

JJG

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

JJG 705-2014

Liquid Chromatography (English Translation)

Issued on: February 14, 2014

Implemented on: August 14, 2014

Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the PRC

Table of Contents

Introduction...	4
1 Scope...	5
2 Normative references...	5
3 Overview...	5
4 Metrological performance requirements...	6
4.1 Delivery System...	6
4.2 Column oven...	6
4.3 Detector...	6
4.4 Overall instrument performance...	7
5 General technical requirements...	7
5.1 Instrument appearance...	7
5.2 Instrument circuit system...	7
6 Control of measuring instruments...	8
6.1 Verification conditions...	8
6.2 Verification items and methods...	9
6.3 Verification results...	20
6.4 Verification cycle...	20
Appendix A Chromatographic column performance testing...	21
Appendix B Performance testing of monochromator filters...	23
Appendix C Flow phase density at different temperatures...	25
Appendix D Verification certificate (inner page) format...	27
Appendix E Calibration record format of liquid chromatograph...	28

1 Scope

This Regulation applies to the initial verification, subsequent verification, in-service inspection of liquid chromatographs, which are equipped with UV-Vis detectors, diode array detectors, fluorescence detectors, differential refractive index detectors, evaporative light scattering detectors.

2 Normative references

JJG 537-2006 Verification regulation of fluorescence spectrophotometer

3 Overview

A liquid chromatograph (hereinafter referred to as the instrument) is an analytical instrument, which is composed of a delivery system, an injection system, a separation system, a detection system, a data processing system. Figure 1 is a block diagram of its components. The liquid chromatograph utilizes the differences in the distribution or adsorption characteristics of various components in the sample, between the stationary phase and the mobile phase, within the chromatographic column. The mobile phase carries the sample into the chromatographic column for separation; the sample is detected by the detector; the chromatogram is recorded by the data processing system.

Qualitative and quantitative analysis is performed based on the retention time and response value (peak area or peak height) of each component.

4.1 Delivery System

4.1.1

The delivery pipeline interfaces must be tight and secure, with no leakage within the specified pressure range.

4.1.2

The pump flow rate setpoint error SS and flow rate stability SR shall meet the requirements of Table 1.

4.1.3

Maximum permissible gradient error Gc. $\pm 3\%$.

4.2 Column oven

4.2.1

Maximum permissible error of column oven temperature setpoint. $\pm 2\text{ }^{\circ}\text{C}$.

4.2.2

Column oven temperature stability. Not greater than $1\text{ }^{\circ}\text{C/h}$.

4.3 Detector

The main technical indicators of the instrument's detector are shown in Table 2.

4.4 Overall instrument performance

The overall performance of the instrument is expressed by the repeatability of qualitative and quantitative measurements. The requirements are shown in Table 3.

5.1 Instrument appearance

The instrument shall have a nameplate, displaying the instrument name, model, manufacturer's name, product series number, exit-factory date, etc. Domestically produced instruments shall have a manufacturing license mark for metering instruments.

5.2 Instrument circuit system

The instrument's power cord and signal cables are securely connected. All switches, knobs, buttons function normally. Indicator lights are sensitive. The display is clear.

6 Control of measuring instruments

Control of measuring instruments includes initial verification, subsequent verification, in-use inspection.

6.1.1 Environmental conditions

6.1.1.1

The verification chamber shall be clean and dust-free, free of flammable, explosive, corrosive gases, and well-ventilated.

6.1.1.2

Room temperature (15 ~ 30) °C, with temperature changes not exceeding 3 °C during verification (for differential refractive index detectors, room temperature changes not exceeding 2 °C); relative humidity 20% ~ 85%.

6.1.1.3

The instrument shall be placed stably on the workbench, free from strong mechanical vibrations and electromagnetic interference sources, meanwhile properly grounded.

6.1.1.4

The power supply voltage is (220 ± 22) V; the frequency is (50 ± 0.5) Hz.

6.1.2 Verification equipment

6.1.2.1

Stopwatch. Minimum scale division not greater than 0.1 s.

6.1.2.2 Analytical balance. Maximum weighing capacity not less than 100 g, minimum

scale division not greater than 1 mg.

6.1.2.3 Digital thermometer. Measuring range (0 ~ 100) °C, maximum permissible error

±0.3 °C.

All the above instruments must be certified by metrological verification.

6.1.3 Certified reference materials.

Naphthalene-methanol solution reference material. Certified value 1.00×10^{-4} g/mL, expanded uncertainty < 4%, $k = 2$;

Naphthalene-methanol solution reference material. Certified value 1.00×10^{-7} g/mL, expanded uncertainty < 4%, $k = 2$;

Cholesterol solution in methanol reference material. Certified value 200 µg/mL, expanded uncertainty < 2.0%, $k = 2$;

Connect the detector and data processing system; set the detector wavelength to

254 nm. After power-on stabilization, inject 2% isopropanol-water solution directly into the detection cell to flush it, until the reading stabilizes. Record this value. Then, following the same method, sequentially inject a series of acetone-

2% isopropanol aqueous solutions (acetone content of 0.1%, 0.2%, ..., 1.0%) into the detection cell. Record the stable response signal value corresponding to each solution. Each solution is measured 3 times. Take the arithmetic mean. Plot a standard curve, using 5 acetone solution concentrations (0.1%, 0.2%, 0.3%, 0.4%,

0.5%) and their corresponding response signal values. Find the readings at each point on the curve where the acetone solution concentration is greater than 0.5%;

compare it with the measured values at the corresponding concentration points.

The concentration at which the value differed by 5% is taken as the upper limit of detection (CH). The minimum detection concentration, which is obtained according to 6.2.2.4 c), is taken as the lower limit of detection (CL). The CH/CL ratio is taken as the linear range.

6.2.2.5 Fluorescence detector performance

a) Wavelength indication error and repeatability

For the calibration of the wavelength indication error and repeatability of the fixed-wavelength fluorescence detector, the filter in the detector needs to be removed. Referring to the method 5.3.3 in JJG 537-2006 "Verification regulation of fluorescence spectrophotometer" (see Appendix B), measure the wavelength corresponding to the maximum transmittance on a calibrated UV-Vis spectrophotometer. The difference between this wavelength and the wavelength marked on the filter is the wavelength indication error.