Product Information

Sigma-Aldrich® Kilo-Lab® Cylinder System
Cylinder Packaging System and Recommended Transfer Procedures

Technical Bulletin AL-263

TECHNICAL BULLETIN

Product Description
The cylinder packaging system provides a convenient method for storing and dispensing development and pilot-plant scale quantities of high-hazard liquids. The properties of these products require personal exposure be kept to a minimum. Many of the liquids must be handled and stored without exposure to atmospheric moisture or oxygen. Fortunately, the design of the Kilo-Lab® cylinder system (see Figures 1-3) enables convenient transfer of liquids using the equipment and techniques described in detail in this bulletin. Sigma-Aldrich® provides the special equipment (see Appendix) needed for handling Kilo-Lab cylinders and their contents.

A complete and detailed listing of equipment is furnished in the Appendix of this bulletin. Throughout the bulletin catalog numbers are provided for parts and equipment. Please refer to the Aldrich Catalog/Handbook of Fine Chemicals or our website, sigmaaldrich.com, for these listings.

There is a complete cylinder maintenance program to ensure each customer receives a cylinder in the best possible condition. Each cylinder is stamped with a unique serial number and a detailed individual history is maintained for each container. Naturally, these cylinders meet or exceed all applicable shipping regulations. A deposit is required with the purchase of a chemical packaged in these highly specialized cylinders. If the cylinder is returned in good condition within six months by the purchaser, credit will then be issued for the deposit amount.

Figure 1.
90 Liter Kilo-Lab Cylinder

Figure 2.
200 Liter Kilo-Lab Cylinder
Components
Kilo-Lab cylinders are available in 4 sizes. Technical Bulletin AL-235 covers cylinders with a nominal capacity of 18 Liters. This bulletin covers the cylinders with nominal capacity of 90, 200, and 400 Liters. Table 1 provides detailed specifications for these larger cylinders. The differences between the three sizes are the overall dimensions, capacity, liquid outlet valve, and dip tube (see Figure 5).

The standard liquid outlet valve is a CGA 350 valve with 1/4 inch I.D. dip tube. Except for cylinders containing materials which are poisonous by inhalation, 200 and 400 Liter cylinders can also be equipped with a 3/4 inch ball valve with 7/8 inch I.D. dip tube. The larger outlet on this option allows the cylinder to be emptied in a short period of time. All cylinders are shipped with a cover to protect the valves. Thus, no additional packaging is required when they are shipped by common carrier.

The Kilo-Lab cylinder valves are shown in Figure 4. The pressure relief valve and nitrogen-inlet/vent valve are connected to the vapor space inside the cylinder. The CGA 350 liquid outlet valve is connected to a dip tube inside the cylinder. The 200 and 400 Liter cylinders, except those containing chemicals, which are poisonous by inhalation, also have a rapid-empty liquid outlet valve, which is connected to a second dip tube inside each cylinder.
Table 1. Specifications of Kilo-lab Cylinders

<table>
<thead>
<tr>
<th>Specification</th>
<th>90 Liter</th>
<th>200 Liter</th>
<th>400 Liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material of construction, cylinder</td>
<td>carbon steel</td>
<td>carbon steel</td>
<td>carbon steel</td>
</tr>
<tr>
<td>DOT Specification</td>
<td>4BW240</td>
<td>4BW240</td>
<td>4BW240</td>
</tr>
<tr>
<td>Maximum cylinder pressure (psig)</td>
<td>240</td>
<td>240</td>
<td>240</td>
</tr>
<tr>
<td>Cylinder test pressure (psig)</td>
<td>480</td>
<td>480</td>
<td>480</td>
</tr>
<tr>
<td>Shipping pressure, approximate (psig)</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Suggested maximum use pressure (psig)</td>
<td>50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Pressure relief valve set point (psig)</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>Tare weight, with valves (Kg)</td>
<td>35</td>
<td>100</td>
<td>162</td>
</tr>
<tr>
<td>Total Capacity (Liters)</td>
<td>108</td>
<td>215.9</td>
<td>453.6</td>
</tr>
<tr>
<td>Working capacity (Liters)</td>
<td>97</td>
<td>200</td>
<td>408</td>
</tr>
<tr>
<td>Shipping dimensions, L × W × H, inches</td>
<td>15 × 15 × 50</td>
<td>28 × 28 × 47</td>
<td>30 × 30 × 58</td>
</tr>
<tr>
<td>Recommended operating temperature</td>
<td>ambient</td>
<td>ambient</td>
<td>ambient</td>
</tr>
<tr>
<td>Fork truck/forklift access</td>
<td>none</td>
<td>standard</td>
<td>standard</td>
</tr>
</tbody>
</table>

**Outlet Specifications, stainless steel**

<table>
<thead>
<tr>
<th>Specification</th>
<th>90 Liter</th>
<th>200 Liter</th>
<th>400 Liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard outlet valve</td>
<td>CGA 350</td>
<td>CGA 350</td>
<td>CGA 350</td>
</tr>
<tr>
<td>Standard outlet fitting</td>
<td>CGA 350</td>
<td>CGA 350</td>
<td>CGA 350</td>
</tr>
<tr>
<td>Rapid-empty outlet valve - Not available for materials that are poisonous by inhalation</td>
<td>N/A</td>
<td>3/4 in. Ball</td>
<td>3/4 in. Ball</td>
</tr>
<tr>
<td>Rapid-empty outlet valve fitting - Not available for materials that are poisonous by inhalation</td>
<td>N/A</td>
<td>3/4 in. Female NPT</td>
<td>3/4 in. Female NPT</td>
</tr>
</tbody>
</table>

**Dip Tube Specifications, stainless steel**

<table>
<thead>
<tr>
<th>Specification</th>
<th>90 Liter</th>
<th>200 Liter</th>
<th>400 Liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard I.D. (included with standard outlet)</td>
<td>1/4 in.</td>
<td>1/4 in.</td>
<td>1/4 in.</td>
</tr>
<tr>
<td>Rapid-empty I.D. (included with rapid-empty outlet)</td>
<td>N/A</td>
<td>7/8 in.</td>
<td>7/8 in.</td>
</tr>
</tbody>
</table>

Figure 5. Cylinders
Figure 6.
Cylinder Schematic and Legend

Legend:
A: CGA 350 liquid outlet valve
B: Handle
C: Cap
D: Rapid-empty liquid outlet valve
E: Handle
F: Plug
G: Nitrogen-inlet/vent valve
H: Handle
I: Cap
J: Nitrogen-inlet adapter
K: Nitrogen-flushing adapter
L: ¾ inch Transfer line
M: Ferrule lock fitting
N: Nitrogen-flushing valve
O: Handle
P: Nitrogen-flushing adapter
Q: ¼" Transfer line
R: Nitrogen-flushing valve
S: Handle
T: Pressure Relief Valve

*Not available on 90l. cylinders or cylinders containing materials which are poisonous by inhalation.
Precautions and Disclaimer
Please consult the Material Safety Data Sheet for information regarding hazards and safe handling practices.

Caution: Due to the hazardous nature of many of the products packaged in the returnable cylinders, all users must read this bulletin carefully and completely before starting any actual laboratory/production work. If unsure of any of these procedures or need assistance, please contact Sigma-Aldrich at 1-800-213-1206 prior to use.

Storage/Stability
Cylinders containing product should be stored upright under 5 psig nitrogen pressure in a cool, well-ventilated location, away from heat and direct sunlight. Refrigerate when appropriate. All valves should be closed, and capped or plugged, and the cylinder cover should be closed. Cylinders must be clearly labeled with the product name and hazard labels. Please do not mark directly on or place any additional labels on the cylinders. Instead use tags to label as partially full or empty.

Procedure
Reading and closely following the transfer procedure described in this bulletin, regardless of the quantity needed or the chemical transferred, is recommended. A wide variety of adapters and transfer equipment needed for handling Kilo-Lab cylinders and their contents are offered (see Appendix). Since the products packaged in Kilo-Lab cylinders are typically highly hazardous, and may be reactive or sensitive to water, oxygen or both, they must be handled while using appropriate personal protective equipment and must never be exposed to the atmosphere.

The user must be a fully qualified and experienced laboratory or chemical production worker to handle these reagents without problems. All users must be aware of the very hazardous nature of many of these products. The Material Safety Data Sheet (MSDS) must be read and understood by the user prior to starting any work with the product contained in a Kilo-Lab cylinder. In general, handle only under an inert atmosphere and exercise caution to prevent inhalation of vapors or direct contact with the skin. Operators must wear appropriate personal protective equipment throughout the entire transfer procedure.

During all nitrogen-pressure transfers, the flowing liquid can generate a static charge. Therefore, the cylinder and receiver must be connected to a suitable ground (Catalog Numbers Z197866, Z197904, or Z197912, see Appendix).

Technical experts are available by telephone to answer questions regarding the proper handling of products in Kilo-Lab cylinders. Contact Sigma-Aldrich immediately at 1-800-213-1206 if an inadvertent deviation from the recommended transfer procedure occurs during use of a Kilo-Lab cylinder.

The separate transfer procedures are provided for cylinders with a CGA 350 Liquid outlet valve A and for cylinders with a rapid-empty valve D.

The rapid-empty liquid outlet valve D is available only on 200 and 400 L cylinders, except on those containing materials which are poisonous by inhalation, and should be used only if the cylinder is going to be emptied prior to disconnecting the transfer line and adapters from the cylinder. If a partial charge is required, our recommendation is to use the CGA 350 liquid outlet valve A.

Liquid Transfer - Cylinders with a CGA 350 Liquid Outlet Valve A
Read all steps before starting and do not work alone.
1. Place the cylinder in a secure and upright position in a safe and well-ventilated area. If the product is to be delivered by weight, secure the cylinder on a floor scale. The cylinder must be connected to a suitable ground (Catalog Numbers Z197866, Z197904, or Z197912, see Appendix).
2. Open the protective cover.
3. Make sure the CGA 350 liquid outlet valve A is closed by turning handle B clockwise.
4. Make sure the rapid-empty valve D (200 and 400 L cylinders only) is closed. The valve is closed when the handle E is in the horizontal position perpendicular to the valve body.
5. Make sure the nitrogen-inlet/vent valve G is closed by turning handle H clockwise.
6. Carefully remove cap I from the nitrogen-inlet/vent valve G. Be sure to save this cap for later replacement.
7. Wrap PTFE sealing tape (Catalog Number Z104388 or Z148814) around the threads of nitrogen-inlet/vent valve G.
8. Connect a nitrogen-inlet adapter J (Catalog Number Z251313) to a suitable dry nitrogen (or dry argon) source using flexible tubing. Secure the tubing in place. The nitrogen needs to be pressure-regulated at 5–10 psig.

9. Adjust the nitrogen source to give a slow stream exiting from nitrogen-inlet adapter J and connect it to the nitrogen-inlet/vent valve G. Tighten with a wrench and check the connections using a water/soap solution to ensure there are no leaks. 

**Note:** When connecting the nitrogen-inlet adapter J to the nitrogen-inlet/vent valve G, it may be helpful to use a 90° elbow coupling (Catalog Number Z253235) to make the connection.

10. Assemble or prepare a complete liquid-transfer line Q with nitrogen-flushing adapter P (Catalog Number Z251372) which is clean and dry.

11. Slowly and carefully remove cap C (left-hand threads, turn clockwise to remove) from CGA 350 liquid outlet valve A.

**Caution:** Occasionally there maybe liquid or solid under the cap, and some fuming may occur with the more reactive reagents. Remove any solid from the valve opening by washing with a stream of odorless mineral spirits (Catalog Number 262560) from a wash bottle and, if necessary, use a wire brush to clean the inside threads. Wear appropriate protective equipment.

12. Wrap PTFE sealing tape around the outlet threads of valve A. This is not done to improve the seal but to aid in later removal of adapter P.

13. Connect a suitable nitrogen source using flexible tubing to a nitrogen-inlet adapter J (Catalog Number Z251313) and connect the adapter J to the nitrogen-flushing valve R. Secure the tubing in place. The nitrogen should be pressure-regulated at 5–10 psig.

14. Open nitrogen-flushing valve R on the nitrogen-flushing adapter P and adjust the nitrogen source to give a steady stream of nitrogen exiting from the open end of the transfer line Q and nitrogen-flushing adapter P.

15. While the transfer line is being flushed, connect the adapter P with transfer line Q to CGA 350 liquid outlet valve A and tighten with a wrench (left-hand threads, turn counter-clockwise to install).

**Note:** Install the adapter P such that the nitrogen-flushing valve R is pointing up.

16. Allow the adapter P and transfer line Q to flush with nitrogen for at least 10 minutes.

17. With nitrogen still flushing through the nitrogen-flushing adapter P and the transfer line Q, adapt the open end of the transfer line such that it can be connected to the process line/vessel. Tighten the connection and check all connections in the transfer line Q and nitrogen-flushing adapter P using a water/soap solution to ensure no leaks.

**Notes:**
- Use PTFE sealing tape on any pipe threads.
- Avoid kinking the transfer line. Kinking the transfer line may damage it and result in potential leaks.
- The process line/vessel to which the transfer line is attached should have a valve in-line in the closed position. The process system must be previously flushed with nitrogen and vented appropriately.
- Should the liquid from the cylinder be introduced to the process system at a level below that in the process vessel, a back flow prevention device is recommended. Suck-back into the cylinder may result in a serious incident.
- Sigma-Aldrich offers a variety of connectors to suit the needs for end fittings on the transfer line (see Appendix).

18. Close the nitrogen-flushing valve R by turning handle S clockwise. The transfer line is now ready for the liquid transfer.

19. Verify that the process system has been prepared for the transfer and then open the valve in-line on the process system at the end of the transfer line Q.

20. Cautiously open the CGA 350 liquid outlet valve A by turning the handle B counter-clockwise. Liquid should begin to flow from the cylinder through the transfer line and into the process system. Flow can be detected by visual inspection through a sight glass on the process system or by weight loss from the cylinder if installed on a floor scale. If flow is not detected or if the flow is slow, continue to step 21.

**Note:** The seal on the valve handle will need to be broken before operating the valve.

**Caution:** Wear appropriate personal protective equipment throughout the entire transfer procedure.

21. With the nitrogen source set at 5–10 psig and connected to the nitrogen-inlet/vent valve G, open the nitrogen-inlet/vent valve G by turning handle H counter-clockwise. This will pressurize the vapor space in the cylinder to 5–10 psig of nitrogen. If necessary, the nitrogen pressure can be increased up to 20 psig to increase the rate of flow.

**Caution:** Do not pressurize the cylinder to more than 50 psig.
22. When the desired amount of liquid has been transferred, close the CGA 350 liquid outlet valve A by turning handle B clockwise and then close the nitrogen-inlet/vent valve G by turning handle H clockwise.  
Note: During the actual transfer, the CGA 350 liquid outlet valve A can be opened and closed as needed.

23. With the nitrogen source set at 5–10 psig and connected to the nitrogen-flushing valve R, open the nitrogen-flushing valve R by turning handle S counter-clockwise. This will flush residual liquid from the transfer line Q into the process system. Flush the line for 5–10 minutes while lifting the transfer line Q to prevent any liquid from remaining in low areas of the line.

24. Close the nitrogen-flushing valve R on the nitrogen-flushing adapter P and then close the valve on the process system at the end of the transfer line Q. Continue to Clean-up Procedure.

Liquid Transfer - Cylinders with a Rapid-empty Valve D
Read all steps before starting and do not work alone. If a partial charge is required, use of the CGA 350 liquid outlet valve A is recommended.

1. Place the cylinder in a secure and upright position in a safe and well-ventilated area. If the product is to be delivered by weight, secure the cylinder on a floor scale. The cylinder must be connected to a suitable ground (Catalog Numbers Z197866, Z197904, or Z197912, see Appendix).

2. Open the protective cover.

3. Make sure the CGA 350 liquid outlet valve A is closed by turning handle B clockwise.

4. Make sure the rapid-empty valve D (200 and 400 L cylinders only) is closed. The valve is closed when the handle E is in the horizontal position perpendicular to the valve body.

5. Make sure the nitrogen-inlet/vent valve G is closed by turning handle H clockwise.

6. Carefully remove cap I from the nitrogen-inlet/vent valve G. Be sure to save this cap for later replacement.

7. Wrap PTFE sealing tape (Catalog Number Z104388 or Z148814) around the threads of nitrogen-inlet/vent valve G.

8. Connect a nitrogen-inlet adapter J (Catalog Number Z251313) to a suitable dry nitrogen (or dry argon) source using flexible tubing. Secure the tubing in place. The nitrogen needs to be pressure-regulated at 5–10 psig.

9. Adjust the nitrogen source to give a slow stream exiting from nitrogen-inlet adapter J and connect it to the nitrogen-inlet/vent valve G. Tighten with a wrench and check the connections using a water/soap solution to ensure there are no leaks. Note: When connecting the nitrogen-inlet adapter J to the nitrogen-inlet/vent valve G, it may be helpful to use a 90° elbow coupling (Catalog Number Z253235) to make the connection.

10. Assemble or prepare a complete nitrogen-flushing adapter K (Catalog Number Z251232) which is clean and dry.

11. Remove the plug F from the rapid-empty valve D. Be sure to save this plug for later replacement. Note: Use one wrench to remove plug F and another to hold valve D. This will prevent the rapid-empty valve from unthreading from the cylinder. Caution: Occasionally there maybe liquid or solid under the cap, and some fuming may occur with the more reactive reagents. Remove any solid from the valve opening by washing with a stream of odorless mineral spirits (Catalog Number 262560) from a wash bottle and, if necessary, use a wire brush to clean the inside threads. Wear appropriate protective equipment.

12. Wrap PTFE sealing tape around the male pipe threads of the nitrogen-flushing adapter K and then thread this adapter into the rapid-empty valve D. Be careful not to cross-thread this adapter. Tighten with a wrench while using a second wrench to prevent overtightening the valve in the cylinder.

13. Connect a suitable nitrogen source using flexible tubing to a nitrogen-inlet adapter J (Catalog Number Z251313) and connect the adapter J to the nitrogen-flushing valve N. Secure the tubing in place. The nitrogen should be pressure-regulated at 5–10 psig.

14. Connect a clean and dry transfer line L (Catalog Number Z251348) to the nitrogen-flushing adapter K via the ferrule lock fitting M. Tighten with a wrench.

15. Open nitrogen-flushing valve N on the nitrogen-flushing adapter K and adjust the nitrogen source to give a steady stream of nitrogen exiting from the open end of the transfer line L.

16. Allow the adapter K and transfer line L to flush with nitrogen for at least 10 minutes.
17. With nitrogen still flushing through the nitrogen-flushing adapter K and the transfer line L, adapt the open end of the transfer line such that it can be connected to the process line/vessel. Tighten the connection and check all connections in the transfer line L and nitrogen-flushing adapter K using a water/soap solution to ensure no leaks. **Notes:**
- Use PTFE sealing tape on any pipe threads.
- Avoid kinking the transfer line. Kinking the transfer line may damage it and result in potential leaks.
- The process line/vessel to which the transfer line is attached should have a valve in-line in the closed position. The process system must be previously flushed with nitrogen and vented appropriately.
- Should the liquid from the cylinder be introduced to the process system at a level below that in the process vessel, a back flow prevention device is recommended. Suck-back into the cylinder may result in a serious incident.
- Sigma-Aldrich offers a variety of connectors to suit the needs for end fittings on the transfer line (see Appendix).

18. Close the nitrogen-flushing valve N by turning handle O clockwise. The transfer line is now ready for the liquid transfer.

19. Verify that the process system has been prepared for the transfer and then open the valve in-line on the process system at the end of the transfer line L.

20. Cautiously open the rapid-empty liquid outlet valve D by turning handle E from the horizontal position to the vertical position. Valve D is not to be used as a flow regulator. It must be fully open during transfer or fully closed to stop the transfer. Liquid should begin to flow from the cylinder through the transfer line and into the process system. Flow can be detected by visual inspection through a sight glass on the process system or by weight loss from the cylinder if on a floor scale. If flow is not detected or if the flow is slow, continue to step 21. **Note:** The seal on the lock-out device on the valve handle will need to be broken and the device pulled away from the valve stem before the valve can be operated. **Caution:** Wear appropriate personal protective equipment throughout the entire transfer procedure.

21. With the nitrogen source set at 5–10 psig and connected to the nitrogen-inlet/vent valve G, open the nitrogen-inlet/vent valve G by turning handle H counter-clockwise. This will pressurize the vapor space in the cylinder to 5–10 psig of nitrogen. If necessary, the nitrogen pressure can be increased up to 20 psig to increase the rate of flow. **Caution:** Do not pressurize the cylinder to more than 50 psig.

22. When the cylinder is empty, close the nitrogen-inlet/vent valve G by turning the handle H counter-clockwise. Allow the nitrogen pressure in the cylinder to relieve through the transfer line and into the process vessel; then close the rapid-empty liquid outlet valve D by turning handle E from the vertical position to the horizontal position. **Notes:**
- The lock-out device will need to be pulled away from the valve stem to operate and completely close the rapid-empty valve.
- When using the rapid-empty liquid outlet valve, it is recommended the entire contents of the cylinder be removed before disconnecting the transfer line L and nitrogen-flushing adapter K. During the actual transfer, the rapid-empty liquid outlet valve D can be opened and closed as needed. If it becomes necessary to disconnect the transfer line L and nitrogen-flushing adapter K before the cylinder has been emptied, please contact Sigma-Aldrich at 1-800-213-1206 for information on how to proceed. If it is necessary to remove partials from the cylinder, use of the CGA 350 liquid outlet valve A is suggested.

23. With the nitrogen source set at 5–10 psig and connected to the nitrogen-flushing valve N, open the nitrogen-flushing valve N by turning handle O counter-clockwise. This will flush residual liquid from the transfer line L into the process system. Flush the line for 5–10 minutes while lifting the transfer line L to prevent any liquid from remaining in low areas of the line.

24. Close the nitrogen-flushing valve N on the nitrogen-flushing adapter K and then close the valve on the process system at the end of the Transfer line L. Continue to Clean-up Procedure.
Clean-up Procedure

Caution: Due to the hazardous nature of many of the products contained in Kilo-Lab cylinders, appropriate personal protective equipment must be worn throughout the entire transfer procedure, including the Clean-up Procedure.

1. Verify that nitrogen-inlet/vent valve G is closed and then remove the nitrogen-inlet adapter J from the nitrogen-inlet/vent valve G. Replace cap I on the nitrogen-inlet/vent valve G and tighten with a wrench.
   Note: Wrap the threads on the nitrogen-inlet/vent valve G with PTFE sealing tape before replacing cap I.
2. Verify that the nitrogen-flushing valve R or N on the nitrogen-flushing adapter P or K, respectively, is closed and then remove the attached nitrogen line.
3. Verify both liquid outlet valves A and D are closed and then cautiously disconnect the end of the transfer line Q or L from the process system. Immediately cap both the open end of the transfer line and the open connection of the process system.
   Caution: If the reagent is highly reactive with air, some fuming may result. It is necessary to cap the open ends immediately to prevent a fire. Be sure to take the appropriate precautionary measures when cleaning the process line/system. Wear personal protective equipment. Fire retardant clothing is highly recommended when handling pyrophoric materials.
   Note: Sigma-Aldrich offers a variety of connectors to suit needs for end caps and plugs (see Appendix).
4. If the reagent in the cylinder is pyrophoric or water-reactive, carefully move the cylinder with attached transfer apparatus to a safe location, preferably outside, away from all flammables or combustibles.
5. Again verify that the CGA 350 liquid out-let valve A or rapid-empty valve D, as applicable, are closed, and then cautiously remove the corresponding transfer line and nitrogen-flushing adapter P or K, from the liquid outlet valve.
   Note: Use one wrench to remove the adapter K and another to hold valve D. This will prevent the rapid-empty valve from unthreading from the cylinder.
   Caution: A small amount of liquid may remain behind in the valve opening. If the reagent is highly reactive with air, fuming or a small fire of short duration may result.
6. If the reagent is pyrophoric or water-reactive, place the transfer line and nitrogen-flushing adapter in an empty, dry metal tub and carry to a safe distance from the cylinder, preferably outside, away from other flammables/c combustibles. If the reagent is not pyrophoric or water-reactive, move the transfer line and nitrogen-flushing adapter to a well-ventilated area. See steps 11–14 for clean-up of the transfer line and nitrogen-flushing adapter.
   Note: The transfer line should be cleaned as soon as possible after it is removed from the cylinder. If this is not done, partially hydrolyzed material will often plug the ends of the line while the inner material remains active. This can cause problems later, when the transfer line is cleaned.
7. Return to the cylinder, and if no reaction is evident, rinse the inside of the valve opening with a stream of odorless mineral spirits (Catalog Number 262560) from a wash bottle. Clean the threads if necessary using a small wire brush to remove any solid.
   Note: Do not open any valves while cleaning.
8. If necessary, clean the threads on cap C (or plug F if the rapid-empty liquid outlet was used) and replace cap C (or plug F) on the appropriate liquid outlet valve. Tighten cap C (or plug F) with a wrench.
   Note: Wrap the threads of plug F with PTFE sealing tape before replacing. Be careful not to cross-thread this plug. Use one wrench to tighten plug F and another to hold valve D to prevent it from turning when tightening the plug. Be careful not to overtighten.
9. Verify that all valves on the cylinder are closed and that all caps and plugs are in place and tightened. Remove any chemical residue from the top and sides of the cylinder and then close the protective cover and secure it in place.
10. The Kilo-Lab cylinder can now be returned to storage or shipped back to Sigma-Aldrich. See the storage and cylinder return sections in this bulletin for details. The transfer apparatus should be cleaned as described in steps 11–14.
11. If the reagent is not pyrophoric or water-reactive, clean the transfer line and nitrogen-flushing adapter following normal procedures for process equipment. Work in a well-ventilated area using appropriate personal protective equipment. However, if the reagent is pyrophoric or water-reactive, keep the transfer line and nitrogen-flushing adapter outside. Remove the cap from the end of the transfer line and open the nitrogen-inlet valve on the nitrogen-flushing adapter. While still outdoors, rinse the transfer line and nitrogen-flushing adapter with a stream of odorless mineral spirits into an empty, dry metal bucket. 

Caution: If the product is pyrophoric, this sometimes results in a fire.

12. Now flush the transfer line and nitrogen-flushing adapter with water (or dilute acid, if needed to dissolve solids formed on hydrolysis) and finally with methanol or acetone. 

Caution: If the product is pyrophoric, this sometimes results in a fire.

Note: Be sure water contacts all surfaces of the transfer line and nitrogen-flushing adapter to ensure complete hydrolysis of reactive material. Operate all valves on the transfer line while running water through the valves to ensure complete hydrolysis within the valve housing. After cautiously rinsing the transfer line and nitrogen-flushing adapter with water, it is recommended to completely submerge the equipment in water.

13. The transfer line and nitrogen flushing adapter can be brought inside for a final clean-up following normal procedures for process equipment.

14. Verify that all rinses in the bucket(s) have been completely hydrolyzed before following the appropriate disposal procedures.

Cylinder Return
To return a cylinder to Sigma-Aldrich, the cylinder must be empty (except for residual product) with all valves in the closed position and all valves capped or plugged. Any chemical residue on the outside of the cylinder must be removed. The cylinder cover must also be secured in place. Empty cylinders contain product residue, which is fully regulated as hazardous material and must be shipped in accordance with all applicable shipping regulations. Please do not mark or place any additional labels on the cylinders; use tags to label as empty.

Return empty cylinders to:

Aldrich Chemical Company, Inc.
5485 County Road V
Sheboygan Falls, WI 53085-2814.

Cylinders must be returned in good condition within six months of the date of purchase to receive a full credit of the deposit amount. Please contact our Customer Service Department at 1-800-213-1206 with any further questions regarding the return of Kilo-Lab cylinders.
Appendix

The equipment offered for transferring liquids from the Kilo-Lab cylinders is extensive. The use of these cylinders and the related equipment should be as straightforward and trouble-free as possible. As an added benefit, customers may find this equipment useful for other situations where need to transfer of air-sensitive liquids is needed.

Kilo-Lab cylinders are constructed of carbon steel, while all valves and fittings are stainless steel. A wide variety of adapters and liquid-transfer apparatus needed for safe and convenient transfer of all products contained in these cylinders is offered. The use of stainless steel adapters and valves, and PTFE transfer lines with stainless steel overbraid for all transfers is recommended.

Several options exist for transferring from the Kilo-Lab cylinders. Most of the adapters and transfer lines are offered individually to provide for custom-designed transfer systems. Pre-designed transfer systems are also offered for those who prefer this option.

The requirements for a custom-designed transfer system are as follows:
1. Valves - cavity-filled ball valves are recommended and should be equipped with PTFE seats and seals.
2. Pipe - stainless steel is recommended.
3. Flexible tubing – PTFE tubing with stainless steel overbraid is recommended.
4. Thread sealant - exclusive use of PTFE sealing tape is recommended for all national pipe threaded connections.

The nitrogen-inlet/vent valve on all Kilo-Lab cylinders is equipped with a 1/4 inch male-NPT fitting. A nitrogen-inlet adapter (Catalog Number Z251313, see Figure 7) is offered for this inlet, which provides for easy connection to 1/4 inch I.D. flexible tubing. A 90° elbow coupling (Catalog Number Z253235, see Figure 8) is also offered, which may be helpful when connecting the nitrogen-inlet adapter to the nitrogen-inlet/vent valve on 200 and 400 L cylinders.

The 200 and 400 L cylinders, except those containing materials which are poisonous by inhalation, are equipped with a second outlet valve, the rapid-empty outlet valve. When transferring from the rapid-empty outlet valve, the liquid outlet fitting is 3/4 inch female-NPT. A rapid-empty outlet/flushing tee adapter (Catalog Number Z251240, see Figure 11) is offered. A rapid-empty cylinder outlet/flushing tee adapter assembled with a 1/4 inch needle valve and 3/4 inch ferrule lock fitting (Catalog Number Z251232, see Figure 12) is also offered, which allows connection to the rapid-empty valve via a 3/4 inch NPT and directly to a transfer line with 3/4 inch tube end.
PTFE transfer lines with stainless steel overbraid in several sizes and lengths are offered (see Table 2). Care must be taken when using these transfer lines to prevent kinking, which may cause damage and possibly leaking.

**Table 2.**

Transfer Lines For Liquid Removal from Kilo-Lab Cylinders

<table>
<thead>
<tr>
<th>Inner Diameter (inches)</th>
<th>Length (feet)</th>
<th>End Connection</th>
<th>End Connection</th>
<th>Catalog Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/4</td>
<td>4</td>
<td>1/4 in. nut/ferrule</td>
<td>1/4 in. male-NPT</td>
<td>Z251356</td>
</tr>
<tr>
<td>1/4</td>
<td>4</td>
<td>1/4 in. nut/ferrule</td>
<td>–</td>
<td>Z251372</td>
</tr>
<tr>
<td>3/4</td>
<td>8</td>
<td>3/4 in. nut/ferrule</td>
<td>3/4 in. nut/ferrule</td>
<td>Z251348</td>
</tr>
</tbody>
</table>

All transfer lines are PTFE with stainless steel overbraid and end fittings.
If a longer transfer line is needed, two lines can be connected together to give the desired length. A variety of couplings (see Table 3) are offered to connect transfer lines together, if necessary, as well as to the process system. Several of these couplings are pictured in Figure 15.

**Table 3.**
Stainless Steel Couplings

<table>
<thead>
<tr>
<th>End Connection</th>
<th>End Connection</th>
<th>Catalog Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/4 in. Ferrule lock</td>
<td>1/4 in. Ferrule lock</td>
<td>Z251445</td>
</tr>
<tr>
<td>1/4 in. Female NPT</td>
<td>1/4 in. Ferrule lock</td>
<td>Z251453</td>
</tr>
<tr>
<td>1/4 in. Male NPT</td>
<td>1/4 in. Male NPT</td>
<td>Z251461</td>
</tr>
<tr>
<td>1/4 in. Tube end</td>
<td>1/4 in. Male NPT</td>
<td>Z251488</td>
</tr>
<tr>
<td>3/4 in. Ferrule lock</td>
<td>3/4 in. Male NPT</td>
<td>Z251291</td>
</tr>
<tr>
<td>1/4 in. Replacement</td>
<td>1/4 in. Replacement</td>
<td>Z251534</td>
</tr>
</tbody>
</table>

Figure 15.
Couplings

A cap and plug (see Table 4) are offered to seal the end of the transfer lines and/or process and cylinder valves. Several of these caps and plugs are pictured in Figure 16.

**Table 4.**
Stainless Steel Cap and Plug

<table>
<thead>
<tr>
<th>Description</th>
<th>Material</th>
<th>Function</th>
<th>Catalog Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/4 in. Cap</td>
<td>stainless steel</td>
<td>Seals 1/4 in. tube end</td>
<td>Z251518</td>
</tr>
<tr>
<td>1/4 in. Plug</td>
<td>stainless steel</td>
<td>Seals 1/4 in. ferrule lock end</td>
<td>Z251526</td>
</tr>
</tbody>
</table>

Figure 16.
Cap and Plug
A variety of valves (see Table 5 and Figure 17) are individually offered.

**Table 5. Stainless Steel Valves**

<table>
<thead>
<tr>
<th>Inlet</th>
<th>Outlet</th>
<th>Valve Type</th>
<th>Catalog Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/4 in. Male NPT</td>
<td>1/4 in Male NPT</td>
<td>Packed, Needle, in-line</td>
<td>Z146064</td>
</tr>
<tr>
<td>1/4 in. Male NPT</td>
<td>1/4 in. Male NPT</td>
<td>Packed, Needle, 90° angle</td>
<td>Z146056</td>
</tr>
<tr>
<td>3/4 in. Male NPT with 1/4 in. Female NPT tap for dip tube</td>
<td>CGA350</td>
<td>Diaphram 90° angle</td>
<td>Z251208</td>
</tr>
</tbody>
</table>

**Figure 17. Valves**

Miscellaneous items which are useful when assembling and transferring products from Kilo-Lab cylinders:

- PTFE sealing tape - 520 inch roll for wrapping male pipe threads to give air-tight seals.
  - 1/4 inch wide roll (Catalog Number Z148814)
  - 1/2 inch wide roll (Catalog Number Z104388)

**Table 6. Grounding Straps**

<table>
<thead>
<tr>
<th>Description</th>
<th>Length</th>
<th>Catalog Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexible bonding wire with 2 copper alligator clips</td>
<td>3 ft.</td>
<td>Z197866</td>
</tr>
<tr>
<td>Flexible bonding wire with 2 hand clamps</td>
<td>3 ft.</td>
<td>Z197904</td>
</tr>
<tr>
<td>Flexible bonding wire with 1/4 in. terminal and hand clamp</td>
<td>10 ft.</td>
<td>Z197912</td>
</tr>
</tbody>
</table>
Sigma-Aldrich reserves the right to substitute products and parts, and to make changes and revisions to dimensions and other specifications described herein at its sole discretion without prior notice to purchaser.

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The foregoing discussions and procedures are given to assist our customers in the transfer and use of our products supplied in Kilo-Lab cylinders. The user must read this bulletin carefully, consult the figures, and follow each step in the transfer procedure. Only a technically qualified individual should perform this procedure. If you need further assistance at any time, please contact us at 1-800-213-1206.

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