For more information on the Outlier Test, see *Outlier Test*, p. 147.

Chapter Eight Method Validation

1. Method Validation Overview

The Method Validation software tests the variance and the linearity of the data. It also determines performance characteristics according to the requirements of DIN 38402-Part 51.

For method validation according to DIN regulations, follow these steps:

1. Select the estimated working range.
2. Prepare 10 calibration standards, with concentrations distributed uniformly over the complete working range.
3. Create a sample table, with 10 injections of the highest and lowest standard concentrations and a single injection for all other standards. If more than one injection is made of the intermediate standards, the linearity test is calculated with the results of the first replicates only.
4. Analyze these standards. The results are saved in a *.cal* file.
5. Select Method Validation from the TOC Control Validation menu, or select DIN 38402 from the Toccntr program folder. The Method Validation screen appears.
6. From the Options menu, ensure that *Validation Strictly Following DIN* is enabled. If not, select the menu item.
7. Select Open from the File menu.
8. Select *.cal* from the file type drop-down list.
9. From the File Name list, select the *.cal* file where the measurement data was saved. Then select OK.
10. The calculations are automatically performed, and the validation data displayed.

Complete Datasheet

Smpl [i]	Smpl.ID	Xi [ppm]	Yi,1	Yi,2	Yi,3	Yi,4
1	1: KHP	10.000	4280	4231	4244	4249
2	2: KHP	20.000	8306	8372	8432	
3	3: KHP	30.000	12667	12599	12777	
4	4: KHP	40.000	16993	17005	17032	
5	5: KHP	50.000	20693	20784	21058	
6	6: KHP	60.000	25914	26046	25311	
7	7: KHP	70.000	29725	30039	30645	
8	8: KHP	80.000	37448	38098	37857	
9	9: KHP	90.000	39045	39515	39804	
10	10: KHP	100.000	44569	43574	44698	44163

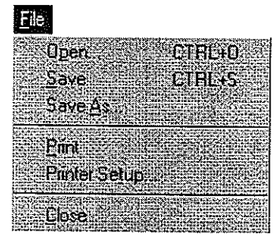
The validation data is displayed

11. Select Save from the File menu, assign a file name in the Save As dialog box, and select OK. The validation data is saved with a .val file name extension.

Note: *When the actual calibration function doesn't meet the DIN specification, check the instrument for system errors, or reduce the working range, then repeat the Validation.*

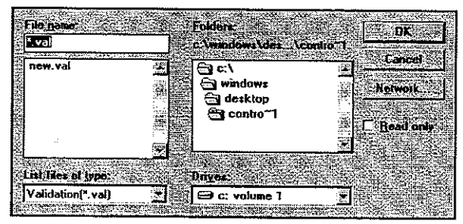
For details, refer to the TOC 5000 / TOC 5050 instruction manual, shipped with the instrument.

2. File Menu



The File menu

Open



The Open dialog box

Select the directory where the desired file is located.

Select the type of file desired from the drop-down list. The available file types are listed below (identified by file name extension):

.val

.val files are created by the DIN38402 program. They contain results of validation calculations performed on data.

.cal

.cal data files are created by the TOC Control program. When these files are opened by the DIN38402 program, validation calculations are performed automatically.

Select the desired file from the list of file names that appears.

Click OK.

Save

Choose Save to save the displayed data in a previously-named file. If the file has not been saved previously, the Save As dialog box opens.

Save As

Choose Save As to save the current data under a new file name or location. File type is determined automatically.

Print

Select Print to print the displayed data according to the Print Options dialog box specifications (in the Options menu).

Printer Setup

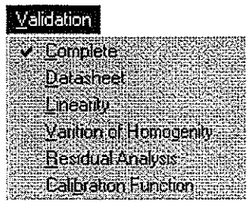
Select Printer Setup to display the Printer Setup dialog box. Options vary according to the system printer.

Close

Choose Close to exit the DIN38402 program.

3. Validation Menu

Several charts and tables are available for viewing in the Validation program. Select desired items from this menu.



The Validation menu

Complete

All the information represented by the five menu items below this command is displayed.

Datasheet

Measurement results of all samples and their replicates are displayed in the table. When *Validation Strictly Following DIN* is enabled (Options menu), only the first replicate of each sample is used for computing the linearity test and calibration function. In that case, the unused replicates are displayed over a light blue background color.

Complete Datasheet

Smpl (i)	Smpl.ID	Xi [ppm]	Yi,1	Yi,2	Yi,3	Yi,4	Yi,5	Yi,6
1	100 ppm	100.000	4336	4232	4205	4206	4222	4236
2	200 ppm	200.000	8430	8352	8380	8285	8401	8372
3	300ppm	300.000	11710	11747	11734	11716	11720	11897
4	400ppm	400.000	16609	16668	16689	16665	16778	16461
5	500 ppm	500.000	23596	23832	23850	23742	23795	23649
6	600ppm	600.000	23766	23603	23592	23713	23596	23473
7	700ppm	700.000	27887	27782	27835	27803	27641	27716
8	800ppm	800.000	31323	31230	31522	31267	31405	31375
9	900ppm	900.000	35122	35255	35292	35273	35425	35251
10	1000.00 ppm	1000.000	39760	39568	39880	39823	39619	39461

The Complete data sheet

Linearity

Calculated results of the linearity test are displayed in this spreadsheet.

On the left are the results of the linear regression calculation; on the right, the corresponding results of the quadratic calibration are displayed. The footer displays the comparison between these two calculation methods.

Result of the regression analysis

Linear Regression		Regression 2nd order	
Slope(b):	350.76970	Regression coeff. (a):	-279.51667
Intercept(a):	-321.93333	Regression coeff. (b):	348.64886
		Regression coeff. (c):	0.01928
Residual SD: (Sy1):	117.33117	Residual SD: (Sy2):	124.30957
Process SD: (Sx1):	0.33450	Process SD: (Sx2):	0.35439
Rel. Process CV: (Vx1):	0.61%	Rel. Process CV: (Vx2):	0.64%
Coeff. of Correlation(r^2):	0.999892	Sensitivity E():	350.76970
Lower limit of determination (Xp):	1.50182		
Difference betw. variations(DS^2):	1962.73485		
Test value(PW):	0.12701		
Value from F-table:	12.25000		

The difference beween variance DS^2 and Sy2^2 is not significant
The calibration function may be regarded as linear in the working range

The Linearity data sheet

Homogeneity of Variance

The replicates used, and the results of the homogeneity of variance test, are displayed in this spreadsheet.

Testing the homogenity of variance

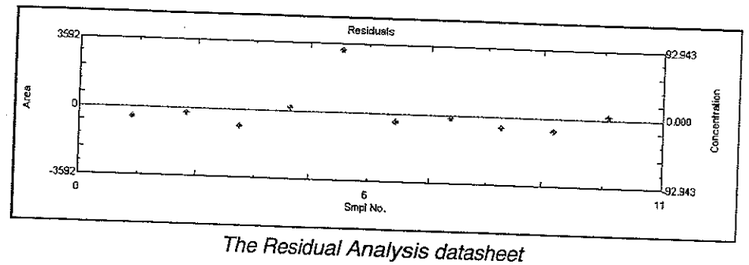
Smpl (i)	Smpl.ID	Xi [ppm]	Yi,1	Yi,2	Yi,3	Yi,4	Yi,5	Yi,6
1	100 ppm	100.000	4336	4232	4205	4206	4222	4236
2	1000.00 ppm	1000.000	39760	39568	39880	39823	39619	39461

Test value: 1.468E+001
F-Value: 5.350E+000
The difference between s1^2 and s2^2 is significant

The Homogeneity of Variance datasheet

Residual Analysis

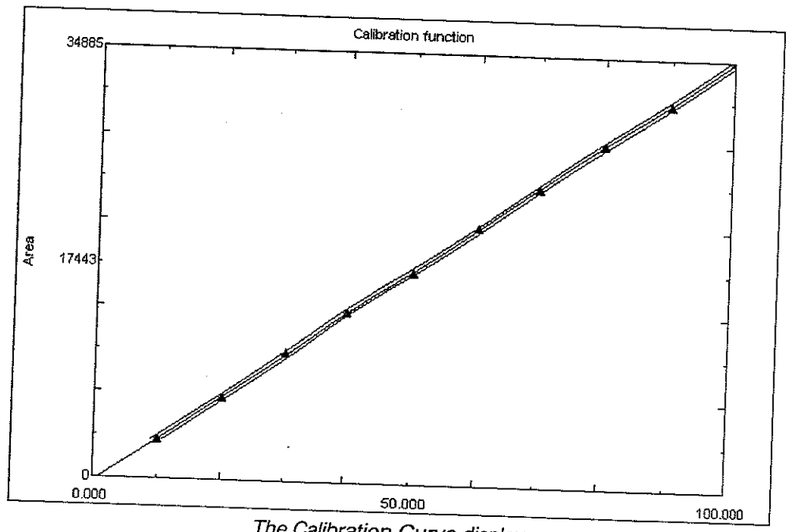
This graph displays the deviations of the calibration points from the calculated linear regression curve. It is used to determine whether there is an outlier in the data or if there is a significant problem over the working range of the calibration.



The Residual Analysis datasheet

Calibration Function

This graph displays the linear regression of the calibration curve, as well as the levels of confidence above and below the calibration curve. This graph can be customized; see p. 142 for more information.



The Calibration Curve display

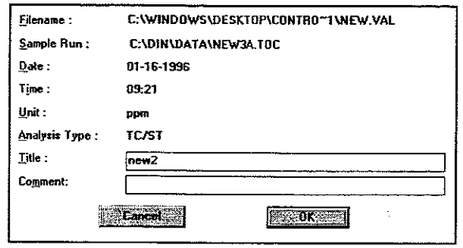
4. Options Menu

The Options menu items are used to set parameters for different features of the program.

The Options menu

General Parameters

The General Parameters dialog box appears, where general information about the current file is displayed. A title and comment can be entered, if desired.



The General Parameters dialog box

File Name

Displays the name of the file where the Validation results are stored.

Sample Run

Displays the name of the file where the data for the validation calculations are stored.

Date / Time

Displays the date and time of the validation.

Unit

Displays the analysis unit of measurement (ppb, ppm, etc.).

Analysis Type

Displays the analysis method (TC, IC, etc.) of the injections.

Title

Enter a title, if desired.

Comment

Enter comments, if desired.

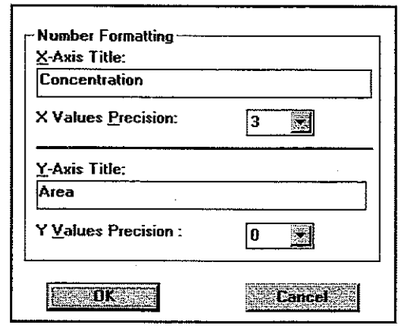
Press OK to save the entries, or press Cancel to exit the dialog box without saving.

Display Font

Opens the Font dialog box to customize the on-screen fonts.

Display Options

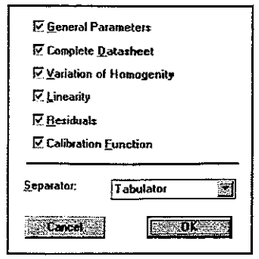
Define the precision of the X- and Y- values, and change the X- and Y-axis titles.



The Display Options dialog box

File Export Options

Enables the user to select items of information to be exported to an ASCII file.

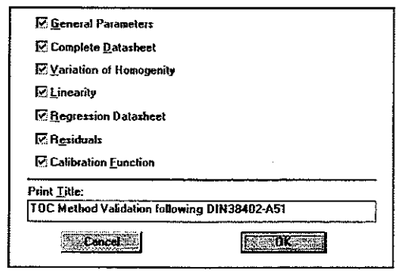


The File Export Options dialog box

In the Separator drop down list, select the character for separating items in the ASCII file: space, tab, comma, or semicolon.

Print Options

Designate the items to be printed.



The Print Options dialog box

Recalculation

Select this item to recalculate all results of the current Validation.

Validation strictly following DIN

When enabled, the validation calculation strictly follows DIN 38402 requirements, i.e., only the first injection of every standard is used for calibration (except for the first and last standards, which are averaged). Otherwise, the mean value of all area counts is used for calculating the calibration.

Save Settings on Exit

When enabled, the display parameters are saved.

5. Help Menu

Content

Opens the table of contents of the help file.

About

Displays version number and registration information.

6. Calculations

The following is a list of calculations performed by the Validation program.

Checking the Homogeneity of Variance

For both points under consideration, the variances are calculated using the following equations:

$$s_i^2 = \frac{\sum_{i=1}^{10}\left(y_{ij} - \overline{y_i}\right)^2}{n_i - 1}$$

where:

$$\overline{y_i} = \frac{\sum_{i=1}^{10} y_{ij}}{n_i}$$

Next, for the F-test, the following test value is calculated:

$$TV = \frac{s_{10}^2}{s_1^2} \text{ with } TV = \frac{s_{10}^2}{s_1^2} \text{ or}$$

$$TV = \frac{s_1^2}{s_{10}^2} \text{ with } s_1^2 > s_{10}^2$$

and compared with the value taken from the F-table for

$$f_1 = f_2 = n - 1 = 9 .$$

Results

If $TV < F_{f_1 f_2 : 99\%_1}$, then the difference between variances s_1^2 and s_{10}^2 is not significant.

If $TV \geq F_{f_1 f_2:99\%_1}$, then the difference between variances s_1^2 and s_{10}^2 is significant.

Linearity Test

For this test, use the first injection of all calibration points. The following equations are used:

The linear calibration equation

$$y = a + b \bullet x$$

where:

$$b = \frac{\sum\left[\left(x_i - \bar{x}\right)\left(y_i - \bar{y}\right)\right]}{\sum\left(x_i - \bar{x}\right)^2}$$

$$a = \bar{y} - b\bar{x}$$

Standard deviation:

$$s_y = \sqrt{\frac{\sum\left(y_i - \hat{y}_i\right)^2}{N-2}}$$

where:

$$\hat{y} = a + bx_i$$

Process standard deviation:

$$s_{x0} = \frac{s_y}{b}$$

Relative process standard deviation:

$$V_{x0} = \frac{s_{x0}100\%}{\bar{x}}$$

Coefficient of correlation:

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

The quadratic calibration equation

$$y = a + b \bullet x + c \bullet x^2$$

where:

$$a = \left(\sum y_i - b\sum x_i - c\sum x_i^2\right) / N$$

$$b = \frac{Q_{xy} - cQ_{x^3}}{Q_{xx}}$$

$$c = \frac{Q_{xy}Q_{x^3} - Q_{x^2y}Q_{xx}}{\left(Q_{x^3}\right)^2 - Q_{xx}Q_{x^4}}$$

$$Q_{xx} = \sum x_i^2 - \left(\left(\sum x_i\right)^2 / N\right)$$

$$Q_{xy} = \sum\left(x_i y_i\right) - \left(\left(\sum x_i\right)\left(\sum y_i\right) / N\right)$$

$$Q_{x^3} = \sum x_i^3 - \left(\left(\sum x_i\right)\left(\sum x_i^2\right) / N\right)$$

$$Q_{x^4} = \sum x_i^4 - \left(\left(\sum x_i^2\right)^2\right) / N$$

$$Q_{x^2y} = \sum\left(x_i^2 y_i\right) - \left(\left(\sum y_i\right)\left(\sum x_i^2\right) / N\right)$$

Standard deviation:

$$s_y = \sqrt{\frac{\sum\left(y_i - \hat{y}_i\right)^2}{N-3}}$$

Sensitivity:

$$E\left(\bar{x}\right) = b + 2c\bar{x}$$

Process standard deviation:

$$s_{x0} = \frac{s_y}{E\left(\bar{x}\right)}$$

Relative process standard deviation

$$V_{x0} = \frac{s_{x0}100\%}{\bar{x}}$$

F-test

For the F-test, the following test value is calculated:

$$DS^2 = (N-2)s_{y1}^2 - (N-3)s_{y2}^2$$

$$TV = \frac{DS^2}{s_{y2}^2}$$

This value is compared with the value obtained from the F-table for

$$f_1 = 1 \text{ and } f_1 = N-3.$$

Criterion:

If $TV < F_{f_1,f_2;99\%}$, then the difference between DS^2 and s_{y2}^2 is not significant. The calibration may be regarded as linear in the working range.

If $TV \geq F_{f_1,f_2;99\%}$, then the difference between DS^2 and s_{y2}^2 is significant. The calibration is not linear in the working range.

Chapter Nine Control Charts

1. Overview of Control Charts Software

	Smpl. ID	Control Value	Comment	Excluded	Date
1	control sample	50.27			05/30/97 14:56:38
2	control sample	50.19			05/30/97 15:02:48
3	control sample	50.17			05/30/97 15:08:58
4	control sample	50.29			05/30/97 15:15:06
5	control sample	50.18			05/30/97 15:21:20
6	control sample	50.03			05/30/97 15:27:30
7	control sample	50.17			05/30/97 15:33:44
8	control sample	50.08			05/30/97 15:39:50
9	control sample	50.23			05/30/97 15:45:56
10	control sample	50.33			05/30/97 15:49:00
11	control sample	50.56			05/30/97 13:50:50
12	control sample	50.80			05/30/97 14:06:16
13	control sample	50.42			05/30/97 14:33:02
14	control sample	50.40			05/30/97 14:43:00

The Control Chart window

The Control Charts software monitors precision and accuracy of a defined data set.

The software supports four Control Chart types:

- Mean Value Control Charts
- Recovery Control Charts
- Blind Value Control Charts
- Spanwidth Control Charts

Each Control Chart type may be used to create Control Charts for different working ranges and analysis methods.

Control Charts are valid for a specific period of time; after that, a new Control Chart must be created.

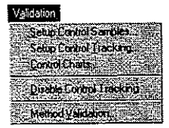
The limits for a new chart are copied from the previous Control Chart of the same working range and analysis method.

For each Control Chart, a file is created which contains general information about the type, data structure and other global data. This file also contains all data required

for sample identification as well as data required for display and calculation of the entire Control Chart.

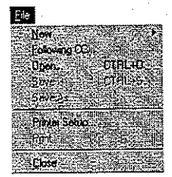
Note: *Once samples are added to a Control Chart, they cannot be deleted.*

Either select Control Charts from the Toccntr program folder or select Control Charts from the TOC Control Validation menu.



The Validation menu

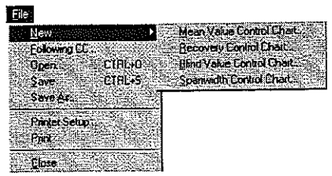
2. File Menu



The File menu

New

The New command opens a sub-menu from which one of the file types described below is selected.

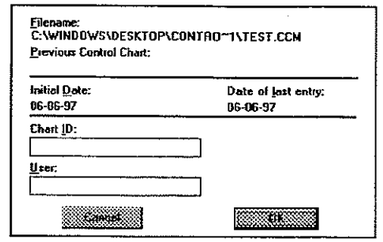


The New menu

Select any of these options to open the Open dialog box. To create a new Control Chart, enter a new name in the File Name text box, then select OK.

When OK is selected in the Mean Value, Blind Value, or Spanwidth Control Chart Open dialog boxes, the following Control Chart Options dialog box is displayed.

When OK is selected in the Recovery Control Chart Open dialog box, the Recovery Control Charts Option dialog box opens first. Fill in that dialog box as described on p. 172, then click OK to open the Control Charts Options dialog box shown below.



The Control Charts Options dialog box

Filename

The file name and path displays in the non-modifiable *filename* field.

Previous Control Chart

The name of the control chart previously used to track this data displays in the non-modifiable *Previous Control Chart* field.

Initial Date

The date the control chart was created displays in the non-modifiable *Initial Date* field.

Date of Last Entry

The date which the control chart was last modified displays in the non-modifiable *Date of Last Entry* field.

Chart ID

Enter a brief description of the control chart in the *Chart ID* text box.

User

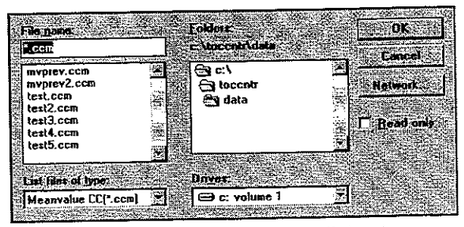
Enter the name of the user of this control chart in the *User* text box.

Mean Value Control Chart

The Mean Value Control Chart (.ccm extension) checks the accuracy of an analytical method on an instrument. In some special cases this chart can be used to detect system errors. For example, when using certified standard for the control samples over a long period of time, control value drifts indicate a change in system performance.

Recovery Control Chart

The Recovery Control Chart (.ccr extension) tests an analytical method for matrix influences and provides a restrictive control of accuracy by testing proportional system errors. We recommend using certified standards for Recovery Control Charts, which will test the accuracy of the system as well as the recovery rate.



The Recovery Control Chart dialog box

Recovery Control Chart Options Dialog Box

The Recovery Control Charts dialog box, shown below, opens when File/New/Recovery Control Chart is selected to open the Open dialog box, then OK is pressed.

Using the radio buttons, select either Certified Standard or a Real Sample as control samples.

Certified Standard

When using certified standards, choose between standards with fixed or variable carbon contents. For a fixed content standard, verify that the

standard will be available for the entire time the Control Chart is valid.

If using a fixed concentration, select *Fixed Theoretical Concentration* and enter the concentration of this standard in the text box below.

To use standards with variable amounts of carbon, do **not** select *Fixed Theoretical Concentration.* Enter the standard concentration in the Setup Control Tracking Setup dialog box, accessed by selecting Validation/Setup Control Tracking in the TOC Control application.

Real Sample

When using real samples as control samples, process them in two different ways.

a) For samples spiked with a fixed concentration of carbon, select *Fixed Concentration of the Spike Component* and enter the spiked content in the text box.

b) For control samples with different spiked amounts, enter the spiked content under Setup Control Tracking on the Validation menu in the TOC Control program.

Blind Value Control Chart

The Blind Value Control Chart (.ccb extension) is a special form of the Mean Value Control Chart used for quality assurance of the instrument and variable elements.

This Control Chart can be used with analytical methods where the blind value is different from zero and normally distributed, i.e., -BOD, COD-determination and special photometric methods. Here, the control sample is the blind sample for the respective analytic method.

Unlike the mean value Control Chart, the control value is the information value (absorbance, peak height, peak area) received from the analysis.

Spanwidth Control Chart

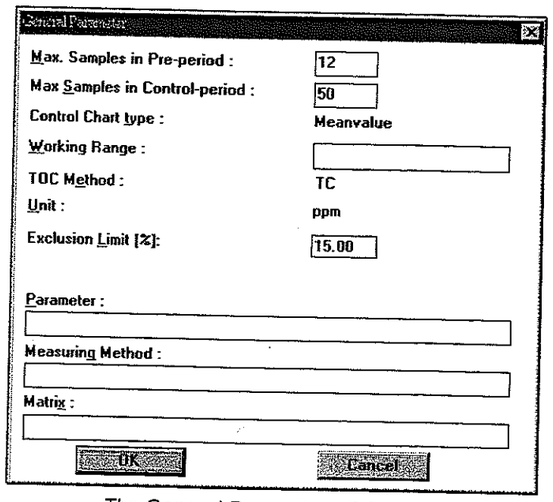
The Spanwidth Control Chart (.ccs extension) tests the precision of an analytical method and monitors the

accuracy under actual conditions (Drift Control). For this type of Control Chart, there are several existing models for calculating the control and warning limits.

We recommend calculating only the upper control limit; defining the upper exclusion limit is optional. The lower control limit is zero during measurement of multiple determination with up to six samples.

General Parameters dialog box

This dialog box opens when OK is selected on the Control Chart Options dialog box. Enter additional information and parameters for the new Control Chart in this dialog box.



The General Parameters dialog box

Max. Number of Samples in Pre-Period

In the *Max Number of Samples in Pre-Period* text box enter the number of samples to be used for the preparation period. This value is important because the samples are used to calculate the limits (upper and lower warning limit, upper and lower control limit) after all samples of the preparation period are added to the Control Chart. The default value is 12.

Max. Number of Samples in Control-Period

Enter the estimated maximum number of samples in the Control Chart; the default is 50.

Working Range

Enter the working range for the current Control Chart. Match the range with the concentration of the samples used for control tracking and the concentration range of the calibration curve selected. To check the linearity of the calibration, see the Method Validation chapter.

TOC Method

Enter the TOC analysis method (TC, IC, etc.) for this Control Chart. During data addition, the samples are checked when the analysis type matches; otherwise the sample will not be added to the Control Chart.

☛ *Note:* *When an Out of Control Event or an error occurs during data addition, a message is displayed in the Message Log.*

Unit

Define the unit of the control samples, ensuring that only samples with the same unit are added to the Control Chart.

Exclusion Limit (%)

This value defines the upper and lower exclusion limit of the Control Chart. This limit is only defined once and will not be changed with the recalculation of the other limits.

No. of Injections (Spanwidth Control Chart only)

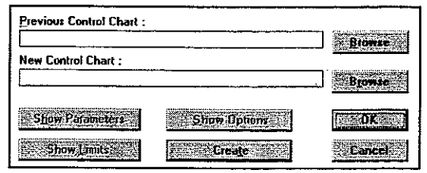
For the Spanwidth Control Chart it is recommended that only control samples with the same number of replicates be used as that value defines this number. The control sample is added to the Control Chart, even when the number of replicates differs.

Parameter / Measuring Method / Matrix

These are information fields for your reference only. Enter relevant information.

Following CC

Select this menu item to create the Following Control Chart.



The Following Control Chart dialog box

Previous Control Chart

When a Control Chart is already loaded, the file name is added automatically to this dialog box. The BROWSE button can be used to locate a different file.

New Control Chart

Enter the file name of the Following Control Chart in the Edit field. The BROWSE button is used to select a directory where the new Control Chart will be saved.

Create

Press CREATE to read the data of the previous Control Chart and calculate the new limits from the samples of the control period stored in the previous Control Chart.

Show Parameters / Show Options / Show Limits

After creating the new Control Chart, the SHOW PARAMETERS, SHOW OPTIONS, and SHOW LIMITS buttons are enabled. Use them to view information on the created Control Chart.

Press OK to save the new, empty Control Chart under the assigned file name in preparation for use.

Open

Select Open to access the Open dialog box. Use this dialog box to open an existing Control Chart.

Save

Choose Save to save the current data. If the data has not been saved previously, the Save As dialog box opens.

Save As

Choose Save As to open the Save As dialog box. Use this dialog box to save the current data to a new file name or location.

Printer Setup

Select Printer Setup to open the Printer Setup dialog box. Options will vary depending on the system printer.

Print

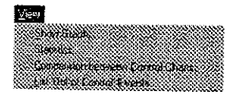
Select Print to print the current control chart.

Close

Exits the Control Charts program.

3. View Menu

These menu items are used to display the features and parameters of the Control Charts.



The View menu

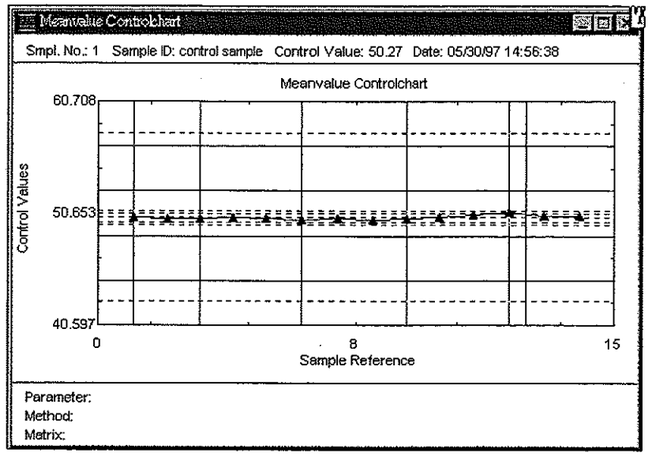
Show Graph

Select Show Graph to open a dialog box where the table data is displayed as a graph. This graph can be customized; see p. 142 for more information.

The header contains information about the selected control sample. The footer contains information about the current Control Chart.

To change the current sample information, move the mouse pointer to the solid vertical gray line. When the mouse pointer changes to a double arrow, hold the left mouse button down and move the line to the desired point

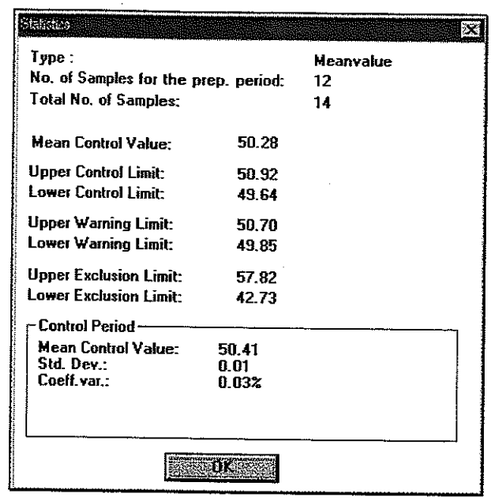
on the graph. When the left mouse button is released, new information is displayed in the header.



The Graph display

Statistics

Select Statistics to display information relevant to the loaded Control Chart. Select OK to close the dialog box.



The Statistics dialog box

Type

Type displays the type of Control Chart currently open (Mean Value, Recovery, Blind Value, or Spanwidth).

No. of Samples for the Prep. Period

No. of Samples for the Prep. Period displays the number of samples used for the preparation period. This number was set when the chart was first created. For Following Control Charts, this number will always be zero.

Total No. of Samples

Total Number of Samples displays the total number of sample entries. The total includes samples used during the preparation and control periods.

Mean Control Value

The Mean Control Value is the average control value calculated from all samples of the preparation period. This value is calculated from the samples in the preparation period or, for a new

chart, carried over from the control period of the previous Control Chart.

Upper/Lower Control Limits and Upper/Lower Warning Limits

The Upper and Lower Control Limits and Warning Limits are the calculated limits of the current Control Chart. The values are calculated from the samples in the preparation period or, for a new chart, taken from the control period of the previous Control Chart.

Upper/Lower Exclusion Limit

The displayed values are set during the creation of the Control Chart. They must not be changed during the lifetime of a Control Chart system.

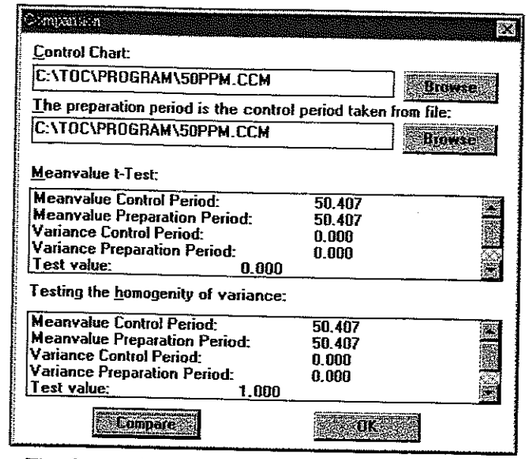
Mean Control Value / Standard Deviation / Coefficient of Variation

The Mean Control Value, Standard Deviation, and Coefficient of Variation are calculated from all samples of the control period (except excluded samples), and are updated with every new data addition. The difference between this mean value and the mean value stored in the Control Chart shows long term changes of the control samples during the lifetime of the Control Chart.

Comparison Between Control Charts

After finishing a complete Control Chart, the current results of the control period must be compared with the results from the preparation period. For Control Charts without a preparation period, the current values must be compared with the results of the preparation period taken from the previous Control Chart.

Enter the file names of the Control Charts or use the BROWSE button to locate the files. When there is a Control Chart open, the file names are automatically entered from that chart.



The Comparison Between Control Charts dialog box

Select COMPARE to begin the statistical comparison. The following two methods are used to check long-term deviations in the Control Chart.

Mean Value T-Test

The mean values and variances are calculated from the control and preparation period. Then a test value is calculated and compared with the corresponding value from the T-table. This comparison shows whether the long term deviation of the control samples is significant.

The equations are:

$$TV = \left| \frac{\bar{x}_1 - \bar{x}_2}{s_d} \right| \sqrt{\frac{N_1 N_2}{N_1 + N_2}}$$

with:

$$s_d = \sqrt{\frac{(N_1 - 1)s_1^2 + (N_2 - 1)s_2^2}{N_1 + N_2 - 2}}$$

for : $N_1 = N_2 = N \; f_1 = f_2; f = 2N - 2$

Criterion:

TV <= t(f, P = 95%): deviation is not significant

TV > t(f, P = 95%): deviation is significant

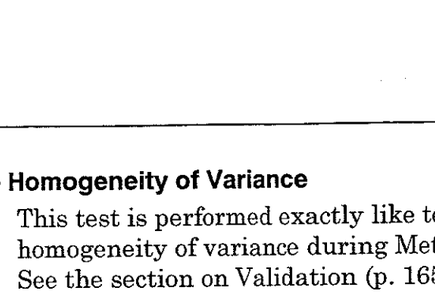
Testing the Homogeneity of Variance

This test is performed exactly like testing the homogeneity of variance during Method Validation. See the section on Validation (p. 165) for the equations.

List Out of Control Events

After adding new control samples to the control period of a Control Chart, the control value is compared with the defined limits. Several events can occur which show that there are long-term changes in the analytical performance of the instrument. When an Out-of-Control event occurs, the instrument has to be checked for system errors. After fixing the problem, re-calibrate the instrument and perform Method Validation again.

During measurement, a message is recorded each time an Out-Of-Control event occurs. In this message box, all Out-Of-Control events are listed which occurred during the lifetime of the current control period.



The Out of Control Events dialog box

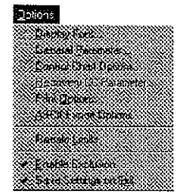
The following Out-of-Control Events can occur during the lifetime of a Control Chart:

- One control value exceeds the control or exclusion limits
- Two out of three control values exceed the warning limits
- Seven consecutive control values are above or below the mean control value
- Seven consecutive control values show an increasing or decreasing tendency

☛ ***Note:*** *When an Out of Control Event or an error occurs during data addition, a message is displayed in the Message Log.*

4. Options Menu

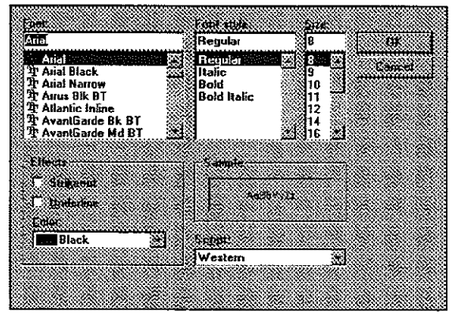
The Options menu items are used to set parameters for different features of the Control Chart software.



The Options menu

Display Font

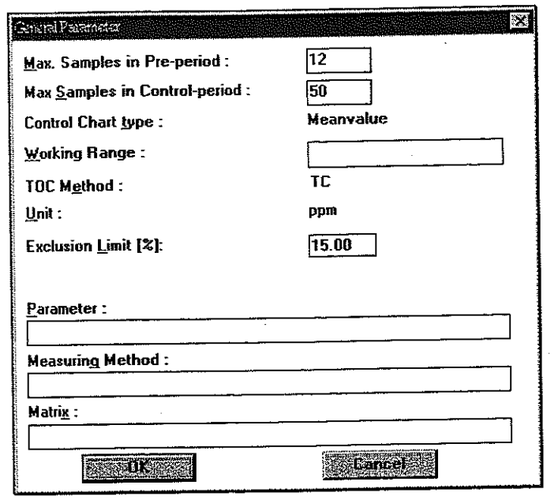
Select Display Font to open the Font dialog box. Use this dialog box to select a new screen font. See *Printer Font*, p. 62, for more information on the Font dialog box.



The Display Font

General Parameters

The General Parameters dialog box displays the parameters for the current Control Chart. Some items cannot be changed after the Control Chart is created.



The General Parameters dialog box

Max. Number of Samples in the Preparation Period

The *Max Number of Samples in the Preparation Period* displays the number of samples used for the preparation period. These samples are used to calculate the limits (upper and lower warning limit, upper and lower control limit) after all samples of the preparation period are added to the Control Chart. This field is modifiable. The default is 12.

Max. Number of Samples in the Control Period

The *Max Number of Samples in the Control Period* field displays the maximum number of samples in the Control Chart. This field is modifiable. The default is 50.

Control Chart Type

The *Control Chart Type* field displays the current type of Control Chart.

Working Range

In the *Working Range* text box enter the working range for the current Control Chart, e.g., 10 – 100 ppm. The range should correlate with the concentration of the samples used for control

tracking and also with the concentration range of the used calibration curve. To check the linearity of the calibration, see the section on Validation (p. 155).

TOC Method

The TOC Method displays the analysis type (TC, IC, etc.) for this Control Chart. During data addition, only samples of the specified analysis type are added to the Control Chart.

☛ ***Note:*** *When the Message Log displays "Checking Controls" and the analysis type does not match, an error message is added to the Message Log.*

Unit

Displays the unit of measurement. Ensure that only samples of the same units are added to the Control Chart.

Exclusion Limit (%)

This value defines the upper and lower exclusion limit of the Control Chart. This limit is only defined once and cannot be changed with the recalculation of the other limits.

No of injections (Spanwidth Control Chart only)

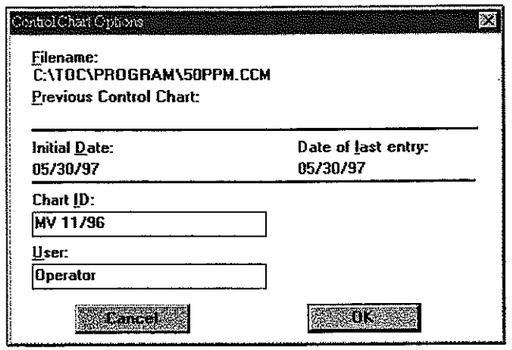
For the Spanwidth Control Chart, it is recommended that only control samples with the same number of replicates be used, as that value defines this number. However, the control sample is added to the Control Chart even when the number of replicates differs.

Parameter / Measuring Method / Matrix

Enter common parameters for sample conditions, sample origin, and sample matrix.

Control Chart Options

Common parameters for the current Control Chart are displayed in this dialog box. Fields that cannot be changed after a Control Chart is created are dimmed.



The Control Charts Options dialog box

File name

The file name and location of the current Control Chart is displayed.

Previous Control Chart

The file name of the previous Control Chart is displayed.

Initial Date / Date of Last Entry

The Initial Date and Date of Last Entry display the current system date when a new Control Chart is being set up. When a saved Control Chart is open, the relevant dates are shown.

Chart ID

Enter an identification for the Control Chart.

User

Enter the name of the user.

Recovery CC Parameters

The items in this dialog box display the same parameters as those when the new Control Chart was created. Fields that cannot be changed after a Control Chart is created are dimmed.

Previous Control Chart

When a Control Chart is open, the file name is displayed in this dialog box. The BROWSE button can be used to locate a different Control Chart.

New Control Chart

Enter the file name of the Following Control Chart. The BROWSE button can be used to locate the directory where the new Control Chart will be saved.

Create

Click CREATE to read the data of the previous Control Chart and calculate the new limits from the samples of the control period stored in the previous Control Chart.

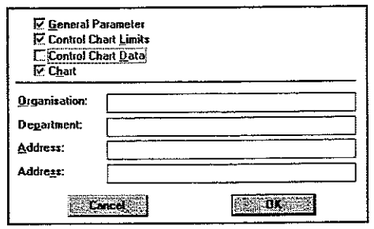
Show Parameters / Show Options / Show Limits

After the new Control Chart is created, these buttons are enabled. They may be used to look at the parameters of the created Control Chart.

Press OK to store the new, empty Control Chart under the defined file name.

Print Options

Select Print Options to open the Print Options dialog box. Select information to be included in a printout. An organization, department, and address can be entered. These items will be included on the printout.



The Print Options dialog box

General Parameters

Select General Parameters to print the information displayed in the General Parameters dialog box. This dialog box can be viewed by selecting Options/General Parameters.

ASCII Export Options

Select a separator from the drop-down list. During ASCII export, items of information are separated by the selected separator. When importing the ASCII file into another program, the separator used must be specified.

The ASCII Export Options dialog box

ReCalc Limits

After excluding samples from calculation, select this menu item to recalculate the limits of the current Control Chart.

Select OK to recalculate the limits of the current Control Chart

Enable Exclusion

When a check mark is displayed next to this menu item, exclusion is enabled. When control samples are excluded, they are not used for calculations or the determination of Out-of-Control events.

☛ *Note:* *Add a comment regarding the reasons for the exclusion in the Control Chart.*

Save Settings on Exit

When a check mark appears next to this menu item, all program settings are saved when the program is closed.

5. Help Menu

Content

Select Content to open the table of contents for the Help file.

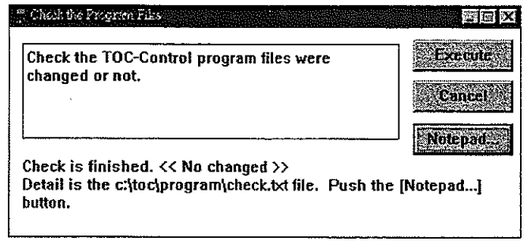
About

Select About to view software registration and version information.

Chapter Ten Program Check

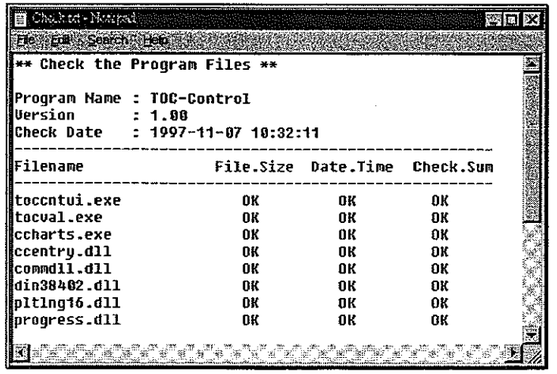
To ensure data integrity, this auxiliary program checks whether any program files have been modified,.

Select the Program Check item from the Toccntr folder. This opens the Check the Program Files window.



Ensure data integrity with the Program Check

Select EXECUTE to perform the check. When the check is finished, the results will be displayed in the window. For details on the check, select NOTEPAD.



View the details of the Program Check in Notepad

Chapter Eleven Verification

The TOC Control software supplies verification data so the operator can verify that the software is working correctly and that data is being processed correctly.

The verification files are listed below:

File Name	File Size (KB)	Date Stamp	Time Stamp
VAL_TEST. PKT	29,498	11-07-97	16:57
VAL_TEST.MET	310	11-07-97	15:13
VAL_TEST.CAL	353	11-07-97	16:51
VAL_TEST.TOC	1,132	11-07-97	16:58

These files are available on Disk 3 of the program installation disks. Create a directory for the files (e.g. \toccntr\test), then copy the files to the directory.

Verification Procedure:

1. Start the TOC Control program.
2. From the File menu, choose Open, and open the "val_test.toc" file.
3. Select Attach Data File from the File menu, and choose the "val_test.pkt" file. This enables the program to locate the Peak Profile data of the analyses.