URG-9000D Tips and Observations

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1. Injection Timing and Chromatograms

a. 1-hour sampling rate

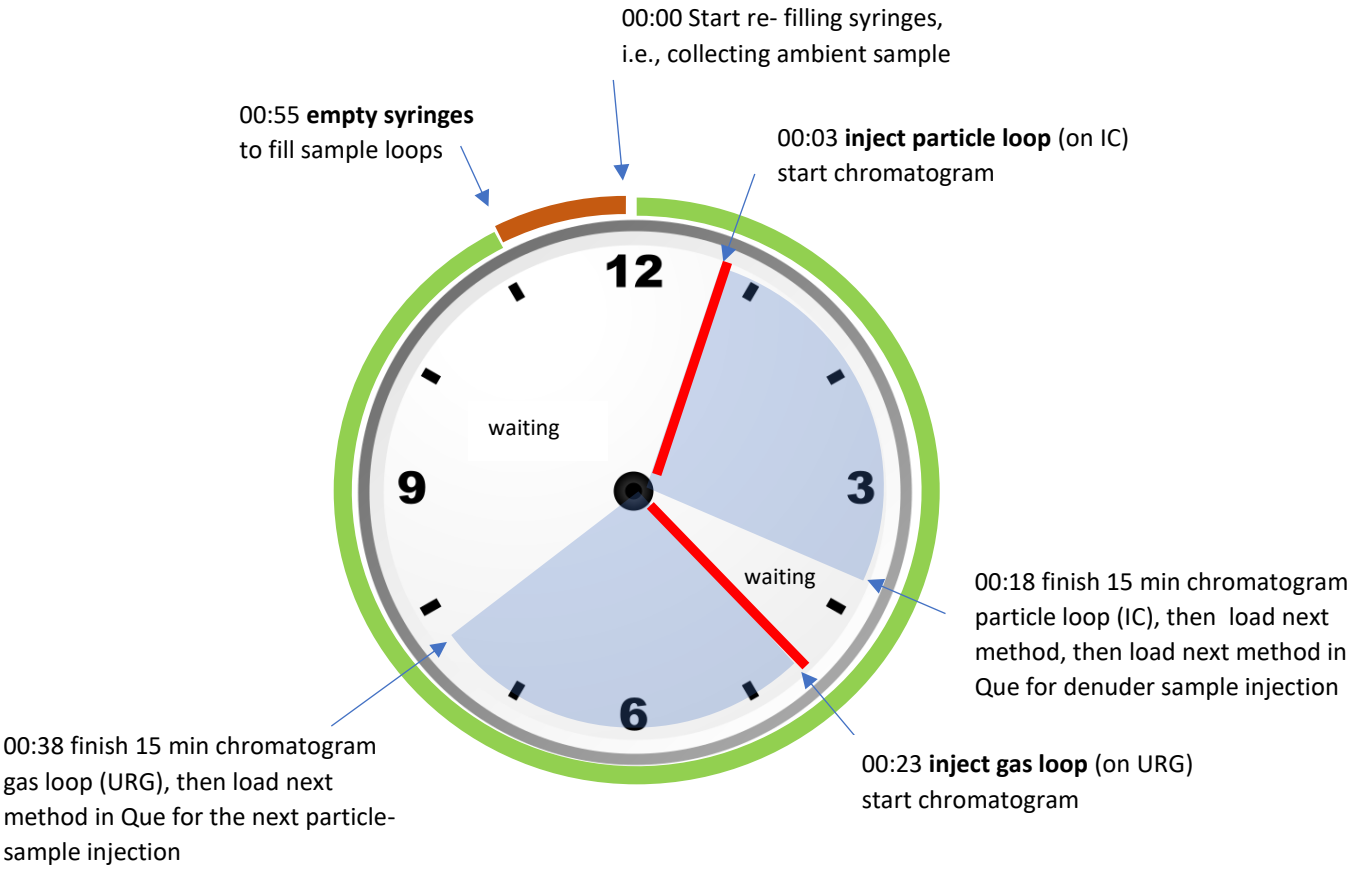
(Timing for 60 minute sampling with 15 minutes chromatograms):

Collecting ambient sample (from droplet cyclone & denuder liquid) into respective syringes

Syringes reverse, transferring collected sample to the respective loops for injection

Chromatogram running (method running*) actual method will be loaded at an earlier time, when the previous method/chromatogram was finished.

Inject sample loop into IC



Text version of the above: please check this

60 min. sample rate (avec 15 min. chromatogram)

(00:00 – 00:55 Syringes are filling with aerosol sample from the liquid cyclone (or denuder).

From 0:55’ to 0:00 the syringes switch direction and push the collected liquid into the loops on the IC and the URG.

Then start loading again at 00:00 until 00:55.

In the meantime:

At 0:03 min: inject sample loop (on IC) – start 15 minute chromatogram for particle anions

At 0:18 min: finish 15 minute chromatogram – particle anions. Load next method in Que, for the upcoming denuder sample injection.

At 0:23 min: inject sample loop (on urg) - start 15 minute chromatogram for gas denuder sample

At 0:38 min: finish 15 minute chromatogram – gas denuder sample. Load next method in Que, for the upcoming particle sample injection.

From 00:38 – 00:55: Waiting only until Syringes are filled again at 0:55 min. Then loops are filled.

(the same happens for 27 minute chromatograms, aside from the inject timing. Same sample loop volume injected.)

b. 30-minute sampling rate

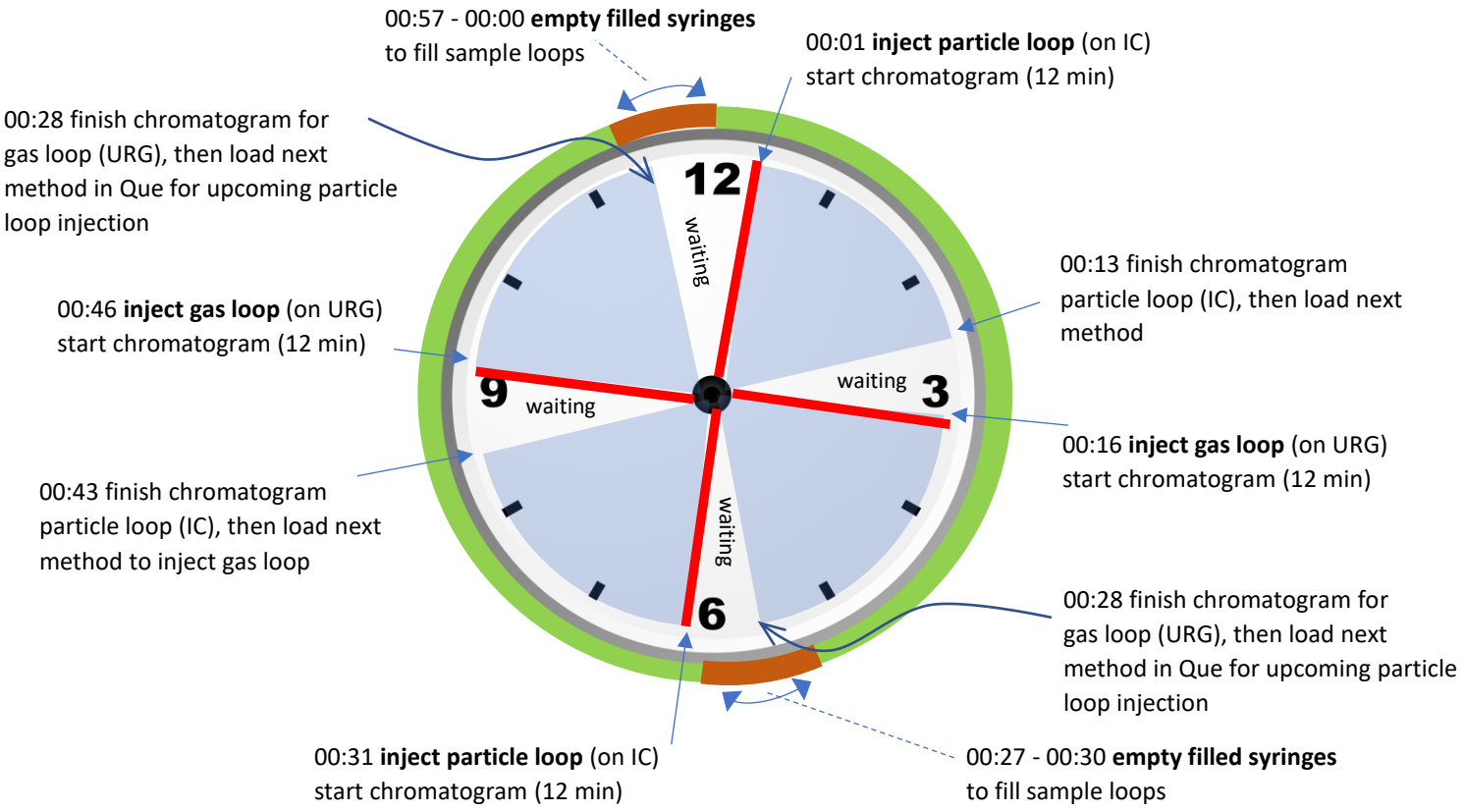
(with 12 minute chromatograms)

Collecting ambient sample (from droplet cyclone & denuder liquid) into respective syringes

Syringes reverse, transferring collected sample to the respective loops for injection

Chromatogram running (method running*) actual method will be loaded at an earlier time, when the previous method/chromatogram was finished.

Inject sample loop into IC



Text version of above (please check):

Syringes are filling with aerosol sample from the liquid cyclone, from time 0:00 to 00:28'. From 0:28' to 0:31 they switch direction and push the sample into the loops on the IC and the URG.

Then start loading again.

At 0:01 min: inject sample loop (on IC) – start 12 minute chromatogram for particle

At 0:13 min: finish 12 minute chromatogram – particle. Load next method in Que, for the upcoming denuder/gas sample injection.

At 0:16 min: inject sample loop (on urg) - start 12 minute chromatogram for gas denuder sample

At 0:28 min: finish 12 minute chromatogram – gas. Load next method in Que, for the upcoming particle sample injection.

… loops are filled again until 27 min.

------ one 30 minute measurement completed

At 0:31 min: inject next sample loop (on IC) – start 12 minute chromatogram for particle

At 0:43 min: finish 12 minute chromatogram – particle. Load next method in Que, for the upcoming denuder/gas sample injection.

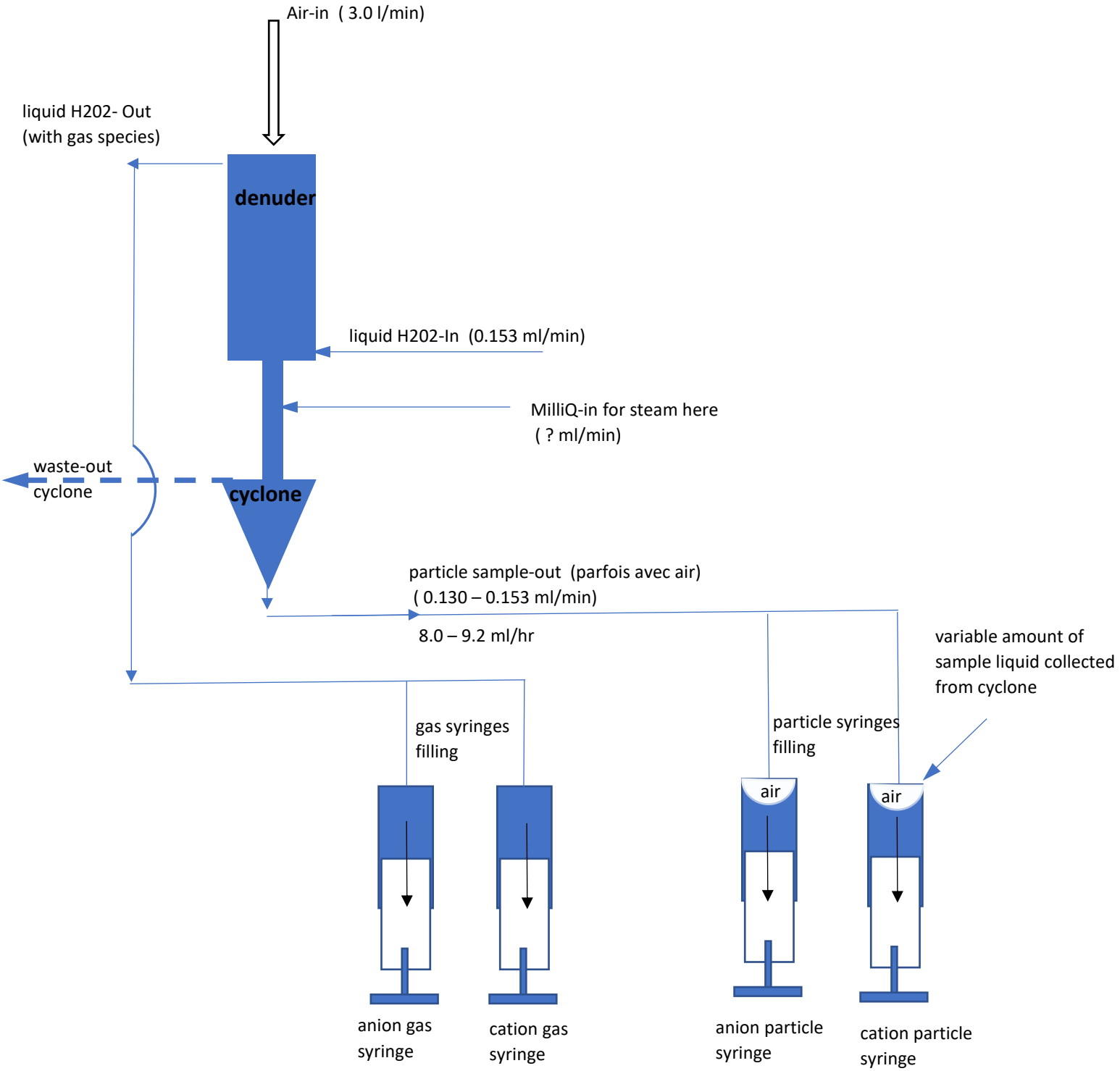
At 0:46 min: inject sample loop (in urg) - start 12 minute chromatogram for gas denuder sample

At 0:58 min: finish 12 minute chromatogram – gas. Load next method in Que, for the upcoming particle sample injection.

… loops are filled again until 00 min.

2. Flow diagrams

a. syringes are collecting sample



for 1 hour sampling approx volume of liquid collected = 9 ml

for 30 minute sampling approx volume = 4.7 ml

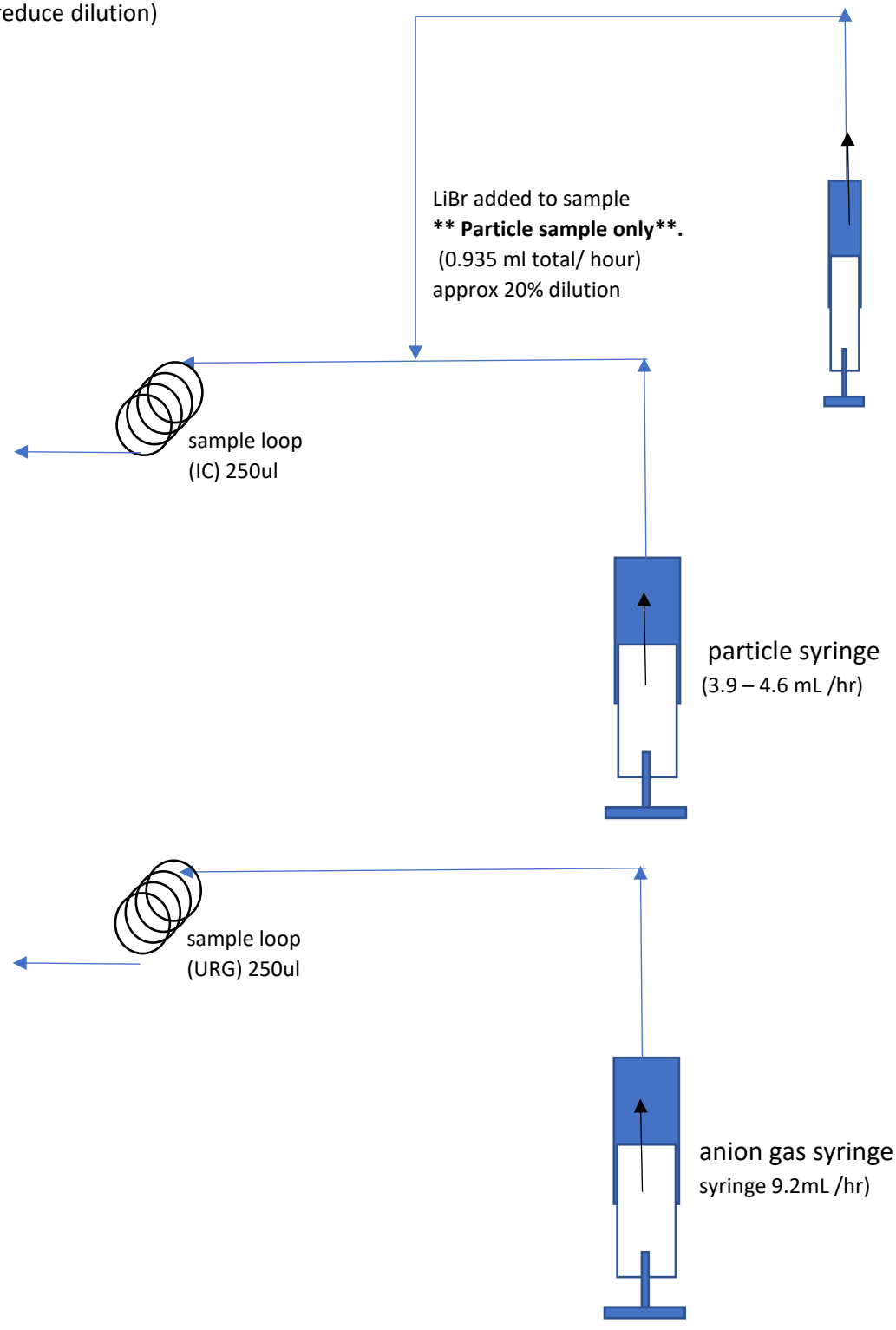
Total Collected Liquid Vol/ Hr
= 8.0-9.2 ml/hr
approx max 18% dilution
effect, not measured

2. Flow diagrams (cont'd)

b. syringes are transferring collected sample to loops or concentrator columns

(Shown here for 1 syringe.)

(Note: the Libra was disconnected to reduce dilution)



for 250 ul sample loops:
0.250ml of the 8 or 9 mL
collected. Or .03125 - .0272

3. Timing Issues

Below are example times recorded by URG in 30 minute sample runs (12 min chromatogram cycles):

Times saved in Report Designer

Injection Name	Sample Start Time	Sample End Time	Inject Time	**Actual****
1-Particle	7/7/2021 11:00:00 AM	7/7/2021 11:30:00 AM	07.07.21 10:31	??
2-Gas	7/7/2021 11:00:00 AM	7/7/2021 11:30:00 AM	07.07.21 11:43	11:46
3-Particle	7/7/2021 11:30:00 AM	7/7/2021 12:00:00 PM	07.07.21 11:58	12:01
4-Gas	7/7/2021 11:30:00 AM	7/7/2021 12:00:00 PM	07.07.21 12:13	12:16
1-Particle	7/7/2021 12:00:00 PM	7/7/2021 12:30:00 PM	07.07.21 12:28	12:31
2-Gas	7/7/2021 12:00:00 PM	7/7/2021 12:30:00 PM	07.07.21 12:43	12 :46
1-Particle	7/7/2021 12:30:00 PM	7/7/2021 1:00:00 PM	07.07.21 12:58	13 :01
4-Gas	7/7/2021 12:30:00 PM	7/7/2021 1:00:00 PM	07.07.21 13:13	13:16

** **Actual (real) times** are NOT recorded

Pay attention to the above recorded times. There are a few problems.

a. Injection times are not correct. Instead they represent when the previous method (chromatogram) finishes and the new method is loaded. This is called the “injection time”, when the new method is loaded. After loading, the URG is waiting to inject and begin a new chromatogram.

If you use these, you must correct them. In this case (30 minute sampling) the offset is three minutes. But in the hour sampling case, they alternate See diagram #1 on the timing for 1-hour chromatograms.

b. Initial particle injection time is often not correct, no matter what you do.

c. “Sample start time” and “sample end time” - IMPORTANT

As an example, in the case of 30 minute sampling, the particle sample stop and end times should be the half hour interval before the injection. In order to achieve this, the following steps must be followed. (Otherwise the times recorded will not be correct.)

Before starting a new que, open the control panel for the URG

Press “ Stop Immediately” (option to start at top of next hour)`

Then start the Que.

If you do not do this, the Sample start and End times will be wrong and shifted.

You could just forget all this and use the injection times only, keeping in mind that they must be corrected also.

d. Syringe movement timing (for the previous same injections)

Inject Name	Sample Start Time	Sample End Time	Inject Time	Actual	**Syringe movement**
1-Particle	7/7/2021 11:00:00 AM	7/7/2021 11:30:00 AM	07.07.21 10:31	??	
2-Gas	7/7/2021 11:00:00 AM	7/7/2021 11:30:00 AM	07.07.21 11:43	11:46	
					11:57 empty to loops
					12:00 begin filling
3-Particle	7/7/2021 11:30:00 AM	7/7/2021 12:00:00 PM	07.07.21 11:58	12:01	
4-Gas	7/7/2021 11:30:00 AM	7/7/2021 12:00:00 PM	07.07.21 12:13	12:16	
					12:27 empty to loops
					12:30 begin filling
1-Particle	7/7/2021 12:00:00 PM	7/7/2021 12:30:00 PM	07.07.21 12:28	12:31	
2-Gas	7/7/2021 12:00:00 PM	7/7/2021 12:30:00 PM	07.07.21 12:43	12 :46	
					12:57 empty to loops
					13:00 begin filling
1-Particle	7/7/2021 12:30:00 PM	7/7/2021 1:00:00 PM	07.07.21 12:58	13 :01	
4-Gas	7/7/2021 12:30:00 PM	7/7/2021 1:00:00 PM	07.07.21 13:13	13:16	
					13:27 empty to loops
					13:30 begin filling

4. Thoughts on possible sampling and dilution problems in the URG

(this comes after what seems to be (based on prelim data) a 10-15% under-representation of the sampled mass.)

Case 1 :

Cyclone loses mass, i.e., collected droplets exit out exhaust of liquid cyclone instead of being pulled-out the bottom. This is definitely the case when no air is pulled out the bottom of the liquid cyclone and syringes are full. One possible solution is to lower the steam input rate.

result: -for concentrator column, total mass is not collected and lost

-for sample loop, same, but only if mass collected in sample-liquid cyclone, is not well-mixed.

Case 2:

Liquid collected in vertical column in syringe is not well mixed, i.e., there is layering of species inside the syringe.

result: - for concentrator column, no effect since all liquid (and mass) is injected.

- for sample loop, the volume interval of loop may not represent the sample avg. though the length of the syringe column

Case 3:

Dilution Factor: URG does not measure the dilution factor caused by varying temperature and humidity conditions. The only way to have an idea of the liquid volume of the sample is to observe the volume collected in the syringes. Often the particle syringes do not contain air, which means they are not collecting all the mass (see case 1). In observing the range of the syringes, a full syringe collects max. 4.6 mL (for a 1 hour measurement). For 30 minutes it collects max 2.35 mL. These are estimates based on observations.

***Note:**

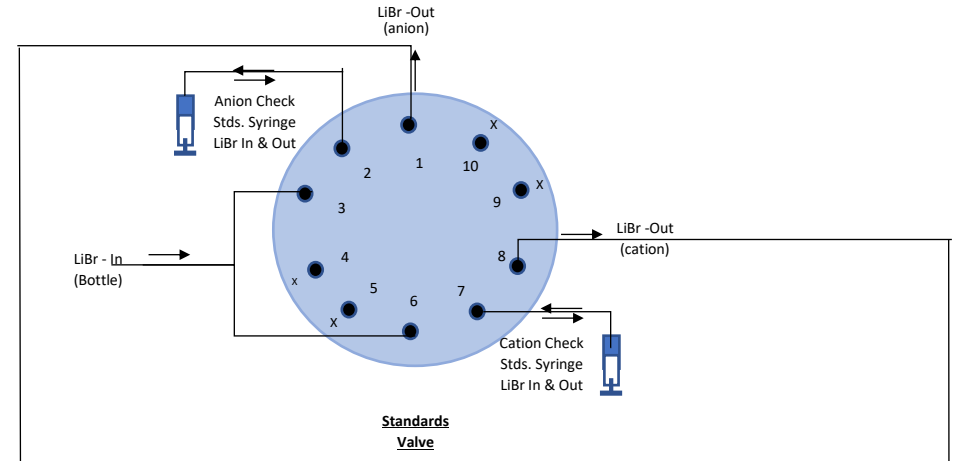
The "dilution factor" mentioned in the URG manual is not really a dilution factor. It is a multiplication factor to account for the analyzed volume/mass as a fraction of the total liquid collected.

For loops: the loop volume as a fraction of the total collected liquid (for 250 ul loops, this is approx 35.2),

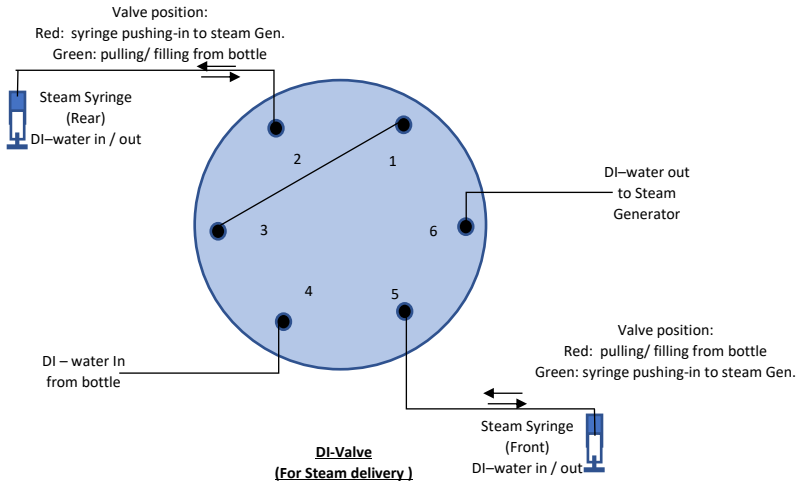
For concentrator columns: the sample is split into two channels(factor of 2.)

5. General Valve-flow Configurations on the URG

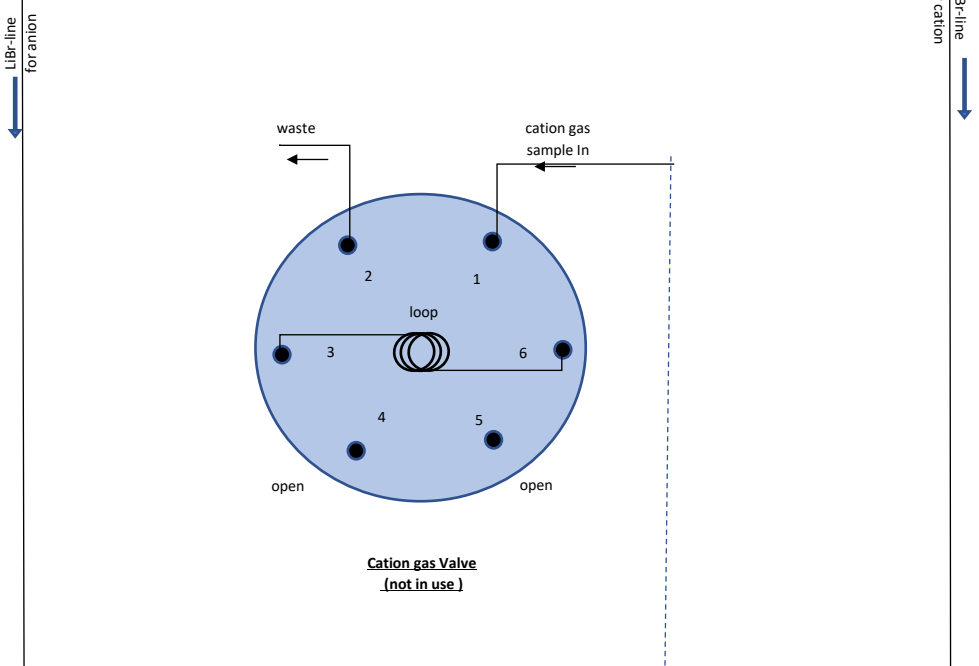
a. Valve configurations and connections



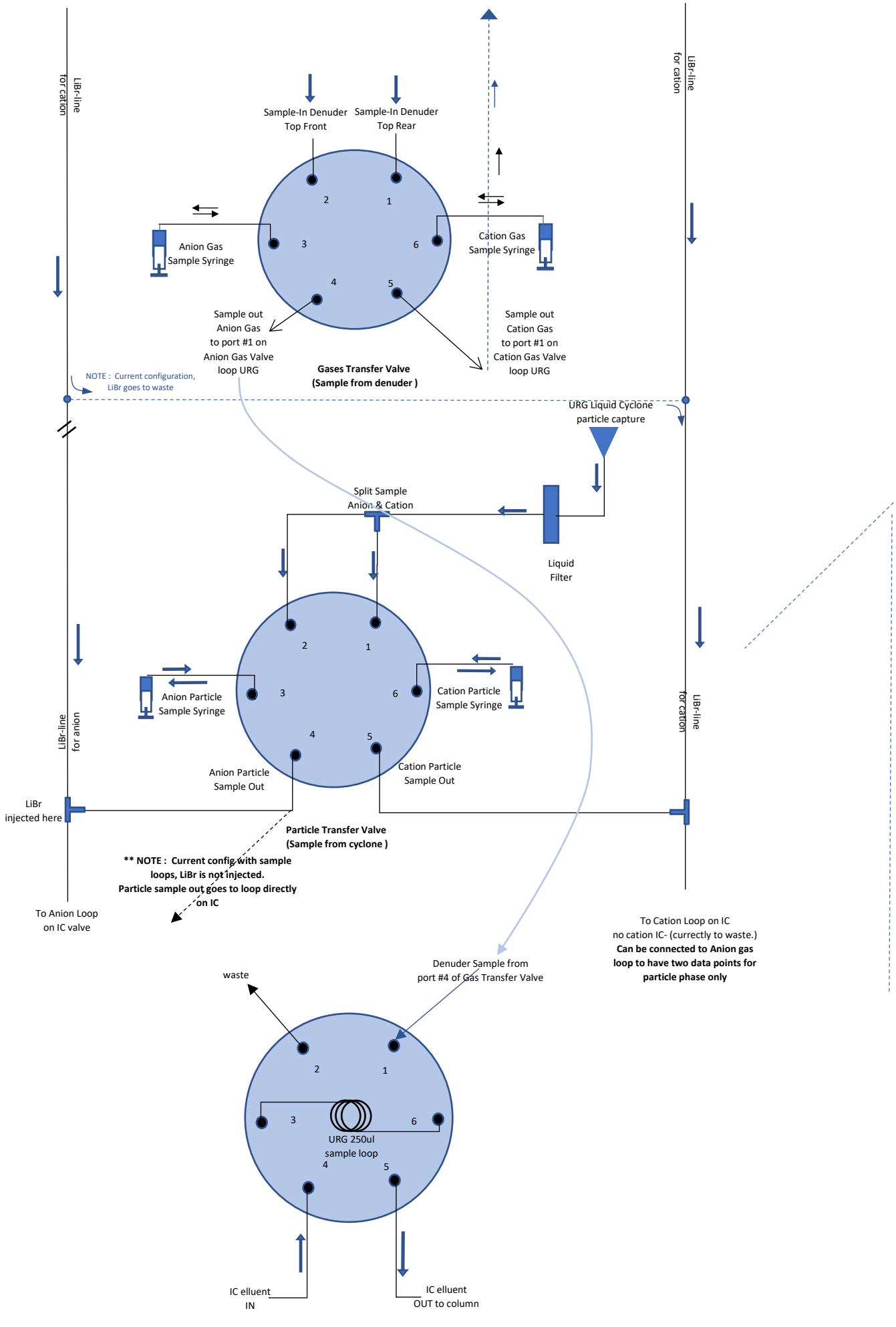
Standards Valve



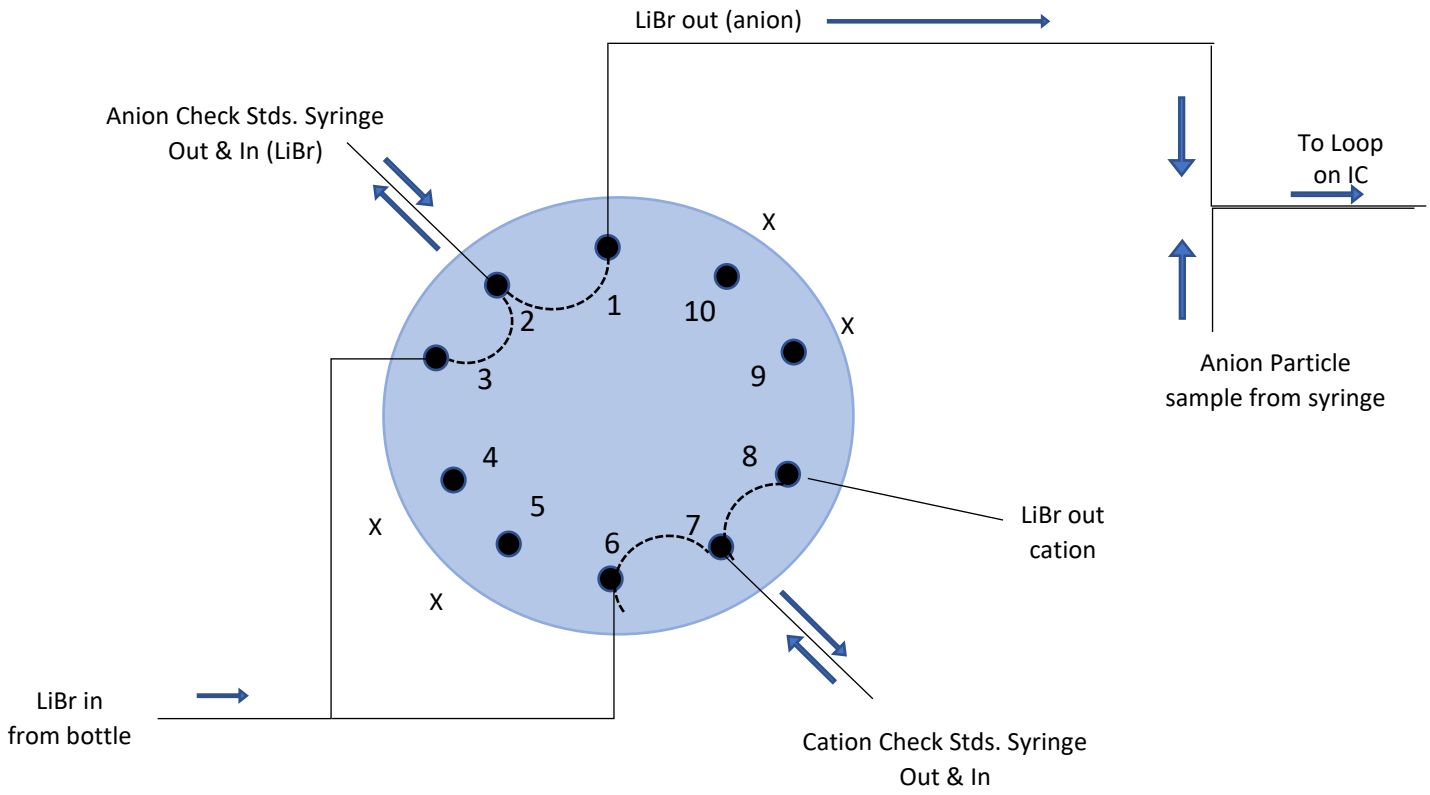
DI-Valve (For Steam delivery)



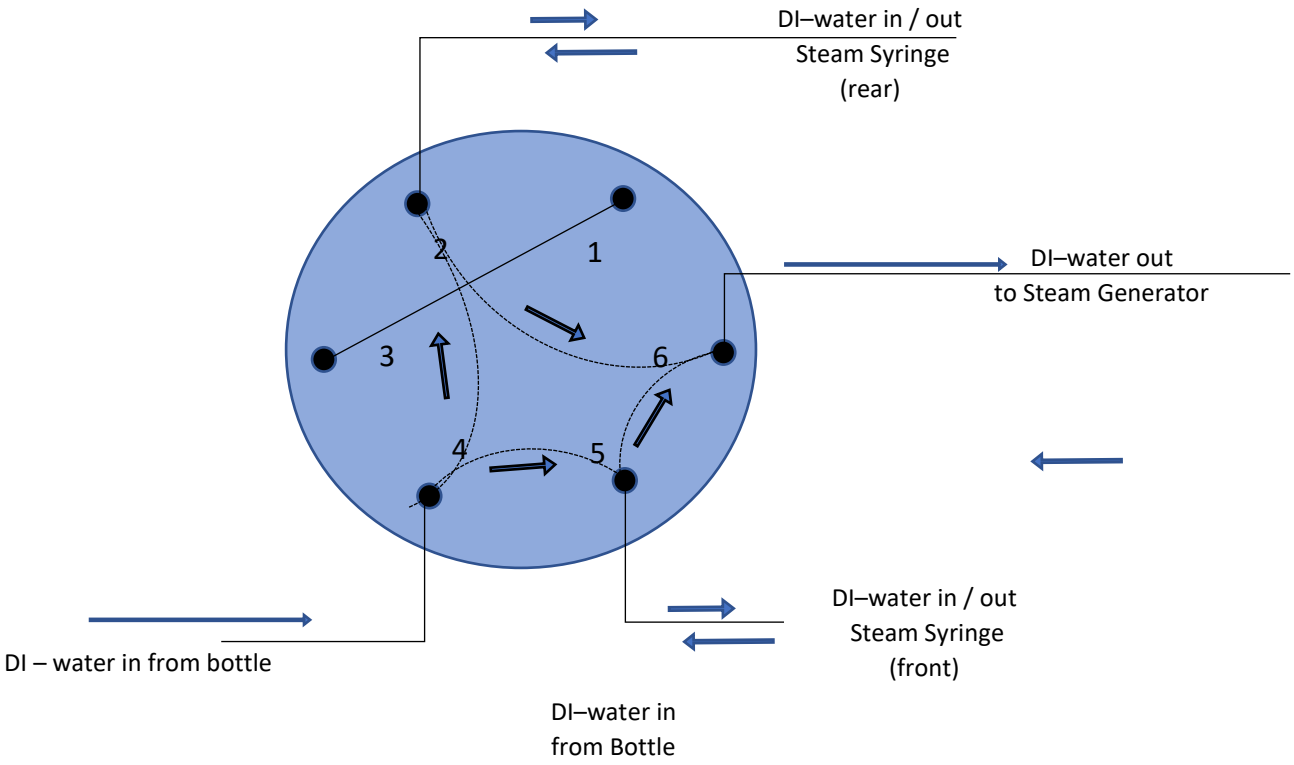
Cation gas Valve (not in use)


LiBr-line for cation
Sample-In Denuder Top Front
Sample-In Denuder Top Rear
2
1
3
6
4
5
Anion Gas Sample Syringe
Cation Gas Sample Syringe
Sample out Anion Gas to port #1 on Anion Gas Valve loop URG
Gases Transfer Valve (Sample from denuder)
Sample out Cation Gas to port #1 on Cation Gas Valve loop URG
NOTE : Current configuration, LiBr goes to waste
URG Liquid Cyclone particle capture
Split Sample Anion & Cation
Liquid Filter
Anion Particle Sample Syringe
Cation Particle Sample Syringe
LiBr-line for anion
Anion Particle Sample Out
Cation Particle Sample Out
LiBr injected here
Particle Transfer Valve (Sample from cyclone)
** NOTE : Current config with sample loops, LiBr is not injected. Particle sample out goes to loop directly on IC
To Anion Loop on IC valve
To Cation Loop on IC
no cation IC- (currectly to waste.)
Can be connected to Anion gas loop to have two data points for particle phase only
Denuder Sample from port #4 of Gas Transfer Valve
waste
URG 250ul sample loop
IC elluent IN
IC elluent OUT to column

5a. Individual Valves (just guesswork)

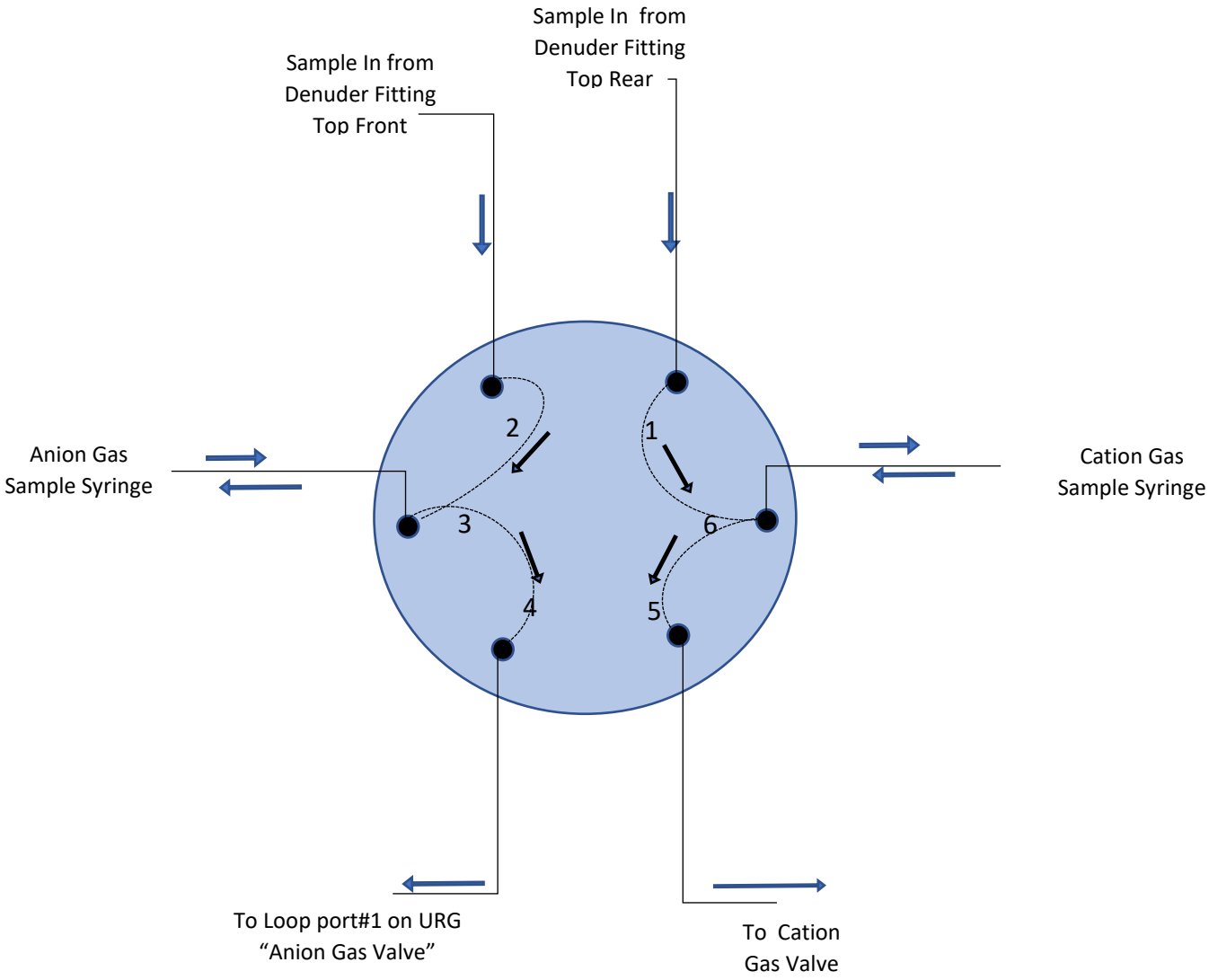


Standards
Valve
(For delivery of Lithium Bromide)



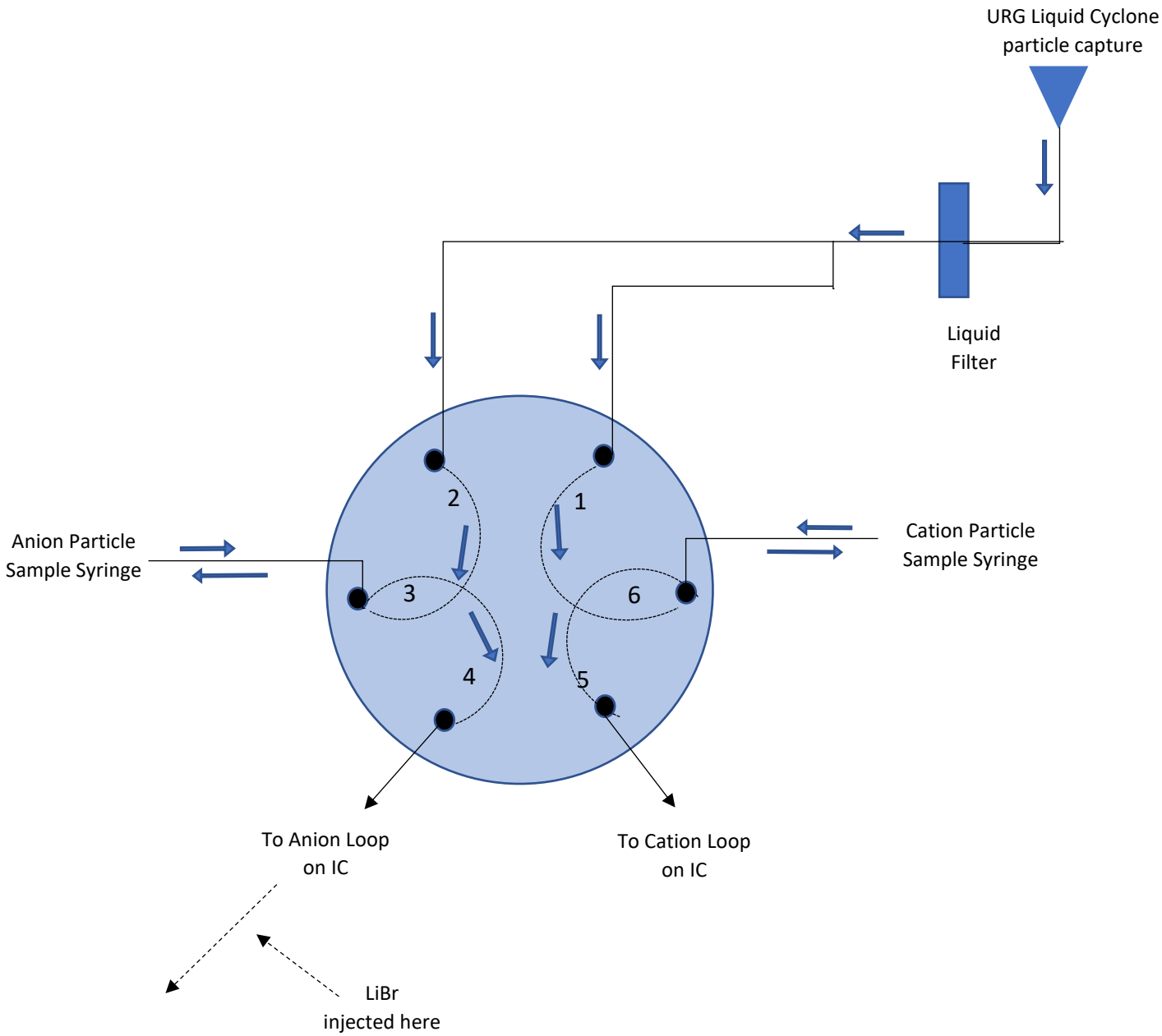
DI-Water

(For Steam delivery)

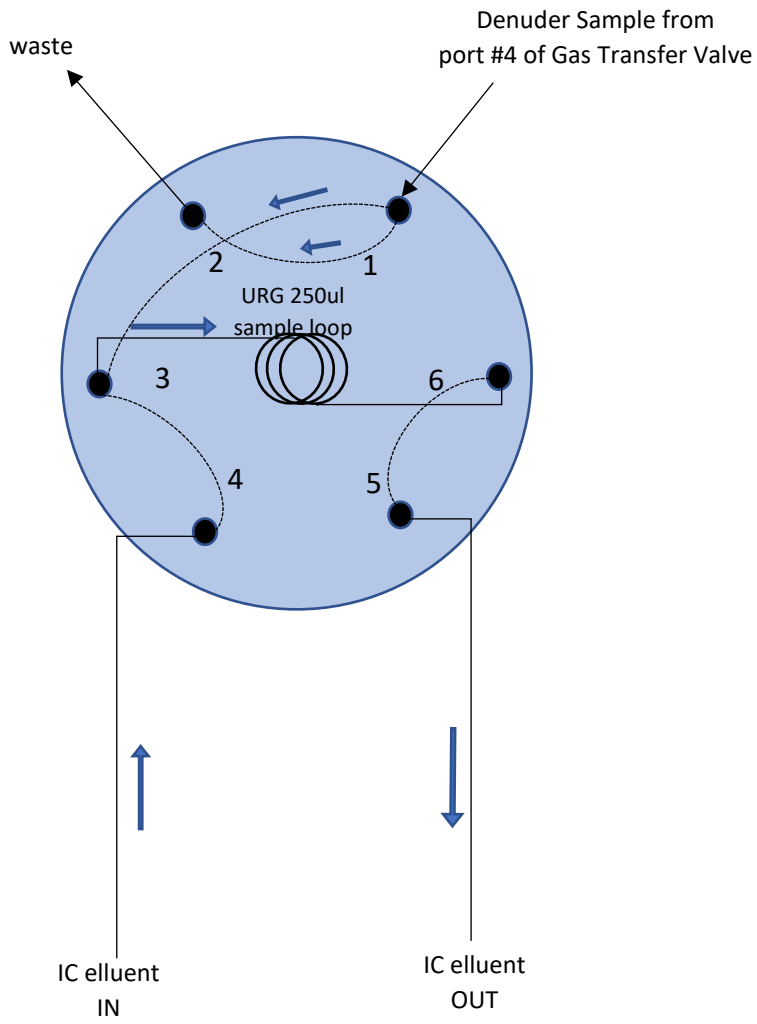


Gases Transfer Valve

(Sample from denuder)



Particle Transfer Valve
(sample from liquid cyclone)



Anion Gas Valve
(sample from denuder)

6. The report designer

The report designer is the interface containing the URG-data and what can be exported as a text or excel file.

Normally configured (see manual), the report designer outputs peak and concentration data after each injection.

Plots can also be generated. The template report designer provided by URG contains tabular data, charts, and graphs.

The user can change (and should) the formulas in the spreadsheet according to preference.

The main issue with the report designer is the CPU/ memory it requires.

It can easily disrupt and seize the chromeleon software.

Therefore make your own report designer as efficient as possible.

I personally removed all graphs, charts, and unnecessary peak data.

**** Note:**

Running a Sequence in the Que with more than a single day of injections appears to burden the report designer and software significantly. Even adding blank injections to a sequence (to be completed) will slow the data processing significantly.