

LABODEST 104

Operation Manual

- General Instructions for the Assembly of LABODEST and AUTODEST-Systems
- Labodest 104 : Drawings and Legends
- Distillation Control System D 401 incl. Adapter Unit

General Instructions for the Assembly of LABODEST- and AUTODEST-Systems

The systems are assembled in accordance with the corresponding drawing. Prior to the description of the assembly itself, please find below some general advice and information.

1. Mounting Frame, Clamps and Supports

For mounting the systems, we use grid-type framework made of welded steel tube material, i.e. for the frame rectangular tubes are used and for the grid tubular rods with a diameter of 13 mm. The framework should be anchored in the ceiling, in the wall or in the floor.

For mounting the glass parts, standard laboratory clamps will be sufficient holders. Flasks and larger receivers will be placed on rings.

The assembly of systems with flanged connections requires a reference point in the frame.

2. Handling of Ground-in and Flanged Connections

Depending on size and intended use, the glass parts will be equipped with taper-ground joints, ground-in ball-and-socket joints and flanged joints.

Any and all ground-in and flanged joints have to be cleaned very carefully before the assembly. They must be absolutely dustfree. Ground-in joints, which have to be greased, must be cleaned by means of solvents. If for some reason or other a joint has to be undone, it has to be cleaned and newly greased before re-assembly.

Taper-ground joints are greased by means of a high-vacuum grease of good quality. Apply several thin, parallel strokes on the cone. When the joints are put together, the air can escape between these grease layers. The grease layers have to be as thin as possible. In case the cone is scarcely accessible, the sleeve is to be greased. The cone is inserted into the sleeve without canting, respectively the sleeve is pushed over the cone. Then the two ground sections are worked into each other by turning until they are completely transparent and free from reams. Please ensure that the ground sections are not canted against each other, when they are clamped together, otherwise the ground section will become leaky. Moreover there will be the risk of breakage.

Ground-in ball-and-socket joints are greased very slightly and regularly with a high-vacuum grease. Afterwards the ground-in ball-and-socket joints are nested into each other. Turn the ball in the shell until this ground section will be transparent and free from reams. Contrary to taper-ground joints, the ground-in ball-and-socket joints can slightly be canted against each other.

Grease-free ground-in ball-and-socket joints. This flexible joint consists of two semi-spherical joining pieces which are clamped together. For sealing purposes, the ball is fitted into a silicone rubber gasket, which is coated with either Teflon or Viton.

Flanged joints. These joints can be of various types and shapes, depending on the customer's requirements. We mainly use KF-joints as ground-in ball-and-socket joints.

Flanged joints are always assembled by means of metallic clamp straps. The clamp straps are slid over the glass tubes with flanges. The interior of the ring is provided with an insert; by means of screws, the ring is then pressed onto the glass flange. In case of KF-flanges a spiral or plastic ring is used as insert which transfers the clamp strap's pressure onto the arched glass rim of the tube. Before the assembly, the surfaces of the plane, resp. spherical flanges have to be cleaned very carefully. Furthermore the sealing must be dustfree. It is placed between the two ground front surfaces of the glass tubes.

The clamp straps are turned such that the screw holes at both tube ends are situated opposite each other. KF-spherical flanges may slightly be canted against each other. The screws of the flange rings are tightened carefully one after the other so that unilateral strains on the glass tubes will be avoided.

The screws have to be fastened thoroughly, but carefully. After the initial operation, the screws have to be tightened again due to the shrinkage of the sealing. This is to be done with utmost care. In case of a too strong or unilateral tightening of the screws, the flanges might be crushed.

Screwed joints. The screwed joints consist of thread, screw cap and Teflon-coated silicone sealing. Such screwings are used preferably for lines as well as for thermometers and other connection nozzles.

3. Reference Point of the Column

At one point of its lower part the column has to be firmly clamped or has to be positioned in such a way that the respective support will hold the whole system. All other clamps above this reference point will only serve for maintaining the column in vertical position. They must not be tightened to an extent which will impede the thermal extension.

Distillation flask with flanged joints can be arranged on a platform with a clamp strap. In case of larger flasks, it will, however, be better to place the flask in a support at the bottom of the oil-bath.

Rotary evaporators are equipped almost always with clamp straps. They are placed onto a platform. Evaporators of larger size are placed on a platform which at the same time holds the clamp strap of the immersion heater.

In case of batch distillations, a frequent exchange of the flask will be necessary. In this case, the upper end of the lower column section is to be the reference point; the flask is mounted from below to the column section.

4. Assembly of the System

The assembly starts either with the distillation flask, the evaporator or the lower column section.

In case of discontinuous distillations, the flask is in general heated by means of an oil-bath. In exceptional cases, the oil-bath must be substituted by a direct electrical heating. Besides, metallic flasks with electrically heated oil-jackets, which are welded all over, are applied, too.

Distillation flask or evaporator as reference point. If the flask itself is chosen as reference point, the assembly has to start with the oil-bath. By means of a platform lift, the oilbath is placed into the framework and is adjusted such that - when lifting the platform - the guiding tube, which is welded to the frame, will go into the sleeve fixed at the external pot of the oil-bath. Then the magnetic stirrer is placed into the oil-bath.

The flask is placed into the oil-bath; for larger-sized flasks, the oil-bath is equipped with an adequate support. Smaller flasks with taper-ground joints are clamped in the ground section. In case of ground-in ball-and-socket joints, the clamp is attached directly beneath the ball. In both cases, the rim of the ground section, resp. the ball must rest on the clamp in order to prevent the flask from slipping down. For flanged joints, a platform is used with the clamp strap placed on rails. Rotary evaporators are fixed correspondingly.

Then the column is mounted to the flask (distillation flask or evaporator). First of all, the heating jacket (if any) is fastened to the frame. Please ensure that the internal tube is in an exactly vertical position above the flask. The column section is inserted from above. It may not contact the heating jacket. At any rate, please ensure that the column section is arranged vertically and without strain inside the heating jacket. The upper end is fitted with a clamp, which is, however, not intended to bear the weight of the column, but to keep it in vertical position.

In the same way, further column courses and column tops, coolers, etc. are installed. Subsequently, accessories, like e.g. receivers, water feed heatings, etc. are mounted. Finally the thermometers are inserted and, in case of systems with vacuum operation, vacuum pipings are arranged. Subsequently a leakage test is carried out.

Lower column course as reference point. If the flask of the system must be exchangeable, because it is to be taken out for cleaning or because flasks of different sizes will be used or because the system is intended for continuous or discontinuous operation, the weight of the column may not act upon the flask. In this case, the lowest column section is used as reference point.

The assembly starts with the heating jacket for this section. It is screwed to the frame vertically and at an adequate height. The column section is inserted into the heating jacket and is fixed so as to carry the weight of the system.

Then the oil-bath is mounted from below as well as the other column sections, coolers, etc. (see above) one after the other.

5. Leakage Test

The equipment is evacuated to final vacuum by means of the vacuum pump. Then a shut-off device in the vacuum piping is shut and the manometer has to be observed. The pressure increase inside the system shall not exceed a value of 2-3 mbar/15 min.; in case of high-vacuum systems, the requirements are even more rigorous. Also in case of systems operating at normal pressure, we recommend a vacuum test.

When the system is sufficiently tight, the cooling water hoses and electric lines are arranged. For this purpose the respective wiring and mounting plans have to be followed.

If all coolers are fed with cold water, the water is first of all conveyed through receivers, distillate coolers, second condensers and finally through the main condenser. The water feed has to be connected to the coolers such that the cooling spirals and cooling jackets are filled without bubbles. In general, the water has to flow upwards. In case of intensive coolers with spirals and external cooling jacket, the water should first pass the spirals and then the cooling jacket. The cooling water safety device is to be installed in the discharge. Coolers of very different cross-sections should not be arranged in series.

In case a cooling water heating facility is available, the heating jackets of the receiver, the distillate cooler and the main condenser are flushed one after the other with warm water, while the second condenser is fed with cold water.

Flexible Joints

Rotulex™-System

This flexible joint, patented by SOVIREL consists of two semi-spherical joining pieces which crush an elastic and inert silicone rubber sealing with either PTFE- or VITON-protective coating.

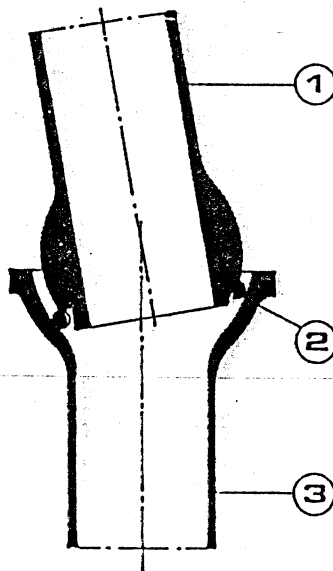
Output Data:

Operation Temperature : Max. 200°-250°C

Pressure Resistance : 0.5-6 bar depending on
size and accessories (nippers or
flanges)

Dynamic Vacuum Stability : 10⁻⁴ Torr with compound sealing
10⁻⁷ Torr with VITON-sealing

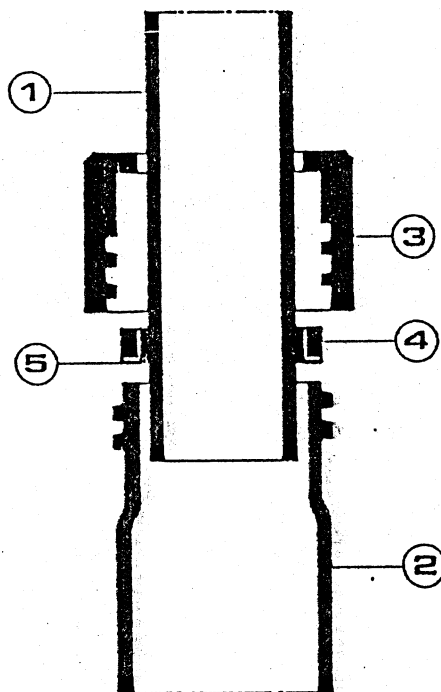
Chemical Inertness : 732-01 (the same as for
glass) and PTFE
resp. VITON



Screwed Joint System

Screwed joint cap (3) as well as silicone sealing (4) with PTFE-sleeve packing (5) to be slid over the object, which is to be pushed through (thermometer, tube, etc.). The PTFE-surface must face the reaction space.

Screwed joint cap (3) to be slid over the silicone sealing with PTFE-sleeve packing and screwed tightly on the threaded tube (2).



Couplings and Gaskets

An essential factor for operational safety and extent of maintenance regarding glass installations is - apart from the coupling flange systems - the correct choice of the clamp straps and gaskets. The criteria to be considered are:

- Nominal width of the components
- Pressure
- Temperature
- Kind of media to be processed
- External corrosion.

The joints consist of split and one-piece clamp straps imparting the screwing forces via shims onto the glass flange.

Clamp Straps and Shims

- For nominal widths ranging from DN 15 to DN 300:

For this purpose, one-piece clamp straps made of the following materials are used:

: Plastic-type, identification letter "K"

Material: Acid- and solvent-proof phenolic resin

Admissible Operating

Temperature: 130°C

Shims: Made of plastic material (DUROPLAST)

: Silumin-type, identification letter "S"

Material: GK Al Si 12 - DIN 1725

For increased interior pressure and temperatures up to 200°C

Shims: Up to DN 100 = plastic material

DN 150 - DN 300 = SILUMIN with asbestos strips

Moreover in case of extreme stress, the Silumin-rings may also be provided with a corrosion-resistant coating and/or the shims may be of special steel.

- **For nominal widths ranging from DN 300 to DN 600:**

In the field of system engineering, grey cast iron clamp straps (identification letter "G") of split execution are used for the entire admissible range of pressures and temperatures.

The shim consists of a glued caoutchouc-asbestos-strip.

- **For a nominal width of DN 1000:**

Sectional steel ring of split execution with glued caoutchouc-asbestos-shim, again for the entire range of pressures and temperatures.

Gaskets

On account of its universal chemical stability, PTFE is used as sealing material.

Screws and Nuts

The standard equipment of such couplings includes screws of cadmium-plated execution. For extreme operational conditions, the screws and nuts are made of special steel.

Assembly and Commissioning

After the assembly of the glass parts, the following procedure is to be carried out in order to safeguard a constant soundness of the sealings:

- If possible, heat the all glass parts to the intended operating temperature.
- Tighten the joints again in cold state by means of the indicated torques.

This procedure guarantees the best possible setting of the PTFE-gasket such that the cold flow, which will still occur, will be compensated for by the flexibility of the joint.

The admissible torques for thightening the KF-joints can be taken from the following table:

Screw-tightening Torque in Nm for clamp straps made of			
Nominal Width	Plastic (K)	Silumin (S)	Grey Cast Iron (G)
15	1.0	1.0	-
25	2.5	2.5	-
40	3.0	4.5	-
50	3.0	4.5	-
80	3.0	4.5	-
100	4.5	6.0	-
150	4.5	6.0	-
200	4.5	6.0	-
300	4.5	6.0	6.0
400	-	-	9.0
450	-	-	9.0
600	-	-	15.0
1000	-	-	22.0*
			* = sectional steel ring
	glass part clamp strap gasket		glass part clamp strap with shim gasket
joint DN 15-300 "ball cup"		joint DN 300-600 "plane"	

