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Schlenk Tube Technique

(Supervised by Ph. D. Masato Tanaka)

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Schlenk Tube Technique

Original work and supervision
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Chemical Resources Laboratory

1. Introduction

Experimental methods in inert atmospheres have become indispensable not only in inorganic and organometallic chemistry, but also in synthetic organic chemistry and materials science. The number of glovebox-equipped laboratories has risen in recent years, allowing experiments using this equipment to be conducted.

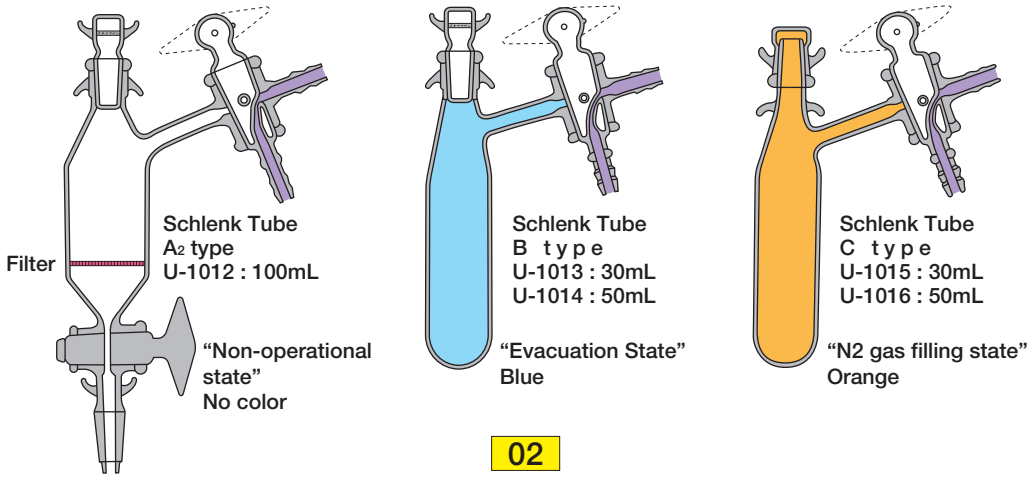
The **glovebox method** is incredibly convenient, as it allows experiments to be conducted under controlled internal conditions, with inert gases from which oxygen and moisture have been removed circulating throughout the box. Frankly however, not only are the procedures for introducing reagents and glasswares to the glovebox main body and antechamber, etc., complicated, and time-consuming. In addition, the operator's face can become itchy at a crucial point in the procedure.

In order to use the glovebox method on a regular basis, laboratories must have as many gloveboxes as the number of workers and pay high prices for the equipment itself and running costs. Furthermore, there is no hope for a sufficient number of the equipment installed, where the space is highly limited. Under such circumstances, the **Schlenk-tube method** remains fundamental in experiments using oxygen- and moisture-sensitive substances, and especially in experiments handling organometallic compounds.

A glass apparatus invented by **Dr. W. Schlenk** is used for the Schlenk method. This apparatus is called the **Schlenk tube** and has been developed into a wide variety of more convenient apparatuses by subsequent researchers and glass craftsmen. To do a good job in any field, whether it is science, art, or sports, it is essential to select, master the use of, and apply the proper tools.

Likewise, to do a good job using the **Schlenk method**, it is essential to select, master the use of, and apply the proper tools. This manual will explain the manipulation of the **Schlenk tube** and the basic apparatus that can be used with it.

Note that in the following explanations, all manipulations under inert gas are given with **N_2 gas**, but argon is used in exactly the same way.



Text will be color-coded in the following way to allow easier understanding:

- "Non-operational state" prior to start of the experiment – No color
- "Evacuated state" with vacuum pump – Blue
- "N_2 gas filled state" – Orange

More comprehensive explanation on manipulations under inert gas is found in the references given below. *

Procedures are fundamentally identical, but some differences may arise depending on individual cases (and the researcher's preferences).

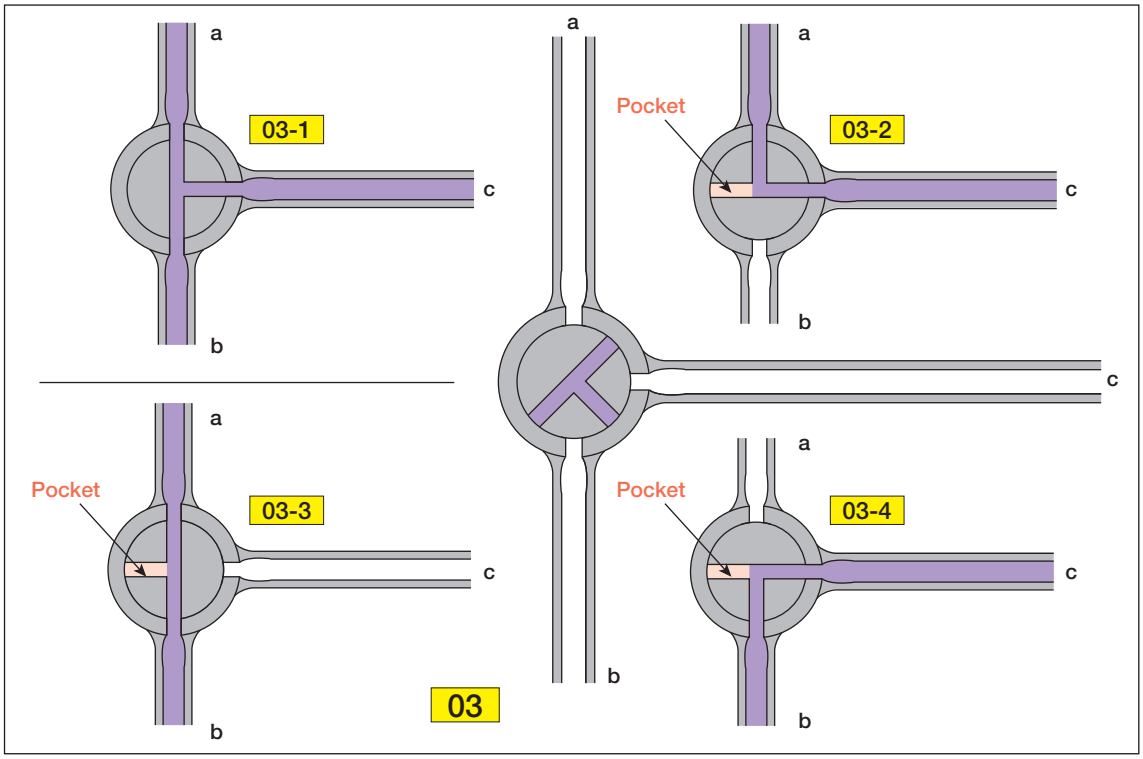
In addition, by understanding, mastering and applying various techniques, readers can expect to see more efficient progress in research and development.

*Note: D.F Shriver, translated by: Yoshito TAKEUCHI, Hitoshi MIKUNI, and Shūji TOMODA, "Manipulation of Air Sensitive Compounds", Hirokawa Shoten, 1972. 12; Akio Yamamoto, "Organometallic Chemistry", Shōkabō Publishing, 1982, p.178.

2. Schlenk Cock

The reader is likely be already familiar with the common **Schlenk Tube** (referred to below as **Tube**).

The Schlenk Cock (referred to below as **Cock**) used here is a **Stopcock** made of glass and is an indispensable component for simple, rapid, and reliable operations in a perfect inert atmosphere. The structure and operation of the **Stopcocks** are described below. In the Schlenk method, the **atmosphere inside the Tube or related vessels is replaced with N_2 gas by repeated evacuation and introduction of N_2 gas.** The **Cock** is used to select between the vacuum and **N_2 gas**.Its difference from the T-shaped-3-way **Stopcock** is displayed in 03 and 04.



1. As shown in 03-1, with the T-shaped-3-way **Stopcock**, all 3 directions (**a**, **b**, and **c**), can be connected at the same time. In this case, evacuation and **N_2 gas** input can occur at the same time.
2. Moreover, as shown in 03-2, when **a⇔c** is connected and **N_2 gas** is passed through, a pocket (dead space) arises in the flow path. Because it is impossible to completely remove the air (oxygen) in the pocket by simply passing **N_2 gas**, air contamination may occur.
3. In contrast, with the **Cock**, only 2 direction circuits, **A⇔C** / **B⇔C** / **A⇔B** are possible since the **Cock** is not a three way **Stopcock**, but comprises 3 sets of 2-way **Stopcocks**. Accordingly, there is no pocket (dead space). Even when the stopper is removed for sampling and addition of chemicals during the reaction. These operations can be carried out without air contamination by introducing **N_2 gas** through the **Cock**.

Accordingly, use of the **Cock** is preferable over the T-shaped-3-way **Stopcock** for the Schlenk glass apparatus.

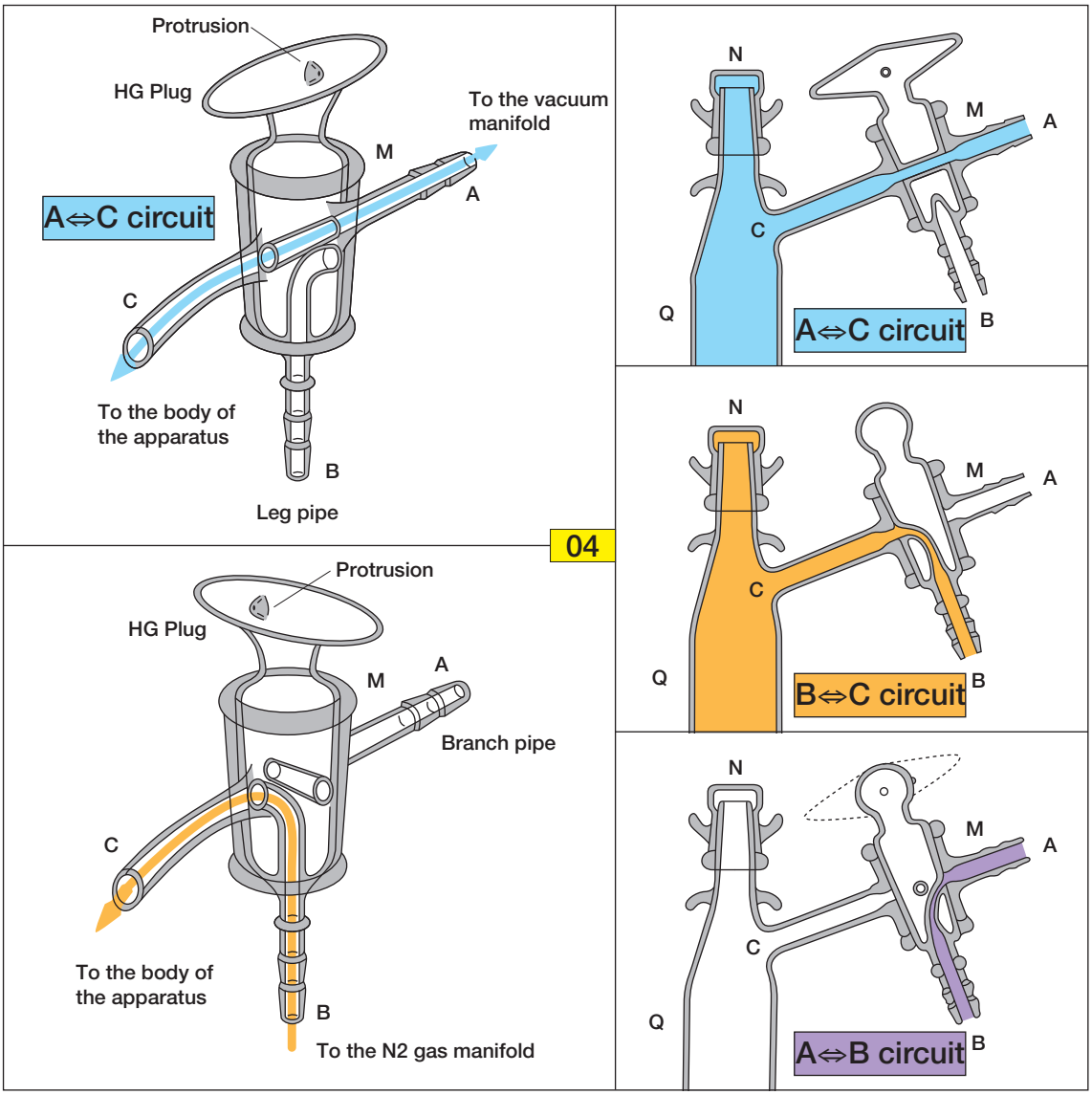
It is possible to replace the vacuum and **N_2 gas lines** connected to the **branch pipe A** and **leg pipe B** of the **Cock** depending on the handling and ease of operation of equipment concurrently in use, but in this manual, the explanations will generally follow the configuration illustrated in 04, where **branch pipe A** is connected to the vacuum line and **leg pipe B** to the **N_2 gas line** with branch pipe C fused to the body of the apparatus.

In this case, the connections and functions of each circuit are as follows:

A. Turn the Hollow Glass Plug (reffered to below as **HG Plug**) to circuit **A⇔C** to evacuate the apparatus.

B. Turn the **HG Plug** to circuit **B⇔C** to introduce **N_2 gas** into the apparatus.

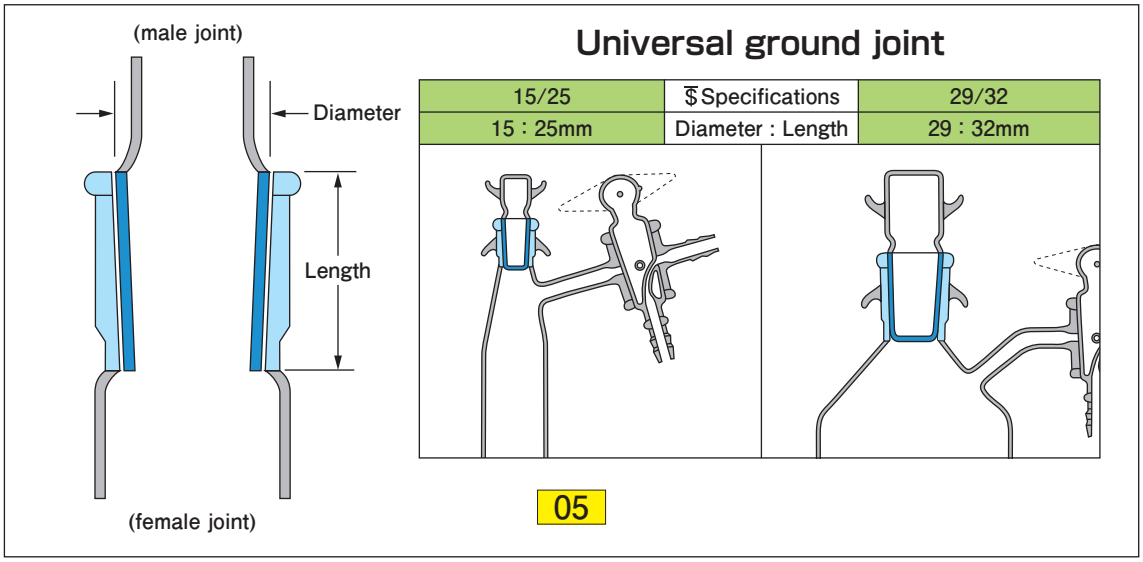
C. After the **gas has been displaced** completely by alternating between evacuation and **introduction of N_2 gas**, remove the rubber tube from A and turn **HG Plug** to circuit **A⇔B** to isolate it from the outside.*



* When the **HG Plug** has been turned to circuit **B⇔C**, the pressure inside the vessel is slightly higher than that outside (vide infra). Hence, when you turn the **HG Plug** to circuit **A⇔B**, air does not infiltrate from outside, unless you turn it too slowly. Note that the procedure mentioned above in **A, B** and **C** is not the easiest way to replace air by nitrogen. The easiest way is shown in 10 ,if the vessel is empty.

3. Transparent and opaque joints

The ground tapered joint of the glass apparatus joins the male joint (referred to below as the **Plug**) to the female joint (referred to below as the **Socket**) 05. Typically, the joints are opaque, but **transparent** ones are standard for joints in the **Sugiyama-gen** glass apparatus.



Transparent joints are precisely polished and have high airtightness. As such, they can frequently be used without greasing. However, they do not allow maintenance of a precise level of pressurization and decompression over an extended time period. Even very fine emery powder is coarser than gas, so gas will leak through the bumps on the ground surface. To tightly seal the joint portion, the bumps on the ground surface must be filled with a thin coating of grease, which must stay put.

However, grease tends to be lost from the joints when the vessel is evacuated because the ground scratches in transparent joints are very shallow.

So, when putting the Schlenk apparatuses together, using a transparent joint, it is important that the other joint must be opaque, and that the opaque joint must be thinly coated with grease to fill the joint surface scratches, and to ensure the grease be stayed on .

Do not put too much grease; the excess grease oozes out, contaminating the sample, ending up with unsuccessful recrystallization.

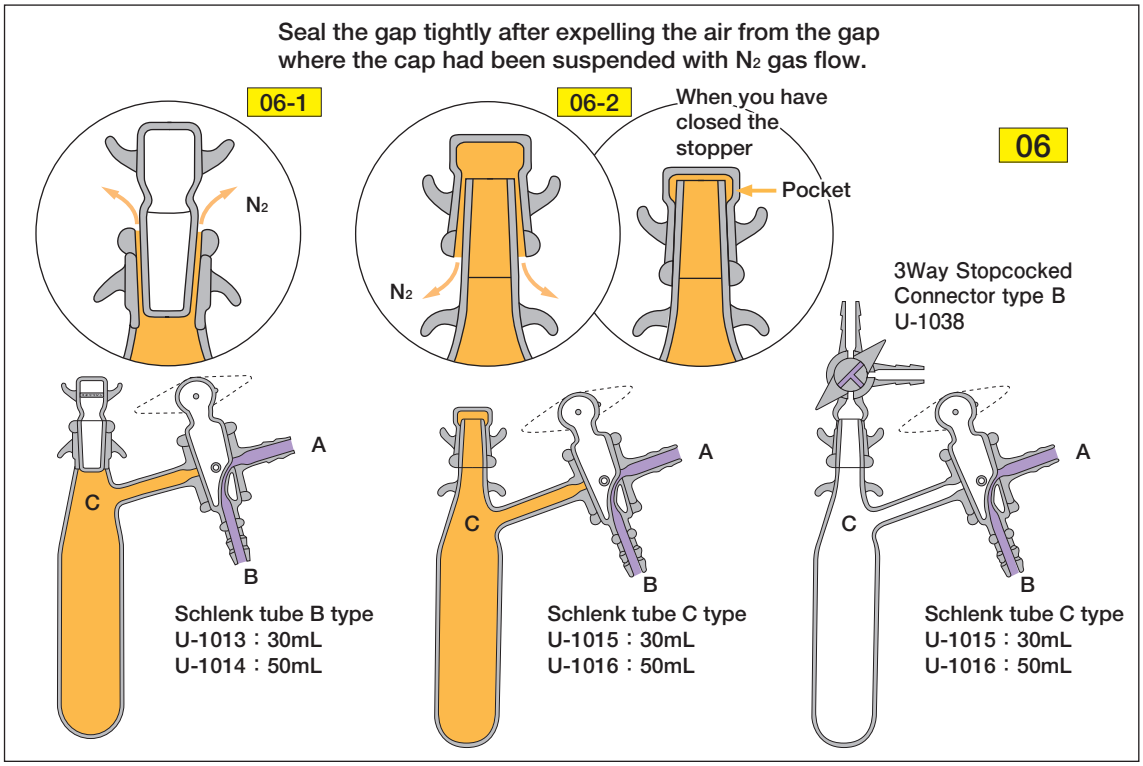
So how do you determine the right amount of grease? To get the right amount of grease, put **Plug** and **Socket** using a very thin layer of grease, then press lightly and turn. If the glass at the jointing portion appears transparent without streaks, it is OK. This is just an abstract explanation. To gain more realistic explanation, do it yourself.

To become skilled in the procedure, the reader must master this technique through repeated practice.

4. Preventing Grease Contamination

What can be done to prevent contamination of the solution in the **Tube** with the grease applied to prevent leaks from the stopper and allow smooth **HG Plug** rotating operation ?

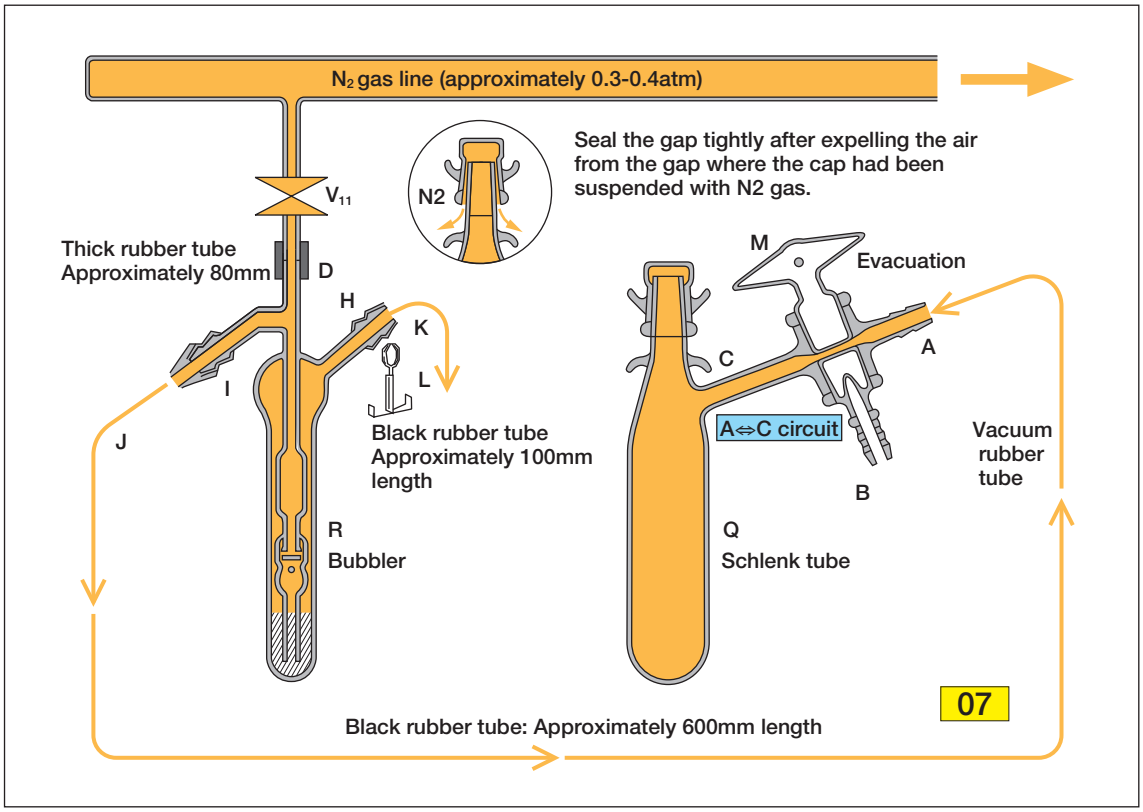
1. It may be effective, for example, to use a greaseless-type screw valve in place of a **Cock** However, because greaseless screw valves can only function as 2-way stopcock, it is necessary to use a glovebox or attach the **Cock** separately to the **vacuum and N_2 gas lines** to get a perfect inert atmosphere.



2. Grease enters from the **Cock** and stopper joint, but contamination from the stopper is more serious. In order to avoid the contamination from the stopper, use Schlenk tube C type with a male joint and female stopper. The point is that, when the stopper is placed, there can be a pocket, which prevents grease or a solution of grease from flowing down into the vessel, as shown in 06-2. In order to allow for pocket formation when the covering stopper is closed, the **Socket** of the stopper is made to be slightly shorter than the length of the **Tube**'s **Plug** . It is also designed to minimize the ceiling space when the stopper is closed (approximately 2mm).
3. Low boiling solvents in the **Tube** may condense in the pockets. If this can happen, use a stopper attached with stopcock (⇒**U-1038**). When condensation has indeed happened, insert a syringe needle to the pocket to remove the condensate before it overflows into the **Tube**. This requires a high level of skill.

5. Points to Consider When Closing the Stopper

1. For the **Plug** stopper such as those shown in 06-1 **Tube**, suspend the stopper for some time with a finger. While allowing the micro pressurized (0.3-0.4atm) **N_2 gas** to escape from the small gap thus created, extrude the remaining air, close the stopper when it has been completely flushed, thus closing the stopper while blocking air contamination.
2. For the **Socket** stopper (lid cover) such as those shown in the 06-2 **Tube**, suspend the stopper for some time with a finger. While allowing the micro pressurized (0.3-0.4atm) **N_2 gas** to escape from the small gap thus created, extrude the remaining air from the stopper, flush it out completely, and close the stopper, blocking air contamination.
3. Connect black rubber tube **J** to the **Tube** shown in 07. When black rubber short tube **K**, attached to the part **H** of the U-1003 type **Bubbler** containing a small amount of liquid paraffin (referred to below as **Bubl**) is closed with pinchcock **L**, either the **Plug** or the **Socket** can be used to lightly cover the **Tube** opening. The stopper will float up due to the micro pressurized (0.3-0.4atm) **N_2 gas**. Extrude the remaining air from the small gap created to close the stopper while blocking air contamination.
4. Even when the short rubber tube **K** attached to the part **H** is left open, rather than closed with the pinchcock **L**, "super" micro pressurization corresponding to the liquid paraffin height is maintained, allowing the flow of **N_2 gas** through the gap made with fingers.



6. How to Avoid Over-greasing

Contamination of the sample with grease must be minimized, especially when crystallization is desired and when handling silicon compounds.

What is an efficient way to precisely maintain decompression and pressurization without using any grease ?

In this case, glass apparatuses fitted with finely ground transparent **Plugs** and **Sockets** are used. However, this method does not allow for the maintenance of high air tightness. Hermetic tightness is maintained by sealing with white Teflon tape instead of using grease.

Specifically, wipe the **Plug** well, apply the Teflon tape to it, and press on that end with your left finger (if right-handed). Wind around once while pulling on the tape with your right hand. Wind around a second time and pull tightly to tear the tape off. *

This **Plug** is fitted to the **Socket**. Pushing strongly while turning in the direction the tape was wrapped will result in a hermetic seal.

Do not apply too much force, or you will break the glass. For you safety, work with gloves.

* Do not cut Teflon tape with scissors.

When scissors are used, no matter how hard you push the **Plug** to the **Socket**, level differences in the thickness of the parts where the tape overlaps remain, allowing leaks from the gaps and inhibiting air tightness.

It is important to wrap the tape around the **Plug** for about one-and-a-half turns and tear the tape off at an angle as right as possible.

A highly hermetic seal can be obtained by pressing down strongly on the torn portion of the tape which was stretched thin. This allows the level difference to disappear.

It is important to stretch while wrapping to ensure the two ends of the tape (made fibrous through tearing) do not overlap. It takes some getting used to, but skill in these procedures can only be obtained through repeated practices.

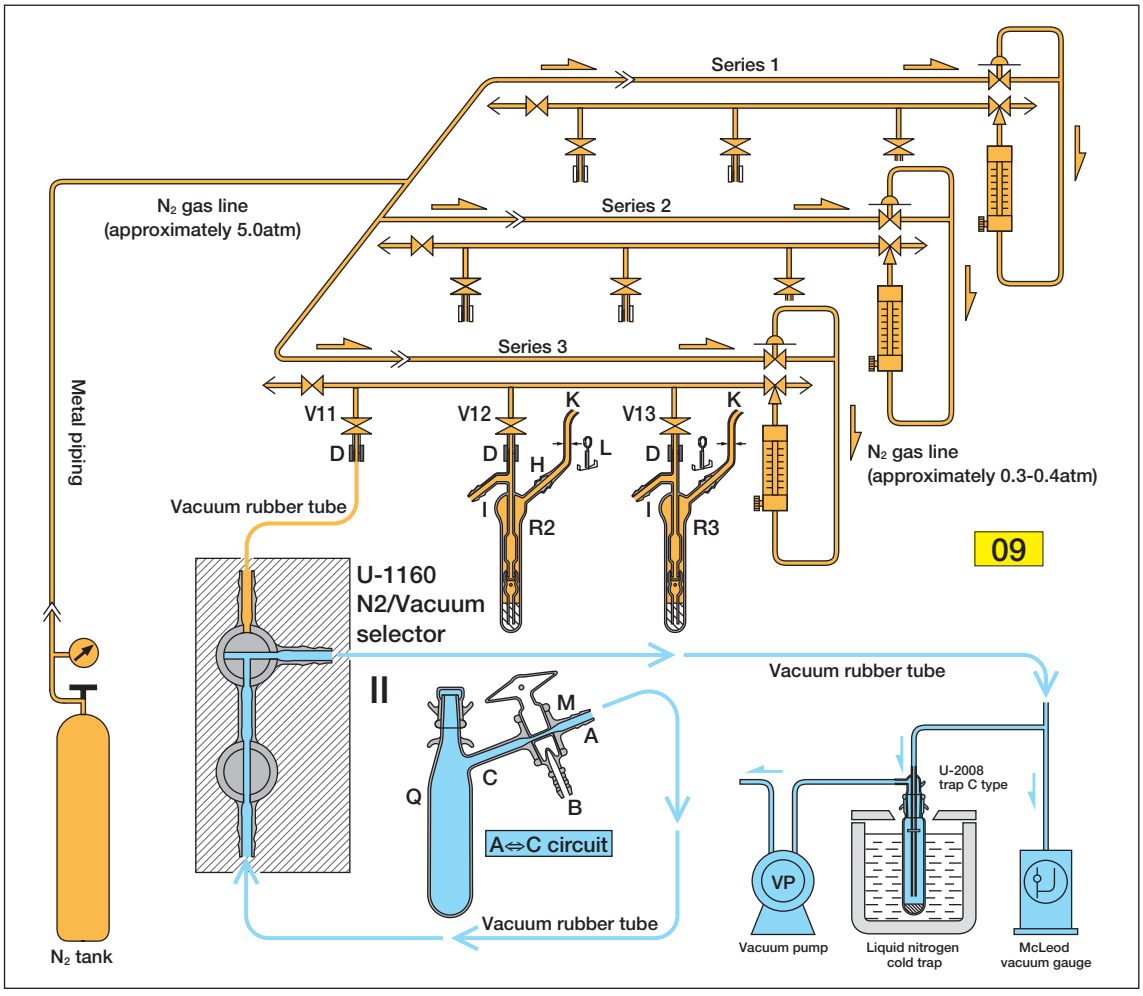
This way of sealing is not only limited to the operations of the **Tube** and **Schlenk flask**, but also can be applied to the reflux condenser, dropping funnel, 3-way **Stopcock** and a variety of other apparatuses (excluding the **Stopcock** portion). It allows for high air tightness without the use of grease. Though the Teflon **Stopcock-Plug** appears useful as it saves one the trouble of having to wash the grease from the apparatus after use, it does not allow for high air tightness regardless of how much the inelastic Teflon Cockplug is tightened. Accordingly, apart from typical organic synthesis using the Grignard reagent, Teflon **Stopcocks** should not be used in procedures using those materials as immediately decompose in oxygen.

7. Nitrogen Line Layout

Typical lab bench layout when the Schlenk tube technique is frequently used is illustrated in 09 . For example, in settings such as university laboratories, where a large number of students carry out the similar procedures at the same time, a large number of nitrogen cylinders cannot be set up at the same time due to High Pressure Gas Safety Act regulations.

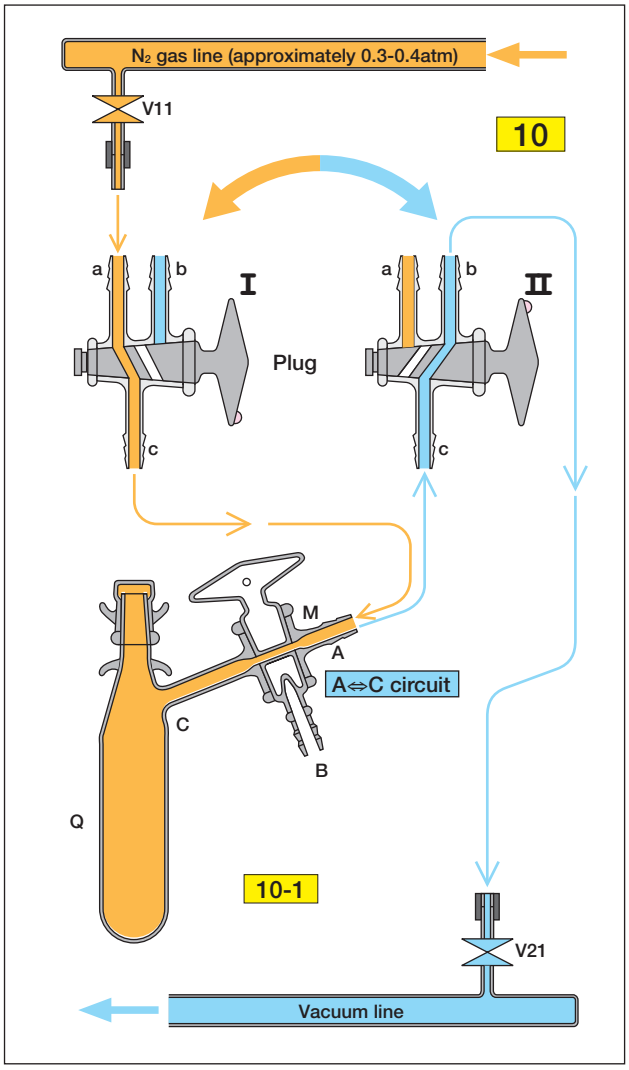
1. Typically, thick metal piping is laid out across the entire room from a **single N_2 cylinder**. This is divided into a number of **N_2 gas line series** (in the figure, these are **series 1, 2, and 3**).
2. The 2nd pressure of nitrogen in the piping depends on the number of series, but the pressure-reducing valve is adjusted to keep the pressure around 5 atm.
3. The following devices are connected to the **N_2 gas lines** of each series in the specified order: **gas pressure regulator⇒flow meter** (float-type ones are easier to read) **N_2 gas outlet** (It is convenient to have around 5-6 outlets every 15-25 cm in each series although only 3 outlets are shown in 09). The gas pressure regulator is adjusted to maintain a micro pressure of around 0.3-0.4 atm. Do not change this pressure without permission.

   Beware of making the pressure higher: it may make the attached stoppers, dropping funnels and other glasswares attached may fly off and break.



A gas pressure regulator installed before the flow meter is set for each series in 09, but installation of one regulator for every 3 or 4 piping series is acceptable. However, it is best to set a flow meter to each series individually.

4. Typically the **N_2 gas** outlet **V12** and **V13** (the use of metal valves make it easier to adjust the flow) is connected to **Bubl R2**, **R3**, as exemplified by the "**Series 3**" piping in 09 .
5. Installation of a **vacuum pump** for each **N_2 gas line** series makes the experimental operations easier and convenient. But if a number of vacuum sources are often required, install vacuum lines equipped with glass **Stopcock** or metal valves.
6. In the "**series 3**" piping example in 09, **outlet V11** on the left end of the **N_2 gas line** is connected to the **N_2**/vacuum selector (referred to below as "selector"; for details, see 14. The upper 3-way **Stopcock** shown in 09 works as a valve for switching between the evacuation and **N_2 gas** introduction and the lower 2-way **Stopcock** works as an opening and closing valve. Figure 10 shows operation tips for the switch. Below is provided an explanation with reference to the figure. Further procedure that follows is exemplified by using selector 14R .



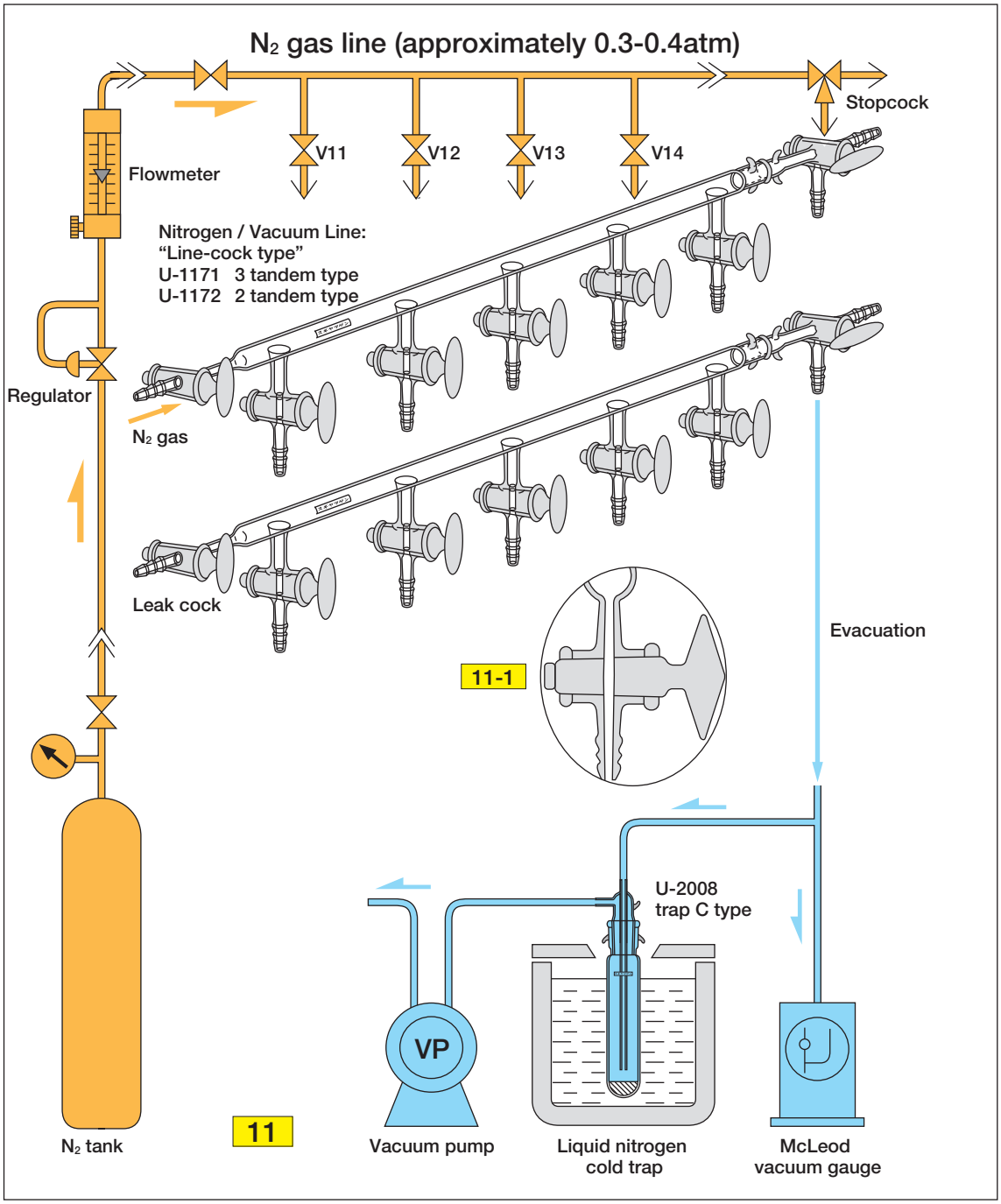
7. Connect **V11** of the nitrogen line and leg pipe **a** of the selector using evacuationpressure resistant rubber tubing. Likewise the leg pipe **b** and vacuum valve **V21** are connected using evacuation-pressure resistant rubber tubing.
8. Then, connect the leg pipe c of the selector and the side pipe A of **Tube** with evacuation-pressure resistant rubber tubing.
   Turn the **HG Plug M** towards circuit **A⇔C**.
9. Turn the **HG Plug** of the selector from pattern **I** to **II** to evacuate the **Tube**.
10. Turn the **HG Plug** of the selector back to pattern **I**. Now **N_2 gas** is introduced to **Tube** from **V11** .
11. Repeat 9. and 10. several times ; the gas in the **Tube** will be turned into pure **N_2**.
    Note that you can follow the same procedure even when **Tube** contains non-volatile solid.
    However, you should take account of cautions given in 15 5.

Nitrogen/Vacuum Line Type I: "Straight bore two way Stopcock type"

This type of single-column glass line is inconvenient, as it can be used only for the vacuum manifold or the **N_2 gas manifold**, but not both. However, because it is reasonably priced, it is suitable for student training.

Depending on the purpose, use either the a) **single-column line × 1 unit** or b) **single-column line × 2 units** (for nitrogen and vacuum).

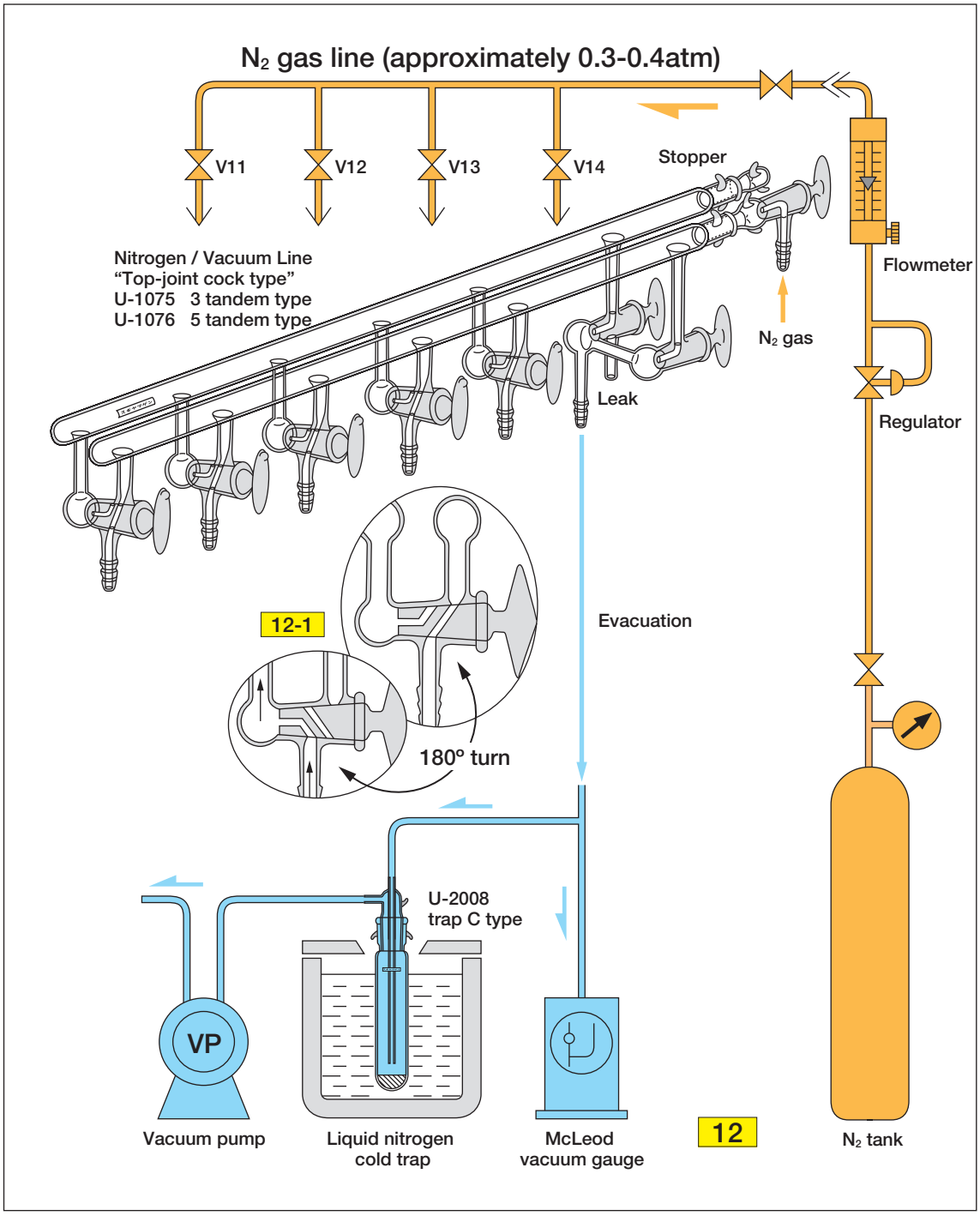
Even when you use one unit of the single-column, place a selector as shown in 09 to make the line more convenient.



Nitrogen/Vacuum Line Type II: "Three Way Stopcock with Double Oblique Hollow Glass Plug and Vacuum Chamber type"

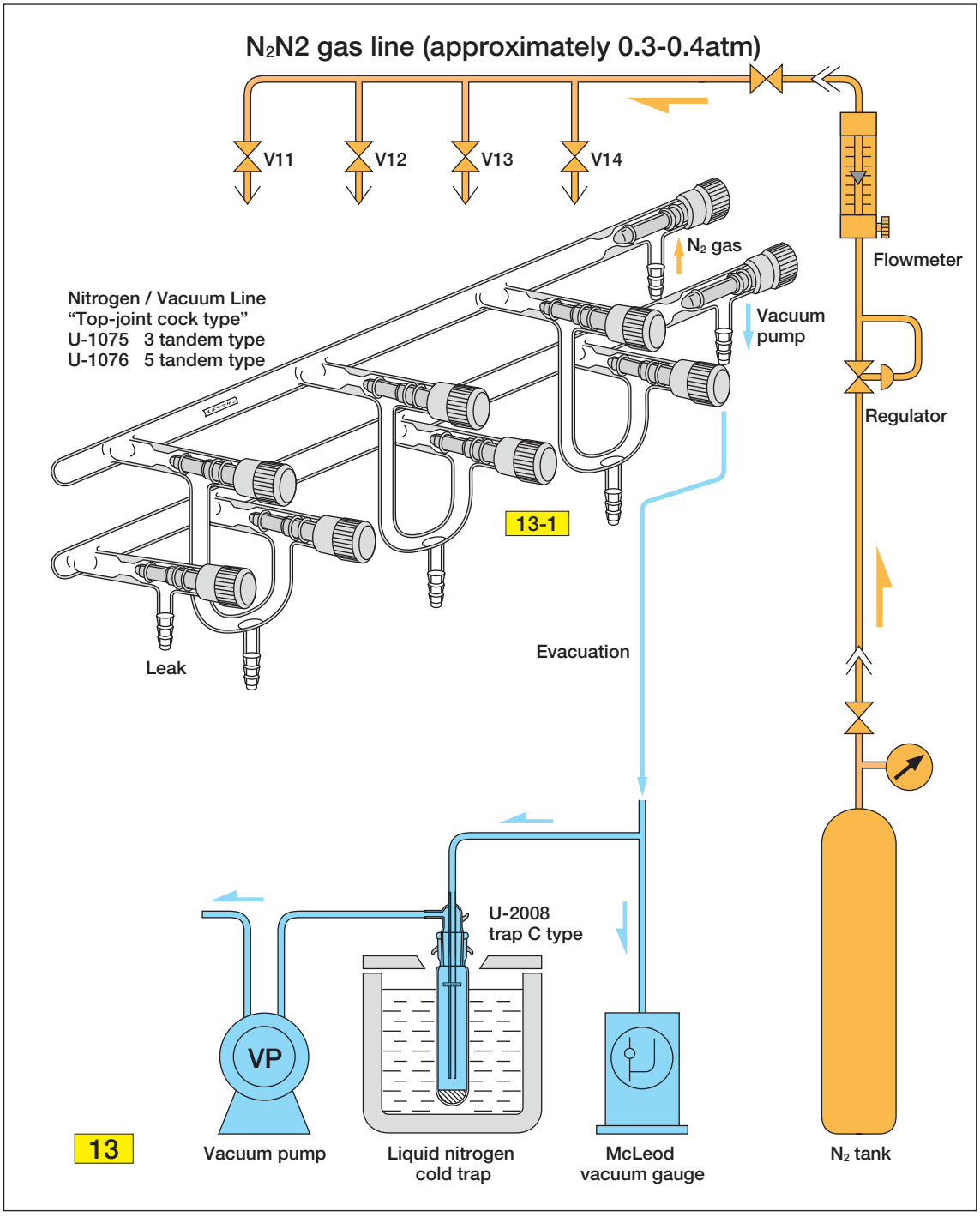
With this type of double-column line, one can be used for the vacuum manifold and the other for the **N_2 gas manifold**. With a single manipulation of the **Stopcock-Plug**, continuous switching between evacuation and **N_2 gas** input can be conducted from any nozzle.

Do not apply too much grease for smooth operation of **Stopcock-Plug**.



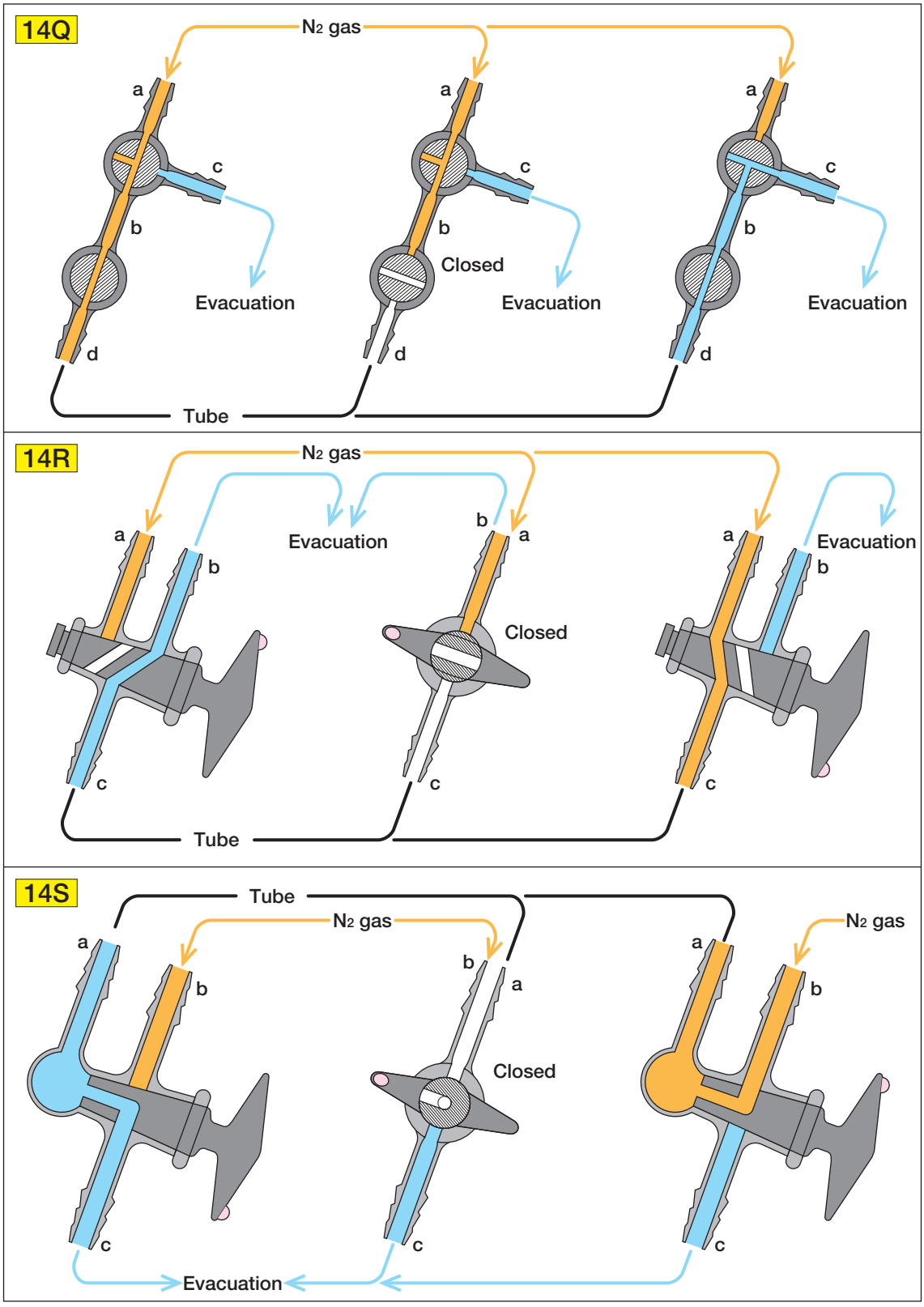
Nitrogen/Vacuum Line Type III: "Greaseless type Screw Valve with PTFE Plug "

If grease is objectionable at any cost, the greaseless-type **Screw Valve** can be used, as shown in 13-1. However, these valves function only as 2-way **Stopcocks**. As such, the convenience of being able to continuously switch between evacuation and **N_2 gas input** from any **nozzle** with a single manipulation of the **Stopcock**, as with the oblique bore three way **Stopcock** must be sacrificed.



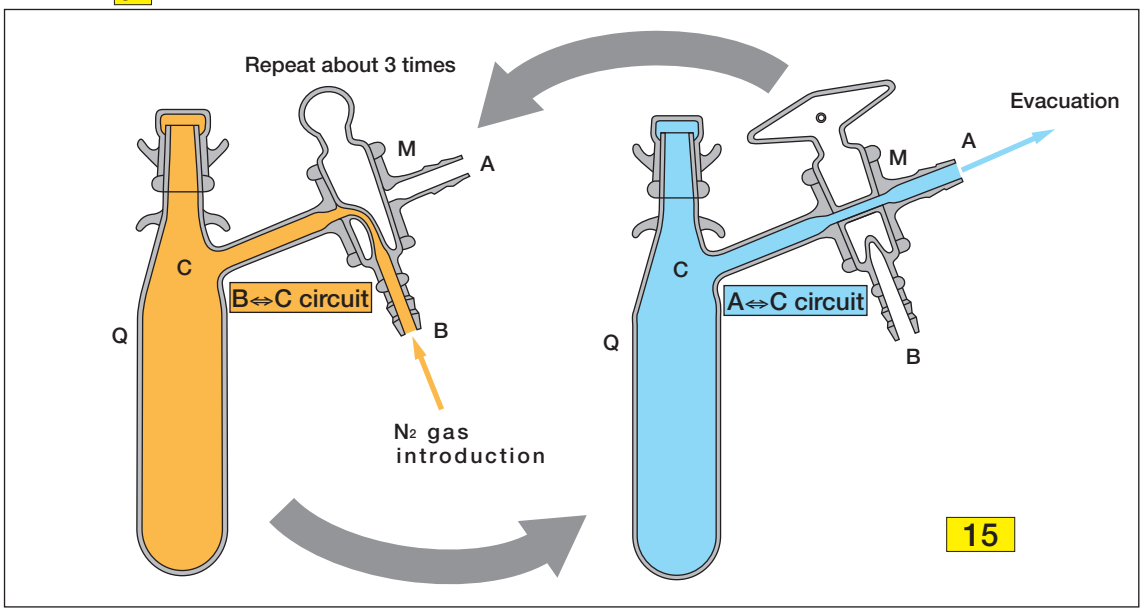
8. Three Types of Selectors

Three types of selectors to displace the atmosphere from air to N2 gas are introduced in 14Q, 14R and 14S. Especially 14R and 14S comprises 3 sets of 2-way **Stopcock** just like **Cock** does shown in 4 . Select one as you like it.



9. N_2 Gas Substitution in Empty Vessels or Solid-containing Vessels

The method displayed in 10 is commonly used, but the same effect can be obtained by manipulating the **Cock** of the **Tube**. The following is an explanation with reference to 15 and also 07



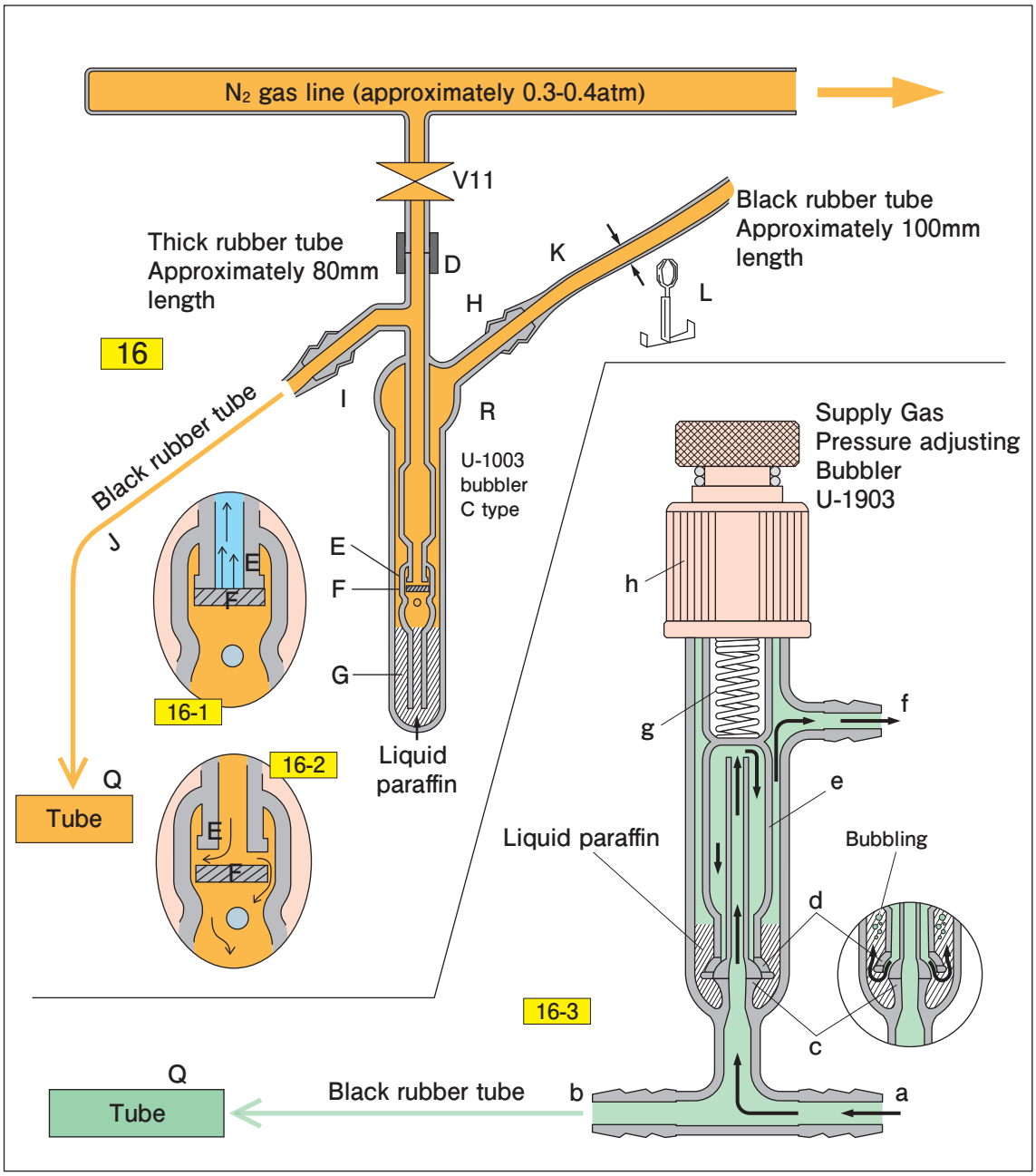
1. Put the evacuation pressure-resistant rubber tube (referred to below as vacuum rubber tube) extended from the **vacuum** valve to **branch pipe A** of the **Cock** of **Tube Q**. Put on the **black rubber tube** from the **N_2 gas outlet** to **leg pipe B**.
2. Turn **Hollow Glass Plug**(or **HG Plug**) **stopper** towards circuit **A⇔C** and evacuate **Q**.
3. Turn **HG Plug M** towards circuit **B⇔C** to introduce **N_2 gas** into **Q**.
4. By switching between 2.: **Evacuation** and 3.: **N_2 gas introduction** 3 times, the inside of Q becomes a near-perfect **N_2 gas atmosphere**.
   This takes only 30 seconds at most.
5. It's OK to follow exactly the same procedures with a solid in the vessel. However, particularly when the solid is a micro-powder, carefully and slowly manipulate the **HG Plug** to ensure that the solid is not blown up by the flow of the incoming **N_2 gas** or sucked into the pump upon **evacuation**.
   …Here, lets return to 07 to explain what happens when the **Bubl** is used.
6. Put on the **vacuum rubber** tube extended from **vacuum valve V21** on **branch pipe A** of the **Cock**, turn **M** towards circuit **A⇔C** , and evacuate **Q**.
7. The **black rubber tube J** extended from **I** of **Bubl** is connected to **branch pipe B** of the **Cock**. Turn **HG Plug M** towards circuit **B⇔C** and introduce **N_2 gas** into **Q**.
8. By switching between 6.: **Evacuation** and 7.: **N_2 gas introduction** 3 times, the inside of **Q** becomes a near-perfect **N_2 gas atmosphere**.

10. Bubbler Operation

As an example, we will describe the operation of the **bubbler (U-1003)** using in combination with **Schlenk round-bottom flasks**.

Above all, the most unique feature of this device is that it has a **glass disk F**, which acts as "valve" or "shutter" to prevent the counterflow of the liquid paraffin.

Operation is straightforward and effective.



1. Arrangement

1. An **N2 gas line** gas pressure is set at 0.3 ~ 0.4atm.
2. An **N2 gas outlet V11** and **Bubl** is connected by a short vacuum rubber tube **D** and a **black rubber tube J** (ca. 600 mm) is connected to **branch pipe I**.

*Note: When not conducting an experiment or when there is nothing connected to the end of J, outside air can enter into J and result in oxygen adsorption by the inner walls of the tube. In order to avoid this situation, do not leave the tip of J open.

3. Put a short (100mm) **black rubber tube** on **Bubl** **branch pipe H**. Place **pinch cock L** on **H**, so that it can be immediately opened and closed depending on need.
4. Inject liquid paraffin into **Bubl** in advance. The right amount to inject is slightly more (≈2ml) than the Inner volume of the inner tube **G** below **E**.
5. Evacuate **Tube Q**.
   At this point, sudden decompression of the inside of the flask and bubbler leads instantaneous movement of the disk F up to the stopper **E** 16-1. This movement prevents the infiltration of liquid paraffin instantaneously, which in turn prevents the influx of external air from **H**. Time required for evacuation depends on the size of the vessel, but a couple ~10 seconds is enough. If the need arises, it is advisable to check the pressure by a McLead vacuum gauge situated between the **vacuum line** and vacuum pump.
6. Introduce **N_2 gas** into **Q**. **N_2 gas** is introduced though **D→I→J** and once the **Schlenk flask** is filled, change direction and flow **through** to depress **F** 16-2 and flow out from **H** with bubbling through the liquid paraffin. Even though **L** has been removed and **K** open, the inside of the apparatus is maintained at the "super" micro pressurization corresponding to the distance between the bottom of **G** and the surface of the liquid paraffin (=liquid paraffin column). This helps minimize the influx of external air.
   *Note 2: Because the inside of the apparatus is still in a micro pressurized state (0.3-0.4atm), use a stainless spring or a thick rubber band to tightly shut middle stopper **M** to prevent it from lifting and flying off.

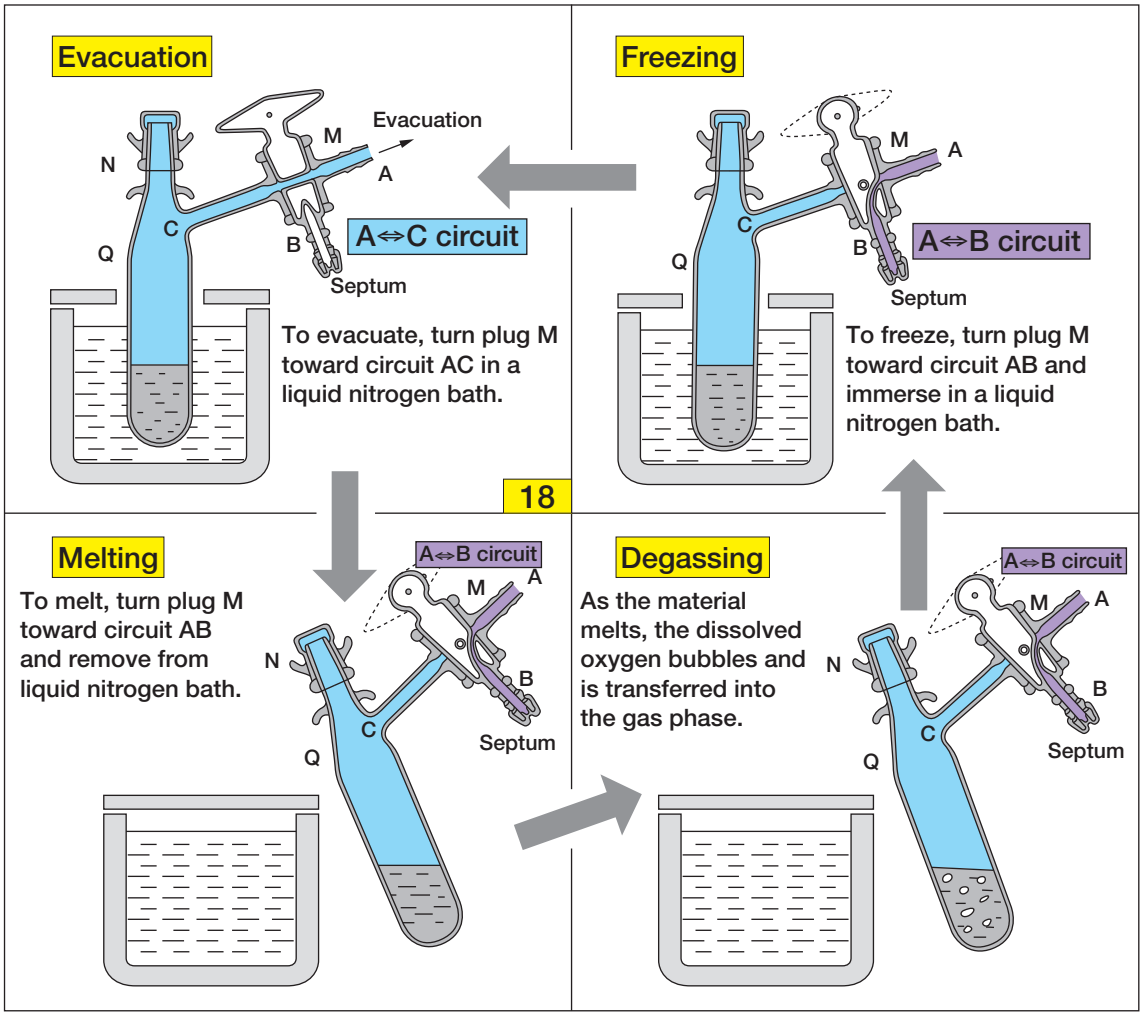
2. Supply Gas Pressure adjusting Bubbler

Shown in the former page 16-3 is another type of **Bubl** U-1903, which is utterly different in function and application from U-1003.

1. It is applicable in case of flowing a certain gas from branch pipe **a** to **b**.
   By rotating the cap **h**, restoring strength of the spring **g** can be adjusted to desired extent, so as to previously control the supply gas pressure in an expected degrees.
   Inject liquid paraffin into the well **d** in advance. The right amount to inject is slightly more than the Inner volume of the inner tube. For detail see the illustration of 16-3.
2. If the supply gas should carelessly rush flowing from the branch pipe **a** to **b**, the excessive gas pressure push up the oppressing edge **d** of the lifter **e** from the semi-ball type joint **c**, making gap between **c** and **d**. The excessive gas expels through this gap with bubbling through the liquid paraffin out of the leak pipe **f**, keeping the previously controlled gas pressure degrees.
3. In the meantime, you should again check and adjust the **Gas Pressure Regulator** ⇒ the **Flow Meter**, recovering into normal conditions.

11. Degassing of Solvents and Reagents

Solvents and reagents used in an **N_2 gas atmosphere** must have been removed dissolved oxygen beforehand. When using a large amount of reaction solvent, dissolved oxygen is removed by refluxing under a **stream of N_2 gas**. In a small scale experiment, however, operate in accordance with the figure below for degassing.



As in the above figure, repeat the **Freezing ⇒ Evacuation ⇒ Melting** leading to bubbling (**Degassing**) procedures 3 times. After checking that there is no effervescence at the melting-degassing stage and all dissolved oxygen appears to have been removed, remove the **vacuum rubber tube** from **A**, put a black rubber tube from the nitrogen line to **A**, remove the septum attached to **B**, switch **HG Plug** M to flow **N_2 gas** through circuit **A⇔B** to **I**, and switch **M** to circuit **A⇔C** to introduce **N_2 gas** into **Q**. Note that degassing can be more easily carried out by using the selector. 10

A solvent with dissolved oxygen completely removed can be obtained in this way.

*Note: In degassing water, when the water turns to ice, its volume increases. Accordingly it is safer to use a round-bottom vessel than a cylindrical **Tube**. Even in this case, it is safer to freeze onto the vessel wall by agitating its contents all the time. When using a cylindrical vessel, agitation of the vessel contents while the vessel is immersed in liquid nitrogen must not be neglected. Note well that the vessel will break otherwise unless you agitate.

12. Evaporation of the Solvent in the Schlenk Reaction Vessel

1. When evaporating solvent in accordance with 10

1. Connect branch pipe **A** and the selector leg pipe **D** with the vacuum rubber tube.Turn the **HG Plug M** of the **Tube**'s **Cock** from **A⇔B** circuit to **A⇔C** .
   *Note 1: You are going to evaporate a solvent, so ensure that the cold trap is placed before the vacuum pump.
2. Open vacuum valve V21 and while stirring the reaction solution inside the **Tube** with a **magnetic stirrer**, gradually move the **switch** over to **setting II**, and evaporate the solvent under reduced pressure.
   *Note 2: When evaporating under a reduced pressure, the temperature of the solution decreases due to the solvent's latent heat of vaporization. To facilitate the evaporation process, sustain the temperature through immersion of **Tube** in a water bath, heating with a heater when needed, etc.
   *Note 3: By continuing the evaporation of the cold solution without sustaining the temperature, the solution itself may foam under a reduced pressure and flow out from **Q**. To avoid this problem, in addition to sustaining the temperature, adjust the **HG Plug M**, the vacuum line **metal valve V21**, etc., to set the most appropriately reduced pressure, while keeping close watch over the state of evaporation.
3. After the evaporation has progressed to a desired extent, change the **switch** to **setting I** to introduce **N_2 gas** into the **Tube** from **V11**.
   *Note 4: When the amount of the solution is too large, it may overflow. To prevent such a situation, evaporate after transferring excess solution to a separate tube. After the first aliquot has been evaporated, add another small aliquot and evaporate it. It is advisable to carry out this procedure several times.

2. When evaporating solvent in accordance with layout 16.

1. Put **vacuum rubber tube** on from **vacuum valve V21** to **branch pipe A** of the **Cock** of the **Schlenk round-bottom flask Q**. Also connect **branch pipe B** of **Q**'s **Cock** and **branch pipe I** of the **Bubl** with the **black rubber tube**.
2. While stirring the solution in **Q** with a **magnetic stirrer**, gradually turn the middle stopper **M** to circuit **A⇔C** to evacuate the inside of **Q**, and evaporate the solvent under reduced pressure. If the need arises, it is advisable to check the pressure by a MacLeod type vacuum gauge situated between the **vacuum line** and vacuum pump.
3. After evaporation has progressed to a desired extent, turn **HG Plug M** towards circuit **B⇔C** and introduce **N_2 gas** into **Q**.
   *Note: Precautions are the same as 1. Notes 1-4 above.

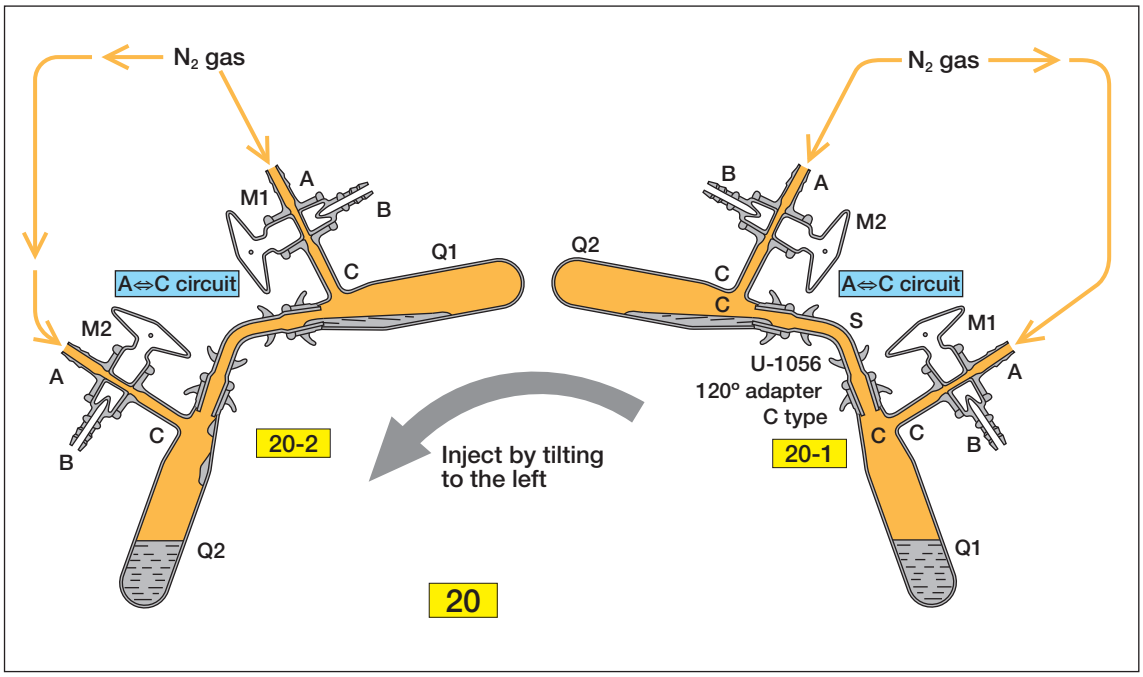
13. Method of Transfer Liquid Between Schlenk Tubes

1. Liquid Transfer Methods through Tube Manipulation

In this method, turn the **Tube**'s **Cock** of both the donating and receiving flasks towards circuit **A⇔C** , and carry out the transfer procedures under **N_2 gas** flow without the intrusion of outside air.

The following is an explanation with reference to 20. Although not explicitly illustrated in the figure, the **N_2 gas** flows though the **Bubl** for each of **Q1** and **Q2**.

1. Using a syringe, transfer a liquid sample to **Tube Q1**, 20-1
2. Connect **Q1** and **Q2** with a **120°** adapter (**U-1056**), 20-2
3. With the **120°** adapter as the axis transfer a liquid sample from **Q1** to **Q2** while tilting the entire connected apparatus to the left (this is the way it is displayed below). ⇒20-3



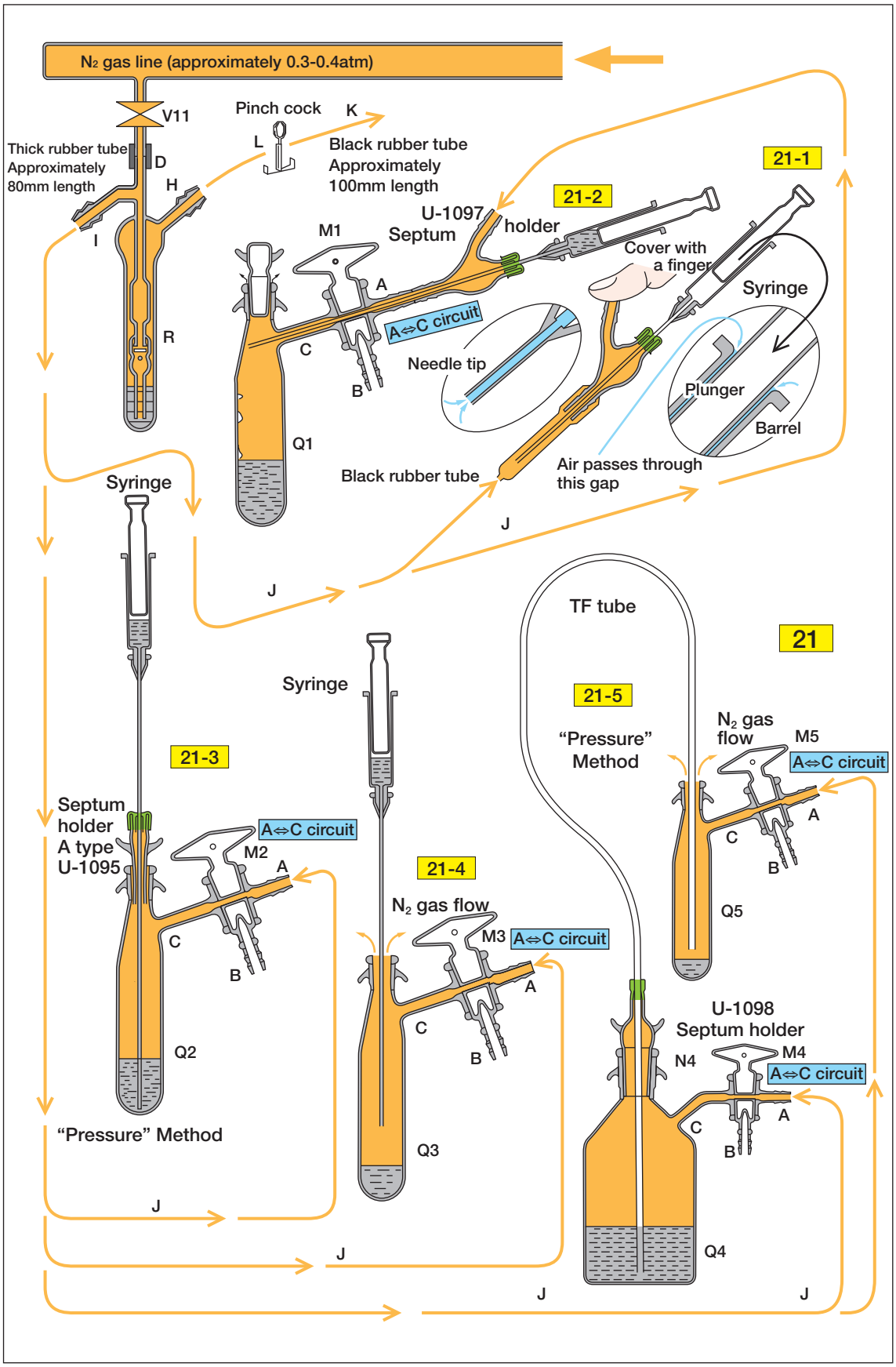
2. Methods for Syringe Transfer of Liquid

When using a **Syringe**, steps to prevent the infiltration of air must be taken in advance. There are two paths through which air can enter. One is intrusion through the gap between the **Barrel** and the **Plunger**. The other is air infiltration through the tip of the long needle.

The traces of air that infiltrate through these pathways decompose the valuable sample (if not completely, at the very least to some extent). Color change can make it obvious that decomposition has occurred at a particular part of the sample. To get **N_2 gas** displacement from the inside of the **Barrel** and from the **needle** while keeping air out, place the tip of the **needle** into **N_2 gas** and pull and push the **Plunger** several times to introduce and purge the gas.

These procedures will be described with reference to 21-1, 21-2.and 33-A .

Note: It takes time to make a hole through a thick rubber stopper to insert the needle so an adapter using a commercially available septum is presented.


N2 gas line (approximately 0.3-0.4atm)
V11
Thick rubber tube
Approximately
80mm length
D
H
I
R
Pinch cock
K
L
Black rubber tube
Approximately
100mm length
21-1
21-2
U-1097
Septum
holder
M1
A
A⇔C circuit
C
B
Q1
Needle tip
Cover with
a finger
Syringe
Plunger
Barrel
Black rubber tube
Air passes through
this gap
J
Syringe
J
TF tube
21
21-5
"Pressure"
Method
N2 gas
flow
M5
A⇔C circuit
A
C
B
Q5
Syringe
21-3
Septum
holder
A type
U-1095
A⇔C circuit
M2
A
C
B
Q2
"Pressure" Method
21-4
N2 gas flow
M3
A⇔C circuit
A
C
B
Q3
U-1098
Septum holder
N4
M4
A⇔C circuit
A
C
B
Q4
J
J
J
J
J

1. Push the **Plunger** to the bottom of the **Barrel** in advance. Then, pierce the needle through **septum holder** connected to the **black rubber tube J**.
2. Open the **N_2 gas line valve V11**, close **rubber tube K** connected to **Bubl R** with **pinch cock L**. Then the **N_2 gas** will flow through **D→I→J** and into **the septum holder**.
3. Cover the **septum holder branch pipe** with a finger. Doing this will lead the micro pressurized (0.3-0.4atm) **N_2 gas flow** to push back the **Plunger**, force its way into the space or gap between the **Barrel** and **Plunger**, and remove residual air in this portion.
4. When the **Plunger** has been pushed back to the upper range of its scale markings, remove the **needle** from the **septum holder**. Stand by after forcing back the **Plunger** to the middle range of scale markings, then push the **Plunger** to the bottom of the **Barrel** right before you draw up liquid in the next procedure. In this way, when the **Syringe** is transferred in the atmosphere, possible air infiltration can be extruded by pushing back the **Plunger** even if air may infiltrate through the **needle**.

There are two liquid transfer methods of using **Syringe** – **"suction"** and **"pressurization"**.

a. "Suction" Method 21-4

1. Turn **Cock M3** towards circuit **A⇔C** to release the flow of **N_2 gas** and with the **N_2 gas** overflowing from the opening of **Tube Q3** (stopper removed),
2. get the needle to the bottom of **Q3**, draw up the liquid sample by suction,
3. and gently transfer by inserting the needle into a separate **Tube**. 21-4

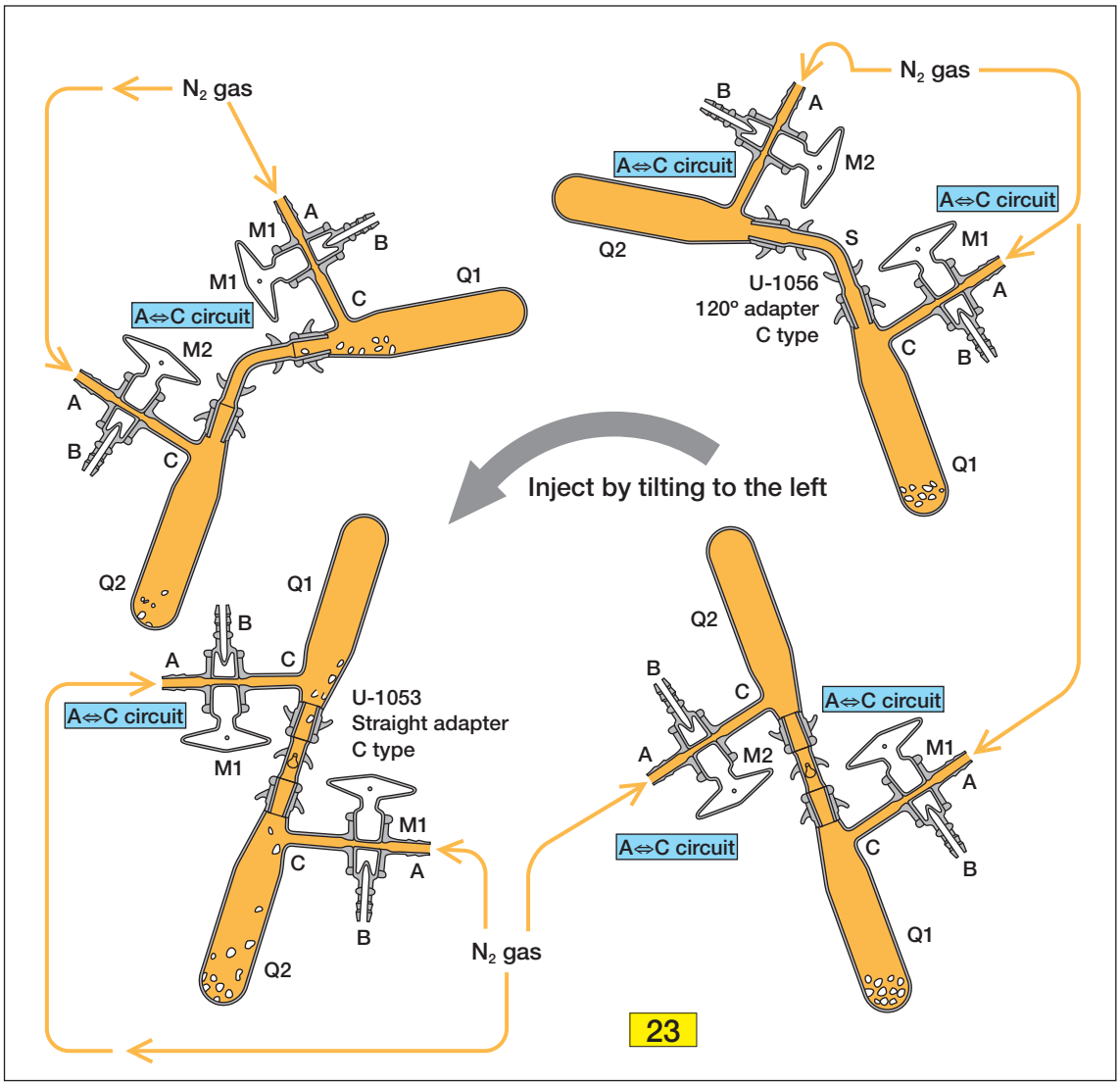
b. "Pressurization" Method 21-3

1. Pierce the **needle** through **septum holder N1** fitted to the opening of **Tube Q2** , and turn **M2** towards circuit **A⇔C** to supply the micro pressure of the **N_2 gas**.
2. When the **needle** reaches the bottom of **Q2**, the surface of the liquid gets pushed down by the 0.3-0.4atm **N_2 gas**, and the liquid sample is transferred into the **Barrel** through the **needle**.
3. Transfer the syringe to a separate **Tube**. Gently inject the liquid sample. ⇒21-2, 21-3
4. When applying this method (for example when solvents and reagents that are being saved or reserved are subdivided in a separate **Tube Q5**), fit a **septum holder** on **Schlenk bottle Q4** and insert a **TF tube** with inner diameter of 2mm into the mounted septum. When the tip of the TF tube reaches the bottom of **Q4** and the other end has entered **Q5**, turn **Cock M4** to circuit **A⇔C**. The liquid surface of **Q4** will be pushed down by the 0.3-0.4atm **N_2 gas** and the solvent or reagent pushed out into **Q5** through the **TF tube**. 21-5

14. Method of Transfer Solids Between Schlenk Tubes

The method for transferring solids from **Tube Q1** to **Q2** is described hereafter.

1. Tilt the two **Tubes** connected with a **straight-type adaptor** or **120° adaptor** from right to left (in the figure) to transfer the solids.

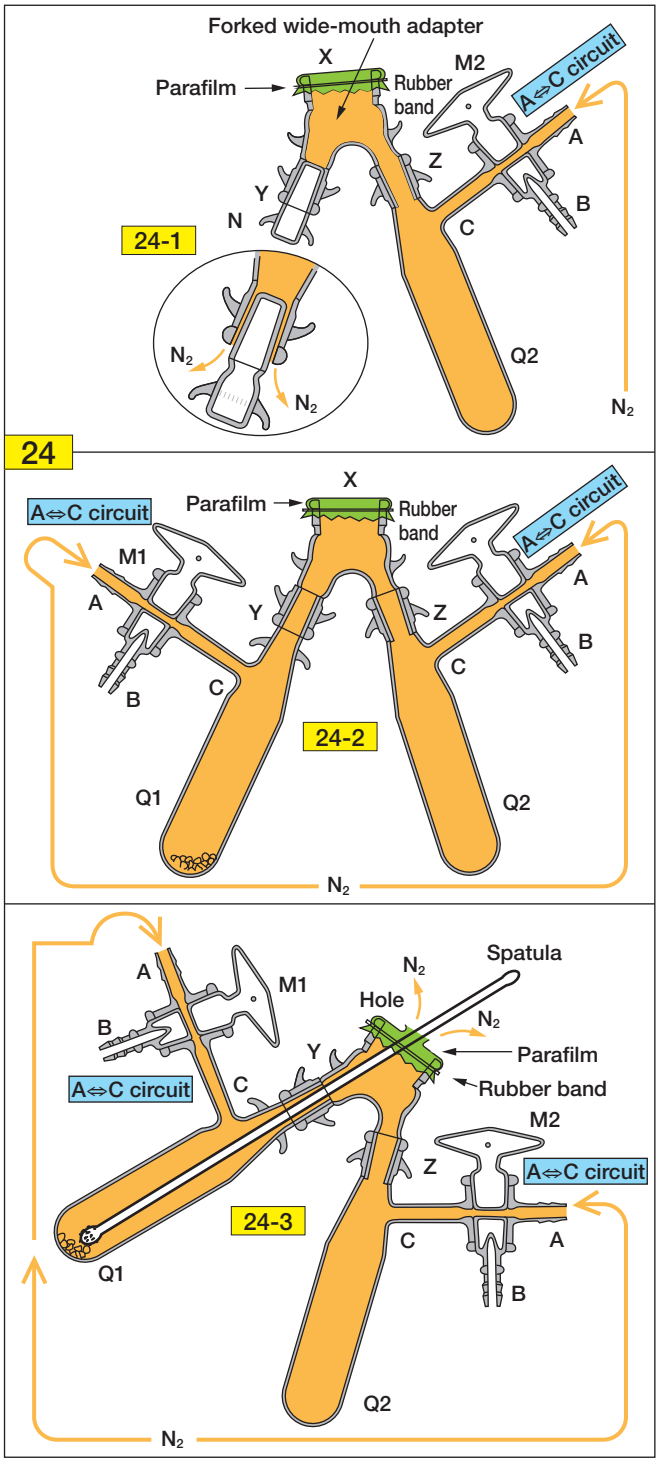


2. Transfer Using Forked Wide-mouthed Adapter

Cover the opening of the forked **wide-mouth adapter** with a **plastic film** such as **Parafilm**, and affix it with a rubber band around the adapter. Then, make a small hole by inserting a **spatula** to simply and precisely transfer a small amount of solid. 24

Do not overstretch the film. Stretch it with enough slack that allows the hole to work as an outlet through which the **N2 gas** is overflowing.

Turn **Cocks Q1** and **Q2** to their respective circuits **A⇔C** to release only a small amount of **N_2 gas**. With this, the **N_2 gas** will constantly overflow from the small hole that was created with **spatula** insertion, allowing prevention of outside air infiltration into the apparatus.
The following is an explanation with reference to 24. Although not explicitly illustrated in the figure,the **N_2 gas** flows though the **Bubl** for each of **Q1** and **Q2** as in 07.



1. Mount **Tube Q2** (receiver of the transferred solid), to **Z** and mount jointed-stopper to **Y** in advance. ⇒24-1
2. Allowing for some slack, cover the upper wide-mouth opening **X** with a **plastic film**, such as **Parafilm**, and affix it with a rubber band from the top.BC circuit
3. Next, turn **HG Plug M2** towards circuit **A⇔C** to introduce **N_2 gas** and then turn the **HG Plug** to circuit **B⇔C**, Repeat the same operation several times to achieve pure **N_2 gas** atmosphere in **Q2**.
4. With the **N_2 gas** overflowing, remove the stopper that had been fitted to Y, mount the donating **Tube Q1** (before mounting, turn M1 to circuit **A⇔C** , and leave the **N_2 gas** overflowing). 24-2
5. Make a small opening by inserting a **spatula** from the center of the film and pry it open so that it makes the shape of the **N_2 gas** outlet. This will allow you to move the **spatula** around freely.
6. Insert the **spatula** into donating **Tube Q1**, pull up the scooped solid sample up inside the adapter to transfer from **Q1→Q2**.
   All operations are carried out under an **N_2 gas atmosphere** inside the connected vessels.

15. Solid-liquid Separation Method

Filtration is basic in the separation of crystal from a suspended mixture (solid-liquid separation), but this method is somewhat troublesome.

Use of the so-called “decantation method” circumvents troublesome operations whether filtrate or solid is desired.

In this method, the mixture reacted in a **Tube**-type vessel is left to stand without disturbance. The supernatant liquid is then taken away with care so as not to disturb the precipitate.

This is an ideal method for recrystallization in which crystals that come out after heating and cooling operations. Even when the crystal is a micro-powder, solid-liquid separation can be done with ease, if the mother solution is separated gently.

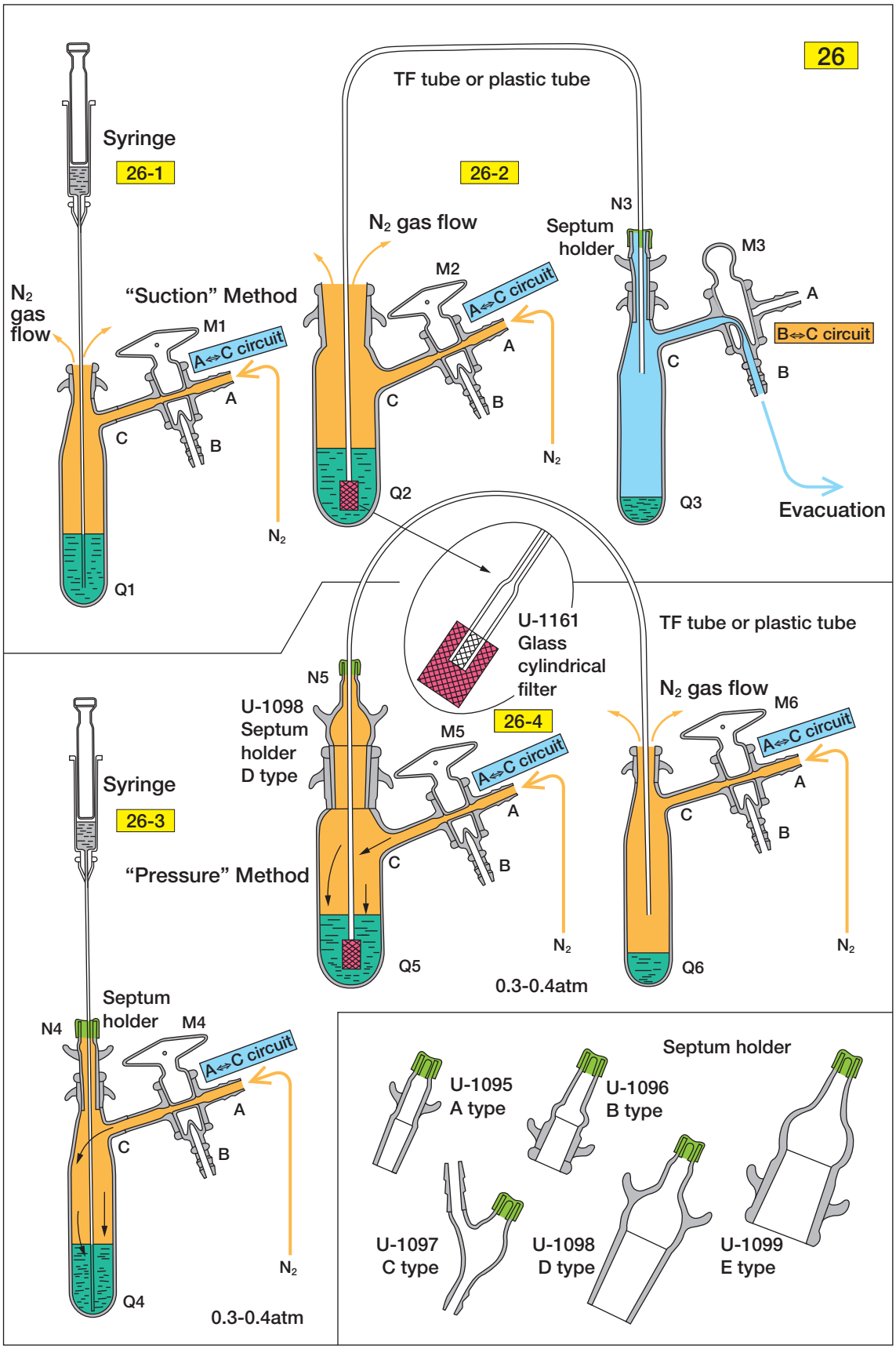
There are **“suction”** and **“pressurization”** methods by using a syringe or Teflon (TF) tube.

1. “Suction” Method

1. When using a syringe, turn **M1** to circuit **A⇔C** and leave the **N_2 gas** overflowing from the opening of **Q1**. After placing a needle with inner diameter 2mm (henceforth referred to as "needle") into the liquid in **Q1**, raising the "inner cylinder" will allow the liquid sample to be suctioned gradually into the **Barrel**. ⇒26-1
2. Put a ball filter on one end of a **TF** tube with a 3mm outer diameter and leave it in the liquid inside **Q2**. Penetrate the other end of the **TF** tube through the septum holder **N3**, fitted to the opening of **Q3**, and after it has been inserted into **Q3**, turn **M3** to circuit **A⇔C**, depressurize **Q3** to allow gradual suction of the supernatant liquid. ⇒26-2

2. “Pressurization” Method

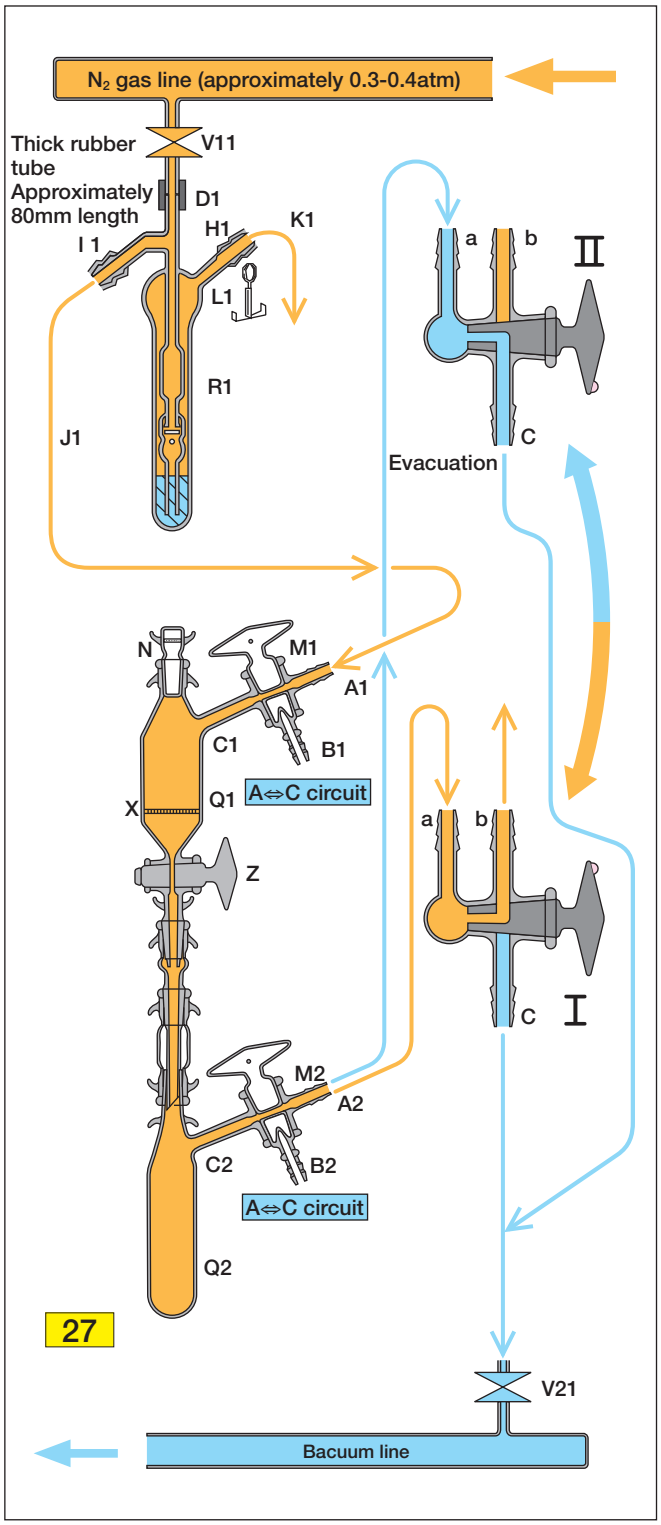
1. When using a syringe, pierce septum holder **N4** fitted to the opening of **Q4** with a needle. Once the needle has reached the bottom of **Q4**, turn **M4** to circuit **A⇔C** to Introduce **N_2 gas**. The surface of the liquid in **Q4** will be pushed down by the 0.3-0.4atm **N_2 gas**, leading the liquid sample through the needle into the **Barrel**. If there is no filter attached to the tip of the needle, extract only the supernate, taking care not to disturb the precipitate. Note that you should remove the needle from the **septum holder** once the liquid sample has been transferred; leaving it as it is can result in the **Plunger** flying off due to the pressure of the gas. ⇒26-3
2. Turn **M5** and **M6** to circuit **B⇔C** in advance and leave the **N_2 gas** overflowing from the opening of **Q5** and **Q6**. Next, fit **septum holder N5** to the opening of **Q5** and insert a **TF** tube (outer diameter of ca. 3 mm) through **N5** Put a glass or other ball filter on one end of the **TF tube** and leave it in the liquid inside **Q5** after inserting the other end into **Q6**. The surface of the liquid in **Q5** will be pushed down by the 0.3-0.4atm **N_2 gas**, leading the liquid sample to **Q6** through the **ball filter**. ⇒26-4



*Note: The TF or plastic tube is not included in our products.

16. Filtration in an N_2 Gas Atmosphere

Filtration is conducted when, for example, the removal of micro-powder from a mixture with a micro-powder suspension (solid-liquid separation) is desired. The filtration using **U-1012** and **U-1015/1016**-type **Tubes** connected through an adapter with reference to the below diagram is explained hereafter.



Arrangement

1. Put black rubber tube **J1** on from **V11** via **R1** to **A1** of **Q1**.
2. Put **vacuum rubber tube** from vacuum valve **V21** to leg pipe **C** of the selector.
3. A vacuum rubber tube is put between side pipe **A2** of **M2** and side pipe a of the selector.
4. After the atmosphere in the whole system has been changed from air to **N_2 gas**, inject the suspension onto the sintered glass filter **X** in **Q1**(How to inject a suspension will be explained separately.)

   The following three methods are used for filtration, depending on the conditions.

   *1 To transfer a suspension using a **Syringe**, you have to use a rather thick needle. Depending on the case, cannulae technique using tubing made of metal, polyethylene, Teflon , or glass can be easier.

   Note, however, that cannulae for medical use is not appropriate.

1. Gravity Filtration

In gravity filtration, you have to use another bubbler **Bubl R2**. Connect the black rubber tubing from **R2** to side pipe **A2** of **Q2**. In this method, filtration of the suspension is carried out with neither pressurization nor evacuation. The only thing you have to do is slowly introduce **N_2 gas** to **Q1** through **R1**.You need not apply **N_2 gas** from **R2**. A drawback of this procedure is that it takes a rather long time.

2. Pressure Filtration

The procedure is nearly the same as the gravity filtration.

Also in this procedure, you have to use another **Bubl R2** and connect its black rubber tubing to **A2** of **Q2**. The only difference is to close **K1** with a pinch cock; then the suspension in **Q1** is pressed out by the pre-adjusted pressure of the nitrogen line (0.3-0.4 atm) through the sintered glass filter **X** into **Q2** to obtain a filtrate.

As the progress of the filtration, the pressure in **Q2** is released through **A2**, **R2** and **H2**.

3. Filtration under Reduced Pressure

In this procedure, the receiver **Q2** is partially evacuated by a vacuum pump.

Depending on the extent of the evacuation and the vapor pressure of the solvent, the sintered glass filter can get stuck due to vaporization of the solvent.

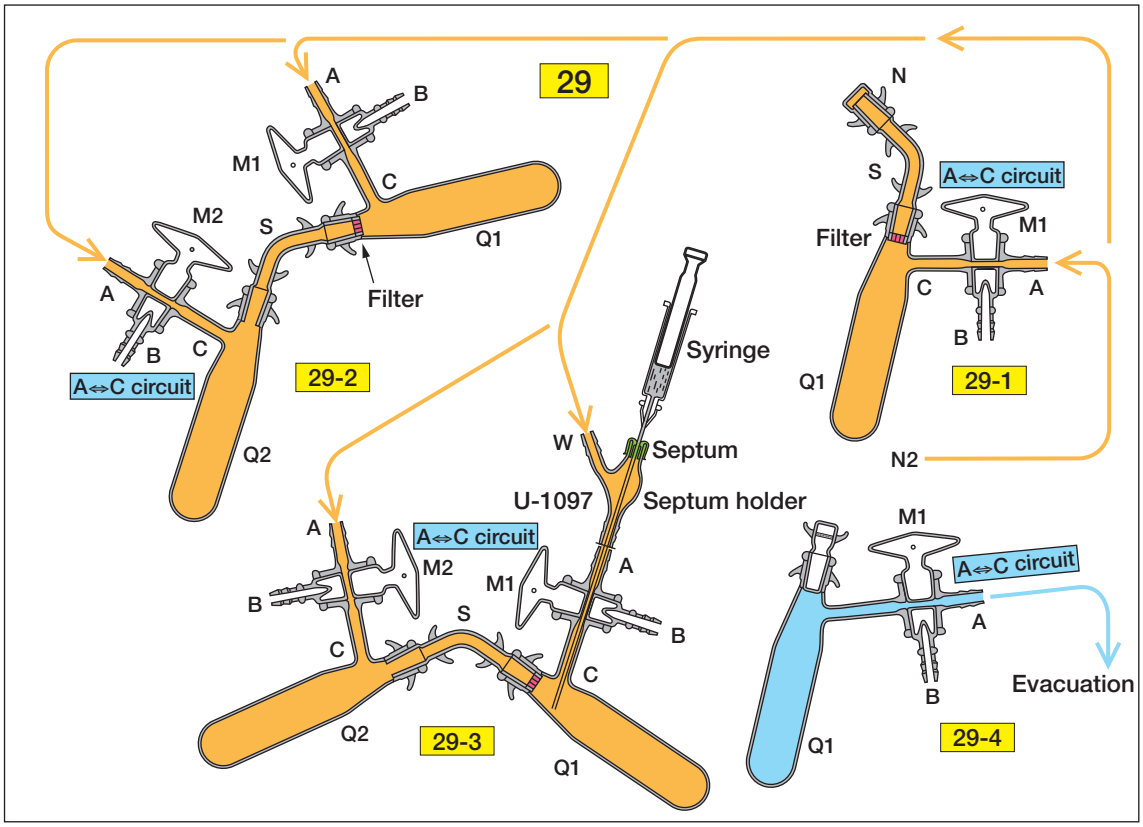
Accordingly , pressure filtration is preferable.

1. Place the suspension onto **X**.
2. Partially evacuate **Q2** by manipulating the **HG Plug** of the selector and then close **M2**.
   If you open the **Cockplug Z**, filtrate is collected in **Q2**.
   Depending on the case, you may have to evacuate again.
   If you do need to further evacuate, do not fail to place a cold trap between the selector and the vacuum pump.

4. Filtration Method through Tube Manipulation

● **When filtrate is desired**

1. Turn **M1** to circuit **A⇔C** to allow **N_2 gas** to go through **Q1**, remove stopper **N** while **N_2 gas** is running through **Q1**. ⇒29-1
2. Using **120° adapter with a sintered glass filter**, connect the donating **Q1** and **Q2** (**N_2 gas displacement** of **Q2** already completed) 29-2, turn **M2** to circuit **A⇔C**, release the **gas**, and tilt the **Q1⇔S⇔Q2** linked system from right to left to **filter by gravity as shown in** 29-2.



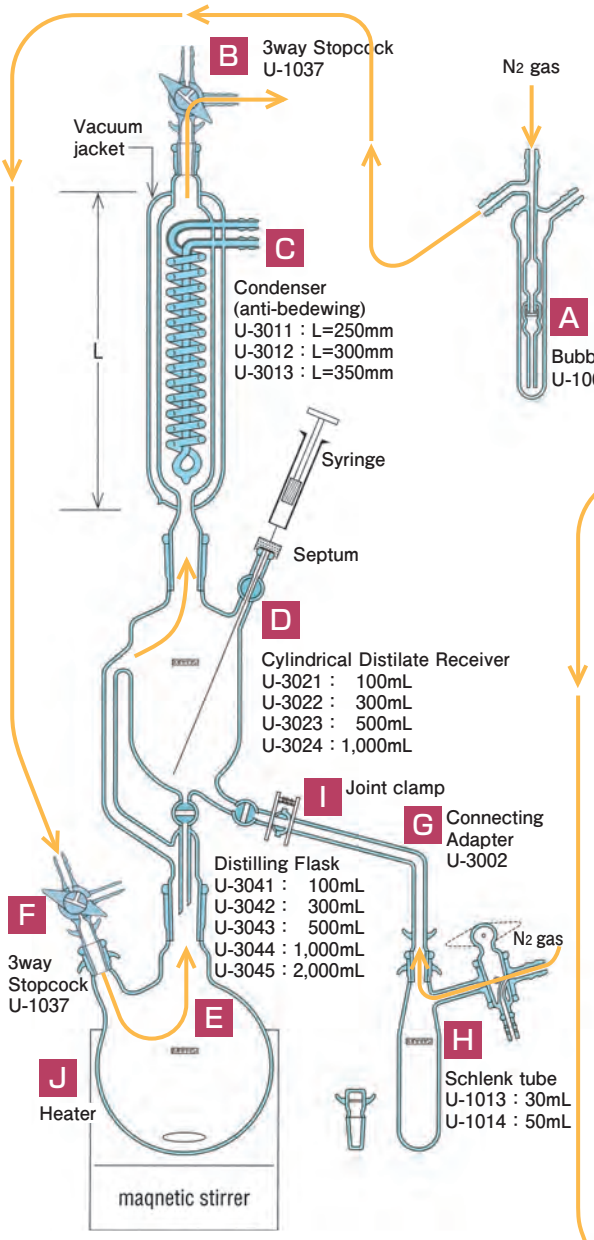
● **When solid is desired**

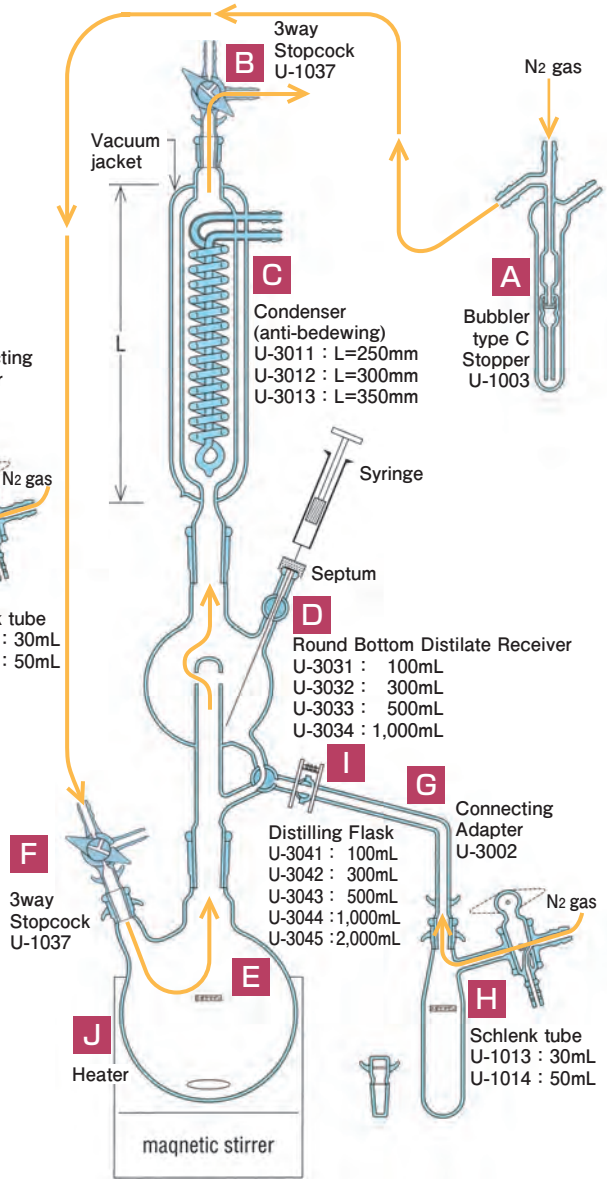
1. When a solid (crystal) is required, tilt the **Q1⇔S⇔Q2** linked system to filtrate 29-2. While releasing **N_2 gas** from **septum holder branch pipe W** on **branch pipe A** of **M1**, pierce the needle of the **Syringe** through the septum and circuit **A⇔C** of **M1**. Place it so that the tip of the needle reaches the inside of **Q1** and inject the solvent and shake the apparatus to wash the solid. Then filter the mixture through the sintered glass filter 29-3.
2. Remove **S** and **Q2** from linked system **Q1⇔S⇔Q2** and fix stopper **N** to **Q1** to close it.
   Turn **M1** to circuit **A⇔B**. Remove the septum holder from **branch pipe A**.
   After that, turn **M1** to circuit **A⇔C** and apply vacuum through **A**, to desiccate the solid to isolate it, as illustrated in 29-4.

17. Distillation Apparatus for dry and N2 -Free Solvents

Convenient distillation apparatus for dry and N_2 gas free solvents is illustrated herein.
The condenser is vacuum-jacketed to prevent from bedewing in summer or wet season.
For details, see illustrations below.

* For details, see page 53, 54.





#	Name of article
A	Bubbler type C
B	3way Stopcock
C	Vacuum jacketed Condenser (anti-bedewing)
D	Distilate Receiver
E	Distilling Flask (2 necked)
F	3way Stopcock
G	120° bent Adapter
H	Schlenk Tube
I	Joint clamp
J	Support / Heater assembly

18. Example Operations in Synthesis Reaction

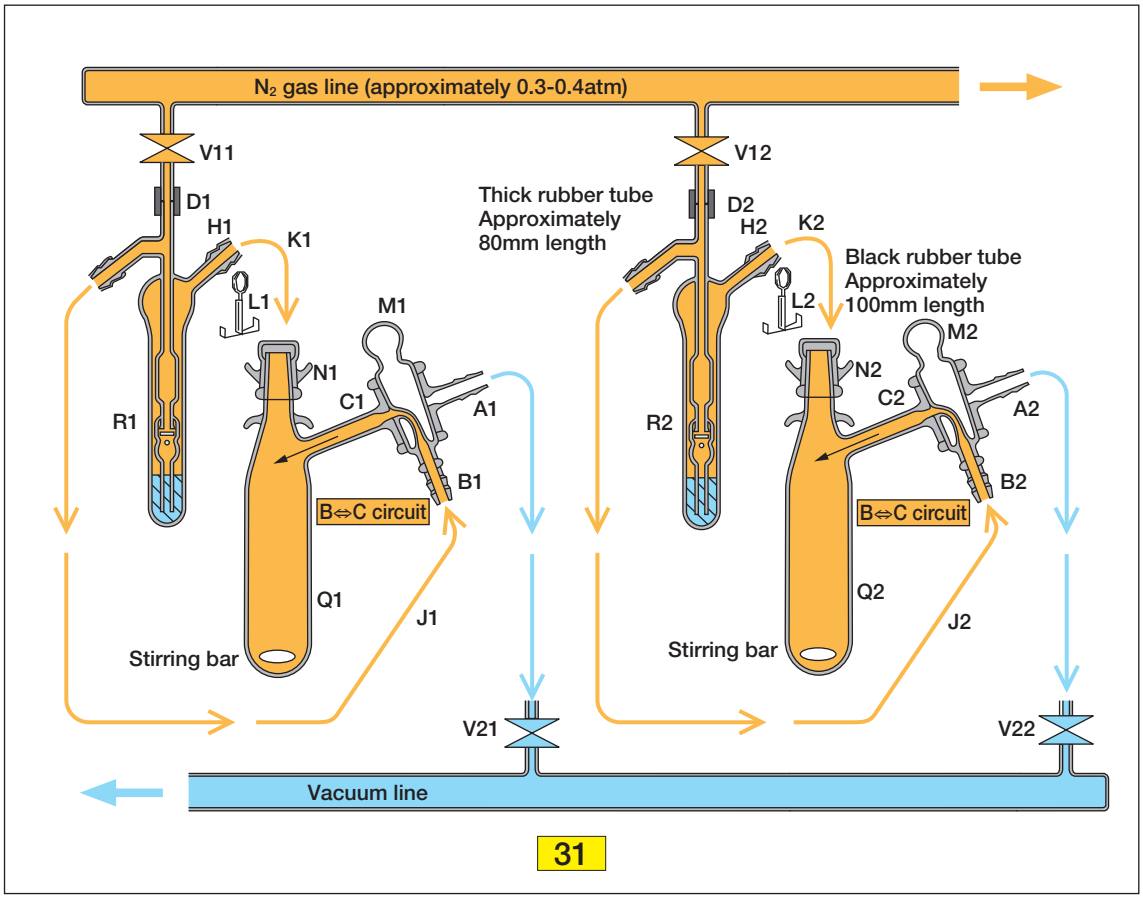
With the synthesis of $Pt(PEt_3)_4$ as an example, we will explain operation procedures in synthesis reactions.

$$K_2PtCl_4 + 4PEt_3 + 2KOH + C_2H_5OH \longrightarrow Pt(PEt_3)_4 + 4KCl + CH_3CHO + 2H_2O$$

Reactant PEt_3 and product $Pt(PEt_3)_4$ are both highly sensitive to oxygen. The former not only gives off a strongly offensive odor, but is also a poison. Thus, it is necessary to use it in an **N_2 gas atmosphere** under a fume hood. For this, the **N_2 gas** line fitted with **Bubl** must be installed under a fume hood as well. The PEt_3 will certainly be stored in an **N_2 gas atmosphere**, but generally, the solvents, reagents, etc., used for the reaction, recrystallization, extraction, and washing, must have oxygen removed through distillation under a **stream of N_2 gas**, vacuum distillation, degassing, etc.

Operation Procedure

1. Set up two 100 mL-**Tubes** **Q1** and **Q2** with a magnetic stirring bar as follows (illustrated in 31).



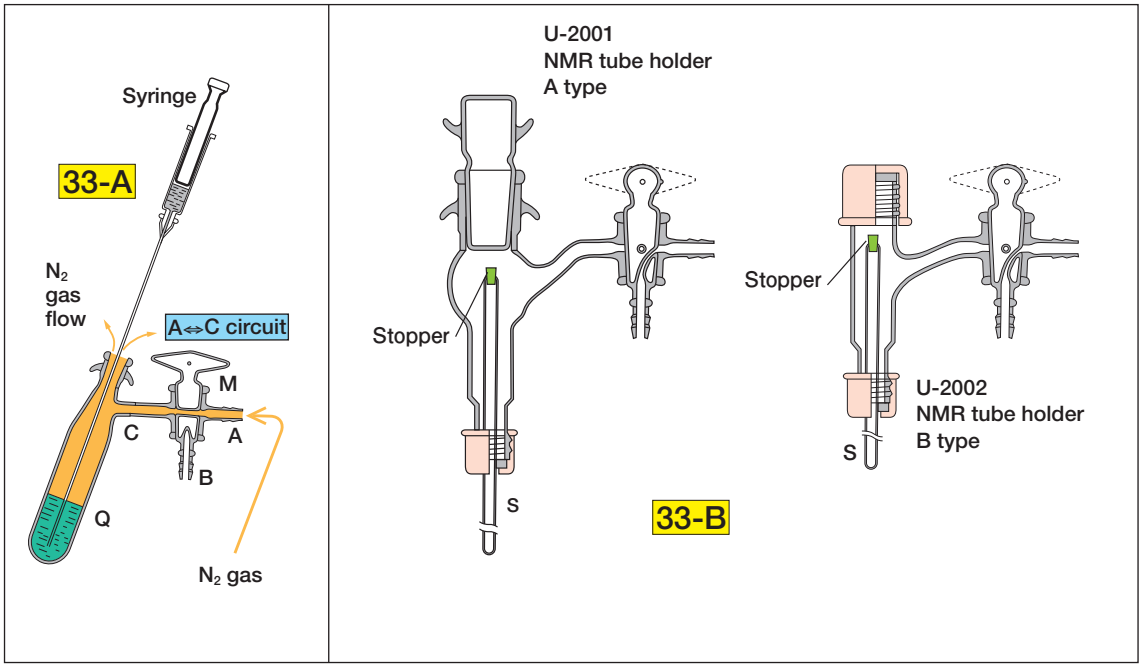
2. Place KOH (0.7 g, 12.5 mmol) to **Q1**, replace the air in it by **N_2 gas** as explained in 10, and connect black rubber tube **J1** from **R1** to the branch pipe **A1**. Open the valve **V11**, then **N_2 gas** flows out through **J1**, **A1** and **B1**. Turn **M1** to circuit **A⇔C** and remove the stopper **N1**. **N_2 gas** through **J1** is now flowing out from the opening of **Q1**, through which you can introduce water (1 mL; degassed and stored in a separate **Tube** under **N_2 gas**)

and EtOH (30 mL; degassed and stored in another separate **Tube** under **N_2 gas**) by means of **Syringes**. Stir the mixture until a homogeneous solution is obtained and then add PEt_3 (3.0 mL, 20mmol;stored in a separate **Tube** under nitrogen). Put the stopper N1 back to the opening this operation makes **N_2 gas** flow out through **R1** and **H1**; you may leave the **N_2 gas** flowing out

3. To the other 100 mL-**Tube** **Q2**, place K_2PtCl_4 (1.5 g, 3.6 mmol; this is air stable and no need to store under **N_2 gas**) and replace the air in **Q2** as explained in 10, and connect black rubber tube **J2** from **R2** to the branch pipe **A2**. Open the valve **V12**, then **N_2 gas** flows out through **J2**, **A2** and **B2**. Turn **M2** to circuit **A⇔C** and remove the stopper **N2**. **N_2 gas** is now flowing out through **J2** and the opening of **Q2**, through which degassed water (10 mL) is added to dissolve K_2PtCl_4. Withdraw the resulting solution by means of a **Syringe**, put the stopper **N_2** back to the opening and close **V12**
4. Now you add the solution taken in the **Syringe** obtained in the previous operation to the solution in **Q1** as follows. Open the stopper **N1**; **N_2 gas** is now flowing out through **J1** and the opening of **Q1**. Insert the needle of the **Syringe** and add the solution in the **Syringe** to the solution in **Q1** slowly (over some five min). During this operation, **N_2 gas** is flowing out from the opening. There is no need to flow rapidly, but if the flow rate is too low, air may come in through the opening. You can check the flow rate by inspecting the bubbling through **R1** simply by covering the opening with your finger; if the rate is too rapid or slow, control by manipulating **V11**.
5. After the solution has been added after some five min, close **N1** and stir at room temperature for 1 h, then at 60 oC for 3 h. Then, cool the solution to room temperature and the resulting mixture is concentrated as follows. Connect the vacuum rubber tube (fitted to **V21**) to pipe **A1** and turn **M1** of **Q1** slowly to circuit **A⇔C** to evaporate the solvent and volatiles under reduced pressure. An orange oil containing a solid is obtained after the evaporation.
6. Turn **M1** of **Q1** to circuit **A⇔B** and connect **A1** to the **black rubber tube J1** to flow **N_2 gas** through **J1**, **A1** and **B1**, then turn **M1** to circuit **A⇔C** to introduce **N_2 gas** into **Q1**. Take away the stopper **N1** to allow **N_2 gas** to overflow from the opening of **Q1**. Add degassed hexane (15 mL) from the opening by means of a **Syringe** and stir the mixture thoroughly to extract the product.
7. The extract taken out by a **Syringe** is a suspension, which can be filtered as described in 27(Note that the whole glassware must be assembled under **N_2 gas**).
8. Thus, the suspension is placed onto the sintered glass filter fitted to a **Tube** at the top of the setup to allow the extract is filtered into the bottom **Tube**. Repeat the extraction and filtration using the same apparatus to collect the second filtrate also in the same **Tube**.
9. Remove the top part of the assembly (i.e. **Tube** fitted with a sintered glass filter) and evaporate the combined filtrate under vacuum as described in 5. until the volume is reduced to 7~8 mL and **N_2 gas** is introduced by manipulating **M**. Open the stopper **N** to allow **N_2 gas** to overflow, add PEt3 (0.5 mL, 3.4 mmol) to the residue, close **N** and cool the **Tube** to -78°C to precipitate colorless solid product.
10. Remove the supernatant liquid using a **Syringe** as illustrated in 26-1. Wash the solid with 3mL of degassed hexane that has been cooled to -78ºC. Remove the washing using a **Syringe**. Repeat the same washing procedure, connect the pipe **A** to a vacuum pump and apply vacuum at -40 ºC to dry the solid. By manipulating **M**, introduce **N_2 gas**. In general, 2.0-2.2 g of the product can be obtained in this way.

19. Introducing a Sample into the NMR Tube

1. Pierce **NMR** tube S through the hole in the lower screw cap of **"NMR tube holder"** (**U-2001**) and tighten securely. In this way, the rubber seal fitted inside the screw cap will hermetically seal and tightly fix S., thus, the whole apparatus assembled is turned into a **"Schlenk tube with an NMR tube"**.
2. Next, turn the **Cock** to carry out the operation shown in 10 to replace the air with **N_2 gas**.
3. Remove the stopper from the socket on top. Turn **M** to circuit **A⇔C** and leave the **N_2 gas** overflowing from the opening top of **Q** , using a **Syringe**, inject the sample into S as illustrated in 33-A.
4. When the plastic stopper to shut S is male, pick it up with tweezers and put it to the opening of S to close it tightly. Loosen and uncap the lower screw cap of the **"NMR tube holder"** and pull S out. Just in case, stretch and wrap **Parafilm** over the point of connection between the stopper and **S** opening, to ensure hermeticity, and measure NMR.
5. When the plastic stopper to shut S is female, displace the air with the stopper inserted into the interior of the **"NMR tube holder"** in advance. After removing the air inside the stopper as well, pick it up with tweezers to place it over the opening of S. Close tightly and follow the same procedure as in 4.
6. Hermeticity of the connecting point between the opening of S and the stopper after the **"NMR tube holder"** has been removed is not maintained perfect, so be careful when measurements take a long time to carry out.



7. In view of the comment in 6., it is necessary to take more secure procedure, for instance, when the time course of the reaction is followed and the temperature is changed during the reaction.

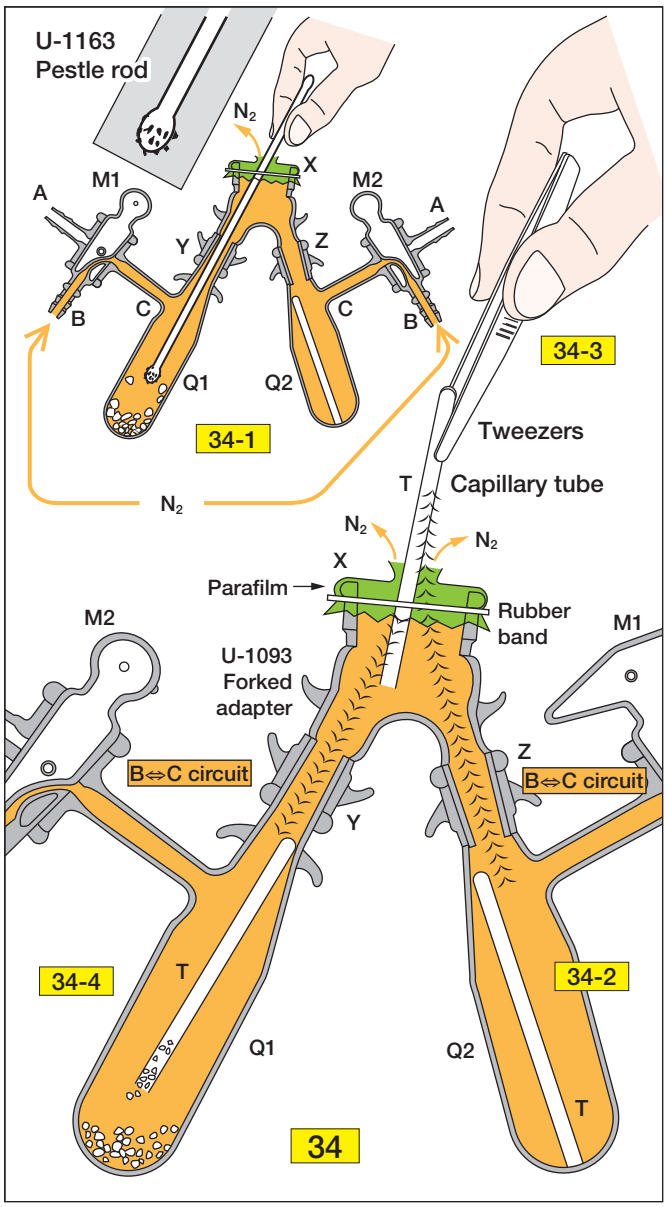
   In such cases, custom-made **NMR tube holder** with a leg made of the same material, thickness, and diameter as **S** (**"NMR tube holder"** should be used. After connecting **S**

to this leg by glassblowing technique and introducing the sample according to the tips described in 2. and 3., melt and seal off S from the leg by gas flame. Note that **S** must be symmetrical in all directions; you need some technique of glassblowing. The tube **S** obtained in this way is completely airtight.

20. Melting Point Measurements in Inert Gases

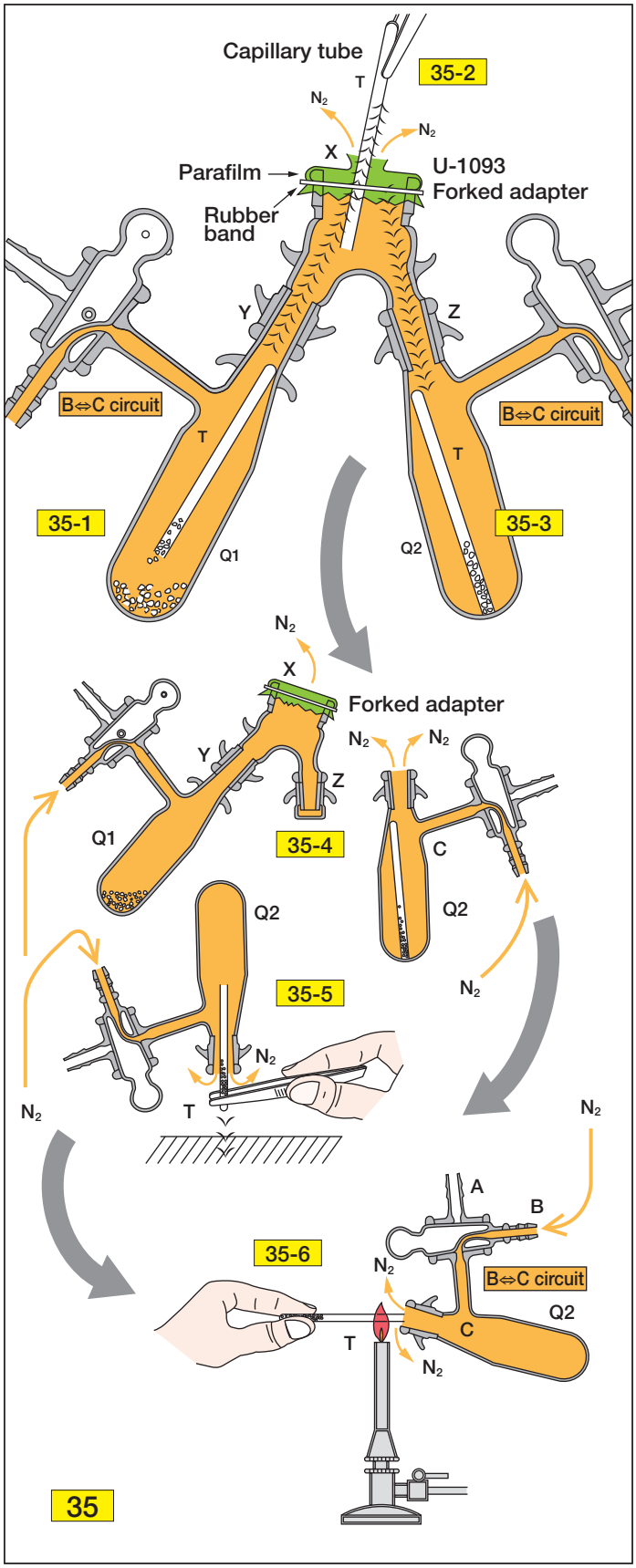
When you are preparing organometalic complexes, you may have to measure their melting points of air sensitive complexes. The technique is somewhat similar to that of transfer of solid materials shown in 24, which you should keep in mind.

You should select an appropriate folked adapter, **Q1** where a sample is stored under **N_2** and empty **Tube Q2**.



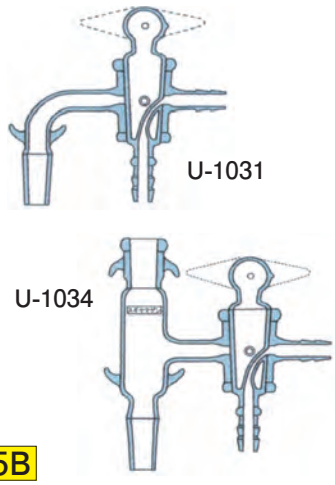
1. The sample in **Q1** must be crushed into fine powder in advance by means of a glass pestle.
2. An empty capillary tube **T**, one end of which is sealed, is placed into **Q2** with the open end facing down.
   Then, replace air with **N_2 gas** in **Q2** containing **T** by manipulating **Cock M2**.
3. **Q1** and **Q2** thus prepared are set to the legs of an appropriate forked adapter, while **N_2 gas** is overflowing from the forked adapter through its top opening. The opening is then covered with a plastic film (**Parafilm**) and make a small hole to insert tweezers.
4. While **N_2 gas** is overflowing from the forked adapter through the small hole, pick **T** up with tweezers and transport it to **Q1**. The open end of **T** is pushed to the powder sample at the bottom several times to stuff the sample into the capillary.
   These consecutive operations are illustrated in 34-2, 34-3, and 34-4.

Read the following procedures while referring to 35.



5. The following is just the opposite to 4. The capillary tube **T** is returned back to **Q2** by means of tweezers.
   These consecutive operations are illustrated in 35-1, 35-2, and 35-3.
6. Remove **Q1** and **Q2** from the forked adapter. **Q1** is not needed anymore; put a stopper on it and store safely.
5. **Q2** is then turned upside down and shake or vibrate the capillary to get the sample to go towards the bottom of T. ⇒35-5
   During the operation, the open end of **T** is located in the stream of **N_2 gas** through **Q2**. There is no risk to be exposed to external air.
6. Finally, **Q2** is turned to a horizontal position, pull capillary T out a little more with tweezers and seal quickly with a gas flame at the middle part about 20mm down from the open end. ⇒35-6

All the operations have now been completed.



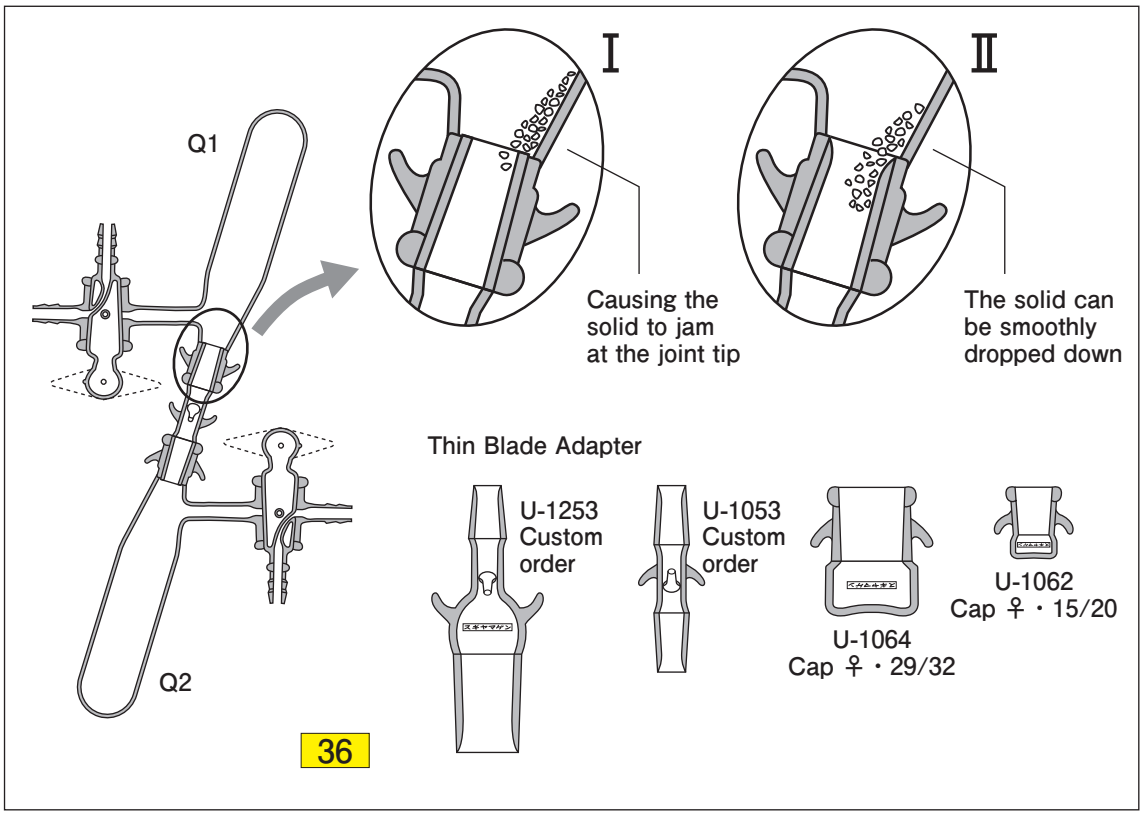
35B

Illustrated for **21** on the next page.

21. Thin Blade Adapter

Apart from liquids, using standard adapters for transfer of solid samples between **Tubes** can cause the solid to jam at the thick joint tip, preventing smooth transfer.

For example, as in 30, when the reaction is carried out in **Q1**, with a **Socket** opening, some sort of adapter is required to transfer the solid sample obtained to another vessel. If a standard straight-type adaptor (**U-1053**) is used in this situation, the thickness of the joint portion acts as a barrier. The valuable solid and crystal gets jammed and cannot be transfered smoothly. In such cases, it is convenient to use a custom-made thin blade adapter (**Custom order**), with the tip of its joint portion as thin as a blade, such as the one shown in 36. It is necessary to carefully remove the grease from the opening of **Q1** with a tissue prior to attaching this adapter. The blade can be nicked easily, so while it is not in use, cover it with a stopper to protect it.



22. To Transform the Flask Into a Schlenk Tube-Type Reaction Vessel

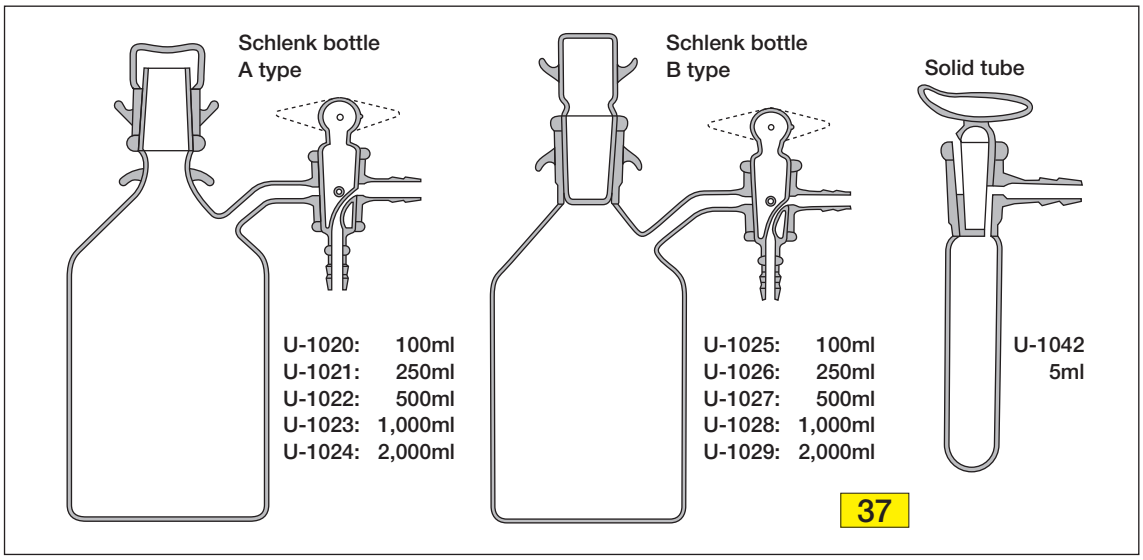
In some cases, one may want to have another **Cock** during a procedure, or to use a large 3-necked flask as a **Tube**-type 3-necked flask during the experiment. The above illustrated adapter with a **Cock** (**U-1031**,**U-1034**) will come in handy in such cases. This adapter is very convenient as it allows you to add another **Cock** without reducing the number of flask openings.

*Due to editing reasons, the cock (**U-1031**,**U-1034**) is illustrated in the former page. 35-B

23. Solvent Container to avoid frequent distillation

Many research labs use distillation apparatuses to dry or distill common solvents under **N_2 gas**. However, there are approximately 10-20 common solvents, including those used only occasionally, thus it doesn't make sense to reflux and distill every time. Also, for operations like recrystallization, one may encounter a sudden need for a solvent not frequently used. Apart from the case in which only a small quantity, readily available by simple degassing, is needed, doing reflux and distillation at that time would cause a large loss of time. By having many different kinds of solvents and water saved and reserved in Schlenk bottles 37 for use at a time like this, you can avoid the troublesome procedures for degassing and drying of solvents and reagents described in 09. This allows experiments to proceed simply, reliably, and rapidly.

The solvents stored in Schlenk bottles can be used without a time limit if you operate carefully as you treat the **Tube**. When you close the stopper after taking the needed amount of solvent, thoroughly carry out the procedures required not to allow air to infiltrate into the bottle as explained in 07



Trust in the group is indispensable for shared use of solvents stored in Schlenk bottles. Absolutely avoid sharing with individuals who tend to skip over the operating tips or those with a weak sense of responsibility. **Schlenk bottles U-1020~1024** have **Socket** opening with a slightly shorter **Socket** on the cap. In this design, care has been taken to strictly prevent the grease from coming into the bottle. ⇒06

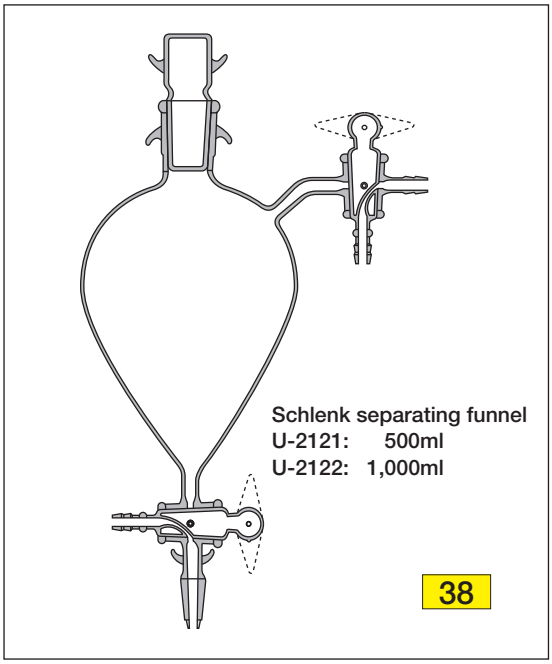
Some bottles like **U-1120~1124** have greaseless valves. Because the Schlenk bottle is a large flat-bottomed glass vessel, take into account the possibility of rupturing when evacuated. Just in case, it is recommended to evacuate after putting the bottle into a cloth bag with a string tying the opening.

24. Storage Vessels for Solids

Solid tube (**U-1042**) illustrated in 37 is a storage vessel for samples that decompose by oxygen or moisture. This vessel has a simple structure that allows switching the flow of **N_2 gas** simply by turning the **Cockplug** that also serves as a stopper.

If the sample sticks to the female joint when transferring from this vessel into another by means of spatula, wipe the joint with a paper-tissue. Thoroughly carry out the evacuation-nitrogen introduction procedures and ensure the complete displacement with **N_2 gas** inside the solid tube. When storing the sample, turn the stopper from the position shown in 37 180º to shut it tightly and isolate the interior from outside air.

If you make large crystals of compounds like Pd(PPh3)4, the vessel can be used to store them for a number of years. However, this vessel is unsuitable for compounds that require strict exclusion of air or moisture, and use **Tube** like **U-1015** with **Cock**.



25. Separating Funnel

The separating funnel shown in 38 is only used for large-volume extraction or washing. For small-volume extraction and washing, add extraction solvent or water to the **Tube** or Schlenk flask that contains a sample solution. Consecutively shake or stir and then leave it to stand. It is much easier to remove upper or lower layer with a syringe after that. Also, this ensures the strictness.

26. Handling Silica Gel etc. Used as a Desiccant and in Column Chromatography

In extraction and washing described one of the in aqueous in most cases. If you need to get an organic layer, you must desiccate with a drying agent, which contains oxygen. The silica gel and alumina for the column chromatography also contain oxygen. So adding them as such would lead to oxygen contamination. Thus, prior to use, you must insert the silica gel and alumna into a **Tube**-type vessel, heat at a temperature of about 100-150ºC, and evacuate for several hours to deoxygenate them (do not forget to set up a cold trap). You must only use the silica gel or aluminum after carrying out these procedures, cooling to room temperature and introducing **N_2 gas**. Because moisture is also removed during the procedure, the silica and alumina for the column chromatography become highly active (the adsorptive power increases); you must add an appropriate amount of water to reduce their activity after the procedure.

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