

The Nikhef gas filling system

Gas mixture in light-weight JSP bottle

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Why using premixed gases in JSP bottle?

- ◆ No need for gas mixing system on site
- ◆ Content 12.3 l
- ◆ Pressure up to 20 bar gauge
 - Useful for small flows (like 0.1 – 1.0 l/h)
 - => running time 10 – 100 days
 - Relaxed safety requirements
 - When filled with flammable mixture less dangerous than a simple spray can
 - Possibly may be placed in test beam area
 - => short and thin pipes, low dead volume
- ◆ Using premixed from commercial vendors not attractive
 - Expensive
 - Long delivery time (5 – 6 weeks)
- ◆ => We built a gas filling system in house (Nikhef)



Gas bottle

Empty weight 4.1 kg


- ◆ Volume: 12.3 l
- ◆ Special version of light weight bottle
 - Fabricate JSP
 - Originally intended for butane and propane
- ◆ Material: AISI 304 (stainless steel)
- ◆ Test pressure: 30 bar
- ◆ Burst pressure: 140 bar
 - Controlled burst location (cylinder bottom)
- ◆ Equipped with safety valve opening at 27 bar gauge
 - => good filling pressure: 20 bar gauge
- ◆ Seals of outlet valve and safety valve: NBR (nitrile)
 - Outlet valve: only gas exposure to metals when closed
- ◆ Valve thread for regulator: LU-1 (W21,8x1/14"L)
 - (commonly used left thread for flammable gases)



Certificates



- ◆ TPED 1999/36 (pi)
- ◆ EN14140
- ◆ ADR (international transport of dangerous goods by rail)
- ◆ RID (international transport of dangerous goods by train)
- ◆ IMDG (international marine code of dangerous goods)
- ◆ RRP (pressure container regulation)
- ◆ RAP (pressure machine regulation)
- ◆ Certified to conform to EN 14140; Directive (ADR) 94/55/EC and Directive (TPED) 99/36/EC
- ◆ All certificates apply for bottle filled with liquefied flammable gas
 - LPG, Butane, Propane

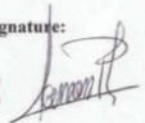





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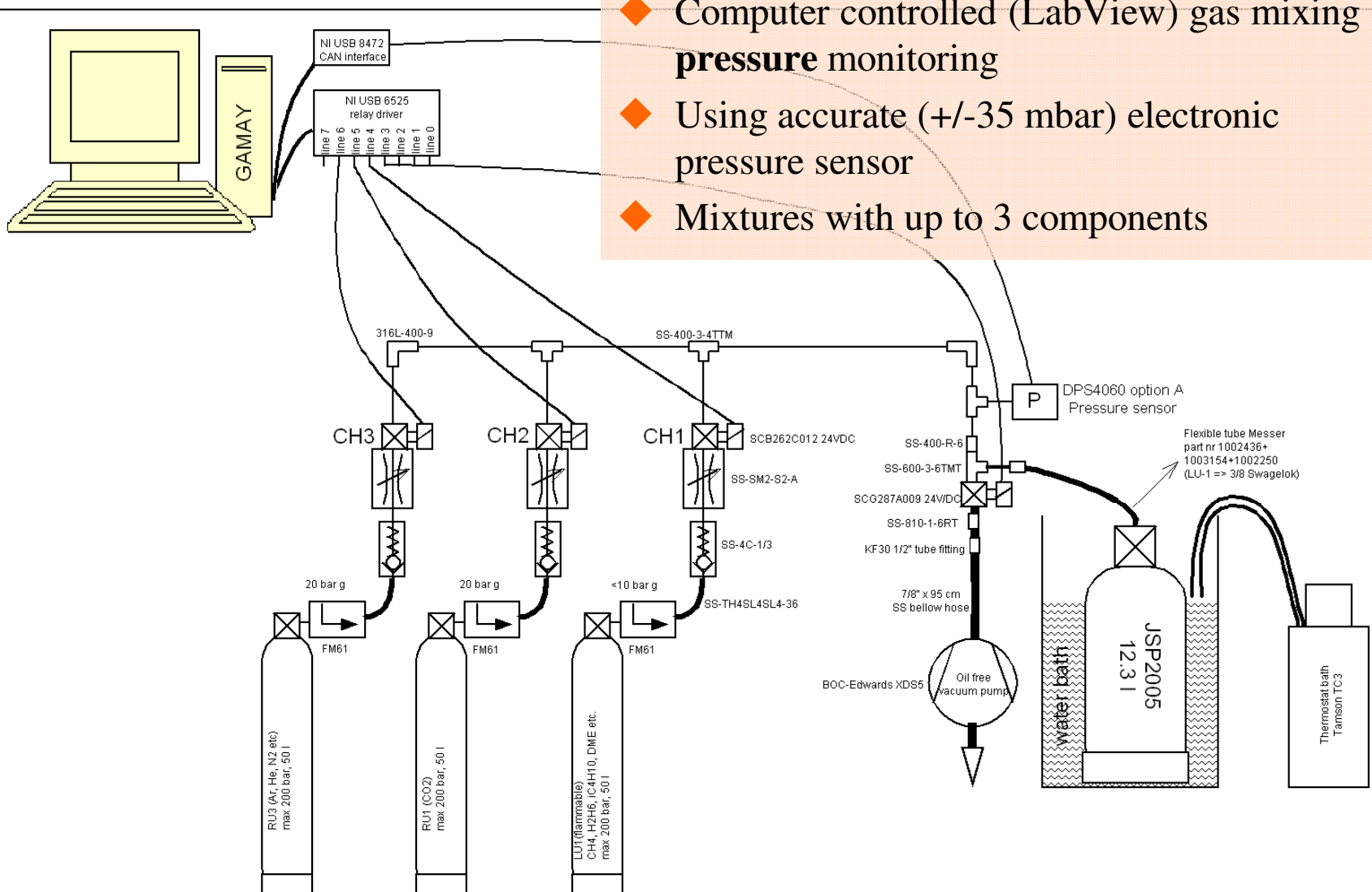



NOTIFIED BODY EC Design EXAMINATION CERTIFICATE In accordance with module B1 of the Transportable Pressure Equipment Directive 1999/36/EC		Certificate N°: <u>04/SP/767-Index 0-REV 3</u>																						
Manufacturer: JSP/PORTINOX, S.A.																								
Concerned Equipment: Transportable refillable welded stainless steel gas cylinders. Drawing N°: <u>101.000.17</u> of 03 rd March. 2004																								
<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: left;">Type N°:</th> <th style="text-align: left;">GL/30/229/X/12.3/Y</th> </tr> </thead> <tbody> <tr> <td>Test Pressure</td> <td>30 bar</td> </tr> <tr> <td>Min. Burst Pressure</td> <td>67.5 bar</td> </tr> <tr> <td>Capacity</td> <td>12.3 L</td> </tr> <tr> <td>Wall thickness min (Cyl part)</td> <td>1.07 mm</td> </tr> <tr> <td>Wall thickness min (Bottoms)</td> <td>0.91 mm</td> </tr> <tr> <td>Nominal diam. (inside)</td> <td>229 mm</td> </tr> <tr> <td>Withdrawal Tube</td> <td>NO</td> </tr> <tr> <td>Welding of the threaded neck</td> <td>MIG</td> </tr> <tr> <td>Welding of the semi bodies</td> <td>TIG</td> </tr> <tr> <td>Gas</td> <td>LPG</td> </tr> </tbody> </table>			Type N°:	GL/30/229/X/12.3/Y	Test Pressure	30 bar	Min. Burst Pressure	67.5 bar	Capacity	12.3 L	Wall thickness min (Cyl part)	1.07 mm	Wall thickness min (Bottoms)	0.91 mm	Nominal diam. (inside)	229 mm	Withdrawal Tube	NO	Welding of the threaded neck	MIG	Welding of the semi bodies	TIG	Gas	LPG
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The undersigned, acting within the scope of its notification for the purpose of the European Council Directive 99/36/EC, certifies that the type of these gas cylinders conforms to EN 14140; Directive (ADR) 94/55/EC and Directive (TPED) 99/36/EC, on the base of the supplied information and performed controls on cylinders.																								
Date: 10/03/2005	Name: Herman Ph	Position: Resp. Cyl. Dept.																						
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Diagram Nikhef gas filling system

- ◆ Computer controlled (LabView) gas mixing by **pressure monitoring**
- ◆ Using accurate (± 35 mbar) electronic pressure sensor
- ◆ Mixtures with up to 3 components



Nikhef gas mixing station

15-1-2010
Fred Hartjes

Planned filling sequence

1. Put required mixture in LabView sequencer
 - Partial pressures will be calculated
2. Evacuate bottle and tubing to < 1 mbar
3. Fill to ~ 1 bar abs with “background” gas (argon, CO₂, He, N₂,)
4. Re-evacuate
5. Fill with component 1 to desired partial pressure and stop automatically
6. Measure obtained pressure after few minutes
7. Add component 2 to desired summed pressure and stop automatically
8. Measure obtained pressure
9. Even so for component 3
10. Calculate obtained mixing ratio
11. Print bottle label and write data in log file



Vapour pressures vs temperature

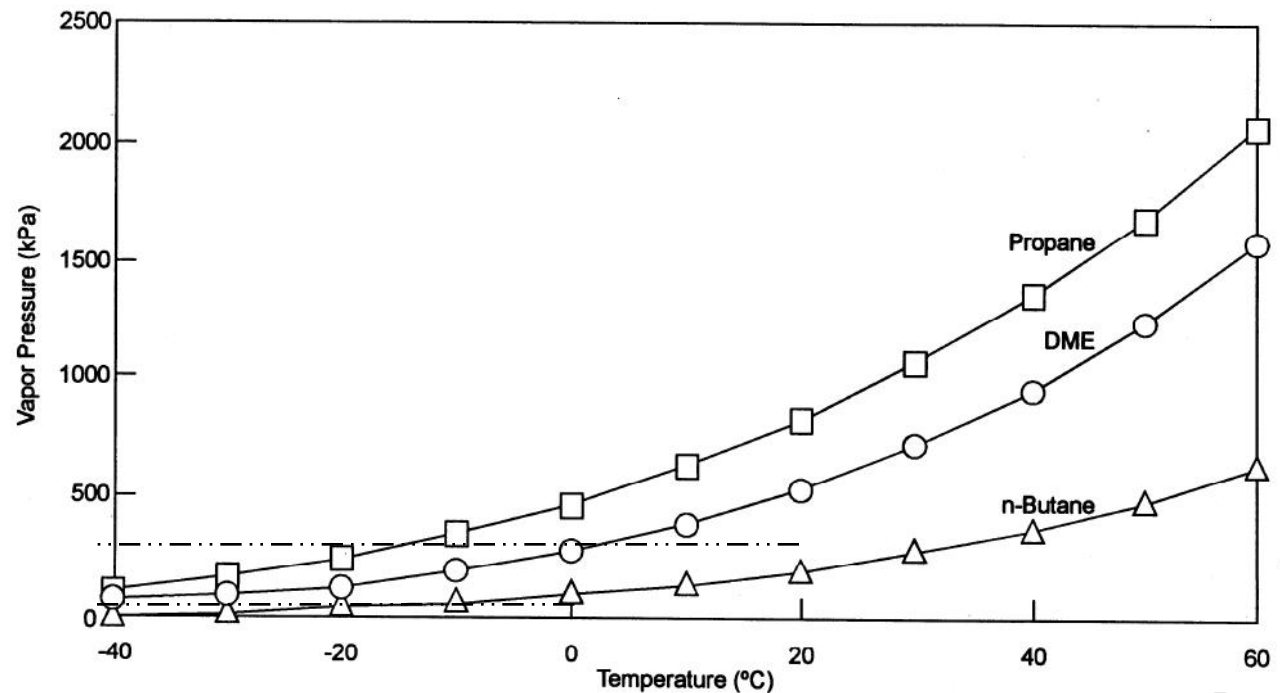
◆ Maximum bottle pressure often determined by condensation point

- Isobutane 2.6 bar @ 15 °C

■ \Rightarrow Ar/ i C₄H₁₀ 50/50 can be no more than 5.2 bar abs or **4.2 bar gauge**

- DME 5.1 bar @ 20 °C

■ Boiling point – 24.8 °C



Expected performance

- ◆ Accuracy of the component fraction 0.1% of total mixture
- ◆ Deviation from required mixture 1% of total mixture
- ◆ Example:
 - Target mixture Ar/CH₄ 90/10
 - Realized mixture will be something like Ar/CH₄ (90.08±0.1)/(9.92±0.1)
- ◆ Contamination
 - O₂, N₂, H₂O on ppM level
 - To be confirmed
- ◆ There will be no independent analysis of the mixture or to traces of contamination
 - Measuring oxygen on ppM level might be possible

Possible contamination by materials filling system

◆ Gas bottle

- Stainless steel AISI 304

◆ Valves

- NBR
- Teflon tape
-

◆ Piping, fittings

- stainless steel

◆ Cleaning

- Piping, fittings: well cleaned
- Gas bottle
 - Etched, flushed and neutralised after high temperature manufacturing
 - Baking out not standard but possible (130 °C under vacuum)
 - High temperature baking (300 – 800 °C possible when dismantling valve)

- **=> for critical operation adequate filtering (molecular sieve) mandatory**
 - ageing tests



Security rules for the gas mixing station at Nikhef

◆ Creating mixtures with flammable gases at Nikhef allowed if

- Done in well vented space
- Explosion detection available
- Proper grounding of equipment
- Only accessible for limited number of persons
- These persons are well trained
- Additional fire extinguisher is present
- Risk analysis has been made
- Flammable gas indication is outside
- Security staff has been informed and instructed

Using JSP bottle with flammable gas mixtures at a CERN test beam



- ◆ Pressure of DME and isobutane mixtures limited by vapour pressure

Mixture	P1 (bar abs)	P2 (bar abs)	P _{tot} (bar gauge)	Net content (l)	H ₂ eq. mass(g)	Running time (days) at 0.3 l/h
Ar/CH ₄ 90/10	18.9	2.1	20	246	7.0	33.2
CO ₂ /DME 50/50	4.0	4.0	7.0	86	25.4	12.0
Ar/iC ₄ H ₁₀ 80/20	8.8	2.2	10	123	26.0	17.1

Safety in perspective



Premixed JSP bottle with DME/CO₂ 50/50
25.5 g H_{eq}



Spray can/lighter filler
68.4 g H_{eq}



50 l bottle with isobutane
~6750 g H_{eq}



Butane container for cooking
~2500 g H_{eq}

⇒ JSP bottle accepted in beam area?

⇒ JSP bottle may be safely transported in a car

~~Airplane~~


Using JSP bottles with flammable mixture at CERN

- ◆ Not more than $\sim 30 \text{ g H}_{\text{eq}}$
- ◆ Well within Risk Class 1 ($< 400 \text{ g H}_{\text{eq}}$)
- ◆ One should wish having another Risk Class ($< 40 \text{ g H}_{\text{eq}}$)
 - Range of spray can

FLAMMABLE GAS SAFETY CODE Annex A

Flammable Gas Safety Manual

List of



Risk Class 0	: $Q_{\text{tot}} = 0$ the use of non flammable gases or mixture(s)
Risk Class 1	: $Q_{\text{tot}} < 0.4 \text{ kg}$ (approximately equivalent to 1 kg of hydrocarbon) risk of a small local flash fire or explosion
Risk Class 2	: $0.4 \text{ kg} < Q_{\text{tot}} < 40 \text{ kg}$ (approximately equivalent to 100 kg of hydrocarbon): risk of a local fire or explosion
Risk Class 3	: $Q_{\text{tot}} > 40 \text{ kg}$ (approximately equivalent to 100 kg of hydrocarbon): risk of a general fire or explosion

Commissioning

- ◆ Gas heated by filling process

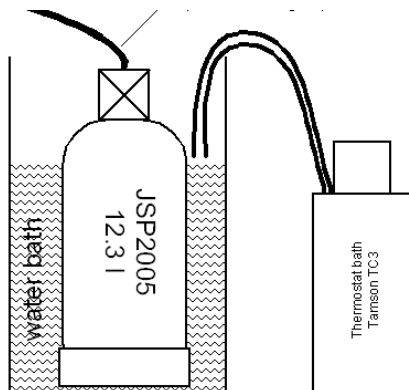
- Cooled by wall of bottle

- ◆ Bottle temperature stabilized by water

- ◆ Double exponential decay to saturation value

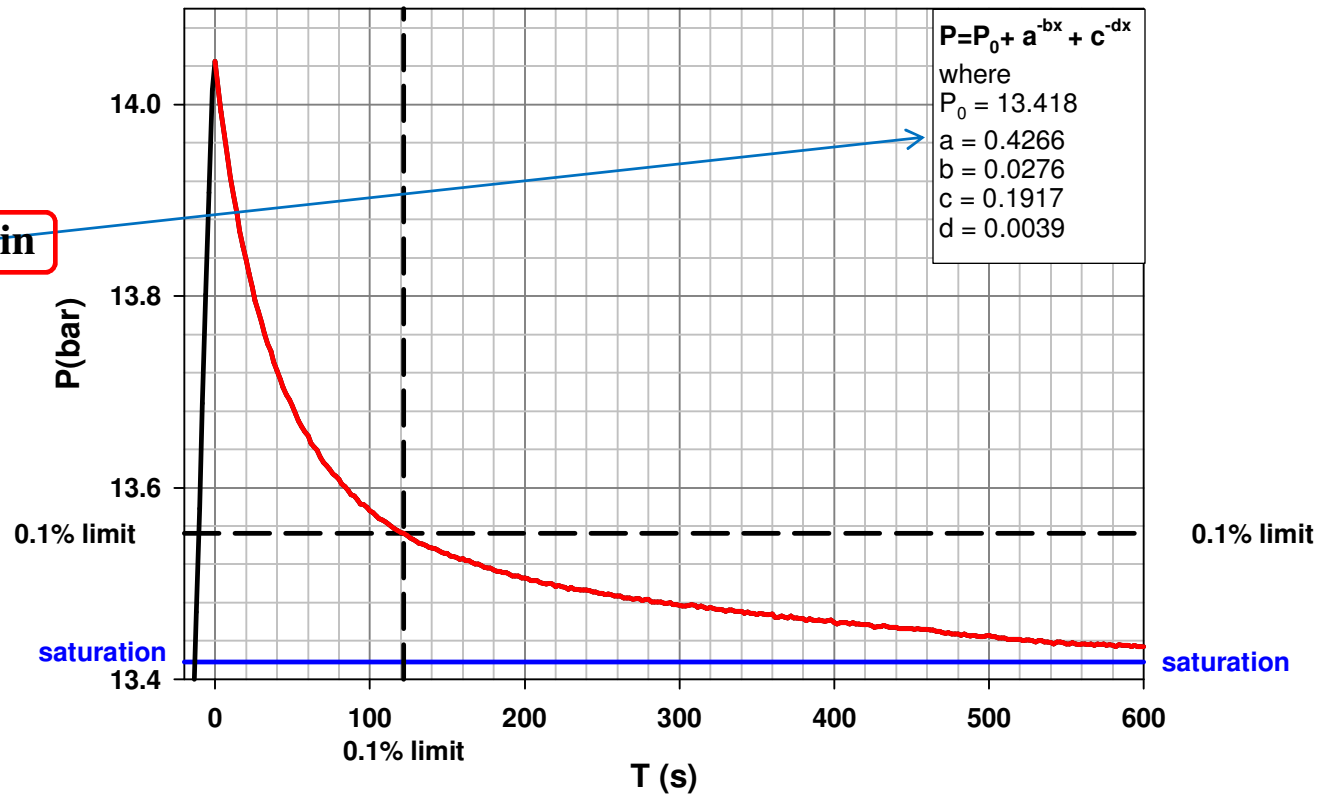
- $\tau = 36 \text{ s}$ and $\sim 4 \text{ min}$

- ◆ After 2 min within 0.1% limit



Pressure(P) vs time (T) after filling

0.1% limit



Present status 22-2-2010

◆ Finished

- Hardware installation
- Remote control operational
 - Electromagnetic valves
 - Vacuum pump
 - Pressure meter

◆ To be done

- Commissioning
- Operation program (LabView)
 - Sequencing
 - Logging
- CE certification



Conclusions

- ◆ Using premixed gases in JSP bottles has many advantages for low (< 1 l/h) flow applications
 - Thin gas pipes (1/8")
 - Premix gas bottles in lab/ test beam area
 - Simple, non critical (1 channel) gas regulation
 - Easy and cheap experimental set-up

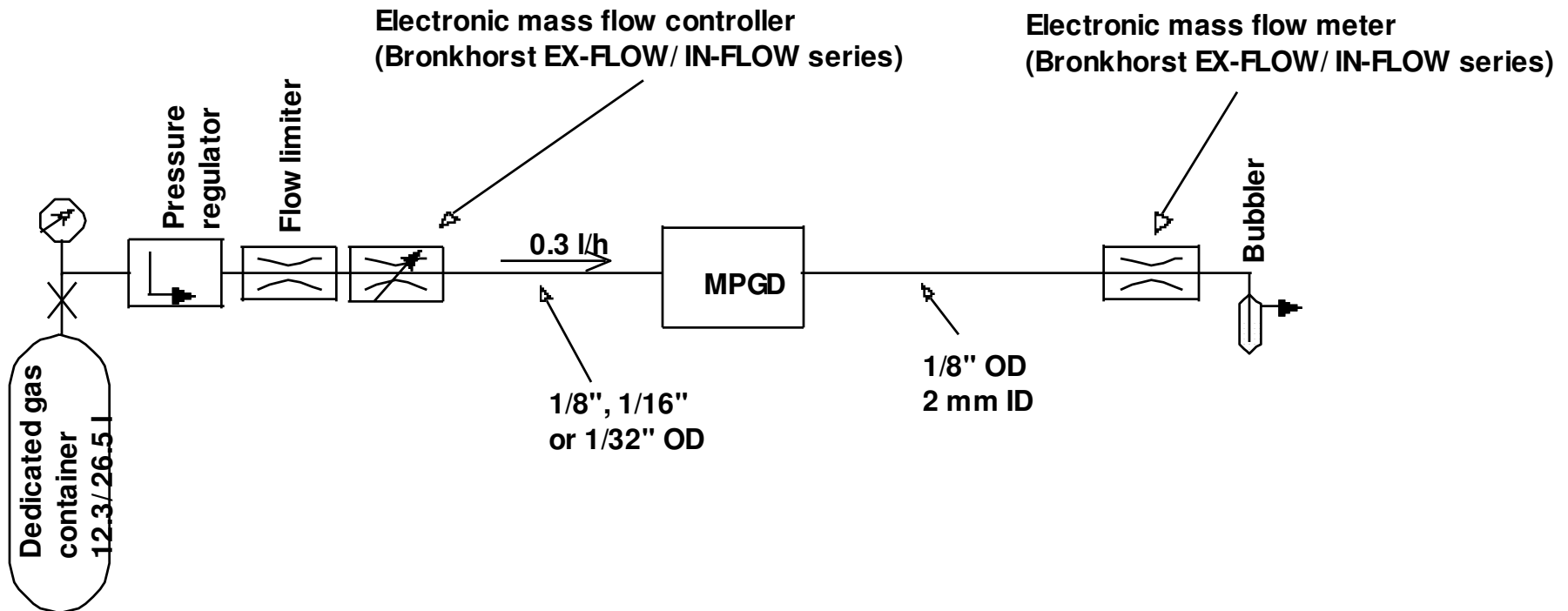
- ◆ Nikhef installation will be soon ready to fill the JSP bottles



Spare

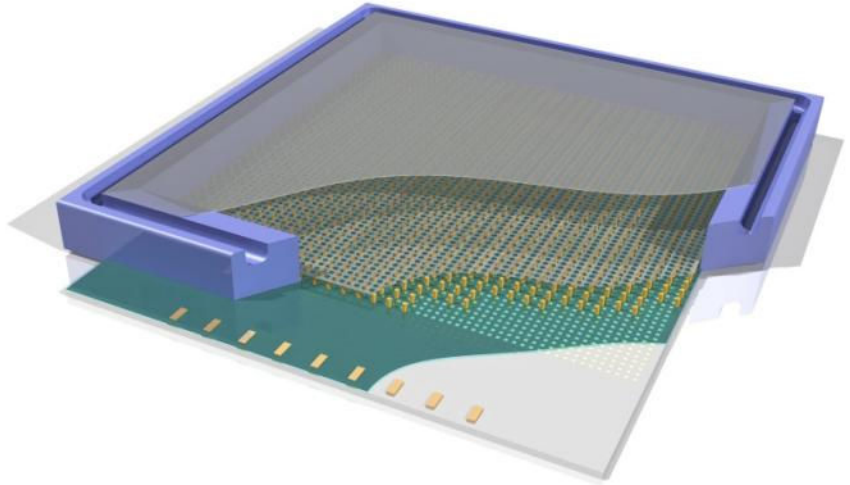
Test beam gas system with premixed bottles

- ◆ Assume small flow (≤ 0.3 l/h)
- ◆ Flow regulated by electronic mass flow controller (explosion proof)
- ◆ Flow check (electronic mass flow sensor) at exhaust
 - => verifying leaks
- ◆ Upstream: thin pipes may be used (1/8", 1/16", 1/32")
- ◆ Downstream: thicker pipes (1/8")



What is different for single chip MPGDs?

- ◆ Very small detector volumes
 - 1 Gossip detector \approx **0.2 ml** (15 x 15 x 1 mm³)
- ◆ \Rightarrow very small gas flows may be used
 - 10 volume changes/hour
 - \Rightarrow 33 $\mu\text{l}/\text{min}$ (**2 ml/h**)
 - Commercial mass flow controllers go down to $\sim 2 \text{ ml}/\text{min}$ FS
 - \Rightarrow permit flows down to 100 $\mu\text{l}/\text{min}$
- ◆ For practical reasons we normally use bit larger flows
 - 2 - 5 ml/min (**0.12 – 0.3 l/h**)



Advantages of small flows (0.12 – 0.3 l/h)

◆ Permitting very thin gas lines

- Gas lines 1/32" (≈ 0.8 mm OD) well feasible
 - 3 m tubing **0.5 mm ID** with CO₂ and 0.12 – 0.3 l/h
 - \Rightarrow Back pressure 10 – 24 mbar
- Gas line of 1/64" (≈ 0.4 mm OD) not excluded
 - 3 m tubing **0.25 mm ID** with CO₂ and 0.12 – 0.3 l/h
 - \Rightarrow Back pressure 0.15 – 0.4 bar
- (Using normal size gas pipes (6 mm OD or larger) would lead to very long reaction times)

◆ On site mixing of small flows hard

- Long flow measurement times
- (almost) out of range of commercial mass flow controllers

◆ \Rightarrow use premixed gas bottles

1/32" tubing

