

Some laboratory methods

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	page
<u>Preface</u>	1
<u>General</u> Laboratory notes. Water. Glassware. Removal of CO <sub>2</sub> and O <sub>2</sub> . Recrystallization.	2
<u>Weights and volumes</u> Weights. Volumetric glassware.	3
<u>Acids and bases</u>	
<u>Preparation and purity.</u> HClO <sub>4</sub> , HCl, HNO <sub>3</sub> , Na <sub>2</sub> CO <sub>3</sub> , NaOH, KOH, Ba(OH) <sub>2</sub>	5
<u>Analysis.</u> Acid-base-titrations. Strong acids. Strong bases. Carbonate. KHCO <sub>3</sub> , Ti <sub>2</sub> CO <sub>3</sub> .	8
<u>Ionic media</u>	
<u>Preparation and purity.</u> NaClO <sub>4</sub> , NaCl, NaBr, LiBr, KNO <sub>3</sub> , Ba(ClO <sub>4</sub> ) <sub>2</sub> .	9
<u>Analysis.</u> NaClO <sub>4</sub> , Ba(ClO <sub>4</sub> ) <sub>2</sub> , other ionic media.	11
<u>Other stock solutions</u>	
<u>Preparation.</u> AgClO <sub>4</sub> . Other metal perchlorates.	11
<u>Analysis.</u> Metal. Perchlorate. Silver-halogenide titrations. Redox titrations.	12
<u>Emf work</u>	
<u>Thermometers and potentiometers.</u> Thermometers. Potentiometers. Wilhelm and other glassware.	13
<u>Electrodes for [H<sup>+</sup>].</u> General. H <sub>2</sub> . Quinhydrone. Glass.	15
<u>Redox and metal electrodes.</u> Redox. Mercury and amalgam. Silver.	18
<u>Reference electrodes.</u> Calomel. Ag <sup>+</sup> /Ag. X <sup>-</sup> /AgX, Ag.X <sup>-</sup> , HgX <sub>4</sub> <sup>2-</sup> /Hg.	20
<u>Determination of H and h.</u> Correcting H. Equations for E. Gran dia- gram. Calculating E <sub>0</sub> , j, and h. H in so- lutions of metal salts. Determination of E <sub>0</sub> . E <sub>0</sub> for metal, amalgam, and redox elec- trodes.	21

## Preface

The present notes are based on the experience of workers in this laboratory up to June 1959. They will be revised from time to time in the future; suggestions for additions or improvements will be most welcome.

For the type of work we have chosen a high standard of accuracy must be maintained. This little collection of methods is not a substitute for the personal instruction of new co-workers, nor for experimental judgement. It goes without saying that nobody need feel obliged to follow the procedures described here if he wants to try other methods. This manual may be useful in bringing attention to certain good standard methods, and also in encouraging discussions and the testing of new methods.

It is important to be accurate and careful in the right place. Therefore you should try to realize what is the accuracy of your various determinations, and how possible errors may influence the final results. Double checks must be made, if possible using methods as different as possible, so that errors in reading or mixing (which may happen to anyone) cannot get as far as the final results. Still worse are systematic errors, such as impurities in the reagents used, or systematic errors in the analytical methods. You may remember that the determination of total perchlorate with a cation exchanger, which we once considered reliable, seems to introduce systematic errors in the presence of some polyvalent cations.

The methods given above are certainly not perfect, and may be improved. If you have information <sup>or</sup> experience which differs from the statements made here, or which you think ought to be brought to the attention of others anyway, will you please let me know so that it can be introduced into the next edition. Thank you.

Lars Gunnar

General

Laboratory notes: please keep your notes in such a state that someone else (such as yourself three years later) can understand what the notes mean and find what he wants. It is especially important that the primary data (analyses, calibrations, emf-data) which form the basis for all the following work, are kept in such a way that they do not disappear or are not mixed with secondary calculations. Preferably use a bound book, or numbered loose leaves, with your name, and date of the experiment on them. Never rely on your memory; its half-life is shorter than you think.

Water. Distilled water should be tested for impurities that may influence measurements:  $\text{CO}_2$ , silicate,  $\text{Cl}^-$ .  $\text{CO}_2$  is removed by boiling, or by bubbling through  $\text{N}_2$ , free from  $\text{CO}_2$ . Chloride is detected with  $\text{Ag}^+$  (compare with known concentrations). Silicate is detected by the yellow molybdate test (yellow color on addition of  $\text{NH}_3$  and  $\text{NH}_4^+$  molybdate Nils, Georg II): More sensitive (too sensitive for some purposes) is the molybdate-benzidine test (Vogel, Qualitative Analysis 1945, p. 292, first method, Nils). For some purposes (e.g. rinsing bottles and under fortunate circumstances, even mixing equilibrium solutions), distilled water from the tap may be used; in general it is safer to use the doubly distilled water from Frans E:s room. Even that must be tested!

Glass-ware. For acid solutions use Jena Geräte or Pyrex which, however, will dissolve in the course of time to give a few tenths of a mM  $\text{Si}(\text{OH})_4$ . Alkaline solutions should be kept in polythene bottles with thick walls (which let through less  $\text{CO}_2$  and  $\text{H}_2\text{O}$  vapor). Usually the screw stopper is tight; this can be tested by squeezing the bottle a little, while screwing the stopper into position; if the bulge is still there next time you use the bottle, you can be certain the stopper is tight. Rubber stoppers will stain the polythene and probably contaminate the alkali; therefore, screw stoppers or smooth polythene stoppers are preferable. (Nils)

For short periods, however, one may keep equilibrium solutions with not more than 0,1 M  $\text{OH}^-$  in Jena Geräte glass vessels (Gösta, anions III, A C S 1959, 722). In the course of a titration, only some 0.01 mM Si will dissolve, and glass has many advantages, for instance it is possible to see a faint opalescence.