

Quantitative NMR Tutorial

Commands: [nmrq], [nmrquant]

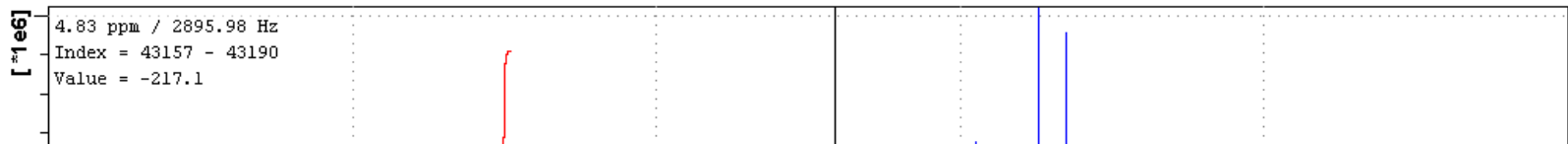


We can quantify three different scenarios:

- We know the molecular weight and the proton distribution of the integral ranges.
→ We can calculate mass content.
- We know the molecular weight, the proton distribution and the amount of substance.
→ We can calculate relative amount of substances.
- We know the molecular weight, the proton distribution and the amount of substance and we know the purity of a reference compound.
→ We can calculate relative amount and purity of substances.

Quantitative NMR [nmrq], [nmrquant]

Scenario 1a: one integral per component



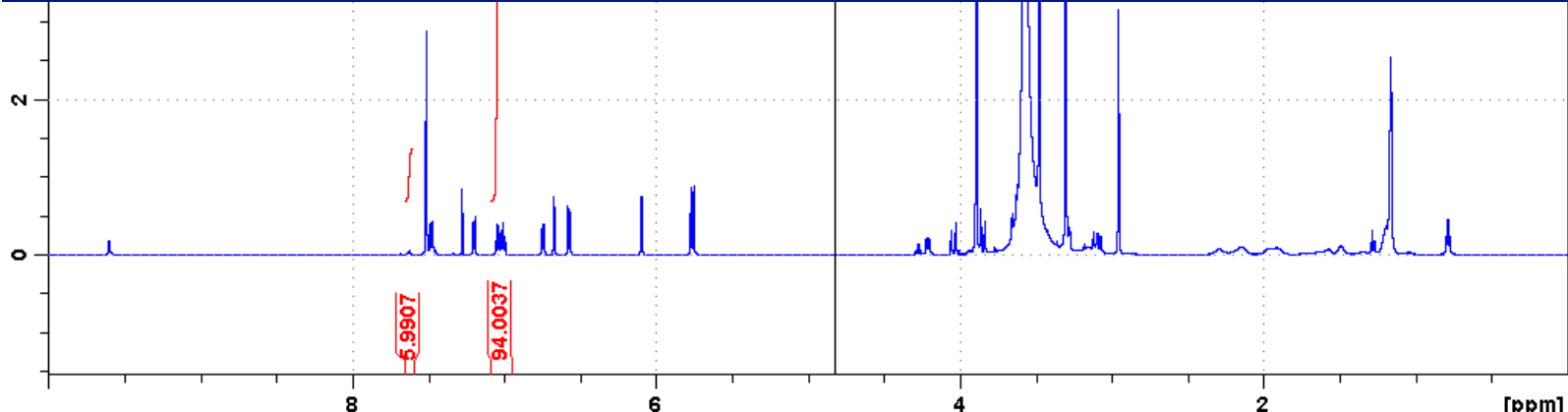
NMR Quantitative Analysis

Matrix

Enter proton contribution for samples in integral region, Enter additional information for components

	5.9907	94.0037	Molecular Wt /g	Component Wt /mg	Purity of Internal Standard	
Probe X	0	2	382.42	0.0		<input type="checkbox"/>
Toluol	5	0	92.14	0.0	1.0	<input type="checkbox"/>

OK Cancel





Quantitative NMR [nmrq], [nmrquant]

Scenario 1a: one integral per component

```
8
9
10 Component      Mass      Mole      Integral  Number  Mw
11              % (w/w)   %          of H      of H    (g/mol)
12 -----
13 Probe X        99.39     97.5142   94.0037   2       382.4200
14 Toluol         0.61      2.4858    5.9907    5       92.1400
15
16 'Probe X' has a mass content of 99.39 % (w/w)
17 'Toluol ' has a mass content of 0.61 % (w/w)
18
19 The contributions of all components have been added up to 100 % (w/w)
20
21 No   Low   High   Slope  Bias   Integral  Obs-Calc  Number
22                               of H
23 -----
24 1    7.655  7.598  0.000  0.000  5.9907    0.000     5
25 2    7.094  6.946  0.000  0.000  94.0037   0.000     2
26
```

Quantitative NMR [nmrq], [nmrquant]

Scenario 1b: several integrals per component



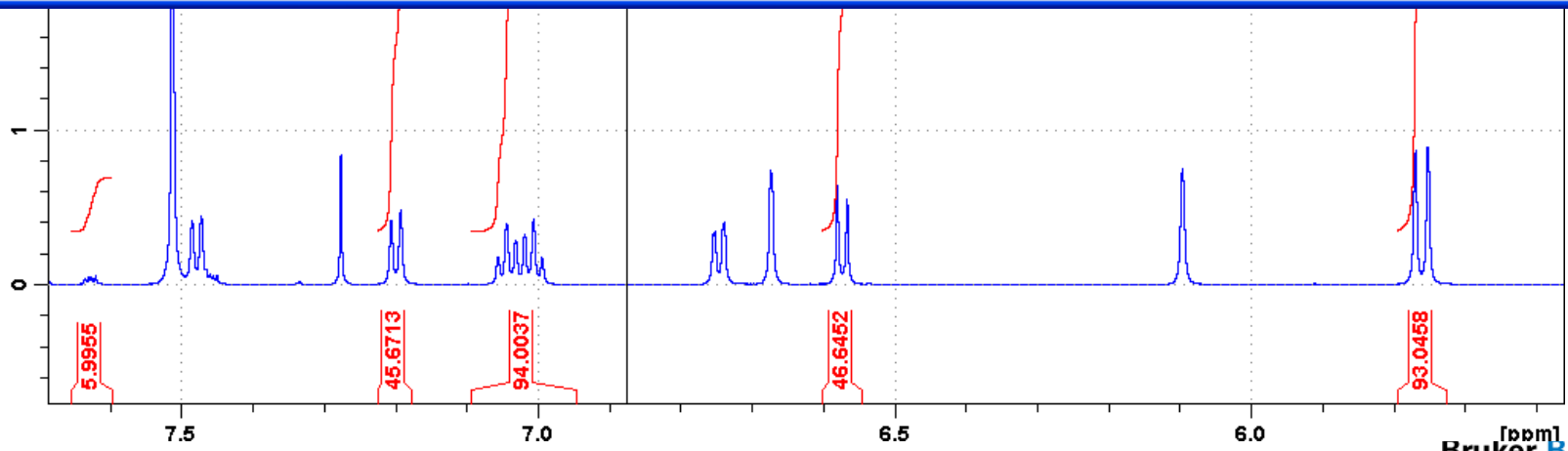
NMR Quantitative Analysis

Matrix:

Enter proton contribution for samples in integral region, Enter additional information for components

	5.9907	45.6713	94.0037	46.6452	93.0458	Molecular Wt /g	Component Wt /mg	Purity of Internal Standard
Probe X	0	1	2	1	2	382.42	0.0	
Toluol	5	0	0	0	0	92.14	0.0	1.0

OK Cancel



Quantitative NMR [nmrq], [nmrqquant]

Scenario 1b: several integrals per component

Component	Mass % (w/w)	Mole %	Integral	Number of H	Mw (g/mol)
Probe X	99.38	97.4955	279.3660	6	382.4200
Toluol	0.62	2.5045	5.9907	5	92.1400

'Probe X' has a mass content of 99.38 % (w/w)

'Toluol' has a mass content of 0.62 % (w/w)

The contributions of all components have been added up to 100 % (w/w)

No	Low	High	Slope	Bias	Integral	Obs-Calc	Number of H
1	7.655	7.598	0.000	0.000	5.9907	0.000	5
2	7.225	7.178	0.000	0.000	45.6713	0.970	1
3	7.094	6.946	0.000	0.000	94.0037	-0.721	2
4	6.602	6.546	0.000	0.000	46.6452	-0.004	1
5	5.795	5.726	0.000	0.000	93.0458	0.237	2

Quantitative NMR [nmrq], [nmrquant]

Scenario 2a: components plus weights



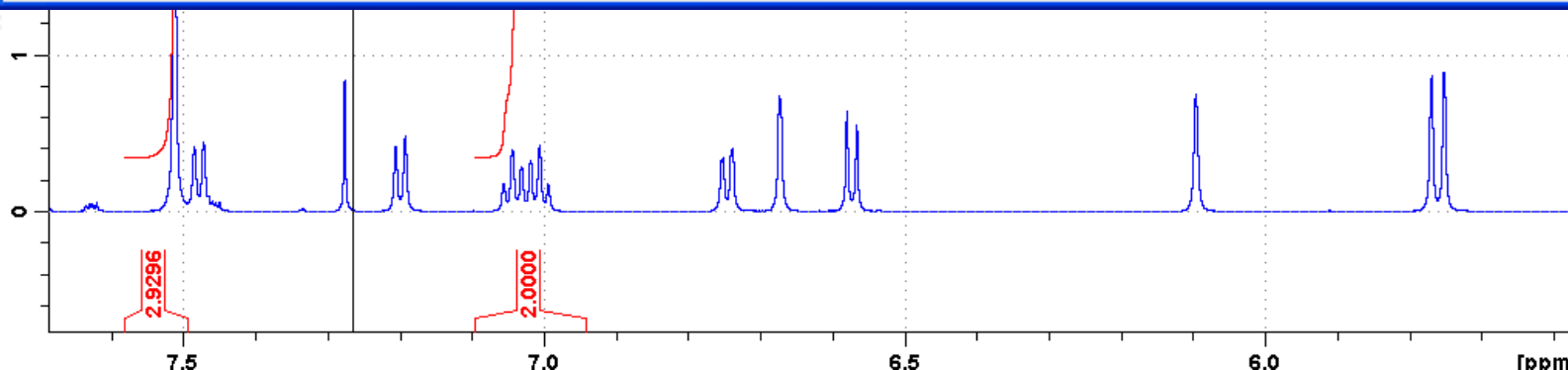
NMR Quantitative Analysis

Matrix

Enter proton contribution for samples in integral region, Enter additional information for components

	2.9296	2.0	Molecular Wt /g	Component Wt /mg	Purity of Internal Standard	
Probe X	0	2	382.42	99.3		X
Koffein	1	0	194.19	14.4	1.0	X

OK Cancel



Quantitative NMR [nmrq], [nmrquant]

Scenario 2a: components plus weights

9

10 The purity of the Internal Standard (Koffein) is 100.0 % (w/w)

11

12 Component	13 Assay % (w/w)	Integral	Number of H	Mw (g/mol)	Component mass (mg)
15 Probe X	9.748	2.0000	2	382.4200	99.3000
16 Koffein	100.000	2.9296	1	194.1900	14.4000

17

18 'Probe X' compared to 'Koffein' has a purity of 9.748 % (w/w)

19

21 No	Low	High	Slope	Bias	Integral	Obs-Calc	Number of H
24 1	7.581	7.494	0.000	0.000	2.9296	0.000	1
25 2	7.097	6.943	0.000	0.000	2.0000	0.000	2

26

Quantitative NMR [nmrq], [nmrquant]

Scenario 2b: components plus weights

6.702 ppm / 4022.315 Hz
 Index = 37017 - 37024
 Value = 3830

5

NMR Quantitative Analysis

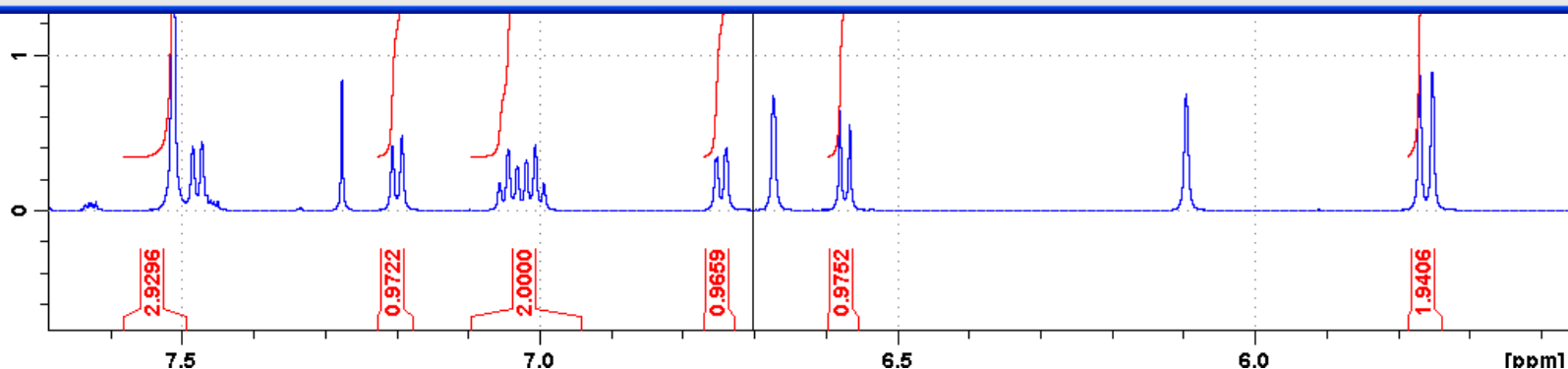


Matrix

Enter proton contribution for samples in integral region, Enter additional information for components

	2.9296	0.9722	2.0	0.9659	0.9752	1.9406	Molecular Wt /g	Component Wt /mg	Purity of Internal Standard
Probe X	0	1	2	1	1	2	382.42	14.4	
Koffein	1	0	0	0	0	0	194.19	99.3	1.0

OK Cancel



Quantitative NMR [nmrq], [nmrqquant]

Scenario 2b: components plus weights

9

10 The purity of the Internal Standard (Koffein) is 100.0 % (w/w)

11

12 Component Assay Integral Number Mw Component
 13 % (w/w) of H (g/mol) mass (mg)

14

15	Probe X	9.477	0.9722	1	382.420	99.3000
16	Probe X	9.748	2.0000	2	382.420	99.3000
17	Probe X	9.415	0.9659	1	382.420	99.3000
18	Probe X	9.506	0.9752	1	382.420	99.3000
19	Probe X	9.458	1.9406	2	382.420	99.3000
20	Koffein	100.000	2.9296	1	194.190	14.4000

21

22 The purity of 'Probe X' is 9.521 % (w/w), SD= 0.13 % (w/w), RSD= 1.38 %

23

24

25 No Low High Slope Bias Integral Obs-Calc Number
 26 of H

27

28	1	7.581	7.494	0.000	0.000	2.9296	0.000	1
29	2	7.227	7.178	0.000	0.000	0.9722	0.009	1
30	3	7.097	6.943	0.000	0.000	2.0000	-0.037	2
31	4	6.771	6.728	0.000	0.000	0.9659	0.015	1
32	5	6.598	6.555	0.000	0.000	0.9752	0.006	1
33	6	5.786	5.739	0.000	0.000	1.9406	0.022	2

Quantitative NMR [nmrq], [nmrquant]

Scenario 3: components plus weights



[1e6] 3.349 ppm / 1674.715 Hz
 Index = 39977 - 40009
 Value = -1067

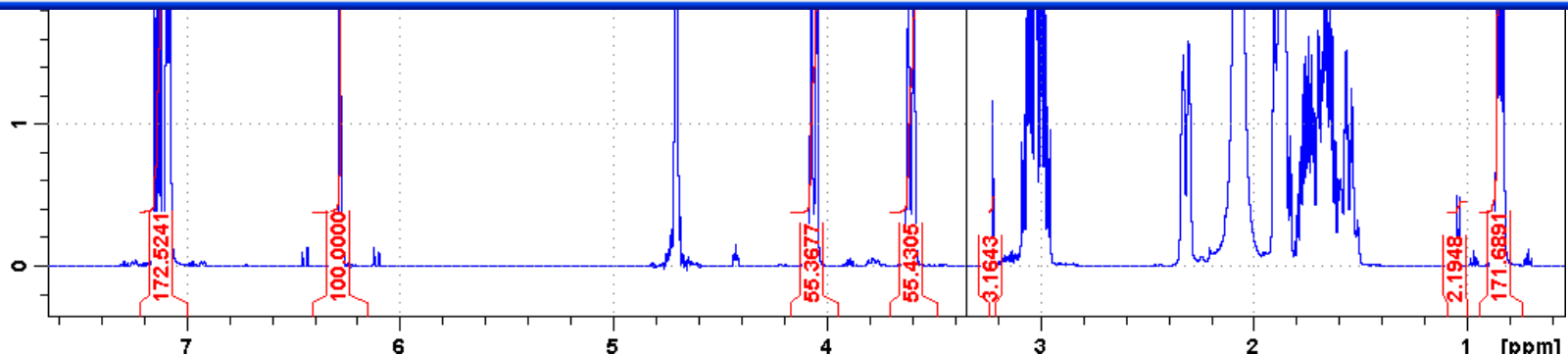
NMR Quantitative Analysis

Matrix:

Enter proton contribution for samples in integral region, Enter additional information for components

	0.1766	172.5241	100.0	55.3677	55.4305	3.1643	2.1948	171.6891	Molecular Wt /g	Component Wt /mg	Purity of Internal Standard	
probe X	0	3	0	1	1	0	0	3	328.88	11.3738		X
Isopropanol	0	0	0	0	0	0	6	0	60.1	11.3738		X
Methanol	0	0	0	0	0	3	0	0	32.04	11.3738		X
Internal Standard	0	0	2	0	0	0	0	0	116.07	3.4879	1.0	X

OK Cancel



Quantitative NMR [nmrq], [nmrquant]

Scenario 3: components plus weights

```

10
11 The purity of the Internal Standard is 100.0 % (w/w)
12
13 Component          Assay          Integral      Number      Mw           Component
14                   % (w/w)              of H          (g/mol)      mass (mg)
15 -----
16 probe X            99.939         172.5241      3           328.880      11.3738
17 probe X            96.219         55.3677       1           328.880      11.3738
18 probe X            96.328         55.4305       1           328.880      11.3738
19 probe X            99.455         171.6891      3           328.880      11.3738
20 Isopropanol        0.116          2.1948        6            60.100       11.3738
21 Methanol           0.179          3.1643        3            32.040       11.3738
22 Internal Standard 100.000        100.0000      2            116.070       3.4879
23
24 The purity of 'probe X' is          97.985 % (w/w) , SD= 1.99 % (w/w) , RSD= 2.03 %
25 The purity of 'Isopropanol' is      0.116 % (w/w)
26 The purity of 'Methanol' is         0.179 % (w/w)
27


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Quantitative NMR [nmrq], [nmrquant] in Automation



The toolbar contains 2 additional buttons:



 Implements the command "Calculate tabulated data and show report". It runs a quantitative analysis using tabulated data, stored in nmrquant.txt (format see below). The result is being displayed in the system notepad and stored in a quant file. The internal format of the tabulated data file is in Appendix A.

 Implements the "Save Automated calculation file" button: Enables storing of the tabulated data in an arbitrary file. The file contents can be calculated using the kernel nmrquant application by means of the "nmrqa" command.

nmrqa command syntax

nmrqa – the command without arguments performs the quantitative analysis within the opened data set using default file name "nmrquant.txt" as a source of the tabulated data and "quant" as an output file.

nmrqa "input_filename" <"output_filename"> - performs the quantitative analysis within the opened data set using input_filename as a source of the tabulated data and output_filename or "quant" as an output file.

nmrqa "directory_name" – the command performs the quantitative analysis from any location. The directory should contain default file "nmrquant.txt" as a source of the tabulated data and "quant" file will be created as an output file.

nmrqa "input_path_name" <"output_pathname"> – the command performs the quantitative analysis from any location. The directory should contain file input_path_name as a source of the tabulated data and output_pathname or "quant" file will be created as an output file.

nmrqa command generates prompts, if any necessary file absent or syntax is wrong, but it doesn't hangs an application. All the syntax errors within the input data file are being stored in the output file.

A sample of the file content is shown below.

It is similar to internal nmrquant data format, so the user can create draft the nmrquant file using the internal component matrix editor.

The file may contain several tables, delimited with START and END lines.

The first value is a number of integrals. The next one is a list of the integral values, delimited with ":" symbols.

The next integer value defines the number of components. The component description has a following format:

"Title" : "Molecular weight" : "Component weight".

The undefined values should be equal to 0.0.

The component table followed by proton array, which define contribution to a corresponding integral.

The line number corresponds to a sample number (in a sample the 1:0:1... line describes the proton contribution of the sample 1 and the 0:1:0... line describes the proton contribution of the sample 2), the number of array's elements should be equal to a number of integrals. "Foreign's integrals" should be filled with the zero number of protons, like in component table of the nmrquant application.

If the component description

block contains an error, the corresponding warning appears in a report and does not stop calculations on other blocks.

Nmrquant.txt file sample



START

12

19.3481:1.14:19.53:19.4955:1.1168:1.2597:3.6792:40.75:60.72750:39:39:58.88

2

sample 1:139.22:10

sample 2:126.11:1

1:0:1:1:0:0:0:2:3:2:2:3

0:1:0:0:1:1:2:0:0:0:0:0

END

START

10

19.3481:1.14:19.53:19.4955:1.1168:1.2597:3.6792:40.75:60.72750

2

Sample 3:139.22:0

Sample 4:126.11:0

1:0:1:1:0:0:0:2:3:2

0:1:0:0:1:1:2:0:0:0

END