The Nikhef gas filling system

Gas mixture in light-weight JSP bottle

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RD51 Mini workshop
CERN, 25 February 2010

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°: 04/SP/767-Index0-REV.3.

Manufacturer: JSP/PORTINOX, S.A.

Concerned Equipment: Transportable refillable welded stainless steel gas cylinders.
Drawing N°: 101.000.17 of 03rd March. 2004

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

Type approval mark: B04/180

The manufacturer is allowed to affix the π mark (followed by our n° 0029) to approved equipment in the conditions described in article 10 of the Directive, only if the requirements stated in module D or F are fully complied with.
The approval is valid until 01st April 2014

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.

Notified body identification number: 0029 **Signature:**

Notified body stamp: APRAGAZ Belgium Inspecting Authority

Eng. Ph. HERMAN
Cylinders Dept. Head

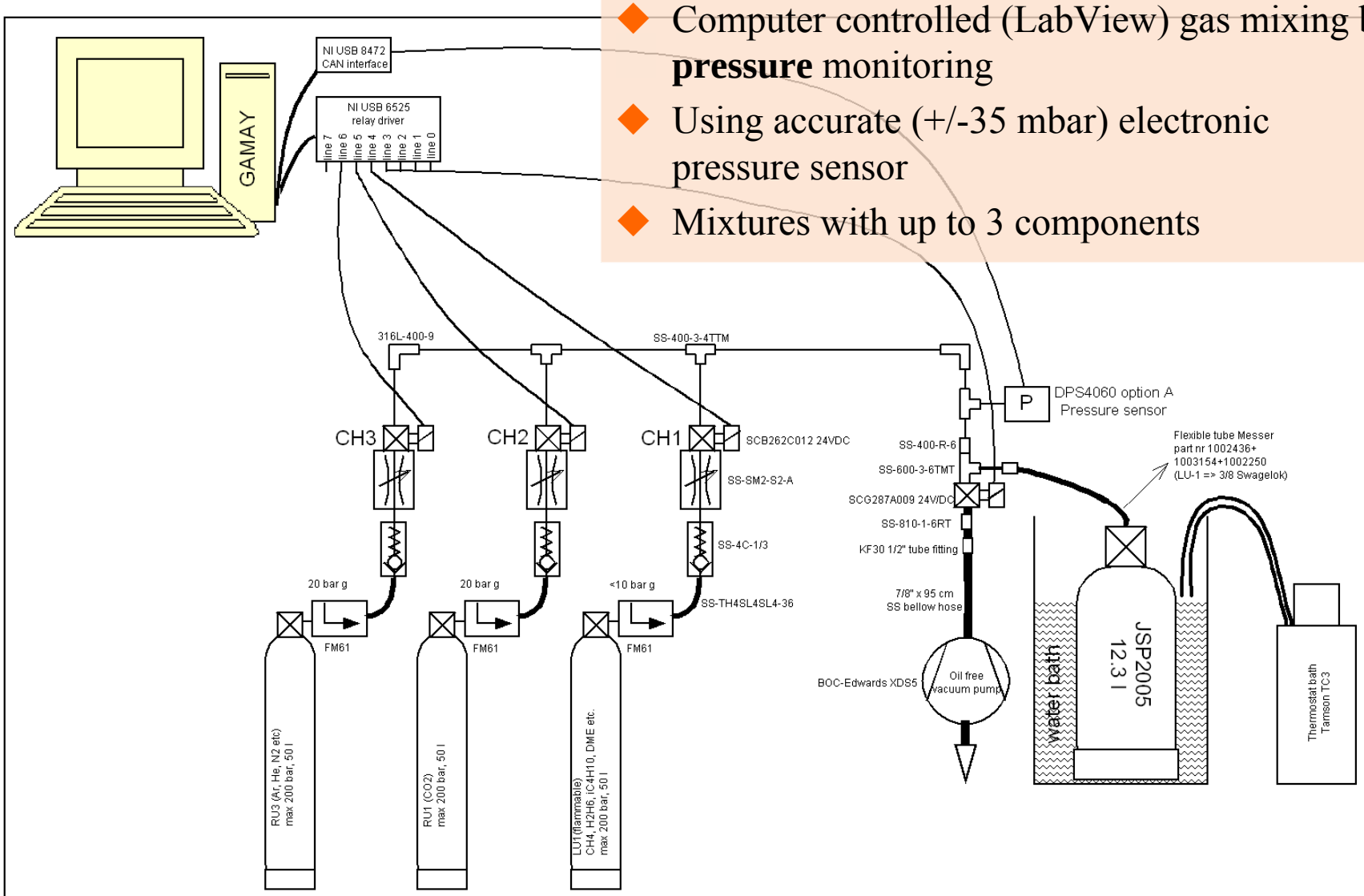
Notified body reference: 0405/B.0165

BELTEST EN 45004 Cert.N°147-I

BELCERT EN45011 Cert.N°039 PR

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

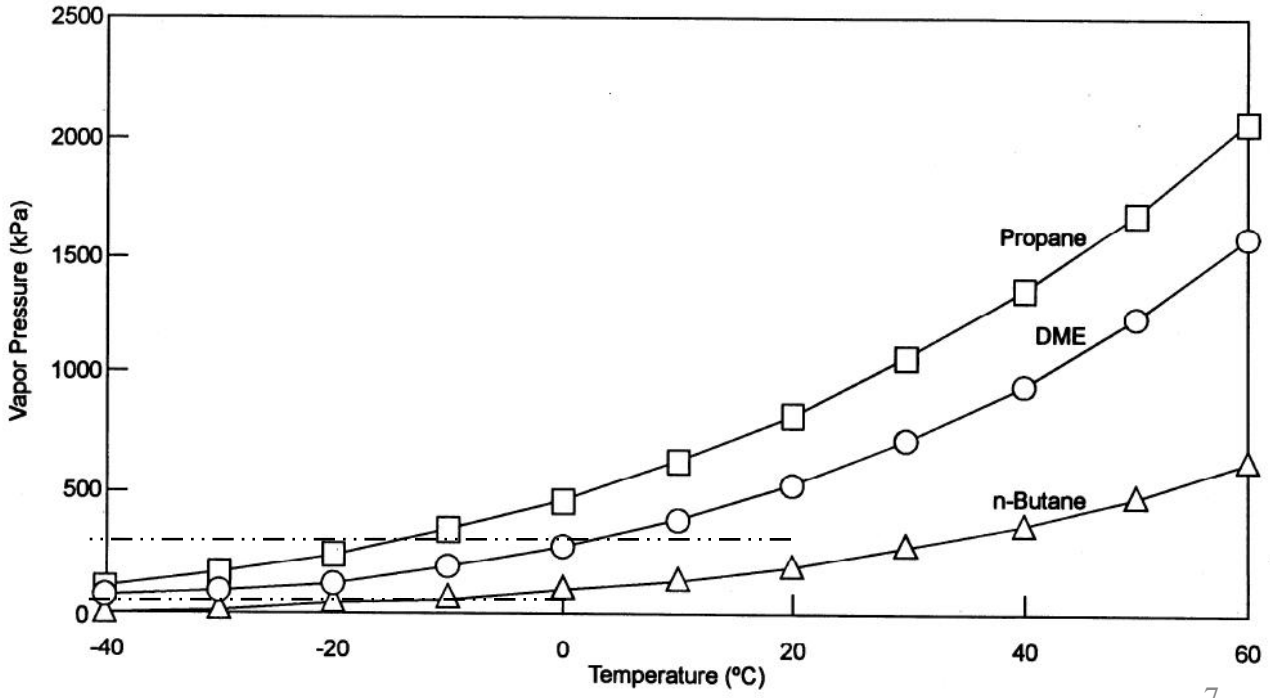
Fred Hartjes

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 ºC
    - => Ar/iC_4H_{10} 50/50 can be no more than 5.2 bar abs or **4.2 bar gauge**
  - DME 5.1 bar @ 20 ºC
    - Boiling point – 24.8 ºC



Expected performance

- Accuracy of the component fraction 0.1% of total mixture
- Deviation from required mixture 1% of total mixture
- Example:
  - Target mixture Ar/CH_4 90/10
  - Realized mixture will be something like Ar/CH_4 (90.08±0.1)/(9.92±0.1)
- Contamination
  - O_2, N_2, H_2O 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_4H_{10} 80/20	8.8	2.2	10	123	**26.0**	17.1

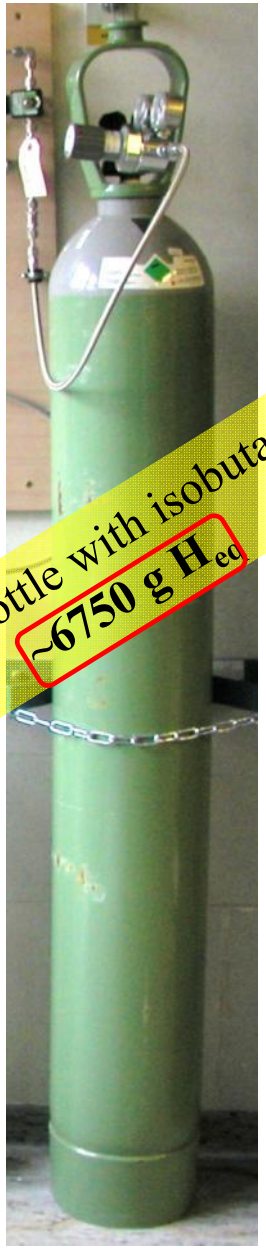
Safety in perspective

Premixed JSP bottle with DME/CO_2 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 ~ 30 g H_{eq}
- Well within Risk Class 1 (< 400 g H_{eq})
- One should wish having another Risk Class (< 40 g H_{eq})
  - Range of spray can

FLAMMABLE GAS SAFETY CODE
Annex A

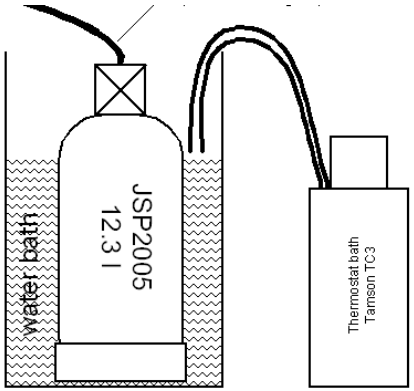
Flammable Gas Safety Manual

List of

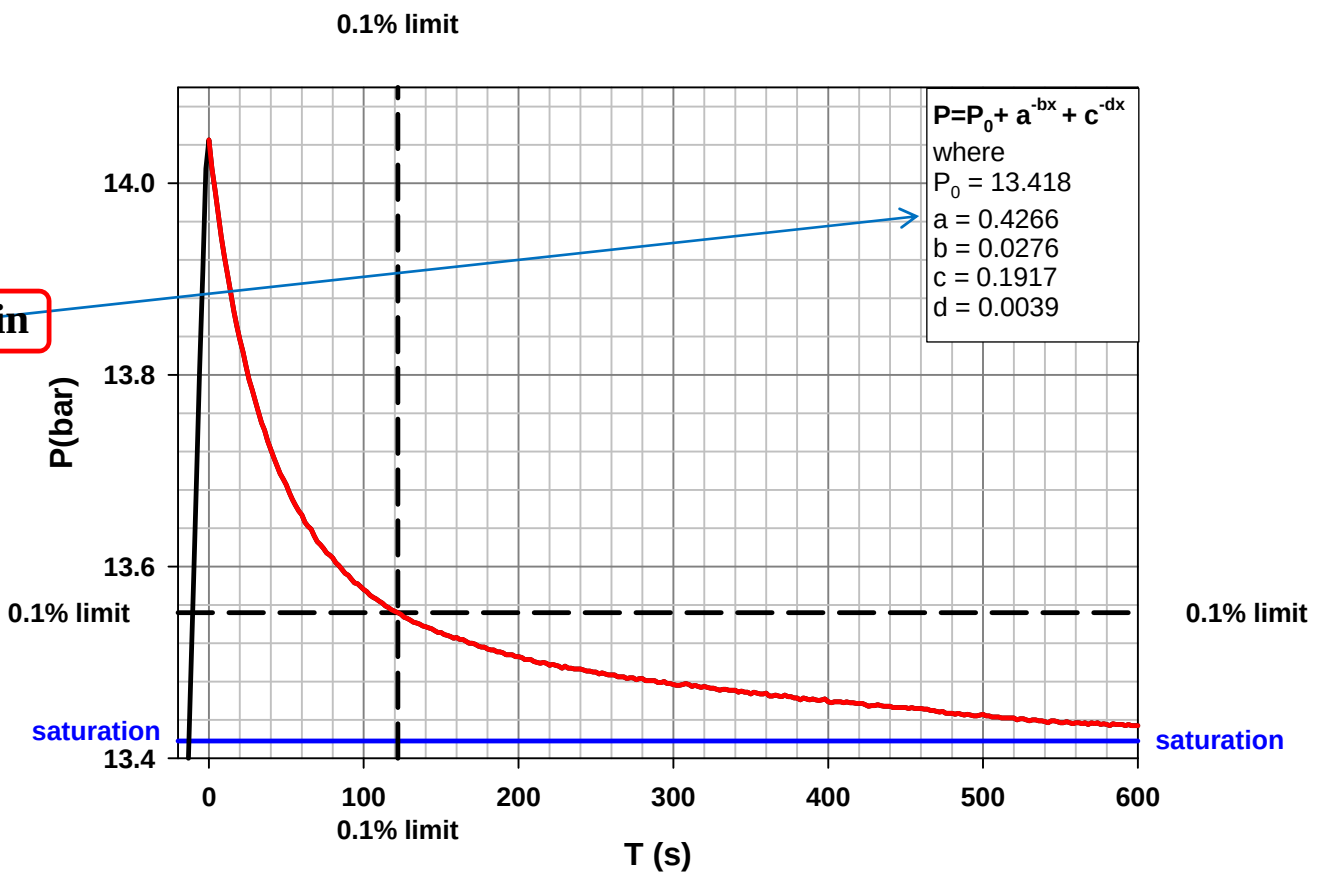
Risk Class 0	: Q_{tot} = 0 the use of non flammable gases or mixture(s)
Risk Class 1	: Q_{tot} < 0.4 kg (approximately equivalent to 1 kg of hydrocarbon) risk of a small local flash fire or explosion
Risk Class 2	: 0.4 kg < Q_{tot} < 40 kg (approximately equivalent to100 kg of hydrocarbon): risk of a local fire or explosion
Risk Class 3	: Q_{tot} > 40 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
  - **τ = 36 s and ~ 4 min**
- After 2 min within 0.1% limit



Pressure(P) vs time (T) after filling



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

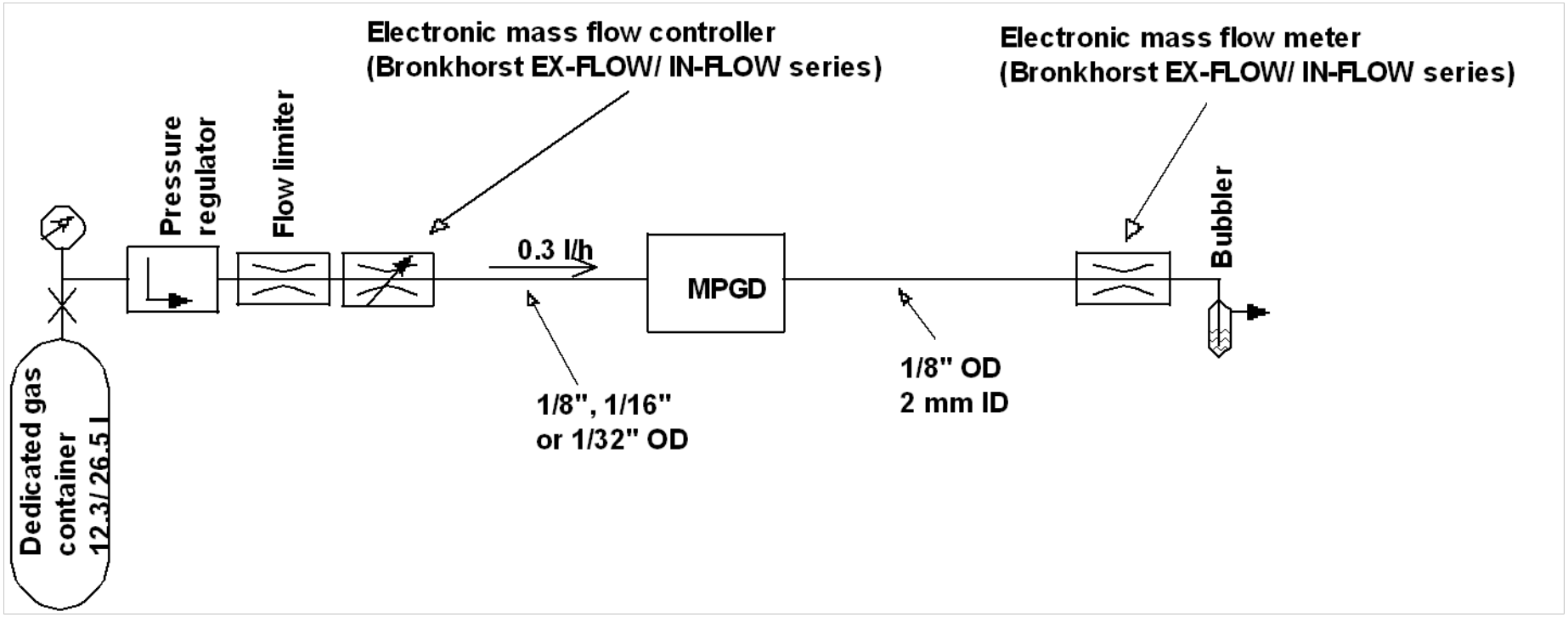
- Using premixed gases in JSP bottles has many advantages for low (< 1 l/h) flow applications
  - Thin gas pipes (1/8”)
  - Premix gas bottles in lab/ test beam area
  - Simple, non critical (1 channel) gas regulation
  - Easy and cheap experimental set-up

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

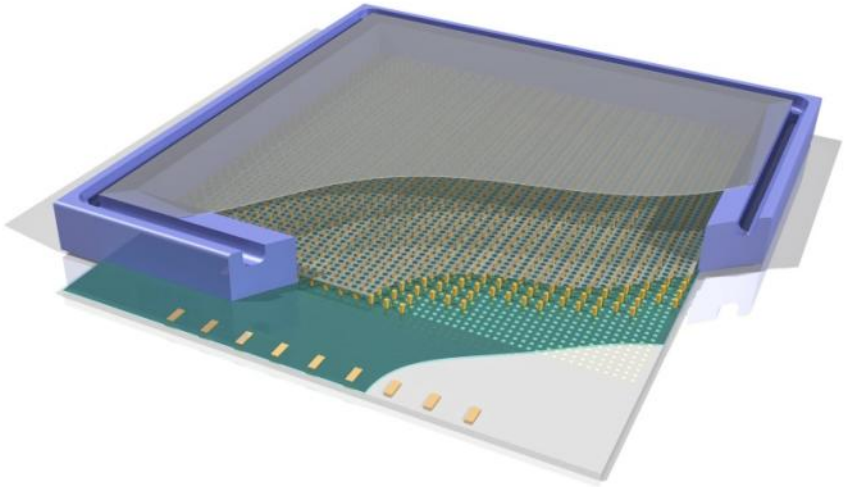
Spare

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”)

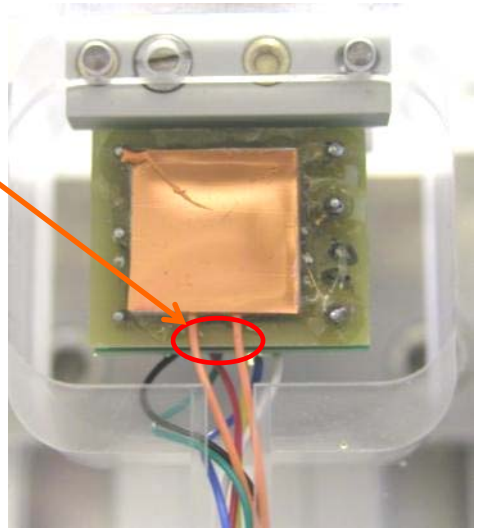


What is different for single chip MPGDs?



- Very small detector volumes
  - 1 Gossip detector ≈ **0.2 ml** (15 x 15 x 1 mm^3)
- => very small gas flows may be used
  - 10 volume changes/hour
  - => 33 µl/min (**2 ml/h**)
  - Commercial mass flow controllers go down to ~ 2 ml/min FS
    - => permit flows down to 100 µl/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 CO_2 and 0.12 – 0.3 l/h
    - => Back pressure 10 – 24 mbar
  - Gas line of 1/64" (≈ 0.4 mm OD) not excluded
    - 3 m tubing **0.25 mm ID** with CO_2 and 0.12 – 0.3 l/h)
    - => 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
- => use premixed gas bottles