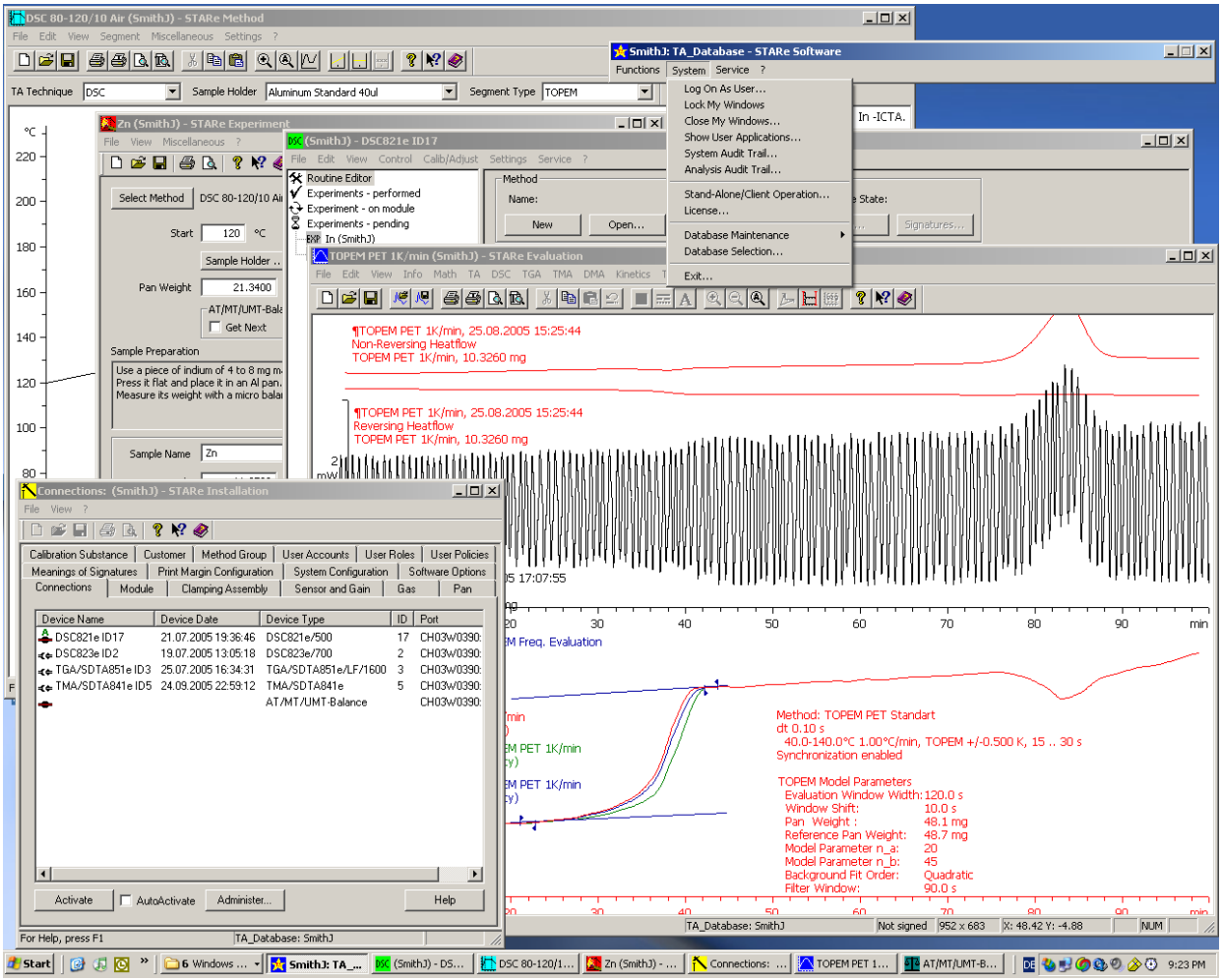
STARe Software

Version 9.00 for Windows® 2000 and Windows® XP

METTLER TOLEDO STARe Thermal Analysis System



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What's new in version 9.00?

Summary of the new features

This chapter gives you an overview of the new features and improvements of the STARe Software in version 9.00 compared with the earlier versions 6.*xx*, 7.*xx* and 8.*xx*.

The new possibility of running the STARe Software in a client/server configuration is explained in section *Client/server operation of the STARe Software, page A-2*. For sake of completion, the possibility of Terminal Server operation, which has been available in previous STARe Software versions, is also explained.

TOPEM® The new temperature modulated DSC technique is explained in section *TOPEM® — the new TMDSC technique, page A-8*.

The new possibility of localizing the STARe Software user interface thanks to Unicode support is introduced in section *Graphical user interface supports Unicode, page A-9*.

Further new features since version 8.10 are introduced in section *Miscellaneous new features, page A-10*.

The last section *List of known errors in version 9.00, page A-12* contains a list of the errors known to occur in version 9.00.

Client/server operation of the STARe Software

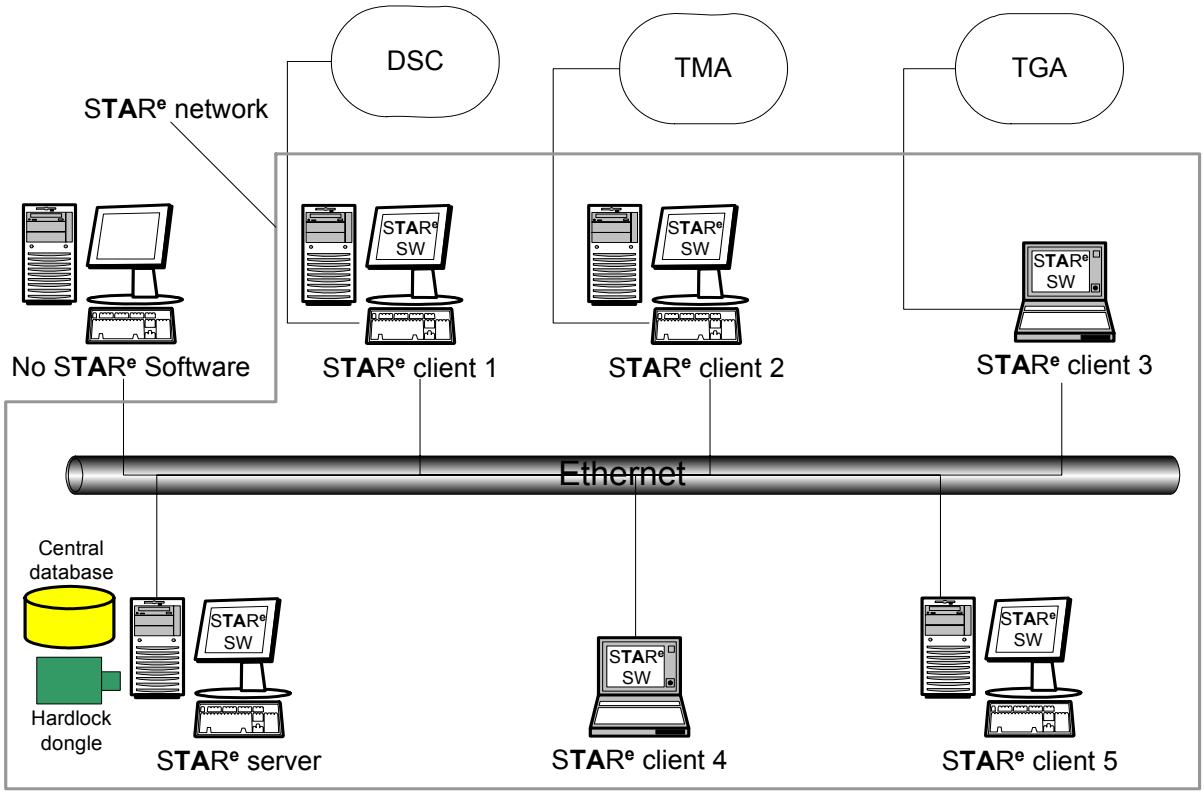
It is now possible to run the STARe Software in a client/server configuration. The configuration of several client computers and a server computer is called a STARe network. A STARe network uses the infrastructure of an existing IT network but is otherwise independent. For sake of clarity, we introduce the following terminology:

Terminology

- A STARe server is the computer on which the central STARe database is stored and administration of STARe Software licenses takes place.
- A STARe client is a computer on which the STARe Software is run in so-called "client operation mode". To obtain access to the central STARe database and to administer licenses, the STARe client is connected to the STARe server.
- In stand-alone operation mode, the STARe database is stored on the same computer on which the STARe Software is run and administration of licenses takes place.

STARe network

The central STARe database is stored on the STARe server and can be accessed from any client within the a STARe network as the following figure illustrates:



The above illustration shows the following connections and installations:

- The STAR^e Software is installed and running on every STAR^e client and on the STAR^e server.
- The Hardlock dongle and the central STAR^e database are on the STAR^e server.
- All the clients can access the central STAR^e database on the STAR^e server using the physical Ethernet connections of the existing IT network.
- STAR^e clients 1, 2 and 3 are each connected to a TA module (DSC, TMA and TGA, respectively). STAR^e clients 4 and 5 are not connected to a TA module.
- Additionally, a computer on which no STAR^e Software is installed is shown in the IT network.
- Please note that a STAR^e client or a STAR^e server cannot communicate with a TA module via a remote connection. For example, STAR^e clients 4 and 5 cannot communicate with the TA-Modules indicated in the figure. To establish communication, a direct connection to a TA module via a physical cable must be installed.

The STAR^e Software must be installed on every computer within a STAR^e network. On a STAR^e client computer the installation procedure is the same as for a stand-alone installation. On a STAR^e server computer, however, some special components and services have to be installed additionally.

There are two modes of operation: Client operation mode and stand-alone operation mode. In a typical client/server configuration, the STAR^e Software is run in stand-alone operation mode on the STAR^e server and in client operation mode on each STAR^e client. While running in client operation mode, a STAR^e client accesses the Hardlock dongle, the license file and the database on the STAR^e server and the IT network connection must be available at all times.

The licensing of the "Server Mode" and "Concurrent Users" software options is a basic requirement for operating the STAR^e Software in a client/server configuration within a STAR^e network. The "Server Mode" software option provides the basic functionality for client/server operation. The "Concurrent Users" software option includes the licensing for the desired number of client sessions (i.e. main menu bars) which can be run simultaneously.

If a Hardlock dongle and a valid license file are installed additionally on the STAR^e client, the STAR^e Software can also be run in stand-alone operation mode. A user can then switch between the two operation modes. When running in stand-alone mode, the STAR^e client accesses its local Hardlock dongle, license file and database.

♣ It is possible to operate more than one STAR^e network on the same physical network.

♣ The section *Interruption of server operation, page A-4* contains information about possible problems that may occur during server operation.

Interruption of server operation

If the IT network connection to the STAR^{e} server is interrupted, for example in the event of a problem on the STAR^{e} server, a STAR^{e} client can no longer operate normally. Safeguards have been implemented to provide for this situation: When the network connection is interrupted, the experiment continues on the TA module and, on completion, the Module Control Window saves the measurement curve locally in a file. After the connection to the server has been resumed, the curve is automatically saved in the central STAR^{e} database the next time an Evaluation Window is opened.

A similar situation can occur on a standalone system, for example when the Ingres II has to be ended because of a problem. The curve can then not be saved in the STAR^{e} database at the end of an experiment and is saved in a file. When the connection to the STAR^{e} database is available again, the measurement curve is automatically saved in the STAR^{e} database the next time an Evaluation Window is opened.

STARe Software and Terminal Server

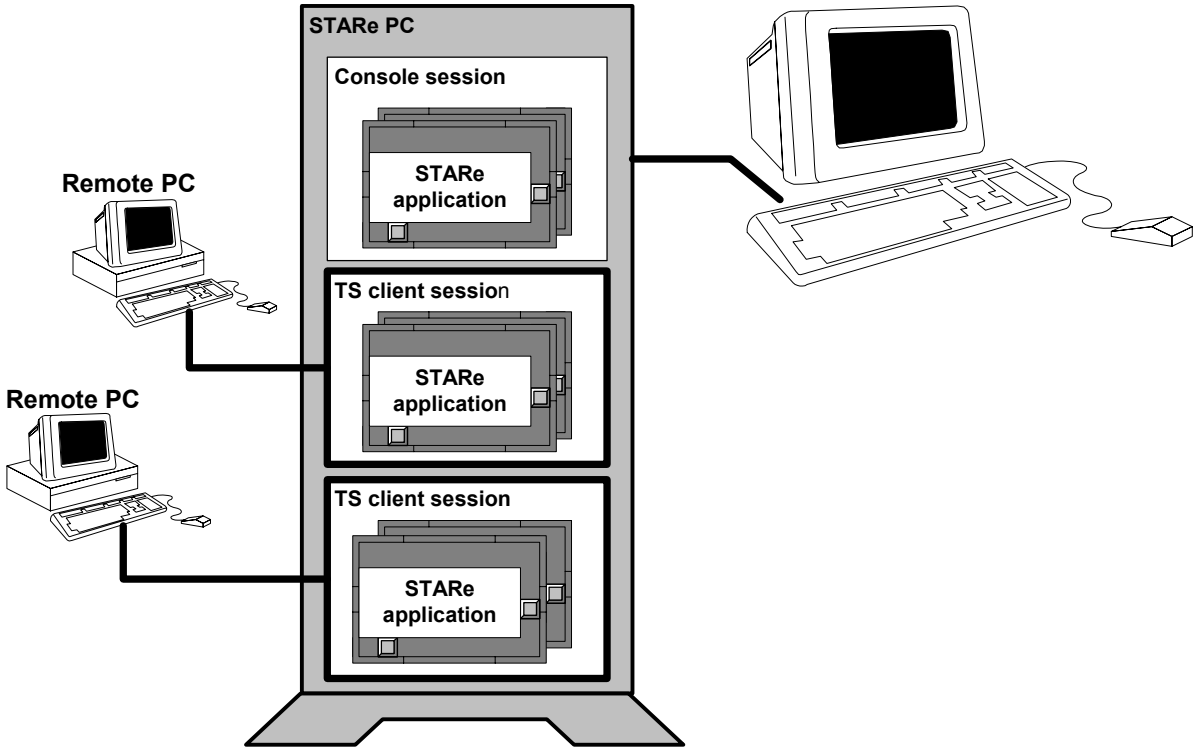
As previous STARe Software versions, STARe Software version 9.00 supports Terminal Server operation. In certain cases this may be considered as an alternative to the new Client/server operation possibility described in the preceding section.

It is possible to run several STARe Software sessions simultaneously with the help of the Terminal Services component in the server version of Windows. Terminal Services offers the possibility of running several Windows sessions simultaneously via so-called Terminal Server client sessions. The STARe Software can be run in each client session. The text below describes how this is accomplished. To understand the text you should be familiar with the following terminology:

- **Windows server**: The server version of Windows 2000 or Windows XP. It is called Windows 2000 Server or Windows Server 2003, respectively, and includes the Terminal Services component.
- **Terminal Services**: Component of a Windows terminal server installation that allows you to run multiple Windows client sessions. Terminal Services is an optional service, which can be enabled on any version of Windows 2000 Server and on Windows Server 2003.
- **Terminal Server**: Windows server installation with installed Terminal Services
- **TS**: Acronym for Terminal Server
- **Terminal Services client**: Program for running a client session. Typically, a client session is installed and run on a remote computer, i.e. one that is not at the same location as the computer on which Terminal Server is installed. The Terminal Services client software transmits only keyboard, mouse and display information over the network. The client program of Windows 2000 Terminal Server is called "Terminal Services Client" whereas the Windows Server 2003 client program is called "Remote Desktop Connection (RDC)".
- **Terminal server computer**: Computer on which Terminal Server is installed
- **Console**: The keyboard, mouse and monitor of the server computer on which Terminal Server is installed.

Installation of the STAR^{e} Software

The S**TA**R**e** Software is installed only once, namely on the terminal server computer. The different sessions of the S**TA**R**e** Software running in Windows client sessions are independent and do not interfere with each other. They access the same database system on the computer on which Terminal Server and the S**TA**R**e** Software are installed.



The Terminal Server client window looks very similar to that of the desktop on the console. Within the Terminal Server client, the S**TA**R**e** Software can be started in the usual way. The user sees only his or her individual S**TA**R**e** Software session which is independent of any other client session.

Required software options

The basic requirement for running the S**TA**R**e** Software on Terminal Server via a Terminal Server client is that the Server Mode and Concurrent User software options are installed. The software option Concurrent Users includes the number of additional licenses required to run a desired number of sessions of the S**TA**R**e** Software simultaneously.

Restrictions

The S**TA**R**e** Software has the following restrictions when it is run with a Terminal Server client:

- The Experiment Window is not available.
- The Module Control Window and the Balance Control Window are not available.
- In the Install Window, the Connections tabbed page is not available.
- A limited number of other commands on the user interface are also unavailable.

Recommended use

We recommend using the STARe Software with Terminal Server in the following way:

Lab PC:

Terminal Server is installed on your lab PC. You can run the STARe Software on the lab PC "as usual" and all the STARe Software version 9.00 commands are available. You can use the Experiment Window or the Routine Window experiment editor to set up experiments and run them in the Module Control Window. You can also perform all database maintenance tasks.

Office PC:

To run the STARe Software on the PC in your office you must install the Terminal Server client program via the network on your office PC. A network connection has to be available to the lab PC where Terminal Server is running.

With the STARe Software running on the Terminal Server client on your office PC, you can create methods in the Method Window. When you save methods they are stored in the database on the lab PC and must also be run there. In the Evaluation Window, you can access the measurement curves stored in the database on the lab PC and evaluate them.

You can also perform different user management tasks on your office PC in the Install Window.

TOPEM® — the new TMDSC technique

TOPEM®, the new advanced multi-frequency temperature modulated DSC (TMDSC) technique, is now available with the new TOPEM® software option.

TMDSC methods allow both temperature-dependent and time-dependent processes to be separated. The basic idea of TOPEM® is to overlay the isothermal or ramped temperature with a time series of stochastic (random) temperature pulses of different durations.

Sample properties can be determined as a function of time and temperature over a wide frequency range using PEM, a state-of-the-art mathematical method, which is implemented in the Evaluation Window. Thanks to the frequency information, effects that shift with frequency can easily be distinguished from frequency-independent effects. This greatly simplifies the interpretation of samples that exhibit overlapping effects.

Besides new evaluation facilities in the Evaluation Window, new TOPEM®-related features appear also in the Method Window and Experiment Window when the TOPEM software option is installed. A special method has to be created in the Method Window to perform TOPEM® measurements.

For a more detailed explanation of the TOPEM® technique, please refer to the following publications:

- "TOPEM® — the new advanced multi-frequency TMDSC technique", METTLER TOLEDO product datasheet, order number ME-51724435.
- Schubnell, M., et al., "TOPEM® — the new advanced multifrequency temperature modulated DSC technique", *Proceedings of the 33rd NATAS Conference, 2005*.
- Schawe, J.E.K., et al., "TOPEM® — the latest innovation in temperature modulated DSC", *Proceedings of the 33rd NATAS Conference, 2005*.

Working databases and archive databases

STARe Software version 9.00 provides a number of new database maintenance features.

Two types of databases can be used: Working databases and archive databases.

To store any data that is produced within a STARe Software session and to perform measurements and evaluations, a **working database** must be loaded and activated with the new "Load" command (the previous "Restore" command is no longer available). Only one working database can be used at a time (previously up to five databases could be used).

An **archive database** is used mainly for reference purposes and is a read-only database. When an archive database is selected as the current database, it is not possible to perform measurements and evaluations or to save or delete any data in the database.

Graphical user interface supports Unicode

The graphical user interface (GUI) has now been adapted to support the Unicode character set. It is now possible to translate the GUI into virtually any language.

As in previous versions of the STARe Software, you can define the GUI language setting in Windows Control Panel under Regional Options. The user interface of the STARe Software will appear in the language that corresponds to this setting if a set of dynamic-link library files (files with the extension DLL) can be loaded for that language. The dynamic-link libraries contain all the texts in a particular language that appear on the GUI.

The installation CD-ROM of STARe Software version 9.00 includes only the English GUI. Language packs for German, French and Chinese will be made available as download.

Please note that the language setting of the graphical user interface (GUI) of the STARe Software is independent of the language of the database.

Miscellaneous new features

State of reduced operation

It is now possible to run the S**TA**R^{e} Software in a state of reduced operation. Such a state is assumed when no working database is loaded, no user is logged on or the Hardlock dongle is not accessible. In such a situation, a limited number of commands are available which can be used to resume normal operation.

For example it is possible to start the main menu bar when no database is loaded. In this situation the commands to load a database are available. This was different in previous versions: the main menu bar was available only when a database was loaded.

Segments without data possible for most TA techniques

It is now possible to create methods that include segments in which no data is collected for all TA techniques except for TGA. For details, see the online help in the Method Window.

Please note, however, that only the newer DSC823^{e}, TMA/SDTA841^{e} and DMA/SDTA861^{e} modules can execute methods containing segments without data. Older instruments cannot run such methods.

DSC823^{e}

S**TA**R^{e} Software version 9.00 supports the new DSC823^{e}.

Improved text editing possibilities in the Evaluation Window

The text editing possibilities have been improved in the Evaluation Window. A text item can now be edited with a simple text editor by double-clicking (or by clicking and pressing ENTER).

Synchronization and trigger on the new DSC823^{e} module

An external device, for example a mass spectrometer, a pump or an UV lamp, can now be switched on and off automatically directly from the new DSC823^{e} during an experiment. This is done via an electric cable which is connected from the DSC module to the external device. The new "synchronization" and "trigger" features within a method now provide two possibilities to switch an external device on and off:

- With the synchronization feature, an external device is switched on for the duration of an experiment. The device is switched off again at the end of the experiment.
- With the trigger feature, an external device can be switched on and off within individual segments of a method. The cycle in which an external device is switched on and off once is called a trigger event.

When synchronization is switched on it overrides any previous trigger setting and vice versa.

Changes in the Module Control Window

Changing parameters of an experiment in the Module Control Window's experiment buffer is restricted to the owner of the experiment.

If you have installed the User Rights software option and you are using the STARe Software in a multi-user environment, a user can now only make changes to parameters of an experiment if he is the "owner" of the respective experiment (i.e. the user who created it) and is assigned the user right "Exper: Save and Run".

The following parameters can be changed with the mentioned restrictions:

- Temperature at the end of an experiment (temperature end behavior, TEB)
- PosItion number
- Furnace open permission, FOP
- Values for the sample size and pan weight

The Module Control Window is now assigned to the current user and can be locked. When a Module Control Window is locked it is not minimized. In the locked state the menu bar is not available and appears dimmed.

Contents of experiment buffer is saved

The contents of the experiment buffer is now saved. If the Module Control Window is closed, the contents of the experiment buffer (i.e. experiments pending in the experiment buffer queue) will appear again the next time the same Module Control Window is started. Any changes made to experiments, for example to temperature end behavior, position number, furnace open permission or sample size and pan weight, are also saved.

Base software now includes more features

Three earlier software options have now been incorporated in the base software. These software options were previously named "Online Evaluations", "DOS File Import" and "Application Database". They have been discontinued for version 9.00 but remain available for earlier version of the STARe Software.

User right for viewing audit trail

A new user right named "View Audit Trail" has been added to the list of user rights in the Install Window. A user can now only view the audit trail if he is assigned this new user right. The audit trail is available with the 21 CFR 11 Compliance software option and user rights can be assigned in the Install Window with the User Rights software option.

New USB Hardlock dongles

A green USB Hardlock dongle is now shipped with the STARe Software which is plugged into the USB port of your computer. The parallel-port Hardlock dongle shipped with previous STARe Software versions can still be used for standalone installations. For client/server operation, however, the new green USB Hardlock dongle must be used.

List of known errors in version 9.00

The following list describes a number of the known errors that are known to occur with S**TA**R^{e} Software version 9.00.

Problem	Details and possible remedy
General	
Problem when path of the installation folder of the S**TA**R^{e} Software includes some special characters	The path of the installation folder in which the S**TA**R^{e} Software is installed is specified during the installation procedure. If its name includes certain types of special characters, load operations of databases will fail. These types include for example German umlaut characters (ä, ö or ü) or French accent characters (such as é, è or à). We strongly recommend to avoid such characters in the path name.
Running the S**TA**R^{e} Software in client operation mode on the S**TA**R^{e} server computer will cause access problems to the database in a certain case.	If you attempt to run the S**TA**R^{e} Software in the client operation mode on the S**TA**R^{e} server computer by entering the name of the same computer for the S**TA**R^{e} server computer, the database cannot be accessed. The S**TA**R^{e} Software should be run in standalone mode on the S**TA**R^{e} server.
S**TA**R^{e} Software interactive windows accessing an archive database cannot be locked. This may cause a security problem when working in a 21 CFR 11 compliant environment.	If an archive database has been loaded and selected and there are interactive windows accessing it, it is not possible to lock these interactive windows using the Lock My Windows command on the main menu bar. Only interactive windows accessing a working database can be locked with this command. To prevent any security problem because of this, users working in a 21 CFR 11 compliant environment must be advised to manually close all interactive windows accessing an archive database. We recommend to define this in a Standard Operating Procedure (SOP) of an organisation.

Problem	Details and possible remedy
Database Maintenance	
Temporary problems with database maintenance operations in a STARe network may occur after unorderly shutdown of a STARe client.	If a STARe client in a STARe network is shut down in an unorderly manner (for example by disconnecting the mains supply), most database maintenance operations cannot be performed temporarily on other STARe clients until the STARe client has been started again (database backup operations are not affected by this). Meanwhile, after the shutdown, the name and process identifier of the STARe client will still appear on the list in the Running Applications dialog box on other STARe clients or on the STARe server. We strongly recommend not to shut down STARe clients in an unordelry manner.
Install Window	
It is possible to select more than one connection on the list on the Connections tabbed page but only one connection can be activated at a time.	Several entries on the list on the Connections tabbed page can be selected while pressing the CTRL or SHIFT keys but only the last entry selected is activated when clicking the Activate button. Each connection must be activated individually.
It is possible to leave the text box of a signature meaning empty when defining the meaning of a signature.	On the Meaning of Signatures tab in the Install Window, it is possible to create empty signature meanings by selecting the check box of a signature level and deleting the text in the signature meaning box. Such a signature would appear without a meaning. We recommend not to use such "blank" signature meaning.
Conflicts with connections may occur in a STARe network when the same TA module is used by different STARe clients in succession.	If different STARe clients in a STARe network use the same TA module in succession, it is possible that their connections to the TA module come into conflict with each other. This can occur when there is an active connection on one particular STARe client and the user on this STARe client closes the Module Control Window by closing the main menu bar. Then, another connection to the same TA module cannot be activated immediately on a different STARe client. This is because the Connections tab in the Install Window is not automatically updated. A way to update or "refresh" the connections tab list is to click another tab and then click the Connections tab again.

Problem	Details and possible remedy
Method Window	
Problems with sampling intervals with more than one decimal place after the point.	Sampling intervals with more than one decimal place after the point can produce erroneous measurement curves. We therefore do not recommend to enter more than one decimal place after the point for the sampling interval (except in methods for a DSC823^{e} in which also the four individual interval values 0.02, 0.04, 0.06 and 0.08 s are possible).
If a method includes several short segments and the sampling interval is set smaller than 0.1 s, measurement data may be lost due to communication problems with TA module.	If a method is run which includes several segments as short as 1 s and the sampling interval is set smaller than 0.1 s (possible in DSC823^{e} methods), the Module Control Window may not be able to keep up with the rate at which the measurement data is produced on the TA module. This can result in loss of measurement data. We recommend not to use very short segments and sampling intervals at the same time.
Module Control Window	
It is not possible to edit more than one experiment at a time in the experiment buffer.	Certain parameters of an experiment (i.e. position number, furnace open position, temperature end behavior, sample size and pan weight) pending in the experiment buffer queue can be changed in the Module Control Window. Several experiments in the queue can be selected but only the parameters of the first experiment in the queue, i.e. the experiment at the top of the list, can be edited within a selection. Each experiment must therefore be edited individually.
If, in a STAR^{e} network, the connection to the STAR^{e} server is interrupted, any Module Control Window running on a STAR^{e} client cannot be closed in an orderly manner.	If you are running the STAR^{e} Software in client operation mode on a STAR^{e} client in a STAR^{e} network and the connection to the STAR^{e} server is interrupted, any Module Control Windows currently running cannot be closed in an orderly manner. It can only be closed by ending its processes using Windows Task Manager. If the connection to the STAR^{e} server can be resumed, the Module Control Window can be closed in an orderly manner in the Install Window.

Problem	Details and possible remedy
Evaluation Window	
Wrong keyboard shortcuts with Chinese user interface	With the Chinese version of the user interface the following keyboard shortcuts do not work properly: ALT + Z : Zoom ALT + A : Automatic scaling ALT + B : Automatic ordinate scaling We recommend that you click the corresponding menu commands.
No error message when presentation of curve versus sample temperature fails	If a curve does not include sample temperature data and you nevertheless attempt to display it versus the sample temperature, no error message is displayed and the curve is erroneously displayed as a horizontal line through the origin.
The STARe Software cannot be closed on a STARe server or a STARe client while a backup of STARe database is in progress.	In a STARe network, the STARe Software cannot be closed on the STARe server while a database backup operation is in progress on a STARe client. If you nevertheless attempt to close the STARe Software on the STARe server in this situation, the backup operation is not interrupted but continues to completion. No error message is displayed. The STARe Software is closed on the STARe server as soon as the backup operation is completed.
The Autoscale command does not work properly on a curve with very small curve values.	It is possible that a curve consisting of very small numeral values cannot be rescaled using the Autoscale command. After attempting to automatically scale the curve, it may be erroneously displayed as a horizontal line through the origin. A workaround for this problem is to enlarge the curve using the Zoom command.
On a TMA module, the mechanical adjustment data printed or exported with a measurement curve may not correspond to the actual adjustment at the time of the measurement.	You can include adjustment data of the TMA module in the printout or exported text file when you print out or export as text a measurement curve using the Print or Export Other Format... commands. The mechanical adjustment data that appears in the printout or exported text file however correspond to the current adjustment of the TMA module and not to the adjustment at the time of the measurement. We recommend to printout or export adjustment data shortly after the measurement, before an adjustment is carried out on the corresponding TMA module.

1 Preface

1.1 Welcome to the METTLER TOLEDO STARe system!

Windows®

This User Handbook refers to version 9.00 of the STARe Software which can be run on Windows® 2000, Windows® XP Professional and Windows® Server 2003. It contains supplementary information to the operating instructions incorporated in the online Help to the STARe Software.

The STARe system comprises the STARe Software and the following TA modules: DSC823e, HP DSC827e, TGA/SDTA851e, TMA/SDTA840, TMA/SDTA841e and DMA/SDTA861e.

- The older modules DSC20, DSC25, DSC27HP, DSC30, TMA40, TG50, DSC820, DSC821e, DSC822e and TGA850 were included in the STARe system earlier but are now no longer available. They can however be operated with version 9.00 of the STARe Software.

The DSC823e, being the latest module in the STARe system, can only be run with STARe Software version 9.00.

The STARe software consists of a base software that can be expanded by adding software options according to your requirements.

- As far as it is necessary to understand the workings of the STARe Software, this User Handbook contains some information that specifically relates to individual TA modules of the STARe system. The information related to operating the hardware of your TA module is included in the operating instructions shipped with your TA module.
- The DSC25, DSC27HP, DSC30, TG50 and TMA40 modules require a TC15 TA Controller when operating with the STARe software. You can find instructions for operation with the TC15 TA Controller in the TC*15* TA Controller Operating Instructions (see chapter *Accessories*).

1.2 Typographical conventions

The following typographic conventions are used in this User Handbook:

safety notes

Safety notes are marked with safety triangles and draw your attention to points concerning safety and danger. Ignoring this information could endanger the user and result in damage to the instrument and other malfunctions.

Caution

Risk of electrical shock

NOTICE

The text NOTICE enclosed in a box indicates important remarks concerning data recording.

Times New Roman

Times New Roman type is used to mark text that appears on the screen of the computer (e.g. menu items, error messages, etc.). For example
File/Open Curve
means: click the menu File menu and then the sub menu Open Curve.

Italic

Italic font style is used for the titles of documents.

(1)
(2)

(1), (2), (3), etc. mark instructions that the user has to carry out. Explanatory text is not numbered.

♣

The clover symbol ♣ signifies additional remarks

CTRL+ALT+B indicates shortcuts for menu commands.

☞V6.0+

Information related to a particular version of the software (for example version 6.0 and later).

CTRL, SHIFT, ALT

Keys to be pressed on the keyboard or keypad of a TA module appear as capital letters. If joined with a plus (+) they are to be pressed simultaneously. Especially shortcuts are indicated in this way.

Examples:

- Press CTRL + C (to execute the Copy command on the Edit menu).
- Press OK on the keypad (denotes that the OK key on the TA module's keypad is to be pressed).

2 Introduction: Windows and STARe Software

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2 Introduction: Windows and STARe Software

2.1 Configuring the desktops

In the following, the name “Windows” refers to the three operating system Windows® 2000, Windows® XP Professional and Windows® Server 2003 on which the STARe Software can be run.

You can customize the Windows desktop according to your own requirements. The following settings can be made in Windows Control Panel.

Items to change	Program in Control Panel:
Color, font, screen background:	Display
Keyboard	Keyboard
Mouse	Mouse
Sound	Acoustic signals, Multimedia
Screen display and windows	Display

♣ The appearance of text displayed in the window area of the Evaluation Window can be changed on the Edit menu by using the Color..., Line Style... and Font... commands.

2.2 STARe Software

The STARe Software is the control and evaluation software of the STARe system. The STARe Software controls all the measuring modules connected to the system.

interactive windows

The STARe Software is operated using different interactive windows. You can create methods, control experiments and perform evaluations with these windows. Each user has his own interactive windows.

installation

To link a TA module to the STARe Software and establish communication, first of all some settings must be made in the Install Window.

method

A **method** defines the temperature program for the measurement. Each experiment requires a method. You can use a method already defined in the database or create a new method. A method can be created in the Method Window (software option) or in the Routine Window (software option). You can use a method as many times as you like.

experiment

Each **measurement** requires an experiment. An **experiment** is defined by a method and parameters related to the experiment (e.g. sample size, sample name, customer, etc). The measurement can be started as soon as the experiment has been sent to the TA module. An experiment can be defined in the Experiment Window (software option) or the Routine Window (software option).

evaluation

After the measurement, the measured curve can be opened and evaluated in the Evaluation Window. A large number of **evaluation possibilities** are available.

modular software

The STARe Software is modular and can be configured to suit your individual needs. If later on your requirements change, you can expand the software with additional software options.

To perform a measurement with the STARe system you need at least:

base software

- the STARe base software
  (includes the Install Window and Evaluation Window)
- the Routine Window software option (including a method editor and an experiment editor) **or** the Experiment Window and Method Window software options.

2.3 Structure of the interactive windows

The STARe system features seven different interactive windows:

- Install Window
- Module Control Window
- Balance Control Window
- Evaluation Window
- Routine Window, integrated in the Module Control Window (option)
- Method Window (option)
- Experiment Window (option)

All windows and functions are opened by clicking the corresponding command on the Functions menu in the main menu bar.

- Evaluation Window
- Install Window

If the software options “Method Window“ and “Experiment Window“ are installed, the following menu commands are available additionally:

- Method Window
- Experiment Window

Each window has a special function. The basic characteristics of a window are however the same for all interactive windows.

Normally, a window has the following layout:

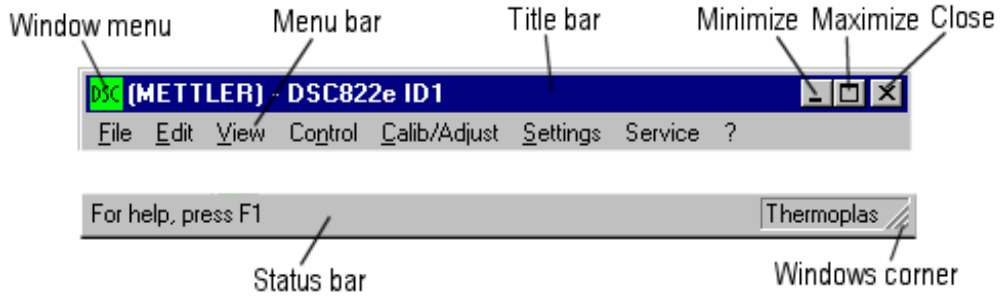


Fig. 2-1. Features of interactive windows

The **title bar** is used to identify a window. To **move the whole window**, you can position the cursor on the title bar pressing the left mouse button and drag the window to the desired position.

To close the interactive window directly you can double-click the window menu button or click the Close button in the top right window corner. To change the the size of an interactive window: Click on the Window corner and, while holding down the mouse button, pull the corner until the window has the desired size.

Please consider the following points to make entries in interactive window:

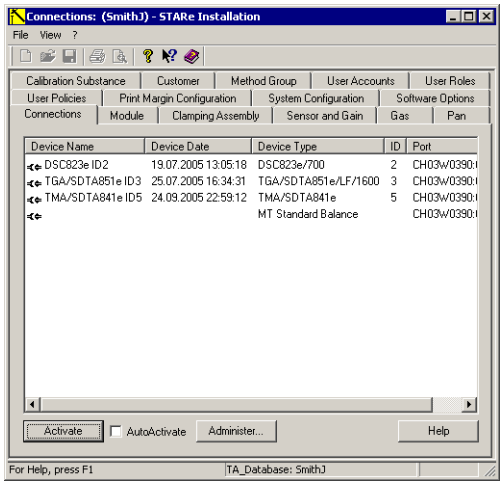
double-clicking
- With a double click in an entry box, you can mark the entire box and overwrite entries directly.

backspace key
- With the backspace key, you can erase incorrect entries.

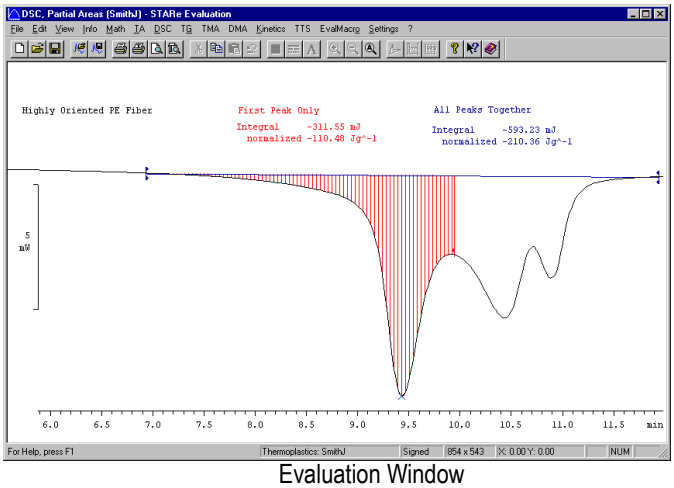
arrow key
- With the arrow key, you can change from one box to another.

ENTER key
- With the ENTER key, you confirm your entries. You can confirm your entries by clicking OK or by pressing the ENTER key.

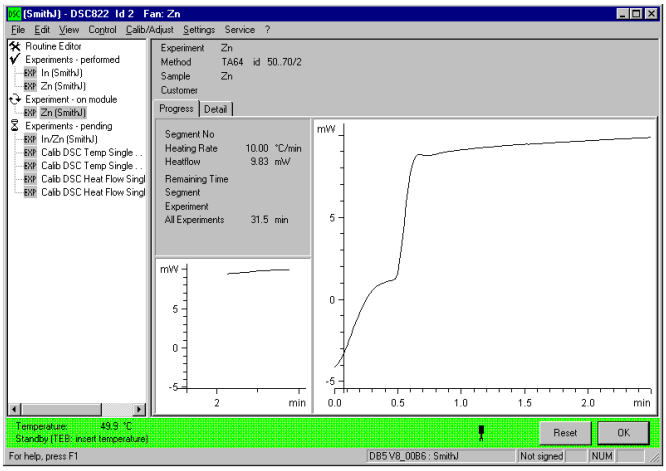
NUMLOCK key
- The NumLock keypad on the right hand side of the keyboard can be used to enter numbers.



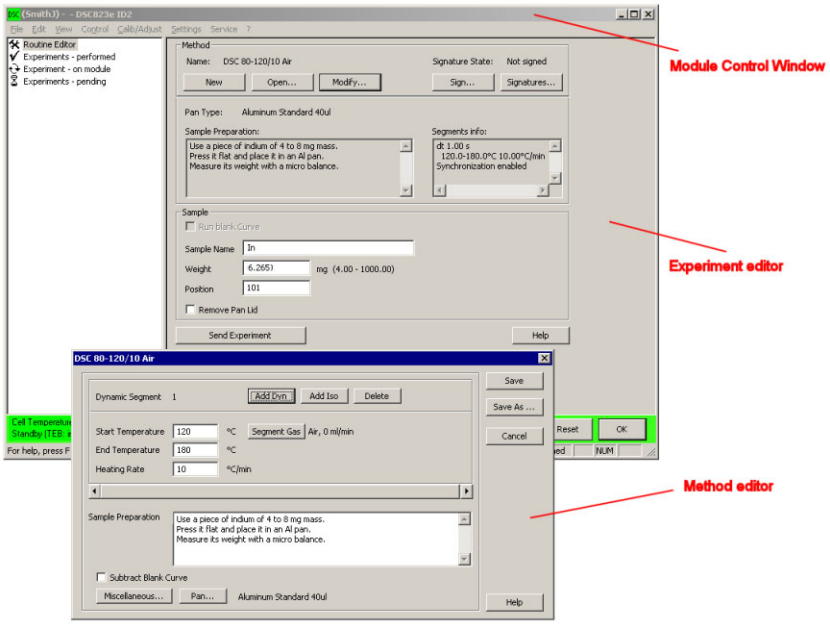
Install Window



Evaluation Window

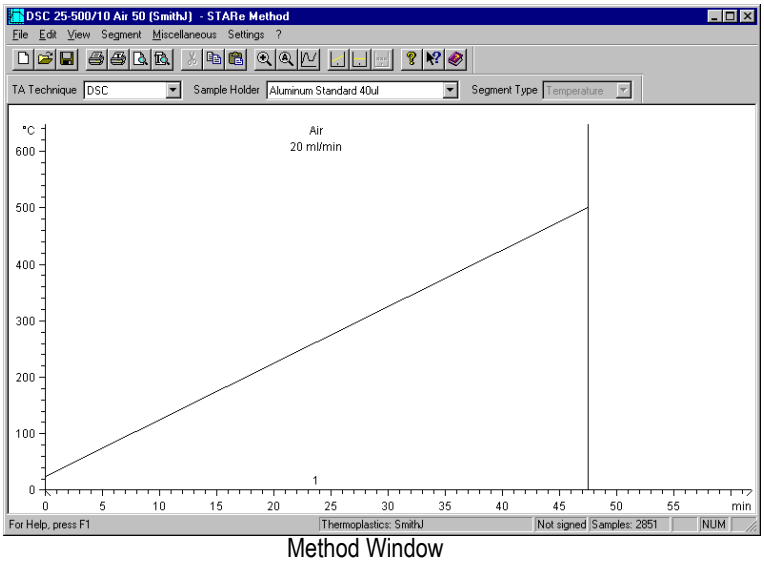


Module Control Window

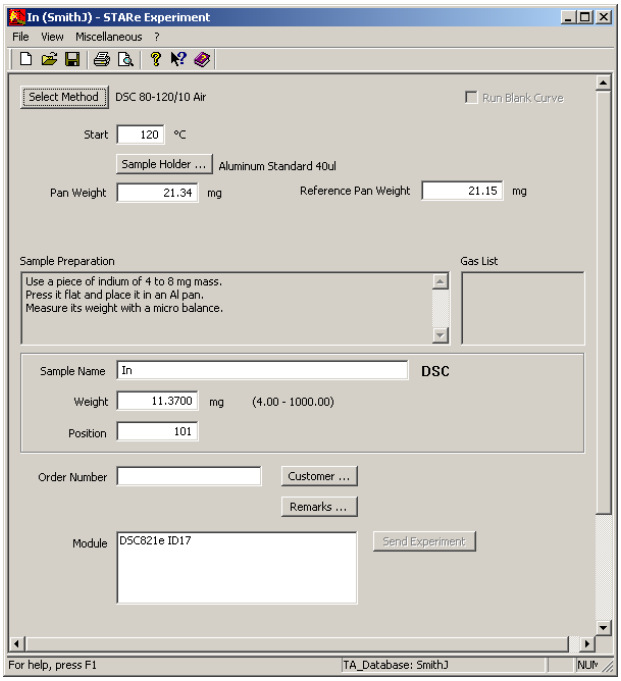


Module Control Window and Routine Window

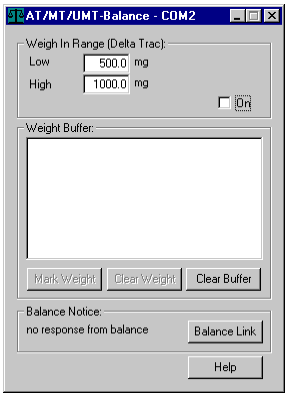
Fig. 2-2. Interactive windows in the STARe Software



Method Window



Experiment Window



Balance Control Window

Fig. 2-2 (contd). Interactive windows in the STARe Software

2.3.1 Maximizing the window

maximize

To display a window over the whole screen:

- Click the maximize button.

  The window is displayed over the whole screen (this command is equivalent to the maximize command in the window menu).

buttons of the minimized programs in the taskbar

Buttons of the minimized programs

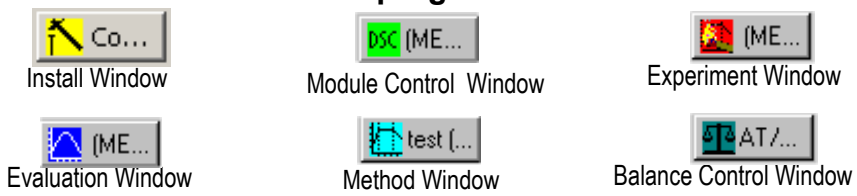


Fig. 2-3. Buttons of different interactive windows

By minimizing interactive windows, you can make room on your desktop and also increase the speed of your computer.

♣ It is quicker to open a window from its minimized state than to open an a new window.

The symbol on the taskbar button shows you what type of window it is. Depending on the interactive window, the name of the module, “install” for an install window or the name of the user is written next to the graphic. It is possible to open interactive windows for different users on the screen at the same time. To define more than one user, the software option “Install Plus“ must be installed.

opening a minimized window

To open a minimized window:

- Click the taskbar button of the particular window.

  The window is opens.

Each module connected is automatically shown in the taskbar as a button.

2.4 Meaning of the interactive windows

2.4.1 **Install Window** (base software)

Install Window

The Install Window is used to link all instruments and options with the STARe Software and to define new parameters of a more general nature, e.g. not related to just one window.

- All instruments (modules, balance) connected must be assigned to an interface so that the system is able to recognize them.
- Hardware options must be assigned to the module in this window.
- The default user name is “METTLER”. The user name can be changed in the Install Window.

♣ With the base software, only one user can be defined. If you want to define more than one user, the option “Install Plus” must be installed.

- The presentation of DSC effects is defined here, i.e whether the exothermic or endothermic DSC effects are displayed upward or downward.

Install Plus

2.4.2 Enhanced Install Window with the Install Plus option

With the **Install Plus** software option you can define additional user accounts so that multiuser operation is possible. Customers, types of crucibles, gases or method groups can also be defined.

2.4.3 **Module Control Window** (Base software)

Module Control Window

The Module Control Window allows you to run an experiment on your TA module and monitor the progress of the measurement.

An experiment can be started and interrupted via the Module Control Window. The current measurement in progress is shown as an online curve in the Module Control Window. It is not possible to create or modify methods or experiments in the Module Control Window.
As soon as an experiment is sent to the module, its name appears in the Buffer of the Module Control Window. After the sample is inserted, the measurement can be started and the measured curve followed online. The measurement can be aborted at any time.

In the Routine Window software option, the Module Control Window includes an additional method editor and experiment editor. The Routine Window can be used to create and modify simple methods and experiments.

The operation and display functions on the module are also available in the Module Control Window. The only exception is the opening of the furnace lid. The furnace lid can only be opened on the module.

2.4.4 Balance Control Window (Base software)

Balance Control Window

If you connect a METTLER TOLEDO balance to the STARe system, the button of the Balance Control Window appears automatically in the taskbar. Sample weight data can be sent directly from the balance to the STARe Software (Experiment Window/Experiment Editor).

2.4.5 Routine Window (software option)

Routine Window

The Routine Window is incorporated in the Module Control Window and consists of an Experiment Editor and a Method Editor.

You can create simple methods (maximum 10 segments) and define experiments with the Routine Window.

- To run measurements with the STARe system, either the Routine Window or the Experiment Window must be installed.

2.4.6 Experiment Window (software option)

Experiment Window

An experiment is defined in the Experiment Window. Experiments can be run directly in the Experiment Window. You can also define order numbers and customers.

If the Experiment Window is installed, the File menu in the Module Control Window includes an additional menu command Send Experiment.. which can be used to send to the experiment buffer experiments stored in the database.

2.4.7 Method Window (Software option)

Method Window

Methods can be created in the Method Window.

With the software options Relative Loop, Conditional Experiment Termination and MaxRes, DMA Evaluations and TOPEM® additional commands and features are available in the Method Window.

2.4.8 Evaluation Window (Base software)

Evaluation Window

Measurement curves can be opened, processed and evaluated in the Evaluation Window. The following evaluation possibilities are available in the base software:

- Onset and Endset (with and without threshold)
- Integration
- Peak, Step (with horiz. or tang. baselines)
- Table (e.g. to show curves in tabular form)
- Normalization to sample size (shown in W/g or %)
- Deconvolution (deconvolution of the signal by using crucibles with large time constants)
- Display of curves with respect to time, reference or sample temperature
- Show sample temperature
- SDTA (Single Differential Temperature Analysis, calculation of the DTA curve)

Additional evaluations are available if the following software options are installed:

Mathematics	Divide, multiply, add, subtract
ADSC	Fast Fourier Transformation, Steady State ADSC, ADSC
ADSC cp	Calculation of Cp from temperature modulated curves
DSC Evaluations	Glass transition, content, conversion
TOPEM®	Evaluations based on an advanced multi-frequency TMDSC technique
Specific Heat	Specific heat, cp with sapphire
IsoStep	Accurate evauation of specific heat. Kinetic effects can be separated from changes in heat capacity.
DSC Purity	Calculation purity according to simplified or complete equation Van't Hoff
TGA Evaluations	Content, conversion
DMA Evaluations	Evaluation of DMA curves by the TTS (time temperature superposition)
TMA Evaluations	Glass transition, conversion
nth Order Kinetics	Nth order Kinetics, ASTM E698, ASTM E1641, simulation, conversion plot and isoconversion plot
Model Free Kinetic	Activation energy curve, simulation, conversion plot and isoconversion plot
Advanced Model Free Kinetic	Model free kinetic analysis with isothermal and dynamic curves
Auto Evaluations and Validation	Define automatic evaluations in the Method Window

2.5 Structure of the Database

documentation

The S**TA**R**e** system ensures that measurement data is exactly and completely recorded. This means that your experimental records comply with GLP, EN45001 and other standards.

data base

All parameters defined, measured curves and evaluations are stored and managed in the **database**.

2.5.1 Database and interactive windows

The S**TA**R**e** system offers a variety of database features. Each interactive window is linked to the database. This enables data to be readily exchanged between the different interactive windows (see *Fig. 2-4*). Only one database can be accessed at any one time. The database can be selected in the system menu with the Database Selection... command. The default name of the database can be changed.

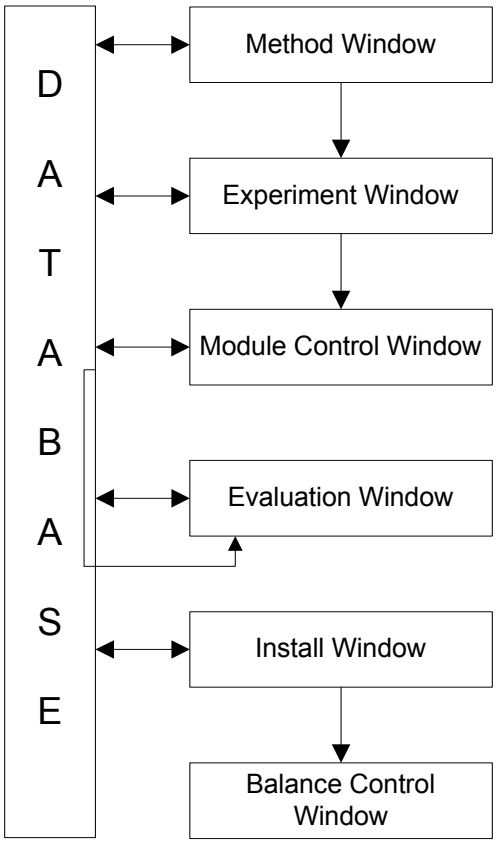


Fig. 2-4. Data exchange between the database and interactive windows

In addition, default values are defined in the database. These default values are used in the S**TA**R**e** system if you do not define your own settings.

2.5.2 Database diagram

data base

The database diagram (see *Fig. 2-5*) shows the interrelationship between the different interactive windows and of other terms.

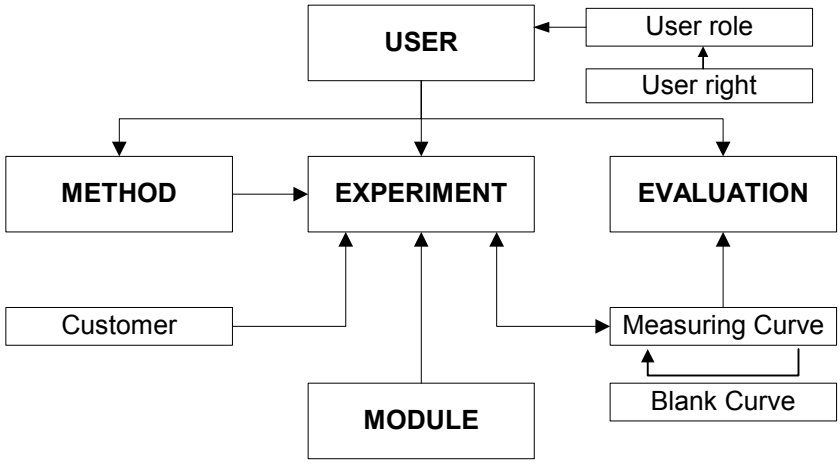


Fig. 2-5. Database diagram: The interrelationship between the various interactive windows is indicated by arrows.

The arrow (→) shows the direction from the stronger term to the weaker term. This means that:

- the user defines a method, but a method does not define the user.
- the user defines an experiment, but an experiment does not define the user.
- the user defines an evaluation, but an evaluation does not define the user.
- the customer defines an experiment, but an experiment does not define the customer.
- the method defines an experiment, but an experiment does not define the method.
- the module defines an experiment, but an experiment does not define the module.
- the measured curve defines an evaluation, but an evaluation does not define the measured curve.
- the user defines user roles and user rights, but user roles and user rights do not define the user.

Most of the information and data are **interlinked with the experiment.**

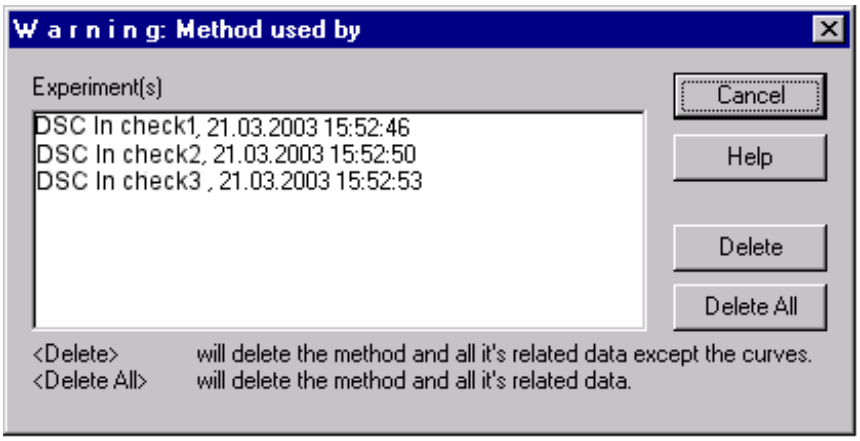
2.6 Deleting data

When deleting records containing data you must take into account that **all records linked to the deleted record** are also deleted

The relationships within the database are very important when deleting data (see the section *Database diagram*).

You can delete records (eg. methods, curves, evaluatations,etc.) with the Delete command. If any other records are linked to the record you are about to delete, you have the options of deleting the record with or without the measurement curves linked to it.

The following type of dialog box will appear containing a list of the linked records. It gives you the options of clicking the Delete button (to delete all the linked records *without* any measured curves) or the Delete All button to delete all records *including* the measured curves):



Example:

A method is linked to an experiment and the experiment is linked to a measurement curve. On attempting to delete the method the above mentioned dialog box appears. If you click Delete, the experiment and the measurement curve will all be deleted. You can however cancel the delete operation by clicking Cancel.

Examples of deletion processes

delete user

The user account is always the starting point of any data acquisition. When the user John Smith creates a method, the method is assigned to his name. The same applies for experiments or evaluations.

If the user account “John Smith” is deleted, all methods, experiments and evaluations linked to its name are deleted as well. (It is however not possible to delete user accounts when the 21 CFR 11 compliance mode is switched on.)

delete method

An experiment is defined by a method. If John Smith‘s method "Example" is deleted, then the method and all experiments defined by this method are also deleted.

If another user has used the method "Example" (made by John Smith) for his experiments, then the experiments and evaluations of that user are also deleted.

Deleting the method "Example" does however not modify the user account John Smith.

delete measurement curve

The experiment and the evaluation are dependent on the measurement curve. If the measurement curve is deleted, the experiment and all the evaluations connected with this curve are also deleted.

EvalMacro
delete evaluations

If an experiment is evaluated automatically with an EvalMacro, the experiment and the evaluation are dependent on each other. If you delete the evaluation, the experiment will also be deleted. If you delete the experiment, the evaluation will also be deleted.

delete blank curves

Measurement curves and thier corresponding blank curves are dependent on one another. If you delete a blank curve, the data of the original measurement curve without blank curve subtraction is lost. However the blank corrected curve is not deleted and remain stored.

3 Installation Instructions for version 9.00

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3 Installation Instructions for version 9.00

3.1 Introduction

The following installation instructions show you how to install the STARe Software and the Ingres II 2.6 database software. The Ingres II 2.6 database software includes the database management system used by the STARe Software and is shipped on the STARe Software CD-ROM.

One of the following Windows operating systems should be properly installed before you install version 9.00 of the STARe Software.

- Windows® 2000 (any edition)
- Windows® XP Professional
- Windows® Server 2003

In sections of the following text that apply to all three of the above mentioned operating systems, the name "Windows" is used for short, referring to all three operating systems. Where it is necessary to refer to only one particular operating system the full name is specified.

♣ Please note that Asian language versions of the STARe Software are not supported by Windows 2000.

3.2 Scope of Delivery

Delivered by METTLER TOLEDO:

- 1 CD ROM with
  - S**TA**R^{e} Software
  - Ingres II 2.6 software
  - Hardlock driver
  - MATLAB Component Runtime
  - Hardlock service
  - Sequences files software for the TGA-MS coupling
  - Readme-en.txt (text file)
- Paper documents
  - User Handbook including installation instructions
  - User Guide
  - Installation protocol
  - Declaration of System Validation
- 1 Hardlock

3.3 Before you begin installing...

Before you begin installing the Ingres II 2.6 software and the STARe Software we recommend that you read the following sections on system requirements.

3.3.1 Hardware requirements

hardware requirements

- Make sure that the hardware meets the following requirements:
  - For normal operation:

	Recommended minimum:
CPU	1 GHz
RAM	512 MB

  - For 21 CFR 11 compliant operation: *

	Recommended minimum:
CPU	1 GHz
RAM	512 MB

* With software option 21 CFR 11 Compliance installed and 21 CFR 11 compliance mode switched on

  - For the TOPEM® software option:

	Recommended minimum:
CPU	3 GHz
RAM	1 GB

  - For the STARe server or Terminal Server operation:

	Recommended minimum:
CPU	2 / 3 GHz
RAM	512 MB / 1 GB

- To install the PC, follow the instructions for your Personal Computer. The power cable should be laid in such a way that it cannot be accidentally disconnected.

peripheral device

- Make sure the following peripheral devices are installed: CD-ROM drive, SCSI or IDE
- Please use officially supported hardware only.
- Make sure that the system's peripheral devices are configured correctly.

3.3.2 Supplements to the standard Windows installation

NTFS partition

- We strongly recommend that you install the S**TA**R**e** Software on an NTFS partition (this is imperative for 21 CFR 11 compliant operation). A FAT partition can be converted into a NTFS partition by entering the following command at the Windows command prompt:
  CONVERT <drive>: /FS:NTFS

Windows user account ingres

- A local Windows user account ingres must be implemented. This user account must permanently be assigned administrator rights and the user name ingres must not contain capital letters. The password of this user account must never be changed and never expire. Otherwise the Ingres II 2.6 database management system may not start up properly.

Windows user accounts for users of the S**TA**R**e** Software

- A Windows user account must be set up for each user of the S**TA**R**e** Software.

required disk space:

- Sufficient space must be available on your hard disk. The following free space is required:
  for non-21 CFR 11 compliant operation:

	Recommended
Free disk space	2 GB

for 21 CFR 11 compliant operation: *

	Recommended
Free disk space	3 GB

* With software option 21 CFR 11 Compliance installed and 21 CFR 11 compliance mode switched on

♣ Enough disk space for your own entries in the database must be provided. Typically, at least 300 MB extra space is required. Depending on usage, more space may have to be provided. For example 21 CFR 11 compliant operation, DMA or TOPEM® measurements or client/server operation may require considerably more additional disk space.
Please take into account that twice the amount of disk space as required for the database must be available additionally to perform a backup of a database.
In general, not more than 90% of the hard disk space should be used.

3.4 Overview of the installation procedure

The following procedure is recommended for the initial installation of the S**TA**R^{e} Software. Besides the actual S**TA**R^{e} Software program components, auxiliary software components and Ingres II 2.6 database management system (DBMS) software must also be installed. The Ingres II 2.6 software must be installed first.

The installation of the entire S**TA**R^{e} Software is carried out by the user ingres. This user must be installed and assigned Windows administrator rights (see *Supplements to the standard Windows installation, page 3-4*).

The following list summarizes the installation steps:

Stages of the installation	Log on to Windows as
1 Install Ingres II 2.6	• ingres
2 Start Ingres II 2.6	• ingres
3 Install S**TA**R^{e} Software with Hardlock device driver and optional components	• ingres
4 Start S**TA**R^{e} Software	• ingres
5 Reboot	

3.5 Installation of the Ingres II 2.6 software

Before installing the STAR[e] Software you must install the Ingres II 2.6 database management system. The version required by STAR[e] Software version 9.00 is Ingres II 2.6 SP2 ("SP2" stands for Service Pack 2).

A local Windows user account ingres must be implemented. This user account must permanently be assigned administrator rights and the user name ingres must not contain capital letters. The password of this user account must never be changed and never expire. Otherwise the Ingres II 2.6 database management system may not start up properly.

3.5.1 Installing Ingres II 2.6 software

When you perform the installation of the Ingres II 2.6 software, the Ingres II Setup Wizard guides you through the installation procedure.

To install the Ingres II 2.6 software:

(1) Log on to Windows as user ingres.

(2) Insert the STAR[e] CD-ROM in the CD-ROM drive.

(3) Start the installation program by selecting the CD-ROM drive from My Computer or the Windows Explorer and double-clicking the file Ingres II 2.6 SP2\Setup.exe.

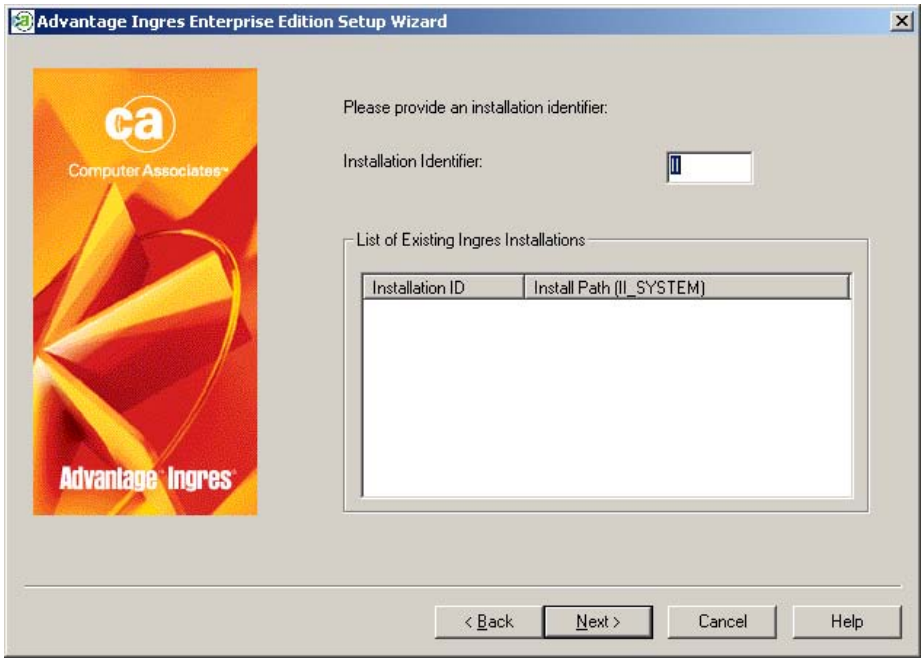
The Ingres II Setup Wizard starts up showing the initial Welcome dialog box.

(4) Click Next to continue.

A dialog box appears prompting you to enter your name and company name.

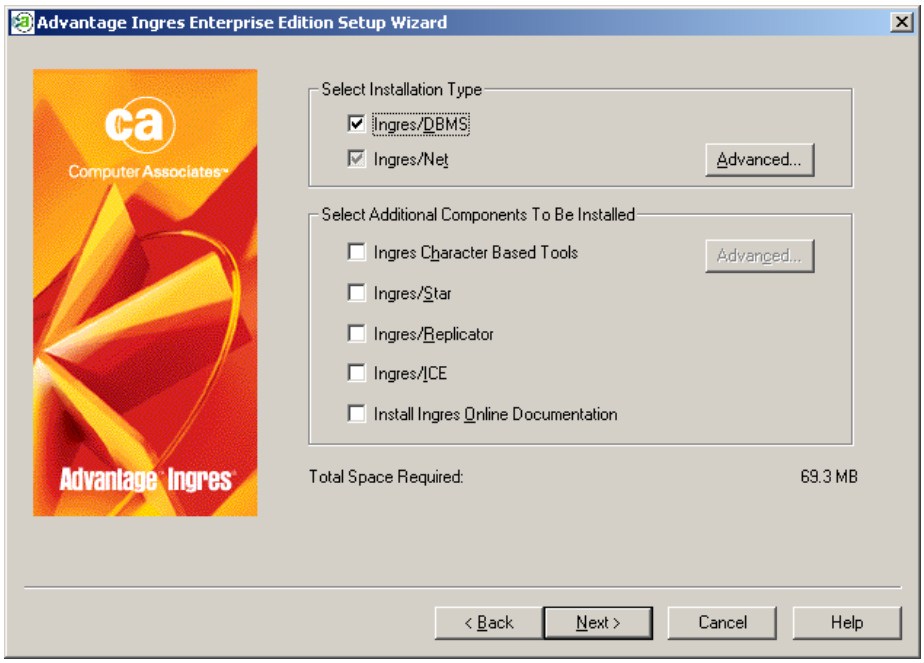
(5) Make your specific entries in the Name and Company boxes and click Next to continue.

The following dialog box appears.



(6) Make sure that II is entered in the Installation Identifier text box. Then click Next to continue.

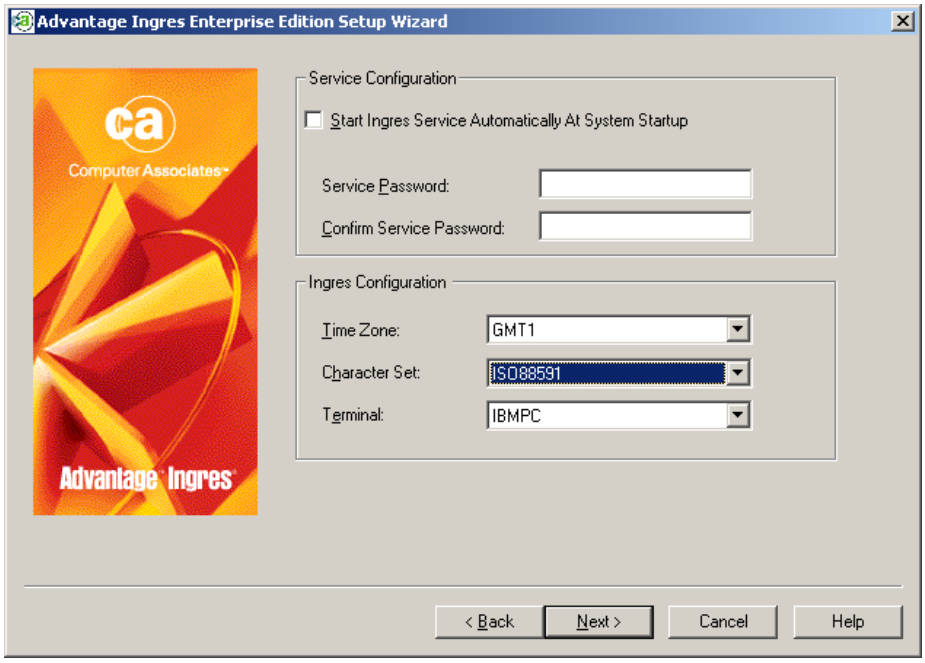
The following dialog box appears:



♣ The default setting of the check box Ingres/Net as shown above specifies the use of the TCP/IP protocol and should be adopted.

(7) Make the settings as shown above. Clear all the check boxes under Select Additional Components To Be Installed. Then click Next to continue.

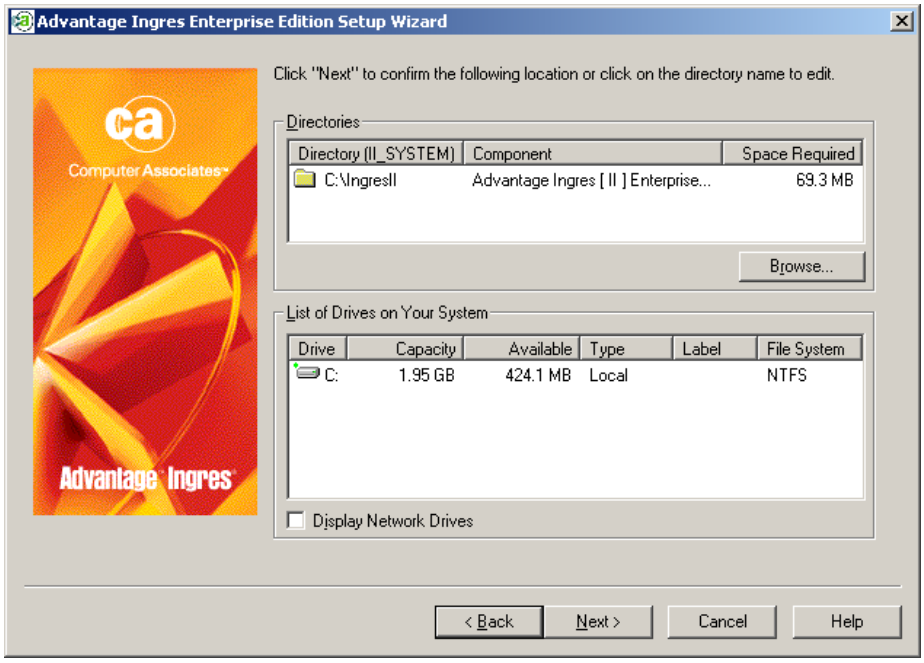
The dialog box for entering the Ingres II 2.6 software settings appears.



(8) Make the following settings:

- Under Service Configuration, deactivate the feature Start Ingres Service Automatically At System Startup by clicking so that the check disappears. Leave the Service Password and Confirm Service Password text boxes blank.
- Under Ingres Configuration:
  Select your time zone in the Time Zone text box with the arrow buttons (for example select GMT1 for Greenwich Meantime + 1 hour).
  Select the character set ISO88591 in the Character Set box.
  Select IBMPC in the Terminal box
- Click Next to continue.

A dialog box for defining the installation folder of the Ingres II 2.6 software appears.

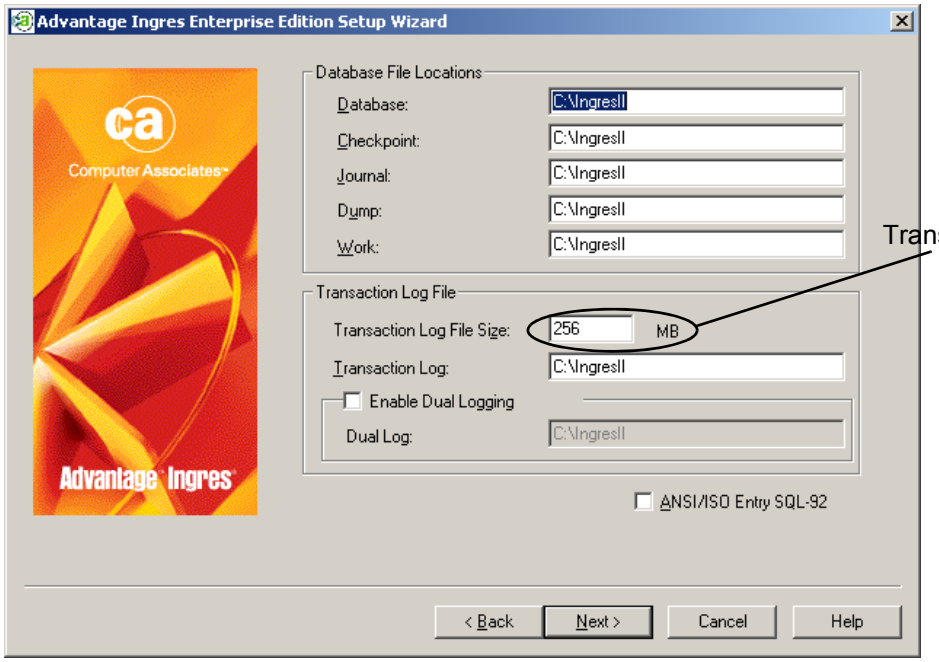


(9) Check the path of the folder under Directory. In the default installation the path is C:\IngresII as shown above. Click Next>.

or

If you want to install the Ingres II 2.6 software in a different folder: Use the Browse... button to navigate to the desired folder, then click Next>.

A dialog box displaying the specified folders (locations) of data base files appears.



Make sure that the size of the Transaction Log file is at least 256 MB.
The STAR^{e} Software setup program will not run if the Transaction Log file is smaller than 256 MB.

(10) Change the file size by entering at least 256 MB in the Transaction Log File Size box.

(11) If the database file locations are as desired click Next to continue and start the installation of the Ingres II 2.6 software.

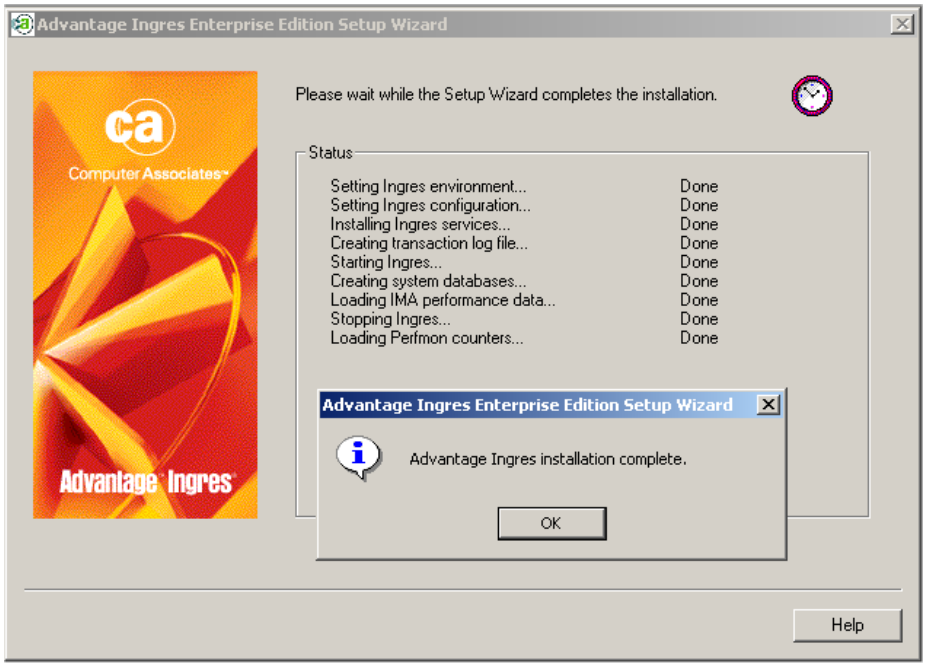
The final dialog box of the Ingres II Setup Wizard appears.

or

Otherwise click Back until you reach the dialog box for entering the Ingres II 2.6 software settings and repeat from step 8 onward.

(12) Click Finish to start the installation of Ingres II 2.6.

The Ingres II 2.6 software is installed. Different windows and dialog boxes appear on your screen during the installation procedure.
At the end of the installation the following message appears: Advantage Ingres installation complete.



(13) Click OK to exit the installation program.

The Ingres II 2.6 software has now been installed.

3.5.2 Prevent automatic startup of the Ingres Visual Manager

The Ingres Visual Manager program is installed automatically with the Ingres II 2.6 software. When the Ingres Visual Manager is running, a number of messages from the Ingres II 2.6 software may appear on the screen. These messages do however not concern users of the STARe Software and can be avoided by preventing the Ingres Visual Manager from starting up automatically.

To prevent automatic startup of the Ingres Visual Manager:

(1) Right-click the Start button in the Windows taskbar.

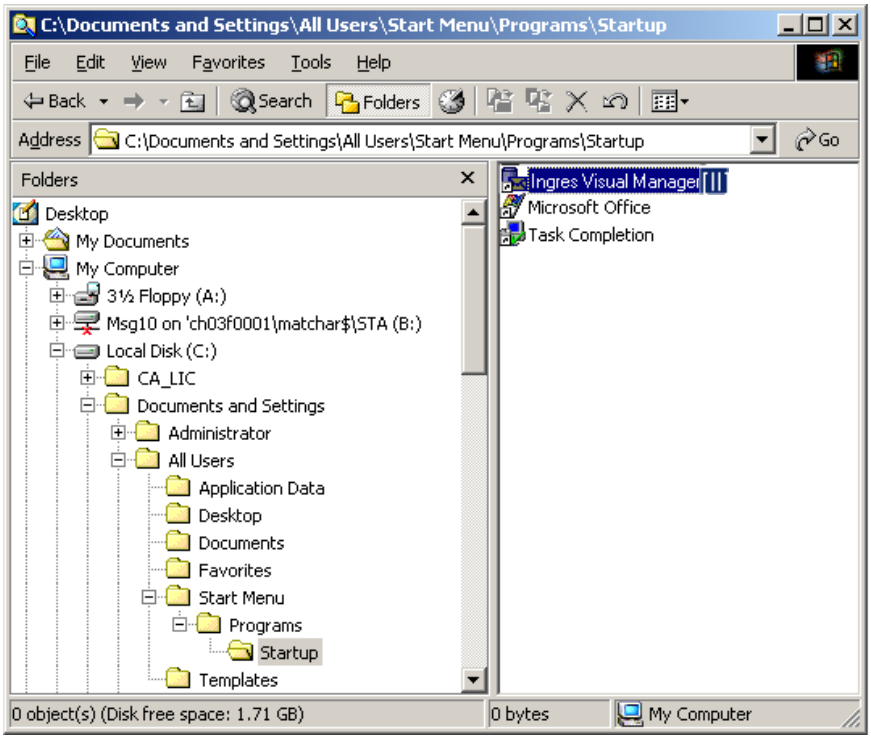
A submenu appears including the Open All Users and Explore All Users commands.

(2) Click Explore All Users.

The Windows Explorer opens and automatically navigates you to the Start Menu folder.

(3) In the Start Menu folder, open the folder Programs and then the subfolder Startup.

The contents of the Startup folder is displayed in the right pane.



(4) Delete the shortcut Ingres Visual Manager [II]in the Startup folder.

The shortcut Ingres Visusal Managers is deleted. The Ingres Visual Manager will now not start up automatically.

3.6 Configure and startup of the Ingres Intelligent Database [II] service

To start up the Ingres II 2.6 software automatically on starting your computer, make the following changes in Windows Control Panel:

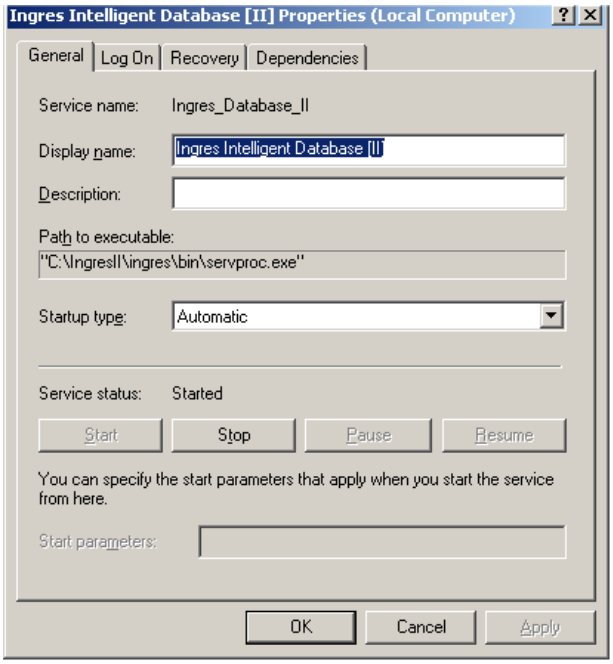
(1) Click Start/Settings/Control Panel to open Control Panel, double-click Administrative Tools and then Services on the corresponding lists.

(2) Mark Ingres Intelligent Database [II] in the list of services.

(3) Choose Action/Properties.

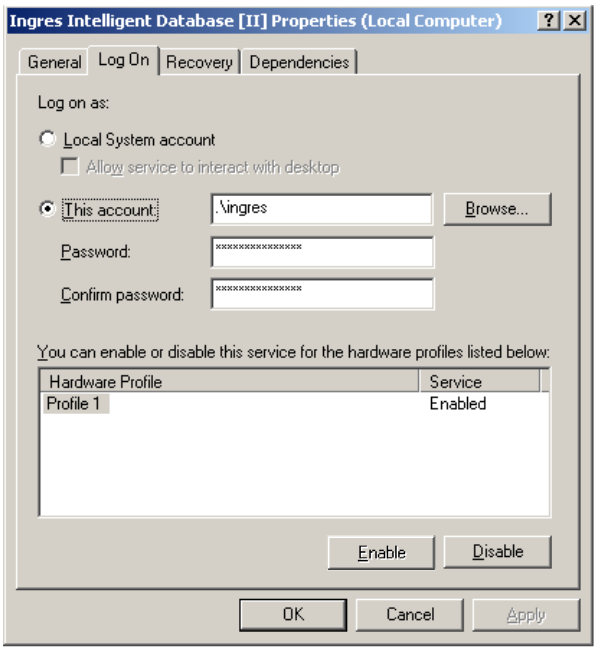
The Ingres Intelligent Database [II] Properties dialog box appears

- Click the General tab.



- Ensure that Automatic is selected in the Startup Type box.

- Click the Log On tab.



(4) Select the This Account check box and make the following entries:

- Ensure that the This account box contains the entry .\ingres.
- Enter the password of user ingres in the text box Password and confirm the password in the text box Confirm Password.

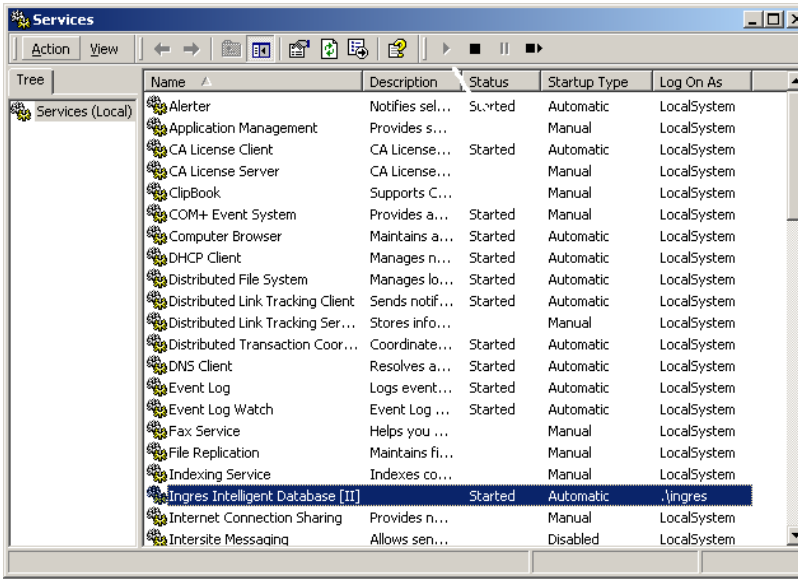
♣ The Password and Confirm Password text boxes always contain a number of Asterisks (******).

- Click OK to apply the settings and to close the dialog box.

The program returns to the Services dialog box.

(5) Start the Ingres Intelligent Database [II] service by clicking Action/Start.

If the settings have been made correctly, the status Started is displayed in the Services dialog box.



(6) Close the Services and Administrative Tools dialog boxes.

The startup settings for the Ingres II 2.6 software are completed. You can now proceed to install the STARe Software components as explained in section *Installation of the STARe Software, page 3-15*.

3.7 Installation of the STARe Software

After you have installed the Ingres II 2.6 software you can install the STARe Software including the STARe Software program components and some auxiliary components (Hardlock software).

To install the STARe Software and auxiliary components:

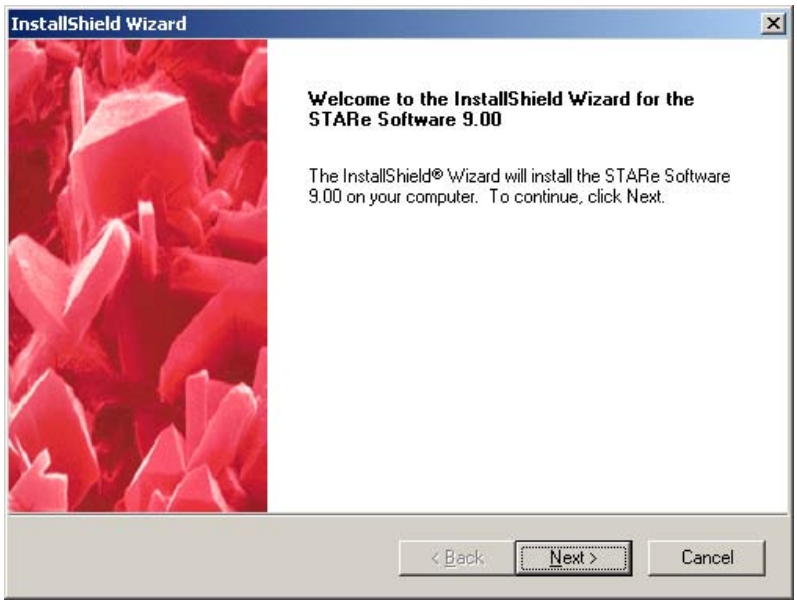
(1) Log on to Windows as user ingres.

(2) Insert the STARe CD-ROM in the CD-ROM drive.

(3) Select the CD-ROM drive from My Computer or Windows Explorer.

(4) Double-click the setup.exe file to start the setup program and the installation procedure.

The initial dialog box of the InstallShield Wizard installation program appears.

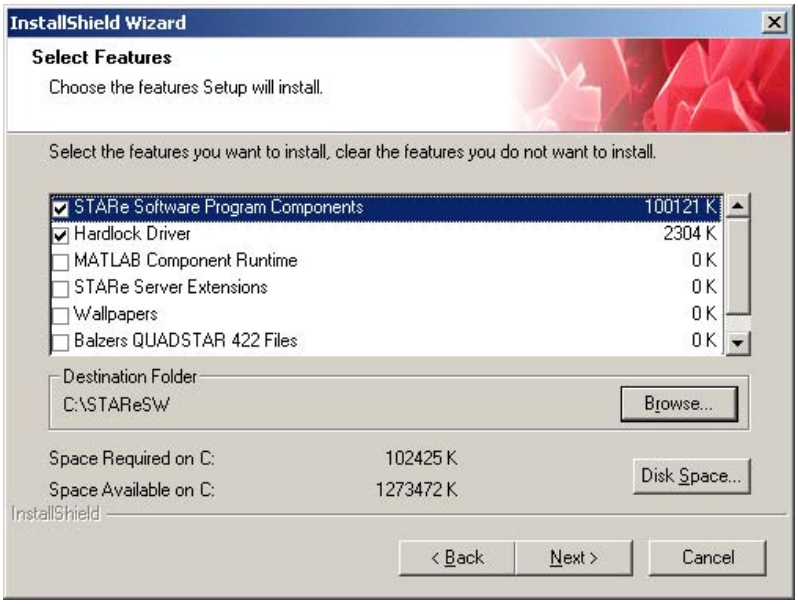


(5) Click Next> in the initial “Welcome” dialog box.

A dialog box appears containing the license agreement text.

(6) Confirm the License Agreement box with Yes after reading the agreement text.

The Select Features dialog box for the selection of the software components appears.



The setup installation program automatically recognizes the components that need to be installed. For the initial installation of the S**TA**R**e** Software, the setup installation program automatically selects the items STARe Software Program Components and Hardlock Driver. This is the minimum requirement for the installation.

The item MATLAB Component Runtime is required if you wish to install the TOPEM software option.

The item STARe Server Extensions is necessary if you wish to use this S**TA**R**e** Software installation as a S**TA**R**e** Server.

The items Wallpaper, Balzers QUADSTAR 422 Files and Balzers QUADSTAR 32-Bit Files are optional.

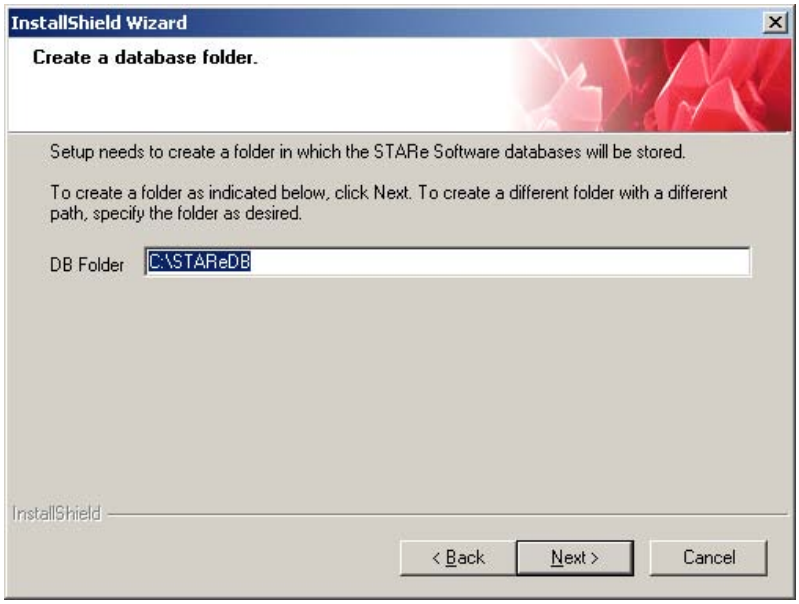
Please note that not more than **80 characters** are allowed in the path name.
Do not install the S**TA**R**e** Software on a network drive. The software will not run on a network drive.

(7) If you want to install the program in the default folder, confirm the default folder C:\STAReSW with Next> in the Program Folder box.

or

To perform the installation in a folder other than the default folder: Navigate to the desired folder with Browse... and confirm with Next>.

The Create a database folder dialog box appears. A folder in which the database of the S**TA**R**e** Software will be stored has to be created.

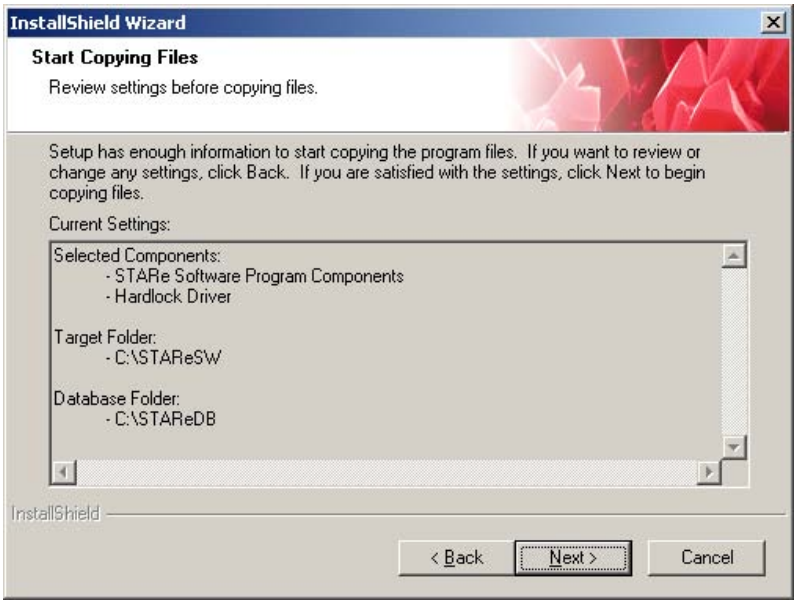


(8) To create a folder as indicated in the DB Folder box, click Next>.

or

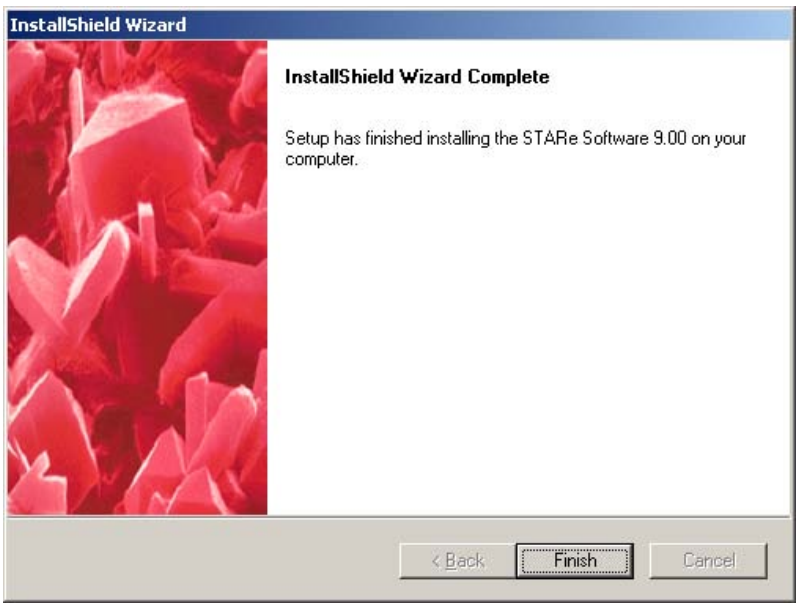
To create a different folder with a different path or name, enter the path and name of the desired folder in the DB Folder box.

The Start Copying Files dialog box appears. You can review or change the settings by clicking Back> to return to the pervious dialog box.



(9) If you are satisfied with the settings, click Next> to copy the files.

The Setup Status dialog box appears showing the progress of the installation. The STARe Software is automatically installed together with the Hardlock Driver and any optional items (if selected) are copied to your hard disk.
At the end the final dialog box of the STARe Software installation procedure appears:



(10) Click Finish to conclude the setup program.

The components of the STARe Software are installed.

The installation of the STARe Software is now completed.

3.8 Checking the Ingres II 2.6 license

When you have completed the installation of the STARe Software and Ingres II 2.6 software we recommend that you check your Ingres II 2.6 license.

To check your Ingres II 2.6 license:

(1) Open the Command Prompt window in Windows. Change to the folder Ingres II 2.6 SP2 which includes the license check program LicCheck.exe as follows:

- Insert the STARe CD-ROM in the CD-ROM drive.
- <CD drive letter>:, ENTER

  where <CD drive letter> denotes the letter of your CD-ROM drive on your PC (for example D).
- cd Ingres II 2.6 SP2, ENTER

(2) Enter the command
LicCheck
at the command prompt and press ENTER to start the license check program.

The license check program starts. The output should be as in the following example:

```
D:\Ingres II 2.6 SP2>liccheck

Querying Service 'LogWatch'...
Stopping Service 'LogWatch'...
'LogWatch' Service Stopped Successfully.
License Check for '2H30' returned: 7300 (All OK)

License Is Valid!

Starting Service 'LogWatch'.....
'LogWatch' Service Started Successfully.
```

Your license is only valid if the note All OK appears. The text License Is Valid however is always displayed regardless of the validity of your license.

The check of the Ingres II 2.6 software license has been completed.

3.9 First start of the S**TA**R**e** Software after installation

Before you start the S**TA**R**e** Software for the first time after installing it, make sure that the Hardlock (dongle plug) shipped with the software is plugged into the parallel port or USB port of your computer.

Please take note of the following points about language setting and localization of the S**TA**R**e** Software:

language of database

- The language version of the S**TA**R**e** Software database can be changed (localized) within a session of the S**TA**R**e** Software. You can select the language version of the database on the System menu of the main menu bar by clicking Database Maintenance/Localize.

language of user interface

- The language version of the S**TA**R**e** Software user interface is set independently of the language of the database when you start up the S**TA**R**e** Software. It corresponds to the language setting in Windows Control Panel under Regional Options at the time of the startup.

3.9.1 Starting normal operation

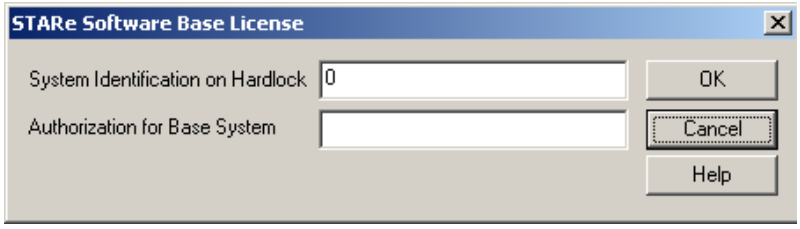
The following procedure describes how you start the STARe Software for normal operation for the first time after installation. By "normal operation" we refer to an operation state in which all the features included in the base license can be used. You must enter your Hardlock number and authorization code and load a working database for this.

To start the STARe Software for the first time proceed as follows:

(1) Log on to Windows as user ingres.

(2) Start the STARe Software by clicking the STARe Software icon on the desktop or by clicking Start/ Programs/STARe software.

The STARe Software Base License dialog box appears prompting you to enter your Hardlock number and your authorization code for the STARe Software.



♣ The Hardlock number is printed on the label on your Hardlock. The authorization code is printed on the license sheet that is delivered with the Hardlock for the STARe Software.

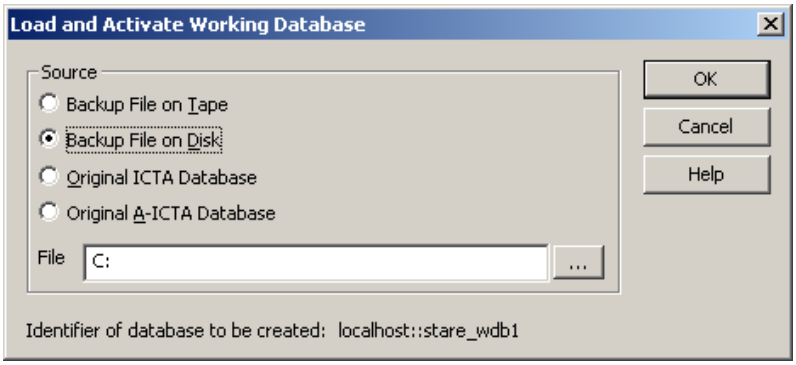
The authorization code is a hexadecimal number and also includes, besides numbers, letters from A to F. Take care not to confuse the number (0) zero and the letter O on your keyboard (a hexadecimal number never contains the letter O). It is not necessary to enter blanks between the groups of characters, they serve only to improve the readability on the license number sheet and can be entered if preferred. The dialog box is not case sensitive.

♣ Since the authorization code is rather long, we recommend you to enter the characters in pairs to avoid false entries.

(3) Enter your Hardlock number and authorization code and close the box with OK.

The main menu bar appears with only the System menu available..

(4) Click
System/Database Maintenance\Load Working Database...
The Load and Activate Working Database dialog box appears:



(5) To load an existing database: Select the desired source by clicking the corresponding text under Source. Then click Browse and navigate to the device or folder where the database is stored.

♣ You can reinstall a database of an earlier version of the STARe Software using the Load Working Database or the Load Active Database command. However, only databases created by STARe Software version 5.0 and higher can be loaded. Detailed information on the Load Working Database and Load Archive Database commands can be found in the online Help to the STARe Software.

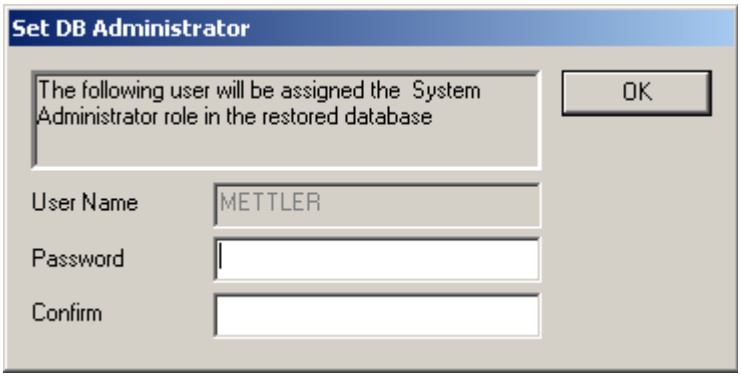
♣ If no database from an earlier version of the STARe Software is available you must load the original version of the database as described below.

or

To restore the original version of a database: Select Original ICTA (ICTA sign convention) or Original A-ICTA (Anti-ICTA sign convention).

(6) Click OK to load the database.

The database is loaded and the messages Decompressing zip file and Reloading database are displayed. When the load procedure is completed successfully the Set DB Administrator dialog box appears.



You must define a password for an administrator in the newly loaded database. The user name of this administrator is displayed in the User Name box but cannot be changed. The default administrator name is METTLER.

(7) Enter a password for the database administrator in the Password box and confirm it in the Confirm box.

The main menu bar appears. All the menus are now available..

(8) Close the STARe Software by clicking System/Exit...

The STARe Software is closed.

(9) Reboot your PC.

You have now completed the installation procedure of STARe Software version 9.00. If you have purchased any software options to the STARe Software you can install these now within the STARe Software. See Install Window Online Help to read how to install the software options.

On starting the STARe Software main menu bar a number of checks are performed automatically. Any problems that occur during these checks are recorded in a log file. The name of this log file begins with the text "STARe" and includes the date and time of its creation.

Example:

– STARe - 20050906_150709_6.log
the date format is "year, month, day" (2005, 09, 06)
the time format is "hours, minutes, seconds" (15:07h, 09.6 s)

3.9.2 Starting in a reduced operation state

If a valid Hardlock number and authorization code are entered but no working database is loaded or if, the S**TA**R^{e} Software starts in a reduced operation state in which only a limited number of tasks can be performed. On the main menu bar, the Functions and Service menus are not available and appear dimmed. On the System menu, however, the following commands are available:

- Switch between stand-alone and client operation mode **
- Enter a (different) base license
- Database maintenance operations:
  - Load a working database
  - Activate a working database
  - Show all databases loaded
  - Define database folder *

  * This command is not available in client operation mode.

  ** This command is also available when Hardlock is not inserted.

3.9.3 Reduced operation with archive databases

When a read-only archive database is selected, operation of the S**TA**R^{e} Software is also reduced. It is not possible to perform measurements and evaluations and to use the Backup… command. It is also not possible to save or import data using "Save" and "Import" commands in interactive windows. It is however possible to export data using "Export" commands. Furthermore, there is no need to log on to the S**TA**R^{e} Software on selection an archive database.

If you are using the software option User Rights, please note that the user right "Select Databases" is required to select an archive database.

If you are working in the 21 CFR 11 compliance mode please take note of the following:
Because an archive database is read-only, no entries are made in its audit trail when the 21 CFR 11 compliance mode is switched on. It is also not possible for any user to view the audit trail.

3.10 Installation of the PDF file creation tool

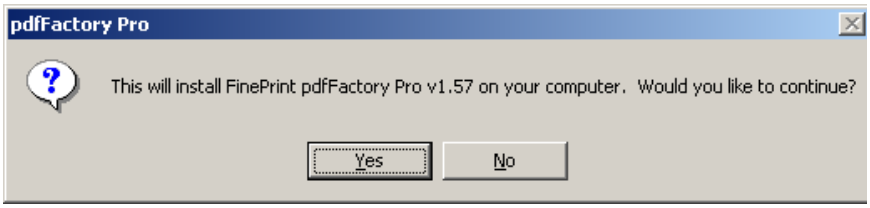
Please note: The FinePrint pdfFactory Pro PDF file creation tool is intended for use with the 21 CFR 11 Compliance software option. The license serial number required to use the tool is shipped only on purchase of the 21 CFR 11 Compliance software option. Therefore you need not install FinePrint pdfFactory Pro until you purchase the 21 CFR 11 Compliance software option.

To install FinePrint pdfFactory Pro:

(1) Insert the STARe CD-ROM in the CD-ROM drive.

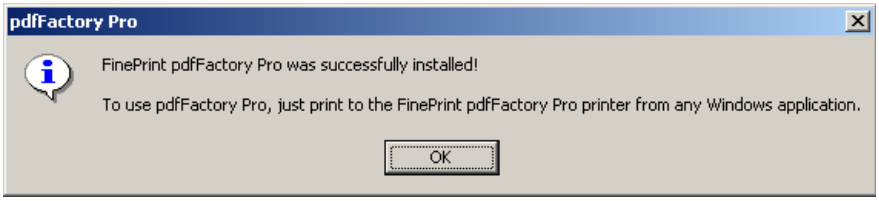
(2) Double-click the file fpppront157.exe in the folder pdfFactroy on the STARe CD-ROM.

The file fpppront157.exe is self-extracted. The following dialog box appears:



(3) Click Yes and then in the FinePrint pdfFactory Pro License Agreement dialog box click I accept the terms to accept the agreement and start the installation.

The FinePrint pdfFactory Pro is installed. At the end of the installation the following dialog box appears:



(4) Click OK.

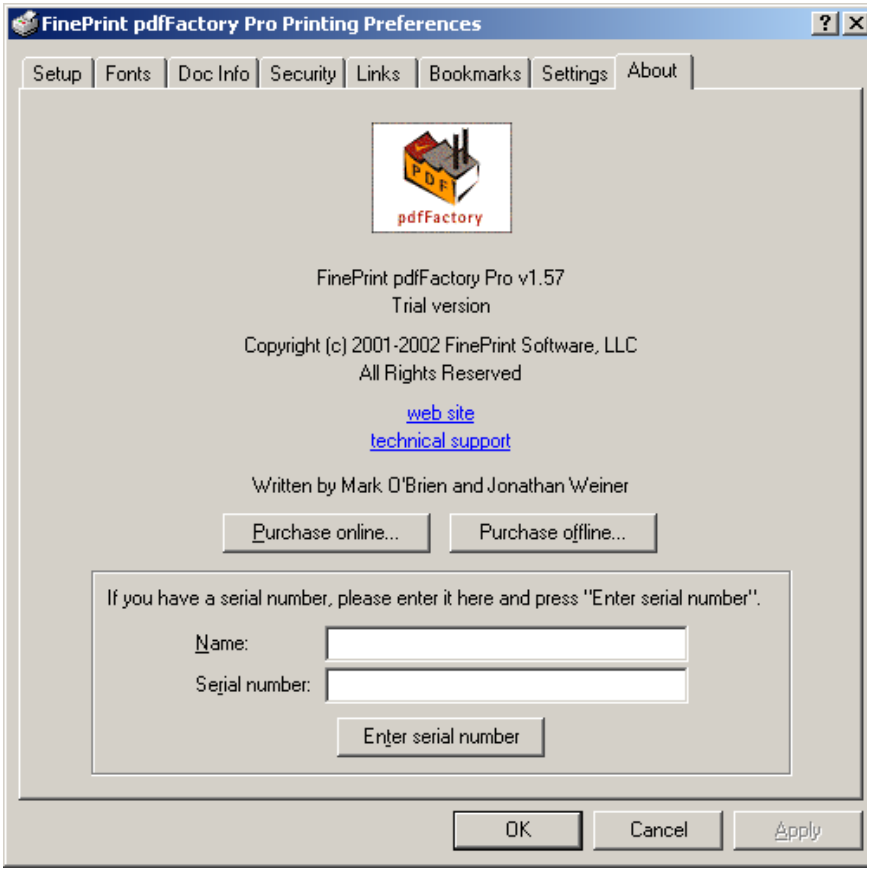
The installation is completed. You must now enter a serial number for the FinePrint pdfFactory Pro license as follows:

(5) Click Start/Settings/Control Panel to open Control Panel, double-click Printers, then right-click FinePrint pdfFactory Pro on the list of available printer devices and click Properties on the submenu.

The FinePrint pdfFactory Pro Printing Preferences dialog box appears.

(6) Click the About tab.

The About tabbed page appears.



♣ The license serial number is shipped on purchase of the 21 CFR 11 Compliance software option.

(7) Enter the serial number in the Serial Number box and your name in the Name box. Then click OK to confirm.

The installation of the FinePrint pdfFactory Pro tool is completed.

FinePrint pdfFactory Pro is a printer driver. This means you can print to it from your application, just as you would with any other printer. All STARe Software interactive windows except the Balance Control Window and the main menu bar have a Print... command available in the File menu. You can print STARe Software records to a PDF file (for example to meet requirements of 21 CFR Part 11) using the Print... command.

You can customize the security settings for the PDF files you create on the Security tab of the FinePrint pdfFactory Pro Printing Preferences dialog box. This is may be necessary to meet requirements of 21 CFR Part 11.

3.11 Uninstalling the STARe Software completely

The following procedure describes how you completely uninstall the STARe Software. Completely uninstalling the STARe Software means that all the software components including the STARe Software and the Ingres II 2.6 software are removed from your PC.

Backup your databases by using the backup function before you uninstall the STARe Software. The databases are destroyed on uninstalling of the STARe Software.

To uninstall the STARe Software and Ingres II 2.6 software completely:

(1) Log on to Windows as user ingres and start the STARe Software.

(2) Backup the current working database and all inactive working databases as follows: backup databases

- Backup the current working database by clicking System/Database Maintenance... on the main menu bar and using the Backup... command. Detailed information on Backup... can be found in the online Help to the STARe Software.
- Activate each inactive working database using the Activate... command on the System/Database Maintenance... submenu. Then backup the database with the Backup... command.

You have backed up all your databases.

(3) Quit the STARe Software by clicking System/Exit.

(4) Stop the Ingres Intelligent Database [II] service (See *Stopping the service for the Ingres II database management system, page 3-37* for more details).

The Ingres Intelligent Database [II] service is stopped.

(5) From Windows Control Panel, remove the S**TA**R**e** Software and the auxiliary software components as follows:

- Double-click Add-Remove Programs.
- Select the program in the list you want to remove and then click Change/Remove. Repeat this operation to remove the following programs.
  - STARe Software
  - Advantage Ingres [II] Enterprise Edition *
  - Hardlock (if on the list)
  - MATLAB Component Runtime
- Click OK and close Control Panel.

The S**TA**R**e** Software and its auxiliary software components have now been removed.

* Please take note of the following points when uninstalling the Ingres II 2.6 software: The Ingres II Uninstaller program will guide you through the uninstallation process. A dialog box with the message that one of the files cannot be deleted (for example the file plist p8219_1.dat) may appear. This is because the file is write-protected. In this case proceed as follows:

- Do **not** abort the Ingres Uninstaller program.
- Open the Windows Explorer or My Computer, navigate to the file and remove its write protection.
- Continue the uninstallation by clicking Retry in the Ingres Uninstaller program dialog box.
- At the end of the uninstallation process you should receive the message "Ingres successfully uninstalled".

(6) With Windows Explorer or in My Computer, delete all the folders of the S**TA**R**e** Software and its databases as well as the Ingres II folder from your hard disk. In the standard installation the default folders are as follows:

C:\STAReSW

C:\STAReDB

C:\IngresII

The S**TA**R**e** Software and its auxiliary software components have now been completely removed from your PC.

3.12 Updating the STAR^e Software from earlier versions 6.00, 6.01 or 6.10 to version 9.00

To update the STAR^e Software versions 6.00, 6.01 or 6.10 to version 9.00 all the previous software components including the database management software must be completely uninstalled. STAR^e Software version 9.00 can then be installed.

♣ Versions 6.00, 6.01 and 6.10 of the STAR^e Software run under Windows NT. Please note that this version of Windows is no longer supported with STAR^e Software version 8.00 and later. It is therefore necessary to update Windows NT to Windows 2000 or Windows XP within the following procedure.

Backup your databases with the backup function first before you perform an update. All databases of your STAR^e Software installation are destroyed when you perform an update.

backup databases

To update the STAR^e Software from version 6.00, 6.01 or 6.10 to version 9.00:

(1) Log off and log on to Windows as the user with whom you have previously installed the STAR^e Software (do not log on as user ingres).

(2) Backup all your databases as follows:

- Start the STAR^e Software and select each database with the command System/Database Selection... on the main menu bar.
- Backup each database by clicking Database Maintenance... and using the Backup command. Detailed information on Backup can be found in the operating instructions to your previous version of the STAR^e Software.

You have made backups of your databases.

(3) Log on to Windows as user ingres. Insert the CD-ROM of version 6.00, 6.01 or 6.10 of the STAR^e Software into the CD-ROM drive.

(4) Stop the OpenIngres Intelligent Database service as follows:

- Open Control Panel by selecting Start/Settings/Control Panel
- Start the program Services by double-clicking Services in the Control Panel window.

  The Services dialog box appears.
- Mark OpenIngres Intelligent Database in the list of services and stop this service by clicking Stop.
- Close the Services dialog box and Control Panel.

The OpenIngres Intelligent Database service is stopped.

(5) Remove the OpenIngres Intelligent Database service as follows:

- Click Start/Programs/Command Prompt to open the Command Prompt window.
- Enter the command opingsvc remove and press ENTER.

The OpenIngres Intelligent Database service is removed.

(6) Remove program components that are no longer required as follows:

- On the Start menu, click Settings/Control Panel to open Control Panel.
- Double-click Add/Remove Programs.

The Add/Remove Programs dialog is started.

- Click the Install/Uninstall tab.
- Using the Add/Remove button, remove the following programs from the list of installed programs.
  - NuTCRACKER Operating Environment
  - XVision
  - STARe Software
- Click OK and close Control Panel.

You have removed the program components that are no longer required.

(7) Using Windows Explorer or in My Computer, delete the following folders including their contents from your hard disk.:

C:\OPING

C:\CA_APPSW

C:\STAReSW

(8) Update Windows NT to Windows 2000 or Windows XP.

(9) Follow the instructions in sections 3.5 through 3.8 to install the Ingres II 2.6 software and the S**TA**R**e** Software.

(10) Reboot your computer and log on as a S**TA**R**e** Software user.

Your S**TA**R**e** Software version has now been updated to version 9.00. You can now continue with the procedure in section *First start of the STARe Software after installation, page 3-20*.

3.13 Updating the STAR^e Software from earlier versions 6.20 or 7.0x to version 9.00

To update the STAR^e Software versions 6.20 or 7.0x to version 9.00 all the previous software components including the database management software must be completely uninstalled. STAR^e Software version 9.00 can then be installed.

♣ Your Versions 6.20 and 7.0x of the STAR^e Software may be running under Windows NT. Please note that Windows NT is no longer supported with STAR^e Software version 8.00 and later. It is therefore necessary to update Windows NT to Windows 2000 or Windows XP within the following procedure in case the Version 6.20 or 7.0x of the STAR^e Software is installed on Windows NT.

Backup your databases with the backup function first before you perform an update. All databases of your STAR^e Software installation are destroyed when you perform an update.

backup databases

To update the STAR^e Software from version 6.20 or 7.0x to version 9.00:

(1) Log off and log on to Windows as the user with whom you have previously installed the STAR^e Software (do not log on as user ingres).

(2) Backup all your databases as follows:

- Start the STAR^e Software and select each database with the command System/Database Selection... on the main menu bar.
- Backup each database by clicking Database Maintenance... and using the Backup command. Detailed information on Backup can be found in the operating instructions to your previous version of the STAR^e Software.

You have made backups of your databases.

(3) Log on to Windows as user ingres. Insert the CD-ROM of version 6.00, 6.01 or 6.10 of the STAR^e Software into the CD-ROM drive.

(4) Stop the Ingres Intelligent Database [II] service as explained in section *Stopping the service for the Ingres II database management system, page 3-37*.

The Ingres Intelligent Database [II] service is stopped.

(5) From Control Panel remove program components that are no longer required as follows:

- Double-click Add-Remove Programs.
- Select the program in the list you want to remove and then click Change/Remove. Repeat this operation to remove the following programs:
  - MKS Platform Components
  - SCO XVision Eclipse
  - Ingres *
  - STARe Software
- Click OK and close Control Panel.

* Please take note of the following points when uninstalling the Ingres II 2.5 software: The Ingres II Uninstaller program will guide you through the uninstallation process. A dialog box with the message that one of the files cannot be deleted (for example the file plist p8219_1.dat) may appear. This is because the file is write-protected. In this case proceed as follows:

- Do **not** abort the Ingres Uninstaller program.
- Open the Windows Explorer or My Computer, navigate to the file and remove its write protection.
- Continue the uninstallation by clicking Retry in the Ingres Uninstaller program dialog box.
- At the end of the uninstallation process you should receive the message "Ingres successfully uninstalled".

(6) Using Windows Explorer or in My Computer, delete the folders of the STAR^e Software as well as the Ingres II, MKS Toolkit and XVision software including their contents from your hard disk. In the standard installation the default folders are as follows:

C:\CA_LIC

C:\IngresII

C:\STAReSW

(7) Update Windows NT to Windows 2000 or Windows XP.

(8) Follow the instructions in sections 3.5 through 3.8 to install the Ingres II 2.6 software and the STAR^e Software.

(9) Reboot your computer and log on as a STAR^e Software user.

Your STAR^e Software version has now been updated to version 9.00. You can now continue with the procedure in section *First start of the STARe Software after installation, page 3-20*.

3.14 Updating the STARe Software from earlier versions 8.00 or 8.01 to version 9.00

To update the STARe Software versions 8.00 or 8.01 to version 9.00 the database management software must be completely uninstalled. STARe Software version 9.00 can then be installed.

backup databases

Backup your databases with the backup function first before you perform an update. All databases of your STARe Software installation are destroyed when you perform an update.

To update the STARe Software from version 8.00 or 8.01 to version 9.00:

(1) Log on to Windows as the user ingres.

(2) Backup all your databases as follows:

- Start the STARe Software and select each database with the command System/Database Selection... on the main menu bar.
- Backup each database by clicking Database Maintenance... and using the Backup command. Detailed information on Backup can be found in the operating instructions to your previous version of the STARe Software.

You have made backups of your databases.

(3) Stop the STARe Ingres Control Service as explained in section *Stopping the service for the Ingres II database management system, page 3-37* .

The STARe Ingres Control Service is stopped.

(4) From Control Panel, remove the Ingres II 2.5 software as follows:

- ▸ Double-click Add/Remove Programs in the list.
- ▸ Select Ingres in the list and then click Change/Remove.

♣ Please take note of the following points when uninstalling the Ingres II 2.5 software: The Ingres II Uninstaller program will guide you through the uninstallation process. A dialog box with the message that one of the files cannot be deleted (for example the file plist p8219_1.dat) may appear. This is because the file is write-protected. In this case proceed as follows:

- Do **not** abort the Ingres Uninstaller program.
- Open the Windows Explorer or My Computer, navigate to the file and remove its write protection.
- Continue the uninstallation by clicking Retry in the Ingres Uninstaller program dialog box.
- At the end of the uninstallation process you should receive the message "Ingres successfully uninstalled".

- ▸ Click OK and close Control Panel.

(5) Using Windows Explorer or in My Computer, delete the folders of the Ingres II 2.5 software including their contents from your hard disk. In the standard installation the default folders are as follows:

C:\CA_LIC

C:\IngresII

(6) Follow the instructions in sections 3.5 through 3.8 to install the Ingres II 2.6 software and the STARe Software.

(7) Reboot your computer and log on as a STARe Software user.

Your STARe Software version has now been updated to version 9.00. You can now continue with the procedure in section *First start of the STARe Software after installation, page 3-20*.

3.15 Updating STARe Software version 8.10 to version 9.00

To update the STARe Software version 8.10 to version 9.00, the Ingres II 2.6 software installation can be retained. However, all databases of your STARe Software installation will be destroyed when you perform the update.

backup databases

Backup your databases with the backup function first before you perform an update. All databases of your STARe Software installation are destroyed when you perform an update.

To update the STARe Software from version 8.10 to version 9.00:

(1) Log on to Windows as the user ingres.

(2) Backup all your databases as follows:

- Select each database with the command System/Database Selection... on the main menu bar of the STARe Software.
- Backup each database by clicking Database Maintenance... and using the Backup command. Detailed information on Backup can be found in the operating instructions to your version of the STARe Software.

You have made backups of your databases.

(3) Follow the instructions in section 3.7 to install the STARe Software.

(4) Reboot your computer and log on as a STARe Software user.

Your STARe Software version has now been updated to version 9.00. You can now continue with the procedure in section *First start of the STARe Software after installation, page 3-20*

3.16 Stopping the service for the Ingres II database management system

The service for the Ingres II database management system must be stopped in several steps of installation, uninstallation or update procedures of the STARe Software. This service is named Ingres Intelligent Database [II] or STARe Ingres Control Service depending on which version of the STARe Software you have previously installed. The service is stopped with the Services program in Windows Control Panel as follows:

(1) Click Start/Settings/Control Panel to open Control Panel, double-click Administrative Tools and then Services on the corresponding lists.

(2) Mark Ingres Intelligent Database [II] or STARe Ingres Control Service in the list of services and stop this service by clicking Action/Stop.

(3) Close the Services dialog box.

The service for the Ingres II database management system has been stopped.

3.17 Installing and repairing components of the STARe Software

You can install auxiliary components of the STARe Software you have not installed previously during the initial installation without having to reinstall the whole software. (The reinstallation of the entire STARe Software is however necessary if you want to change the installation folder as explained in section *Changing the installation folder, page 3-43*.)

You can also repair components of the STARe Software. If, for example, the Hardlock software is not working properly you can repair the Hardlock installation or you can rectify wrong environment variables or registry settings in Windows if they have been changed.

To install auxiliary software components

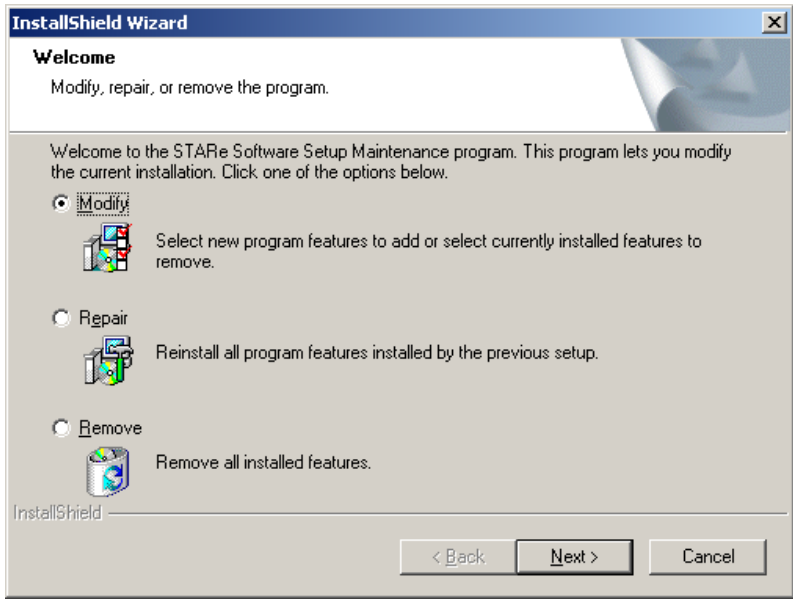
(1) Log on to Windows as user ingres.

(2) Insert the STARe CD-ROM in the CD-ROM drive.

(3) Select the CD-ROM drive from My Computer or the Windows Explorer.

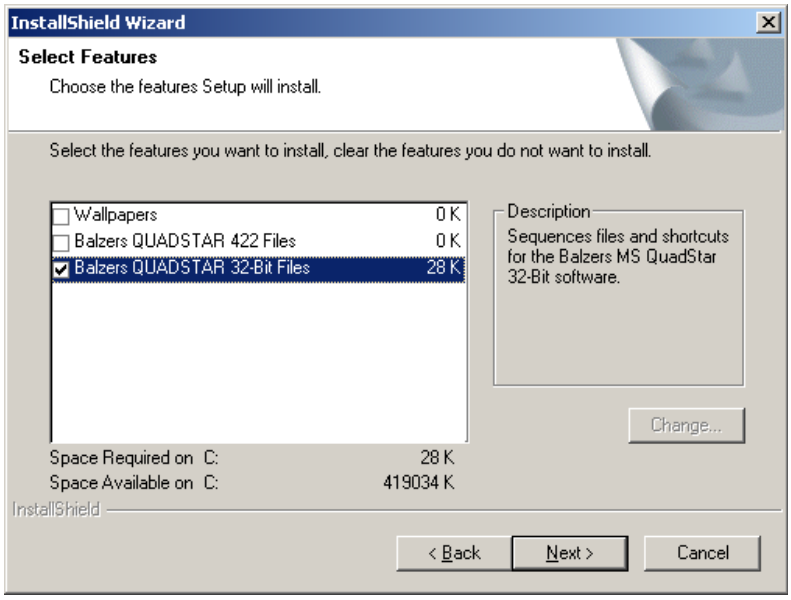
(4) Double-click the setup.exe file to start the installation.

The Welcome dialog box of the InstallShield Wizard installation program appears.



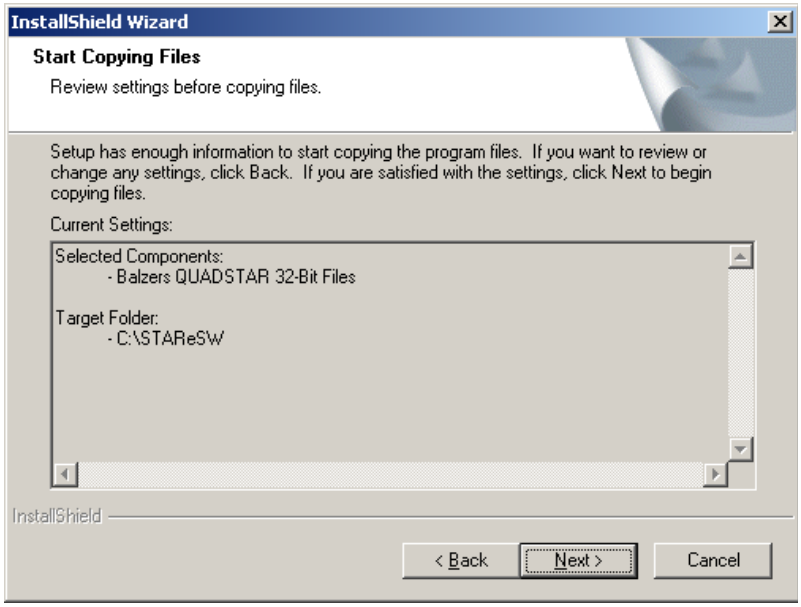
(5) Click the Modify option and then Next>.

The Select Features dialog box for the selection of the software components appears.



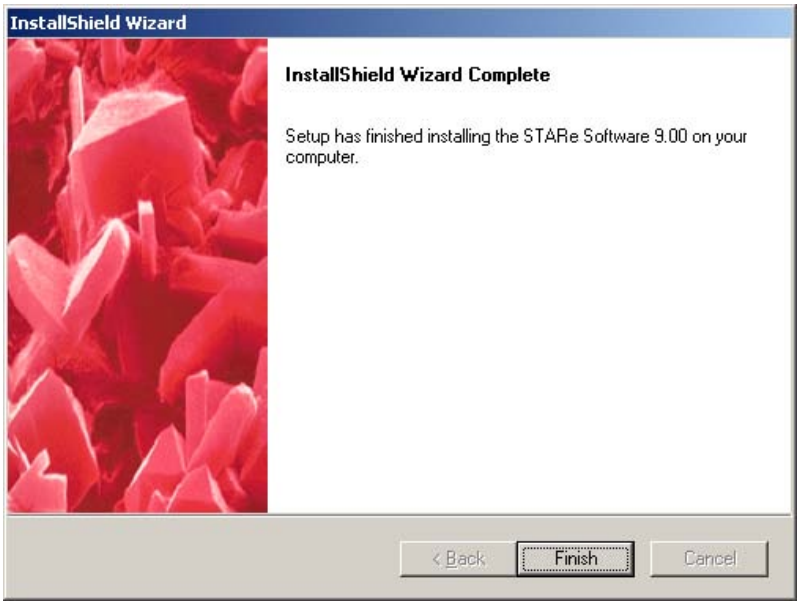
(6) Select the items you want to install by selecting the corresponding check boxes (the above screen shot shows an example).

The Start Copying Files dialog box appears. You can review or change the settings by clicking Back>.



(7) If you are satisfied with the settings, click Next> to start copying the files.

The Setup Status dialog box appears showing the progress of the installation. The components are automatically installed. Please wait for the following dialog box to appear:



(8) Click Finish to conclude the setup program.

The auxiliary software components of the S**TA**R^{e} Software are installed.

You have now installed the selected auxiliary software components.

To repair components of the STARe Software:

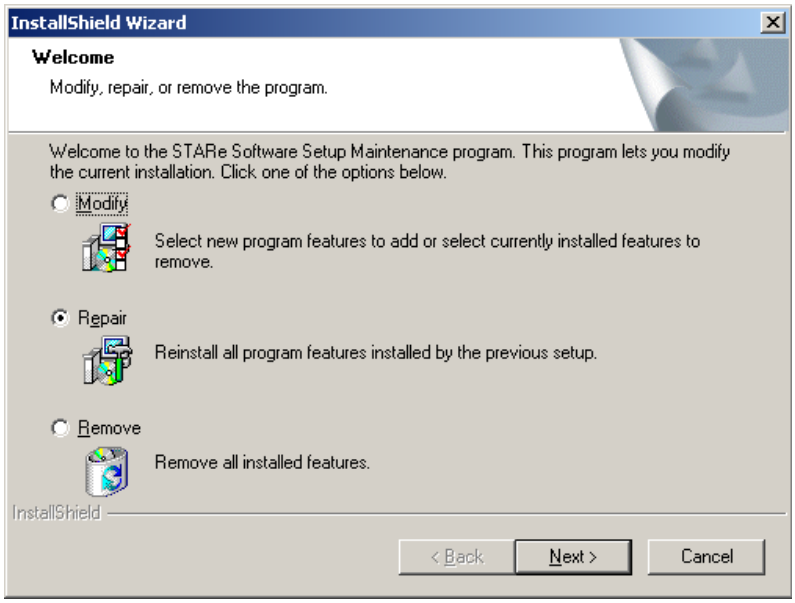
(1) Log on to Windows as user ingres.

(2) Insert the STARe CD-ROM in the CD-ROM drive.

(3) Select the CD-ROM drive from My Computer or the Windows Explorer.

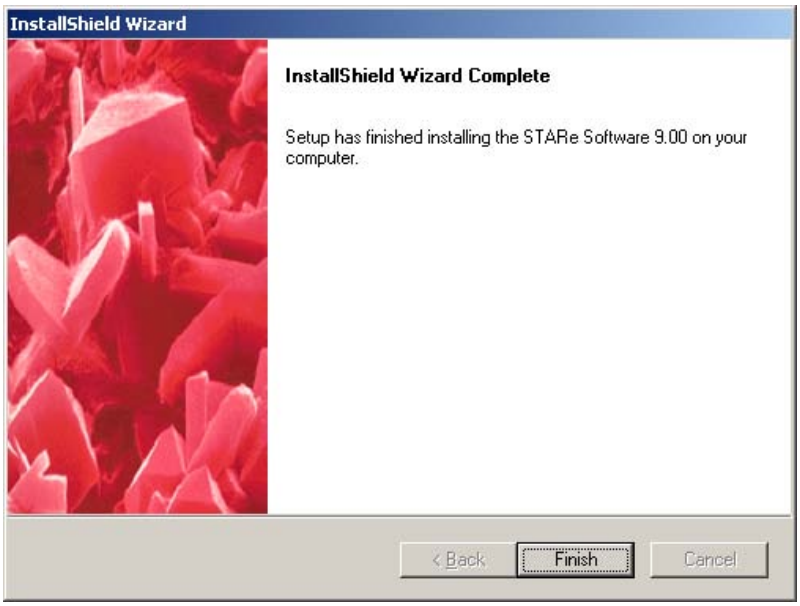
(4) Double-click the setup.exe file to start the installation.

The Welcome dialog box of the InstallShield Wizard installation program appears.



(5) Click the Repair option and then Next>.

The Setup Status dialog box appears showing the progress of the installation. The Hardlock software is automatically installed. Please wait for the following dialog box to appear:



(6) Click Finish to conclude the setup program.

You have repaired components of the S**TA**R^{e} Software.

3.18 Changing the installation folder

If you have already installed the STARe Software but want to change the installation folder you must reinstall the STARe Software (but not the Ingres II 2.6 SP2 database software).

The STARe Software configuration files are copied to the sys subfolder of the new installaton and reused.

Backup your databases by using the backup function before you uninstall the STARe Software.

backup databases

(1) Log on to Windows as user ingres.

(2) Backup the current working database and all inactive working databases as follows:

- Backup the current working database by clicking System/Database Maintenance... on the main menu bar and using the Backup... command. Detailed information on Backup... can be found in the online Help to the STARe Software.
- Activate each inactive working databases using the Activate... command on the System/Database Maintenance... submenu. Then backup the database with the Backup... command.

You have backed up all your databases.

(3) With Windows Control Panel, remove the STARe Software as follows:

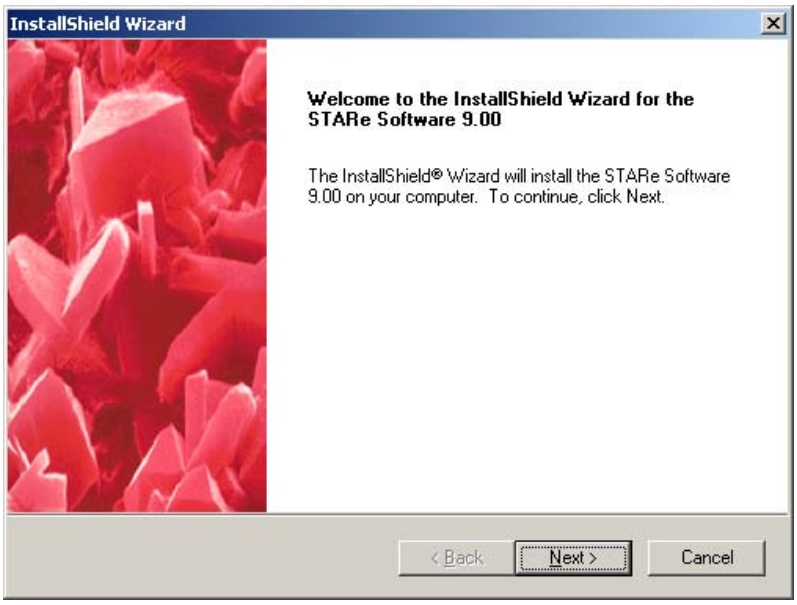
- Click Start/Settings/Control Panel to open Control Panel and double-click Add-Remove Programs in the list.
- Select the STARe Software program in the list and then click Change/Remove.
- Click Yes to confirm.

(4) Insert the STARe CD-ROM in the CD-ROM drive.

(5) Select the CD-ROM drive from My Computer or the Windows Explorer.

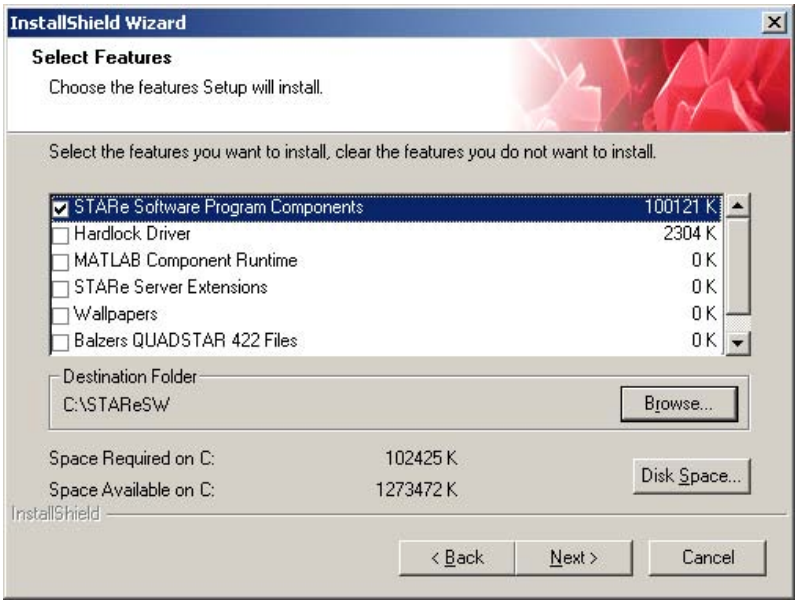
(6) Double-click the setup.exe file to start the setup program for the installation.

The initial dialog box of the InstallShield Wizard installation program appears.



(7) Confirm the License Agreement box with Yes after reading the agreement text.

The Select Features dialog box for the selection of the software components appears.



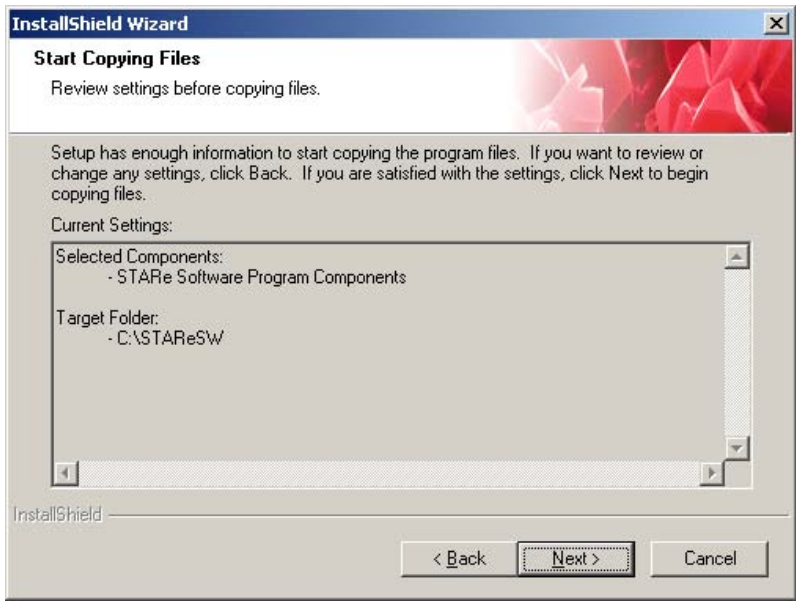
The setup installation program automatically recognizes the software components that need to be installed. Only the feature STARe Software Program Component is selected here because the other features are already installed.
The folder of your current STARe Software installation is indicated under Destination Folder.

Please note that not more than **80 characters** are allowed in the path and folder name.
Do not install the STARe Software on a network drive. The software will not run on a network drive.

---

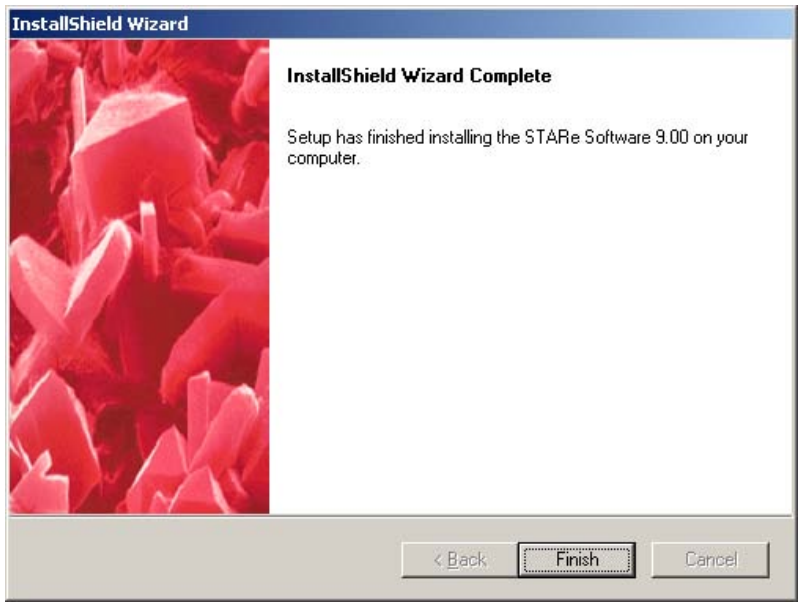
(8) Navigate to the desired folder with Browse... and confirm with Next>.

The Start Copying Files dialog box appears. You can review or change the settings by clicking Back>.



(9) If you are satisfied with the settings, click Next> to start copying the files.

The Setup Status dialog box appears showing the progress of the installation. The S**TA**R^{e} Software is automatically installed. Please wait for the following dialog box to appear:



(10) Click Finish to conclude the setup program.

The components of the STARe Software are installed.

♣ We recommend you to reboot your PC at the end of the STARe Software installation procedure.

The change of the STARe Software installation folder is now completed. You must now restore the STARe Software configuraion files.

(11) Restore the STARe Software configuration files by copying all files from the old sys subfolder of your old installation into the sys subfolder of your new installation.

If you do not have a licenses file available, you must reenter the your authorization code for the base software and your Hardlock number (see *First start of the STARe Software after installation, page 3-20*).

The authorization codes for the STARe Software options you have purchased and installed previously are also included in the licenses file. You must reenter them in the STARe Software Install Window if your licenses file is no longer available (see Install Window online Help for more details).

(12) Delete the folder of your old STARe Software installation (in the default installation this folder is named C:\STARe-SW).

You have now changed the installation folder of the STARe Software and can start and use the STARe Software again.

3.19 Changing the host name of your PC

Changing the host name of your PC will require a few changes to the Ingres II 2.6 installation so that Ingres II 2.6 will still start up properly and can be used by the S**TA**R**e** Software. To make the changes you have perform the following steps.

♣ Changing the IP address of your PC after you have installed Ingres II 2.6 will not have any effect on the Ingres II 2.6 installation.

To configure Ingres II for the new hostname

(1) Log on to Windows under the user name ingres.

(2) Stop the Ingres Intelligent Database [II] service (See *Stopping the service for the Ingres II database management system, page 3-37* for more details).

(3) Open the Command Prompt window in Windows.

(4) Enter the following command at the command prompt:
ingsetenv II_GCNII_LCL_VNODE <NEWHOSTNAME>
where <NEWHOSTNAME> stands for the new host name that must be entered here.

(5) Open the config.dat file in the C:\IngresII\ingres\files folder in a text editor and replace all the text strings containing the old host name by text strings containing the hew host name text string. Use the Find/Replace function of the text editor to do this.

♣ On non-standard Ingres II installations the config.dat file is located in the corresponding subfolder of the Ingres II installation folder.

(6) In the folder C:\IngresII\ingres\files\name, rename all _<OLDHOSTNAME> file name endings to _<NEWHOSTNAME>. <OLDHOSTNAME> refers to the previously defined host name and <NEWHOSTNAME> to the new host name.
If there are existing files with a _<NEWHOSTNAME> ending, delete them before renaming any files with a _<OLDHOSTNAME> ending.

(7) Start the Ingres Intelligent Database [II] service again (as explained in section *Configure and startup of the Ingres Intelligent Database [II] service, page 3-12*), in step 5.

Ingres II has now been configured for the new hostname.

3.20 Setting up a STARe network

The following procedures explain how to set up a STARe network to operate the STARe Software in a client/ server configuration. A descripton of such a configuration is given in chapter "What's new in version 9.00?" in section "Client/server operation of the STARe Software". The following procedures show you how to set up the STARe server and the STARe clients.

To set up the STARe server:

(1) Insert the green USB Hardlock dongle in the USB port of your computer.

♣ The black parallel-port Hardlock dongle cannot be used to set up a STARe network.

(2) Follow the instructions in sections 3.5 through 3.8 to install the Ingres II 2.6 software and the STARe Software. Be sure to include the feature STARe Server Extensions in the Select Features dialog box of the setup program.

♣ The time zone setting for the Ingres II software must be the same on the STARe server and all STARe clients. The time zone for Ingres II must be set during installation as explained on page 3-8. Also, the computer time setting must be the same throughout the STARe network.

♣ STARe Software setup must be allowed to create certain exceptions in Windows Firewall on Windows XP SP2 or Windows Server 2003 SP1 installations. If the STARe Software cannot create or activate these exceptions (for example because of the limitation on group policies), the exceptions must be configured manually as explained below.

To configure the Windows firewall exceptions manually:

- Enable the exception File and Printer Sharing.
- Create an exception for the Hardlock service:
  Allow communication with the program C:\WINDOWS\system32\hls32svc.exe
- Create an exception for Ingres/Net:
  Allow communication with the program %II_SYSTEM%\ingres\files\iigcc.exe.
  or:
  Allow communication with the TCP port 21064.

♣ The STARe Software creates a network share STAReSYS$ which STARe clients require to access global configuration files. Any user is allowed to access this share. From within a Microsoft Windows network, however, it is not visible as a shared folder.

(3) Increase the limit on the number of STARe Software applications as described in the section *Increasing the limit on the number of STARe Software applications, page 3-51*.

(4) Start the STARe Software by following the procedure in section *First start of the STARe Software after installation, page 3-20*. Be sure to enable the standalone operation mode of the STARe Software.

To set up a STARe client:

(1) Follow the instructions in sections 3.5 through 3.8 to install the Ingres II 2.6 software and the STARe Software.

- The time zone setting for the Ingres II software must be the same on the STARe server and all STARe clients. The time zone for Ingres II must be set during installation as explained on page 3-8. Also, the computer time setting must be the same throughout the STARe network.
- STARe Software setup must be allowed to create certain exceptions in Windows Firewall on Windows XP SP2 or Windows Server 2003 SP1 installations. If the STARe Software cannot create or activate these exceptions (for example because of the limitation on group policies), the exceptions must be configured manually as explained below.
  - Enable the exception File and Printer Sharing.

(2) Start the STARe Software and switch to client operation mode. Specify the host name of the STARe server computer.

- No Hardlock dongle is required to operate the STARe Software in client operation mode.
- The STARe client must establish a connection to the STARe server. If both computers are in the same Windows domain and the user is logged on to Windows on the STARe client as a domain Windows user, the connection can be established automatically. Otherwise, the connection must be established manually, for example with the command net use \\<STARe server host name> /user:<windows user known to the STARe server computer>.

3.21 Increasing the limit on the number of STARe Software applications

To set up a STARe Software version 9.00 installation as a STARe Server, the Ingres II database management system has to be configured so that a larger number of STARe Software applications can access the database simultaneously.

To configure the DBMS for a larger number of STARe Software applications:

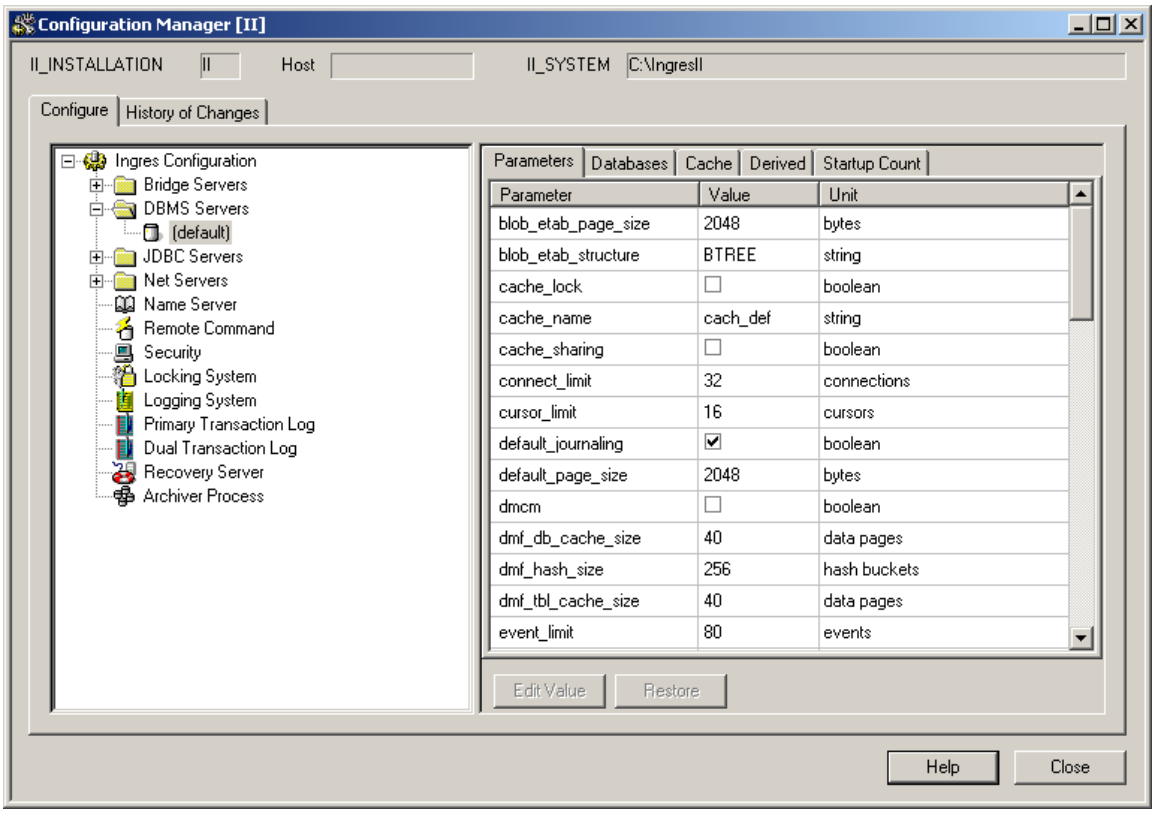
(1) Make sure that the STARe Software is not running. Close it if necessary.

(2) Stop the Ingres Intelligent Database [II] service as explained in section *Stopping the service for the Ingres II database management system, page 3-37*.

The Ingres Intelligent Database [II] service is stopped.

(3) Start the Ingres II Configuration Manager by clicking Start/Programs/Ingres II [II]/Ingres Configuration Manager.

The Configuration Manager [II] dialog box appears.

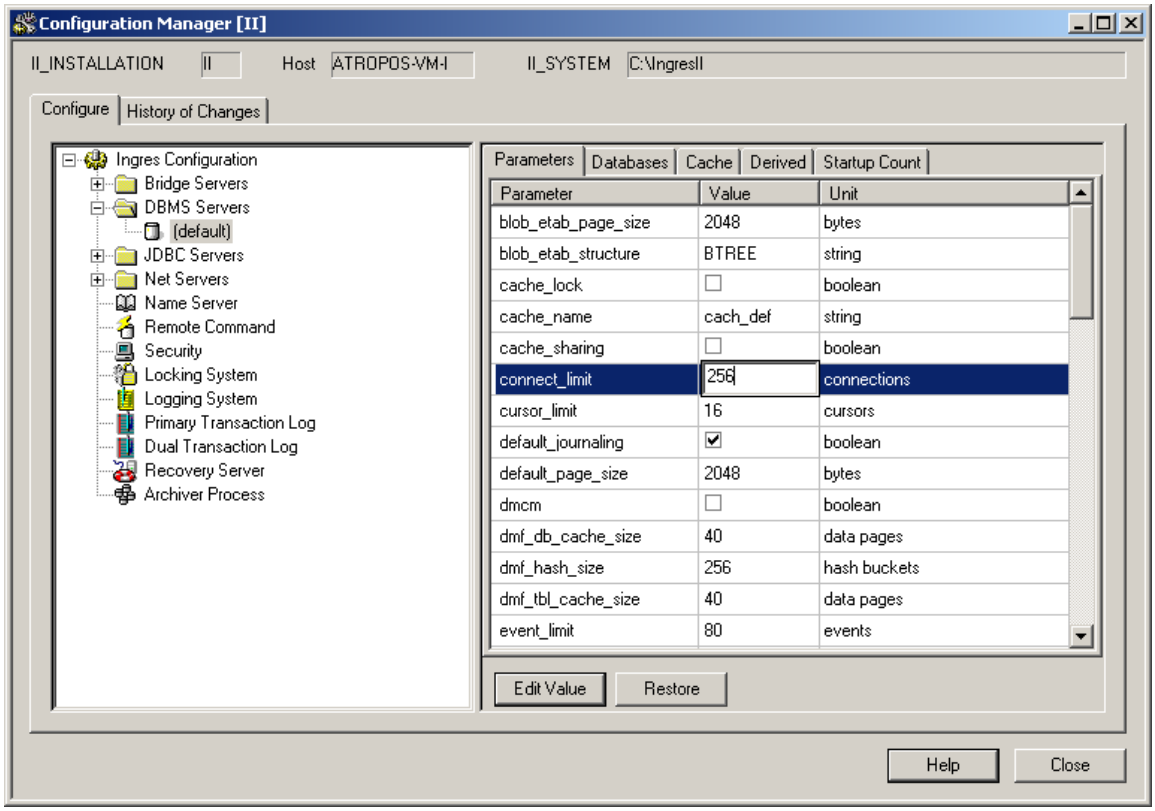


(4) In the left pane, open the DBMS Servers folder and click (default).

(5) In the list in the right pane, click the entry connect_limit.

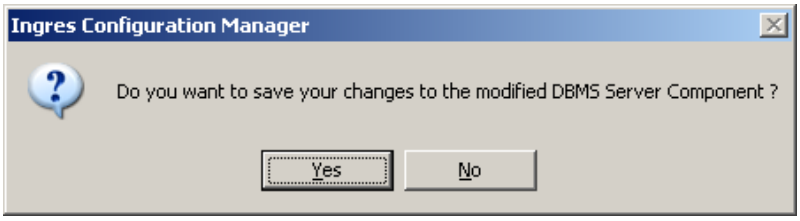
(6) Click Edit Value and the enter 256 as the new value.

♣ If the dialog box is small so that not all of the buttons in the right pane are visible, you can move the border in the middle to the left to make them accessible.



(7) Click Close to close the Configuration Manager [II] dialog box by clicking Close.

A following dialog appears.



(8) Clicking Yes to confirm.

(9) Restart the Ingres Intelligent Database [II] service as explained in section *Configure and startup of the Ingres Intelligent Database [II] service, page 3-12*, in step 5.

The DBMS is now configured for a larger number of STARe Software applications.

3.22 Installing the STARe Software on Terminal Server

Windows Terminal Services offers the possibility of running several Windows sessions simultaneously in so-called Terminal Server client sessions. The STARe Software can be run in each client session. The workings of this is explained in section *Terminal Server* of the chapter *What's new in version 9.00?*

Since we consider it the user's responsibility to install Terminal Server this is not explained here. To run the STARe Software on Terminal Server, the following points have to be considered additionally.

Operating Systems

One of the following operating systems is required:

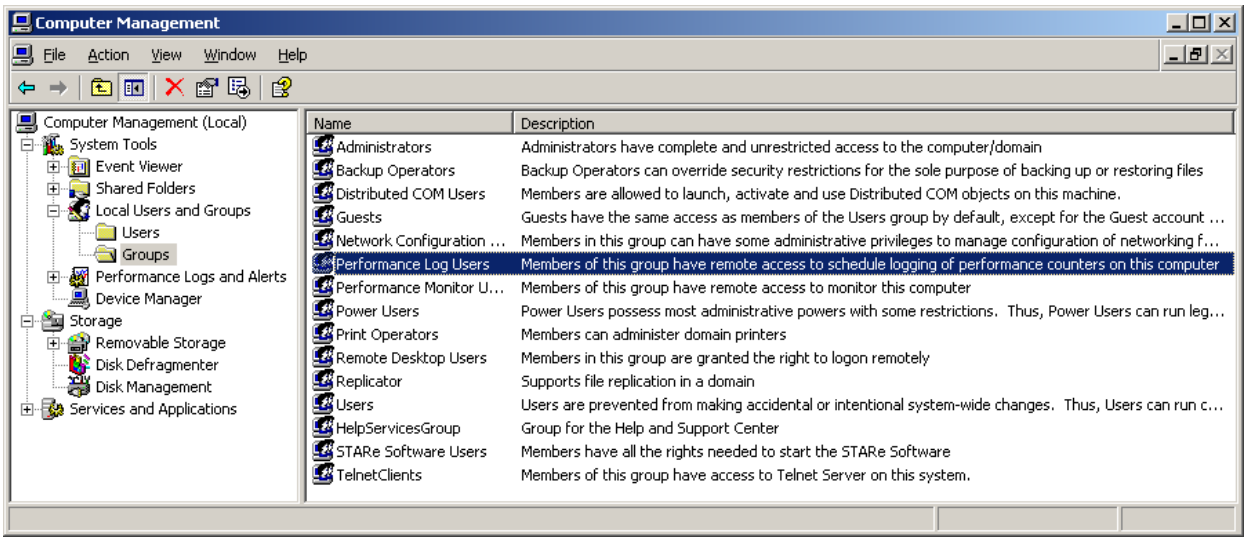
- Windows 2000 Server with Terminal Services and SP4 or later

or

- Windows Server 2003, any Edition (the STARe Software has been tested on the Windows Server 2003 Standard Edition)

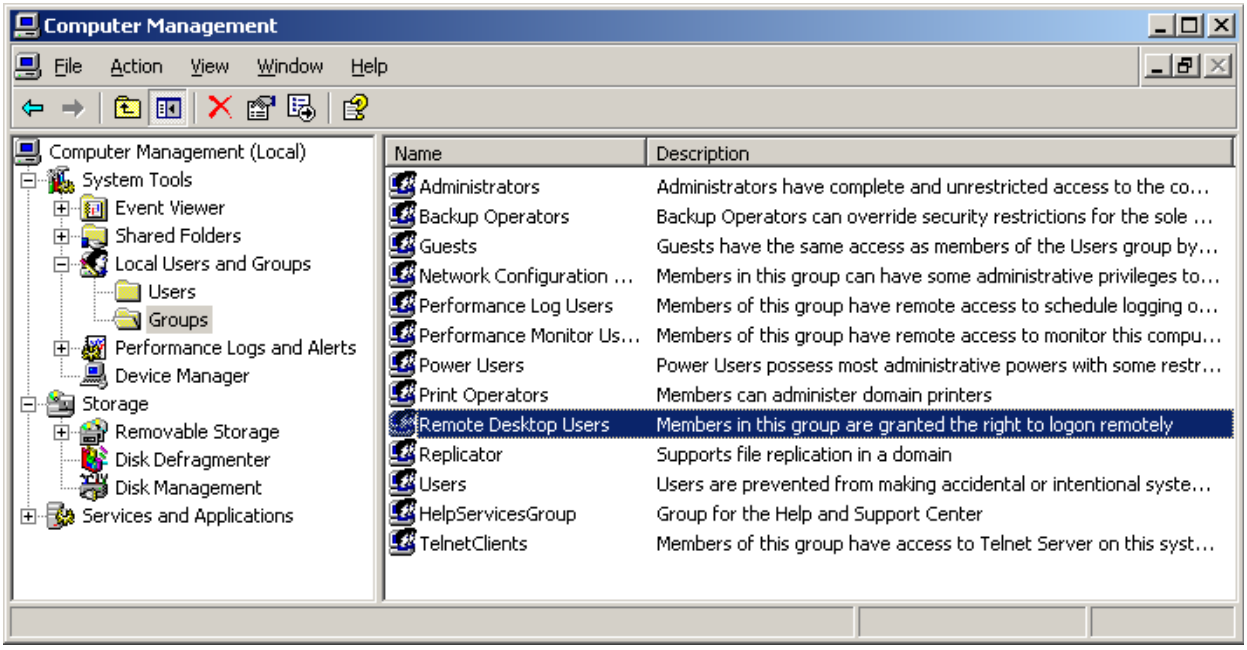
Windows Performance Data Access

Windows Server 2003 only: All Windows users using the STARe Software must have access to the Windows performance data. To grant Windows users access to the performance data, you must include them in the Performance Log Users group in the Computer Manager dialog box shown below.

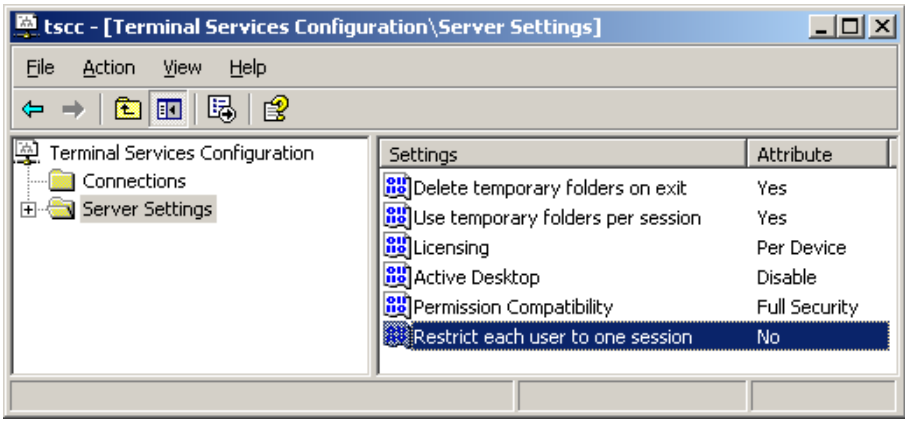


Log on with a Remote Desktop Connection (Windows Server 2003 only)

In Windows Server 2003, Terminal Server is accessed via a Remote Desktop Connection. All Windows users or user groups using the STARe Software must be included in the Remote Desktop Users group. You can do this in the Computer Mangement dialog box shown below.



To allow each Windows user to run more than one remote session at a time, make the following setting in the Terminal Services Configuration dialog box.



STARe Software installation procedure

The installation procedure of the STARe Software on Terminal Server is almost the same as on a standard Windows operating system. The only difference is that a special install mode must be activated in Terminal Server to install the STARe Software. This is done by using the **Add/Remove Programs** feature in Windows Control Panel. The setup program of the STARe Software must therefore be started using the **Add/Remove Programs** feature in Windows Control Panel as explained in the following.

- ♣ The **Add/Remove Programs** feature is named **Add or Remove Programs** in Windows Server 2003.
- ♣ Windows Server 2003 detects programs called setup.exe and activates the install mode automatically. However, Microsoft recommends using the **Add or Remove Programs** feature in Control Panel to install applications on Windows Server 2003.

To start the setup program Terminal Server:

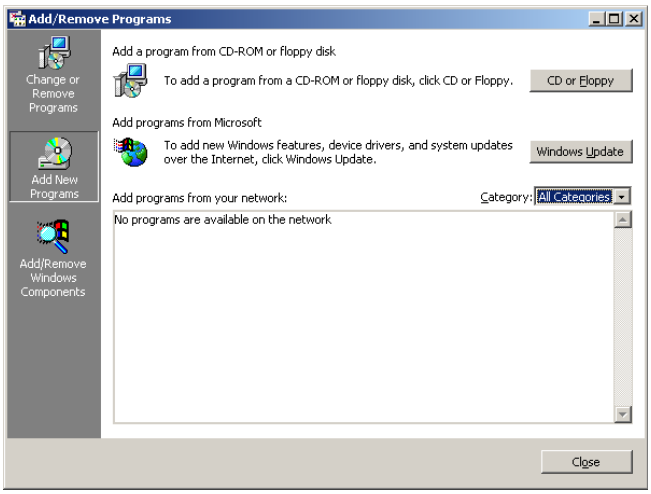
(1) On the Start menu, click Settings/Control Panel to open Control Panel.

(2) In **Windows 2000 Server**: Double-click Add/Remove Programs.

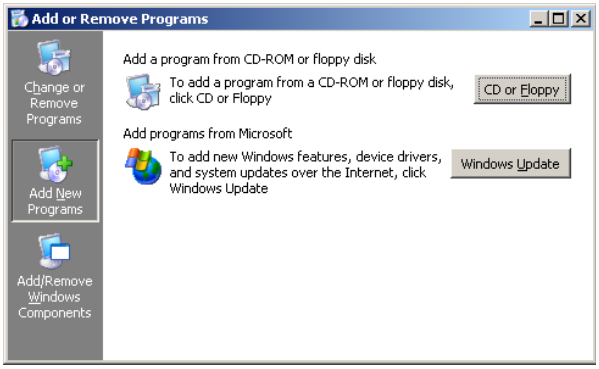
or

In **Windows Server 2003**: Double-click Add or Remove Programs.

The Add/Remove Programs or Add or Remove Programs dialog box appears.



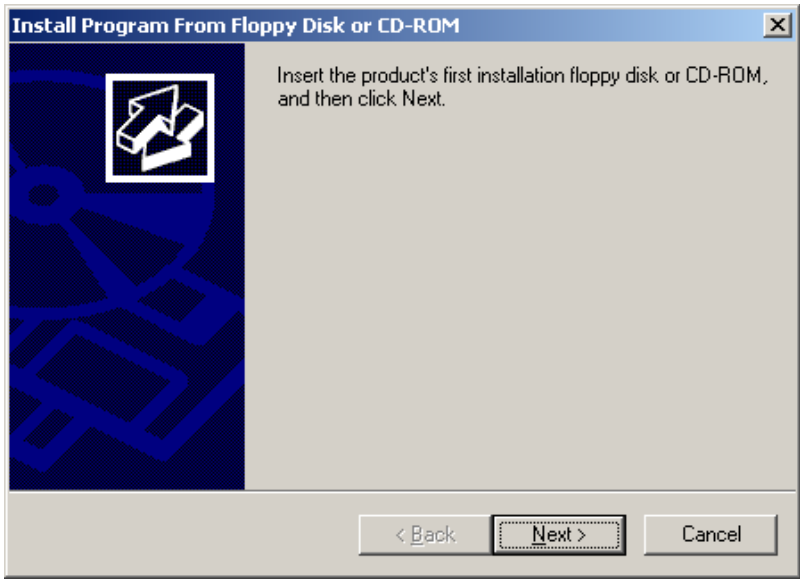
Windows 2000 Server



Windows Server 2003

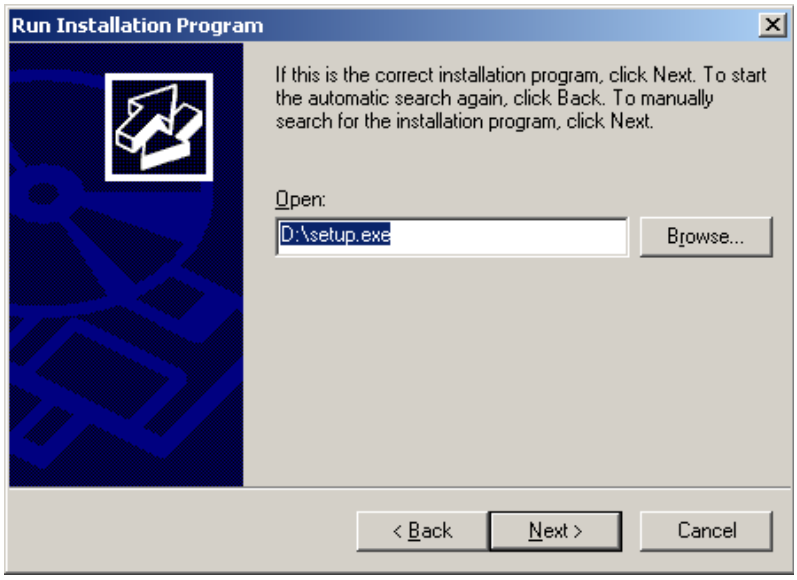
(3) Click Add New Programs and then click CD or Floppy.

(4) The installation wizard starts and the following dialog box appears:



(5) Click Next.

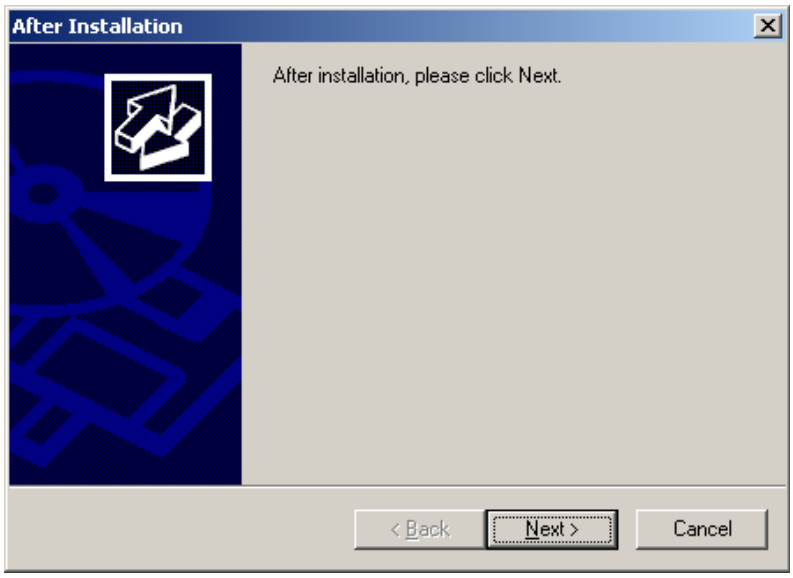
The following dialog box appears in which the name of the setup program is entered in the Open box.



♣ The installation wizard searches the floppy disk drive and CD-ROM drive for the STARe Software setup program. If it fails to find the set-up program file, you must click Browse and navigate to it manually.

(6) Click Next.

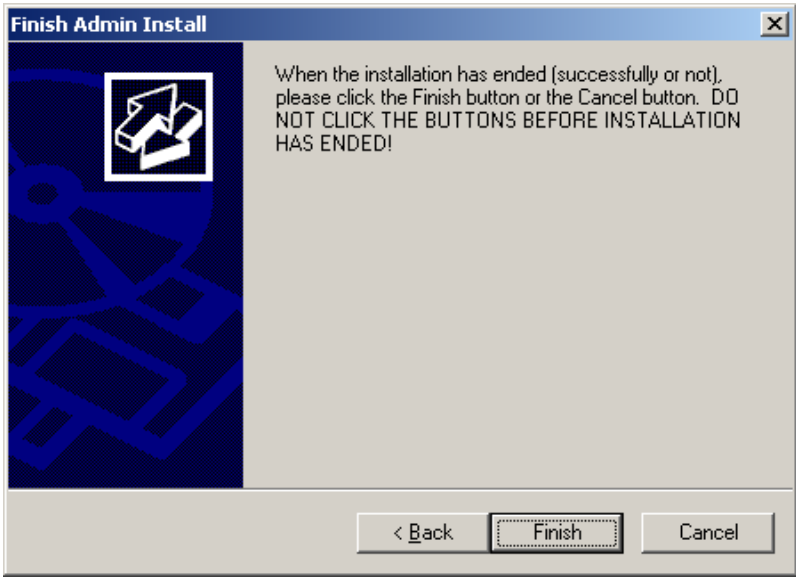
The following dialog box appears and the STARe Software setup program starts.



You must not click Next in this dialog box until the installation is completed.
You can now proceed accordingly as explained in chapter *Installation of the STARe Software, page 3-15*.

(7) Click Next when the installation is completed.

At the end of the installation the following dialog box appears. It includes a text requesting you to click Finish when the installation has ended. You must not click Finish before the installation has ended.



4 Starting and Exiting the STARe Software

4.1 Starting the STARe Software

(1) Switch on the devices in the following order:
Printer, PC, screen, TA module.
The main switch is at the rear in the center of your TA module. If you do not need the module immediately, you can switch it on later.

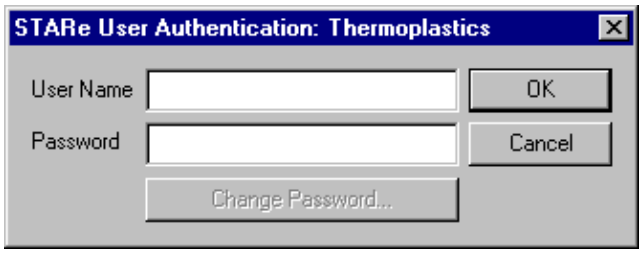
(2) Log on to Windows with your user name and password.

(3) Ensure that you have the correct language setting in Windows: If necessary, start Windows Control Panel and select under Regional Settings the language English. (You can select any version of English).

select language version

(4) Double-click the STARe Software icon on the Windows desktop.

The STARe User Authentication dialog box appears:



(5) Enter your user name and password and click OK.

The STARe Software main menu bar appears.

♣ If you start the S**TA**R^{e} Software for the first time after the installation, you must enter the authorization code. See the chapter *Installation* Instructions, section *First Start of the S**TA**R^{e} Software after the installation*.

Install Plus

♣ With the **Install Plus** software option you can define additional user accounts so that multiuser operation is possible.

Interactive window

(6) Open the desired interactive window by clicking the corresponding command on the Functions menu in the main menu bar.

With the base software the following menu commands are available:

- Evaluation Window
- Install Window

If the software options “Method Window“ or “Experiment Window“ are installed, the following menu commands are available, respectively:

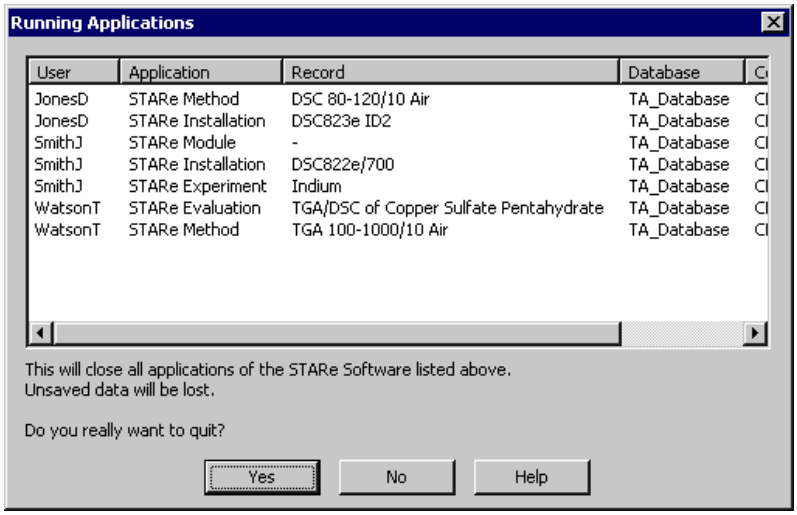
- Method Window
- Experiment Window

4.2 Quitting the STARe Software

Before quitting the STARe Software you should ensure that no measurements are running. If there are still measurements running you can either wait until the measurement is completed or terminate the measurement prematurely as described below in section *Terminating an experiment before quitting the STARe Software, page 4-4*.

(1) Click System/Exit... in the main menu bar.

The Running Applications dialog box appears. It contains a list of all interactive windows that are still open.



(2) Click Yes.

The STARe Software application is closed. All open interactive windows are closed.

If you quit the STARe Software during measurements the current measurement is reset. The part of the curve already measured is saved (except if the Save Curve check box is not selected under Control/Save Curve... in the Module Control Window).

4.3 Terminating an experiment before quitting the STARe Software

terminate experiment

To quit the STARe Software, it might be necessary to terminate an experiment prematurely with the RESET-key on the keypad of your TA module or the Reset button in the corresponding Module Control Window. The measurement curve is stored up to the termination point. The curve is not stored if the Save Curve check box is cleared in the Configuration dialog of the Module Control Window (this dialog box can be called up by clicking Control/Configuration... in the Module Control Window).

If you terminate a measurement before 10 curve points have been measured (for example with the default sampling interval of 1 s, within the first 10 seconds of the current measurement), the respective experiment returns to the first position in the queue under Experiments pending. You can start the experiment again with Start Experiment command on the Control menu.

5 End-User License Agreement

End-User License Agreement

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5. LIMITED WARRANTY FOR THE HARDLOCK

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Failure to comply with these terms and conditions will result in loss of support from METTLER TOLEDO and may lead to legal action.

6 Sofware Options and Accessories

Standard Accessories	Order Number
• STARe Software CD-ROM (base software)	51 141 630
• STARe Software User Guide, english	51 710 258
• STARe Software User Handbook, english	51 710 263
Optional Accessories	
• Operating Instructions, DSC823e, english	51 710 377
• Operating Instructions, HP DSC827e, english	51 710 242
• Operating Instructions, TGA/SDTA821e, english	51 709 673
• Operating Instructions, TMA/SDTA840, english	51 709 740
• Operating Instructions, TMA/SDTA841e, english	51 710 315
• Operating Instructions, DMA/SDTA861e, english	51 710 069
• TC15 TA Controller	709 188
• RS232C cable, 9 pins,female with bi-directional handshake to connect an AT/MT/UM - Balance to the PC	210 493
• Assembly instructions for the RS232C cable	704 044

Software Options	Order Number
• ADSC	51 119 679
• ADSC cp	51 140 657
• DSC Evaluations	119 457
• TOPEM®	51 141 971
• DSC Purity	51 119 492
• Mathematics	51 140 658
• Specific Heat	119 458
• TGA Evaluations	119 459
• 21 CFR 11 Compliance	51724329
• Advanced Model Free Kinetics	51141434
• Auto Evaluations and Validation	119 462
• Conditional Experiment Termination	119 463
• DMA Evaluations	51141351
• Experiment Window	51 119 875
• Install Plus	51 119 877
• IsoStep™ DSC	51141466
• Kinetics, nthOrder	51 119 493
• MaxRes	51 140 478
• Method Window	51 119 874
• Model Free Kinetics	51 119 664
• Multi Module Operation	119 461
• Relative Loop	51 119 494
• Routine Window	51 119 876
• TMA Evaluations	51 119 460
• User Rights	51724330
• Server Mode	51141647
• Concurrent Users	51141648

7 Reporting Errors and Requesting New Features

This chapter gives general instructions for reporting errors or for requesting new features with the STARe Software. Please consider the following points:

- **Provide us with as much information as possible**

Always supply as much relevant information as possible and explain what you mean as precisely as possible.

This helps us to find out the possible reason(s) for an error and, in the case of a feature request, to determine what you would really like to have.

- **Version number**

Always specify the version number of the STARe Software.

It makes no sense for us to try to reproduce an error that has already been eliminated and is no longer present in the latest version of the software.

- **Version of the module software**

If the problem occurs when you are performing an experiment, please specify the version of the TA module software.

- **Accurate description**

Describe precisely what you have done, the order in which you did it, and what the result was.

In many cases, there are often several different ways of doing something. We may not be able to reproduce undesirable effects or behavior without knowing precisely what you did. We have to know exactly how to proceed.

As an example: When working with the Evaluation Window, behavior often depends on the result mode setting.

- **The log file**

Always have a look at the log files.

They are located in the folder `log` below the installation folder of the STARe Software (e.g. C:\STAReSW\log) and their names depend on the window in which they were created.

Each interactive windows creates an individual log file when it is started. The name of this log file begins with the name of the interactive window and includes the date and time of its creation.

Example:

– STARe - 20050906_150709_6.log
the date format is "year, month, day" (2005, 09, 06)
the time format is "hours, minutes, seconds" (15:07h, 09.6 s)

Even if you do not understand every entry in the log files, the time stamp added to each entry may indicate whether an entry has something to do with your problem.

If the problem is related to the database management system (Ingres II), the log file is errlog.log in the folder ingres\files below the installation folder of Ingres (e.g. C:\IngresII\ingres\files).

- **Reproducibility**

Try to find out how reproducible an error is. Does it depend on certain settings or objects (such as curves, methods, etc.)? Does it depend on the order in which you performed your actions?

- **Export of STARe objects**

If a problem depends on certain objects (e.g. an evaluation fails only with certain curves), export the objects to an empty folder in STARe format. This will result in a list file and one further file (named 0, 1, 2, etc.) per object.

- **Composing an e-mail error report**

When you report errors please:

- Provide all the information mentioned above (version, description, reproducibility)
- Take a **snapshot** (Main Menu Bar: Service/Snapshot). This will integrate all relevant log files into the zip-file, snapshot.zip, which is written to the installation folder of the STARe Software.
  Include this file in your e-mail report.
- Include the exported objects in your e-mail report, i.e. the list file and all the other files in that folder.

8 Calibration and Adjustment

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8 Calibration and Adjustment

8.1 Introduction

This chapter explains the theoretical principles on which the calibration and adjustment of your STARe module are based, and shows you how to perform a calibration or adjustment using the STARe Software.

8.2 Definitions

In these operating instructions, the terms calibration and adjustment are used according to the definitions given below:

Calibration: "The set of operations that establish, under specified conditions, the relationship between a quantity indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values released by standards." [1]

Adjustment: "Operation of bringing a measuring instrument into a state of performance suitable for its use.

Note: Adjustment may be automatic or manual." [2]

[1,2] Reference: International Vocabulary of Basic and General Terms in Metrology, DIN Deutsches Institut for Normung, 2nd Edition 1994, Beuth Verlag GmbH

8.3 Basic principles of calibration and adjustment

This section describes the thermoanalytical principles that underlie the calibration and adjustment of the STARe module, as well as individual types of calibration and adjustment.

According to the definitions given above, the calibration and adjustment of the STARe module consists of the following tasks:

Calibration For the module in question, the determination of the actual deviation of measured values of reference substances from the standard literature values by means of calibration measurements.

Reference substance: a substance that is suitable for the calibration measurement and whose thermoanalytical property values are well established in the literature

Adjustment Adapting the specific module parameters so that the measured values of the calibration measurements performed afterward are within the limits of permissible error

Limits of permissible error: the specified extreme values for permitted deviations (positive and negative) from a set value.

8.3.1 Tau lag adjustment

The temperature sensors in STARe modules cannot measure the sample temperature directly because they are not in direct contact with the sample.

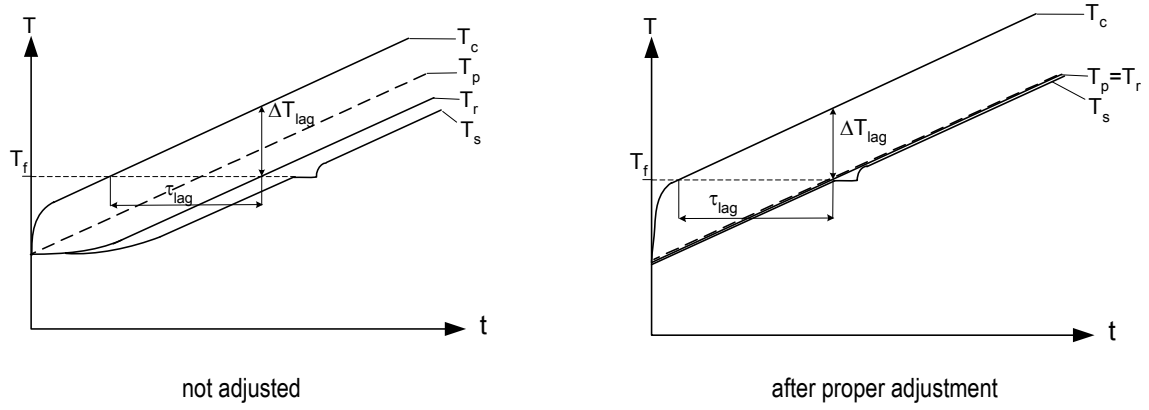
In a dynamic thermoanalytical experiment, a time delay occurs between the measured temperature and the reference temperature because of the thermal inertia arising from the heat capacities and thermal resistances of the crucible and DSC sensors.

The so-called reference temperature is used as the reference value for the experiment. It is defined as the temperature at the sample position in the empty measuring cell. With the DSC module, for example, it is the temperature of the empty reference crucible.

To make sure that the reference temperature agrees with the temperature set in a temperature program, the furnace temperature must be in advance of the reference temperature during heating and cooling.

The purpose of the tau lag adjustment in a dynamic experiment is to match the reference temperature T_r to the program temperature T_p. The tau lag adjustment corrects the dynamic behavior of the measuring cell.

Fig. 8-1 shows the temperature behavior in the measuring cell before and after adjustment during the same calibration measurement. The aim of adjustment is to get the program temperature, the reference temperature and the sample temperature to agree with each other.



Key		Subscripts:	
T_c :	furnace temperature	c :	cell
T_r :	reference temperature	r :	reference
T_s :	sample temperature	s :	sample
T_p :	program temperature (according to temperature program)	p :	program
T_f :	transition temperature (temperature of fusion)	lag :	lag
ΔT_{lag} :	the extent to which the reference temperature lags behind the furnace temperature		
τ_{lag} :	tau lag of the furnace temperature		

Fig. 8-1. Temperature behavior in a measuring module before and after (right) adjustment

♣ A sample of very low heat capacity is used for the calibration measurement. This effectively corresponds to the reference state. In most experiments, however, the heat capacity cannot be neglected. This results in the sample temperature lagging slightly behind the reference temperature. In the individual STARe modules, the sample temperature is calculated from the measured values in different ways (see the section *Determination of the sample temperature, page 8-12*).

The quantity tau lag (τ_{lag}) corresponds to the time delay between the furnace temperature and the reference temperature. Tau lag is a characteristic parameter of the measuring cell and depends on its design. Tau lag is determined for each measuring cell in a calibration experiment. Just like other physical characteristic quantities, it is also temperature dependent.

The following relationship exists between the time delay, tau lag, and ΔT_{lag}, the difference in temperature between the furnace temperature and the reference temperature:

$$\Delta T_{lag} = \beta \cdot \tau_{lag} \qquad (1)$$

where β is the heating rate in K/min

If the tau lag parameter of a measuring module is not adjusted, or is incorrectly adjusted, then the onset temperature depends on the heating rate β.

You can determine whether tau lag adjustment is necessary by performing two simple calibration measurements, for example the onset temperature of the melting of indium at two different heating rates, e.g. at 5 and 20 K/min). If the onset temperatures of the melting curve are significantly different, then a tau lag adjustment must be performed. The following diagram demonstrates how the onset temperature varies with the heating rate when the measuring cell is not adjusted. Depending on the heating rate, the error in the onset temperature can be up to 2.5% of the true value.

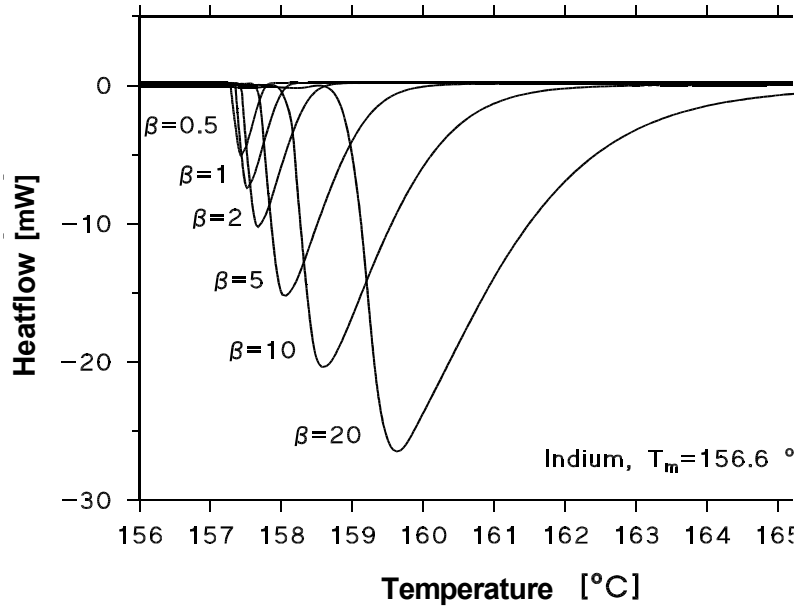


Fig. 8-2. The onset temperature measured at different heating rates β (here τ_{lag} is set to 0, which corresponds to a non-adjusted measuring cell)

The following diagram shows the onset temperature plotted as a function of the heating rate, β.

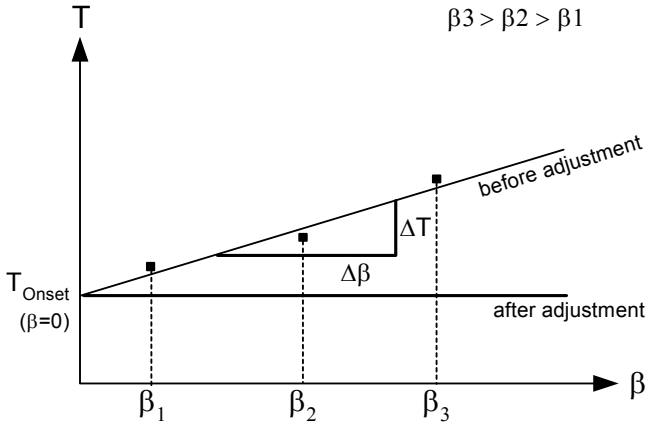


Fig. 8-3. Onset temperature as a function of the heating rate β, before and after adjustment

The relationship between the onset temperature and the heating rate can be determined by linear regression. According to equation (1), the slope of the regression curve represents the deviation of the tau lag value stored in the module with respect to the actual tau lag value of the module.

The intercept of the regression curve with the ordinate corresponds to the value of the onset temperature of the substance measured by the module at a heating rate of β = 0 K/min.

Proper adjustment of tau lag results in the onset temperatures being independent of the heating rate. (See *Fig. 8-4* and compare with *Fig. 8-3*.)

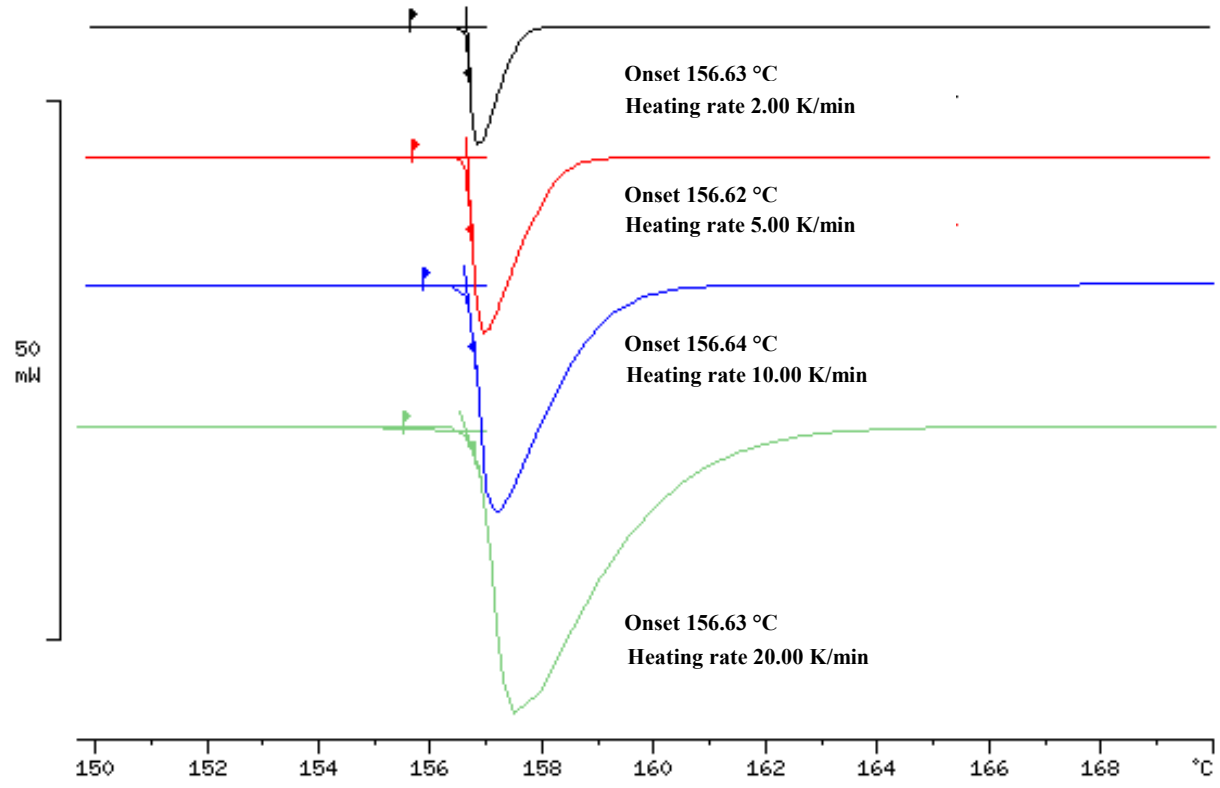


Fig. 8-4. The onset temperatures are independent of the heating rate **after** tau lag adjustment

Tau lag is a function of temperature, which means that the value obtained for tau lag depends on the calibration substance used because the individual transition temperatures are different. We can determine the temperature dependence of tau lag by measuring the onset temperature of different transitions and interpolating the results using a quadratic equation of the type shown below:

$$\tau_{lag} = A + B \cdot T + C \cdot T^2 \qquad (2)$$

With just one reference substance, only the ordinate intercept A is determined. If two or three reference substances are used, the slope, B, is also obtained. Calibration with four or more reference substances is, however, needed to determine the coefficient C.

The definition of tau lag depends on the type of STARe module

The definition of the quantity tau lag depends on the type of STARe module and the actual measurement configuration.

With the STARe DSC module, tau lag describes the extent to which the reference temperature lags behind the furnace temperature according to equation (1).

For the STARe TGA module and the STARe TMA modules, two different tau lag quantities are defined because these modules measure not only the furnace temperature but also the crucible holder temperature (TGA) or sample support temperature (TMA). We therefore distinguish between a tau lag $\tau_{lag\ c}$ for the lag of the reference temperature behind the furnace temperature, and a different tau lag $\tau_{lag\ sh}$ for the lag of the actual sample temperature behind the crucible holder temperature or the sample support temperature.

$$T_s = T_{sh} - \beta \cdot \tau_{lag\ sh} \quad (3)$$

$$T_r = T_c - \beta \cdot \tau_{lag\ c} \quad (4)$$

where:

T_c :	furnace temperature	c :	cell (furnace)
T_r :	reference temperature (temperature program)	r :	reference
T_{sh} :	temperature measured at the crucible holder or sample support	sh :	sample holder (crucible holder or sample support)
T_f :	transition temperature (temperature of fusion)	lag :	lag (time delay)
β :	heating rate		
$\tau_{lag\ sh}$:	tau lag between the reference temperature and the crucible holder or sample support temperature		
$\tau_{lag\ c}$:	tau lag between the furnace and the reference temperature		

Tau lag conversion factors within STARe Software FlexCal concept

For each STARe module, the quantity tau lag depends on the type of gas and crucible used.

The FlexCal concept implemented in the STARe Software enables the STARe Software to determine conversion factors from the standard data stored in the database. These factors allow tau lag for other combinations of gases and crucibles to be calculated. A flow chart showing how the STARe Software does this is shown in *table 8-1*.

- ♣ To obtain the best possible measurement accuracy, we recommend that you perform an adjustment with the desired gas and crucible factors rather than using conversion factors.

Tau lag depends on the type of gas and crucible. The STARe Software takes this into account through factors that are used to multiply the tau lag value of the standard gas and crucible combination (air and the standard 40 µl Al crucible, 40 µl).

The following table summarizes the tau lag values calculated with the conversion factors for different types of gases and crucibles:

	Air τ_{lag} **[s]**	**Helium** τ_{lag} **[s]**	**Gas y** τ_{lag} **[s]**
Standard Al crucible, 40 µl	3.5	2.6	τ_y
Large Al crucible, 160 µl	7.0	5.3	τ_y
High pressure crucible	21	15.7	τ_y
Crucible x	τ_x	τ_x	τ_{xy}

Table 8-1: Tau lag values in the STARe Software

table 8-1 applies to crucibles used for the STARe DSC modules. The tau lag values for STARe TGA modules can be tabulated in a similar way.

A mathematical model in the STARe Software determines the values for the conversion factors for the most common gases and crucibles. The model uses the data from the basic adjustment of the following measurement combinations as input values:

DSC measurements:	standard 40 µl aluminum crucible, air
TGA measurements:	standard 40 µl alumina crucible, air

A tau lag calibration should be performed regularly with all STARe modules to check measurement accuracy. If the limits of permissible error are exceeded, an adjustment should be performed.

8.3.2 Temperature adjustment

Different types of temperature sensors are used in the S**TA**R^{e} TA modules to measure temperatures during a measurement. The following table and diagram summarizes the different temperature sensors:

Temperature	DSC	TGA	TMA	DMA
Furnace (DSC, TGA TMA) Clamping assembly (DMA)	Pt100 (furnace)	R-type thermocouple (furnace)	R-type thermocouple (furnace)	K-type thermocouple (clamp. assembly)
Crucible holder (TGA) Sample support (TMA) Clamp (DMA)	-	R-type thermocouple (crucible holder)	R-type thermocouple (sample support)	K-type thermocouple (clamp)
Reference temperature	is calculated	is calculated	is calculated	is calculated

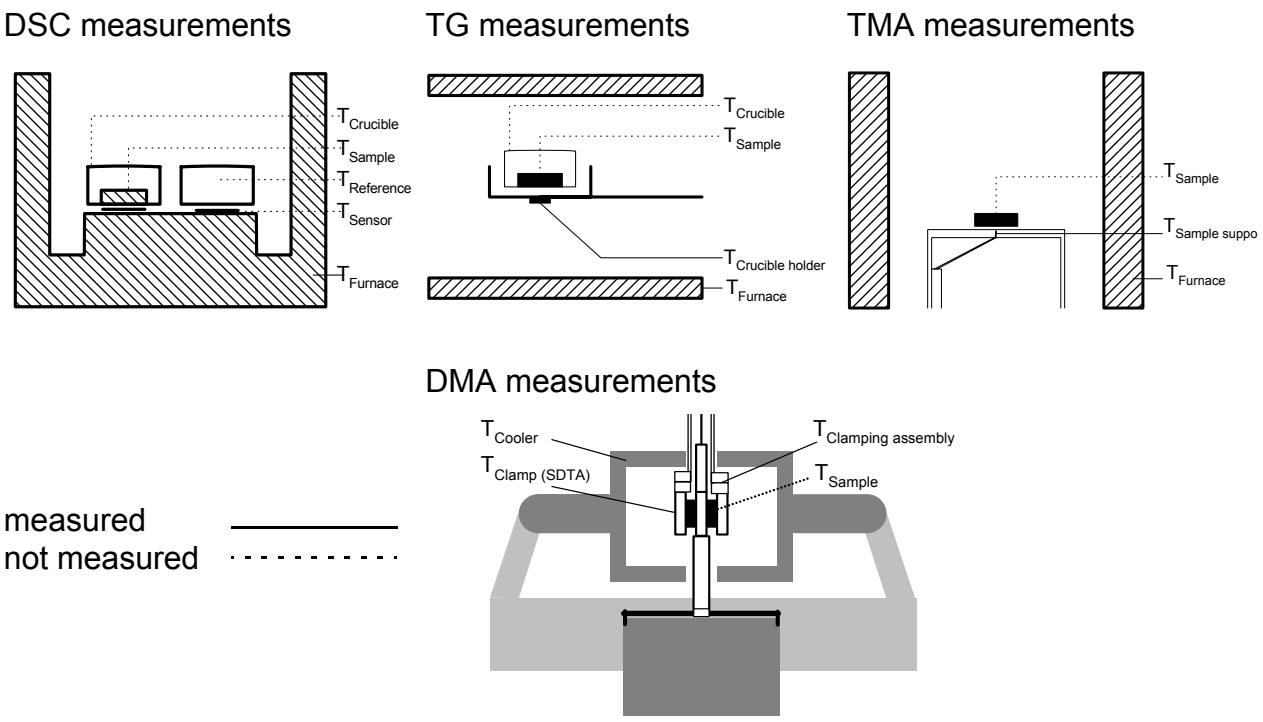


Fig. 8-5. Temperatures in the S**TA**R^{e} modules

Temperature sensors that are not adjusted or not properly adjusted yield incorrect measured values. A temperature calibration determines the deviation (offset) of the sensor temperature values from the values of the reference substances (e.g. the melting point of indium). Temperature adjustment allows you to correct this deviation and ensures that the furnace is heated in such a way that the temperature at the sample position agrees with the program temperature.

The deviation of temperature sensors can be approximated using a mathematical equation.

With the TGA and TMA modules, the temperature deviation of the furnace and the crucible holder temperature or the sample support temperature is determined in the following way.

$$\Delta T = T - T_{lit} = A + B \cdot T + C \cdot T^2 \qquad (5)$$

where T_{lit} is the literature value of the transition point

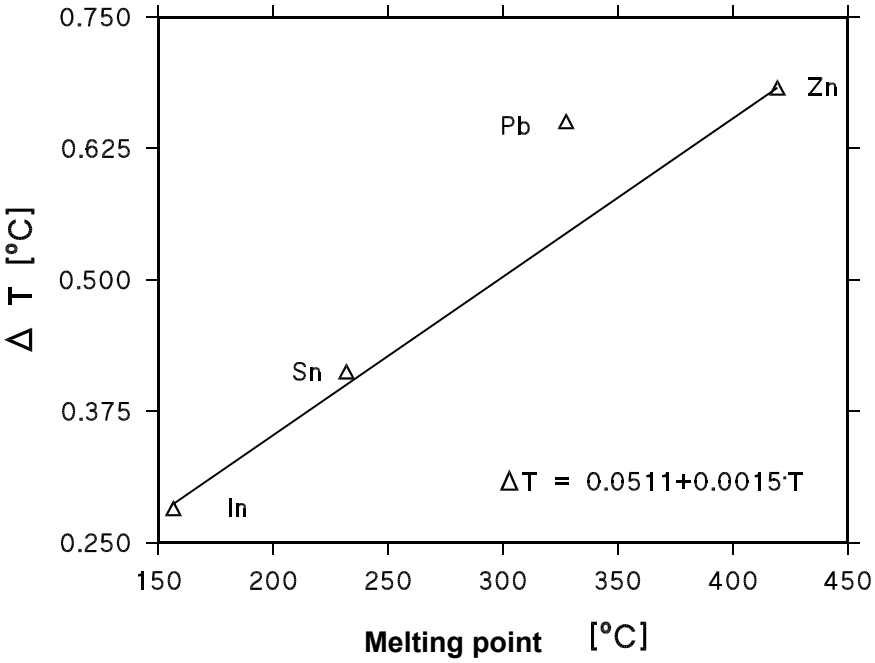


Fig. 8-6. Correction of the temperature deviation (offset) using a linear interpolation of measurements with four substances (In, Sn, Pb and Zn)

In the DSC module, a Pt100 temperature sensor measures the furnace temperature. The following quadratic equation describes the temperature deviation of its electrical resistance (R):

$$R(T) = A + B \cdot T + C \cdot T^2 \qquad (6)$$

For equations (5) and (6) the following applies: With just one reference substance, only the ordinate intercept A is determined. If two or three reference substances are used, the slope, B is also obtained. Calibration with four or more reference substances is, however, needed to determine the coefficient C.

The values of the coefficients A, B and C for a standard Pt100 are as follows:

$A = \quad 100\ \Omega$

$B = \quad 0.3908\ \Omega\ °C^{-1}\ T$

$C = \quad -58.02\ 10^{-6}\ \Omega \cdot C^{-2}\ T^{2}$

Determination of the sample temperature

In the S**TA**R^{e} modules, it is not possible to measure the sample temperature directly. The sample temperature can however be calculated from the measured data. Depending on the type of module, the calculation is performed either by the S**TA**R^{e} Software, or directly by the module software.

With DSC measurements, the S**TA**R^{e} Software determines the sample temperature in the Evaluation Window. Using the heat flow measurement data, the software calculates the temperature difference between the sample and reference temperatures and hence the sample temperature.

With TGA and TMA measurements, the module software determines the sample temperature directly. If we take into account the time delay tau lag (τ_{lag}), the sample temperature can be calculated from the measured temperature T as follows:

$$T_{Sample} = T + \Delta T(T) - \beta \cdot \tau_{lag}(T) \qquad (7)$$

where:

T_{sample}	sample temperature
T	temperature measured by the sensor
ΔT	offset of the temperature sensor
β	heating rate

♣ Note: For TGA measurements, we recommend that you always use the same purge gas flow rate.

To check the measurement accuracy, we recommend that you perform a temperature calibration at regular intervals with all S**TA**R^{e} modules. Depending on the type of module, the temperature calibration can refer to the furnace temperature (DSC, TGA and TMA), the crucible holder (TGA) and/or the sample support temperature (TMA).

8.3.3 Heat flow adjustment of DSC modules

Heat flow adjustment is only performed with the DSC modules of the STARe system because only these modules measure heat flow directly. The sensitivity of the sensor is the decisive quantity for the measurement of the heat flow and is the parameter that is adjusted. We call this quantity the calorimetric sensitivity E. It is the product of the thermal resistance R_{th} and the sensitivity S of the DSC sensor:

$$E(T) = R_{th} \cdot S \qquad (8)$$

where $$S = \frac{U}{\Delta T}$$

and U : electrical voltage at the sensor

ΔT : temperature difference across the sensor

Both the factors R_{th} and S are temperature dependent.

- The heat flow measurement of the DSC FRS5 sensor is based on the measurement of a temperature difference using 56 Au/AuPd thermocouples.

Example:

- The value of the sensitivity S of the DSC822e FRS5 sensor is ≈ 560'0000 digital points/K (which corresponds to 330 µV/K) and the value of the thermal resistance R_{th} of this type of sensor is 0.04 K/mW.

- You can enter your own calibration value for the sensitivity S (see the appendix Calibration of the sensitivity of the sensor of a STARe DSC module).

According to the so-called "Thermal Ohm's Law", the heat flow $\dot{Q}$ is calculated as follows:

$$\dot{Q} = \frac{\Delta T}{R_{th}}$$

since $$\Delta T = \frac{U}{S}$$

this gives $$\dot{Q} = \frac{U}{R_{th} \cdot S} = \frac{U}{E(T)}$$

For practical reasons, so that we can calculate E as a function of temperature in the calibration, we divide the quantity E into a temperature-independent (constant) component (E_{In}) and a temperature-dependent component (E_{rel}):

$$E(T) = E_{In} \cdot (E_{rel}(T) + dE_{rel}(T)) \quad (9)$$

$E_{(T=156.6\,°C)}$ is the value of the calorimetric sensitivity at the melting point of indium, i.e. at 156.6 °C. For the DSC822^{e} module, we use a value of 25,216 mW^{-1} for the default value of E_{In}.

1/ $E_{(T=156.6\,°C)}$ is the maximum signal resolution of the sensors. The default value is:

$$1/E_{(T=156.6\,°C)} = 0.04\ \mu W$$

♣ The calorimetric sensitivity of the sensor, E(T), can be adjusted at the melting point of a reference substance other than indium in order to optimize the accuracy of the sensor in the desired temperature range.

The components $E_{rel}(T)$ and $dE_{rel}(T)$ are described by quadratic equations of temperature:

$$E_{rel}(T) = A + B \cdot T + C \cdot T^2 \quad (10)$$

and

$$dE_{rel}(T) = da + db \cdot T + dc \cdot T^2 \quad (11)$$

$E_{rel}(T)$ is scaled to 1 at the melting point of indium: E_{rel} (156.6 °C) = 1

Only the coefficients *da*, *db* and *dc* of the expression for dE_{rel} can be adjusted. The coefficients A, B and C for the calculation of E_{rel} are stored as fixed values in the database of the S**TA**R^{e} Software and are not part of the adjustment.

With just one reference substance, only the ordinate intercept, *da*, is determined. If two or three standard substances are used, the slope, *db*, is also obtained. Calibration with four or more standard substances is, however, needed to determine the coefficient, *dc*.

The effect of the type of crucible and gas on heat transfer is taken into account by the factors t_t and f_g, which are used to multiply E(T):

$$\dot{Q} = \frac{U}{E(T) \cdot f_T \cdot f_G}$$

where f_t : crucible factor, f_g : gas factor

♣ For air and the standard aluminum crucible $f_t = f_g = 1$

With (10) we obtain:

$$\dot{Q} = \frac{U}{E_{In} \cdot (E_{rel}(T) + dE_{rel}(T)) \cdot f_T \cdot f_G} \qquad (12)$$

For the heat flow calibration, we use reference substances that are well documented in the literature (e.g. indium) and whose heats of fusion ΔH_f are accurately known. The heat of fusion ΔH_f of indium is:

$$\Delta H_{f\,In} = 28.45 \text{ J/g}$$

We determine the new value of E_{In} by comparing the measured heat of fusion with the literature value:

$$E_{In\,neu} = \frac{\Delta H_{f\,Lit}}{\Delta H_{f\,gemessen}} \cdot E_{In\,alt} \qquad (13)$$

The coefficients *da* and *db* in equation (11) for dE_{rel} (T) are obtained from the following equation in which we make the integral of the heat flow across the melting peak equal to the literature value of the heat of fusion:

$$\int \dot{Q} \cdot dt = \Delta H_{m\,Lit} \qquad (14)$$

You can determine the coefficients *da* and *db* by performing two or more single sample calibrations. *da* alone is also be determined as part of a total adjustment.

The heat flow calibration yields a new value for E_{In} and using the coefficients *da* and *db* determines the temperature dependence of the calorimetric sensitivity, E(T).

The following table shows the default values stored in the STARe Software database for the calorimetric sensitivity E for different types of gases and crucibles: The value of E_{In} is multiplied by the corresponding crucible and gas factors f_t and f_g.

	Air $E_{in} \cdot f_g f_t$ [mW^{-1}]	**Helium** $E_{in} \cdot f_g f_t$ [mW^{-1}]	**Gas y** $E_{in} \cdot f_g f_t$ [mW^{-1}]
Standard Al crucible, 40 µl	25,220	18,915	E_y
Large Al crucible, 160 µl	22,194	16,645	E_y
High pressure crucible	22,194	16,645	E_y
Crucible x	E_x	E_x	E_{xy}

Table 8-2: Values of the calorimetric sensitivity E for the DSC822e in the STARe Software

A mathematical model in the STARe Software determines the values for the crucible and gas factors f_t and f_g for the most common gases and crucibles.

If you use crucibles or gases that are not included in the calculation model, you must perform a separate adjustment in order to determine the corresponding value of the product $E f_t f_g$.

- The type of gas atmosphere influences the heat flow measurement. When gases of good thermal conductivity are used, e.g. helium, the DSC sensor measures a smaller heat flow to the sample than with gases of poor thermal conductivity. Good thermal conductivity results in part of the heat flow being transferred to the gas instead of to the DSC sensor.
- The measurement values also change if you use other types of crucibles under otherwise identical conditions.

8.3.4 Non-thermal adjustment

Besides the adjustments that are directly related to the measurement of thermal quantities, some non-thermal quantities are also adjusted for particular STARe modules.

You can find detailed information on this in the corresponding STARe module operating instructions.

Weight adjustment

Weight adjustment is performed only with the STARe TGA modules in order to adjust the internal balance. On switching on the TGA module, the internal module software automatically performs an adjustment of the balance (this takes about three minutes). Two known weights incorporated in the TGA module serve as reference weights.

Adjustment with STARe TMA modules

The following adjustments are performed only with the STARe TMA modules. They are started from the STARe Software in the Module Control Window in the Calib/Adjust menu by means of the following drop-down menu:

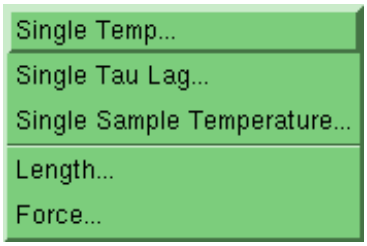


Fig. 8-7. Drop-down menu with the Length Calibration and the Force-Calibration menu items

The additional dialog and user input take place via the LCD display and the keypad.

An **adjustment of the length measurement** is performed to adjust the length sensor. This adjustment is based on difference measurements of reference lengths that are set with gauge blocks.

The **adjustment of the force** is performed using a reference weight incorporated in the TMA module.

8.4 Calibration and adjustment of your S**TA**R^{e} module

The purpose of calibration measurements is to check that the S**TA**R^{e} module is performing to specifications, i.e. is within the defined limits of permissible error. If the calibration values are outside the preset limits, then an adjustment of your module is necessary. The next section describes how you calibrate and adjust your S**TA**R^{e} module.

The following flow chart summarizes the procedure.

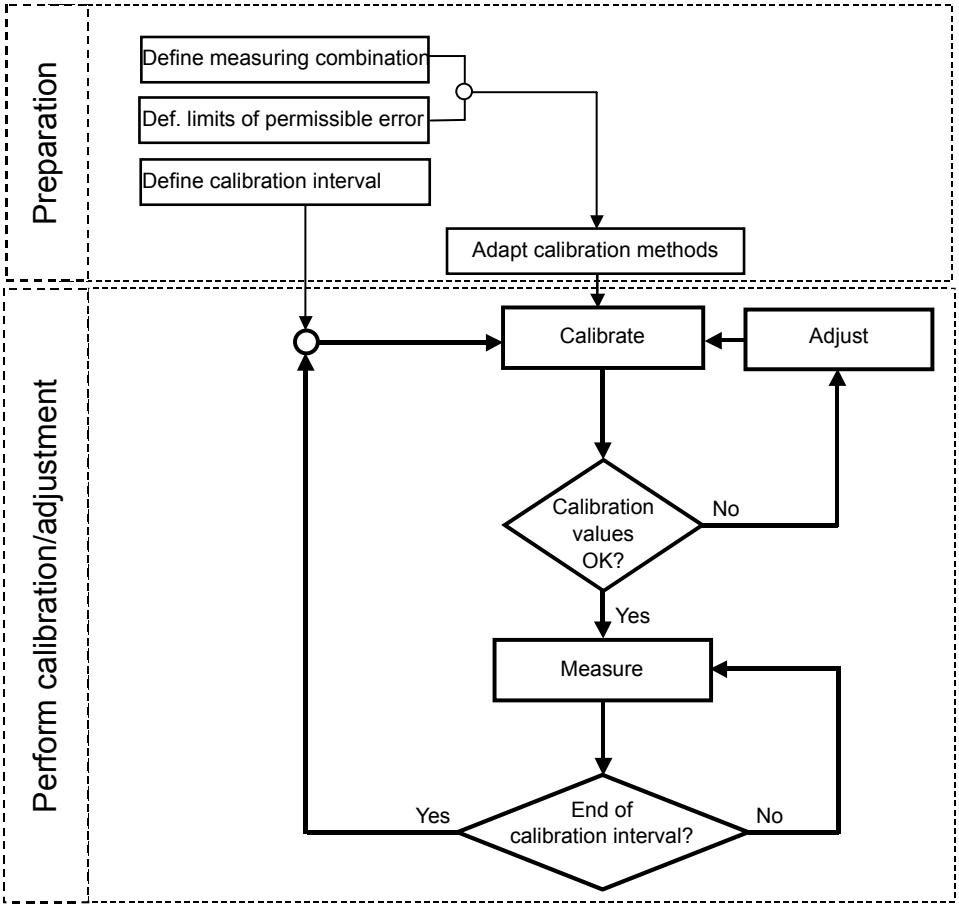


Fig. 8-8. Overview of the adjustment procedure

Before the actual adjustment and calibration are performed, there is a preparation stage in which you define the basic parameters, i.e. the desired combination of crucible and gas, the limits of permissible error for measurement accuracy and the calibration interval. The preparation stage also includes setting up the calibration method. We recommend that you do not begin the calibration or adjustment until you have completed these important preparations.

8.4.1 Factory adjustments

Before shipping a module, various standard adjustments are performed on your STAR^e module in the factory. The adjustment dates can be found on the diskette delivered with your module. You can load the data from the floppy disk to your STAR^e Software (see also the STAR^e Software online Help).

The following table summarizes the factory adjustments:

Module	Sample holder	Calibration substances		Gas	Adjustment type
DSC82x^e	aluminum crucible, 40 µl	In (T_f = 156.6 °C)	Zn (T_f = 419.6 °C)	air	tau lag temperature (Pt100) heat flow
TGA/SDTA85x^e	alumina crucible, 70 µl	In (T_f = 156.6 °C)	Al (T_f = 660.4 °C)	N_2	tau lag temperature of the furnace temperature of the crucible holder
TMA/SDTA84x	aluminum crucible, 20 µl	In (T_f = 156.6 °C)	Zn (T_f = 419.6 °C)	air	tau lag furnace temperature sample support temperature
DMA/SDTA86x	• small CA shear clamp • large CA bending clamp	In (T_f = 156.6 °C)	H_2O (T_f = 0 °C)	air	sample temperature CA temperature Dewar

Table 8-3: Factory adjustment of STAR^e TA modules

8.4.2 Measuring combination

module, crucible and purge gas combination

You can store a set adjustment data in the database for each combination of module, crucible and purge gas (i.e. measuring combination). Depending on the experimental conditions, the S**TA**R^{e} Software transfers the corresponding adjustment data to the module for the measurement, so that you still obtain correct measurement results even if you for example switch from helium to oxygen.

♣ For TGA and TMA measurements: Please note that the purge gas flow rate for the measurement and for the adjustment must be practically the same because flow rate affects the reference temperature in TGA and TMA modules.

8.4.3 Calibration

Good Laboratory Practice (GLP) and measurement accuracy

Calibration is a very important way of checking the measurement accuracy of your STARe module for GLP purposes. The calibration of your STARe module is based on the Single and Multi calibration methods that are described in the section *Calibration methods in the STARe Software, page 8-23*. They offer the following advantages:

- The STARe Software records the calibration data in a table in a dialog box that you can open in the Module Control Window in the Calib/Adjust menu.
- For calibration you only have to perform one measurement.
- You can perform a calibration at several heating rates. This allows you, for example, to adjust tau lag with just one calibration measurement.

♣ Only the numerical values of the calibration parameters are obtained as the result of the calibration. There is no possibility, as there is with Checks, of checking whether the results are within the limits of permissible error, or of providing additional text about the result of the calibration (see section *Calibration using Checks, page 8-52*).

The following criteria and reference values are used for the individual types of calibration:

Tau lag calibration:	measured onset temperatures independent of the heating rate.
Temperature calibration:	literature values of the melting points.
Heat flow calibration:	literature values of the heats of fusion.

We recommend that you perform a calibration once a month.

♣ If the calibration measurements are within the limits of permissible error during two successive months, you can double the interval for the calibration measurements; otherwise you must halve it. If you want the best possible accuracy, you can perform calibration measurements before and after each sample measurement.

According to GLP regulations, you should also check the measurement accuracy of your module if you make the following changes:

- change the installation location of your S**TA**R^{e} module
- replace the DSC sensor (only DSC module)
- replace the crucible holder or the sample support (TGA and TMA modules)

If the values of the calibration measurements are within the limits of permissible error, there is no need to perform an adjustment. If the values are outside the limits, you must adjust your S**TA**R^{e} module again.

You can change the limits of permissible error according to your own individual requirements. In this respect, we recommend that you abide by the principle “as accurately as required”. The control methods available in the S**TA**R^{e} are based on the S**TA**R^{e} instrument specifications and have very narrow limits of permissible error.

8.4.4 Calibration methods in the STARe Software

The STARe database contains a number of methods that have been specially prepared for the calibration of your STARe module. In the Method Window you can identify these special calibration methods in the list in the dialog box, e.g. when you open methods whose names begin with the abbreviation Calib.

description of the calibration methods

The name of the calibration method also includes the following information:

- Type of module (DSC, TGA, TMA, DMA etc.)
- Type of calibration
  - Tau lag
  - Temperature
  - Heat flow
  - Sample holder
  - Total
- Multiple or single sample crucible
  - Single
  - Multiple

single and multiple sample crucibles

- Calibration substance (e.g. In or Zn)

Example:

– The method name for an indium check for the DSC module is:
Calib DSC Heat Flow Single In

Since there are very many measurement combinations, only the calibration methods for the most important standard cases are stored in the database: the standard 40 µl aluminum crucible for the DSC; the 70 µl alumina crucible for the TGA and air atmosphere.

You can use the methods provided as templates and adapt them to suit your own special measurement combination(s) in the Method Window.

♣ In contrast to the Checks (see the section *Calibration using Checks, page 8-52*), the calibration methods do not include automatic evaluation.

Types of calibration methods (the Type feature)

the Type box in the Method Window

There are different types of calibration corresponding to the individual types of modules. You can perform a particular calibration in different ways. The Method Window of the S**TA**R^{e} Software presents many calibration methods under the feature Type (Calibration dialog box, in the menu Miscellaneous/ Calib/Adjust). You can adapt these methods to meet your own requirements.

automatic evaluations

If you assign your calibration method to a particular type in the Method Window, the S**TA**R^{e} Software automatically links the corresponding automatic evaluations with your methods and activates different display possibilities in the Experiment Window and Module Control Window. Under the Type feature, we distinguish between the following types of calibration:

- **Temperature calibration with multiple samples**

  Type: Temp. with multiple samples

  Temperature calibration based on the measurement of a multiple sample crucible containing several different reference substances. Typically you use two or three samples in one crucible (e.g. In and Zn) in this type of calibration.

- **Temperature calibration with single samples**

  Type: Temp. with single samples

  Temperature calibration based on the measurement of a single sample crucible, i.e. a crucible containing only one reference substance. Typically several single sample crucibles are measured one after the other. Calculation of the mean value of several individual measurements results in improved measurement accuracy. (See also the *Comments on single sample measurements, page 8-27*.)

- **Temperature calibration for the TA4000**

  Type: Temp TA4000

TA4000 family

  Temperature calibration for TA4000 series measuring cells. In this case you can use the Mettler-Toledo calibration crucible containing three metals.

TC15 TA Controller

♣ With S**TA**R^{e} Software versions < 4.0: When using the TA4000 DSC measuring cell with the TC15, you must perform a temperature calibration of the above type at least once. For calibrations later on, we recommend that you use standard temperature and heat flow calibrations and your own calibration samples. This results in better temperature accuracy.

ADC calibration

♣ With S**TA**R^{e} Software versions $\geq$ 4.0: When using the TA4000 DSC measuring cell with the TC15, you must perform an ADC calibration at least once.

- **Tau Lag calibration with a single sample**

  Type: tau lag with single sample

  tau lag and onset temperature

  Tau lag calibration with a crucible containing one sample. The calibration determines tau lag for the melting temperature of the reference substance (e.g. indium) and makes the onset temperature independent of the heating rate. If you want to calibrate the temperature dependence of tau lag, you have to perform several experiments with different substances, e.g. In or Zn. (See also the *Comments on single sample measurements, page 8-27*)

- **Heat flow calibration**

  Type: heat flow

  calorimetric sensitivity

  Heat flow calibration with a crucible containing one sample only. The sample consists of an accurately known quantity of the reference substance, e.g. indium. The STARe Software then determines the calorimetric sensitivity E from the heat flow measurement (see also the *Comments on single sample measurements, page 8-27*).

- **Heat flow calibration with single samples**

  Type: heat flow

  Heat flow calibration using several measurements with one sample (calculation of the mean value) and several samples of different substances, e.g. In and Zn. In each case, the sample consists of an accurately known quantity of the reference substance.

Example:

– Heat flow calibration with single samples using three measurements of a crucible containing 6.18 mg indium, and two measurements of a second crucible with 9.13 mg zinc.

- **Total adjustment**

  Type: Total

  Methods of the type Total do not include any calibrations. They are only used for the adjustment of your STARe module.

  Total adjustment adjusts your module completely in one measurement and at the same time performs the corresponding tau lag-, temperature and sensor adjustment for your module (see also the section *Total adjustment, page 8-35*).

- **Crucible holder or sample support**

  Type: sample holder

sample temperature-measurement

  TGA and TMA specific calibration. It calibrates the sample temperature measured under the crucible holder or sample support on the basis of one measurement.

- **Crucible holder or sample support with single samples**

  Type: sample holder with single sample

  TGA and TMA specific calibration. It calibrates the sample temperature measured under the crucible holder or sample support by calculating the mean value of several measurements of the same reference substance, or of each of several different reference substances. (See also the *Comments on single sample measurements, page 8-27*.)

- **ADC**

  Type: ADC

TC15 and ADC calibration

  Special calibration method for the TA4000 module. From the S**TA**R^{e} Software version 4.0 onward, a calibration of the type ADC must be performed to calibrate the analog to digital converter (ADC). (With earlier versions of the S**TA**R^{e} Software, the AD converter of the TC15 was calibrated with each temperature calibration.)

- **Calibration with a ordinary method**

  Type: None

automatic evaluations and display possibilities

  The None type corresponds to an ordinary method. You can use ordinary methods as a basis for calibrations just as you normally do in an experiment. The None type calibration suppresses automatic evaluations and display possibilities in the Experiment Window and Module Control Window. You obtain the data for the calibration through the evaluation in the Evaluation Window (see the appendix *Adjustment through manual entry of adjustment data).*

TG50

♣ The control of temperature accuracy of TG50 modules is based on the measurement of the Curie transition (point of inflection) of nickel using the adjustment magnet.

Comments on single sample measurements

You can include additional measurements with single samples in the calibration later on by performing the corresponding calibration measurements. The STARe Software stores the calibration data. You can look at the data in a dialog box that you call up in the Calib/Adjust menu in the Module Control Window of the STARe Software.

The actual adjustment of your module is not performed until the end of the calibration procedure when you click OK in the information box corresponding to the type of calibration desired.

8.4.5 Adapting and creating calibration methods

The calibration methods supplied with the STARe Software enable you to adjust your STARe module to a high degree of accuracy. If you want the best possible accuracy, you can adjust your STARe module with methods adapted from existing methods or methods that you create yourself.

The following shows you how to adapt existing calibration methods and to create new calibration methods.

To adapt an existing calibration method:

(1) Open a method window.

(2) Choose File/Open...

A list with all the methods stored appears.

display calibration methods

(3) To find calibration methods more quickly: click the Filter button and then select the Only calibration methods function. Click OK.

A list with all the calibration methods appears.

(4) Click the desired adjustment method.

The adjustment method selected is displayed in the Method Window.

(5) Make the necessary changes to the method. Edit the adjustment method just like a normal method and pay attention to the following instructions and notes:

heating and cooling rates in the method

- Make sure that your module is able to achieve the defined heating and cooling rates. If need be, adapt the rates accordingly.

crucible type in the method

- If you are not using the standard crucibles, change the type of crucible in the method as appropriate.

crucibles of large thermal inertia

- When using crucibles of large mass and/or poor thermal conductivity, expand the temperature range of the heating and cooling segments to take the larger thermal inertia of such crucibles into account.

temperature range of your measurement

- Choose reference substances whose melting points correspond to the temperature range of your measurement (otherwise the results would be less accurate due to extrapolation).

purge gas

- Select the desired purge gas for your application.

♣ Detailed instructions on how to edit a method can be found in the STARe Software online Help to the Method Window or Routine Window.

measurement accuracy

♣ To obtain the best possible accuracy, you should adjust the module for each of its specific measurement combinations before performing the measurement. If you do not perform an adjustment, the STARe Software calculates adjustment data for a non-adjusted measurement combination on the basis of the standard adjustment data and factors stored in the database. You do not, however, achieve the same degree of measurement accuracy that you would if you performed specific adjustment beforehand.

(6) Click File/Save as... and enter the name of the method with the following information in the Name box:

method name

- Module type (DSC, TGA, TMA, DMA etc.)
- Type of calibration: tau lag, temperature, heat flow, sample holder or total
- Multiple (multiple) or single sample crucible (single)

multiple / single

- Calibration substance (e.g. In or Zn)

(7) Click OK to store your method.

Example:

– The method name for a DSC method: DSC Heat Flow Single Sn

You have now adapted an calibration method to your requirements and stored it.

To create a new calibration method:

(1) Open a method window.

(2) Choose File/Open..., to open a new method.
A new empty method appears in the method window.

(3) Choose Miscellaneous/Calib/Adjust.
The Calibration dialog window appears.

(4) Mark the reference substance you want to use for your calibration so that their melting points cover the temperature range of your measurement (otherwise the results would be less accurate due to extrapolation).

temperature range of your measurement

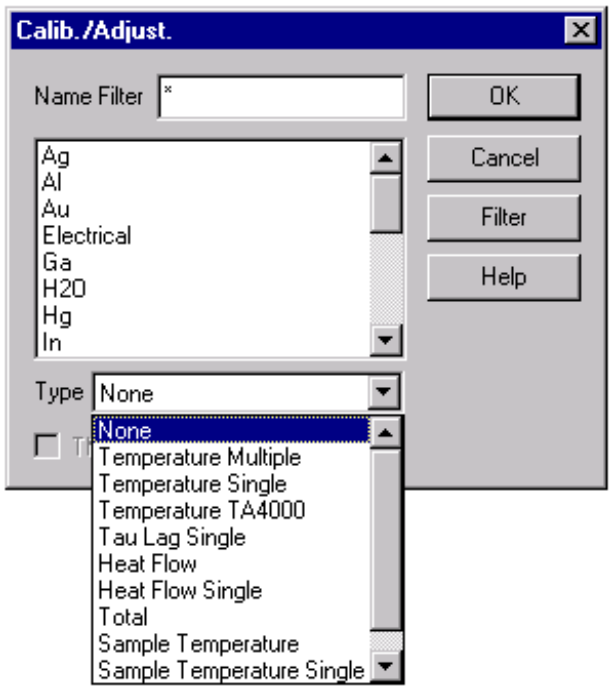
♣ You can select several reference substances at the same time by holding down the CTRL key and marking the desired substances with the mouse.

selecting several substances at the same time

(5) Click the Type button to define the type of adjustment method.

Type button

A drop-down list with the types of calibration methods appears.



♣ The definition of the type results in macros being attached to your method. These macros automatically evaluate the measured values and produce displays corresponding to the type of calibration. It is only at this stage that the method becomes an actual calibration method. The None type corresponds to an ordinary method without any attached macros.

(6) Mark the desired type of calibration method and click OK.

(7) Define the temperature program for your calibration. Pay attention to the following instructions and notes:

start and end temperature of the heating segment

- Set the start temperature of your heating segment to the expected value of the melting temperature of the substance less about five times the numerical value of the temperature in the heating rate (in K/min).
- Set the end temperature of your heating segment to the expected value of the melting temperature plus about twice the numerical value of the temperature in the heating rate (in K/min).

reference points for the calibration

- Use only first order transition points as reference points for the calibration. The melting peaks of high quality reference substances are best. The substances normally used are already in the database. You can however add other substances in the Install Window under Install/Calibration Substance. Typical heating rates are 1…10 K/min, but this depends on the heating rates you use for your actual sample measurements.

Example:

– Gallium (29.8 °C): 25 °C to 32 °C at 1 K/min
Benzoic acid (122.4 °C): 100 °C to 135 °C at 5 K/min

▸ With a tau lag adjustment method: define an additional heating and cooling segment before the segments of the actual calibration. Use at least two different heating rates for the calibration measurement (e.g. 2 and 5 K/min).

premelting the sample

♣ Heat transfer to the sample is usually greatly improved by melting and recrystallizing the sample beforehand. A slightly lower onset temperature is obtained.

heating and cooling rates in the method

▸ Make sure that your module is able to achieve the defined heating and cooling rates. If need be, adapt the rates accordingly.

crucibles of high thermal inertia

▸ When using crucibles of large mass and/or poor thermal conductivity, expand the temperature range of the heating and cooling segments to take the larger thermal inertia of such crucibles into account.

measurement accuracy

♣ To obtain the best possible accuracy, you should adjust the module for each of its specific measurement combinations before performing the measurement. If you do not perform an adjustment, the STARe Software calculates adjustment data for a non-adjusted measurement combination on the basis of the standard adjustment data and factors stored in the database. You do not, however, achieve the same degree of measurement accuracy that you would if you performed specific adjustment beforehand.

method names

(8) Choose File/Save as... and enter the name of the method with the following information in the Name box:

- Type of module (DSC, TGA, TMA, DMA etc.)
- Type of calibration: tau lag, temperature, heat flow, sample holder or total
- Multiple (multiple) or single sample crucible (single)

multiple / single

- Calibration substance (e.g. In or Zn)

(9) Click OK to store your method.

Example:

– The method name for a DSC method: DSC Heat Flow Single Sn

You have now created and stored a new method.

8.4.6 Sample preparation

The following section contains instructions and notes on sample preparation:

General instructions (for all measuring techniques):

heat transfer to the sample

- Press the sample to a flat disk before you put it in the crucible in order to ensure good heat transfer. Do not press the sample against the bottom of the crucible. It could form a low melting eutectic with the crucible material.
- With DSC, TGA/SDTA and TMA/SDTA measurements: Make two measurements with the same crucible. The heat transfer to the sample is usually greatly improved after the sample has melted and recrystallized in the first measurement. A slightly lower onset temperature is then obtained for the second measurement.

temperature range of the measurement

- Tau lag adjustments: We recommend that you use at least two different substances for the tau lag adjustment. The onset temperatures of the substances should cover the desired temperature range.

multiple sample crucible

Instructions for preparing multiple sample crucibles:

- Place the samples in the crucible as far apart as possible from each other.
- If you perform measurements with indium and zinc, wrap the sample of zinc in a small piece of aluminum foil in order to prevent it fusing with the indium sample (risk of eutectic formation).
- Press a flat piece of aluminum foil onto the sample disks with a suitable rubber cylinder in order to fix them securely.

DSC samples

Instructions for the preparation of DSC samples:

- Seal the crucible hermetically in order to prevent the sublimation and evaporation of substances with high vapor pressures. If the temperature of the experiment exceeds 250 °C, pierce a small hole in the lid of the crucible **before** sealing.

♣ The tau lag: for S**TA**R^{e} DSC modes is between -3 and +10 seconds.

8.4.7 Adjustment

If the calibration measurements show that the measured values of your STARe module are outside the limits of permissible error, then you must readjust your module.

Important notes

Please pay attention to the following points before you adjust your STARe module:

- Only specially trained personnel should be allowed to adjust your STARe module. Incorrect adjustment leads to inaccurate or false results.
- You can check whether the adjustment has been done properly by performing a calibration immediately after the adjustment.

checking with a calibration

- To achieve the best possible measurement accuracy with your STARe module should be adjusted for each measurement configuration

measurement accuracy

♣ With DSC and TGA modules, the adjustment data for crucibles and gases are stored in the database.

- Total adjustment allows you to adjust your STARe module in one measurement. With total adjustment, you obtain good measurement accuracy for your module and save a lot of time compared with single adjustments.

Single adjustments

There are specific single adjustments for each STARe module depending on the measuring principle involved. We distinguish between tau lag, temperature and sensor adjustment. The total adjustment of your module also includes these three types of adjustment and guarantees a degree of measurement accuracy that is good enough for most normal applications.

If need be, you can however achieve an even better degree of measurement accuracy for your S**TA**R^{e} module with single adjustments. We recommend that you perform the different types of adjustment in the following order:

sequence of calibrations and adjustments

1 Tau lag-adjustment

2 Temperature adjustment

3 Sensor adjustment (heat flow, sample temperature, force or length)

Pt100 temperature sensor

♣ DSC: The measurement of the furnace temperature with the Pt100 temperature sensor is independent of the choice of purge gas and crucible. If you change the type of crucible or purge gas, you do not have to readjust the furnace temperature sensor.

purge gases for TGA and TMA

♣ TGA and TMA: With the TGA and TMA modules, the purge gas has a slight influence on the measurement of the furnace temperature.

Total adjustment

Total adjustment allows you to adjust your STARe module in one measurement. With total adjustment, you obtain good measurement accuracy for your module and save a lot of time compared with single adjustments.

As shown in *table 8-4*, for each STARe module there is a corresponding total adjustment that includes all the module specific single adjustments.

STARe module:	DSC	TGA	TMA
Tau lag adjustment	✓	✓	✓
Furnace temperature adjustment	✓	✓	✓
Sample temperature adjustment	-	✓	✓
Heat flow adjustment	✓	-	-

Table 8-4: Total adjustment of STARe modules

♣ An automatic total adjustment with one measurement cannot be performed on STARe DMA modules.

Total adjustment requires at least **two substances**. The temperature program for total adjustment includes at least **three different heating rates** for each calibration substance. The coefficient of calorimetric sensitivity, *da*, is calibrated. The measurements in the first two segments are discarded. They serve to melt and recrystallize the sample for the first time in order to improve the heat flow to the sample.

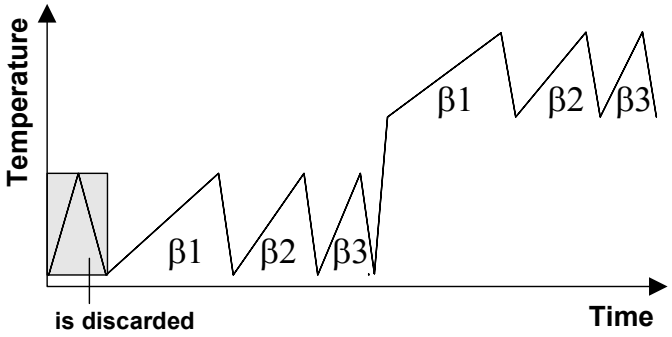


Fig. 8-9. Temperature program for total adjustment

♣ **With DSC total adjustment:** The heat flow adjustment in a total calibration is based on the measurement of a single substance and is therefore a one-point calibration. In the Experiment Window, you can only enter the weight of one substance, and therefore have to enter the weight of the substance with the lowest melting point. We recommend that you perform at least two single sample heat flow calibrations (type single) with different reference substances after each DSC total adjustment.

The following diagram shows the central part of the flow chart given in *Fig. 8-8* for total adjustment.

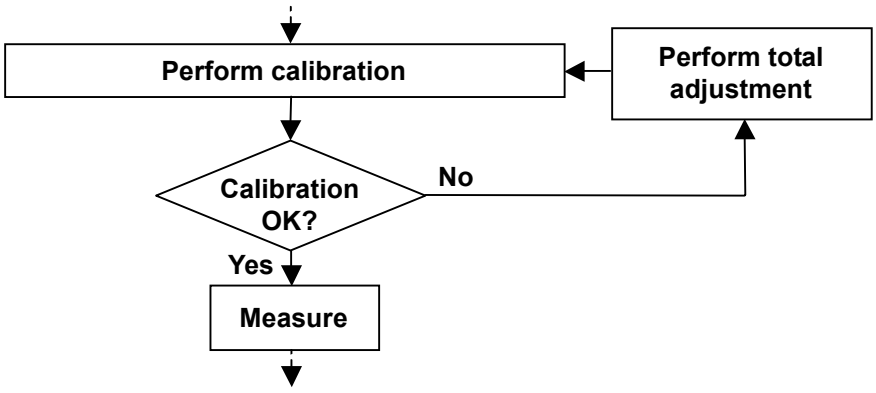


Fig. 8-10. Flow chart for total adjustment

♣ Total adjustment proceeds automatically. This means that you can adjust your STAR^{e} module overnight.

Measuring configuration for adjustment

You only need perform a temperature adjustment for your standard measuring configuration.

However, if you want to achieve the highest degree of measurement accuracy, we recommend that you adjust your STARe module for each measuring configuration (i.e. for each combination of gas and crucible). A data set corresponding to each measuring configuration is then stored in the STARe Software database.

Adjustment data in the Install Window

You can look at the values of the gas and crucible factors in the STARe Software Install Window, in addition to other adjustment data.

To display the adjustment data in the Install Window:

(1) Choose Topic/Module.

(2) Click the Adjustment Overview button

adjustment overview

The Adjustment Overview dialog box appears. If you have not performed an adjustment on your STARe module, the dialog box shows the default data from the STARe Software database. The following figures show the dialog boxes of the individual STARe modules with the corresponding adjustment data.

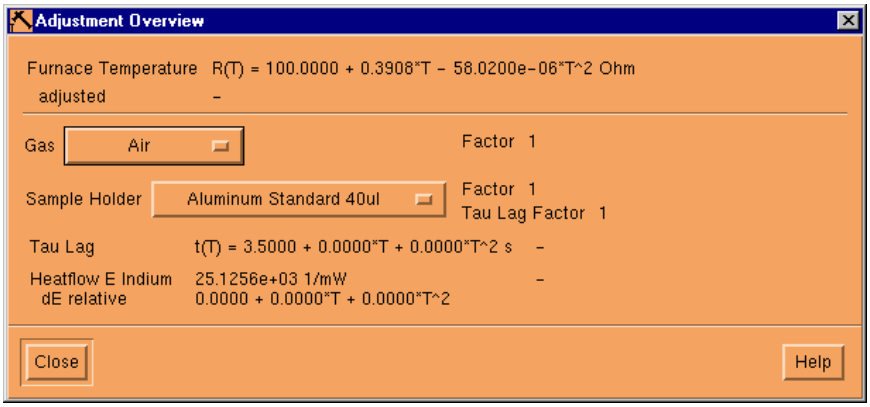


Fig. 8-11. Adjustment data of a STARe DSC module (default values of the STARe database)

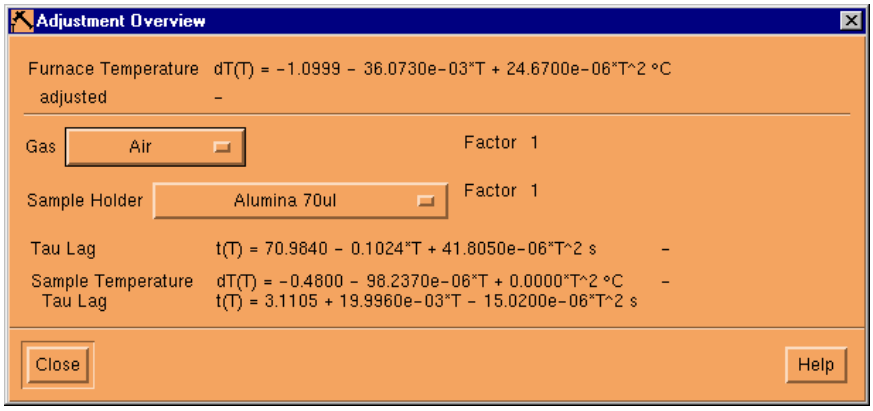


Fig. 8-12. Adjustment data of a STARe TGA module

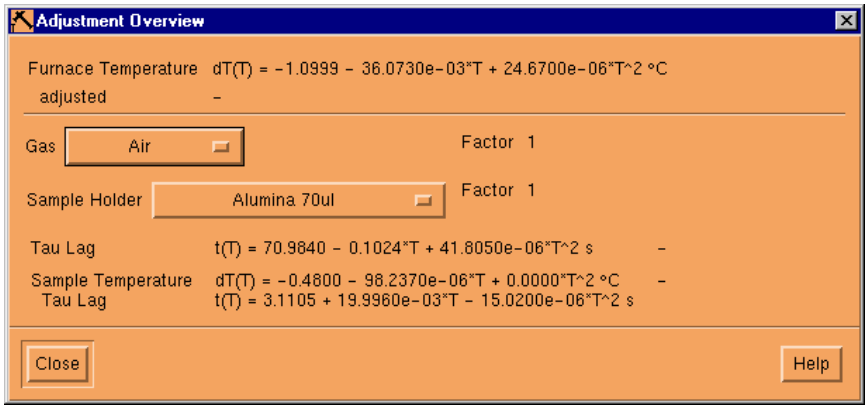


Fig. 8-13. Adjustment data of a STARe TMA module.

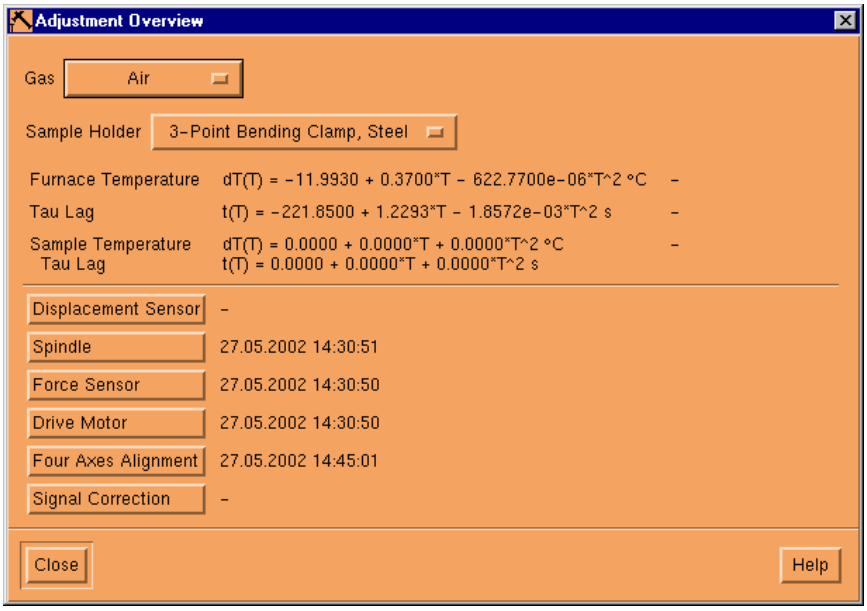


Fig. 8-14. Adjustment data of a STARe DMA module.

♣ The dates of the furnace adjustment and the crucible holder or sample support are shown separately in the dialog box. If the STARe Software determined the adjustment parameters according to the FlexCal calculation model, then the calibration date appears in brackets. If you have not yet adjusted your STARe module, no calibration date appears, and the STARe Software uses the default values.

gas and crucible factors

(3) To display other gas and crucible factors, click the Gas or the Crucible button and select the type of gas or crucible desired.

The values stored in the STARe Software for the crucible or gas factors selected are displayed in the dialog box.

8.4.8 Performing the adjustment

The result of a calibration measurement determines whether you have to adjust your STARe module. With all STARe modules, the first part of the adjustment procedure is a calibration that determines the deviation of your module from a reference state. At the end of the calibration, you can confirm the new calibration values in an information box and proceed with the adjustment, or else discard the new calibration values (see also *Fig. 8-8*).

An exception to this is total adjustment. Here the deviation from the actual adjustment state is displayed and not the deviation from the reference state.

adjustment possibilities

The STARe Software offers the following possibilities for performing adjustments:

- Adjustment with a multiple sample crucible
- Adjustment using the mean value of several measurements with single samples
- Adjustment using one measurement of a single sample
- Adjustment through the manual entry of your own results or calibration data

♣ The table at the end of this chapter presents an overview of the different calibration and adjustment possibilities.

The following sections describe the first three adjustment possibilities. Instructions for the manual entry of calibration data can be found in the appendix *Adjustment through the manual entry of adjustment data*.

Adjustment with a multiple sample crucible

EvalMacro

You can adjust your STARe module automatically using a calibration method labeled Total, Multi or, with TGA and TMA modules, SampleH. In the multiple sample crucible, i.e. a crucible containing several different samples, there are at least two reference substances. Your module performs the temperature program defined by the method, and the STARe Software evaluates the adjustment measurement in automatic evaluations. In a dialog box of the Module Control Window of the STARe Software, you have the possibility of performing or canceling the adjustment.

total adjustment

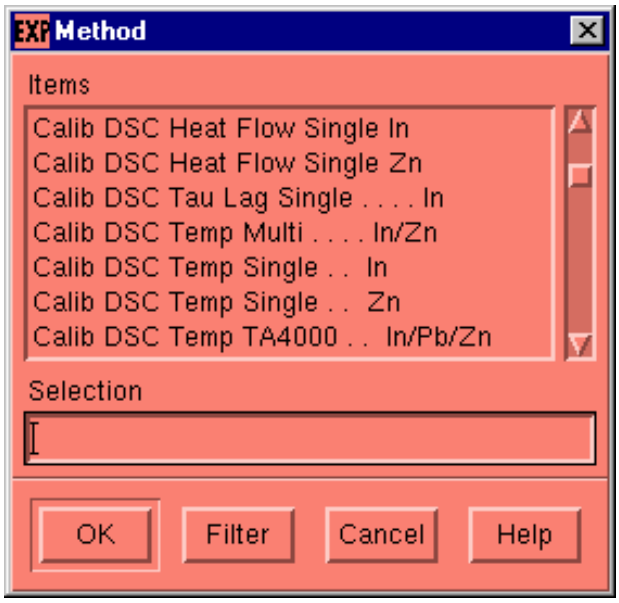
A typical example of an adjustment performed using a multiple sample is a total adjustment using a method named Total.

To adjust your module with a multiple sample crucible:

The desired adjustment method is in the database and your STARe module is ready.

(1) Open an Experiment Window. Click the select method button and select the item select method in the drop-down list.
The Method dialog box appears.

Experiment Window



(2) In the list, select the desired adjustment method labeled Multi, Total or, with the TGA and TMA modules, SampleH, and then click OK.
The data relating to the selected method is transferred to the Experiment Window. The corresponding entries appear in the input and text boxes. In the middle text box instructions on sample preparation appear. Besides the measuring method (TG, DSC, TMA etc.), the label Calibration appears next to the text box to indicate that it is a calibration experiment.

(3) Prepare your sample for the experiment by following the instructions on sample preparation in the middle text box.

sample preparation

(4) Supply the information on crucible weight, sample weight or sample length missing from the Experiment Window by weighing or measuring the sample and entering the data in the appropriate Experiment Window box.

crucible weight,
sample weight
sample length

send experiment

(5) Send the experiment to your S**TA**R^{e} module by selecting the module name in the Module list box and clicking Send Experiment.

The S**TA**R^{e} Software sends the experiment to the module, which then performs the adjustment measurement and evaluates the adjustment measurement automatically.

With a tau lag adjustment:

coefficients for the adjustment

An information box appears with the values and coefficients determined from the measurement for the adjustment. The old values are shown in brackets.

With a temperature adjustment:

An information box appears with the values and coefficients determined from the measurement for the adjustment. The old values are shown in brackets.

With a heat flow calibration:
An information box with the measured heats of fusion appears. The literature values are displayed in brackets next to the measured values. The box also contains the new value for E_{In} with the old value next to it in brackets.

With a total adjustment:
An information box appears with the calibration values. The old values are given in brackets.

In all information boxes: The deviations of the calibrated quantities (tau lag, temperature or heat flow) from the actual state of adjustment are given for 25 °C and 500 °C.

♣ With DSC calibrations it is possible that the automatic evaluations give incorrect calibration values because of artifacts in the calibration measurement curves. For example, for a total adjustment it is possible that values appear in the information box that deviate from the usual values (e.g. of the coefficient A in the tau lag correction function). You can overwrite these values by entering you own standard values manually in the corresponding boxes of the information box.

♣ You can look at the current adjustment values of your module in the Install Window under Topic/Module by clicking the Adjustment Overview button, and use them as standard values.

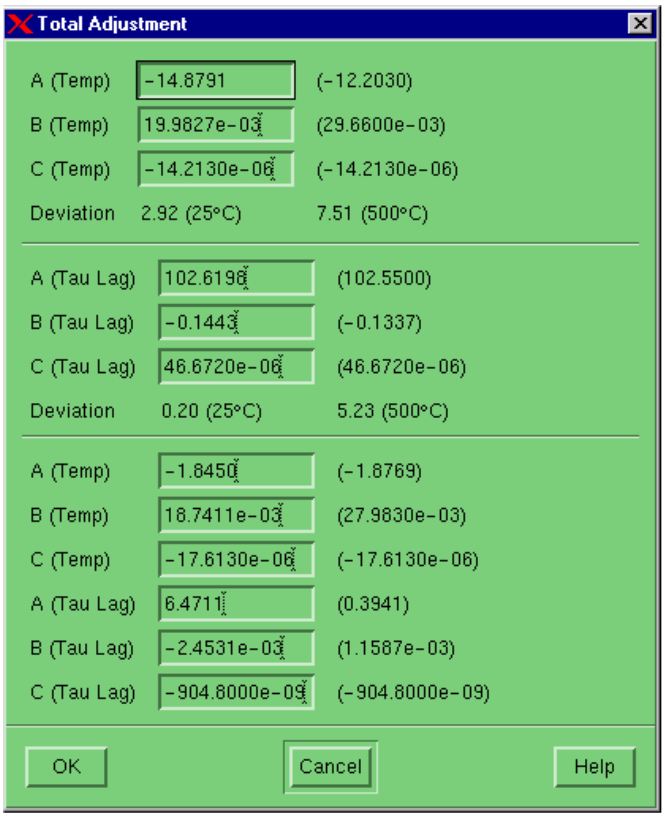


Fig. 8-15. Example of an information box at the end of a TGA total adjustment

(6) Confirm or cancel adjustment values:

If the newly determined calibration values are outside the set limits of permissible error: click OK to accept the new calibration values and to adjust your module.

The STARe Software adjusts your module with the new values.

or

If you want to retain the current calibration state: click Cancel to reject the new calibration values.

The STARe Software discards the new values and retains the current state of adjustment.

♣ The information box stops the sample robot. You can proceed to the next experiment by clicking OK or Cancel.

stopping the sample robot

Adjustment by means of several measurements with single sample crucibles

You can adjust your STARe module by measuring several single samples. This type of adjustment allows you to determine all the coefficients of the quadratic approximation functions for the calibration of temperature, tau lag or heat flow.

measurement accuracy and mean value calculation

To achieve the best possible measurement accuracy with your STARe module, you can adjust the module with a mean value of several single measurements. The measurement values are obtained from repeated measurements with a single sample crucible. Typically two or three measurements are performed with each reference substance.

Your module executes the temperature program defined by the method. The STARe Software evaluates the individual adjustment measurements and stores the results in a table. You can reject individual results in a dialog box in the Module Control Window. The STARe Software uses all the entries in the table up to the current position for the calibration.

- The calibration does not take any entries after the current position into account.
- At the end of the calibration process, you can call up an information box containing the results of the calibration. The STARe Software does not readjust your module until you confirm the values in this dialog box with OK. In this type of adjustment, the dialog box does not appear automatically after the measurement.

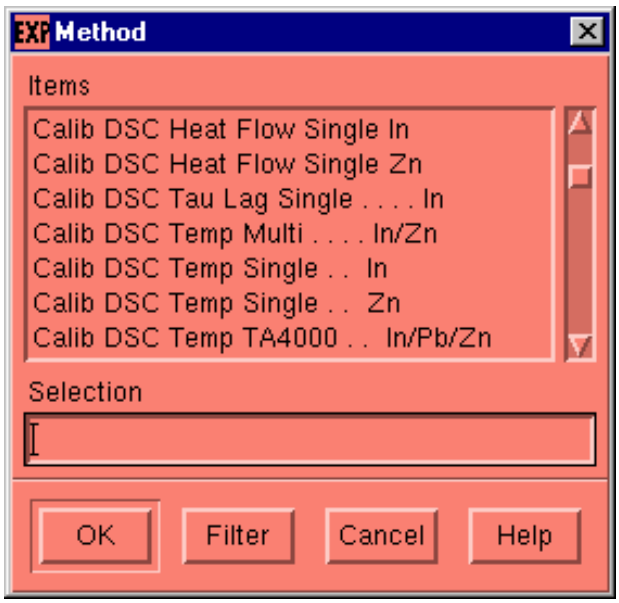
To adjust your module with several measurements of single samples:

The desired direct adjustment method is available in the database and your STARe module is ready.

selecting the adjustment method in the Experiment Window

(1) Open an Experiment Window. Click the select method button and select the item select method in the drop-down list.

The Method dialog box appears.



(2) Select the desired adjustment method labeled Single in the list and click OK.

The Experiment Window accepts the data of the selected method. The corresponding entries appear in the text boxes. In the middle text box, instructions on sample preparation appear.

sample preparation

(3) Prepare your sample for the experiment by following the instructions for sample preparation given in the box.

crucible weight, sample weight sample length

(4) Supply the information on crucible weight, sample weight or sample length missing from the Experiment Window by weighing or measuring the sample and entering the data in the appropriate box of the Experiment Window.

(5) Send the experiment to your STARe module by selecting the module name in the Module list box and clicking Send Experiment.

The STARe Software sends the experiment to the module, which then performs the adjustment measurement, and evaluates it automatically. The software does not, however, display the measurement result directly in an information box.

mean value

(6) To perform several experiments with the same sample to obtain the mean value: repeat step 5 several times.

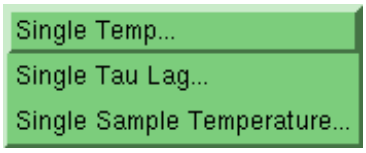
(7) To perform experiments with another sample: repeat steps 2 to 6.

(8) When you have completed the measurements, select the Calib/Adjust menu in the Module Control Window

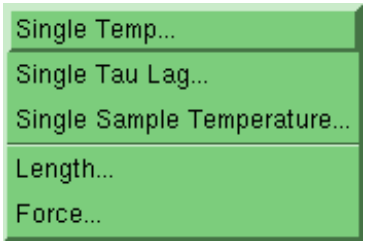
A drop-down list appears with the different types of calibration available for your module:

DSC:

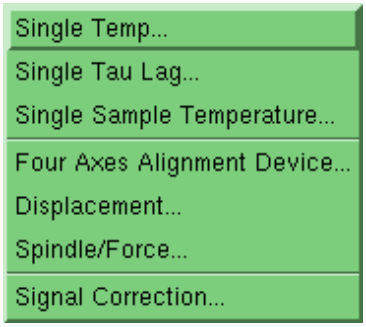
TGA:



TMA:



DMA:



(9) Select the type of calibration you want to perform.

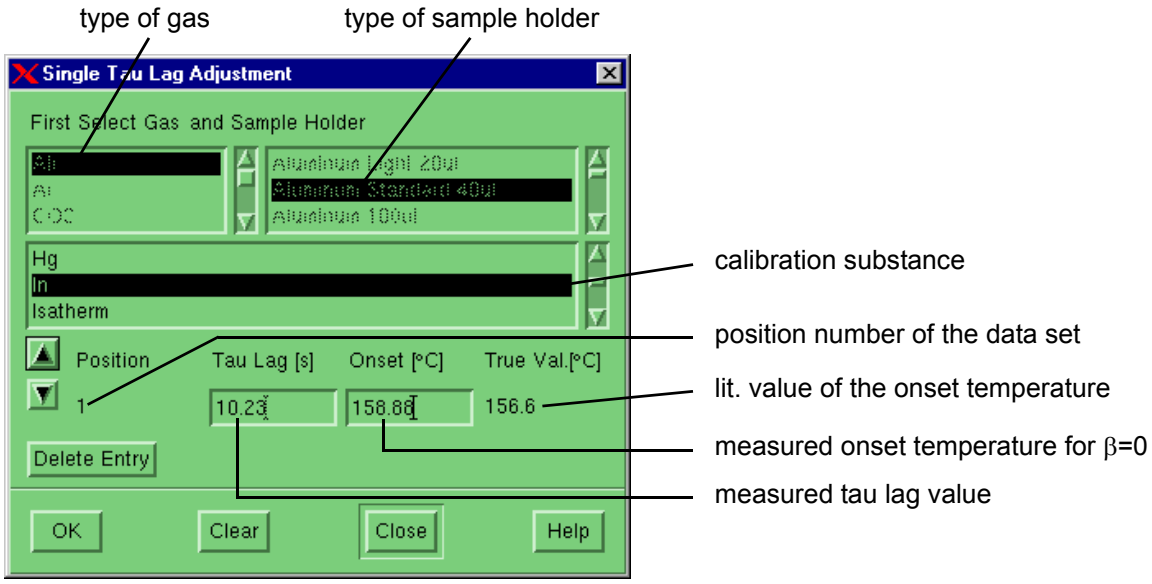
A dialog box appears with the measurement values of your calibrations.

The name of the dialog box corresponds to the type of calibration (e.g. Temperature Calibration) and contains the data sets from your latest calibration measurements. You can move from one data set to the other by clicking the ▲ and ▼ arrow buttons. The number of each dataset is given under Position.

sample changer ♣ This dialog box does not stop the sample changer.

♣ Only the data from the latest calibration measurements appear in the boxes of the dialog box. If you have not performed any calibration measurements since the last adjustments, the value 0 (zero) appears in the data boxes. After an adjustment, the new calibration data are sent to the STARe module and the data sets in the dialog box deleted.

- Dialog box for the tau lag adjustment of the DSC, TGA and TMA:



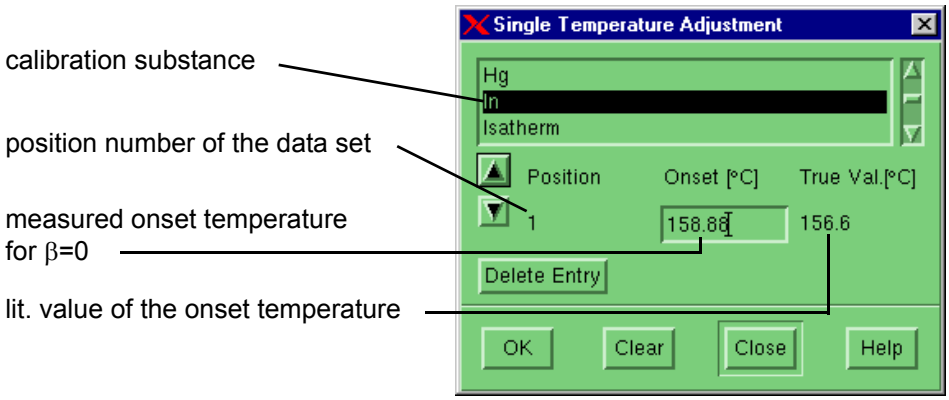
The boxes displaying the type of gas, crucible and reference substance are in the upper part of the dialog box. The boxes for the type of gas and crucible remain unchanged when the arrow button is clicked.

The box labeled Tau Lag in the lower part of the dialog box displays the measured value of tau lag and the measured onset temperature for β=0. The measured value of the onset temperature does not affect the tau lag calibration.

The box labeled True Val. displays the literature value of the onset temperature.

♣ The STARe Software only includes the data sets up to the current position number in the adjustment.

- Dialog box for the temperature adjustment of the DSC, TGA and TMA:

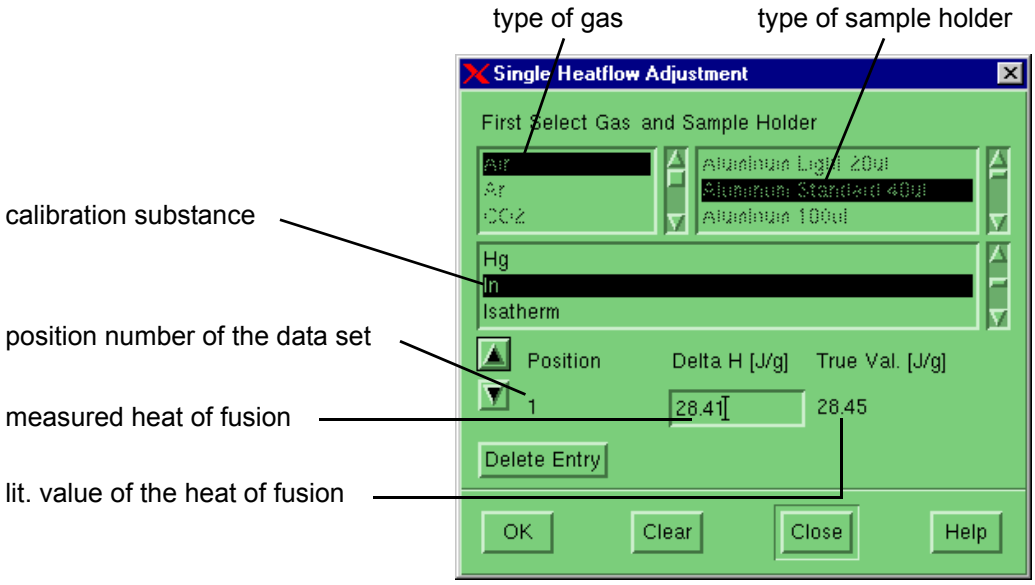


The box displaying the calibration substance is in the upper part of the dialog box.

The box labeled Onset displays the measured onset temperature of the calibration substance. The literature value of the onset temperature is displayed next to this under True Val.

♣ The S**TA**R^{e} Software only includes the data sets up to the current position number in the adjustment.

- Dialog box for the heat flow adjustment of the DSC:



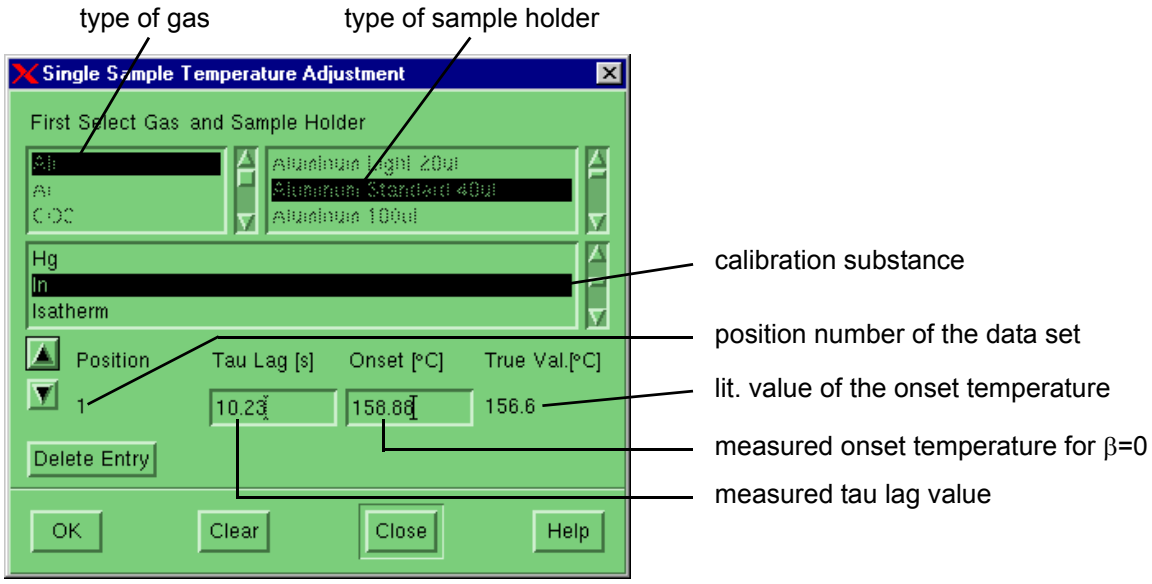
The boxes displaying the type of gas, crucible and calibration substance are in the upper part of the dialog box. The boxes for the type of gas and crucible remain unchanged when the arrow button is clicked.

The box labeled Delta H in the lower part of the dialog box displays the measured value of heat of fusion of the calibration substance. The literature value of the heat of fusion is displayed next to this under True Val.

♣ The S**TA**R^{e} Software only includes the data sets up to the current position number in the adjustment.

- Dialog box for adjustment of the TGA crucible holder and TMA sample support:

♣ This dialog box allows you to perform a tau lag adjustment and a crucible holder or sample support adjustment simultaneously.



The boxes displaying the type of gas, crucible and calibration substance are in the upper part of the dialog box. The boxes for the type of gas and crucible remain unchanged when the arrow button is clicked.

The box labeled Tau Lag in the lower part of the dialog box displays the measured tau lag and onset temperature values. Next to this, the box labeled True Val. displays the literature value of the onset temperature.

(10) Select the desired data set by clicking the ▲ and ▼ arrow buttons. To delete one or more data sets:

- To delete one data set: click Delete entry.
- To delete all the data sets of your adjustment measurements: click Delete.

Clicking the Delete button deletes all the data of your adjustment measurements.

(11) If you want to take several single measurements into account:

mean value

- Change to the last entry of the series of measurement data using the ▲ and ▼ arrow buttons. Make sure that you include all the desired data sets in the adjustment by selecting the appropriate position number. The STARe Software only includes data sets up to the current position number in the adjustment. The current position number is given under Position.
- Click OK

  The STARe Software calculates the mean value from all the measured data up to the current position. If you have performed calibration measurements with several substances, the STARe Software calculates the mean value for each substance.

  An information box with the newly calculated calibration data appears. The values of the current adjustment state appear next to these in brackets. The deviations of the calibrated quantities (tau lag, temperature or heat flow) from current state of adjustment at 25 °C and 500 °C are also displayed.

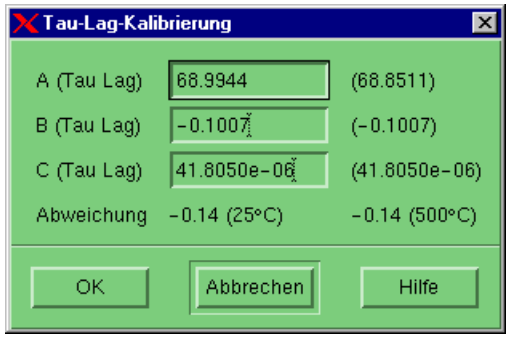


Fig. 8-16. Example of an information box at the end of a tau lag calibration

♣ You can look at the current values of the adjustment of your module in the Install Window under Topic/Module by clicking the Adjustment Overview button, and can use them as standard values.

(12) Confirm or reject adjustment values:

If the newly determined calibration values are outside the limits of permissible error that you set: click OK to accept the new calibration values and to adjust your module.

The STARe Software adjusts your module with the new calibration values.

or

If you want to retain the current calibration state: click Cancel to reject the new calibration values.

The STARe Software discards the new values and retains the current state of adjustment.

Calibration using Checks

The S**TA**R^{e} Software allows you to perform calibrations with so-called Checks, in addition to the calibration methods described in the section *Calibration methods in the STARe Software, page 8-23*. Checks are normal methods of the "None" type to which automatic evaluations (EvalMacros) have been added. EvalMacros perform evaluations on the measurement curve (e.g. onset determinations) and check whether the measured values of your S**TA**R^{e} module are within the limits of permissible error. At the end of the check, a text informs you about the result of the evaluation. In addition, you obtain the graphical evaluation of the calibration in the Evaluation Window.

limits of permissible error of the measurement accuracy

The checks supplied with the S**TA**R^{e} Software database are based on the S**TA**R^{e} instrument specifications and therefore have very narrow limits of permissible error for measurement accuracy as the following table shows:

Substance	Onset temperature (temperature calibration)	Heat of fusion (heat flow calibration)
Indium	156.6 ± 0.3 °C	28.45 ± 0.6 J/g
Zinc	419.6 ± 0.7 °C	107.5 ± 3.2 J/g

Table 8-5: The limits of permissible error for measurement accuracy included in the S**TA**R^{e} Software Checks

You can change the limits of permissible error according to you individual requirements. When setting the limits of permissible error, keep to the principle “as accurate as necessary”.

To perform a Check:

(1) Click Select method in the Routine Window or Experiment Window and Select method in the drop-down list.

The Method dialog box appears with a list of the methods included in the STARe database. The names of calibration methods begin with Check.

Checks

DSC Checks:	TG Checks:	TMA Checks:
Check DSC^endo In	Check TG50...Ni	Check TMA40...In
Check DSC^end Zn	Check TGA851.....In/Al	Check TMA840/841...In/Zn
Check DSC^exo In	Check TGA851.....Pd	
Check DSC^exo Zn		

(2) Select the desired method.

♣ To find the desired method more quickly, you can use the filter function and enter Check* in the method name box. You can also select the method group Checks in the method filter.

filter function

(3) Prepare the sample for the calibration measurements. The sample preparation varies according to the type of module and measurement. Follow the instructions in the text box in the Experiment Window. Pay attention to additional instructions on sample preparation in the section *Sample Preparation*:

(4) Start the experiment by clicking Send Experiment.

The STARe Software calibrates your module by sending the experiment to the module for execution. With STARe DSC and TMA modules the STARe Software evaluates the measurement automatically. If the measured calibration values are within the limits of permissible error, the message The temperature is within specifications! appears. If the measured values are outside the limits of permissible error, the message Please calibrate the module! appears.

(5) With S**TA**R^{e} TGA modules:

- Open the calibration curve in the Evaluation Window after the calibration measurements.

SDTA curve

- Click TA/SDTA.

  The S**TA**R^{e} Software generates an SDTA curve.

- Remove the TGA curve by clicking the ordinate axis and selecting Edit/Delete or simultaneously pressing the SHIFT and DELETE keys.

  Only the SDTA curve remains displayed on the screen.

- Select File/EvalMacro open/AAA

The S**TA**R^{e} Software evaluates the calibration measurement. If the measured calibration values are within the limits of permissible error, the message The Module is within specifications! appears. If the measured values are outside the limits of permissible error, the message Please calibrate the module! appears

TG50 module

♣ With the TG50 module the calibration measurements are evaluated automatically. It is therefore not necessary with this module to evaluate the calibration measurements in the Evaluation Window.

(6) If the measured calibration values are outside the limits of permissible error: adjust your S**TA**R^{e} module.

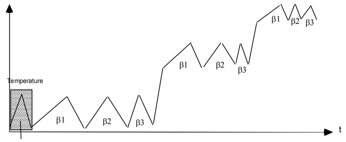
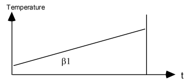
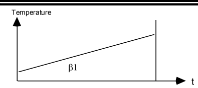
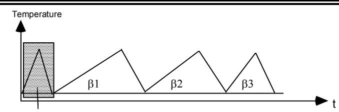
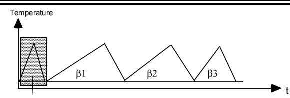
or:

If the calibration measured values are within the limits of permissible error: you do **not** need to adjust your S**TA**R^{e} module.

You have performed the Check and now know whether your S**TA**R^{e} module meets the specified limits of permissible error of measurement accuracy. The procedure for adjustment is described in the section above.

8.5 Summary of adjustment possibilities

Summary

Adjustments	Number of measurement	Number of substances	Temperature program	Limits
1. Total Adjustment Determination of all needed adjustments for the module with one measurement. All adjustments are performed simul-taneously. The Total adjustment includes three adjustments (temperature, tau lag and heat flow for the DSC module; temperatur-, tau lag and sample holder adjustment for the TGA module.				
• Total Adjustment	1	≥ 2	for each substance ≥ 3 different heating rates	Measured values displayed in an information window
2. Temperature Adjustment Determination of Onset temperatures of metals (Indium, Zink, Aluminum).				Indium DSC: 156.6 ± 0.3°C TGA: 156.6 ± 1.0°C
• Temperature Multiple	1	≥ 2	1 dynamic segment	Measured values displayed in an information window
• Temperature Single	≥1	1	1 dynamic segment	Measured values saved in table *
• Temperature TA4000	1	≥3	1 dynamic segment	Measured values displayed in an information window
3. Heat Flow Adjustment Determination of the calorimetric sensitivity E_{In} of the DSC sensor.				Indium DSC: 28.45 ± 0.6J/g
• Heat Flow	1	1	1 dynamic segment	Measured values displayed in an information window
• Heat Flow Single	≥ 1	1	1 dynamic segment	Measured values saved in table *
4. Sample Holder Calibration				Indium TGA: 156.6 ± 1.0°C Difference between measurements (β1 - β2) TGA: ± 0.5°C
• Sample Holder	1	1	≥ 3 different heating rates (β)	Measured values displayed in an information window
• Sample Holder Single	≥ 1	1	≥ 3 different heating rates (β)	Measured values saved in table
5. Tau Lag Calibration Determination of the time constant Tau Lag.				Difference between measurements (β1 - β2): DSC: ± 0.2°C TGA: ± 0.5°C
• Tau Lag Single	≥1	1	≥ 3 different heating rates (β)	Measured values saved in table *

* To perform the calibration, you must open the dialog box of the Corresponding „Single“ calibration in the Module Control Window on the Calibration menu and confirm with OK.

9 Glossary

In this section you find the terms applied in conjunction with the user interface of the STARe Systems.

♣ An arrow (→) refers to another term in the glossary.

blank curve

Blank Curve: The blank curve is measured under exactly the same conditions as a subsequent sample curve except without the sample. These conditions include the type of crucible, the type and flow rate of the purge gas, the → sampling interval and the temperature program. Blank and sample experiments use the same → method. The blank curve should be reproducible within a narrow range and contain all systematic deviations from the ideal zero line, e.g. caused by thermal asymmetries in a DSC cell.

blank subtraction

Blank Subtraction: Subtracting the → blank curve from a sample curve measured under identical conditions results in a curve free from systematic deviations caused by the equipment. Blank subtraction is mainly important for c_p determination and thermogravimetry.

In the STARe Software automatic blank subtraction takes place during the measurement: the online curve visible in the module control window and the saved curve are blank corrected. The **automatic blank subtraction** requires a method with Subtract Blank Curve under Miscellaneous. In the Experiment Window the Run Blank Curve check box is selected when running the blank experiment. The subsequent sample measurements (run with Run Blank Curve check box cleared) all are corrected by the latest blank curve available.

Manual blank subtraction is also possible by using the Difference command on the Math menu in the Evaluation Window. But in this case the user is responsible to keep identical conditions.

controlled power down sequence

Controlled power down sequence: a procedure that stops orderly all processes running in the STARe Software. You click Exit on the System menu in the main menu bar and confirm the dialog box with the question Do you really want to quit? with Yes.

coordinate system

Coordinate system: In the coordinate system of a diagram two coordinates (ordinate and abscissa) specify the location of a curve point in a plane. A diagram contains one or several coordinate systems. Each system has its ordinate and abscissa in the respective units and color. All graphic objects belong to exactly one coordinate system. A new coordinate system is automatically generated when a new curve is displayed whose units don't fit in the existing coordinate system. You delete a coordinate system by selecting its ordinate or abscissa scale and clicking Edit/Cut. This deletes all graphic objects that belong to the respective coordinate system.

Curve: The output of a sensor gives a measured curve. It consists of a series of values of the property of interest. In the STARe Software there are also calculated curves, such as the integral or a first derivative curve. They can be evaluated just like measured curves.

equidistance

Equidistance: Equal distance between a series of values, e.g. curve points (→ sampling interval) or table rows. Measured curves are time equidistant. Tables can be time, reference temperature, sample temperature, or ordinate equidistant.

evaluation

Evaluation: The STARe Software offers **graphic evaluations** e.g. zooming a section of a curve to full screen and **general TA evaluations** such as integrating over a certain baseline or an onset determination. In addition, **software options** can be purchased separately.

An evaluated curve (= diagram) can be stored in the data base as an “evaluation”.

EvalMacro

EvalMacro: The evaluation macro is a listing of evaluation instructions obtained by an interactive evaluation of an example curve. You can store the EvalMacro and apply it to another curve. For later routine use you save the EvalMacro and include it in a method. Furthermore there is a possibility to validate (→ validation) the analysis automatically by defining an OK range for the results in question.

The EvalMacro is a software option and can be purchased separately. Without this option a method with an EvalMacro (e.g. one of the tutorial methods) can still be applied.

experiment

Experiment: The STARe experiment is the complete description of a particular measurement. The experiment is composed in the experiment window by selecting a method and entering the sample name and weight. Automatically, the user name, i.e. the operator or owner of the experiment, is included. The experiment is sent to and performed on an connected TA module. The primary goal of the measurement is to obtain a thermoanalytical curve. After finishing an experiment, the information is completed by the results of the experiment (measured curve, optional automatic evaluation). The experiment automatically is stored under the name of the sample.

digits

Digits: Numerical results are calculated with a large number of digits. When displayed, the number of digits after the decimal point has to be limited to avoid unnecessary digits (for example an onset value of 5.7198 °C). Usually, the number of decimals is two, e.g. an Onset of 5.72 °C. If a figure is smaller than 0.10, this rule would lead to bad resolution (for example a delta cp of 0.03 $Jg^{-1}K^{-1}$). To solve the problem, figures < 0.1 are displayed in scientific format, e.g. 37.19e -03 $Jg^{-1}K^{-1}$. (= $37.19 * 10^{-3}$ $Jg^{-1}K^{-1}$).

filter

Filter: In a database a filter is used to efficiently find the desired information. In the STARe System it serves for instance to retrieve measured curves. You can sort out your curves according to the following criteria:

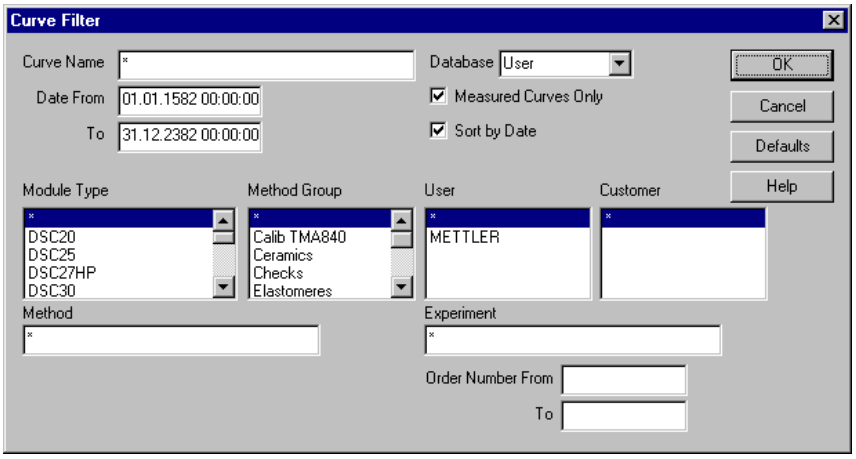


Fig. 9-1: The curve filter with its different selection boxes.

With the highlighted module type DSC822e and the default settings (*) for all the other items just the curves measured with the DSC822e will be sorted out. The asterisk (*) is used as a “wild card“ and represents any text.

Example: If you remember the beginning of a curve name only, use e.g. Copper*. Try also copper* because the filter distinguishes upper and lower case (or simply try *opper*). If it's the middle part of a curve name you remember, use e.g. *sulfa*. The second * stands for any ending.

If you want all curves named test1 through test9 enter test?. But this does not find e.g. test 11.

In the default setting the check box Measured Curves is selected and only measured curves (but not calculated curves) are displayed.

Forced Start

Forced Start: is a means to skip certain stages before and after the actual measurement by pressing the OK key on the module. For more information see the online Help to the STARe software.

FPO

FPO: Furnace *P*ower switched Off to save energy at the end of an experiment series. For more information see Module Control Window Help.

interactive window

Interactive window: An application within the STARe Software with an individual Windows interface in which particular tasks can be performed. Windows often overlap each other like sheets of paper on your desk. You can regroup interactive windows, moving them to the front or the back of the other interactive windows.

FOP

FOP: Furnace Open Permission (default: off): During a measurement, you may not open the automatic cover of the DSC822e furnace. FOP = on means that the opening is possible even during a measurement (key FURNACE on the DSC822e keypad). For more information see Module Control Window Help.

method

Method: A method is the sum of all instructions which define the execution of a measurement (e.g. temperature program) and the evaluation of the measured → curve. Complex temperature programs are split into several → segments. A new method is composed in the **method editor** (or Method Window).

pan

Pan: container for the sample to be measured. Also called crucible.

Purge Gas: The purge gas creates a defined furnace atmosphere in the sample chamber by flushing out air and the volatile products evolved by the sample. The purge gas and its flow rate, e.g. 50 ml/min, is defined in the → segments of a → method.

routine analysis

Routine Analysis: Any repetitive investigations in R+D, product development, competitive product analysis, or quality control. Routine analyses justify the creation of a → method prior to the → experiment.

sampling Interval Scroll Bar

Sampling Interval: Time distance between stored data points. It is also called → equidistance. Default is 1 s; minimum is 0.1 s; maximum is 3600 s. Sampling Interval is accessible under Miscellaneous in the Method Window.

segment

Segment: Each dynamic or isothermal part of a temperature program is called a segment.

TEB

TEB: The temperature end behavior can be defined in the experiment buffer. For more information see Module Control Window Help.

user

User: When you start S**TA**R**e** Software, a dialog box with the STARe User Authentication dialog box appears (this dialog box is also available under System/Log On As User...). After entering username and password and confirming with OK all windows you open afterwards are assigned to your username.

If the software option Install Plus is installed, several users can use the S**TA**R**e** Software simultaneously. One user can open, say, an Experiment Window and an Evaluation Window. Another user can open the windows assigned to his name.

When you use your own Experiment Window the measured curves or created methods are assigned to the right user.

user interface

User Interface: Interface between the program and the user of the PC. The primary goal of the user interface is to increase the user effectiveness and satisfaction. The color screen with its window technique is the most important part of the user interface.

validation

Validation: To validate the analysis automatically, an OK range for the results in question can be defined. If all results are within the specified ranges, an "OK Text" will be displayed, such as Examination passed. With one or more results being outside the ranges, a "KO Text" appears, such as Failed.

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STARe Software User Handbook

Subject to technical changes and to the availability of the accessories supplied with the instruments

ME-51710263D

Printed in Switzerland 0509/2.12

Mettler-Toledo GmbH, Sonnenbergstrasse 74, CH-8603 Schwerzenbach, Switzerland, Tel. (01) 806 77 11, Telefax (01) 806 73 50, Internet: http://www.mt.com

A	**Mettler-Toledo GmbH., A-1100 Wien**	**A**	**Mettler-Toledo Ltd., Port Melbourne, Victoria 3207**	**B**	**N.V. Mettler-Toledo s.a., B-1651 Lot**
T	**Tel. (01) 604 19 80, Fax (01) 604 28 80**	**U**	**Tel. (03) 9644 57 00, Fax (03) 9645 39 35**	**E**	**Tel. (02) 334 02 11, Fax (02) 378 16 65**
C	**Mettler-Toledo (Schweiz) AG, CH-8606 Greifensee**	**C**	**Mettler-Toledo (Shanghai) Ltd., Shanghai 200233**	**C**	**Mettler-Toledo, spol, s.r.o., CZ-12000 Praha 2**
H	**Tel. (01) 944 45 45, Fax (01) 944 45 10**	**N**	**Tel. (21) 6485 04 35, Fax (21) 6485 33 51**	**Z**	**Tel. (02) 25 49 62, Fax (02) 242 475 83**
D	**Mettler-Toledo GmbH, D-35353 Giessen**	**D**	**Mettler-Toledo A/S, DK-2600 Glostrup**	**E**	**Mettler-Toledo S.A.E., E-08038 Barcelona**
E	**Tel. (0641) 50 70, Fax (0641) 507 128**	**K**	**Tel. (43) 270 800, Fax (43) 270 828**	**S**	**Tel. (03) 223 22 22, Fax (03) 223 02 71**
F	**Mettler-Toledo s.a., F-78222 Viroflay**	**H**	**Mettler-Toledo (HK) Ltd., Kowloon**	**H**	**Mettler-Toledo, d.o.o., HR-10000 Zagreb**
R	**Tel. (01) 309 717 17, Fax (01) 309 716 16**	**K**	**Tel. (02) 744 12 21, Fax (02) 744 68 78**	**R**	**Tel. (01) 230 41 47, Fax (01) 41 233 63 17**
H	**Mettler-Toledo, KFT, H-1173 Budapest**	**IT**	**Mettler-Toledo S.p.A., I-20026 Novate Milanese**	**J**	**Mettler-Toledo K.K., Yokohama 231**
U	**Tel. (01) 257 70 30, Fax (01) 257 21 75**		**Tel. (02) 333 321, Fax (02) 356 29 73**	**P**	**Tel. (45) 633 53 50, Fax (45) 664 96 50**
K	**Mettler-Toledo (Korea) Ltd., Seoul (135-090)**	**K**	**Mettler-Toledo CA, 480009 Almaty**	**M**	**Mettler-Toledo (M) Sdn. Bhd., 47301 Petaling Jaya**
R	**Tel. (02) 518 20 04, Fax (02) 518 08 13**	**Z**	**Tel. (07) 3272 608 834, Fax (07) 3272 608 835**	**Y**	**Tel. (03) 703 27 73, Fax (03) 703 17 72**
M	**Mettler-Toledo (S.E.A.), 47301 Petaling Jaya**	**M**	**Mettler-Toledo S.A. de C.V., México C.P. 06430**	**N**	**Mettler-Toledo B.V., NL-4000 HA Tiel**
Y	**Tel. (03) 704 17 73, Fax (03) 703 17 72**	**X**	**Tel. (05) 547 57 00, Fax (05) 541 22 28**	**L**	**Tel. (0344) 638 363, Fax (0344) 638 390**
P	**Mettler-Toledo, Sp. z o.o., PL-02-929 Warszawa**	**R**	**Mettler-Toledo C.I.S. AG, 10 1000 Moskau**	**S**	**Mettler-Toledo AB, S-12008 Stockholm**
L	**Tel. (22) 651 92 32 Fax (22) 651 71 72**	**U**	**Tel. (95) 921 92 11, Fax (95) 921 63 53**	**E**	**Tel. (08) 702 50 00, Fax (08) 642 45 62**
S	**Mettler-Toledo (S) Pte. Ltd., Singapore 139944**	**S**	**Mettler-Toledo, SK-83103 Bratislava**	**SI**	**Mettler-Toledo, d.o.o., SI-1236 Trzin**
G	**Tel. 7 786 779, Fax 7 764 904**	**K**	**Tel. (07) 522 74 96, Fax (07) 237 190**		**Tel. (06) 116 218 01, Fax (06) 116 217 89**
T	**Mettler-Toledo (Thailand) Ltd., Bangkok 10310**	**T**	**Mettler-Toledo Pac Rim AG, Taipei**	**U**	**Mettler-Toledo Ltd., Leicester, LE4 1AW**
H	**Tel. (02) 719 64 80, Fax (02) 719 64 79**	**W**	**Tel. (2) 2579 5955, Fax (2) 2579 5977**	**K**	**Tel. (0116) 235 70 70, Fax (0116) 236 63 99**
U	**Mettler-Toledo, Inc., Worthington, OH 43085**				
S	**Tel. (614) 438 4511, Fax (614) 438 4900**				

For all other countries: Mettler-Toledo GmbH PO Box VI-400, CH-8606 Greifensee Phone +41-1-944 22 11, Fax +41-1-944 31 70