



Prep and Load Platform

-xt SYSTEM

-xt

Specifications for PAL-*xt* Systems

Valid for PAL-*xt* System
models only

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Gravimetric Test

Syringe Volume	Specification	Test Conditions
10 µL	Repeatability Relative Standard Deviation s-rel : 0.20 %	15 times 8 µL Water transferred into target vial.
100 µL	Repeatability Relative Standard Deviation s-rel : 0.15 %	100 µL Water transferred in a target vial.
	Linearity Correlation Coefficient R: ≥0.9999	6 levels and 3 replicates of each level. 5,10,25,50,75,100 µL
1000 µL	Repeatability Relative Standard Deviation s-rel : 0.10 %	500 µL Water transferred in a target vial.
	Linearity Correlation Coefficient R: ≥ 0.9999	6 levels and 3 replicates of each level. 50,100,250,500,750,1000 µL
2500 µL	Repeatability Relative Standard Deviation s-rel : 0.10 %	1000 µL Water transferred in a target vial.
	Linearity Correlation Coefficient R: ≥ 0.9999	8 levels and 3 replicates of each level. 125,250,500,750,1000,1500,2000,2500 µL

General Remarks:

- Number of specimens for repeatability tests: n = 7
- For each target vial a source vial is specified.
- Temperature and barometric pressure not taken into consideration.
- Repeatability but not Accuracy determined
- Balance: Precisa Switzerland Model XB 120A (d = 0.1 mg)

Gas Chromatographic Test: High Split Ratio | Retention Gap | Solvent iso-Octane

Specification:

- Repeatability: Relative Standard Deviation s-rel: **0.30 %**
- Carry-over: **not applicable**

General Remarks:

- Number of specimens for Repeatability Tests: n = 20
- Carry-over: Test: Remaining solvent in syringe can not be eliminated from injection to injection.

Test Conditions:

Test Sample: iso-Octane puriss., 2 mL vials filled to 50 % of nominal content
 Syringe: Hamilton PNo.: 203205 | CTC Analytics PNo.: SyrC L10-26S-AS

PAL-xt Parameter Settings:

CYCLE	GC-Inj	Fill Strokes	5
SYRINGE	10 µL	Pullup Del	5 s
Sample Volume	1.0 µL	Inject to	GC Inj1
Air Volume	0.5 µL	Inject Speed	50 µL/s
Pre Cln Slv1	3	Pre Inj Del	500 ms
Pre Cln Slv2	0	Pst Inj Del	500 ms
Pre Cln Spl	2	Pst Cln Slv1	3
Fill Volume	5 µL	Pst Cln Solv2	0
Fill Speed	2 µL/s		

Eject speed 50 µL/s (Syringe Parameter)

GC Agilent 6890:

- Injector: split/splitless, Liner: Single Taper 4 mm ID, Needle Penetration 45 mm
 Silanized glass wool packing (PNo. HP 5062-3587)
 Temperature: 200 °C
 Split Ratio = 1:1000
- Detector: FID
 Temperature 300 °C
 Fuel Gases: Make-up Gas N2 25 ± 2 mL/min, Air 400 ± 30 mL/min, H2 30 ± 2 mL/min
- Column: Retention Gap (uncoated fused silica column).
 Length 5m, OD 0.375 mm, ID 0.100 mm,
 Supplier: BGB Switzerland, PNo. BGB TSP 1003755
 Column Oven Temperature: 200 °C, Runtime 0.5 min
 Carrier Gas: Helium 0.2 mL/min, approx. 23 psi column head pressure.
 Wash Solvent: iso-Octane

Data Acquisition System: Agilent ChemStation Software Version 6.03

Gas Chromatographic Test: Split Mode | Analytical Column | Alkene Test Mix

Specification:

- Repeatability: Relative Standard Deviation s-rel: **0.80 %**
- Carry-over: **< 0.080 %**

General Remarks:

- Number of specimens for Repeatability Tests: n = 8
- Repeatability determination based on C14 Peak
- Carry-over Test: Peak < 1 area count, not detectable.

Test Conditions:

Test Sample: Alkane Test Mix C14,C15,C16 | 0.3 µg dissolved in iso-Octane.
2 mL vials filled to 50 % of nominal content

Syringe: Hamilton PNo.: 203205 | CTC Analytics PNo.: SyrC L10-26S-AS

PAL-xt Parameter Settings:

CYCLE	GC-Inj	Fill Strokes	5
SYRINGE	10 µL	Pullup Del	5 s
Sample Volume	1.0 µL	Inject to	GC Inj1
Air Volume	0.5 µL	Inject Speed	50 µL/s
Pre Cln Slv1	3	Pre Inj Del	500 ms
Pre Cln Slv2	0	Pst Inj Del	500 ms
Pre Cln Spl	2	Pst Cln Slv1	3
Fill Volume	5 µL	Pst Cln Solv2	0
Fill Speed	2 µL/s		

Eject speed 50 µL/s (Syringe Parameter)

GC Agilent 6890:

- Injector: split/splitless, Liner: Single Taper 4 mm ID, Needle Penetration 45 mm
silanized glass wool packing (PNo. HP 5062-3587)
Temperature: 250 °C
Split Ratio = 1:50
- Detector: FID
Temperature 300 °C
Fuel Gases: Make-up Gas N2 25 ± 2 mL/min, Air 400 ± 30 mL/min, H2 30 ± 2 mL/min
- Column: 5% Phenyl-/95% Methyl-Silicon. HP5. Length 30m, ID 0.320 mm, Filmthickness 0.25 mm
Supplier: Agilent, PNo. HP 5182-9733
Column Oven Temperature: 80 °C, hold 1 min, 15 °C/min to 200 °C, hold 1 min, Runtime 10 min
Carrier Gas: Helium 2.0 mL/min
Wash Solvent: iso-Octane

Data Acquisition System: Agilent ChemStation Software Version 6.03

Gas Chromatographic Headspace Test: High Split Ratio | Retention Gap | Solvent iso-Octane

Specification:

- Repeatability: Relative Standard Deviation s-rel: < 1.00 %
- Carry-over: < 0.050 %

General Remarks:

- Number of specimens for Repeatability Tests: n = 7
- 10 µL are measured by using a glass capillary, Drummond Microcaps (Supplier Supelco, PNo. P-1924)

Test Conditions:

Test Sample: Iso-Octane, puriss.,
Microcaps Glass Capillary hold with Tweezers, filled and put into 20 mL Headspace vial, sealed immediately.

Syringe: Hamilton PNo.: 203084 | CTC Analytics PNo.: SyrC HS 2.5 -23-5

PAL-xt Parameter Settings:

CYCLE	HS-Inj	Fill Speed	100 µL/s
SYRINGE	2.5HS	Pullup Del	1 sec
Sample Volume	500 µL	Inject to	GC Inj1
Incubat Temp	80 °C	Inject Speed	500 µL/s
Incubat Time	7 minutes	Pre Inj Del	500 ms
Agi Speed	250 rpm	Pst Inj Del	1 s
Agi On Time	5 s	Syr Flushing	90 s
Agi Off Time	2 s	GC-Runtime	60 s
Syringe Temp	85 °C		

GC Agilent 6890:

- Injector: split/splitless, Liner: Single Taper 4 mm ID, Needle Penetration 40 mm
Silanized glass wool packing (PNo. HP 5062-3587)
Temperature: 250 °C
Split Ratio = 1:100
- Detector: FID
Temperature 300 °C
Fuel Gases: Make-up Gas N2 25 ± 2 mL/min, Air 400 ± 30 mL/min, H2 30 ± 2 mL/min
- Column: Retention Gap (uncoated fused silica column). Length 5m, OD 0.375 mm, ID 0.100 mm
Supplier: BGB Switzerland, PNo.: BGB TSP 1003755
Column Oven Temperature: 200 °C, Runtime: 1.0 min
Carrier Gas: Helium 0.1 mL/min, approx. 14 psi column head pressure.
Purge Gas: Helium

Data Acquisition System: Agilent ChemStation Software Version 6.03

HPLC Chromatographic Test: Loop Overfill and Partial Loop Filling Standard Injection Technique

Specification:

Syringe Volume	Specification	Test Conditions
100 µL	Repeatability Loop Overfill Relative Standard Deviation s-rel : 0.10 %	Loop size 20 µL Loop fill volume: 80 µL
100 µL	Repeatability Partial Loop Filling Relative Standard Deviation s-rel : 0.20 %	Loop size 20 µL Loop fill volume: 10 µL
100 µL	Linearity Correlation Coefficient R: > 0.9999	5 levels and 3 replicates of each level. 4,6,8,10,12 µL (20 to 60 % of loop size)

General Remarks:

Number of specimens for Repeatability Tests: n = 9
Carry-over: Test: Peak < 1 area count, not detectable.

Test Conditions:

Test Sample: Benzophenone, Supplier Fluka PNo. 12750,
50 µg/mL dissolved in in Water : Methanol = 50 : 50
Blank Solution: Water : Methanol = 20 : 80
Vials: 2 mL standard Autosampler vials, crimped. Vials filled to 50% of nominal content.
Syringe: Hamilton PNo.: 203077 | CTC Analytics PNo.: SyrC G100-22S-3

PAL-xt Parameter Settings:

CYCLE	LC-Inj	Pullup Del	500 ms
SYRINGE	100 µL	Inject to	LC Vlv1
Sample Volume	80 / 10 µL	Inject Speed	10 µL/s
Air Volume	0 nL	Pre Inj Del	500 ms
Pre Cln Slv1	1	Pst Inj Del	500 ms
Pre Cln Slv2	0	Pst Cln Slv1	2
Pre Cln Spl	2	Pst Cln Slv2	0
Fill Speed	10 µL/s	Vlv Cln Slv1	2
Fill Strokes	3	Vlv Cln Slv2	0

Eject Speed 200 µL/s (Syringe Parameter)

HPLC System Agilent 1100

HPLC Pump: Binary system, Flow Rate: 1.0 mL/min
HPLC Detector: Variable UV-VIS , Wavelength for Detection: 258 nm
Without Degassing and Column Heater
Column: Zorbax XDB-C8, Length: 150 mm, ID 4.6 mm, 5 µm particle size, end-capped
Suppliers: Agilent, PNo.: HP 993967906
Mobile Phase: Water : Methanol = 20 : 80
Wash Solvent: Water : Methanol = 50 : 50

Data Acquisition System: Agilent ChemStation Software Version 6.03

HPLC Chromatographic Test: Loop Overfill and Partial Loop Filling DLW Injection Technique

Specification:

Syringe Volume	Specification	Test Conditions
100 µL	Repeatability Loop Overfill Relative Standard Deviation s-rel : 0.50 %	Loop size 2 µL Loop fill volume: 7 µL
100 µL	Repeatability Partial Loop Filling Relative Standard Deviation s-rel : 0.50 %	Loop size 20 µL Loop fill volume: 10 µL
100 µL	Carry-over: Test < 40 ppm	Blank after full loop injection Loop size 2 µL Loop fill volume: 7 µL

General Remarks:

Number of specimens for Repeatability Tests: n = 10 for Loop Overfill
Number of specimens for Repeatability Tests: n = 10 for Partial Loop Filling

Test Conditions:

Test Sample Loop Overfill:	Caffeine, 520 mg/L dissolved in H ₂ O : ACN = 90 : 10, 0.1% TFA
Test Sample Partial Loop Filling:	Caffeine, 52 mg/L dissolved in H ₂ O : ACN = 90 : 10, 0.1% TFA
Test Sample Carry-over:	Chlorohexidine, 0.6 mg/mL dissolved in H ₂ O : ACN = 90 : 10, 0.1% TFA
Blank Solution:	H ₂ O : ACN = 90 : 10, 0.1% TFA
Vials:	2 mL standard autosampler vials, crimped. Vials filled to 50 % of nominal content.
Syringe:	100 µL
PAL Cycle Time :	67 seconds

PAL-*xt* Parameter Settings:

CYCLE	LC-Inj DLW Standard Rev01	Pullup Del	500 ms
SYRINGE	100 µL DLW	Inject to	LC Vlv1/2
Sample Volume	7 / 10 µL	Inject Speed	5 µL/s
Airgap Volume	3 µL	Pre Inj Del	500 ms
Front Volume	5 µL	Pst Inj Del	500 ms
Rear Volume	5 µL	Valve Cln Slv2	2 sec
Needle Gap Valve	Clean 3 mm	Pst Cln Slv2	2 sec
Fill Speed	5 µL/s	Vlv Cln Slv1	3 sec
Fill Strokes	0	Pst Cln Slv1	2 sec
Stator Wash	0		

HPLC System Dionex Summit

HPLC Pump: P580, LPG	Flow Rate: 0.5 mL/min (integrated degasser)
HPLC Detector: 340U	Wavelength for detection Caffeine: 273 nm Wavelength for detection Chlorohexidine: 257 nm
Column Repeatability Test:	Zorbax SB-C18, 2.1x50 mm, 1.8 µm
Column Carry-over Test:	Halo C18 2.1x50 mm, 2.7 µm (at ambient temperature, without Column Heater)
Mobile Phase Repeatability Test:	H ₂ O : ACN = 90 : 10 + 0.1% TFA, isocratic
Mobile Phase Carry-over Test:	A: H ₂ O + 0.1% TFA B: ACN + 0.1% TFA Gradient: 0.5 min. 10% B 7.5 min B = 90% hold 1 min B = 90%
Wash Solvents DLW System:	Solvent 1: H ₂ O + 0.1% TFA Solvent 2: ACN + 0.1% TFA

Data Acquisition System: Chromeleon, Software Version 6.8.

HPLC Chromatographic Test: Loop Overflow and Partial Loop Filling DLW-2 Low volume Injection

Specification:

Syringe Volume	Specification	Test Conditions
100 µL	Repeatability Loop Overflow Relative Standard Deviation s-rel : 0.5 %	Loop size 5 µL Loop fill volume: 15 µL
100 µL	Repeatability Loop Overflow Relative Standard Deviation s-rel : 0.5 %	Loop size 2 µL Loop fill volume: 7 µL
100 µL	Repeatability Partial Loop Filling Relative Standard Deviation s-rel : 0.5 %	Loop size 5 µL Loop fill volume: 2.5 µL
100 µL	Repeatability Partial Loop Filling Relative Standard Deviation s-rel : 0.5 %	Loop size 2 µL Loop fill volume: 1 µL
100 µL	Carry-over: Test < 40 ppm	Blank after full loop injection Loop size 2 µL Loop fill volume: 7 µL

General Remarks:

Number of specimens for Repeatability Tests: n = 10 for Loop Overflow

Number of specimens for Repeatability Tests: n = 10 for Partial Loop Filling

Test Conditions:

Test Sample Loop Overflow:	Caffeine, 80 mg/L dissolved in H ₂ O : ACN= 90 : 10 0.1% TFA
Test Sample Partial Loop Filling:	Caffeine, 80 mg/L dissolved in H ₂ O : ACN= 90 : 10 0.1% TFA
Test Sample Carry-over:	Chlorohexidine, 0.6 mg/mL dissolved in H ₂ O : ACN = 90 : 10, 0.1% TFA
Blanks Solution:	H ₂ O : ACN = 90 : 10, 0.1% TFA
Syringe:	100 µL

PAL-*xt* Parameter Settings:

CYCLE	LC-Inj DLW Standard Rev04	Pullup Del	3000 ms
SYRINGE	100 µL DLW	Inject to	LC Vlv1
Sample Volume	1, 2.5, 7, 15 µL	Inject Speed	1 µL/ s
Airgap Volume	1 µL	Pre Inj Del	3000 ms
Front Volume	5 µL	Pst Inj Del	3000 ms
Rear Volume	5 µL	Valve Cln Slv2	2 s
Needle Gap Valve	Clean 3 mm	Pst Cln Slv2	2 s
Fill Speed	1 µL/ s	Vlv Cln Slv1	3 s
Stator Wash	0	Pst Cln Slv1	2 s

Note: For 'Front Volume' and 'Rear Volume' Sample Solution has been used.

HPLC System Dionex Summit

HPLC Pump: P580, LPG	Flow Rate: 0.5 mL/min (integrated degasser)
HPLC Detector: 340U	Wavelength for detection Caffeine: 273 nm Wavelength for detection Chlorohexidine: 257 nm
Column Repeatability Test:	Zorbax SB-C18, 2.1x50 mm, 1.8 µm
Column Carry-over Test:	Halo C18 2.1x50 mm, 2.7 µm (at ambient temperature, without Column Heater)
Mobile Phase Repeatability Test:	H ₂ O : ACN = 90 : 10 + 0.1% TFA, isocratic
Mobile Phase Carry-over Test:	A: H ₂ O + 0.1% TFA B: ACN + 0.1% TFA Gradient: 0.5 min. 10% B 7.5 min B = 90% hold 1 min B = 90%
Wash Solvents DLW System:	Solvent 1: H ₂ O + 0.1% TFA Solvent 2: ACN + 0.1% TFA

Data Acquisition System: Chromeleon, Software Version 6.8.

Injection Precision - Repeatability Relative Standard Deviation

Mean Value (average). Average of measured area counts.

$$\bar{x} = \frac{\sum x_i}{n}$$

\bar{x} = Mean Value of area counts.

x_i = Peak area counts, i th observation of n

n = Number of injections, calibration points.

Standard Deviation

$$SD = \sigma_{n-1} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

σ_{n-1} = Standard Deviation

Relative Standard Deviation

$$\text{Precision \%RSD} = \frac{SD \times 100}{\bar{x}}$$

RSD = Relative Standard Deviation