



Instruction

Liquid Nitrogen filling from the storage tank in
26.34.U2.064

Date: September 26th 2025

1. Supply Concept
2. Material Hazards
 - Cryogenic Temperature
 - Inert Gas
3. Tank System Hazards
 - Storage Containers
 - Storage Tank System
4. Safety Devices
 - Technical Measures
 - Personal Protective Equipment (PPE)
5. Filling Process



Conceptual planing

- Higher supply security through stockpiling on campus
- Lower liter price by eliminating personnel provision from external companies
- Procurement and billing centrally by the ZCL
- Improved availability through:
 - automated reordering
 - extended filling hours
 - faster availability
 - filling of containers independent of external companys

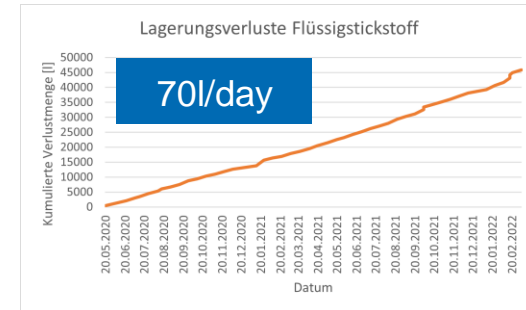
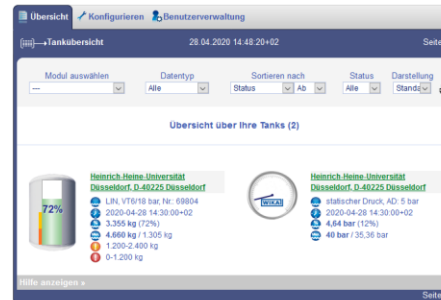
Implementation

- The tank system capacity is sufficient for approximately 4 weeks
- Reduction from 0,70 €/l in the container service to 0,30-0,40 €/l in the tank
- Operator responsibility for the tank system centrally managed, responsibility for internal transports decentralized.
- User-independent resupply
- Filling system available on business days
- No preorder deadline or waiting time
- Filling by trained employees of HHU from the using operational units



Tank system

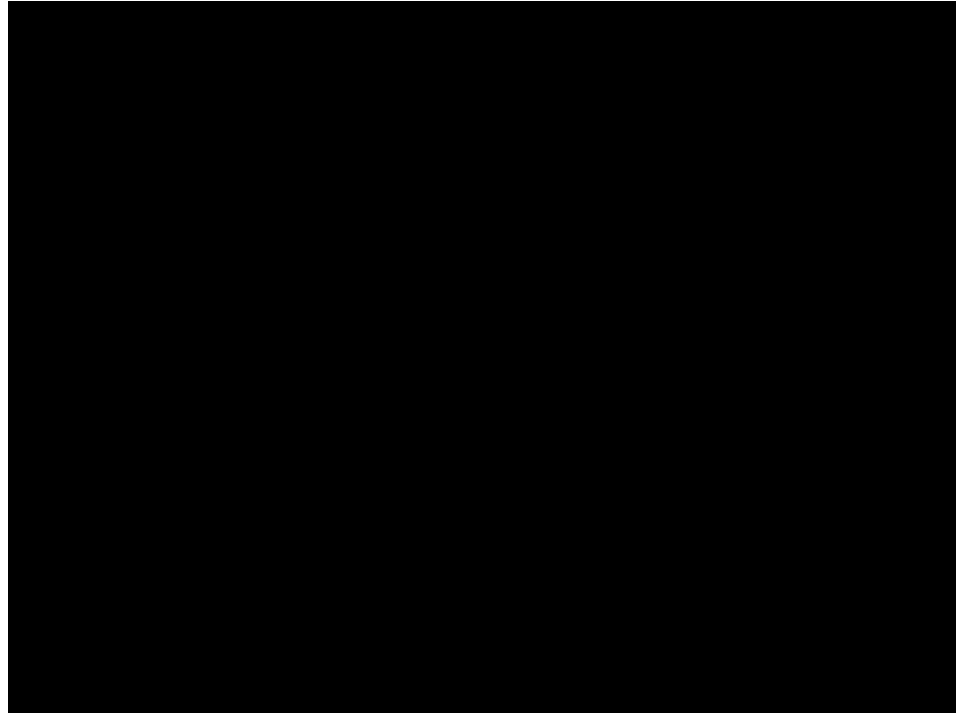
- 6.000 l storage tank for ~80.000 l/year
- Storage filling from the delivery area
- Only liquid phase filling
- Remote monitoring via GSM
- Warning at 50% and 25%
- Loss rate of the tank 0.29 %/day
- Total loss rate ~20%/year



Any questions
regarding the concept?

Hazards due to ultra low temperatures

- The boiling point is at -196°C / 77 K
- Frostbite
 - The Leidenfrost effect creates an insulating gas cushion at a high temperature difference.
 - Good heat conductors (e.g. a wedding ring) quickly interrupt the effect.
 - Frostbite can occur, particularly with jewelry.
- Ice formation on uninsulated surfaces.
- Materials will become brittle



Hazard due to phase transition

- Liquid with a density of 0,807 kg/l
- Rapid volume expansion during the phase transition
 - One litre of liquid phase produces 694 l or 0,694 m³ of gas phase
- Pressure build-up in closed vessels and systems
- Displacement of breathable air
 - The content of a 50 l container generates 34.7 m³ of gas phase.
 - A 50 liter container is sufficient to make three-person office unusable.
- Cold gases sink to the ground, displacement of the breathable air starts at the bottom of the room.



Hazard due to oxygen deficiency

- Nitrogen is odorless and reduces the oxygen level.
- ASR A3.6 specifies a minimum of 19% oxygen for workspaces
- DGUV I 205-006 states that a level above >17% is considered safe
- < 12% health risks cannot be excluded (DGUV I 205-026)
- The adjacent table is translated from „BAUA Gefährdungsfaktoren Erstickten, Ertrinken“

O ₂ -content in the air	Hazards and Effects
21 – 18 Vol.-%	Affected persons may not exhibit any noticeable symptoms
18 – 11 Vol.-%	Without the affected individual noticing, physical and mental performance abilities are impaired.
11 – 8 Vol.-%	Possible unconsciousness within minutes without warning. Below 11%, risk of death.
8 – 6 Vol.-%	Unconsciousness after a short time. Resuscitation is possible if performed immediately.
6 – 0 Vol.-%	Immediate unconsciousness. Brain damage, even with rescue.

Hazards due to careless handling of the equipment and containers

- Mechanical hazard due to tipping or impact.
- Thermal hazard due to cold components and released contents.
- Suffocation hazard due to unintentional release (tipping, opening of the discharge valve).
- Explosion hazard due to faulty or blocked safety valve.
 - Only for containers with threaded connection/ pressure build-up device.

Preliminary: Protective measures

- Qualified personnel
- Instructed personnel
- Tested material
- Personal protective equipment
- Risk assessment: e.g. pushing loads

Hazards due to tank system

- Bursting of the system
- Cold surfaces
- Formation of Ice layers
- Unintentional release leading to
 - Suffocating Atmosphäre
 - Extremely cold temperatures
- Power connection of the dead man's switch, 230 volts
- Above the filling station is a line for compressed air with ~6 bar pressure





Questions regarding the
hazards?

Safety measures: Instruction

- Only instructed personnel may perform the filling operation:
 - Key distribution only to instructed personnel
 - No key transfer between users
- The instruction must be repeated annually and documented
 - Personnel instructed in the ZCL are considered multipliers and may instruct one level of additional personnel for a 12 months period.
- Permissions are stored in the ZCL, and email reminders are sent before expiration.

Unterweisung Flüssigstickstoffabfüllung

Institut / Dezernat / Arbeitsgruppe	ZCL-Kundenkennung
[Analytische Chemie]	[CO2-Beispielheimer]

Es wurde durch einen im ZCL unterweisenen Multiplikator eine Unterweisung der folgenden Personen zur eigenständigen Nutzung der Flüssigstickstoffabfüllung im Bereich 26.34.U2.064 mit folgenden Themen durchgeführt:

1. Grundlegender Aufbau der Abfülleinrichtung und Versorgungskonzept
1. Stoffliche Gefährdung durch Flüssigstickstoff
2. Anlagengefährdungen durch Dewargefäße
3. Anlagengefährdungen durch die Tank- und Abfüllanlage
4. Sicherheitsvorrichtungen der Anlage
5. Einbindung in die Gaswarnanlage
6. Bedienelemente der Abfüllrichtungen
7. Persönliche Schutzausrüstung
8. Notfallmaßnahmen, Rettungsweg und Notruf
9. Organisatorische und praktische Durchführung des Abfüllvorgangs

UW-Nr.	Anrede	Vorname	Nachname	Unikennung	Unterschrift
1	[Frau]	[Doris]	[Mustermann]	[must001]	
2					
3					
4					
5					
6					
7					
8					
9					
10					

Die Unterweisung ist für ein Jahr gültig und bei fortbestehendem Bedarf anschließend zu wiederholen. Die Daten werden im MACH-System erfasst, es erfolgt eine Information per E-Mail vor Auslaufen der Berechtigung.

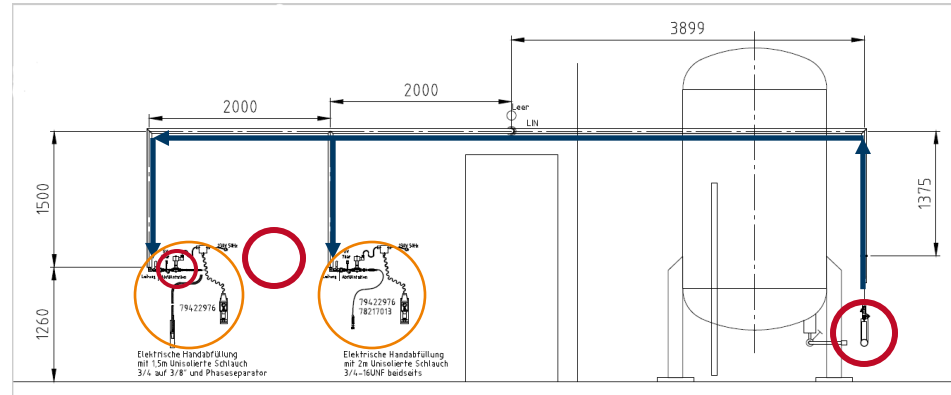
UnterweisendeR	Datum	Unterschrift
[Max Mustermann]		

v22/1

Form and Presentation are available at zcl.hhu.de

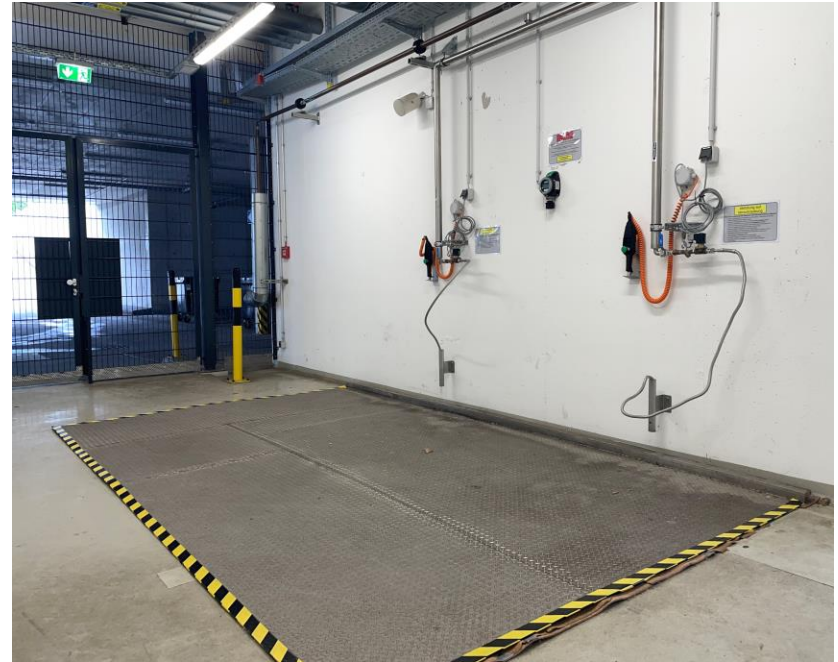
Basics: Structure of the filling system

- Vacuum-insulated pipeline to the filling station
- Pressure limiter 3-5 bar
- Two positions for simultaneous filling
- Left: sintered metal tip as phase separator
- Right: Connection tread $\frac{3}{4}$ "-16 UNF (corresponds to Cryotherm Apollo series, transition pieces to NPT $\frac{3}{8}$ "-18 and R $\frac{1}{2}$ " available for order in the ZCL)
- Oxygen sensor
- Pneumatic shutdown in case of oxygen deficiency warning
- Ball valve and dead man's switch



Structural safety measures

- Access restriction to the filling area and tank system
- Cross ventilation through lattice doors
- Blowing out excess nitrogen from the filling line into the outdoor area
- Touch protection for the filling line up to 2 meters in height
- Touch protection for rigid filling line through vacuum insulation
- Spatially limited metal plate for protection of the floor coating



Caution: Trip edge on the metal plate.

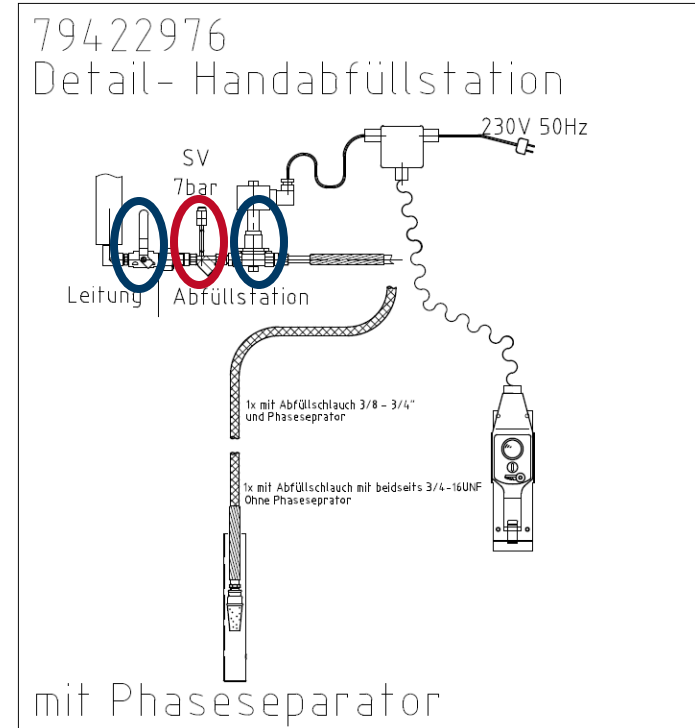
Gas detection system (GDS)

- Sensor for monitoring oxygen content at chest height
- Connection to the general gas detection system:
 - Automatic activation of an emergency valve on the tank and stoppage of the supply
 - Pre-alarm 19%: Visual notification
 - Main alarm 17%: Audible notification
 - Forwarding of the main alarm to the hazard alert station D6
 - Forwarding of alarms to ZCL personnel in 26.34
- After the main alarm: release of the tank system in the technical area required



Filling station

- Ball valve
 - The line is only open in the fully opened position.
- Solenoid valve with dead man's switch
 - Substance leakage due to unattended filling is prevented.
- Safety valve
 - In case of overpressure between the valves, the line automatically vents.





■ Safety glasses

- Goal: Eyes are protected against splashes caused by pressure surges

■ Laboratory coat

- Goal: Arms are covered, splashes are diverted

■ Cryogenic gloves

- Goal: In case of issues, the cold-exposed parts can still be operated at any time

■ Full length trousers

- Goal: Splash protection, liquid phase cannot enter the footwear from above

■ Sturdy closed footwear

- Goal: No pooling under the sole, mechanical protection during container movement

■ Hearing protection

- **Optional:** For containers with very small pressure relief.

Hearing protection & emergency call

- Hearing protection for containers with small pressure relief openings and high noise generation
- Applying according to the illustrated instructions
- Emergency phone:

112



Earplugs are provided in the filling area via a dispenser. Noise reduction rating: 36dB (SNR)

Suitable containers

- No visible damage
- No ice formation in the jacket area
- Stable and securely upright
- Wheels freely rotatable
- All wheels running smoothly
- Corks present and easily operable
- Pressure relief valve activates
- For small filling openings: Ensure sufficient immersion depth of the sintered metal tip.

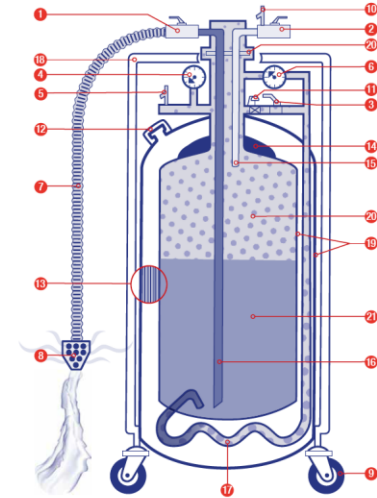


Fotos: KGW-Isotherm



Structure of a typical container

- Doppel walled vacuum insulation in the jacket area(12, 13, 19)
- Transport facilities and mechanical protection (9, 18)
- Safety devices (4, 5, 12)
- Optional: Lifting/Risiers (16) with pressure buildup mimic (17) and valves (1 ,2 ,3)
- Optional and under certain conditions useful accessories (6, 10, 11, ...)



Wesentliche Konstruktionselemente und Ausrüstungsteile

- | | |
|-----------------------------|---|
| 1 Entnahmeventil | 12 Vakuumverschuß- und Sicherheitsventil |
| 2 Abgas- und Überlaufventil | 13 Superisolation |
| 3 Druckaufbauventil | 14 Absorbens (Langzeit-Vakuumschutz) und Spezial-Getter |
| 4 Manometer | 15 Abgas- und Überlaufleitung |
| 5 Sicherheitsventil | 16 Entnahmeleitung |
| 6 Füllstandsanzeige | 17 Druckaufbauverdampfer |
| 7 Abfüllschlauch | 18 Fahrgestell und Schutzrahmen |
| 8 Phasenseparator | 19 Innen- und Außenbehälter |
| 9 Lenkrollen | 20 gasförmiger Stickstoff |
| 10 Druckabbauregler (opt.) | 21 flüssiger Stickstoff |
| 11 Druckaufbauregler (opt.) | |


Refer to the manual of the specific container!
Keywords: Transport, maximum capacity, operation,...



Tool for testing the pressure valve

Inspection of the pressure relief device

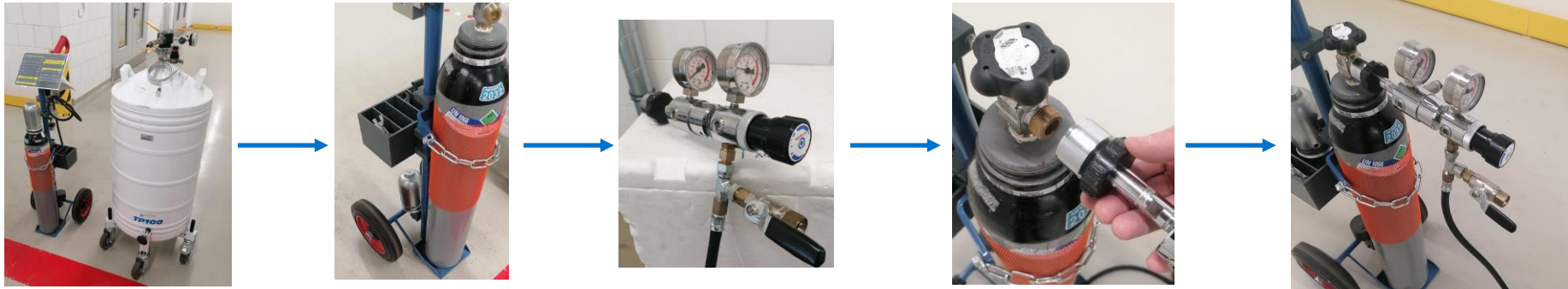
- Device for the annual inspection of the safety device on cryogenic containers with pressure buildup, consisting of:
 - Pressure gas cylinder
 - Pressure regulator
 - Connection nozzle and transition piece
 - Operating manual
 - Transport tool
- Available for daily rental in the ZCL
- Test documentation recommended both in the operating unit and on the container.

Überdrucksicherungsprüfung bei Flüssigstickstoffkanne mit Druckaufbau	
Prüfung nur an ungefüllter Kanne vornehmen.	Druckminderer über schwarzen Drehknopf weiter öffnen, maximal sind 3 bar möglich.
Prüfwagen und Kanne regengeschützt und standsicher nebeneinander platzieren.	Beim auf der Kanne aufgedruckten Überdruck  – in der Regel 0,5 bis 1,3 bar – soll die Sicherheitseinrichtung für eine erfolgreiche Prüfung ansprechen und hörbar abblasen.
Schutzkappe der Druckgasflasche abschrauben und auf dem Zylinder zwischen den Rädern ablegen.	Gasflasche am Hauptventil schließen.
Druckminderer aus der Halterung nehmen und ohne Werkzeug aufschrauben.	Druckminderer über Drehknopf schließen.
Prüfen, daß der schwarze Drehknopf zur Gasentnahme geschlossen ist.	Restdruck über Kugelhahn vorsichtig ablassen: Hebel mit der Flußrichtung!
Prüfen, daß der Kugelhahn geschlossen ist: Hebel quer zur Flußrichtung!	Flansch mit Dichtung und Klemme ODER Übergangsstück demontieren.
Flansch mit Dichtung auf der Kanne befestigen. ODER Übergangsstück aufschrauben.	Druckminderer demontieren und in Halterung einlegen.
Gasflasche am Hauptventil öffnen.	Schutzkappe wieder auf die Gasflasche aufschrauben.
Horchen: Zischt es am Aufbau? Falls ja, Verschraubung verbessern.	Prüfung auf der Kanne und in der Betriebseinheit dokumentieren.
Druckminderer über schwarzen Drehknopf leicht öffnen und etwa 0,3 bar einstellen.	Generelle Anforderungen an Flüssigstickstoffkannen: - Keine sichtbaren Beschädigungen - Keine Eisbildung im Bereich des Mantels - Lenkrollen frei drehbar - Alle Rollen leicht laufend - Stopfen vorhanden und leichtgängig - Überdruckventil spricht an
Horchen: Zischt es am Aufbau? Falls ja, Verschraubung verbessern.	

Grundlage der jährlichen Sicht- und Funktionsprüfung ist die BetrSichV.

Preparation for testing

- Set up the test device and the empty container side by side.
- Unscrew the protective cap of the gas cylinder and place it on the cylinder between the wheels.
- Take the pressure reducer from the holder and screw it onto the gas cylinder without any tools.



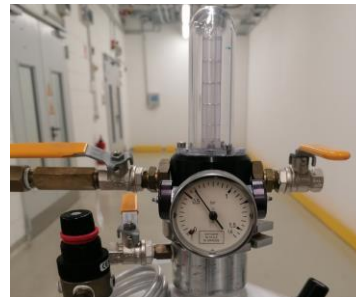
Preparation for testing

- Connect the black hose to the nitrogen tank
- Close the ball valve (1)
- Check that the valve for gas extraction is closed (turned out). (2)
- Careful open the main valve of the gas bottle (3) and check if everything is tight.
- The first manometer on the pressure reducer indicates the bottle pressure (4) after opening.
- The manometer for the final pressure should not show any pressure (5) and no gas should escape.



Conducting the test

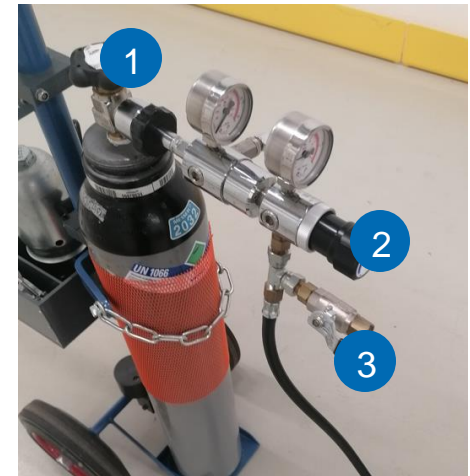
- Carefully turn the knob in until gas flows and pressure is applied to the kettle (1) (pay attention to the back pressure of the valve just before the flow opens)
- Increase the pressure until about 0.3 bar is set. (2)
- Listen to see if it hisses at the assembly. If so, improve the sealing.
- If not, increase the pressure in the container to just below the printed activation pressure for the safety device (usually 0.5 to 1.3 bar)
- Carefully increase the pressure further beyond the activation pressure, the safety device should audibly vent the excess pressure.



Dismantling of the test device

- Close the gas bottle at the main valve. (1)
- Close the pressure regulator by turning the knob (unscrew it) (2)
- Carefully release any residual pressure through the ball valve.(3)
- Disconnect the black hose from the can.
- Unscrew the pressure reducer and put it back into the holder.
 - (Note: If not done beforehand, also relieve pressure by briefly turning the black knob (2) to remove all pressure from the system pressure less, so that the connection to the gas bottle can be loosened by hand. Otherwise, up to 200 bar of pressure will work against you.)
- Screw the protective cap back onto the gas bottle.
- Document the test results on the container and in the operating unit.
- Return the testing device to the ZCL

**Dismantling
after the end of use
only depressurised!**



It is not allowed to tamper the safety devices Under any circumstances!

Section 145 of the German Penal Code

(2) Anyone who intentionally or knowingly(...)

2. removes, alters or renders protective devices useless, that are intended to prevent accidents or common dangers,

will be punished with imprisonment for up to two years or a fine

What if something unexpected happens?

<https://www.youtube.com/watch?v=U2QzpyQOIAg>

**No panic.
Stay calm.**

- In case of malfunctions: Interrupt the filling
- In case of main alarm from the gas detection system: leave the area
- In case of tipping over a filled container: leave the area
- Re-entry only after clearance measurement and deactivation of the alarm
- Support available by the ZCL personnel and the operations center (112)
- First aid kit and first responders are available in the ZCL
- Escape route: Proceed to the delivery yard and up the exit ramp



Questions regarding the safety
aspects?

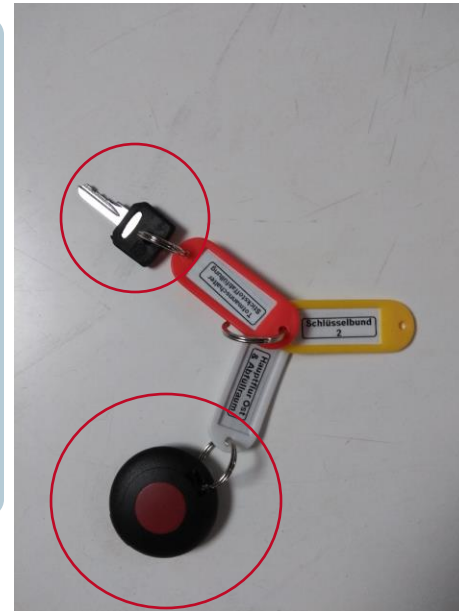
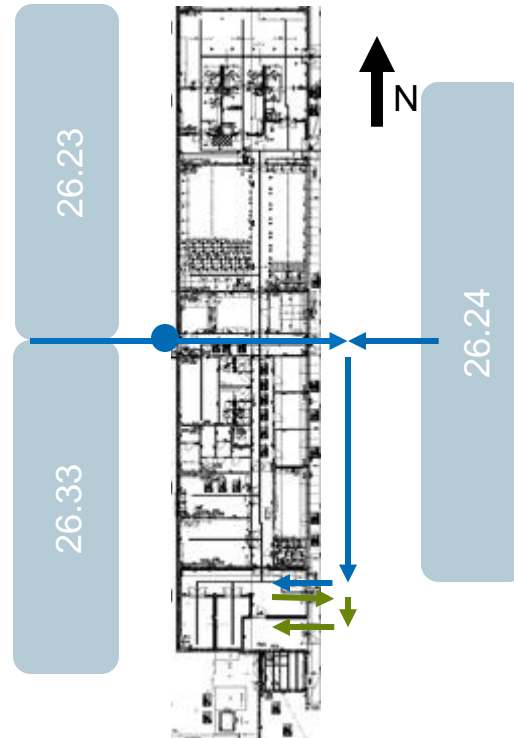
Preparations

- Filling is allowed :
 - Mon-Thu 8:00-11:30 AM and 1:30-2:30 PM
 - Friday 8:00-11:30 AM and 1:00-2:00 PM
- Fill out the chemical request form based on the nominal volume of the container
- Bring the empty container and the request form to the gas distribution area
- Submit the chemical request form
- Recieve the key ring in exchange for a deposit
 - For example, the employee/student ID
 - **No cash, no personal ID card**



Entrance

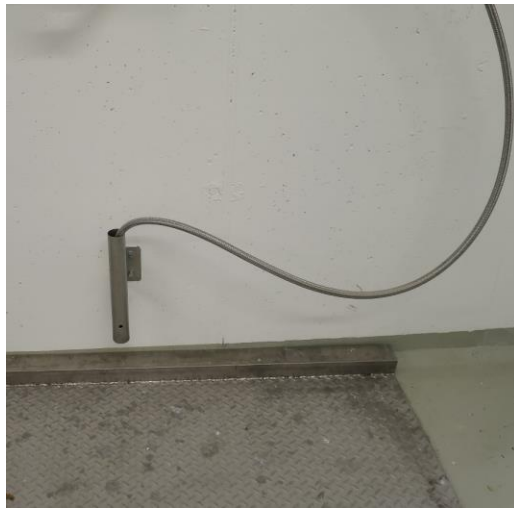
- From corridor 26.23/33.U2
 - Lift table system with „Chemikalienfahrt“
- Or from 26.24.U2
 - Via the delivery yard
- To the gas supply area
- Into the filling area
- Set of keys
 - 1: Gate to the filling area
 - 2: Dead man´s switch
 - If needed you can get a second key for the dead man´s switch



Filling process

Connection

- Note: Perform all work in the room while standing, never sit on the floor.
- Expose the filling opening of the container
- Take the flexible hose from the holder
- For free filling: Hang the phase separator into the neck of the container
- For threads connections:
 - Open the valve to the gas phase and turn it away from the oxygen sensor
 - Open the valve to the riser pipe
 - Close any pressure build-up valves
 - Screw the hose onto the riser pipe connection, a 24 mm wrench is required



Filling

- Open the ball valve: 'Lever parallel to the pipeline.'
- Remove the dead man's switch from the wall mount, unlock it with the key.
- **Manually** hold the dead man's switch pressed until droplets of liquid phase exit the can.
 - While free filling: the filling level can be slightly improved towards the end by slowly pulling the hose out.
 - Note: All hot components must be cooled down at the beginning of the filling process.



Filling duration: approximately 15 minutes for a warm 100 L can, when both positions are in use simultaneously.

Appearance: Free filling

- During all filling processes, freezing moisture in the air can cause significant fog formation.
- The fog simultaneously serves as an indicator for areas with reduced oxygen levels.
- The level of the gas detection system sensor and breathing zone must remain clear
- Observe the manufacturer's filling instructions: Liquefied gas vessels must never be filled to 100%!



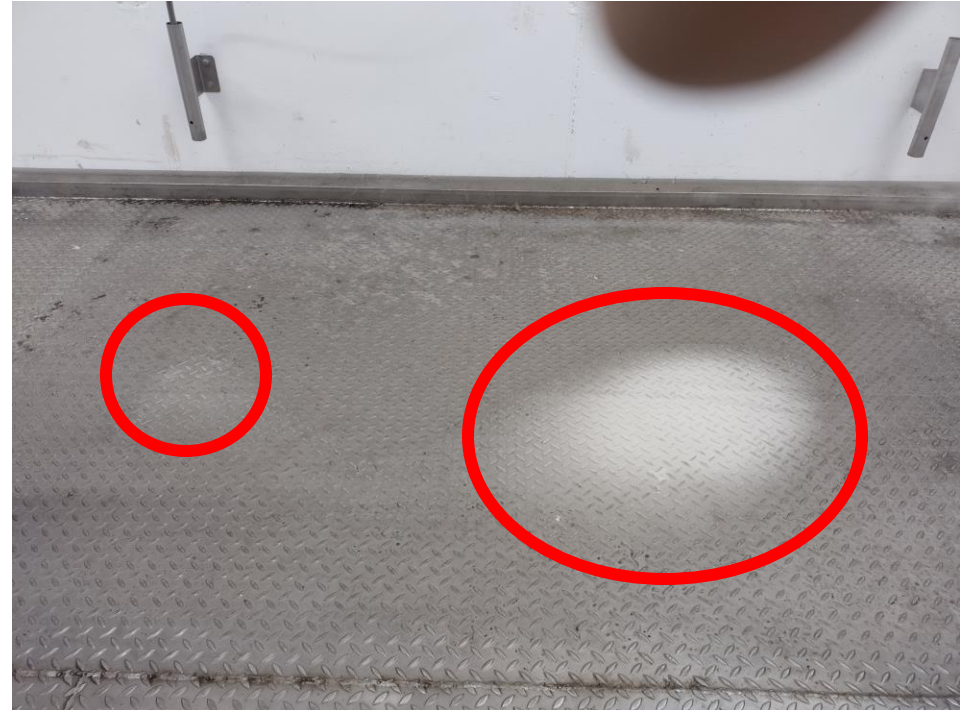
Appearance: Fixed connection

- Orientation never towards the personnel on the left side.
- Orientation never towards the wall below the gas detection system sensor
- Less fog due to higher flow velocity and faster distribution.
- If a large amount of fog forms: risk of unintentional liquid phase release.
 - Check the can's manual to see if the manufacturer specifies filling and transport without a dip tube!



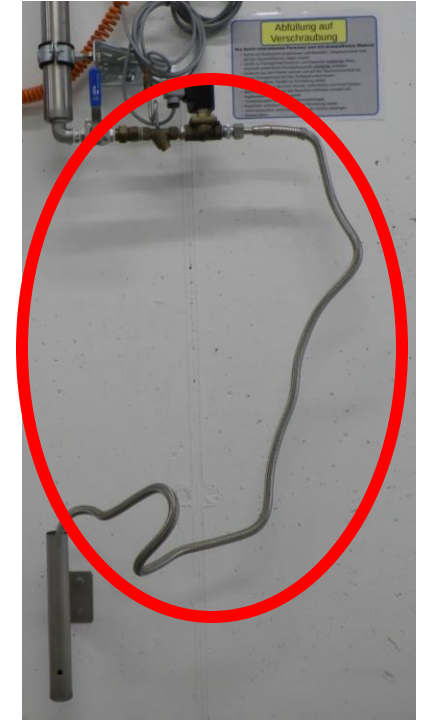
Damage potential 1

- Only small amounts of loss are harmless.
- Continued release overcomes the Leidenfrost effect.
- The liquid phase can overflow the protection
- Cooling leads to thermal shock for the plastic coating of the floor.
- Freezing does not occur while purging with dry nitrogen flow
- Frost formation occurs delayed after addition of normal moist ambient air.



Damage potential 2

- The threads of the fixed connection become cryogenically cold during the filling process.
- The union nut on the flexible hose can also freeze.
- Loosen the threaded connection using a 24 mm open-end wrench and a counter wrench. **Do not strike it!**
- Important: The union nut must be rotatable! Otherwise, the hose will be twisted and damaged.



Clean up after the filling process

- Lock the dead man's switch and remove the key.
- Hang the flexible hose in the holder
- Close the ball valve: „Handle perpendicular to the direction of the pipeline“
- Collect the carried equipment
- Leave the room
- Close the door
- Return the key ring to the gas distribution area



Any final questions?



Tour and signature