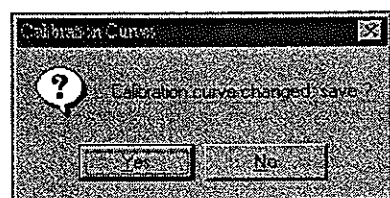


Attach the peak profile data

4. Select View/Calibration Curve to open the Calibration Curve dialog box. This automatically opens val_test.cal.
5. Choose "Point to point without zero shift" as the Calculation Method. Select OK.
6. When prompted, save the calibration curve.



Save the calibration curve

7. From the Edit menu, select Recalculate. This recalculates the data based on the new calibration curve calculation parameter. In this way, the calculation function of the software can be evaluated.
8. From the File menu, choose Page Setup, and select the Default button for every tab.
9. From the File menu, select print. Confirm the printer setup and select OK.

When the report has printed, compare it to the printout given here. The reports should be identical, except for the General Information. Identical reports verify that the software is operating properly and has not been corrupted. If the reports are not the same, ensure that the procedure was followed correctly, and that the Page Setup settings are correct. Contact your Shimadzu representative for further assistance.

Verification report for comparison

General Information

Organization: Shimadzu
 User: Tanaka
 Filename: D:\COMMON\TOCCNTRF\SOFT_VALIVAL_TEST.TOC

Instrument Conditions

Instrument Attachments: TOC 5000A + ASI 5000
 TC-Cat Type: Normal sense TC catalyst
 Bubble Removal: OFF
 Syringe Size: 250 µl
 Injection Speed: STD
 Syringe Wash: ON

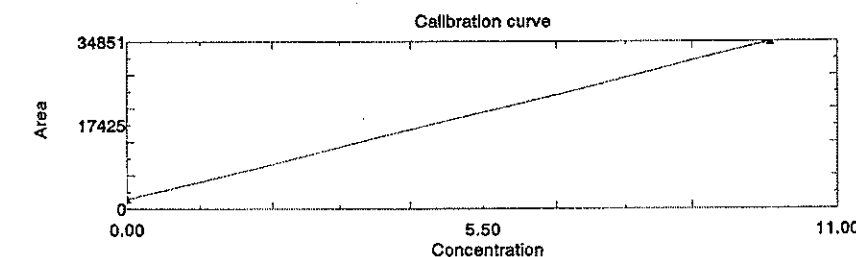
Calibration Curves

Filename: val_test.cal
 Title: 0-10ppm

Analysis	No. of Washes	Unit	Range	Sparge time	Inj. Vol	Max SD	Max CV
TC	4	ppm	1	0	50	200	2.00%

Sample Name	Sample ID	Conc.	No. of Inj.	Max No. of Inj.	Mean Area	SD	CV
KHP	0	0.000	3	5	2050	138	6.73%
KHP	10	10.00	3	5	34851	393	1.13%

Slope: 3280.1
 Intercept: 2049.7
 R²: 0.00000



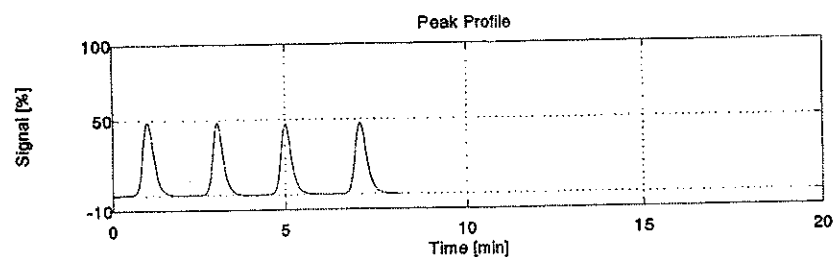
Samples

Sample Name: sample
 Sample ID: 1
 Method: val_test.met
 Cal Curve: 1: val_test.cal

Type	Analysis	Spurge Time	Date/Time
Unknown	TC	0	1997/11/07 18:57:54

Mean Area	Conc	Result	No. Of Washes	SD	CV
18641	5.058 ppm		4	0.04766	0.943%

No.	Range	Inj. Vol.	Area	Conc	Excl.	Date/Time	Cal Curve
1	1	53	18478	5.009		1997/11/07 18:51:00	val_test.cal
2	1	53	17974	4.855	E	1997/11/07 18:53:16	val_test.cal
3	1	53	18791	5.104		1997/11/07 18:55:39	val_test.cal
4	1	53	18654	5.062		1997/11/07 18:57:54	val_test.cal



Statistics / Summary

Sample Name	Analysis	Conc.	Result
sample	TC	5.058 ppm	

Appendix A Error Messages

Error Message Severity Levels

1. For information only
2. When the problem is fixed, the action can be completed; otherwise, a save alternative will occur
3. The requested action cannot be completed, but the user can continue to use the software
4. System Failure: exit the application and restart
5. Instrument Error: instrument maintenance is required
6. Fatal Instrument Error: contact your Shimadzu representative

Error Messages

Message	Severity	Reason	Action
Acid addition volume is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
Argument Error	4	Communication	Check the parameters for the current action
ASI not initialized	5	Instrument	Execute [Options][Maintenance][Initialize ASI]
Calibration curve not found	2	File System	Check the path
Cannot create directory!	2	PC	Check the path Check the hard drive for free space
Cannot delete file	2	File System	Check the path and file name
Cannot open COM	3	Communication	Check the communication parameters Check the RS232 cable for correct installation Check the DIP switches on the instrument RS232 board
Cannot open method file	4	Methods	Check the path and file name
COM already opened	3	Communication	Use another COM port

Error Messages

Message	Severity	Reason	Action
Command not sent	5	Communication	Retry this function later
Control data list is not valid, function cannot be executed.	1	File system	Does the file CCHART.DAT exist in the applications program directory?
CV is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
Dilution factor is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
Error in receiving data	3	Communication	Check the communication parameter Check the RS232 cable for correct installation Check the DIP switches on the instrument RS232 board
Error in sample table line:	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
ERROR!! Arm horizontal home position	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Arm horizontal pulse count	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Arm lower limit	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Arm vertical home position	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Arm vertical pause position	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Arm vertical pulse count	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Cannot find syringe zero point	5	Instrument error	Check the instrument Process "Zero Point Detection"
ERROR!! Illegal power off	5	Instrument error	Check the instrument

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Message	Severity	Reason	Action
ERROR!! Position detection for syringe pump four-port valve abnormal	6	Instrument error	Shut down the instrument
ERROR!! RAM / ROM error	6	Instrument error	Shut down the instrument
ERROR!! SSM IC furnace not ready	3	Instrument error	Is the SSM connected? Check the settings of the temperature controller
ERROR!! SSM sample boat position	5	Instrument error	Try to move the sample rod Shut down the instrument Perform SSM maintenance
ERROR!! SSM sample boat position detector	5	Instrument error	Try to move the sample rod Check the SSM sample boat slider for correct position
ERROR!! SSM TC furnace not ready	3	Instrument error	Is the SSM connected? Check the settings of the temperature controller
ERROR!! Syringe pump error	6	Instrument error	Shut down the instrument
ERROR!! Syringe pump home position not detected	3	Communication	Perform zero point detection or change the syringe
ERROR!! TC furnace overheat	6	Instrument error	Shut down the instrument
ERROR!! Thermocouple disconnected	6	Instrument error	Shut down the instrument
ERROR!! Thermocouple shorted	6	Instrument error	Shut down the instrument
ERROR!! Turntable detector	5	Instrument error	Shut down the instrument Perform ASI maintenance
ERROR!! Turntable home position	5	Instrument error	Shut down the instrument Perform ASI maintenance

Error Messages

Message	Severity	Reason	Action
ERROR!! Turntable pulse count	5	Instrument error	Shut down the instrument Perform ASI maintenance
Error: COM not ready	4	Communication	Check the communication settings of the software Check the DIP switches on the instrument communication board Check the RS232 cable for correct connection to the PC
Error: Communication port not opened	4	Communication	Check the communication settings of the software Check the DIP switches on the instrument communication board Check the RS232 cable for correct connection to the PC
Error: Get EOT	4	Communication	Check the communication settings of the software Check the DIP switches on the instrument communication board Check the RS232 cable for correct connection to the PC
Error: Parity error	4	Communication	Check the DIP switches on the instrument communication board Check the RS232 cable for correct connection to the PC Check the communication settings of the software
Error: Time Out	4	Communication	Check the DIP switches on the instrument communication board Check the RS232 cable for correct connection to the PC Check the communication settings of the software
Error: Unknown Communication error	4	Communication	Check the communication settings of the software Check the DIP switches on the instrument communication board Check the RS232 cable for correct connection to the PC

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Message	Severity	Reason	Action
File cannot be opened! The application creates a new empty control data list.	4	File system	Does the file CONTROL.DAT exist in the applications program directory? Check the file path.
File not found! The application creates a new empty control data list.	1	File system	Does the file CCHART.DAT exist in the applications program directory? Check the file path.
Filenames must be different!	1	File system	Select another file name
Hardware error	5	Communication	Check the communication parameter Check the RS232 cable for correct installation Check the DIP switches on the instrument RS232 board
Illegal Command	2	Communication	The instrument is not in the correct status to accept this action. Check the function parameter Check the instrument status Execute the "ESCAPE" command Retry this function later
Illegal Mode	1	Communication	The instrument is not in the correct status to accept this action. Check the instrument status Execute the "ESCAPE" command Retry this function later
Illegal Parameter	2	Communication	The instrument is not in the correct status to accept this action. Check the function parameter Check the instrument status Execute the "ESCAPE" command Retry this function later
Injection volume exceeds the upper limit	2	Wrong Parameter	Check the Method conditions according to the current instrument settings Check the syringe size
Injection volume is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings Check the syringe size
Injection volume is too small	2	Wrong Parameter	Check the Method conditions according to the current instrument settings

Error Messages

Message	Severity	Reason	Action
			Check the syringe size
Instrument Busy	3	Communication	Retry this function later
Instrument error notification	5	Instrument error	Check additional error messages
Instrument is not connected. Cannot execute this function!	2	Communication	Check the RS232 settings of the software Connect the instrument
Instrument not ready	2	Instrument	Check the instrument conditions Retry this function later
Max. No. of injection is not valid	2	Wrong Parameter	Check the Method parameters according to the current instrument settings
Memory allocation error! Application will be terminated!	4	Internal Windows error	Close other applications Restart the computer Increase the RAM size
No of washes too high	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
No. of injection not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
No. of washes is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
Not Connected	2	Communication	Connect the instrument
Restart time is not OK	1	Instrument	Check the instrument date and time The restart time has to be greater than one hour from the current instrument time
RS232 busy error	5	Communication	Retry this function later
SD is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
Spurge time is not valid	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
Spurge time is too long	2	Wrong Parameter	Check the Method conditions according to the current instrument settings
The catalyst defined in the calibration curve is not suitable for the current instrument conditions!	1	Calibration curve	Does the calibration curve match the current instrument conditions? Instrument conditions must be changed before creating a curve
The type of the calibration curve doesn't match with the method!		Method	Does the calibration curve match the current instrument conditions? Ensure that the analysis type is the same in both the calibration curve and method

TOC Control Software Manual

Message	Severity	Reason	Action
This file is not in the correct format or corrupted! It can't be opened	2	File system	Is the file suitable for the TOC-Control application. (*.toc, *.cal, *.met, *.pkt)?
Timeout error	3	Communication	Retry this function later
Unknown error	3	Communication	Restart the application
Vial is not defined	2	Wrong Parameter	Check the Method conditions according to the current instrument settings Select appropriate tray
Zero point of the sample syringe is not detected, cannot execute function!	2	Instrument error	Check the instrument conditions (Syringe Size) Execute the [Options]-[Maintenance]-[Zero Point Detection]

Appendix B

Automatic Calibration Curve Selection

When more than one Calibration Curve has been selected in the Method dialog box, the optimum Calibration Curve is selected automatically according to the following procedure.

1. Measurement is initially conducted using the first calibration curve in the list (CAL#1).

When the sample is lower in concentration than CAL#1:

2. If the sample concentration (SAMP) is lower than the highest standard concentration of CAL#1, and higher than the other calibration curve standards (CALOther), the first calibration curve is used.

$CAL\#1 > SAMP > CALOther \rightarrow CAL\#1$ used

3. If two calibration curves have been designated, the sample concentration is lower than the highest standard concentrations for both CAL#1 and CAL#2, and the concentration of CAL#2 is lower than that of CAL#1, then the second calibration curve is used.

$CAL\#1 > CAL\#2 > SAMP \rightarrow CAL\#2$ used

4. If three calibration curves have been designated, the selection is essentially the same; the closest concentration of standard Calibration Curve that is still higher than the sample concentration will be used.

$CAL\#1 > CAL\#2 > SAMP > CAL\#3 \rightarrow CAL\#2$ used

$CAL\#1 > CAL\#2 > CAL\#3 > SAMP \rightarrow CAL\#3$ used

When the sample concentration is higher than Cal#1, and Cal#2 or Cal #3 (CALOther) are higher in concentration than Cal#1:

5. If the sample concentration is marked with an "H" (indicating that the peak height exceeded the full scale of CAL#1), the other calibration curve is used.

$CALOther > SAMP\ H > CAL\#1 \rightarrow CALOther$ used

6. If SAMP is not marked with an H, the program performs a ratio calculation. The ratio of the measured sample concentration and CAL#1 is compared to the ratio of the sample concentration and CALother. In most cases, CAL#1 is selected, because the NDIR is linear for any range setting, and a concentration within full scale range will not be far greater than CAL #1. Therefore, using CAL#1 will be at least as accurate as if another calibration curve is used.

CALother > SAMP > CAL#1 → CAL#1 usually used

7. If SAMP is higher than that of both CAL#1 and CALother, the other calibration curve is used.

SAMP > CALother > CAL#1 → CALother used

8. If SAMP is higher than CAL#1, and the other two concentration curves have higher concentrations than CAL#1, measurement will be calculated based on the lower concentration calibration curve that is closest to the sample concentration. Then, the evaluation described in Steps 5, 6 and 7 is conducted.

CAL#2 > SAMP > CAL#3 > CAL#1 → CAL#3 usually used

SAMP > CAL#2 > CAL#3 > CAL#1 → CAL#2 usually used

The following points about automatic optimum calibration curve selection should also be considered:

- ◆ The procedures described above are valid for both single and multi-point calibration curves.
- ◆ For multiple injections, when the first peak is within the full scale, while succeeding peaks exceed the scale, operation will continue based on the first injection. The amount that the successive peaks exceed the range is considered too small to affect the measurement.
- ◆ The TC catalyst type (Normal or High sensitivity) and the syringe size must be the same for the calibration curves selected.
- ◆ Different units can be used for the calibration curve concentrations, provided the units are of the same grouping (i.e., a combination of ppm and ppb, or mg/L and µg/L).

- ◆ When analyzing samples with an expected wide range of concentrations, set the concentration ratio between two calibration curves to between 5 – 10. Since the measurement always begins with CAL#1, this should be the curve most likely to be used. The selection of CAL#2 and CAL#3 depends upon their concentrations and the expected sample concentrations.
- ◆ Automatic selection of the optimum calibration curve cannot be used in measurement based on the carbon equivalent.

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