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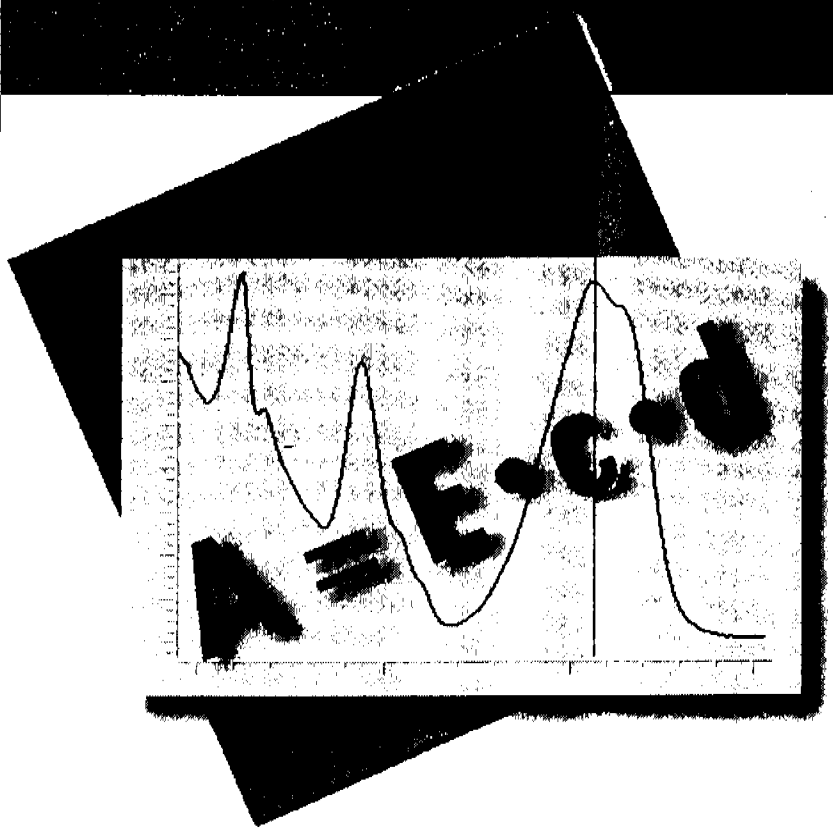

Standard Operating Procedure

Operational Qualification/ Performance Verification for HP 8453 UV-visible Spectroscopy Systems

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Warning

For details of safety, see "Safety Information" in the *HP 8453 Reference Manual.*

Hewlett-Packard GmbH
Chemical Analysis Group Europe
Hewlett-Packard-Strasse 8
D-76337 Waldbronn
Germany

Standard Operating Procedure

Operational Qualification / Performance Verification for HP 8453 UV-visible Spectroscopy Systems

General Information

This handbook is intended for use by the technical reader requiring operating procedures for operational qualification or performance verification (OQ/PV) of the HP 8453 UV-visible spectrophotometer, HP ChemStation software for UV-visible spectroscopy, and optional accessories.

This document describes the purpose of these procedures, and provides guidelines in the preparation, setup, and evaluation of OQ/PV tests and procedures. The electronic files which accompany this manual can be used to develop customized standard operating procedures, which may be specific to your laboratory environment and requirements.

The handbook lists the specifications of the instrument, as well as procedures for verifying instrument performance based on these specifications. These detailed procedures, intended for less experienced users, describe the handling requirements for various chemicals, cuvettes, flow cells, and consumables. Strict adherence to these procedures is required, in order to insure success in verifying instrument performance. This document includes part numbers and ordering information for supplies from Hewlett-Packard as well as other companies.

For information about installation of the system hardware, including the spectrophotometer, computer, and additional accessories, see the *Installing Your UV-visible Spectroscopy System* handbook. For detailed repair and maintenance information as well as instrument and accessory part numbers, see the *online help* in your HP ChemStation software or the *HP 8453 Reference Manual.*

Fill-in Form 1

Protocol Approval

Customer protocol approval

I agree that the OQ/PV procedures in this document are applicable to the equipment defined in the table of contents.

Name	Signature	Date

Hewlett-Packard protocol approval

I agree that the OQ/PV procedures, assembled by Hewlett-Packard Analytical Services are appropriate for the equipment defined in the table of contents.

Name	Signature	Date

Installing the Electronic Version

To install your own copy of the manual on your hard disk perform the following steps:

1 Insert the CD-ROM that comes with this document in your CD-ROM drive.

2 Use the Windows File Manager or Windows 95 Explorer to install Adobe® Acrobat Reader and optionally, a copy of the manual, by selecting the \oqpv\setup.exe program.

3 When the system restarts, new icons appear in your HP ChemStation program group. Selecting the HP 8453 OQ/PV Handbook opens the d:\oqpv\oqpvbook.pdf file in Adobe Acrobat Reader. From this program you can print this manual. In addition, Adobe Acrobat Reader provides full Windows-compliant copy-and-paste capability.

Exceptional Conditions

Any exceptional conditions encountered during the administration of this protocol will be documented at the time of occurrence and reviewed by Hewlett-Packard personnel. Exceptional conditions will be investigated and the appropriate course of action determined, for example, repairs will be completed if instrument failure is covered contractually.

Fill-in Form 2

Document Revision History

Revision Number	Changes	Date	First Approval	Second Approval	Final Approval

Fill-in Form 3

System Test Laboratory Location

The equipment for which the OQ/PV procedures in this document are performed can be found in the location specified below as of the date this protocol was performed.

Company Name

Location

Department	Building #	Room

Date this OQ/PV was last performed

Verified by (signature)	Date

Use Fill-in Form 4 after you have completed all tests for the operational qualification of the HP 8453 spectroscopy system.

Fill-in Form 4

Certification of Completion of Operational Qualification

The protocol documented in the following pages, performed on the given date, determined the operational suitability for the equipment as named.

Hewlett-Packard HP 8453 spectroscopy system			
Instrument Name			
Initial	Requalification	Yes	No
Certification?		Sticker attached to instrument?	
Name		Signature	Date
Engineer Analytical Services			
Name		Signature	Date
Laboratory Supervisor/Manager			

In This Book

Chapter 1 describes the purpose of operational qualification and performance verification testing, the prerequisites for testing, an overview of the individual tests, and a list of the parts and materials required.

Chapter 2 describes the handling and preparation of tubing, chemicals, flow cells, standards, and software, and completion of the performance verification procedure.

Chapter 3 contains the standard operating procedure for checking and setting the pump time for maximum acceptable cross contamination when using a sipper system or sipper/autosampler system.

Chapter 4 contains the SOP for testing and optimizing the alignment of each position of the multicell transport.

Chapter 5 contains the SOP for checking the temperature accuracy of the Peltier temperature-controlled cell holder using an external temperature measuring device.

Chapter 6 describes verification and revalidation of software components.

Chapter 7 describes verification and revalidation of the different online sampling systems for dissolution testing.

Chapter 8 helps you locate and identify parts and their part numbers.

Contents

1

Introduction

Introduction

This chapter describes the purpose of operational qualification and performance verification testing, the prerequisites for testing, an overview o the individual tests, and a list of the parts and materials required.

Purpose

Good laboratory practice (GLP) and good manufacturing practice (GMP) require that all laboratory instrumentation shall be adequately inspected, cleaned, and maintained. Instruments used for the generation, measurement, and evaluation of data shall be adequately tested, calibrated, and standardized. This protocol defines the methods and documentation that can be used to evaluate the HP 8453 UV-visible spectroscopy system for operation in accordance with the intended use and published specifications. Successful completion of this protocol will verify that the HP 8453 UV-visible spectroscopy system is performing to published specifications.

Scope

This document describes the operational qualification and performance verification (OQ/PV) procedure for the HP 8453 UV-visible spectroscopy system. This procedure applies to the following:

- HP 8453 spectrophotometer
- Sipper system including peristaltic pump and tubing
- Autosampler system including peristaltic pump and tubing
- Multicell transport
- Peltier temperature-controlled cell holder
- General purpose software for HP ChemStation
- Advanced software for HP ChemStation
- Biochemical software for HP ChemStation
- Dissolution testing software for HP ChemStation
- Dissolution testing hardware

NOTE This procedure does not apply to any spectroscopy system or module that has been altered or modified such that it no longer meets Hewlett-Packard design specifications.

Prerequisites

It is recommended that you do routine user maintenance immediately prior to running the OQ/PV procedure. User maintenance includes the following steps:

- For the HP 8453 spectrophotometer: clean the source lens and the spectrograph lens according to the procedures given in the *Reference Handbook* and the online help.
- To ensure a stable system, the spectrophotometer lamp should be switched on for a minimum of 45 minutes. For more consistent results, a 2-hour warm-up period is recommended prior to beginning this procedure.
- For the sipper and autosampler: check the quality of your pump tubing and replace it if required.
- Adjust the multicell transport mechanically or store the optimum positions in the software using the multicell transport cell adjustment tool (89075-23800).
- For the Peltier temperature-controlled cell holder there is no special maintenance required.
- For the dissolution testing system: disconnect the spectrophotometer from the bath, remove probes from the bath, exchange all pump tubings and probe filters.

Frequency of Testing

The OQ/PV procedure should be scheduled at least once each year, or after significant repair. However, you should determine the frequency of testing based on your laboratory's particular operating environment.

Maintenance/Calibration Contracts and Warranties

A warranty statement is included in the *HP 8453 Reference Manual.* If a failure is encountered during the OQ/PV procedure, the instrument will be removed from use until the appropriate repair is made. After the repair is completed, the reason for repair and any applicable data generated to show the repair corrected the existing failure should be logged according to current laboratory procedures. The instruments will be serviced by trained HP service representatives.

Simple maintenance can be performed by a trained analyst using the manuals or the online help system. Hewlett-Packard has trained service representatives who perform maintenance or repairs on an as-called basis.

Spare parts are listed in the individual manuals. Some parts are kept with the instrument and some are exchanged by Hewlett-Packard service representatives.

Declaration of Change Control

Change control procedures are in place in order to maintain the validation process. Any changes to the instrument hardware, or computer hardware or software must be clearly specified. A control system will determine the degree of revalidation required according to the extent of the changes. All details of the changes are thoroughly recorded and documented, together with details of completed tests and their results.

Records of OQ/PV

For the majority of the tests of the HP 8453 spectrophotometer, test results are printed automatically in a report on completion of the procedure.

If required, fill out the appropriate attachment form for each test.

Overview of the Procedures

The OQ/PV procedures use a combination of internal test functions and analytical methods (using certified samples) to evaluate instrument performance, and to verify the instrument is operating according to performance specifications agreed upon by you and Hewlett-Packard. The HP ChemStation software for UV-visible spectroscopy provides software for semiautomated OQ/PV of the HP 8453 spectrophotometer.

You can select to have this procedure performed by qualified staff within your organization, by Hewlett-Packard trained service engineers, or by an independent service provider. Typically, the complete OQ/PV procedure can be completed within 3 hours.

Tests

The OQ/PV procedure comprises the tests shown in Table 1.

Table 1 **OQ/PV Tests**

Instrument	Type of Test	Alternatives / Test Medium	Time Required
Spectrophotometer	Photometric Accuracy Test	NIST 930e solid standard	2 minutes
		Potassium dichromate solution	5 minutes
	Wavelength Accuracy Test	NIST 2034 holmium oxide standard	2 minutes
		Holmium oxide solution	5 minutes
	Stray Light Test	Sodium nitrite solution	5 minutes
		Sodium iodide solution	5 minutes
		Potassium chloride solution	5 minutes
	Resolution Test	Toluene-hexane solution	5 minutes
	Baseline Flatness Test	Through software	1 minutes
	Photometric noise test	Through software	2 minutes
	Photometric stability test	Through software	60 minutes

Table 1 **OQ/PV Tests, continued**

Instrument	Type of Test	Alternatives / Test Medium	Time Required
Peltier Temperature Controlled Cell Holder	Temperature accuracy	QuaT 100 temperature measuring device	15 minutes
Sipper System	Flow test	Through software/caffeine sample	5 minutes
Sampler system	Flow test	Through software/caffeine sample	5 minutes
Multicell transport	Position reproducibility	Through software with the multicell adjustment tool	5 minutes
General purpose software revalidation	Uses methods and data from the CD-ROM and a printout of results in this document	Through software	5 minutes
Advanced UV-Visible ChemStation Software Revalidation	Uses methods and data from the CD-ROM and a printout of results in this document	Through software	5 minutes
Biochemical analysis software revalidation	Uses methods and data from the CD-ROM and a printout of results in this document	Through software	5 minutes
Single-bath dissolution testing system based on the multicell transport	Spectrophotometer tests, multicell transport test, flow test, flow rate test, cross-contamination test	Through software/caffeine sample	210 minutes
Single-bath dissolution testing system based on the 8-port valve	Spectrophotometer tests, valvet test, flow test, flow rate test, cross-contamination test	Through software/caffeine sample	210 minutes
Dissolution testing multibath sampling system	Spectrophotometer tests, multicell transport test, valve test, flow test, flow rate test, cross-contamination test	Through software/caffeine sample	225 minutes
Dissolution testing software	Uses methods and data from the CD-ROM and a printout of results in this document	Through software	5 minutes
DDE Interface	Uses methods and data from the CD-ROM	Through software	5 minutes

Spectrophotometer

All of the following tests for the HP 8453 spectrophotometer are supported by a procedure in the HP ChemStation software.

Photometric Accuracy Test

The photometric accuracy test can be done two alternate ways, using

- the SRM 930e neutral density glass filter from NIST, or
- the potassium dichromate solution obtained from Hewlett-Packard sealed in an ampule, or a solution made up as specified by the European Pharmacopoeia (EP). A flow cell is used with PTFE tubing and a syringe to bring the solution into the light path.

Depending on the availability of the standards and the wavelength range of interest, one or the other or both ways can be used. The SRM 930e filter at 1 AU, uses wavelengths at 440.0, 465.0, 546.1, 590.0, and 635.0 nm, whereas the potassium dichromate standard uses wavelengths at 235, 257, 313, 350 nm.

Wavelength Accuracy Test

The wavelength accuracy test can be done two alternate ways, using

- the SRM 2034 holmium oxide standard from NIST which is a sealed cuvette containing holmium oxide in perchloric acid, or
- holmium oxide solution can be obtained from Hewlett-Packard sealed in an ampoule, or a solution can be made up as specified by the European Pharmacopoeia (EP). A flow cell is used with PTFE tubing and a syringe to bring the solution into the light path.

Depending on the availability of the standards one or the other ways can be used.

Stray Light Test

Stray light testing can be done using any one or any combination of solutions of:

- Sodium nitrite solution (ASTM)
- Sodium iodide solution (ASTM)
- Potassium chloride solution (EP)

These standards can be obtained from Hewlett-Packard sealed in an ampoule, or a solution can be made up as specified by the European

Pharmacopoeia (EP) or ASTM. A flow cell is used with teflon tubing and a syringe to bring the solution into the light path.

Resolution Test

The resolution test according to the European Pharmacopoeia is done using:

- Toluene-hexane solution

This solution obtained from Hewlett-Packard sealed in an ampoule, or a solution made up as specified by the European Pharmacopoeia (EP). A flow cell is used with PTFE tubing and a syringe to bring the solution into the light path.

Baseline Flatness Test

This test utilizes software only. It takes a scan without a cell in the cell holder and checks that the noise level of the complete spectrum (190–1100 nm) is lower than 0.001 AU rms.

Photometric Noise Test

This test utilizes software only. It takes 60 consecutive scans without a cell in the cell holder and checks that the noise level of all scans at 500 nm is lower than 0.0002 AU rms.

Photometric Stability Test

This test utilizes software only. It scans without a cell in the cell holder every 60 s, over 1 hour, after minimum warm-up time of 1 hour, and checks that the noise level of all scans at 340 nm is lower than 0.001 AU/h.

Peltier Temperature-Controlled Cell Holder Test

Temperature Accuracy

The temperature accuracy test uses a calibrated, external measuring device to verify temperature accuracy of the cell holder. An additional tool is required and can be obtained from Hewlett-Packard.

Sipper/Autosampler Pump Time Test

Flow Test

The flow test allows to check that the sampling system is operating correctly and indicates the presence of flow, restrictions of air leaks, low flow rates or high dead volumes which could lead to errors in measurement. This test uses a test solution obtained from Hewlett-Packard, or a solution made up according to the description in the procedure.

Multicell Transport Reproducibility Test

This test uses the diagnostic part of the HP ChemStation software to check the reproducibility of the cell positions of the multicell transport. The cell positions are checked initially, then the multicell transport is moved to random positions for 5 minutes. Afterwards a second check is made and compared with the initial results to check the positioning reproducibility. The required test tool can be obtained from Hewlett-Packard

General Purpose Software Revalidation

Revalidation uses methods and data from the support subdirectory on the CD-ROM and a printout of results in this document to check the integrity of the file storage, calculation and printout routines.

Advanced Software Revalidation

Revalidation uses methods and data from the support subdirectory on the CD-ROM and a printout of results in this document to check the integrity of the file storage, calculation and printout routines.

Biochemical Analysis Software Revalidation

Revalidation uses methods and data from the support subdirectory on the CD-ROM and a printout of results in this document to check the integrity of the file storage, calculation and printout routines.

Dissolution Testing Software Revalidation

Revalidation uses methods and data from the support subdirectory on the CD-ROM and a printout of results in this document to check the integrity of the file storage, calculation and printout routines.

Test for Single-bath Dissolution Testing System based on a Multicell-transport Sampling System

Flow Test (Peristaltic Pump 8VS / Multicell Transport)

The flow test allows to check that each channel of the sampling system is operating correctly, and indicates the presence of flow, restrictions of air leaks, low flow rates or high dead volumes which could lead to errors in measurement. This test uses a test solution obtained from Hewlett-Packard, or a solution made up according to the description in the procedure.

Flow Rate Test

The flow rate test allows to check that each channel of the sampling system is operating correctly, and indicates the presence of flow, restrictions of air leaks, low flow rates or high dead volumes which could lead to errors in measurement. This test uses distilled water and a beaker to measure the amount of water pumped through each channel.

Cross-Contamination Test

The cross-contamination test allows to check that each channel of the sampling system is operating correctly, and indicates the presence of carry over from successive pumping events. This test uses a test solution obtained from Hewlett-Packard, or a solution made up according to the description in the procedure.

Test for Single-bath Dissolution Testing System based on a Valve Sampling System

Flow Test (8-Port Valve / Sipper)

The flow test allows to check that each channel of the sampling system is operating correctly, and indicates the presence of flow, restrictions of air leaks, low flow rates or high dead volumes which could lead to errors in

measurement. This test uses a test solution obtained from Hewlett-Packa or a solution made up according to the description in the procedure.

Flow Rate Test

The flow rate test allows to check that each channel of the sampling syst is operating correctly, and indicates the presence of flow, restrictions of a leaks, low flow rates or high dead volumes which could lead to errors in measurement. This test uses distilled water and a beaker to measure the amount of water pumped through each channel.

Cross-Contamination Test

The cross-contamination test allows to check that each channel of the sampling system is operating correctly, and indicates the presence of carr over from successive pumping events. This test uses a test solution obtain from Hewlett-Packard, or a solution made up according to the description the procedure.

Test for Multibath Dissolution Testing System

Flow Rate Test for each Bath

The flow rate test allows to check that each channel of the sampling syster is operating correctly, and indicates the presence of flow, restrictions of al leaks, low flow rates or high dead volumes which could lead to errors in measurement. This test uses distilled water and a beaker to measure the amount of water pumped through each channel.

Cross-Contamination Test for each Bath

The cross-contamination test allows to check that each channel of the sampling system is operating correctly, and indicates the presence of carry over from successive pumping events. This test uses a test solution obtaine from Hewlett-Packard, or a solution made up according to the description i the procedure.

Acceptance Criteria

Test limits and acceptance criteria are provided in the software and in this document.

NOTE Note that the limits specified in this document and in the HP ChemStation online help system are for new instruments and for instruments which have been maintained and repaired with the intention of ensuring performance according to Hewlett-Packard specifications. Instruments in routine use over longer periods may not continue to meet all Hewlett-Packard specifications. Therefore, acceptance criteria may be assigned according to analytical requirements, and agreed to by yourself and your local Hewlett-Packard representative.

Performance specifications are measured after a minimum of 45 minutes from cold start or from lamp turn-on, with no cell or filter unless specified. Cold start in this context means that the spectrophotometer had been stored for some hours at room temperature.

NOTE For detailed procedures to verify the performance of the instrument, see Chapter 2 "Spectrophotometer Performance Verification" through Chapter 5 "Peltier Temperature- Controlled Cell Holder Performance Verification". For sources of standards and parts required, see Chapter 8 "Parts and Materials".

Table 2 Limits of Acceptance Criteria for the HP 8453 Spectrophotometer

Type	Specification	Comments
Resolution	> 1.6	Ratio of absorbance of peak/valley around 269 and 266 nm is greater than 1.5, blank scan on hexane; a 9-point spline function is used; 0.5 s integration time; (EP* method)
Stray light	< 1.0%	At 200 nm, solution of 1.2% KCl, blank scan on distilled water, 5 s integration time; (EP method)**
	< 0.05%	At 220 nm, solution of 10 g/l NaI, blank scan on distilled water, 5 s integration time; (ASTM method)
	< 0.03%	At 340 nm, solution of 50 g/l $NaNO_2$, blank scan on distilled water, 5 s integration time; (ASTM method)
Wavelength accuracy	< ± 0.5 nm	NIST 2034 standard, using transmittance peak minima; wavelength in NIST certificate are interpolated for 1.5 nm bandwidth from the values given for 2 nm and 1 nm bandwidth; uncertainty of standard from NIST certificate (typically ±0.1 nm) is added to the specification; 99-point spline function is used; 0.5 s integration time
Wavelength reproducibility	< ± 0.02 nm	Ten consecutive scans with NIST 2034 standard; 0.5 s integration time
Photometric accuracy	± 0.005 AU	NIST 930e standard at 1 AU, at 440.0, 465.0, 546.1, 590.0, and 635.0 nm, the expanded uncertainty from NIST certificate is added to the specification; 0.5 s integration time
Photometric accuracy	± 0.01 AU	Potassium dichromate in 0.01 N H_2SO_4 at 235, 257, 313, 350 nm; blank scan on 0.01 N H_2SO_4; 0.5 s integration time; blank scan on solvent; (EP method)
Photometric noise	< 0.0002 AU rms	Sixty consecutive scans on air with 0.5 s integration time at 0 AU, 500 nm; 11-point moving average: using equation: Noise(rms)=SQRT((SUM(X-x)^2)/n) where x are measured values, X is a 11-point moving average, n is the number of points
Photometric stability	< 0.001 AU/h	Scan on air at 0 AU, 340 nm, after 1-hour warm up, measured over 1 hour, every 60 s, integration time 5 s; difference between maximum and minimum values are compared to specification; at constant ambient temperature
Baseline flatness	< 0.001 AU rms	Scan on air at 0 AU, 190 - 1100 nm, 0.5 s integration time

* EP stands for European Pharmacopoeia

** Apparent absorbance is strongly affected by dissolved oxygen. According to ASTM, bubble pure nitrogen through liquid for several minutes immediately before use. Use only recently distilled water (not demineralized water).

OQ/PV Certification

This document provides a protocol to verify and record configuration and operation of analytical laboratory equipment from Hewlett-Packard. It has been prepared from best practices of Hewlett-Packard customers and has been found to be of use to them in satisfying the configuration and operational verification and recording requirements of various laboratory certification programs.

NOTE Certification depends upon many factors and use of this protocol alone does not assure certification and Hewlett-Packard makes no promises or representations as to its sufficiency for any specific certification program.

Spectrophotometer Performance Verification

Spectrophotometer Performance Verification

This chapter describes the handling and preparation of tubing, chemicals, flow cells, standards, and software, and completion of the performance verification procedure. The chapter has two sections:

- "Using the Performance Verification Procedure in the HP 8453 UV-Visibl Software" on page 32, and
- "Performance Test Procedures" on page 42.

Using the Performance Verification Procedure in the HP 8453 UV-Visible Software

This section guides you through the procedure to prepare flow cell, tubing, and standards and to configure the software for a performance verification. The performance verification procedure describes how to use the general purpose software and the spectrophotometer. Additional information is required to handle the standards and chemicals, see "Performance Test Procedures" on page 42.

Preparing the Flow Cells and Tubings

This chapter is only required if you are using the liquid holmium oxide, potassium dichromate, stray light and resolution standards in combination with the OQ/PV hardware kit from Hewlett-Packard.

Contents of the OQ/PV Hardware Kit

The parts included in the OQ/PV hardware kit are shown in Table 3. There are two sets of flow cells, tubings, and syringes given, one for the organic phase (toluene-in-hexane test), and one for the water phase. In addition, there are two tools provided, one for verification of the multicell transport and one to support measuring temperatures in the Peltier temperature-controlled cell holder with an external temperature measuring device.

Table 3 **Contents of OQ/PV Hardware Kit for UV-Visible (5063-6523)**

Quantity	Description
2	Flow cell, 3.5 × 11-mm aperture, 360-µl volume
1	Tubing, 40-cm long, one black fitting, one orange fitting
1	Tubing, 40-cm long, one black fitting, one white fitting
1	Tubing, 40-cm long, one black fitting with orange ring
1	Tubing, 40-cm long, one black fitting with white ring
2	Luer lock to screw fitting adapter
2	Syringe, 20 ml
1	Cell passivating and cleaning fluid, 1000 ml
1	Multicell transport adjustment tool
1	Temperature sensor support
1	Performance verification procedures handbook (including 3.5-inch flexible disk)

Assembling the Tubings

WARNING

To avoid contamination the flow cell and tubing for the organic pha test must never be used for the water phase test. Similarly, the flov cell and tubing for the water phase test must never be used for the organic phase test.

1 Use the set with the orange fittings for the organic phase (*O*range = *O*rgani phase) and the set with the white fittings for the water phase (*W*hite = *Wat* phase).

2 Use the long tubing with different fittings at either end and connect the blac hand-tight fitting to the outlet of the flow cell. The outlet of the flow cell is opposite of the inlet which is marked with an arrow on the quartz window.

3 Connect the other end of this tubing to the Luer-lock adapter and fix the adapter to the syringe.

4 Use the short tubing with one open end and connect the fitting to the inlet o the flow cell. (The inlet is marked with an arrow on the quartz window.)

5 Check that the quartz windows of the flow cell are clean. If not, wipe the optical surfaces with a lint free optical tissue.

6 Place the flow cell in the cell holder of the spectrophotometer.

After you have assembled the tubings flow cells and syringes, leave all parts together to avoid mixing the parts up.

Preparing for the Tests

The parts included in the verification standards kits are shown in Table 4 and Table 5.

Table 4 **Contents of the OQ/PV Standards (1) Kit for UV-Visible (5063-6503)**

Quantity	Description
2	0.01 N sulfuric acid
2	Potassium dichromate solution
1	Sodium nitrite solution
1	Sodium iodide solution
1	Potassium chloride solution
2	Hexane
1	Toluene solution

Table 5 **Contents of the OQ/PV Standards (2) Kit for UV-Visible (5063-6521)**

Quantity	Description
1	10% perchloric acid
1	Holmium oxide solution

1 To avoid spill of chemicals while drawing sample from an open ampule use a beaker which is small enough to hold an ampule in upright position. Position this beaker in reach of the inlet tubing of the flow cell.

2 Get a waste container with 500-ml volume.

3 Get a beaker with approximately 250-ml of distilled water, HPLC grade.

4 Position the ampules on your lab bench in the sequence how they will be us during the verification procedure:

sulfuric acid,
potassium dichromate solution,
perchloric acid,
holmium oxide solution,
sodium nitrite solution,
sodium iodide solution,
potassium chloride solution,
hexane,
toluene solution.

Cleaning Your Flow Cell and Tubings

Apply the following steps every time before you do a verification to make sure your flow cell for the water phase is clean and does not trap any air bubbles.

NOTE

This procedure applies only for the flow cell and tubings used for the wate phase. Do not use it for the flow cell and tubings which are reserved for the hexane-toluene test. There is no cleaning procedure necessary for these flo cell and tubings.

Draw 50 ml of cell passivating and cleaning fluid through flow cell.

1 Prepare minimum 50 ml a solution of 5 % cell passivating and cleaning fluid (part number 5062-8529) in water.

2 Place the inlet tubing into the beaker with the 5 % cell passivating and cleanin fluid and use the syringe to slowly draw the 50 ml of cell passivating and cleaning fluid through the flow cell.

NOTE

You may observe a high amount of air coming through the flow cell, because the solution contains a detergent.

Empty the syringe.

3 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing which you disconnected at the same level as the inlet tubing to avoid emptying of the tubing.

4 Empty the syringe into the waste container and re-connect the syringe to the Luer-lock adapter at the tubing.

5 Repeat steps 2, 3 and 4 until you have drawn the 50 ml of cell passivating and cleaning fluid through the flow cell and tubings. For extensive cleaning you may leave the fluid in the flow cell and tubings for an extended time period.

Draw 100 ml of water through flow cell.

6 Place the inlet tubing into a beaker with water and draw 100 ml of water through the flow cell.

7 Empty the syringe into the waste container and reconnect the syringe to the Luer-lock adapter at the tubing.

8 Repeat steps 6 and 7 until you have drawn the 100 ml of water through the flow cell and tubings.

Performance Verification of the HP 8453 Spectrophotometer

Scope

The following procedure describes how to configure the verification procedure on the HP ChemStation for UV-visible spectroscopy.

Frequency

Follow appropriate procedure:

- when you first install your HP 8453 UV-visible spectroscopy system,
- on a regular base at intervals of a minimum of 6 months,
- if you connect a different HP 8453 spectrophotometer to your HP ChemStation.

Instrumentation and software

This SOP applies to a HP 8453 UV-Visible spectroscopy system comprising HP 8453 spectrophotometer and general purpose software for the HP ChemStation.

Requirements

Different tests have different environmental temperature conditions according to their original specifications by NIST and the various Pharmacopoeias. An environmental temperature range between 20–21 °C meets all specifications on which the test in this manual are based.

All liquid standards should be stored at environmental temperature for approximately 3 hours. The spectrophotometer should have been turned or for at least 45 minutes—preferably for 2 hours.

To perform configure and subsequently perform the verification procedure you may need the following standards from NIST or as liquid standards in ampules from Hewlett-Packard depending upon the test set that you select.

For sources of standards, see "Standards from External Sources" on page 202.

- Photometric accuracy: NIST 930e or potassium dichromate solution
- Wavelength accuracy: NIST 2034 or built-in deuterium lamp emission lines test or holmium oxide solution
- Stray light: liquid potassium chloride, and sodium nitrite, and sodium iodide solutions
- Resolution: toluene solution
- Photometric noise: software only, no standards required
- Baseline flatness: software only, no standards required
- Photometric stability: software only, no standards required

Where appropriate the 930e NIST standards should have been calibrated or recalibrated within two years of use. Each standard should be clearly labeled with an identity number for positive identification.

Procedure

1 If the spectrophotometer is not already turned on, switch the spectrophotometer on.

2 Start the HP ChemStation.

3 Select Verification and Diagnostics from the Mode menu or tool bar.

4 Select the Verification task in the graphical interface or by using the Task menu.

5 Under sampling system select Setup and make sure the path length is set to 1 cm. Choose OK to leave this dialog box.

6 Select the verification task Setup. A table showing the tests available is displayed.

7 Select the tests which you want to perform which are appropriate to your specific verification requirements and, where appropriate, enter information on the standards you will use using the Setup buttons.

Because the tests listed are of different complexity and duration it is recommended to do the following groups of tests together in two differe subsequent performance verification procedures:

all tests requiring external standards, e.g. wavelength accuracy, stray ligh potassium dichromate, and

1-hour photometric stability test, noise test, baseline flatness, absorbanc stability test, deuterium lamp emission lines test.

8 When you have finished entering the data, select OK.

9 Check that the environmental temperature is between 20 and 21 °C and th the liquid standards have been stored at this temperature for approximatel hours.

10 Make sure the spectrophotometer has been turned on for at least 45 minutes—preferably 2 hours.

11 Start the verification procedure by clicking on the Start button in the graphi interface.

12 Follow exactly the instructions displayed on your monitor ensuring that th identity numbers of the standards you use match those displayed.

Use the appropriate test procedures for each individual standard on the following pages when applying the performance verification tests in the software:

Photometric Accuracy Test For the photometric accuracy test apply the procedures "Potassium Dichromate Solution Test" on page 43, or perfor the NIST 930e photometric accuracy test and follow the procedure as prompted by the software.

Wavelength Accuracy Test For the wavelength accuracy test apply th procedures "Holmium Oxide Solution Test" on page 46, perform the NIST 2034 holmium oxide test and follow the procedure as prompted by the software, or perform the deuterium lamp emission lines test and follow the procedure as prompted by the software. Do not forget to remove the flow ce or cuvette from the sample area for this test.

Stray Light Tests For the stray light tests apply the procedures "Sodiun Nitrite Stray Light Test" on page 49, "Sodium Iodide Stray Light Test" on page 52, and "Potassium Chloride Stray Light Test" on page 55.

Resolution Test For the resolution test apply the procedure "Toluene Resolution Test" on page 58.

Noise Test
Baseline Flatness Test
Absorbance Stability Test These tests will run unattended. Follow the procedure as prompted by the software.

13 Print the report and sign it.

14 If you want to save the verification configuration as part of the instrument configuration, close the HP ChemStation software by selecting Exit from the File menu and make sure that the Save Configuration box has been selected.

15 To clean the flow cell and tubings, perform the procedure "Before you Store the Flow Cell and Tubings" on page 60.

Acceptance

The instrument passes the verification test if the report show pass for each individual test. Use Fill-in Form 5 on page 61 and Fill-in Form 6 on page 62 to document your results.

Troubleshooting

If the spectrophotometer fails the verification test, refer to the standard operating procedures (SOPs) given on the support subdirectory on the CD-ROM which comes with your general purpose software. Run the instrument intensity spectrum diagnostic and, if it is low, replace the lamp according to the SOP "Changing the Lamp of the HP 8453" and clean the source lens according to the SOP "Cleaning the Source Lens of the HP 8453" and repeat the verification procedure.

If, after completing the above, the instrument does not pass the verification test, call Hewlett-Packard.

Performance Test Procedures

This section lists the procedures to correctly handle the chemicals, flow c and standards. For successful performance verification great care must b taken to follow exactly these procedures. Very small contaminations of standards, flow cell or the chemicals may result in failures of the procedur Advice about waste disposal are given to avoid chemical reactions when disposing the used chemicals, because the concentrations of the chemical are relatively high.

Potassium Dichromate Solution Test

Always use the flow cell and tubings for water phase (white fittings) for this standard. Dispose the waste according to the local safety regulations.

WARNING **Wear eye glasses and gloves when breaking the ampules, because small glass particles may come off. Observe the warning symbols and labels on the ampules and their packing material and act accordingly.**

CAUTION Never remove the flow cell from the cell holder between blank measurement and sample measurement. This may cause errors which will give wrong results.

Doing the Blank Measurement

At the prompt:

```
Verification Test 2: Photometric Accuracy

Rinse flow cell or cuvette with water and fill
with 0.01N sulfuric acid for blank measurement.
```

perform the following steps:

Draw 15 ml of water through flow cell.

1 Place the inlet tubing in the beaker with water and use the syringe to draw 15 ml of water into the flow cell.

Empty the syringe.

2 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing you disconnected at the same level as the inlet tubing to avoid emptying the tubing and getting air in the flow cell. Empty the syringe in the waste container.

Re-connect the syringe.

3 Reconnect the syringe to the Luer-lock adapter at the tubing.

4 To observe that the flow cell is free from air you may remove it from the holder of the spectrophotometer. If there is air in the flow cell which is difficult to remove, position it that the outlet tubing is at the highest poir while drawing water through it.

Break ampule with Sulfuric acid.

5 Select an ampule which contains the 0.01 N sulfuric acid. Make sure the up part of the ampule which will be broken off does not contain any liquid. T remove any liquid, turn the ampule upside down to fill it entirely and slov turn it back that the liquid in the upper part can flow down.

6 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of Sulfuric acid through flow cell.

7 Remove the inlet tubing from the beaker with water and wipe off any liqui the end using a tissue.

8 Place the inlet tubing in the ampule and draw 9 ml of 0.01 N sulfuric acid us the syringe.

9 Make sure the flow cell is in the cell holder and the lever is locked (in dov position).

Start Blank measurement.

10 Before you start the blank measurement, make sure you are prepared to c the sample measurement immediately afterwards.

CAUTION You have only 4 minutes to do the sample measurement after the blank. If four minutes elapse you will be asked to repeat the blank measurement ag

11 Select OK to start the blank measurement.

Doing the Potassium Dichromate Measurement

Break ampule with Potassium Dichromate solution.

1 Break the ampule which contains potassium dichromate in 0.01 N sulfuric acid and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of sample through flow cell.

2 Take the inlet tubing and wipe any liquid off the end using a tissue. There should be an air plug between 1 mm and 10 mm inside the tubing to avoid contamination of the standard when you put it into the ampule.

3 Place the inlet tubing in the ampule and draw 9 ml of sample using the syringe.

Start measurement.

4 Start the measurement in the software, selecting the OK button.

Draw 3-5 ml of water through flow cell.

5 When the measurement has finished, remove the inlet tubing from the ampule and wipe any liquid off the end using a tissue.

6 Place the inlet tubing in the beaker with water and draw up 3–5 ml of water to flush the tubings.

Empty the syringe.

7 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing which you disconnected at the same level as the inlet tubing to avoid emptying of the tubing, i.e. air getting into the flow cell. Empty the syringe into the waste container.

Reconnect the syringe

8 Reconnect the syringe to the Luer-lock adapter at the tubing.

If this was the last performance test in your test sequence, clean the flow cell before storing it, see "Before you Store the Flow Cell and Tubings" on page 60.

Holmium Oxide Solution Test

Always use the flow cell and tubings for water phase (white fittings) for standard. Dispose the waste according to the local safety regulations.

WARNING **Wear eye glasses and gloves when breaking the ampules, because si glass particles may come off. Observe the warning symbols and lal on the ampules and their packing material and act accordingly.**

CAUTION Never remove the flow cell from the cell holder between blank measuren and sample measurement. This may cause errors which will give wrong results.

Doing the Blank Measurement

At the prompt:

```
Verification Test 1: Wavelength Accuracy

Rinse flow cell or cuvette with water and fill
with 10 % perchloric acid for blank measurement.
```

perform the following steps:

Draw 15ml of water through flow cell.

1 Place the inlet tubing in the beaker with water and use the syringe to drav 15 ml of water in the flow cell.

Empty the syringe.

2 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position t tubing you disconnected at the same level as the inlet tubing to avoid empty the tubing and getting air in the flow cell. Empty the syringe in the waste container.

Re-connect the syringe.

3 Reconnect the syringe to the Luer-lock adapter at the tubing.

4 To observe that the flow cell is free from air you may remove it from the cell holder of the spectrophotometer. If there is air in the flow cell which is difficult to remove, position it that the outlet tubing is at the highest point while drawing water through it.

Draw 9ml of 10% perchloric acid through flow cell.

5 Select an ampule which contains the 10% perchloric acid. Make sure the upper part of the ampule that will be broken off does not contain any liquid. To remove any liquid, turn the ampule upside down to fill it entirely and slowly turn it back that the liquid in the upper part can flow down.

6 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

7 Place the inlet tubing into the ampule and use the syringe to draw 9 ml of 10 % perchloric acid through the flow cell.

8 Make sure the flow cell is in the cell holder and the lever is locked (in down position).

Start Blank measurement.

9 Select OK to start the blank measurement.

Doing the Holmium Oxide Solution Measurement

Break ampule with Holmium Oxide solution.

1 Select an ampule which contains the holmium oxide in 10 % perchloric acid. Make sure the upper part of the ampule which will be broken off does not contain any liquid. To remove any liquid, turn the ampule upside down to fill it entirely and slowly turn it back that the liquid in the upper part can flow down.

2 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of sample through flow cell.

3 Take the inlet tubing and wipe any liquid off the end using a tissue. There should be an air plug between 1 mm and 10 mm inside the tubing to avoid contamination of the standard when you put it into the ampule.

4 Place the inlet tubing in the ampule and draw 9 ml of sample using the syri

Start measurement.

5 Start the measurement in the software, selecting the ok button.

Draw 3-5 ml of water through flow cell.

6 When the measurement has finished, remove the inlet tubing from the amp and wipe any liquid off the end using a tissue.

7 Place the inlet tubing in the beaker with water and draw 3–5 ml of water t flush the tubings.

Empty the syringe.

8 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position t tubing you disconnected at the same level as the inlet tubing to avoid emptyi the tubing and getting air in the flow cell. Empty the syringe in the waste container.

Reconnect the syringe.

9 Reconnect the syringe to the Luer-lock adapter at the tubing.

If this was the last performance test in your test sequence, clean the flow ce before storing it, see "Before you Store the Flow Cell and Tubings" on page 60.

Sodium Nitrite Stray Light Test

Always use the flow cell and tubings for water phase (white fittings) for these standard. Dispose the waste according to the local safety regulations.

WARNING

Wear eye glasses and gloves when breaking the ampules, because small glass particles may come off. Observe the warning symbols and labels on the ampules and their packing material and act accordingly.

CAUTION

Never remove the flow cell from the cell holder between blank measurement and sample measurement. This may cause errors which will give wrong results.

Doing the Blank Measurement

At the prompt:

```
Verification Test 3: Stray Light

Fill flow cell or cuvette with
water for blank measurement.
```

perform the following steps:

Draw 20 ml of water through flow cell.

1 Place the inlet tubing into the beaker with water and use the syringe to draw 20 ml of water into the flow cell.

Empty the syringe.

2 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing you disconnected at the same level as the inlet tubing to avoid emptying the tubing and getting air in the flow cell. Empty the syringe in the waste container.

Reconnect the syringe.

3 Reconnect the syringe to the Luer-lock adapter at the tubing.

4 To observe that the flow cell is free from air you may remove it from th holder of the spectrophotometer. If there is air in the flow cell which is difficult to remove, position it that the outlet tubing is at the highest po while drawing water through it.

5 Make sure the flow cell is in the cell holder and the lever is locked (in d position).

Start Blank measurement.

6 Select OK to start the blank measurement.

Doing the Sodium Nitrite Measurement

Break ampule with stray light standard.

1 Select an ampule with sodium nitrite in water.

2 Make sure the upper part of the ampule which will be broken off does nc contain any liquid. To remove any liquid, turn the ampule upside down tc it entirely and slowly turn it back that the liquid in the upper part can flo down.

3 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of sample through flow cell.

4 Take the inlet tubing and wipe any liquid off the end using a tissue. There should be an air plug between 1 mm and 10 mm inside the tubing to avoic contamination of the standard when you put it into the ampule.

5 Place the inlet tubing in the ampule and draw 9 ml of sample using the syri

Start measurement.

6 Start the measurement of the respective verification procedure in the software, selecting the OK button.

Draw 3–5 ml of water through flow cell.

7 When the measurement has finished, remove the inlet tubing from the ampule and wipe any liquid off the end using a tissue.

8 Place the inlet tubing in the beaker with water and draw up 3–5 ml of water to flush the tubings.

Empty the syringe.

9 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing you disconnected at the same level as the inlet tubing to avoid emptying the tubing and getting air in the flow cell. Empty the syringe into the waste container.

Reconnect the syringe

10 Reconnect the syringe to the Luer-lock adapter at the tubing.

If this was the last performance test in your test sequence, clean the flow cell before storing it, see "Before you Store the Flow Cell and Tubings" on page 60.

Sodium Iodide Stray Light Test

Always use the flow cell and tubings for water phase (white fittings) for th standard. Dispose the waste according to the local safety regulations.

WARNING **Wear eye glasses and gloves when breaking the ampules, because sn glass particles may come off. Observe the warning symbols and lab on the ampules and their packing material and act accordingly.**

CAUTION Never remove the flow cell from the cell holder between blank measurem and sample measurement. This may cause errors which will give wrong results.

Doing the Blank Measurement

At the prompt:

```
Verification Test 3: Stray Light

Fill flow cell or cuvette with
water for blank measurement.
```

perform the following steps:

Draw 20 ml of water through flow cell.

1 Place the inlet tubing into the beaker with water and use the syringe to dra 20 ml of water into the flow cell.

Empty the syringe.

2 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position tl tubing you disconnected at the same level as the inlet tubing to avoid emptyi the tubing and getting air in the flow cell. Empty the syringe in the waste container.

Reconnect the syringe.

3 Reconnect the syringe to the Luer-lock adapter at the tubing.

4 To observe that the flow cell is free from air you may remove it from the cell holder of the spectrophotometer. If there is air in the flow cell which is difficult to remove, position it that the outlet tubing is at the highest point while drawing water through it.

5 Make sure the flow cell is in the cell holder and the lever is locked (in down position).

Start Blank measurement.

6 Select OK to start the blank measurement.

Doing the Sodium Iodide Measurement

Break ampule with stray light standard.

1 Select an ampule with sodium iodide in water.

2 Make sure the upper part of the ampule which will be broken off does not contain any liquid. To remove any liquid, turn the ampule upside down to fill it entirely and slowly turn it back that the liquid in the upper part can flow down.

3 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of sample through flow cell.

4 Take the inlet tubing and wipe any liquid off the end using a tissue. There should be an air plug between 1 mm and 10 mm inside the tubing to avoid contamination of the standard when you put it into the ampule.

5 Place the inlet tubing in the ampule and draw 9 ml of sample using the syringe.

Start measurement.

6 Start the measurement of the respective verification procedure in the software, selecting the OK button.

Draw 3-5 ml of water through flow cell.

7 When the measurement has finished, remove the inlet tubing from the am and wipe any liquid off the end using a tissue.

8 Place the inlet tubing in the beaker with water and draw up 3–5 ml of wat flush the tubings.

Empty the syringe.

9 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you positio tubing you disconnected at the same level as the inlet tubing to avoid empt the tubing and getting air in the flow cell. Empty the syringe in the waste container.

Reconnect the syringe

10 Reconnect the syringe to the Luer-lock adapter at the tubing.

If this was the last performance test in your test sequence, clean the flow before storing it, see "Before you Store the Flow Cell and Tubings" on page 60.

Potassium Chloride Stray Light Test

Always use the flow cell and tubings for water phase (white fittings) for these standard. Dispose the waste according to the local safety regulations.

WARNING **Wear eye glasses and gloves when breaking the ampules, because small glass particles may come off. Observe the warning symbols and labels on the ampules and their packing material and act accordingly.**

CAUTION Never remove the flow cell from the cell holder between blank measurement and sample measurement. This may cause errors which will give wrong results.

Doing the Blank Measurement

At the prompt:

```
Verification Test 3: Stray Light

Fill flow cell or cuvette with
water for blank measurement.
```

perform the following steps:

Draw 20 ml of water through flow cell.

1 Place the inlet tubing in the beaker with water and use the syringe to draw 20 ml of water into the flow cell.

Empty the syringe.

2 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing which you disconnected at the same level as the inlet tubing to avoid emptying the tubing and getting air in the flow cell. Empty the syringe into the waste container.

Reconnect the syringe.

3 Reconnect the syringe to the Luer-lock adapter at the tubing.

4 To observe that the flow cell is free from air you may remove it from the c holder of the spectrophotometer. If there is air in the flow cell which is difficult to remove, position it that the outlet tubing is at the highest point while drawing water through it.

5 Make sure the flow cell is in the cell holder and the lever is locked (in dow position).

Start Blank measurement.

6 Select OK to start the blank measurement.

Doing the Potassium Chloride Measurement

Break ampule with stray light standard.

1 Select an ampule with Potassium Chloride in water. Make sure the upper pa of the ampule which will be broken off does not contain any liquid. To remo any liquid, turn the ampule upside down to fill it entirely and slowly turn it back that the liquid in the upper part can flow down.

2 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of sample through flow cell.

3 Take the inlet tubing and wipe any liquid off the end using a tissue. There should be an air plug between 1 mm and 10 mm inside the tubing to avoid contamination of the standard when you put it into the ampule.

4 Place the inlet tubing in the ampule and draw 9 ml of sample using the syring

Start measurement.

5 Start the measurement of the respective verification procedure in the software, selecting the ok button.

Draw 3-5 ml of water through flow cell.

6 When the measurement has finished, remove the inlet tubing from the ampule and wipe any liquid off the end using a tissue.

7 Place the inlet tubing in the beaker with water and draw up 3-5 ml of water to flush the tubings.

Empty the syringe.

8 To empty the syringe, leave the inlet tubing in the beaker with water and disconnect the Luer-lock adapter from the syringe. Make sure you position the tubing which you disconnected at the same level as the inlet tubing to avoid emptying of the tubing, i.e. air getting into the flow cell. Empty the syringe into the waste container.

Re-connect the syringe

9 Reconnect the syringe to the Luer-lock adapter at the tubing.

If this was the last performance test in your test sequence, clean the flow cell before storing it, see "Before you Store the Flow Cell and Tubings" on page 60.

Toluene Resolution Test

Always use the flow cell and tubings for organic phase (orange fittings) fc this standard. Dispose the waste according to the local safety regulations.

WARNING **Wear eye glasses and gloves when breaking the ampules, because sm glass particles may come off. Observe the warning symbols and labe on the ampules and their packing material and act accordingly.**

CAUTION Never remove the flow cell from the cell holder between blank measureme and sample measurement. This may cause errors which will give wrong results.

Doing the Blank Measurement

At the prompt:

```
Clear sample area for Blank Measurement
```

use the following steps:

Break ampule with Hexane.

1 Select an ampule which contains hexane. Make sure the upper part of the ampule which will be broken off does not contain any liquid. To remove any liquid, turn the ampule upside down to fill it entirely and slowly turn it back that the liquid in the upper part can flow down.

2 Break the ampule and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of Hexane through flow cell.

3 Place the inlet tubing in the ampule and draw 9 ml of hexane using the syringe

4 To observe that the flow cell is free from air you may remove it from the cell holder of the spectrophotometer. If there is air in the flow cell which is difficult to remove, position it that the outlet tubing is at the highest point while drawing water through it.

5 Leave the inlet tubing in the ampule until the blank measurement has finished to avoid air entering the tubing.

6 Make sure the flow cell is in the cell holder and the lever is locked (in down position).

Start Blank measurement.

7 Select OK to start the blank measurement.

Doing the Toluene Measurement

Break ampule with Toluene-Hexane.

1 Break the ampule which contains toluene solution and position the open ampule in a beaker next to the instrument that it cannot fall over.

Draw 9 ml of sample through flow cell.

2 Take the inlet tubing and wipe any liquid off the end using a tissue. There should be an air plug between 1 mm and 10 mm inside the tubing to avoid contamination of the standard when you put it into the ampule.

3 Place the inlet tubing in the ampule and draw 9 ml of sample using the syringe.

Start measurement.

4 Start the measurement in the software, selecting the ok button.

Empty the syringe.

5 Empty the syringe into the waste container.

6 Clean the flow cell before storing it, see "Before you Store the Flow Cell and Tubings" on page 60.

Before you Store the Flow Cell and Tubings

Storing the Water Phase Flow Cell and Tubings

CAUTION Do not apply this procedure to the organic phase flow cell and tubings (orang color code).

1. After the last test in your test sequence is finished, purge the flow cell with 2 ml of water from the beaker.
2. Position the flow cell on a tissue and draw as much air as possible through th flow cell into the syringe. Hold the flow cell upside down that any liquid in th flow cell is drained.
3. Empty the syringe into the waste container if necessary. Repeat this and the previous step 3–4 times.
4. Disconnect the Luer-lock adapter from the syringe and store all items away.

Storing the Organic Phase Flow Cell and Tubings

CAUTION Do not apply this procedure to the water phase flow cell and tubings (white color code).

1. After the toluene solution test, draw air through the flow cell to empty it.
2. Break the second ampule which contains n-Hexane and draw the n-hexane through the flow cell.
3. Position the flow cell on a tissue and draw as much air as possible through the flow cell into the syringe. Hold the flow cell upside down that any liquid in the flow cell is drained.
4. Empty the syringe into the waste container if necessary. Repeat this and the previous step 3–4 times.
5. Disconnect the Luer-lock adapter from the syringe and store all items away.

Spectrophotometer OQ/PV Attachment Forms

Use Fill-in Form 5 and to record the results of the spectrophotometer performance verification.

Fill-in Form 5

Spectrophotometer Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	G1103A
Manufacturer	Model #

Serial #	Asset #	System #

Use the printout of the software to record your results in detail. Fill-in Form 6 gives you an overview of all available tests.

If a test is not useful for your particular situation, you can mark it with n/a for not applicable.

Performed by	Checked by

Fill-In Form 6

Spectrophotometer Test Results Record

Test	Test Procedure	n/a	Pass	Fail
	Photometric Accuracy Tests:			
1	Potassium dichromate solution test			
2	NIST 930e photometric accuracy test			
	Wavelength Accuracy Tests:			
3	Holmium oxide solution test			
4	NIST 2034 Holmium OxideTest			
5	Deuterium lamp emission line test			
	Stray Light Tests:			
6	Sodium nitrite stray light test			
7	Sodium iodide stray light test			
8	Potassium chloride stray light test			
	Resolution Test:			
9	Toluene solution test			
	Unattended Instrument Tests:			
10	Noise test			
11	Baseline flatness test			
12	Absorbance stability test			

Test	Reason for n/a for any of the above tests

Comments | Completed by

3

Sipper/Autosampler Performance Verification

Sipper/Autosampler Performance Verification

This chapter contains the standard operating procedure for checking and setting the pump time for maximum acceptable cross contamination when using either a sipper system (order number 89068D) or sipper/autosampler system (order number 89068D and 89072A, or 89068D and Gilson 221/222/223).

Sipper/Autosampler Pump Time Test

Scope

The following procedure describes how to determine the pumping time required to achieve an appropriate acceptable cross contamination between consecutive samples when using a sipper or autosampler system with the HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure when:

- you install or reinstall the sipper system, and
- at daily intervals.

Instrumentation and Software

This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer with a sipper system (order number 89068D) and general purpose software for the HP ChemStation (order number G1115AA) . The sipper system may be used alone or in combination with an autosampler.

Requirements

To perform this SOP you will require:

- approximately 50 ml of a test sample solution consisting of 0.01 mg/ml of caffeine. This concentration of caffeine has an absorbance of 1.4 AU at 205 nm and 0.5 AU at 273 nm (another sample may be substituted which absorbs at the analytical wavelength of your specific analysis. This sample should have an absorbance of approximately 1 AU), and
- 100 ml of the solvent you used to prepare the sample solution.

Procedure

1 The sipper system should be properly installed as described in the manual.

2 Switch on the HP 8453 spectrophotometer and allow it to warm up for at least 20 minutes before making any measurements.

3 Start the HP ChemStation software.

4 Select the Verification and Diagnostics mode using the tool bar Mode section (or use the Mode menu).

5 Select the Flow Test Task in the graphical user interface (or use the Task menu).

6 Ensure that the system is properly configured for the appropriate sipper accessory using the Sampling section of the graphical user interface.

7 Set the parameters for operation of the sipper by selecting Setup and Parameter in the graphical interface (or use the Instrument menu, Setup Sampling System Parameter) and entering the following:

Pump time: 20 s

Pump Direction: CW

Wait time: 3 s

Sample Return: 0 %

Wash time: 0 s

Air Segment: 0 s

8 Set the wavelength for your test sample and the desired level of purity using the Setup button in the Flow Test window of the graphical user interface.

9 Put the end of the sipper tube (or the end of the autosampler probe) into the reservoir containing the solvent.

10 Switch on the pump by clicking on the pump icon in the instrument panel of the graphical user interface and flush the system for approximately twice as long as the pump time you set. Switch off the pump by clicking on the pump icon.

11 Put the end of the sipper tube (or the end of the autosampler probe) into the reservoir containing the test sample and start the test using the Run button in the Flow Test window. The test will take 50% longer than the pump time you

have entered. You should get a graphic display of the absorbance versus time trace and a display of the pump time in seconds required to achieve the entered % purity.

12 If the estimated pump time is more than 20% different from your initial pump time, enter the estimated pump time under step 7 and repeat steps 9 through 11. Repeat until you get consecutive results within 10% or 1 second, whichever is greater.

Acceptance

Optimal pump time has been achieved, when the flow test indicates that you get consecutive results within 10% or 1 second, whichever is greater. Use Fill-in Form 7 through Fill-in Form 10 to document your results. If the test fails, refer to "Troubleshooting" on page 67.

Troubleshooting

If good results cannot be obtained:

- Check if there is a bubble in the flow cell, if yes, gentle tapping of the cell will help to dislodge it.
- Check the flow rate of your pump and replace the pump tubing if necessary.
- Check for air being sucked in at one of the fittings.
- Check rate of your pump and replace the pump tubing if necessary.

Sipper/Autosampler Attachment Forms

Use Fill-in Form 7 and Fill-in Form 8 to record the results of the sipper/autosampler performance verification.

Fill-in Form 7

Sipper Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	89068C
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 8

Sipper Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	89072A
Manufacturer	Model #

Autosampler Serial #	Asset #	System #

Fill-in Form 9 and Fill-in Form 10 give you an overview of all available tests.

Fill-in Form 9

Sipper Test Results

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time				
	Passed		Failed	

Fill-in Form 10

Autosampler Test Results

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time				
	Passed		Failed	

4

Multicell Transport Performance Verification

Multicell Transport Performance Verification

This chapter contains the standard operating procedure for testing and optimizing the alignment of each position of the multicell transport (order number 89075D or G1120A).

Multicell Transport Reproducibility Test

Scope

The following procedure describes how to achieve optimal alignment of the multicell transport when used with a HP 8453 UV-visible spectroscopy system.

Frequency

Follow the appropriate procedure:

- when you install or reinstall the multicell transport assembly,
- after repairing the multicell transport assembly, and
- at monthly intervals.

Instrumentation and Software

This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer with a multicell transport (order number 89075D or G1120A) and general purpose software for HP ChemStation (order number G1115AA). The multicell transport is used in place of a single cell holder.

Requirements

To perform this SOP you will require:

- MCT adjustment tool (part number 89075-23800) as the target used to check alignment of the multicell transport.

Procedure

1 The multicell transport assembly should be properly installed as described in the manual.

2 All flow cells and/or cuvettes should be removed from the multicell transport assembly.

3 Switch on the spectrophotometer and allow it to warm up for at least 20 minutes before attempting to align the multicell transport assembly.

4 Start the HP ChemStation software.

5 Select the Verification and Diagnostics mode using the tool bar Mode selection (or use the Mode menu).

6 Select the Maintenance Task from the graphical user interface task panel (or use the Task menu).

7 Select the MCT Test/Recalibration function from the graphical interface task panel.

8 If you are verifying a multicell transport of type 89075D, open the locking lever on the multicell transport assembly.

9 Insert the multicell adjustment tool into position 2 of the multicell transport. Orient the indicator on the top of the multicell adjustment tool towards the light source. If you are verifying a multicell transport of type 89075D, close the locking lever. During this step, position 1 must remain free and clear of all obstructions to allow for a successful blank measurement.

10 In the multicell transport adjustment test panel, deselect all cell positions with the exception of cell position 2.

11 Start the test.

12 When the test has finished, select recalibrate, and repeat the test. The results should indicate that this cell position is now optimally aligned.

13 Repeat steps 8 through 11 using cell positions 3, 4, 5, 6, 7, 8 (only when a multicell transport of type G1120A is installed) and 1, ensuring optimal alignment of all cell positions within the multicell transport assembly.

Acceptance

Optimal alignment of the multicell transport of type 89075C/Dhas been successful when the multicell transport adjustment test indicates that all cell positions require no further adjustment, that means the turns necessary to adjust the multicell transport are less than 0.2 turns. Use Fill-in Form 12 on page 77 to document your results. The multicell transport of type G1120A is recalibrated by the firmware of the spectrophotometer and needs no mechanical adjustment. If the test fails, refer to "Troubleshooting" on page 75.

Troubleshooting

If good results cannot be obtained:

- Check to be sure the sampling system is properly configured as a multicell transport.
- Check to be sure that the multicell transport is properly mounted within the sample compartment.
- Check to be sure the multicell transport adjustment tool is seated and locked properly into the multicell transport assembly.
- For a multicell transport of type 89075D, use the MCT alignment tool in cell position 4, complete steps 8 and 9 to ensure alignment of the MCT. Using a 1.5 mm hex key (part number 8710-0909), turn the home sensor switch adjustment screw the recommended number of turns. Repeat the multicell transport reproducibility test.
- Check to be sure that the multicell transport assembly is not defective.
- Call Hewlett-Packard.

Multicell Transport OQ/PV Attachment Forms

Use Fill-in Form 11 to record the hardware for which the performance verification was carried out.

Fill-in Form 11

Multicell Transport Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89075C/D or G1120A
Manufacturer	Model #

Serial MCT#	Asset #	System #

Use Fill-in Form 12 to record the performance verification results of the multicell transport.

Fill-in Form 12

Multicell Transport Test Results (89075C/D only)

	Limit		Turns Required	
Cell 1	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 2	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 3	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 4	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 5	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 6	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 7	Passed		Failed	
Turns required (only 89075C/D)	< 0.2 turns			
Cell 8 (only G1120A)	Passed		Failed	

5

Peltier Temperature-Controlled Cell Holder Performance Verification

Peltier Temperature-Controlled Cell Holder Performance Verification

This chapter contains the standard operating procedure for checking the temperature accuracy of the Peltier temperature-controlled cell holder (order number 89090A) using an external temperature measuring device.

Temperature-accuracy Test of Peltier Temperature-Controlled Cell Holder

Scope

The following procedure describes how to verify the temperature accuracy of the Peltier temperature-controlled cell holder (order number 89090A) when used with a HP 8453 UV-visible spectroscopy system.

Frequency

Follow the appropiate procedure:

- after repairing the Peltier temperature-controlled cell holder, and
- on a regular base, with recommended intervals of 6 months.

Instrumentation and Software

This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer with a Peltier temperature-controlled cell holder (order number 89090A) and general purpose software for HP ChemStation (order number G1115AA). The Peltier temperature-controlled cell holder is used in place of the single cell holder.

Requirements

To perform this SOP you will require:

- Temperature sensor support tool, part number 89090-84700, for mounting the temperature sensor in the Peltier temperature-controlled cell holder.
- Heraeus QuaT 100 handheld temperature measuring unit.
- Heraeus QuaT 340 external temperature sensor with a temperature accuracy of 0.1 K.

Procedure

1 The Peltier temperature-controlled cell holder should be properly installed as described in the manual.

2 Using the Configuration Editor of the HP ChemStation software, configure your system for use with the Peltier temperature-controlled cell holder. Save this configuration.

3 Switch on the spectrophotometer and allow it to warm up for at least 20 minutes before attempting to verify the temperature accuracy of the Peltier temperature-controlled cell holder.

4 Switch on the Peltier temperature-controlled cell holder and allow it to warm up for at least 20 minutes before attempting to verify the temperature accuracy of the Peltier temperature-controlled cell holder.

5 Open the locking lever on the Peltier temperature-controlled cell holder.

6 Insert the QuaT 340 external temperature sensor into the temperature sensor support tool, with the flat portion of the sensor facing towards the center of the tool.

7 Insert the temperature sensor support tool and temperature sensor into the cell holder. Insure that the assembly is seated firmly in the cell holder. Close the locking lever.

8 Start the HP ChemStation software.

9 Using the task panel of the HP ChemStation software, set the temperature of the Peltier temperature-controlled cell holder to 15 °C. Allow 5 minutes for the cell holder, tool, and sensor to equilibrate.

10 Record the cell temperature by monitoring the sampling task panel display.

11 Record the cell temperature displayed by the Heraeus QuaT 100 temperature measurement unit.

12 Repeat steps 9 through 11 using temperature settings of 35 and 50 °C.

Acceptance

Successful verification of the performance of the Peltier temperature-controlled cell holder has been achieved if the two recorded values at all three temperatures do not differ by more than 0.3 degrees

Celsius. Use Fill-in Form 14 on page 85 to document your results. If the test fails, refer to t"Troubleshooting" on page 83.

Troubleshooting

If good results cannot be obtained:

- Check to be sure that the Peltier temperature-controlled cell holder and the external temperature sensor are installed correctly.
- Check to be sure that Peltier temperature-controlled cell holder is activated and that the temperature has been set to the appropiate value.
- Check to be sure the temperature sensor support tool and the QuaT 340 external temperature sensor are seated and locked properly into the Peltier temperature-controlled cell holder.
- Verify that the Heraeus 100/340 handheld temperature measurement unit has recently been calibrated against a NIST-traceable standard. This information should be listed on the test certificate accompanying the unit.
- Check to be sure that the Peltier temperature-controlled cell holder is not defective.
- Call Hewlett-Packard.

Peltier Controlled Cell Holder OQ/PV Attachment Forms

Use Fill-in Form 13 to record the hardware for which the performance verification was carried out.

Fill-in Form 13

Peltier Controlled Cell Holder Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	89090A
Manufacturer	Model #

Serial #	Asset #	System #

Use Fill-in Form 14 to record the results of the Peltier temperature-controlled cell holder performance verification.

Fill-in Form 14

Peltier Temperature-controlled Cell Holder Test Results

Temperature	Maximum Deviation QuaT + Cell Holder	Measured	OK
at 15 °C	0.1 + 0.3 = 0.4°C		
at 35 °Ct	0.1 + 0.2 = 0.3°C		
at 50 °C	0.1 + 0.3 = 0.4°C		
	Passed	Failed	

6

Software Performance Verification

Software Performance Verification

This chapter contains the standard operating procedure for verification and revalidation of software components:

- General scanning software
- Advanced software
- Biochemical analysis software

The verification procedure for the dissolution testing software is described in "Dissolution Testing System Performance Verification" on page 107.

Revalidation of General Scanning Software for HP ChemStation

Scope

The following procedure describes how to validate the general scanning software on a HP ChemStation of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure if:

- you have installed or reinstalled your software,
- you have upgraded your software to a new revision, or
- you have had a software or system crash.

Instrumentation and Software

- This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer and general scanning software for HP ChemStation (order number (G1115AA).
- The CD-ROM on which the general scanning software was delivered.
- A printer connected to the system.

Procedure

1 Start Windows.

2 Start the HP ChemStation software by clicking the Instrument # Online icon of the HP ChemStations group, where # is the number of the instrument you chose in the configuration process.

3 Insert the CD-ROM in your disk drive.

4 Load the sample data from the svalid.sd data file of the support\uv\sops\data subdirectory using the Files menu.

Fixed Wavelengths Task

5 Load the fvalid.m method file from the support\uv\sops\8453 subdirectory of the supplemental disk using the Files Menu or the Load Method icon in the tool bar.

6 Print a report using the File menu or the printer icon in the tool bar.

7 Compare the report with the report in "Exhibit A: Validation Results for General Scanning Software—Fixed Wavelengths" on page 91. With the exception of operator name and report date and time they should be identical.

Spectrum/Peaks Task

8 Load the svalid.m method file from the support\uv\sops\8453 sub-directory of the supplemental disk using the Files Menu or the Load Method icon in the tool bar.

9 Print a report using the File menu or the printer icon in the toolbar.

10 Compare the report with the report in "Exhibit B: Validation Results for General Scanning Software—Spectrum/Peaks" on page 92. With the exception of operator name and report date and time they should be identical.

Ratio/Equations Task

11 Load the rvalid.m method file from the support\uv\sops\8453 sub-directory of the supplemental disk using the Files Menu or the Load Method icon in the toolbar.

12 Print a report using the File menu or the printer icon in the toolbar.

13 Compare the report with the report in "Exhibit C: Validation Results for General Scanning Software—Ratio/Equation" on page 92. With the exception of operator name and report date and time they should be identical.

Quantification Task

14 Load the qvalid.m method file from the support\uv\sops\8453 sub-directory of the supplemental disk using the Files Menu or the Load Method icon in the tool bar.

15 Print a report using the File menu or the printer icon in the tool bar.

16 Compare the report with the report in "Exhibit D: Validation Results for General Scanning Software—Quantification" on page 93. With the exception of operator name and report date and time they should be identical.

Acceptance

Check that the results in the printed reports are identical with the numbers in "Exhibit A: Validation Results for General Scanning Software—Fixed Wavelengths" on page 91 through "Exhibit D: Validation Results for General Scanning Software—Quantification" on page 93 of this SOP. Use Fill-in Form 16 on page 95 to document your results. If the test fails, refer to "Troubleshooting" on page 91.

Troubleshooting

If the results are not identical reinstall the software and repeat the revalidation procedure. If the results are still not identical, call Hewlett-Packard.

Exhibit A: Validation Results for General Scanning Software—Fixed Wavelengths

```
Method file : FVALID.M    Last update: Date 05/15/95 Time 14:30:49
Information : Fixed Wavelengths task validation method
Data File   : D:\SUPPORT\UV\SOPS\DATA\SVALID.SD
Created     : 7-Jun-93 10:36:22
Overlaid Spectra:
[Spectral Graphic]
          #  Name                Abs<300nm>
         ---------------------------------
          1  300_40                 1.00080
          2  300_40+offset          1.04980
          3  300_40+scatter         1.16190
   Report generated by : HP     Signature: ..............
-------------------------------------------------------------------
                  *** End Fixed Wavelength Report ***
-------------------------------------------------------------------
```

Exhibit B: Validation Results for General Scanning Software—Spectrum/Peaks

```
Method file : SVALID.M    Last update: Date 05/15/95 Time 14:29:00
Information : Spectrum/Peaks task validation method
Data File   : D:\SUPPORT\UV\SOPS\DATA\SVALID.SD
Created     : 7-Jun-93 10:36:22
Overlaid Spectra:
                          [Spectral Graphic]
  #  Name               Peaks(nm) d1(Abs)(AU) Valleys(nm) d1(Abs)(AU)
  ---------------------------------------------------------------------
  1  300_40               280.0      3.0390E-2    320.0  -3.0226E-2
  2  300_40+offset        280.0 3.0396E-2     320.0  -3.0307E-2
  3  300_40+scatter       280.0 2.7231E-2     320.0  -3.2040E-2
Report generated by : HP                  Signature: ................
---------------------------------------------------------------------
                       *** End Spectrum/Peak Report ***
---------------------------------------------------------------------
```

Exhibit C: Validation Results for General Scanning Software—Ratio/Equation

```
Method file : RVALID.M    Last update: Date 05/15/95  Time 14:26:45
Information : Ratio/Equation task validation method
Data File   : D:\SUPPORT\UV\SOPS\DATA\SVALID.SD
Created     : 7-Jun-93 10:36:22
Overlaid Spectra:
[Spectral Graphic]
Equation : Ratio = WL1/WL2
Where    : WL1 = Abs(252nm), WL2 = Abs(300nm), Wt = Weight, V =
Volume
# Name    Dilut. Factor  Weight  Volume Ratio    Abs<252nm> Abs<300nm>
---------------------------------------------------------------------
1 300_40        1.00000 10.00000 1.00000 5.7114E-2 5.7159E-2   1.00080
2 300_40+offset 1.00000  5.00000 1.00000 0.10123   0.10628     1.04980
3 300_40+scatter 1.00000  1.00000 1.00000 0.32712   0.38009     1.16190
Report generated by : HP                  Signature: ................
---------------------------------------------------------------------
                     *** End Ratio/Equation Report ***
---------------------------------------------------------------------
```

Exhibit D: Validation Results for General Scanning Software—Quantification

```
Method file : QVALID.M    Last update: Date 05/15/95  Time 14:24:40
Information : Quantification task validation method
Data File   : D:\SUPPORT\UV\SOPS\DATA\SVALID.SD
Created     : 7-Jun-93 10:36:22
Overlaid Sample Spectra
                          [Spectra Graphic]
Analyte name        : 300_40
Calibration equation: Conc. =    -32.48500 A * d2(Abs)
Calibrated at       : Date 05/15/95  Time 14:24:40 Operator: Tony Owen
 #  Name             Dilut. Factor        300_40(A)    d2(Abs)<300nm>
-------------------------------------------------------------------------
1  300_40                  1.00000      7.9399E-2       -2.4442E-3
2  300_40+offset           1.00000      8.0633E-2       -2.4822E-3
3  300_40+scatter          1.00000      8.0937E-2       -2.4915E-3
Report generated by : HP                Signature: ..............
-------------------------------------------------------------------------
                      *** End Quantification Report ***
-------------------------------------------------------------------------
```

General Scanning Software OQ/PV Attachment Forms

Fill-in Form 15

Software Revision Description

This chapter describes the OQ/PV tests to be performed using the HP ChemStation software revision below (running under the given operating system).

HEWLETT PACKARD	G1115AA
	Product #

Manufacturer

General scanning software

HP ChemStation software module

License #	Revision #

Operating System

Revision #

Fill-in Form 16

General Scanning Software

Type of Test	Criteria	Report OK?
Fixed Wavelength	according to Exhibit A	
Spectrum/Peaks	according to Exhibit B	
Ratio/Equation	according to Exhibit C	
Quantification	according to Exhibit D	

Passed		Failed		

Revalidation of Advanced Software for HP ChemStation

Scope

The following procedure describes how to validate the advanced software on the HP ChemStation of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure if:

- you have installed or reinstalled your software,
- you have upgraded your software to a new revision, or
- you have had a software or system crash

Instrumentation and Software

- This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer and advanced software for HP ChemStation (order number (G1116AA).
- The CD-ROM on which the advanced software was delivered.
- A printer connected to the system.

Validation Procedure

1 Start Windows.

2 Start the HP ChemStation software by clicking the Instrument # Online icon of the HP ChemStations group, where # is the number of the instrument you chose in the configuration process.

3 Insert the CD-ROM in your disk drive.

4 Select the Advanced mode using the Mode Menu (or by using the Mode combo-box on the Toolbar).

5 Load the Automation Table file avalid.a from the support\uv\sops\8453 subdirectory of the CD-ROM using **Load. Automation Table** from the File Menu.

6 Select Run Automation from the Automation Menu.

7 Wait until the Automation finished message appears on the message line and two reports have been printed.

Acceptance

Check that the results in the printed reports are identical with the numbers in "Exhibit A: Validation Results for Advanced Software—Report 1" on page 98 and "Exhibit B: Validation Results for Advanced Software—Report 2" on page 99 of this SOP. Use Fill-in Form 18 on page 100 to document your results.

Troubleshooting

If the results are not identical, reinstall the software and repeat the revalidation procedure. If the results are still not identical, call Hewlett-Packard.

Exhibit A: Validation Results for Advanced Software—Report 1

*** Results Report ***

Method file
AGVALID.M
Number of Samples 3
Operator HP

Results Summary

Sample Name	Analyte	Method	WL Result	Value	Std.Dev.	Unit
300_40	Result	Equation 1	3.6059E-3	100.00000	***	AU
		Equation 2	2.0701E-4	100.00000	***	AU
		Equation 3	3.6059E-3	100.00000	***	AU
		Equation 4	2.0747E-4	100.00000	***	
300_40+offset	Result	Equation 1	3.5815E-3	100.00000	***	AU
		Equation 2	1.9839E-4	100.00000	***	AU
		Equation 3	3.5815E-3	100.00000	***	AU
		Equation 4	1.9954E-4	100.00000	***	
300_40+scatter	Result	Equation 1	3.5945E-3	100.00000	***	AU
		Equation 2	2.0606E-4	100.00000	***	AU
		Equation 3	3.5945E-3	100.00000	***	AU
		Equation 4	2.0688E-4	100.00000	***	

*** End Results Report ***

Exhibit B: Validation Results for Advanced Software—Report 2

```
                        *** Results Report ***
Method file
    AQVALID.M
    Number of Samples       2
    Operator                HP
```

Results Summary

Sample Name	Analyte	Method	WL	Result	Value	Std.Dev.	Unit
Mixture	300_40	MCA		***	1.00030	8.2514E-5	A
		MCA 1		***	1.00030	8.2692E-5	A
		SCA 2	1.00010	1.00040		1.3524E-4	A
		SCA 3	1.00010	1.00030		7.2487E-5	A
	600_80	MCA		***	1.00020	5.8871E-5	A
		MCA 1		***	1.00030	5.9013E-5	A
Mixture+offset	300_40	MCA		***	1.00030	8.3452E-5	A
		MCA 1		***	1.00040	8.3408E-5	A
		SCA 2		0.99979	1.00010	1.3522E-4	A
		SCA 3		0.99979	1.00010	7.2477E-5	A
	600_80	MCA		***	1.00030	5.9540E-5	A
		MCA 1		***	1.00030	5.9524E-5	A

```
                    *** End Results Report ***
```

Advanced Software OQ/PV Attachment Forms

Fill-in Form 17 **Software Revision Description**

This chapter describes the OQ/PV tests to be performed using the HP ChemStation software revision below (running under the given operating system).

HEWLETT PACKARD	G1116AA
Manufacturer	Product #
Advanced software	
HP ChemStation software module	
License #	Revision #
Operating System	
Revision #	

Fill-in Form 18 **Advanced ChemStation Software**

Type of Test	Criteria	Report OK?
Report 1	According to Exhibit A	
Report 2	According to Exhibit B	

Passed		Failed		

Revalidation of Biochemical Analysis Software for HP ChemStation

Scope

The following procedure describes how to validate the biochemical analysis software on a HP ChemStation of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure if:

- you have installed or reinstalled your software,
- you have upgraded your software to a new revision, or
- you have had a software or system crash.

Instrumentation and software

- This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer and biochemical analysis software for HP ChemStation (order number (G1117AA).
- The CD-ROM on which the biochemical analysis software was delivered.
- A printer connected to the system.

Validation Procedure

1 Start Windows.

2 Start the HP ChemStation software.

3 Insert the CD-ROM in your disk drive.

Kinetics Mode

4 Select the Kinetics mode from the Mode selector box in the graphics panel (or by using the Mode Menu).

5 Select the sampling system Multicell in the instrument panel or instrument menu.

6 Load the klnvalid.kd file from the \sops\data sub-directory of the supplemental disk using Load Data from the Files menu. The results are automatically calculated.

7 Print a report using the Print Report icon on the tool bar (or using Print Report from the Files menu).

Thermal Denaturation Mode

8 Select the Thermal Denaturation mode from the Mode selector box in the graphics panel (or by using Mode Menu).

9 Load the method file tdvalid.m from the support\uv\sops\8453 subdirectory of the CD-ROM using the Load Method icon on the Toolbar (or using Load Method from the Files menu).

10 Load the tdvalid.sd file from the support\uv\sops\data subdirectory of the CD-ROM using Load Data from the Files menu. The results are automatically calculated.

11 Print a report by using the Print Report Tool on the Toolbar (or using Print Report from the Files Menu).

Acceptance

Check that the results in the printed reports are identical with those in "Exhibit A: Validation Results for Biochemical Analysis Software—Kinetics" on page 103 for kinetics and "Exhibit B: Validation Results for Biochemical Analysis Software—Thermal Denaturation" on page 103 for thermal denaruration of this SOP. Use Fill-in Form 20 on page 105 to document your results.

Troubleshooting

If the results are not identical reinstall the software and repeat the revalidation procedure. If the results are still not identical, call Hewlett-Packard.

Exhibit A: Validation Results for Biochemical Analysis Software—Kinetics

```
                          *** Results Report ***
Method file : <untitled>
Information : Default Method of Kinetics Mode
Data File   : D:SUPPORT\UV\SOPS\DATA\KINVALID.KD
Created     : 1/9/95  16:31:54
Used Wavelength       : 400 nm
Background correction : subtract average over range from 550 nm to
            570 nm
Run Time    :    2500.0 s
Start Time  :       0.0 s
Cycle Time  :     125.0 s
Time Trace  :[Graphic Time traces]
Used cell layout:                S S S - - - -
Rate Calculation Type    :    First order
Calculation Time Range   :    0 s to Run Time
Cell #   Name            Factor     Rate(1/s)      Std.Dev     Comment
-------------------------------------------------------------------------
1        Trace 1          1.0000    1.2743E-3     2.0051E-5
2        Trace 2          1.0000    1.2399E-3     5.8056E-6
3        Trace 3          1.0000    1.2529E-3     5.1011E-6
Report generated by : HP                  Signature: ..............
-------------------------------------------------------------------------
                 *** End Kinetics Results Report ***
-------------------------------------------------------------------------
```

Exhibit B: Validation Results for Biochemical Analysis Software—Thermal Denaturation

```
-------------------------------------------------------------------------
                          *** Results Report ***
Sample Information
Operator                : cg
Sample Name             : DNA
Solvent                 : Citrate buffer
Molarity                : 0.0015 mol/l
DNA Length              : ---
File                    : TDVALID.TD     Created : 1/24/95 9:45:06
Comment                 : 0.01 ml Sample diluted to 1ml
Acquisition Parameters
Instrument              : OFFLINE
Acquisition range       : 190 to 1100 nm
Integration Time        : 0.5 s
Idle Temperature        : 45.0 °C
```

```
Used Sensor              : External Sensor
Ramping Speed            : Fast
Stirrer Status           : Off
Autosave File            : dna.td
Temperature Ramp
No.        Start          Stop     Increment     Hold Time
-------------------------------------------------------------
 1        45.0 °C       62.0 °C       1.0 °C      1.00 min
 2        62.0 °C       77.0 °C       0.5 °C      1.00 min
 3        77.0 °C       85.0 °C       1.0 °C      0.50 min
 4        85.0 °C       77.0 °C      -1.0 °C      0.50 min
 5        77.0 °C       62.0 °C      -0.5 °C      1.00 min
 6        62.0 °C       45.0 °C      -1.0 °C      1.00 min
Calculation Parameters
Used Wavelength          : 260 nm
Background Correction    : Single reference wavelength at 320 nm
Calculation Method       : Derivative
Filterlength             : 55
Sensitivity              : ---
%GC                      : 2.44*(TM-81.5-16.66*log(M))
Volume Correction        : ---
Whole Temperature Absorbance Trace
          [Graphic Whole Temperature Absorbance Trace]
Interpolated Heating Trace 1
          [Graphic Interpolated Heating Trace 1]
Derivative of Heating Trace 1
          [Graphic First Derivative of Heating Trace 1]
Results of Heating Trace  1
Operator                 : HP
Sample Name              : DNA
SolventName              : Citrate buffer
Molarity (M)             : 0.0015 mol/l
DNA Length (K)           : ---
Date                     : 1/24/95
Time                     : 9:45:06
Calculation Range        : From 44.3 °C to 82.9 °C
Delta Absorbance         : 42.25 %
Delta Temperature        : From 50.2 °C to 73.3 °C
Melting Temperature (1): 58.90 °C
%GC (1)                  : 59.65 %
Report generated by :    HP              Signature: .....
-------------------------------------------------------------
                *** End Thermal Denat. Results ***
-------------------------------------------------------------
```

Biochemical Analysis Software OQ/PV Attachment Forms

Fill-In Form 19

Software Revision Description

This chapter describes the OQ/PV tests to be performed using the HP ChemStation software revision below (running under the given operating system).

HEWLETT PACKARD	G1117AA
	Product #

Manufacturer

Biochemical analysis software

HP ChemStation software module

License #	Revision #

Operating System

Revision #

Fill-In Form 20

Biochemical Analysis Software

Type of Test	Criteria	Report OK?	
Kinetics	According to Exhibit A		
Thermal	According to Exhibit B		

Passed		Failed		

7

Dissolution Testing System Performance Verification

Dissolution Testing System Performance Verification

This chapter has the following sections:

Performance Verification of Offline Sampling Systems for Dissolution Testing

This section guides you briefly through the procedure of how to do a performance verification on the different offline sampling systems for dissolution testing.

Performance Verification of Offline Sampling Systems

Scope

The following procedure describes how to carry out the verification procedure on an offline sampling system of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure:

- when you first install your dissolution testing sampling system,
- on a regular base at intervals of a minimum of 6 months, and
- if you change or exchange any software or hardware of your dissolution testing sampling system.

Instrumentation and Software

This SOP applies to a HP 8453 UV-visible dissolution testing system comprising a HP 8453 spectrophotometer, a sipper/autosampler sampling system (order number 89068D with HP 89072A, or 89068D with Gilson 221 or Gilson 222), general purpose software for HP ChemStation (order number G1115AA) and dissolution testing software for HP ChemStation (order number G1118AA).

Requirements

Different tests have different environmental temperature conditions according to the various pharmacopeias. An environmental temperature range between 20–21 °C meets all specifications on which the test in this manual are based.

To perform this SOP you will require:

OQ/PV Standards (1), (2)

- OQ/PV standards (1) and (2), part numbers 5063-6503 and 5063-6521

respectively,

50 ml distilled water and 50 ml caffeine/water sample

- approximately 50-ml of distilled water, HPLC grade,
- approximately 50-ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml). Another sample may be substituted which absorbs at the analytical wavelength of your specific analysis. This sample should have an absorbance of approximately 1 AU.

Preparing the Offline Sampling Systems for the Test

The section describes the different steps for preparing the offline sampling systems for the performance verification.

Preparing the Spectrophotometer and the Sipper/Autosampler System

NOTE

The verification of the individual hardware components has to be carried out during an installation of one of the offline sampling system, or a change/repair of one of the hardware components.

NOTE

For an existing and unchanged system, begin the OQ/PV with Chapter 2 "Spectrophotometer Performance Verification".

Performance verification of the spectrophotometer and the sipper/sampler system

Prior to the performance verification of the sipper/autosampler system, the performance of the HP 8453 spectrophotometer and the sipper/autosampler system have to be verified.

Refer to Chapter 2 "Spectrophotometer Performance Verification" for doing a spectrophotometer performance verification.

Refer to the Chapter 3 "Sipper/Autosampler Performance Verification" for doing a flow test on the sipper/autosampler system.

NOTE

These procedures do not verify the performance of any additional hardware used with the off-line sampling systems.

Preparing the Offline Sampling System

Renew pump tubing

1 Exchange the pump tubing against a new one (part number 5041-2166 for pump tubing 2.06 mm id).

Cleaning Flow Cells and Tubings of Offline Dissolution Testing Systems

Apply the following steps every time before you do a verification to make sure your flow cell is clean and does not trap any air bubbles.

Draw 50 ml of cell passivating and cleaning fluid through flow cell.

1 Prepare minimum 50 ml a solution of 5% cell passivating and cleaning fluid (part number 5062-8529) in water for each cell.

2 Place the tubing/needle into the beaker with the 5% cell passivating and cleaning fluid and pump the 50 ml of cell passivating and cleaning fluid through each flow cell.

NOTE You may observe a high amount of air coming through the flow cell, because the solution contains a detergent.

Draw 100 ml of water through flow cell.

3 Place the tubing/needle into a beaker with distilled water and draw 100 ml of water through the flow cell.

NOTE Before proceeding with the performance verification, the tubing has to be filled with distilled water.

Performance Verification Procedures for Offline Sampling Systems

Procedure I

1 If the HP 8453 spectrophotometer is not already turned on, switch the spectrophotometer on.

2 Start the HP ChemStation.

3 Select Verification and Diagnostics from the Mode menu or tool bar.

Select dissolution test and sipper/autosampler system

4 Select Dissolution Test in the Task drop down box of the graphical user interface or by using the Task menu.

5 In the Sampling system drop down box choose the offline system in use (Sipper, Autosampler 89072A, Autosampler Gilson 221/222).

6 Press the Setup button and check the path length setting. Choose *Parameter* and check the settings of the pump. The default settings for the sipper system and the HP 89072A autosampler are:

Set pump parameters for sipper system and autosampler

Pump time: 20 s
Pump Direction: CW
Wait time: 3 s
Reverse time: 0 s
Sample Return: 0%
Wash Time: 0 s
Air Segment: 0 s

The default settings for the Gilson 221/222 autosamplers are:

Pump time: 20 s
Pump Direction: CW
Wait time: 3 s
Sample Return: 0%
Air Segment: 0 s

NOTE For this test, the same wavelength as specified in the "Sipper/Autosampler Performance Verification" on page 63, is used.

Put probe in distilled water

7 Put the tubing/needle into a beaker filled with 50 ml of distilled water.

8 Put outlet of the pump into a volumetric flask to measure the amount of water pumped through the flow cell.

9 In the graphical user interface of the sampling system press the Flow Rate-button. Set the parameters of the flow rate test by entering the following:

Set flow rate values

Duration: 2 min
Direction: CW
Limits: 6 ml/min, +/- 10%

Start flow rate measurement and measure the amount of liquid pumped through the cell

10 Start the flow rate test by pressing the OK-button.

11 Measure the volume you collected in the beaker and enter it, in units of ml, into the edit box coming up at the end of the flow rate test.

12 Check in the test result table coming up, if the test is passed.

Acceptance I

The calculated flow rate must be within the limits specified. This is indicated by passed in the *result* column of the test result table. Use Fill-in Form 23 on page 119 to document your results. If the test fails, refer to "Troubleshooting I" on page 116.

Troubleshooting I

- Check if cells and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as

described under .

- Check if the pump tubing, has been renewed and that the tubing clamps are closed.
- Depending on whether the pumped volumes are to high or to low, lower or raise the pump speed of the peristaltic pump 1FS.

Offline Sampling Systems OQ/PV Attachment Forms

Use Fill-in Form 21 and Fill-in Form 22 to record the hardware for which the performance verification was carried out.

Fill-in Form 21

Peristaltic Pump 1FS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	G1103-60004 / G1103-60006
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 22

Autosampler Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89072A or Gilson 221/222
Manufacturer	Model #

Autosampler Serial #	Asset #	System #

Use Fill-in Form 23 and to record the results of the offline system performance verification.

Fill-in Form 23

Sipper / Autosampler Flow Rate Test Results

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate				
	Passed		Failed	

Performance Verification of Online Sampling Systems for Dissolution Testing

This section guides you through the procedure of how to do the following performance verification tests:

- "Performance Verification of Multicell Transport Sampling System" on page 121,
- "Performance Verification of Valve Sampling System" on page 137,
- "Performance Verification of Multibath Sampling System" on page 152.

Performance Verification of Multicell Transport Sampling System

Scope

The following procedure describes how to carry out the verification procedure on a multicell transport sampling system (order number G1127A) of the HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure:

- when you first install your dissolution testing sampling system,
- on a regular base at intervals of a minimum of 6 months,
- if you change or exchange any software or hardware of your dissolution testing sampling system.

Instrumentation and Software

This SOP applies to a HP 8453 UV-visible dissolution testing system comprising a HP 8453 spectrophotometer, the multicell transport sampling system (order number G1127A), general purpose software for HP ChemStation (order number G1115AA) and dissolution testing for HP ChemStation (order number G1118AA).

Requirements

Different tests have different environmental temperature conditions according to the various pharmacopeias. An environmental temperature range between 20–21 °C meets all specifications on which the test in this manual are based.

To perform this SOP you will require:

OQ/PV Standards (1), (2)

- OQ/PV Standards (1) and (2), part numbers 5063-6503 and 5063-6521 respectively.

MCT adjustment tool

- MCT adjustment tool part number 89075-23800.

600 ml distilled water and 200 ml caffeine/water sample

- 3 beakers filled with approximately 200 ml of distilled water, HPLC grade.
- 2 beakers filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml). Another sample may be substituted which absorbs at the analytical wavelength of your specific analysis. This sample should have an absorbance of approximately 1 AU.

Preparing the Multicell Transport Sampling System for the Test

The chapter describes the different steps for preparing the multicell transport sampling system for the performance verification.

Preparing the Multicell Dissolution Sampling System

Disconnect bath and remove probes from bath

1 To avoid any problems coming from the dissolution bath itself, unplug the communication cable connecting the bath with the PC.

2 Remove the probes from the dissolution bath.

NOTE

The performance verification test is best applicable when HP probes (dissolution probe kit, part number 5062-8537) are in use. If possible, exchange any non-HP probes against those coming with the dissolution testing sampling system. In case non-HP probes are used for the verification, adjust the pump times according to your hardware.

Renew probe filters

3 Replace all probe filters (part number 5181-1246) with new ones.

Renew pump tubings

4 Replace all pump tubings with new ones (part number 5041-2166 for pump tubing 2.06 mm id).

Cleaning Flow Cells and Tubings of Multicell Dissolution Testing System

Apply the following steps every time before you do a verification to make sure your flow cells is clean and does not trap any air bubbles.

Draw 50 ml of cell passivating and cleaning fluid through flow cell

1 Prepare a minimum of 50 ml of a solution of 5% cell passivating and cleaning fluid (part number 5062-8529) in water for each cell.

2 Place all probes (with the filter) into the beaker with 400 ml of the 5% cell passivating and cleaning fluid and pump 50 ml of cell passivating and cleaning fluid through each flow cell.

NOTE You may observe a high amount of air coming through the flow cell, because the solution contains a detergent.

Draw 100 ml of water through each flow cell

3 Place all probes (with the filter) into a beaker with 800 ml distilled water and draw 100 ml of water through each flow cell.

NOTE Before proceeding with the performance verification, every channel has to be filled with distilled water.

Preparing the Spectrophotometer, Multicell Transport and Peristaltic Pump 8VS

NOTE The verification of the individual hardware components has to be carried out during an installation of a multicell transport sampling system, or a change/repair of one of the hardware components, or at least every 6 months.

NOTE For an existing and unchanged system, which is checked out on a daily or weekly basis, begin the OQ/PV with "Procedure I" on page 125.

Performance Verification of the Spectrophotometer and the Multicell Transport

Prior to the performance verification of the multicell transport sampling system, the performance of the HP 8453 spectrophotometer, the multicell transport and the peristaltic pump 8VS have to be verified.

Refer to Chapter 2 "Spectrophotometer Performance Verification" for doing a spectrophotometer performance verification.

Refer to the Chapter 6 "Software Performance Verification" for doing a multicell transport performance verification.

Procedure I

1 The peristaltic pump 8VS and multicell transport should be properly installed as described in the manual.

2 Switch on the spectrophotometer and allow it to warm up for at least 20 minutes before making any measurements.

3 Start the HP ChemStation software.

4 Select the Verification and Diag*nostics* mode using the tool bar Mode section (or use the Mode menu).

5 Select the Flow Test task in the graphical user interface (or use the Task menu).

6 Set the wavelength for your test sample and the desired level of purity using the Setup button in the Flow Test window of the graphical user interface. The default values are the following:

Wavelength: 273 nm
Percent: 99.5%

7 Select Online Multicell Transport (7 cells) or Online Multicell Transport (8 cells) in the sampling system drop down box.

8 Set the parameters for operation of the pump by selecting Setup and Parameter in the graphical interface (or use the Instrument menu, Setup Sampling System. Parameter) and entering the following:

Pump time: 40 s
Pump Direction: CW
Wait time: 3 s
Sample Return: 0%

Wash time: 0 s
Air Segment: 0 s

9 Select the Cell you want to test in the Flow Test window of the graphical user interface.

10 Put the end of the probe of the channel you want to test into the reservoir containing the solvent.

11 Switch on the pump by clicking on the pump icon in the instrument panel of the graphical user interface and flush the system for approximately twice as long as the pump time you set. Switch off the pump by clicking on the pump icon.

12 Make a Blank measurement.

13 Put the end of the probe of the channel you want to test into the reservoir containing the test sample and start the test using the Run button in the Flow Test window. The test will take 50% longer than the pump time you have entered. You should get a graphic display of the absorbance versus time trace and a display of the pump time in seconds required for every channel to achieve the entered % purity.

14 If the estimated pump time is more than 20% different from your initial pump time, enter the estimated pump time under step 8 and repeat steps 10 through 13. Repeat until you get consecutive results within 10% or 1 second, whichever is greater.

15 Repeat steps 9 through 14 for every cell in use.

Acceptance I

Optimal pump time has been achieved, when the flow test indicates that you get consecutive results within 10% or 1 second, whichever is greater. Use Fill-in Form 26 on page 134 to document your results. If the test fails, refer to "Troubleshooting I" on page 126.

Troubleshooting I

If good results cannot be obtained:

- Check if there is a bubble in the flow cell, if yes, gentle tapping of the cell will help to dislodge it.

- Check the flow rate of your pump and replace the pump tubing if necessary.
- Check for air being sucked in at one of the fittings.

Performance Verification Procedures for Multicell Transport Sampling System

Procedure II

1 If the HP 8453 spectrophotometer is not already turned on, switch the spectrophotometer on.

2 Start the HP ChemStation.

3 Select Verification and Diagnostics from the Mode menu or tool bar.

Select Dissolution Test and Multicell as sampling system

4 Select Dissolution Test in the Task drop down box of the graphical user interface or by using the Task menu.

5 In the *Sampling* system drop down box choose Online Multicell (7-cell) or Online Multicell (8-cell), depending on your multicell transport.

NOTE For this test, the same wavelength is used as specified in Chapter 4 "Multicell Transport Performance Verification".

Put probe in distilled water

6 Put probe 1 into a beaker filled with 200 ml of distilled water and select the cell in the graphical user interface accordingly.

7 Put return of probe 1 into a volumetric flask to measure the amount of water pumped through the flow cell.

8 In the graphical user interface of the sampling system press the *Flow Rate*-button. Set the parameters of the flow rate test by entering the following:

Set flow rate values

Duration: 2 min
Direction: CW
Limits: 6 ml/min, +/- 10%

Start flow rate measurement and measure the amount of liquid pumped through each cell

9 Start the flow rate test by pressing the OK-button.

10 Measure the volume you collected in the volumetric flask and enter it, in units of ml, into the edit box coming up at the end of the flow rate test.

11 Check in the test result table coming up, if the test is passed.

12 Repeat steps 6 through 11 for each cell of the multicell transport.

Acceptance II

The calculated flow rate must be within the limits specified. This is indicated by passed in the *result* column of the test result table. Use Fill-in Form 27 on page 135 to document your results. If the test fails, refer to "Troubleshooting II" on page 129.

Troubleshooting II

- Check if cells and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as described in "Cleaning Flow Cells and Tubings of Multicell Dissolution Testing System" on page 123.
- Check if all pump tubings, filter tips have been renewed and that the tubing clamps are closed.
- Depending on whether the pumped volumes are to high or to low, lower or raise the pump speed.

Procedure III

1 Press the Cross Contamination-button in the graphical user interface. Set the parameters of the cross contamination test by entering the following:

NOTE 273 nm is set-up as default wavelength when the caffeine sample (part number 5063-6524) is used for the test and the path length of the cell 10 mm. For 1 mm path length cells use 205 nm as default wavelength.

Wavelength: 273 nm
Minimum limit: 99.5%
Maximum limit: 0.5%

Mark all cell positions you want to verify.

2 Start the cross-contamination test by pressing the OK-button.

Put all probes into distilled water

3 Put all probes into a beaker filled with approximately 200 ml of distilled water (HPLC grade), or your Blank medium, and press OK.

4 Put all probes into a beaker filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml), or your Sample medium, and press *OK*.

5 Repeat steps 3 and 4 as prompted by the software.

Acceptance III

For each of the cells the table should show *passed* in the result column. Use Fill-in Form 28 on page 136 to document your results. If one of the cells failed the test, refer to "Troubleshooting III" on page 130.

Troubleshooting III

- Check if cells and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as described in "Cleaning Flow Cells and Tubings of Multicell Dissolution Testing System" on page 123.

- Check if all pump tubings, filter tips have been renewed and that the tubing clamps are closed.
- Go back to Flow Task test and check the pump time. Depending on whether the pump time is to high or to low, lower or raise the pump time.

Multicell Transport Sampling System OQ/PV Attachment Forms

Use Fill-in Form 24 and Fill-in Form 25 to record the hardware for which the performance verification was carried out.

Fill-in Form 24

Peristaltic Pump 8VS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89092A
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 25

Multicell Transport Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	HP 89075C/D or G1120A
Manufacturer	Model #

MCT Serial #	Asset #	System #

Use Fill-in Form 26 through Fill-in Form 28 to record the performance verification results of the multicell transport.

Fill-in Form 26

Peristaltic Pump 8VS / Multicell Transport Flow Test Results (Procedure I)

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time Channel 1				
	Passed		Failed	
Pump Time Channel 2				
	Passed		Failed	
Pump Time Channel 3				
	Passed		Failed	
Pump Time Channel 4				
	Passed		Failed	
Pump Time Channel 5				
	Passed		Failed	
Pump Time Channel 6				
	Passed		Failed	
Pump Time Channel 7				
	Passed		Failed	
Pump Time Channel 8				
	Passed		Failed	

Fill-in Form 27

Peristaltic Pump 8VS / Multicell Transport Flow Rate Test Results (Procedure II)

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate Channel 1				
	Passed		Failed	
Flow Rate Channel 2				
	Passed		Failed	
Flow Rate Channel 3				
	Passed		Failed	
Flow Rate Channel 4				
	Passed		Failed	
Flow Rate Channel 5				
	Passed		Failed	
Flow Rate Channel 6				
	Passed		Failed	
Flow Rate Channel 7				
	Passed		Failed	
Flow Rate Channel 8				
	Passed		Failed	

Fill-In Form 28

Peristaltic Pump 8VS / Multicell Transport
Cross-Contamination Test Result (Procedure III)

	Min. for 100% Absorbance	Measured	Max. for 0% Absorbance	Measured
Relative Abs. % Channel 1				
	Passed		Failed	
Relative Abs. % Channel 2				
	Passed		Failed	
Relative Abs. % Channel 3				
	Passed		Failed	
Relative Abs. % Channel 4				
	Passed		Failed	
Relative Abs. % Channel 5				
	Passed		Failed	
Relative Abs. % Channel 6				
	Passed		Failed	
Relative Abs. % Channel 7				
	Passed		Failed	
Relative Abs. % Channel 8				
	Passed		Failed	

Performance Verification of Valve Sampling System

Scope

The following procedure describes how to carry out the verification procedure on a valve sampling system (order number G1128A) of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure:

- when you first install your dissolution testing sampling system,
- on a regular base at intervals of a minimum of 6 months,
- if you change or exchange any software or hardware of your dissolution testing sampling system.

Instrumentation and Software

This SOP applies to HP 8453 UV-visible dissolution testing system comprising a HP 8453 spectrophotometer, the valve sampling system (order number G1128A), general purpose software for HP ChemStation (order number G1115AA) and dissolution testing software for HP ChemStation (order number G1118AA).

Requirements

Different tests have different environmental temperature conditions according to the various pharmacopeias. An environmental temperature range between 20–21 °C meets all specifications on which the test in this manual are based.

To perform this SOP you will require:

OQ/PV Standards (1), (2)

- OQ-PV Standards (1) and (2), part numbers 5063-6503 and 5063-6521 respectively.

600 ml distilled water and 200 ml caffeine/water sample

- 3 beakers filled with approximately 200 ml of distilled water, HPLC grade,
- 2 beakers filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml). Another sample may be substituted which absorbs at the analytical wavelength of your specific analysis. This sample should have an absorbance of approximately 1 AU.

Preparing the Valve Sampling System for the Test

The section describes the different steps for preparing the valve sampling system for performance verification.

Preparing the Valve Dissolution Testing System

Disconnect bath and remove probes from bath

1 To avoid any problems coming from the dissolution bath itself, unplug the communication cable connecting the bath with the PC.

2 Remove the probes from the dissolution bath.

NOTE The performance verification test is best applicable when HP probes (dissolution probe kit, part number 5062-8537) are in use. If possible, exchange any non-HP probes against those coming with the dissolution testing sampling system. In case non-HP probes are used for the verification, adjust the pump times according to your hardware.

Renew probe filters

3 Exchange all probe filters (part number 5181-1246) against new ones.

Renew pump tubing

4 Exchange the pump tubing against a new one (pump tubing 2.06 mm id, part number 5041-2166).

Cleaning Flow Cells and Tubings of Valve Dissolution Testing System

Apply the following steps every time before you do a verification to make sure your flow cell is clean and does not trap any air bubbles.

Draw 50 ml of cell passivating and cleaning fluid through flow cell

1 Prepare a minimum of 50 ml of a solution of 5% cell passivating and cleaning fluid (part number 5062-8529) in water for each cell.

2 Place the probe according to the selected channel (with the filter) into the beaker with the 5% cell passivating and cleaning fluid and pump the 50 ml of cell passivating and cleaning fluid through each flow cell.

NOTE You may observe a high amount of air coming through the flow cell, because the solution contains a detergent.

3 Repeat step 1 and 2 for each of the channels.

Draw 100 ml of water through flow cell

4 Place the probe according to the selected channel (with the filter) into a beaker with distilled water and draw 100 ml of water through each flow cell.

5 Repeat step 4 for each of the channels.

NOTE Before proceeding with the performance verification, every channel has to be filled with distilled water.

Preparing the Spectrophotometer, Eight-port Valve and Peristaltic Pump 1VS

NOTE The verification of the individual hardware components has to be carried out during an installation of a valve sampling system, or a change/repair of one of the hardware components, or at least every 6 months.

NOTE For a existing and unchanged system, which is checked on a daily or weekly basis, begin the OQ/PV with "Procedure I" on page 141.

Performance Verification of the Spectrophotometer and the Valve

Prior to the performance verification of the valve sampling system, the performance of the HP 8453 spectrophotometer, eight-port valve and peristaltic pump 1VS have to be verified.

Refer to Chapter 2 "Spectrophotometer Performance Verification" for doing a spectrophotometer performance verification.

Procedure I

1 The eight-port valve and peristaltic pump 1VS should be properly installed as described in the manual.

2 Switch on the HP 8453 spectrophotometer and allow it to warm up for at least 20 minutes before making any measurements.

3 Start the HP ChemStation software.

4 Select the Verification and Diagnostics mode using the tool bar Mode section (or use the Mode menu).

5 Select the Flow Test task in the graphical user interface (or use the Task menu).

6 Set the wavelength for your test sample and the desired level of purity using the *Setup* button in the Flow Test window of the graphical user interface. The default values are the following:

Wavelength: 273 nm
Percent: 99.5%

7 Select Online Valve System in the sampling system drop down box.

8 Set the parameters for operation of the pump by selecting Setup and Parameter in the graphical interface (or use the Instrument menu, Setup Sampling System. Parameter) and entering the following:

Pump time: 47 s
(fixed, depending on method loaded previously in dissolution module)
Pump Direction: CW

NOTE

The pump time is fixed to 47 seconds whenever a cycle time of 7.5 minutes has been chosen. For a cycle time of 5 minutes, the pump time is 29 seconds, whereas for a cycle time of 10 minutes you will get a pump time of 58 seconds.

9 Select the Channel you want to test in the Flow Test window of the graphical user interface.

10 Put the end of the probe of the channel you want to test into the reservoir containing the solvent.

11 Switch on the pump by clicking on the pump icon in the instrument panel of the graphical user interface and flush the system for approximately twice as long as the pump time you set. Switch off the pump by clicking on the pump icon.

12 Make a Blank measurement.

13 Put the end of the probe of the channel you want to test into the reservoir containing the test sample and start the test using the Run button in the Flow Test window. The test will take 50% longer than the pump time you have entered. You should get a graphic display of the absorbance versus time trace and a display of the pump time in seconds required for every channel to achieve the entered % purity.

14 Repeat the test for each of the channels.

Acceptance I

Optimal pump time has been achieved, when the flow test indicates that you get consecutive results within 10% or 1 second, whichever is greater. Use Fill-in Form 31 on page 149 to document your results. If the test fails, refer to "Troubleshooting I" on page 142.

Troubleshooting I

If good results cannot be obtained:

- Check if there is a bubble in the flow cell, if yes, gentle tapping of the cell will help to dislodge it.
- Check the flow rate of your pump and replace the pump tubing if necessary.
- Check for air being sucked in at one of the fittings.
- Check rate of your pump and replace the pump tubing if necessary.

Performance Verification Procedures for Valve Sampling System

Procedure II

1 If the HP 8453 spectrophotometer is not already turned on, switch the spectrophotometer on.

2 Start the HP ChemStation.

3 Select Verification and Diagnostics from the Mode menu or tool bar.

Select dissolution test and multicell as sampling system

4 Select Dissolution Test in the Task drop down box of the graphical user interface or by using the Task menu.

5 In the Sampling system drop down box choose Online Valve System.

6 Press the Setup button and check the settings of the pump. The default settings are:

Set pump parameters

Pump time: 47 s
(fixed, depending on method loaded previously in dissolution mode)
Pump Direction: CW
Wait time: 3 s
Reverse time: 0 s

NOTE

The pump time is fixed to 47 seconds whenever a cycle time of 7.5 minutes has been chosen. For a cycle time of 5 minutes, the pump time is 29 seconds, whereas for a cycle time of 10 minutes you will get a pump time of 58 seconds.

Put probe in distilled water

7 Put probe 1 into a beaker filled with 200 ml of distilled water and select the channel in the graphical user interface accordingly.

8 Put return of probe 1 into a beaker to measure the amount of water pumped through the flow cell.

9 In the graphical user interface of the sampling system press the Flow Rate-button. Set the parameters of the flow rate test by entering the following:

Set flow rate values

Duration: 2 min
Direction: CW
Limits: 6 ml/min, +/- 10%

Start flow rate measurement and measure the amount of liquid pumped through each channel

10 Start the flow rate test by pressing the OK-button.

11 Measure the volume you collected in the beaker and enter it, in units of ml, into the edit box coming up at the end of the flow rate test.

12 Check in the test result table coming up, if the test is passed.

13 Repeat steps 7 through 12 for each channel of the valve.

Acceptance II

The calculated flow rate must be within the limits specified. This is indicated by passed in the *result* column of the test result table. If the test fails, refer to "Troubleshooting II" on page 144.

Troubleshooting II

- Check if cells and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as described in "Cleaning Flow Cells and Tubings of Valve Dissolution Testing System" on page 139.
- Check if the pump tubing, filter tip have been renewed and that the tubing clamp is closed.
- Depending on whether the pumped volumes are to high or to low, lower or raise the pump speed.

Procedure III

1 Press the Cross Contamination-button in the graphical user interface. Set the parameters of the cross contamination test by entering the following:

Wavelength: 273 nm
Minimum limit: 99.5%
Maximum limit: 0.5%

NOTE 273 nm is setup as default wavelength when the caffeine sample (part number 5063-6524) is used for the test and the path length of the cell 10 mm. For 1 mm path length cells use 205 nm as default wavelength.

2 Start the cross contamination test by pressing the OK-button.

3 Put probe 1 into a beaker filled with distilled water or Blank medium. Press the OK-button.

4 Put probe 1 into a beaker filled with a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml) or the Sample medium. Press the OK-button.

NOTE Ensure having chosen a wavelength where your sample shows an absorbance of approximately 1 AU.

Put probes 1,3,5,7 into the blank

5 Put probes 1,3,5,7 into a beaker filled with approximately 200 ml of distilled water (HPLC grade), or your Blank medium.

Put probes 2,4,6,8 into the sample

6 Put probes 2,4,6,8 into a beaker filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml), or your Sample medium, and press *OK*.

Put probes 2,4,6,8 into the blank

7 Put probes 2, 4, 6 and 8 into a beaker filled with approximately 200 ml of distilled water (HPLC grade), or your blank medium.

Put probes 1,3,5,7 into the sample

8 Put probes 1, 3, 5 and 7 into a beaker filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml), or your Sample medium, and press OK.

Acceptance III

For each of the channels the table should show *passed* in the result column. If one of the channels failed the test, refer to "Troubleshooting III" on page 146.

Troubleshooting III

- Check if the cell and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as described under .
- Check if the pump tubing, filter tip have been renewed and that the tubing clamp is closed.
- Go back to Flow Task test and check the pump time. Depending on whether the pump time is to high or to low, lower or raise the pump time.
- Check if the valve fittings tightened. To ensure proper installation of the fittings, tighten them gently with the wrench shipped with the valve sampling system.

Valve Sampling System OQ/PV Attachment Forms

Use Fill-in Form 29 and Fill-in Form 30 to record the hardware for which the performance verification was carried out.

Fill-in Form 29

Peristaltic Pump 1VS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	G1103-60004 / G1103-60006
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 30

Valve Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89079A
Manufacturer	Model #

Valve Serial #	Asset #	System #

Use Fill-in Form 31 through Fill-in Form 33 to record the performance verification results of the valve system.

Fill-in Form 31

Eight-port Valve / Peristaltic Pump 1VS Flow Test Results (Procedure I)

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time Channel 1				
	Passed		Failed	
Pump Time Channel 2				
	Passed		Failed	
Pump Time Channel 3				
	Passed		Failed	
Pump Time Channel 4				
	Passed		Failed	
Pump Time Channel 5				
	Passed		Failed	
Pump Time Channel 6				
	Passed		Failed	
Pump Time Channel 7				
	Passed		Failed	
Pump Time Channel 8				
	Passed		Failed	

Fill-in Form 32

Eight-port Valve / Peristaltic Pump 1VS Flow Rate Test Results (Procedure II)

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate Channel 1				
	Passed		Failed	
Flow Rate Channel 2				
	Passed		Failed	
Flow Rate Channel 3				
	Passed		Failed	
Flow Rate Channel 4				
	Passed		Failed	
Flow Rate Channel 5				
	Passed		Failed	
Flow rate Channel 6				
	Passed		Failed	
Flow Rate Channel 7				
	Passed		Failed	
Flow Rate Channel 8				
	Passed		Failed	

Fill-in Form 33

Eight-port Valve / Peristaltic Pump 1VS
Cross-Contamination Test Results Procedure III)

	Min. for 100% Absorbance	Measured	Max. for 0% Absorbance	Measured
Relative Abs. % Channel 1				
	Passed		Failed	
Relative Abs. % Channel 2				
	Passed		Failed	
Relative Abs. % Channel 3				
	Passed		Failed	
Relative Abs. % Channel 4				
	Passed		Failed	
Relative Abs. % Channel 5				
	Passed		Failed	
Relative Abs. % Channel 6				
	Passed		Failed	
Relative Abs. % Channel 7				
	Passed		Failed	
Relative Abs. % Channel 8				
	Passed		Failed	

Performance Verification of Multibath Sampling System

Scope

The following procedure describes how to carry out the verification procedure on the multibath sampling system (order number G1129A or G1130A) of the HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure:

- when you first install your dissolution testing sampling system,
- on a regular base at intervals of a minimum of 6 months,
- if you change or exchange any soft- or hardware of your dissolution testing sampling System.

Instrumentation and Software

This SOP applies to a HP 8453 UV-visible dissolution testing system comprising a HP 8453 spectrophotometer, a multibath sampling system (order number G1129A or G1130A), general purpose software for HP ChemStation (order number G1115AA) and dissolution testing software for HP ChemStation (order number G1118AA).

Requirements

Different tests have different environmental temperature conditions according to the various pharmacopeias. An environmental temperature range between 20–21 °C meets all specifications on which the test in this manual are based.

To perform this SOP you will require:

OQ/PV Standards (1), (2)

- OQ/PV Standards (1) and (2), part numbers 5063-6503 and 5063-6521 respectively.

MCT adjustment tool

- MCT adjustment tool part number 89075-23800

600 ml distilled water and 200 ml caffeine/water sample for each bath

- 3 beakers filled with approximately 200 ml of distilled water, HPLC grade, for each bath
- 2 beakers filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml) for each bath. Another sample may be substituted which absorbs at the analytical wavelength of your specific analysis. This sample should have an absorbance of approximately 1 AU.

Preparing the Multicell Transport of the Multibath Sampling System for the Test

The chapter describes the different steps for preparing the multibath sampling system (order number G1129A or G1130A) for the performance verification.

Preparing the Multibath Dissolution Testing System

Disconnect bath and remove probes from bath

1 To avoid any problems coming from the dissolution bath itself, unplug the communication cable connecting the bath with the PC.

2 Remove the probes from the dissolution baths.

NOTE

The performance verification test is only applicable when HP probes (dissolution probe kit, part number 5062-8537) are in use. Exchange any non-HP probes against those coming with the dissolution testing sampling system.

Renew probe filters

3 Replace all probe filters (part number 5181-1246) with new ones.

Renew pump tubings

4 Replace all pump tubings with new ones (pump tubing 2.06 mm id, part number 5041-2166).

Cleaning Flow Cells and Tubings of multibath Dissolution Testing System

Apply the following steps every time before you do a verification to make sure your flow cell is clean and does not trap any air bubbles.

Draw 50 ml of cell passivating and cleaning fluid through flow cell

1 Prepare a minimum of 50 ml of a solution of 5% cell passivating and cleaning fluid (part number 5062-8529) in water for each cell.

2 Place the probe of the selected channel (with the filter) into the beaker with the 5% cell passivating and cleaning fluid and pump the 50 ml of cell passivating and cleaning fluid through each flow cell.

3 Repeat step 1 and 2 for each channel of each bath.

NOTE You may observe a high amount of air coming through the flow cell, because the solution contains a detergent.

Draw 100 ml of water through flow cell

4 Place the probe (with the filter) into a beaker with distilled water and draw 100 ml of water through each flow cell.

5 Repeat step 4 for each channel of each bath.

NOTE Before proceeding with the performance verification, every channel has to be filled with distilled water.

Preparing the Spectrophotometer, Multicell Transport, Eight-port Valve and Peristaltic Pump 1VS

NOTE The verification of the individual hardware components has to be carried out during an installation of a multicell transport sampling system, or a change/repair of one of the hardware components, or at least every 6 months.

NOTE For an existing and unchanged system, which is checked on a daily or weekly basis, begin the OQ/PV with "Procedure I" on page 156.

Performance verification of the spectrophotometer and the multicell transport

Prior to the performance verification of the multibath Sampling System, the performance of the HP 8453 spectrophotometer and the multicell transport have to be verified.

Refer to Chapter 2 "Spectrophotometer Performance Verification" for doing a spectrophotometer performance verification.

NOTE

The following procedure has to be carried out for each of the valve systems in use.

Procedure I

1 The eight-port valve and peristaltic pump 1VS system should be properly installed as described in the manual.

2 Switch on the HP 8453 spectrophotometer and allow it to warm up for at least 20 minutes before making any measurements.

3 Start the HP ChemStation software.

4 Select the Verification and Diagnostics mode using the tool bar Mode section (or use the Mode menu).

5 Select the Flow Test task in the graphical user interface (or use the Task menu).

6 Set the wavelength for your test sample and the desired level of purity using the Setup button in the Flow Test window of the graphical user interface. The default values are the following:

Wavelength: 273 nm
Percent: 99.5%

7 Select Online Valve System in the sampling system drop down box.

8 Set the parameters for operation of the pump by selecting Setup and Parameter in the graphical interface (or use the Instrument menu, Setup Sampling System. Parameter) and entering the following:

Pump time: 47 s
(fixed, depending on method loaded previously in dissolution module)
Pump Direction: CW

NOTE The pump time is fixed to 47 seconds whenever a cycle time of 7.5 minutes has been chosen. For a cycle time of 5 minutes, the pump time is 29 seconds, whereas for a cycle time of 10 minutes you will get a pump time of 58 seconds.

9 Select the Channel you want to test in the Flow Test window of the graphical user interface.

10 Put the end of the probe of the channel you want to test into the reservoir containing the solvent.

11 Switch on the pump by clicking on the pump icon in the instrument panel of the graphical user interface and flush the system for approximately twice as long as the pump time you set. Switch off the pump by clicking on the pump icon.

12 Make a Blank measurement.

13 Put the end of the probe of the channel you want to test into the reservoir containing the test sample and start the test using the Run button in the Flow Test window. The test will take 50% longer than the pump time you have entered. You should get a graphic display of the absorbance versus time trace and a display of the pump time in seconds required for every channel to achieve the entered % purity.

14 Repeat the test for each of the channels.

Acceptance I

Optimal pump time has been achieved, when the flow test indicates that you get consecutive results within 10% or 1 second, whichever is greater. Use Fill-in Form 37 on page 166 to document your results. If the test fails, refer to "Troubleshooting I" on page 157.

Troubleshooting I

If good results cannot be obtained:

- Check if there is a bubble in the flow cell, if yes, gentle tapping of the cell will help to dislodge it.
- Check the flow rate of your pump and replace the pump tubing if necessary.

- Check for air being sucked in at one of the fittings.
- Check rate of your pump and replace the pump tubing if necessary.

Performance Verification Procedures for Multibath Sampling System

NOTE The procedures described in the following have to be carried out for each of the baths, in order to achieve a complete verification of the multibath system.

Procedure II

1 If the HP 8453 spectrophotometer is not already turned on, switch the spectrophotometer on.

2 Start the HP ChemStation.

3 Select Verification and Diagnostics from the Mode menu or tool bar.

Select Dissolution Test and Multibath as sampling system

4 Select Dissolution Test in the Task drop down box of the graphical user interface or by using the Task menu.

5 In the Sampling system drop down box choose Multibath.

6 Press Setup and Parameter for checking the settings of the pump. The default settings are:

Set pump parameters

Pump time: 47 s
(fixed, depending on method loaded previously in dissolution module)
Pump Direction: CW

NOTE The pump time is fixed to 47 seconds whenever a cycle time of 7.5 minutes has been chosen. For a cycle time of 5 minutes, the pump time is 29 seconds, whereas for a cycle time of 10 minutes you will get a pump time of 58 seconds.

Put probe in distilled water

7 Put probe 1 into a beaker filled with 200 ml of distilled water and select the channel in the graphical user interface accordingly.

8 Put return of probe 1 into a beaker to measure the amount of water pumped through the flow cell.

9 In the graphical user interface of the sampling system press the *Flow Rate*-button and select the bath to verify.

10 Set the parameters of the flow rate test by entering the following:

Set flow rate values

Duration: 2 min
Direction: CW
Limits: 6 ml/min, +/- 10%

Start flow rate measurement and measure the amount of liquid pumped through each channel

11 Start the flow rate test by pressing the OK-button.

12 Measure the volume you collected in the beaker and enter it, in units of ml, into the edit box coming up at the end of the flow rate test.

13 Check in the test result table coming up, if the test is passed.

14 Repeat steps 7 through 13 for each channel of the valve and each bath.

Acceptance II

The calculated flow rate must be within the limits specified. This is indicated by passed in the *result* column of the test result table. Use Fill-in Form 38 on page 167 to document your results. If the test fails, refer to "Troubleshooting II" on page 160.

Troubleshooting II

- Check if cells and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as

described under .

- Check if all pump tubings, filter tips have been renewed and that the tubing clamps are closed.
- Depending on whether the pumped volumes are to high or to low, lower or raise the pump speed.

Procedure III

1 Press the Cross Contamination-button in the graphical user interface. Set the parameters of the cross contamination test by entering the following:

Wavelength: 273 nm
Minimum limit: 99.5%
Maximum limit: 0.5%

NOTE 273 nm is set-up as default wavelength when the caffeine sample (part number 5063-6524) is used for the test and the path length of the cell 10 mm. For 1 mm path length cells use 205 nm as default wavelength.

2 Press OK and select the bath to verify by activating the according radio button accordingly.

3 Start the cross contamination test by pressing the OK-button.

4 Put probe 1 into a beaker filled with distilled water or Blank medium. Press the OK-button.

5 Put probe 1 into a beaker filled with a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml) or the Sample medium. Press the OK-button.

NOTE Ensure having chosen a wavelength where your sample shows an absorbance of approximately 1 AU.

Put probes 1,3,5,7 into the blank

1 Put probes 1, 3, 5 and 7 into a beaker filled with approximately 200 ml of distilled water (HPLC grade), or your Blank medium.

Put probes 2,4,6,8 into the sample

2 Put probes 2, 4, 6 and 8 into a beaker filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml), or your Sample medium, and press OK.

Put probes 2,4,6,8 into the blank

3 Put probes 2, 4, 6 and 8 into a beaker filled with approximately 200 ml of distilled water (HPLC grade), or your Blank medium.

Put probes 1,3,5,7 into the sample

4 Put probes 1, 3, 5 and 7 into a beaker filled with approximately 200 ml of a mixture of caffeine/distilled water, HPLC grade (part number 5063-6524 for 50 ml), or your Sample medium, and press OK.

Acceptance III

For each of the channels the table should show *passed* in the result column. Use Fill-in Form 39 on page 168 to document your results. Whenever one of the channels failed the test, refer to "Troubleshooting III" on page 162.

Troubleshooting III

- Check if the cell and tubings are free of bubbles. When there is a bubble in the flow cell, tap the flow cell gently on the table to remove the bubble. Sticking bubbles can only be removed by cleaning the flow cell as described under .
- Check if all pump tubings, filter tips have been renewed and that the tubing clamps are closed.
- Go back to Flow Task test and check the pump time of the peristaltic pump. Depending on whether the pump time is to high or to low, lower or

raise the pump time.

- Check if the valve fittings tightened. To ensure proper installation of the fittings, tighten them gently with the plastic wrench shipped with the valve sampling system.

Multibath Sampling System OQ/PV Attachment Forms—Bath 1

Use Fill-in Form 34, Fill-in Form 35 and Fill-in Form 36 to record the hardware for which the performance verification was carried out.

Fill-in Form 34

Peristaltic Pump 1VS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	G1103-60004 / G1103-60006
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 35

Eight-port Valve Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89079A
Manufacturer	Model #

Valve Serial #	Asset #	System #

Fill-in Form 36

Multicell Transport Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89075C/D or G1120A
Manufacturer	Model #

MCT Serial #	Asset #	System #

Use Fill-in Form 38, Fill-in Form 38 and Fill-in Form 39 to record the results of the multibath system performance verification.

Fill-in Form 37

Eight-port Valve / Peristaltic Pump 1VS Flow Test Results (Procedure I)

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time Channel 1				
	Passed		Failed	
Pump Time Channel 2				
	Passed		Failed	
Pump Time Channel 3				
	Passed		Failed	
Pump Time Channel 4				
	Passed		Failed	
Pump Time Channel 5				
	Passed		Failed	
Pump Time Channel 6				
	Passed		Failed	
Pump Time Channel 7				
	Passed		Failed	
Pump Time Channel 8				
	Passed		Failed	

Fill-in Form 38

Eight-port Valve / Peristaltic Pump 1VS Flow Rate Test Results (Procedure II)

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate Channel 1				
	Passed		Failed	
Flow Rate Channel 2				
	Passed		Failed	
Flow Rate Channel 3				
	Passed		Failed	
Flow Rate Channel 4				
	Passed		Failed	
Flow Rate Channel 5				
	Passed		Failed	
Flow Rate Channel 6				
	Passed		Failed	
Flow Rate Channel 7				
	Passed		Failed	
Flow Rate Channel 8				
	Passed		Failed	

Fill-in Form 39

Eight-port Valve / Peristaltic Pump 1VS Cross-Contamination Test Results

	Min. for 100% Absorbance	Measured	Max. for 0% Absorbance	Measured
Relative Abs. % Channel 1				
	Passed		Failed	
Relative Abs. % Channel 2				
	Passed		Failed	
Relative Abs. % Channel 3				
	Passed		Failed	
Relative Abs. % Channel 4				
	Passed		Failed	
Relative Abs. % Channel 5				
	Passed		Failed	
Relative Abs. % Channel 6				
	Passed		Failed	
Relative Abs. % Channel 7				
	Passed		Failed	
Relative Abs. % Channel 8				
	Passed		Failed	

Multibath Sampling System OQ/PV Attachment Forms—Bath 2

Use Fill-in Form 40, Fill-in Form 41 and Fill-in Form 42 to record the hardware for which the performance verification was carried out.

Fill-in Form 40

Peristaltic Pump 1VS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	G1103-60004 / G1103-60006
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-In Form 41

Eight-port Valve Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89079A
Manufacturer	Model #

Valve Serial #	Asset #	System #

Fill-In Form 42

Multicell Transport Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89075C/D or G1120A
Manufacturer	Model #

MCT Serial #	Asset #	System #

Use Fill-in Form 43, Fill-in Form 44 and Fill-in Form 45 to record the results of the multibath system performance verification.

Fill-in Form 43

Eight-port Valve / Peristaltic Pump 1VS Flow Test Results (Procedure I)

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time Channel 1				
	Passed		Failed	
Pump Time Channel 2				
	Passed		Failed	
Pump Time Channel 3				
	Passed		Failed	
Pump Time Channel 4				
	Passed		Failed	
Pump Time Channel 5				
	Passed		Failed	
Pump Time Channel 6				
	Passed		Failed	
Pump Time Channel 7				
	Passed		Failed	
Pump Time Channel 8				
	Passed		Failed	

Fill-In Form 44

Eight-port Valve / Peristaltic Pump 1VS Flow Rate Test Results (Procedure II)

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate Channel 1				
	Passed		Failed	
Flow Rate Channel 2				
	Passed		Failed	
Flow Rate Channel 3				
	Passed		Failed	
Flow Rate Channel 4				
	Passed		Failed	
Flow Rate Channel 5				
	Passed		Failed	
Flow Rate Channel 6				
	Passed		Failed	
Flow Rate Channel 7				
	Passed		Failed	
Flow Rate Channel 8				
	Passed		Failed	

Fill-in Form 45

Eight-port Valve / Peristaltic Pump 1VS Cross-Contamination Test Results

	Min. for 100% Absorbance	Measured	Max. for 0% Absorbance	Measured
Relative Abs. % Channel 1				
	Passed		Failed	
Relative Abs. % Channel 2				
	Passed		Failed	
Relative Abs. % Channel 3				
	Passed		Failed	
Relative Abs. % Channel 4				
	Passed		Failed	
Relative Abs. % Channel 5				
	Passed		Failed	
Relative Abs. % Channel 6				
	Passed		Failed	
Relative Abs. % Channel 7				
	Passed		Failed	
Relative Abs. % Channel 8				
	Passed		Failed	

Multibath Sampling System OQ/PV Attachment Forms—Bath 3

Use Fill-in Form 46, Fill-in Form 47 and Fill-in Form 48 to record the hardware for which the performance verification was carried out.

Fill-in Form 46

Peristaltic Pump 1VS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	G1103-60004 / G1103-60006
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 47

Eight-port Valve Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	89079A
Manufacturer	Model #

Valve Serial #	Asset #	System #

Fill-in Form 48

Multicell Transport Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	89075C/D or G1120A
Manufacturer	Model #

MCT Serial #	Asset #	System #

Use Fill-in Form 49, Fill-in Form 50 and Fill-in Form 51 to record the results of the multibath system performance verification.

Fill-in Form 49

Eight-port Valve / Peristaltic Pump 1VS Flow Test Results (Procedure I)

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time Channel 1				
	Passed		Failed	
Pump Time Channel 2				
	Passed		Failed	
Pump Time Channel 3				
	Passed		Failed	
Pump Time Channel 4				
	Passed		Failed	
Pump Time Channel 5				
	Passed		Failed	
Pump Time Channel 6				
	Passed		Failed	
Pump Time Channel 7				
	Passed		Failed	
Pump Time Channel 8				
	Passed		Failed	

Fill-in Form 50

Eight-port Valve / Peristaltic Pump 1VS Flow Rate Test Results (Procedure II)

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate Channel 1				
	Passed		Failed	
Flow Rate Channel 2				
	Passed		Failed	
Flow Rate Channel 3				
	Passed		Failed	
Flow Rate Channel 4				
	Passed		Failed	
Flow Rate Channel 5				
	Passed		Failed	
Flow Rate Channel 6				
	Passed		Failed	
Flow Rate Channel 7				
	Passed		Failed	
Flow Rate Channel 8				
	Passed		Failed	

Fill-In Form 51

Eight-port Valve / Peristaltic Pump 1VS Cross-Contamination Test Results

	Min. for 100% Absorbance	Measured	Max. for 0% Absorbance	Measured
Relative Abs. % Channel 1				
	Passed		Failed	
Relative Abs. % Channel 2				
	Passed		Failed	
Relative Abs. % Channel 3				
	Passed		Failed	
Relative Abs. % Channel 4				
	Passed		Failed	
Relative Abs. % Channel 5				
	Passed		Failed	
Relative Abs. % Channel 6				
	Passed		Failed	
Relative Abs. % Channel 7				
	Passed		Failed	
Relative Abs. % Channel 8				
	Passed		Failed	

Multibath Sampling System OQ/PV Attachment Forms—Bath 4

Use Fill-in Form 52, Fill-in Form 53 and Fill-in Form 54 to record the hardware for which the performance verification was carried out.

Fill-in Form 52

Peristaltic Pump 1VS Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT PACKARD	G1103-60004 / G1103-60006
Manufacturer	Model #

Pump Serial #	Asset #	System #

Fill-in Form 53

Eight-port Valve Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89079A
Manufacturer	Model #

Valve Serial #	Asset #	System #

Fill-in Form 54

Multicell Transport Description

This chapter describes the OQ/PV tests to be performed for the HP 8453 spectroscopy system component below.

HEWLETT® PACKARD	89075C/D or G1120A
Manufacturer	Model #

MCT Serial #	Asset #	System #

Use Fill-in Form 55, Fill-in Form 56 and Fill-in Form 57 to record the results of the multibath system performance verification.

Fill-in Form 55

Eight-port Valve / Peristaltic Pump 1VS Flow Test Results (Procedure I)

	Set Point	Upper Limit	Lower Limit	Measured
Pump Time Channel 1				
	Passed		Failed	
Pump Time Channel 2				
	Passed		Failed	
Pump Time Channel 3				
	Passed		Failed	
Pump Time Channel 4				
	Passed		Failed	
Pump Time Channel 5				
	Passed		Failed	
Pump Time Channel 6				
	Passed		Failed	
Pump Time Channel 7				
	Passed		Failed	
Pump Time Channel 8				
	Passed		Failed	

Fill-in Form 56

Eight-port Valve / Peristaltic Pump 1VS Flow Rate Test Results (Procedure II)

	Set Point	Upper Limit	Lower Limit	Measured
Flow Rate Channel 1				
	Passed		Failed	
Flow Rate Channel 2				
	Passed		Failed	
Flow Rate Channel 3				
	Passed		Failed	
Flow Rate Channel 4				
	Passed		Failed	
Flow Rate Channel 5				
	Passed		Failed	
Flow Rate Channel 6				
	Passed		Failed	
Flow Rate Channel 7				
	Passed		Failed	
Flow Rate Channel 8				
	Passed		Failed	

Fill-In Form 57

Eight-port Valve / Peristaltic Pump 1VS Cross-Contamination Test Results

	Min. for 100% Absorbance	Measured	Max. for 0% Absorbance	Measured
Relative Abs. % Channel 1				
	Passed		Failed	
Relative Abs. % Channel 2				
	Passed		Failed	
Relative Abs. % Channel 3				
	Passed		Failed	
Relative Abs. % Channel 4				
	Passed		Failed	
Relative Abs. % Channel 5				
	Passed		Failed	
Relative Abs. % Channel 6				
	Passed		Failed	
Relative Abs. % Channel 7				
	Passed		Failed	
Relative Abs. % Channel 8				
	Passed		Failed	

Performance Verification of the Dissolution Testing Software

This section guides you through the performance verification of the dissolution testing software.

Dissolution Testing UV-Visible ChemStation Software Revalidation

Scope

The following procedure describes how to validate dissolution testing software on a HP ChemStation of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure if:

- you have installed or reinstalled your software,
- you have upgraded your software to a new revision, or
- you have had a software or system crash.

Instrumentation and Software

- This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer, general scanning software for HP ChemStation (order number G1115AA) and dissolution testing software for HP ChemStation (order number G1118AA).
- The CD-ROM including the method files you need, is delivered with the general scanning software.
- A printer connected to the system.

Procedure

1 Start Windows.

2 Start the HP ChemStation software by clicking on the Instrument # Online icon of the HP ChemStations group, where # is the number of the instrument you chose in the configuration process.

3 Select Dissolution Testing Mode from the Mode drop-down box.

4 Insert the CD-ROM in your CD-ROM drive.

5 Load the sample data from the disvalid.sd data file of the support\uv\sops\data subdirectory using the Files menu.

Single-Bath Dissolution Testing Software

6 Load the disvalid.m method file from the support\uv\sops\8453 subdirectory of the CD-ROM using the Files Menu or the Load Method icon in the tool bar.

7 Print a report using the File menu or the printer icon in the tool bar.

8 Compare results of the report with the values in "Exhibit A: Validation Results for Dissolution Testing Software" on page 187. With the exception of operator name and report date and time they should be identical.

Multibath Dissolution Testing Software

9 Select Multibath Dissolution Testing form the Mode drop-down box.

10 Load the dimvalid.m method file from the support\uv\sops\8453 subdirectory of the CD-ROM using the Files Menu or the Load Method icon in the tool bar.

11 Print a report using the File menu or the printer icon in the tool bar.

12 Compare results of the report with the values in "Exhibit A: Validation Results for Dissolution Testing Software" on page 187. With the exception of operator name and report date and time they should be identical.

Acceptance

Check that the results in the printed reports are identical with the values in "Exhibit A: Validation Results for Dissolution Testing Software" on page 187 of this SOP. Use the fill-in forms at the end of this chapter to document your results. If the test fails, refer to "Troubleshooting" on page 186.

Troubleshooting

If the results are not identical reinstall the software and repeat the revalidation procedure. If the results are still not identical, call Hewlett-Packard.

Exhibit A: Validation Results for Dissolution Testing Software

Individual Tablet Weights (mg)	Vessel	Weight
	1.00	100.0
	2.00	100.0
	3.00	100.0
	4.00	100.0
	5.00	100.0
	6.00	100.0

Weight of 100% released

Component Name	Weight (mg)
Comp	900.00

Data Analysis: (routine)

Used Wavelength: 360 nm
Process Spectrum as: Absorbance
Background correction: none
Use Calibration: Yes
Analyte name : Comp
Calibration curve : Linear
Units : mg/mL
Do Spectral Match: No

Calibration:

Comp	Standard	Value	Predicted	Error(%)
	Standard	1.00000	1.00000	0.0

Dissolution Table: Comp

Time	V 1	V 2	V 3	V 4	V 5	V 6	Average	Std.Dev	%
0.0	0.0	-0.0	0.0	-0.0	-0.0	-0.0	-0.0	0.0	-118.3
15.0	95.2	93.5	94.1	93.8	95.5	92.2	94.0	1.2	1.3
30.0	102.1	99.6	99.4	99.5	100.7	100.5	100.3	1.0	1.0

Quality Control Requirements:	Time	Minimum	Maximum	Result
	30.0	85.0	115.0	passed

Dissolution Testing Software OQ/PV Attachment Forms

Fill-in Form 58

Software Revision Description

This chapter describes the OQ/PV tests to be performed using the HP ChemStation software revision below (running under the given operating system).

HEWLETT® PACKARD	G1118AA
Manufacturer	Product #

Dissolution testing software

HP ChemStation software module

License #	Revision #

Operating System

Revision #

	Completed by

Fill-in Form 59

Dissolution Testing Software

Type of Test	Criteria	Report OK?
Single-bath dissolution test bath 1	According to Exhibit A	
Multibath dissolution test bath 1	According to Exhibit A	
Multibath dissolution test bath 2	According to Exhibit A	
Multibath dissolution test bath 3	According to Exhibit A	
Multibath dissolution test bath 4	According to Exhibit A	

Passed		Failed		

Performance Verification of the DDE Interface for Bath Drivers

This section guides through the way a performance verification of your DDE interface has to be carried out, in order to ensure a correct communication with your bath driver.

Revalidation of the DDE Interface for Dissolution Testing Software

Scope

The following procedure describes how to validate the DDE interface of dissolution testing software on a HP ChemStation of a HP 8453 UV-visible spectroscopy system.

Frequency

Follow appropriate procedure if:

- you have installed or reinstalled your software,
- you have upgraded your software to a new revision,
- you have changed your bath driver, or
- you have had a software or system crash.

Instrumentation and Software

- This SOP applies to a HP 8453 UV-visible spectroscopy system comprising a HP 8453 spectrophotometer, general scanning software for HP ChemStation (order number G1115AA) and dissolution testing software for HP ChemStation (order number G1118AA).
- The CD-ROM including the method files you need, is delivered with the general scanning software.
- A printer connected to the system.

Procedure

1 Start Windows.

2 Insert the HP ChemStation CD-ROM in your CD-ROM drive.

3 Start Windows Explorer.

4 Install the dummy bath driver from the CD-ROM by clicking on setup.exe in the directory support\uv\sops\bathdrv into the directory c:\hpchem\bathdrv. This directory will be created by the installation process.

5 Start the HP ChemStation software by clicking on the Instrument # Online icon of the HP ChemStations group, where # is the number of the instrument you chose in the configuration process.

Single-Bath Dissolution Testing Software

6 Select Dissolution Testing Mode from the Mode drop-down box.

7 Select New Method from the File menu.

8 Configure the bath driver by selecting Bath from the Config menu and specify in the Enter command line of bath driver dialog box the command:

```
c:\hpchem\bathdrv\dummy16.exe
```

Click OK to close the dialog box.

9 To activate the bath driver a mode switch has to be done. Select Standard Mode from the Mode drop-down box. Select Dissolution Testing Mode from the Mode drop-down box.

10 Select Edit Product Info & Bath Param in the Method menu. Click on the tab for Option & Info and activate the option Bath Parameter are controlled by ChemStation. Close the dialog box with OK to activate the option.

11 Select Dissolution Bath Status from the Instrument menu. Position the window of dissolution bath driver that way to the bottom of the screen, that you can see the value field of the bath for current temperature (default 37 °C), current stirrer speed (default 075), current pH (default 7.00) and current volume (default 900).

12 Select Edit Product Info & Bath Param in the Method menu. Click on the tab for Bath and modify the values as follows:

Temperature 35.0 °C
pH 9.0
Volume 800 ml
Stirrer speed 100 rpm

13 Close the dialog box with OK and verify that the Dummy Bath Status screen reflects this change in parameters.

NOTE The fields for the parameters of the individual vessels are for future enhancements.

14 Click on the button Blank to measure a blank spectrum.

15 Click on the button Dissolution Run to start a dissolution run. In case the Consistency check results screen pops up, click on OK to continue.

16 Verify that the actual parameters for pH, temperature and stirrer speed, which are read back from the bath driver, are identical with the set values.

NOTE The volume is not read back from the dummy bath driver.

17 Close the dialog box with Cancel to abort the dissolution run.

Multibath Dissolution Testing Software

18 Select Multibath Dissolution Testing Mode from the Mode drop-down box

19 Select New Method from the File menu.

20 Configure the bath driver by selecting Bath from the Config menu, select all four baths, and specify in the Enter command line of bath driver dialog box the command:

```
c:\hpchem\bathdrv\dummy16.exe
```

for all four baths

Click 'OK' to close the dialog box.

21 To activate the bath driver a mode switch has to be done. Select Standard Mode from the Mode drop-down box. Select Multibath Dissolution Testing Mode from the Mode drop-down box.

22 Select Spectrophotometer Reference from the Measure menu to measure a reference.

23 Click on the button Bath 1 to switch to bath 1.

24 Select Edit Product Info & Bath Param in the Method menu. Click on the tab for Option & Info and activate the option Bath Parameter are controlled by ChemStation. Close the dialog box with OK to activate the option.

25 Select Dissolution Bath Status from the Instrument menu. Position the window of dissolution bath driver that way to the bottom of the screen, that you can see the value field of the bath for current temperature (default 37 °C), current stirrer speed (default 075), current pH (default 7.00) and current volume (default 900).

26 Select Edit Product Info & Bath Param in the Method menu. Click on the tab for Bath and modify the values as follows:

Temperature 35.0 °C
pH 9.0
Volume 800 ml
Stirrer speed 100 rpm

27 Close the dialog box with OK and verify that the Dissolution Bath Status screen reflects this change in parameters.

NOTE The fields for the parameters of the individual vessels are for future enhancements.

28 Click on the button Blank to measure a blank spectrum.

29 Click on the button Dissolution Run to start a dissolution run.

30 Verify that the actual parameters for pH, temperature and stirrer speed, which are read back from the bath driver, are identical with the set values.

NOTE The volume is not read back from the dummy bath driver.

31 Close the dialog box with Cancel to abort the dissolution run.

32 Repeat step 23 through step 31 for Bath 2 to Bath 4.

Acceptance

Check that the values set in the method for the bath parameters pH, temperature and stirrer speed are identical with the values shown in the dissolution bath status screen. Check that the actual values for pH, temperature and stirrer speed are identical with the set values for the bath parameters in the dissolution run parameter screen then starting a dissolution run.

Troubleshooting

If the results are not identical reinstall the software and repeat the revalidation procedure. If the results are still not identical, call Hewlett-Packard.

Revalidation of the DDE Interface for Dissolution Testing Software OQ/PV Attachment Forms

Fill-in Form 60

Software Revision Description

This chapter describes the OQ/PV tests to be performed using the HP ChemStation software revision below (running under the given operating system).

HEWLETT PACKARD	G1118AA
Manufacturer	Product #

Dissolution testing software

HP ChemStation software module

License #	Revision #

Operating System

Revision #

Fill-in Form 61 **Dissolution Testing Software**

Parameter	Method	Dissolution Bath Status		Values OK?		Dissolution Run Parameter Actual Value		Values OK?	
Temperature	35.0								
pH	9.0								
Stirrer speed	100								
		Passed				Failed			

8

Parts and Materials

Parts and Materials

This chapter gives part numbers for all Hewlett Packard parts that are necessary to do the performance verification and it gives a list of sources for standards that cannot be obtained through Hewlett-Packard.

Parts List

Table 6 **OQ/PV Kits and Chemicals**

Description	Part Number
OQ/PV standards (1) kit for UV-visible spectroscopy (liquid standards in ampules)*	5063-6503
OQ/PV standards (2) kit for UV-visible spectroscopy (liquid standards in ampules, containing all standards with perchloric acid)**	5063-6521
OQ/PV hardware kit for UV-visible spectroscopy (including tubings, 2 flow cells, 2 syringes, 2 Luer-lock adapters, multicell transport adjustment tool, temperature sensor support, 3.5-inch flexible disk and handbook)	5063-6523
Caffein sample, 0.01 mg/ml, 50 ml solution	5063-6524

* For names and chemical formulae of the liquid standards, see Table 8 on page 203
** For names and chemical formulae of the liquid standards, see Table 8 on page 203

Table 7 **Replacement Parts**

Description	Part Number
Flow cell, 11 × 3.5 mm aperture, 360 µl volume, 10 mm path length	5061-3398
OQ/PV Tubing kit	5063-6522
Cell passivating and cleaning fluid, 1000 ml	5062-8529
Multicell transport adjustment tool	89075-23800
Temperature sensor support	89090-84700

Standards from External Sources

The following standards can be obtained from external sources. Use the information below for ordering.

Wavelength Accuracy

The NIST 2034 holmium oxide solution is available from:

U.S. Department of Commerce
National Institute of Standards and Technology
Standard Reference Materials Program
Bldg. 202, Room 204
Gaithersburg
MD 20899
USA
Tel. (301) 975 6776

Photometric Accuracy

The NIST 930e standard is available from NIST, see above address.

Other Standards

All other standards can be prepared using the appropriate material recommended in the EP or ASTM procedures.

Temperature Accuracy

QuaT 100 handheld temperature measuring unit with a QuaT 340 temperature probe, available from:

Heraeus Sensors GmbH
Reinhard-Heraeus Ring 23
63801 Kleinostheim
Germany
Tel. (+49) (6027) 503-0

Names and Chemical Formulae of Hewlett-Packard Liquid Standards

Table 8 **Names and Chemical Formulae of Hewlett-Packard Liquid Standards**

Name	Chemical Formula
OQ/PV Standards (1) Kit	
Potassium dichromate	$K_2Cr_2O_7$
Sulfuric acid	H_2SO_4
Sodium nitrite	$NaNO_2$
Sodium iodide	NaI
Potassium chloride	KCl
Toluene	C_7H_8
Tolune / hexane	C_7H_8 / C_6H_{14}
OQ/PV Standards (2) Kit	
Holmium oxide	Ho_2O_3
Perchloric acid	$HClO_4$

HEWLETT® PACKARD

Expanding Possibilities

In This Book

This handbook is intended for the technical reader who needs an operating procedure for the performance verification of the instrument.

The handbook contains specifications of the instrument as well as procedures of performance verifications. These procedures are listed in great detail concerning handling of chemicals and cuvettes, because they are intended also for less experienced users. Following these procedures exactly is mandatory for the success of the performance verification. Part numbers and ordering information for parts from Hewlett-Packard and from other companies are given in a separate section.

For information about installation of the system including the spectrophotometer, computer and accessories, see the *Installing Your UV-Visible System* handbook.

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