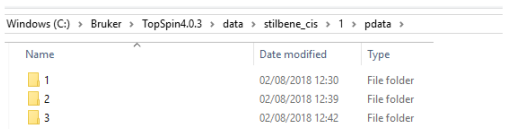
ANATOLIA: NMR Spectrum Analysis

Loughborough University

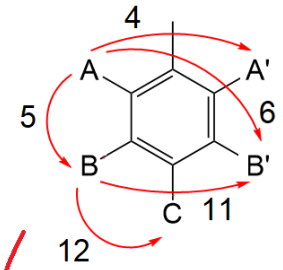
In Windows:
Copy pdata "1" and paste as "2", then repeat ans ave as "3"
C:\Bruker\TopSpin4.0.3\data\stilbene_cis\1\pdata
original FID/spectrum in "1"
broadened spectrum in "2"
calculated spectrum in "3"



In Windows copy the "ANATOLIA" executable file into:
"C:\Bruker\TopSpin4.0.3\prog\anatolia\"

In Windows copy the "ANATOLIA" AU file into:
"C:\Bruker\TopSpin4.0.3\exp\stan\nmr\au\src\user

The command "ANATOLIA" should now run in the TOPSPIN command line.




```
1   FreqA       2915.01
2   FreqB       2904.5
3   FreqC       2890.76
4   4J12           2.049
5   3J13           7.92
6   5J14           0.6
7   4J15           1.22
8   3J23          0.6
9   3J24          7.92
10  4J25           1.22
11  4J34           1.424
12  3J35           7.47
13  3J45           7.47
14   LW            0.5
15   Magn          1e8
```

"0" means optimise parameters listed at the bottom
"1" means calculate the spectrum using the input data
"Nspins" how many spins have been defined in the data file

```
SimMode 0

Spin System
NSpins 5
Chemical shift indices
1  1  2  2  3
J-coupling indices
  4  5  6  7
     8  9  10
        11 12
           13

Spectra parameters
PathToDataSet            ./
ExpProcNo                1
ExpBroadedProcNo         2
CalcProcNo               3

Optimization parameters
InputParameters                 parameters_start.txt
OutputParameters                parameters.txt
SpectraTextOutput               output.txt
LBs                             3.0 2.0 1.0 0.8 0.6 0.4 0.2 0.1 0.0
MagnitudeFromExpSpec            1

List of optimized parameters
1 2 3
```

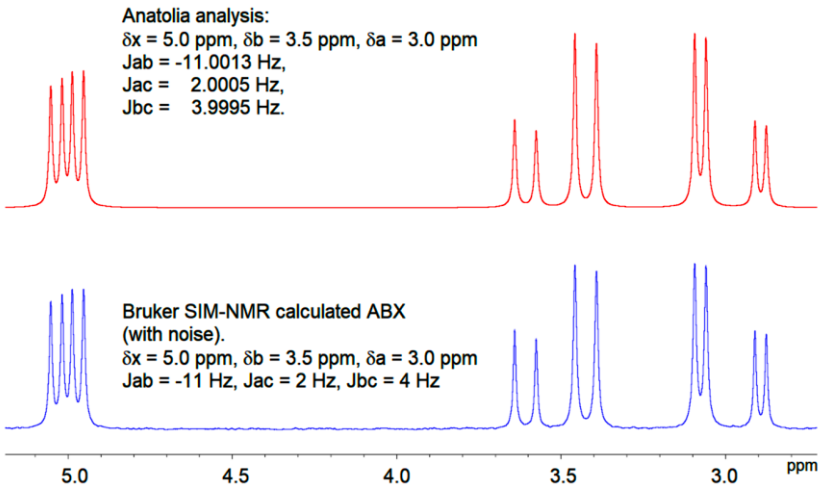
Chemical Shift: 1 1 2 2 3 an AA'BB'C spin system
this is linked the chemical shift values in the data file

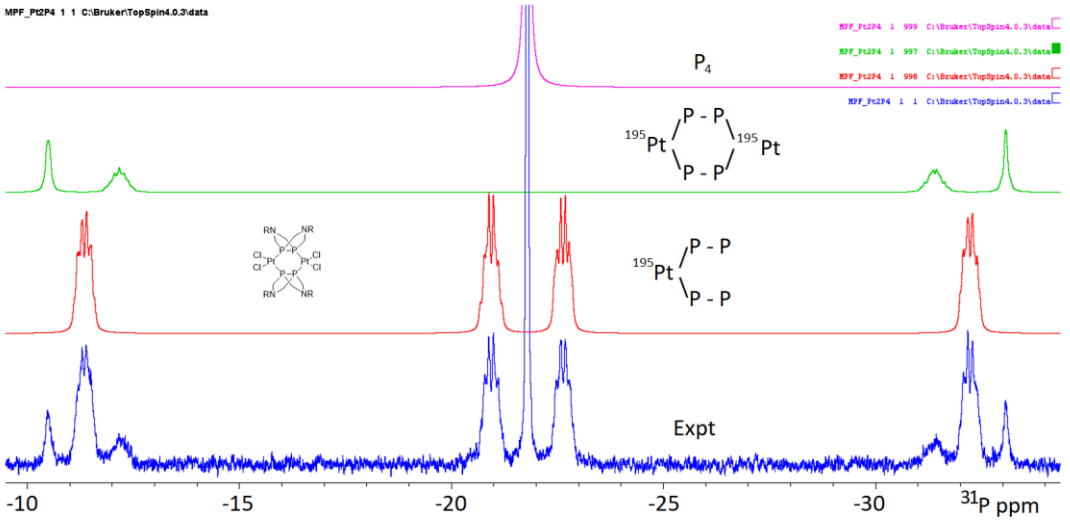
Coupling constants: these are listed in the data file
4 5 6 7 refers to J 1-2 1-3 1-4 1-5
AA' AB AB' AC
8 9 10 refers to J 2-3 2-4 2-5
A'B A'B' A'C
11 12 refers to J 3-4 3-5
BB' BC
13 refers to J 4-5
B'C

This input data file is placed in :
C:\Bruker\TopSpin4.0.3\data\spectrum\1
this is the same as ./ (Windows)

Broadening applied to spectrum saved in "2"

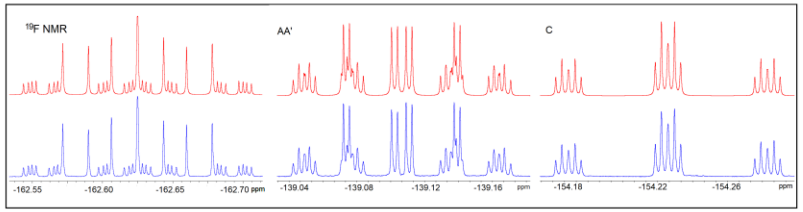
Optimised parameters:
these numbers refer to the line-number in the "parameter-start" file.
So, 1 2 3 means optimise the three chemical shift values.

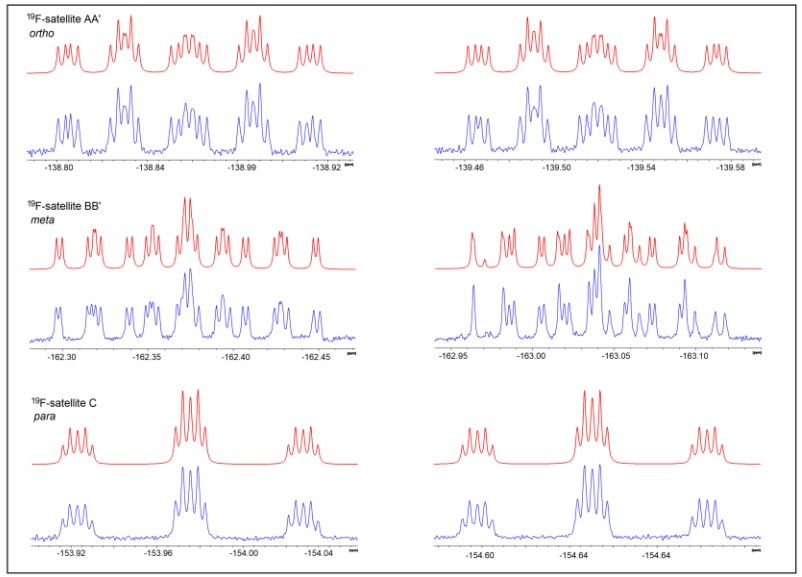


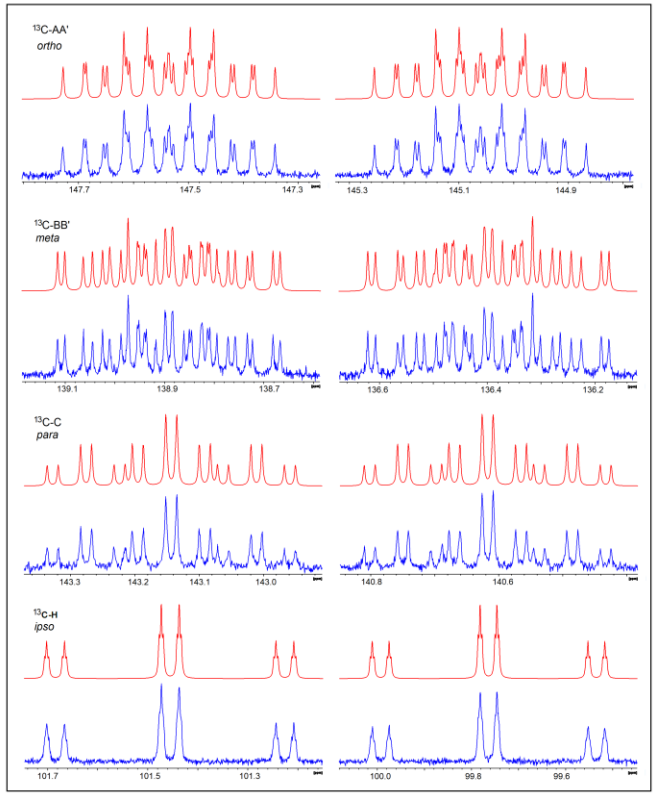


D. A. Cheshkov, K. F. Sheberstov, D. O. Sinitsyn, V. A. Chertkov, *Magn. Reson. Chem.* 56 (**2018**) 449-457.

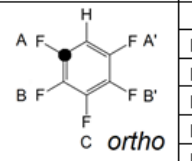
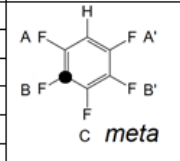
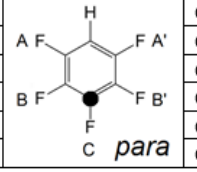
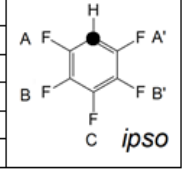
Dr. Mark Edgar

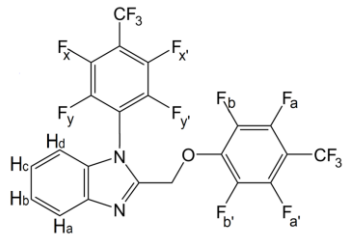


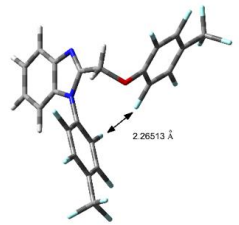


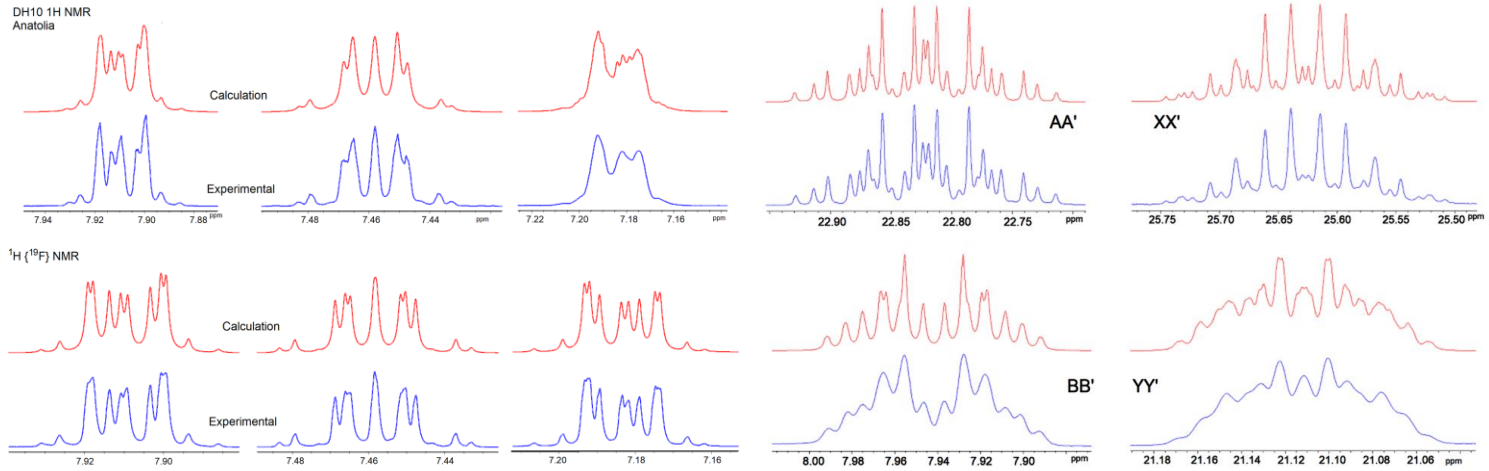


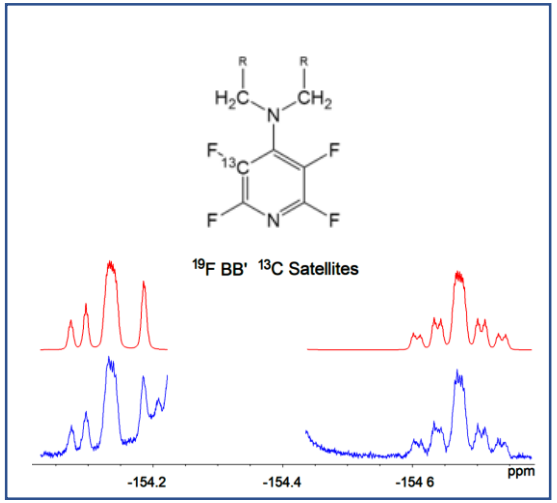
		1965[a]	2011 LC[b]	ANATOLIA[d]	G-03[f]
1,2	^4J AA'	± 2.4	−1.748	-2.173	5.86
1,3	^3J AB	± 20.6	−21.224	-21.781	-21.39
1,4	^5J AB'	± 8.8	8.667	8.738	10.73
1,5	^4J AC	± 1.3	1.141	1.235	-1.50
1,6	^3J AH	± 10.2	10.688	10.02	7.14
3,4	^4J BB'	± 1.2	−1.235	-1.195	-4.23
3,5	^3J BC	± 18.8	−19.381	-20.110	-21.14
3,6	^4J BH	± 6.9	7.147	6.873	5.28
5,6	^5J CH	± 2.7	−2.636	-2.659	-2.44

	ANATOLIA	G-03			ANATOLIA	G-03	
	Ortho				*Meta*		
A-A	-248.72	-318.16		B-A	10.09	9.93	
A-A'	7.38	6.42		B-A'	16.41	5.99	
A-B	11.16	5.15		B-B	-250.63	-319.33	
A-B'	-4.25	5.08		B-B'	-1.42	-2.15	
A-C	-3.76	0.006		B-C	9.14	6.56	
A-H	12.15	6.47		B-H	12.83	8.26	
	Para				*Ipso*		
C-A	5.22	1.44		C-A	23.03	14.16	
C-A'	5.22	1.44		C-A'	23.03	14.16	
C-B	13.31	6.84		C-B	0.42	-2.87	
C-B'	13.31	6.84		C-B'	0.42	-2.87	
C-C	-253.91	-321.89		C-C	-3.60	3.88	
C-H	-1.72	-1.52		C-H	169.98	160.43	

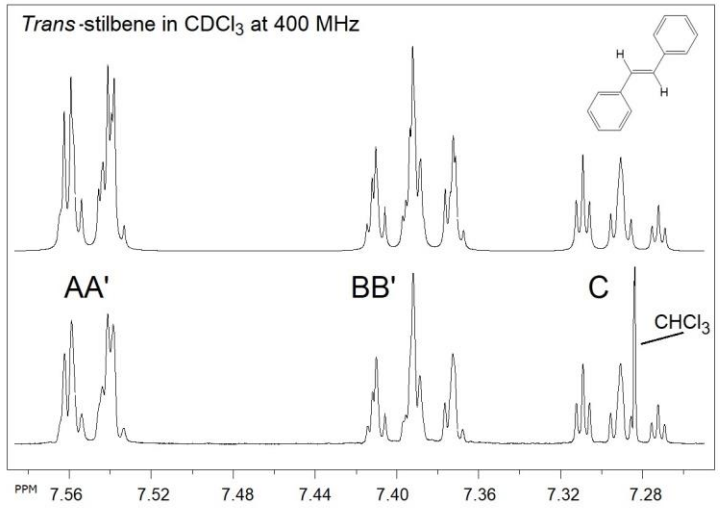


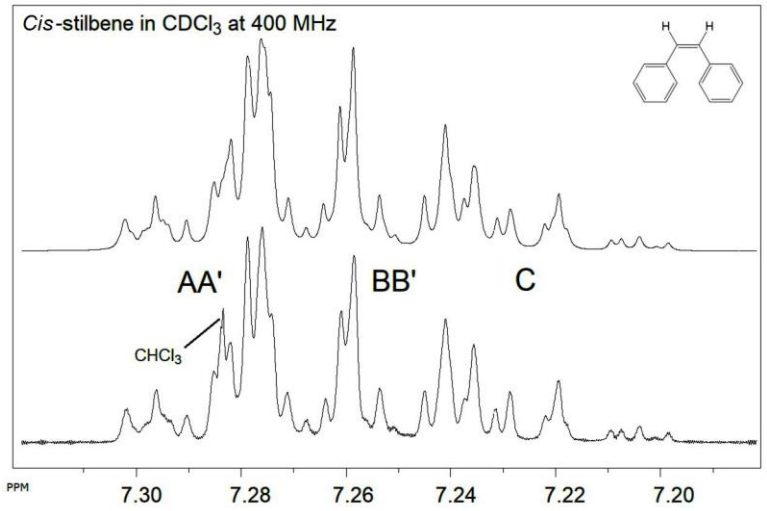


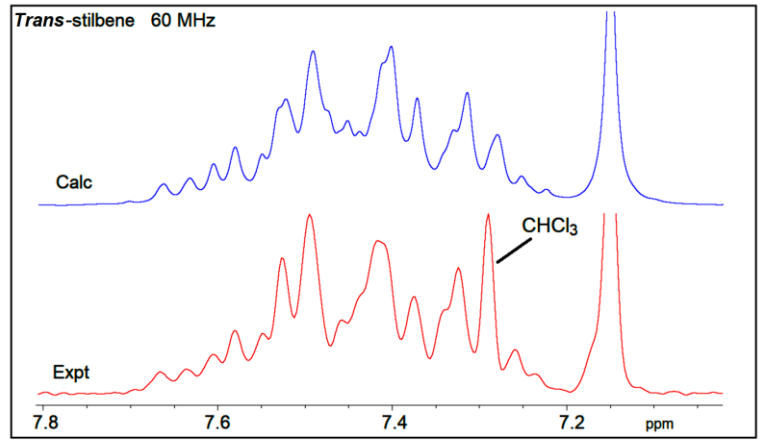


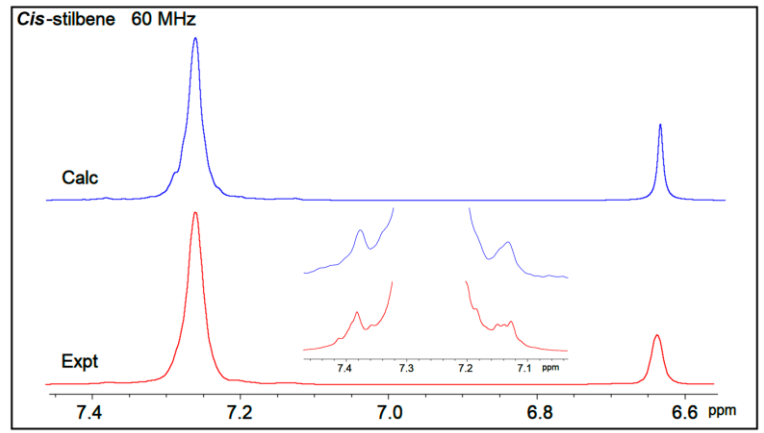


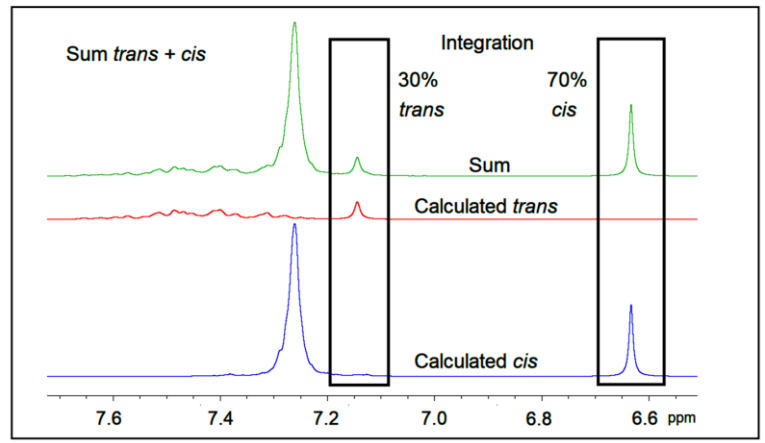
Edgar, M.; Zeinali, F.; Mojally, M.; Hughes, C.; Riaz, S.; Weaver, G. W., *J. Fluorine Chemistry*, 224, **2019**, p35-44.

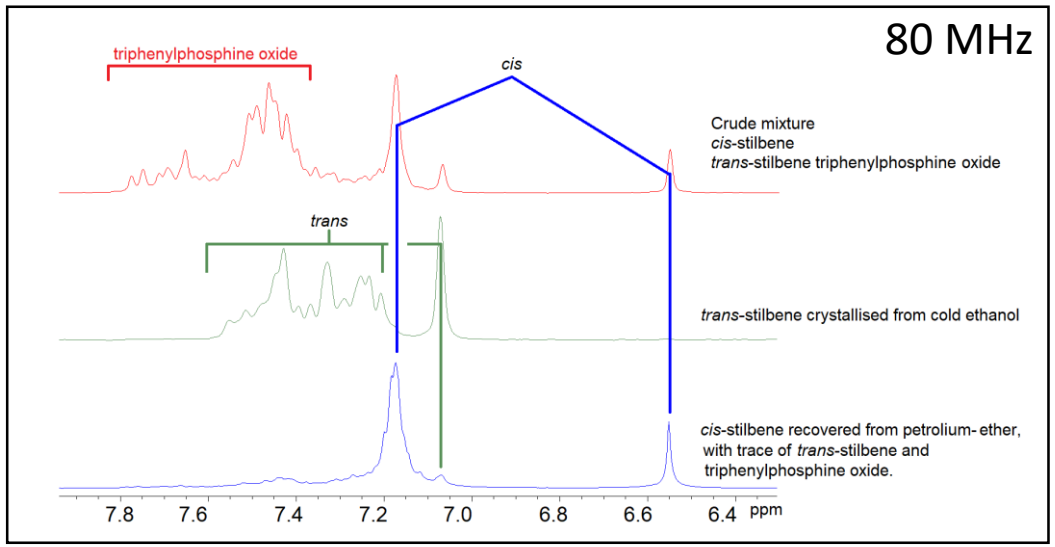

Trans-stilbene in $CDCl_3$ at 400 MHz
AA'
BB'
C
$CHCl_3$
PPM 7.56 7.52 7.48 7.44 7.40 7.36 7.32 7.28

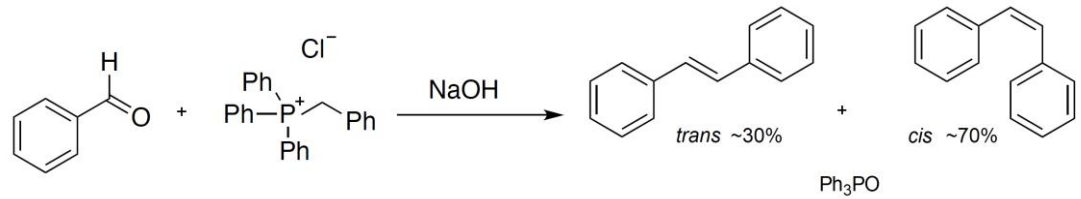

Cis-stilbene in $CDCl_3$ at 400 MHz
AA'
BB'
C
$CHCl_3$
PPM 7.30 7.28 7.26 7.24 7.22 7.20


Trans-stilbene 60 MHz
Calc
Expt
$CHCl_3$
7.8 7.6 7.4 7.2 ppm


Cis-stilbene 60 MHz
Calc
Expt
7.4 7.2 7.0 6.8 6.6 ppm


Sum *trans* + *cis*
Integration
30% *trans*
70% *cis*
Sum
Calculated *trans*
Calculated *cis*
7.6 7.4 7.2 7.0 6.8 6.6 ppm


80 MHz
triphenylphosphine oxide
cis
trans
Crude mixture
cis-stilbene
trans-stilbene triphenylphosphine oxide
trans-stilbene crystallised from cold ethanol
cis-stilbene recovered from petrolium-ether, with trace of *trans*-stilbene and triphenylphosphine oxide.
7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 ppm


H
O
Cl⁻
Ph
Ph—P⁺—Ph
Ph
NaOH
trans ~30%
cis ~70%
Ph_3PO