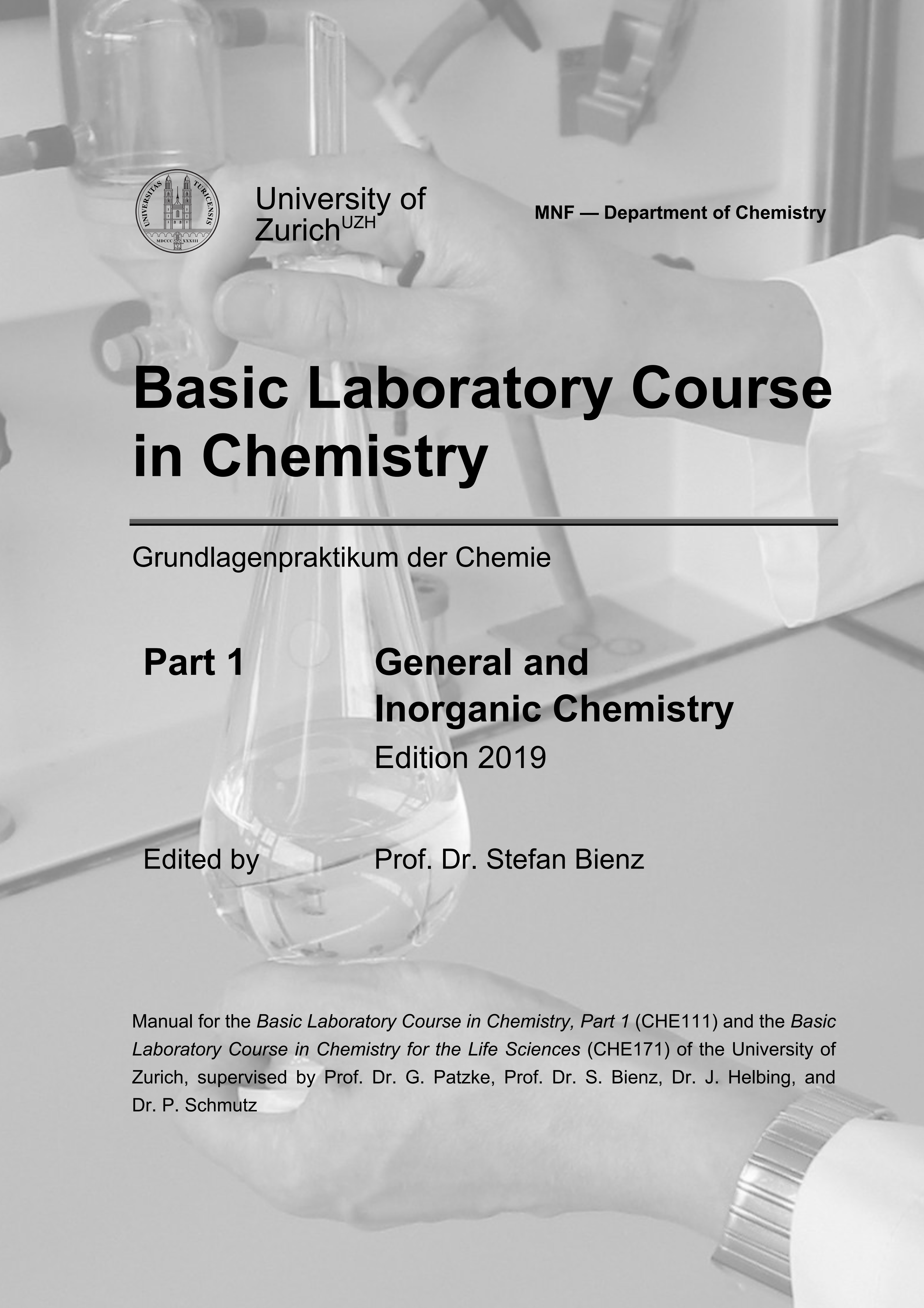


University of
Zurich^{UZH}

MNF — Department of Chemistry

Basic Laboratory Course in Chemistry

Grundlagenpraktikum der Chemie

Part 1 **General and Inorganic Chemistry**

Edition 2019

Edited by Prof. Dr. Stefan Bienz

Manual for the *Basic Laboratory Course in Chemistry, Part 1* (CHE111) and the *Basic Laboratory Course in Chemistry for the Life Sciences* (CHE171) of the University of Zurich, supervised by Prof. Dr. G. Patzke, Prof. Dr. S. Bienz, Dr. J. Helbing, and Dr. P. Schmutz

Welcome to Our Basic Practical Course in Chemistry

We would like to welcome you to the *Basic Laboratory Course in Chemistry, Part 1* (CHE111) and the *Basic Laboratory Course in Chemistry for the Life Sciences* (CHE171), respectively. You have chosen to study Chemistry as your major or minor, and the modules CHE111 and CHE171 are compulsory supplements to the lecture modules *Fundamentals of Chemistry, Part 1* (CHE101), *Fundamentals of Chemistry for Students of Biology* (CHE170), and *Fundamentals of Chemistry for Students of Biomedicine* (CHE175). We recommend attending our practical classes parallel to the lecture courses mentioned above or after their completion. The laboratory classes will be continued with second parts (CHE112 and CHE173, respectively), for which the completion of CHE111 or CHE171 is a prerequisite.

The major goals of the practical courses are that you

- Acquire basic chemical laboratory skills and learn the safe manipulation of chemicals and chemical reactions.
- Become skilled in precisely observing and documenting experiments.
- Learn and practice basic chemical principles by performing experiments in Inorganic, Organic, and Physical Chemistry by yourself.
- Acquire knowledge pertaining the disposal and recovery of chemicals as well as to gain awareness of environmental protection from a chemical point of view.

These are ambitious aims, but you may count on our support in achieving them.

We hope you will enjoy this practical course!

Greta Patzke
Stefan Bienz
Jan Helbing
Paul Schmutz

Admission and Group Assignments

Admission

The number of working benches in the GPC laboratories is limited, and so is the number of students that can take part in the course. For this reason, the booking of the modules CHE111 and CHE171 counts as provisional reservation of a laboratory bench only, and we have defined the following rules for definite admittance to the courses:

CHE111 (offered only in the Fall Semester)

- Students that have booked CHE111 need to confirm their participation by personal signature on the occasion of the information meeting (“Einführungsveranstaltung”) that takes place in the week before the semester starts. Without this confirmation, they lose their reservation of a laboratory bench, which might mean that admission could be refused.
- Students that were not able to book CHE111 — for which reason whatsoever — need to register with us on the occasion of the information meeting to get admission to the course and access to a laboratory bench.
- Latecomers and students that did neither confirm their participation nor register with us on the occasion of the information meeting will be admitted only as far as the remaining space allows. The principle “first come, first served” will be followed.

CHE171 (offered in the Fall Semester and in the Spring Semester)

- Prerequisite for admission to CHE171 is passing the exam of CHE170 or CHE175.
- If not all students that have passed CHE170/175 can be accommodated due to shortage of laboratory space, the laboratory benches are assigned according to the performance in the exams.
- Students that did not pass CHE170/175 can still be admitted to CHE171 if sufficient laboratory space is available and the grade in the exam is ≥3.5. The remaining places are assigned according to the individual performances in the exams. Students that did not participate in the exams have lowest priority.

Group Assignments

- Admitted students of CHE111 that have booked a specific group will be assigned to a respective laboratory group if ever possible.
- Admitted students of CHE111 that have booked the *Wartegruppe* or that have registered with us at the information meeting will be assigned to any of the available groups.

- Admitted students of CHE171 will be assigned to the booked group or, if no group could be specified upon booking, to any of the available groups.
- If you need a special solution regarding your assignment due to compelling reasons (and only then!), please contact us in due time: on the occasion of the information meeting or before the end of the booking period. Be aware that reassignments become very difficult when the group assignments and the organization of the course are completed and all groups are full.

The group assignments will be published in due time *via* our information page on the web.

Scope of Work and Requirements to Earn the Credit Points

Scope of Work

The work requested within the practical course includes your:

1. Adequate self-preparation for the experiments by means of the manual and, if necessary, textbooks, internet, etc., and timely submission of the answers to the *Preparative Questions*.
2. Participation and cooperation during introductions and discussions.
3. Proper execution of all the experiments of the program and attaining their objectives.
4. Proper and complete documentation of the experimental procedures and your observations in the laboratory notebook while in the laboratory.
5. Proper evaluation and discussion of the experiments and their results in the laboratory notebook and/or in separate reports.

Program

The GPC is a structured course. This means that all students of a given group will perform the same experiments — sometimes with small variations — at the same time. The order of the experiments is fixed, and the respective organigram is published at the beginning of the semester (see also our information page).

Absence

The practical course has to be followed strictly according to the specified program, and the full program has to be completed to pass. If you have to be absent due to medical reasons, you have to inform your teaching assistant immediately (by e-mail) and provide a physician’s attest (to be shown to the teaching assistant).

Your teaching assistants are not allowed to grant leaves of absences. If you desire or need a leave of absence, you have to submit a well-founded, written request to Prof. Dr. *S. Bienz* as early as possible but not later than two weeks in advance (preferentially by e-mail: stefan.bienz@chem.uzh.ch). Such requests will be granted very restrictively and only for compelling reasons. Be aware that we will not respond to absence messages that are supplemented with requests for opportunities to make up for missed experiments.

In order to complete the course, you have to make up for any missed experiment, which will be possible with our full support, if your absence was approved or properly excused. If you miss experiments without our approval or without providing a physician's attest, we will not offer opportunities to make up for them. Since the scope of work for receiving the proficiency certificate can no longer be fulfilled, you have to leave the course. The module will be graded as failed, and you need to repeat it partially or completely.

Proficiency Certificate

You receive the proficiency certificate for CHE111or CHE171 when you have completely fulfilled the scope of work mentioned above within the given time frames, your work in the laboratory was assessed as "passed" by your teaching assistants (grade of 4 or higher), and you have paid the fee. Criteria for the assessments are: experimental skills, precision, cleanliness, chemical understanding, quality of the documentations, and keeping deadlines. The final rating of the course is "passed" or "failed" without disclosure of a grade.

Ethical Conduct

Scientific and safe working in a laboratory not only requires suitable laboratory techniques but also responsible and ethically correct conduct. Particularly with regards to plagiarism and data handling, the age of internet and electronic data processing has led to increased problems. Plagiarism and scientific fraud have also been observed in our practical courses. Typical examples are: the use of direct copies or translations of texts from Wikipedia, research publications, reports, or other sources or the use of data that were not acquired by oneself but copied from colleagues or invented and fictional.

We consider as unethical, for example:

- To enter data into the laboratory notebook that were not recorded from the own experiments (faked data, data copied from colleagues, or external data without indication of the source).

- To copy solutions to *Preparative Questions*, evaluations, or laboratory reports (completely or only in parts; electronically or hand-written). This includes, for example, also the use of third-party documents that are just adapted by “fitting-in the own raw data” or by “re-formatting”.

  It is acceptable, of course, to discuss evaluations and reports with colleagues. However, the reports will always be different in details of the content, because your observations will never match exactly those of your peers. If very similar documents result from close (but still independent) collaborations, refer to all people involved.
- To insert text blocks from external sources (*e.g.*, Wikipedia, text books, or others) into reports instead of using own phrasing or to insert graphics, text blocks, or data interpretations from external sources into your documents without giving proper reference to the origin.

Unethical conduct cannot be tolerated, even if it may appear of minor magnitude. It leads inevitably to sanctions, which might include the exclusion from the practical course with the module being assessed as “failed” and having to be repeated in its entirety. In serious cases, disciplinary action will be taken with the involvement of the dean of the faculty and/or the president of the university.

Exclusion

You will be excluded from the practical course, if any of the following applies:

- The proficiency certificate can no longer be achieved due to unsatisfactory performance or unauthorized absences.
- The preparation to the experiments is insufficient.
- The safety regulations or the instructions of the teaching assistants, technicians, or lecturers are violated out of intent or of gross negligence.
- The individual working space and the laboratory are not kept clean.
- Unethical conduct is detected.
- The fee for the practical course is not paid on time.

Equipment and Costs

Fee

The current fees for the practical courses are CHF 160.00 (CHE111) and CHF 120.00 (CHE171) and will be collected by invoice. The fees do not cover the full costs of the course and cannot be reduced — not even if you have completed the program only partially and have canceled the course within the allowed time frame. You owe the fee in any circumstance and in its entirety if you start the course (by attending at least one afternoon).

We will provide you without further costs with:

- Safety brochure
- Protective gloves
- Laboratory notebooks
- Chemicals for the experiments
- Glassware and equipment/instruments (as a loan)
- Replacement of broken glassware (as far as appropriate)
- Additional laboratory consumables

Personal Equipment

As personal equipment you need:

- Laboratory coat (long sleeves, covering the knees, and flame retardant)*
- Pocket calculator and a ruler
- Safety goggles, over-the-glasses spectacles for eyeglass wearers*
- *Peleus* ball (safety pipetting aid)*

* These articles can be purchased from *Atomoi* (chemistry student's union)

Equipment Service

Defective equipment will be repaired or substituted free of charge, provided that you did not break it deliberately or through gross negligence. You get replacements (also for missing instrumentation) from the technicians in the preparation room 13 M 50. Please notify the teaching assistants about any defective equipment in the laboratory, particularly larger stationary equipment such as rotary evaporators, vacuum pumps, melting point instruments, etc. It is imperative that this information is passed on to the teaching assistants and technicians as quickly as possible to ensure that the instruments are repaired timely and made again available for you and your colleagues.

Work Hygiene and Safety

Regulations

Health and Medical Constraints

- If you are pregnant or medically impaired, contact your physician and the head of the practical course.

Clothing

- It is mandatory to always wear safety glasses in the laboratory regardless of what you are doing. Safety glasses must have side protection shields. Normal glasses are not accepted.
- Always wear protective clothing in the laboratory: long trousers, closed-toed shoes, and a laboratory coat buttoned up, with the sleeves not rolled up. You will not be admitted to the laboratory if you do not wear appropriate clothing.
- You should wear gloves when there is a risk that you could come into contact with problematic chemicals. This is the case when you perform experiments that involve toxic or irritant chemicals or chemicals with unknown properties. Do not permanently wear gloves in the laboratory.

  Be aware that gloves are not long-term barriers for chemicals. If gloves get contaminated, they have to be replaced immediately. Thus, you always wear clean and uncontaminated gloves! This ensures that you are fully protected and also that you cannot contaminate items that might be touched later with unprotected hands.
- Confine long hair, loose sleeves, and scarves. These could cause accidents or catch fire when worn loosely.
- Do not wear large rings, bracelets, or necklaces, because these may get caught on laboratory equipment and thereby cause accidents. Moreover, contact with electrical sources may lead to electric shock.
- Do not wear laboratory clothing (laboratory coat and gloves) outside of the laboratories. In particular, do not visit the cafeteria “in full gear”!

General Behavior in the Laboratory

- Use your common sense.
- It is forbidden to deposit coats and other overgarments in the laboratories due to fire protection laws. Use the lockers in the corridor (beware of thieves).

- Smoking is strictly forbidden in the whole laboratory wing including corridors and balconies. Eating, drinking, and chewing is forbidden in the laboratories.
- Do not sit on the benches or on the floor.
- Do not place books, pocket calculators, backpacks etc. onto the benches. Only the laboratory notebook shall be used there for immediate recording of your measurements and observations. But keep the notebook still at a sufficient distance from your experiment.
- Never leave an ongoing experiment unattended. If you have to leave the laboratory temporarily, ask first your teaching assistant for permission.
- Do not perform any unauthorized experiments. This is strictly forbidden.
- Keep your work place always safe, clean, and in operable conditions. Remove unused chemicals and instrumentation from the working place.
- Clean dirty glassware and instrumentation even if you did not cause the soiling (the last user will strictly be responsible for improper relicts).
- Replace defective and missing glassware with the technicians.
- Store chemicals and instrumentation safely at your bench. *E.g.*, keep the chemical containers safely closed when not in use and do not place *Pasteur* pipettes or other small glass instruments onto the bench top (use a beaker or a rack!).
- Do not receive visitors in the laboratory.
- Turn off your mobile phone.
- Do not wear earbuds and do not listen to music in the laboratory.
- Do not rush, do not run, and do not push!
- Never work alone in a laboratory.
- Windows and escape doors to the outside may only be opened in case of an emergency. If windows or doors are opened, the air in the laboratory is no longer circulating properly, and fumes are dispersed throughout the building.

Preparation and Behavior while Performing Experiments

- Familiarize yourself with the safety equipment of the laboratories and its use: emergency shower, eye wash, fire extinguisher, first aid kits, emergency exits, telephone with emergency number etc. Observe the respective instructions.
- Prepare yourself carefully for your experiment:
  - Before you start and experiment, familiarize yourself with the equipment and the risk potential of the chemicals (flammability, reactivity, stability, toxicity).
  - Retrieve the required equipment and chemicals prior to performing the experiments and check carefully whether the glassware is free of damage.

  - If you do not find all the necessary equipment and glassware at your bench or if part of the equipment is damaged or soiled, immediately ask the technicians for replacement. You shall not remove equipment from your neighbor's bench! Dirty equipment has to be cleaned.
- Always work in a fume hood and with the sash pulled down as low as possible, unless you are instructed otherwise.
- Close chemical containers properly and immediately after you have removed the amount of material you need for your experiment.
- Never withdraw any chemicals with a *Pasteur* pipette directly from an original chemical container. Provide a local stock.
- Never return any chemicals, *e.g.*, from a local stock or from an unused batch, back to the original chemical container.
- Follow the safety instructions given specifically for the individual experiments in the book or by the teaching assistants.
- Monitor the progress of your experiments precisely.
- Focus on your experiments. Always be aware of what you are doing and do not allow yourself to be distracted (*e.g.*, by deep discussions).
- Be aware of what your neighbors do. If they perform an unsafe operation, make him or her aware of it and inform the teaching assistants, if necessary. Prevention is always the best medicine!
- Never use an open flame, except after approval by your instructors.

Before Leaving the Laboratory

- Clean your work place and equipment. You share it with colleagues who cherish a clean place just like you do. You will be held responsible for soiled equipment and/or a dirty workplace you leave behind even if you have inherited it from your predecessor (the devil takes the hindmost).
- Properly clear away all chemicals and equipment. Chemicals are not to be kept in the laboratory cabinets but are stored in trays on the shelves. Always return borrowed equipment to where you got it from.
- Close the sash of the fume hood.
- Turn off all electrical equipment and pull the plugs.
- Wash your hands carefully and use the hand lotion.

Disposal of Waste

- Never dump problematic waste in the sink. If in doubt, seek advice from your teaching assistants.

- Use the provided containers to collect specifically marked residues.
- Dispose of glass waste and injection needles — according to the instructions of the teaching assistants — in the correspondingly designated collecting containers and never in the normal waste bin. You would compromise the safety of the cleaning staff and of your colleagues.
- Do not overfill any collecting containers (chemicals, solvents, waste, glass, etc.). You may obtain replacements at any time from the technicians.

Emergency Procedures

- Notify the teaching assistants about all kinds of accidents; in case of major accidents also notify the head of the practical course and call the internal emergency unit by the emergency number.

  Tel. 144
- Also notify your teaching assistants of events considered as harmless, such as:
  - Unintentional inhaling of gases and vapors.
  - Skin contact with chemicals.
  - Smaller cuts.
- First aid measures are initiated by the teaching assistants and technicians. Further care will be performed on-site by trained staff (paramedic or physician, organized by the university's first aid organization). If external services were called (emergency unit, physician, hospital and so on), even when done later, the head of the course has to be informed.
- All incidents considered as accidents have to be documented by the teaching assistant. Respective report forms are available with the technicians.

Environmental Protection

In 1988, the *Practical Course in General Chemistry* was completely remodeled under the aspect to meet common ecological requirements. The amounts of chemicals were dramatically reduced, chemical products were separated and re-used to a great extent, problematic waste was avoided as far as possible, or otherwise separated and appropriately disposed of. These ecological principles are still observed in the current programs, even though some compromises had to be made due to the constantly increasing number of students attending our courses. We, unfortunately, can no longer offer “recycling experiments” and make students directly aware of ecological problems and their impact regarding “working-up” efforts.

However, we still want to separate our products and wastes to keep our disposals low. This needs your uncompromised collaboration. To reach our goals, you have to:

- Follow strictly the instructions given in the sections *Collection*. Use the correct collection containers for the several types of waste.
- aqueous solutions of heavy metals in the designated containers.
- organic solutions with halogenated organic compounds in the container "not recyclable halogenated organic waste"
- organic solutions with not halogenated organic compounds in the container “not recyclable not halogenated organic waste”
- ethanol and acetone washing solutions in the container “solvent waste (ethanol/acetone)”
- salts and powders in “dry chemical waste”
- Do not use organic solvents such as ethanol or acetone at the sink. Rinse glassware only when absolutely needed with organic solvents and collect the solvent in a large beaker or crystallizing dish, and pour it then into container “solvent waste (ethanol/acetone)”.

Questions Concerning Work Hygiene and Safety

1. Packages with chemicals are marked with pictograms that warn about the potential risks of the contents to health and safety. The older European hazard symbols, which you still find on many bottles and jars and which are described in your safety brochure, are being gradually replaced by new symbols according to the *Globally Harmonized System* (GHS).

   What are the meanings of the following hazard symbols (upper row = older European pictograms, lower row the equivalent GHS symbols)?

   Look up the information in the safety brochure or, if you have not received the brochure yet, in the internet.

2. Mister Felix Merryman, a somewhat priggish, partially absent minded, but motivated student of natural sciences likes organic synthesis, classical music, and outdoor activities. On an unusually warm summer morning, he slips into his favorite blue shorts and his T-Shirt with the inscription *„Survival of the Fittest"* in screaming red, puts on his new sandals, and sets out for the university. At noon, before setting off for his running training, he decides to just start the experiment for the afternoon. He enters the deserted and stale-smelling laboratory of the GPC (how on earth did he do this?) and starts to prepare the equipment and the chemicals for his experiment. He finds the potassium cyanide (KCN) that he needs for the cyanohydrin reaction, which had not worked on his first attempt, in his cupboard where he had stored it away the day before. Felix reflects on his experiment and the many possibilities that may have caused the initial failure. Overpowered by thirst and heat, he opens the windows and balcony doors and then takes out a can of Coke from his back pack. Accompanied by jumping electrons, breaking and forming bonds, as well as nucleophilic and electrophilic centers, he gets to work. He plugs in his earbuds and starts the music player on his smart phone. Then, he takes a sip of the Coke and, refreshed and animated, pours the chemicals that he prepared the previous day into the round bottom flask at his work place to the rhythm of *Mussorgskys „Pictures of an Exhibition*". Finally, he wants to weigh the KCN to be added to the mixture. While he is opening the bottle, his mobile phone rings. His friend Ester has just solved the chemistry exercises and needs a break. "I'll be coming to the lounge in a minute," he promises. (He has earned a smoking break anyway, and smoking is forbidden in den laboratories, as you well know!) In a rush, he quickly pours two spoons full of KCN into the round bottom flask — some more will not do any harm after all. —, places the open KCN bottle just below the separation funnel containing the half concentrated hydrochloric acid, which he already had prepared for the work-up of the reaction, and leaves the laboratory. He returns after 30 min, realizes that the ventilation is still not working properly, and goes to check on his experiment. He opens the sash of the fume hood, stoops down a bit and sticks his head into the hood to look into his reaction flask. He can only observe that the solution has turned slightly brown before he loses consciousness. Fortunately, the technician just enters the laboratory and can call a rescue team.

   a) What may have happened to Mr. Merryman? Look-up what could chemically happen if KCN gets in contact with acid.
   b) List as many safety rules as possible that were broken by Mr. Merryman during his work.
   c) Which telephone number alarms the rescue team (emergency number)?

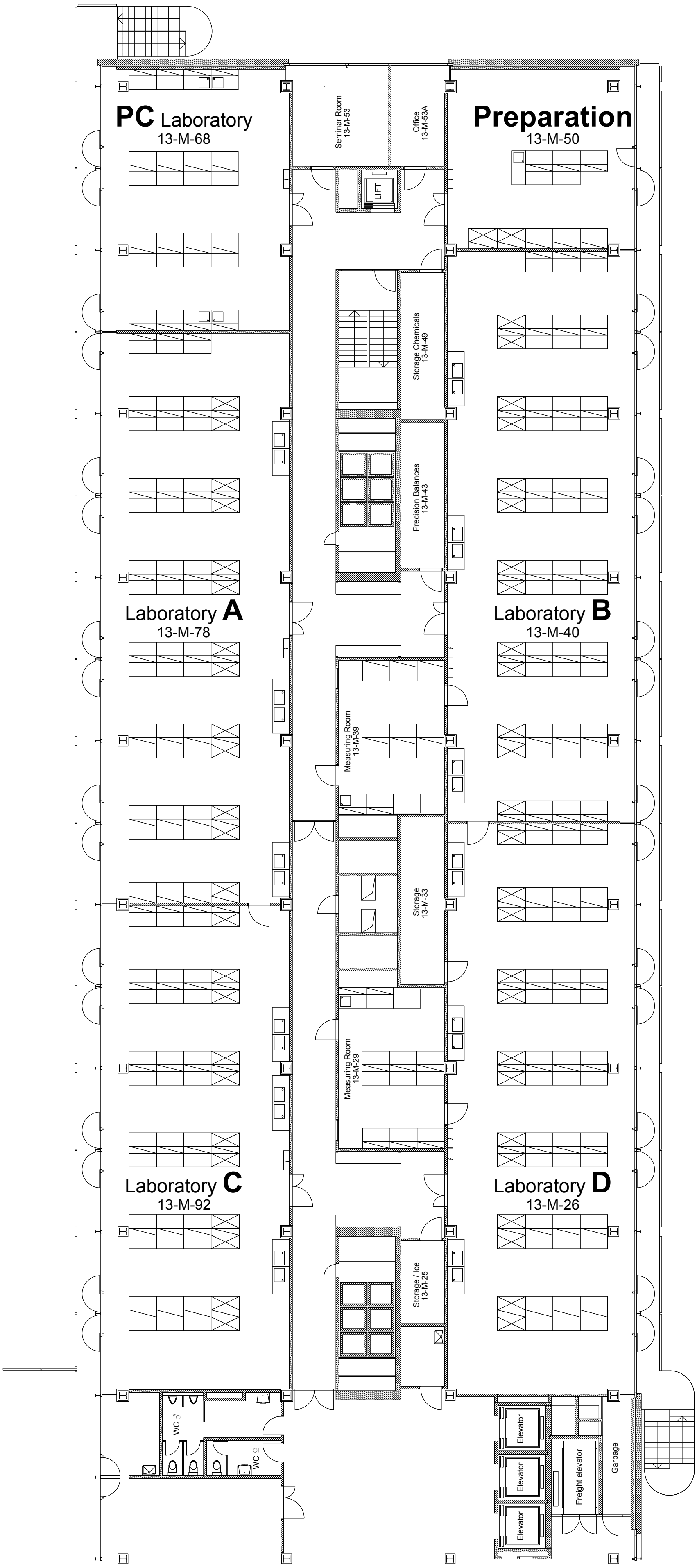
3. During your laboratory safety tour, the first afternoon of the practical class, you will be introduced to the several safety installations of the GPC.

   On the next page you find a map of the GPC laboratories. Answer the questions below during your tour.

   a) Where are the several safety installations of the GPC placed? Enter their locations into the map (just those that are relevant for your laboratory and for the common region you will use).
   b) Where are the emergency escape routes? Mark them with arrows in the map.
   c) Where is the meeting point; where do you have to assemble in case of an evacuation of the laboratories?

Use:

T	telephone	FA	first aid kit
EW	eye washing	ES	emergency shower
FB	fire blanket	FS	fire extinguisher sand
FE	fire extinguisher		


PC Laboratory
13-M-68
Seminar Room
13-M-53
Office
13-M-53A
Preparation
13-M-50
LIFT
Storage Chemicals
13-M-49
Precision Balances
13-M-43
Laboratory A
13-M-78
Laboratory B
13-M-40
Measuring Room
13-M-39
Storage
13-M-33
Measuring Room
13-M-29
Laboratory C
13-M-92
Laboratory D
13-M-26
Storage / Ice
13-M-25
WC ♂
WC ♀
Elevator
Elevator
Elevator
Freight elevator
Garbage

Preparation for an Experiment

Aim

In order to perform a chemical experiment successfully, understanding the theoretical background is important, but above all, you need to prepare well for all practical and specific tasks. If you prepare yourself well, not only will you be more efficient and obtain better results, but you will also contribute to a safer working environment in the laboratory. You will better understand what you are doing, gain time, and keep yourself and your colleagues safe.

Preparation before the Start of the Class

- Study the manual and, if necessary, use also other resources to acquire sufficient knowledge about the theory and practice of the experiment.
- Answer the *Preparative Questions* and submit the answers to the teaching assistants within the deadlines.
- Perform the *Preparative Task* before you enter the laboratory. In particular, watch the videos provided for some of the experiments on the OLAT platform.
- Summarize the experiments (flow chart, sketches of instrumentation) on a separate sheet of paper (not in the laboratory notebook).
- Prepare the laboratory notebook (header, tables for data collection, etc. **but no templates for the procedures**!) whenever appropriate.

The in-depth study of the documents is the first and most important part of every preparation for an experiment. You have to be aware of the chemical, technical, and experimental procedures to be able to perform the experiments sensibly and to understand them. We demand that you acquire sufficient theoretical background for each experiment. Such background information is given in this manual, but you may need to complement it with textbooks or internet researches. The preparation for an experiment includes also that you answer the *Preparative Questions* found in the manual. Hand over your answers to the teaching assistants according to the given deadlines — generally a week befor you start with the corresponding experiment. Your answers to the *Preparative Questions* serve, on the one hand, as a control for the teaching assistants whether you prepare yourself for the experiments. On the other hand, — and more importantly — they shall reveal your state of knowledge and in particular also your problems relating to the experiments. The teaching assistants should get a feedback about the issues they will have to clarify during the introductory meetings. Thus, it makes no sense to just copy the answers to the *Preparative Question* from colleagues: this is first of all unethical, and it can no longer serve the intended purpose.

In the initial phase of your experimental work — mandatorily in the modules CHE111 and CHE171 — we also demand you to briefly sketch all experimental details on a separate sheet of paper (to be done at home, before entering the laboratory). Compose your notes in a way so that they help you to work efficiently during the experiment. Preparing experiments also includes preparing the laboratory notebook (see the following chapter).

> *We have assembled our course manuals so that the procedures and techniques are described at the beginning in minute detail and only later more concisely, when you have to apply the techniques you have learned before. The detailed description of the experiments at the beginning of this manual has the advantage (and the aim as well) that we can teach you a consistent and correct laboratory technique, which you can, in principle, put into practice autonomously by means of this document alone. The short summaries of the experiments we demand from you (only in the Fall Semester) shall help you to work more efficiently and to keep track of your experiments!*

Preparative Work in the Laboratory

- Empty the drying cabinet.
- Check (and get hold of) the required equipment.
- Clean or replace equipment if necessary.
- Provide (partly weigh out) the chemicals, prepare local stocks of chemicals and solvents (you may share these with your colleagues).

When entering the laboratory, your very first task is removing your glassware from the drying cabinet. Do this well before the instruction meetings with the teaching assistants begin. This saves time and avoids unnecessary searches for missing equipment.

After the instruction meetings (not before!), after you have received the demonstration of the laboratory techniques and additional information related to the experiments, get all required equipment and chemicals ready. Make a layout of the glassware needed on the bench. Only when all glassware and chemicals are fully ready (and clean) may you start with the experiment.

> *You share your bench with other students. Therefore, it may happen — and it will happen! — that you will not find all your equipment at the place and in the condition you have left it. Thus, it is imperative to double-check all required material prior to the beginning. Missing and/or defective chemicals and equipment compromize any experiment, and you will most likely have to repeat it. Apart from this: leave your work place in the condition in which you received it originally from us. The last (and only the last!) user is held responsible for the condition of the work place.*

As far as applicable, weigh out the chemicals you want to use into suitable containers prior to the beginning of your experiment. Do this with all chemicals, even if certain substances will be used only later in the afternoon. Provide — best together with your neighboring fellow student — a local stock of solvents and certain other chemicals in appropriate containers and in appropriate amounts. Never ever return surplus chemicals from the stock back into the original storage containers; this is strongly forbidden (you may find reasons for this directive on your own)! Thus, do not build up too large local stocks because otherwise we would have to dispose of too much material.

Now that you are fully prepared for the experiment, assemble the required apparatus and start with your explorations.

Good Luck!

The Laboratory Notebook

Aim and Purpose

One of our most fundamental objectives in this course is that you learn to observe attentively and accurately and to record your observations neatly and completely. For the latter, you need the laboratory notebook. In the course of your scientific work, you will constantly be obliged to document your experiments and their results, regardless of whether you do research in chemistry, biochemistry, biology, biomedicine, or elsewhere. In any case, the time is going to come one day — latest when you have to write a report, a manuscript for publication, or a thesis — that you have to consult your original data, and then you will be thankful to find all pertinent information neatly, completely, and clearly documented in your laboratory notebook. For this reason, we demand right from the beginning that you properly protocol your experiments and keep a correct and organized laboratory notebook. The guidelines given below have to be strictly followed.

> *You might think that we exaggerate with the rules given below and the strictness we ask you to follow them. However, we intend to train you to a uniform routine that shall make your life easier in the long run. You should get drilled to spontaneously record your experiments in a consistent way so that you can later easily find all required information in a suitable form.*

The laboratory notebook is a document that keeps consistent and neat records of your experiments. It is *the* decisive document for the GPC (and not only for the GPC). A scientific experiment is considered as performed only as far as it is documented in a laboratory notebook. Missing or incomplete documentation of an experiment thus means that the experiment is not done.

A record in the laboratory notebook must comprise the description of all procedures and observations, the entries of all measured data (original data!), as well as a minimal evaluation. You have to protocol directly into the notebook the real procedure you followed. Even though, the procedures might be described in very details in our manual, you nevertheless have to describe one-to-one what you have effectively done and observed. This is usually different — at least in detail — from what you find in the documentations, so do not just copy them.

Your notes have to be recorded immediately and directly into the notebook, not onto separate sheets of paper from which transcripts are made later. The entries have to be done in a structured and clear manner so that any outsider would be able to find and follow all your procedures and to find all your obtained data. Reference to the course manual or other sources rather than describing the own procedures and data are not allowed. Thus, entries such as "The experiment was done as described in the manual..." or "NaCl was dissolved in H_2O (for the amounts, see the manual)" are not acceptable.

During this practical course, you will perform some experiments in teams of two. In these cases, both students have to keep their own, complete laboratory notebooks. Experimental data obtained as the result of a joint work are recorded into both notebooks, data acquired by a single person and to be shared for the collaboration is entered only into the notebook of the person that did the experiment. This data is made available to the collaborating party as a copy (integrated into the notebook as an attachment), marked with reference to the source.

The laboratory notebook is a bound notebook in which your entries are recorded doubly by a carbon copy. At the end of each course afternoon, before you leave the laboratory, you have to hand over your notebook entry of the day (the original) directly to your teaching assistants. You do that also when the experiment is not fully completed, *e.g.*, when yields or melting intervals must still be determined. The respective "addenda" are documented separately, on a new page of the notebook.

You only submit your notebook entries to the teaching assistants and not the notebook itself. The notebook is your document that accounts for your work and that you need to prepare evaluations, reports, and upcoming experiments.

The pages of your laboratory notebook are numbered consecutively and must be present without gaps. It is not allowed to tear out individual pages. Additional sheets with graphs, sketches, or other information can be filed in at the appropriate places and have to be numbered XXa, XXb, etc., with XX representing the page number at which the additional sheets added. Indicate the presence of additional sheets in the table of contents. If you have to correct something in an entry, cross the portion to be revised neatly out (do not erase it or cover it by *Tippex*) and rewrite it. You may not conceal anything in the laboratory notebook and, in particular, you also must clearly indicate any failed experiments!

Taking notes with a pencil is allowed and is even recommended because pencil writing does not blur when it comes into contact with water or solvents.

Structure of a Laboratory Notebook

A laboratory notebook consists of a front page (cover and first page of the notebook), a continuously updated table of contents, and the descriptions of the individual experiments with the related observations, data, and evaluations.

Front Page

The front page contains the personal data of the notebook keeper as well as the dates of the first and last entry and the scope of the laboratory notebook.

Notebook No. 1

Michael Muster
CHE 171, General Practical Course in Chemistry, Fall 2019

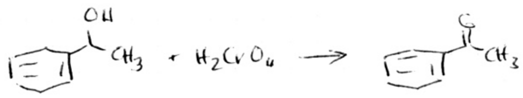
supervised by Jane Expert
Group BIO-IIC, Laboratory 13 M 92

Started: Sept. 19, 2019 with Experiment MM1-05 (1 A)
Finished: ……. with Experiment ……..

Michael Muster (signature)

Table of Contents

Start with the *Table of Content* on page 2 and leave the next three pages empty for the steadily growing index. The entries consist of a code for the experiment, a descriptive title, and the date of the experiment's execution. *E.g.*, it can look like this:

Code	Experiment	Date
……..	……..	……..
MM1-41	2 B: Measurements of volumes (preparation of solutions; density of NaCl solns)	01-10-19
MM1-45		22-11-99

etc.

Take as your code your initials and the number of the laboratory notebook, followed by the number of the notebook page at which the description of the experiment starts. Thus, MM1-41 in the example above refers to the experiment that is described in *Michael Muster*'s first laboratory notebook on p. 41ff. Use the same code for labeling your products and related documents (*e.g.*, spectra, reports, and graphs). If you get more than one product or several fractions in a separation procedure, extend the code appropriately (for instance: MM1-41A, MM1-41B for two products). The stipulated coding is very efficient and allows you to rapidly find the experimental details to any coded sample or spectrum.

The code of the experiment is followed by a short description of the experiment. For the GPC, this is the experiment number and the title of the experiment (shortened if necessary) or, for syntheses, the experiment number and the reaction equation.

Descriptions of the Experiments

Preparation

You may prepare your notebook for a new experiment by creating the header for the planned new entry and by compiling the relevant information related to the experiment. Such information might be safety and physical data for chemicals, literature references, and the like.

Be aware that you are not allowed to pre-write procedures into the notebook. All procedures and observations have to be directly protocolled into the notebook while performing an experiment: not before and not after. You may, however, prepare a check-list or flow-chart for any experiment you intend to do on a separate sheet of paper. In fact, you are even asked to do so for CHE111 and CHE171.

Header

The header for a new experiment is placed onto a new page and consists of a title, a description of the experiment (if not already clear from the title), a reaction scheme (for syntheses), and, for the benefit of your teaching assistants, your name and your group assignment. All chemicals (reactants and products) are enlisted together with their proper names, chemical formulas, and molecular masses. If asked for or needed, also add physical properties to the respective compounds and the source where you found the information (citation).

For more complex experiments, use subtitles to organize your notebook entries. You may not enter the subtitles in advance because you do not know, how extensive your protocol will be.

Description of the Initial Charge and of the Devices to be Used

A batch might be calculated in advance, but it has clearly to be specified as such and set apart from the real batch used in the experiment.

For syntheses and other experiments requiring stoichiometric calculations, describe the intended (planned) initial amounts in units of g or mL (to be measured data) as well as in mol or mmol. Reserve appropriate space to note down the effective amounts you used in the experiment. Mind that the effectively used amounts must be clearly specified as such and must be given even when they match exactly the intended values (cf. above).

If new or more complex devices are used in an experiment, it is appropriate to sketch these — as well as their assembly and their use — in the course of your preparation for the experiment as well.

Description of the Experiments

Following the compilation of all relevant basic information regarding an experiment, you describe by way of a protocol exactly how you have performed the experiment and what you have observed while doing it. The protocol must include all this information and also all original data. Missing or incomplete documentation means that the experiment is not done.

It is appropriate to merely use approximately ¾ of the page width for your protocol and to leave out some space to the right and the left for a "time line" and for additional notes, descriptions, and observations (cf. the example protocols).

Take care to record all parameters of your measurements, the used devices (specifications and inventory number), and all the original data with appropriate precision. Entries such as “approx. 10 drops”, after “10 min” or “the yield is 10.53 g“ (without further indications) are not sufficient. You did not add “approx. 10 drops”, but exactly 9, 10, or 11 drops, *e.g.*, with the *Pasteur* pipette, you did not wait "10 min" but from 14:33 to 14:43 (unless you have really measured the time with a stopwatch — then indicate this as well), and you (most possibly) did not determine the yield of 10.53 g directly but calculated it as the difference of gross and tare masses (= measured values to be noted, which were determined with a certain balance and accuracy).

Note again that you have, to protocol your laboratory work always while preforming an experiment and without exception directly into the laboratory notebook. Temporary recording of procedures and data, *e.g.*, to prepare clean copies later, is not allowed. The laboratory notebook has to accompany you through the laboratory!

The description of the experiments may be done in note form. A clean copy is not required. However, you have to write your entries in a structured and readable way, so that your experiments and their results can be understood and reproduced without additional information.

Evaluation

The description of an experiment always ends with a short evaluation; maybe interim evaluations are already appropriate to be done during the experiment. Distinguish clearly between theoretical information (expectations, chemical knowledge, etc.), your procedures (what was done), observations (what was seen, measuring results, etc.), and interpretations (what do the observed results mean with regards to the expectations). Measurements and observations do not need comments; however, interpretations most often do. Theoretical background is needed to bring your observations and interpretations into context!

In the evaluation, the results of the experiment are briefly summarized and, where appropriate, supplemented with yields, mass balances, graphs, and concise interpretations. You do the evaluation directly after each experiment — before leaving the laboratory. The immediate scrutiny of the experiment serves as a control for you to immediately see whether the results of your experiment are sensible or not and whether you may have to repeat your experiment or supplement it. Familiarize yourself with the required evaluation and objectives before you begin the experiment!

Examples of Notebook Entries (Laboratory Protocols)

You find two examples of notebook entries on the following two pages. They shall be reference and guideline for your own documentations. Note that the form of the entries for “technical experiments” differs significantly from that of “synthesis experiments”. We cannot give you an example that can be applied universally for all types of experiments because the several experiments differ too much from each other. In the Fall Semester, however, you will perform almost exclusively “technical experiments” and thus you will primarily use the example protocol 1 as a guideline for your notebook entries. You will receive additional information from your teaching assistants.

Example 1: Laboratory Notebook Entry for a Technical Experiment

Michael Muster, Grp BIO-IA MMI-25

Versuch 2B: Abmessen von Volumina

Lösung von NaCl herstellen für Dichtebestimmung 7.11.05

Ansatz

NaCl (58.44 g mol^{-1}) aus MM I-22 : 3.253 ± 0.001 g (55.66 mmol)

zu lösen in exakt 100 ml H_2O → Messkolben

Vorgehen

Zeit	Vorgehen	Beobachtungen
13:35	• NaCl in 100 ml Messkolben gegeben	
	• sofort ca 70-80 ml H_2O (ention.) zugegeben; Kolben geschlossen geschüttelt	Löst sich nicht sofort
13:40	• NaCl vollständig gelöst	keine Wärmetönung
	• Zugabe von H_2O (ention.) bis 2 cm unter Markierung (Spritzflasche)	
	• gut Schütteln (auf Kopf gestellt!)	
	• Tropfenweise (Pasteurpipette) Zugabe von H_2O bis Markierung erreicht	

Meniskus

Markierung

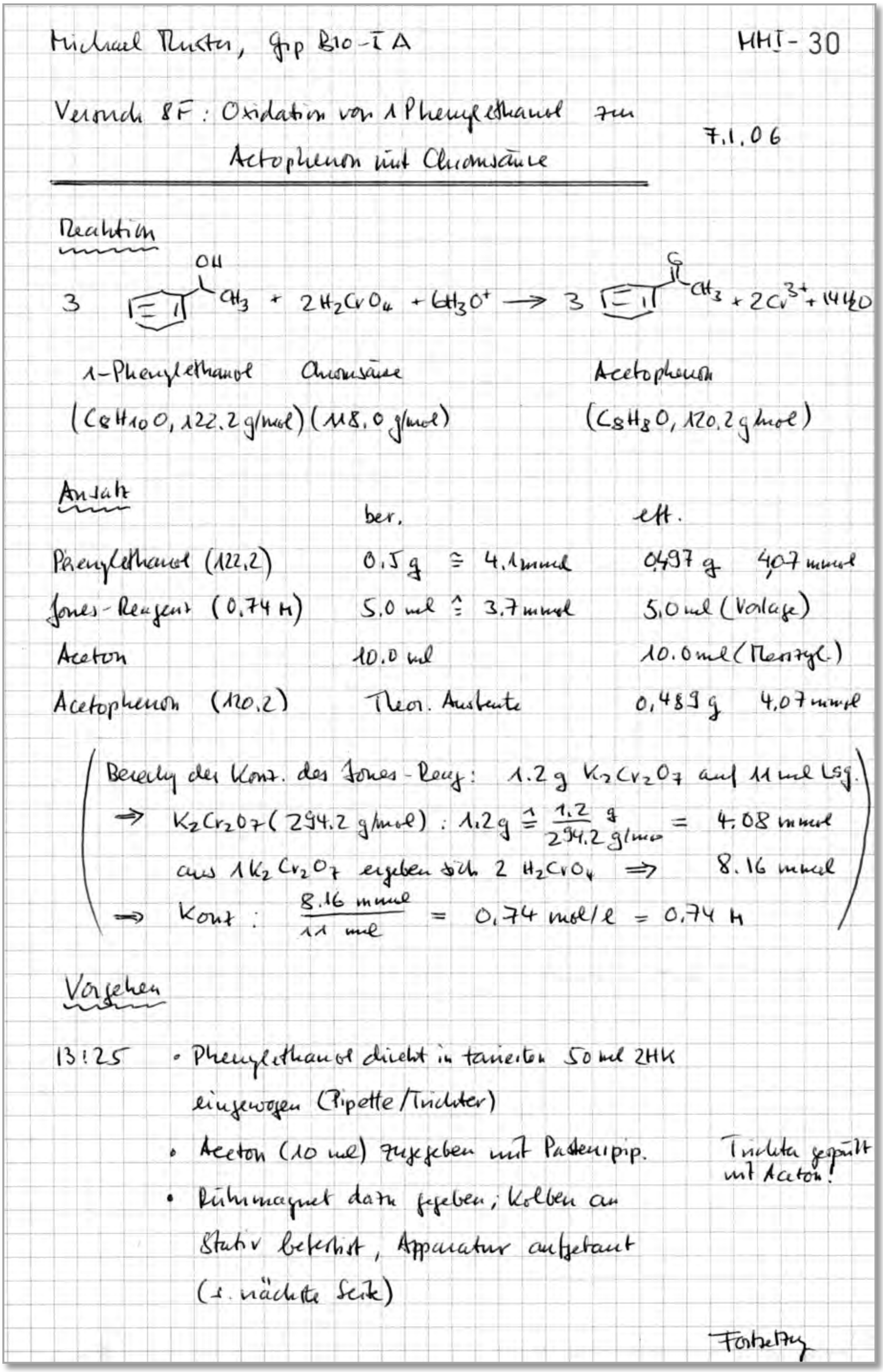
ein paar Tropfen haften am Glas!

Auswertung

Weiterfahren mit Bestimmung der Dichte dieser Lösung

Konzentration: 55,66 mmol / 100 mml $\hat{=}$ 0,557 mol/l (M)

Example 2: Start of a Laboratory Notebook Entry for a Synthesis



Michael Ruster, Grp Bio-I A

HHI-30

Versuch 8F: Oxidation von 1 Phenylethanol zu Actophenon mit Chromsäure

7.1.06

Reaktion

$3\ \ \overset{OH}{|}CH_3 + 2H_2CrO_4 + 6H_3O^+ \longrightarrow 3\ \ \overset{O}{\|}CH_3 + 2Cr^{3+} + 14H_2O$

1-Phenylethanol ($C_8H_{10}O$, 122.2 g/mol) — Chromsäure (118.0 g/mol) — Acetophenon (C_8H_8O, 120.2 g/mol)

Ansatz

	ber.	eff.
Phenylethanol (122,2)	0.5 g ≙ 4.1 mmol	0,497 g 4,07 mmol
Jones-Reagent (0,74 M)	5.0 ml ≙ 3.7 mmol	5,0 ml (Vorlage)
Aceton	10.0 ml	10.0 ml (Meßzyl.)
Acetophenon (120,2)	Theor. Ausbeute	0,489 g 4,07 mmol

(Berechg der Konz. des Jones-Reag: 1.2 g $K_2Cr_2O_7$ auf 11 ml Lsg.

⇒ $K_2Cr_2O_7$ (294.2 g/mol): $1{,}2\,g \mathrel{\hat{=}} \frac{1.2\ g}{294.2\ g/mol} = 4.08\ mmol$

aus 1 $K_2Cr_2O_7$ ergeben sich 2 H_2CrO_4 ⇒ 8.16 mmol

⇒ Konz: $\frac{8.16\ mmol}{11\ ml} = 0{,}74\ mol/l = 0{,}74\ M$)

Vorgehen

13:25
- Phenylethanol direkt in tarierten 50 ml 2HK eingewogen (Pipette/Trichter)
- Aceton (10 ml) zugegeben mit Pasteurpip.
- Rührmagnet dazu gegeben; Kolben an Stativ befestigt, Apparatur aufgebaut (s. nächste Seite)

Trichter gespült mit Aceton!

Fortsetzung

1 First Steps

The first experiments and instructions in this practical course introduce you to basic laboratory activities and techniques. You will learn how to document your experiments and results, and you will become acquainted with your laboratory, its safety facilities, your work place, and your laboratory equipment. You will be taught how to handle glass and how to work with simple glassware and other devices. For instance, you will learn how to fire polish glass edges with the blast burner and how to make glass capillaries from glass tubing. You will also be trained to work with the test tube, to estimate volumes of liquids, to transfer liquids, to handle *Pasteur* pipettes, and to use the *Teclu* burner. The individual experiments in this section are:

Experiment 1 A	Introduction to the Equipment
Experiment 1 B	Handling Glass; Processing Glass
Experiment 1 C	Working with the Test Tube
Experiment 1 D	Estimation of Volumes of Liquids

Experiment 1 A Introduction to the Equipment

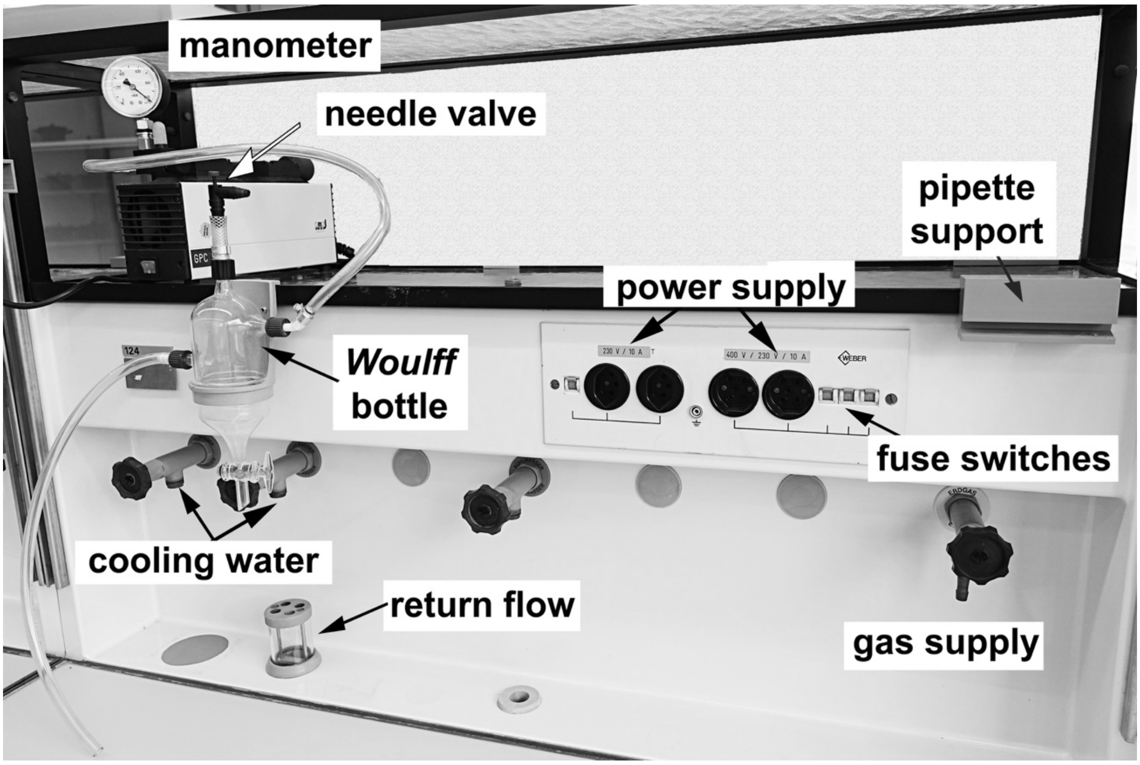
Objective

In this experiment, you become acquainted with the equipment provided at your work bench and in the laboratory. Later, you should be able to find the glassware and the equipment required for the different experiments in your stock again. We do not expect you to know all the glassware and devices by name and function after this experiment; you will improve your knowledge along the course.

Photographic Catalog of the Equipment Provided at Each Bench

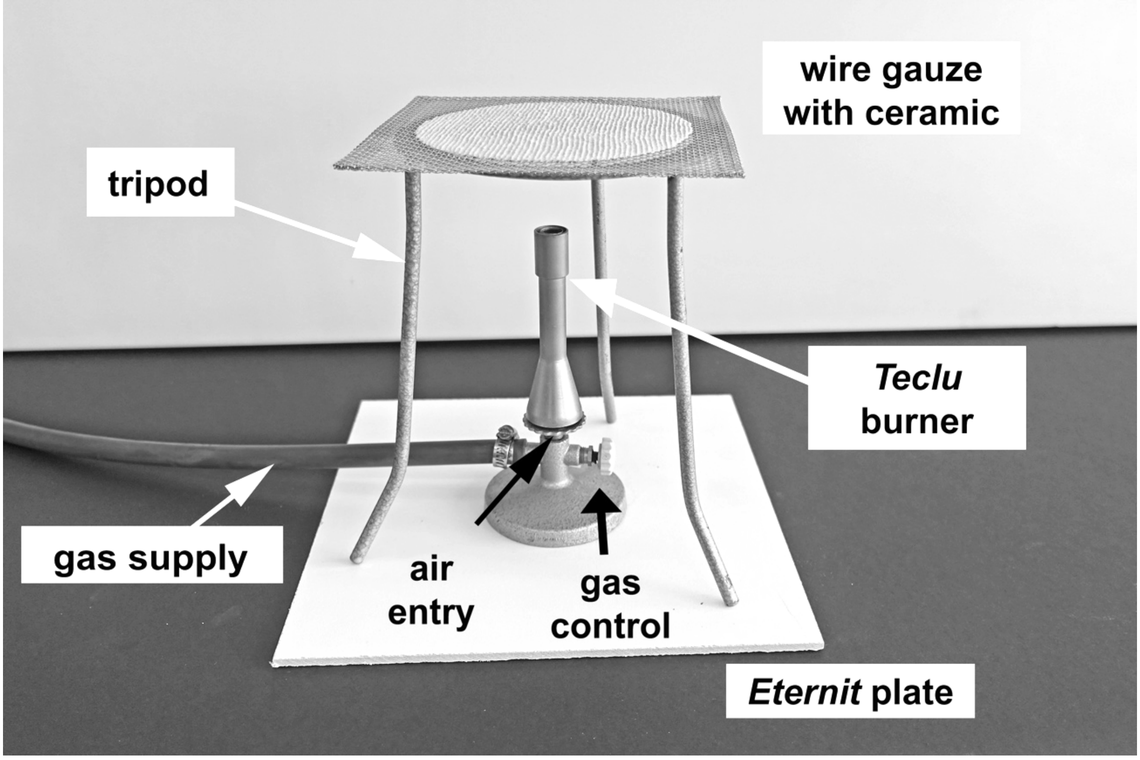
Below you find a list and photographs of the essential devices belonging to the individual stock of each laboratory work bench in the GPC; some of these are shown *in use*.

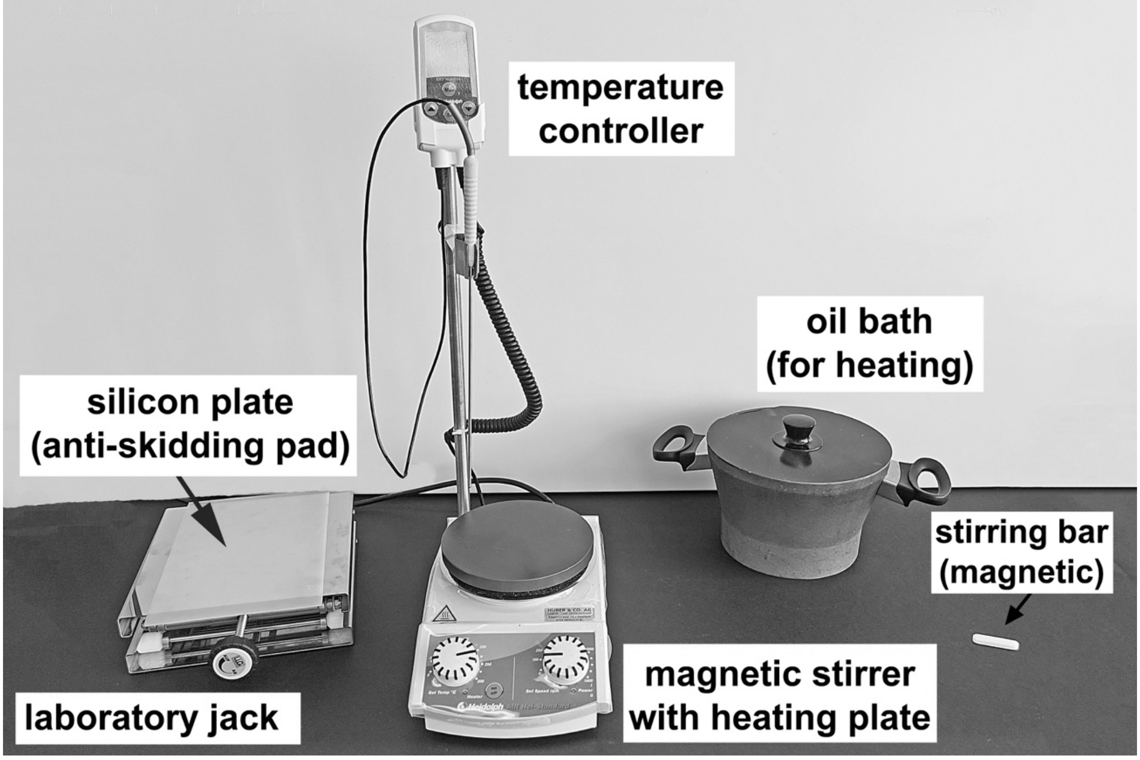
Fixed Installations



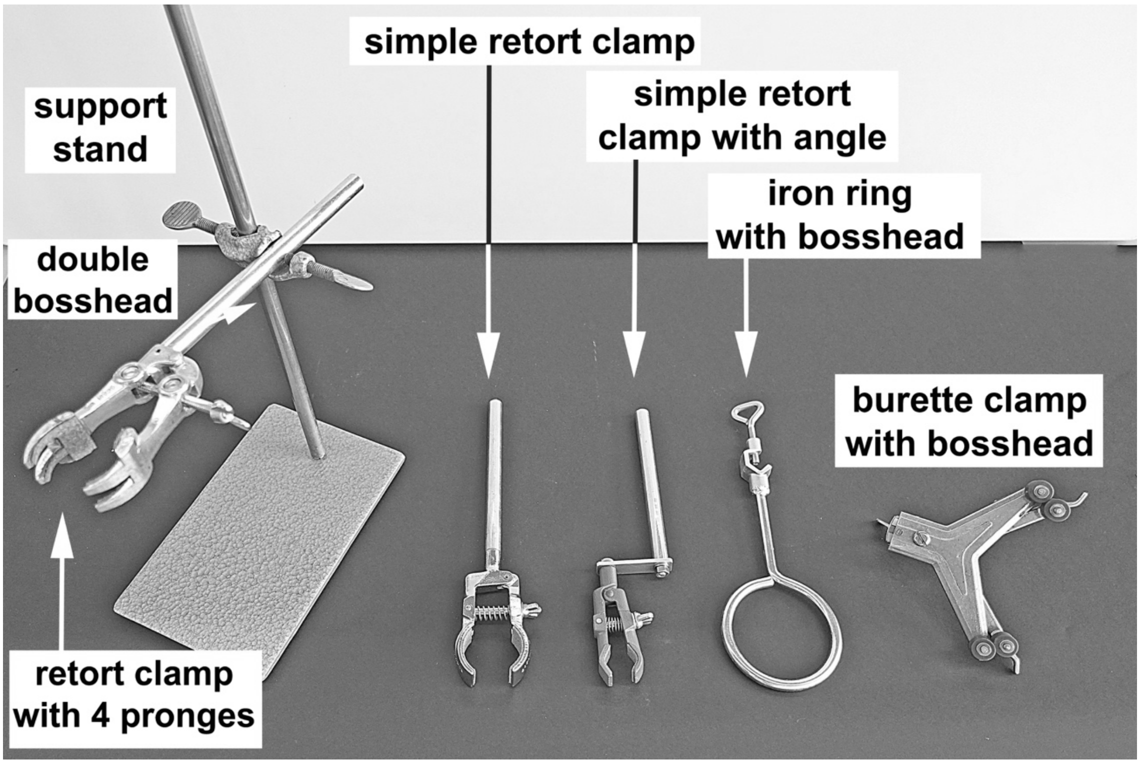
Further installations as well as supplies for gas, cooling water, and electric power are found in and around the fume hood. At the sinks, you not only find normal tap water (cold and warm) but also deionized water to be used as the solvent to prepare solutions of chemicals and to rinse the glassware after their washing. There is also an eye wash station at each sink!

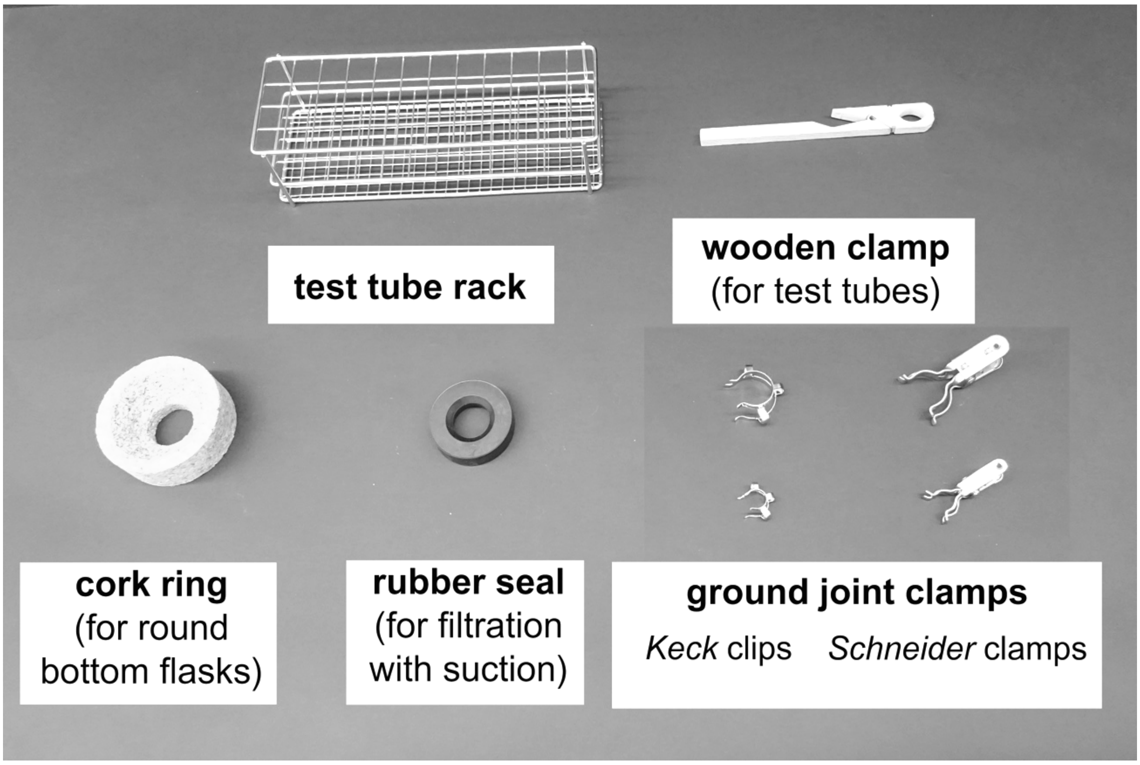
Heating and Stirring



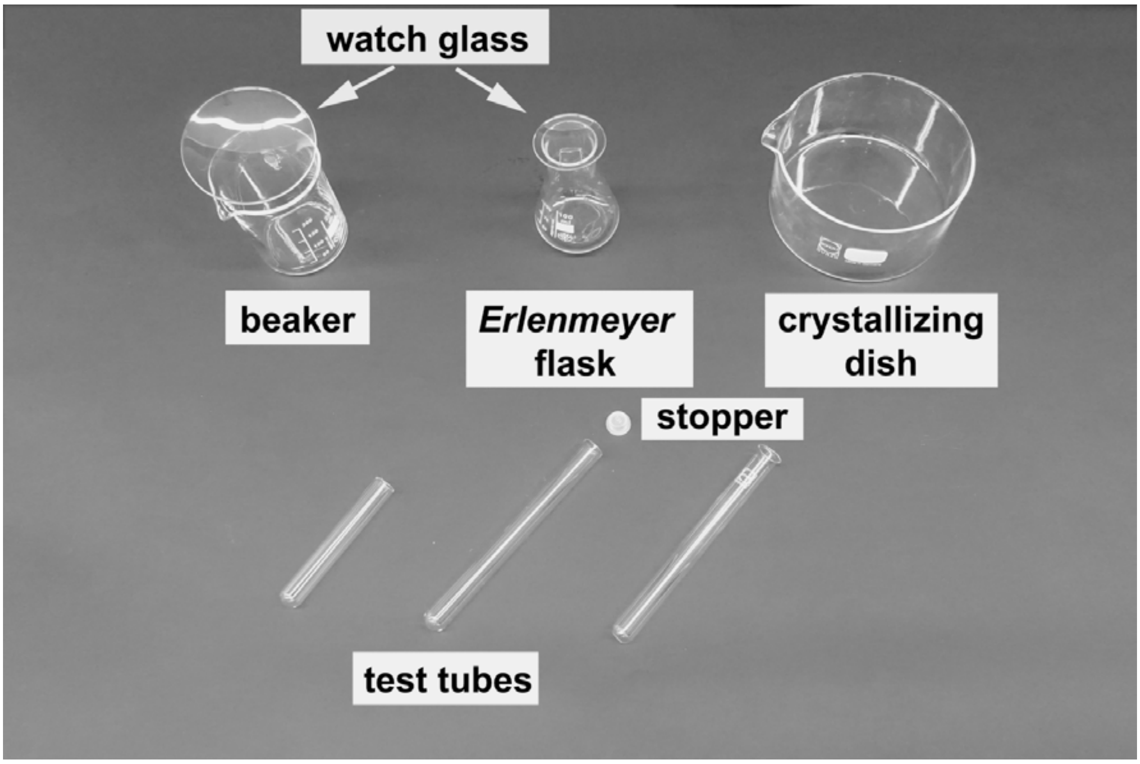


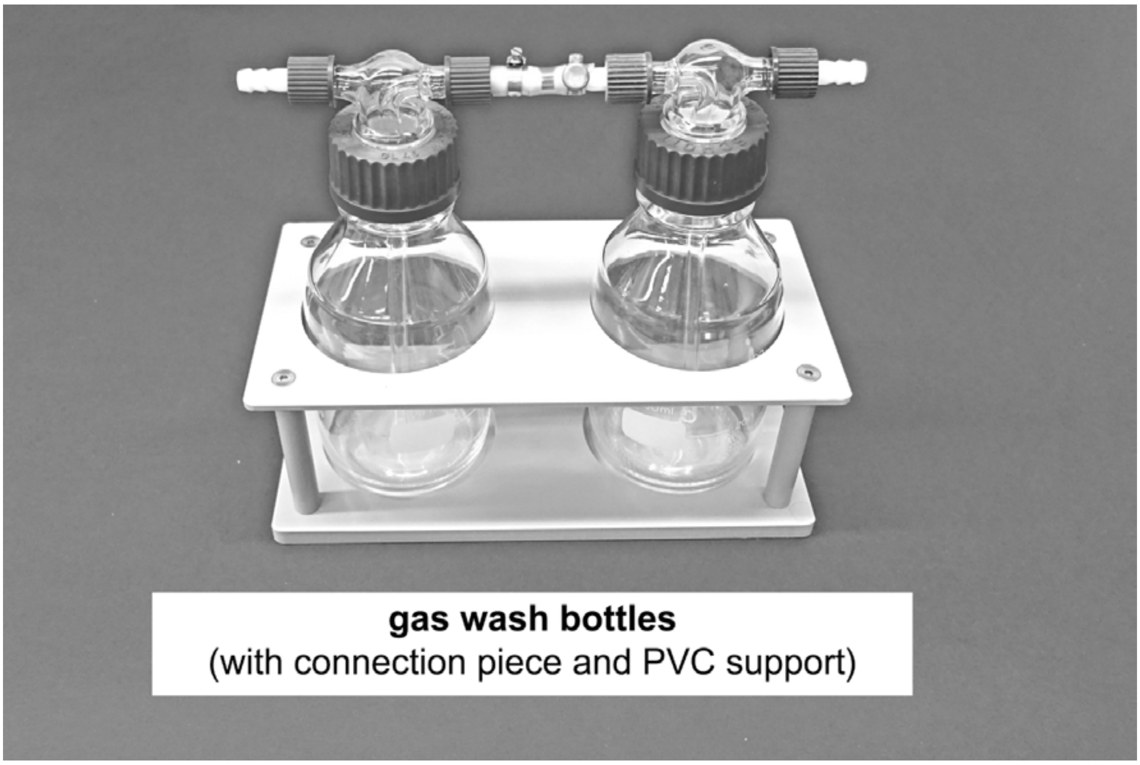
Mountings and Supports



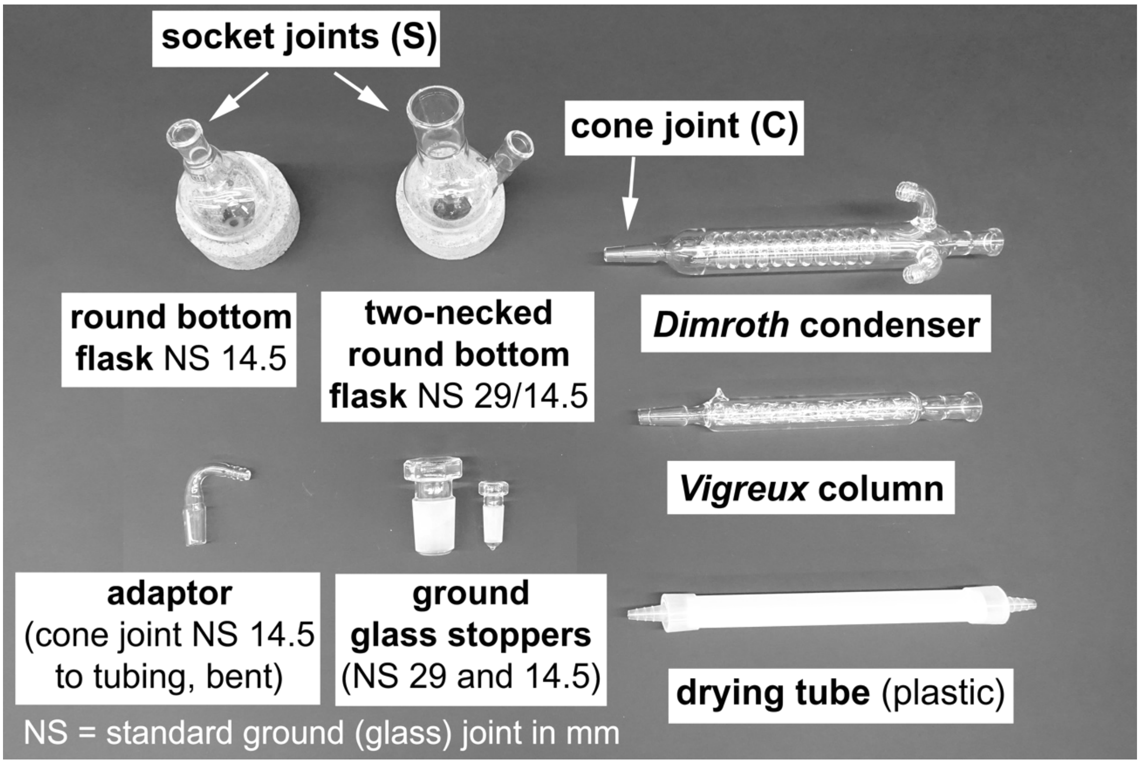


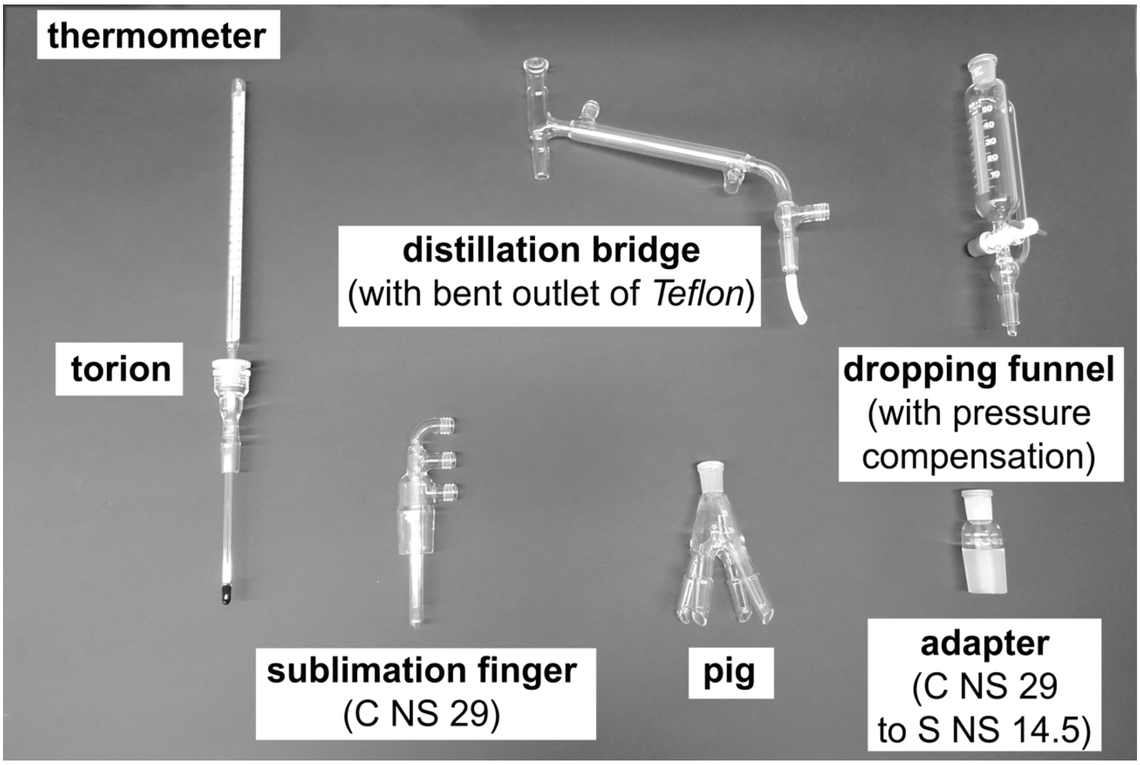
Glassware without Ground Joints



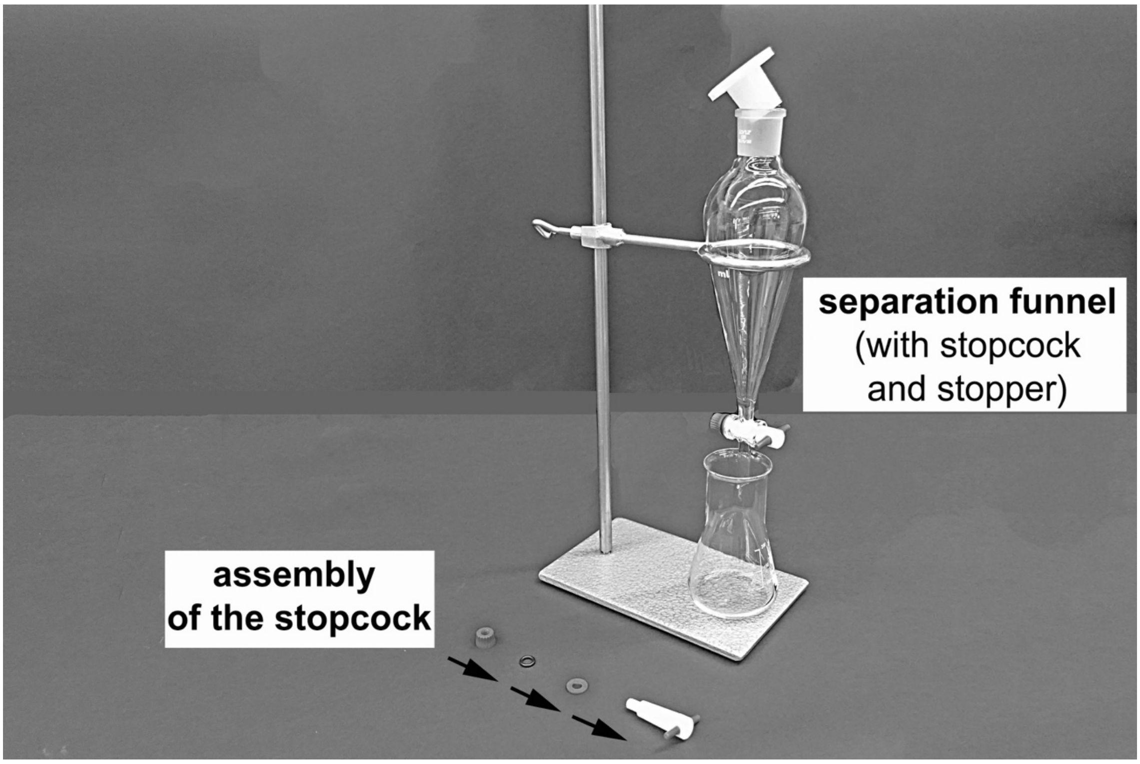


Glassware with Ground Joints

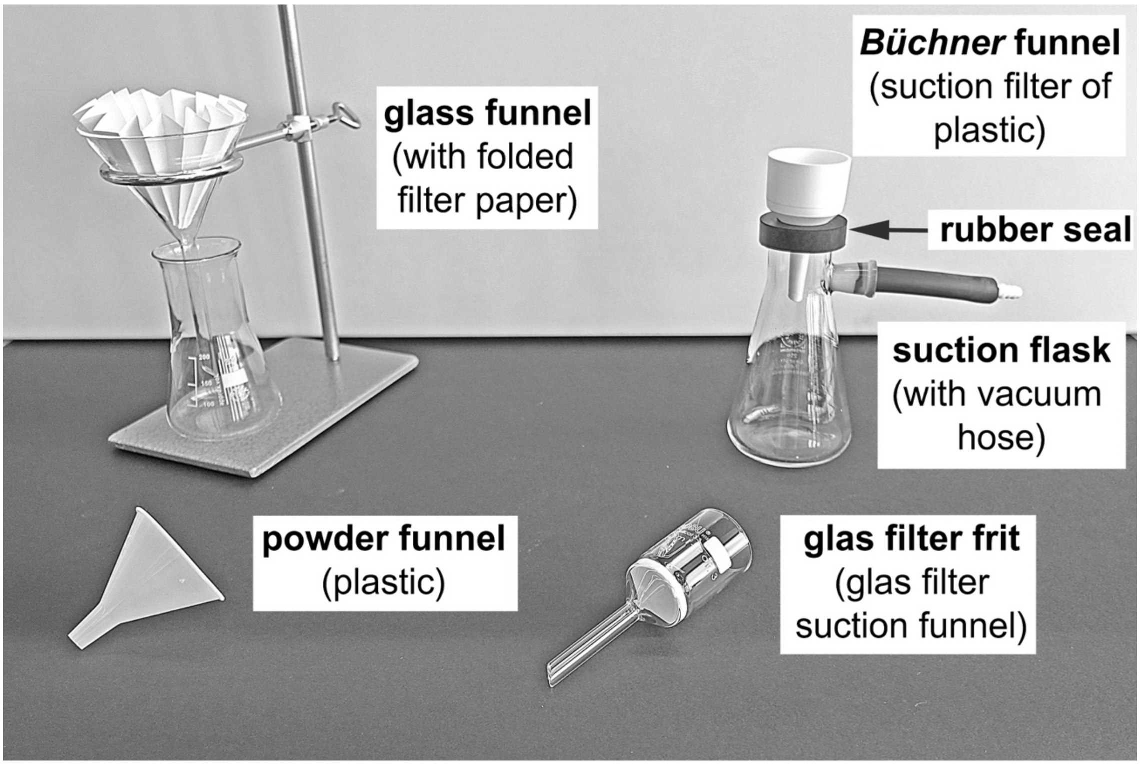




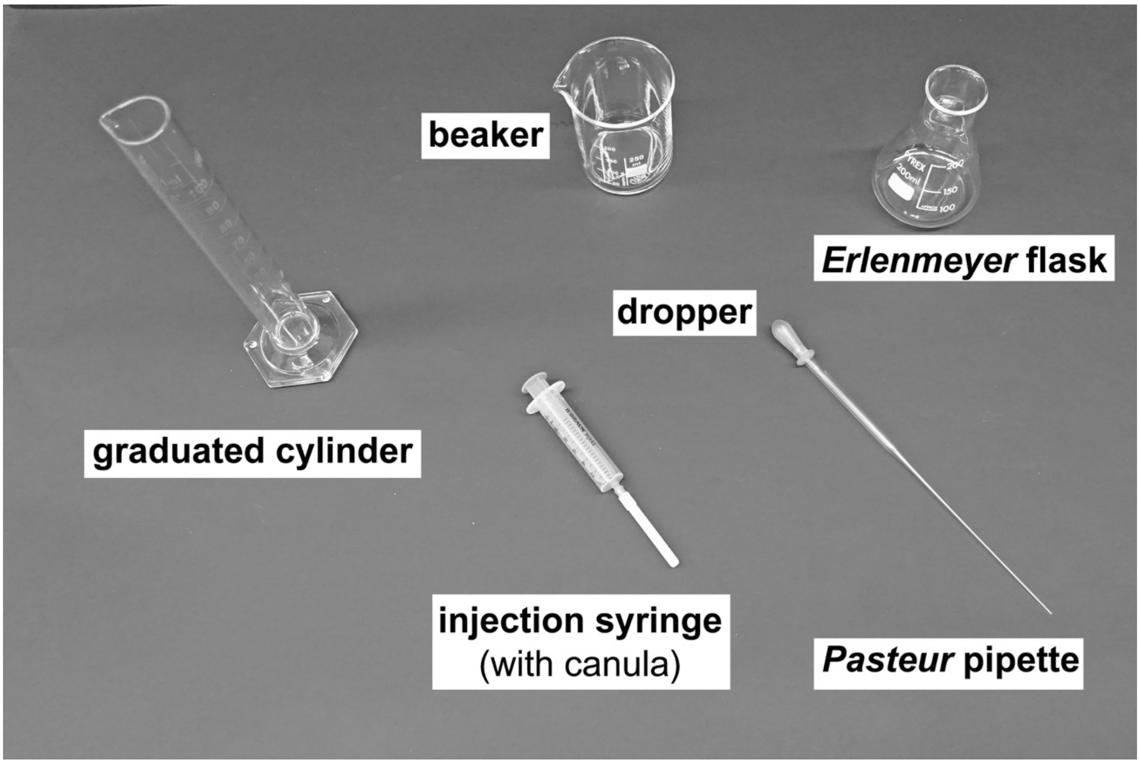
Equipment for Extraction

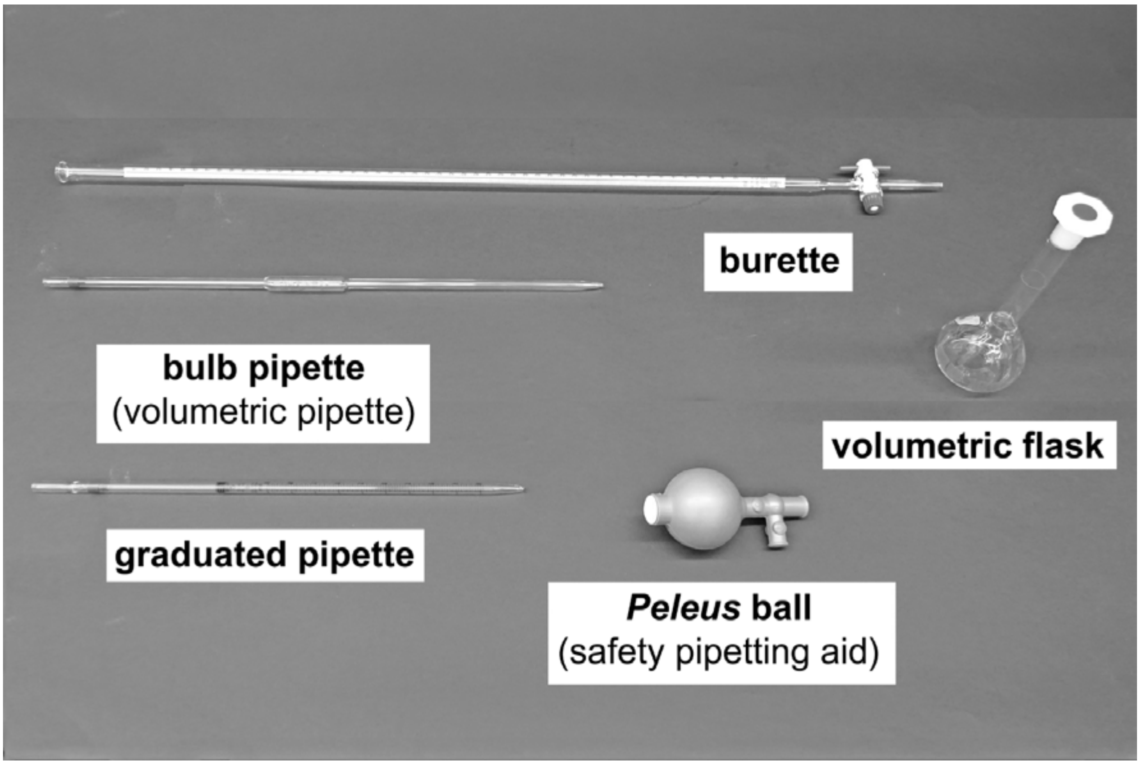


Equipment for Filtration

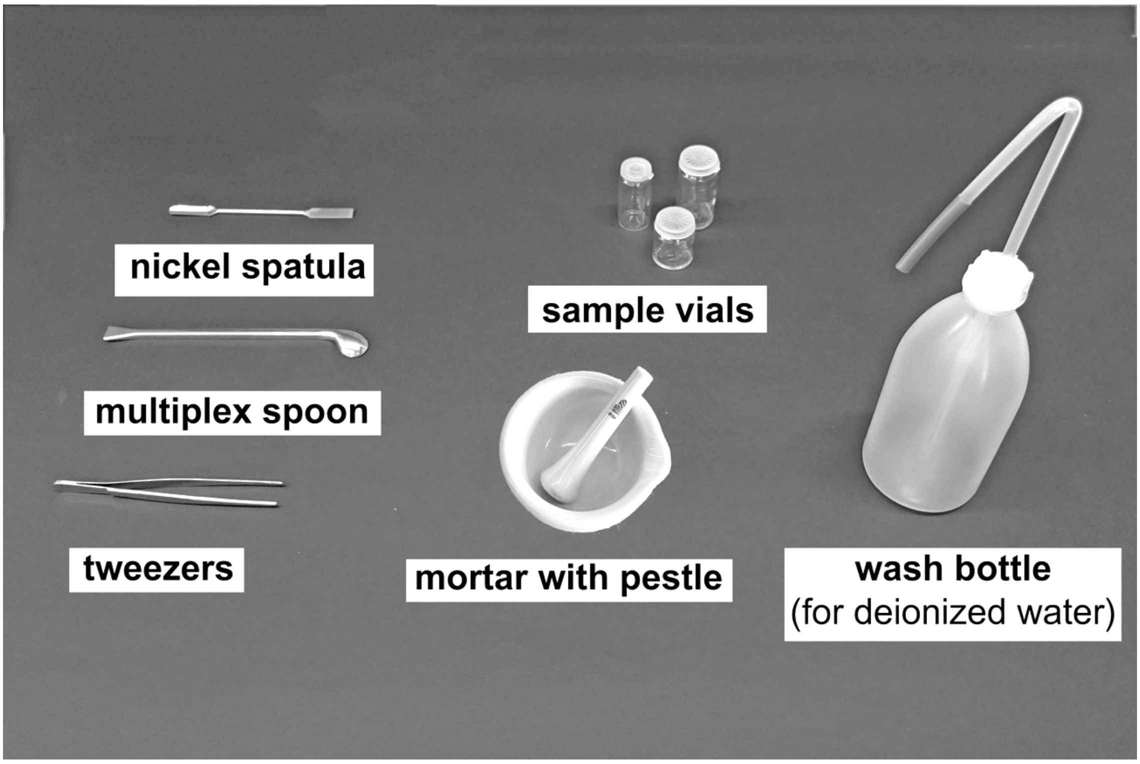


Volume Measurement





Further Accessories



Experimental Instructions

Problem

A: Some equipment was removed from your laboratory bench. Find the missing items.

B: The items supposed to be stored in the drawers are mostly consumables. Fill the drawers with the missing items according to the instructions of your teaching assistants (the numbers given in the inventory list are just approximate values)

Procedure of Inventory Check

Only the inventory contained in the cupboards has to be checked! Do the inventory control stepwise. For instance, start with the plastic box of the cupboard. Clear its content onto your work bench and check the respective stock for completeness by means of the distributed stock list (you can also find this list at the end of this manual). Try to identify the individual devices by their names and start with those you identify spontaneously. When you have found a piece of equipment, inspect it. If it is clean and intact, mark it on the list as “in stock” and place it back into the box. Soiled equipment is to be cleaned, defective material can be exchanged by the teaching assistants or the technicians. Record your observations and actions in your laboratory notebook (*e.g.*, distillation bridge was soiled and washed; missing items are: 1 × 100 mL round bottom flask, ...).

For devices you do not recognize spontaneously, consult the photographic catalog of this manual or the posters in the laboratories. Only if you cannot find it there and your colleagues are not able to help you, ask your teaching assistants for support. In this case, also sketch the “difficult” device into your laboratory notebook and label it with its name.

After you are done with checking the content of the plastic box, place the box back into the cupboard, remove the remaining items of the cupboard, and proceed as above.

Evaluation

No evaluation necessary.

Experiment 1 B Handling Glass; Processing Glass

Objective

Only a trained glassblower can produce new or repair broken glassware. In a chemical laboratory, however, it is often necessary to be able to perform simple tasks by oneself. This includes cutting glass tubes and rods, smoothing glass edges, and making capillaries.

In this experiment, you learn how to handle glass: scratching and breaking, melting and stretching. You will not become a professional glass blower, but you will develop some feeling for glass and learn how to handle glass and the blast burner.

Handling Glass and Glassware

Handling Glassware

Much of the equipment in a chemical laboratory is made of glass. Glass is transparent, smooth, free of pores, easily moldable, and resistant against common chemicals. Therefore, it is very suitable for chemical applications. However, glass also bears risks of injury. It is brittle and breaks readily, forming sharp edges on which you can easily hurt yourself. Thus, always handle glassware with great care.

In particular, you should:

- Never use force while assembling or taking apart glass devices.
- Never expose glass to sudden temperature differences (heating or cooling).
- Never heat containers with thick glass walls only at one spot.
- Introduce pipettes, thermometers, glass rods, and glass tubes without applying force into connecting openings (hoses, pipetting aids, *Torion*, stopper drills). If necessary, use a lubricant like glycerin, grease, or even just water. While assembling, grip the glass piece and its connection piece, each next to the connection site.
- Loosen up stuck ground joints and stoppers without force by gently tapping at the joint with a soft object (wood or cork) only. Do not twist the parts. Heating with a heating gun or with the weak flame of the *Teclu* burner or the use of an ultrasonic bath might help as well.
- Never heat closed containers or apparatuses — especially not when they contain heat-sensitive or reactive substances — because they might explode.
- Always ask the teaching assistants when you are in doubt about anything.

Broken or cracked glassware must not to be used any longer. Put it into the glass waste bucket or into the container for repairing. Note that glass waste is *never* to be disposed of in the normal waste bin. Broken glassware in the waste bin poses a high risk of injury, particularly for the cleaning staff having to empty the bins.

Glassware, especially the devices with ground joints, is rather expensive. It can often be repaired. Thus, do not unnecessarily dispose of glassware. If in doubt, ask the teaching assistants.

Cleaning Glassware

Glassware can generally be cleaned with normal tap water and a household detergent. To remove persistent contaminants, use brushes. For very adhesive (or strong-smelling) organic impurities, you may have to use some wash-grade solvent (usually acetone). However, use these solvents sparingly and only in the fume hood (never at the sink!) and collect the contaminated solvent in the appropriate disposal container.

To avoid glassware showing lime residues after drying, rinse it three times with a little deionized water after appropriate cleaning (please use the deionized water sparingly).

The glassware is dried at 80 °C on your shelf in the drying cabinet. The cabinet is working in an automatic mode and does not need your further attention. Put only clean and disassembled glassware into the drying cabinet (remove labels, stoppers, cocks, etc.). Mind that graduated glassware (such as volumetric flasks, graduated pipettes, bulb pipettes, etc.) and plastic parts (such as plastic stoppers, rubber bulbs, test tube racks, gaskets, etc.) may not be placed into the drying cabinet.

Experimental Instructions

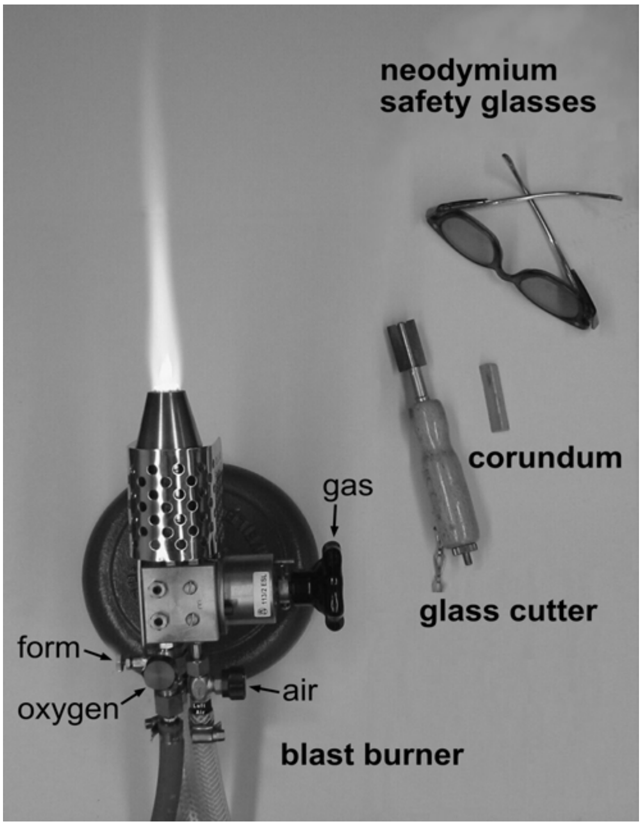
Problem

In the course of your practical work, you will need glass rods of different lengths and glass capillaries (for applying samples onto thin layer plates, cf. later).

In this experiment, you will use a blast burner to make two glass rods with fire-polished ends (about 20 and 30 cm in length, unless you already have them at your place), and at least 60 “good” glass capillaries with an outer diameter of about 0.3 mm and a length of about 15 cm.

Accessories

Blast burner, neodymium safety glasses, glass cutter and corundum (see picture), as well as glass rods and glass tubing with different dimensions are provided in the instrumental rooms I and II.



Safety Instructions

To avoid damage to your eyes, always wear the special neodymium safety glasses found at the glass blowers' bench when working with the blast burner. These glasses filter the intense and damaging Na-D-line of visible light. The neodymium safety goggles have to be worn by wearers of corrected glasses as well!

You cannot see when glass is hot, but touching hot glass can result in severe burns. To avoid burns, never heat a glass item at multiple places. If glassware has to be processed in a way that heating at several places is required, work at one place at a time and let the glass cool down between the steps.

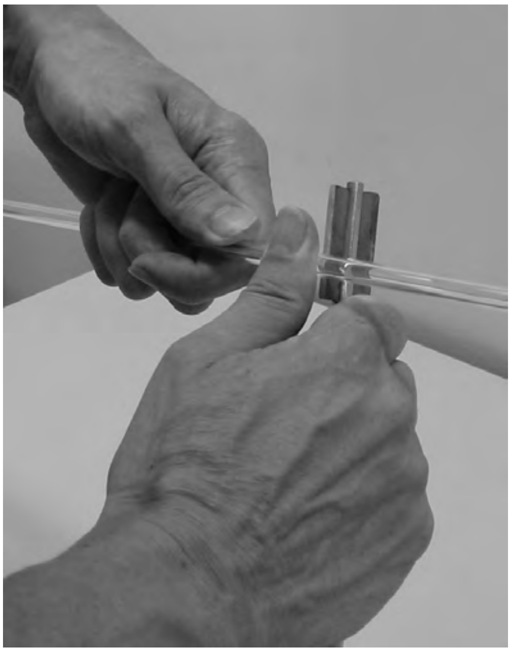
When you leave glassware to cool down, always place it with the hot end turned away from you (against the wall) onto a heat resistant surface. The part of the glassware facing you is thus always cool and can be touched. If you strictly follow this rule, you (and also your colleagues who might try to pick up your work piece) will avoid burns. Carefully determine whether a glass piece is still hot, *e.g.*, by gently breathing across the possibly hot place towards your hand. If the breath is significantly warm, then the work piece is still too hot to touch.

It still might happen, even though you work with great caution, that you burn your fingers on hot glass or cut yourself on sharp edges. Burned skin must be cooled immediately with cold water for at least 15 min. Then, treat the burn with an ointment and maybe place a bandage on it. Smaller cuts should be disinfected and bandaged. Ointment, disinfectant, and bandages can be found in the first aid boxes in the corridor. In case of bigger incidents, alert the house medic through your teaching assistant or the technicians.

Procedure

Cutting and Breaking of Glass Tubes

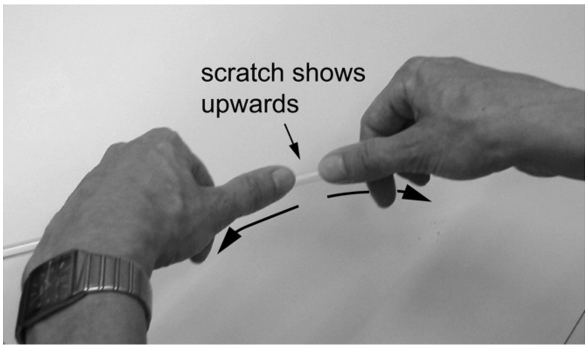
First, you cut four glass tubes to a length of about 30 cm and fire-polish their sharp edges. Use the glass cutter to cut the glass. Moisten the blade of the cutter slightly (*e.g.*, with spit) and then press it quite strongly onto the glass tube opposite to the thumb (see figure below). The glass tube is scratched by slight turning of the cutter around the tube — 2 to 3 mm are sufficient. You should hear a little crunching noise indicating that the glass cutter has worked.



Attention: An insufficiently scratched glass tube does not break cleanly. It can shatter or "frazzle" and result in injuries. Moreover, it is dangerous to perform several scratching attempts at or next to the place of an insufficient scratch-

> *ing attempt. The glass might break in an uncontrolled manner at the already weakened place, and this might lead to injuries.*
>
> *If you are not sure whether or not you have scratched your glass tube correctly, you can wrap the tube into a towel before breaking it and then proceed as described below. In most cases, this procedure is unnecessary, but it is recommended for the more cautious students among you.*

To break the glass tube, grasp it tightly with both hands. The scratch has to point upwards, and both thumbs have to point towards it (see figure below). Now, break the tube by carefully pulling and bending it away from the scratch. If you have scratched the tube correctly, the breakage is clean and smooth. Nevertheless, the edges are sharp and have to be rounded by fire polishing.



Starting the Blast Burner and Adjusting the Flame

Make sure that the valves of the blast burner are closed. Turn on the power switch on the electric board, and then activate the gas supply for the laboratory. Open the oxygen supply for the glass blowing bench by opening the valves starting at the oxygen cylinder going to the glass blowing table. (To close the valves, proceed in the same direction: from the oxygen cylinder to the burner.)

> *Never completely open a valve! If a tap is turned up to the stop, it might get mistaken for a closed tap, tried to be opened with force, and damaged.*

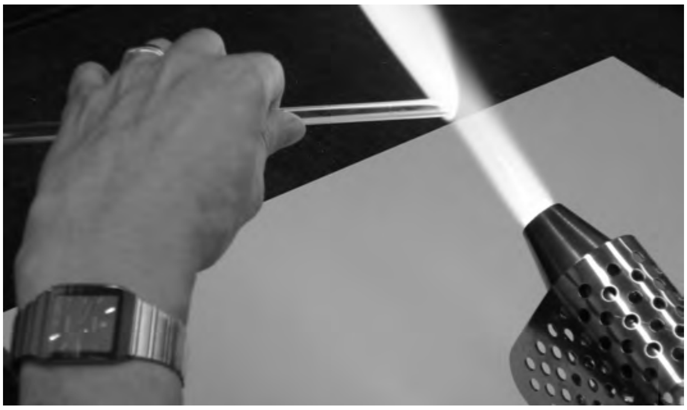
Then, cautiously turn on the supply for gas, air, and oxygen for the blast burner at the glass blowing bench.

Now, slightly open the gas tap at the burner and ignite the emerging gas. Add oxygen until the flame just becomes blue. Then, form the flame by adding oxygen with the aid of the “form” valve. There is no need to add air. Optimize the flame by stepwise adjusting the settings of the three valves “gas”, “oxygen”, and “form”. The flame is good when it has a bluish color, a length of about 20 cm, and a width of about 2 cm, and when it is almost soundless. The flame should be “soft”: it should slightly flicker and easily moves with a draft of air fanned by your hand. Such a flame is sufficiently hot — but not too hot — to work with the glass.

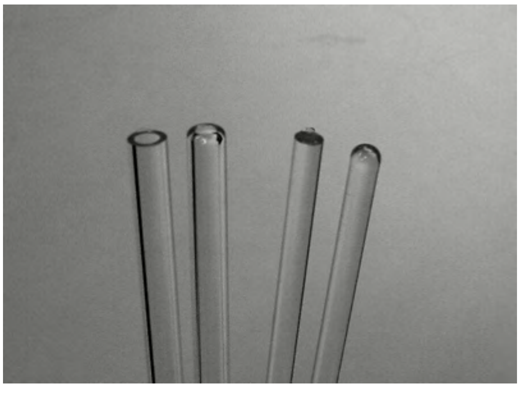
Fire Polishing of Glass Tubes or Glass Rods

Attention! Protect your eyes; wear the provided neodymium safety glasses!

To fire polish the sharp edges of a freshly broken glass tube (for a glass rod, proceed accordingly), hold the glass edges slightly inclined into the brim of the bluish oxygen flame (see figure below). While doing so, rotate the tube continuously along its axis. Heat the glass until the inner and outer edges are “at red heat” and you can see that the sharp edges have become rounded. It is not necessary that the whole cross section of the glass is rounded; it is sufficient to round the edges. Take care not to seal glass tubes by heating them too strongly.



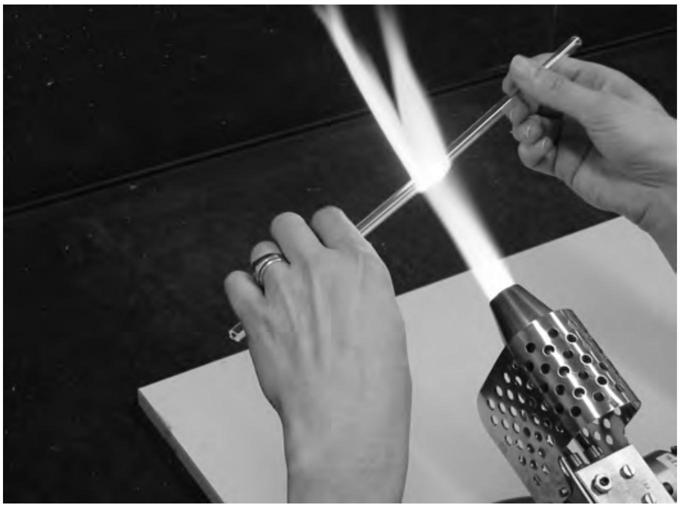
Place the hot glass tube — the hot end against the wall — onto a fire-resistant surface such as one of the *Eternit* plates of the glass blowing table or one of the metal sheets on the benches opposite of them. Do not place the hot glass pieces onto the white bench tables because their surface would get singed. Wait with fire polishing the other end of the tube until the tube is fully cooled down. In the figure below you see a comparison of the raw and polished glass rods and glass tubes.



Making Glass Capillaries

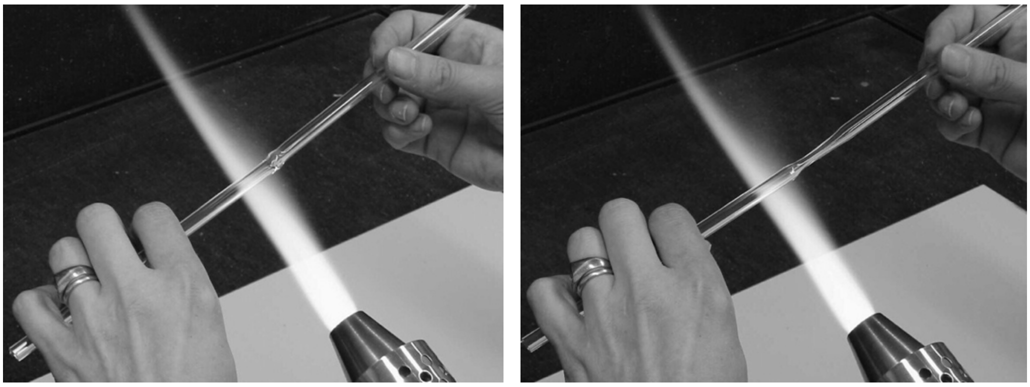
To make glass capillaries from a glass tube, you need to heat a tube in the center — without sealing it — and then to pull the ends apart. Proceed as follows.

Sit comfortably down in front of the blast burner and take a fire-polished glass tube (approximately 30 cm of length) at the two ends into both hands as shown below: your left hand grasps from above, your right from below. Before bringing the glass into the flame, try first to hold the tube slightly inclined (not completely perpendicular to the flame) and to rotate it uniformly around its axis while keeping it in place. If you feel comfortable, bring the center of the glass tube into the broad and "soft" part of the hot flame (ca. 5 cm away from the mouth of the burner) while continuously rotating it around its axis. After a short while, the heated glass part becomes red-hot, and the tube can already be slightly deformed.



Note that you shall not pull the glass tube apart while it is still heated in the flame. If you do that, the tube will become sealed. Rather "push" the ends slightly together so that a small "bulge" is formed (but the tube does not get sealed!). By this, you will obtain capillaries that have somewhat thicker walls and are less fragile.

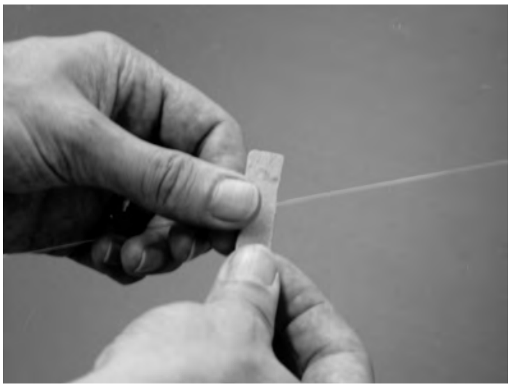
The uniform rotating of the glass tube becomes more difficult when the glass is further heated and becomes very soft. Try to avoid a twisting of the glass. When the heated place is almost white-hot and the melted glass is really soft and mobile, take the tube out of the flame, and only then begin to pull the ends apart: slowly at first, and with increasing resistance more strongly. This way, you can obtain capillaries with a length of up to 2 m. Cut the capillaries cautiously, without burning yourself, off from the glass tube and place the hot glass parts onto a heat-resistant surface for cooling (with the hot ends against the wall).



Cutting and Capillaries

Use the corundum to cut the glass capillaries to a length of about 15 cm. Grasp the raw capillary with your left hand between your thumb and index finger, and scratch the glass carefully by sliding the corundum over the glass without any pressure (see figure below). Then, break the capillary by slightly bending it against the scratch (same movement as with breaking the glass tube). Check the break: it should be smooth and without protrusive edges. Capillaries with sticking out edges are useless. They can, however, be "repaired" by breaking away a small piece of the capillary.

Hand over approximately 60 glass capillaries to the teaching assistants. They will store them for you; you will use them later to apply samples onto thin layer plates in Experiment 5 B.



Collection

Dispose of the cooled glass residues in the respective waste containers provided. The glass will be recycled.

Evaluation

Describe your working with glass in your laboratory notebook with notes and sketches.