# SUPPORTING INFORMATION

# Regioselectivity in reactions between Bis(2-benzothiazolyl)ketone and vinyl Grignard reagents: *C*- versus *O*- alkylation. Part III[&]

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**Content** **Page**

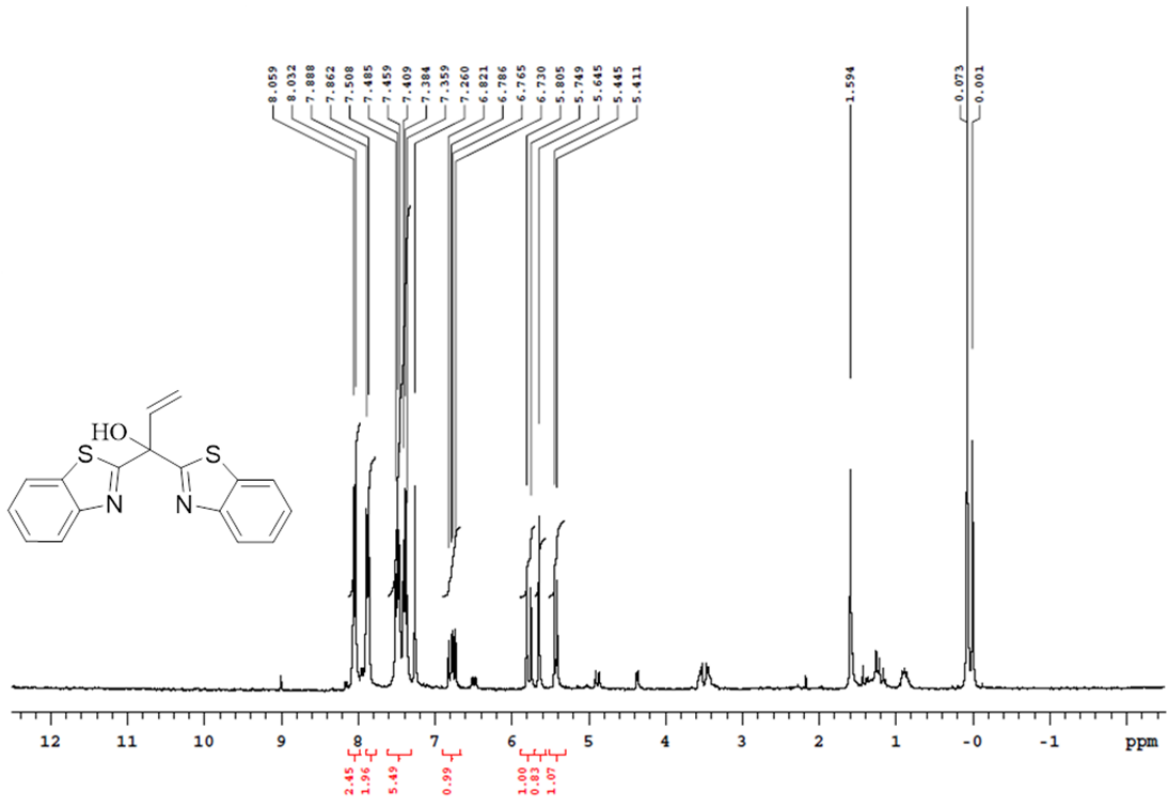


**Figure 1.** $^{1}H$ NMR spectrum ($CDCl_3$) of **3a**.

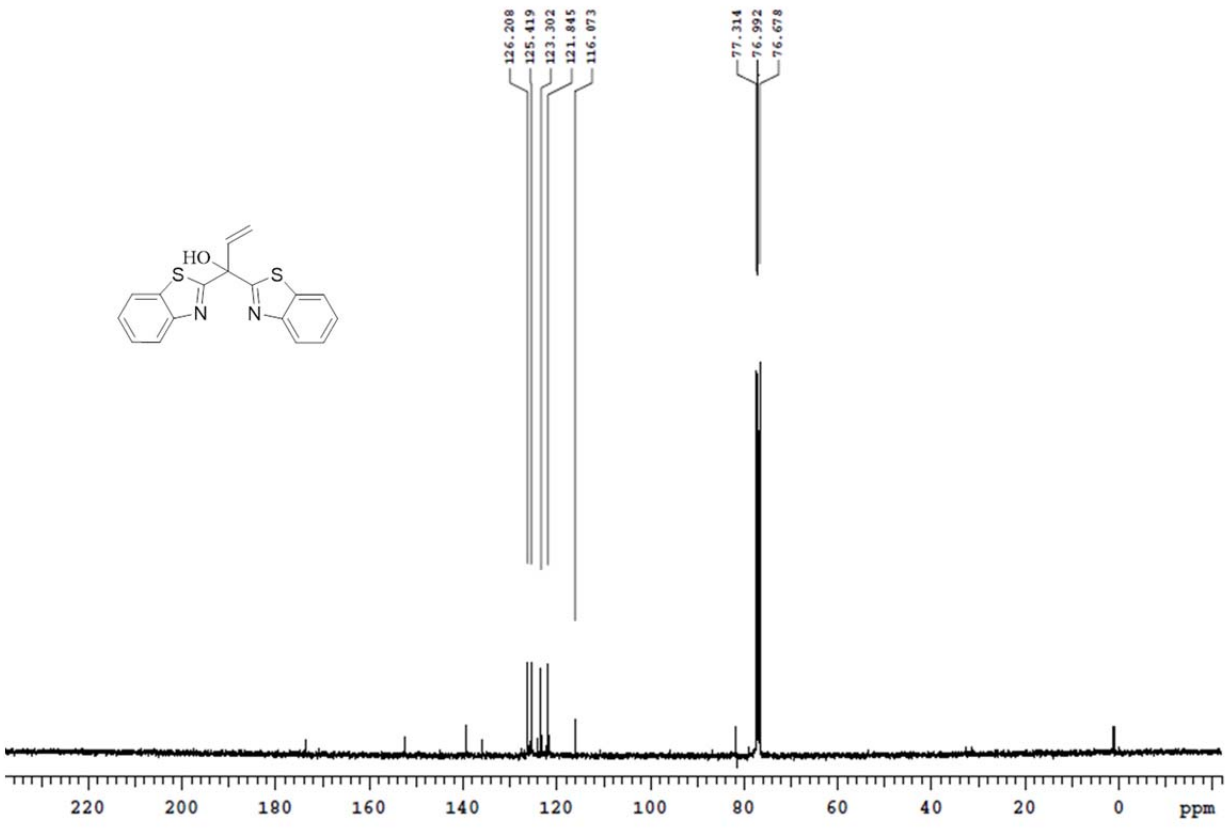


**Figure 2.** $^{13}C$ NMR spectrum ($CDCl_3$) of **3a.**

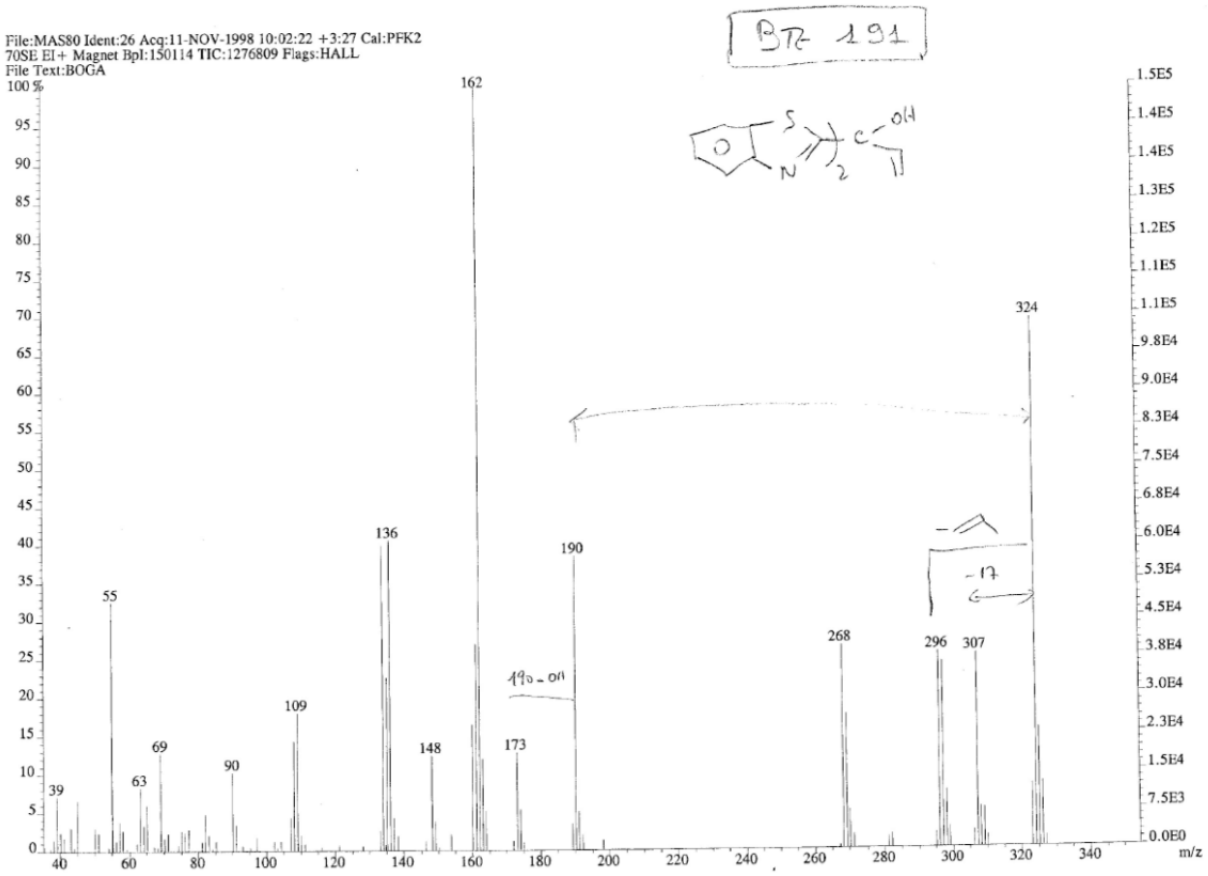


**Figure 3.** MASS Spectrum of **3a**

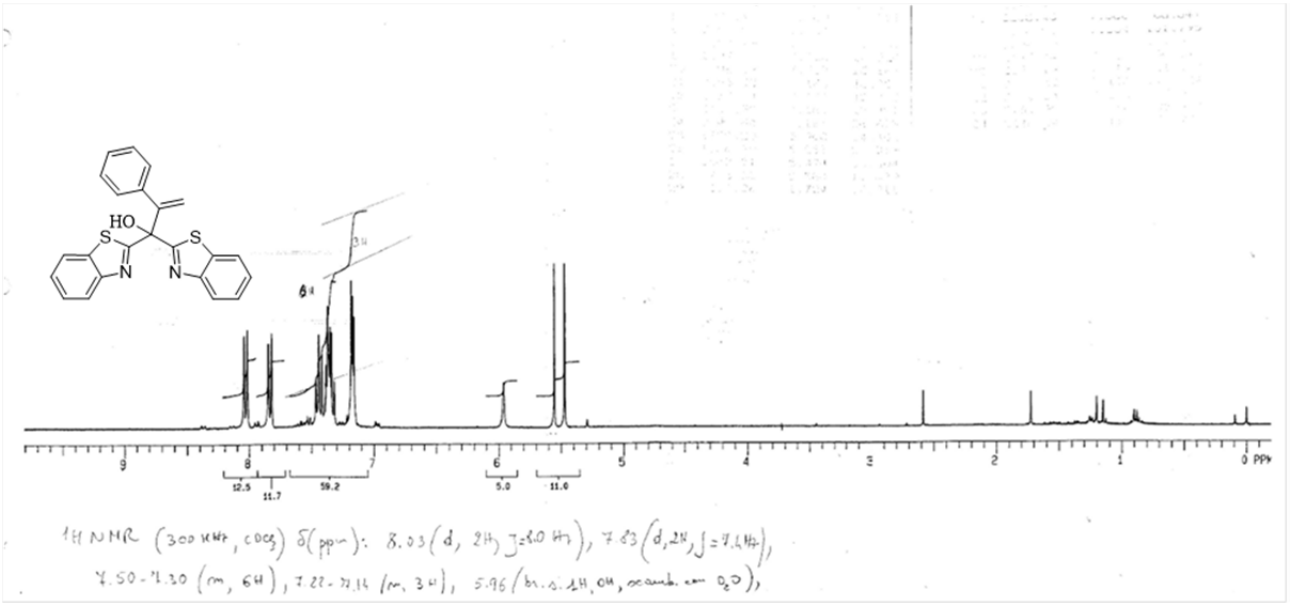


**Figure 4.** $^{1}$**H NMR spectrum (CDCl$_3$) of 3d.**

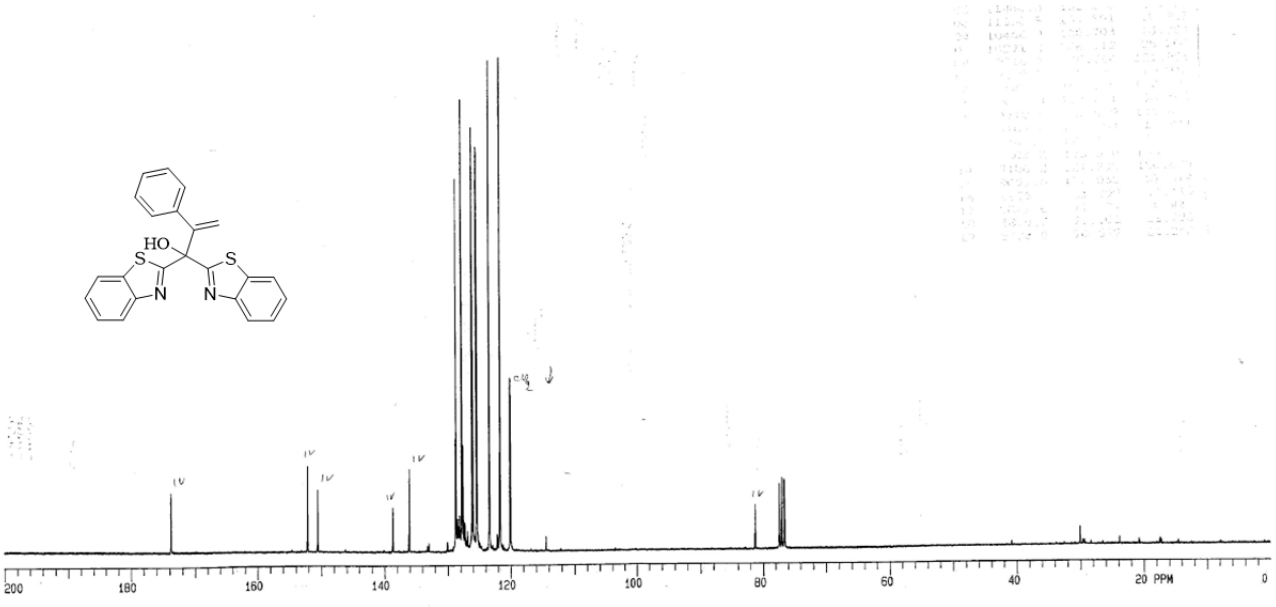


**Figure 5.** $^{13}C$ NMR spectrum ($CDCl_3$) of **3d.**

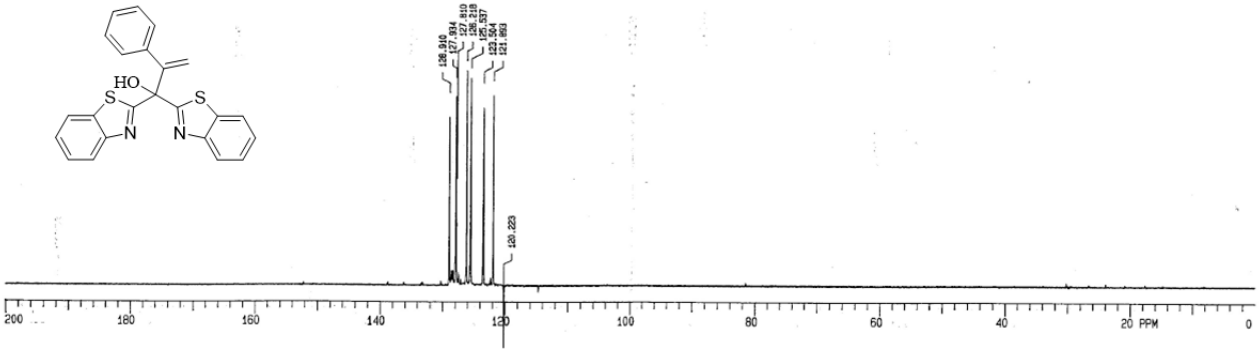


**Figure 6.** DEPT 135 NMR spectrum ($CDCl_3$) of **3d.**

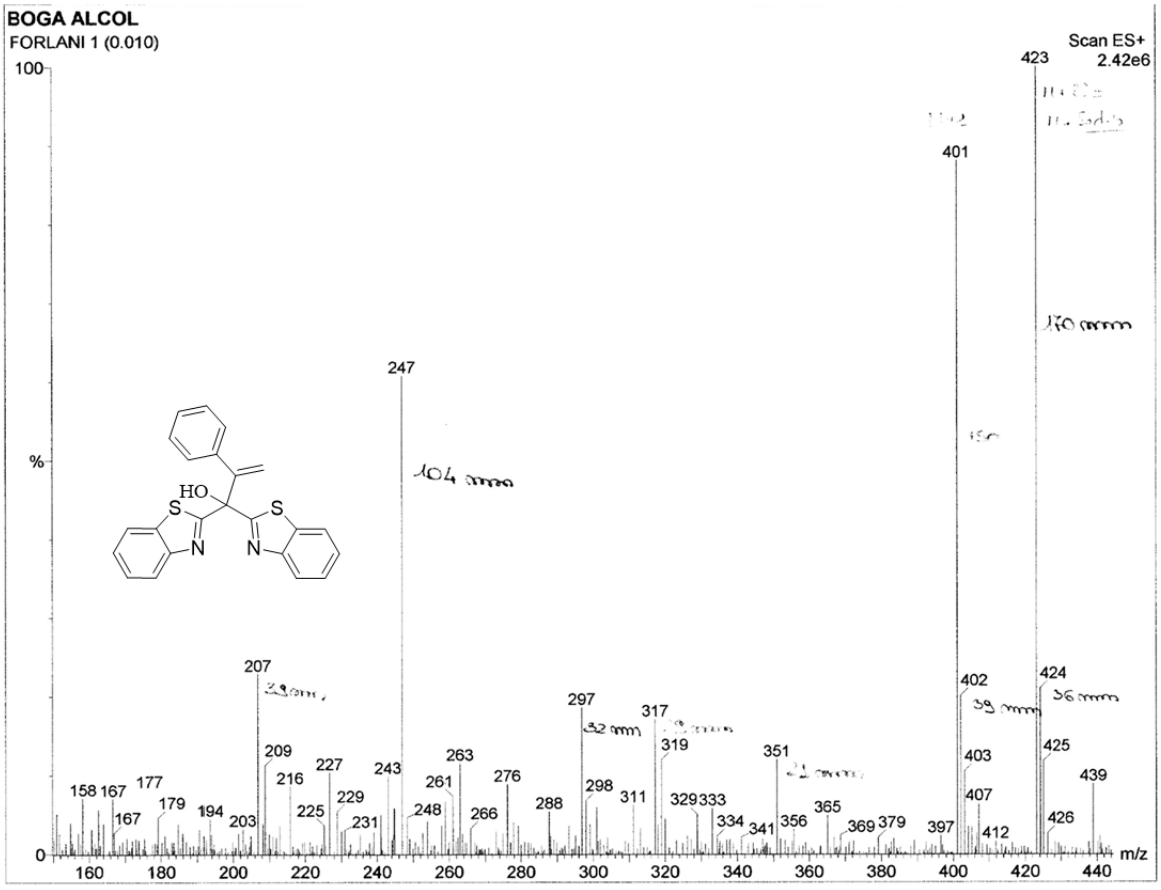


**Figure 7.** Mass spectrum (ESI$^+$) of **3d.**

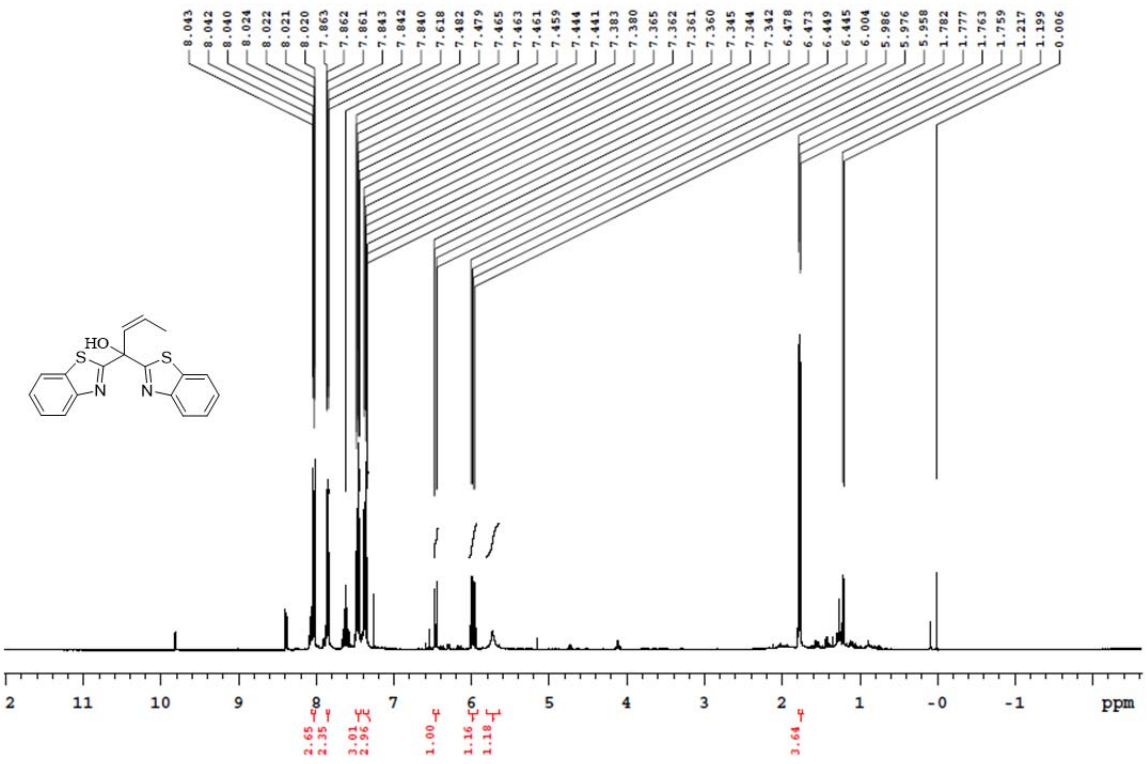


**Figure 8.** $^1$H NMR spectrum ($CDCl_3$) of **3e** with traces of **1**.

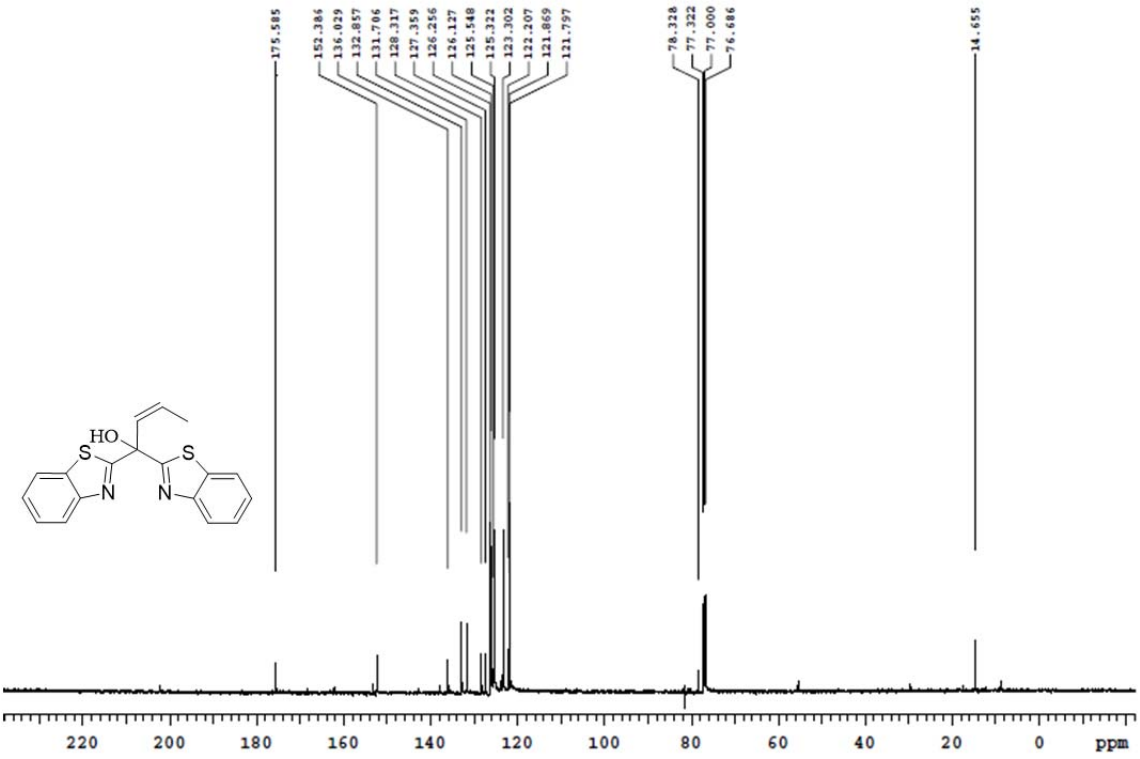


**Figure 9.** $^{13}C$ NMR spectrum ($CDCl_3$) of **3e** with traces of **1.**

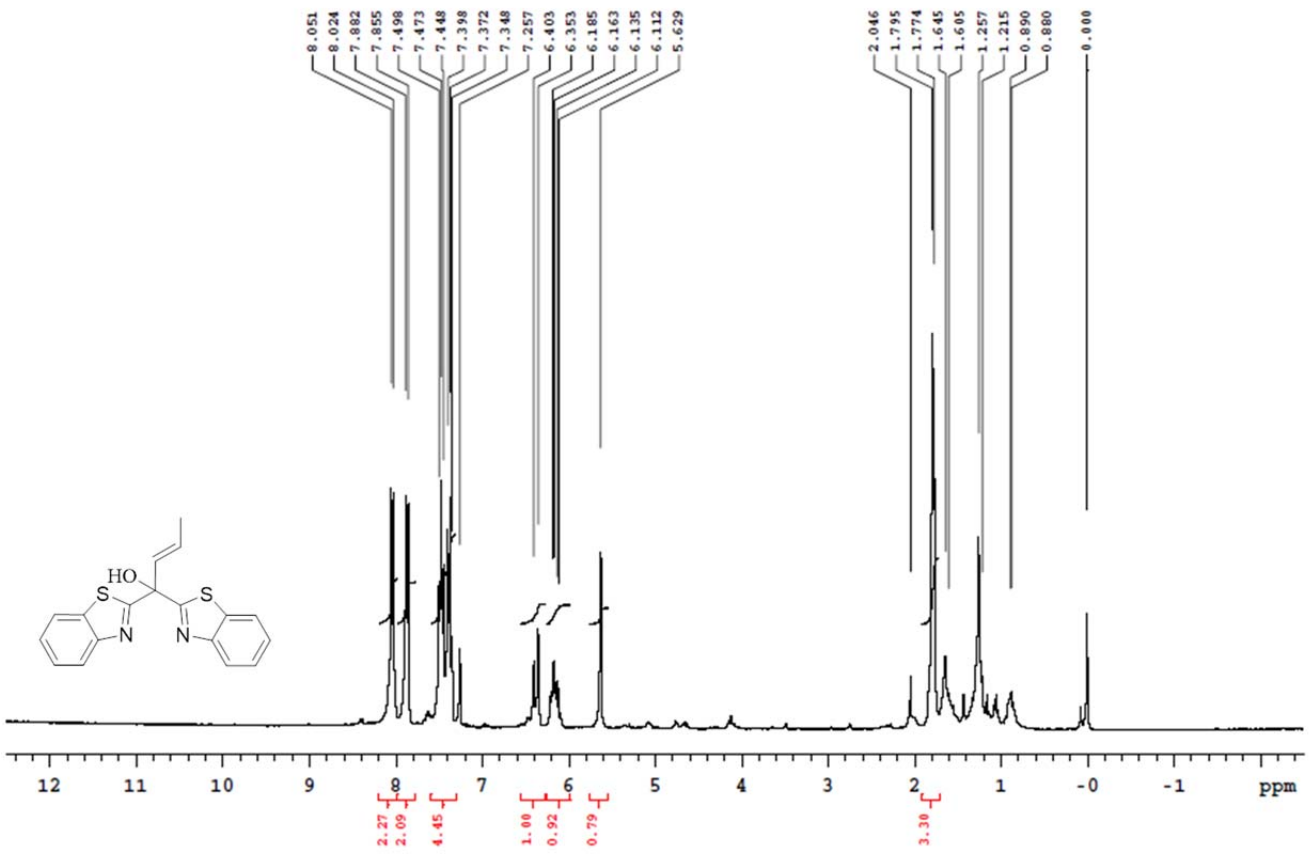


**Figure 10.** $^{1}H$ NMR spectrum ($CDCl_3$) **3f** (traces of water and solvents from FC).

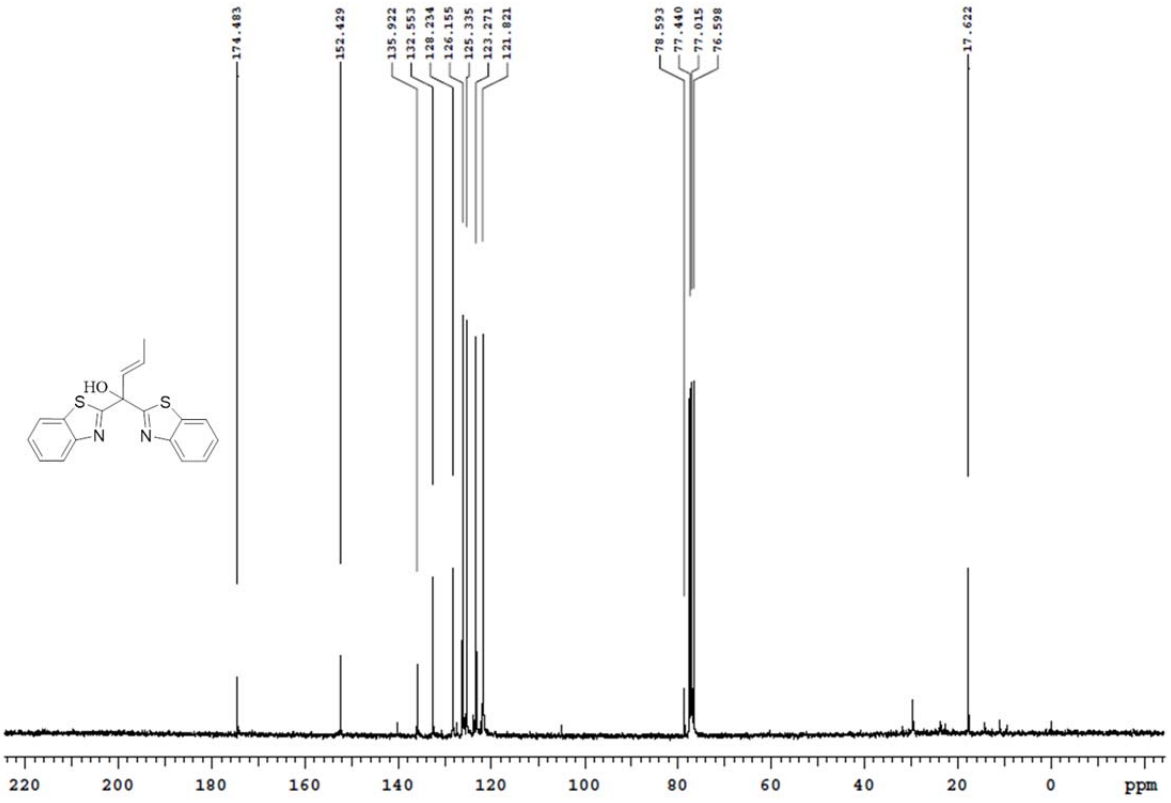


**Figure 11.** $^{13}C$ NMR spectrum ($CDCl_3$) of **3f.**

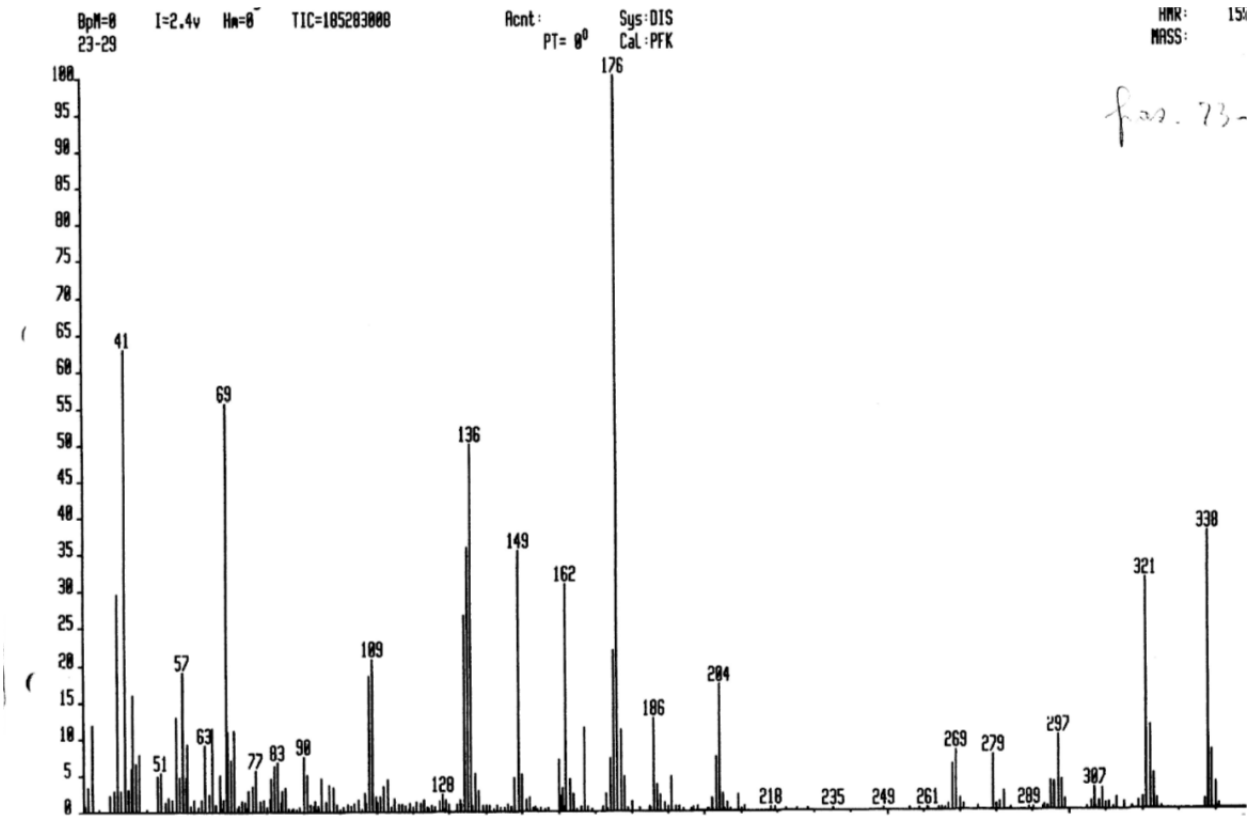


**Figure 12.** MASS Spectrum of **3f**.

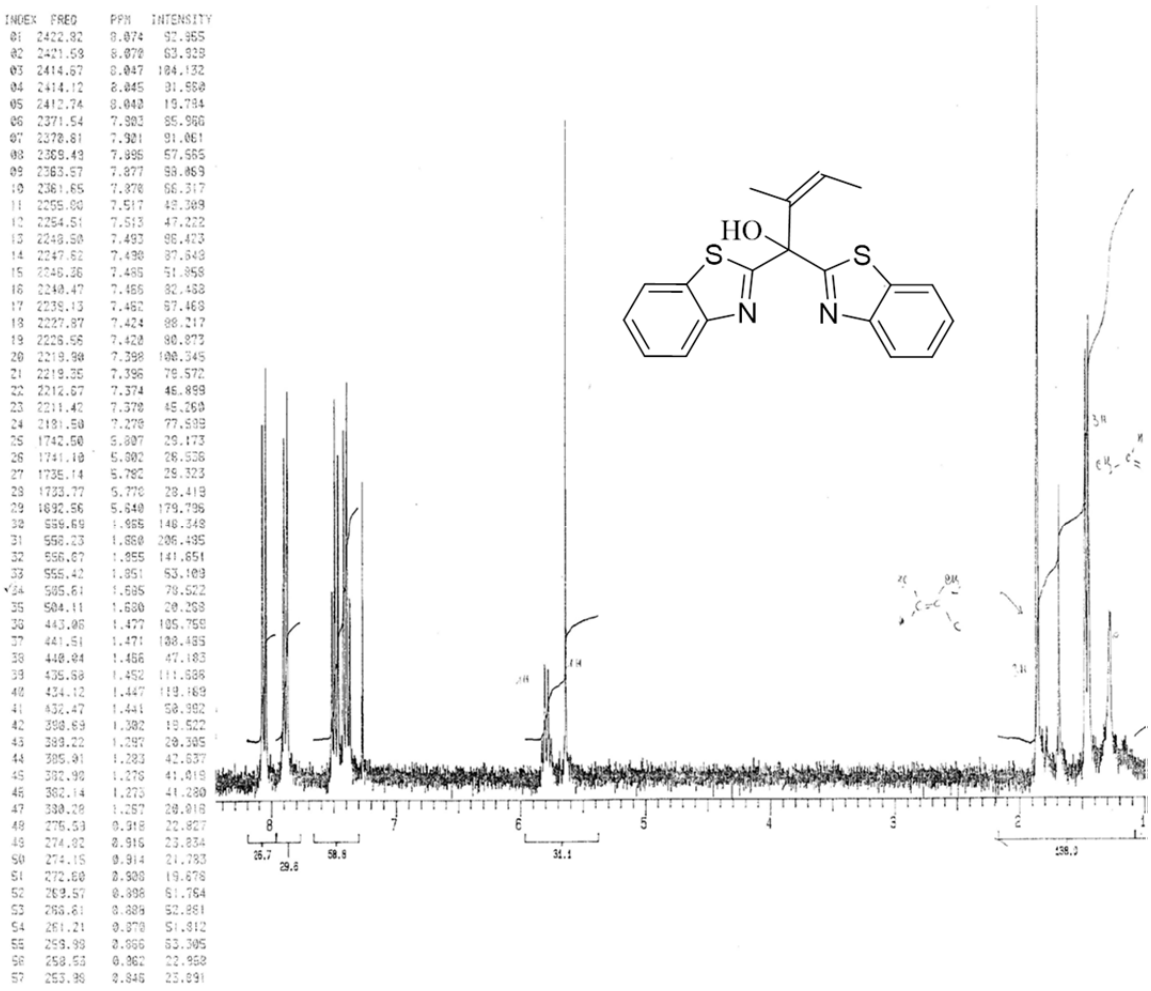

**Figure 13.** $^{1}$H NMR spectrum ($CDCl_3$) of **3g**.

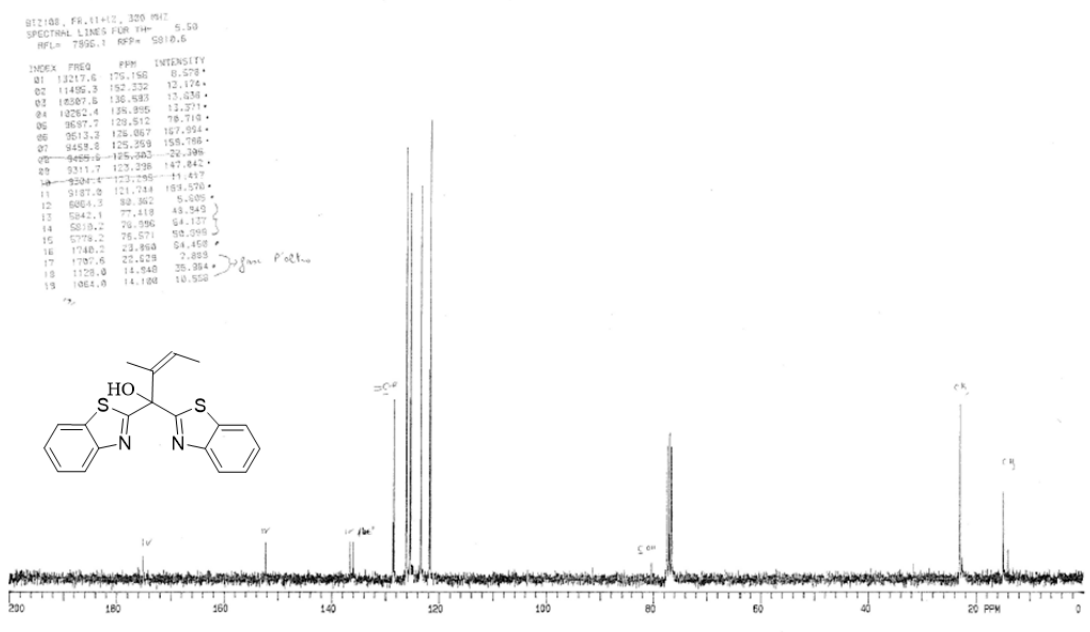

**Figure 14**. $^{13}$C NMR spectrum ($CDCl_3$) of **3g**.

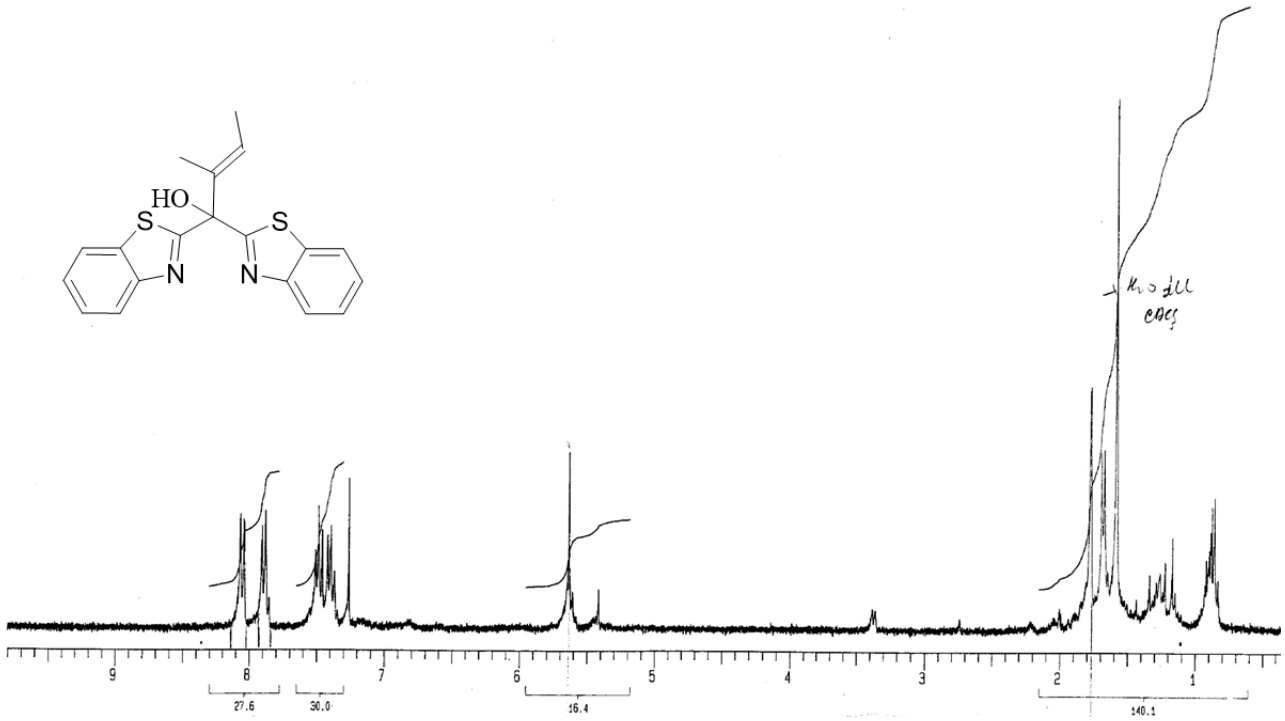


**Figure 15.** $^{1}$H NMR spectrum ($CDCl_3$) of **3h**.

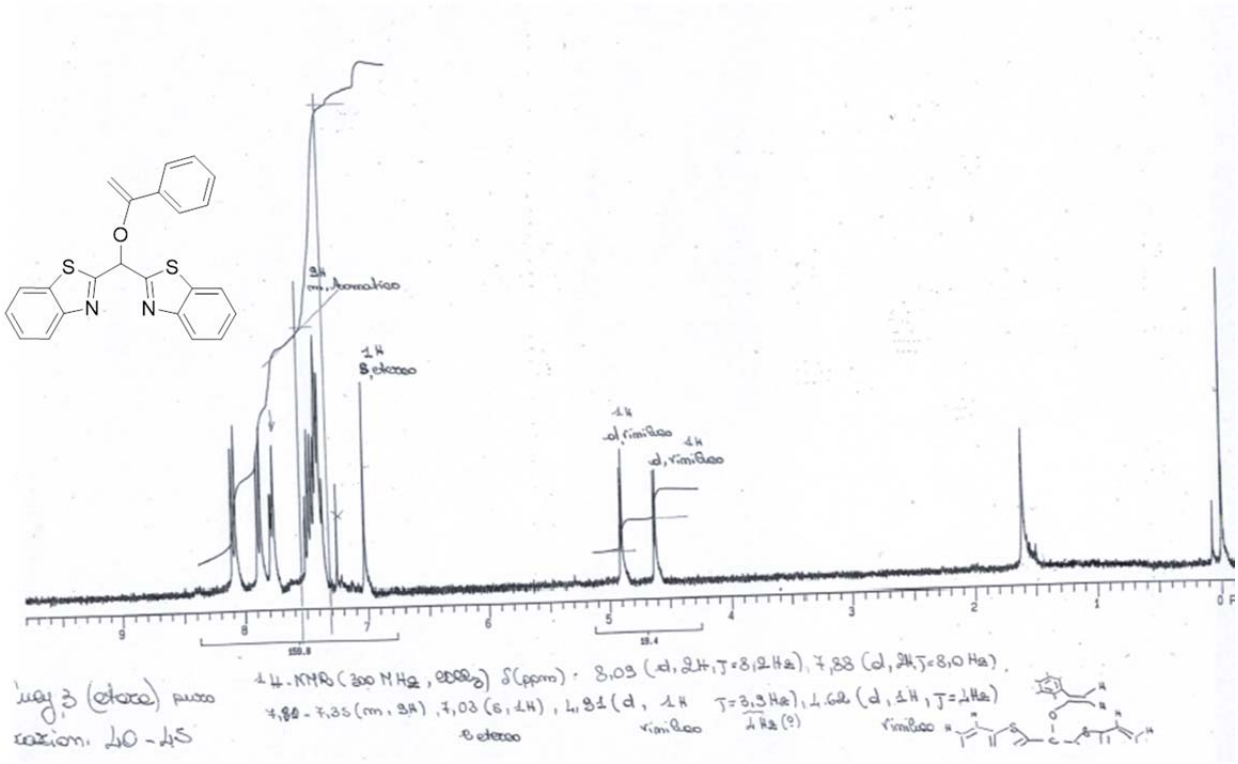


**Figure 16.** $^{1}$H NMR spectrum ($CDCl_3$) of **4d**.

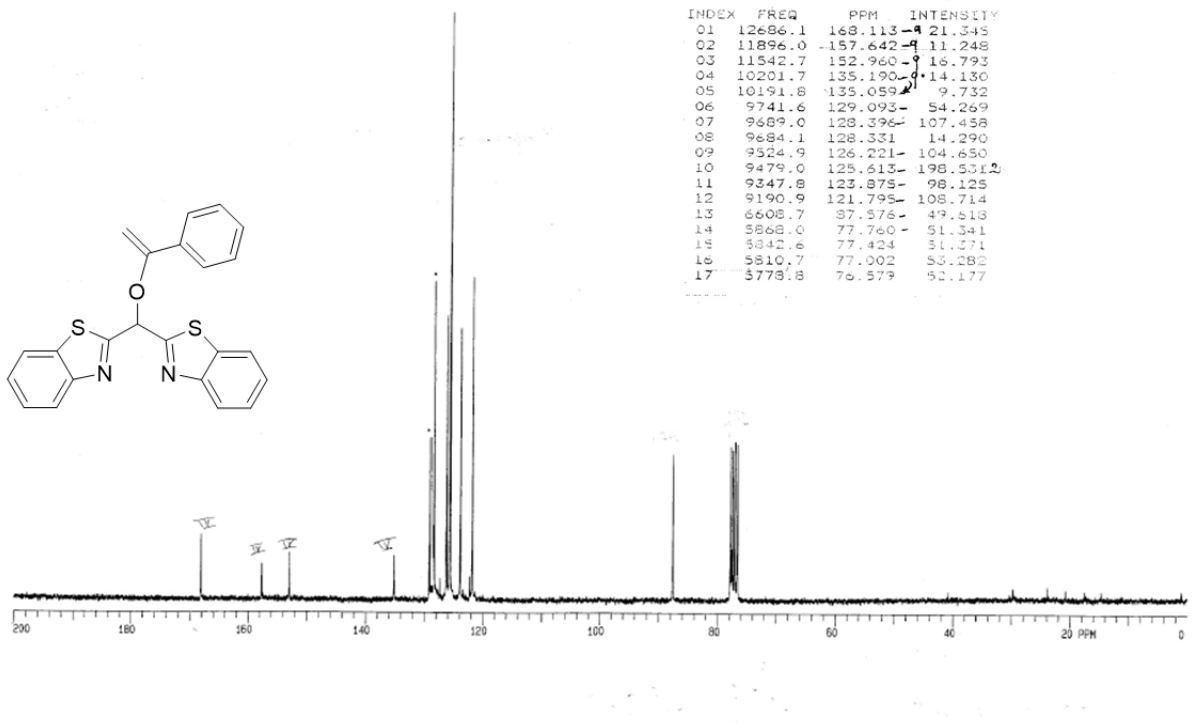


**Figure 17.** $^{13}C$ NMR spectrum ($CDCl_3$) of **4d**.

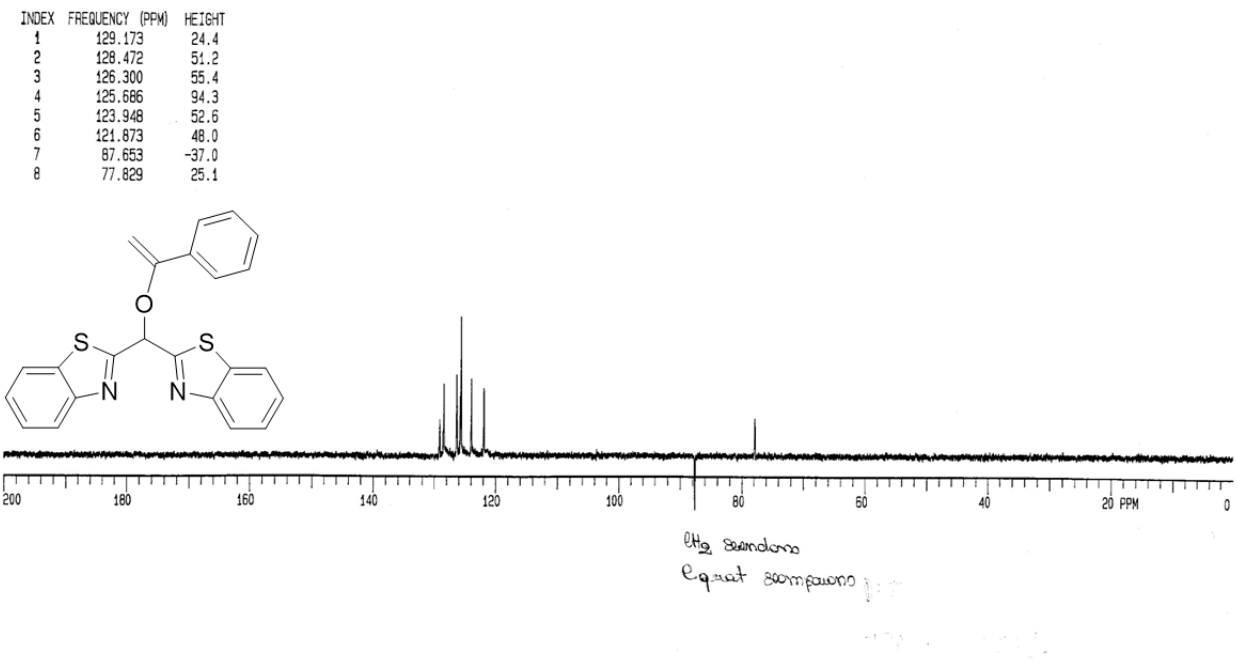


**Figure 18.** DEPT 135 spectrum ($CDCl_3$) of **4d**.

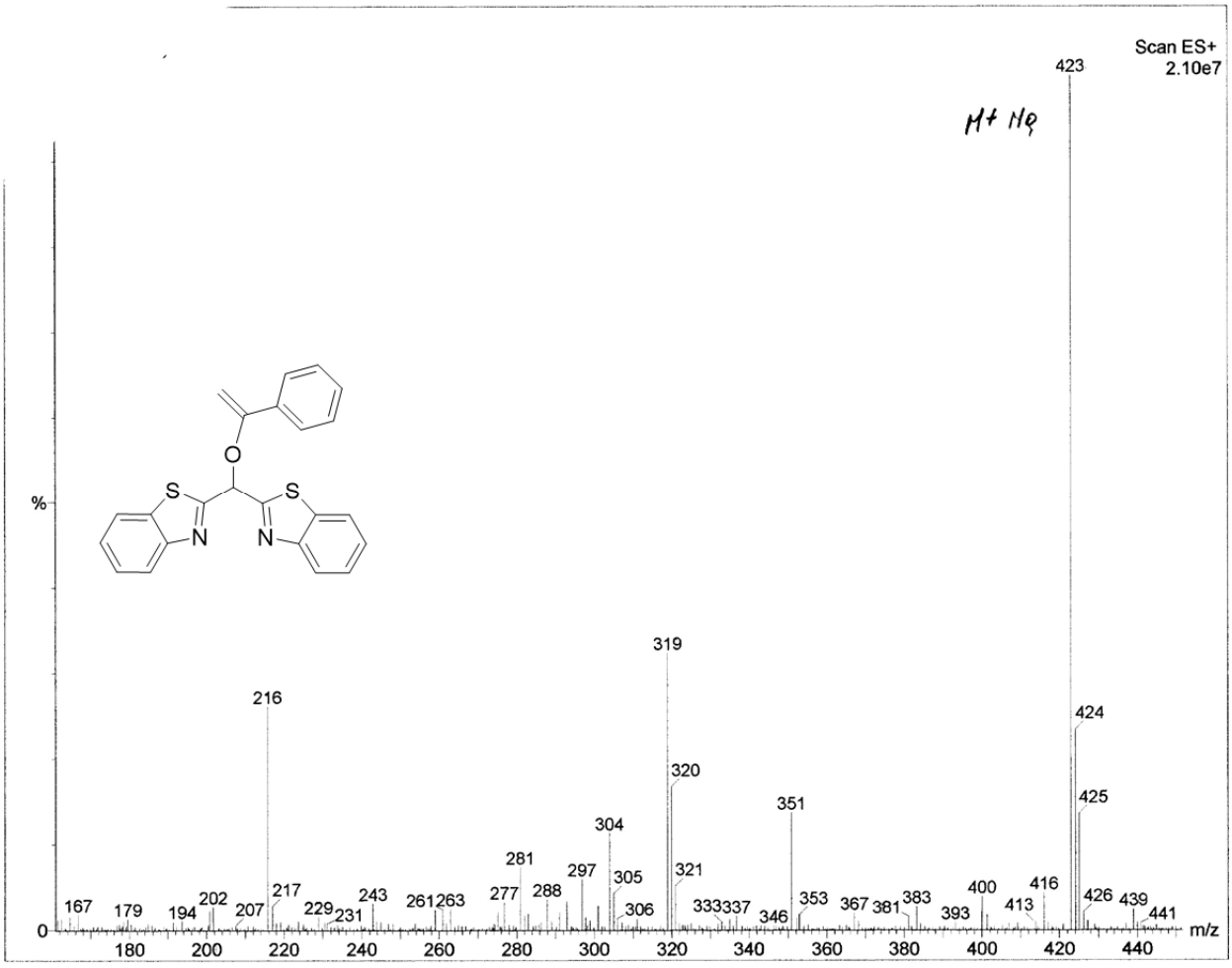


**Figure 19.** $ESI^+$ Mass spectrum of **4d**.

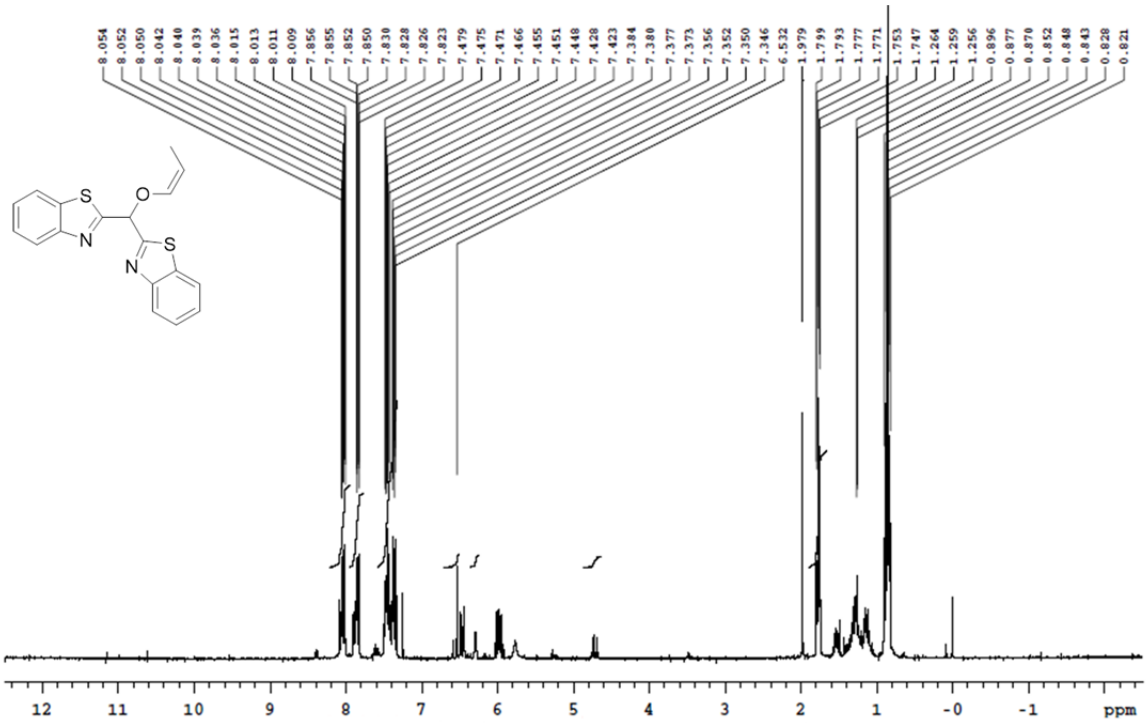


**Figure 20.** $^{1}$H NMR spectrum ($CDCl_3$) of **3e** + **4e**.

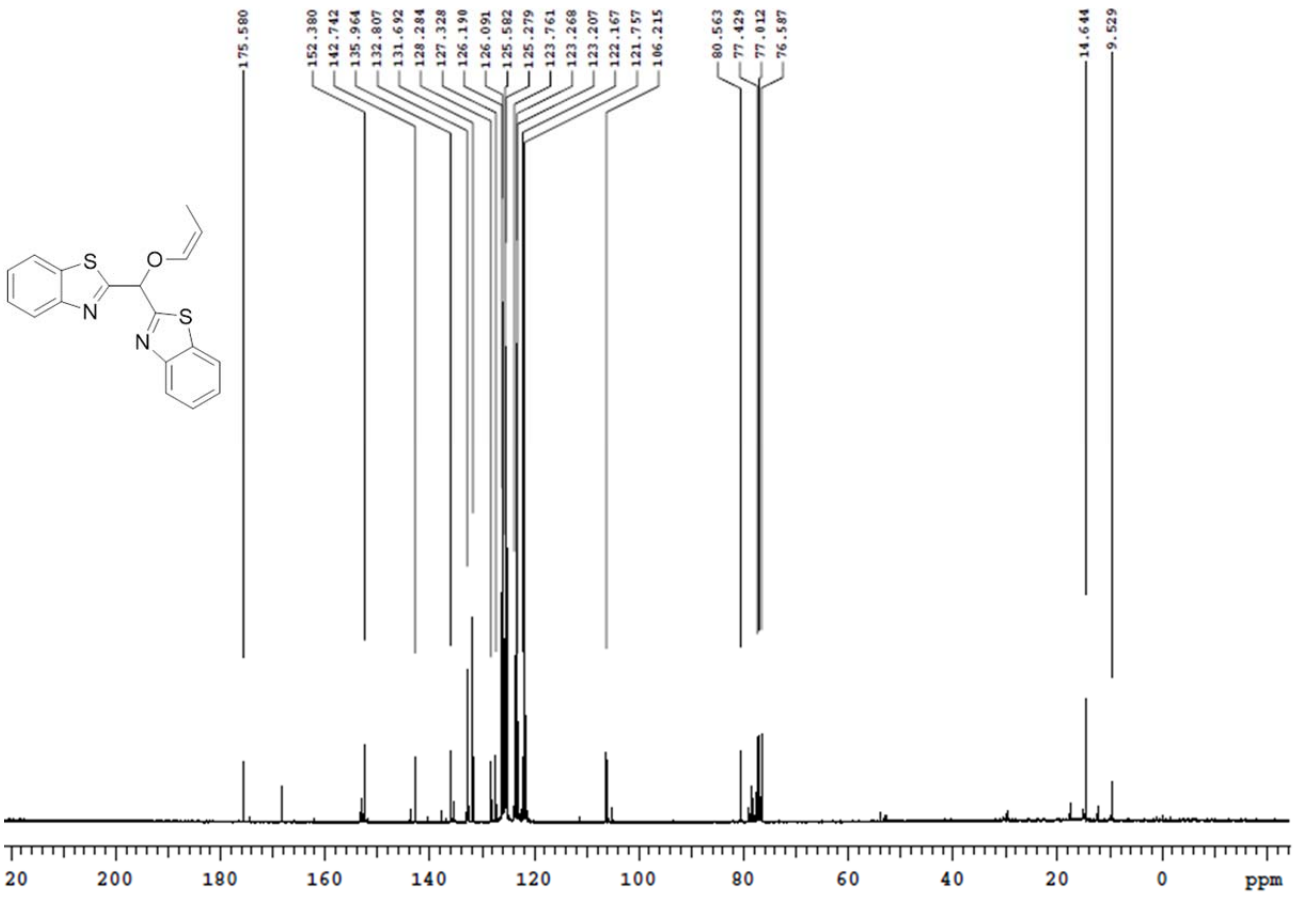


**Figure 21.** $^{13}$C NMR spectrum ($CDCl_3$) of **3e** + **4e**.

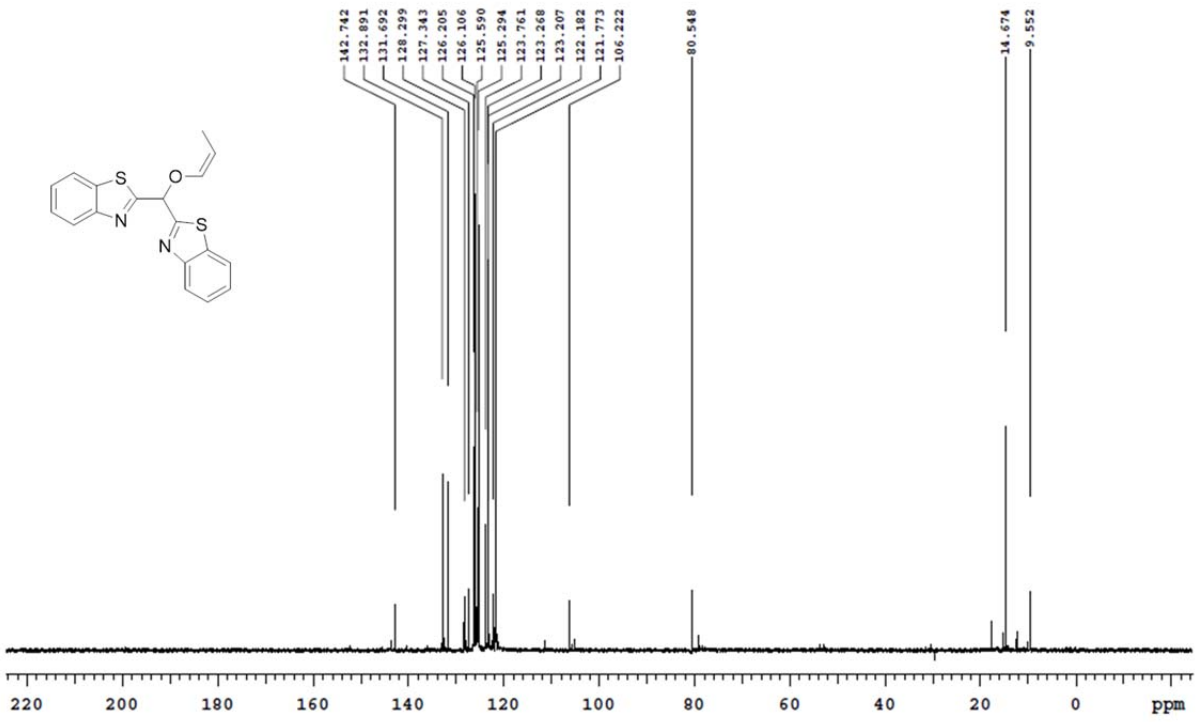


**Figure 22.** DEPT 135 ($CDCl_3$) of **3e** + **4e**.

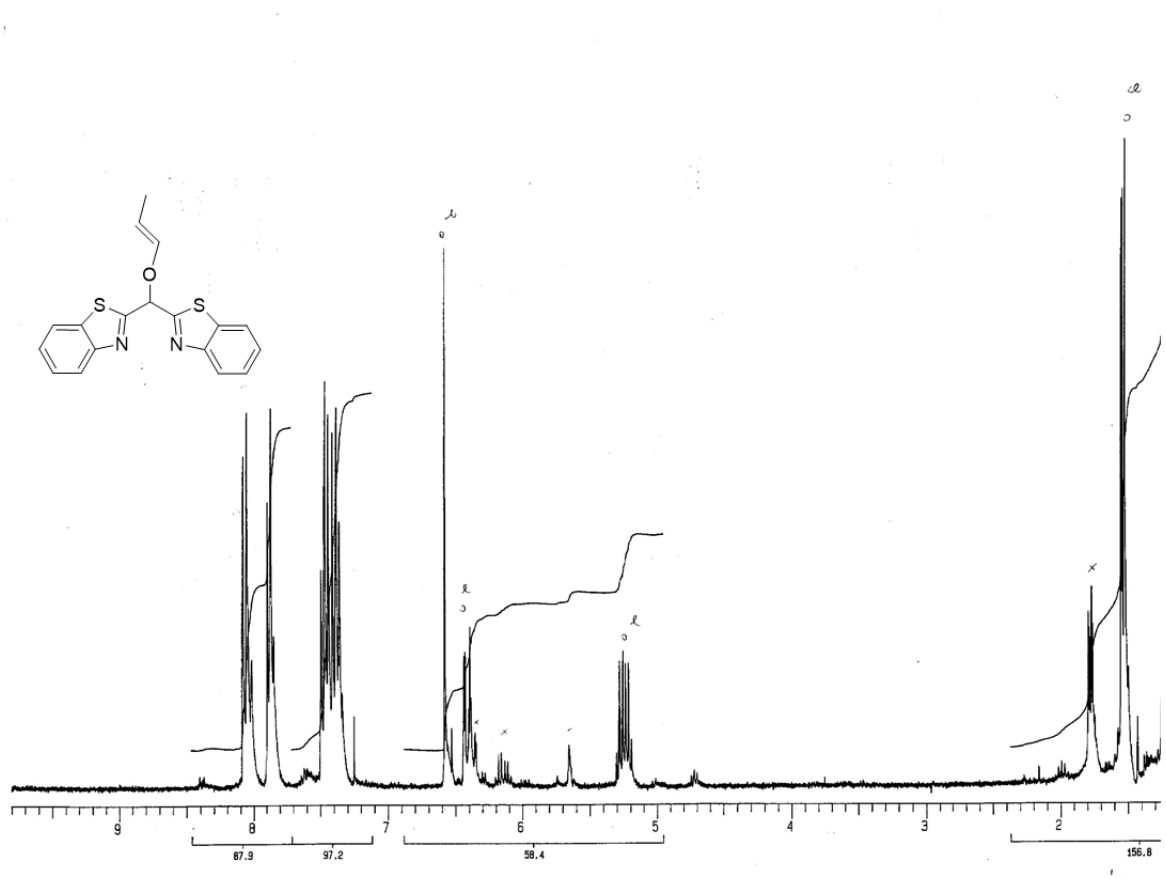


**Figure 23.** $^1$H NMR spectrum ($CDCl_3$) of **4f**.

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No syntax errors found. CIF dictionary Interpreting this report

## Datablock: shelx

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Bond precision:  C-C = 0.0030 A          Wavelength=0.71073

Cell:            a=18.3273(12)     b=16.2782(11)     c=4.2718(3)
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Temperature:     273 K

                 Calculated                Reported
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Hall group       P 2c -2n                  P 2c -2n
Moiety formula   C15 H8 N2 O S2            ?
Sum formula      C15 H8 N2 O S2            C15 H8 N2 O S2
Mr               296.35                    296.35
Dx,g cm-3        1.545                     1.545
Z                4                         4
Mu (mm-1)        0.412                     0.412
F000             608.0                     608.0
F000'            609.19
h,k,lmax         24,21,5                   24,20,5
Nref             3264[ 1844]               3045
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Tmin'            0.891

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AbsCorr = MULTI-SCAN

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R(reflections)= 0.0255( 2843)     wR2(reflections)= 0.0691( 3045)

S = 1.076                 Npar= 181
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The following ALERTS were generated. Each ALERT has the format
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Click on the hyperlinks for more details of the test.

**Alert level G**

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PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)        273 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)        273 Check
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```
   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
   2 ALERT level G = General information/check it is not something unexpected

   2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   0 ALERT type 2 Indicator that the structure model may be wrong or deficient
   0 ALERT type 3 Indicator that the structure quality may be low
   0 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.

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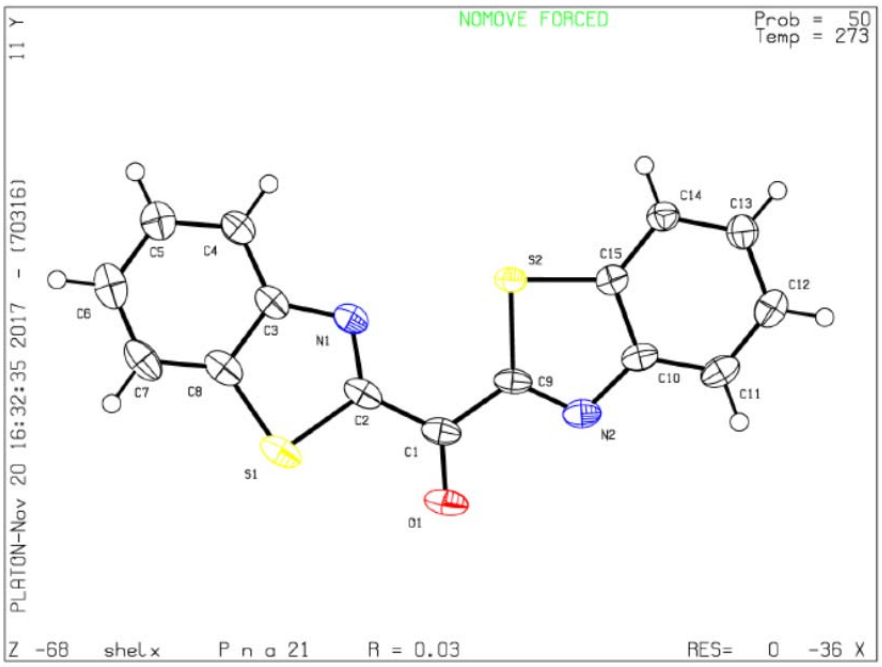
**Datablock shelx** - ellipsoid plot



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No syntax errors found. CIF dictionary Interpreting this report

## Datablock: shelx

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Cell:            a=3.7993(3)     b=29.361(2)        c=13.7502(10)
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                 Calculated                  Reported
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Space group      C c                         C c
Hall group       C -2yc                      C -2yc
Moiety formula   C7 H4 N2 O S2               ?
Sum formula      C7 H4 N2 O S2               C7 H4 N2 O S2
Mr               196.24                      196.24
Dx,g cm-3        1.701                       1.700
Z                8                           8
Mu (mm-1)        0.636                       0.636
F000             800.0                       800.0
F000'            802.22
h,k,lmax         5,38,18                     4,38,18
Nref             3939[ 1979]                 3461
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Tmin'            0.881

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AbsCorr = MULTI-SCAN

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R(reflections)= 0.0239( 3310)      wR2(reflections)= 0.0597( 3461)

S = 1.037                  Npar= 217
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The following ALERTS were generated. Each ALERT has the format
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Click on the hyperlinks for more details of the test.

**Alert level G**

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PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)        273 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)        273 Check
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   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
   2 ALERT level G = General information/check it is not something unexpected

   2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   0 ALERT type 2 Indicator that the structure model may be wrong or deficient
   0 ALERT type 3 Indicator that the structure quality may be low
   0 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.

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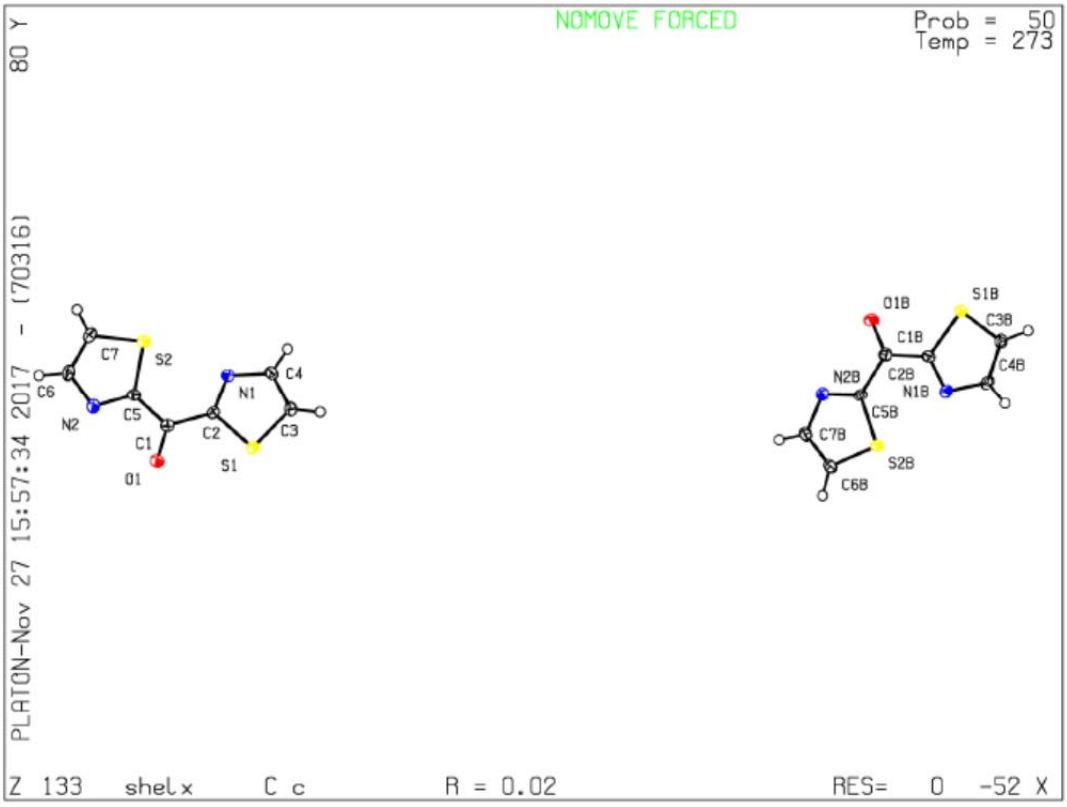
**Datablock shelx** - ellipsoid plot



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No syntax errors found. CIF dictionary Interpreting this report

## Datablock: shelx

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Bond precision:  C-C = 0.0019 A          Wavelength=0.71073

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Temperature:     273 K

                 Calculated                Reported
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Space group      P 21/c                    P 21/c
Hall group       -P 2ybc                   -P 2ybc
Moiety formula   C15 H8 N2 O3              ?
Sum formula      C15 H8 N2 O3              C15 H8 N2 O3
Mr               264.23                    264.23
Dx,g cm-3        1.480                     1.480
Z                4                         4
Mu (mm-1)        0.106                     0.106
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F000'            544.27
h,k,lmax         17,15,10                  16,15,10
Nref             2857                      2785
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S = 0.993                 Npar= 182
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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 B**

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PLAT230_ALERT_2_B Hirshfeld Test Diff for  N1       --C2       ..        9.6 s.u.
PLAT230_ALERT_2_B Hirshfeld Test Diff for  N2       --C9       ..        7.4 s.u.
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**Alert level G**

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PLAT200_ALERT_1_G Reported  _diffrn_ambient_temperature ..... (K)        273 Check
PLAT398_ALERT_2_G Deviating  C-O-C    Angle From 120 for O2            103.4 Degree
PLAT398_ALERT_2_G Deviating  C-O-C    Angle From 120 for O3            103.6 Degree
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```
  0 ALERT level A = Most likely a serious problem - resolve or explain
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  0 ALERT level C = Check. Ensure it is not caused by an omission or oversight
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  0 ALERT type 4 Improvement, methodology, query or suggestion
  0 ALERT type 5 Informative message, check
```

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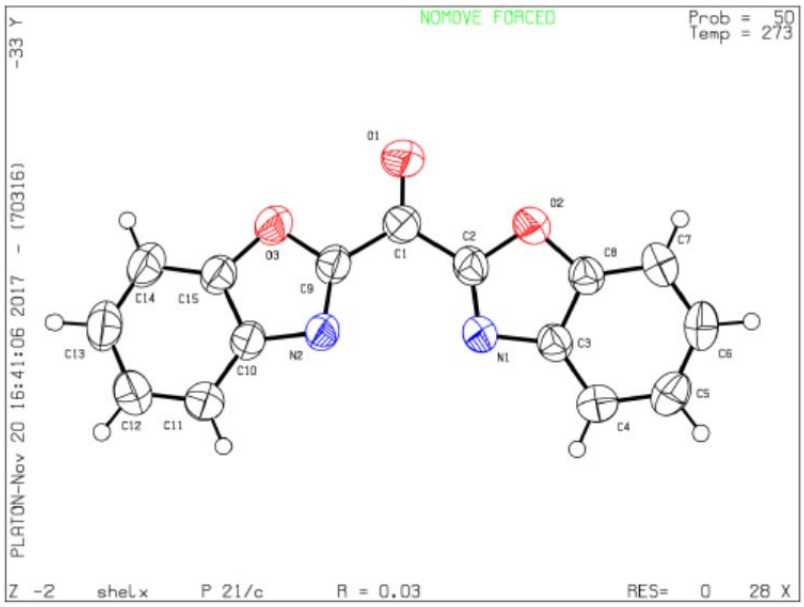

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## Datablock: shelx

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                 Calculated                 Reported
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Space group      P 21/n                     P 21/n
Hall group       -P 2yn                     -P 2yn
Moiety formula   C17 H14 N4 O, 2(H2 O)      ?
Sum formula      C17 H18 N4 O3              C17 H18 N4 O3
Mr               326.35                     326.35
Dx,g cm-3        1.350                      1.350
Z                4                          4
Mu (mm-1)        0.095                      0.095
F000             688.0                      688.0
F000'            688.30
h,k,lmax         9,19,17                    9,19,17
Nref             3239                       3230
Tmin,Tmax        0.983,0.991                0.876,0.945
Tmin'            0.981

Correction method= # Reported T Limits: Tmin=0.876 Tmax=0.945
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 26.237

R(reflections)= 0.0448( 2291)     wR2(reflections)= 0.1366( 3230)

S = 1.090                 Npar= 231
```

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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 B**

PLAT417_ALERT_2_B Short Inter D-H..H-D H2W ..H3W .. 1.93 Ang.
PLAT420_ALERT_2_B D-H Without Acceptor O2W --H3W . Please Check

**Alert level C**

PLAT355_ALERT_3_C Long O-H (X0.82,N0.98A) O2W - H3W . 1.04 Ang.

**Alert level G**

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 273 Check
PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 273 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 4 Note

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

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 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 09/11/2017; check.def file version of 08/11/2017**

Datablock shelx - ellipsoid plot