7. FILLING PROCEDURE DATA SHEET

7.1 Preparation procedures

Preparation procedures							
Date: 27/10/2009		Company: SYSU		Location: CERN		Engineer: SXU / ZZ.	
Step No.	Action and Description			Monitoring	Value	Result	Comment	Time
1.	Take by hand environment conditions							
2.	Temperature: □	Humidity: %RH	Pressure: MPa					
3.	Calibrate the volume of the filling box;(Vbox= 0.021 liter)							
4.	Calculate the volume of the two connecting tubes and valves;							
5.	Select filling environmental temperature, e.g., 22°C;			T_{room}				
6.	Wrap a heating tape around the filling tube outside the box;							√
7.	Prepare an electronic balance with full scale more than 4200g and accuracy of 0.1g;							√
8.	Prepare a bottle of pure N_2 gas for volume calibration							
9.	Prepare high purity (99.99%)CO_2 bottle for filling;							√
10.	Prepare a barrel with ice water mixture for cooling the vessel;							√
11.	Prepare a fan for cooling TTCS accumulator during filling;							√
12.	Connect a dry mechanical pump to the vacuum connector;							√

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7.2 Filling system leak rate examination — Done on 20/10/2009

Filling system leak rate examination

Date: 20/10/2009 **Company:** SYSU **Location:** CERN - CMS area. **Engineer:** SXH/ZZ.

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
1.	Take by hand environment conditions					
2.	Temperature: □ Humidity: %RH Pressure: MPa					
3.	Connect the two SV 1.5s to connector 1 and connector 3, respectively. Close valve 2.					
4.	Measure the background leak rate of the "He mass spectrometer"		$<5\times10^{-10}$ mbar·l/s.	$<1.0\times10^{-10}$ mbar l/s.		12:02
5.	Connect the He mass spectrometer to the vacuum connector					
6.	Open valve1, valve 3 and valve 4 Vacuum the filling system					
7.	Measure the leak rate of the filling system without helium gas.		$<5\times10^{-8}$ mbar·l/s.	9.4×10^{-9}		15:15
8.	Using negative method, blowing helium gas, to measure the leak rate of these connectors, valves. Leak rate for each connector should be below 5×10^{-5} mbar·l/s.		$<5\times10^{-5}$ mbar·l/s.	Values on filling system: $LR_{max} = 5.7\times10^{-9}$. SV1.5(N2) Value $LR = 5.7\times10^{-8}$		
9.	Disconnect the He mass spectrometer and measure the background leak rate of the equipment.		$<5\times10^{-10}$ mbar·l/s.	SV1.5(CO2) Value $LR = 6.0\times10^{-9}$ ∴ replace the valve on SV1.5(CO2). Leak test again. $LR = 5.2\times10^{-8}$ mbar·l/s		

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Filling system leak rate examination

Date: 20/10/2009 **Company:** SYSU. **Location:** CERN-CMS (area) **Engineer:** SXH/ZZ.

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
10.	**Flash the filling system**					
11.	Connect the N_2 bottle to the connector 2					
12.	Open the bottle valve and valve 2 Pressurize the filling system slowly		40±1bar	10bar		10:53
13.	Close valve 2 and open vacuum valve Release the N2 from the system slowly		2±0.5bar			
14.	Open the bottle valve and valve 2 Pressurize the filling system slowly		40±1bar	8bar		10:57
15.	Close valve 2 and open vacuum valve Release the N2 from the system slowly		2±0.5bar			
16.	Open the bottle valve and valve 2 Pressurize the filling system slowly		40±1bar	10bar		11:04
17.	Close valve 2 and open vacuum valve Release the N2 from the system slowly		2±0.5bar			

pressure regulator max is 10bar.

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7.3 Determination of filling system volume

Done with TTCS volume Determinatio

Filling system volume determination							
Date:		Company:		Location:		Engineer:	
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: ☐ 22℃.	Humidity: %RH	Pressure: MPa					
3.	Affix any two of the three PT1000s on the side surface of the SV1.5. and leave the rest one to monitor environmental temperature.							
4.	Connect the SV1.5 (any of the three stand vessels can be used) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **Note: The extend tube between connectors and valves must be supported to reduce strain.** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)							✓
5.	Connect the N_2 bottle to connector 2;							✓
6.	Connect a dry mechanical pump to vacuum connector							✓

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Filling system volume determination						
Date:		Company:	Location:		Engineer:	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
7.	seal the extend filling tube with the other end connected to connector 1 of the filling box; Note: the need of redetermination of the filling system is due to the use of additional extend filling tubes, therefore, the extend filling must be connected to the filling box with the other end sealed. On the other hand, we could skip this procedure if we can rely on the calculation of the volume of the additional extend filling tubes. Then : $V_{FT}=V_{filling_system}+V_{C_extend\ tubes}$		s	Done with TTCS Loop volume determination.		
8.	Close valve 1, valve 4 and the N_2 bottle valve, open valve 2, valve 3, the SV1.5 valve, pressure regulator and the vacuum valve slowly, pump the SV1.5 and the filling box down to 30Pa;	Evacuate Time Vacuum gauge reading	1~2 hours ≤30Pa			
9.	Close the vacuum valve open the valve of the N_2 bottle					

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Filling system volume determination						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
10.	Fill the SV1.5 with N_2 up to 4.5MPa, then close valve 2; wait until pressure and temperature of the standard vessel is stable. Take by hand the pressure as **P1'.** Take by hand the average reading of the three PT1000s in the filling system as T_1'. (here T_1' is comparable to T_{room} with range of $\pm 1^oC$);	Evacuate Time **P1'** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	0.5~1hours $T_{room}\pm 1^oC$ $T_{room}\pm 1^oC$ $T_{room}\pm 1^oC$			
11.	Close the filled SV1.5 valve, valve 3, and the N_2 bottle valve, disconnect the filled SV1.5 from connector 3;					
12.	Weight the filled SV1.5 with an electronic balance (0.1g precision or better), as $M'_{nitrogen1}$;					
13.	Connect the filled SV1.5 back to the connector 3;					
14.	Open valve 3, valve 1,vale 4 and the vacuum valve; open pressure regulator to pump the filling box down to 30Pa;					
15.	Close the vacuum valve, valve 1 and valve 4, switch off the pump. Open the SV1.5 valve and close SV1.5 valve, slowly open valve 1 to fill the N_2 into the filling box and tubes.	DPS reading of the TTCS loop	≤0.5Bar			

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Filling system volume determination						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**		**Monitoring**	**Value**	**Result**	**Comment**	**Time**
16.	Wait about ten mins, take by hand the pressure as **P2'** and the average reading of the three PT1000s in the filling system as T_2'. (here T_2' is comparable to T_{room} with range of ±1°C);		**P2'** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	 T_{room}±1°C T_{room}±1°C T_{room}±1°C			
17.	Close valve 1, valve 3, SV1.5 valve;						
18.	Disconnect SV1.5 from the connector 3;						
19.	Weight the SV1.5 with the electronic balance again, as $M'_{nitrogen2}$;						
20.	The nitrogen mass inside the filling system including tubes is: $M_{FT}= M'_{nitrogen1} - M'_{nitrogen2}$,						
21.	The volume of the filling system including tubes is determined by the ideal gas equation , where $V_{FT} = \frac{M_{FT} R T_2'}{m_{nitrogen} P_2'}$ $m_{nitrogen}$= 28g/mol for nitrogen, and R is the gas constant;				$V_{external}$ =	0.026L.	

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7.4 Determination of TTCS volume

TTCS volume determination							
Date: 27/10/2009		Company: SYSU		Location: CERN		Engineer: XHS/ZZ.	
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
22.	Take by hand environment conditions							
23.	Temperature: □ 22°C.	Humidity: %RH	Pressure: MPa					
24.	Affix any two of the three PT1000s on the side surface of the SV1.5. and leave the rest one to monitor environmental temperature.			Connectors Leak rate: 1. Vacuum the filling system and connected tubes.				13:51.
25.	Connect the SV1.5 (any of the three stand vessels can be used) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **Note: The extend tube between connectors and valves must be supported to reduce strain.** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)			2. LR = 4.9×10⁻⁹ mbar l/s.	Background	LR = 5.0 × 10⁻⁹ mbar l/s.		13:55.
26.	Connect the N_2 bottle to connector 2;							✓

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TTCS volume determination

Date: 27/10/2009	Company: SYSU	Location: CERN	Engineer: SYh/ZZ

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
27.	Connect a dry mechanical pump to vacuum connector					✓
28.	Connect the TTCS loop to connector 1; Longer extend tubes may be used and height difference may exist between the TTCS loop and connector 1. **Note: The extend tube between connectors and valves must be supported to reduce strain.**		N_2 bottle with N_2 inside, no flush for it.			✓
29.	Close valve 1, valve 4 and the N_2 bottle valve, open valve 2, valve 3, the SV1.5 valve, pressure regulator and the vacuum valve slowly, pump the SV1.5 and the filling box down to 30Pa;	Evacuate Time Vacuum gauge reading	1~2 hours ≤30Pa	vacuum. 18pa. flush 1mpa. vacuum. 29pa.		14:13 14:14 14:18
30.	Close the vacuum valve open the valve of the N_2 bottle			flush 1.25mpa.		14:19
31.	Fill the SV1.5 with N_2 up to 4.5MPa, then close valve 2; waiting until pressure and temperature of the standard vessel is stable. Take by hand the pressure as **P1.** Take by hand the average reading of the three PT1000s in the filling system as T_1. (here T_1 is comparable to T_{room} with range of ±1°C);	Evacuate Time **P1** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	0.5~1hours T_{room}±1°C T_{room}±1°C T_{room}±1°C	Vacuum 29pa. Start room temp T_1 = 22.9 T_2 = 22.1 T_3 = 23.0 pressurize to 5.4mpa	pressurize	14:25 14:27 14:30

T_1 = 25.7 —— N_2 bottle
T_2 = 22.0 —— room.
T_3 = 25.6 —— N_2 bottle

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TTCS volume determination

Date: 27/10/2009	Company: SYSU.	Location: CERN	Engineer: SXh./22

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
32.	Close the filled SV1.5 valve, valve 3, and the N_2 bottle valve, disconnect the filled SV1.5 from connector 3;				$T_1 = 23.2$ $T_2 = 22.4$ $T_3 = 23.2$.	14:59
33.	Weight the filled SV1.5 with an electronic balance (0.1g precision or better), as $M_{nitrogen1}$;			$M_{nitrogen1} = 2850.3g$.		
34.	Connect the filled SV1.5 back to the connector 3;		Check leak rate of connector to SV1.5.			
35.	Open valve 3, valve 1,vale 4 and the vacuum valve; open pressure regulator to pump the filling box and TTCS loop down to 30Pa;		BG = 7.9×10^{-9} mbar l/s. LR = 7.6×10^{-9} mbar l/s.			15:16.
36.	Close the vacuum valve, valve 1 and valve 4, switch off the pump. Open the SV1.5 valve and close SV1.5 valve, slowly open valve 1 to fill the N_2 into the TTCS loop to prevent pressure spike to the sensors. Repeating on and off the SV1.5 valve and valve 1 in turn until pressure of the TTCS loop is up 1.5MPa, and then slowly open the SV1.5 valve and valve.	DPS reading of the TTCS loop	≤0.5Bar	Vacuum 15 pa. finish 1.5 Mpa. Vacuum 14 pa		15:25
			extended tube volume determination			15:37
			stable state:	T_{room} = 22.5°C.		

$T_{thermocouple}$ = 22.2 °C.

P_{final} = 5.096 Mpa. 15:42.

Disconned SV1.5 and weight SV1.5: $M_{nitrogen1}$ = 2846.3g.

check leak rate of connector to SV1.5. BG = 1.0×10^{-8} mbar l/s

LR = 9.5×10^{-9} mbar l/s.

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Vacuum TTCS loop. 15:59. 23 pa. 16:35

pressrize to 1.1 Mpa.

vacuum TTCS Loop 16:41
Pa = .19 Pa 17:24
pressrize N_2 from SV1.5 to TTCSloop 17:36

TTCS volume determination

Date: 27/10/2009	Company: SYSU	Location: CERN	Engineer: SXh / ZZ

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
37.	Wait a period of time (0.5~1hours), take by hand the loop pressure as **P2** and the average reading of the three PT1000s in the filling system as T_2. (here T_2 is comparable to T_{room} with range of ±1°C);	**P2** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	 T_{room}±1°C T_{room}±1°C T_{room}±1°C	All pt1000 22~23°C All Dallas 22.19~22.5°C on filling system T_1 = 22.9°C T_2 = 22.4°C T_3 = 23.0°C	P_{final_2} = 2.553 MPa	18:23
38.	Close valve 1, valve 3, SV1.5 valve and the TTCS valve;					
39.	Disconnect SV1.5 from the connector 3;			$M_{nitrogen2}$ = 2802.6g		
40.	Weight the SV1.5 with the electronic balance again, as $M_{nitrogen2}$;	External tube Volume = $M_{nitrogen1} - M_{nitrogen0}$ = 2850.3 − 2846.3 = 4g				
41.	The nitrogen mass inside the loop and the filling system is: $M_{nitrogen} = M_{nitrogen1} - M_{nitrogen2}$,	$P_{final_1} V = \frac{M}{m} RT$	$5.096 \times 10^{-3} V = \frac{4}{28} \times 8.314 \times 295.6$			
42.	The volume of the loop is determined by the ideal gas equation , where $V_{Loop} = \frac{M_{nitrogen} R T_2}{m_{nitrogen} P_2} - V_{FT}$ $m_{nitrogen}$= 28g/mol for nitrogen, and R is the gas constant;			∴ $V_{exter+fillingsystem}$ = 0.068L (~~15.678%~~) ~~$V_{TTCSloop}$ = 1.433L~~ (~~15.874%~~) ∵ $V_{fillsys}$ = 0.042L ∴ $V_{external}$ = 0.068 − 0.042 = 0.026L		
43.	Determine the fill mass by $M_{C_L} = V_{Loop}$FR (where FR = 569.60 g/l).					

Determine max fill mass $M_{C_L_max} = V_{loop} \cdot FR_{max}$ (FR_{max} = 592.39 kg/m³)
Determine min fill mass $M_{C_L_max} = V_{loop} \cdot FR_{min}$ (FR_{min} = 546.82 kg/m³)

Bottle + CO_2 = 3832.3g.

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TTCS volume determination						
Date: 27/10/2009	Company: SYSU	Location: CERN		Engineer: SXh. /ZZ		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
44.	Calculate the filled mass for the standard vessel Set $M_{C_VS} = M_{E_VS} + M_{C_L} + M_{C_R}$; NOTE: $M_{C_R} = M_{C_R_VS} + M_{C_R_FT} = V_{VS} \times \rho_G(T, P) + V_{FT} \times \rho_G(T, P)$ Here ρ_G (T, P) is determined by the chosen final temperature of the standard vessel and the final saturated pressure of the TTCS loop.	TTCS Loop Volume: $M_{TTCSloop+fillingsys} = M_{nitrogen2}$		$T_{ave} = 24°C$. $P_s = 6.279$ MPa. $T_{stan} = 37°C$. $\rho = 167.6$. ~~$M_{CR} = 3831.7$ g~~. bottle + CO_2 + remain = 3823.8 g. $-M_{nitrogen1} = 2846.3 - 2802.6 = 43.7$ g.		
45.	Repeat the above procedure of TTCS volume determination again for confirmation	$PV = \frac{M_{TTCSloop+fillsys}}{m} RT$		$2.553 \times 10^3 \cdot V = \frac{43.7}{28} \times 8.314 \times 295.65$. $\Rightarrow V_{TTCS+fillsys} = 1.5015$ L. $\therefore V_{TTCS} = 1.5015 - 0.068 = 1.433$ L ($\pm 1.615\%$)		

7.5 Fill a standard vessel with CO_2 from a Gas bottle

Remaining: $(0.068 + 1.485) \times 167.6 = 260.4$ g.
TTCS need: $569.6 \times 1.433 = 816.2$ g. $\therefore$ The total: $260.4 + 816.2 + 2747.2 = 3823.9$ g

Fill a standard vessel with CO_2 from a Gas bottle								
Date: 25/10/2009		Company: SYSU		Location: CERN		Engineer: SXU/ZZ		
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: ☐ 27°C (CMS)	Humidity: %RH	Pressure: MPa					

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Fill a standard vessel with CO_2 from a Gas bottle						
Date: 25/10/2009	Company: SYSU	Location: CERN		Engineer: SXh/ZZ		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
3.	Connect the SV1.5 (here anyone of the two SV1.5 can be used) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **<u>Note: The extend tube between connectors and valves must be supported to reduce strain.</u>** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)					✓
4.	Connect a high purity CO_2 gas bottle to connector 2;					✓
5.	Connect a dry mechanical pump to vacuum connector					✓
6.	Close valve 1 ,valve 4 and the gas bottle valve, open valve 3, valve 2 , pressure regulator, the vacuum valve and the SV1.5 valve;					✓

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Done before volume determination, put much more and the release based on volume determination.

Fill a standard vessel with CO_2 from a Gas bottle

Date: 25/10/2009 **Company:** SYSU **Location:** CERN - CMS **Engineer:** SXh/ZZ

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
7.	Open valve 3 until the pressure drop to 30Pa; Close the vacuum valve, open the bottle valve slowly, until the pressure up to 1MPa; Close the bottle valve and open the vacuum valve slowly, pump the SV1.5 until the pressure drop to 30Pa; **Repeat such operations and flush with CO_2 twice (totally three times).**	Evacuate Time Vacuum gauge reading	1~2 hours ≤30Pa ≤30Pa ≤30Pa	vacuum flush 0.9mpa. vacuum 28pa. flush 0.9mpa. vacuum 28pa		12:08 12:10 12:39
8.	Place the vacuumed SV1.5 to a barrel with ice water mixture, switch off the pump;					✓
9.	Open valve 2 and regulate outlet pressure of gas bottle valve slowly until higher than 4MPa;	Outlet pressure of gas bottle	≥ 4MPa	5Mpa.		✓ 14:50

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Fill a standard vessel with CO_2 from a Gas bottle

Date: 25/10/2009 **Company:** SYSU **Location:** CERN-CMS **Engineer:** SXh/ZZ

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
10.	After a period of time (2 hours~3 hours), close the SV1.5 valve, valve 2 and valve 3; Estimate weight by electronic balance without disconnecting SV1.5, check whether it meets required mass in the SV1.5. If CO2 filled into the SV1.5 is less than the required mass, then put the SV1.5 back to the barrel, and open valve 2, valve 3 and the SV1.5valve. During the estimation of weight without disconnecting SV1.5, the extend tube between the SV1.5 and filling box should be put up slightly in order to get real weight of the filled SV1.5 as possible as we can, and even though, the extend tube still account for about 200g in total estimating weight.	Waiting time Estimating weight	2 hours~3 hours $\geqq M_{C_VS}$+200g	weight. bottle + CO_2 = 3940 g. with a scale accuracy ±10g.		16:05.
11.	Disconnect the SV1.5 from the filling box, and dry the SV1.5 surface; Seal the extended tube.	Dry and clean the SV1.5 surface				✓
12.	Weight the filled SV1.5 with a electronic balance, as M_{Filled_VS};	M_{Filled_VS}				
13.	Take by hand the filled CO_2 mass $M_{O_VS} = M_{Filled_VS} - M_{E_VS}$;	M_{O_VS}				
14.	If $M_{O_VS} > (M_{C_VS} - M_{E_VS})$, open the SV1.5 valve very slightly to release a little CO_2 gas, close SV1.5 valve;					

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Fill a standard vessel with CO_2 from a Gas bottle						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
15.	Weight the SV1.5 again; repeats step 12 to 14 until M_{O_VS} value meets the requirement (=$M_{C_VS} - M_{E_VS}$).	M_{O_VS}	(M_{C_VS}–M_{E_VS}) ±1g			
16.	Seal the filled SV1.5 valve for cleanness					

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7.6 Calibration of the filled mass

N/A

Calibration of the filled mass								
Date:			Company:	Location:		Engineer:		
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: □	Humidity: %RH	Pressure: MPa					
3.	Set the filling environment temperature as 22°C							
4.	Connect the filled SV1.5 (the $SV1.5_{new1}$) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **Note: The extend tube between connectors and valves must be supported to reduce strain.** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)							
5.	Connect another standard vessel (**VS_B**) to connector 1 with the same extension filling tube for filling the TTCS loop;							
6.	Connect a dry mechanical pump to vacuum connector							

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N/A

Calibration of the filled mass						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
7.	Close valve 2 and open the vacuum valve, valve 3, pressure regulator, valve 1, valve 4 and the VS_B valve;					
8.	Pump the VS_B until the pressure down to 30Pa;	Vacuum gauge reading	≤30Pa			
9.	Close the vacuum valve and valve 4;					
10.	Open the VS_A valve slowly;					
11.	Switch on the filling box heater with and the standard vessel heater (to control the box temperature to 25°C step by step); If possible, the standard vessel heater should increase the temperature with an interval of 5~10°C. If heat power of the filling box heater is not enough, using additional heat gun to warm up the **VS_A**	No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	≤30°C ≤30°C ≤30°C			
12.	Using the fans to cool the **VS_B**					

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N/A

Calibration of the filled mass						
Date:	Company:	Location:		Engineer:		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
13.	Watch the reading of the digital pressure gauge. When the pressure and temperature is close to the set value, switch on the heating tape on the filling tubes. When the pressure is equal to **5.995MPa**, close the TTCS loop valve, valve 1, the SV1.5 valve, and valve 3, respectively;	APS reading of the filling system Temperatures of the filling box and tubes	*5.995±0.1MPa* *25°C*			
14.	Switch off the filling box heater and the heating tape;					
15.	Unwrap the **VS_A**;					
16.	Disconnect the **VS_A** and the **VS_B** r espectively;					
17.	Weight the **VS_A** and the **VS_B**, as $M_{O_R_VSA}$ and $M_{Filled_VS_B}$, respectively					
18.	From (M_{Filled_VS}– $M_{O_R_VS_A}$), we have the mass of CO_2 out of SV1.5; from ($M_{Filled_VS_B}$ –$M_{E_VS_B}$), we have the mass of CO_2 into **VS_B**;	(M_{Filled_VS}– $M_{O_R_VS_A}$) ($M_{Filled_VS_B}$ –$M_{E_VS_B}$)				
19.	Obtain the mass loss M_{loss}=(M_{Filled_VS}–$M_{O_R_VS_A}$)–(M_{Filled_VSB} –$M_{E_VS_B}$);	M_{loss}				

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N/A

Calibration of the filled mass						
Date:		Company:	Location:		Engineer:	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
20.	Calculate the mass loss based on the filling tube volume $M_{C_R_FT} = V_{FT} \times \rho_G(T)$; where T=25°C	$M_{C_R_FT}$				
21.	Obtain fill loss range of the filling tube by comparing M_{loss} and $M_{C_R_FT}$ to determine performance of the current filling system					
22.	Switch off the pump.					
23.	Seal the filled SV1.5 valve for cleanness					

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7.7 Fill TTCS loop with CO_2 from SV1.5

Fill TTCS loop with CO_2 from SV1.5							
Date: 28/10/2009		Company:		Location:		Engineer:	
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: □	Humidity: %RH	Pressure: MPa					
3.	Set the filling environment temperature as 22°C					Before: bottle + CO_2 mass / mass. = 3832.3g.		
4.	Connect the TTCS loop to connector 1 with an extended filling tube. **Note: Longer extend tubes may be used and height difference may exist between the TTCS loop and connector 1.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**							✓
5.	**Connect a high purity CO_2 gas bottle to connector 2;**							✓

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Fill TTCS loop with CO_2 from SV1.5

Date: 28/10/2009 Company: SYSU. Location: CERN. Engineer: SXh/ZZ.

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
6.	Put three PT1000 vertically by using adhesive tape in turn on the side surface of the SV1.5, then wrap the SV1.5 with heater with Pt1000 in the joint of the heater. **(Note: No.1 PT1000 for control, No.2 PT1000 and No.3 PT1000 for monitor.** **During filling CO_2 from the standard vessel to the TTCS loop, No.1 PT1000 should be paste on the middle of the standard vessel's side face; the other two PT1000s should be pasted on the top and bottom of the standard vessel's side face).**					√
7.	Connect the filled SV1.5 to connector 3; **Note: Longer extend tubes may be used and height difference may exist between the TTCS loop and connector 1.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**					√
8.	Use the He-mass spectrometer to perform leak test before filling. If leak rate more than 5×10^{-5} *mbar·l/s* somewhere, reconnect it until the leak rate is less than 5×10^{-5} *mbar·l/s*	LR	$<5\times10^{-5}$ *mbar·l/s* all connectors.	BG = 5.2×10^{-8} mbar·l/s. LR = 2.6×10^{-8} mbar·l/s		10:20.

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Fill TTCS loop with CO_2 from SV1.5						
Date: 28/10/2009	Company: SYSU	Location: CERN		Engineer: SXh/ZZ.		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
9.	Stop the He-mass spectrometer and replace it with a dry mechanical pump.					✓
10.	Follow the section of 7.5 (**fill a standard vessel with CO_2 to from a Gas bottle**) to fill SV1.5 to $\mathbf{M_{filled_SV}}$;	$\mathbf{M_{filled_SV}}$				
11.	Put three PT1000 vertically on the side surface of the SV1.5 by using adhesive tape. Then wrap the SV1.5 with heater with Pt1000 in the joint of the heater. **Note: check them can work or not** (No.1 PT1000 for control, No.2 PT1000 and No.3PT1000 for monitor. During the filling CO2 from the standard vessel to the TTCS loop No.1 PT1000 should be paste on the middle of the standard vessel's side face; the other two PT1000s should be paste on the top and bottom of the standard vessel's side face).	NO.1 Pt1000 reading NO.2 Pt1000 reading NO.3 Pt1000 reading	Ok or not Ok or not Ok or not			✓
12.	Insulate the filled vessel and connect the SV heater with the socket in the filling box.					
13.	Open the vacuum valve, valve 1, valve2, valve 3, valve 4 , the TTCS loop valve and the pressure regulator				Vacuum TTCS Loop.	9:00.

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Fill TTCS loop with CO_2 from SV1.5

Date: 28/10/2009 **Company:** SYSU. **Location:** CERN. **Engineer:** SXU/ZZ.

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
14.	Wait until pressure of the TTCS loop, the filling box and two extend tubes down to 30Pa or lower. Close valve4, the vacuum valve, and valve 1. then open the gas bottle valve slowly and valve 1 to fill the loop with high purity CO_2 from the gas bottle, until the pressure up to 1MPa (flush the loop)	Evacuate Time Vacuum gauge reading	1~2hours ≤30Pa ≥1MPa	22pa. 1.1 mpa.		10:13. 10:24.
15.	Close the gas bottle valve , open valve 1, valve 4 and the vacuum valve slowly;					
16.	Pump the TTCS loop, the filling box and two extend tubes until the pressure down to 30Pa or lower; Close valve4, the vacuum valve, and valve 1. then open the gas bottle valve slowly and valve 1 to fill the loop with high purity CO_2 from the gas bottle, until the pressure up to 1MPa (flush the loop)	Evacuate Time Vacuum gauge reading	0.6 hour ≤30Pa ≥1MPa	Vacuum. 30pa. 1.2mpa.		10:28. 11:15. 11:15.
17.	Pump the TTCS loop, the filling box and two extend tubes until the pressure down to 30Pa or lower;	Evacuate Time Vacuum gauge reading	0.6 hour ≤30Pa	Vacuum. 27pa.		11:20. 12:13.
18.	Close valve 4, the vacuum valve; switch off the pump.					

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Fill TTCS loop with CO_2 from SV1.5						
Date: 28/10/2009	Company: SYSU.	Location: CERN.		Engineer: SXh/ZZ.		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
19.	Put the SV1.5 with the connected tube on the electronic balance. Open the SV1.5 valve slowly, then close the SV1.5 valve, slowly open valve 1 to fill the CO2 into the TTCS loop to prevent pressure spike to the sensors. Repeating on and off the SV1.5 valve and valve 1 in turn until pressure of the TTCS loop is up to 3MPa, and then slowly open the SV1.5 valve and valve 1.	DPS reading of the TTCS loop	≤0.5bar			✓
20.	Switch on the filling box heater and the standard vessel heater (to control the box temperature to 25°C step by step); If possible, the standard vessel should be heated with a temperature interval of 5~10°C. If heat power of the filling box heater is not enough, using additional heat gun to warm up the SV1.5	NO.1 Pt1000 reading NO.2 Pt1000 reading NO.3 Pt1000 reading	*30±0.5°C* *30±1°C* *30±1°C*	37°C.		
21.	Use the fan to cool the accumulator					✓

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Fill TTCS loop with CO_2 from SV1.5

Date: 28/10/2009 Company: SYSU. Location: CERN. Engineer: SXh/ZZ

Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
22.	Watch the reading of the digital pressure gauge. When the pressure and temperature is close to the set-value, switch on the heating tape of the filling tubes. When the pressure is equal to **5.995MPa**, close the TTCS loop valve, valve 1, the SV1.5 valve, and valve 3, respectively;	Waiting time APS reading of the TTCS loop (P_{TTCB}) APS reading of the filling system ($P_{filling\ system}$)	3~4 hours 5.995±0.1MPa 5.995±0.1MPa			16:05.
23.	Switch off the filling box heater and the standard vessel heater;					✓
24.	Unwrap the heating tape from the SV1.5;					✓
25.	Disconnect the SV1.5 from the filling box, clean surface of the SV1.5					✓
26.	Weight the SV1.5 as ($M_{O_R_VS}$+ M_{E_VS}), compared ($M_{O_R_VS}$+ M_{E_VS}) with ($M_{C_R_VS}$ + M_{E_VS})	($M_{O_R_VS}$+M_{E_VS})	($M_{C_R_VS}$+M_{E_VS}) ±5g	2994.4 g		
27.	Filling finished.					
28.	After the filling is finished, the two extended filling tubes, the SV1.5 valve and the filling system must be carefully disconnected and sealed.			TTCS Loop CO_2 mass: 3832.3 − 2994.4 − 11.6 remaining in tubes = 826.3 g		✓
29.	Carefully packing the two extended filling tubes, the SV1.5 and the filling system.		V_{loop} = 1.433L.	filling rate = 576.6 g/L		✓ (±1.762%)

~~FR * error = 576.6 × 1.026 = 591.6 g/L.~~

Including error highest ρ = FR · error = 576.6 g/L × 1.01762 = 586.76 g/L

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Including error lowest ρ. ρ = 576.6 g/L × 0.98238 = 566.44 g/L

7.8 Venting CO_2 from the TTCS loop

Venting CO2 from the TTCS loop								
Date:			Company:	Location:		Engineer:		
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: °C	Humidity: %RH	Pressure: MPa					
3.	Switch on TTCE Terminal and go to the interface TTCE-2							
4.	Write down the PT01 and PT03 temperatures			PT01 and PT03	>22°C			
5.	Connect the TTCB valve, filter, stainless tube and extended tube							
6.	Open the TTCB valve a little to release CO_2 gas slowly while switch on the ground accumulator heater (GAC) with 30% Monitor PT01 and PT 03, once either temperature is below 18 °C, stop venting by closing TTCB valve.			PT01 and PT03	>17°C			
7.	Use a hot air gun, or hot water bath, to heat up the exit of the stainless exhaust tube							
8.	Repeat steps 6 and 7, until the pressure is down to 1.5 bar			APS reading	1.5±0.1bar			

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Venting CO2 from the TTCS loop						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
9.	Close the TTCB valve.	APS reading	1.5±0.1bar			
10.	Switch off TTCE Terminal					

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8. APPENDIX

8.1 Error analysis of volume determination of a vessel by weighting the gas mass inside

Assumption: no leakage of measuring system, including the container, during the period of measurement; the electronic balance employed has the best accuracy of 0.1g; and the temperature be controlled within 0.5K.

According to the ideal gas equation:

$$PV = \frac{M}{m} RT \qquad \text{(A1.1)}$$

Where m is the mole mass of the gas in use (in g/mole), M is the net gas mass to be measured, and R is the gas constant, T is temperature of 22°C in thermal bath respectively. For nitrogen, m=28g/mole, and a vessel of about 1.5liters, M≈76.3g. From (A1.1), we have

$$\frac{\Delta V}{V} = \frac{\Delta P}{P} + \frac{\Delta M}{M} + \frac{\Delta T}{T} = \frac{\Delta P}{P} + 2\frac{\Delta M_{\mathrm{B}}}{M} + \frac{\Delta T}{T} \qquad \text{(A1.2)}$$

As ΔP is limited by the pressure sensor, with the pressure sensor available with accuracy of ±0.2% FS, by setting P to the FS of the pressure sensor, (e.g., 10MPa), we have the lowest error of 0.44% for $\Delta P/P$. For using nitrogen as an ideal gas, $\Delta V/V$ is about 0.89 %.

Gas used	N_2
m (g/mole)	28
M(g)	76.3
ΔM/M(%)	0.26
ΔT/T(%)	0.17
ΔP/P(%)	0.44
ΔV/V(%)	0.89

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8.2 Error analysis of volume determination of TTCS loop by indirectly measuring the Nitrogen mass inside the loop

Assumption: no leakage to the measuring system, including the TTCS loop and the filling system, during the filling; and the temperature and its uniformity be controlled within ±2K; the volumes of the standard vessel to fill to loop (V_{VS}) is about 1.5 liter, and the volume of the loop is about 1.5 liter ($V_{loop} \approx V_{VS}$); the accuracy of the electronic balance is 0.1g (ΔM_B); no nitrogen absorption on the inner surface of the TTCS loop that made of stainless steel.

1) Error analysis for the measured volume the filling system including the filling tubes.

Fill nitrogen with the SV1.5 to a pressure of 4.5MPa; weight the mass of the filled SV1.5 $M'_{nitrogen1}$. Connect the vessel to the filling system and release the nitrogen slowly. Weight the mass of the SV1.5 again $M'_{nitrogen2}$, the nitrogen mass filled into the filling system can then be calculated: $M_{FT} = M'_{nitrogen1} - M'_{nitrogen2}$, and $\Delta M_{nitrogen} = \Delta M_1 + \Delta M_2 \approx 2\Delta M_B$. To the volume of the filling tube of the filling system is about 0.042 liter. Even with additional tube of 5m long and 3mm in inner diameter, which is about 0.035 liter, the nitrogen mass inside filling tube is about a few percentages of that in the SV1.5 and the pressure inside is about the same as the initial pressure of SV1.5. The estimated N_2 mass is:

$$M_{FT} \approx \frac{1}{20}\frac{P_0 V_{VS} m_{nitrogen}}{RT_0};$$

Assuming $P_{FT} \approx P_0$, the error of V_{FT}

$$\frac{\Delta V_{FT}}{V_{FT}} = \frac{\Delta P_{FT}}{P_{FT}} + 2\frac{\Delta M_B}{M_{FT}} + \frac{\Delta T_{FT}}{T_{FT}}$$

$$= \frac{0.1\% FS}{P_0} + 40\frac{\Delta M_B RT_0}{P_0 V_{VS} m_{nitrogen}} + \frac{\Delta T_{FT}}{T_{FT}}$$

is about 6.3%

2) Error analysis for the measured volume of the loop and filling tube

Similarly, connect the filled vessel to a vacuumed loop and release the nitrogen inside the filled VS1.5 slowly until equilibrium is established. Weight the mass of the SV1.5 again $M_{nitrogen2}$, the

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nitrogen mass filled into the loop can then be calculated: $M_{loop+FT} = M_{nitrogen1} - M_{nitrogen2}$, and $\Delta M_{loop+FT} = \Delta M_1 + \Delta M_2 \approx 2\Delta M_B$. As V_{VS} is about half of the total volume, the nitrogen mass inside the loop and filling tube is about half of the initial filled mass of nitrogen in the SV1.5, and the pressure inside the loop and filling tube is about half of the initial pressure of SV1.5.

$$M_{loop+FT} = \frac{1}{2}\frac{P_0 V_{VS} m_{nitrogen}}{RT_0} \quad \text{And } P_1 \approx \frac{1}{2}P_0;$$

Assuming $T_0 = T_{loop}$, and ΔT_{loop} is about ±2K

The error of $V_{loop+FT}$:

$$\frac{\Delta V_{loop+FT}}{V_{loop+FT}} = \frac{\Delta P_1}{P_1} + 2\frac{\Delta M_B}{M_{loop+FT}} + \frac{\Delta T_{loop}}{T_{loop}}$$

$$= 2\frac{0.2\% FS}{P_0} + 4\frac{\Delta M_B RT_0}{P_0 V_{VS} m_{nitrogen}} + \frac{\Delta T_{loop}}{T_{loop}}$$

is about 2.1%.

3) Error analysis for the measured volume of the loop

The error of V_{loop}:

$$\frac{\Delta V_{loop}}{V_{loop}} = \frac{2.1\% \times 1.5 + 6.3\% \times (0.042 + 0.035)}{1.5}$$

is about 2.4%

In the assumption, no absorption of nitrogen is considered. This is achieved by performing the measurement for at least twice. Let the nitrogen absorption, if there is any, reach its saturated value during the first measurement, so for the following measurement, there should not be additional nitrogen absorption happen.

8.3 Error analysis of the total filled mass of TTCS loop

Assumption: There is no leakage for the filling system, and the TTCS loop during the period of filling and measurement; the temperature is controlled within ±2.0K for filling procedure without thermal bath; and the volume of the loop is about 1.5 liter; the electronic balance employed has the

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best accuracy of ±0.1g (ΔM_B). (Based on our experiments), any mass loss is less than 1g.

According to Annex 8.2, the error of the loop volume is about 2.4%, so the calculated filled mass $m_{cal} = FrV_{loop}$ (≈860g) has also the error of=2.4%.

(1) The error from weighting CO_2 mass is

$\Delta M=\Delta M_2-\Delta M_1=2\ \Delta M_B=\pm 0.2g$.

(2) The error from the filling loss in the filling system, the CO_2 mass remained the filling tube of filling system plus additional tube with length of 5m (0.077L) is about 12.75g based on NIST refrigerant properties database.

The error of total filled mass of TTCS is

$$\frac{\Delta M_{CO2_loop}}{M_{CO2_loop}} = \frac{\Delta V_{loop}}{V_{loop}} + \frac{2M_B + M_{loos}}{V_{loop} \times FR} \approx 2.4\% + 1.2/1.5/569.6 \approx 2.6\%$$

Therefore, we have total CO_2 mass accuracy of ±2.6%, which meets the requirement of ±4%.

Conclusion:

Although the volume error is larger than 2% (required in Ref.1), the mass error (2.6%) is still within that required (4% in the Ref.1)

The impact of using electronic balance with accuracy of 0.1g other than 0.01g is that, the error of loop volume determination is obviously increased, which will be transferred to corresponding mass error. The impact on weighting itself is little.

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8.4 Calculation of the required filling mass into the SV1.5

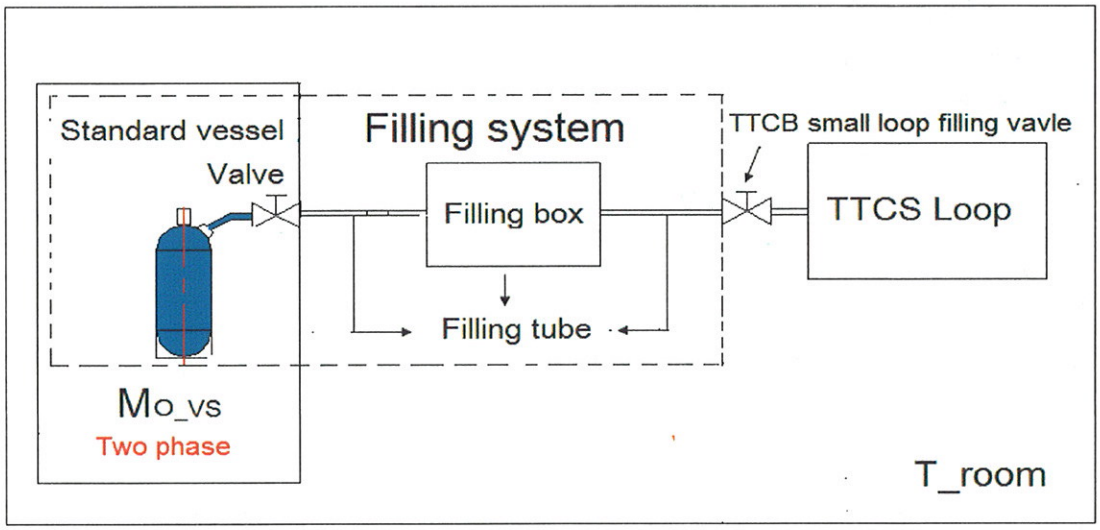

Figure 8-1 CO2 initial state and mass distribution between the filling system and the TTCS loop before starting filling

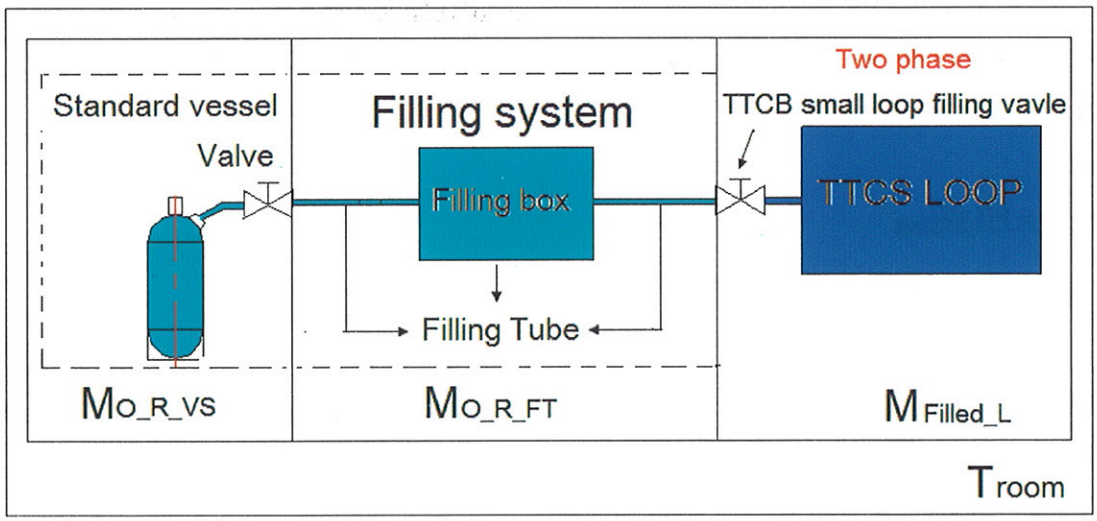

Figure 8-2 CO_2 finial state and mass distribution between the filling system and the TTCS loop after finishing filling

It can be concluded from Figure 8-1 and Figure 8-2 that CO_2 finial mass distribution would exist in the SV1.5, the filling tube of the filling system and the TTCS loop just like shown in Figure 8-3 with ignoring leak mass during filling process.

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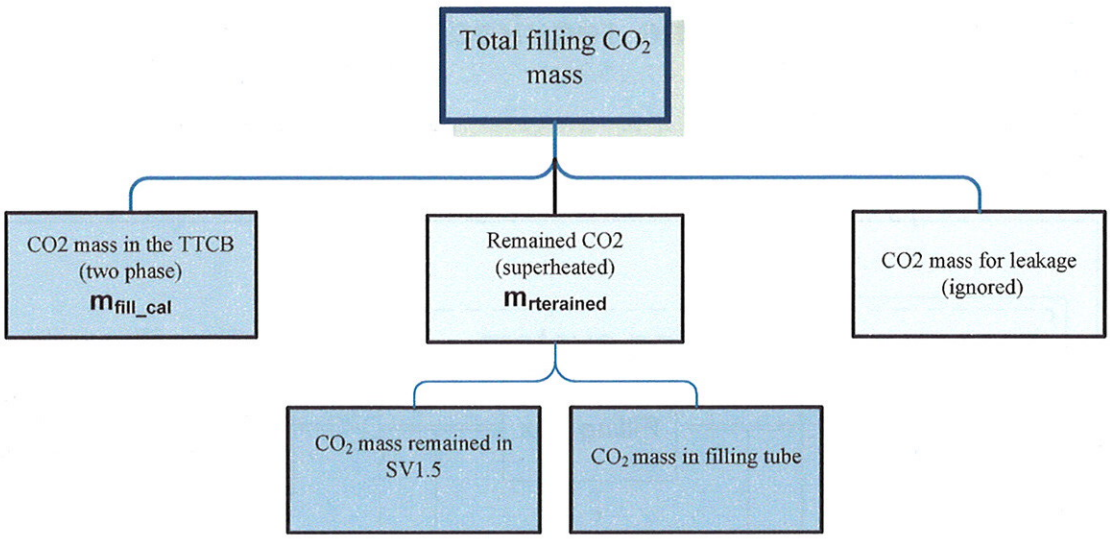

Figure 8-3 CO_2 mass distribution between the filling system and the TTCS loop after finishing filling

8.5 Calculation example

1) Calculation of M_{C_L}

Assumed : V_{Loop}= 1.55L

So

M_{C_L} = FR×V_{Loop}

= 569.6g/L×1.55L

= 882.88g

The above calculated CO_2 must be filled into the TTCS regardless of what temperature you choose for the filling.

2) Calculation of M_{C_R}

If we choose 25°C for the filling, so the retained mass of CO_2 in the filling system including filling box, two extended tube and the stand vessel can be calculated as below:

Known conditions: T_{Loop}(chosen)= 22°C, **P(corresponds toT_{accu}(chosen))**=5.995MPa,

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T_{VS}(controlled)=30°C,

Once **T_{Loop}(chosen)** is fixed, the final pressure in the filling system and the TTCS loop is also fixed. If the chosen T of CO_2 is 22°C, the corresponding saturated pressure is 5.995MPa.

And in the filling box, the SV1.5 and twos extend tubes

ρ_G (30°C, 5.995MPa) = 170.8g/ L

Solution:

And if volume of the used SV1.5 **V_{VS}** is 1.485L , and filling tube of the filling system **V_{FT}** is 0.042Liter, so the CO2 retained in the SV1.5, the filling box and two filling tubes can be calculated as:

$$M_{C_R} = M_{C_R_VS} + M_{C_R_FT}$$

$$= V_{vs} \times \rho_G (30°C, 5.995MPa) + V_{FE} \times \rho_G (30°C, 5.995MPa)$$

$$= (1.485L+0.042L) * 170.8g/L$$

$$= 260.82g$$

According to the above calculation, the temperature chosen for filling will influence the retained mass of CO2 M_{C_R} in the filling system, not influence the mass of CO_2 to be filled into TTCB.

3) Calculation of the total mass

By ignoring the leak mass, the CO_2 mass must filled into the used SV1.5 is

$M_{C_L} + M_{C_R}$=882.88g+260.82g =1143.7g

And it pluses net mass of the used SV1.5 M_{E_VS} is

$M_{C_VS} = M_{E_VS} + M_{C_L} + M_{C_R}$

=2747.23g+882.88g+260.82g =3890.83g

Finally, shown by electronic balance, the total mass should be 3890.83g.

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Table 10 saturated properties of CO2 from 15°C to 29°C with interval of 1°C

Temp [°C]	Pressure[MPa]	Density (L) [kg/m^3]	Density (V) [kg/m^3]	Enthalpy (L) [kJ/kg]	Enthalpy (V) [kJ/kg]	Entropy (L) [kJ/K-kg]	Entropy (V) [kJ/K-kg]
15.00	5.081	821.8	159.6	240.1	418.0	1.136	1.754
16.00	5.204	813.0	165.4	243.1	416.6	1.146	1.746
17.00	5.330	803.9	171.5	246.2	415.0	1.156	1.738
18.00	5.458	794.4	178.0	249.4	413.4	1.167	1.730
19.00	5.589	784.5	184.9	252.6	411.6	1.177	1.721
20.00	5.722	774.2	192.3	256.0	409.7	1.188	1.712
21.00	5.857	763.3	200.2	259.5	407.6	1.199	1.703
22.00	5.995	751.8	208.8	263.0	405.3	1.211	1.693
23.00	6.136	739.5	218.1	266.8	402.8	1.223	1.682
24.00	6.279	726.3	228.3	270.7	400.0	1.236	1.670
25.00	6.425	712.0	239.6	274.9	396.8	1.249	1.658
26.00	6.574	696.2	252.4	279.3	393.2	1.263	1.644

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27.00	6.727	678.4	267.3	284.2	389.0	1.279	1.628
28.00	6.882	657.5	285.1	289.7	384.0	1.296	1.609
29.00	7.042	631.7	307.8	296.3	377.5	1.317	1.586

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1) error of external tube.

$$\frac{\Delta V_{FT}}{V_{FT}} = \frac{\Delta P_{FT}}{P_{FT}} + 2\frac{\Delta M_B}{M_{FT}} + \frac{\Delta T_{FT}}{T_{FT}}$$

$$= \frac{0.1\% \times 10MPa}{5.4MPa} + 2\frac{0.1g}{2850.3g - 2846.3g} + \frac{2k}{295k}$$

$$= \cancel{5.678\%}\ 5.874\%$$

2) error of the measured volume of the loop and filling tube.

$$\Delta V_{loop+FT} = \frac{\Delta P_1}{P_1} + 2\frac{\Delta M_B}{M_{loop+FT}} + \frac{\Delta T_{loop}}{T_{loop}}$$

$$= \frac{0.1 \times 10MPa}{5.096MPa} + 2\frac{0.1g}{2846.3g - 2802.6g} + \frac{2k}{295k}$$

$$= \cancel{1.34}\ 1.332\%$$

3) error of the loop

$$\frac{\Delta V_{loop}}{V_{loop}} = \frac{1.332\% \times 1.433L + 5.874\% \times 0.0692}{1.433L}$$

$$= 1.615\%$$

4) The error of total filled mass of TTCS primary loop.

$$\frac{\Delta M_{CO_2\text{-}loop}}{M_{CO_2\text{-}loop}} = \frac{\Delta V_{loop}}{V_{loop}} + \frac{2M_B + M_{loss}}{V_{loop} \times FR}$$

$$= 1.615\% + \frac{2 \times 0.1g + 1g}{1.433L \times 569.6g/L} = 1.762\%$$

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