Supplementary Material

1,3,4-Thiadiazole-containing Azo Dyes: Synthesis, Spectroscopic Properties and Molecular Structure

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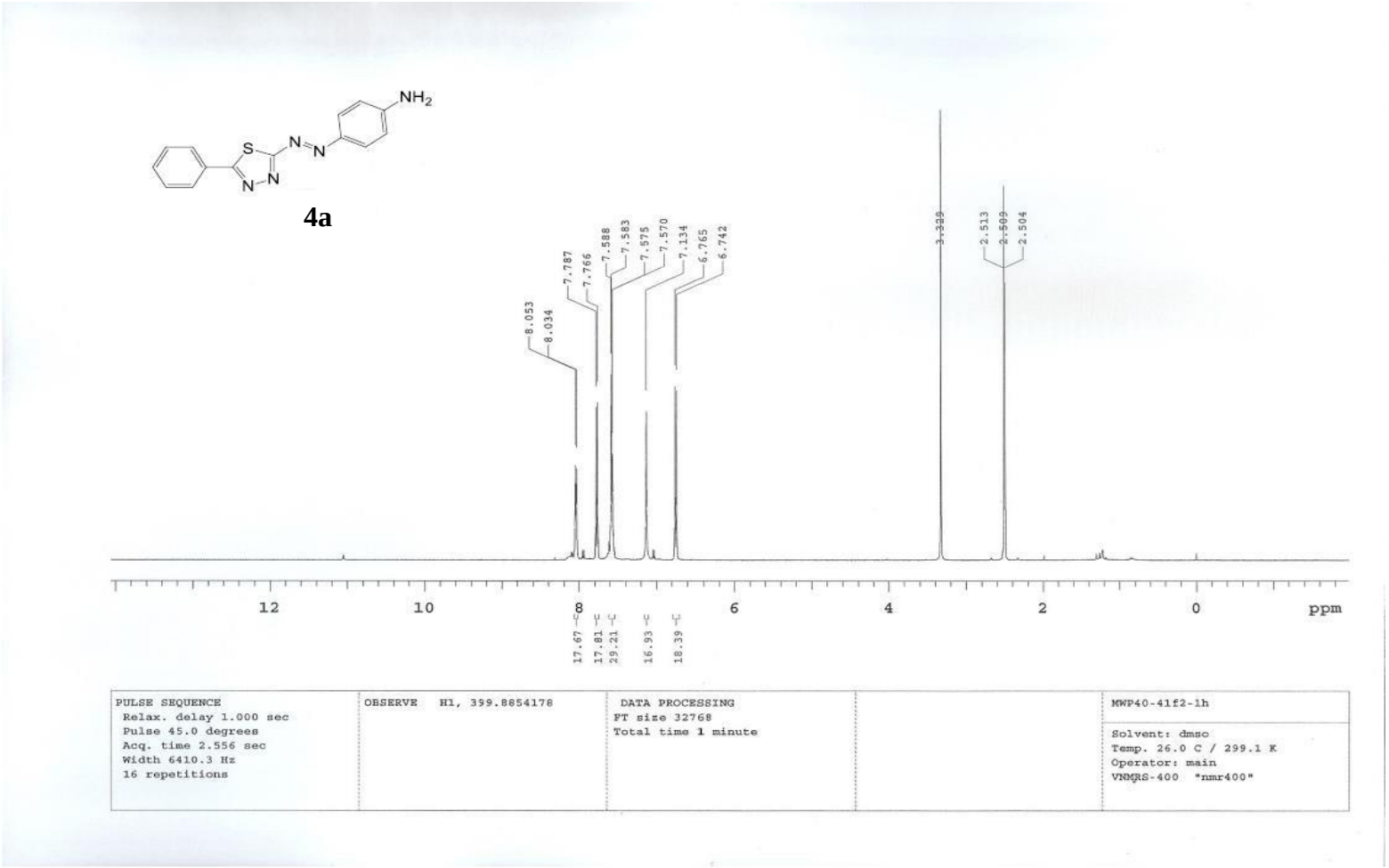
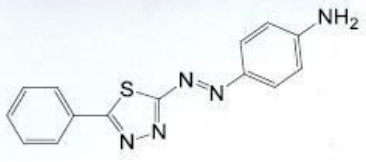
1. ^{1}H and ^{13}C NMR spectra

2-(4-Aminophenylazo)-5-phenyl-1,3,4-thiadiazole (**4a**)

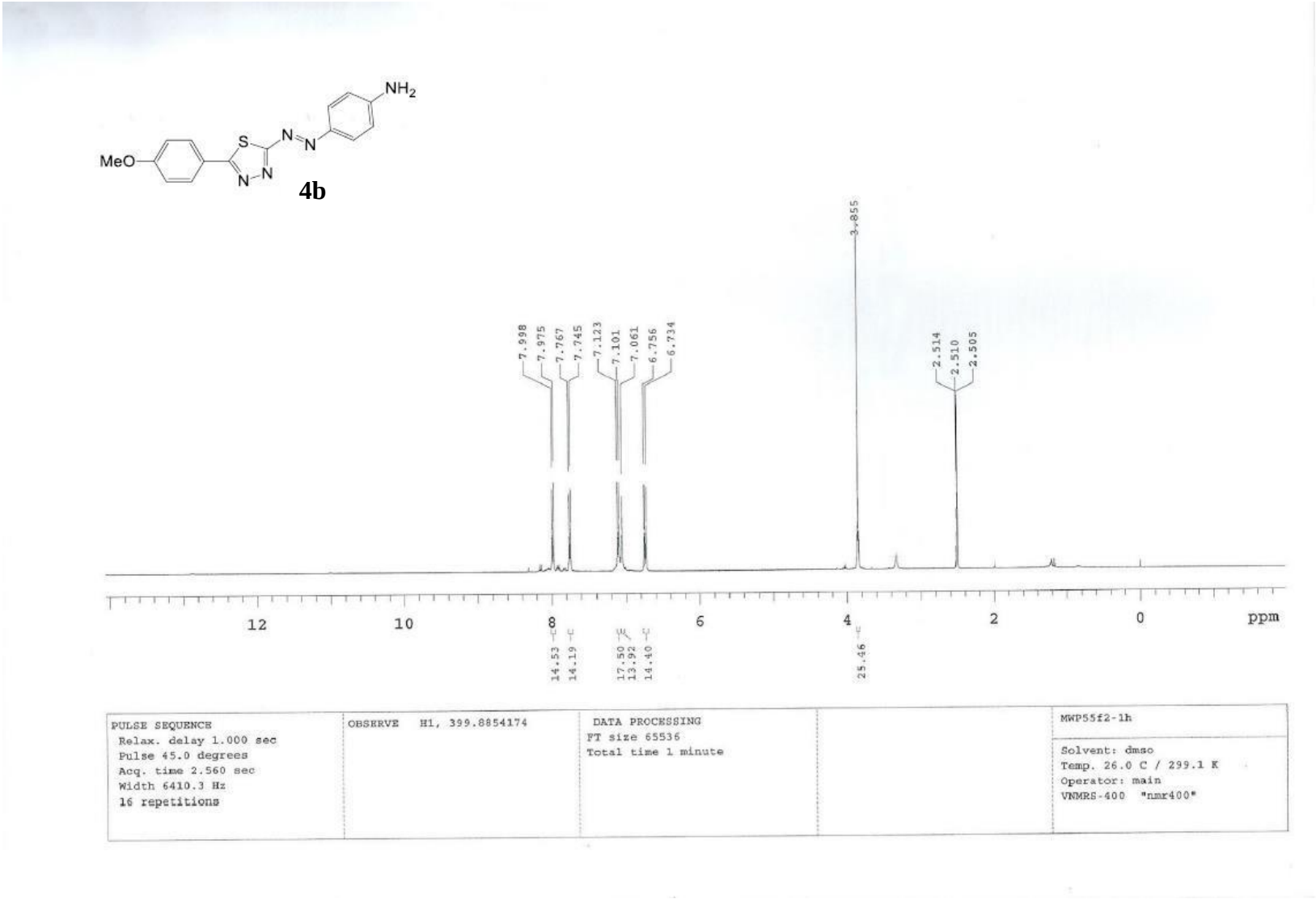


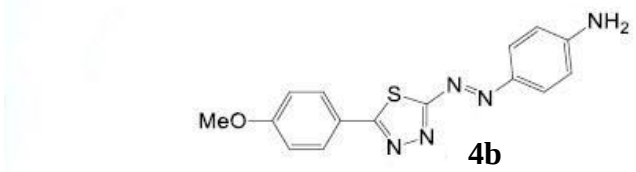


4a

PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.330 sec Width 25000.0 Hz 160 repetitions	OBSERVE C13, 100.5513209 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 131072 Total time 6 minutes		MWP40-41f2-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

2-(4-Aminophenylazo)-5-(4-methoxyphenyl)-1,3,4-thiadiazole (**4b**)





4b

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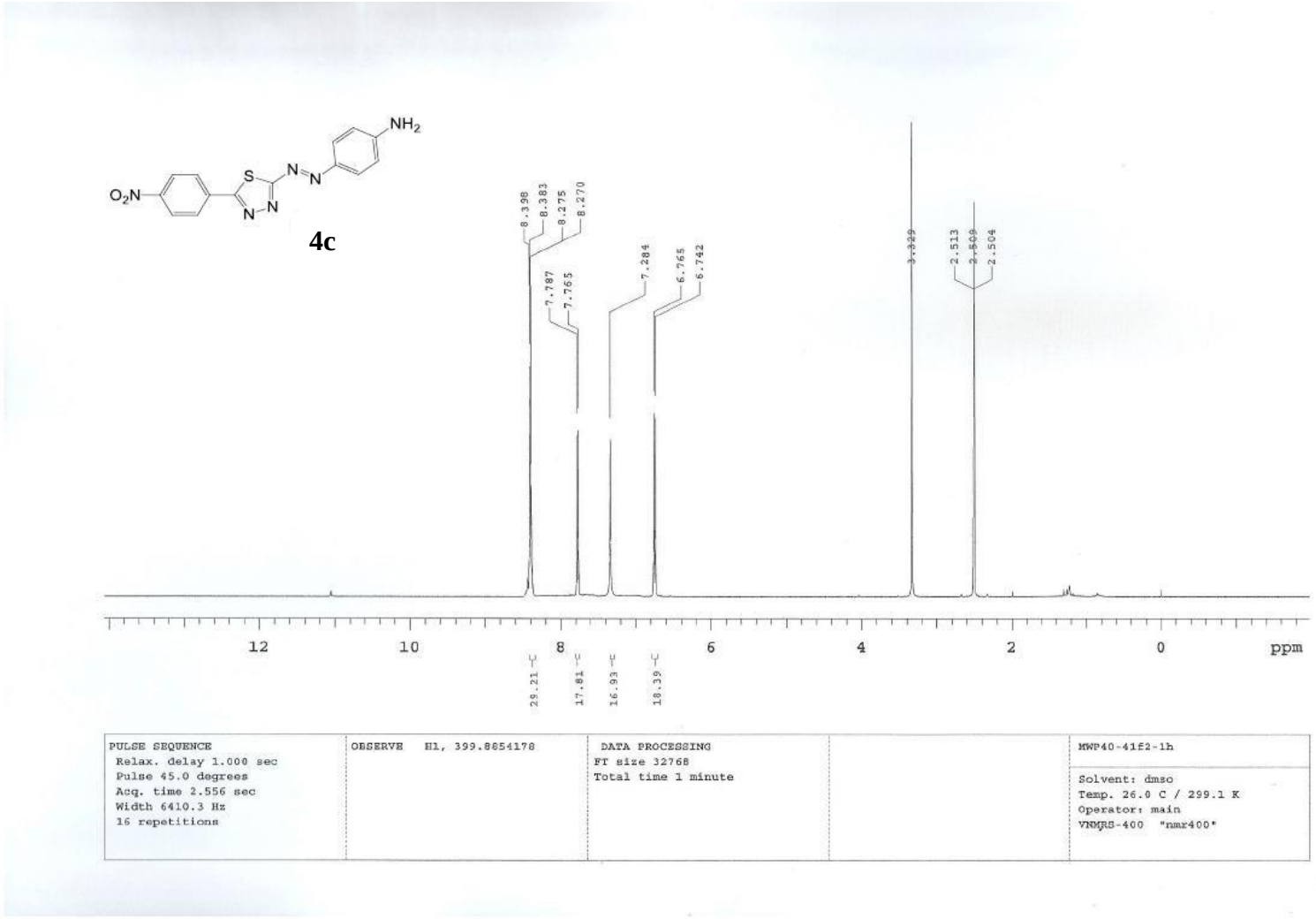
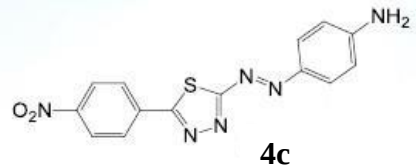
2-(4-Aminophenylazo)-5-(4-nitrophenyl)-1,3,4-thiadiazole (**4c**)





4c

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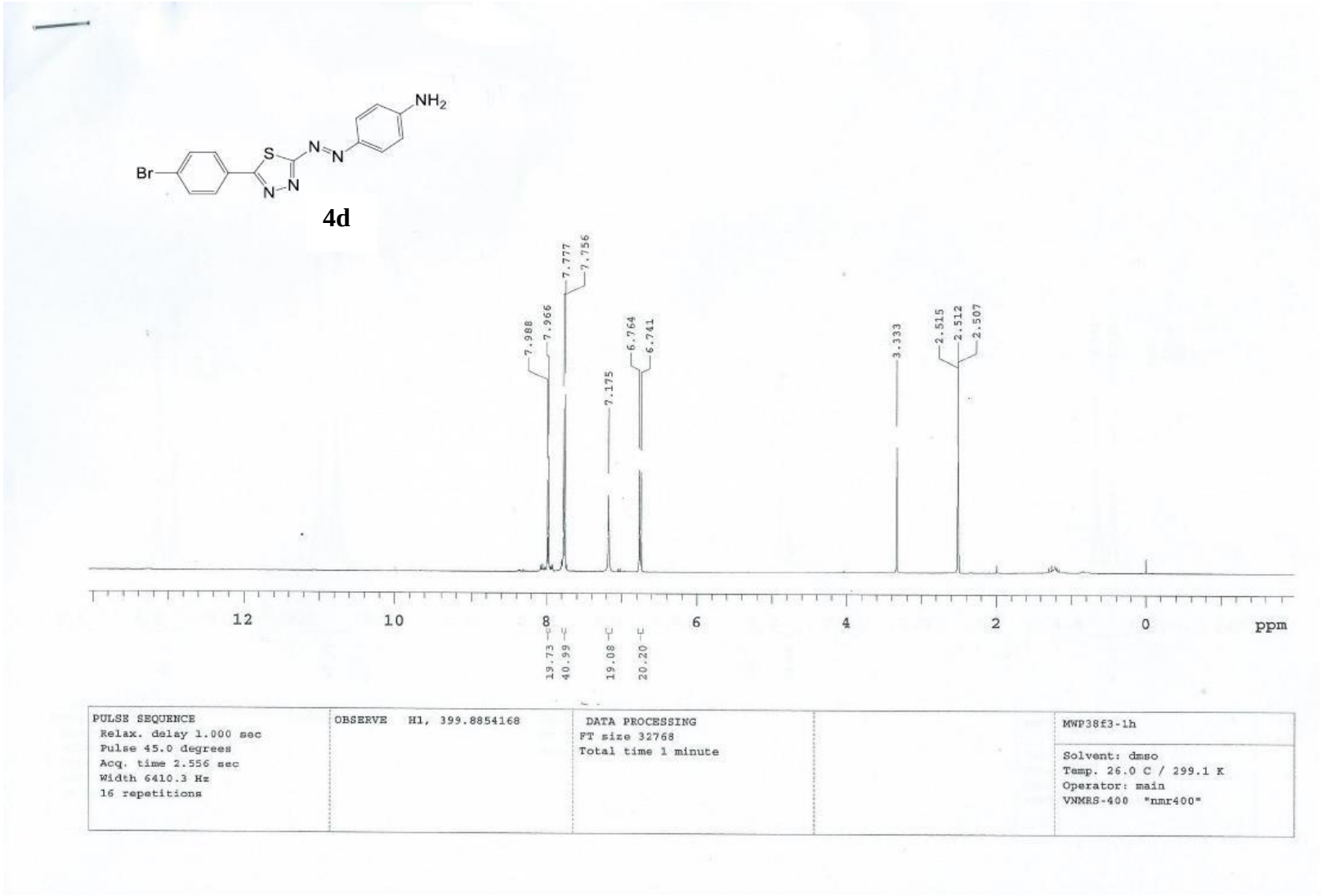
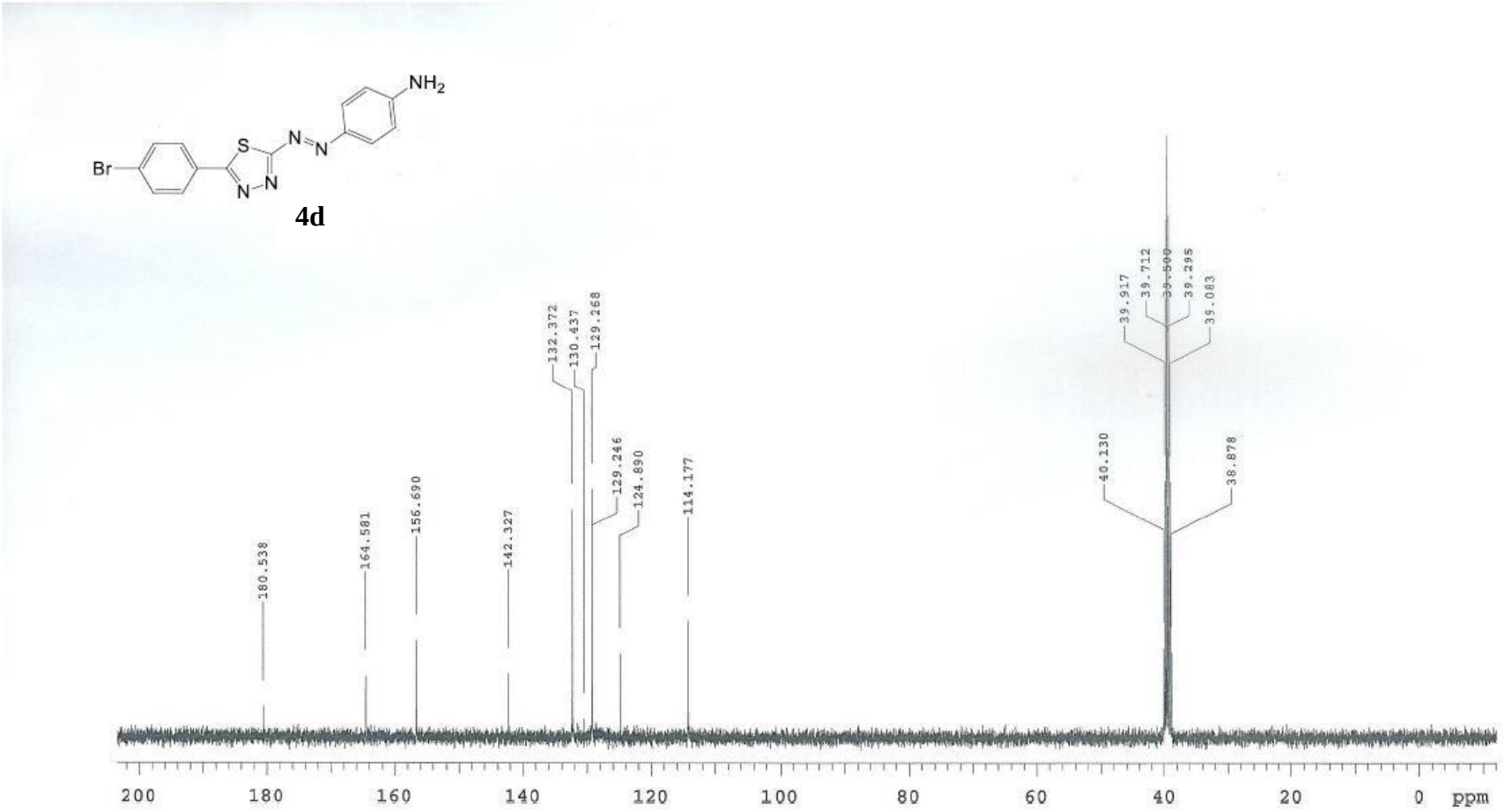
2-(4-Aminophenylazo)-5-(4-bromophenyl)-1,3,4-thiadiazole (**4d**)





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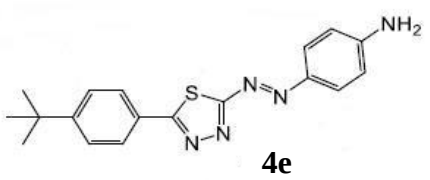
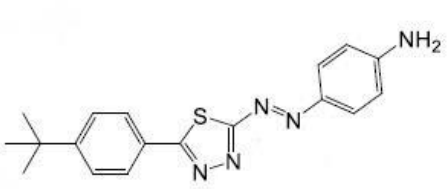
2-(4-Aminophenylazo)-5-(4-t-butylphenyl)-1,3,4-thiadiazole (**4e**)



4e

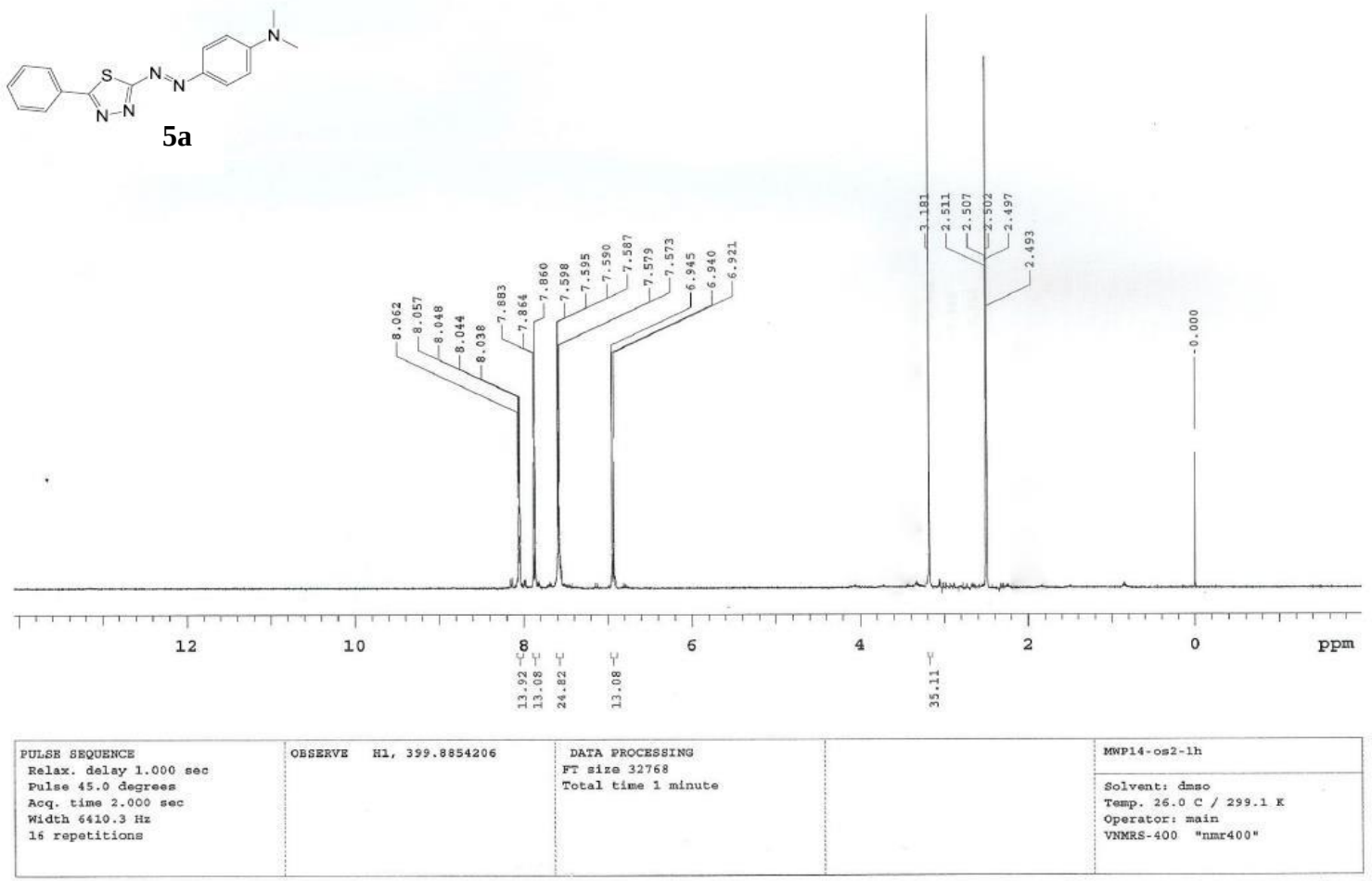
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.556 sec Width 6410.3 Hz 16 repetitions	OBSERVE H1, 399.8854199	DATA PROCESSING FT size 32768 Total time 1 minute		MWP52f2-1h Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

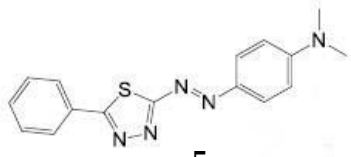


4e

PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.311 sec Width 25000.0 Hz 288 repetitions	OBSERVE C13, 100.5513209 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 11 minutes		MWP52f2-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

2-[4-(*N*,*N*-Dimethylamino)phenylazo]-5-phenyl-1,3,4-thiadiazole (**5a**)

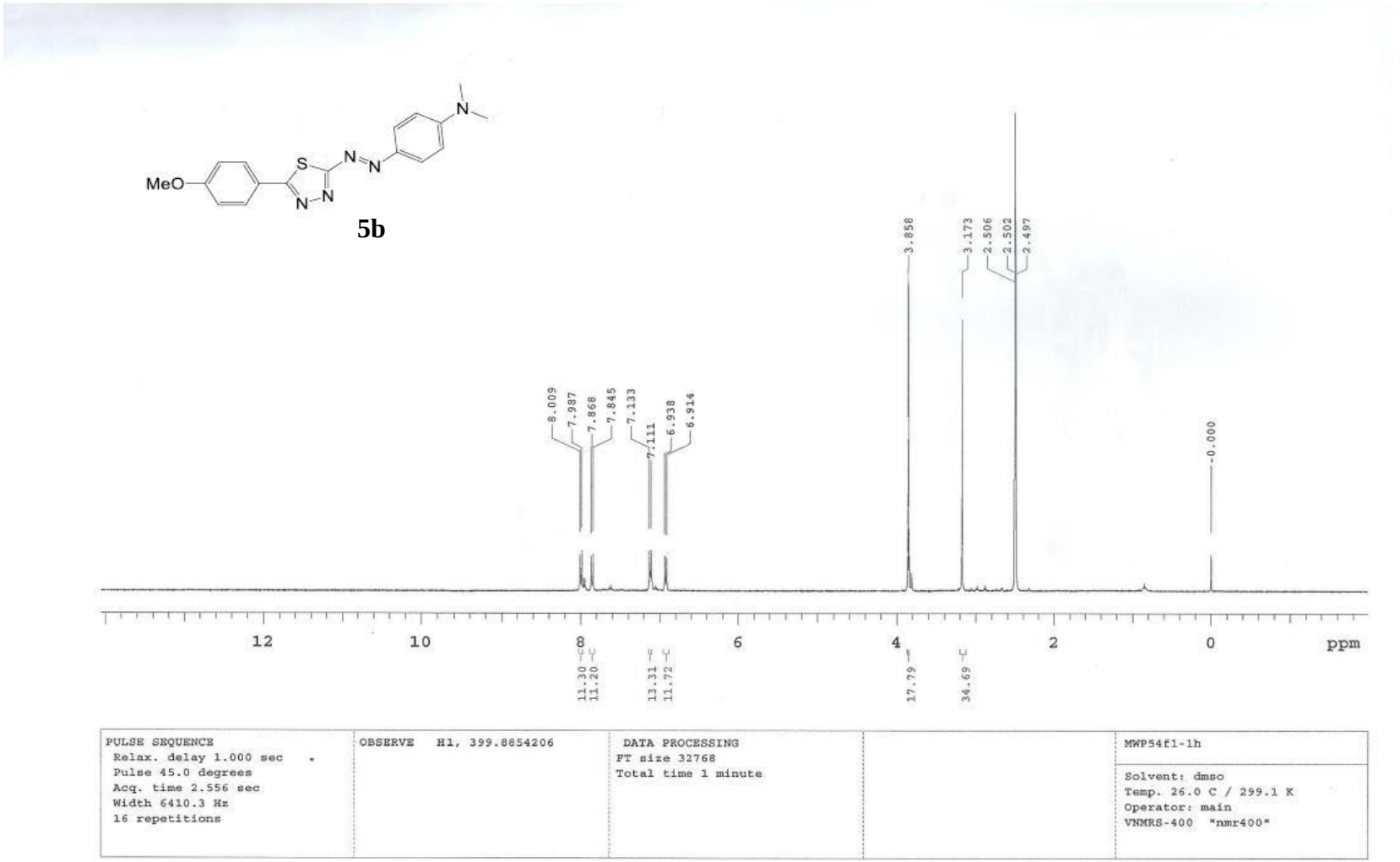


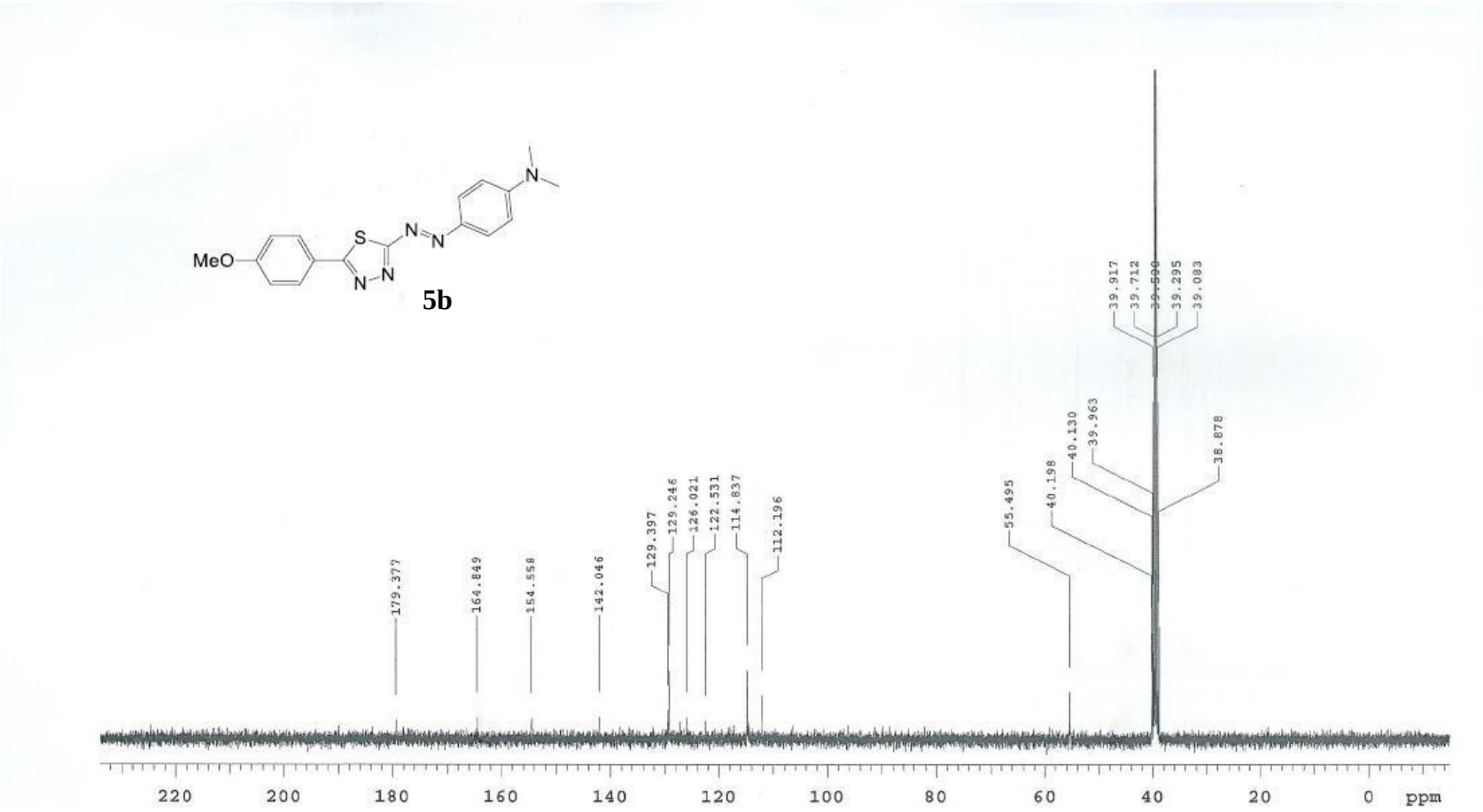


5a

PULSE SEQUENCE Relax. delay 2.000 sec Pulse 45.0 degrees Acq. time 1.311 sec Width 25000.0 Hz 1000 repetitions	OBSERVE C13, 100.5513217 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 55 minutes		MWP14-os2-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

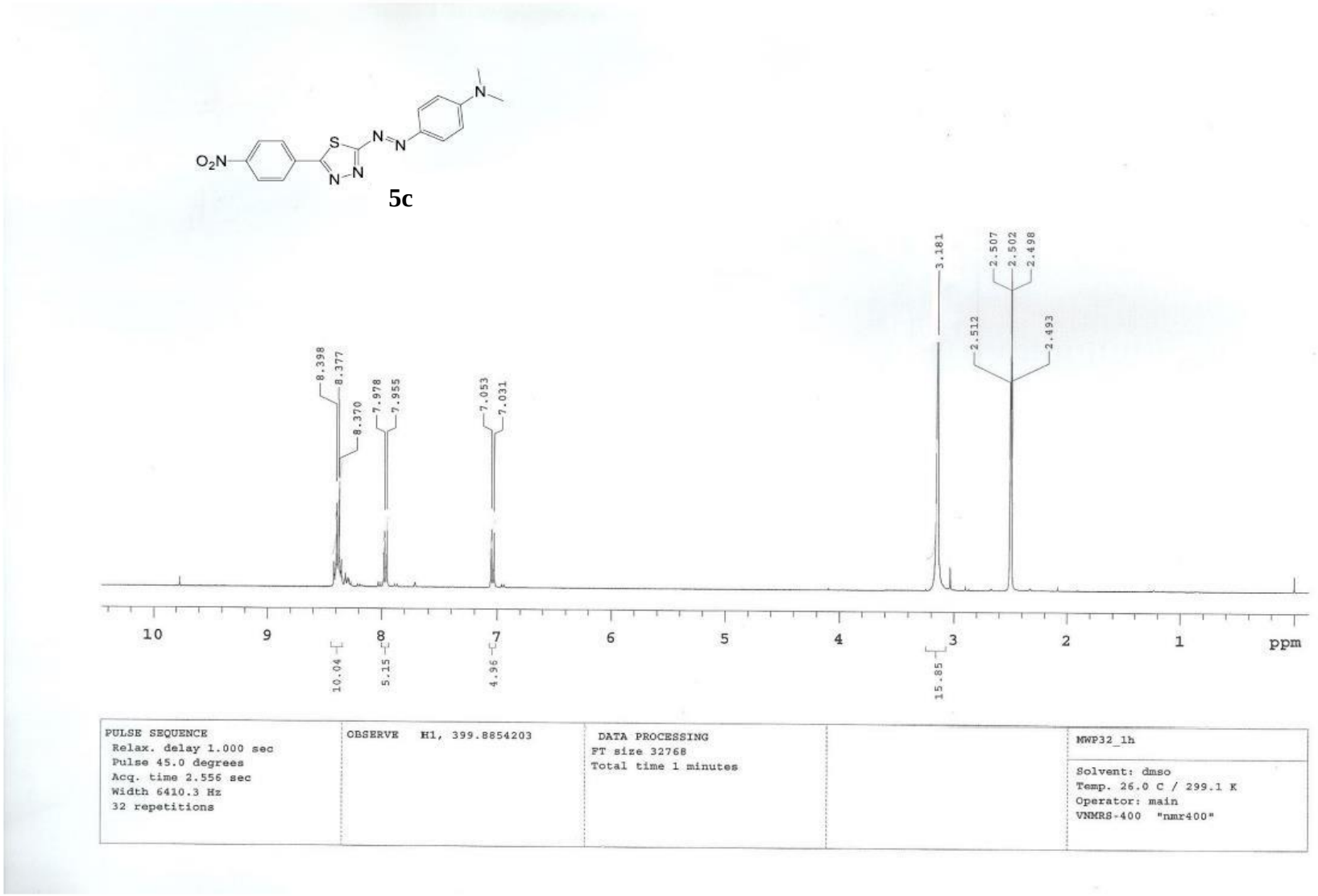
2-[4-(*N*,*N*-Dimethylamino)phenylazo]-5-(4-methoxyphenyl)-1,3,4-thiadiazole (**5b**)

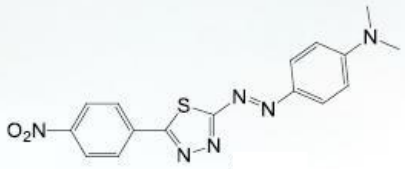




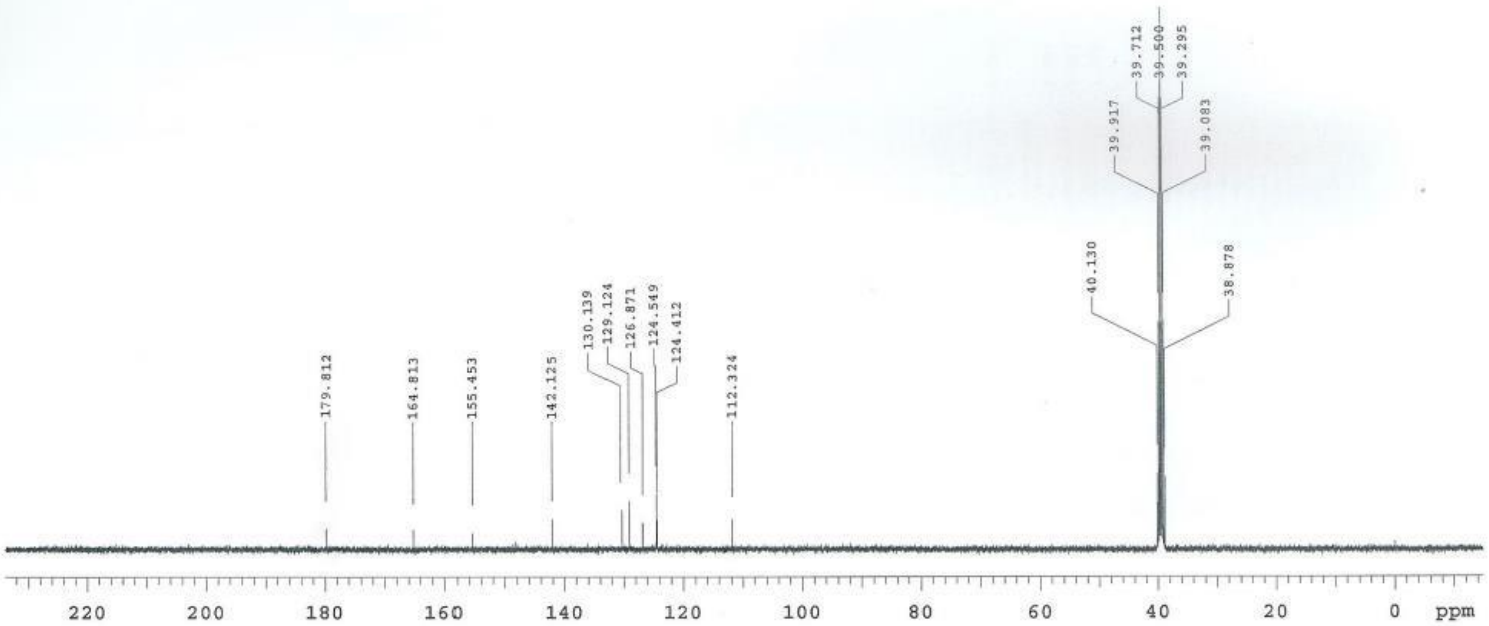
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.311 sec Width 25000.0 Hz 320 repetitions	OBSERVE C13, 100.5513202 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 12 minutes		MWP54-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

2-[4-(*N*,*N*-Dimethylamino)phenylazo]-5-(4-nitrophenyl)-1,3,4-thiadiazole (**5c**)





5c



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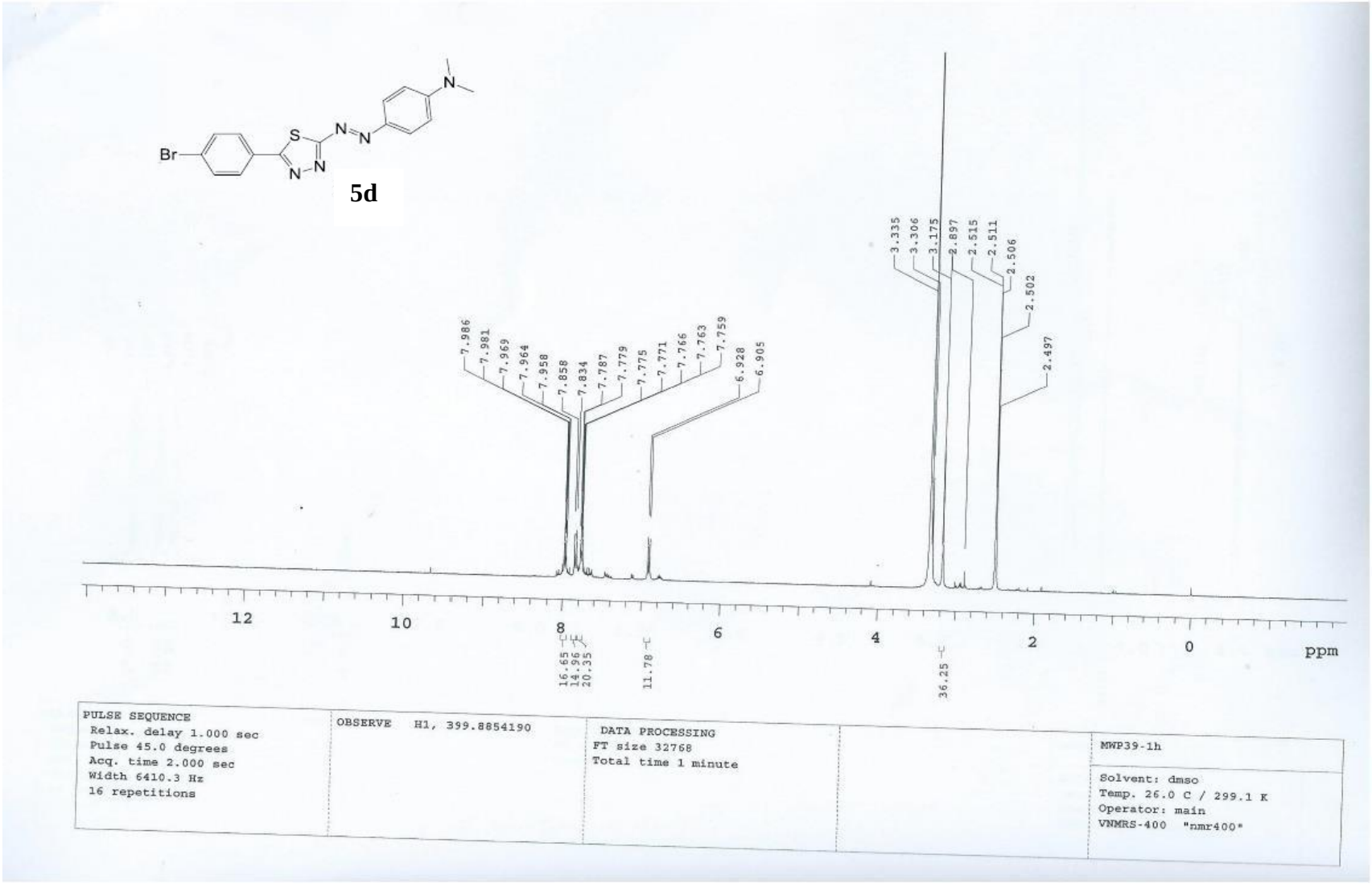
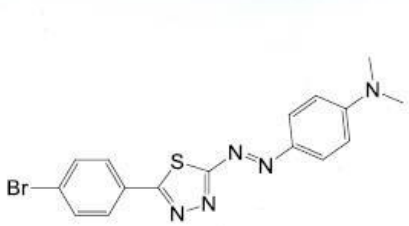
5-(4-Bromophenyl)-2-[4-(N,N-dimethylamino)phenylazo]-1,3,4-thiadiazole (**5d**)

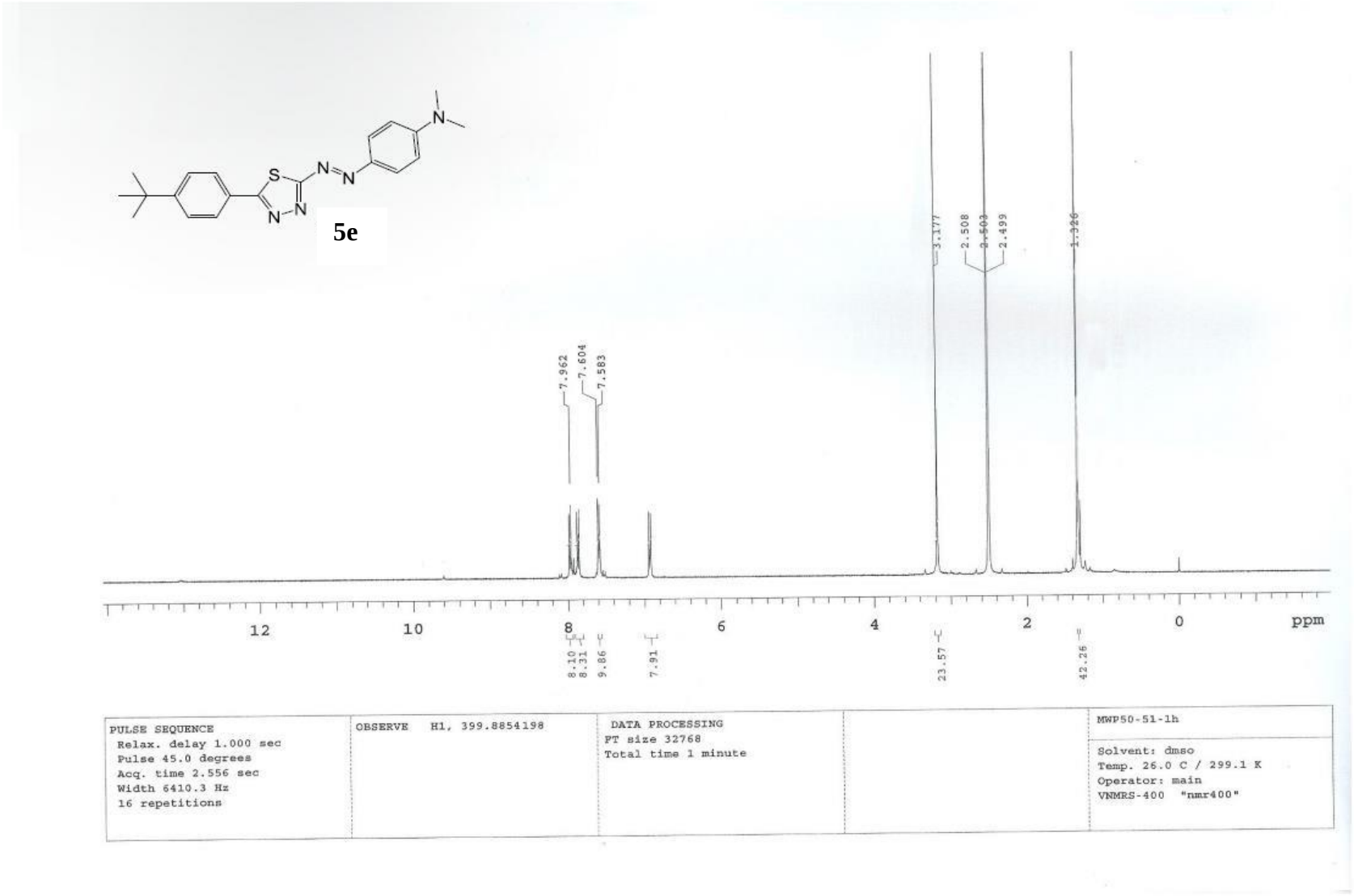


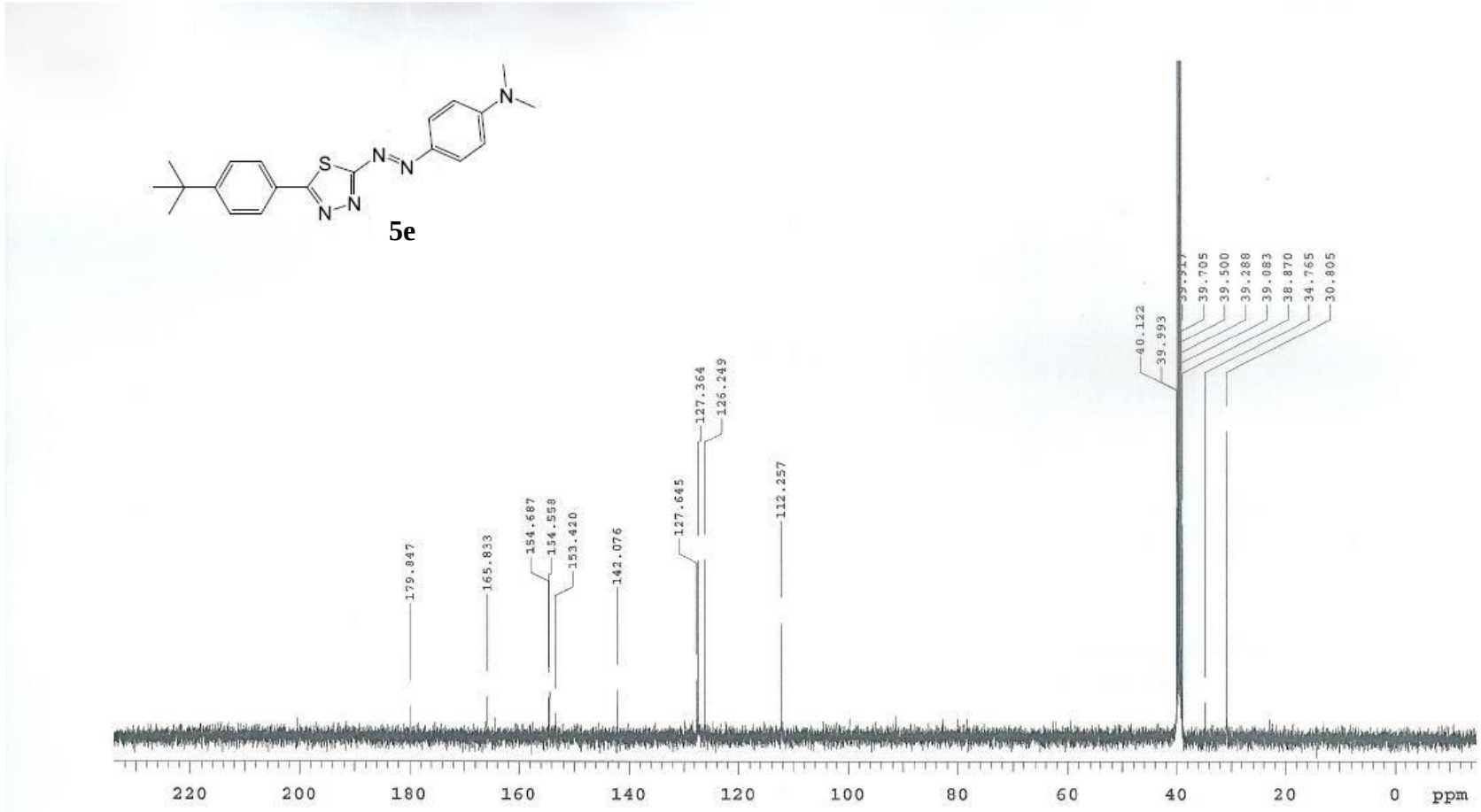


5d

PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.311 sec Width 25000.0 Hz 480 repetitions	OBSERVE C13, 100.5513194 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 18 minutes		MWP39-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

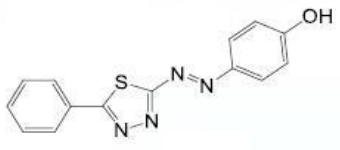
5-(4-*t*-Butylphenyl)-2-[4-(*N*,*N*-dimethylamino)phenylazo]-1,3,4-thiadiazole (**5e**)



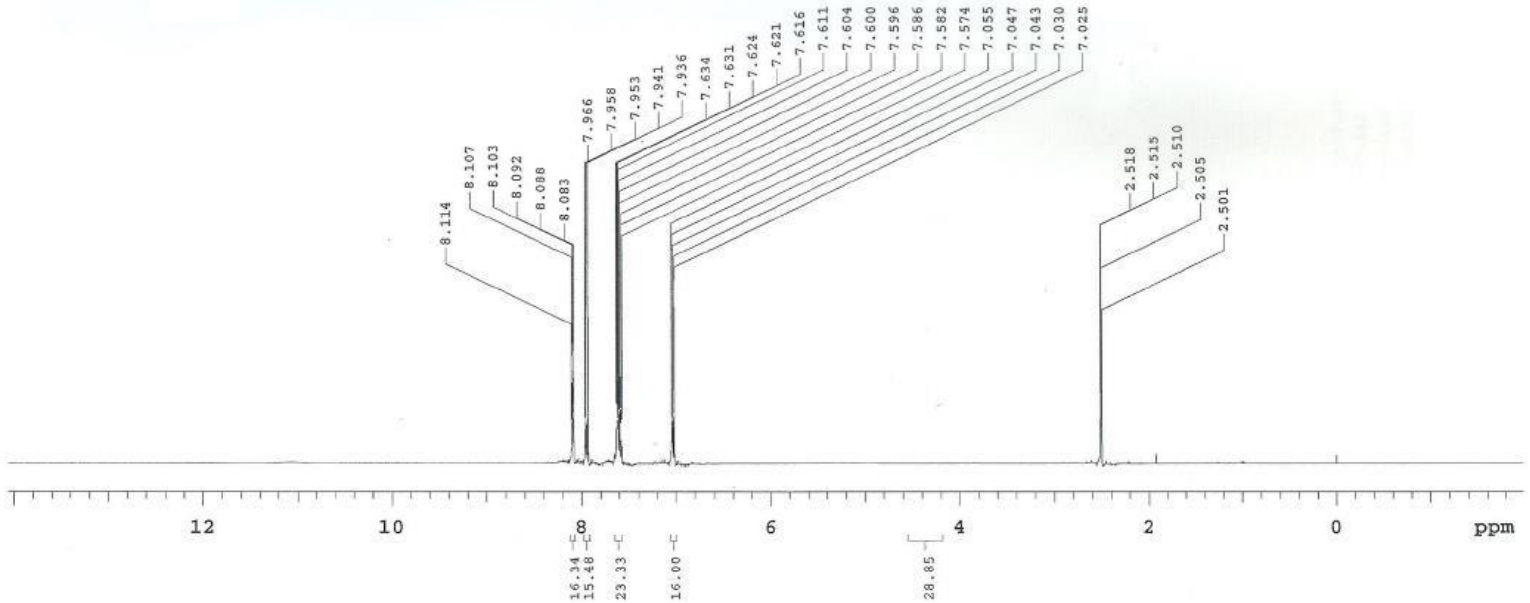


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 0.880 sec Width 25000.0 Hz 1472 repetitions	OBSERVE C13, 100.5513202 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 46 minutes		MWP50-51-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

2-(4-Hydroxyphenylazo)-5-phenyl-1,3,4-thiadiazole (**6a**)



6a



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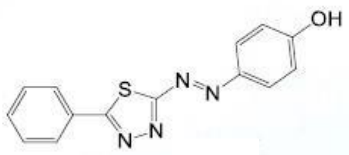
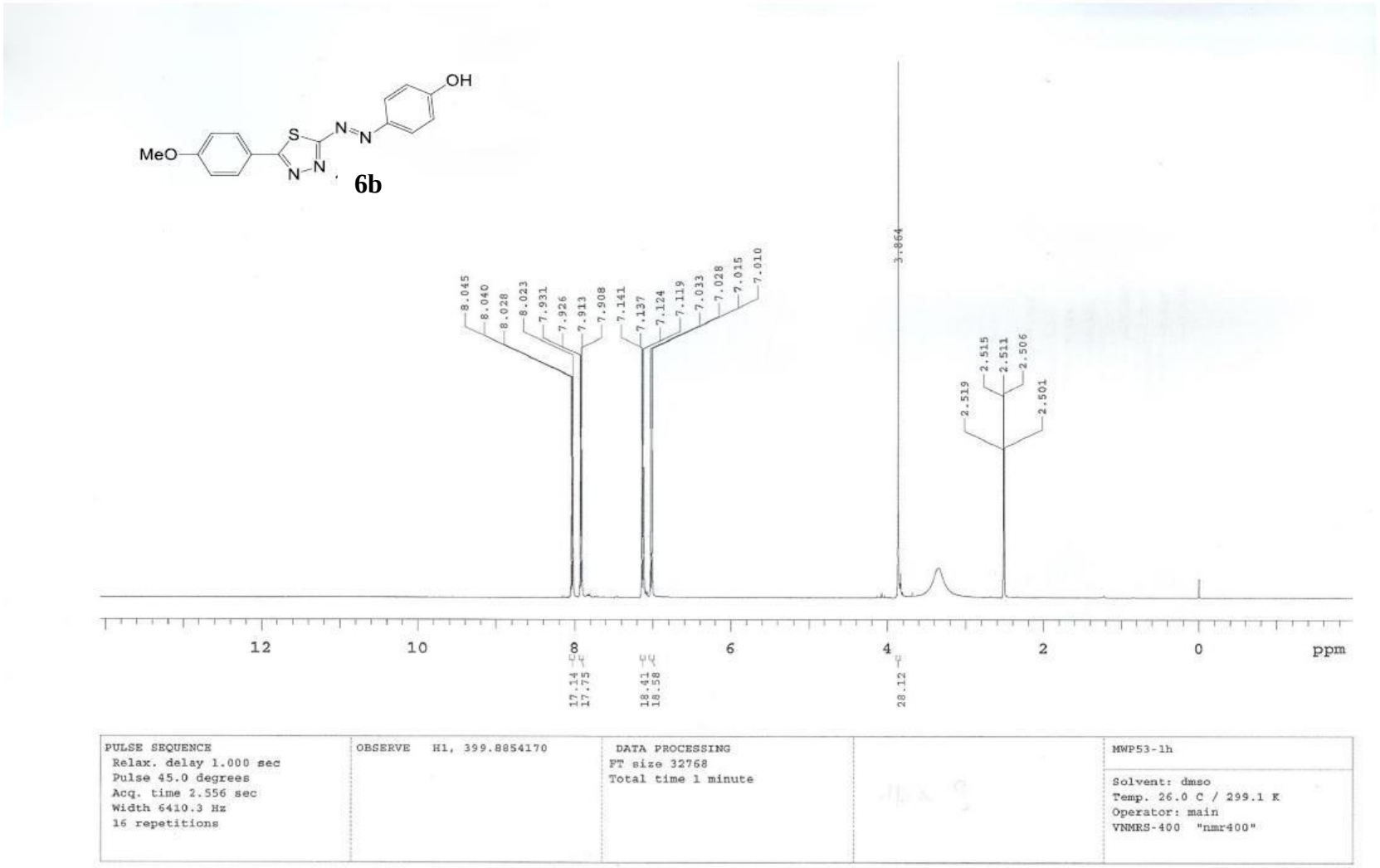
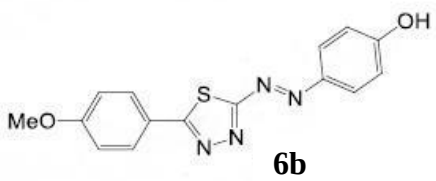


6a

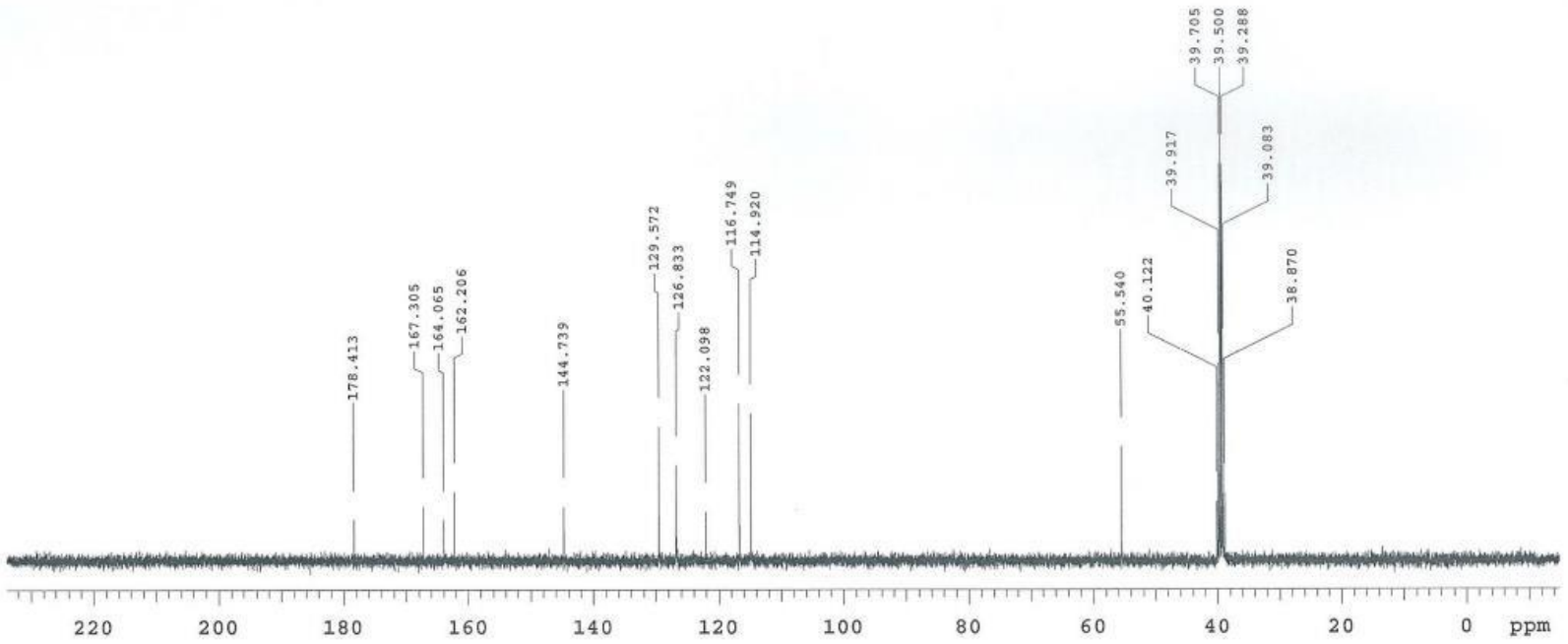
PULSE SEQUENCE Relax. delay 2.000 sec Pulse 45.0 degrees Acq. time 1.311 sec Width 25000.0 Hz 96 repetitions	OBSERVE C13, 100.5513202 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 5 minutes		MWP13-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

2-(4-Hydroxyphenylazo)-5-(4-methoxyphenyl)-1,3,4-thiadiazole (**6b**)





6b



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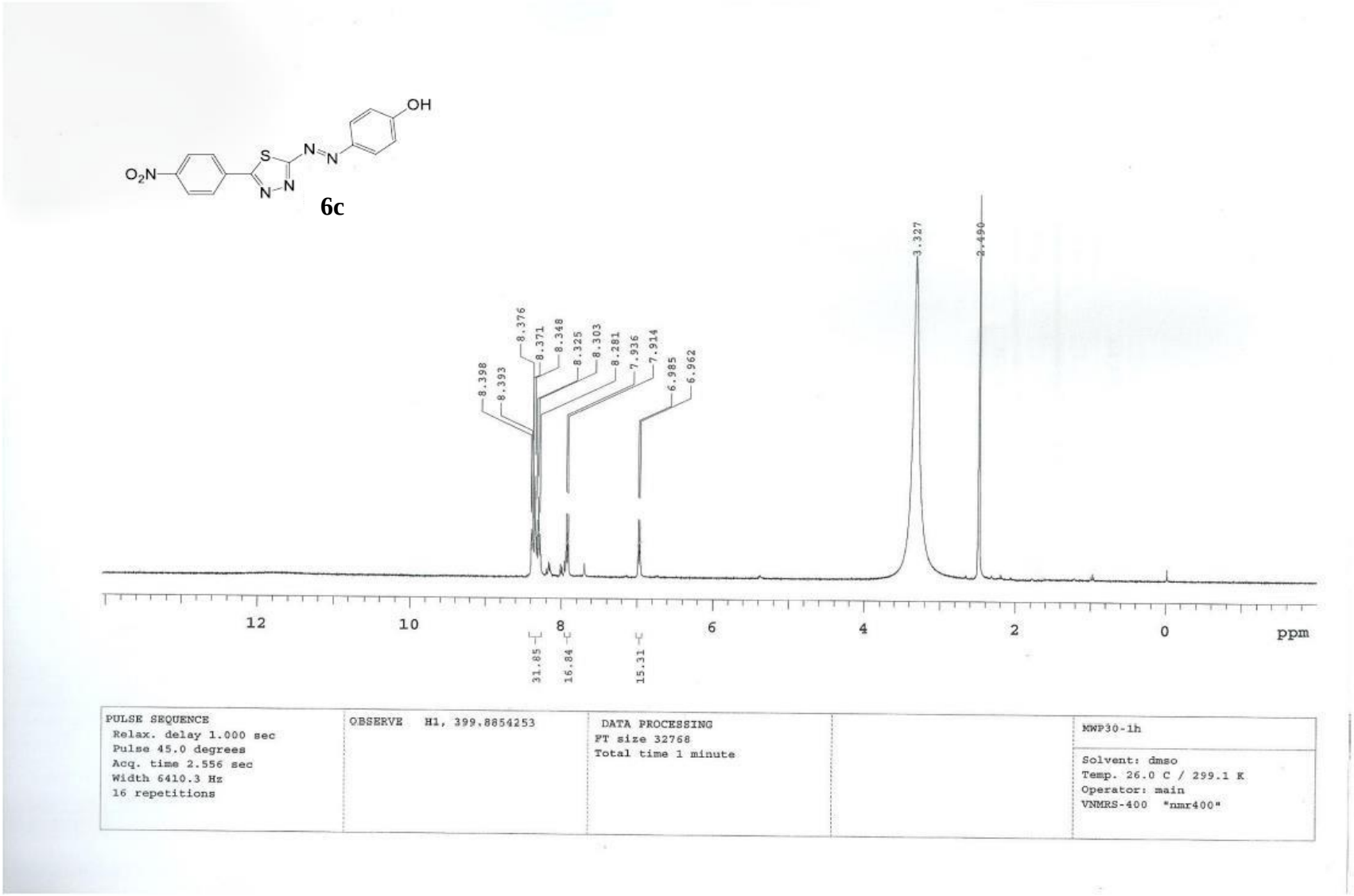
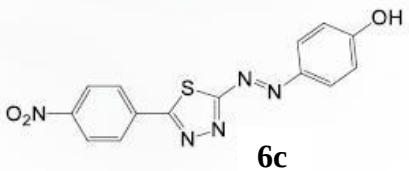
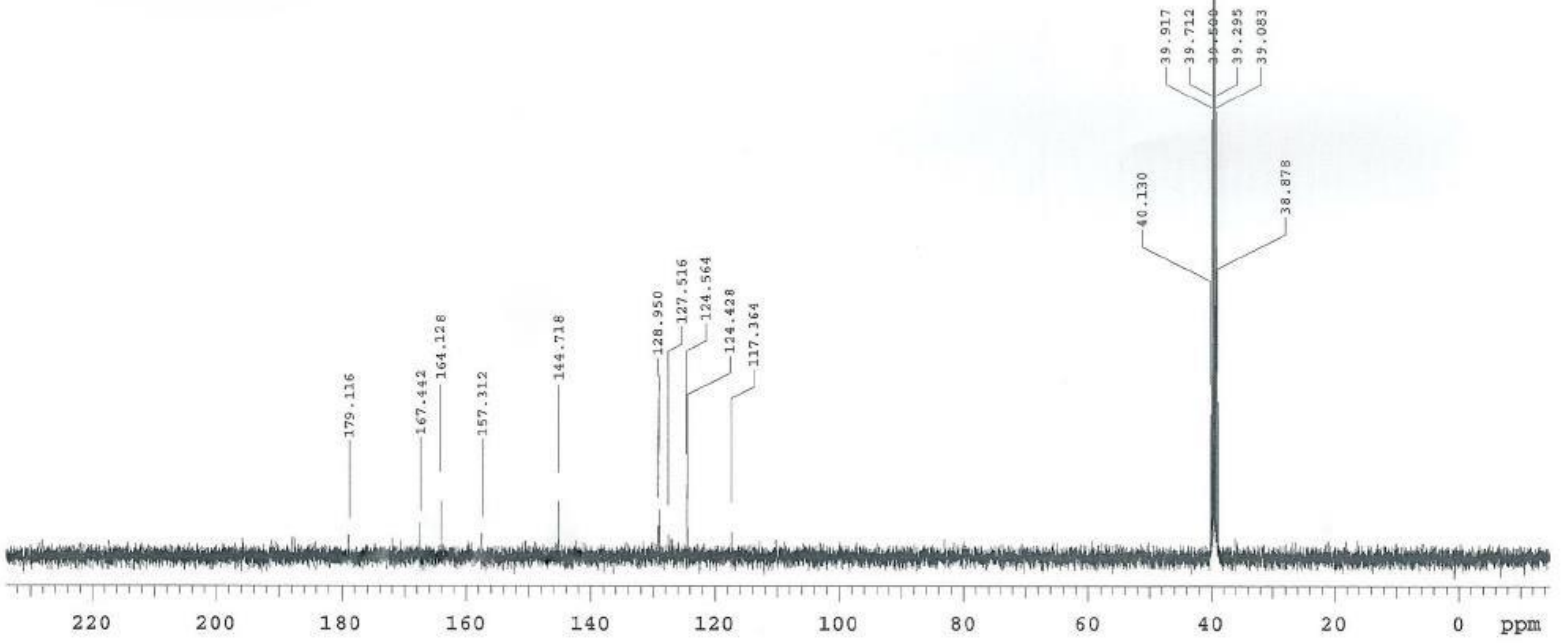
2-(4-Hydroxyphenylazo)-5-(4-nitrophenyl)-1,3,4-thiadiazole (**6c**)





6c



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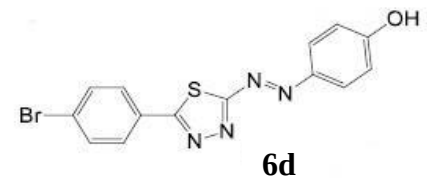
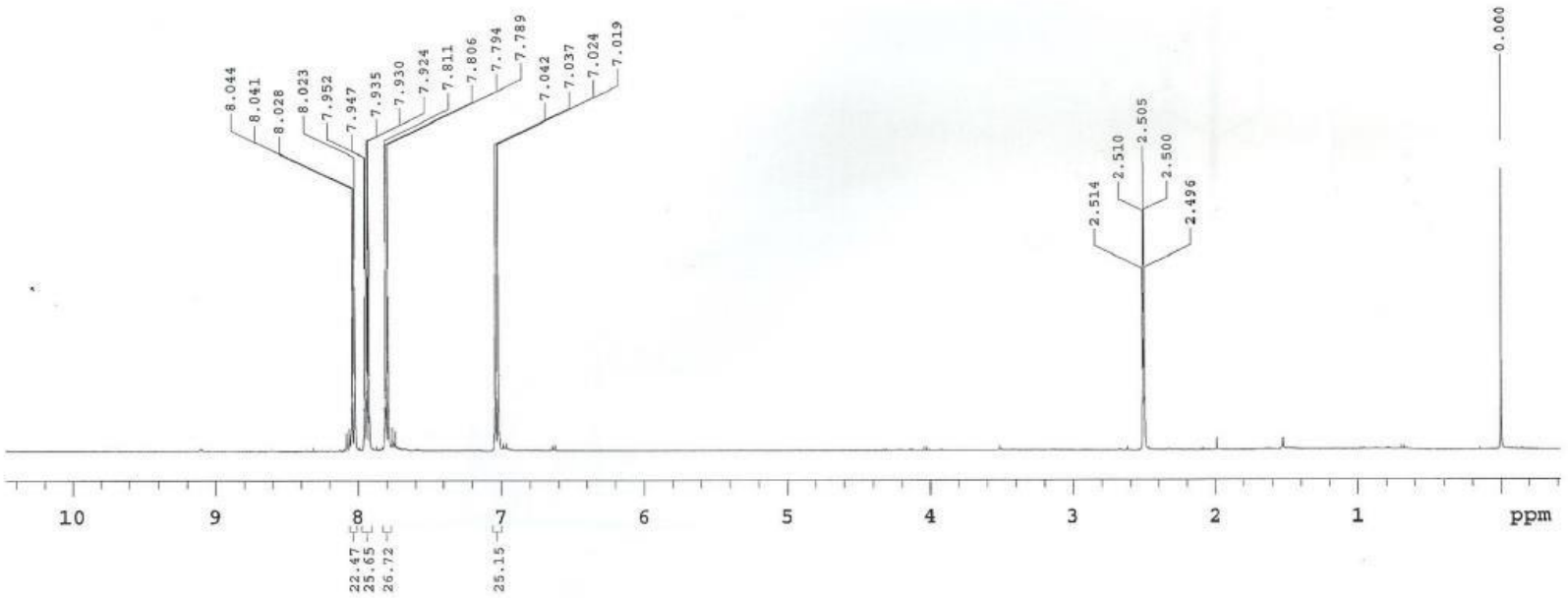
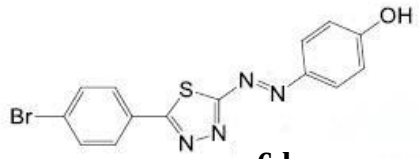
5-(4-Bromophenyl)-2-(4-hydroxyphenylazo)-1,3,4-thiadiazole (**6d**)



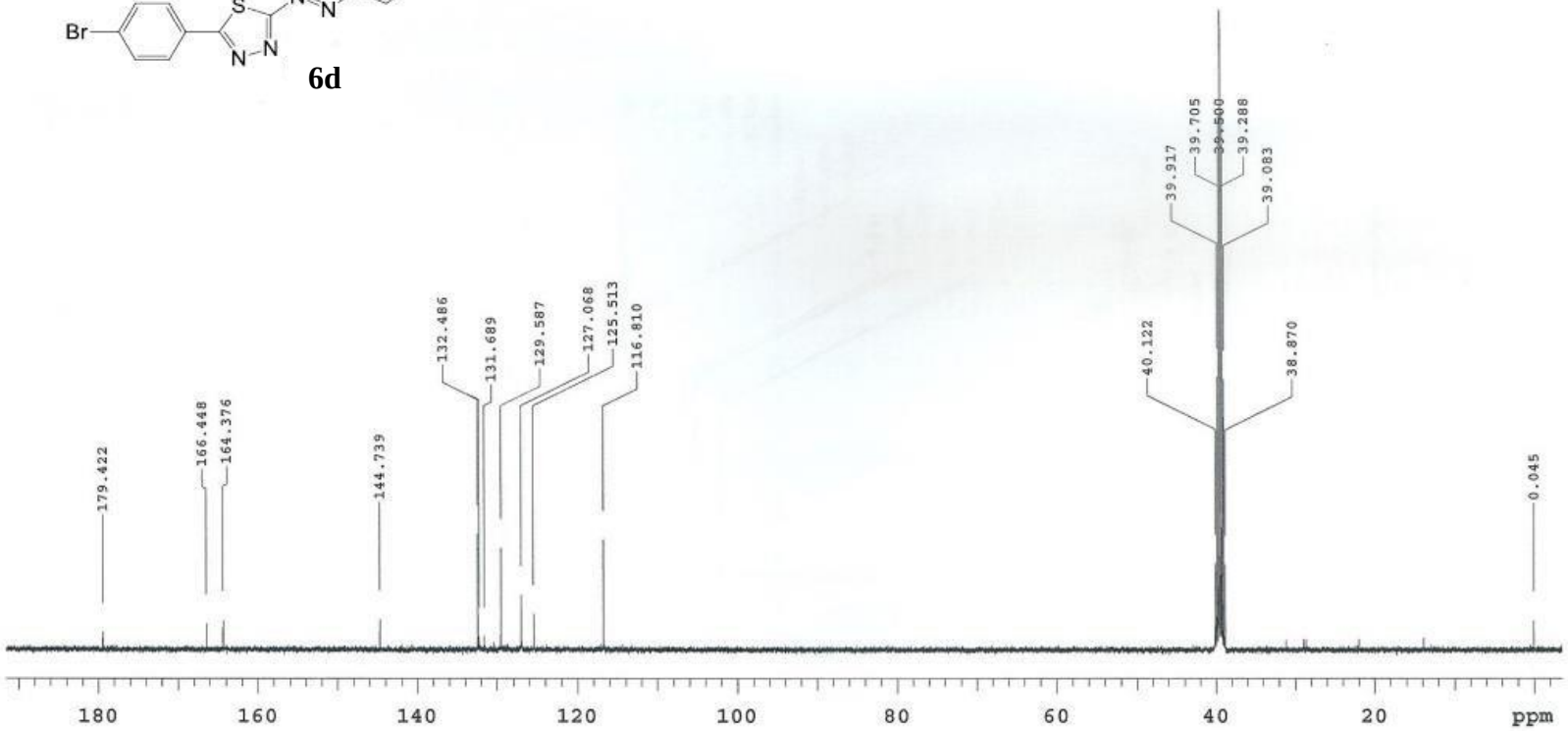
6d



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 90.0 degrees Acq. time 2.556 sec Width 6410.3 Hz 16 repetitions	OBSERVE H1, 399.8854194	DATA PROCESSING FT size 32768 Total time 1 minute		KJp05_1h Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"



6d



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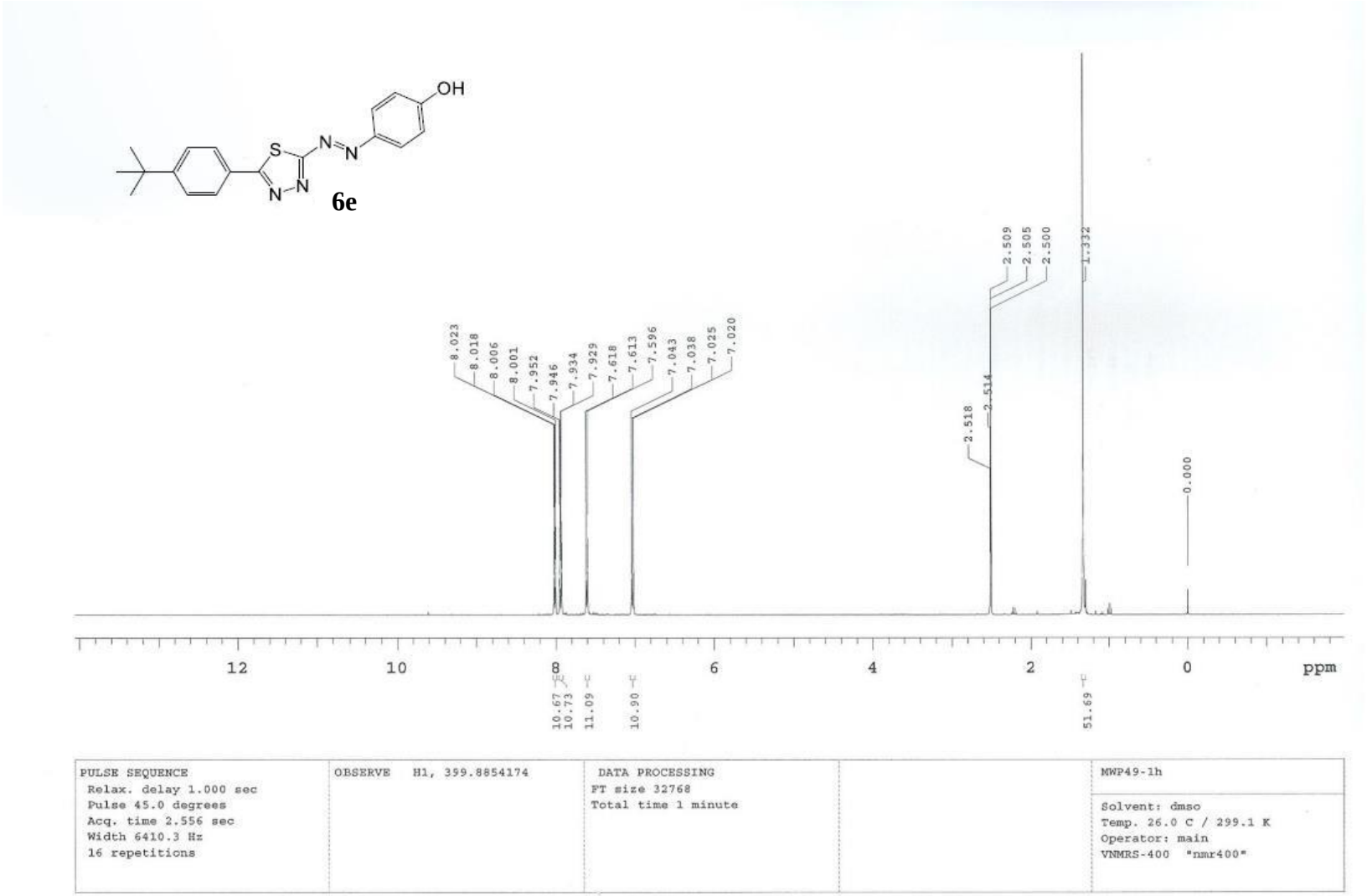
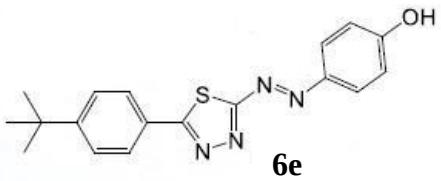
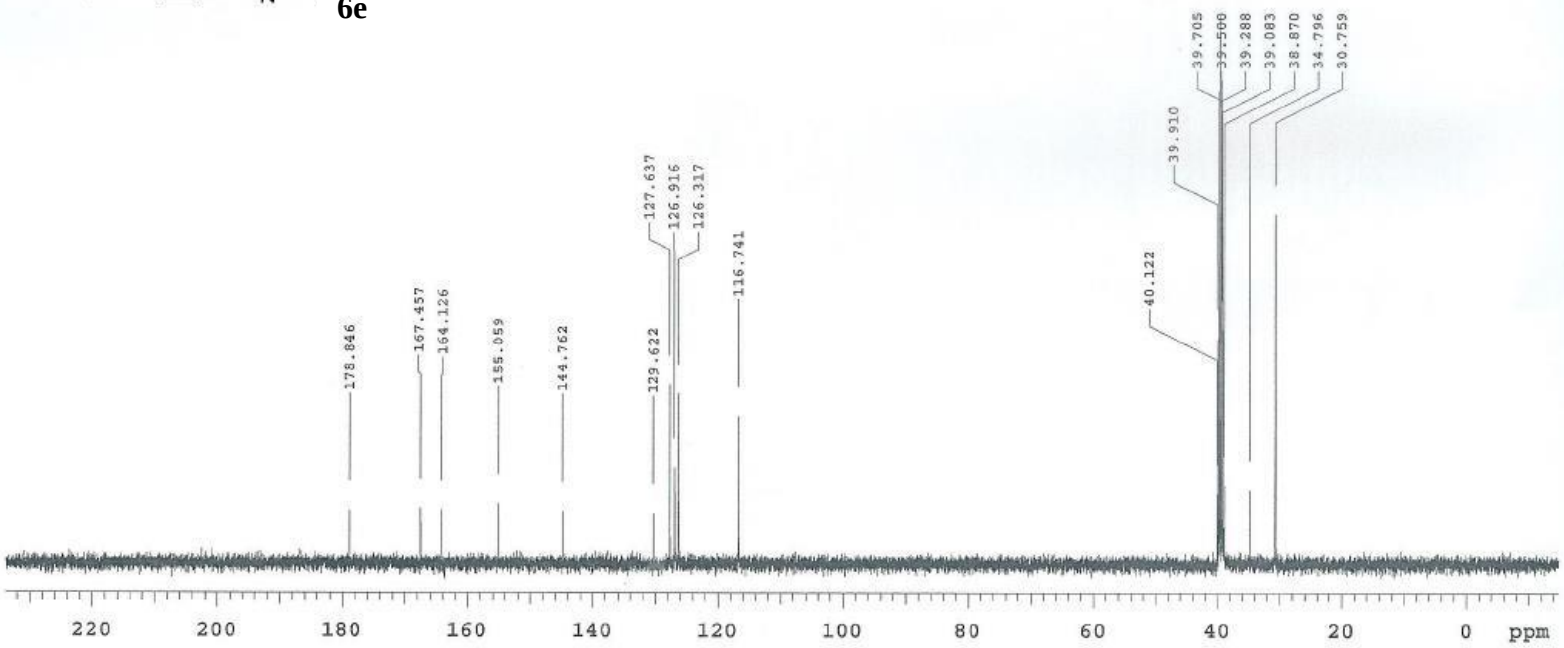
5-(4-t-Butylphenyl)-2-(4-hydroxyphenylazo)-1,3,4-thiadiazole (**6e**)



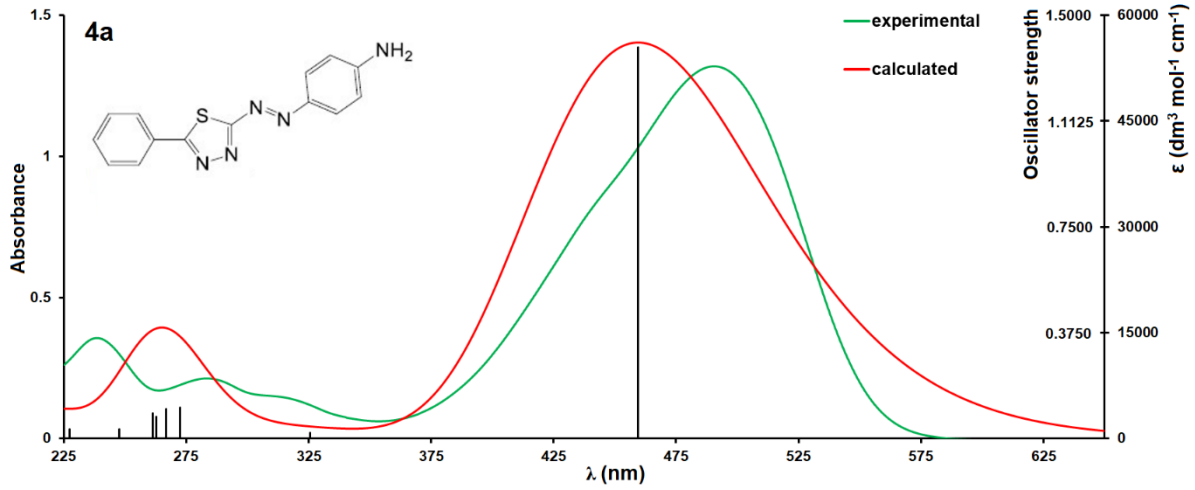


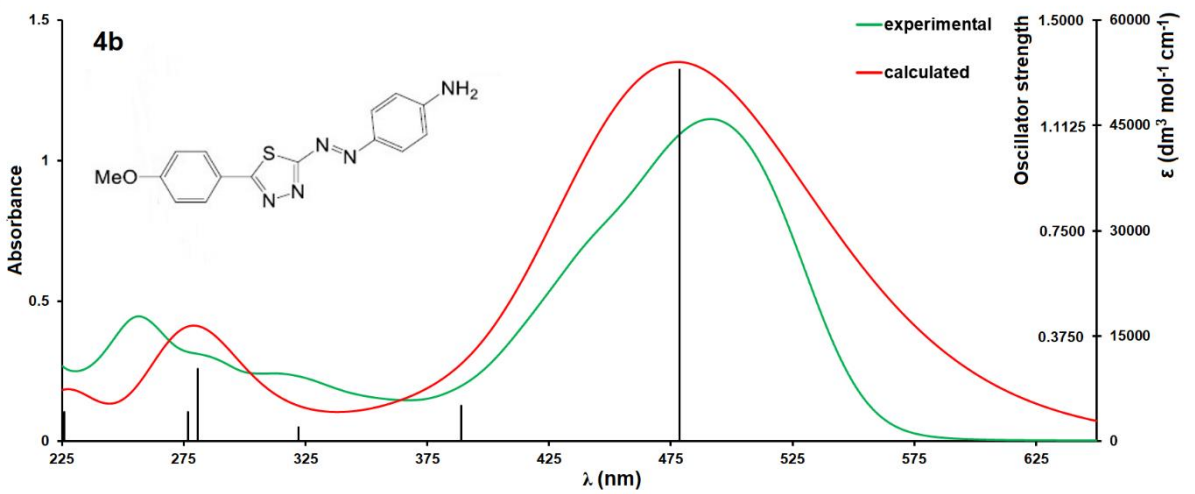
6e

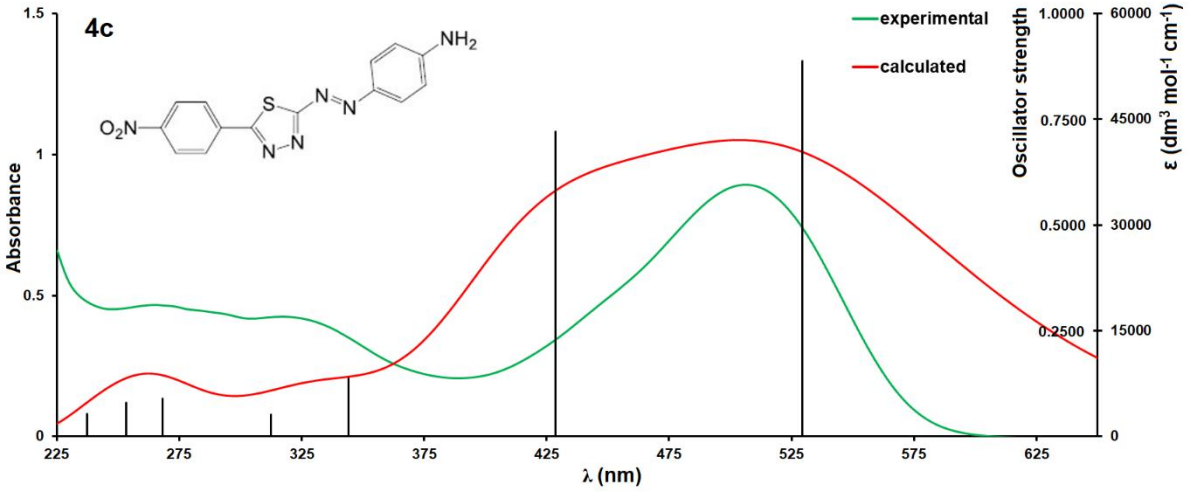


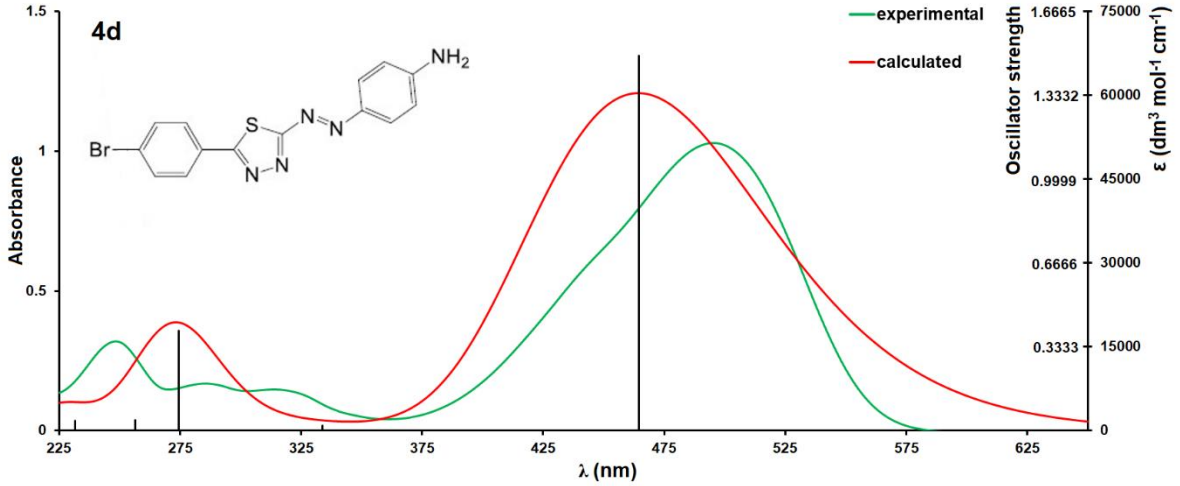
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.311 sec Width 25000.0 Hz 128 repetitions	OBSERVE C13, 100.5513209 DECOUPLE H1, 399.8874340 Power 37 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 4 minutes		MWP49-13c Solvent: dmso Temp. 26.0 C / 299.1 K Operator: main VNMRS-400 "nmr400"

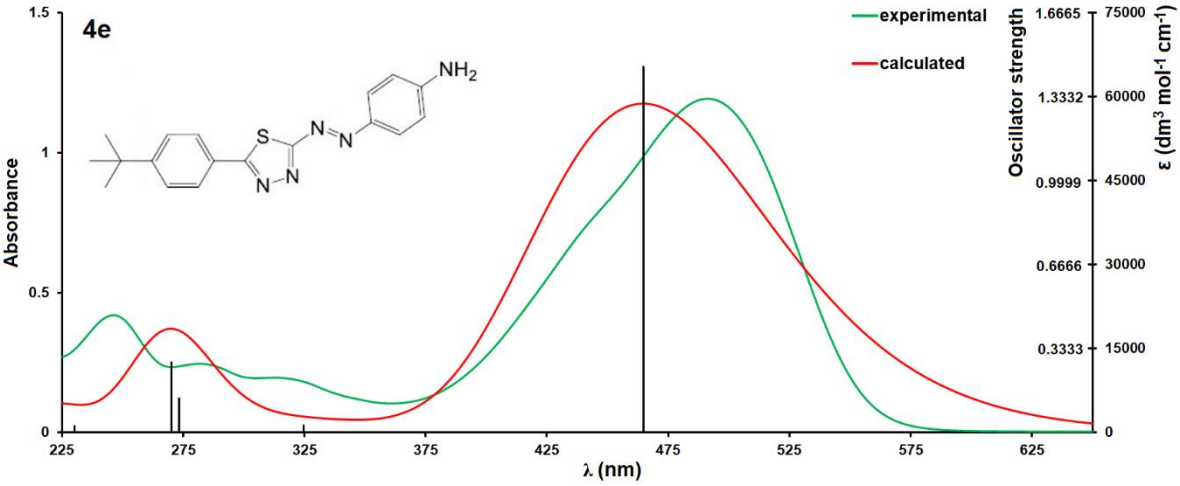
2. UV-Vis absorption data for compounds 4a-e, 5a-e, 6a-e

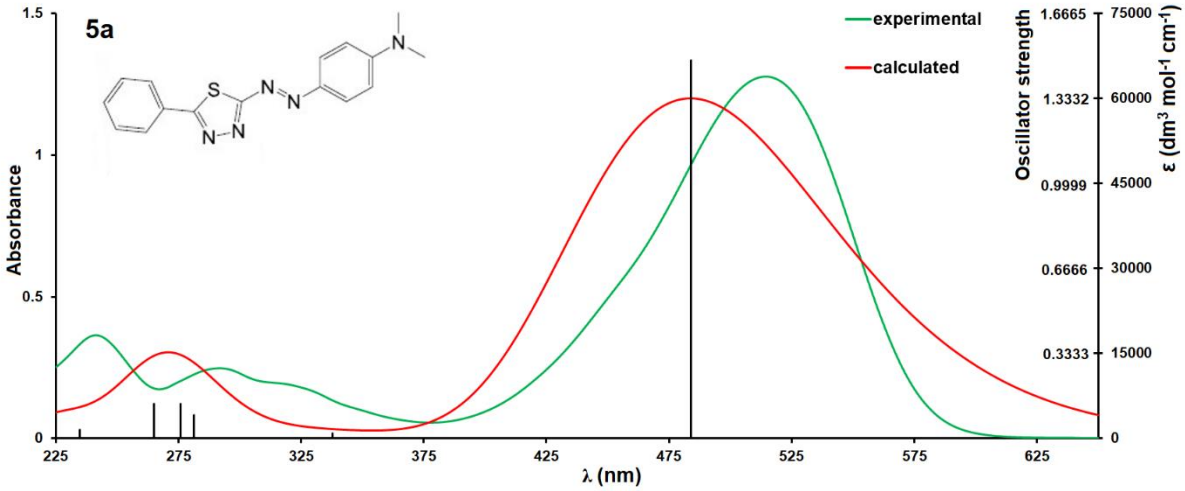


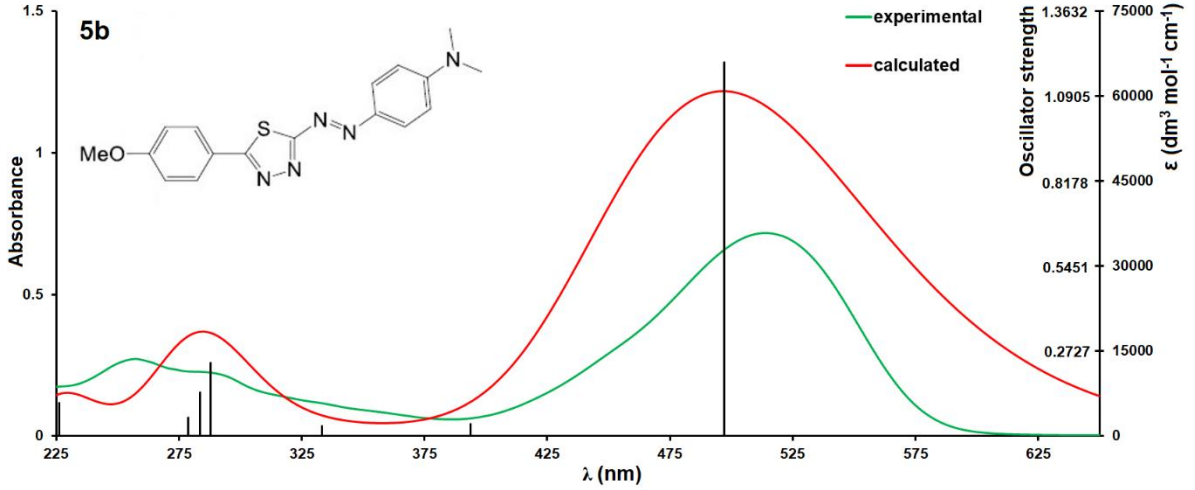


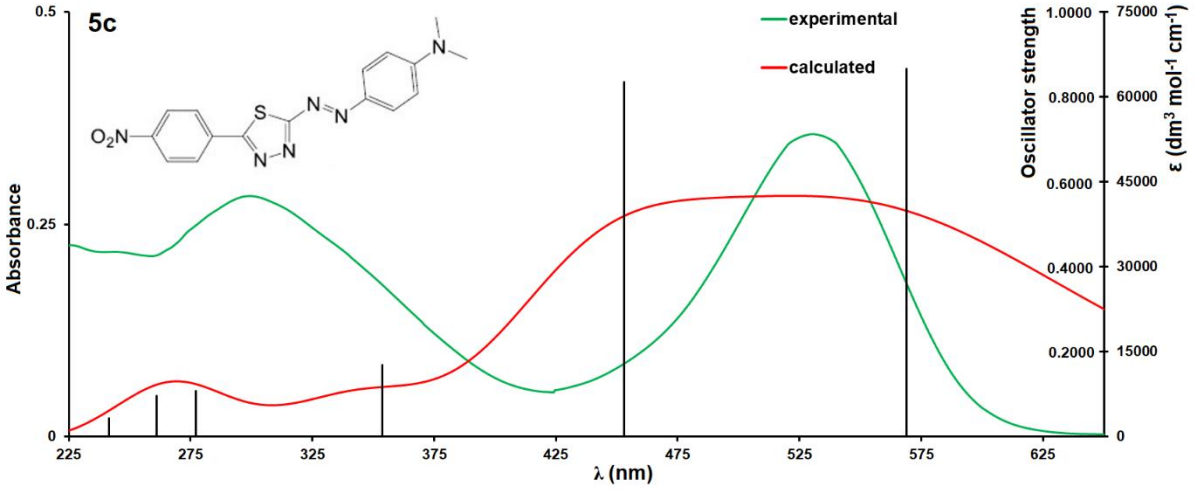


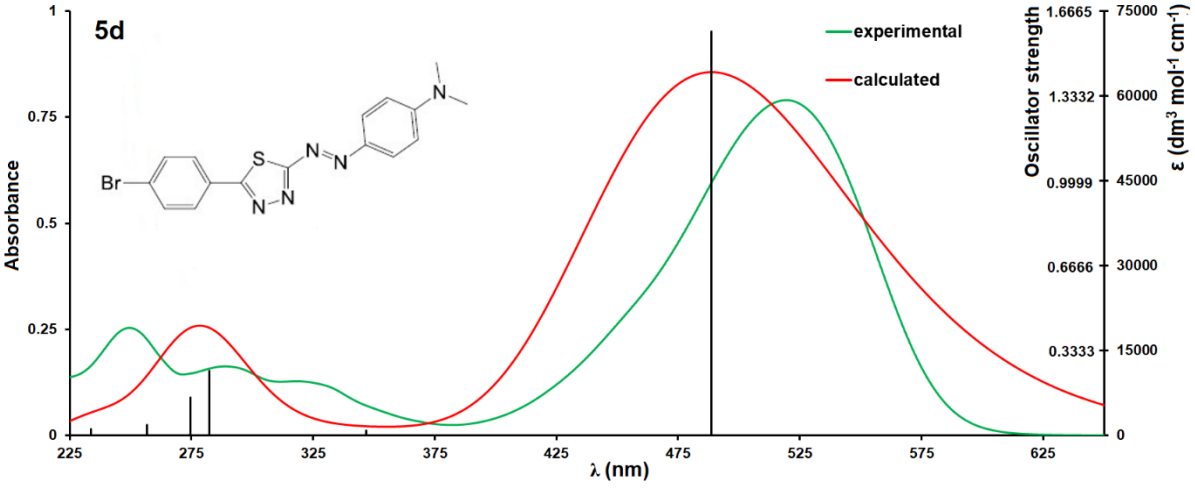

4d
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

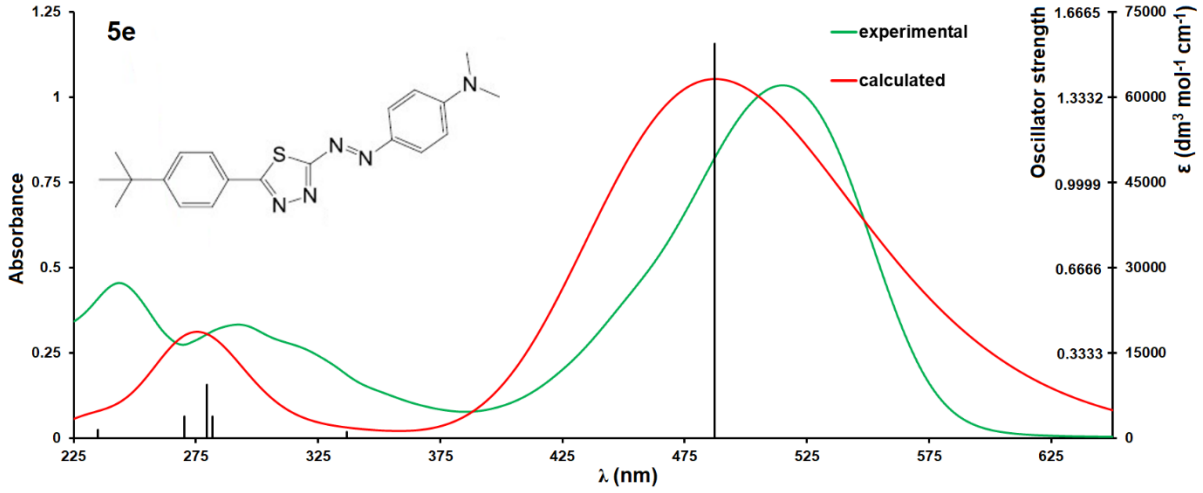

4e
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

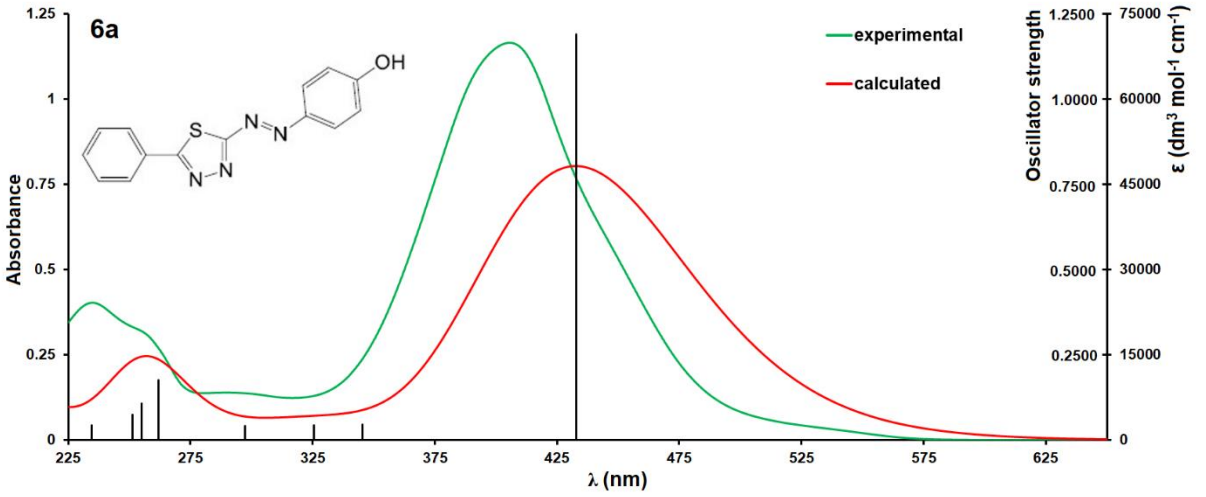

5a
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

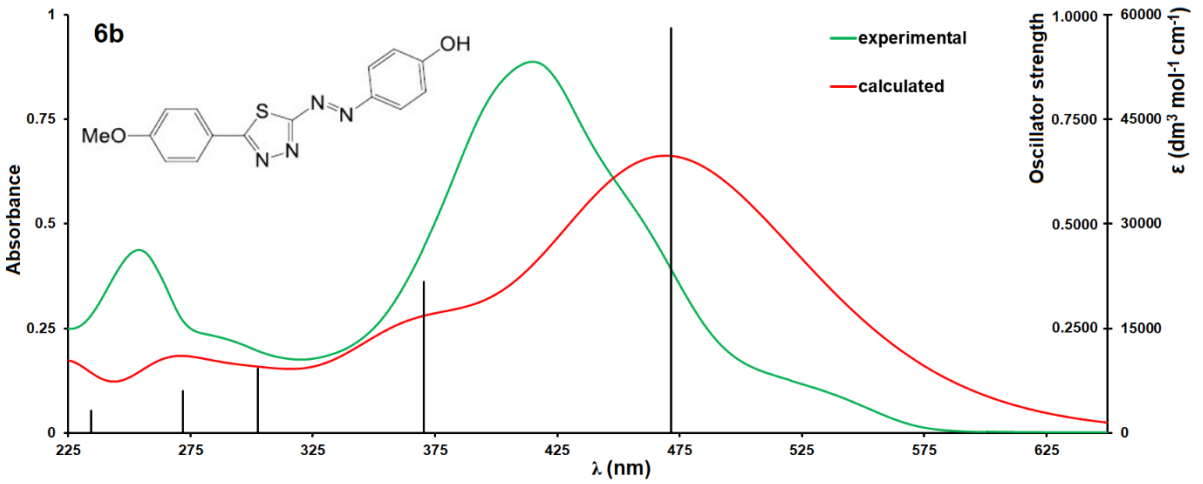

5b
experimental
calculated
MeO
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

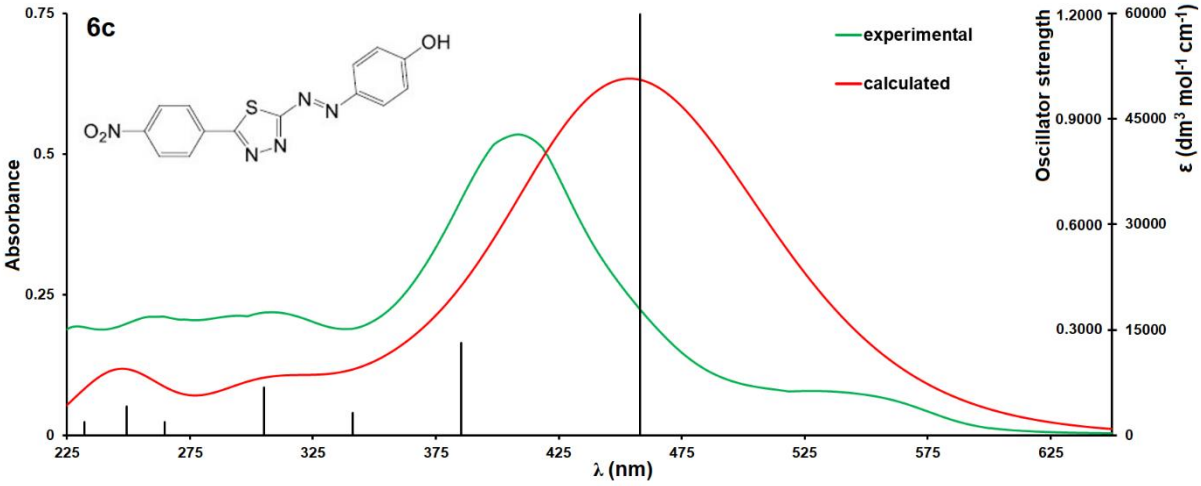

5c
experimental
calculated
O₂N
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

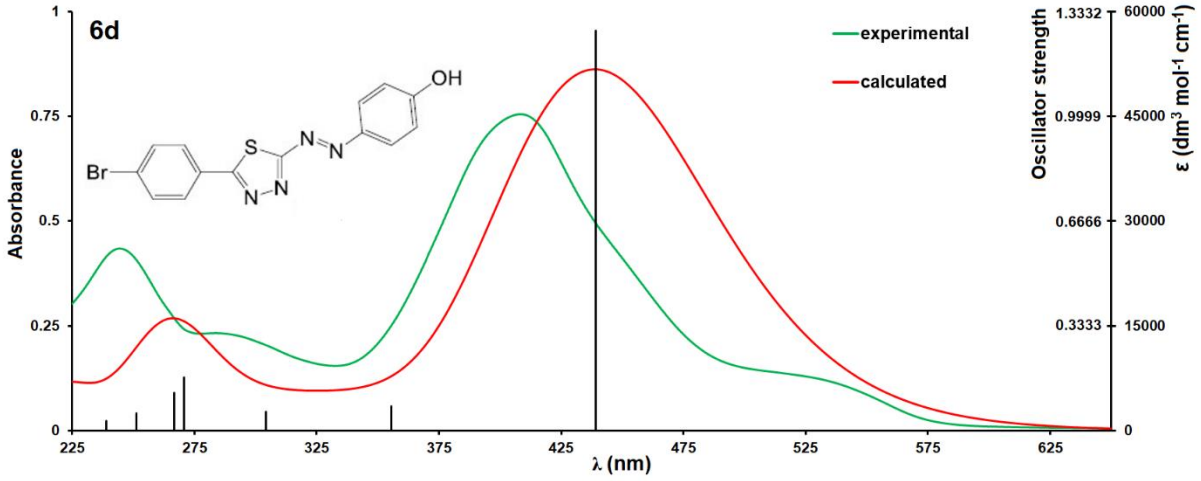

5d
experimental
calculated
Br
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

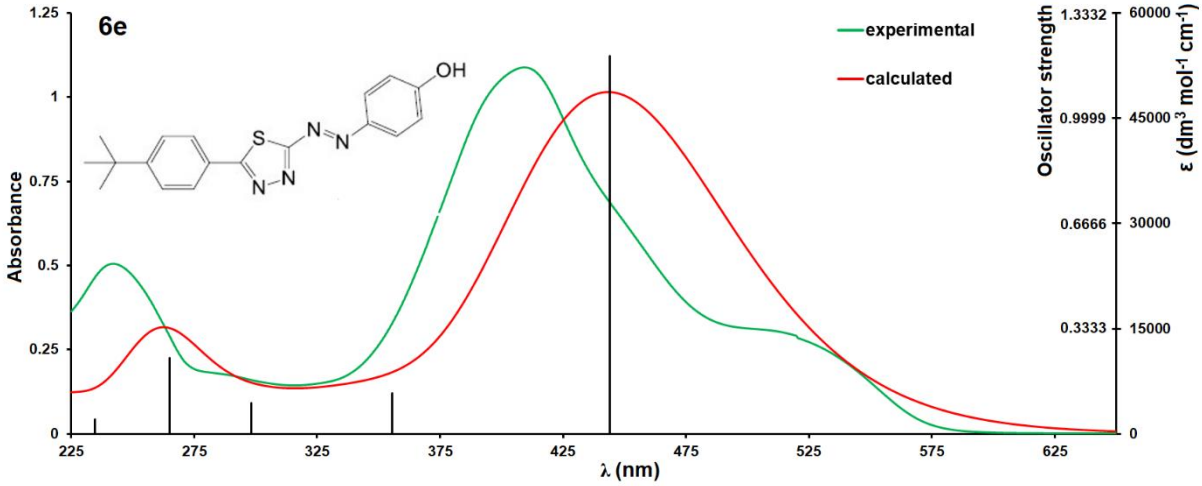

5e
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)


6a
OH
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)


6b
MeO
OH
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)


6c
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)


6d
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)


6e
experimental
calculated
Absorbance
Oscillator strength
ε (dm³ mol⁻¹ cm⁻¹)
λ (nm)

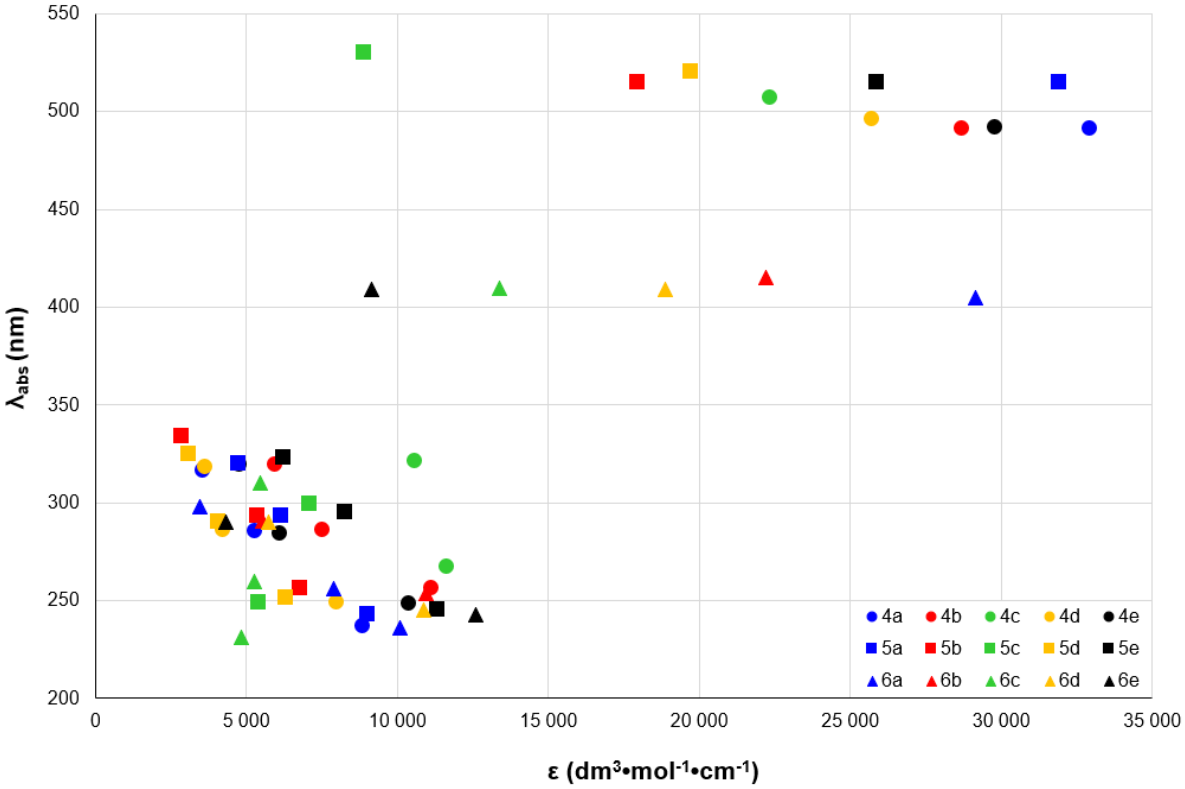


Figure S1. Wavelengths of absorption maxima in relation to their molar absorption coefficients for the studied compounds.

3. Plots of the most important molecular orbitals of compounds 4a-e, 5a-e, 6a-e

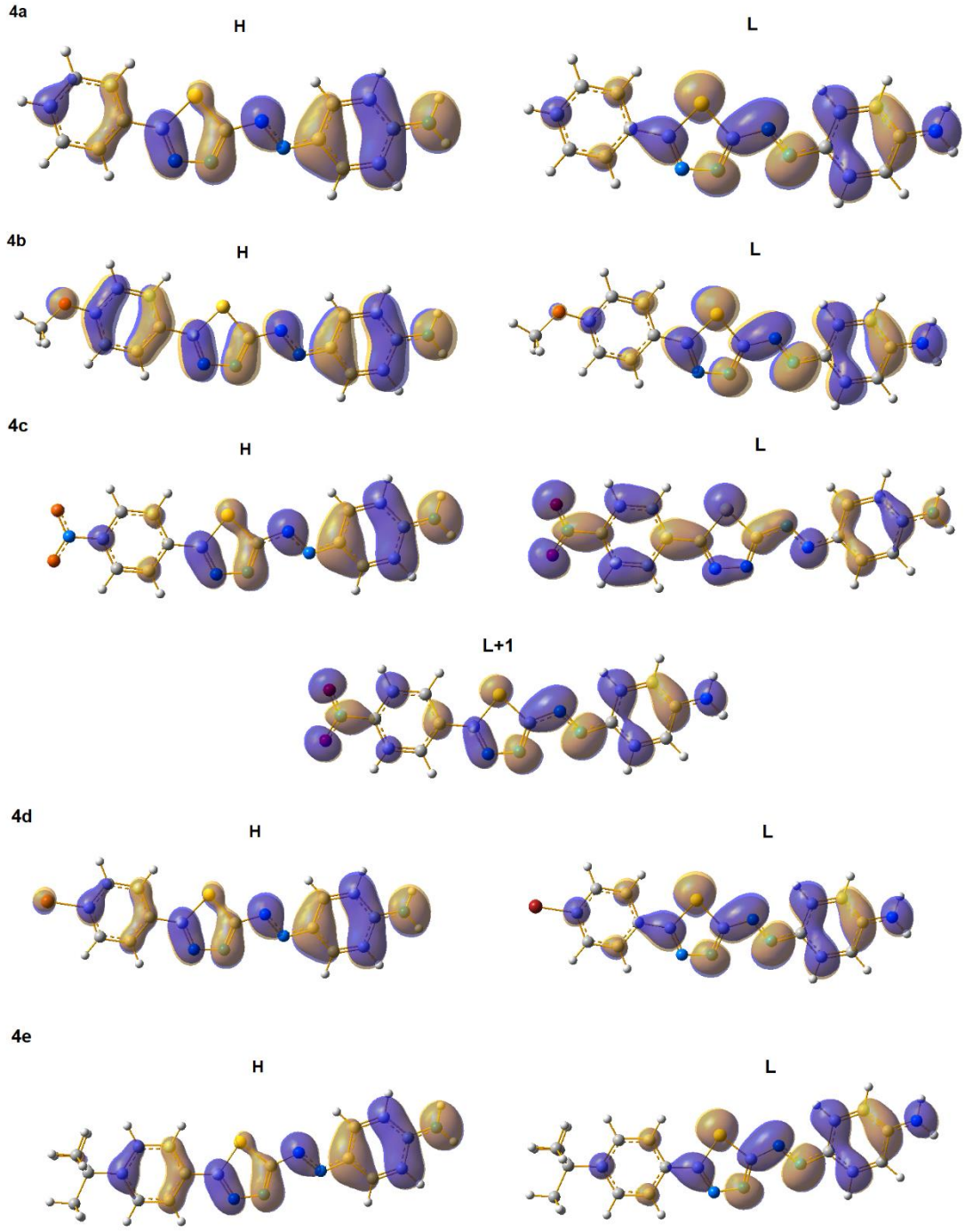


Figure S2. Calculated molecular orbitals for compounds belonging to series 4. The molecular orbitals are plotted with the isovalue equal to 0.02 au.

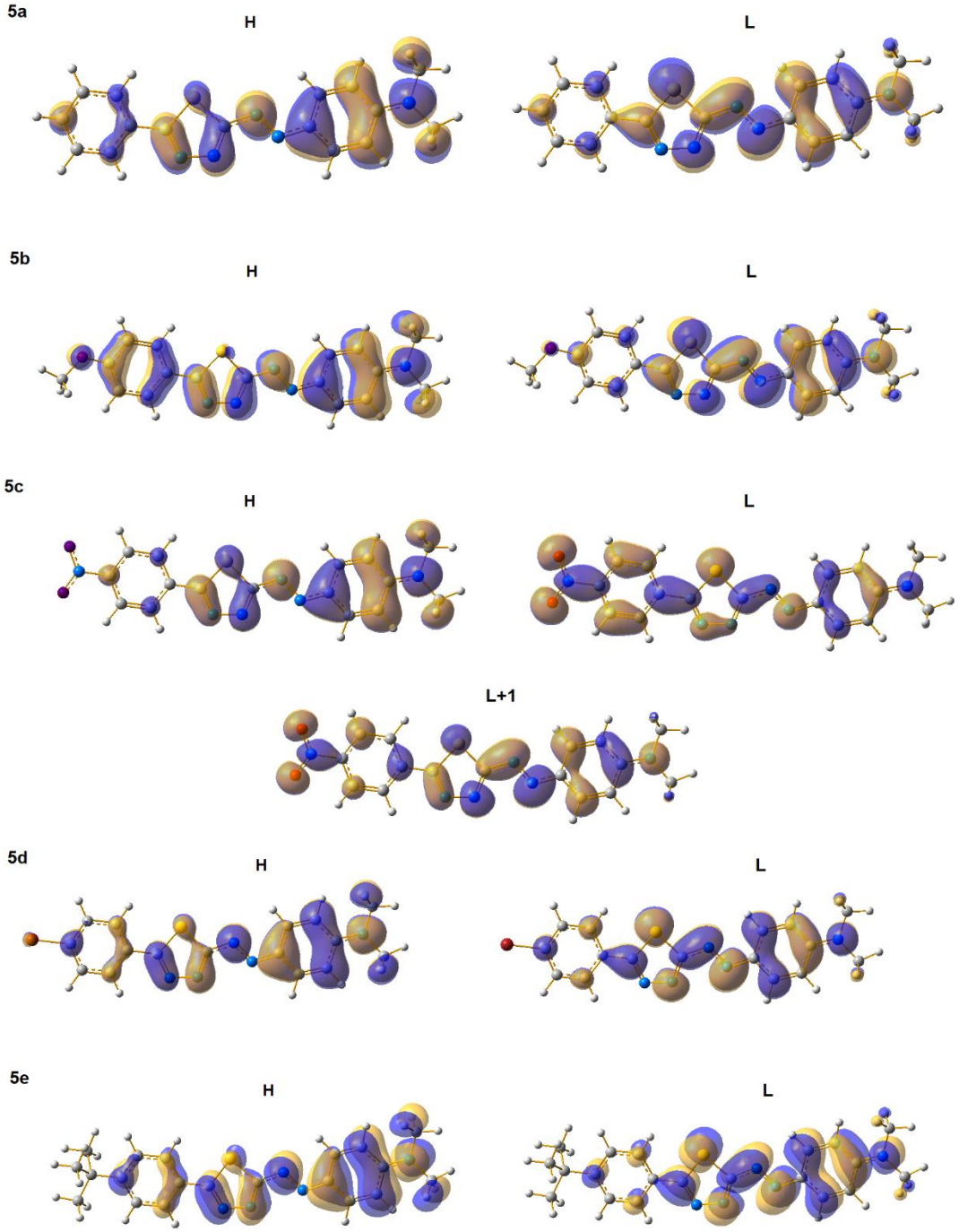


Figure S3. Calculated molecular orbitals for compounds belonging to series 5. The molecular orbitals are plotted with the isovalue equal to 0.02 au.

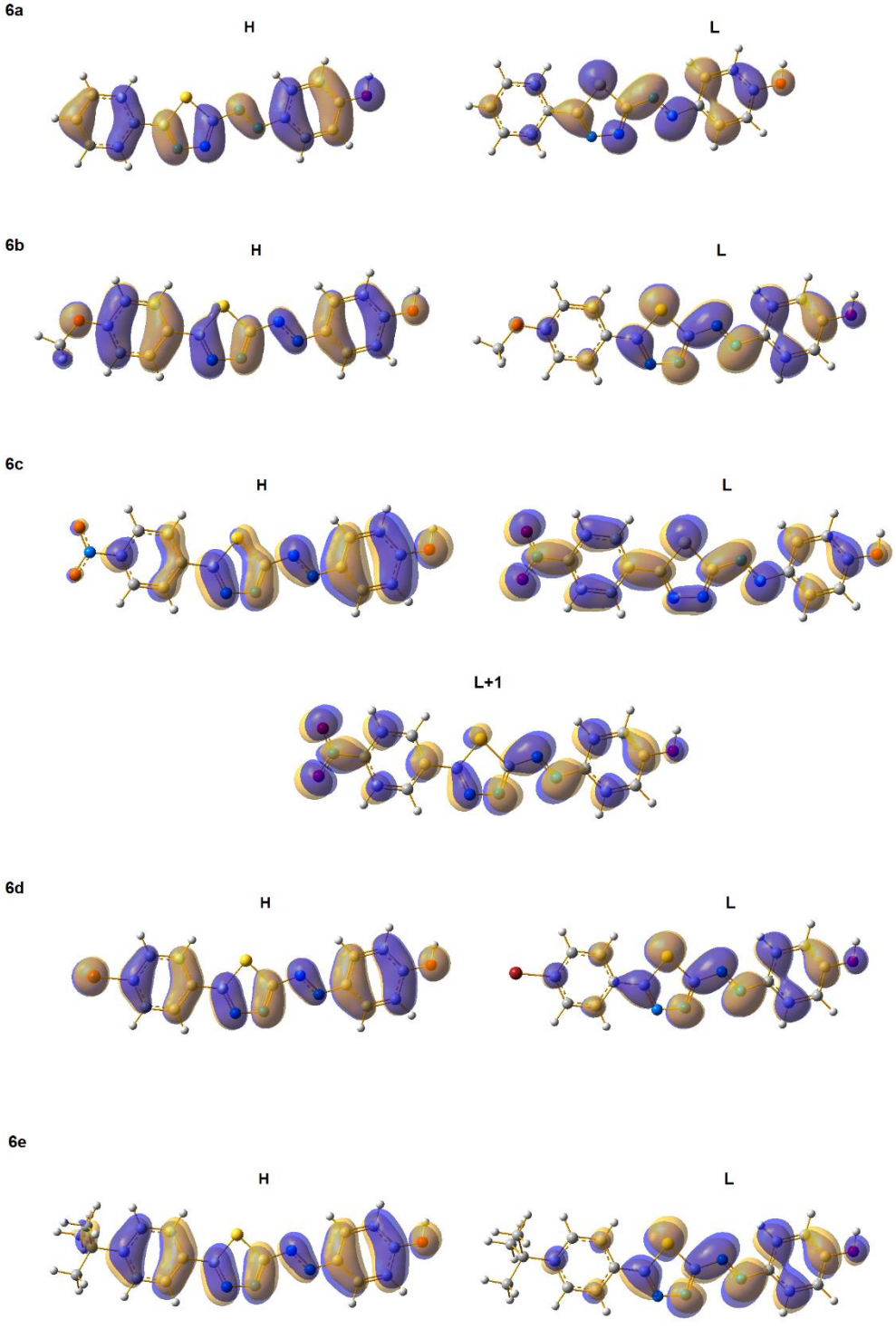
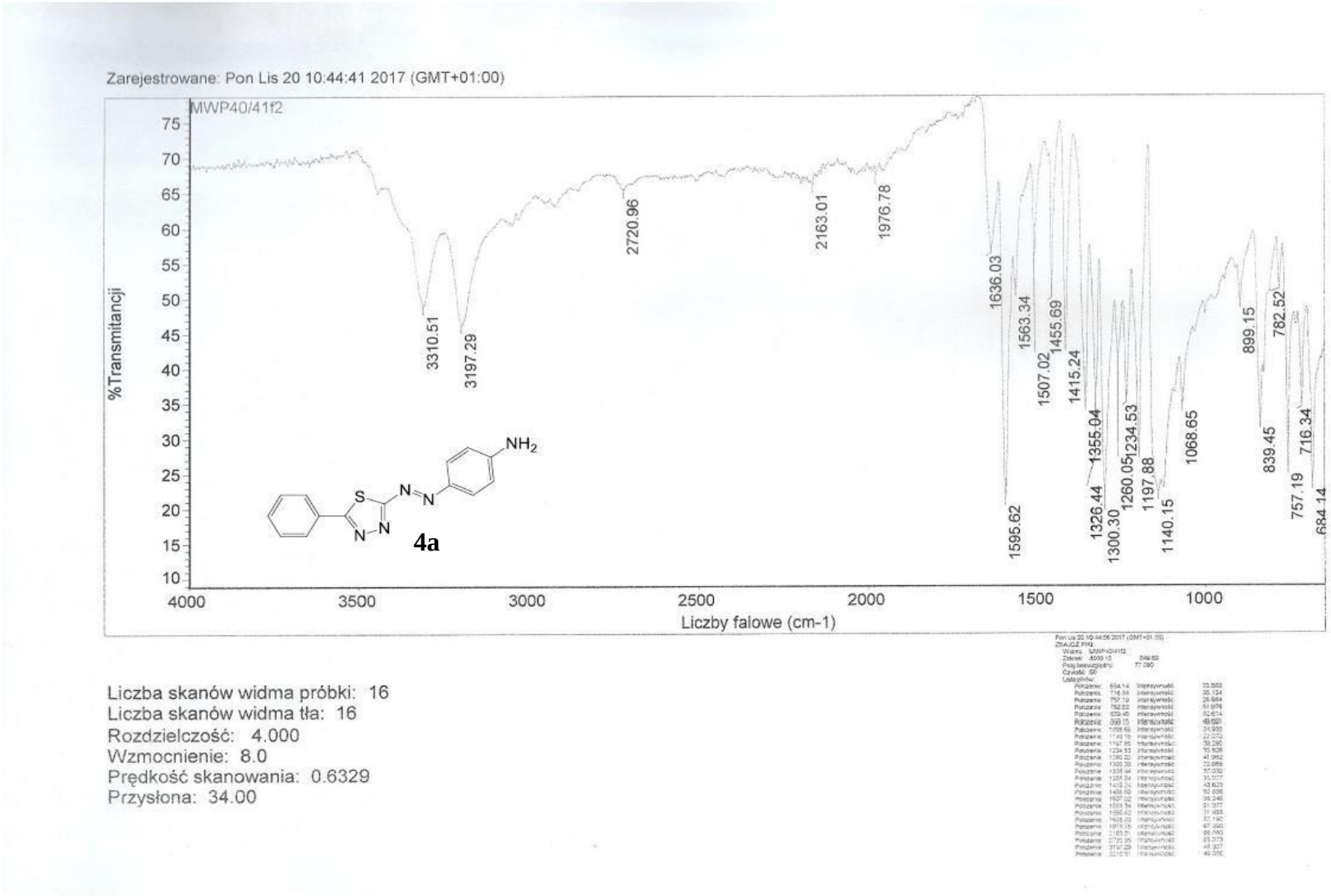
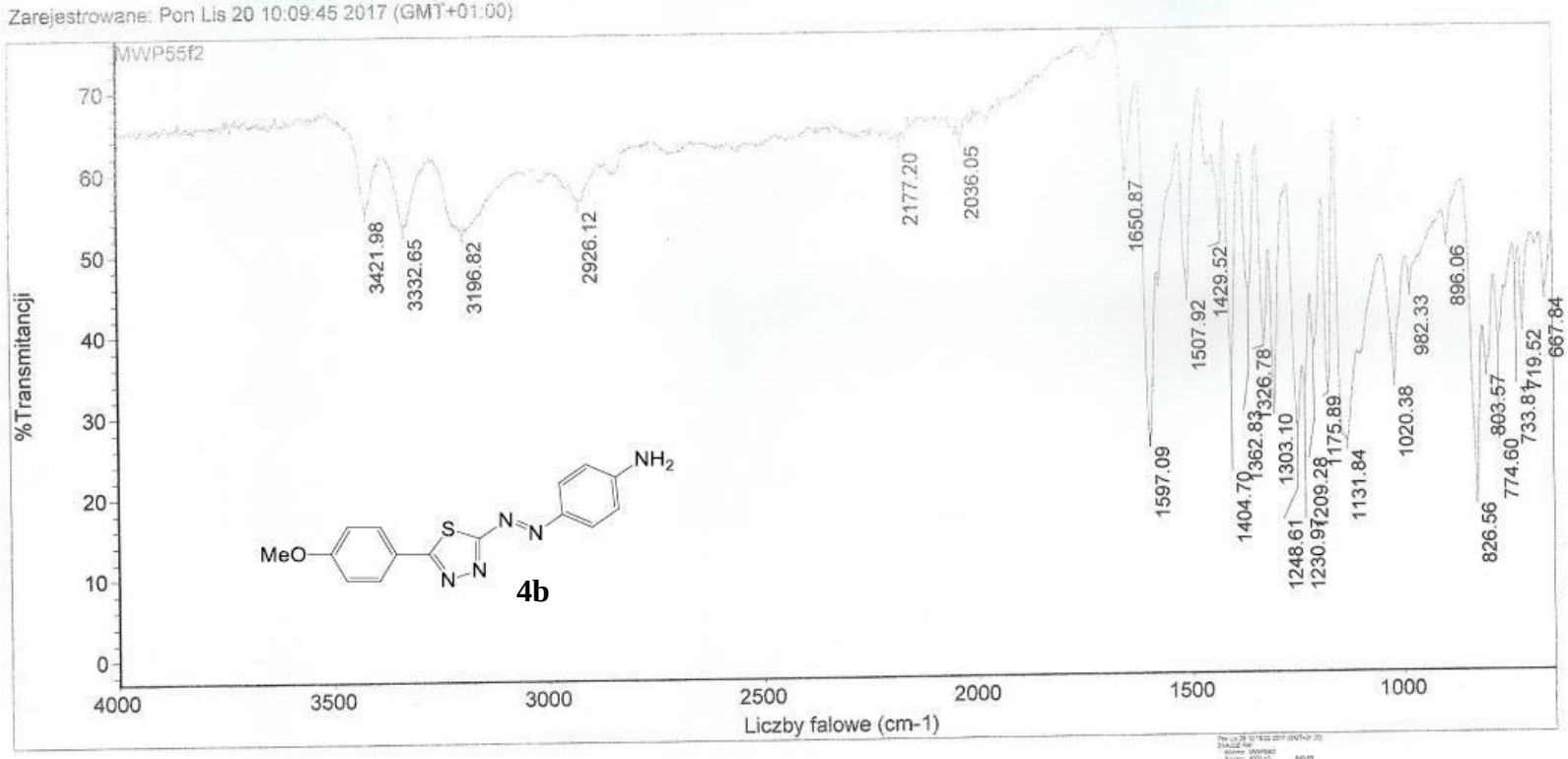


Figure S4. Calculated molecular orbitals for compounds belonging to series 6. The molecular orbitals are plotted with the isovalue equal to 0.02 au.

4. FT-IR spectra of compounds 4a-e, 5a-e, 6a-e



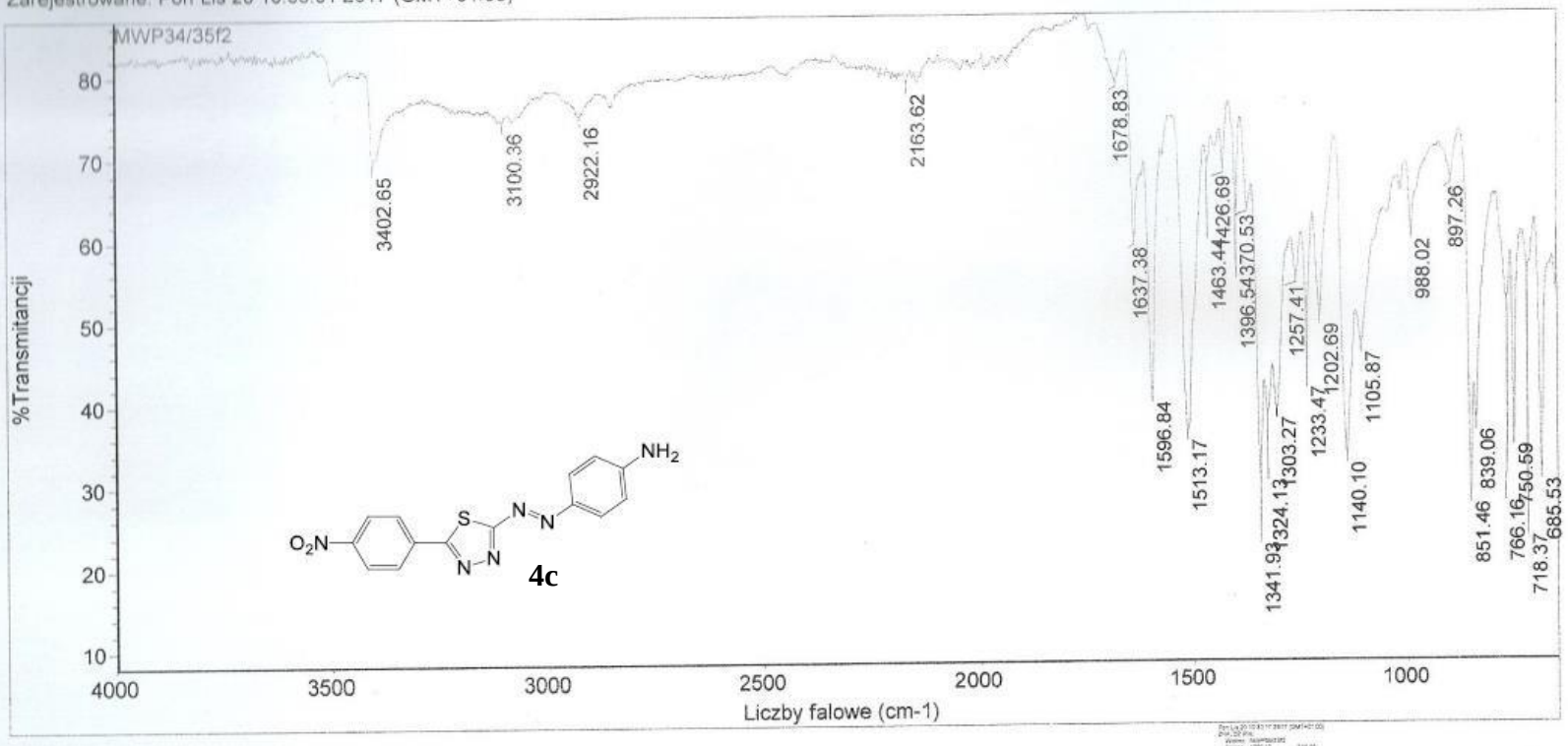
Zarejestrowane: Pon Lis 20 10:09:45 2017 (GMT+01:00)



4b

Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
Rozdzielczość: 4.000
Wzmocnienie: 8.0
Prędkość skanowania: 0.6329
Przysłona: 34.00

Zarejestrowane: Pon Lis 20 10:33:01 2017 (GMT+01:00)



4c

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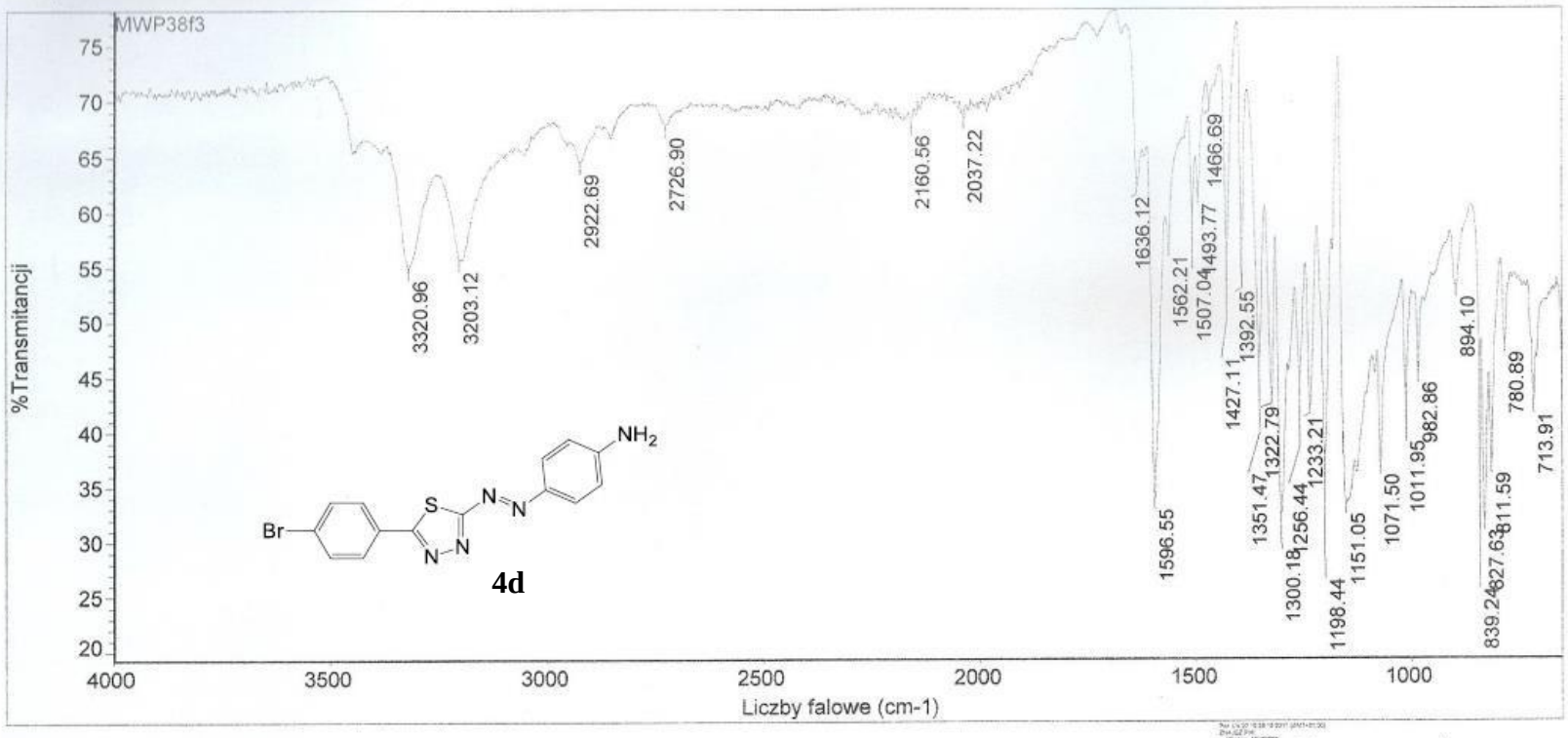

4d

Liczba skanów widma próbki: 16
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Wzmocnienie: 8.0
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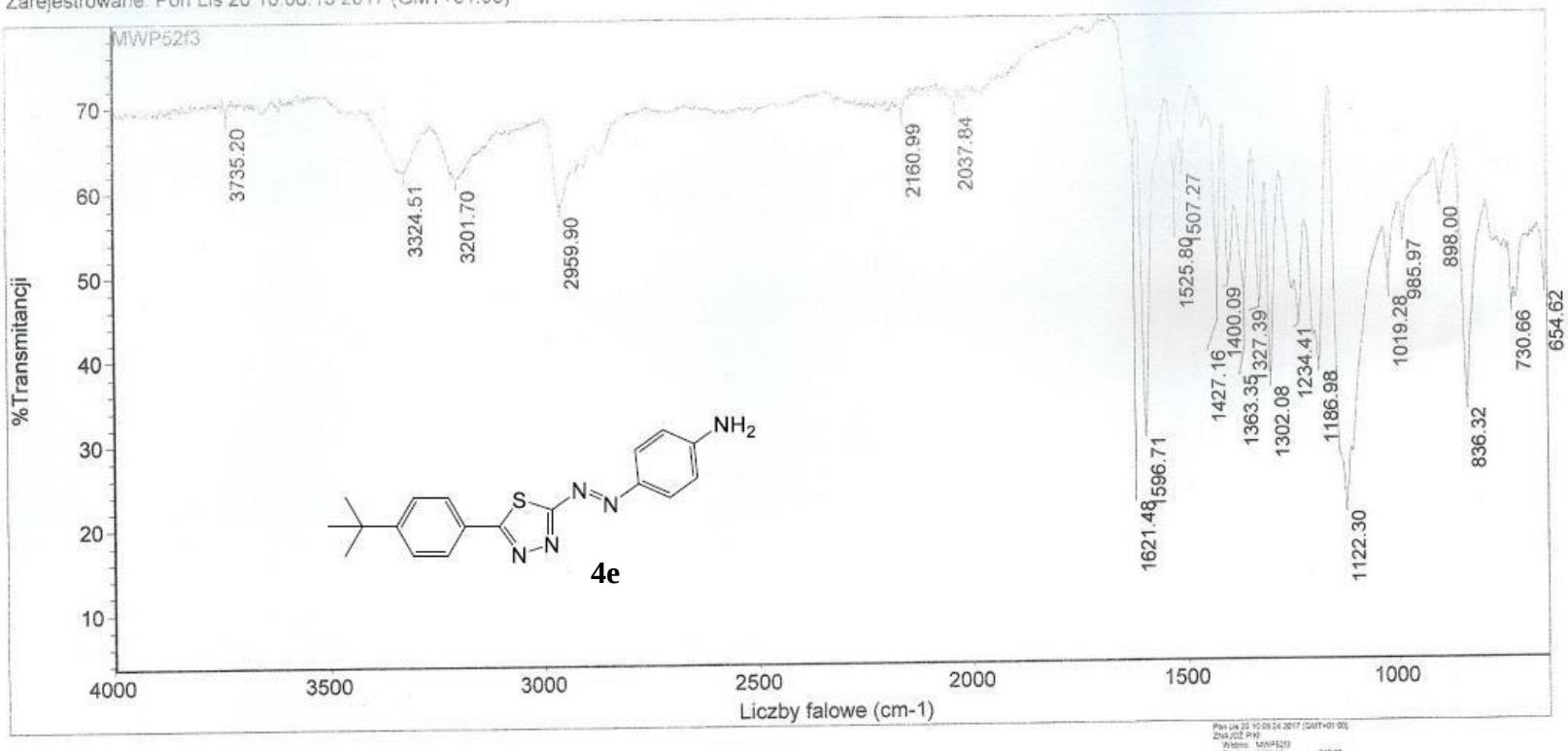

4e

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Wzmocnienie: 8.0
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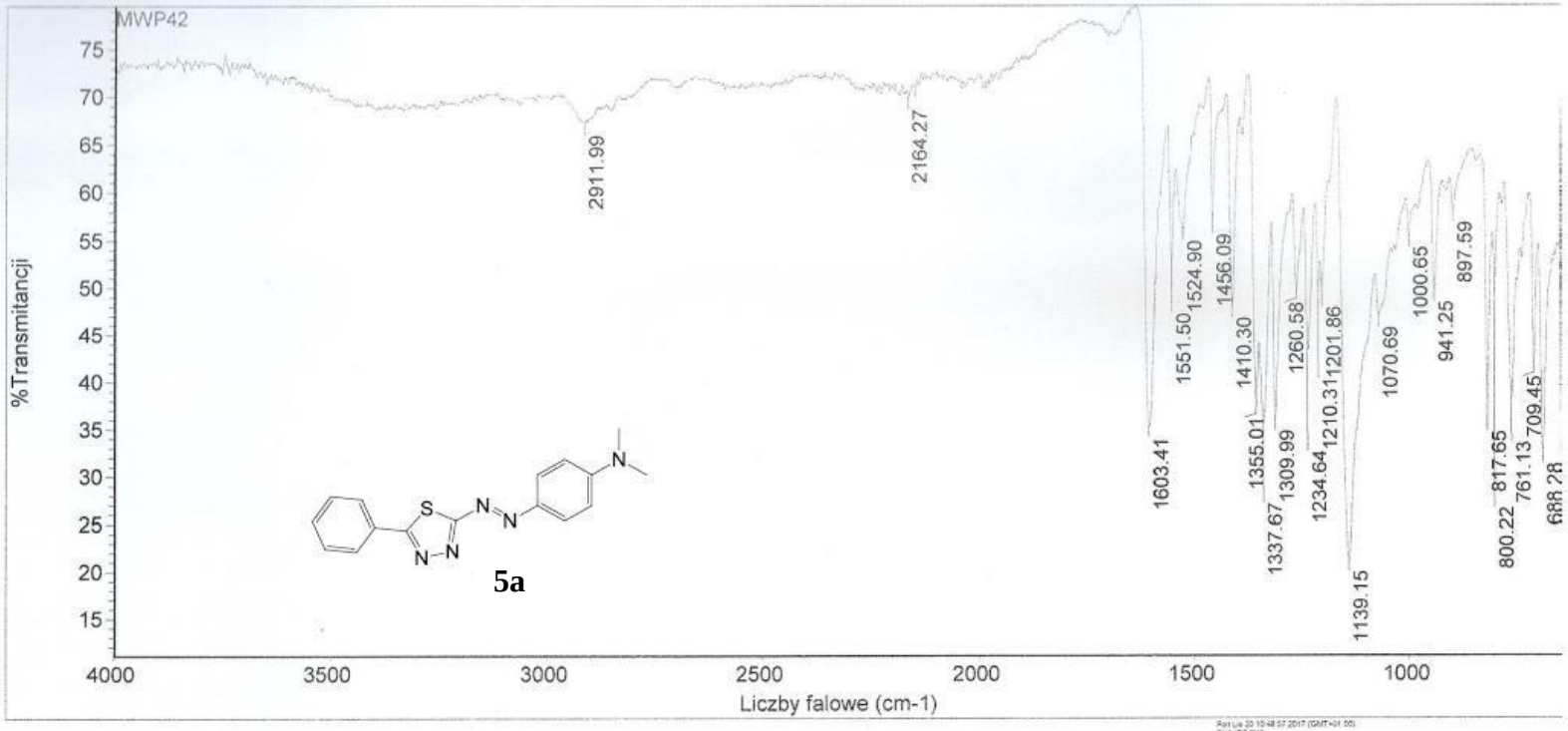

5a

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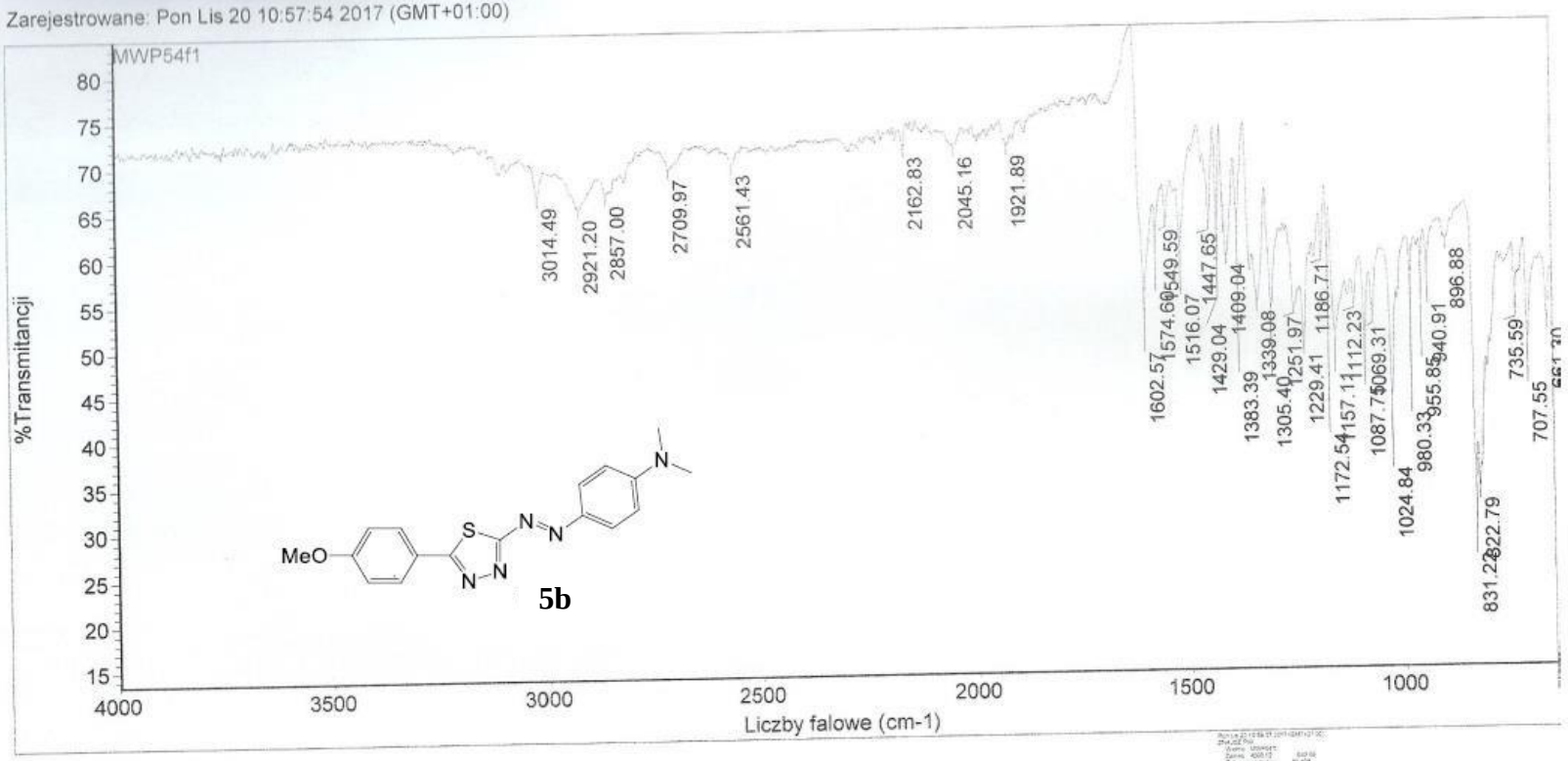

5b

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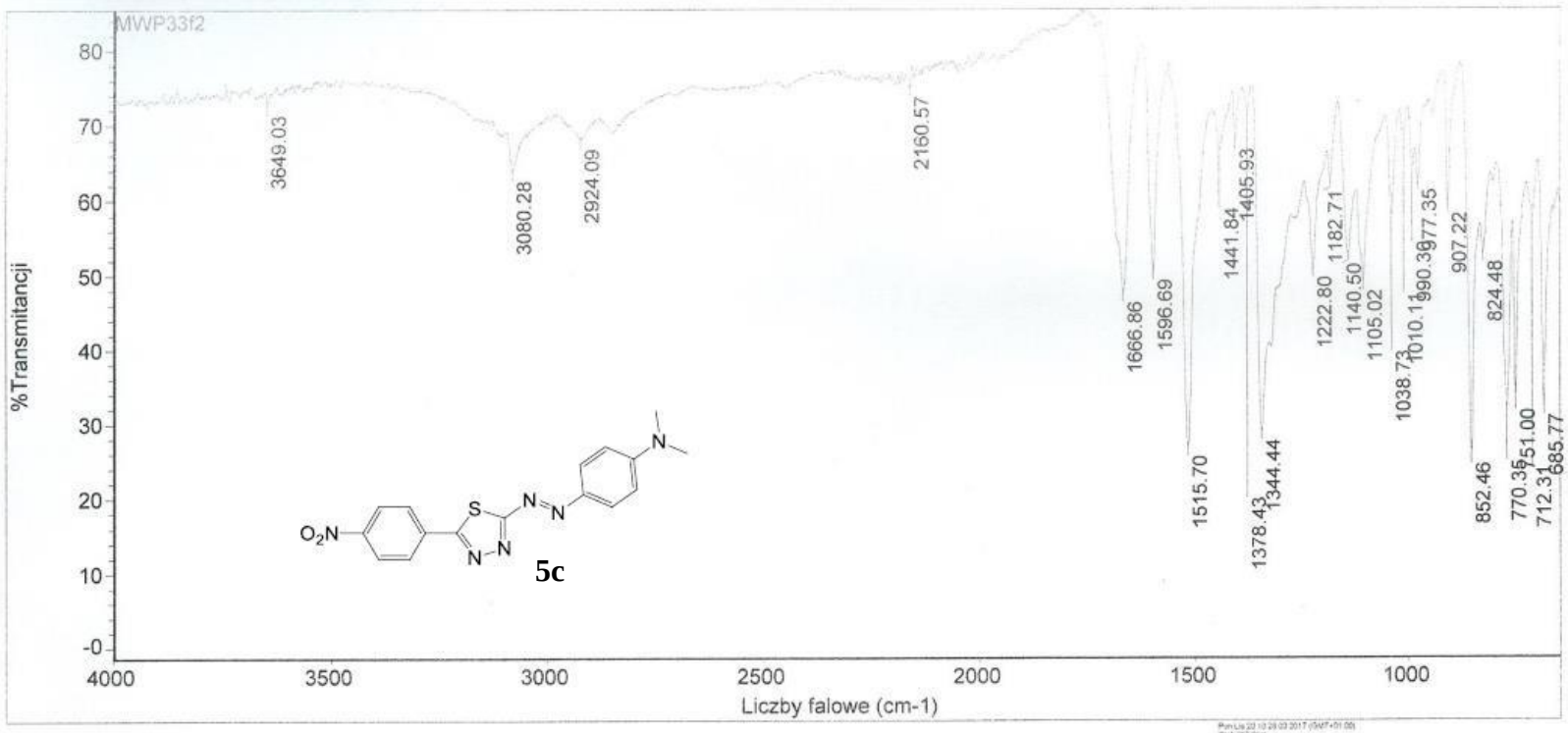

5c

Liczba skanów widma próbki: 16
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Wzmocnienie: 8.0
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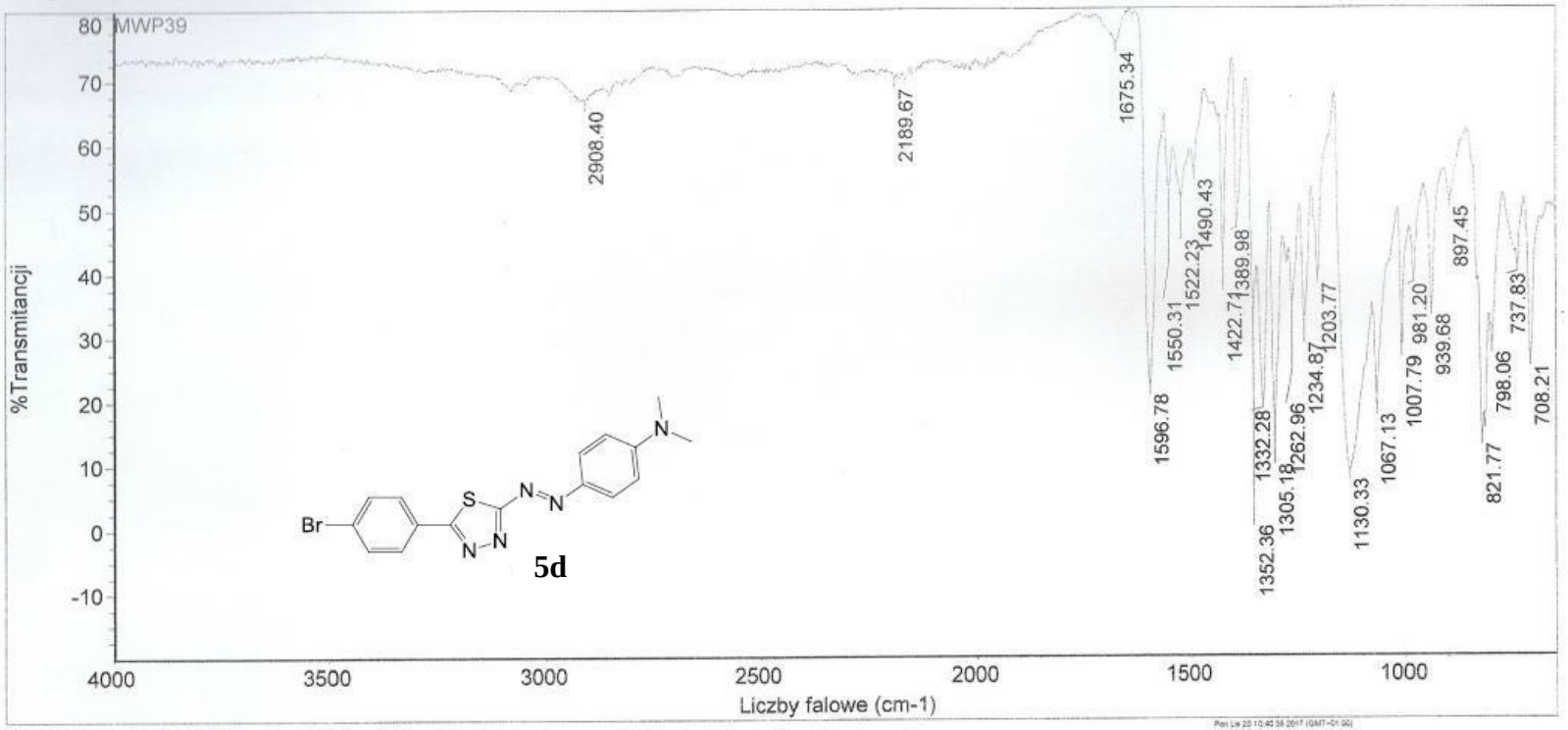
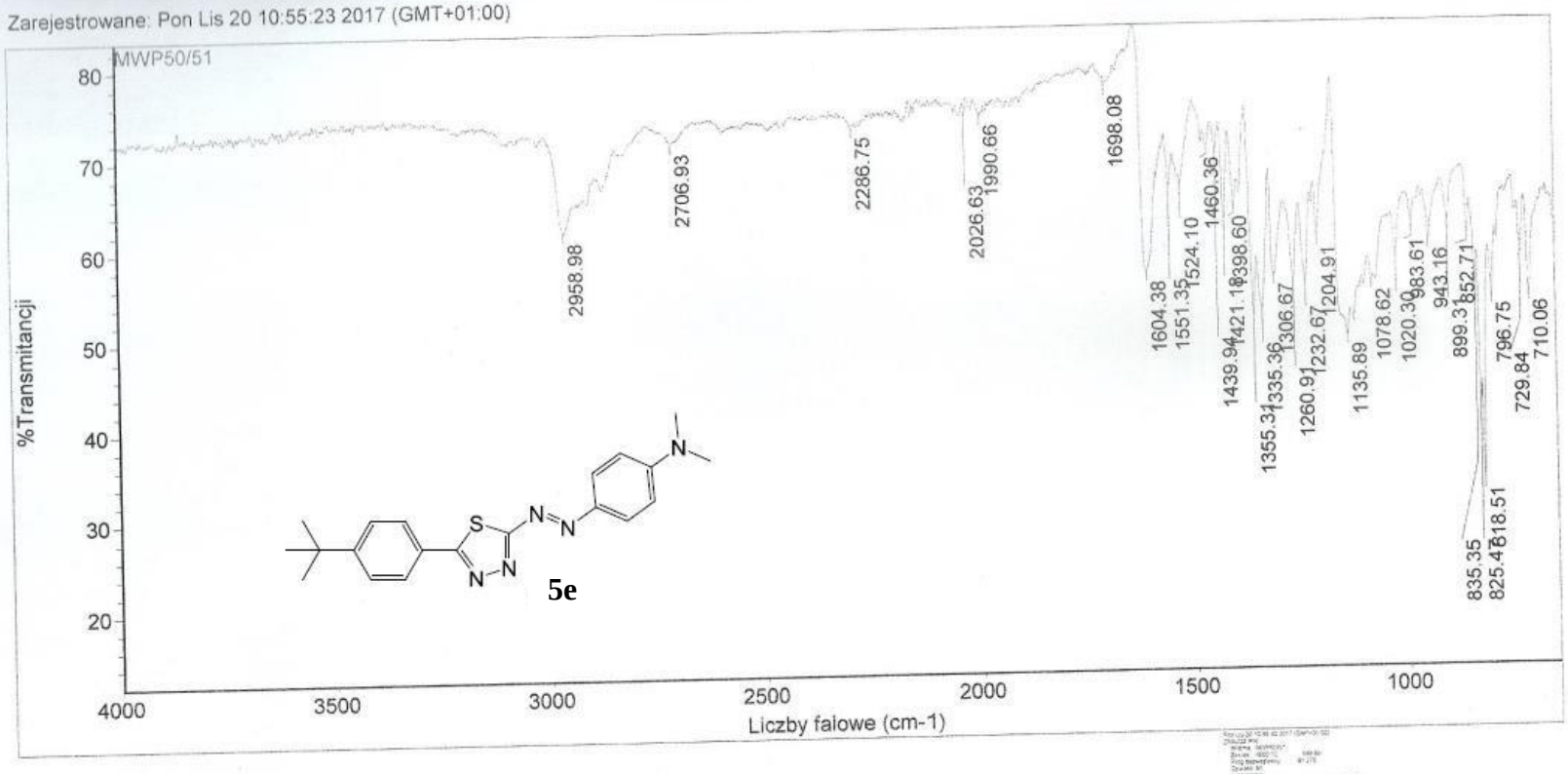
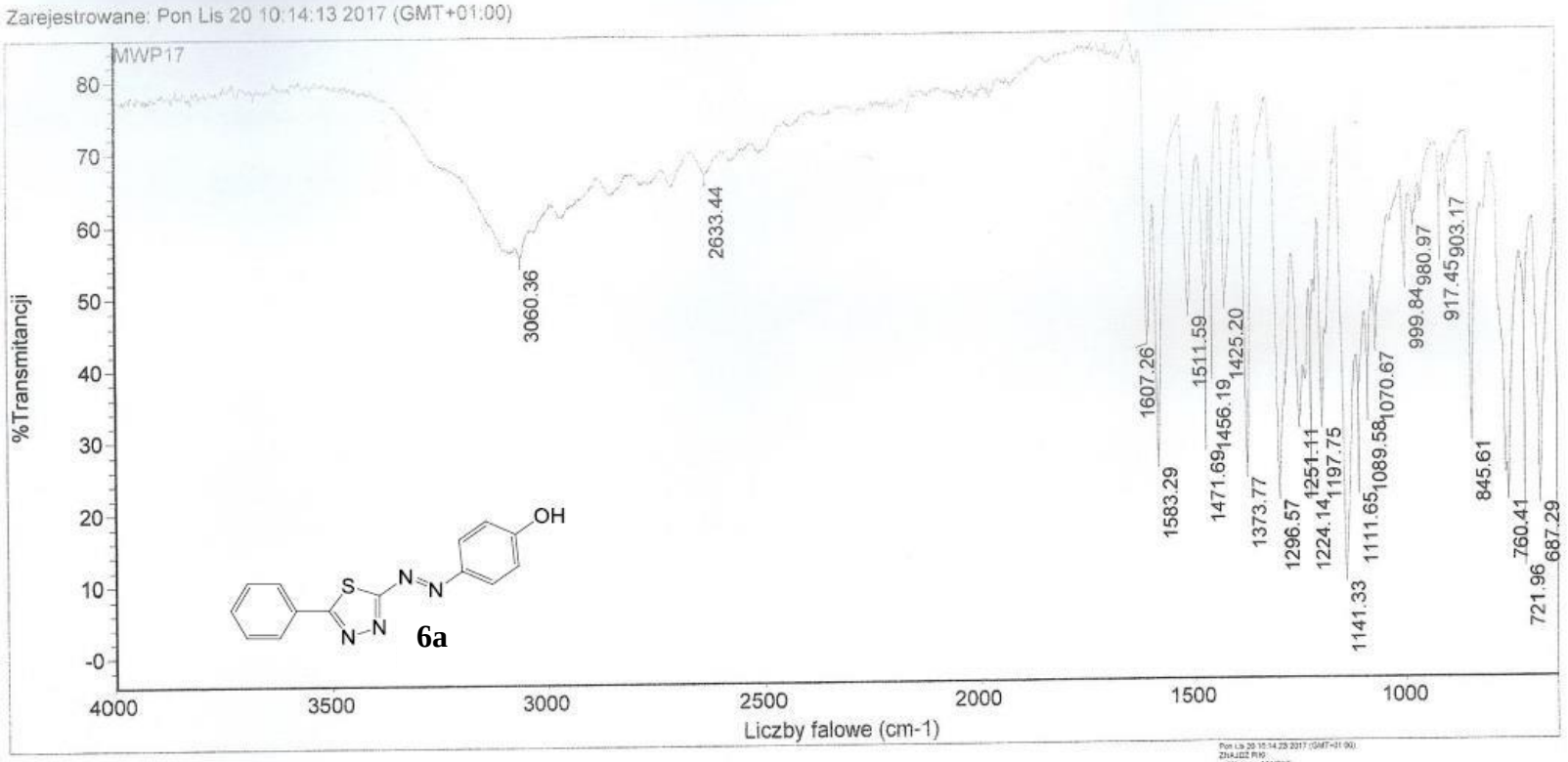


5d

Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
Rozdzielczość: 4.000
Wzmocnienie: 8.0
Prędkość skanowania: 0.6329
Przysłona: 34.00

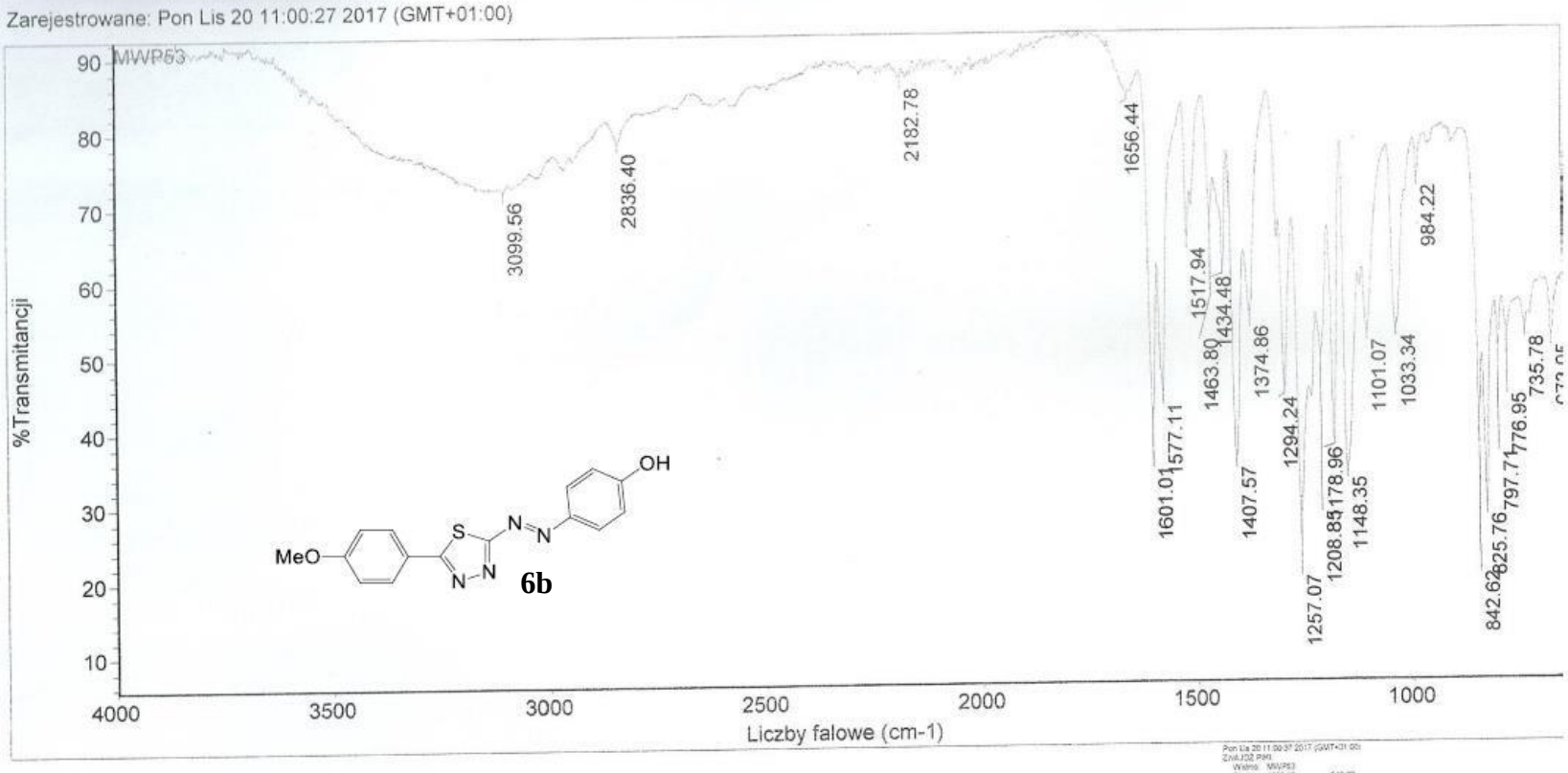


Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
Rozdzielczość: 4.000
Wzmocnienie: 8.0
Prędkość skanowania: 0.6329
Przysłona: 34.00



6a

Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
Rozdzielczość: 4.000
Wzmocnienie: 8.0
Prędkość skanowania: 0.6329
Przysłona: 34.00



Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
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Wzmocnienie: 8.0
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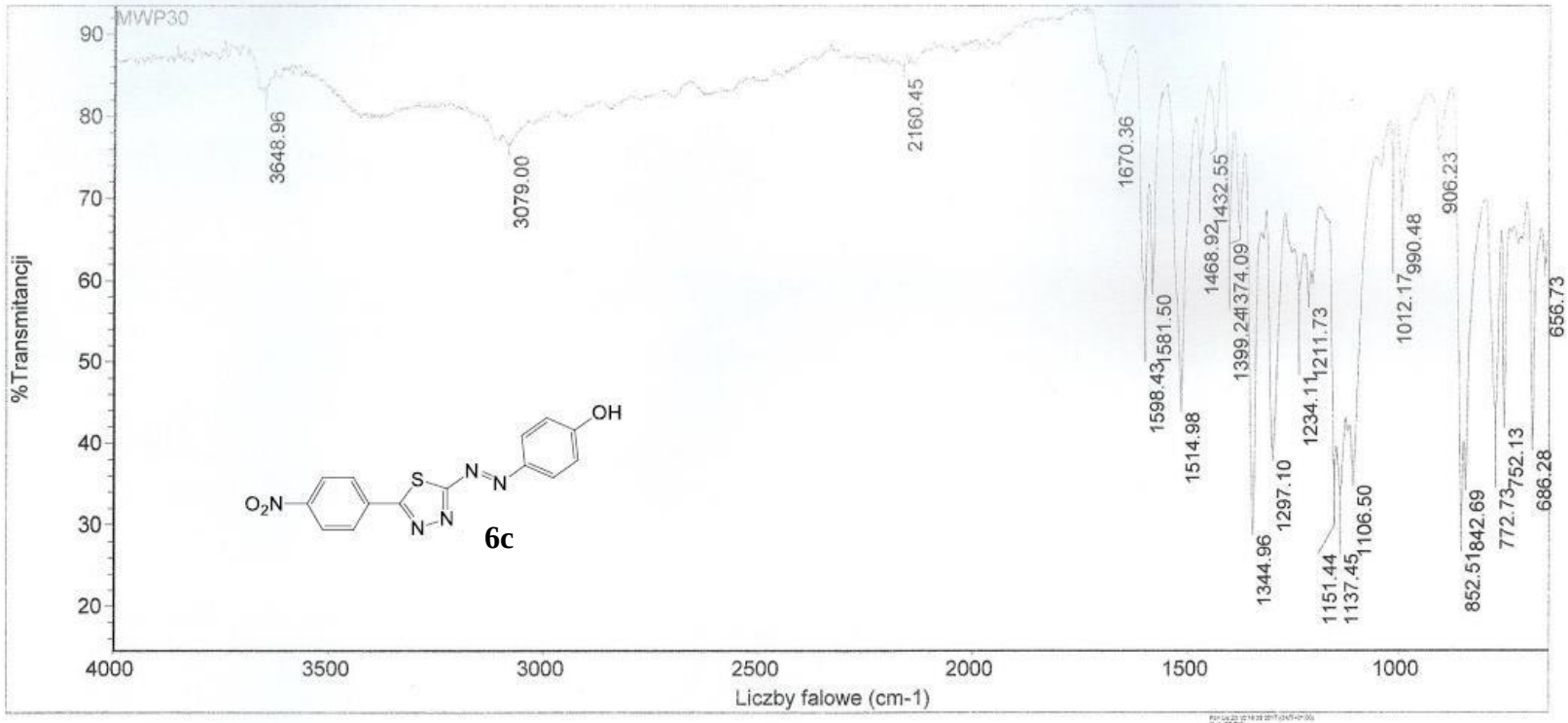

6c

Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
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Wzmocnienie: 8.0
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Przysłona: 34.00

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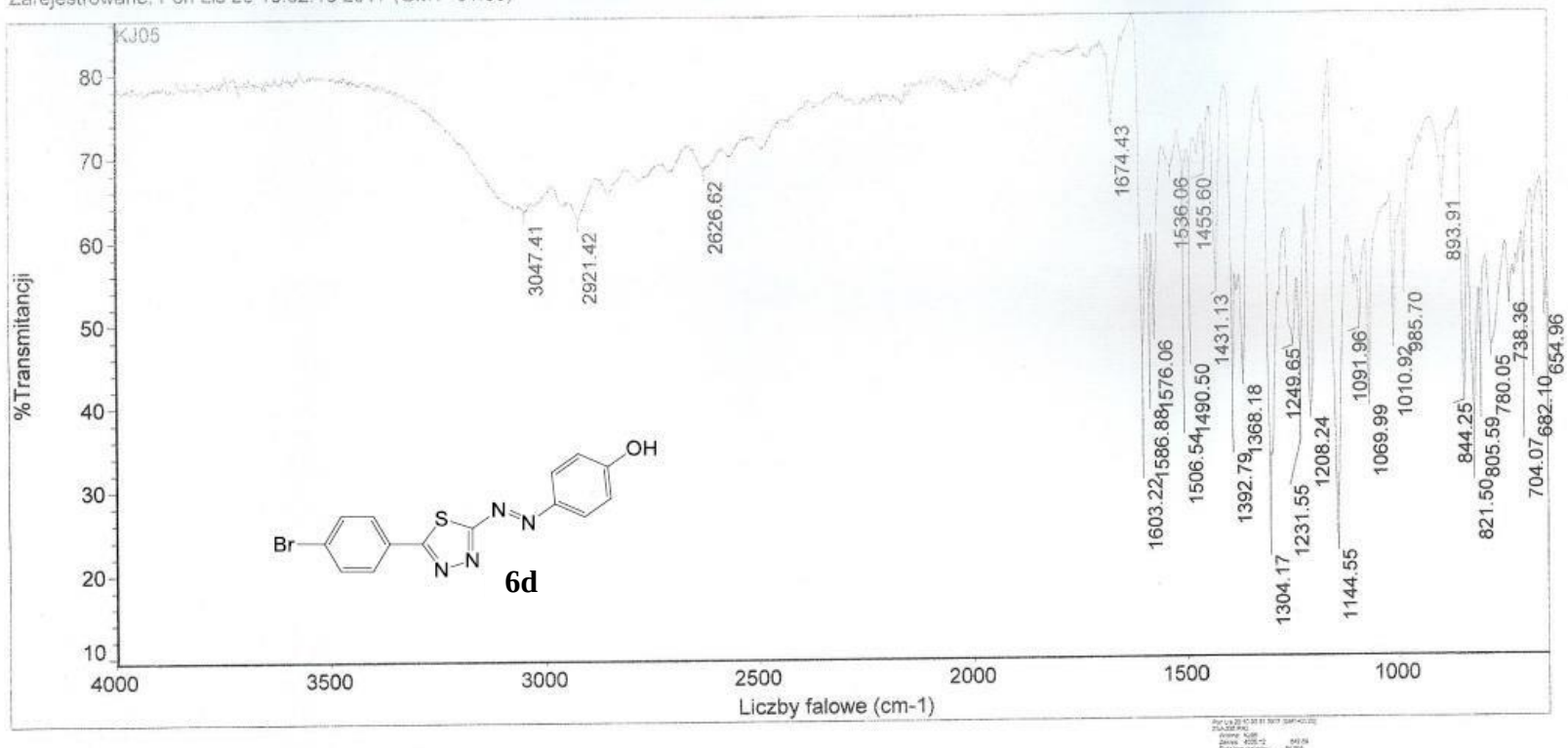
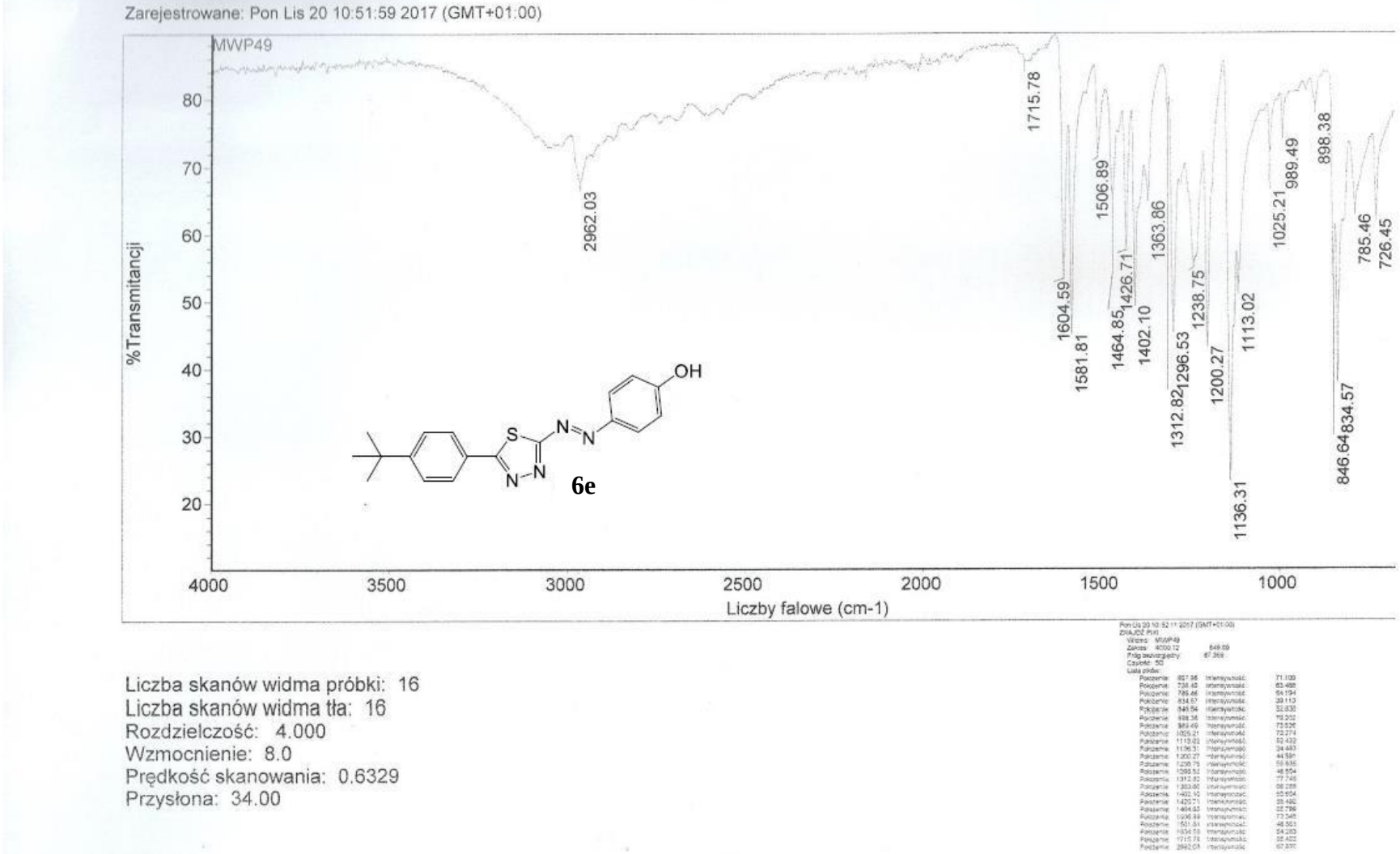


6d

Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
Rozdzielczość: 4.000
Wzmocnienie: 8.0
Prędkość skanowania: 0.6329
Przysłona: 34.00



Liczba skanów widma próbki: 16
Liczba skanów widma tła: 16
Rozdzielczość: 4.000
Wzmocnienie: 8.0
Prędkość skanowania: 0.6329
Przysłona: 34.00

5. Crystal structure determination

A red plate crystal of 2-[4-(*N*,*N*-dimethylamino)phenylazo]-5-(4-methoxyphenyl)-1,3,4-thiadiazole (**5b**) was mounted on a Rigaku Synergy Dualflex automatic diffractometer equipped with Pilatus 300K detector, and used for data collection. X-Ray intensity data was collected with mirror monochromatic CuK_α (λ = 1.54178 Å, micro-focus sealed PhotonJet X-ray tube) radiation at a temperature of 100.0(1) K, with ω scan mode. The shutterless mode was used, and reflections inside the Ewald sphere were collected up to θ = 78.9°. The unit cell parameters were determined from the 13352 strongest reflections. Details concerning crystal data and refinement are given in **Table 1**. Examination of the same reference reflections, obtained before and after measurement, showed no loss in intensity during measurement. Lorentz polarization, and Gaussian absorption (using Gaussian integration over a multifaceted crystal model) corrections were applied during data reduction. The structure was solved by a partial structure expansion procedure. All non-hydrogen atoms were refined anisotropically using a full-matrix, least-squares technique on F2. All hydrogen atoms were identified from different Fourier synthesis after four cycles of anisotropic refinement, and refined as "riding" on the adjacent atom with geometric idealisation after each refinement cycle. Individual isotropic displacement factors equalled 1.2 times the value of equivalent displacement factors of the aromatic carbon atoms, and 1.5 times of parent methyl carbon atoms. SHELXT [1], SHELXL [2], and SHELXTL [3] programs were used for all calculations. Atomic scattering factors were taken from International Tables for Crystallography [4]. Selected interatomic bond distances, dihedral angles and intermolecular interactions are presented in the publication. Tables of crystal data for **5b**, structure refinement, anisotropic displacement coefficients, atomic coordinates, and equivalent isotropic displacement parameters for non-hydrogen atoms, H-atom coordinates, and isotropic displacement parameters, bond lengths, and interbond angles were deposited with the Cambridge Crystallographic Data Centre under No. CCDC1946289.

Table 1. Crystal data and structure refinement details for compound **5b**.

Compound	**5b**
Empirical formula	$C_{17}H_{17}N_5OS$
Formula weight	339.41
Crystal system	Monoclinic
Space group	*P2₁/c* (No. 14)
Temperature [K]	100.0(1)
Wavelength [Å]	λ(Cu*Kα*) 1.54184
Unit cell dimensions [Å, °]	a = 19.0674(2)
	b = 9.7671(1)
	c = 8.7924(1)
	α = 90.00
	β = 102.721(1)
	γ = 90.00
Volume [Å^3]	1597.24(3)
Z	4
Calculated density [Mg/m^3]	1.411
Absorption coefficient [mm^{-1}]	1.923
F(000)	712
Crystal size [mm]	0.013 x 0.060 x 0.129
θ Range for data collection [°]	4.755 to 78.905
Index ranges	$-24 \le h \le 24$,
	$-12 \le k \le 12$,
	$-10 \le l \le 10$
Reflections collected / unique (R_{int})	25674 / 3335 (0.0388)
Completeness to θ = 67° [%]	100.0
Min. and max. transmission	0.765 and 1.000

Data / restraints / parameters	3335 / 0 / 220
Goodness-of-fit on F^2	1.067
Final *R* indices [$I>2\sigma(I)$]	*R1* = 0.0319,
	wR2 = 0.0820
R indices (all data)	*R1* = 0.0360,
	wR2 = 0.0842
Largest diff. peak and hole [e **x** $Å^{-3}$]	0.283 and -0.310

References

1. Sheldrick, G.M. SHELXT - Integrated space-group and crystal-structure determination. *Acta Cryst.* **2015**, A71, 3-8.
2. Sheldrick, G.M. Crystal structure refinement with SHELXL. *Acta Cryst.* **2015**, C71, 3-8.
3. Sheldrick, G.M. A short history of SHELX. *Acta Cryst.* **2008**, A64, 112-122.
4. Cowley, J.M. Scattering factors for the diffraction of electrons by crystalline solids. *In International Tables for Crystallography, Volume C: Mathematical, physical and chemical tables*, 3rd ed.; Prince, E., Ed.; Kluwer Academic Publishers: Dordrecht, Netherlands, 2004; pp. 259-262.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) mwp54_f1

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: mwp54_f1

```
Bond precision:  C-C = 0.0019 A          Wavelength=1.54184

Cell:            a=19.0674(2)    b=9.7671(1)         c=8.7924(1)
                 alpha=90        beta=102.721(1)     gamma=90
Temperature:     100 K

                Calculated                Reported
Volume          1597.24(3)                1597.24(3)
Space group     P 21/c                    P 21/c
Hall group      -P 2ybc                   -P 2ybc
Moiety formula  C17 H17 N5 O S            C17 H17 N5 O S
Sum formula     C17 H17 N5 O S            C17 H17 N5 O S
Mr              339.42                    339.41
Dx,g cm-3       1.411                     1.411
Z               4                         4
Mu (mm-1)       1.923                     1.923
F000            712.0                     712.0
F000'           715.22
h,k,lmax        24,12,11                  24,12,10
Nref            3461                      3335
Tmin,Tmax       0.871,0.975               0.765,1.000
Tmin'           0.780

Correction method= # Reported T Limits: Tmin=0.765 Tmax=1.000
AbsCorr = GAUSSIAN

Data completeness= 0.964          Theta(max)= 78.905

R(reflections)= 0.0319( 2985)     wR2(reflections)= 0.0842( 3335)

S = 1.067                 Npar= 220
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level G

```
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        114 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         10 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   0 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   3 ALERT level G = General information/check it is not something unexpected

   0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   1 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock mwp54_f1 - ellipsoid plot