



Linde AG, division Linde
Engineering

Filling Instruction
MS-Adsorber (radial)
2601.01A.BA1/2601.01B.BA1

Doc.No.: &AB B-PP 260101BA1-3000

Issue: 4

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Instruction for filling - Content

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referred documents:

&AB B-SD 260101BA1-3000 "Technical Specification MOLECULAR SIEVE ADSORBER"
 &AB B-QA 260101BA1-3000 "Filling Protocol MS-Adsorber (radial)"
 &AZ B-PP 2626.LMS (EN) "Site Storage Instruction Adsorbent Material LMS920, LMS930, LMS935"

1. Scope of Application

This instruction was specially established for for first- and refilling of radial type molsieve vessels. It also includes instructions for the handling of molsieve material. Principally the same hints are valid for other types of molsieve vessels.

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	4	13.04.2015	MFCP	Firlbeck	AS/Bader	Firlbeck	completely revised
	3	17.01.2011	PAP	Firlbeck, Kiesel	Weilbuchner	Firlbeck	
	2	30.11.2010	PAP	Firlbeck, Kiesel	Weilbuchner	Firlbeck	
	1	25.11.2010	PAP	Firlbeck, Kiesel	Weilbuchner	Firlbeck	
	Issue	Date	Dept.:	Prepared	Checked	Approved	Remark

2. General remark

Please note:

All filling works (first- and refilling work) must be performed by experienced contractors which possess the required tools and are familiar with the filling works and the local safety precautions.

All filling works (first- and refilling work) and the required documentation in document &AB B-QA 260101BA1-3000 must be supervised by LE assembly personnel or an LE assigned contractor. Refilling works must be supervised by experienced client personnel.

The adsorption material - molsieve - removes humidity, carbon dioxide, hydro carbons (partly) and some components from the process air. These components could cause disturbances (blockage of main heat exchanger and others) in the low temperature section of an air separation plant.

These adsorption materials are very much hygroscopic. They adsorb the humidity from ambient air. This will reduce their process properties, which results in disturbances, while operating the plant later on. Therefore it is mandatory to avoid any unnecessary long contact with ambient air.

The contact with liquid water as well as with any other liquids (aqueous solutions, organic solvents, oils, etc.) must be absolutely avoided, since it will cause an irreversible damage of the adsorption material. This will make the adsorption material useless in the process of an air separation plant. Therefore it is absolutely not permitted to fill during even little rain (or snow). It is very difficult to detect such a damage visually by a change in colour and/or form.

During filling procedure it cannot be avoided, that the adsorption material gets in contact with ambient air. This contact time has to be reduced to an absolute minimum due to the reasons outlined above during the whole filling procedure. This is valid for all the actions from opening of the transport container (drum or big bag) until the materials are in the adsorber vessel and the vessel flanges are closed.

The molsieve material is very fragile. Therefore any mechanical strain (free falling material etc.) has to be avoided.

Basically all activities before, during and after the first filling and the refilling have to be recorded as a quality assurance measurement in the document &AB B-QA 260101BA1-3000.

The reference distance "Y" (refer to 8.3 and 8.4), for monitoring the filling height, has to be determined and recorded in document &AB B-QA 260101BA1-3000 during the first filling.


3. Safety Precautions

In contact with liquid water the adsorption material generates strong heat, which can also cause burns. According to the DIN Safety Data Sheet these materials are harmless regarding their toxic and environmental properties.

The use of tight clothes, gloves, goggles and dust masks while working with molsieve is mandatory.

4. Adsorption Materials

Depending on the size of the adsorber vessel, different types of adsorption material can be used. You can find detailed informations in document &AB B-SD 260101BA1-3000.

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5. Storage at site and inspection of adsorption material prior to filling

5.1 Storage at site

The document "Site Storage Instruction" &AZ B-PP 2626.LMS (EN) must be followed strictly!

5.2 Inspection of incoming goods

Immediately after delivery of the molsieve material the following items must be checked and noted down:

- Type (as marked on drums and/or big bags)
- Supplier (as marked on drums and/or big bags)
- Delivered quantity in comparison to the specified quantity acc. to &AB B-SD 260101BA1-3000
- Condition of the drums and/or big bags (acc. &AZ B-PP 2626.LMS (EN))
- Number of drums and/or big bags per same lot number

In case there are damaged drums and/or big bags (cracks, holes, etc.), they have to be sorted and handled according to document &AZ B-PP 2626.LMS (EN). This is also applicable for drums and/or big bags, which will be damaged later on during storage and/or intermediate transport.

If transport damages occur, a damage report has to be issued and sent to the transport and/or to the assurance company via site manager or project manager.

This material has to be reordered immediately.

5.3 Split of material

The material must be splitted into two portions. All big bags or pallets are continuously numbered. In general the complete material has to be separated in two parts by odd and even numbers. If there is any other separation required, a separate recommendation will be supplied by department RDA (LEHQ).

The material for each vessel shall be marked and stored separately immediately after arrival until filling acc. to &AZ B-PP 2626.LMS (EN).

6. Material quantity

number of units: 1
number of cases per unit: 2

type of filling	refer to "Technical Specification MS-Adsorber (radial)" &AB B-SD 260101BA1-3000
quantity of filling per vessel	refer to "Technical Specification MS-Adsorber (radial)" &AB B-SD 260101BA1-3000

7. Preparations

7.1 Time Schedule

For the filling of a radial adsorber vessel a period of not more than 2 to 4 days shall be considered according to the size of the vessel, the design, the number of working shifts, the available equipment and the availability of workmen. Unpredictable interruptions (weather, power failure, etc.) are not considered in this range.

In any case remember: **CARE HAS PRIORITY TO TIME SCHEDULE !**

7.2 Filling Equipment

The following filling equipment is necessary:

- fork lift truck
- mobile crane
- electric crane installed over the manhole for transport of persons
- transport bucket (same as a concrete bucket), with a filling capacity of 1,0 m³ for 4 drums or 2,0 m³ for 8 drums
- hose (Ø appr. 200 mm or 8", length each appr. 1,5 times the height of the upper platform over ground), the hose has to be smooth inside
- Hopper approx. 500x500mm with a connecting device for the above mentioned hose
- Shelter on the platform (approx. 3x3 m) over the manhole for protection against sudden rainfall
- optional: Covered platform (approx. 4x4 m) on the ground for the transfer from the drums and/or big bags into the bucket
- reinforced plastic foils to seal off of manholes and to cover the hopper and the open drums and/or big bags during interruption of the filling
- Several buckets (metallic with preference, since plastic parts may be charged electrostatically while transferring the adsorption materials)
- brooms
- vacuum cleaner (for cleaning inside the vessel before filling)
- scaffolding boards (without metal frames) or any other wooden or rubber boards (width ~500 mm, length ~2000 mm, thickness ~10 mm)
- lights within the vessel (low voltage, isolating transformer)
- big bags shall be filled in directly via a funnel if they have a molesieve outlet valve at the bottom
- other scaffolding equipment
- optional: laser distance meter for measuring of the reference distance "X" (refer to 8.2 and 8.4)

7.3 Protective Clothing

The following protective clothing are highly recommended:

- gloves
- goggles
- safety shoes
- tight overall
- dust mask (for workers outside the vessel)
- inhalation protection (for workers inside the vessel)

Clothes and shoes shall be so tight, that no adsorption material can enter it (heat is produced, when the adsorption material gets in contact with humid skin).

7.4 Light

Install sufficient lights outside and inside the vessel. Use low voltage and isolating transformer.

7.5 Cleaning Inside the Vessel

The complete supporting floor has to be cleaned (hand broom/vacuum cleaning). All foreign objects (tools, cloth, welding rods, etc.) which are not necessary for the further work have to be taken out. This applies also to the whole area below the filling zone (air inlet nozzle).

Any humidity within the vessel has to be removed.

After cleaning of the vessel no humid object or liquids (incl. any drinks) shall be brought into the vessel. Shoes and clothes must be dry whenever entering the vessel.

7.6 Inspection of Vessel and Internals

The following items have to be checked before filling starts:

- Cleanliness of the filling area inside the vessel
- concentricity of the two perforated cylinders (4 levels horizontally, each with min. 4 measuring points along the circumference)
- distance of the outer perforated cylinder to the inner wall of the vessel (4 levels horizontally, each with min. 4 measuring points - same measuring points as before - along the circumference)

The result of the check has to be recorded in document &AB B-QA 260101BA1-3000; detected defects have to be corrected prior to start of filling.

On both not perforated shells (outside of inner cylinder, and inside of outer cylinder) the filling height for the first filling (+1000mm) - refer to sketch 8.2

and

refilling (+850mm) - refer to sketch 8.3

is marked above the transition of the perforated to the not perforated part of the outer cylinder.

The markings are carried out either by different colours or by welded flat profiles (details see sketch 8.2 and 8.3)

They have to be checked and the result has to be recorded in document &AB B-QA 260101BA1-3000

further information about filling height, control of filling height and refilling see chapter 8.

Below the filling area the air inlet nozzle has to be covered by caps in the T-piece or elbow area, and the drain nozzle has to be plugged in order to avoid blocking by adsorption material during filling.

7.7 Handling of Drums

Take care, that no liquid is on top the cover of the drums that the molsieve material cannot be penetrated by water. Vent and open the drums only just before using them. First open up the small vent screw to release the vacuum, then open the cover.

7.8 Handling of Big Bags

Big bags consist of two layers, the reinforced outer and the gas tight inner bag. At the top part of the outer bag there are 4 hangers for lifting by crane. At the top and at the bottom there are funnel-shaped nozzles in both inner and outer bag, the one at the top is for filling the big bag, and the other at the bottom is the outlet. They can be opened to fill or empty the big bag.

Only at place of discharge (the big bag hanging above the transport bucket or above the hopper) the outlets of the big bag shall be opened. First the outer bag will be opened, then a loop has to be made using a rope around the outlet of the inner bag and finally the inner bag is opened. Using the loop or by lifting or lowering the big bag it is possible to control the discharge of the material.

7.9 Weather conditions

Generally the filling shall only take place during constant dry weather. In case rainfall is forecasted, only a limited number of drums and/or big bags shall be transported to the filling area in front of the adsorber, which are necessary for the actual filling.

Remember:

Any contact of molsieve material with liquidity has to be avoided strictly!

Additional remarks concerning weather conditions and interruption of filling procedure see item 9.

7.10 Safety Precautions

The complete equipment and the execution of works have to comply with the local safety regulations. In addition to that, all works have to be performed according to the "Linde Permit to Work" process.

The following minimum requirements have to be met:

- securing of breathing air
- wearing of safety harness and permanent contact to the flagman
- safeguarding by hoisting device outside the vessel
- necessary lights must be installed in a way, that no obstruction or risk of injury exists (no stumbling over cables, no burning on hot lamps, no short circuits, etc.).
- if welding is necessary, a dedicated "Permit to Work" has to be requested and released prior to work.

If an adsorber was already in operation (e.g. for refilling) at least the following actions (according to the "Linde Permit to Work") have to be considered:

- all inlet and outlet valves in piping to and from the vessel must be secured in closed position.
this includes:
 - blind discs placed
 - instrument air switched off for all valves concerned
 - electric connection to the actuators /solenoid valves interrupted
 - molsieve sequencer stopped and locked
 - signs placed at all places concerned (valves, actuators, electrical equipment, control room etc.)
- continuous controlling including alarm of oxygen content in the vessel (danger of fire and suffocation)
- written permit for work inside the vessel issued by the local safety authority

7.11 Split and Mixture of Material

(only applicable if material delivery contains different batch numbers see 5.3)

If any specific mixture is required, a separate recommendation will be supplied by department RDA (LEHQ). The results have to be recorded in the document &AB B-QA 260101BA1-3000.

7.12 General Remarks

While handling the adsorption material attention has to be paid in order to avoid breakage and dust formation due to abrasion.

Therefore it is important:

- Avoid respective minimize free falling height for adsorption material. This applies for transfer from drum and/or big bag into the transport bucket and into the filling nozzle via the hopper and for further distribution inside the vessel.
- Collect broken material and dump it.

8. Filling of the adsorber vessels

8.1 Execution

Prior to filling please check whether the upper facings of the flanges for manhole and the three filling nozzles are at the same level. If not, LE project management must be informed via LE site management!

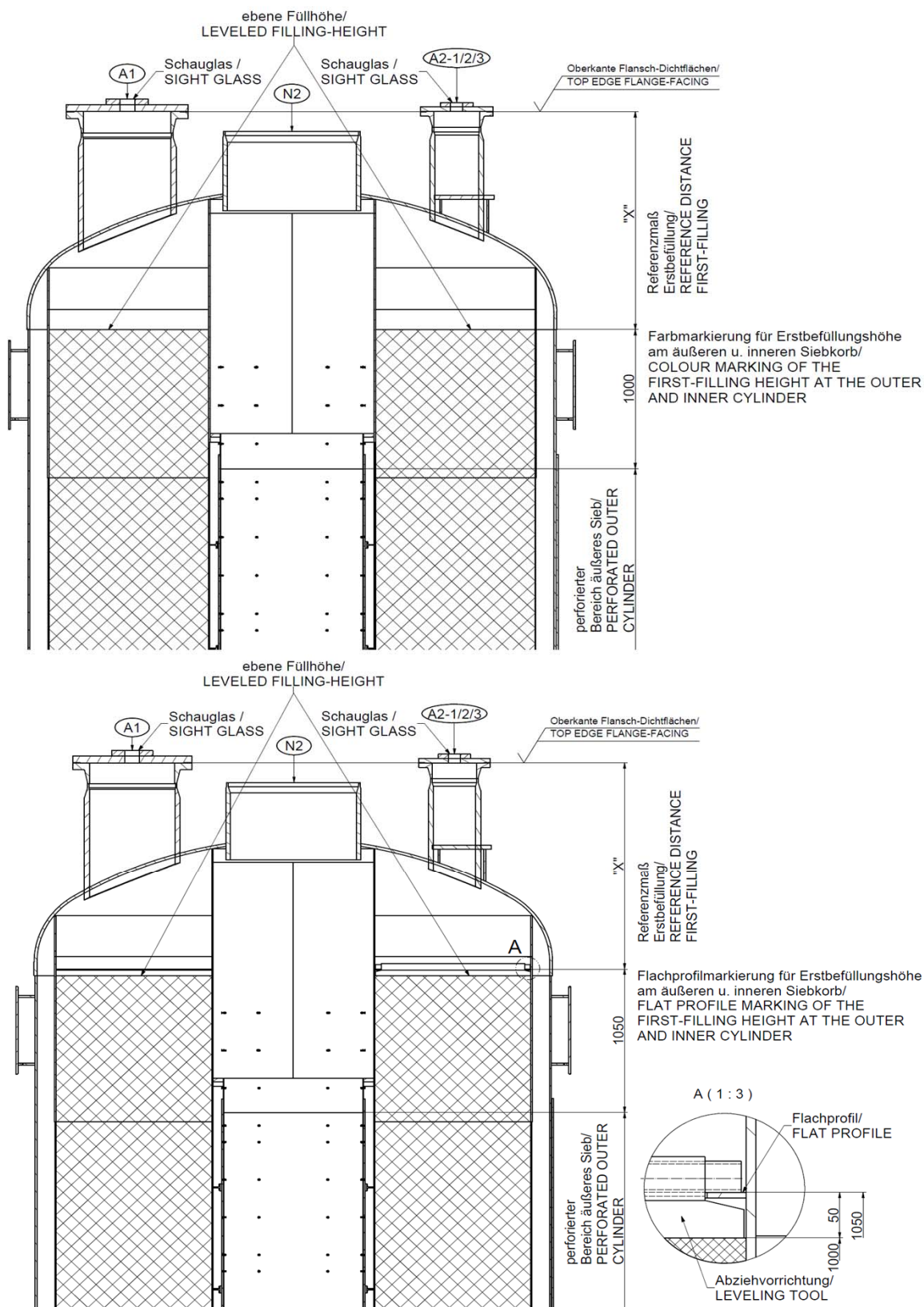
For filling of radial adsorber vessels the following method has been proven:

- a funnel (approx. 500x500mm, 500mm high) is placed right on top of the filling nozzle. Acc. to its dimension it might be necessary to install an additional platform around it to work on
- at the outlet of the funnel a hose (DN200, with smooth inner wall) is fixed and put into the vessel through the filling nozzle
- the length of the hose shall be cut, that it touches just the bottom of the filling area, while the hose is loosely wound around the inner perforated cylinder and its angle with a horizontal line is never more than 45°. Provide hangers for the hose
- Install a shelter above the filling nozzle and the manhole
- Install an electric crane over the manhole for lifting of persons
- transport the drums and/or the big bags on palettes by using a fork lift truck from the storage to the adsorber vessels. Possible requirements for separation and mixture of the material have to be followed
- record batch number of each drum and/or big bag along with number of drum/bigbag and designated vessel, in which the material is filled in and note it down in the document &AB B-QA 260101BA1-3000
- 4-8 drums (equal to 500-1000kg) can be filled into the transport bucket
- lift the transport bucket with the crane right on top of the hopper and fill it into the hopper.
- big bags (alternatively single drums) can also be lifted by crane right on top of the hopper and can be filled in directly.
- the defined filling height is either marked with different colours or by welded flat profiles above the transition of the perforated to the not perforated part of the outer cylinder. This height is also marked on the same height of the inner cylinder to get a plane surface.
- the material flows down the hose and has to be distributed in the vessel by a worker moving the open end of the hose. Differences in filling height of more than 0,1m should be avoided. By piling up layers of about 0,4m thickness the vessel will be filled more and more, meanwhile the hose must be shortened accordingly. The cutted hose pieces must be removed immediately.
- for leveling of the molsieve material at the marked filling height you can use a wooden board or the delivered leveling device (only delivered along with design using welded flat profiles for marking of filling heights).

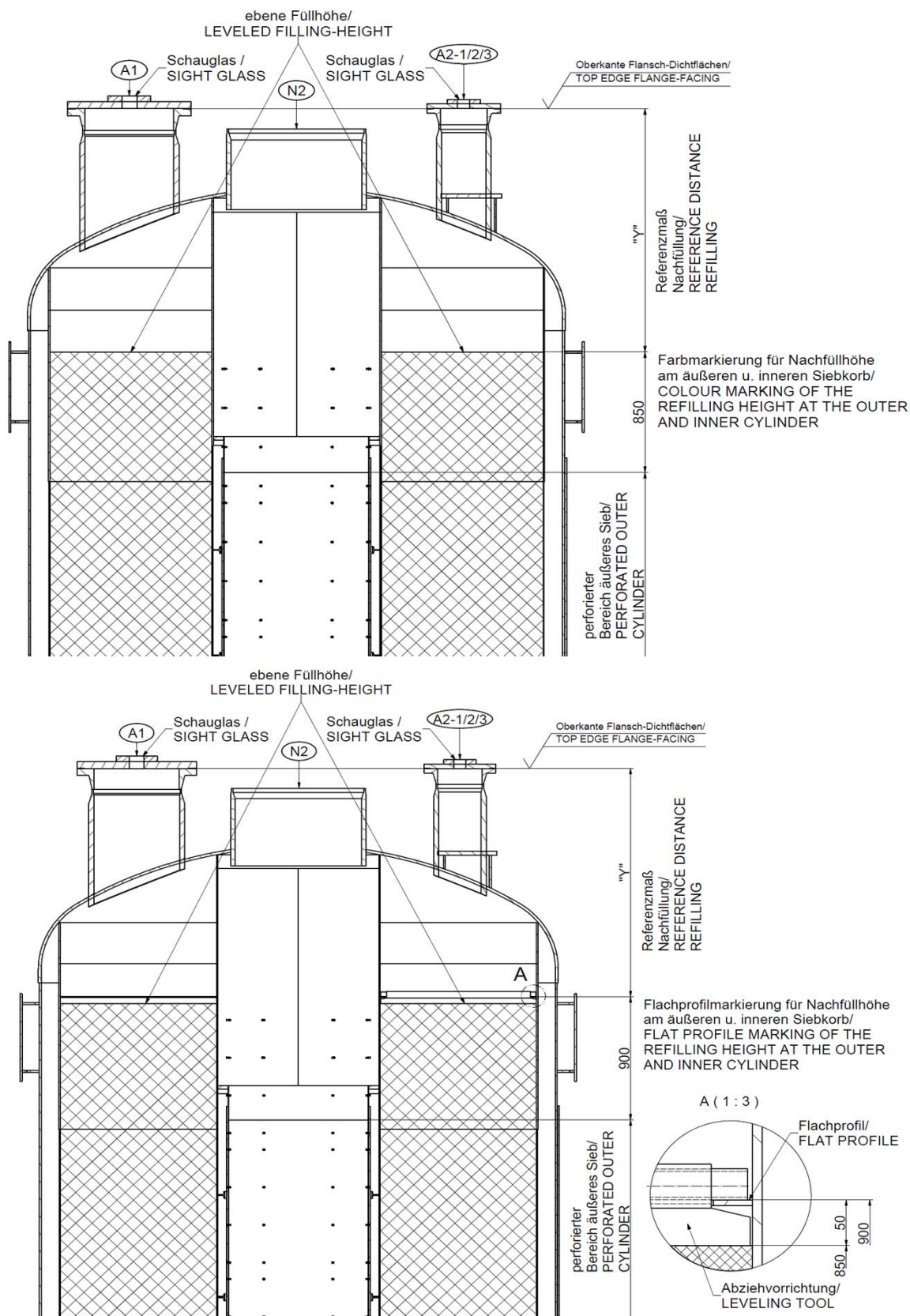
Please note that the surface of the filling level must be completely flat around the whole circumference.

Remark: the delivered quantity of molsieve material always includes a certain safety margin

8.2 Sketch for first filling



8.3 Sketch for refilling



8.4 Determination of reference distances "X" and "Y"

- for the first filling a filling height of +1000mm above the perforated area of the outer cylinder is required to compensate the settling during the initial operating phase.

This filling height for the first filling is marked at the outer and inner cylinder by a coloured line at +1000mm or a flat profile at +1050mm above the perforated area for leveling of the molsieve material (refer to sketch 8.2)

After finishing the filling up to the marking of +1000mm the reference distance "X" between the filling surface and the inspection glass surface has to be measured by a laser measuring device.

Calculation for the reference distance **"Y" (mm) = "X" (mm) + 150mm**

both distances "X" and "Y" have to be recorded in doc. &AB B-QA 260101BA1-3000

- the reference distance "Y" represents the minimum filling height of +850mm above the perforated area of the outer cylinder.
- In case of an overfilling +1000mm above the perforated area an accumulation of H₂O and CO₂ is most likely due to insufficient regeneration of the upper zone.

8.5 Control of filling height

- during the first year of operation a control of filling height (reference line "Y") through the sight glasses by a laser measuring device every 2-3 months is mandatory. Settling of filling height up to max. 250mm below reference line "Y" can be tolerated.
- in the following years the control frequency may be extended to one or two times a year.

8.6 Refilling

- Refilling to reference distance "Y" (+850mm) is necessary at the latest if the level has decreased more than 250mm under reference line "Y".
This design filling height (+850mm) is marked at the outer and inner cylinder by a coloured line or a flat profile above the perforated area for leveling of the molsieve material (refer to sketch 8.3).
- In case of the filling height decreases more than 350mm below the reference line "Y" a CO₂ brake through can occur.

9. Interruption

In case of starting rainfall any filling work has to be interrupted immediately. In such a case as well as during any other longer interruption (like over night) the nozzles have to be closed tight with foils or any gas tight cover.

Already opened drums have to be closed completely with the original cover and fixation. Insert the vent screw. In addition, those drums have to be covered by a plastic foil. Drums and/or big bags have to be stored in a dry shelter.

All equipment, which has been used for filling and remains outside (bucket, hopper, hose etc.), shall be protected against humidity (dew, mist, rain, etc.) by plastic foils duely secured also against wind. Only after all rainfall has stopped completely, the used equipment, nozzles etc. shall be dried carefully and filling can be resumed.

Any contact of molsieve material with liquidity has to be avoided strictly.

10. Retain samples

During the first filling at least three molsieve samples of each vessel shall be taken. The quantity of each sample shall be about two litres. In each vessel at least one sample shall be taken from the bottom, one from the middle, and one from the upper section of the molsieve filling. If possible each sample shall be taken from a different batch of material. Suitable sample containers are dry bottles of polyethylene, PVC or glass, which shall be closed gas tight just after the sample being collected.

All sample shall be marked as follows:

- Date of sample taking:
- LINDE's Project No.:
- LINDE's Code:
- Type of molsieve and supplier:
- Batch number:
- Vessel Tag No.:
- Sampling position in the vessel: (bottom - middle - top)

11. Termination

After all adsorption material has been filled in and the final levelling has been done, all objects, tools boards, lights, cables, etc. have to be taken out of the vessels. In case the final surface has been damaged, it must be rearranged.

The device for flattening must be handed over to the client for refilling works.

The vessel area under the filling area has to be cleaned (vacuum cleaner). The caps in the T-piece or elbow area, and the drain nozzle have to be removed. Special care shall be taken, that no material or other objects/ tools are left in the process nozzle and in the drain line.

All nozzles have to be closed properly by flanges using proper gaskets. Please take care, that the sight glasses are cleaned properly and the filling underneath is visible.

All required informations acc. to document &AB B-QA 260101BA1-3000 have to be recorded including the total filled in quantities per batch and adsorber for the first filling and the remaining rest of adsorption material per vessel.

The filling instruction has to be considered for first filling and for every refilling - as well as all actions have to be recorded in the Filling Protocol &AB B-QA 260101BA1-3000.

The protocols have to be handed over by the responsible supervisor to the following persons:

- startup manager (for implementation into the operation manual)
- client plant manager (for safekeeping of correct filling height during refilling)

A signed copy of the a.m. document &AB B-QA 260101BA1-3000 and the collected samples have to be sent to:

LINDE AG,
Gas and Engineering
Linde Engineering Division
Dep.: RDA Mr. Dr. Grahl
Dr. Carl-von-Linde-Strasse 6-14
D - 82049 Pullach
Germany

12. Extraction of Adsorption Material

12.1 Necessary equipment

For dumping of the adsorption material, the following devices in addition to equipment mentioned above are necessary:

- Long hoses (\varnothing appr. 200 mm or 8", length ~three times the distance of the platform to the ground), the hose has to be smooth inside.
- Vacuum suction vehicle
- Container for transport and dumping

12.2 Actions

12.2.1 Adsorption material will not be reused:

The adsorption material will be sucked off the vessel via a hose by a vacuum suction vehicle, loaded in a container and dumped.

12.2.2 Adsorption material will be reused:

Reusing of adsorption material is only possible if the material can be regenerated adequately and it was not damaged during sucking. In this case please contact "Customer Service Air Separation Plants".