



Specifications PAL RSI (GC)



Imprint

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1 Gravimetric Specifications

1.1 Repeatability

Specifications:

Syringe Type	Specification
10 µL, Gauge: 22s	RSD < 1 %
10 µL, Gauge: 26s	RSD < 1 %
100 µL, Gauge: 22	RSD < 0.1 %
100 µL, Gauge: 26s	RSD < 0.1 %

Conditions:

n=7 x 8 µL transferred with 10 µL syringe

n=7 x 100 µL transferred with 100 µL syringe

Balance used: Mettler Toledo, New Classic MF, MS105DU (d = 0.01 mg)

PAL Sample Control Method: Gravimetric Precision

Parameter	10 µL Syringe	100 µL Syringe
Number of Vials	7	7
Number of Transfers	1	1
Transfer Volume [µL]	8	100
Fill Speed [µL/s]	1	10
Eject Speed [µL/s]	50	200
Fill Volume [µL]	5	50
Fill Strokes	3	3
Air Volume [µL]	0.5	0
Pullup Delay [ms]	3000	3000
Dispense Delay	NA	NA

1.2 Linearity

Specifications:

Syringe Type	Specification
10 µL, Gauge: 22s	$R^2 > 0.9993$
10 µL, Gauge: 26s	$R^2 > 0.9999$
100 µL, Gauge: 22	$R^2 > 0.9999$
100 µL, Gauge: 26s	$R^2 > 0.9999$
100 µL, LCMS-P Tool	$R^2 > 0.9999$

Conditions:

2, 4, 6, 8, 10 µL transferred with 10 µL syringe, 3 replicates

20, 40, 60, 80, 100 µL transferred with 100 µL syringe, 3 replicates

Balance used: Mettler Toledo, New Classic MF, MS105DU (d=0.01 mg)

PAL Sample Control Method: Gravimetric Linearity

Parameter	10 µL Syringe	100 µL Syringe
Number of Vials	5	5
Number of Transfers	1	1
Volume Increment [µL]	2	20
Fill Speed [µL/s]	1	10 (5)*
Eject Speed [µL/s]	50	200 (20)*
Fill Volume [µL]	5	50
Fill Strokes	3	3 (0)*
Air Volume [µL]	0	0
Pullup Delay [ms]	3000	3000

* Numbers in brackets are valid for the LCMS-P Tool only, all other numbers are valid for standard syringes and LCMS-P Tool.

2 Analytical Data GC Liquid Injection

2.1 Repeatability

Specifications:

Alkane	RSD [Area]
C ₁₄	< 0.6 %
C ₁₅	< 0.6 %
C ₁₆	< 0.6 %

Conditions:

Agilent FID Performance Test Solution, PN: 18710-60170
Evaluation Sample Kit, 0.218 g/L C14 / C15 / C16 in hexane
2 mL vials filled with 0.5 mL sample

PAL Sample Control Method: GC Liquid Injection General

Sample Volume [μL]	1
Air Volume [μL]	1
Vial Penetration Depth [mm]	30
Pre Clean with Solvent 1	3
Pre Clean with Sample	1
Fill Volume [μL]	3
Fill Speed [μL/s]	1
Fill Strokes	5
Pullup Delay [ms]	2000
Injector Penetration Depth [mm]	40
Inject Speed [μL/s]	50
Pre Inject Delay [ms]	4000
Post Inject Delay [ms]	2000
Post Clean with Solvent 1	3

GC Method:

SSL (Split / Splitless Injector Model)

Mode	Split
Heater [°C]	280
Pressure [kPa]	52.268
Total Flow [mL/min]	137.6
Septum Purge Flow [mL/min]	5
Gas Saver [mL/min]	20 After 2 min
Split Ratio	50:01:00
Split Flow [mL/min]	130

Oven

Equilibration Time [min]	0.5
Max Temperature [°C]	300
Temperature Programme	80 °C for 1 min, then 25 °C/min to 200 °C for 1 min

Column

Constant Flow	
Pressure [kPa]	52.268
Flow [mL/min]	2.6
Average Velocity [cm/sec]	50.19

FID

Heater [°C]	300
H ₂ Flow [mL/min]	30
Air Flow [mL/min]	400
Make-up Flow [mL/min]	25

GC Column / GC Injector Liner

Column	Phenomenex ZB5 L:15 m, ID: 0.32 mm, df:0.25 µm, 5 % diphenyl, 95 % dimethyl polysiloxane
Liner	Agilent: Split Liner 4 mm x 6.3 x 78.5, glass wool non deactivated

2.2 Carry-over

Specifications:

Alkane	Carry-over in blank 1 [ppm]	Carry-over in blank 2 [ppm]
C14	< 40	not detectable
C15	< 40	not detectable
C16	< 40	not detectable

Conditions:

Alkane Mix, 6.0 g/L in cyclohexane, 1 µL injected
2 mL vials filled to 50 %

PAL Sample Control Method: GC Liquid Injection General

Sample Volume [μL]	1
Air Volume [μL]	1
Vial Penetration Depth [mm]	30
Pre Clean with Solvent 1	3
Pre Clean with Sample	1
Fill Volume [μL]	3
Fill Speed [$\mu\text{L/s}$]	1
Fill Strokes	5
Pullup Delay [ms]	2000
Injector Penetration Depth [mm]	40
Inject Speed [$\mu\text{L/s}$]	50
Pre Inject Delay [ms]	4000
Post Inject Delay [ms]	2000
Post Clean with Solvent 1	3

GC Method:

Identical Method as for Repeatability

3 Analytical Data GC Headspace

3.1 Repeatability

Specifications:

Syringe	% RSD
Headspace 2500 µL	< 1

Conditions:

n=7

iso-Octane, 5 µL out of micro capillary in a 20 mL headspace vial, sealed immediately
500 µL injected

PAL Sample Control Method: Headspace Injection General

Sample Volume [µL]	500
Incubation Temperature [°C]	90
Incubation Time [s]	600
Syringe Temperature [°C]	100
Fill Speed [µL/s]	100
Pullup Delay [ms]	10000
Pre Inject Delay [ms]	2000
Inject Speed [µL/s]	100
Injector Penetration Depth [mm]	40
Post Inject Delay [ms]	5000
Purge Time [s]	90

GC Method:

Oven

Equilibration Time [min]	0.5
Max Temperature [°C]	300

200 °C for 2.5 min, isotherm

SSL (Split / Splitless Injector Model)

Mode	Split
Heater [°C]	250
Pressure [kPa]	56.204
Total Flow [mL/min]	17.55
Septum Purge Flow [mL/min]	5
Gas Saver [mL/min]	20 After 2 min
Split Ratio	1:250
Split Flow [mL/min]	12.5

GC Column

Constant Flow

Pressure [kPa]	56.204
Flow [mL/min]	0.05
Average Velocity [cm/sec]	12.978

FID

Heater [°C]	300
H ₂ Flow [mL/min]	30
Air Flow [mL/min]	400
Make-up Flow [mL/min]	25

GC Column / GC Injector Liner

Column Agilent: Retention Gap, FS deactivated, 0.100 mm 5 m

Liner Thermo: Straight Split Liner, 990 µL volum, 4 mm ID

3.2 Carry-over

Specifications:

Syringe	Carry-over in blank 1 [ppm]	Carry-over in blank 2 [ppm]
Headspace 2500 µL	< 1000	< 500

Conditions:

iso-Octane, 5 µL out of micro capillary in a 20 mL headspace vial, sealed immediately
500 µL injected

PAL Sample Control Method: Headspace Injection General

Sample Volume [µL]	500
Incubation Temperature [°C]	90
Incubation Time [s]	600
Syringe Temperature [°C]	100
Fill Speed [µL/s]	100
Pullup Delay [ms]	10000
Pre Inject Delay [ms]	2000
Inject Speed [µL/s]	100
Injector Penetration Depth [mm]	40
Post Inject Delay [ms]	5000
Purge Time [s]	90

GC Method:

Identical to Method Repeatability

3.3 GC Needle Discrimination

Specifications	Conditions
Peak area C40/C20 > 0.98	Restek Florida Mix, 1 µL injected, 100 msec fast split/splitless injection

Conditions:

Restek Florida mix PN 31266, C8, C10, C12, C14, ...C40, 500 µg/mL in hexane, diluted to 10 ppm with n-hexane
Syringe: PN: 207837, Hamilton 10 µL, Gauge 26s, 57 mm needle length, Point Style AS

PAL Sample Control Method: GC Liquid Injection General

Sample Volume [μL]	1
Air Volume [μL]	1
Vial Penetration Depth [mm]	30
Pre Clean with Solvent 1	1
Pre Clean with Sample	1
Fill Volume [μL]	3
Fill Speed [μL/s]	1
Fill Strokes	10
Pullup Delay [ms]	2000
Injector Penetration Depth [mm]	35
Inject Speed [μL/s]	fast injection mode
Pre Inject Delay [ms]	fast injection mode
Post Inject Delay [ms]	fast injection mode
Post Clean with Solvent 1	1

GC Method:

Oven

Equilibration Time [min]	0.5
70 °C for 1 min, then 25 °C/min to 340 °C for 1 min	

SSL (Split / Splitless Injector Model)

Mode	Splitless
Heater [°C]	280
Mode	Constant Flow, 2.5 mL/min (He)
Septum Purge Flow [mL/min]	5
Gas Saver [mL/min]	20 after 2 min
Split Flow [mL/min]	80
Splitless Time [min]	0.8

GC Column

Constant Flow	
Flow [mL/min]	2.5

FID

Heater [°C]	330
H ₂ Flow [mL/min]	35
Air Flow [mL/min]	350
Make-up Flow [mL/min]	30

GC Column / GC Injector Liner

Column	Thermo PN 26080001, 5% diphenyl, 95% dimethyl polysiloxane L: 7m, ID: 0.32mm, df: 0.25μm
Liner	Thermo PN 453A1925 (ID 4mm, with glass wool, 4.0 x 6.3 x 78.5)



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