D. A. Watson Group Manual

Department of Chemistry and Biochemistry
University of Delaware

Rev 1.01
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Introduction

Welcome to the group!

The purpose of this manual is to give new lab members a resource for information about standard group policies and procedures, as well as instructional information on operation of the most critical lab instrumentation. This document will both answer questions and provide an understanding for expectation for all lab members, in a way that I hope is clear, reasonable and avoids ambiguity. This is also a work in progress, additional information will be added as the group develops. If you think that information needs to be added, please let me know.

It is expected that all persons joining the group will CAREFULLY read this document before they begin work in the lab and refer back to it over the course of their time in the lab, as necessary.

No document is perfect, or all-inclusive. If there are mistakes or omissions, please let me know. Moreover, if policies become outdated due to changes in the lab setting or equipment, they should be updated. However, changes to policies outlined in this manual need to be approved by Don prior to their implementation.

Finally, if there are questions that are left unanswered (as there will be) or disagreements over the contents of this manual, please, by all means, talk to me about it. Nothing can waste more time or lead to more resentment that assuming you know someone's opinion or thoughts. If you want to talk about something, let's talk. That is one of the things I am here for.

Again, welcome to the group. I am excited that you are going to be a part of our research.

Sincerely,

Don

Day 1 Checklist

The following items need to be accomplished on the first day in lab:

- Go to Sue James' Office (104A BRL, suejames@udel.edu, ext 8410) and complete any outstanding HR requirements. International students may need to visit the International Scholar's Office.
- Pickup keys from Sue James. You will need 4 keys (there maybe be a $10/key deposit required)
    - Building Key
    - Lab Key (LDL 207/224)
    - MPW Lab Key
    - NMR Lab
- Get a UDel ID Card from Student Services.
- Obtain UDel Email address. This will be an important communication link.
- Complete online safety training (http://www.udel.edu/ehs/safetyedu.html). Complete the following modules:
    - Chemical Hygiene/Right-To-Know Safety Training for Research Personnel
    - Corrosive Chemical Safety Training
    - Proper Chemical Waste Disposal Training
    - New Graduate Student Orientation - Laboratory Students
- Make an appointment with Dr. Steve Bai (033 BRL, bais@udel.edu, ext 8901) for NMR training.
- Get account setup on group computer
- Read this group manual carefully.
- Setup appointment with Don to discuss group policies.
- Go to Stockroom (BRL 063). Meet Connie and buy a labcoat.
- Get a notebook from DAW.
- Give DAW emergency contact information (mandatory).

Meetings:

Group Meeting: TBA
Organic Journal Club (OJC): Thursday 12:30-1:30pm, 219 BRL
Fox Group Meeting: Friday, 1-2:30p, 219 BRL
Organic/Inorganic Seminar Series: Wednesday, 4pm, BRL

LDL = Lammont DuPont Lab; BRL = Brown Lab; QHD = Drake Hall

Safety

Safety is the utmost importance in the D. A. Watson group. Every precaution needs to be taken to ensure that you and your co-workers are safe at all times in the lab. <u>This means that if an experiment cannot be perform in a way that is safe, it should not be done. No exceptions.</u> Specific safety rules for the lab follow, but please be aware that safety will be taken very seriously. *Gross* lapses in safety protocol, even after a single instance, will constitute grounds for immediate dismissal from the laboratory. Repeat minor violation may also result in a researcher being asked to leave the group. This is not an option that will be exercised lightly, but is necessary for the protection of all persons working in the lab.

You are required to complete EH&S safety training prior to working in the lab.

Use common sense. Not all situations can be spelled out in detail. Use sense when setting up experiments. If you are unsure about how to do something safely, seek help from Don or an experienced group member, or some one in the department with expertise in an area (as needed).

In case of evacuation, report to the departmental evacuation area. See attached map. Report directly to DAW or the Lab Safety Officer. This is mandatory so that we know you are not trapped in the building or otherwise injured.

Safety glasses are required for all persons in the lab. This includes visitors, sales-people, workmen or service contractors, as well as all lab personnel. No exceptions.

The use of gloves is required when handling chemicals. Refer to the glove selection chart if you are unsure of the type to use. Throw away (in chemical waste) dirty, contaminated or used gloves. Do not handle door knobs leading out the lab with gloves. <u>*Only one gloved hand is allowed outside the lab*</u>. If this is not possible to do safely, you must get someone to help you to open doors that has a free, non-gloved hand.

No food, gum or drink is allowed inside the lab.

Labcoats are required for anyone conducting experiments, working at a fume hood, or in contact with chemicals. Labcoats will be provided for lab personnel.

Wear appropriate clothing. Closed toe, close top shoes are required in the lab. Sandals, flip-flops and other like-footwear are not allowed, as they do not protect your feet against spilled chemicals and dropped glassware. Long pants are required for those conducting experiments; shorts, skirts, etc do not provide protection for your legs. Clothing made exclusively of polyester, rayon, or other highly flammable materials is strictly forbidden – recently a student died at UCLA after spilling a small amount of a pyrophoric material on her polyester sweater. Cotton, demin and other natural fiber materials provide better fire resistance. These rules may seem stringent, but they are for your own protection.

Headphones are not allowed in the lab. Headphones in lab present a dangerous situation as they prevent you from hearing what is occurring around you; including trouble in the lab (explosions, etc), calls for help from others, and even what is going on with your own chemistry (e.g., a leak vacuum system). DO NOT WEAR HEADPHONES IN LAB. A stereo system is present in lab, and can be used to listen to music at reasonable (safe and non-disturbing) volume. The use of headphones while at your desk is fine.

Never work alone in lab; the risks are too great. Ideally, someone should be present in the laboratory in which you are working. At minimum, someone MUST be present in an adjacent lab and know that you are there. For our group, this means you cannot work in the lab unless someone else is working in our group, Taber's group or Riordan's group.

Do not work when highly fatigued or exhausted. The chances for mistakes increase greatly when you are sleep deprived, the risks to you and others are too great.

If the ventilation system shuts down, or there is a power failure, DO NOT WORK IN LAB.
Nitric acid needs to be used with extreme care. Nitric acid is a strong oxidizer, when mixed with organics it can (will) explode. *DO NOT PUT NITRIC ACID WASTE IN ORGANIC WASTE CONTAINERS. NITRIC ACID NEEDS ITS OWN WASTE CONTAINER.*

Clean up your glassware and hood at the end of each day. Clean workstations improve safety and the quality of science.

Know where the fire extinguishers and lab showers are! Do not risk yourself attempting to put out a fire that you are not sure that you can extinguish.

Know where the chemical spill kits are, but only use them if it is appropriate and safe to do so. For large or highly toxic spills, call EHS or 911.

Know where the eye wash and lab showers are, know how to use them!

Secondary containers are required for all chemicals and chemical waste.

Know where the MSDS Sheets are and how to use them. We are required to have MSDS Sheets for all chemical housed in the lab.

Use proper care when dealing with sharps. Do not leave exposed sharps out in the lab. Know how to properly disposed of sharps.

Use safety shields for any reaction in glass that is under pressure, vacuum, or is being heated while sealed (high pressure reaction vessel, HPRV). This is especially true of sealed NMR tubes, which have seriously injured many people. If there is any chance of explosion, you need to consult with Don BEFORE setting up the reaction.

Run experiments on the smallest scale possible to obtain needed data. Required scale will depend on the nature of the reaction and how much material, if any, is needed. In general, running reaction on the smallest possible scale is safest. If something goes wrong, a smaller scale means less chance for injury.

Only order the smallest amount of chemical needed. Large bottles may seem cheaper, but only if the whole volume will be used. Buying in bulk and then using only a small amount creates storage, safety and disposal hazards.

All reagents must be dated when opened. Some reagents decomposed over time, for example ethers can form explosive peroxides over time. Date all chemicals when they are opened, and do not store ethers for more than 6 months after opening.

Do not use, make or order alkylmercury reagents. Small amounts of these reagents (particularly dialkyl mercury reagents) have killed personnel in other labs. Do not use, make or order them.

Solvents must be stored in flammable cabinets. Never place solvent bottles on the floor. Do not leave solvent bottles on the bench.

Sealed reactors must be used with extreme care. See safety shield requirement above.

Flame sealing of NMR tubes requires special training. Flame sealing NMR tubes can be a very effective means of running sensitive reactions, however, sealing the tubes must be done very carefully. Many researchers have lost eyes due to exploding NMR tubes that were improperly sealed. DO NOT SEAL NMR TUBES WITHOUT BEING TRAINED BY DON.

Liquid nitrogen needs to be used with great care. Liquid nitrogen is very effective for cooling vacuum traps, but if not used properly can lead to condense liquid oxygen. Liquid oxygen (which is blue in color) can flash boil, leading to explosions. DO NOT USE LIQUID NITROGEN WITHOUT BEING TRAINED BY DON.

Heatguns cause fires if misused. Many serious lab fires have been started by solvent vapors from recrystallizations getting pulled into heatguns. DO NOT USE HEATGUNS TO RECRYSTALLIZE WITHOUT THE USE OF A REFLUX CONDENSER IN PLACE.

Get training before handling pyrophoric reagents. Reagents such as BuLI, *t*-BuLi, Me_2Zn, etc. can ignite upon contact with air. If not handled correctly, even small volumes of these reagents can lead to serious fires, injuries or even death. Read the section of this manual on syringe techniques and get training from Don before using pyrophoric reagents.

Quenching reagents should only be done with more than one person present. When quenching pyrophoric reagents, make sure someone else is present and that the quenching protocol is safe. Consult *Prudent Practices* for safe handling and disposal of chemicals. If you have questions or are unsure, ask before you start.

All chemical containers must be labeled at ALL times and must be closed when not in use. Commercial reagents should have a label when shipped, please make sure that this remains legible. If not, please remark bottle before it can no longer be identified. Chemical waste must be stored in proper bottles, MUST be labeled at all times, and KEPT CLOSED when not in use. This is a safety issue and a regulatory issue (EPA and OSHA fines are >>$1000/PER BOTTLE for open and unlabelled waste!!!)

Chemical Waste bottles and containers must be closed at all times. See above. No exceptions.

Additional Safety Training

In addition to initial safety training required on the first day, the following training must be completed within the first 2 weeks:

Compressed Gas Safety Training
Fumehood Training

Highly Toxic Safety Training
Reactive Materials Safety Training
Syringe Technique and Safety Training

Emergencies

If there is a mishap in the lab, as needed 1) Alert Emergency Personal 2) Provide Appropriate First-aid 3) Use Fire Extinguisher or take other actions to mitigate problem, *if safe to do so.* 4) Call Don, ASAP (day or night). A list of emergency contact information will be maintained separately. If in doubt, call 911.

Scientific Integrity

There is no doubt that numerous pressures exist in our community that press students, post-docs and even faculty to discover astounding facts and invent astonishing reactions/processes. For some, it may seem tempting to "alter" the truth in order to meet these "expectations". Thankfully, our field has a good reputation of calling out bad and falsified science; those who cheat are usually caught.

The objective of our group is to conduct science to understand fundamental chemical processes, as governed by the rules of Nature. We will try, to the best of our ability, to use our time wisely and to study important problems, which will maximize the likelihood of making important discoveries, but the most important objective of this group (in terms of published science) is to report accurate, reproducible results. Fortunately, good science involves understanding what processes do not occur much as it does understanding those that do. And, understanding the boundary limits of a system can often lead to more fruitful results. As such, it is requisite that all co-workers fully, accurately, and above all, honestly report the results of experiments. Fabrication, falsification, or "enhancement" of data will not be tolerated in anyway, and will result in <u>immediate</u> dismissal from the group.

The type of scientific misconduct described above must not be confused with errors. We deal with complex problems and instrumentation on a daily basis; as such, *errors will occur*. Sometimes these are due to systematic problems, such as faulty or miscalibrated instruments; other times they are due to incorrect or overzealous interpretation of data (particularly when dealing with poor quality data). The best way to prevent erroneous results is to work carefully, think critically, examine data skeptically, and above all, ensure reproducibility before drawing conclusions. If you realize that you have reported an error (to Don, in group meeting, in publication, or elsewhere) in some aspect of your research, *it is imperative that you immediately inform Don of the error.* <u>No disciplinary action will ever been taken against co-workers for honestly made errors.</u> Failure to report errors once discovered, however, is academic dishonesty and will be dealt with accordingly.

In order to insure that accurate results are being reported from the group, it is important that co-workers collect, maintain and organize such data as is required to support scientific claims and conclusions. It will be the practice of the group that NMR and other primary data will be presented in discussions in order to support conclusions, and it should be expected that Don will regularly examine such data. In addition, other coworkers may be asked to, on occasion, reproduce results from the lab prior to publication. These steps not only ensure accuracy of results (both for error and fabrication), but also allow critical examination of written documents to ensure that procedures and methods can be followed by others, as written. Specific details regarding data collection, organization, and data reporting will be described below.

Work Environment

Our research labs and offices are a work environment, and it is expected that they be treated as such. Appropriate conduct is expected from all members of the group. Harassment, of any type, will not be permitted or allowed. Computer use should be limited to work related activities. The downloading or viewing of explicit material on group computers, or while in group or department facilities on any computer or device, *is strictly forbidden* and will not be tolerated.

It is expected that all persons will maintain proper personal hygiene as to not affect other's work environment.

Group citizenship is taken seriously. A list of Group Jobs will be disturbed separately, it is expected that everyone will perform their task(s) with diligence. Likewise, the use of common laboratory equipment, group chemicals, and group workspace and office space will be conducted in a courteous and respectful way that does not adversely affect other lab members.

Only with *everyone* working as a research team can we all maximize our research productivity and learning while a part of this group.

Meetings

Group meetings will be held on Tuesday Evening at 6pm in order to discuss both group research and recent literature. A schedule will be distributed several weeks in advance of any meeting, in order to give participants time to prepare.

Attendance of the Organic Division journal club is also expected.

Bi-weekly research meetings, either one-on-one with Don or with a small group, will be scheduled. Please come prepared (hand written document is fine) to discuss important results from the previous weeks and bring copies of important spectra.

Literature Reading

An enormous number of journals now exist that are related to our work in this group, which makes keeping track of everything challenging. At minimum, *all* students and post-docs in this group are expect to regularly read *Nature*, *Science*, *JACS*, *ACIE, Org. Lett.*, *Organometallics* for relevant publications. A schedule will be distributed to assign other journals to individuals to keep tabs on and relevant and interesting papers will be flagged for the group at large.

Vacations

You are allowed up to three weeks (15 days) off per year for vacations. Illness (with in reason) and family emergencies will not count against this. It is *expected* that you will take time off during the year, so that you can stay fresh and focused while in lab, however, strongly preferred that you do not take off the entire period all at once. Please notify Don (via email) at least three weeks before you plan to take vacation so that the group can plan accordingly.

Holidays

You are not required to work on University of Delaware Holidays, however, if you have nothing better to do… come join the fun in lab.

Success in the Group and Field

I am sure that you realize by now that chemistry is a demanding and challenging field, but it is also very rewarding. Success in this field demands critical though, dedication to reading, careful experimentation and willingness to work hard. Unfortunately, no one other than you can make the choice that you that you want to succeed and put the effort in that is required to do so. As an advisor, I will do everything within my power to help you in this goal, but ultimately, the responsibility lies with you to make yourself successful.

Standard Operating Procedures

In the coming weeks, as the group is established, standard operating procedures will be developed to provide instruction of the use of common lab instruments and techniques for laboratory procedures.

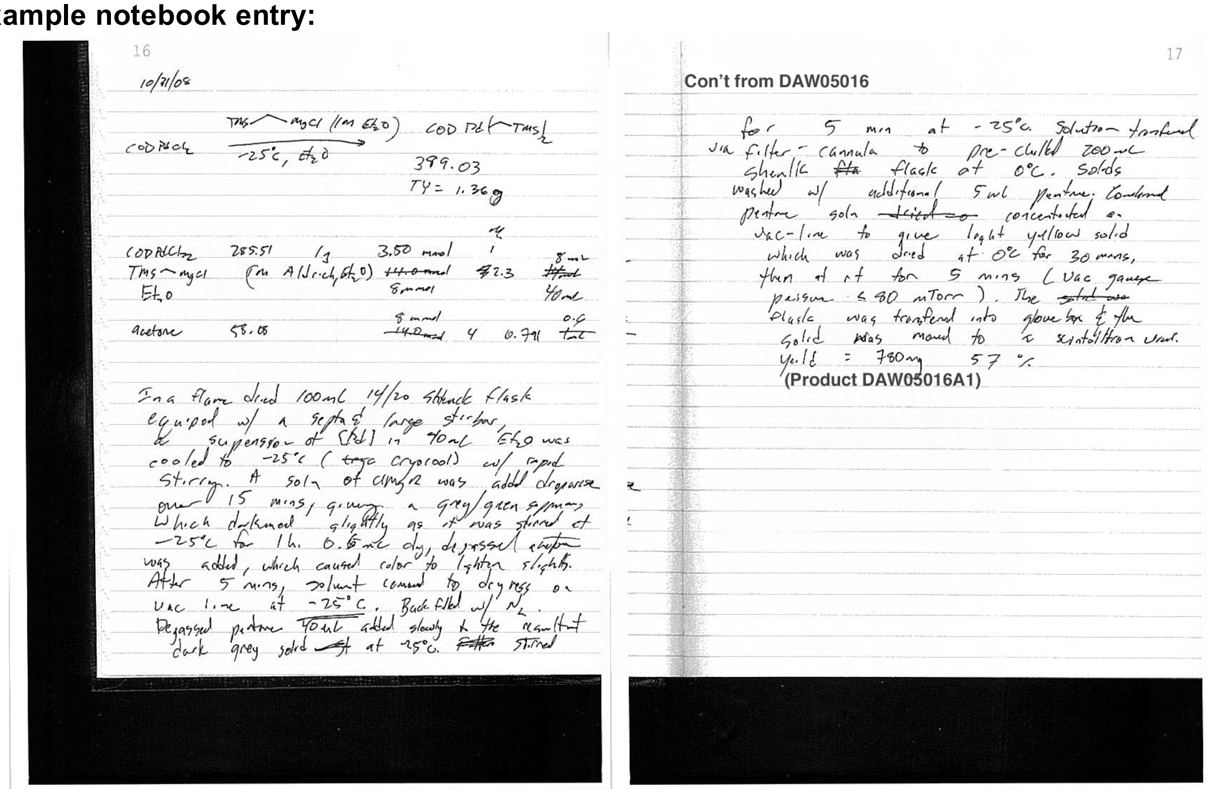
Data Recording and Notebooks

Good record keeping and notebook management is essential to your success in this group. Organized data and meticulous note keeping not only ensures that experiments can be repeated (by yourself and others), but also is indispensable when it come to preparing publications, scrutinizing scientific conclusions and ensuring that experiments need not be repeated unnecessarily. In addition, a properly keep notebook is an important record of when experiments where conceived and executed and is crucial should any of your results be subject to patenting or other IP protection. <u>In short, maintaining a good notebook is expected and required of all co-workers in this group.</u> Periodic "Notebook Checks" will be preformed by DAW to ensure that this expectation is being meet. Be aware, these may not always be announced beforehand.

Notebook Essentials.

An example of a typical notebook page is shown below, however, the following must be included.
<u>**Notebooks belong to the DAW Research Group**</u>*.* Do not remove them from lab; they must be accessible at all times.
Date – Each notebook page must be dated.
Use every page – skipping pages waste money (notebooks are expensive), wastes space (blank pages = more notebooks = more required storage space in the lab). Blank pages also present problems with regard to IP protection. If you need more than one page to properly record an experiment, use more than one. Make sure to mark down where the notes continue; write "Continued on page XX" or "Continued from page XX" on the bottom or top of each page – as appropriate – to ensure you can find your notes. While desirable, contiguous pages are not necessary. If you need to add something and the next page in the notebook is used, go to the next empty page.
Reaction Scheme – include a scheme on each page that shows the reaction(s) you are trying to perform. List all variables in the scheme. This should include equivalents of reagents, temperature, time, etc.
A table of reagents is required for every reaction. This should include: compound name, molecular weight, source or supplier for each chemical (if it was prepared in the group or is otherwise not commercial, include a notebook page; if a chemical was purified before use, state how); mass added, equivalents of each reagent, volume and density (if liquid). ALL REAGENTS, CHEMICALS, CATALYST, SOLVENTS, ETC MUST BE INCLUDED.
Detailed experimental notes must be recorded. The notes are critical and should include details such as where the experiment was setup (bench or glovebox), what size flask, if the flask was dry (if so, how was it dried?), if a stirbar was used, if the reaction was sealed, if the reaction was run under inert-atmosphere (if so, how?), what reagents were added AND IN WHAT ORDER, how reagents were added (was the solid weighed in, was it added via syringe, etc?), how long the reaction was stirred and at what temperature, how the reaction was worked up, how the product was assayed and purified, what the yield or result of the reaction was, any additional notes, conclusions or observations - this can be the color of the reaction, did it exotherm, did it stop stirring, does the experiment need to be changed, did something go wrong such that it should not be repeated, were there precipitates, did we learn anything in particular from the experiment? Any observations that you note about the reaction should be recorded. You never know what seemingly unimportant details might prove crucial later. **Include the yield.**
Page numbers and notebook numbers will be used to uniquely ID each reaction. The first experiment on the page should be numbered "A". **Our convention will be [Initials][two digit Notebook Number][3 digit Page number][Reaction letter].** For example DAW02095A corresponds to reaction A in Don's second notebook (02), experiment page 095.

Multiple nested experiments. It is acceptable to nest several experiments on one page, if and only if, the experiments will be examining a range of a single variable (for example, the same procedure is used to examine 5 different solvents). In this case, each experiment should be marked is a capital number letter after the number above. For example DAW04178B, would be the second experiment (B) on page 178 of Don's 4^{th} notebook. Each such experiment must be clearly labeled and the changed variable must be clearly noted in the experimental notes. If this can not be clearly done, use multiple pages instead. Do not set up so many experiments that you need to use double letters or other characters (ie, do not go to AA or α), use another page.
Spectra Labeling. Each time a spectrum or chromatogram collected during the course of the reaction or a compound is isolated, a new number should be used, and it should be noted in the experimental notes. For example, DAW04178B1 would denote the first compound isolated from experiment B on the given page. Please use "crd" for spectra from crude reaction mixtures (ie DAW04178Bcrd, would be the crude material from second experiment, on page 178 of Don's 4^{th} notebook). Note, the same numbering scheme should be used for the same batch sample of material (ie, the ^{1}H, ^{13}C and GC from first sample you isolate form a reaction should all have the same letter. If you later re-purify that sample, for example, then a new letter should be used and recorded in the notebook. In this way, we can keep track of samples of the same purity and that have been processed similarly.)
Notebook Index. An index of reactions is required. Our notebooks provide space for this at the beginning of each book. Please keep your index up to date.
References. If a literature procedure (or modification thereof) was used to prepare a compound, list it below your table of reagents. If you are repeating on of your own procedures, reference that as well.
Archiving Data. Binders and protector sheets will be provided to you. Please keep a copy of all data (NMR, GC, UV, IR, etc) that you collect in these binders. Collect data from each experiment or page of experiments in a single protector sheet (use multiple if needed) and organize them by notebook and experiment number in the binders. KEEP THIS UP TO DATE.
Example notebook entry:



Determination of Yield by GC: GC Calibration

Except in unusual circumstances, whenever a reaction is being monitored by GC, the yield and conversion of the reaction should be determined using an internal GC standard using the following procedures:

For an “In situ” Internal Standard: A known molar ratio of an appropriate (non-reactive) GC standard is added to the reaction mixture along with starting materials. Usually a fractional stoichiometric quantity is added (1 equiv., 0.5 equiv., or 0.25 equiv. or something similar). This method had the advantage that a solution of starting materials and internal standard and be pre-made and then added to a reaction volumetrically. Analysis of excess of this sample allows for highly accurate calibration of the yield, by pre-determining the exact ratio of internal standard and product. The disadvantage of this method is that you are forced to assume that the internal standard does not interfere with the reaction – which is not always a good assumption (in addition to chemical reactivity – internal standards can alter solvent polarity or other parameters of the reaction).

For an “Ex Situ” Internal Standard: A known amount of an appropriate GC standard is added to a reaction AFTER THE REACTION IS COMPLETED AND PRIOR TO ANY WORKUP. Again, a fractional stoichiometric quantity is best to use. This method, while slightly less accurate, is the preferred method for GC internal standards as it avoids the reactivity issues noted above. **In general and unless otherwise stated, GC “Internal Standard” will refer to this method in the DAW group**.

With either type of internal standard, the GC response factors must be calibrated.

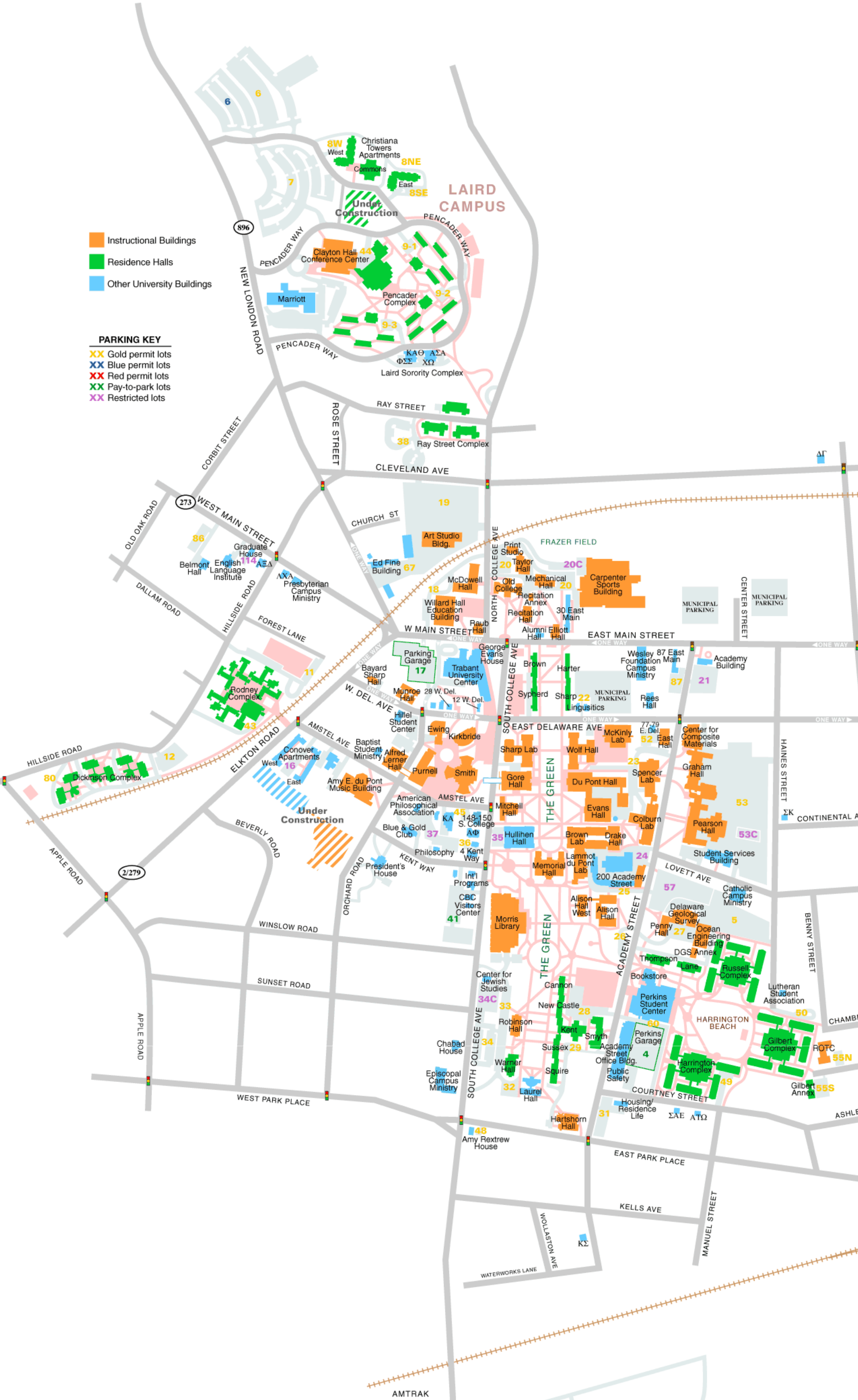
Group Procedure for Calculating Correction Factors for GC Internal Standards

1) Identify each internal standard (ISTD), product, byproduct, and starting material (SM) peak in the GC by independently injecting a sample of each pure material.
2) Make a solution of one analyte (product, byproduct or SM) and in the internal standard in a low boiling solvent (CH_2Cl_2 or EtOAc are usually good choices). It is important that you WEIGH the amounts of analyte and internal standard for this, even if they are liquids. Dilute to concentration appropriate for GC.
3) Repeat step two for all analytes.
4) Run a GC of each solution UNDER THE SAME CONDITIONS (method, GC etc) as your reaction analysis.
4) Calculate moles of each compound (analyte and standard) that were use to make the solution.
5) With each GC trace from step 4, divide the peak area for each compound by the amount number of moles used in the solution to get a response factor R (in area/mol) for each compound.
6) Correction (C) factor ratio by dividing $R_{analyte}/R_{ISTD}$. This new unitless number will be the correction factor for the internal standard for that particular analyte/method combination.

Group Procedure for Calculating GC Yield Using Internal Standard.

1) For each analyte in the GC from your reaction and using the appropriate correction factor “C” from, multiply the area of the internal standard peak by the correction factor C. This number (A) will be equal to the expected area for the analyte on that GC run if you would expect a 1:1 stoichiometric ratio for that product compared to the amount of ISTD used. You may have to correct for stoichiometry at this point.
2) To get yield, divide the area of the $Area_{analyte}/A$.
3) Repeat 1 and 2 for each analyte in the GC trace.

University of Delaware Campus Map



University of Delaware Campus Map

Handling Pyrophoric Reagents

revised 6/95

Fig. 1 Pyrophoric reagents may be packed in a variety of containers.

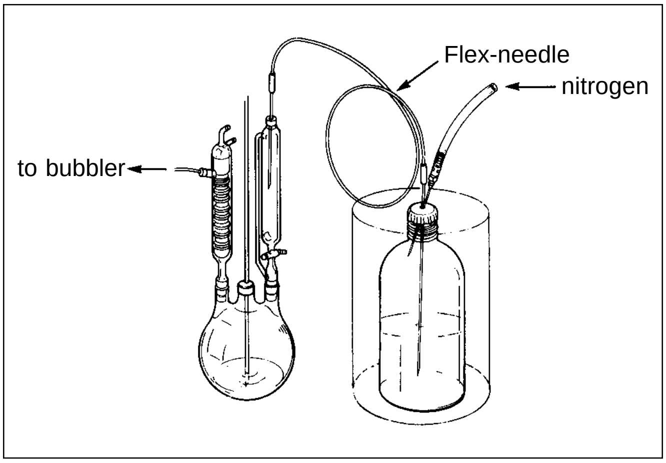


Fig. 2 Double-tipped needle transfer of pyrophoric liquid.

Fig. 3

Fig. 4

NOTE: The metal can in which each bottle is shipped should be retained as a protective container for transporting and storing the bottle of reagent.

I. INTRODUCTION AND PRECAUTION

Due to the hazardous nature of pyrophoric reagents, we strongly recommend that all users read this bulletin carefully and completely before starting any actual laboratory work. If you are unsure of any of these procedures or if you need assistance, please contact us prior to use.

All users of these reagents must be fully qualified and experienced laboratory workers to handle pyrophoric reagents without problems. All users must be made aware of the very hazardous nature of these products.

Users must have read and understood our Technical Information Bulletin No. AL-134 which describes standard syringe and double-tipped-needle transfer techniques before attempting to handle liquid pyrophoric reagents (see Fig. 2).

II. NATURE OF THE REAGENTS

Pyrophoric reagents are extremely reactive toward oxygen and in most cases, water, and must never be exposed to the atmosphere. Failure to follow proper handling techniques could result in serious injury. Exposure of these reagents to air could result in spontaneous combustion, which could cause serious burns or other injuries to the person handling the reagent or others in the immediate area.

In addition, all combustible materials, including paper products, should not be allowed to come in contact with any pyrophoric reagent at any time.

III. HANDLING

Pyrophoric reagents can be handled and stored safely as long as all exposure to atmospheric oxygen and moisture is avoided. Solids must be transferred under an inert atmosphere in an efficient glove box. Liquids may be safely transferred without the use of a glove box by employing techniques and equipment discussed in our Technical Information Bulletin AL-134.

Again, users must have read and understood the accompanying Technical Bulletin AL-134 (call us immediately for a copy if yours has been misplaced), before attempting to handle liquid pyrophoric reagents.

Glass bottles of pyrophoric reagents should not be handled or stored unprotected. The metal can shipped with each bottle should be retained as a protective container for each bottle for transporting and storage (see Fig. 3 and 4).

(OVER)

IV. SPILL

Powdered lime should be used to completely smother and cover any spill that occurs.

A container of powdered lime should be kept within arm's length when working with a pyrophoric material.

IV. DISPOSAL

We feel that the user of the reagent is the person most familiar with the contents and should accept the responsibility for safe disposal of the empty container.

A container with any residual material **MUST NEVER** be opened to the atmosphere. The last traces of reagent must be removed and should be used completely for a chemical reaction; however, if unused and unwanted material must be destroyed, it must be transferred to an appropriate reaction flask for hydrolysis and/or neutralization with adequate cooling.

The essentially empty container is then rinsed three times with an inert dry solvent; this rinse solvent must also be neutralized or hydrolyzed. The solvent must be added to and removed from the container under an inert atmosphere. After adding each rinse, the container is swirled or shaken. The best solvent to use is the same solvent used for the solution of the original reagent. If the container originally contained a **neat reagent, then use a solvent which is completely inert and unreactive toward the reagent.**

After the triple rinse is complete, the container is opened to the atmosphere at a safe location, preferably outdoors or, AT A MINIMUM, IN THE BACK OF A HOOD. After allowing the container to be exposed to the atmosphere for at least a week, the container must be triple-rinsed with water before disposal.

This hazard sheet must remain with the container at all times. If you have any questions, please contact us.

Aldrich Chemical Company, Inc.

1001 West Saint Paul Ave., Milwaukee, WI 53233

Telephone 414-273-3850 / 800-231-8327 — Fax 414-273-4979 / 800-962-9591 — Internet INFO@ALDRICH.COM — TWX 910-262-3052

Technical Bulletin
AL-134

Handling air-sensitive reagents

revised 3/97
8 pages

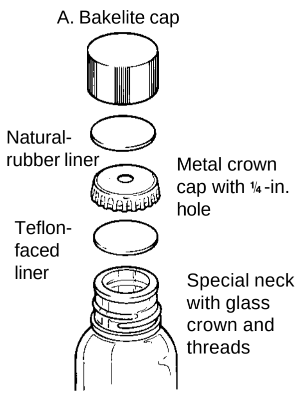


Fig.1.
The Sure/Seal Bottle System

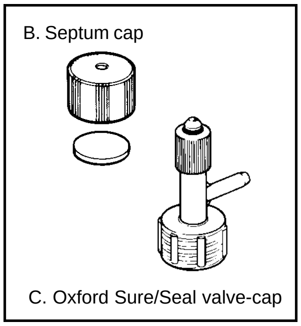
A variety of air-sensitive reagents is available from Aldrich. Specific examples include solutions of borane complexes, organoboranes, borohydrides, Grignard reagents, and organo-aluminums, -lithiums, and -zincs. Since all of these reagents react with water, oxygen, or both, they must never be exposed to the atmosphere.

Most synthetic chemists are familiar with these versatile organometallic reagents. However, because the compounds are air-sensitive or pyrophoric, some workers hesitate to make use of the remarkable chemistry of these reagents. Some chemists still believe that very specialized equipment and complicated techniques are required for handling air-sensitive reagents. This is often not the case.

Air-sensitive materials can be separated into two categories: those which react catalytically with air and/or water, and those which react stoichiometrically. In the latter case- which fortunately includes most of the synthetically useful reagents- the reagents can be handled easily on a laboratory scale using syringe techniques. The catalytically sensitive materials often require the use of more sophisticated apparatus such as vacuum lines, Schlenk apparatus, or inert-atmosphere glove boxes.

Brown and co-workers have described simple, convenient benchtop methods for handling stoichiometrically sensitive compounds on a laboratory scale.[1] Shriver and Drezdzon have presented an excellent description of the more sophisticated techniques used to manipulate catalytically sensitive materials.[2] This data sheet is limited to those techniques necessary for handling air-sensitive reagents on a preparative laboratory scale.

Contents



The Aldrich Sure/Seal™ packaging system

Air-sensitive reagents from Aldrich are packaged in special bottles, normally 800ml in the 32-oz Sure/Seal bottle and 100ml in the 4-oz Sure/Seal bottle. Our exclusive packaging system (Fig. 1) provides a convenient method for storing and dispensing research quantities of air-sensitive reagents. With this bottle, reactive materials can be handled and stored without exposure to atmospheric moisture or oxygen. The reagent comes in contact only with glass and Teflon®, yet it can be readily transferred using standard syringe techniques.

The Bakelite cap on a Sure/Seal bottle can be safely removed because the crown cap, with its Teflon/elastomer liner, is already crimped in place. The reagent can then be dispensed using a syringe or double-tipped needle inserted through the hole in the metal cap. We recommend only small-gauge needles (no larger than 16-gauge) be used and that the Bakelite cap be replaced after each use. After the needle has been withdrawn from the bottle, a small hole will remain in the Teflon/elastomer liner. Under normal circumstances, the hole in the liner will self-seal and the reagent will not deteriorate. However, the possibility exists that once an elastomer liner is punctured, it may leak on long-term storage. This possibility is virtually eliminated with the Sure/Seal system because when the Bakelite cap is replaced, the Teflon/elastomer liner in the cap forms a seal against the top of the metal crown. Thus, the contents are protected from moisture and oxygen in the atmosphere.

A more convenient solution is to use our Sure/Seal septum cap (Fig. 1B). After removing the solid Bakelite cap, a septum cap (a Bakelite cap with a 3/16 in. hole equipped with an elastomer liner) is placed securely on the bottle. The liner is made from white natural rubber, the same material as in our sleeve stoppers. With the septum cap in place, the needle is inserted into the hole in the Bakelite cap and through the septum-cap liner and the crown-cap liner. The Sure/Seal septum cap protects the reagent and works nicely if the reagent is to be used repeatedly over a relatively short period of time (no more than 2 or 3 days).

If an unused portion is to be stored for an extended length of time, the solid Bakelite cap should be replaced on the bottle. For added security, transfer the reagent to a suitable storage vessel or equip the bottle with the Oxford Sure/Seal Valve Cap (Fig. 1C).[3]

Equipment

Reactions involving our air-sensitive reagents can be carried out in common ground-glass apparatus. Other equipment required are a source of inert gas, a septum inlet, a bubbler, and syringes fitted with suitable needles.

Laboratory glassware contains a thin film of adsorbed moisture which can be easily removed by heating in an oven (125°/overnight or 140°/4 hrs). The hot glassware should be cooled in an inert atmosphere by assembling the glassware while hot and flushing with a stream of dry nitrogen or argon. A thin film of silicone or hydrocarbon grease must be used on all standard-taper joints to prevent seizure upon cooling. Alternatively, the apparatus may be assembled cold and then warmed with a heat gun while flushing with dry nitrogen.

The oven-drying procedure is more efficient than using a heat gun because it removes moisture from inner surfaces of condensers and from other intricate parts. Spring clips or rubber bands are required to secure joints during flushing since the nitrogen pressure may open the seals of unsecured standard-taper joints.

Only high-purity, dry nitrogen from a cylinder with a pressure regulator (adjusted to 3-5 psi) should be used for flushing. Plastic tubing can be used to connect the nitrogen line to a tube connector adapter (equipped with a stopcock) on the reaction apparatus. Nitrogen may also be introduced through a rubber septum via a hypodermic needle connected to the end of the flexible tubing on the nitrogen line. The needle-tubing connector provides a simple method for attaching the needle to the tubing. When not in use, this nitrogen-flushing needle should be closed by inserting the needle into a solid rubber stopper to prevent diffusion of air into the needle when the nitrogen is turned off (Fig. 2).

Large rubber septa may be used to cap female joints. However, the use of 6-mm septa and 9-mm o.d./6-mm i.d. medium-wall glass septum inlets is preferred. The small rubber septum provides a more positive reseal after puncture and allows less rubber to be in contact with organic vapors in the reaction vessel. With the recommended medium-wall tubing, the 6-mm septum not only fits the inside diameter of the glass tube but also fits snugly over the outside when the top is folded over (Fig. 3). The glass septum inlet can be built into the reaction flask (Fig. 4) or placed on an adapter (Fig. 5) for use with unmodified glassware.

The rubber septum may be wired in place as shown in Fig. 3. However, if the 6-mm septum is properly fitted to 9-mm medium-wall tubing, the wiring step may be omitted unless high pressures (>10 psi) are expected.

To maintain an air-tight system the reaction vessel must be vented through a mercury or mineral oil bubbler. Drying tubes will not prevent oxygen from entering the system. At all times during the reaction, the system should be under a slight positive pressure of nitrogen as visually indicated by the bubbler. Fig. 6 illustrates a suitable bubbler.

A pressure reversal may cause the liquid in the bubbler to be drawn into the reaction vessel. The enlarged head space in the bubbler will minimize this danger. However, if a large pressure reversal occurs, air will be admitted into the reaction vessel. The T-tube bubbler shown can be used to prevent this problem because nitrogen pressure can be introduced intermittently through the septum inlet. The problem can be completely eliminated by a slow and continuous nitrogen flow.

Small quantities (up to 50ml) of air-sensitive reagents and dry solvents may be transferred with a syringe equipped with a 1-2ft long needle. These needles are used to avoid having to tip reagent bottles and storage flasks. Tipping often causes the liquid to come in contact with the septum causing swelling and deterioration of the septa, and should therefore be avoided.

A rubber septum provides a positive seal for only a limited number of punctures-depending on needle size. Therefore, always reinsert the needle through the existing hole. It is also advantageous to put a layer of silicone or hydrocarbon

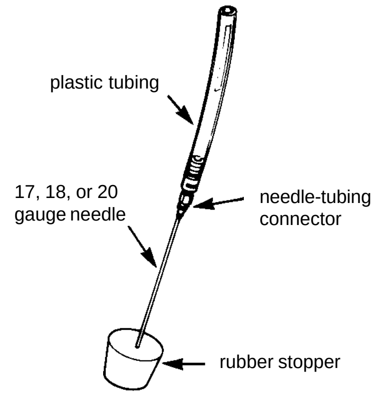


Fig. 2. Nitrogen-flushing needle

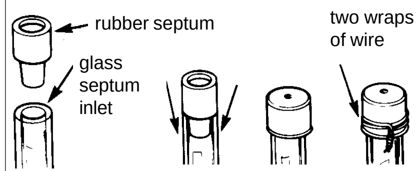


Fig. 3. Use of septum inlet

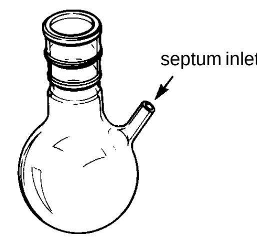


Fig. 4. Flask with septum inlet

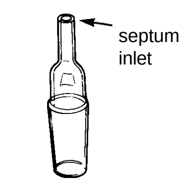


Fig. 5. Septum inlet adapter

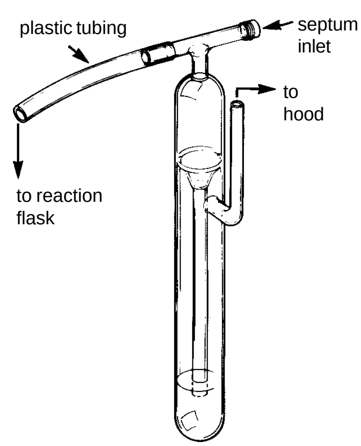


Fig. 6. Bubbler

grease on a rubber septum to facilitate passage of the needle through the rubber and to minimize the size of the hole in the septum. Ideally, the syringe and needle should be dried in an oven prior to use. Naturally, the syringe body and plunger should not be assembled before being placed in the oven. The syringe should be flushed with nitrogen during the cooling. A syringe may also be flushed 10 or more times with dry nitrogen (illustrated in Fig. 7) to remove the air and most of the water adsorbed on the glass. A dry syringe may be closed to the atmosphere by inserting the tip of the needle into a rubber stopper (Fig 2).

The syringe-needle assembly should be tested for leaks prior to use. The syringe is half-filled with nitrogen and the needle tip is inserted in a rubber stopper. It should be possible to compress the gas to half its original volume without any evidence of a leak. A *small* amount of stopcock grease or a drop of silicon oil placed on the Luer lock tip will help ensure tightness.

Reagent transfer with syringe

The syringe transfer of liquid reagents (up to 100ml) is readily accomplished by first pressurizing the Sure/Seal reagent bottle with dry, high-purity nitrogen followed by filling the syringe as illustrated in Fig. 8. The nitrogen pressure is used to slowly fill the syringe with the desired volume plus a slight excess (to compensate for gas bubbles) of the reagent. Note that the nitrogen pressure pushes the plunger back as the reagent enters the syringe. The plunger should not be pulled back since this tends to cause leaks and create gas bubbles. The excess reagent along with any gas bubbles is forced back into the reagent bottle as illustrated in Fig. 9. The accurately measured volume of reagent in the syringe is quickly transferred to the reaction apparatus by puncturing a rubber septum on the reaction flask or addition funnel as shown in Fig. 10. Note: larger syringes are available but are awkward to handle when completely full.

Reagent transfer with a double-tipped needle

To conveniently transfer 50ml or more of reagent, the double-tipped needle technique is recommended. Fig. 11 illustrates liquid-reagent transfer under nitrogen pressure using this technique.

To accomplish the double-tipped needle transfer, the needle is first flushed with nitrogen. The Sure/Seal bottle is pressurized with nitrogen using the nitrogen flushing needle. The double tipped needle is then inserted through the septum on the reagent bottle into the head space above the reagent. Nitrogen immediately passes through the needle. Finally, the other end of the double-tipped needle is inserted through the septum on the reaction apparatus, and the end of the needle in the reagent bottle is pushed down into the liquid. The volume of liquid reagent transferred is measured by using a calibrated flask or addition funnel. When the desired volume has been transferred, the needle is immediately withdrawn to the head space above the liquid, flushed slightly with nitrogen, and removed. The needle is first removed from the reaction apparatus and then from the reagent bottle.

Fig. 7. Flushing a syringe with nitrogen

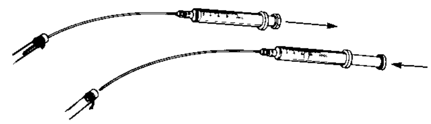


Fig. 8. Filling syringe using nitrogen pressure

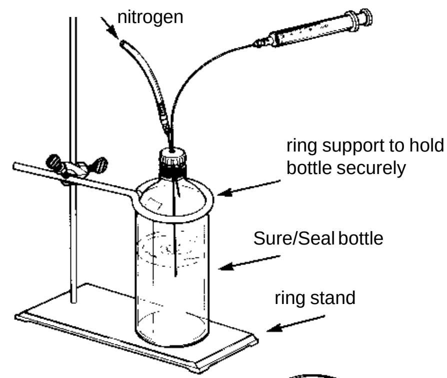


Fig. 9. Removing gas bubbles and returning excess reagent to the Sure/Seal bottle

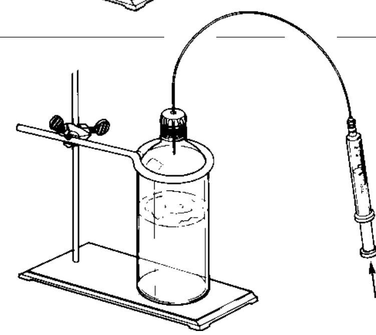


Fig. 10. Syringe transfer of reagent to reaction vessel

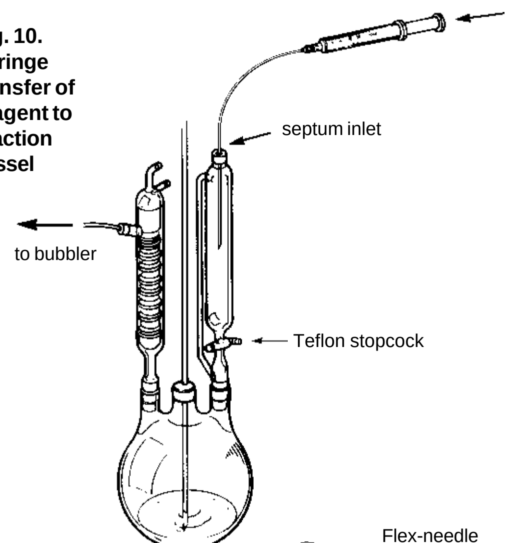


Fig. 11. Double-tipped needle transfer of liquid reagent

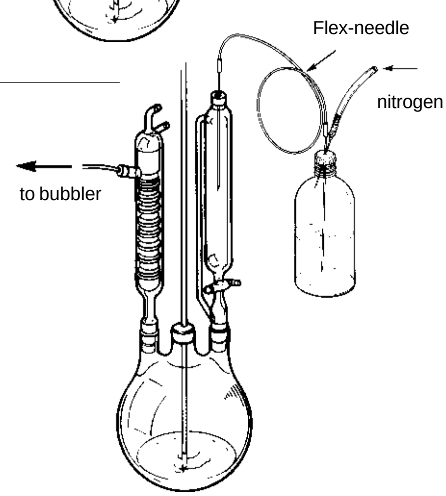


Fig. 12.
Double-tipped needle transfer to graduated cylinder

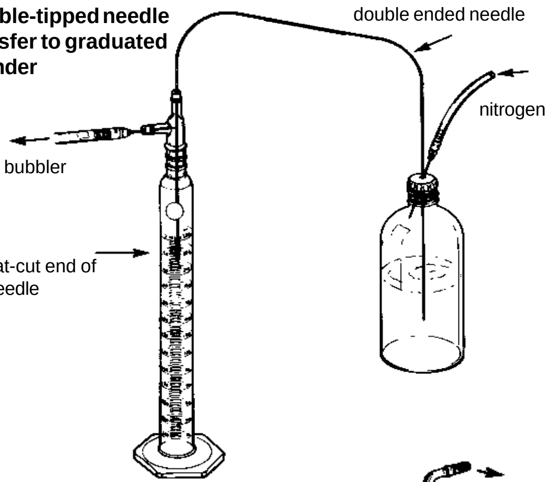


Fig. 13.
Double-ended needle transfer with syringe valve

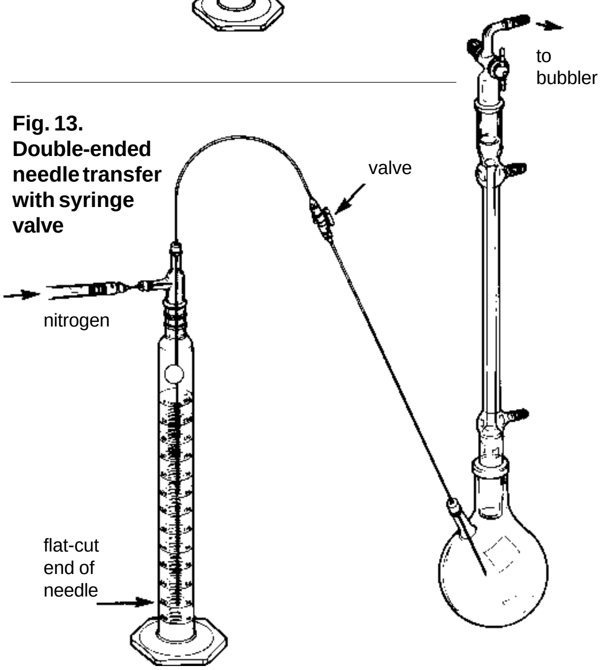


Fig. 14.
Storage bottle equipped with Teflon stopcock

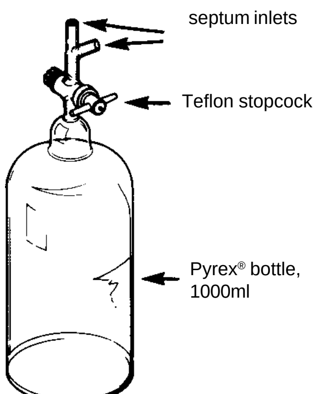
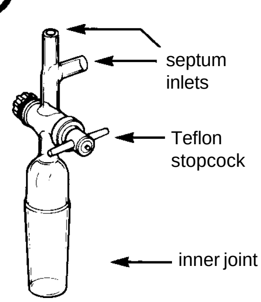


Fig. 15.
Septum inlet adapter for storage flask



An alternative method for transferring measured amounts of reagents is shown in Fig. 12. The reagent is first transferred *via* a double-ended needle from the Sure/Seal bottle to a dry, nitrogen-flushed graduated cylinder (see Fig. 13) equipped with female ₮ joint and a double inlet adapter. Only the desired amount of reagent is transferred to the cylinder. The needle is then removed from the Sure/Seal bottle and inserted through the septum on the reaction apparatus. By applying nitrogen pressure as before, the reagent is added to the reaction apparatus. If it is necessary to add the reagent slowly, a modified transfer needle is constructed from two long standard needles and a male Luer lock to male Luer lock syringe valve. The valve may be opened slightly allowing only a very slow flow of reagent. Thus, the addition funnel is not needed and many reactions can be carried out in single-necked flasks as shown in Fig. 13.

Storage vessels

The 12-gauge stainless steel needles on the Flex-needle provide a rapid means of transferring air-sensitive reagents under nitrogen pressure. However, the needles are so large that once the crown cap liner on the Sure/Seal bottle is punctured, the liner will not self-seal. If only a portion of the contents is to be used, a needle no larger than 16-gauge should be utilized. By using small needles and by always tightly replacing the Bakelite cap, the reagent in a Sure/Seal bottle will not deteriorate even after numerous septum punctures.

However, if the reagent is to be used repeatedly for small-scale reactions or if an unused portion is to be stored for an extended length of time, the material should be transferred from the Sure/Seal bottle to a suitable storage bottle. One type of container for air-sensitive reagents is shown in Fig. 14. Alternatively, an appropriate adapter can be used to convert a round-bottomed flask into a storage vessel (Fig. 15).

The Teflon stopcock on the storage bottle keeps solvent vapors away from the septum, thereby minimizing swelling and deterioration of the septum. Furthermore, the stopcock allows for replacement of the septa. A change of septa is sometimes necessary because they tend to deteriorate on prolonged standing in a laboratory atmosphere.

Cold storage

A problem arises with cold storage in vessels equipped with Teflon stopcocks. Since the thermal expansion coefficient of Teflon is significantly different from that of glass, we have found that special techniques are required when Teflon-plug/glass-barrel stopcocks are used or stored in a coldroom. The Teflon plug contracts more than the glass barrel on cooling, thus, the stopcock can give a good seal at room temperature but leak when moved to a coldroom. Conversely, the stopcock can be tightened in the coldroom giving a good seal, but upon warming to room temperature the Teflon expands, freezing or breaking the stopcock. The simplest solution to this problem is to retighten the stopcock after the apparatus has cooled for about 15 minutes in the coldroom. Thereafter, open and close the stopcock only in the coldroom- *do not attempt to turn the stopcock after it has warmed to room temperature.*

Teflon will cold flow (creep) with time. Therefore, unattended long-term storage of a tightened Teflon stopcock is not recommended. The stopcock should be turned occassionaly (at least once a month) to check for tightness of its seal, regardless of where it is stored.

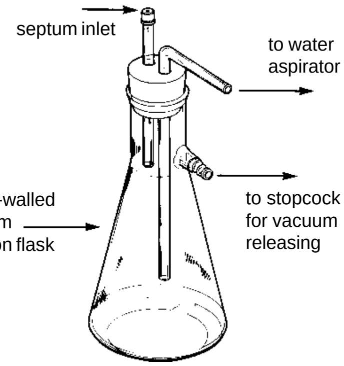


Fig. 16. Needle cleaning and drying technique

Storage or use of Teflon-stopcock-equipped apparatus in a freezer (-20°C or below) presents special problems. If a Teflon stopcock is tightened in a freezer and then allowed to warm to room temperature unattended, the expanding Teflon can break the glass barrel of the stopcock. To prevent this loosen the Teflon plug while turning it as the apparatus warms. This process can be accelerated by warming the glass barrel with the hand. All of the above problems can be avoided by using only all-glass stopcocks whenever an apparatus is to be stored in a coldroom.

A lubricant, such as a silicone or hydrocarbon grease, is required for glass stopcocks. Obviously, the solvents used for our reagents will slowly dissolve most stopcock greases. In cases where this can become a problem, it is advisable to substitute Teflon-clad stopcocks which combine the best of both systems. However, these special Teflon-coated glass plugs are expensive, and once the Teflon is worn, the plug must be discarded.

A better solution to the lubricant problem is to wrap the glass plug with a Teflon tape. The tape must not be overlapped, but wrapped around in one layer and in one continuous spiral with no gaps. The plug should be turned only in one direction to further tighten the tape (*e.g.,* if the tape is wrapped counterclockwise, turn the plug clockwise only). If the Teflon-wrapped plug is held securely in place with a rubber band, a storage vessel can be stored for months in a coldroom or at room temperature without any leakage or freezing of the stopcock.

Equipment cleanup

Clean-up of equipment that has been used to transfer air-sensitive reagents must not be taken lightly. Since many of these reagents react violently with water, fires are a potential hazard. The crown cap and liner of an empty Sure/Seal bottle should be carefully removed and the open bottle left in the hood to allow the last traces of reactive reagent to be slowly air-hydrolyzed and oxidized. After at least a day, the inorganic residue can be rinsed out with water. Empty storage bottles and storage flasks should be treated similarly. Air-hydrolysis in a hood is appropriate only for the last traces of material that remain after a Sure/Seal bottle has been emptied as completely as possible *via* syringe or double-ended needle transfer. The Aldrich Catalog/Handbook or material safety data sheets should be consulted for the recommended disposal procedures for larger amounts of reactive chemicals.

Immediately clean all syringes and needles that have been used to transfer air-sensitive materials. Also, in general, a syringe should only be used for a single transfer. Failure to follow this practice can result in plugged needles and frozen syringes due to hydrolysis or oxidation of the reagents. The double-tipped needles are flushed free of reagent with nitrogen in the transfer system, and then immediately removed and placed in a clean sink. With water running in the sink and in the complete absence of flammable solvents and vapors, the double-tipped needles or Flex-needle can be rinsed with water. When no activity in the rinse water is observed, acetone from a squeeze bottle can be flushed through the needle. Depending on the reagent transferred, it may be necessary to use dilute acid or base from a squeeze bottle to remove inorganic residue that is not water-soluble.

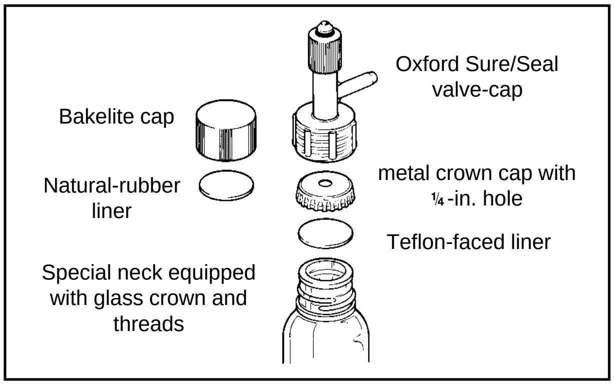
Following its use, a syringe contains a larger amount of residual reagent. It is advisable to rinse out the reactive reagent by first placing a few millimeters of the same solvent that was used for the reagent in a small Erlenmeyer flask in the hood. Keeping the needle tip under the solvent at all times, no more than half the solvent is then drawn into the syringe. The solvent plus dissolved residual reagent is ejected from the syringe back into the same Erlenmeyer flask. Repeat this rinse treatment at least three times. The wash solution can be safely combined with other waste solvents and the syringe may be further cleaned with water and acetone in the sink. Again, treatment with dilute aqueous acid or base may be necessary.

Once the syringe needles and double-tipped needles have been rinsed in a sink, they can be further cleaned and dried using a device similar to that shown in Fig. 16. Needles are cleaned by inserting them through the septum. Vacuum from a water aspirator is used to pull solvents from squeeze bottles through the needles. After pulling air through the system for a few minutes, the syringe plus needle or the double-tipped needle will be dry. The syringe plunger should be replaced in the barrel for storage. If a syringe plunger and barrel are not assembled for storage, dust can settle on the plunger and in the barrel. Upon reassembly, these fine particles will occasionally scratch the barrel or cause seizure of the plunger on the barrel. However, the plunger and barrel must be disassembled before oven drying.

Most of the above techniques were developed for handling various organoborane reagents. However, these methods are applicable to other air-sensitive materials. When handling air-sensitive materials, be prepared for the unexpected. For example, at least one extra set of clean, dry syringes and needles or double-tipped needles should always be available in case the first set of equipment becomes plugged. When working with these air-sensitive reagents keep in mind that these solutions should never be allowed to come in contact with the atmosphere.

Aldrich hopes that by supplying the Sure/Seal packaging system and all the equipment required for syringe-transfer of liquids, chemists will no longer hesitate to use air-sensitive reagents. Our aim at Aldrich is the customers' complete satisfaction. Suggestions for improvement are always welcome.

SURE/SEAL BOTTLE SYSTEM



Sure/Seal bottles

Glass	125ml	**Z11,612-2**
	927ml	**Z11,613-0**

Plastic-coated glass bottles are also available.

Oxford Sure/Seal valve-cap

Screws over Sure/Seal crown cap to permit repeated dispensing of product via syringe while ensuring positive valved closure. Technical Information Bulletin AL-195, with instructions for use of the valve, is included. **Z22,283-6**

Bakelite caps

33-430. Solid tops with liner. **Z10,216-4**
33-430. With 3⁄16-in. hole. **Z10,807-3**

Natural Rubber liner

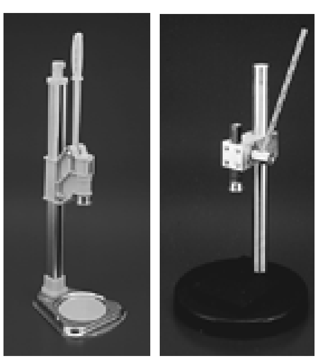
White, 30-mm diameter, 60mil. **Z10,808-1**

Steel crown cap

With ¼-in. hole. **Z10,214-8**

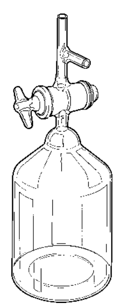
Teflon-faced liner

For crown cap, 25-mm diam. **Z10,215-6**



Crown-cap crimpers

17¼ in. high, 4-in. diam. base, 2in. from crown to pole. (left)
Z11,296-8
Replacement rubber washers.
Z15,154-8
Heavy duty. 21½ in. high, 12-in. diam. base, 3½-in. from crown to pole. (right)
Z11,297-6

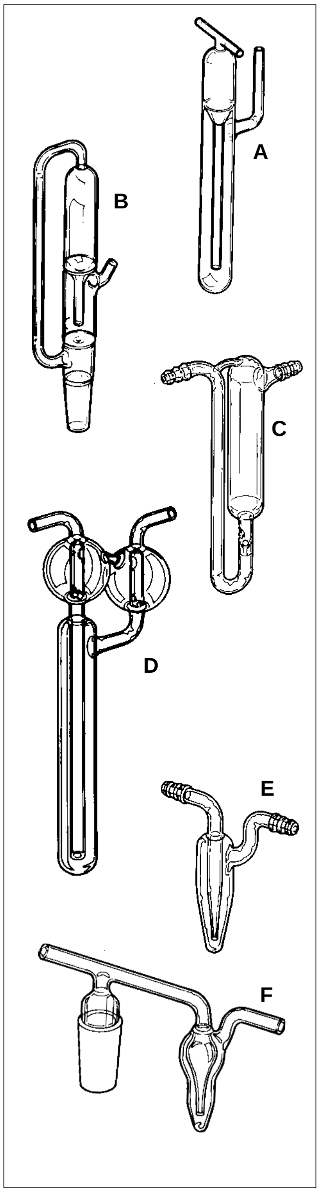


STORAGE BOTTLES

Storage bottles without joints. Clear glass, with stopcock-equipped septum inlet requiring septum Z10,072-2 or Z12,435-4.

Capacity (ml)	Stopcock size (mm)	Cat. No.
Teflon stopcock		
125	2	**Z10,328-4**
250	2	**Z10,329-2**
500	4	**Z10,199-0**
1000	4	**Z10,248-2**
2000	4	**Z10,330-6**
Glass stopcock		
125	2	**Z10,733-6**
250	2	**Z10,734-4**
500	4	**Z10,735-2**

BUBBLERS



Standard

Mineral oil or mercury, 5-7ml. For monitoring gas evolution or rate of flow, or closing off a reaction vessel from the atmosphere. Model (b) has a ₮24/40 joint.

A Z10,121-4
B Z10,432-9

Check-valve bubblers

Permits gas flow under positive pressure. Check-valve ball seats on ground surface under negative pressure preventing oil from being drawn into the purged system.

Single inlet tube, top outlet **C Z22,501-0**

T inlet tube, side outlet **Z22,502-9**

Safety bubbler

The built-in flash arrester bulbs prevents the backflow of mercury and mineral oil to pumps and prevents reactions due to overflow or violent bubbling. 15ml maximum fill mark prevents over-filling.

D Z22,372-7

Mini gas bubbler

For bubble counting. Maximum volume is 4ml.

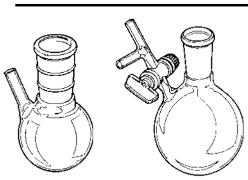
E Z22,371-9

In-line oil bubblers

For precise N_2 pressure control during inert atmosphere reactions. Connect reaction vessel to in-line ₮ joint or use with a ballast bulb to keep pressure constant.

₮14/20 joint	F	**Z22,322-0**
₮19/38 joint		**Z22,334-4**
₮24/40 joint		**Z22,335-2**

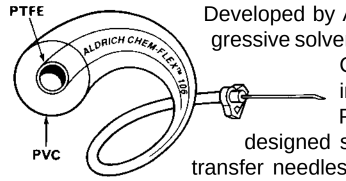
FLASKS



A. Round bottom flask with ground-glass joint and septum inlet.
B. As above but with 2-mm stopcock on septum inlet.

Cap. (ml)	₮ Joint	A Cat. No.	B Cat. No.	Stopcock type
25	14/20	**Z10,217-2**	—	—
50	14/20	**Z10,218-0**	**Z10,725-5**	Glass
50	14/20	—	**Z10,334-9**	Teflon
100	14/20	**Z10,331-4**	**Z10,726-3**	Glass
100	14/20	—	**Z10,335-7**	Teflon
250	14/20	**Z10,332-2**	**Z10,336-5**	Teflon
100	19/22	**Z10,123-0**	**Z10,337-3**	Teflon
250	19/22	**Z10,124-9**	**Z10,338-1**	Teflon
100	24/40	**Z10,125-7**	—	—
250	24/40	**Z10,126-5**	**Z10,729-8**	Glass
250	24/40	—	**Z10,138-9**	Teflon

CHEM-FLEX TUBING



Developed by Aldrich for the safe transfer of aggressive solvents, air-sensitive liquids and gases. Chemflex is constructed of a thin inner-tube of PTFE sheathed in clear PVC. CHEM-*FLEX 106* tubing is designed specifically for use with 12 gauge transfer needles and is suitable for small volume transfers of products packed in Aldrich Sure/Seal bottles.

Features

- chemically inert
- resists crushing and kinking
- available in 25ft & 50ft lengths
- extra strong yet flexible
- cut marks at 6 inch intervals
- color-coded for high visibility

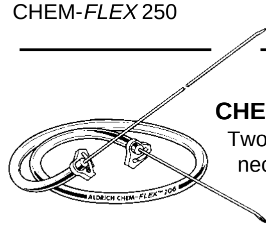
Name/Color-code	I.d. (in.)	O.d.(in.)	Cat. No.
CHEM-*FLEX* 106/Red	0.106	0.380	**Z22,251-8**
CHEM-*FLEX* 125/Blue	0.125 (1/8)	0.400	**Z22,252-6**
CHEM-*FLEX* 187/Green	0.187 (3/16)	0.470	**Z22,253-4**
CHEM-*FLEX* 250/Black	0.250 (1/4)	0.525	**Z22,254-2**

Tubing clamps

Nylon clamps secure CHEM-*FLEX* tubing to transfer needles and fittings.

Tubing size	Diameter range (in.)	Cat. No.
CHEM-*FLEX* 106, 125	23/64 to 25/64	**Z22,417-0**
CHEM-*FLEX* 187	25/64 to 15/32	**Z22,418-9**
CHEM-*FLEX* 250	15/32 to 17/32	**Z22,419-7**

NEEDLES



CHEM*FLEX* *Transfer needle*

Two 12-gauge SS needles (6- and 18-in.) connected to 30-in. of CHEM-*FLEX* 106 tubing with nylon clamps, ready for use. Liquid comes in contact with Teflon and SS only during transfer. **Z23,102-9**

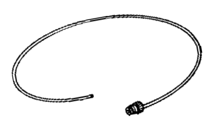
Transfer needles

12 gauge SS, double-ended with one noncoring tip and one flat-cut end. For fabrication of transfer lines with CHEM-FLEX 106 tubing.

6-in. length	**Z11,639-4**
18-in. length	**Z11,640-8**

Teflon syringe needles

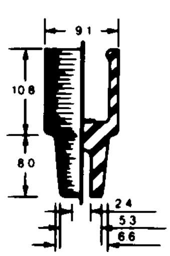
With KEL-F Luer hub.



Gauge	12 in. length Cat. No.	24 in. length Cat. No.
20*	**Z11,731-5**	**Z11,737-4**
18*	**Z11,732-3**	**Z11,738-2**
16*	**Z11,733-1**	**Z11,739-0**
14	**Z11,735-8**	**Z11,740-4**
12	**Z11,736-6**	**Z11,741-2**

* To properly seal the septum around these Teflon needles, first puncture with a SS needle. After threading the Teflon needle through the septa, the SS needle is withdrawn. For 20ga use 14ga SS; 18ga use 13ga SS; 16ga use 12ga SS.

NATURAL RUBBER SEPTA

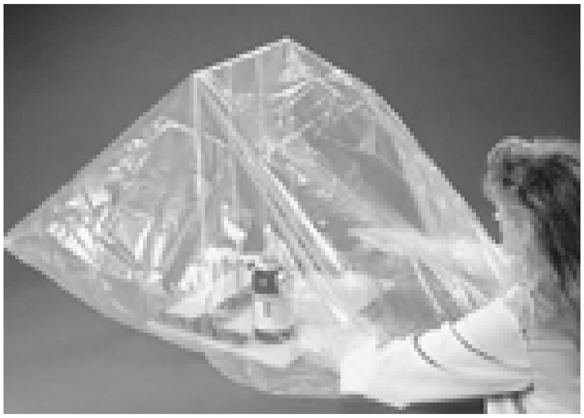


	White Cat. No.	Red Cat. No.
8-mm o.d. wall or 9-mm o.d. wall glass tubing	**Z10,072-2**	**Z12,435-4**
9- and 10-mm o.d. standard-wall glass tubing	**Z10,073-0**	**Z12,436-2**
₮14/20 joints	**Z10,074-9**	**Z12,437-0**
₮19/22 joints*	**Z10,076-5**	**Z11,830-3**
₮24/40 joints	**Z10,145-1**	**Z12,439-7**

*Also fits 125- and 927-ml Sure/Seal bottles.
Cat. No. Z10,072-2 shown

A mixed set of septa with carrying case is also available. Please see the Aldrich Catalog/Handbook for details!

ATMOSBAG — A CONTROLLED - ATMOSPHERE CHAMBER



The Aldrich AtmosBag is a 0.003-in. gauge PE bag that can be sealed, purged, and inflated with an appropriate inert gas, creating a portable, convenient, and inexpensive two handed "glove box" for handling air- and moisture-sensitive as well as toxic materials. Other applications include dust-free operations, controlled-atmosphere habitat, and, for the ethylene-oxide-treated AtmosBag, immunological and microbiological studies. Small AtmosBags have one inlet per side. Includes instructions.

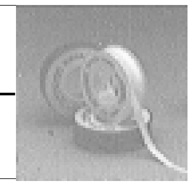
CAUTION: *When handling toxic materials use only in a hood or other controlled system to prevent and protect against exposure in case of leakage. All products made of PE may tear, break, or puncture. To assure that air-sensitive materials do not become exposed to air, follow instructions on package; also test and monitor AtmosBag for leaks before and during use.*

	Uninflated dimensions (in.)			Inflated		Ethylene oxide treated
Size	Opening	Width	Length	volume (in.3)	Cat. No.	Cat. No.
S	12	27	30	3,000 (50L)	**Z11,283-6**	**Z11,837-0**
M	24	39	48	17,000 (280L)	**Z11,282-8**	**Z11,836-2**
L	36	51	58	32,000 (520L)	**Z10,608-9**	**Z11,835-4**

Teflon sealing tape

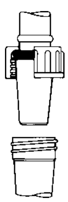
In 520-in. roll.

Width (in.)	Cat. No.
1/4	**Z14,881-4**
1/2	**Z10,438-8**
1	**Z22,188-0**



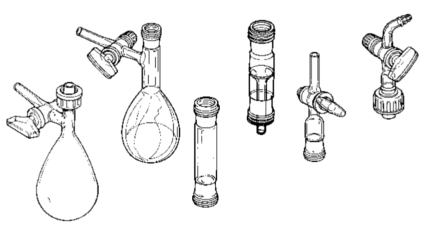
Aldrich Schlenk-type Glassware

- Request Technical Bulletin AL-166

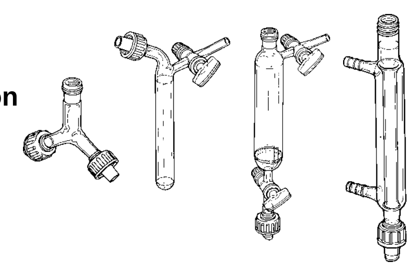


Aldrich Schlenk-type glassware features threaded $\$$ ground glass joints. They require no grease, thus eliminating it as a potential contaminant, and need no cumbersome clamps. The joints consist of a ground glass male joint (**M**) and a ground glass interior female joint (**F**) with exterior threads that allow the male portion to be secured by a septum-type plastic cap using an "O"-ring compression seal. (Cap and "O"-ring are included with all threaded male joints.) Most pieces have stopcock side arms which permit the evacuation of air and the introduction of an inert gas. A high vacuum is not necessary since the purge cycle is repeated a number of times. The versatility of Aldrich Schlenk-type glassware makes the manipulation of air- and moisture-sensitive reagents easier and safer.

Designed for small-scale manipulation of air- and moisture-sensitive reagents



Addition of Liquids • Chemical Reaction
Distillation • Drying • Extraction
Filtration • Recrystallization
Degassing • Transfer of Solids



References

1) Kramer, G.W.; Levy, A.B.; Midland, M.M. in Brown, H.C. "Organic Synthesis via Boranes"; John Wiley and Sons, Inc.: New York, N.Y., 1975 (Aldrich Cat. No. Z10,144-3).
2) Shriver, D.F.; Drezdzon, M.A. "The Manipulation of Air-sensitive Compounds"; John Wiley & Sons: New York, N.Y., 1986 (Aldrich Cat. No. Z16,005-9).
3) See Aldrich Technical Information Bulletin Number AL-195. "Instructions for Using the Oxford Sure/Seal Valve Cap."

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Department of Environmental Health & Safety
Safety Educational Requirements and Recommendations

	Response	Educational Requirement	Frequency
		General Safety	
Are you a new Graduate Student in a Laboratory Science?	No	**New Graduate Student Orientation- Non-Lab**	**Initial**
	Yes	**New Graduate Student Orientation- Lab**	**Initial**
Do you enter or work in Confined Spaces?	Yes	**Confined Space Entry Training**	**Annual**
Will you be using Respiratory Protection?	Yes	**Respirator Training**	**Annual**
Will you be operating a Forklift?	Yes	**Fork Lift Training**	**Initial and Refresher (every 3 yrs)**
Will you generate a hazardous or regulated waste?	Yes	**Chemical/Hazardous Waste Disposal Training**	**Annual**
Is there an AED in your building or do you want to learn CPR?	Yes	Heartsaver AED/CPR Training Healthcare Provided CPR Training	**Biannual**
		Chemical Safety	
Will you be working in a Lab? Addition Guidance available at: http://www.udel.edu/OHS/chptrain/labsafetytrainguide.pdf	No	**Right-to-Know - Basic Training**	**Annual**
	Yes	**Right-to-Know Training** **Chemical Hygiene Plan Training** **Chemical/Hazardous Waste Disposal Training**	**Annual** **Annual** **Annual**
Will you be working with Hydrofluoric Acid?	Yes	**Hydrofluoric Acid Safety Training**	**Annual**
Will you be Transporting or Shipping Dry Ice?	Yes	**DOT Dry Ice Shipping Training**	**Biannual**
Will you be working with Compressed Gasses?	Yes	**Compressed Gas Safety Training**	**Initial**
Using a Fume Hood or Other Laboratory Ventilation?	Yes	Laboratory Ventilation Safety Training	**Initial**
Work with Corrosive Materials?	Yes	Corrosive Materials Safety Training	**Initial**
Working with Highly Toxic or Carcinogenic Materials?	Yes	Highly Toxic Materials Safety Training	**Initial**
Working with Reactive Materials?	Yes	Reactive Materials Safety Training	**Initial**
		Biological Safety	
Will you be working with Biological Materials?	Yes	**Biosafety Training**	**Initial**
Will you be working with Human Samples or Cell Lines?	Yes	**Bloodborne Pathogens Training**	**Annual**
		Radiation Safety	
Will you be working with Radioactive Materials?	Yes	**Radioactive Material Safety Training**	**Annual**
Will you be working with Lasers?	Yes	**Laser Safety Training**	**Initial**
Will you be working with X-ray Producing Devices?	Yes	**X-ray Safety Training**	**Initial**

Bold Red Text Indicates Required Education that must be completed prior to using the materials, working in a laboratory or using a piece of equipment.

These and other educational sessions are available through the Department of Occupational Health & Safety (DOHS). The schedule is available at http://www.udel.edu/ehs/ohstrainsched99.html . Hyperlinked/Underlined classes are also offered through the EHS Online Training Program and can be found at http://ehs.facil.udel.edu:1569/.

Safety education can also be scheduled for your department or group by contacting DOHS.