



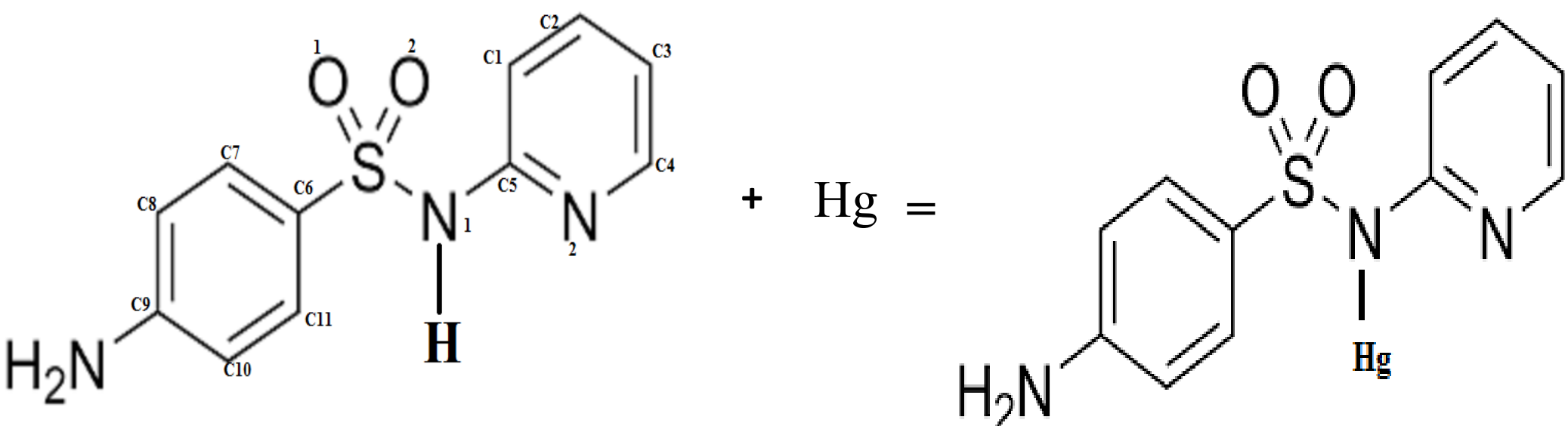
# Synthesis, Spectroscopic characterization and Powder XRD of transition metal complex (mercury) of Sulfapyridine

Vijay D. Bodarya, Tushar M. Lukhi, Ankit D. Vaghasiya, Vibhutiba P. Jethwa, Vaishnavi J. Darji, Dr. (Prof.) U.H. Patel  
Department of Physics, Sardar Patel University, Vallabh Vidyanagar - 388 120, Gujarat, India



## Abstract

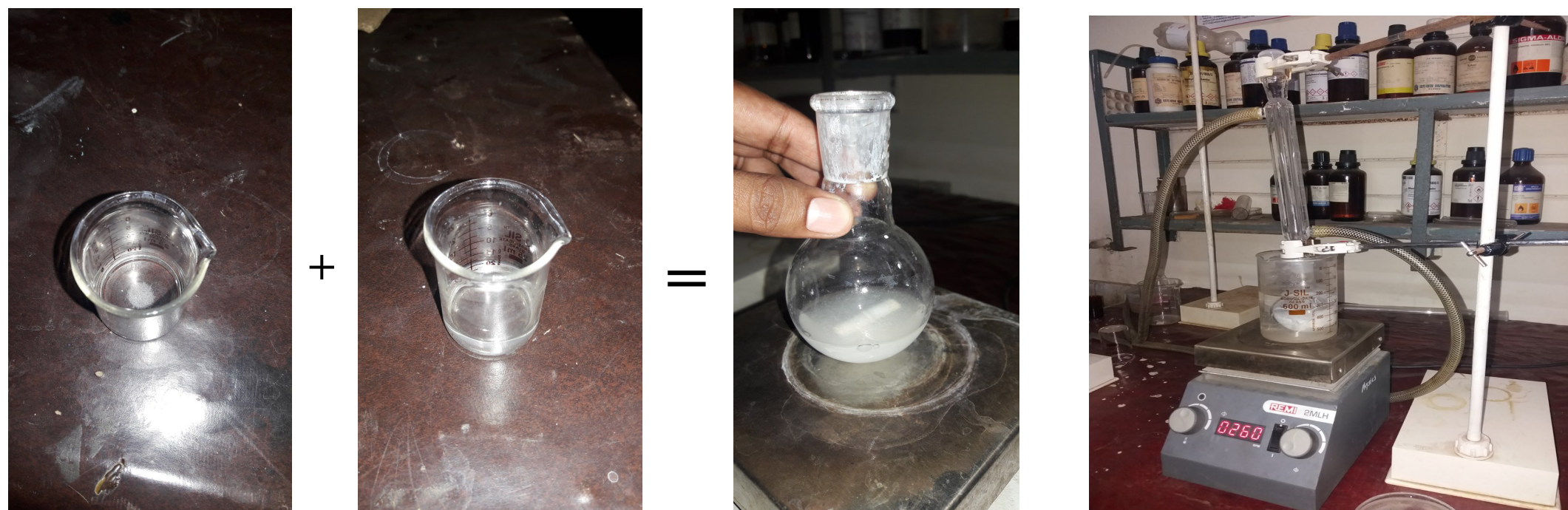
Many drugs possess modified pharmacological and toxicological potentials when administered in the form of their metal complex. All sulfonamides have one free amino group, which offers an extendable chemistry and makes them interesting potential ligand. Sulfapyridine (SPY) is one of the significant members of sulfa family. Sulfapyridine is widely used as an antibacterial, antithyroid and antidiabetic drug. To look for a modified and alternative of SPY, we synthesize its metal (mercury) complex by chemical root and reflux method and characterized by few spectroscopic techniques like FT-IR, NMR and PL. Powder XRD data of the metal (mercury) complex of SPY is recorded using Rigaku Ultima IV X-ray diffractometer at Department of physics.



## Synthesize of metal complex

### Reflux method

The title molecule Hg (II) acitate complex of Sulphapyridine ( $C_{11}H_{11}N_3O_2S$ ) has been synthesize by reflux method. The solution of Hg (II) acitate (1 mmol) in the distilled water was added in the solution of sulphapyridine (2 mmol) in 25 ml methanol and the mixture was reflux for 2 hours. The white precipitate was formed, filtered and washed with water and methanol successively and dried in desiccator.



## IR Spectra

In order to clarify the mode of bonding and the effect of the metal ion on the ligand, the IR spectra of the free ligand and the metal complexes are studied and assigned on the basis of careful comparison of their spectra with that of free ligand. The IR spectra of the complex taken in the region  $4000 - 400 \text{ cm}^{-1}$  are compared with those of the free ligand. Based on some general references and previous studies of complexes with sulphonamide, a tentative assignment of the most important bands is given in Table 1.

The symmetric and antisymmetric bands assigned to  $\nu(\text{NH}_2)$  in the ligand ( $3416.46$  and  $3310.02 \text{ cm}^{-1}$ ) are shifted to higher wave numbers in complex ( $3454.07$  and  $3379.39 \text{ cm}^{-1}$ ), indicating the consequences of the change in their hydrogen bonding scheme. The effect of the coordinated metal is also noticeable on the  $\nu(\text{S-N})$  shifted by about  $15-20 \text{ cm}^{-1}$  to higher wave numbers in complex, this shifting to higher frequencies is in accordance with the shortening of the (S-N) bond length, which has been observed in the crystal of complex.

Table. 1 Characteristics IR bands ( $\text{cm}^{-1}$ ) of the spectra of sulphapyridine and Hg-sulphapyridine complex.

Assignment	SPY	Hg-SPY
N-H	3106.55	-
$\nu_{\text{as}}(\text{NH}_2)$	3416.46	3454.07
$\nu_{\text{s}}(\text{NH}_2)$	3310.02	3379.39
$\Delta(\text{NH}_2)$	1637.81	1624.01
U-phenyl ring	1585.99, 1523.26	1592.96, 1545.47
$(\text{SO}_2)_{\text{as}}$	1323.28, 1302.72	1330.92, 1350.13
$(\text{SO}_2)_{\text{sy}}$	1181.97	1180.55
U(S-N)	998.61, 947.03	1020.81, 969.97

The peak for the sulphonamido (S-N) group in the free ligand at around  $3106.55 \text{ cm}^{-1}$ , is not present in the spectra of the complex, confirming the deprotonation of the  $-\text{SO}_2\text{NH}-$  moiety. All the changes suggest binding of mercury atom to the sulfonamide nitrogen.

Fig. IR spectra of Sulphapyridine

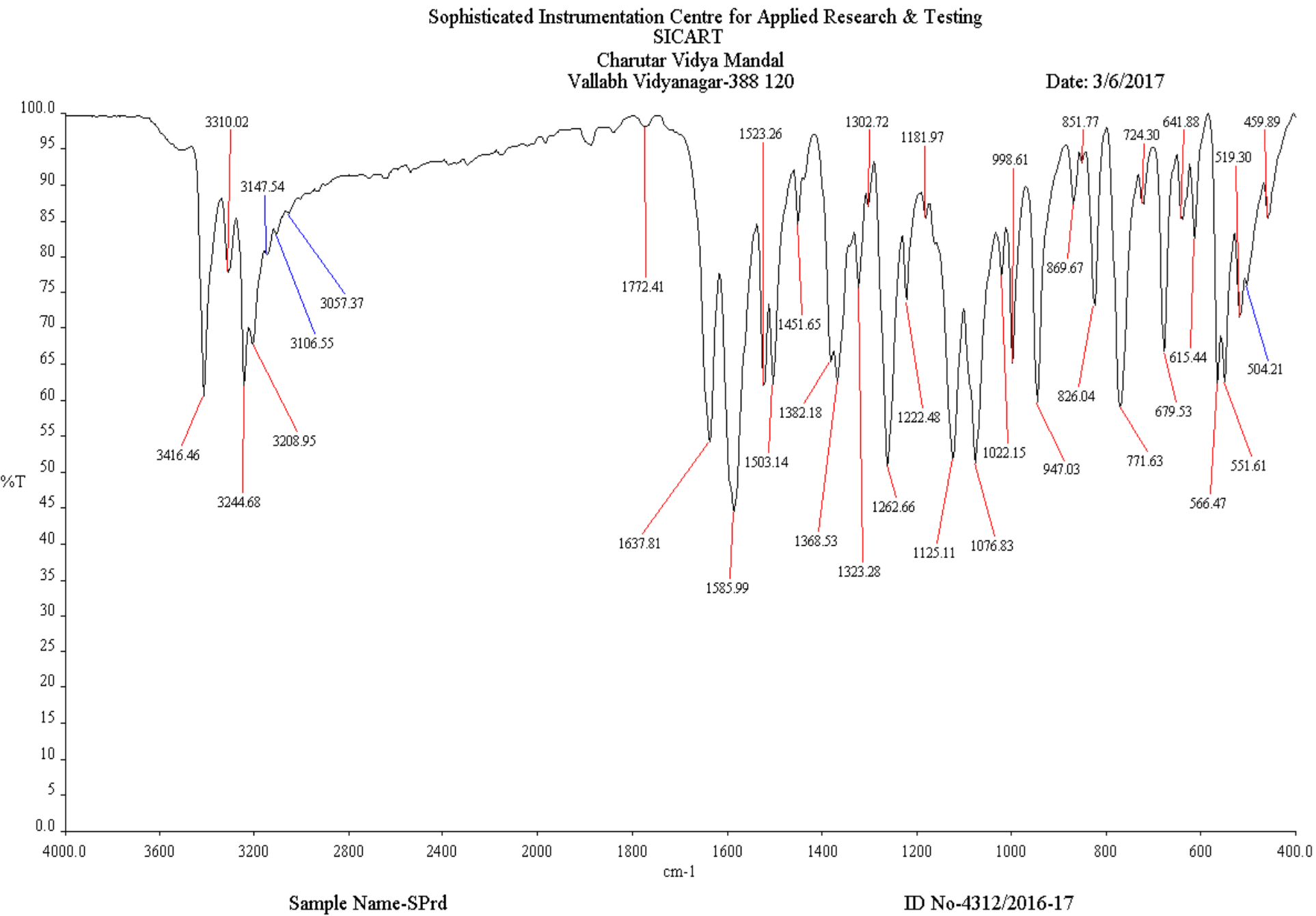
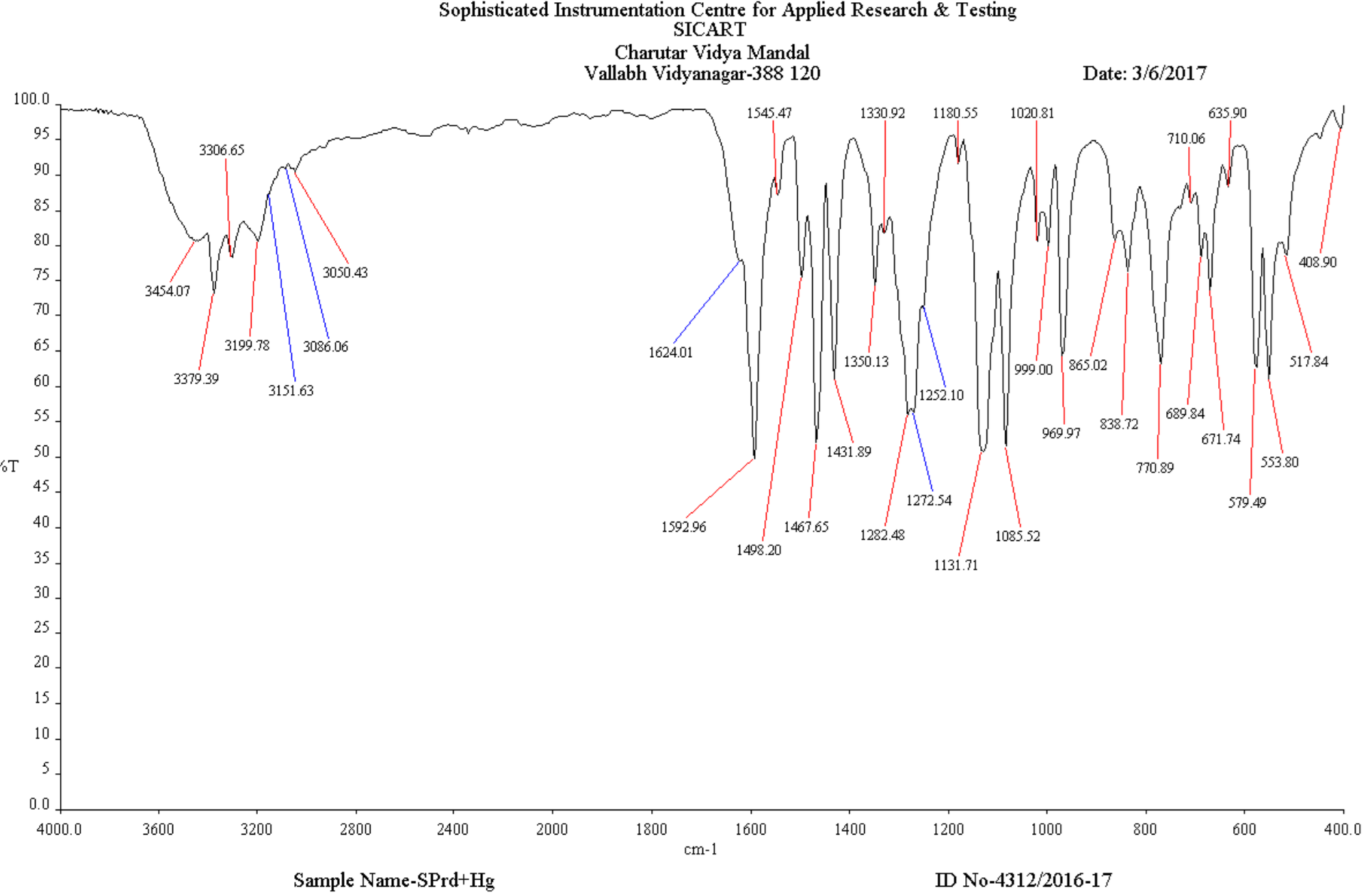


Fig. IR spectra of Hg-Sulphapyridine



## Introduction

- The development of sulphonamides is one of the most fascinating and informative chapter in medical chemistry, highlighting the roles of skilful planning and serendipity in the drug research. The discovery of the antibacterial activity of sulphonamides in the early 1930's was the beginning of the present era of chemotherapy. The subsequent recognition of the relationship between the chemical structure of this compound and their pharmacological response brought into sharp focus the potential power of molecular modification in the drug design [1].
- The sulphonamides [2] represent a large class of antibiotics that have multiple clinical uses. The sulphonamides are the first effective antibiotics to be introduced into clinical medicine and have been in use continuously since the 1930's.
- Sulphapyridine is a sulpha drug. It is used to help control dermatitis herpetiform in (Dhring's disease), a skin problem. It may also be used for other problems. Sulfapyridine is a short-acting sulphonamide antibiotic drug.

## NMR Spectroscopy

Nuclear magnetic resonance spectroscopy, most commonly known as NMR spectroscopy, is a research technique that exploits the magnetic properties of certain atomic nuclei. It determines the physical and chemical properties of atom or the molecule in which they are contained. NMR spectroscopy is an analytical chemistry technique used in quality control and research for determining the content and purity of sample as well as its molecular structure.

The NMR spectrum consists of series of peaks which correspond to different applied field strength and each peak means a set of nuclei at the same magnetic environment. The technique is useful for structure identification of organic, inorganic and polymer compound. In the present study,  $^1\text{H}$  NMR spectra are recorded on a Bruker Avance 111 spectrometer at SICART, Vallabh Vidyanagar.

Due to this complex being insoluble in less polar solvents,  $^1\text{H}$  NMR spectra is recorded from a solution of the complex in  $d^6$ -DMSO. The chemical shifts are expressed in ppm relative to internal TMS. The NMR data is presented in Table. 2.

The data shows that while the ligand is present in the complex, they only display very small shifts in comparison to the free ligand. The most notable feature is the proton magnetic resonance spectrum of the ligand shows a broad singlet at  $10.8 \text{ ppm}$ , which may be assigned to the sulphonamide NH proton. The absence of this proton signal in this spectra of the mercury complex indicates that sulphonamide NH group is deprotonated during complex formation.

Table. 2  $^1\text{H}$  NMR shift assignments of sulphapyridine and the Hg-sulphapyridine complex in DMSO- $d^6$

Assignment	$^1\text{H}(\text{SPY})$	$^1\text{H}(\text{Hg-SPY})$
N(1)-H	10.983	-
C(1)-H/C(3)-H	8.101	8.197
C(2)-H	7.072	7.081
C(7)-H/C(11)-H	7.627	7.657
C(8)-H/C(10)-H	6.566	6.573
$\text{NH}_2$	5.968	5.934

Fig. NMR spectra of Sulphapyridine.

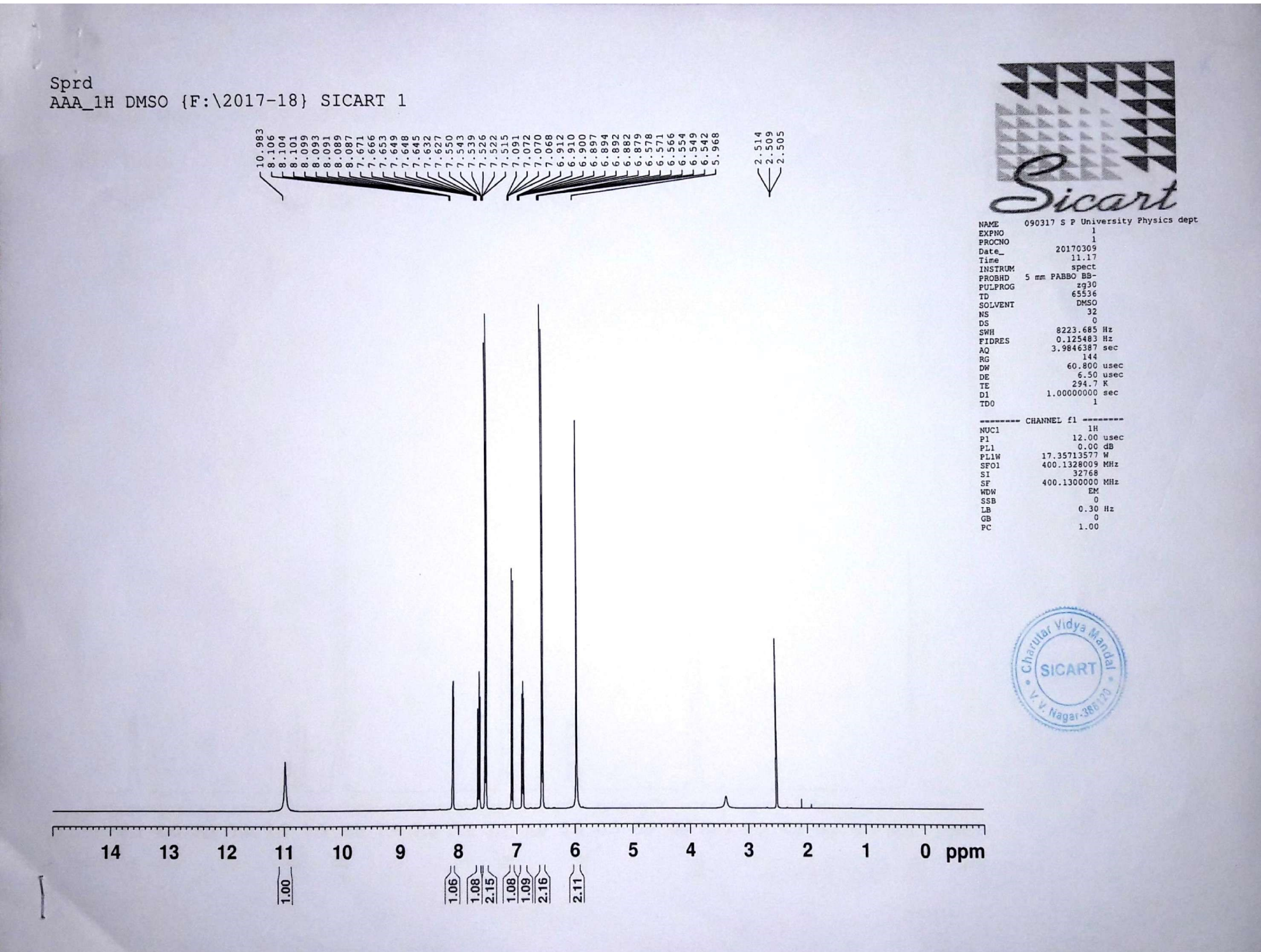
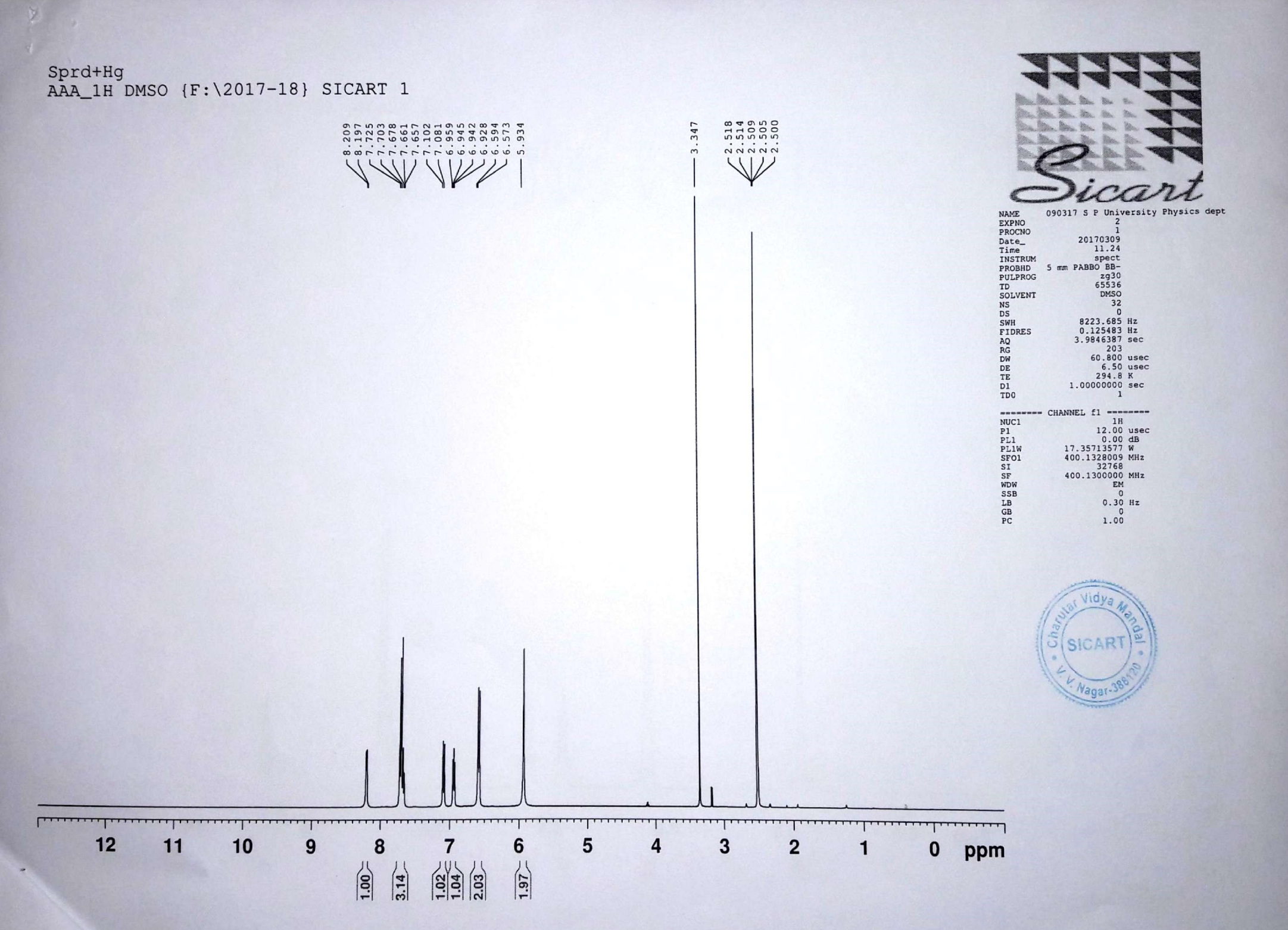


Fig. NMR spectra of Hg-Sulphapyridine



## Feature scope

- Single crystal of the same material can be grown by the slow evaporation method.
- Microbial Inhibitory Concentration (MIC) can be carried out.
- This molecule can be dock with the protein.

## ACKNOWLEDGEMENT

- We are very thankful to our project guide Dr. U. H. Patel for providing her constant guidance. We are thankful to SICART for carried out our data in short time.
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## Why X-ray Study?

- IR and NMR data can not predict 100% accuracy of the molecular structure. Being an unique technique to investigate the 3 - dimensional structure of a molecule, it necessity.
- X-ray crystallography** is a technique used for determining the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder, and various other informations [3].

## POWDER XRD DATA

Table. (A) Sulphapyridine			Table. (B) Hg-Sulphapyridine	
No.	2-theta(deg)	Int. I (counts deg)	2-theta (deg)	Int. I (counts deg)
1	11.680(5)	242(5)	10.633(4)	165(2)
2	15.271(9)	185(6)	16.702(5)	42.5(19)
3	16.011(4)	659(16)	17.301(15)	14.6(16)
4	16.354(2)	817(12)	18.324(13)	36(3)
5	19.9376(19)	199(5)	19.043(9)	77(3)
6	22.351(6)	196(7)	21.129(7)	52(4)
7	23.285(6)	188(13)	23.259(8)	49(2)
8	23.471(8)	276(13)	23.787(12)	56.2(19)
9	25.018(3)	199(41)	26.05(4)	21(4)
10	25.109(5)	133(40)	26.726(10)	98(4)
11	32.959(15)	101(3)	33.34(3)	17(2)
12	38.812(5)	61(2)	37.33(3)	8(2)
13	45.143(7)	65(3)	39.42(3)	16(3)
14	47.25(2)	54(2)	43.06(5)	29(5)

Fig. Graph of Intensity (counts) versus 2θ (degree) for sulphapyridine

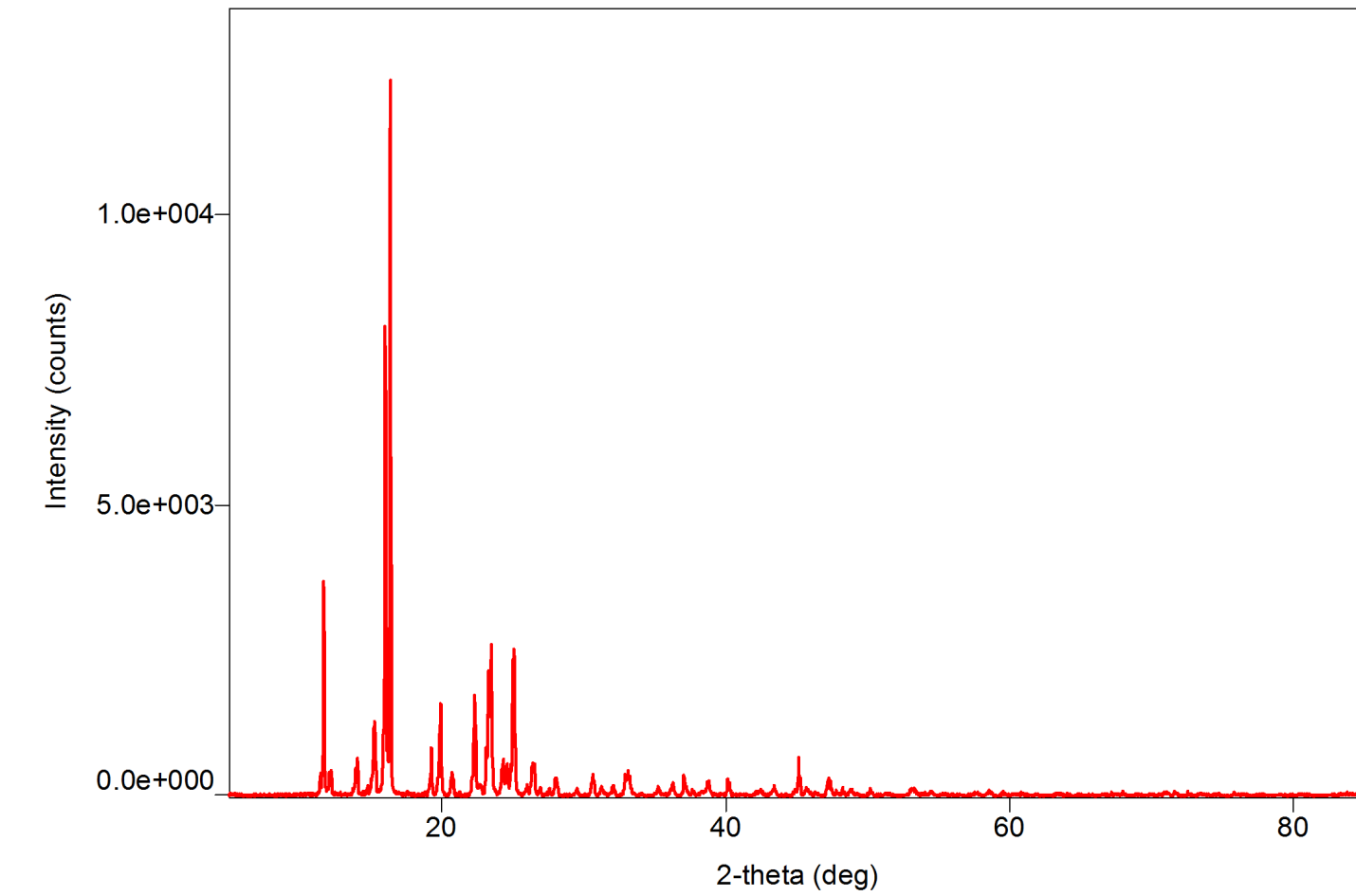
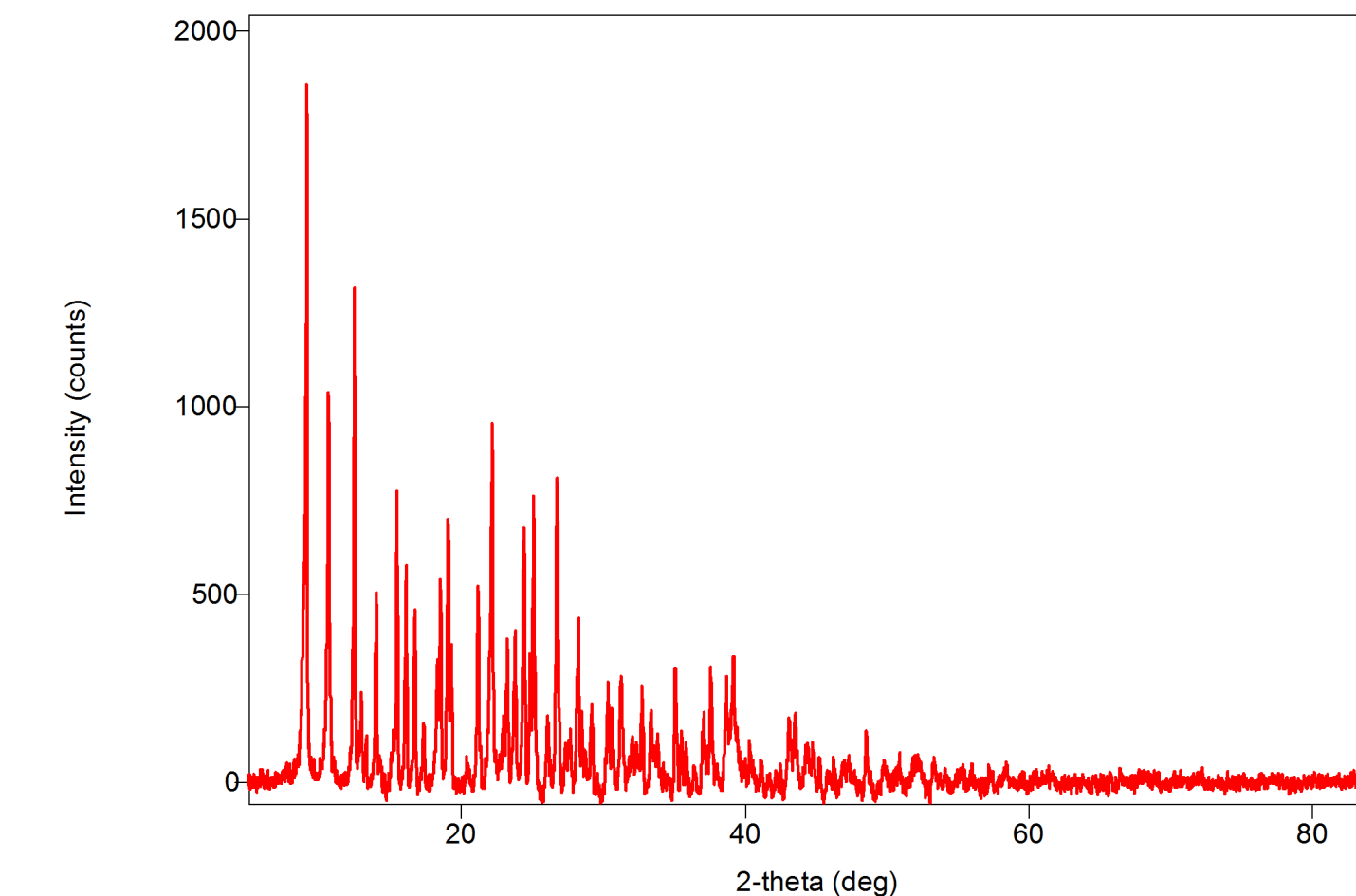


Fig. Graph of Intensity (counts) versus 2θ (degree) for Hg-sulphapyridine



## CONCLUSION

- From the Infrared (IR) spectra, it can be observed that the N-H bond is absent in the complex of Hg-Sulphapyridine and it has been observed a bond around  $408 \text{ cm}^{-1}$  in the metal complex. This clearly indicate the binding of metal with sulphonamido nitrogen.
- From Nuclear Magnetic Resonance (NMR), our prediction reveals that deprotonation occur in the metal complex since N-H peak which is present in the free ligand is absent in the metal complex.
- These two spectroscopic characterization clearly indicates the binding of metal with ligand.
- From powder XRD, it can be observed that the intensity &  $2\theta$  values varies in free ligand & metal complex.

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