

Versatile Three Component Procedure for Combinatorial Synthesis of Biologically Relevant Scaffold Spiro[indole-thiazolidinones] under Aqueous Conditions.

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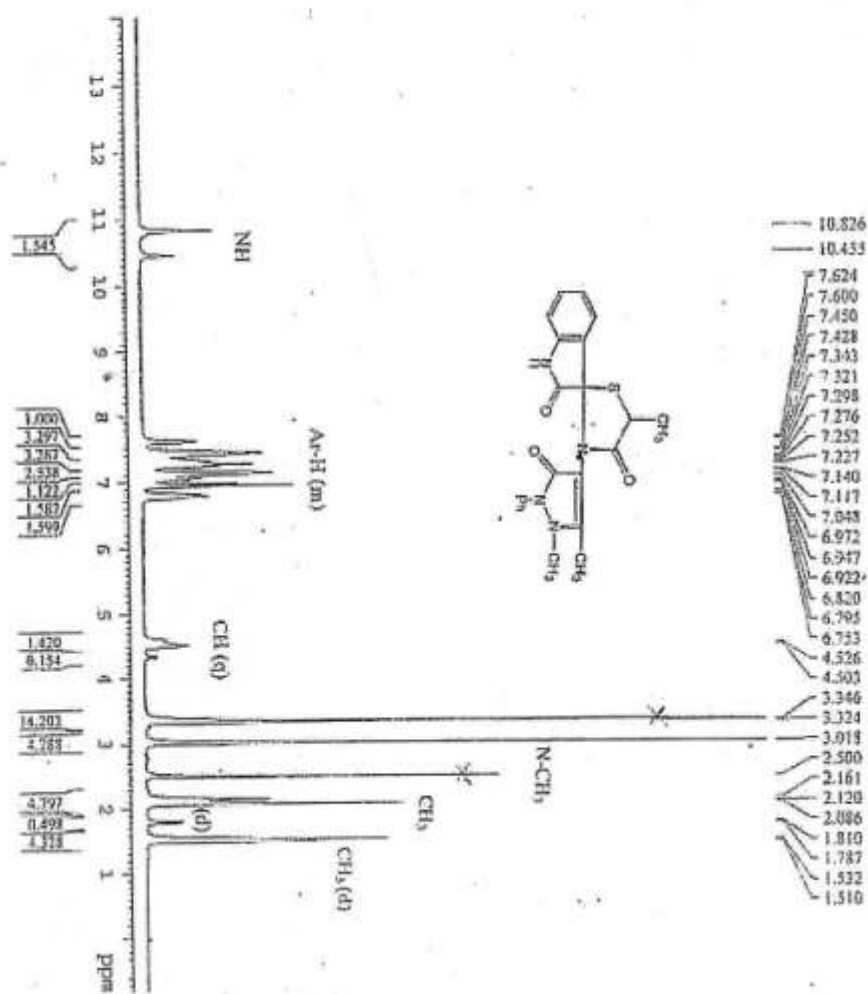
Supporting Information

Experimental: Melting points were determined on a Toshniwal apparatus. The purity of compounds was checked on thin layers of silica gel in various non-aqueous solvent systems, for e.g. benzene: ethylacetate (9:1), benzene: dichloromethane (8:2). IR spectra (KBr) were recorded on a Magna FT IR–550 spectrophotometer and ¹H NMR and ¹³C NMR spectra were recorded on Bruker DRX-300 using CDCl₃ at 300.13 and 75.47, respectively. TMS was used as internal reference. DART Mass spectra of representative compounds were recorded on YOKUDELNA_ES+_2000 spectrometer and EIMS were recorded on Kratos Mass spectrometer. The ultrasound-assisted reactions were carried out in and ultrasonic bath (Bandelin Sonorex) operating at 230 V generating 33 KHz output frequency. The location at which the intensity of ultrasound is maximum checked by Weissler reaction. HIU irradiation was provided by the ultrasonic processor probe system (Processor SONOPROS PR-1000MP, OSCAR ULTRASONICS made) operating at 20 KHz, 750W with 6mm/12 mm tip diameter probes. The synthesized compounds were compared with the authentic samples, prepared by the conventional method.

Typical procedure for preparation of 3'-(2,3-dihydro-1,5-dimethyl-3-oxo-2-phenyl-1H-pyrazol-4-yl)-5'-methyl-spiro[3H-indole-3,2'-thiazolidine]-2,4'(1H)-dione (4a) under HIU irradiation using sonicator:

A mixture of indole-2, 3-dione (3 mmol), 4-aminoantipyrine (3 mmol) and 2-mercaptopropionic acid (3.5 mmol) and cetyltrimethylammonium bromide (20 mol%) in water (10ml) were taken in a flask. The flask was attached to a 12 mm tip diameter probe and the reaction mixture was sonicated for the specified period at 50% power of the processor at 4 s pulse mode. At the end of the reaction period, TLC was checked and the flask was detached from the probe and the content was transferred into a beaker. The formed precipitated product was flittered and washed with water and ethanol to afford the pure white crystalline product (**4a**).

Fig.-1 ¹H NMR of 4a

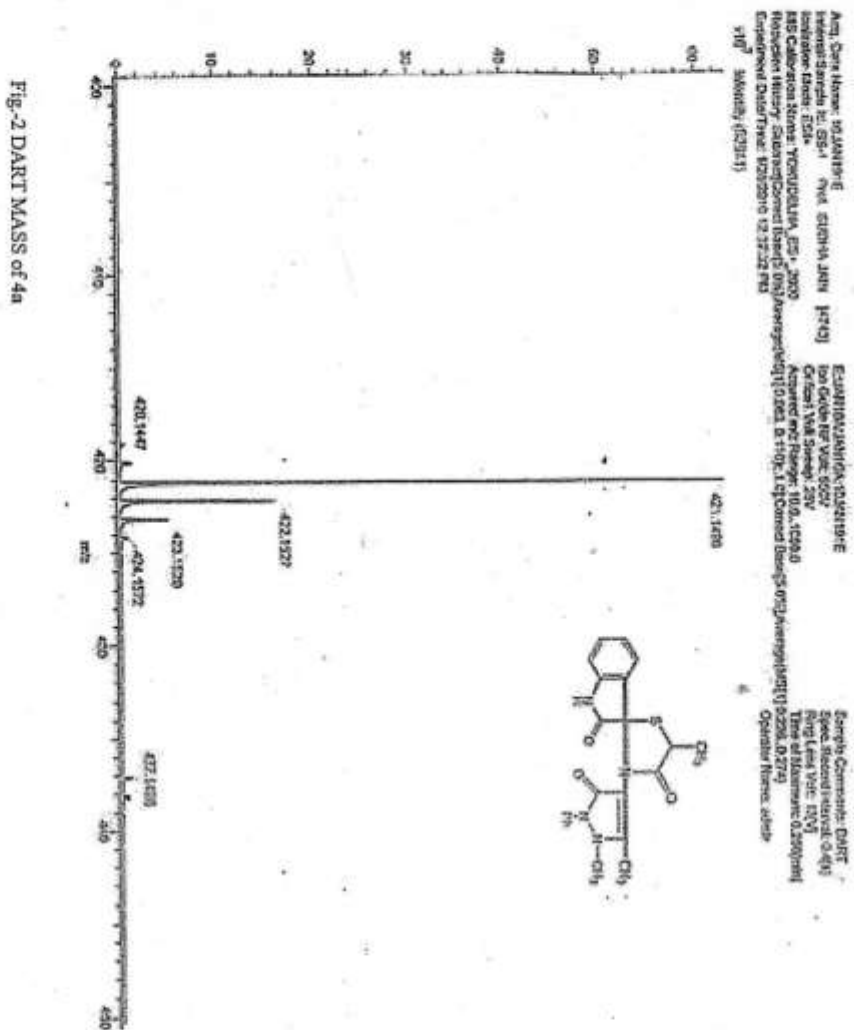


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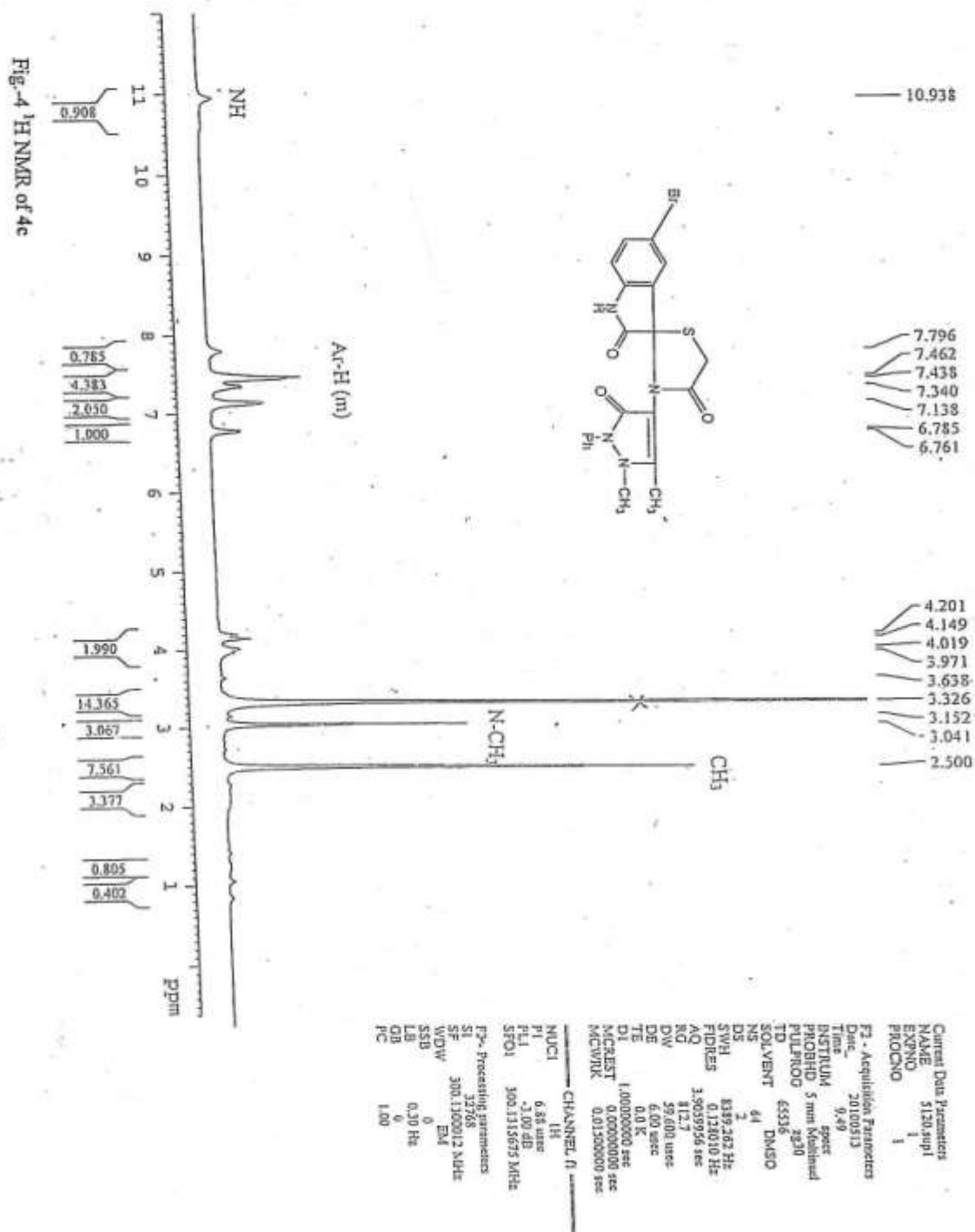
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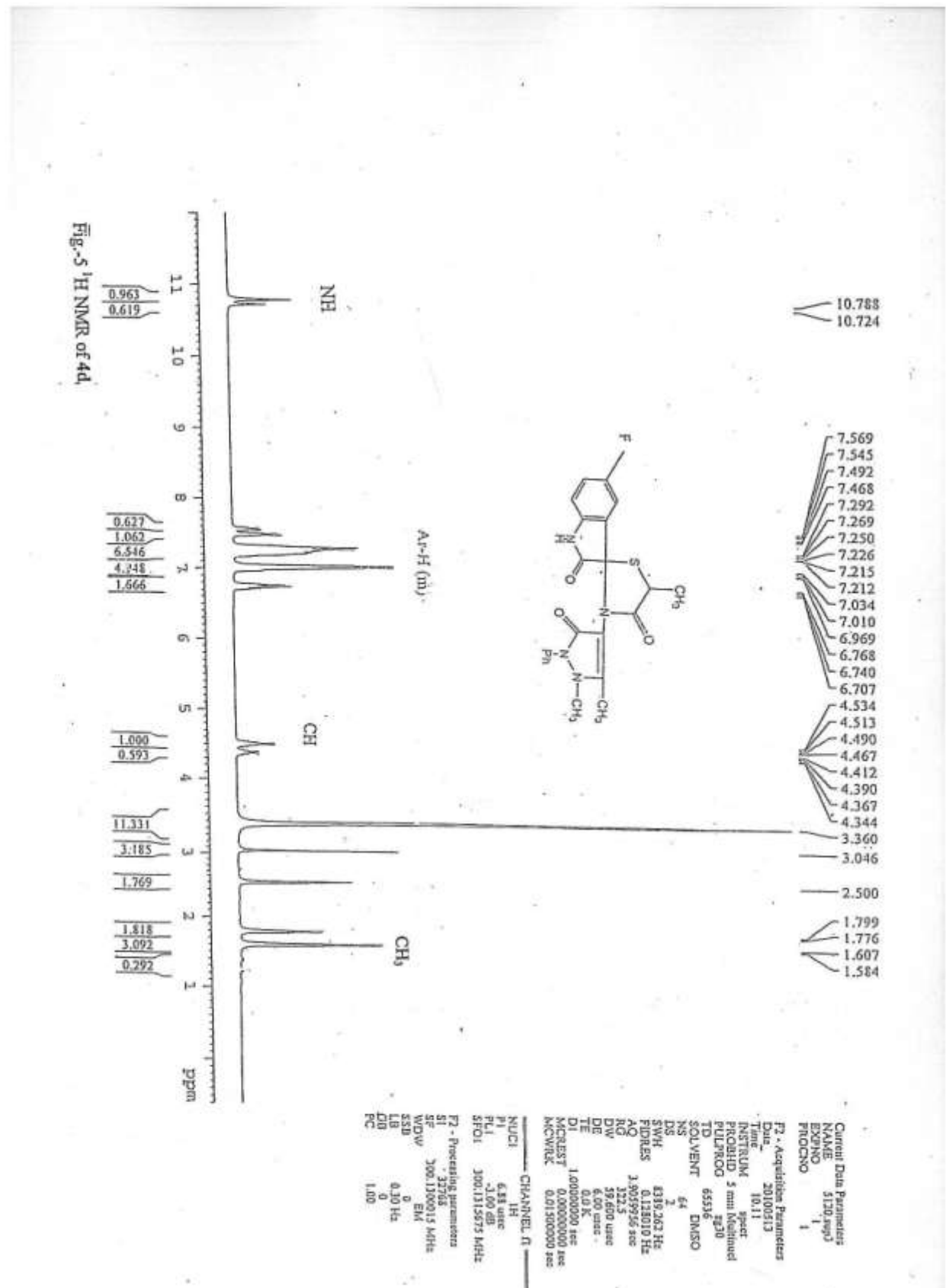
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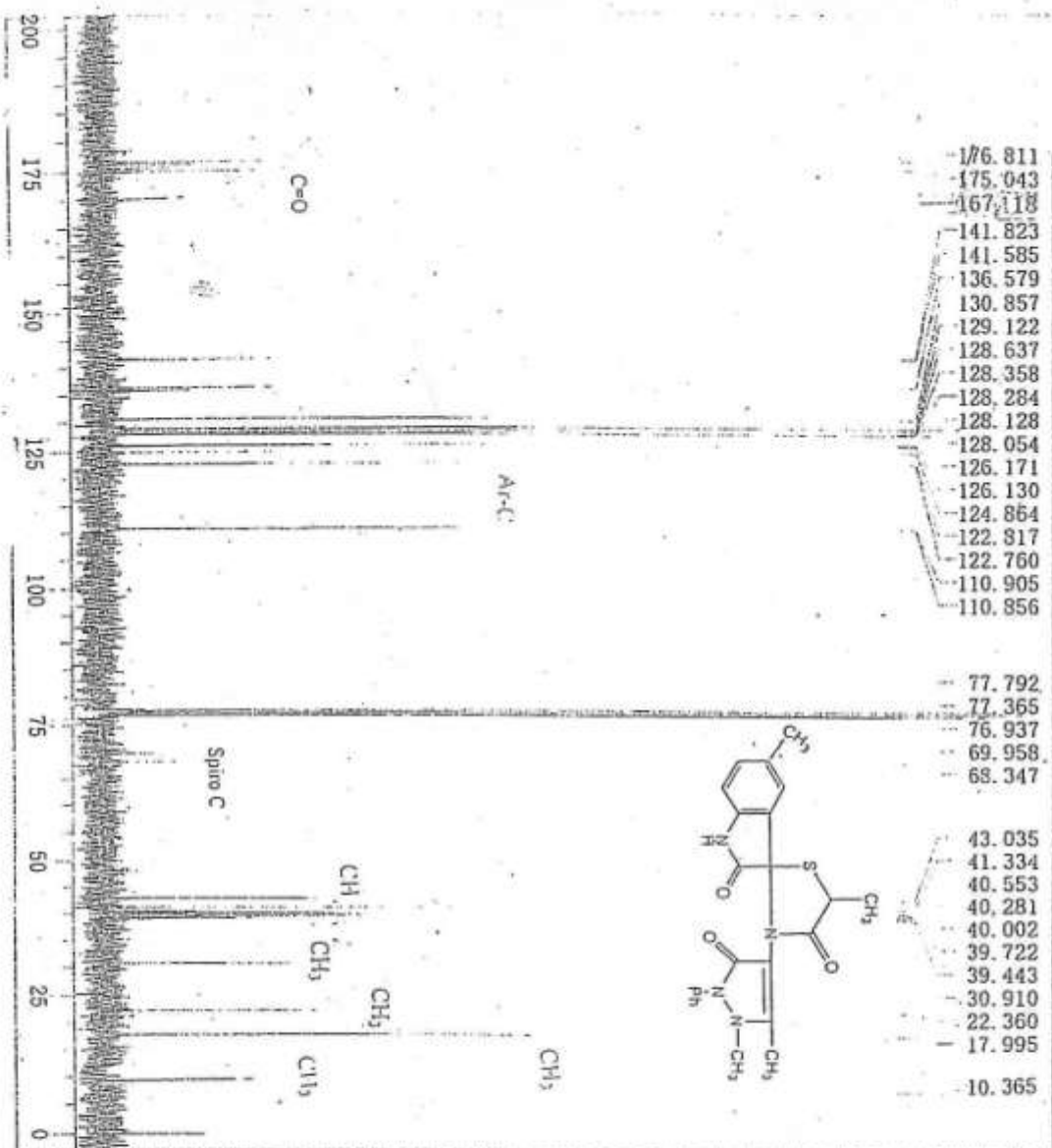
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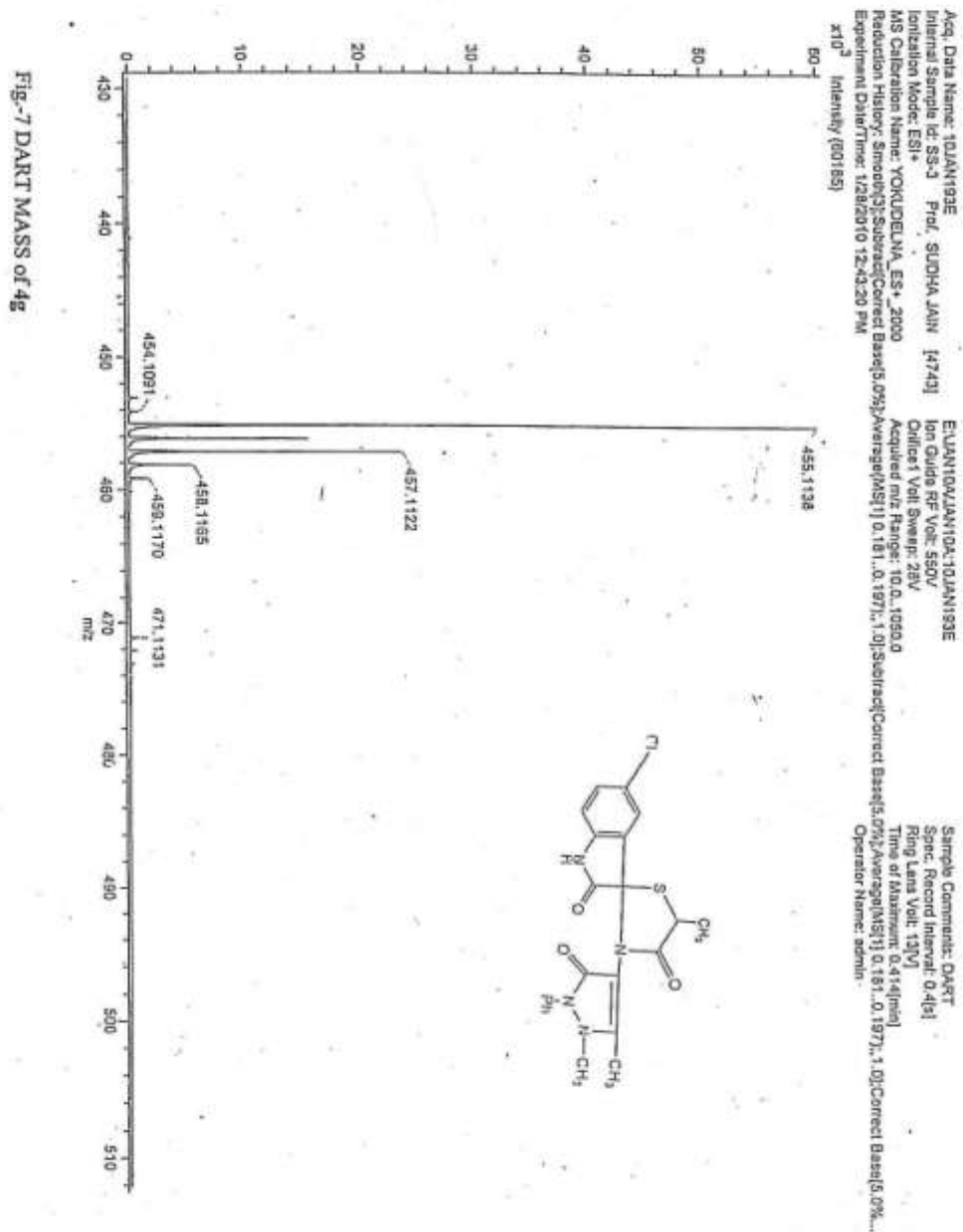


Fig.-7 DART MASS of 4g

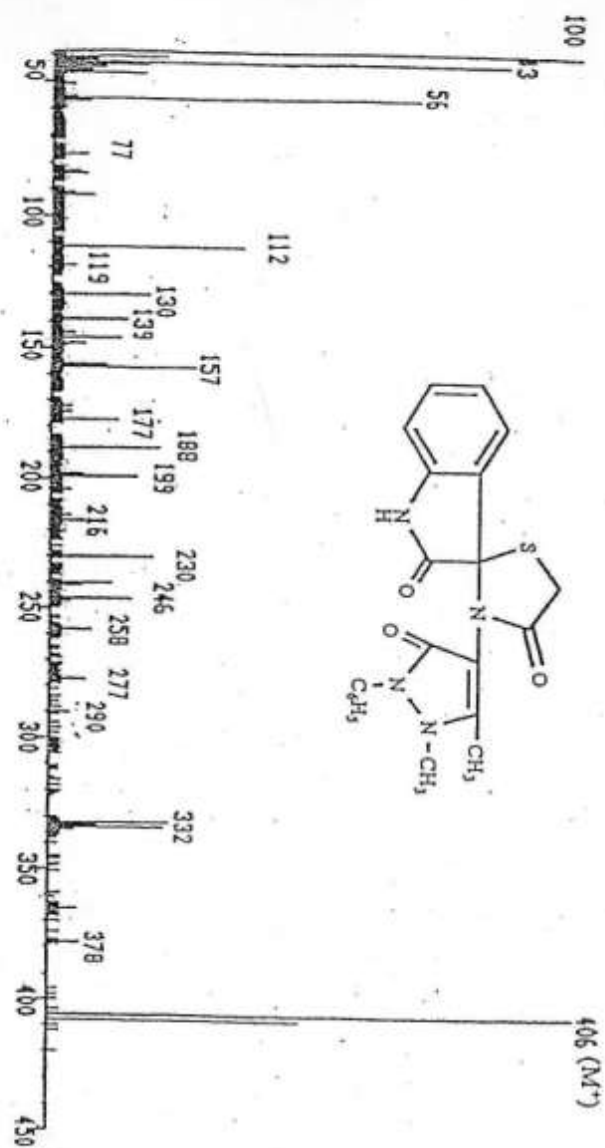


Fig.-8 EIMS MASS of 4h

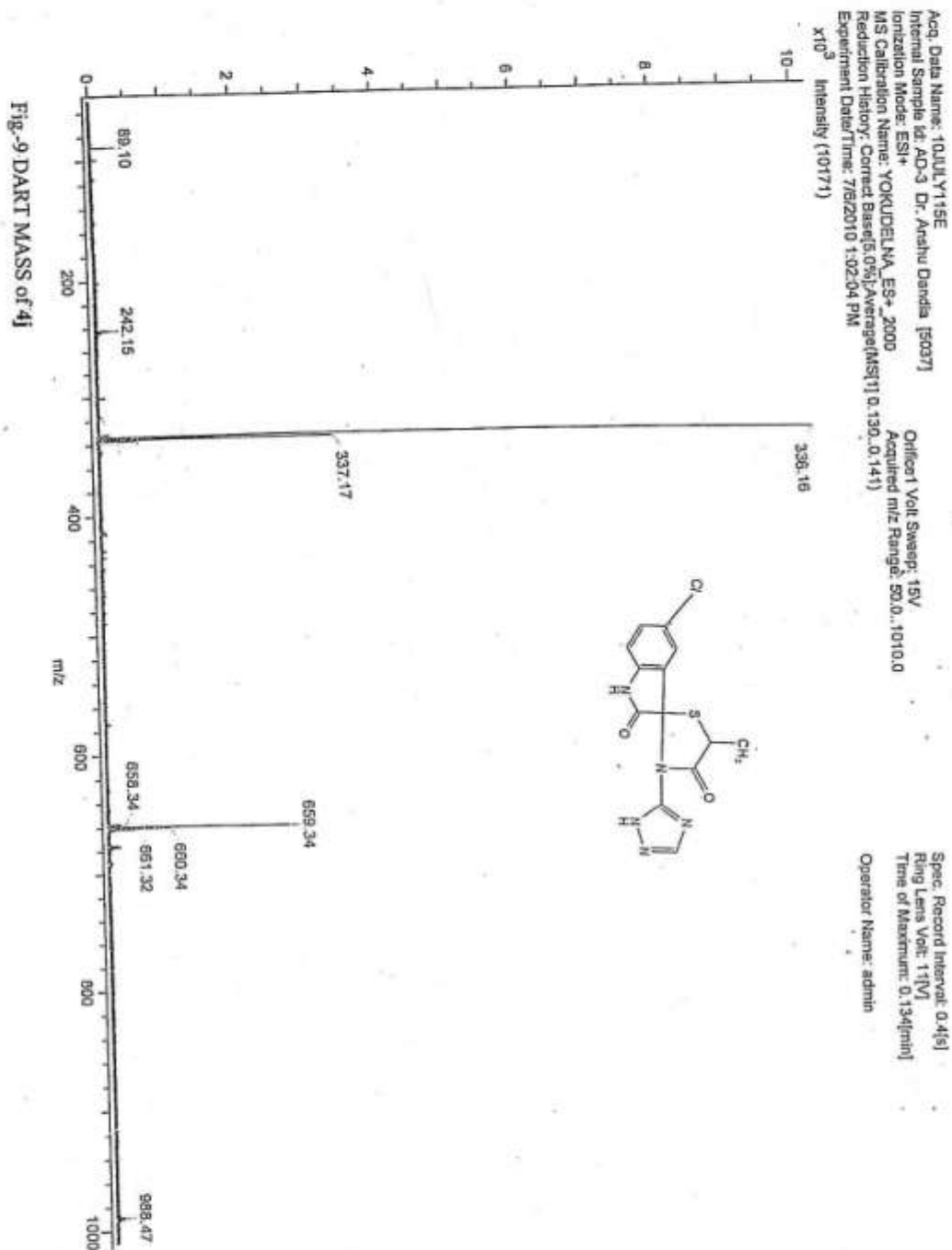


Fig.-9 DART MASS of 4j

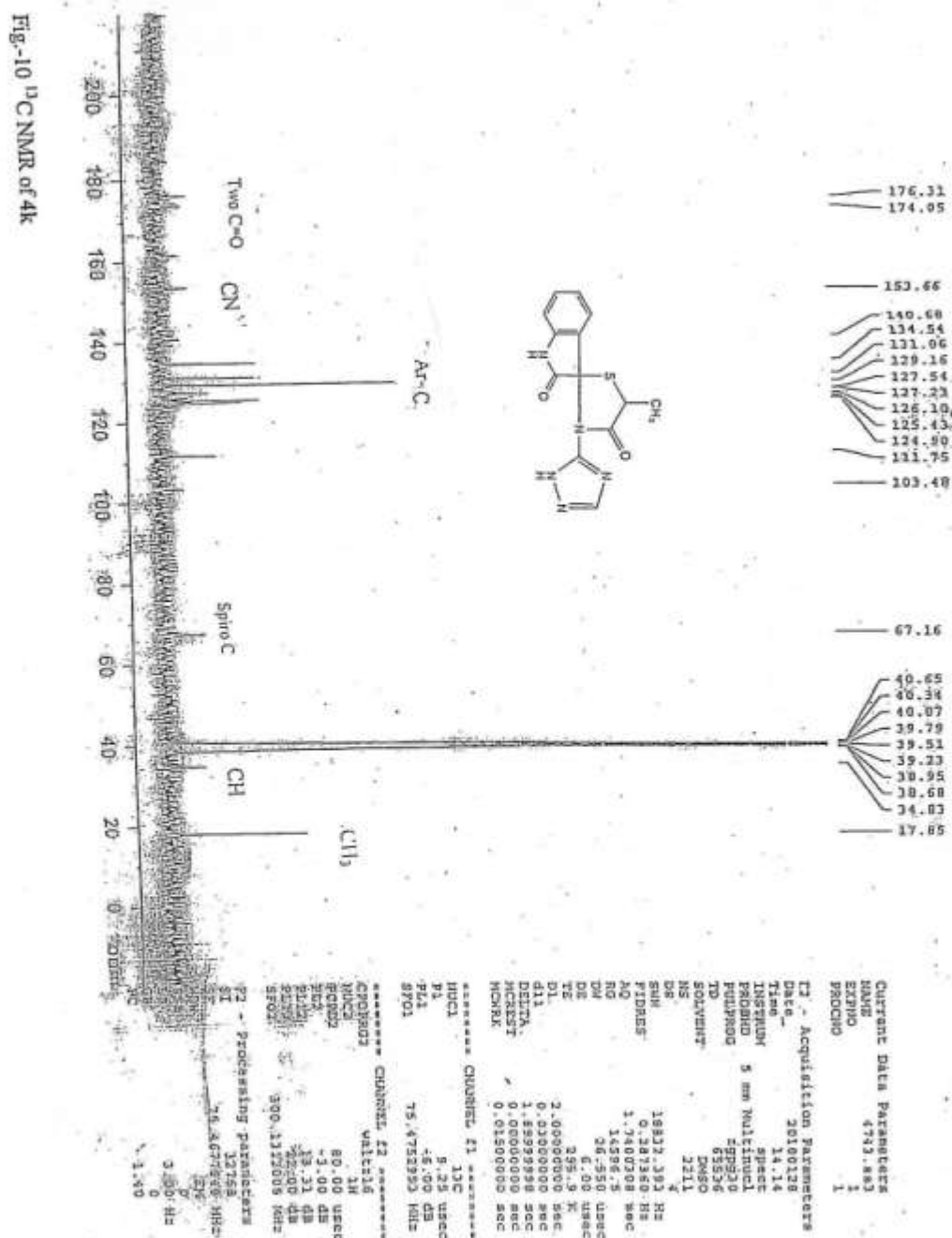


Fig.-11 EIMS MASS of 4k

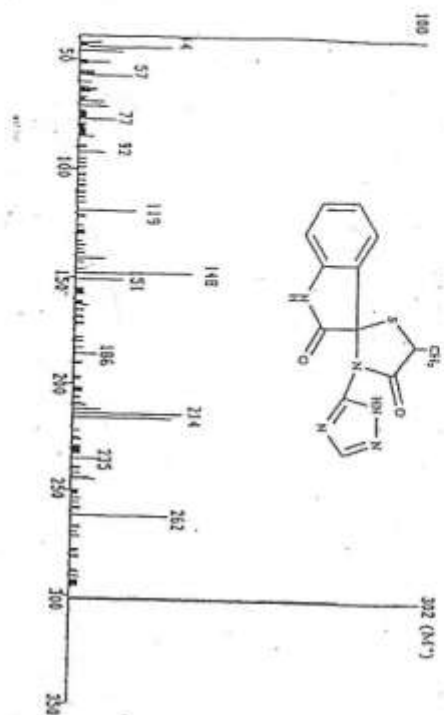
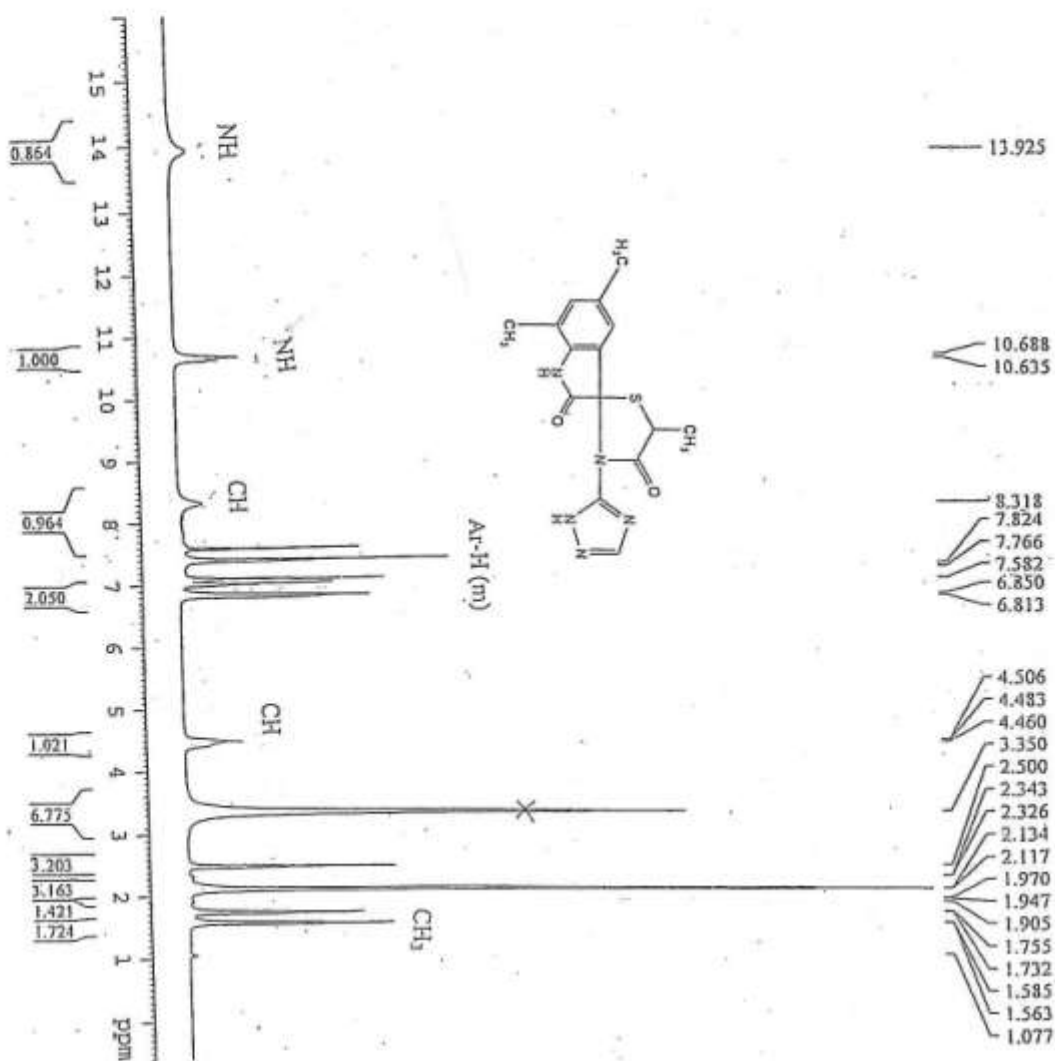


Fig-12 ¹H NMR of 4l

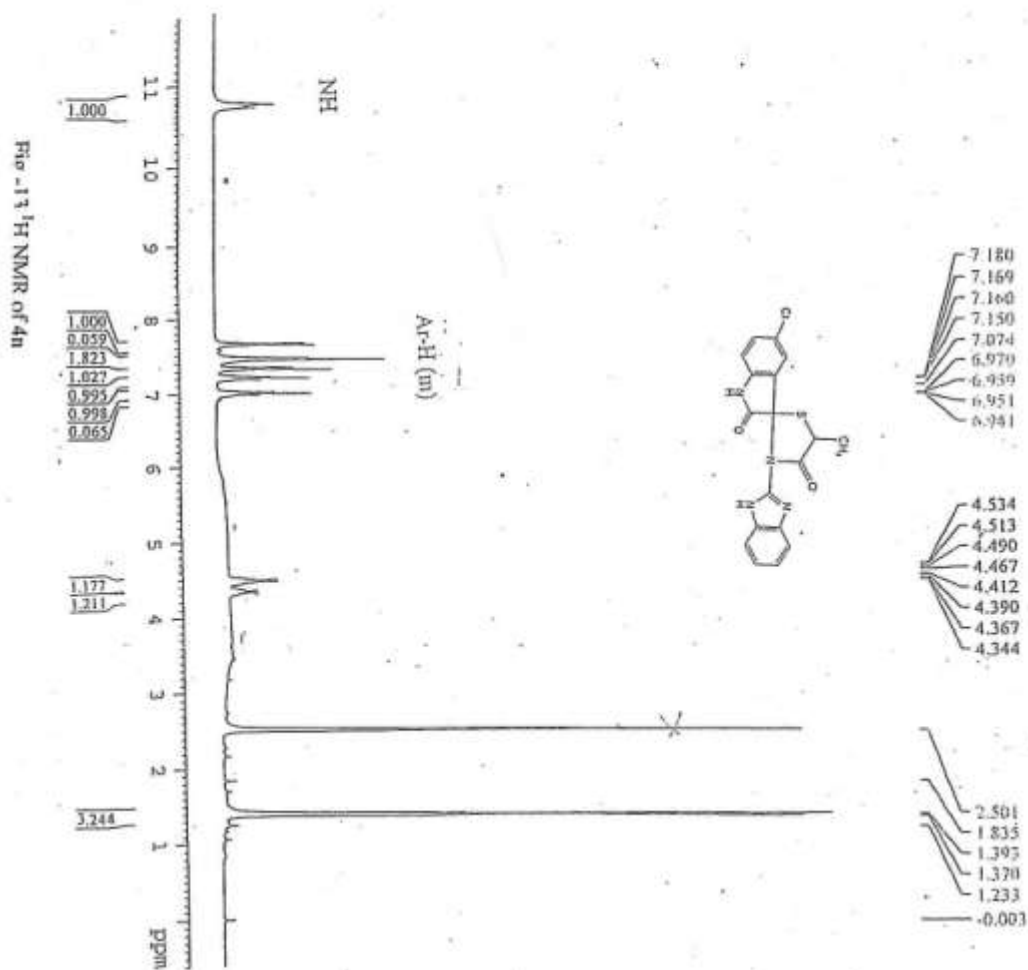


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PC 1.00

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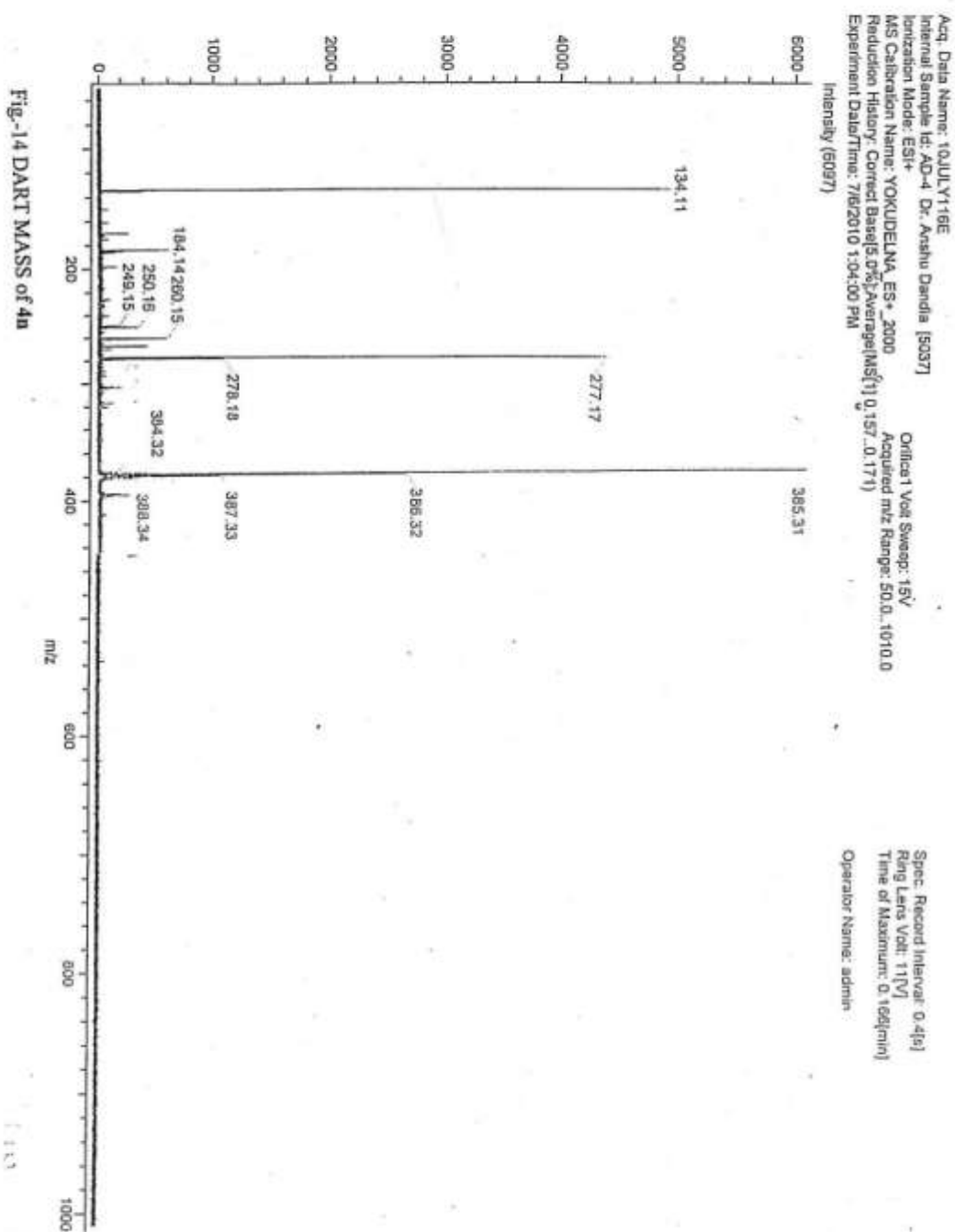
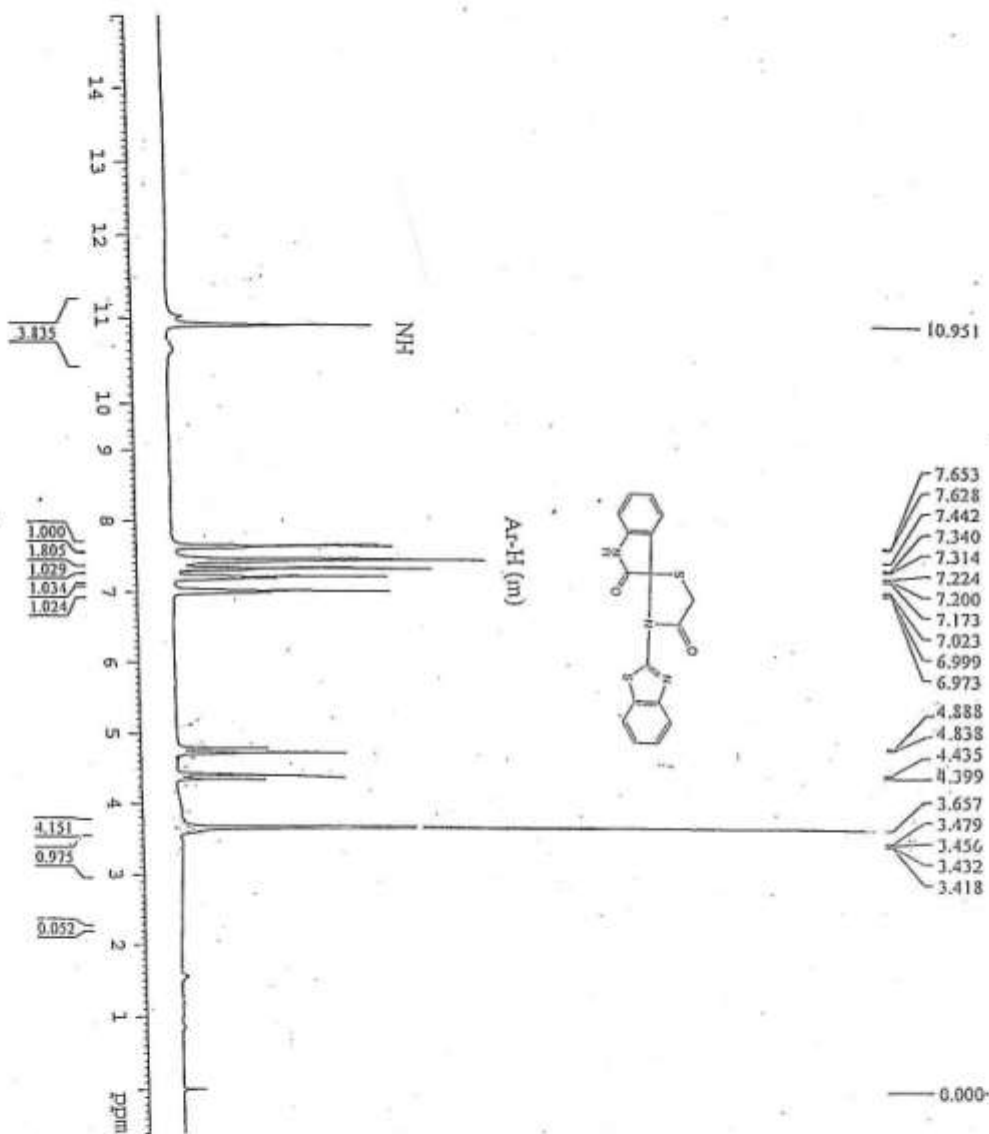




Fig.-16 ¹H NMR of 4s



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FIDRES 0.128016 Hz
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RG 1024
DW 29.600 usec
DE 4.00 usec
TE 300.2 K
T1 0.13 sec
T1RHO 0.0000000
MCREST 1.00000000 sec
MCVARK 0.01300000 sec

CHANNEL f1
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P1 6.48 usec
PL1 -3.00 dB
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F2 - Processing parameters
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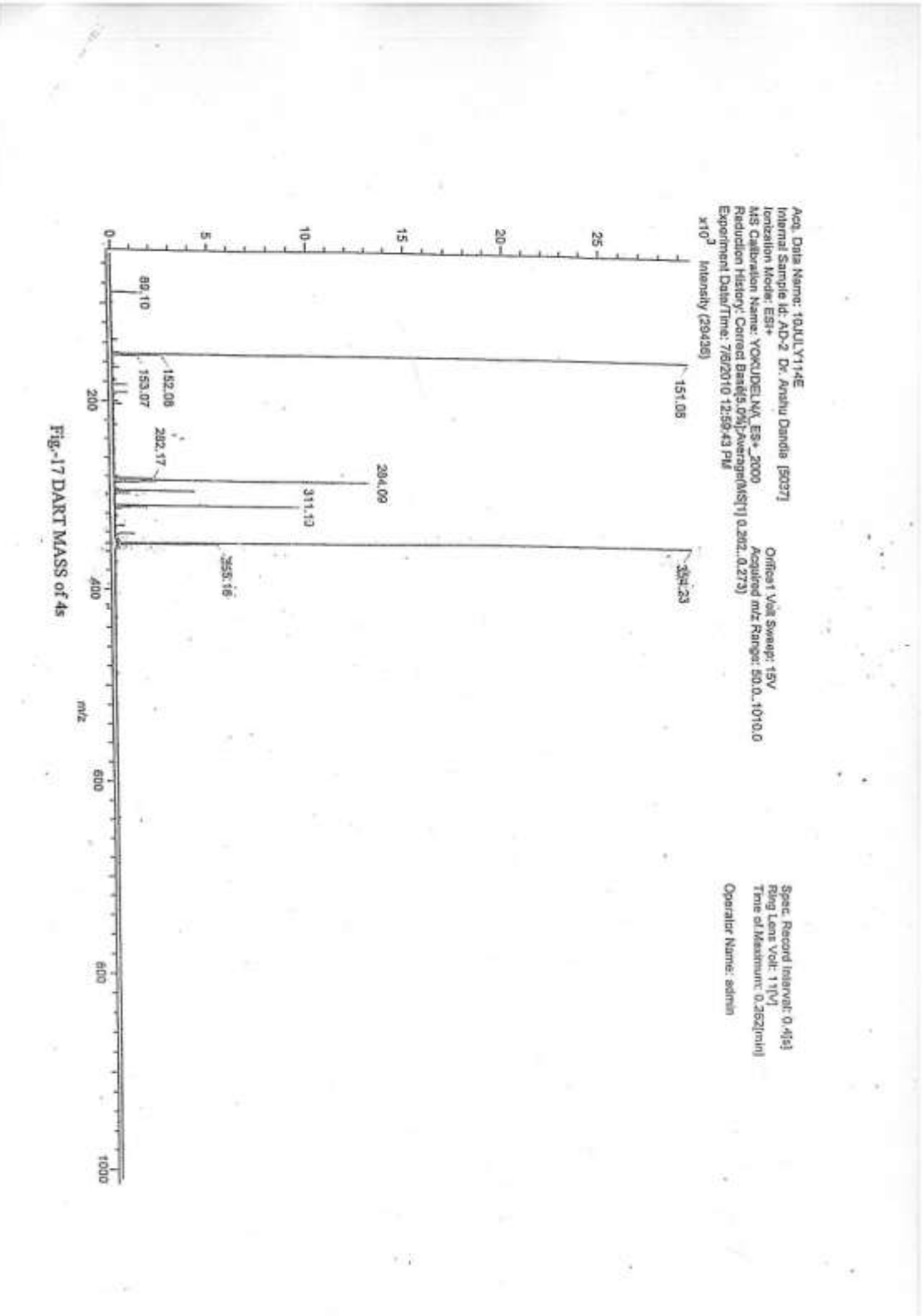
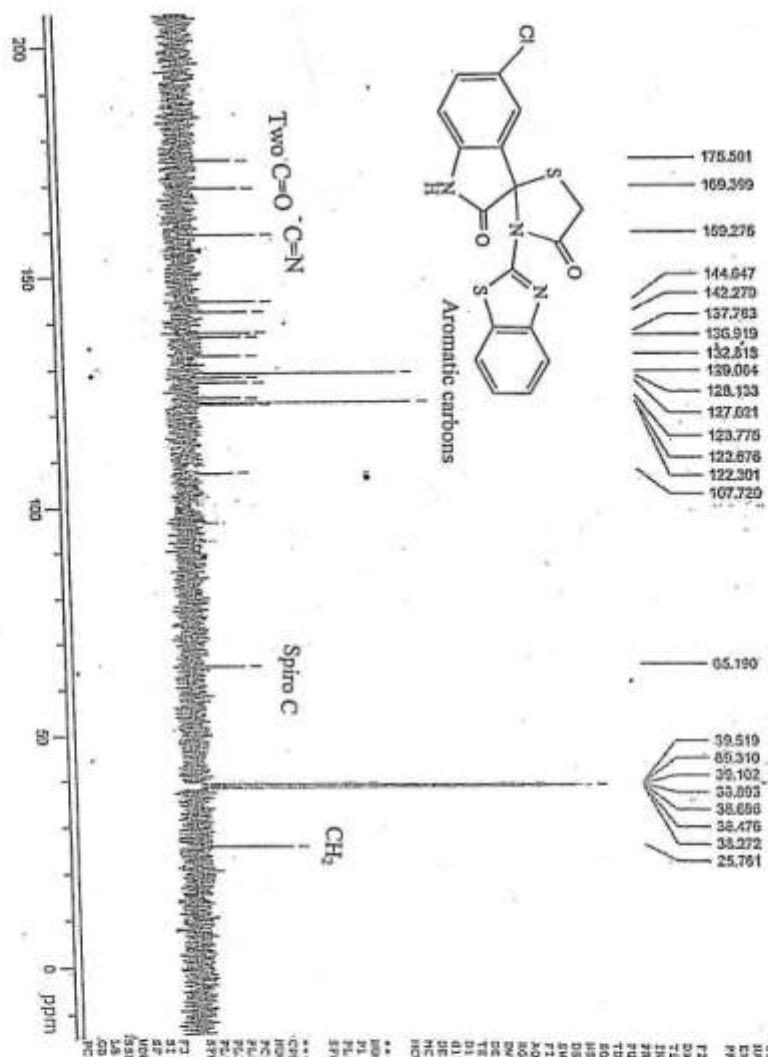


Fig.-17 DART MASS of 4s

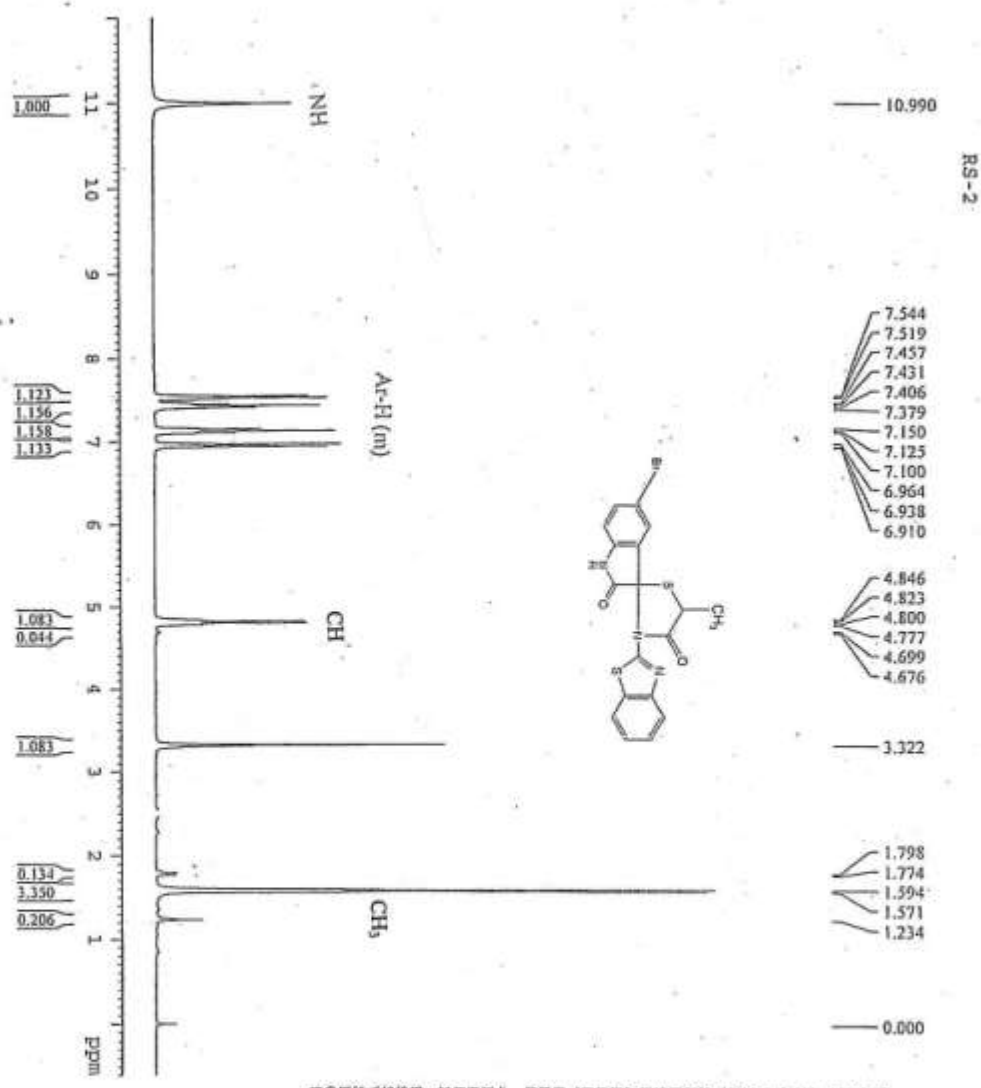
Fig.-18 ¹³C NMR of 4t



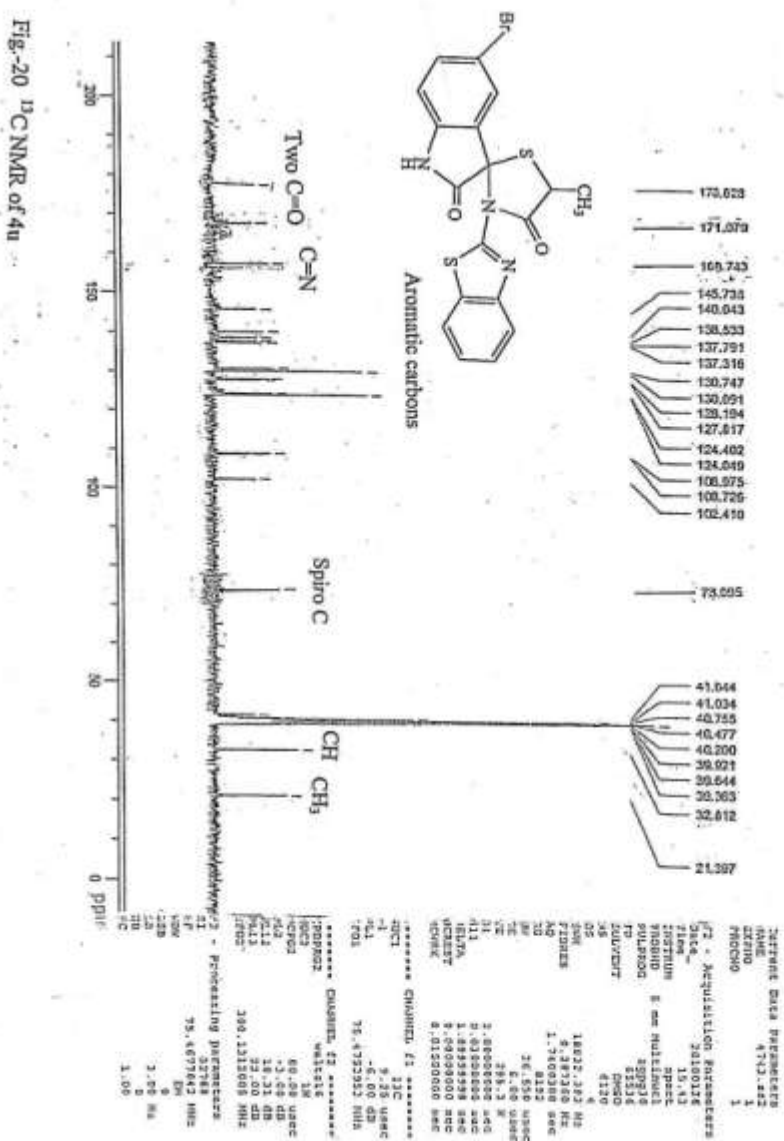
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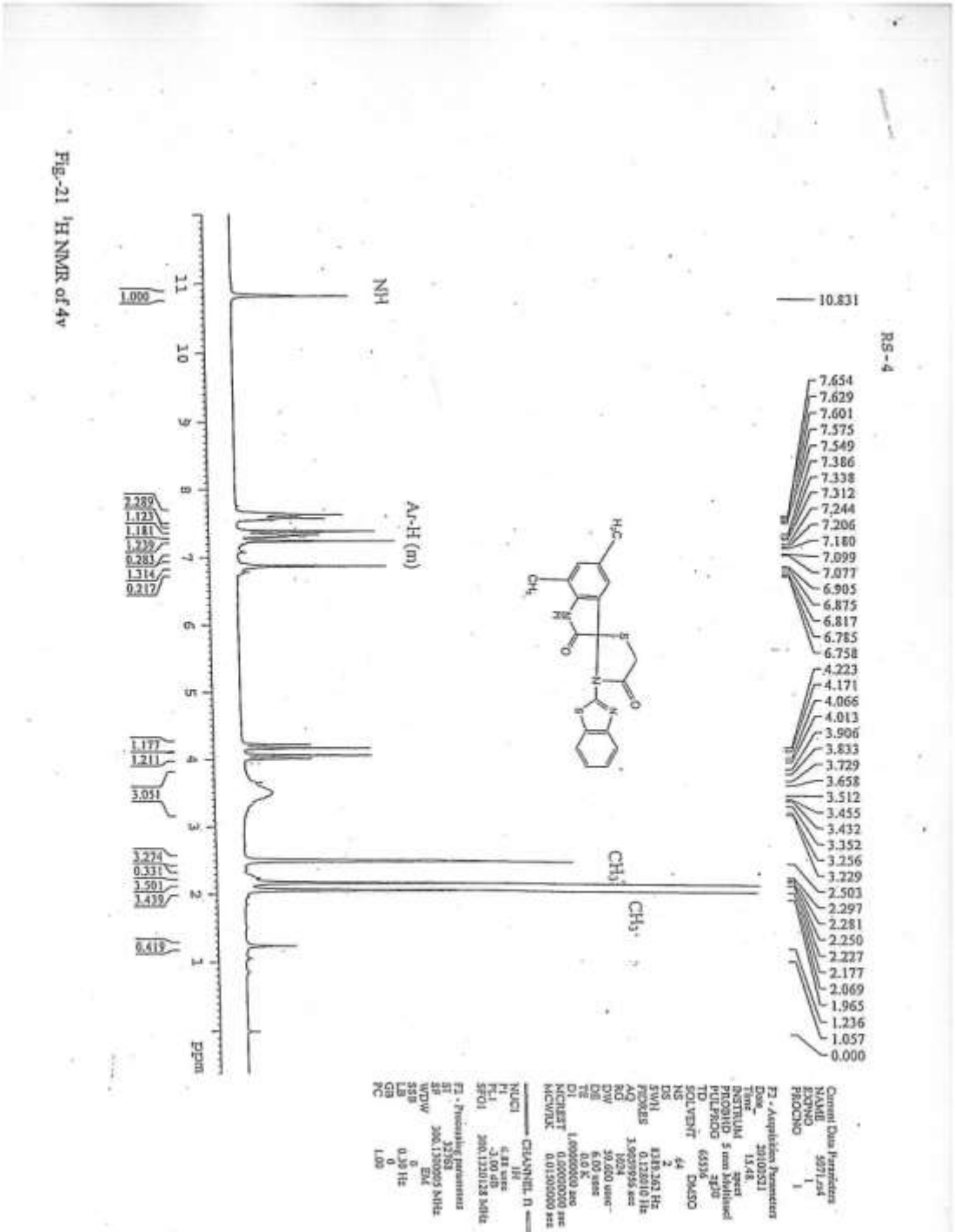
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PL2 -2.00
PL3 -2.00
PL4 20.00
PL5 20.00
SFO2 400.1518005
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NUC3 1H
PCPD3 80.00
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PL4 20.00
PL5 20.00
SFO3 400.1518005

Fig.-19 ¹H NMR of 4u



Current Data Parameters
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PROCNO 1
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DS 3
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FIDRES 0.124010 Hz
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DE 6.00 usec
TE 0.0 K
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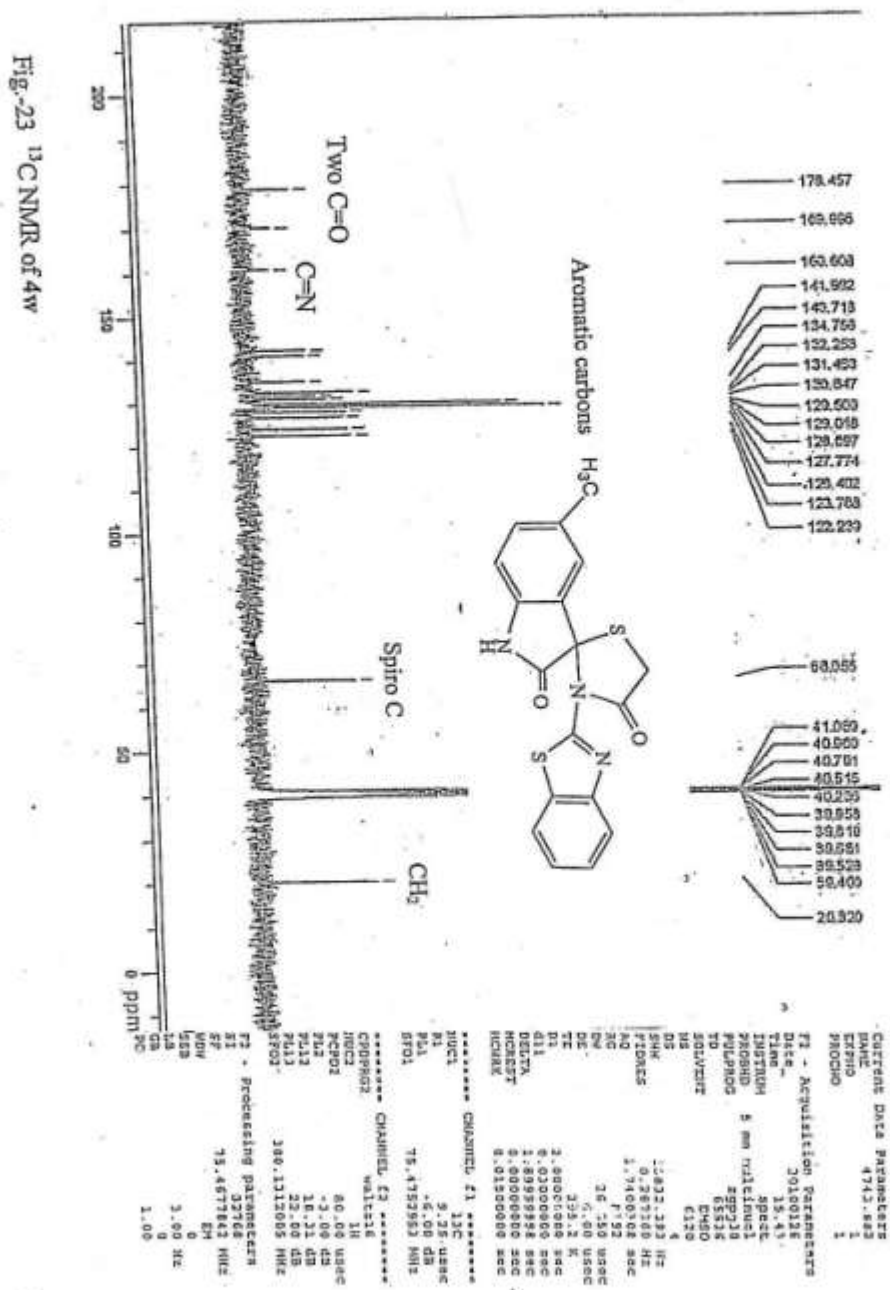
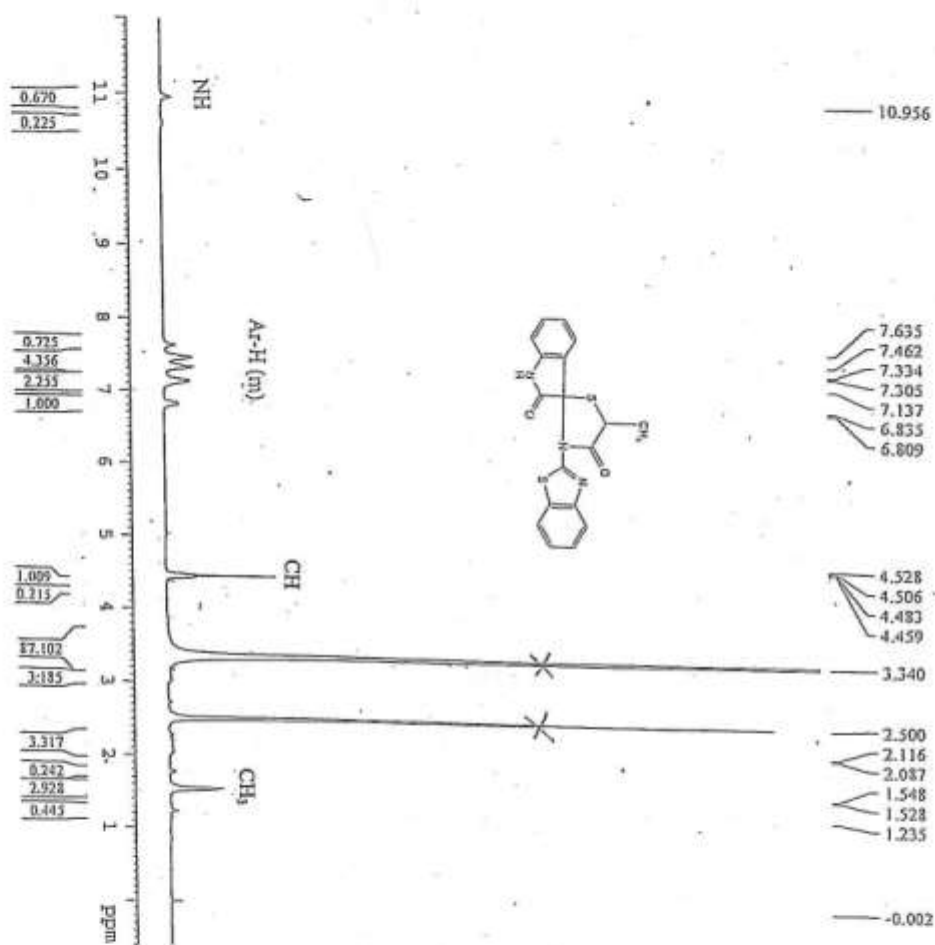


Fig. 24 ¹H NMR of 4x

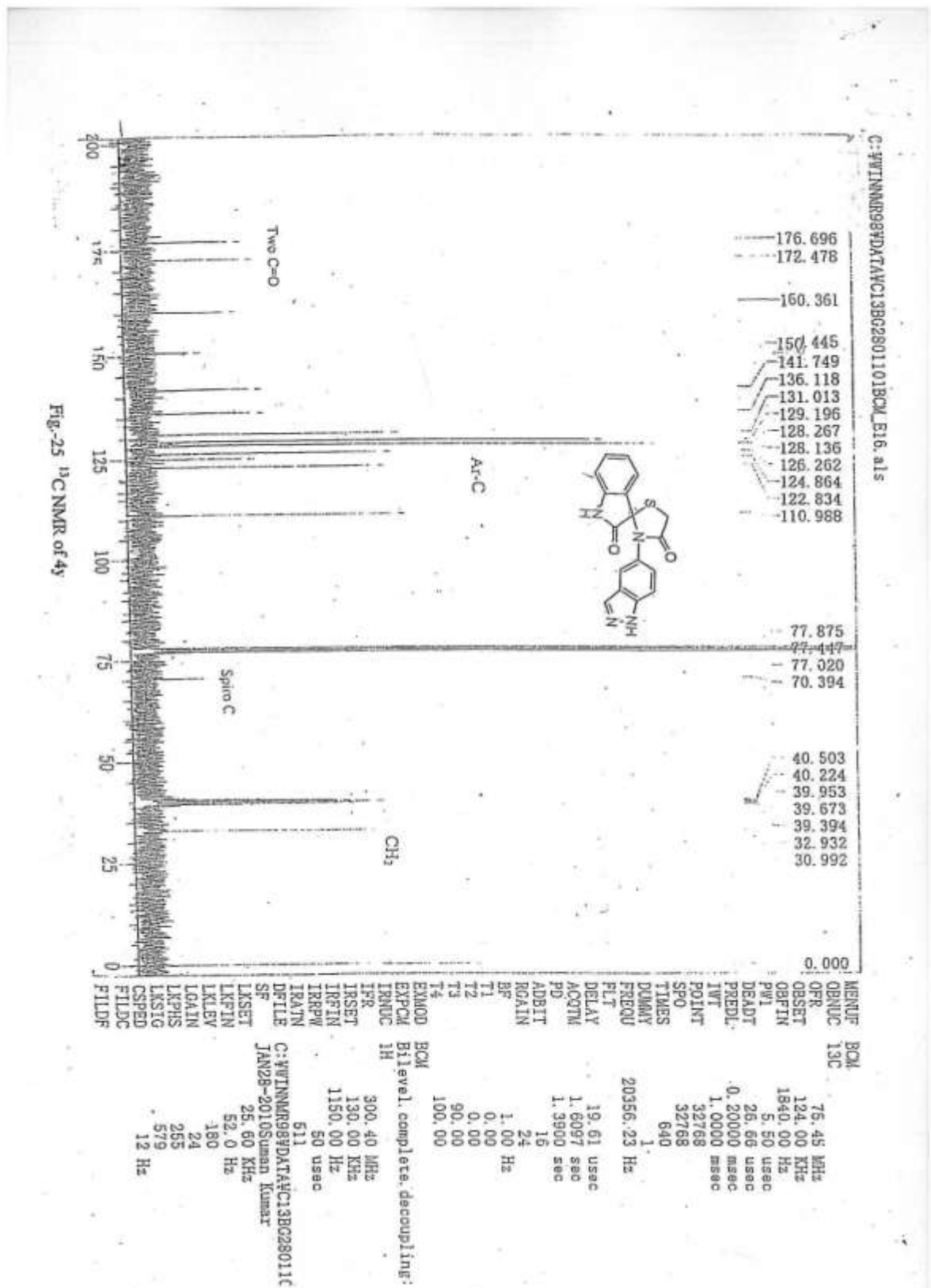


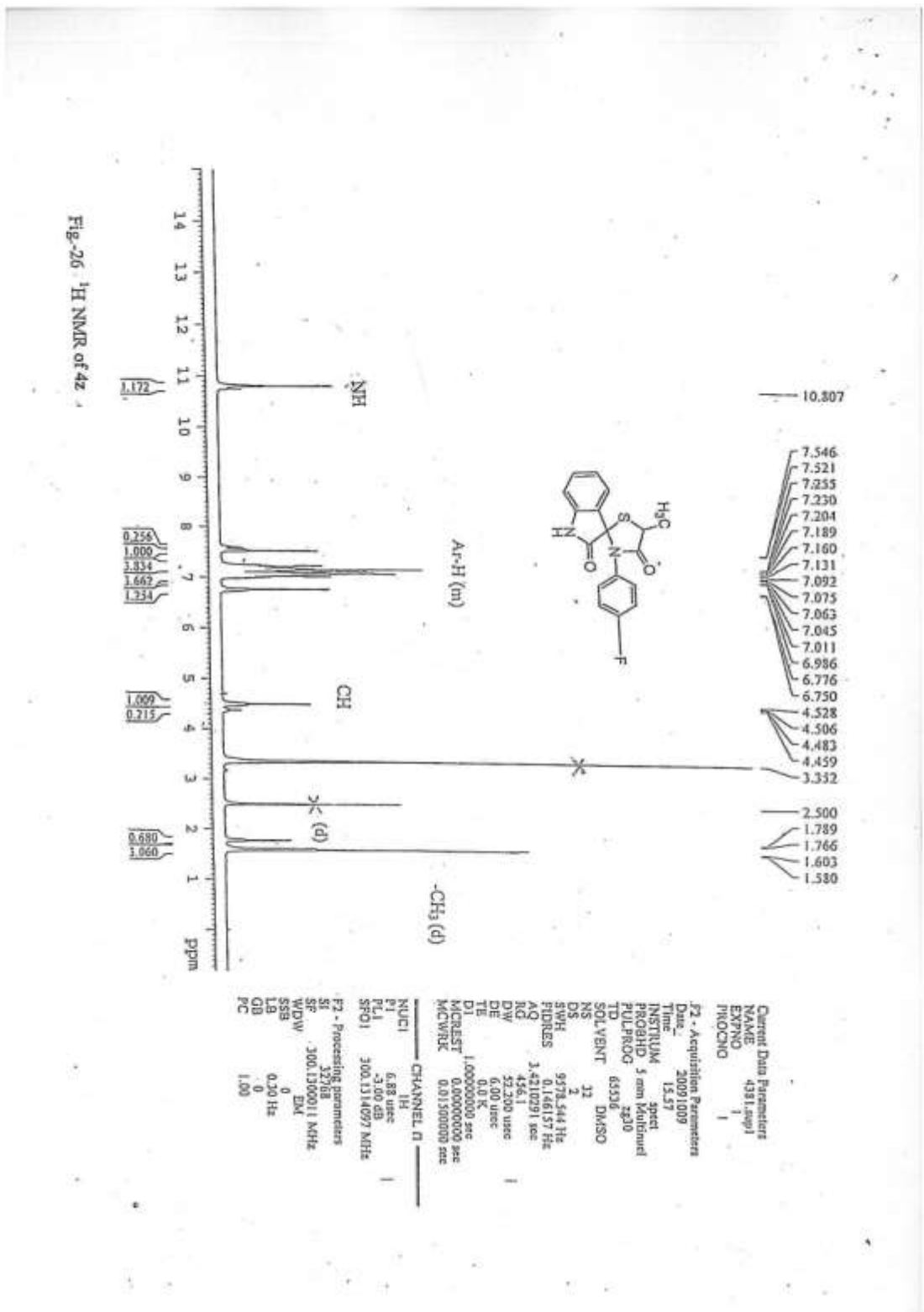
Current Data Parameters
NAME 4998.f62
EXPNO 1
PROCNO 1

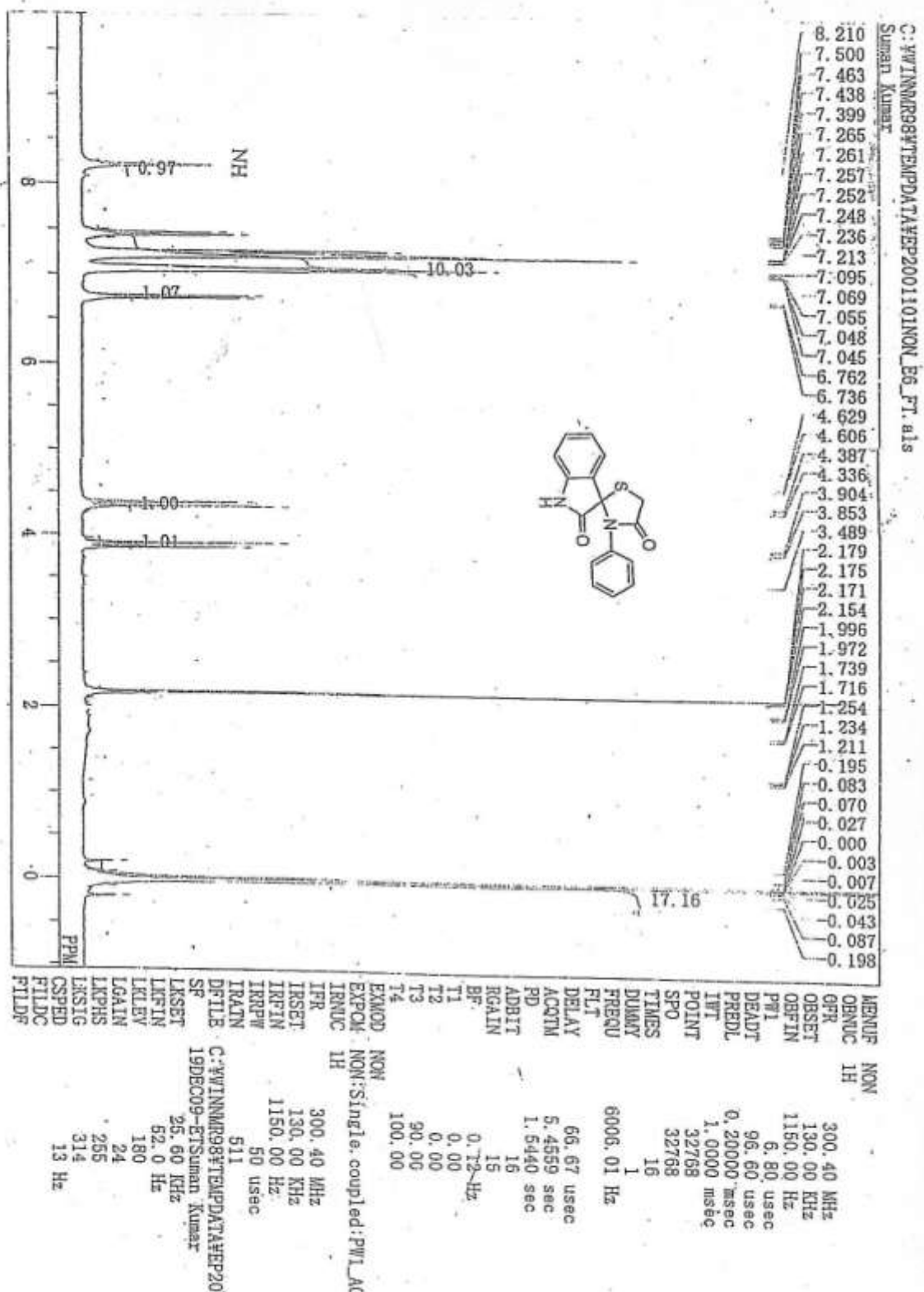
F2 - Acquisition Parameters
Date_ 20100416
Time 10.59
INSTRUM spect
PROBHD 5 mm Multispec
PULPROG zgpg30
TD 65536
FIDRES 0.0000000
SOLVENT DMSO
NS 64
DS 2
SWH 9578.544 Hz
FWHM 0.146157 Hz
AQ 3.4310291 sec
RG 574.7
NO 52.200
DN 4.00 usec
DE 288.0 K
TE 1.00000000 sec
DI 0.00000000 sec
MCHEST 0.01900000 sec
NAMEX

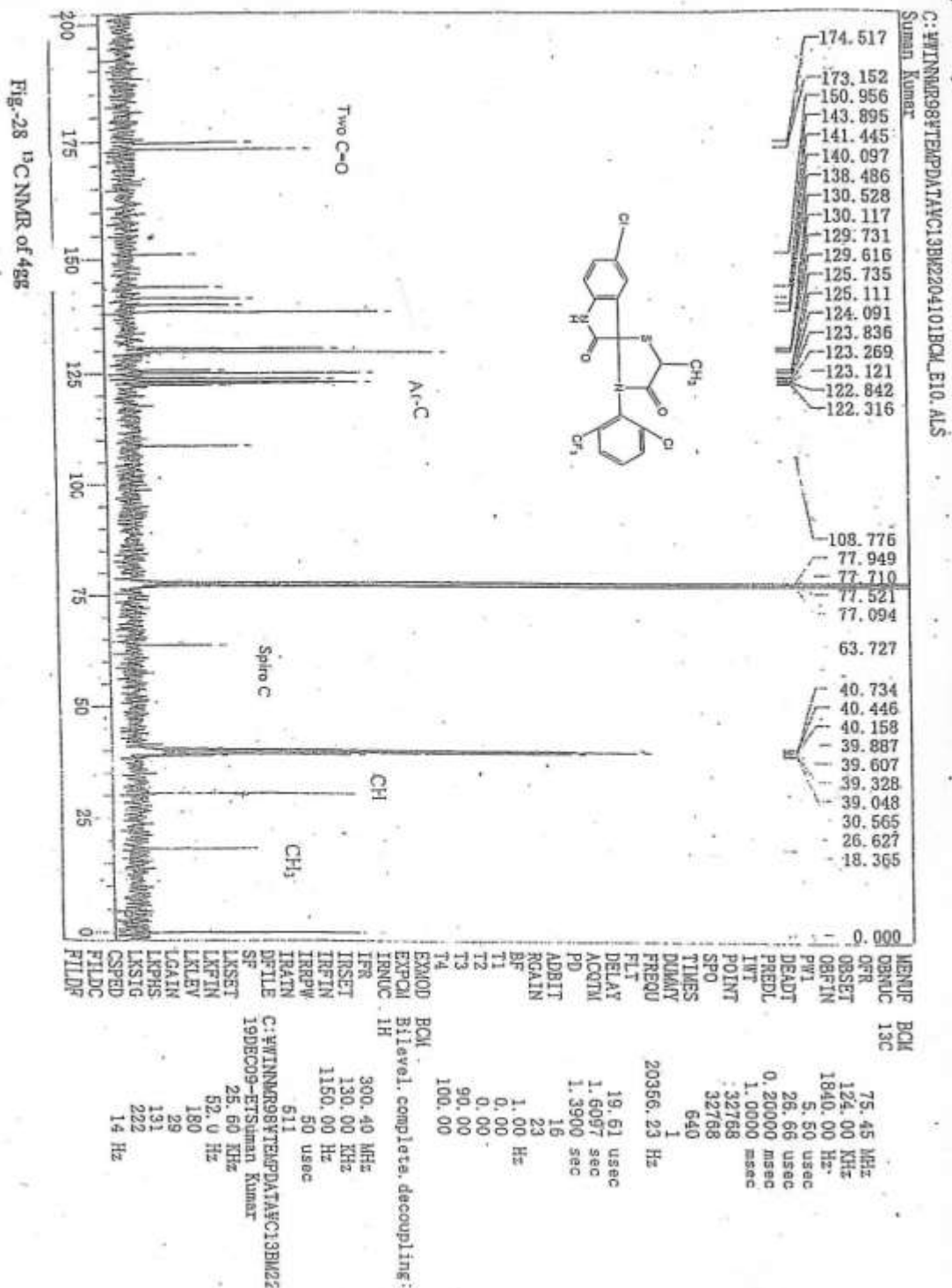
===== CHANNEL f1 =====
NUC1 13X
P1 5.88 usec
PL1 -3.00 dB
SFO1 300.1318534 MHz

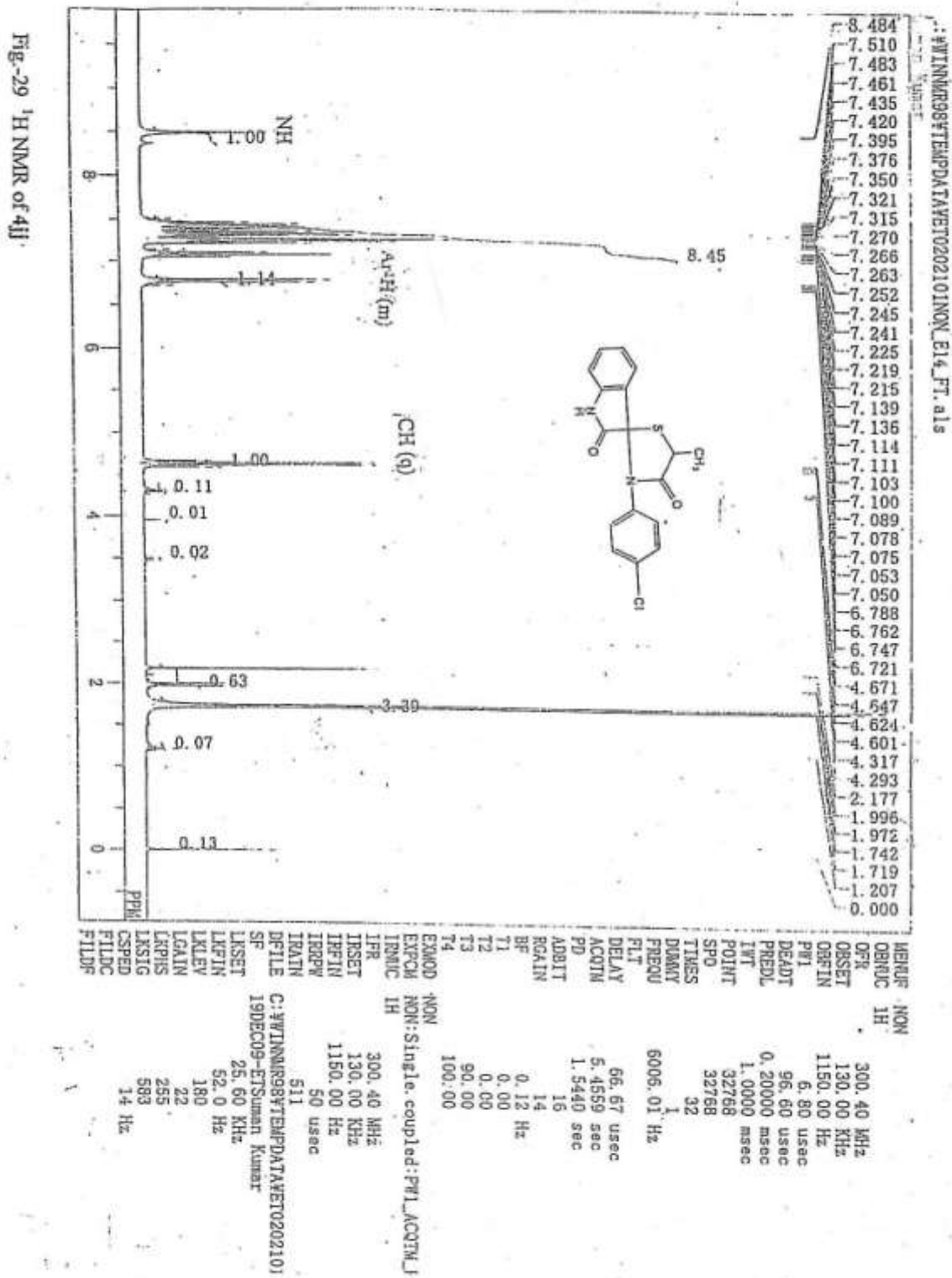
F2 - Processing parameters
SI 32768
SF 300.1300011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

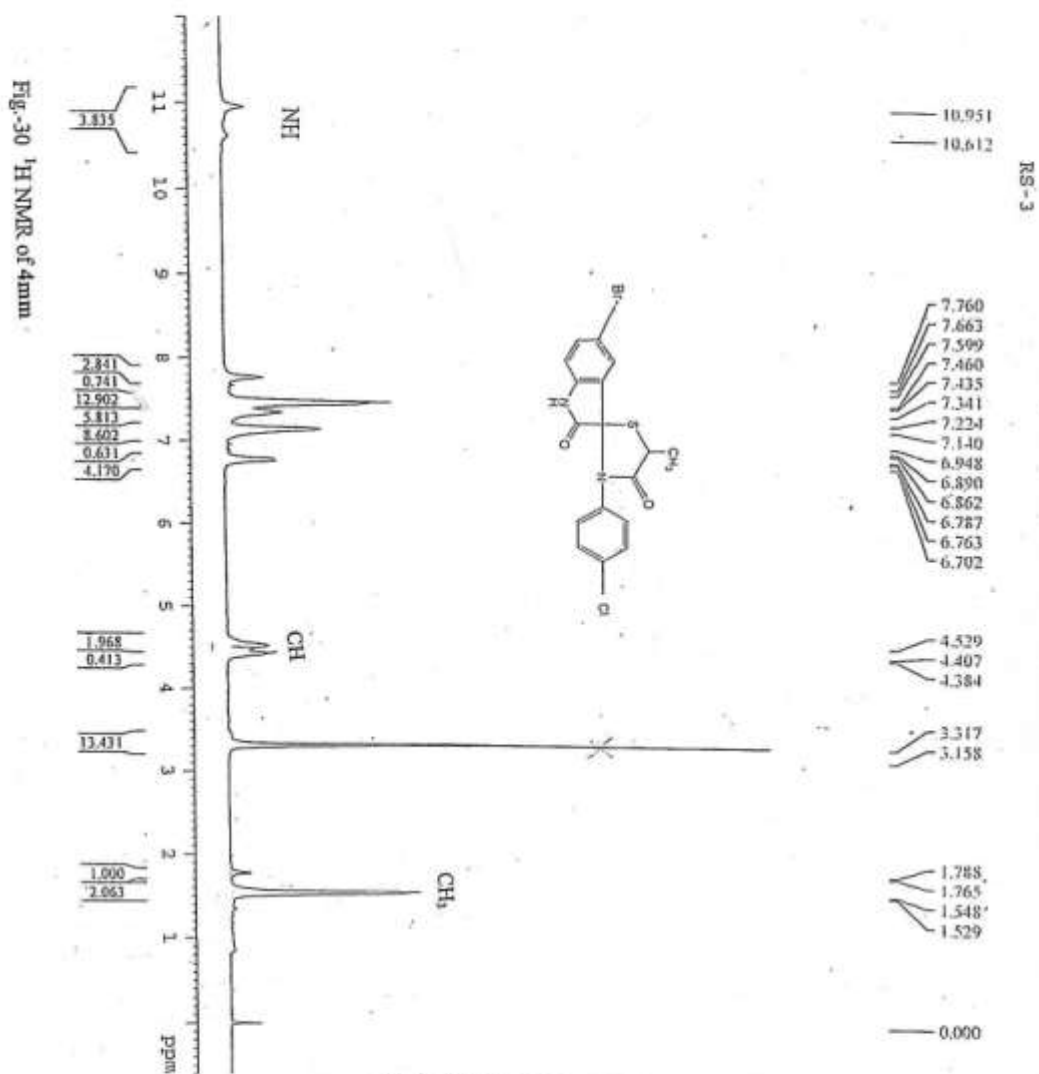


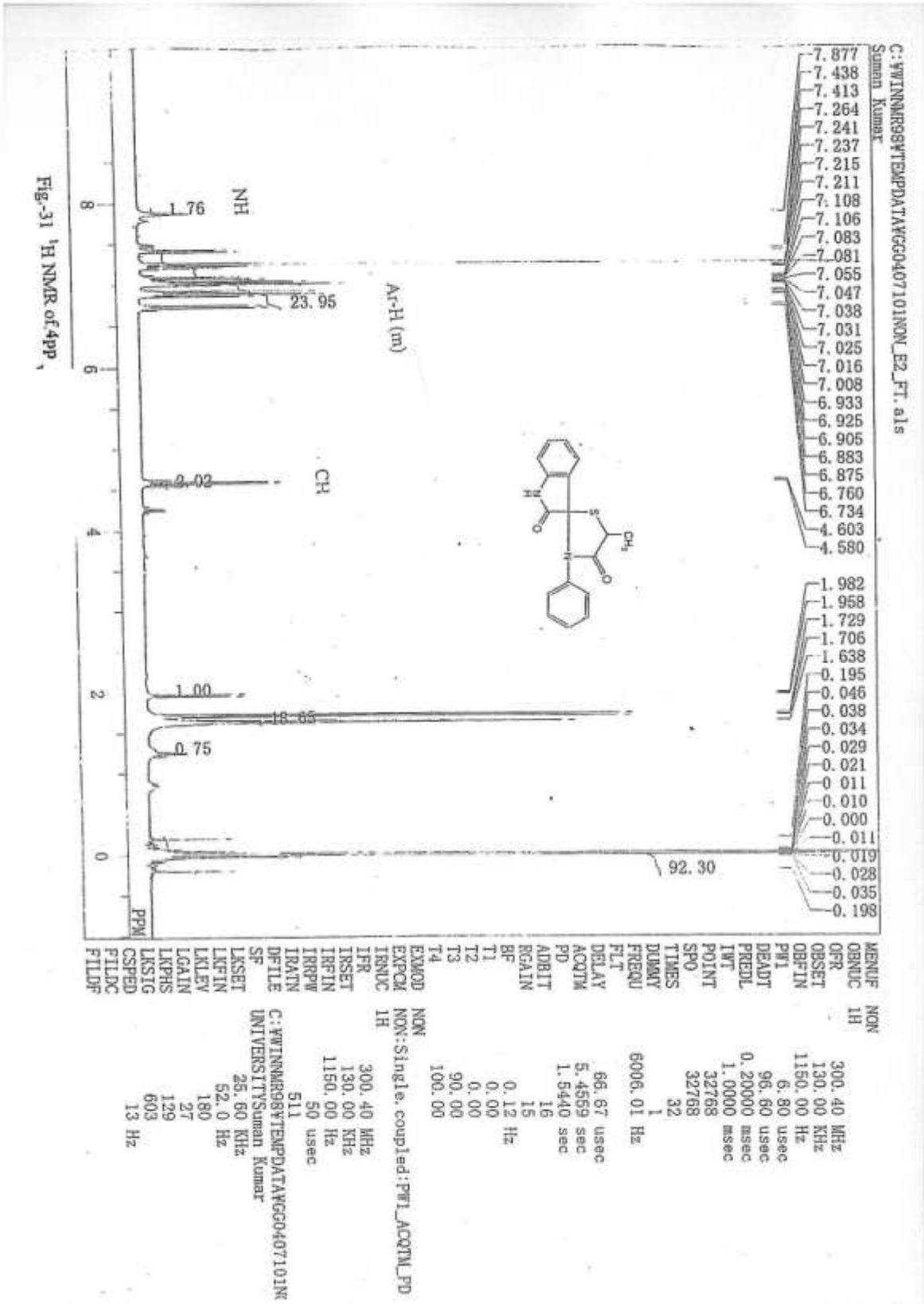




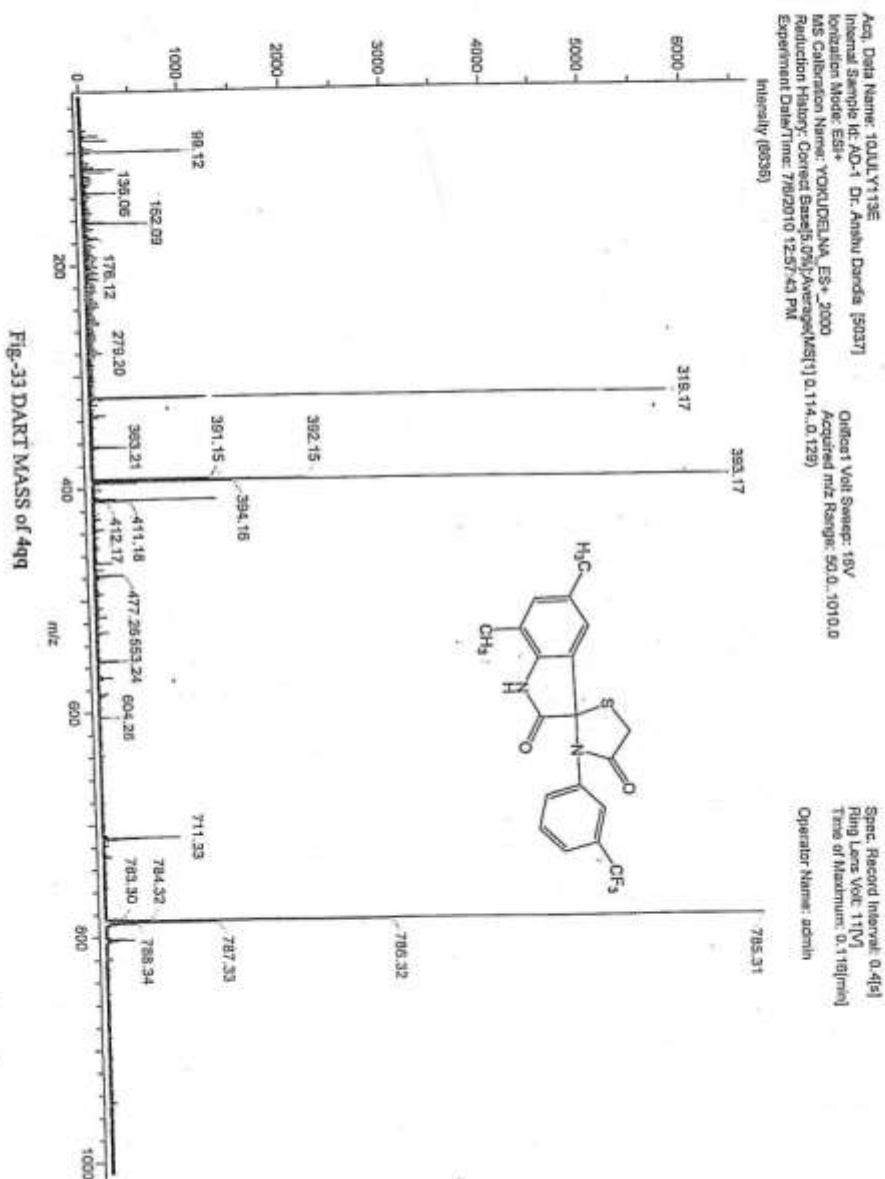


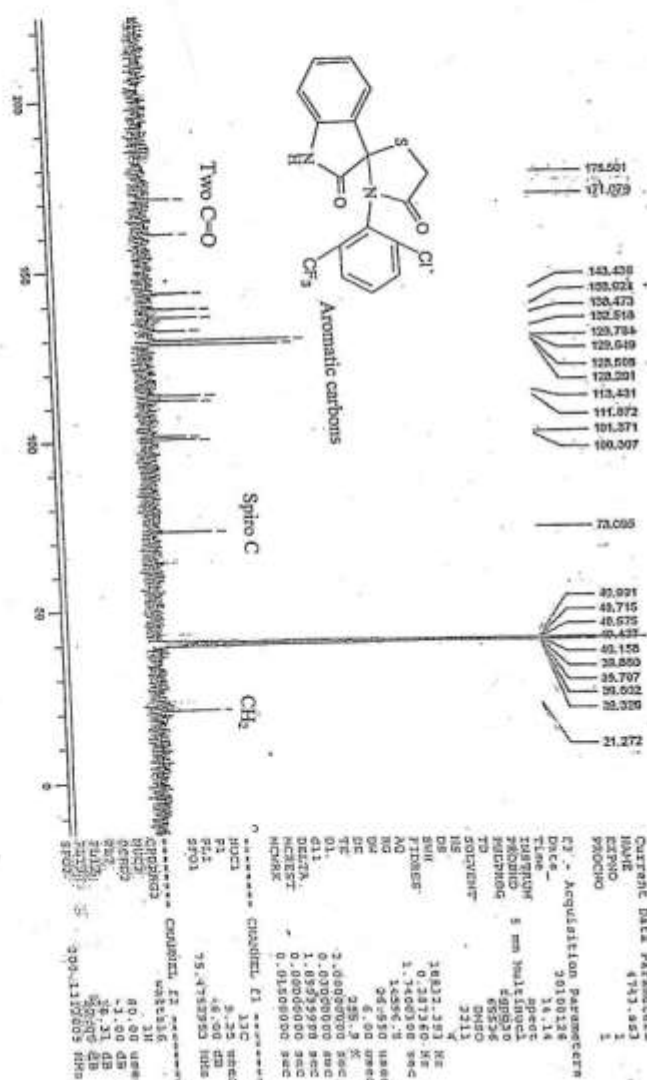


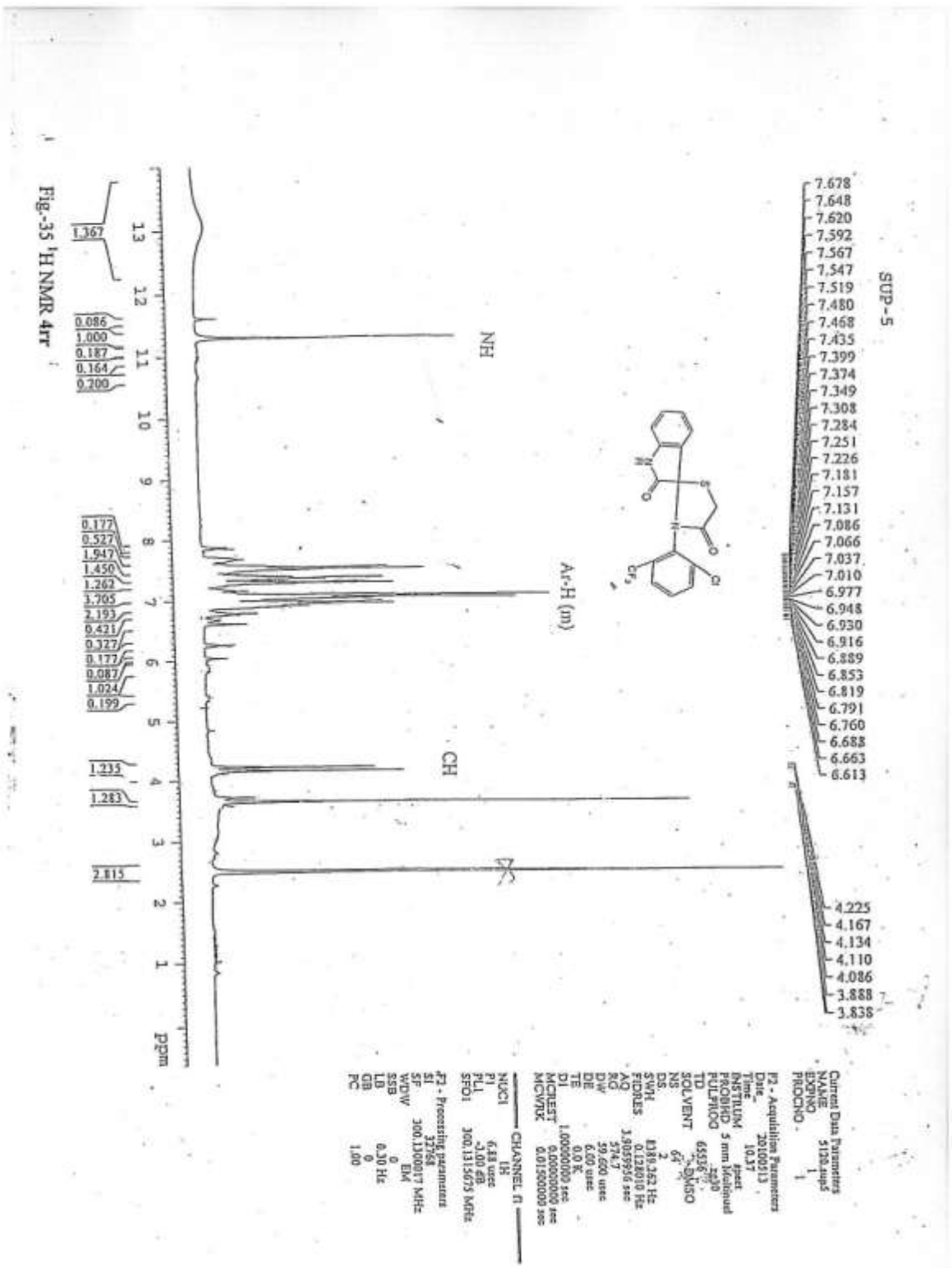


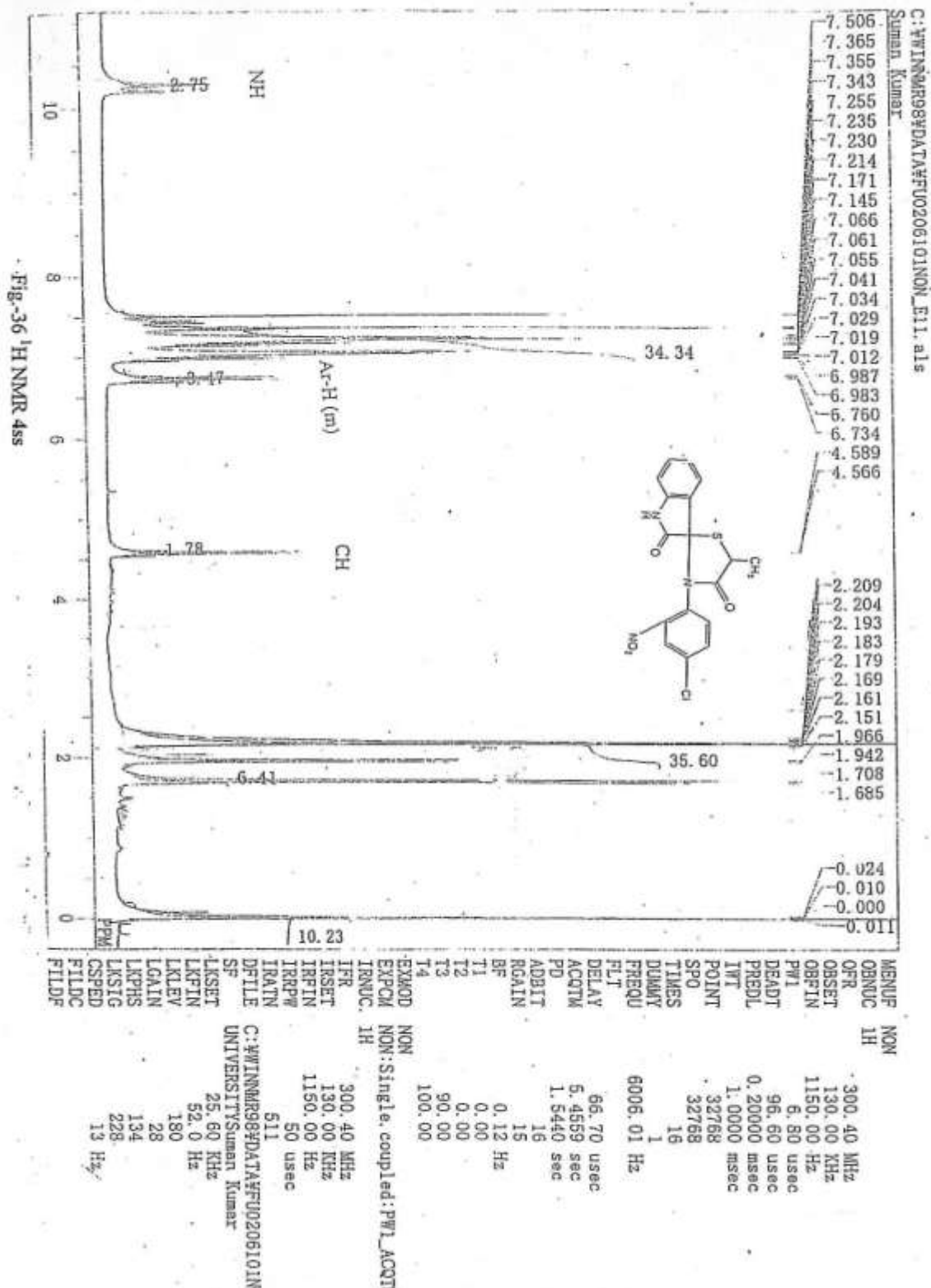


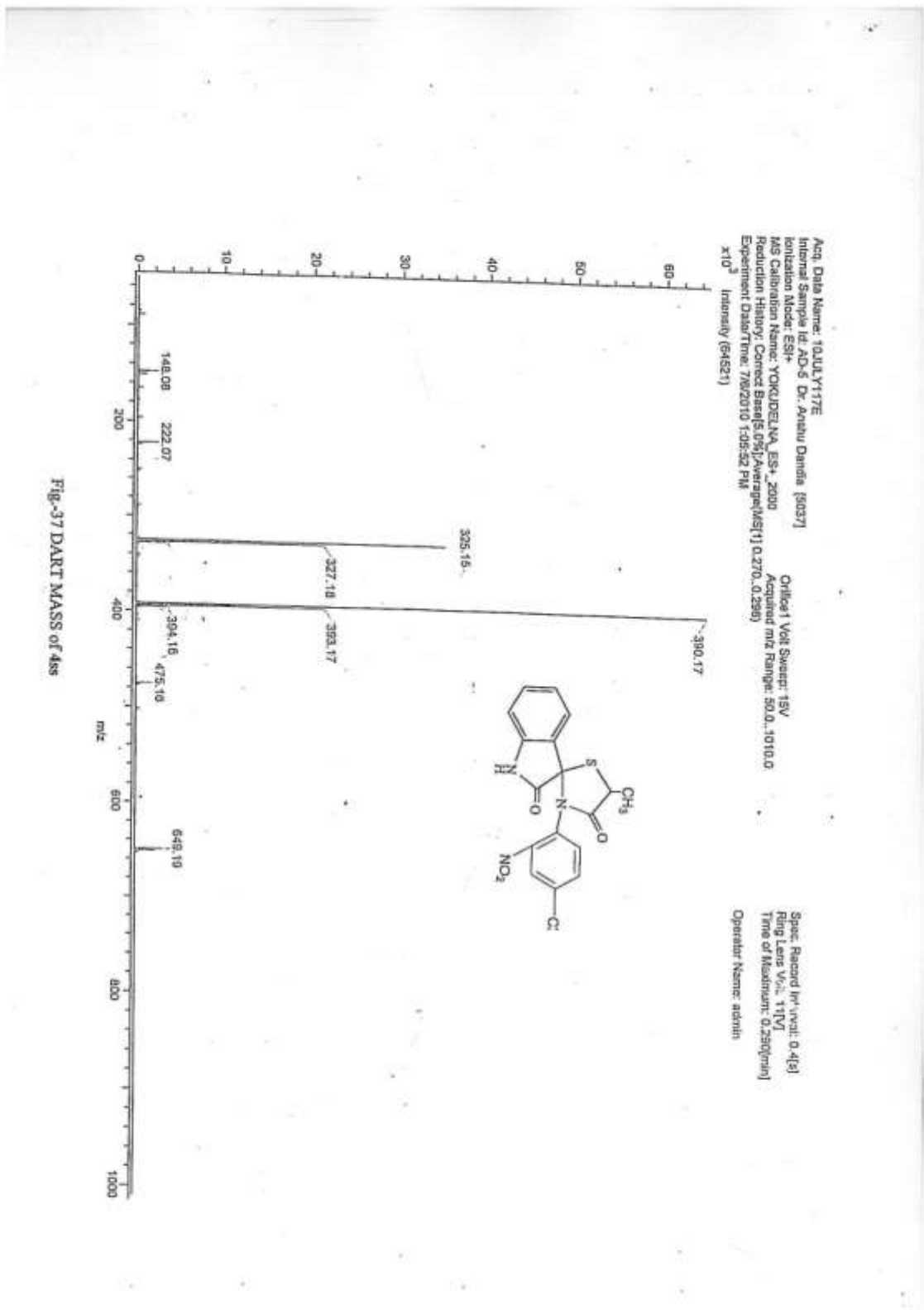












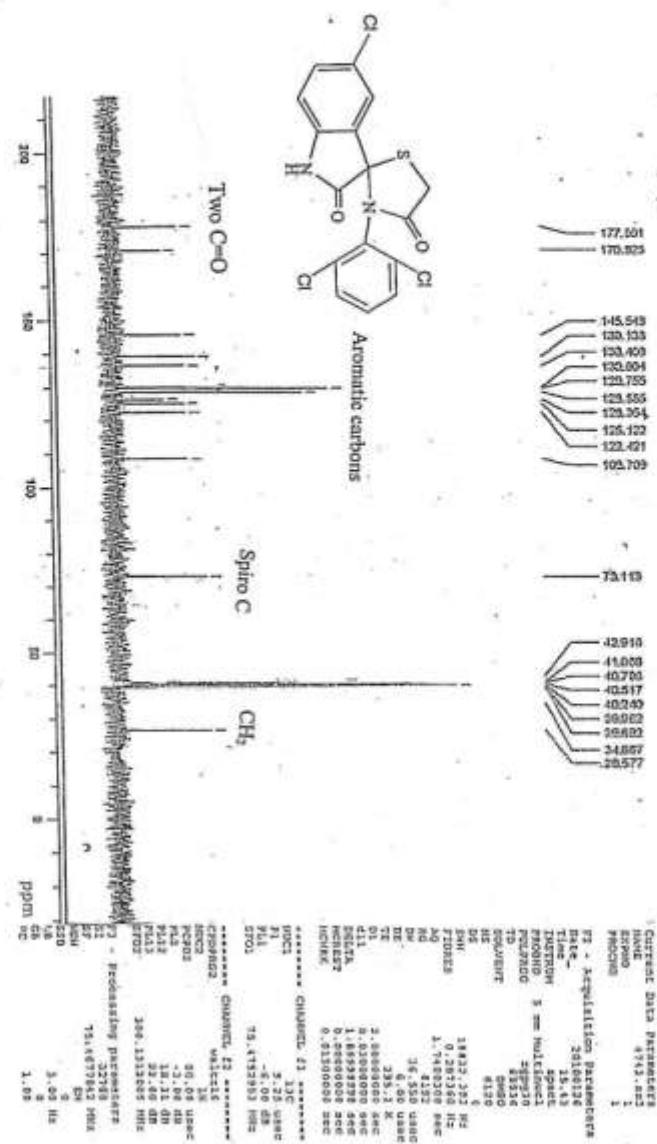
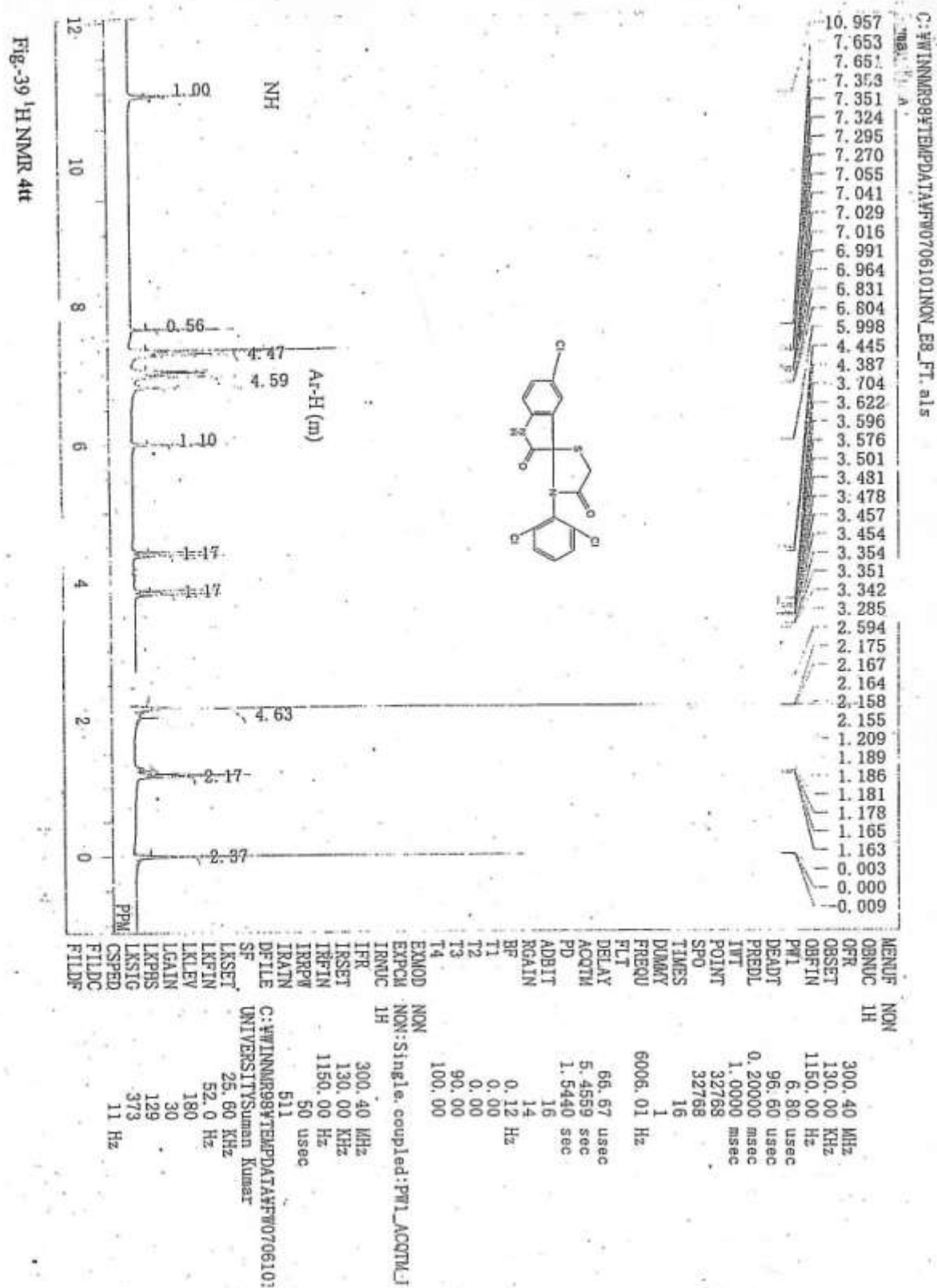


Fig-38 ¹³C NMR 4H



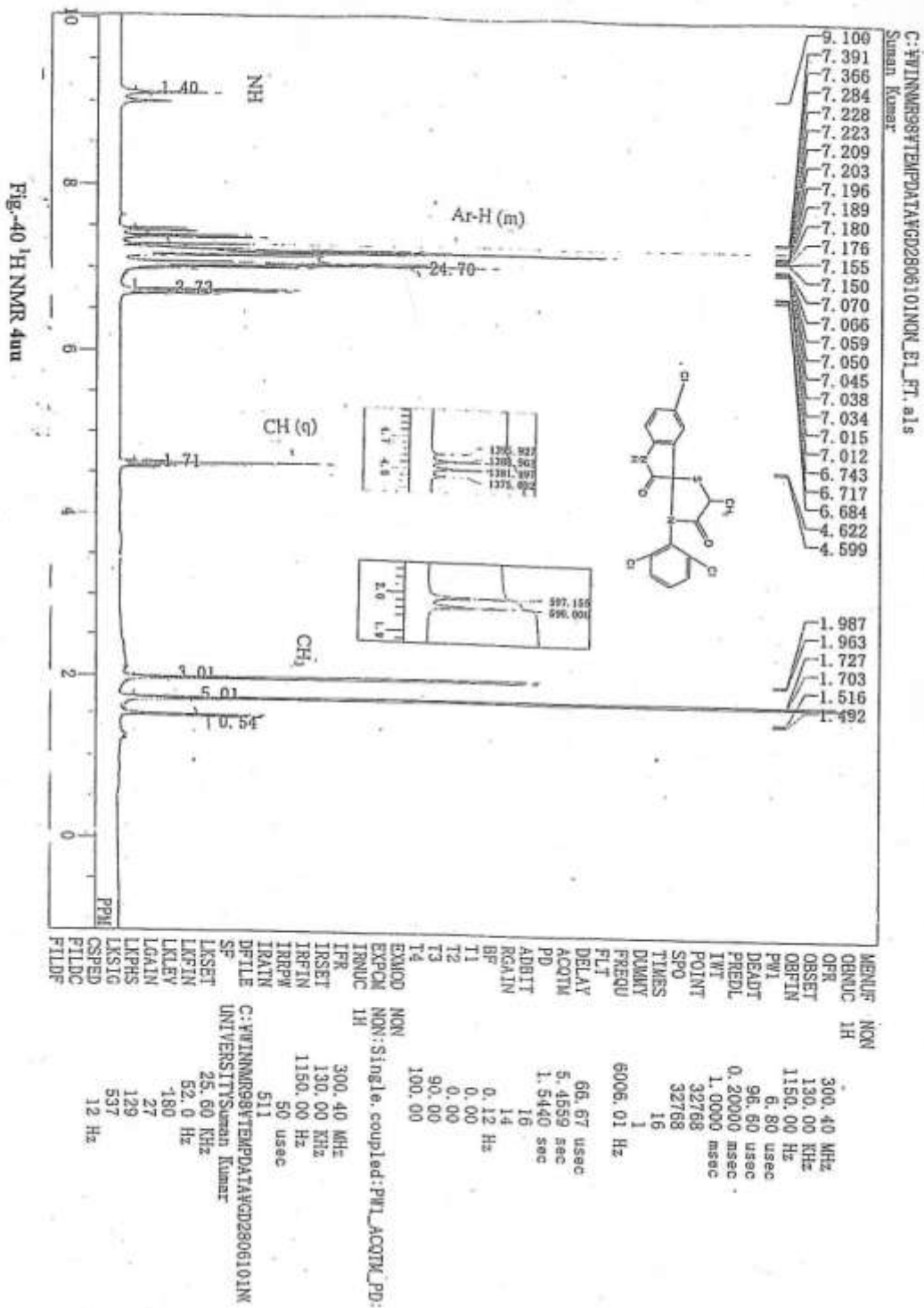


Fig.-41 ^{13}C NMR 4uu

