

ARTISAN®
TECHNOLOGY GROUP

Your **definitive** source for quality pre-owned equipment.

Artisan Technology Group
(217) 352-9330 | sales@artisantg.com | artisantg.com

Full-service, independent repair center
with experienced engineers and technicians on staff.

We buy your excess, underutilized, and idle equipment
along with credit for buybacks and trade-ins.

Custom engineering
so your equipment works exactly as you specify.

- Critical and expedited services
- In stock / Ready-to-ship
- Leasing / Rentals / Demos
- ITAR-certified secure asset solutions

Expert team | Trust guarantee | 100% satisfaction

All trademarks, brand names, and brands appearing herein are the property of their respective owners.

Find the *Oxford Instruments / Resonance Instruments Maran Ultra* at our website: *Click HERE*

RI CALIBRATION AND RI ANALYSIS

USERS MANUAL

RI Calibration Manual v 4.1 23/10/98 (software release version 1.3.1)

Windows 95 is a registered trademark of Microsoft Inc.
Turbo Pascal is a registered trademark of Borland International Inc.

Copyright Resonance Instruments Ltd 1998.
This manual may be reproduced with permission.

Comments, errors and omissions should be reported to :

Product Support,
Resonance Instruments Ltd,
Unit 13, Thorney Leys Business Park,
Witney,
Oxfordshire,
OX8-7GE.
United Kingdom.
Tel. +44 (0) 1993 700442.
Fax. +44 (0) 1993 700363.

E-mail. support@resonance.co.uk

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

CONTENTS

CONTENTS 2

1. INTRODUCTION 4

1.0 Introduction 4
1.1 How to Use This Manual 4
1.2 New Features in RI Calibration 4
1.3 System Requirements 5
1.4 Calibration Concepts 5
1.4.1 Why Calibrate ? 5
1.4.2 Sets of Calibration Standards 6
1.4.3 NMR Data, Calculation Ranges and the Effect of Sample Mass 7
1.4.4 Calibration Summary 9
1.4.5 Restandardisation 9
1.4.6 Common Questions Regarding Calibrations 9

2. OVERVIEW 11

2.0 Introduction 11
2.1 RI Calibration Windows 11
2.1.1 Window Manipulation 11
2.1.2 The RI Calibration Main Window 11
2.1.3 Using the Spreadsheet 12
2.1.4 Calibration Graph Window 13
2.1.5 NMR Data Window 13
2.2 Menus 14
2.2.0 File 14
2.2.1 Edit 15
2.2.2 Page 15
2.2.3 Options 15
2.2.4 Window 15
2.2.5 Help 15
2.3 ToolBar Icons 15
2.4 Importing NMR Data 15
2.5 Concentrations, Masses and Data Type Specification 16
2.6 Viewing Graphs 16
2.7 The NMR Data Window 17
2.7.1 Calculation Range Specification 17
2.7.2 Calculation Range Options 17
2.7.3 Unzoom and Recalc 18
2.8 Specifying the Signal Function 18
2.9 Checking the Calibration 18

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.10 Generating the Calibration File 19
2.10.1 User Options 20
2.10.2 Data Format 20
2.10.3 Output Options 20
2.10.4 Result Text 21
2.10.5 Auto Tune Definition 21
2.10.6 Information Fields 21
2.10.7 Creating the Calibration File 21

3. QUICKSTART TUTORIAL 22

3.0 Introduction 22
3.1 Producing a Calibration 22
3.2 The Example Data Set 22
3.3 Loading the Data and Specifying Sample Details 22
3.4 Specifying the Calculation Ranges 23
3.5 Inspecting the Calibration Graph and Modifying the Data Ranges 24
3.6 Generating the Calibration File for Use With RI Analysis 24

4. APPENDIX 25

4.0 Statistical Definitions 25
4.1 Keyboard Short Cuts 25
4.2 Contact Addresses 25

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1. INTRODUCTION

1.0 Introduction

RI Calibration is Resonance Instruments Windows 95 NMR calibration software for the MARAN and MARAN Ultra instruments. RI Calibration enables users to generate calibrations from calibration standards of known concentrations to enable the routine measurement of unknown samples to take place via RI Analysis.

RI Calibration is designed to be intuitive to use by non expert NMR users, but assumes some user familiarity with Windows 95 and basic statistical concepts, including curve fitting (linear regression). Calibrations produced by RI Calibration may be used in conjunction with RI Analysis by unskilled operators to perform routine measurements on unknown samples.

Users wishing to perform routine measurements using RI Analysis should refer to the RI Analysis Manual for lab personnel.

1.1 How to Use This Manual

RI Calibration has may similarities with the MARAN MS-DOS calibration software described in the MARAN software manual. Users familiar with the original MS-DOS software and calibration concepts may wish to proceed directly to Chapter 3, where a step by step tutorial describes how to create calibrations from a demonstration data set (this set is included on disk with the RI Calibration software).

Users unfamiliar with creating NMR calibrations should first read section 1.3, Calibration Concepts, to familiarise themselves with standard calibration procedures, then proceed to Chapter 2 for a software overview and reference before proceeding to Chapter 3.

1.2 New Features in RI Calibration

RI Calibration embodies many of the concepts introduced in the original MARAN MS-DOS software while offering the ease of use of the Windows 95 environment coupled with significantly increased flexibility. The main change between RI Calibration and the standard MARAN MS-DOS software lies in the way data are handled. In the MS-DOS software, data are acquired within the calibration software. Once the data are acquired they are processed and reduced to a single number which is then used in any subsequent calculations.

RI Calibration retains all the NMR data so that calculation ranges and processing options can be altered without the need for re-acquiring data, which leads a considerable amount of time saving. Re-calculation of the calibration curve following the alteration of calculation ranges is done in real time, allowing the user to optimise the processing parameters with the minimum of effort.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.3 System Requirements

RI Calibration requires the following minimum PC specification to run :

Pentium PC - 75 MHz or better.
16 megabytes of RAM.
10 megabytes of free hard drive space.
Windows 95 operating system.

Users should note that RI Calibration is a stand alone software package and does not require RI NMR or RI Analysis to be present in order to operate. Further copies of RI Calibration may be purchased by contacting Resonance Instruments or your local distributor.

1.4 Calibration Concepts

1.4.1 Why Calibrate ?

NMR is not an absolute measurement technique, i.e one cannot simply place a sample in an NMR analyser and obtain a result for % oil content in seeds, % spin finish or any other quantity.
To perform an NMR analysis, a set of calibration standards (which have a known concentration of the property we are interested in, this may be % oil in seeds, %FOF spin finish, % degree of crystallinity etc.) must first be measured. A record or calibration is generated from the calibration standards. Users may then measure unknown samples and compare them to the calibration produced from the calibration standards.

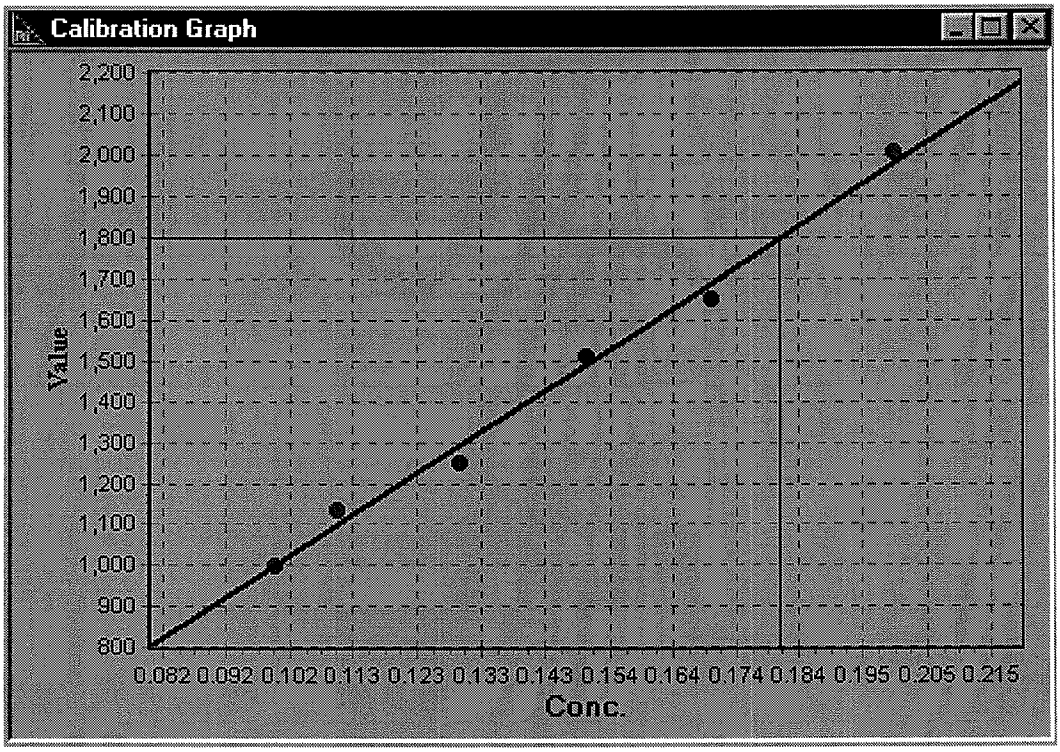
For example, an NMR experiment may yield the following values for 10 known values of spin finish calibration standards :

Concentration (% spin finish)	NMR Value
0.1	998
0.11	1130
0.13	1250
0.15	1510
0.17	1650
0.20	2003

If the NMR values are plotted against the % concentration of the calibration standards, the following graph is produced :

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



The data points (marked with the circles) are fitted using linear regression to produce a calibration fit (solid line). The fit may be a straight line (as in most examples, including the above) or a curve (polynomial calibrations).

Unknown samples may then be measured by establishing where they lie on the fit. In the above graph, for example, an unknown sample producing an NMR value of 1800 would correspond to a concentration of approximately 0.182 %.

1.4.2 Sets of Calibration Standards

A good calibration set should have the following properties :

i) Should ONLY vary in the property associated with the concentration (for example in spin finish measurements only % spin finish, NOT for example textile type. If % spin finish measurement of two different textile types is required, ideally a separate calibration must be performed for each textile).

ii) Should vary in near equal increments between the largest and smallest values of concentrations in unknown samples usually measured.

iii) Should contain duplicates for testing reproducibility and repeatability.

Finally, it is important to note that all calibration standards (and any subsequent measurement of unknown samples) MUST be measured at identical temperatures. The amplitude of a particular samples NMR signal is highly dependent on the sample temperature, variations in the temperature

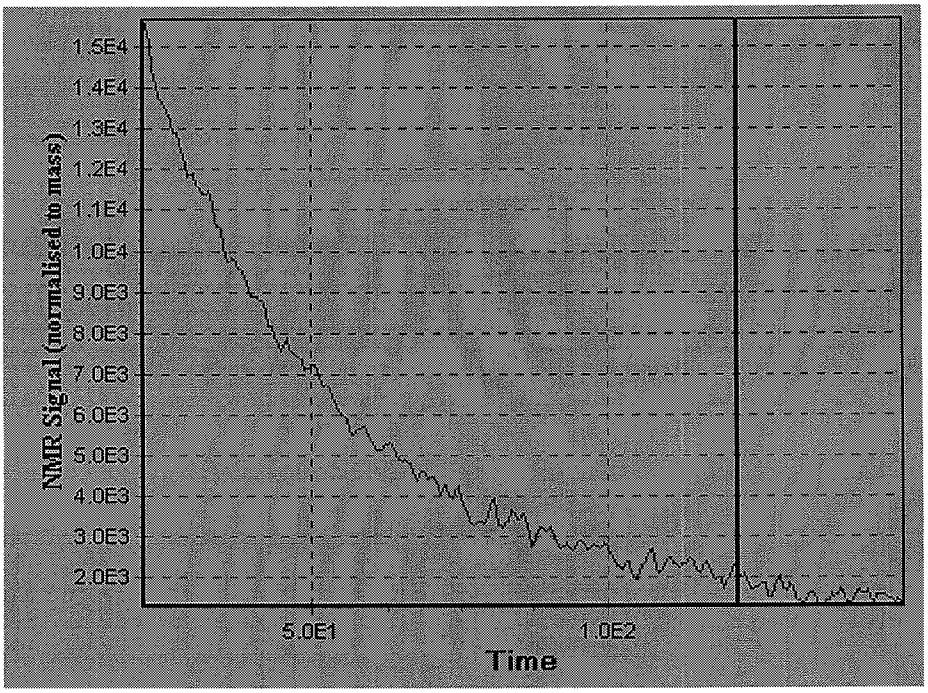
Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

at which calibration standards are measured can lead to significantly increased standard deviations in calibration statistics.

1.4.3 NMR Data, Calculation Ranges and the Effect of Sample Mass

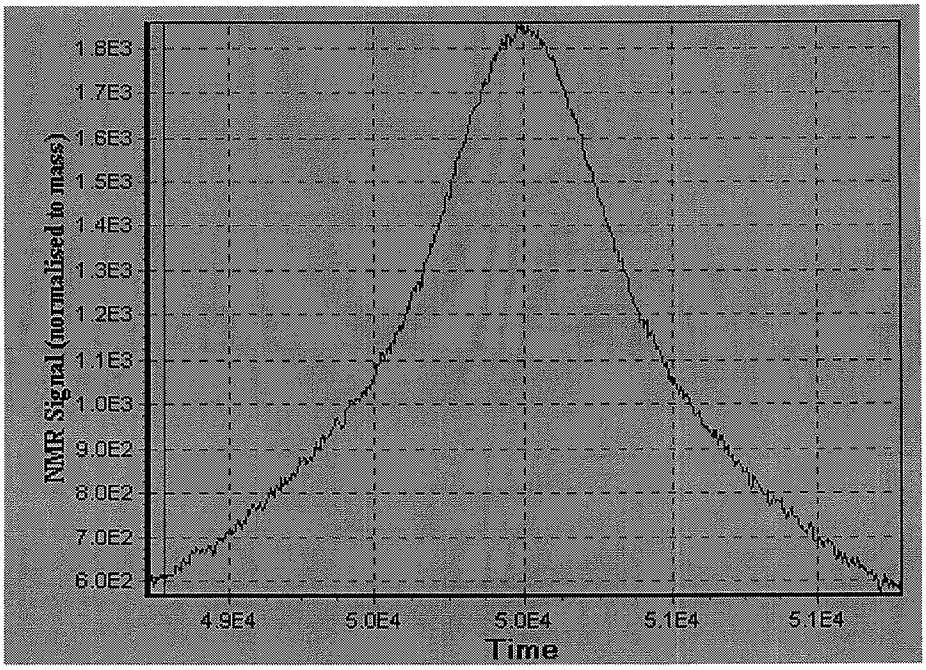
NMR data is acquired in the time following an RF pulse. It is not necessary to discuss how the excitation occurs, or why the data appears as it does (see NMR For Non-Expert Users if more information is required) but sufficient to say that following the excitation the NMR signal amplitude varies as a function of time. A typical NMR signal may look something like this :



Where the signal decreasing as a function of time represents the NMR signal. Another type of NMR signal looks like this :

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



where the signal increases to a maximum value when the time is 50000, then decreases again.

Some areas of the NMR data contain information that we are interested in. Other areas do not. In the first example of NMR data shown above, we are not interested in data from over 125 microseconds following the start of the acquisition (time zero) as this contains only noise. Only the data from approximately 0 microseconds to 125 microseconds contains valuable information which we may be able to correlate to the calibration standards concentration.

Once NMR data has been acquired, a sensitive region or calculation range must be selected for use in the calibration. For example, it may be that the data from 50 to 100 us in the above diagram can be averaged and then correlated with the % spin finish present in the sample. The position of the calculation range necessary to produce a good calibration varies from application to application, and may be found either by experience, by trial and error (by varying the values at which the calculation range starts and finishes and watching the effect on the quality of the calibration) or by referring to information supplied by Resonance Instruments.

Finally, the amplitude of the NMR signal following the excitation pulse is also proportional to the sample mass. For example a if a sample of mass 1 g produces a NMR signal of amplitude 100 at 50 us, a sample of mass 2 g will produce an NMR signal of amplitude 200. Most calibration values must be divided by the sample mass or 'normalised for mass' before the calibration takes place.

Users should note that mass normalisation can take place via the NMR signal, removing the need to weigh the samples. However this sometimes leads to an increase in standard deviation in the results.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.4.4 Calibration Summary

The creation of a calibration may be summarised as follows :

i) Acquire NMR data from a set of samples which have known values (or concentrations) of the property that we are interested in.

ii) Divide this data by the samples mass.

iii) Define a calculation range of the NMR data which relates to the concentration.

iii) Average the data in the calculation range (in some manner) to produce a single data value.

iv) Correlate this value with the concentrations of the calibration standards.

v) Perform a curve fit (usually with least squares minimisation) to produce a calibration fit.

The fit can then be used to measure unknown samples.

1.4.5 Restandardisation

Re-standardisation is not implemented in the current version of RI Calibration.

1.4.6 Common Questions Regarding Calibrations

How Often Should I Perform a Calibration ?

Initially, calibrations should be performed on a routine basis to check instrument stability. Users may wish to perform calibrations once per week to convince themselves of the stability of the calibration with time. From then onwards, the user may wish to reduce the frequency of the calibration once confidence has been established to match local guidelines. Many calibrations show little drift even after several years of use.

How Many Calibration Standards Should I Use in a Calibration ?

The chance that a calibration fit may be weighted by an unsuitable sample is dramatically increased if a small number of calibration standards is measured. However, increasing the number of calibrations standards above a certain level gives little gain in accuracy (in terms of statistical confidence) to the calibration. Users should use their discretion or refer to local guidelines for establishing how many calibration standards are necessary.

Which Parameters Have the Greatest Effect on the Reproducibility of a Calibration ?

Variations in sample temperature have been found to produce the greatest variation in reproducibility for NMR measurements. To ensure that accuracy is maintained, all samples (both while the calibration is taking place and during the measurement of unknown samples) must be measured at the same temperature that the calibration was performed at.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Variations in sample composition can also affect the quality of calibrations. Some changes in sample composition have little or no effect on the calibration quality. Others, however, may invalidate the calibration completely. It is difficult (even for NMR experts) to predict exactly what changes in sample composition are necessary to invalidate a particular calibration. For example, a calibration to measure the % plasticizer in PVC may no longer be valid when the plasticizer type is changed. Users are advised re-calibrate whenever significant changes in sample composition take place.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2. OVERVIEW

2.0 Introduction

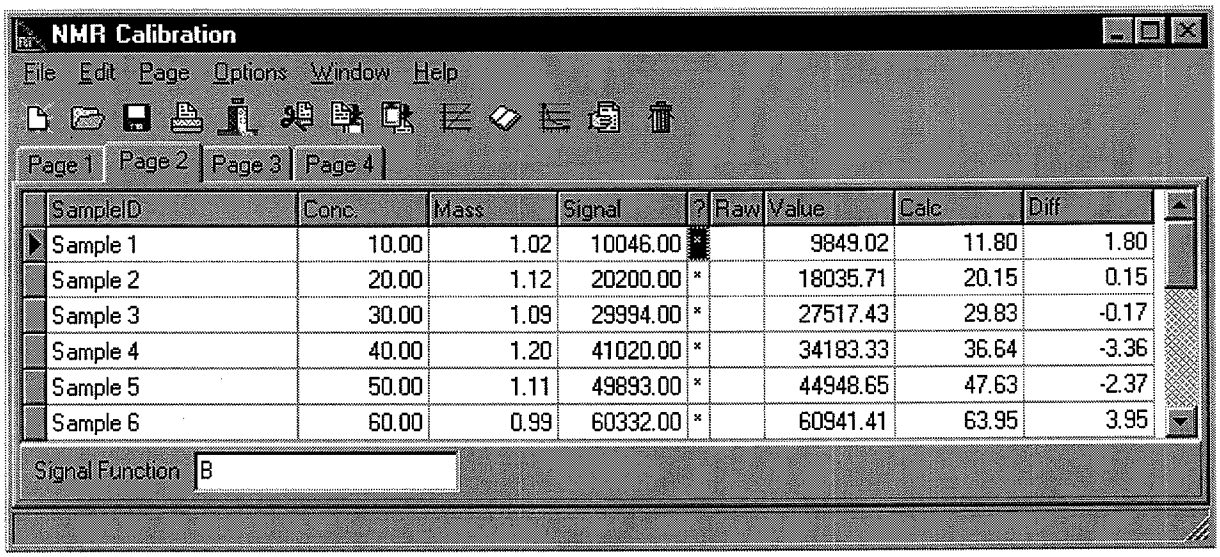
This Chapter provides an overview of RI Calibration and describes the main features of the software.

2.1 RI Calibration Windows

2.1.1 RI Calibration Window Manipulation

RI Calibration behaves as normal Windows 95 application. The windows may be moved, minimised and restored in the usual Windows 95 manner. Users unfamiliar with Windows 95 should refer to the Microsoft manuals supplied with their PC for further information on routine window manipulation. RI Calibration may be started by selecting Start-Programs-MultiQuant-RI Calibration from the Windows 95 Start menu.

2.1.2 The RI Calibration Main Window



The RI Calibration Main Window allows the user to select menu options, enter the data for the samples and to customise the look of the calibration software.

The menu options are situated towards the top of the screen, the ToolBar lies below them. The spreadsheet, containing the data entry cells, resides in the white box in the middle of the window.

The signal function box at the bottom of the window allows the user to specify how the calculation ranges are combined to produce the final value for the calibration.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.1.3 Using the Spreadsheet

9 columns of cells are provided, each cell requires different information about the sample to be entered. To select a cell in a particular row, left click with the mouse on the required cell. The row will be highlighted in blue, the active cell is surrounded by a yellow border. Rows may be cut, copied and pasted using the edit menu (see section 2.2.1). Note that when the row is pasted it is inserted immediately after the currently highlighted row. The column to the left of the Sample ID also indicates the currently active row with an arrow (the row indicator column). Rows can also be deleted / inserted by right clicking on the row indicator column and selecting the appropriate option. Finally the contents of a cell can be copied and pasted automatically by simply left clicking on the required cell and dragging the mouse to the desired location.

Sample Identification - Enter a name for each sample here. Note that this column defaults to the name of the data file that is attached to the row (see section 2.4).

Conc. - The concentration of the calibration standard.

Mass - Sample mass in grams.

Signal - Value of the signal function for that particular data set (NMR data files may be loaded into the signal cells by right clicking on the appropriate signal cell).

? - Sample selector (use sample in calibration (Y/N).

Raw - Allows the user to specify whether or not the data will appear in the NMR Data Window (Y or N).

Value - Mass normalised value of signal used for the calibration.

Calc - Calculated value of sample concentration (from fitted curve).

Diff - Difference between calculated value of sample concentration and given value of sample concentration.

The names of the columns and the decimal places used for each column may be changed by right clicking on the name of the column. The widths of the columns may be altered by placing the pointer on the dividing line between two columns and then left clicking and dragging the mouse to the desired location.

The Signal Function box in the bottom left hand corner of the Main Calibration Window displays the signal function and allows the user to type in a formula containing up to seven data ranges (A to F). Users should refer to section 2.6 for more information on data ranges.

Users may select one of four spreadsheet pages. Different pages may be selected by left clicking on the page tabs at the top of the spreadsheet. The pages exist to enable the user to create several calibrations from the same data set, each with different calculation ranges or signal functions. When the calibration is generated and used with RI Analysis, the measurement software will

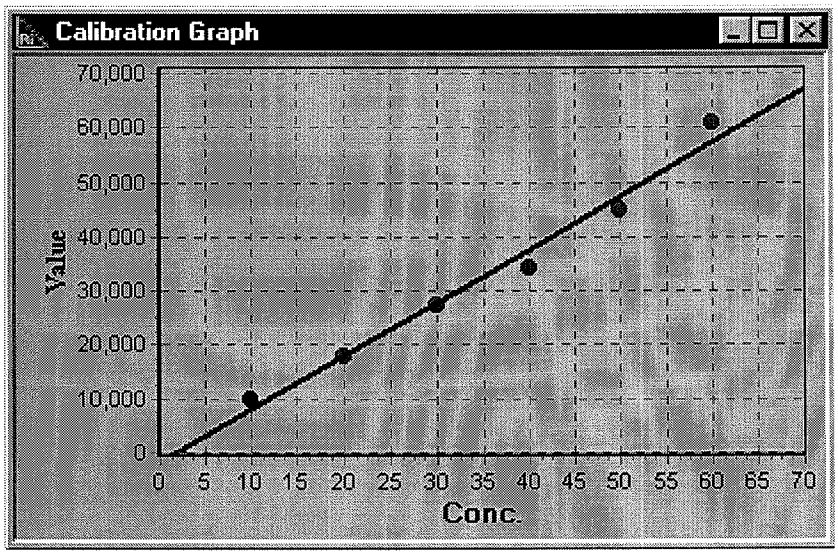
Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

report a value for each spreadsheet page. For example, if a user wishes to create a calibration for measuring oil and water in seeds, the first page can be used to produce the oil content of the seeds, the second page the water content. The page names may be changed by right clicking on the page tab and entering the new name (for example Page 1 could be changed to Oil and Page 2 Water).

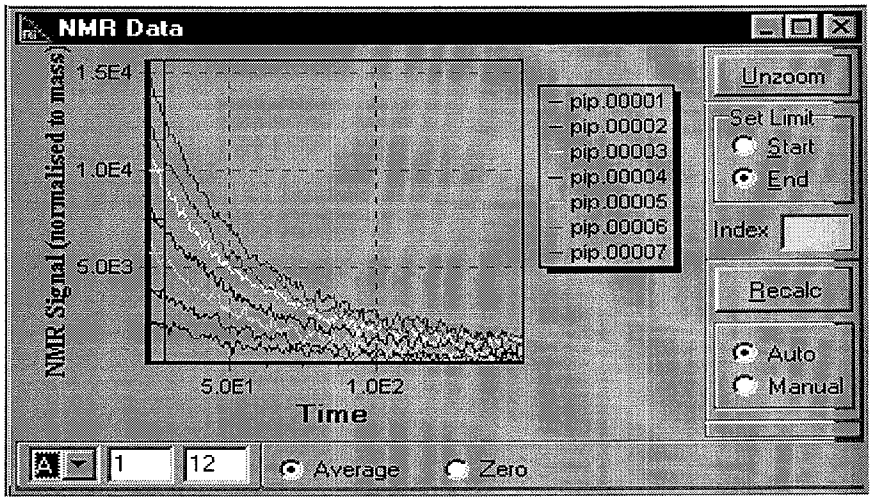
Note that the Calibration Graph Window, the NMR Data Window and the Statistics Window are updated automatically when a new page is selected.

2.1.4 Calibration Graph Window



The Calibration Graph Window displays the current curve fit and data points (Value from the main calibration screen vs conc. from the main calibration screen).

2.1.5 NMR Data Window



The NMR Data Window displays the raw NMR data that has been loaded into the calibration software. The

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

window allows the user to specify data ranges for evaluation and range fitting options. Information of the options available using the NMR Data Window may be found in section 2.5.

2.2 Menus

Six menus may be accessed using the calibration software. Some of the options contained within the menus can be reached by keyboard shortcuts (these are listed in the menus) or via the ToolBar. Click on the relevant icon in the ToolBar to select a menu option. Note that a description of each icons function can be obtained by holding the mouse pointer over the icon.

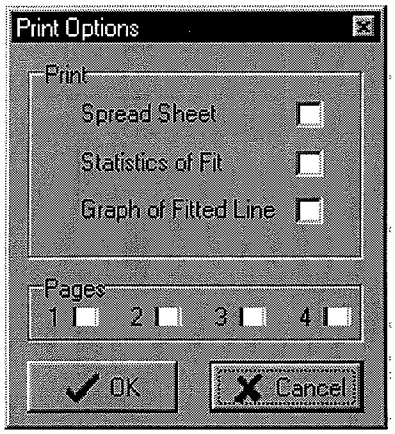
2.2.0 File

The file menu allows users to open and save calibration files, to print the results of a calibration and to generate a calibration file for use with the measurement software. The Exit option allows the user to exit the calibration software. this option may also be performed by the close button in the top right hand corner of the screen in the usual Windows 95 manner.

The New option allows the user to start a new calibration. The user is prompted to save any existing calibration before the new calibration is started.

Open allows the user to open an existing calibration file. Calibration files are saved by default under the filename *.cd in the default calibration directory.

Save allows the user to save a calibration file for use at a later date. Calibration files are saved by default under the filename *.cd in the default calibration directory.



Print allows the user to print out information regarding the calibration. When the print option is selected, several options may be specified. These allow the user to select which windows and also which pages of the calibration are printed.

Generate Calibration generates a calibration from the current data for use with the measurement software (RI Analysis).

The Exit option allows the user to exit the calibration software. this option may also be performed by the close button in the top right hand corner of the screen in the usual Windows 95 manner.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

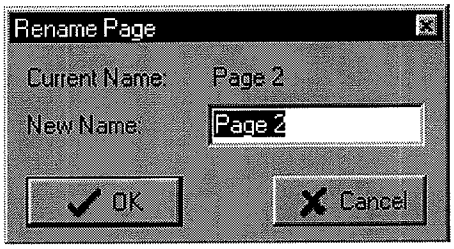
Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.2.1 Edit

This menu allows users to cut, copy and paste data from cells on the data window. To select an area, left click on a cell and drag the mouse in the required direction. A series of cells will be highlighted as the mouse is dragged. After the required cells have been selected, select Cut from the Edit menu to remove the data from the cells and copy it to the clipboard. Select Copy from the edit menu to copy the selected data to the clipboard. Finally, select Paste to re-insert clipboard data into the data entry cells.

2.2.2 Page

Allows the user to rename the current page, or select an active page. Note that the pages may also be selected by clicking on the relevant tabs in the calibration window. Calibration pages may also be renamed by selecting the Rename Calibration option from the page menu.



2.2.3 Options

Allows the user to customise the appearance of the NMR Data Window.

2.2.4 Window

Allows the user to select which of the 3 supplementary windows (Calibration Graph, Statistics and NMR Data) are displayed.

2.2.5 Help

Displays information pertaining to the software version number (which must be quoted in any software enquiries) and addresses for Resonance Instruments and its United States distributor, Universal Systems.

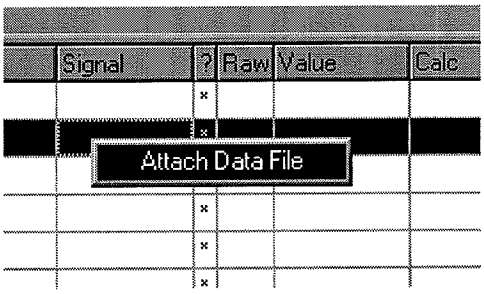
2.3 ToolBar Icons



The ToolBar icons provide shortcuts to the most frequency used menu options. A description of the available shortcuts can be obtained by placing the mouse pointer over each icon in turn.

2.4 Importing NMR Data

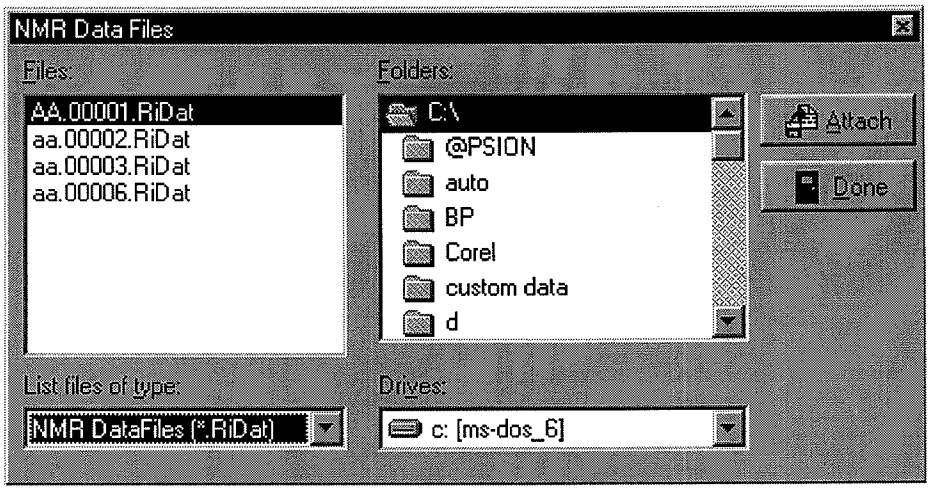
To attach NMR data from an RI NMR file, right click on the sample column of the desired cell. A pop-up box appears which allows the user to attach an RI NMR data file to that particular row of cells. Highlight the 'Attach NMR file' pop-up box and browse the hard drive for the



Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

required file.



Once a file of a given prefix has been loaded further files in the same series can be loaded by simply clicking on the attach button. The file indicator will then increment automatically. If a sample identification has not been entered in the spreadsheet the software will assign the filename of the data file to the Sample ID.

2.5 Concentrations, Masses and Data Type Specification

Once the NMR data files have been attached it is necessary to assign known values (or concentrations) and sometimes masses to the files. To assign concentrations type in the known values of the calibration standards in the Conc. column of the spreadsheet.

In a similar manner the mass of each sample may be typed in the Mass column for mass normalisation.

The Data Type Specification column (?) Informs the program how to handle the data. The data types are as follows :

'*' use the data in the calibration

' ' do not use the data in the calibration (can be used to remove outlying points or bad results).

'Z' use the data to zero the calibration, data cells are highlighted in blue.

'P' use the data to predict the value of the sample from the calibration (for checking 'unknowns') data cells are highlighted in green.

2.6 Viewing Graphs

Once the data has been attached to the cells, bring up the NMR Data Window to view the data. The NMR Data Window contains a plot of the acquired NMR data vs. Time, a list of the .RiDat

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

files loaded, a set of calculation range specification options and other buttons.

The NMR Data Window graph shows the NMR data that has been loaded into the calibration software. The legend to the right of the graph provides a key to the data sets. The data display graph may be manipulated in the usual T-Chart manner :

To zoom an area of the graph, left click on the graph and drag the mouse to the right and down to define a zoom window.

To unzoom the graph left click on the graph and drag the mouse to any position to the left and up from the original point. The graph should return to its original settings.

To move the graph along the x or y axes, right click on the graph and drag the mouse in the desired direction.

Note that in the case of the NMR Data Window, the graph may also be returned to its original settings by left clicking on the unzoom button. Users should note that both the NMR Data Window and the Calibration Graph Window may be manipulated in exactly the same way.

2.7 The NMR Data Window

2.7.1 Calculation Range Specification

Calculation range specification may be performed in a number of different ways. Firstly, click on the arrow in the bottom left hand corner of the data display window to associate a letter with the calculation range. The current start and end points of the data range may be found in the two boxes to the right of the letter.

The beginning and end of the ranges can be specified in two ways :

i) Select 'Start' from the Set Limit box on the left hand side of the NMR data display. Left click anywhere to the LEFT of the green vertical line (this line represents the end of the currently selected data range). The red line (the start of the currently selected data range) will be moved to the value where the mouse pointer was when the left click took place. This process may be repeated for the end of the data range by selecting 'End' from the set limit box and left clicking anywhere to the RIGHT of the red vertical line.

iii) Specify the start and end values for the data range in the two boxes to the right of the calculation range label display. The red and green vertical lines which indicate the beginning and end of the calculation ranges will move to reflect the new values.

2.7.2 Calculation Range Options

One of two calculation options may be specified from each calculation range. The first, average , calculates the average value from all the points in the specified data range and passes it to the main calibration program for calculation. The second option, zero, fits a straight line to the selected data range and extrapolates the line back to time zero. The value of the y-intercept is then

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

passed to the calculation program for calculation.

Once the data ranges have been specified a fit line appears between the start and end limits on the data display graph. The line either reflects the average value of the data between start and end limits, or the straight line that the data has been fitted to.

2.7.3 Unzoom and Recalc

Unzoom, allows the user to reset the NMR Data Window to its original settings. The second function, Recalc, recalculates the calibration based on any new ranges that have been specified. Note that if the Auto radio button is checked, recalculation occurs automatically every time a calculation range is altered or a new calculation range is specified. If the manual radio button is checked, the user needs to left click on the Recalc button before recalculation takes place.

2.8 Specifying the Signal Function

Once the calculation ranges have been specified, the way in which they are combined to form the final value for the calibration is specified in the signal function box which is located in the Main Calibration Window.

Up to 10 calculation ranges may be specified and can be combined with the four operators +,-* and /. The priority of operation is :

()
/ and *
- and +

For example :

A+B

A+(B/D)

1/(A)*A+C/(D-F)+100

are all valid expressions for the signal function.

2.9 Checking the Calibration

Once the signal function has been specified the calibration curve can be viewed in the Calibration Graph Window. This window shows a plot of NMR value (after mass normalisation) vs. Concentration.

The currently selected data point in the main calibration window is highlighted in blue. The curve fit statistics may be viewed in the calibration statistics window. The columns are as follows :

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Type - refers to whether the statistic is for fitted or predicted points '*' or 'P'.

Num Points - the number of points of each type present in the calibration.

Slope - The slope of the line resulting from the linear regression.

Value Intercept - The y-intercept of the line resulting from the linear regression.

Conc. Intercept - The x-intercept of the line resulting from the linear regression.

Correlation Co-efficient - Linear correlation co-efficient (see Appendix A1.0)

Standard Deviation - Standard Deviation σ (see Appendix A1.0).

Users can use the data type column '?' in the spreadsheet to remove outlying or suspect points from the calibration by selecting a ' ' for the data type instead of a '*'. Note that if the data ranges are changed (and the recalc option is set to auto) or the signal function is changed, the effect of the changes on the calibration curve and statistics will be updated automatically. This allows the user to study the effect of varying the data ranges on the calibration and the quality of the results in real time, which significantly facilitates the process of identifying the optimum calculation range for the calibration.

2.10 Generating the Calibration File

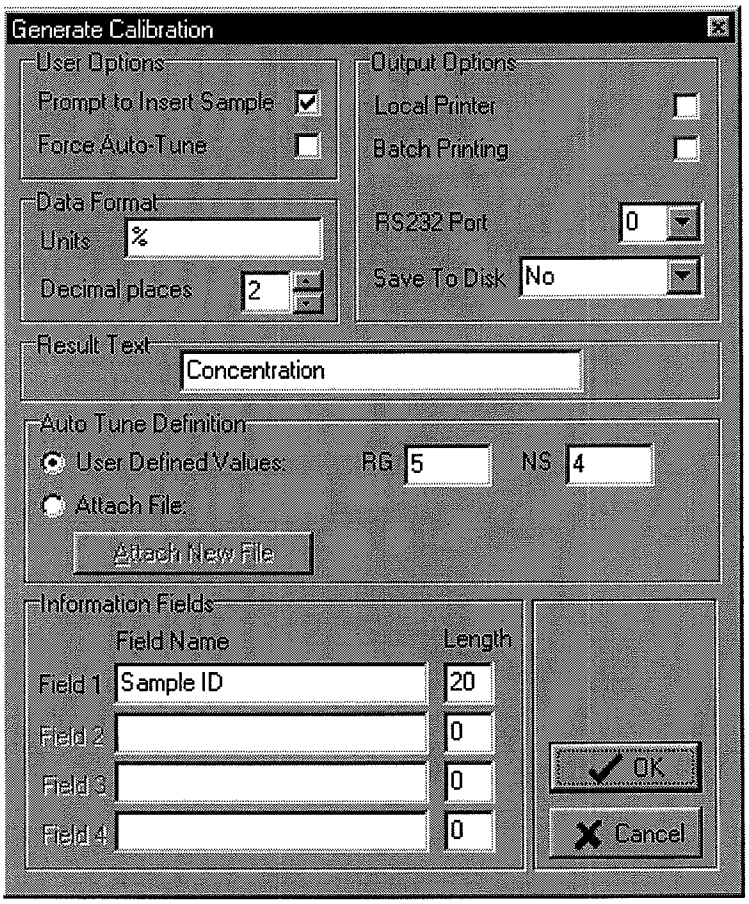
Once the user is satisfied with the quality of the calibration, the calibration may be used to generate a file for use with RI Analysis. RI Analysis allows non expert users to perform routine measurements on unknown samples.

For RI Analysis to function correctly it requires certain details, including the NMR experiment necessary, the NMR parameters, the values of the calculation ranges, the slope of the calibration graph etc. All these details are contained within a file called a calibration file which can be produced via the RI Calibration software.

When satisfied with the results of the calibration software, select the 'Generate Calibration' option from the File menu of the Main Calibration Window. A list of options is presented :

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



2.10.1 User Options

These allow the user performing the calibration to specify details regarding how the non expert user should perform the calibration. Checking the 'Prompt to Insert Sample' box displays a prompt to the non expert user to insert a sample every time a new measurement takes place. Force Auto tune forces the operator to auto tune the instrument.

2.10.2 Data Format

Allows the user to specify what units the measurement is displayed in (%, %FOF, gcm-1 etc). Decimal places allows the user to specify how many decimal places are displayed in the measurement readout.

2.10.3 Output Options

The result of the measurement is displayed to the screen as default. In addition the user may specify that the output is sent to the local printer, to a batch file for printing after all measurements have been completed, to the RS 232 port (specify which port by clicking on the down arrow next to the RS 232 box) or to disk.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.10.4 Result Text

This allows the user to specify the name of the measured property (i.e oil in seeds, % spin finish etc). Where possible, this text should confirm to the operator that he/she is using the correct calibration. For example, if the calibration is for measuring % spin finish in polyester, a good choice for the result test might be : '% spin finish (Polyester)'.

2.10.5 Auto Tune Definition

This section defines how the system auto tunes. The user may specify an RI NMR data file by clicking on the attach file radio button, from which the tuning parameters are extracted.

Alternatively, RG and NS may be specified by clicking on the user defined radio button and entering the appropriate values in the RG and NS boxes.

2.10.6 Information Fields

These fields allow the user to specify whether the operator should enter information about the sample on measurement.

A data field might be required for sample batch number, sample code number and operator. The length parameter specifies the number of characters in each data field.

2.10.7 Creating the Calibration File

Left click on 'OK' to generate the calibration file for use with the measurement software. Note that each time the operator uses the RI Analysis software he/she will need to load in a calibration. Labelling the calibration file with a name such as polyester_1.cd is more likely to ensure correct usage than 12_09_AB4D.cd.

Note that when the calibration is generated and used with RI Analysis, the measurement software will report a value for each individual page that is specified. If test calibrations are generated on pages they must be removed before the calibration is generated.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

3. QUICKSTART TUTORIAL

3.0 Introduction

This section illustrates the use of RI Calibration in creating calibrations. It takes the user through a step by step tutorial in creating a calibration for use with the RI Analysis software.

3.1 Producing a Calibration

The following steps are necessary to produce a calibration :

i) Acquire NMR data using RI NMR.

ii) Specify sample details using the calibration screen.

iii) Load in acquired NMR data.

iv) Specify ranges for data.

v) Check calibration curve and modify data ranges where necessary.

vi) Generate the calibration file for use with RI Analysis.

3.2 The Example Data Set

The example data set consists of five FID files acquired with RI NMR. Four of the samples have known concentrations, the remaining sample is an 'unknown'. The masses, concentrations and filenames are provided in section 3.3.

3.3 Loading the Data and Specifying Sample Details

First select a page to contain the data. Next right click on the Value column of the first row of cells. Move the mouse so that the Attach file box is highlighted. A file browser will appear. Attach the first demonstration file (poly.00001) to the first row of cells. Click again on the Attach file button to repeat the process for all five data files.

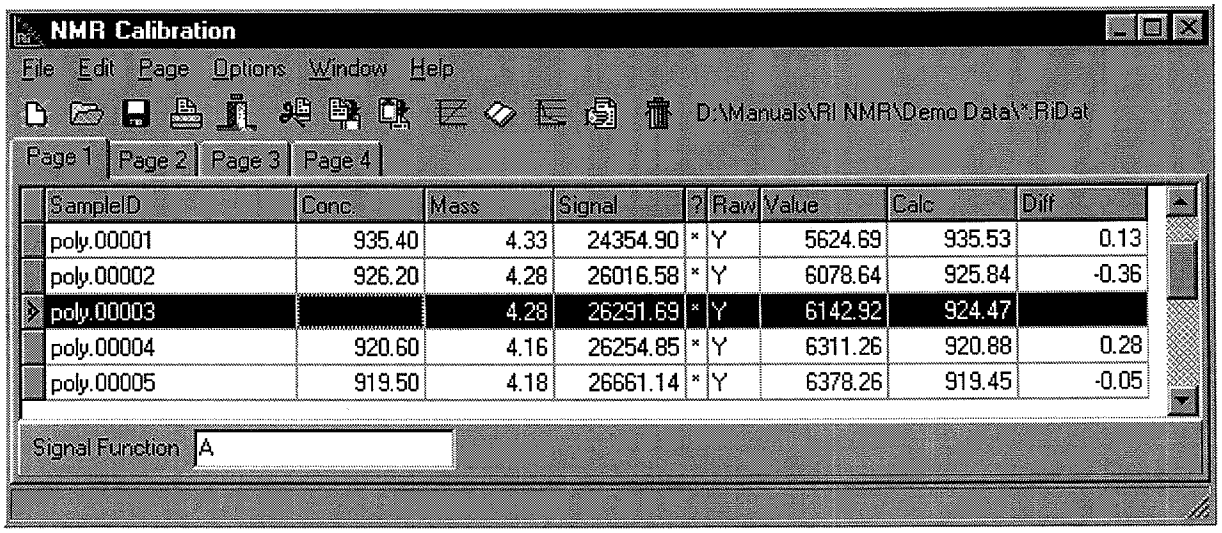
Next the sample masses and concentrations must be specified. Enter the following details in the mass and concentration columns :

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Filename	Mass (g)	Concentration
poly.00001.RiDat	4.33	935.4
poly.00002.RiDat	4.28	926.2
poly.00003.RiDat	4.28	Unknown
poly.00004.RiDat	4.16	920.6
poly.00005.RiDat	4.18	919.5

The final table after all the data has been entered should look like this :



Load in the data by right clicking on the first cell in the 'Signal' column. A file browser will appear in the default NMR data directory. Select the file 'poly.0001' and left click on the attach button. Left click again to attach the next data file to the second 'Signal' cell (the highlighted cell and the highlighted filename increment automatically when files are saved using the RI NMR auto number procedure). Repeat the process until all five demonstration files have been attached.

3.4 Specifying the Calculation Ranges

Once the data has been loaded into RI Calibration, select the View NMR Data icon (third icon from the right on the ToolBar) to view the data.

All five data sets should be displayed on the graph, along with two vertical lines (red and green). These lines define the start and end of the calculation range. All points within the calculation range may be either averaged or fitted to produce a final value which is used with the calibration. Data ranges may be specified in several ways. Either left click on the graph (after selecting either the Start or End Set Limit radio button) or enter new ranges in the boxes below the graph. Ensure the following modes are set :

Fit - Average (averages all points in the calculation range to produce a final value).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

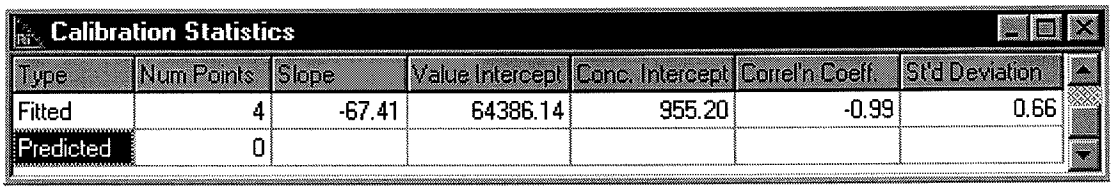
Recalc Mode - Auto (automatically recalculate statistics every time a range is changed).

Up to 10 calculation ranges may be specified for use in a calculation, a letter is associated with each range. Ensure that the 'A' calculation range is specified. Enter the following values for the start and end of the A calculation range :

Start - 50.
End - 100.

Once the calculation range has been specified, return to the main calibration screen and type 'A' in the signal function box.

3.5 Inspecting the Calibration Graph and Modifying the Data Ranges



Type	Num Points	Slope	Value Intercept	Conc. Intercept	Correl'n Coeff.	St'd Deviation
Fitted	4	-67.41	64386.14	955.20	-0.99	0.66
Predicted	0					

The Calibration Graph Window (activated with the fifth icon from the right on the ToolBar) shows the data points and the straight line fit. The details of the fit may be found in the statistics window (activated with the fourth icon from the right on the ToolBar).

It can be seen from the Statistics Window that the standard deviation in the measurement is quite large. This may be reduced by optimisation of the calculation ranges. Returning to the NMR Data Window, alter the values of the start and end parameters of the A calculation range.

Use the variation in the calibration graph and the change in the calibration statistics (which automatically update when the start and end ranges vary) to optimise the calculation range. For example, a standard deviation of 0.20 is achievable if the start and end parameters are set to 5 and 10 respectively. When satisfied with the quality of the calibration return to the Main Calibration Window.

Select the unknown sample (poly.00003) and enter a 'P' in the '?' column. The calibration will now predict the values of the unknown sample (the green point on the calibration graph) from the other 4 data points. The concentration value of the unknown sample should be approximately 924.

3.6 Generating the Calibration File for Use With RI Analysis

The final step is to generate the calibration file for use with the RI Analysis software. Select 'Generate Calibration' from the File menu of the calibration screen and select the appropriate options (a full description of the options may be found in section 2.10). Finally, save the calibration file. This file can now be used with RI Analysis.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

4. APPENDIX

4.0 Statistical Definitions

Correlation co-efficient - Linear correlation co-efficient r defined by the expression :

$$r' \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum (x_i - \bar{x})^2}\sqrt{\sum (y_i - \bar{y})^2}}$$

where x_i and y_i are the values of the ith data point.

Standard Deviation - Standard Deviation σ defined by the expression :

$$\mathbf{F}' \sqrt{\frac{1}{N-1}\sum (x_j - \bar{x})^2}$$

where N is the number of data points and x_j is the jth point.

4.1 Keyboard Short Cuts

<Shift>-<Down Arrow> Inserts a new line in the spreadsheet.
<CNTRL>-<X> Cuts a selection.
<CNTRL>-<C> Copies a selection.
<CNTRL>-<V> Pastes a selection.
<CNTRL>-<Page Number> Selects the specified page (i.e <CNTRL>-<1>).

4.2 Contact Addresses

Resonance Instruments Ltd.
Unit 13, Thorney Leys Business Park
Witney
OX8-7GE
United Kingdom.

Tel +44 (0) 1993 700442. Fax +44 (0) 1993 700363.
E-mail: Support@resonance.co.uk WWW : http://www.resonance.co.uk

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

RI Analysis Manual v3.1 23/10/98 (software release 1.4.1).

Windows 95 is a registered tradename of Microsoft Inc.

Copyright Resonance Instruments Ltd 1998.
This manual may be reproduced with permission.

Comments, errors and omissions should be reported to :

Product Support,
Resonance Instruments Ltd,
Unit 13, Thorney Leys Business Park,
Witney,
Oxfordshire,
OX8-7GE.
United Kingdom.
Tel. +44 (0) 1993 700442.
Fax. +44 (0) 1993 700363.

E-mail. support@resonance.co.uk

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1. INTRODUCTION

1.0 Introduction

RI Analysis is Resonance Instruments Windows 95 NMR analysis software for the MARAN and MARAN Ultra instruments. RI Analysis enables operators to perform routine measurements on samples from calibrations created with RI MultiQuant.

For more information on creating calibrations and acquiring NMR data using RI NMR, users should refer to the RI MultiQuant Manual and the RI NMR Users Manual.

1.1 Instructions For Operators

!!! PLEASE READ THIS SECTION CAREFULLY !!!

MARAN and MARAN Ultra instruments are designed for prolonged use in harsh industrial environments. However, the following points should be noted :

Never, under any circumstances, insert metallic objects into the sample access hole (the round hole in the top of the magnet).

Never force sample tubes into the sample access hole. If a sample tube breaks inside the sample access hole contact Resonance Instruments for advice.

Do not place any magnetic media (floppy disks, videotapes etc) in close proximity to the magnet box.

If the MARAN Ultra magnet box power supply is switched off at any time, the instrument requires a minimum of 12 hours to stabilise.

For information on basic maintenance of the MARAN and MARAN Ultra instruments (including changing fuses, please refer to the MARAN and MARAN Ultra User Installation Manuals).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2. USING RI ANALYSIS

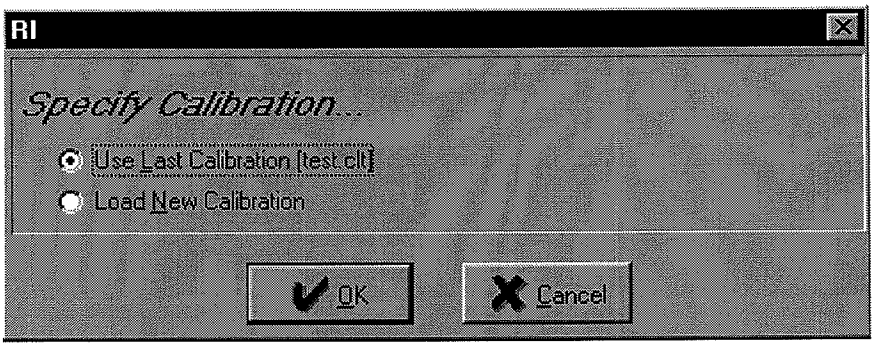
2.0 Introduction

To start RI Analysis, left click on the Start button in the bottom left hand corner of the Windows 95 screen.

Select the Programs menu and select RI MultiQuant, RI Analysis from the start menu.

It is also possible to set up a shortcut to the RI Analysis software that resides on the Windows 95 desktop. To do this, please refer to the Windows 95 menu, or discuss it with your local system administrator.

2.1 Specifying a Calibration



When the RI Analysis program is started one of two options may be selected.

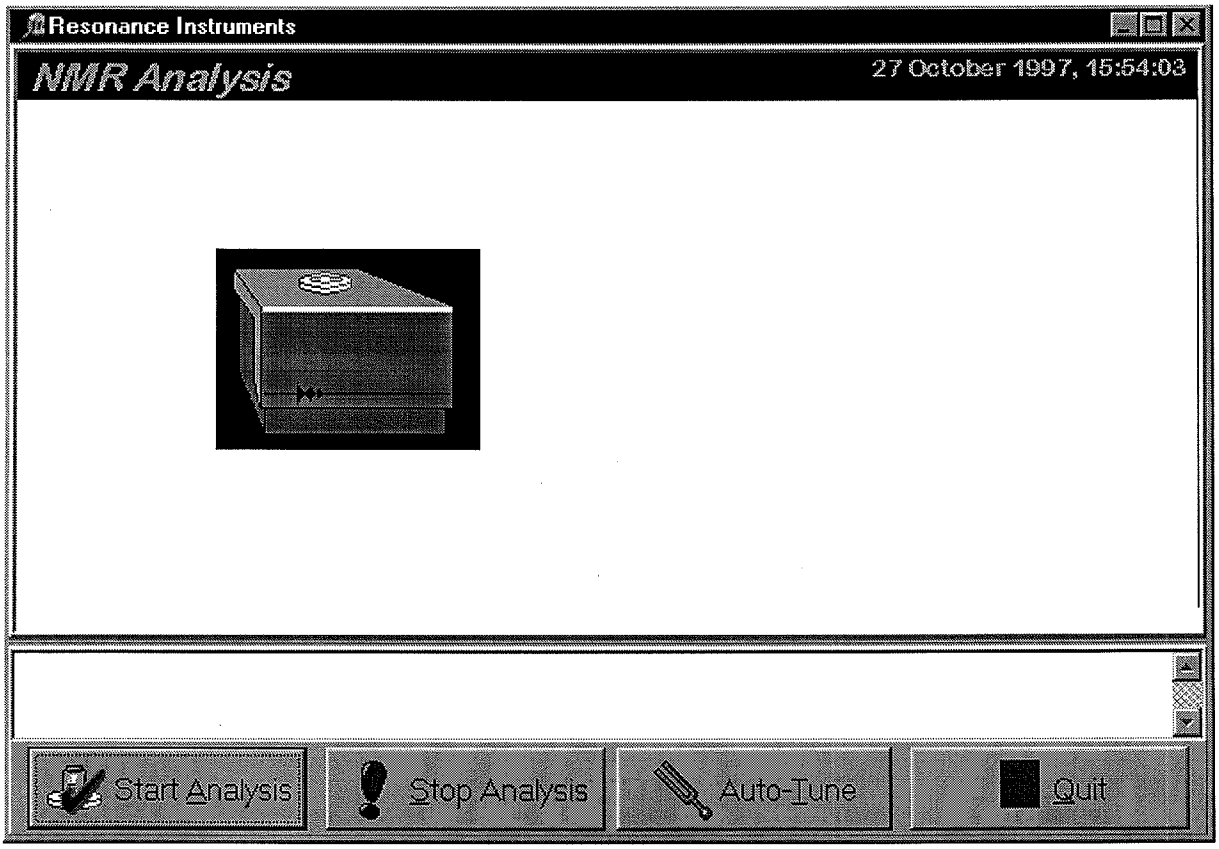
Use last calibration loads in the last calibration that was used with RI Analysis (the name of the calibration is given in brackets).

Load new calibration enables the operator to load in a new calibration file. Click on OK to browse the calibration file directory.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.2 The RI Analysis Measurement Screen



Once the calibration is selected the RI Analysis measurement screen will appear. The screen consists of three sections. The top black window, which contains a picture of the MARAN Ultra/ MARAN analyser, tells the operator when to load samples, prompts for information regarding sample masses and ID's and reports the progress of current measurements.

The central results window (in white) contains a list of all previous measurements made in the current session.

The bottom tool bar offers command options.



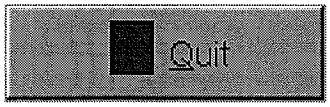
Starts an analysis. Left click on this button to start a measurement.



Left Click on this button to stop an analysis midway through a measurement. Confirm by clicking on the appropriate box in the confirmation window.



Automatically tunes the instrument.



Exits the RI Analysis software

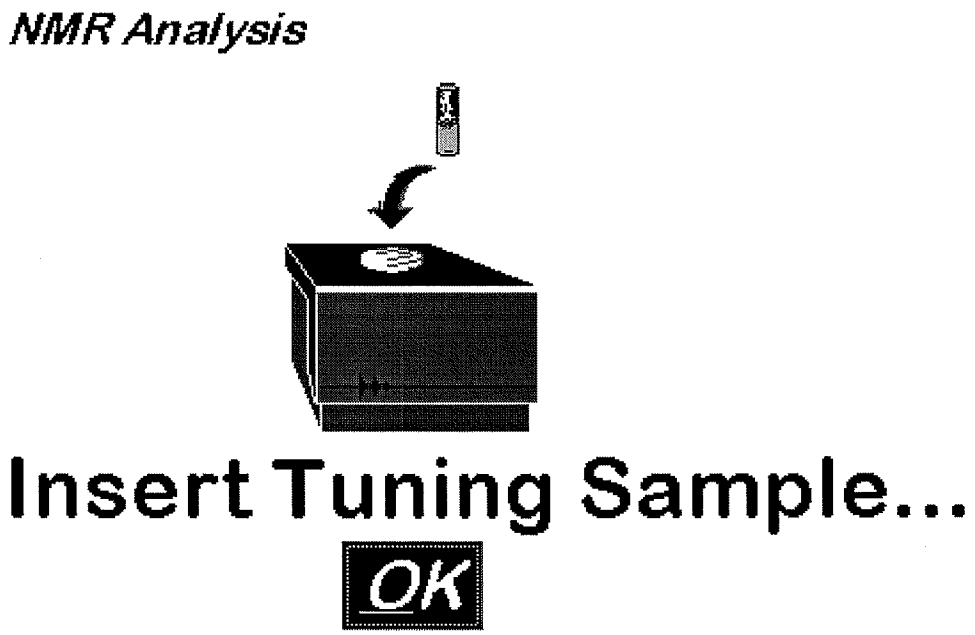
Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.2 Auto Tuning

Once RI Analysis has been started, an auto tune must be performed to tune the instrument.

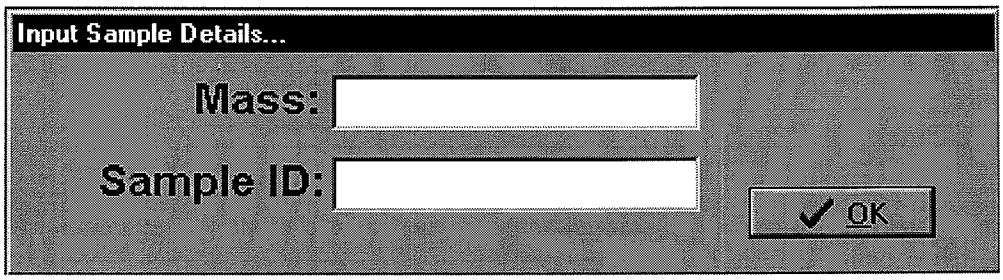
Left click on the Auto Tune button and follow the on screen instructions to auto tune the instrument.



The instrument should be auto tuned at least once per day on the supplied sample. Operators should refer to local guidelines for details on how often auto tune measurements should be performed.

2.3 Making a Measurement

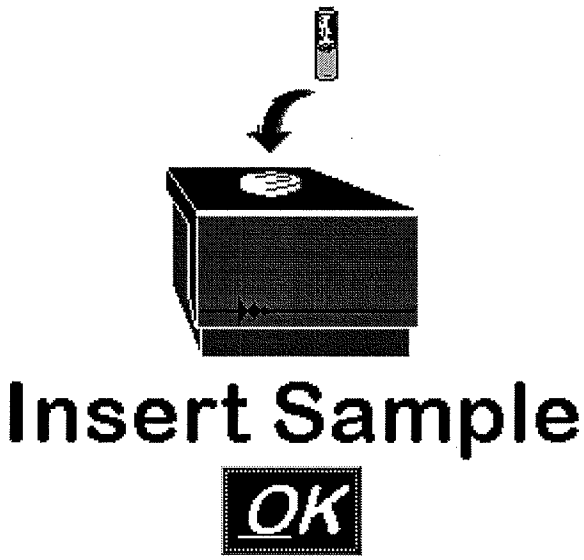
Left Click on the Start Analysis button.



When the prompt appears, enter the appropriate information and follow the on screen instructions to insert the sample.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



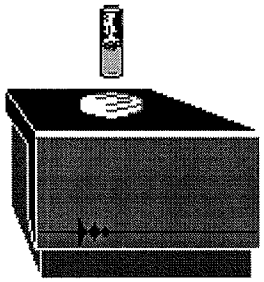
Once the sample has been inserted, click on OK or press <RETURN> if necessary.



The measurement will proceed, after the entire measurement has taken place (i.e when the scans count reaches 512/512 in this example), the measurement will finish and the operator will be prompted to remove the sample.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



Remove Sample

OK

Once the measurement has been performed the measurement result will be reported on screen :



In addition, the result will be logged to the results window and to the printer or to a file on disk if these options were specified when the calibration was created.

2.4 Configuration Options

The configuration options are provided for expert users to examine the NMR data, NMR parameters and the current calibration file settings. It is not necessary for non expert users to have familiarity with the configuration options. The configuration options menu may be accessed by right clicking with the mouse on the MARAN Ultra magnet icon in the centre of the screen.

2.4.1 Display Last FID

Displays the NMR data from the last analysis that was performed.

2.4.2 Instrument Parameters

Displays a list of the current NMR acquisition parameters.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.4.3 Change Analysis Options

This menu allows the user to change certain calibration options which are defined when the calibration is generated using RI MultiQuant. Please refer to the RI MultiQuant manual for more information regarding the calibration options.

2.4.4 About

Provides Resonance Instruments address and the version number of the software. Please quote the version number when contacting Product Support.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group is an independent supplier of quality pre-owned equipment

Gold-standard solutions

Extend the life of your critical industrial, commercial, and military systems with our superior service and support.

We buy equipment

Planning to upgrade your current equipment? Have surplus equipment taking up shelf space? We'll give it a new home.

Learn more!

Visit us at **artisantg.com** for more info on price quotes, drivers, technical specifications, manuals, and documentation.

Artisan Scientific Corporation dba Artisan Technology Group is not an affiliate, representative, or authorized distributor for any manufacturer listed herein.

We're here to make your life easier. How can we help you today?

(217) 352-9330 | sales@artisantg.com | artisantg.com

ARTISAN® TECHNOLOGY GROUP