an atom, molecule, or other object, spectrum is seen from it which is called spectroscopy. We can say that the spectrum that is found here depends entirely on the defined atom and molecule or some other substance. And spectroscopy is also used in physics, chemistry and biology. Red has the lowest frequency. While violet has the shortest wavelength and the highest frequency.

When it comes from the sun's rays it is an ultraviolet wave. The shortest wavelength from above is that of gamma radiation (1 picometer). A total of 22 types of spectroscopy are found. Of which here we will talk about nuclear magnetic resonance spectroscopy. Strong state atomic attractive reverberation (NMR) is a spectroscopic technique that gives results delicate to the nearby atomic climate and is utilized to concentrate on a wide assortment of carbon materials, including peat, humic substances, coal, coke and charcoal. It consequently provides an itemized comprehension of the synthesis of plasma blacks at the nuclear scale is accordingly of interest focused

on their likely viable applications and the ionization improvement of their creation strategies. Subsequently the more extensive test approach utilized in the NMR investigation of these carbon substances utilizes cross polarization (CP) to build the polarization of the carbon cores by their collaboration with them bountiful 1 H cores. Commitments are effectively separated in light of or because of gatherings, normally in the scope of 0 to 90 ppm for aliphatic and 110-160 ppm for sweet-smelling gatherings.

Cross Polarization experiments are less time consuming compared to experiments based on the direct polarization (DP) of the ^{13}C nucleus, as the relative longitudinal relaxation time in CP is associated with an abundant 1 H nucleus (T_{1H}). Which means that the spectral intensity in the ^{13}C NMR spectrum recorded with CP will be affected by differences in the rate of polarization transfer in the chemically differentiated ^{13}C nucleus from 1 H.

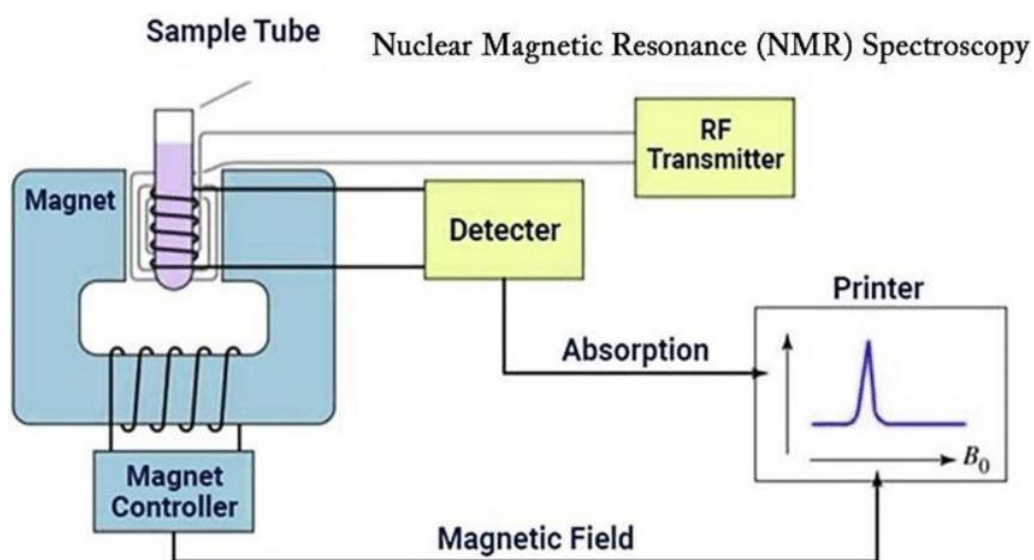


Fig.4.3. NMR Spectroscopy Instrumentation

It possible to determine the fraction of carbon atoms with sp^2 or sp^3 hybridization, observation of chemical reduction of graphene oxide, study of the relationship between chemical transformation in heat-treated carbon and structural parameters, the study of relaxation processes of ^{13}C nuclei interacting with

paramagnetic centres in nanocarbons and many others. the solid-state ^{13}C NMR spectroscopy was used to study plasma black and carbon deposits produced by thermal cracking of natural gas using different types. i.e., different types of plasma reactors.

Assume there is a core wherein a proton is available while the proton addresses the twist second. This makes the proton go about as a little magnet. There should be a clear rule for deciding if atomic attractive reverberation is dynamic for a particle. the atomic attractive reverberation will be dynamic provided that the twist quantum number of a particle is more noteworthy than nothing. What's more, if the quantum number becomes zero then atomic attractive reverberation won't be initiated. This will be conceivable when the nuclear number and the nuclear mass become equivalent for example their twist quantum will be zero. Consequently, one might say that atomic attractive reverberation will be initiated just when the nuclear number and the nuclear mass are not equivalent.

This recommends that the core ought to contain all the data relating to the core that has protons and neutrons in which the proton has a positive charge and the neutron doesn't have a charge. At the point when the electron spins around the core and the actual electron additionally rotates in its place for example the actual electron has some twist. The actual electron can be clockwise or against clockwise twist second. While the other word is attractive, it infers that the attractive field additionally has an impact.

4.3.3 UV Spectroscopy

UV/Vis spectroscopy is regularly utilized in logical science for the quantitative assurance of various analytes, like change metal particles, profoundly formed natural mixtures, and organic macromolecules. Spectroscopic investigation is usually completed in arrangements yet solids and gases may likewise be considered.



Fig:4.4: UV Instrument

Arrangements of progress metal particles can be hued (i.e., retain noticeable light) of the synthetics included. Around here of the electromagnetic range particles and atoms go through electronic changes. Ingestion spectroscopy is integral to fluorescence managing changes from the invigorated state to the ground state.

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Fourier Transform Infrared Spectroscopy (FTIR)

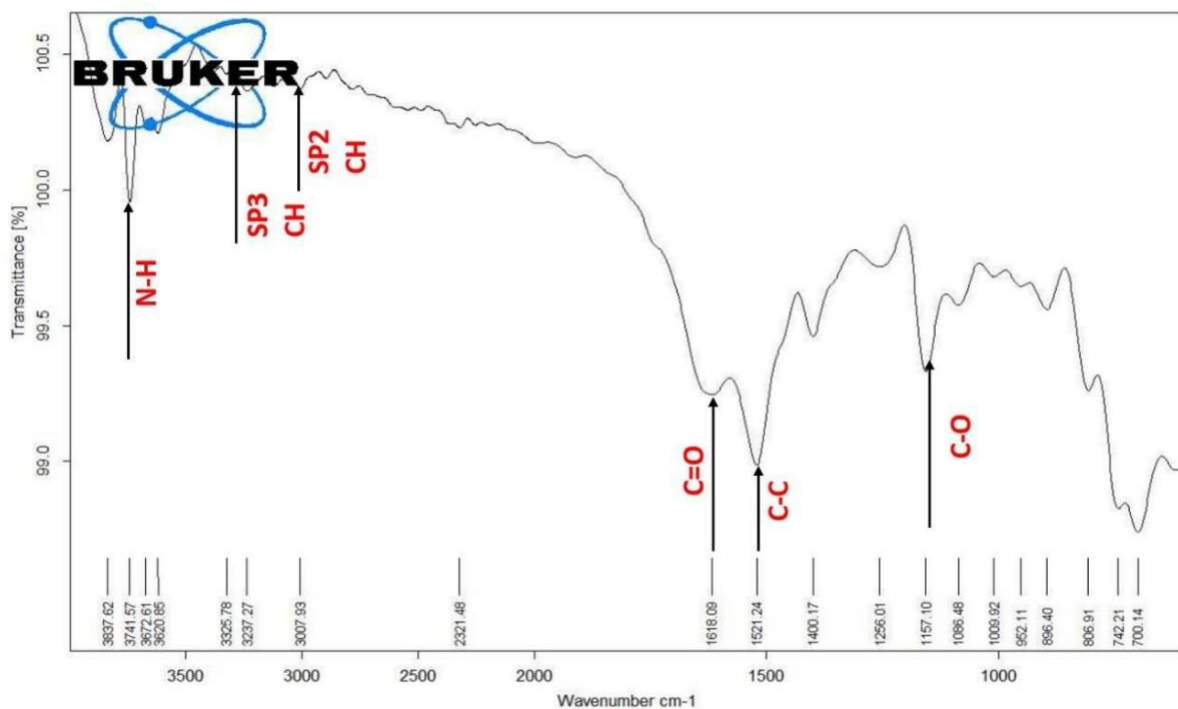


Fig:5.1: Spectral data of FTIR

The measurement of infrared radiations interaction with matter through adsorption, emission, or reflection is known as INFRARED spectroscopy. It is also called IR spectroscopy or vibrational spectroscopy. It is used to research and classify chemical compounds or functional groups that occur in solid, liquid, or gaseous state.

- N-H Stretching at 3741.57
- C-C Stretching at 1521.24
- C=O stretching at 1618.09
- C-O Stretching at 1157

5.2 Nuclear Magnetic Resonance Spectroscopy (NMR)

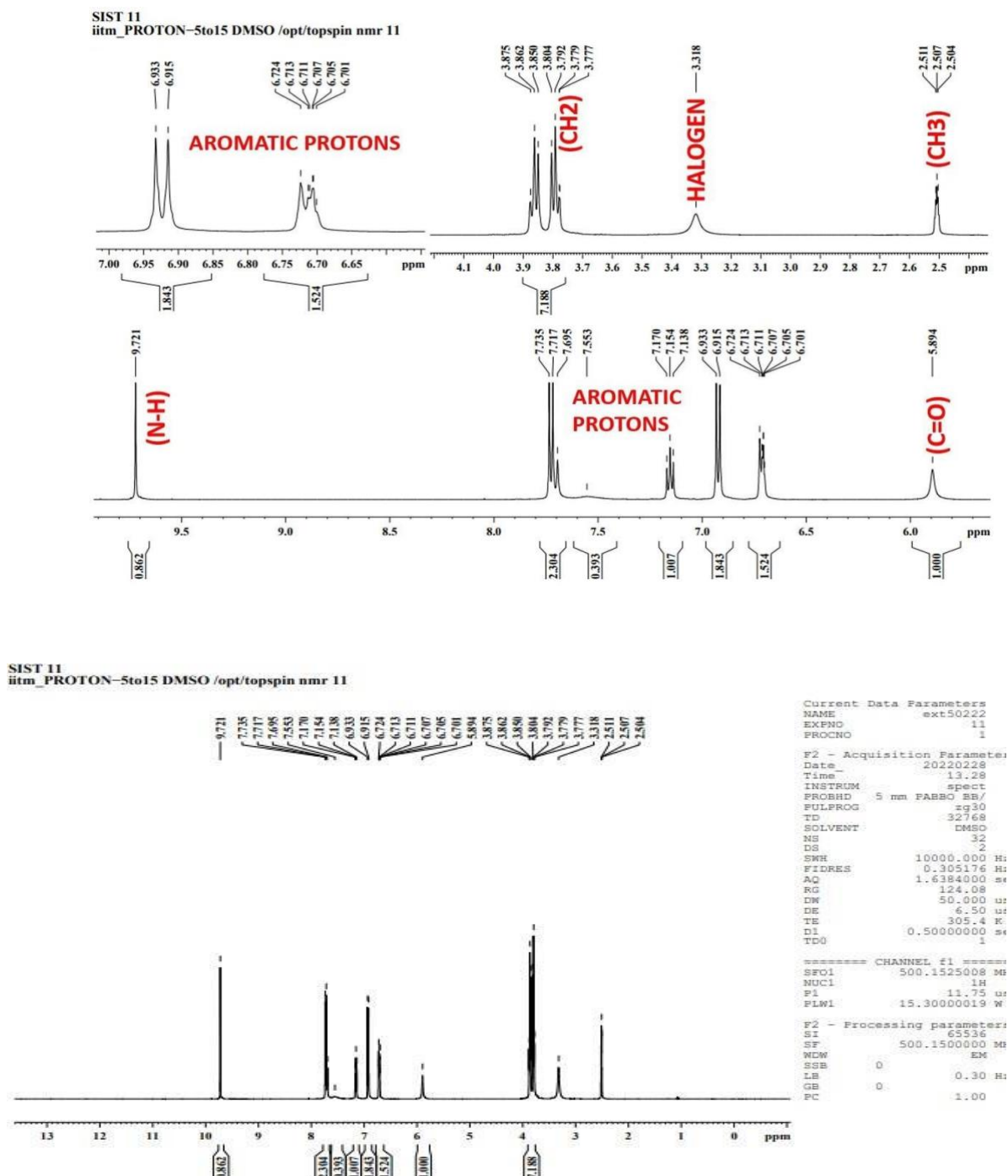
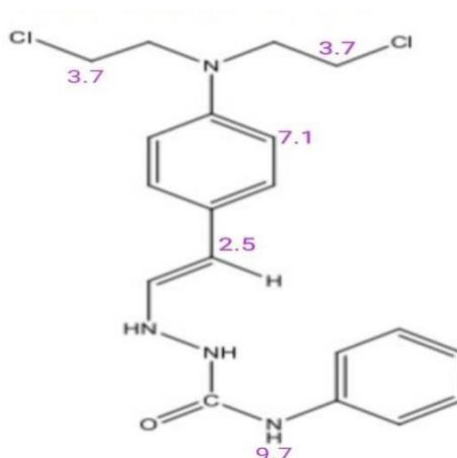


Fig.5.2: Spectral data of NMR

SEMICARBZONE



The NMR Spectrum for semicarbazone are shown in the figure 5.2. the singlet peak obtained at 2.5 corresponds to CH₃, at 3.3 corresponds to Halogens, the peaks from 3.9-3.7 corresponds to CH₂, 6.75-6.65 corresponds to Aromatic protons, the peak at 6.0 is C=O, the peaks from 8.0-6.5 is aromatic protons the peak at 9.7 is N-H peak. These obtained peaks shows that the compound is semicarbazone.

- Three peak at 2.5ppm may be due to CH₃ group
- One peak at 3.3ppm is Halogen group
- Seven peaks at 3.7ppm may be due to CH₂ group
- One peak at 5.8ppm may be C=O group
- One peak at 9.7 ppm may be N-H group

5.2 UV Spectroscopy

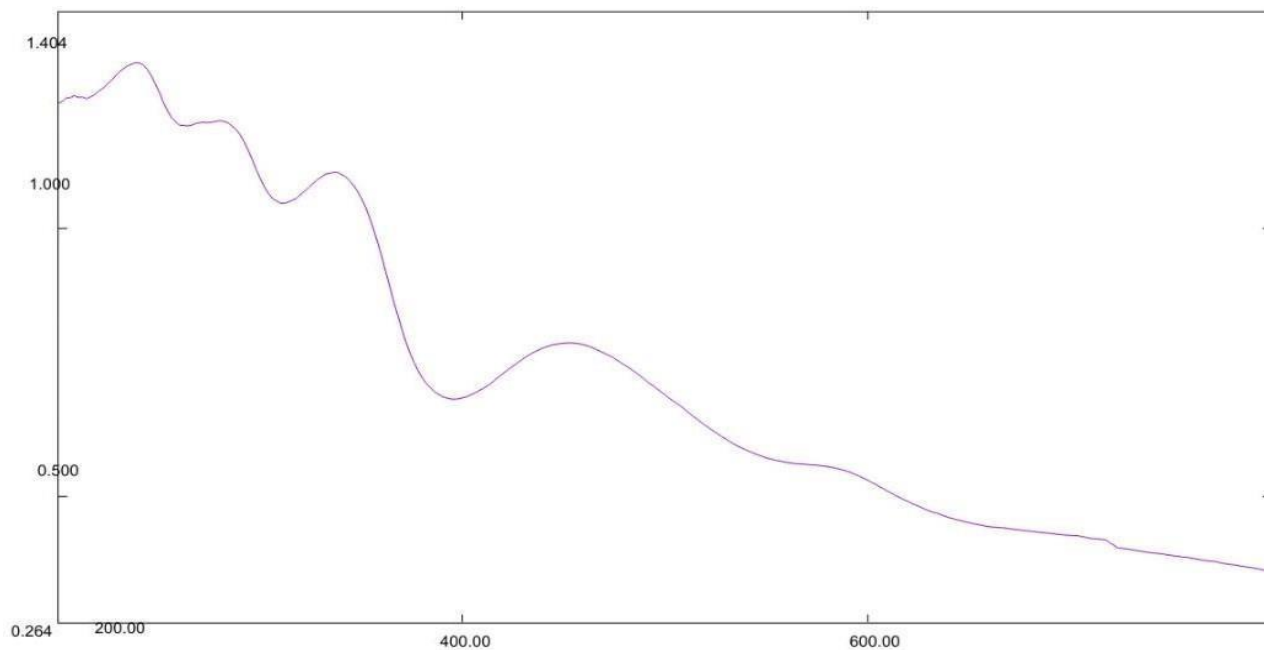


Fig:5.4: Spectral data of UV

- The UV-Visible spectroscopy refers to the adsorption or reflectance spectroscopy used for the quantitative determination of different analytes. The UV-Visible spectra of the compound shows the maximum adsorption peaks at 400nm. This maximum adsorption of phenyl semicarbazone was assigned to the($n- \pi^*$) transition. The UV-Visible spectrum to phenyl semicarbazone is shown in Fig:5.4

CHAPTER 6

CONCLUSION

A new semicarbazone derivative, phenyl semicarbazone with the molecular formula $C_{12}H_{22}Cl_2N_4O$ is synthesized by using N,N-bis (2-chloroethyl) aminobenzaldehyde, from the collected data of IR and UV and NMR represents and its characterization.

The maximum peak range of IR is $700-4000\text{cm}^{-1}$.

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