

Automated Karl Fischer titration for liquid samples



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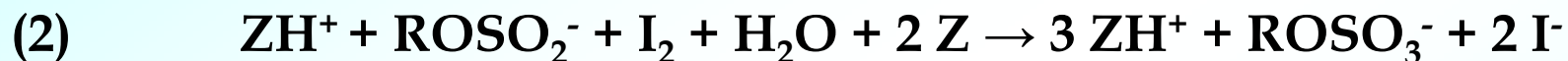
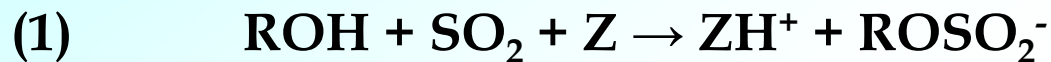
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Outline

- **Karl Fischer titration: theory and techniques**
- **Automation**
- **Volumetric and coulometric KF systems: methods, setup and results**

Karl Fischer Titration



complete equation:



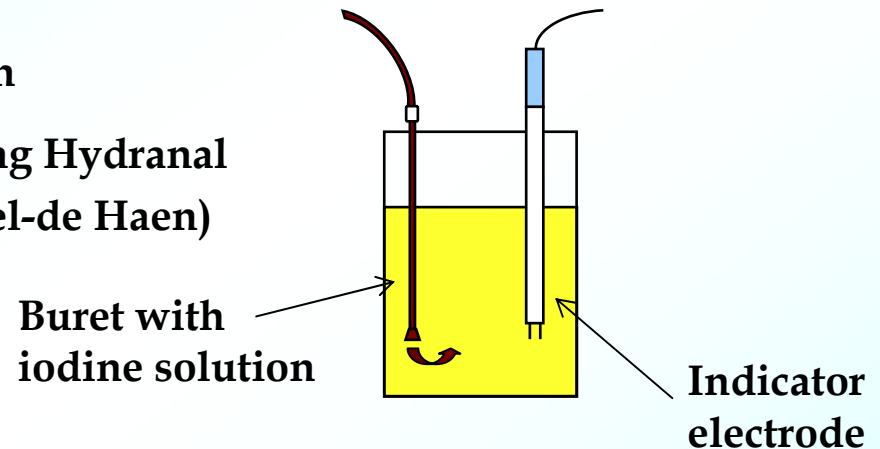
Z = base (e.g. imidazole)

R = -CH₃ (in most reagents)

Techniques of Karl Fischer Titration

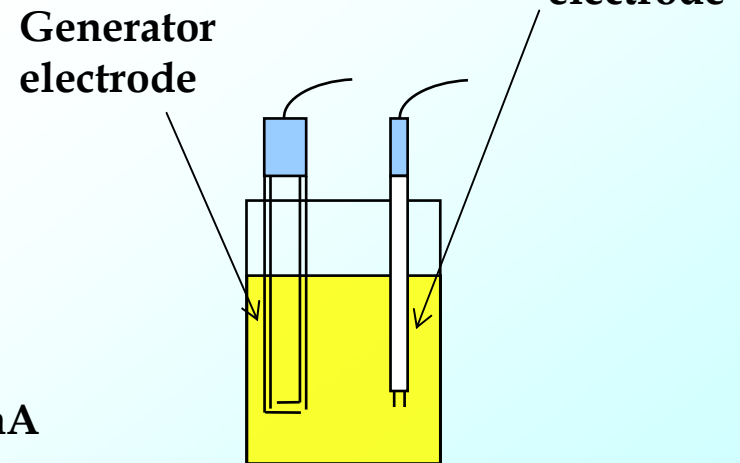
Volumetric titration

- direct titration with iodine solution
- One-component KF technique using Hydranal Composite 2/ Methanol dry (Riedel-de Haen)
- $I_{\text{pol}} 50\mu\text{A} / 250 \text{ mV}$



Coulometric titration

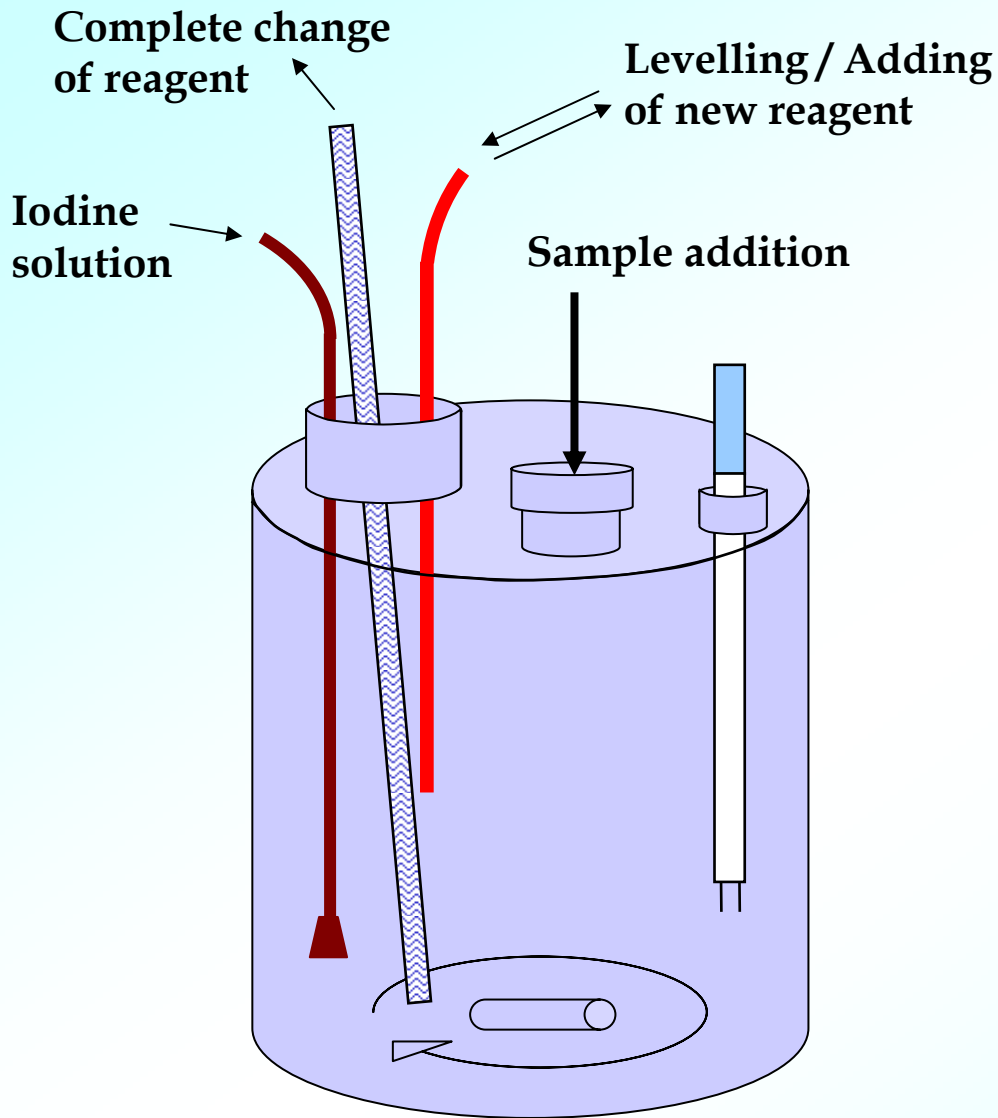
- generation of iodine from iodide; only required amount of iodine produced
- Hydranal Coulomat AD (Riedel-de Haen)
- Generator electrode without diaphragm, $I_{\text{pol}} 10 \mu\text{A} / 50 \text{ mV}$, generator current 400 mA



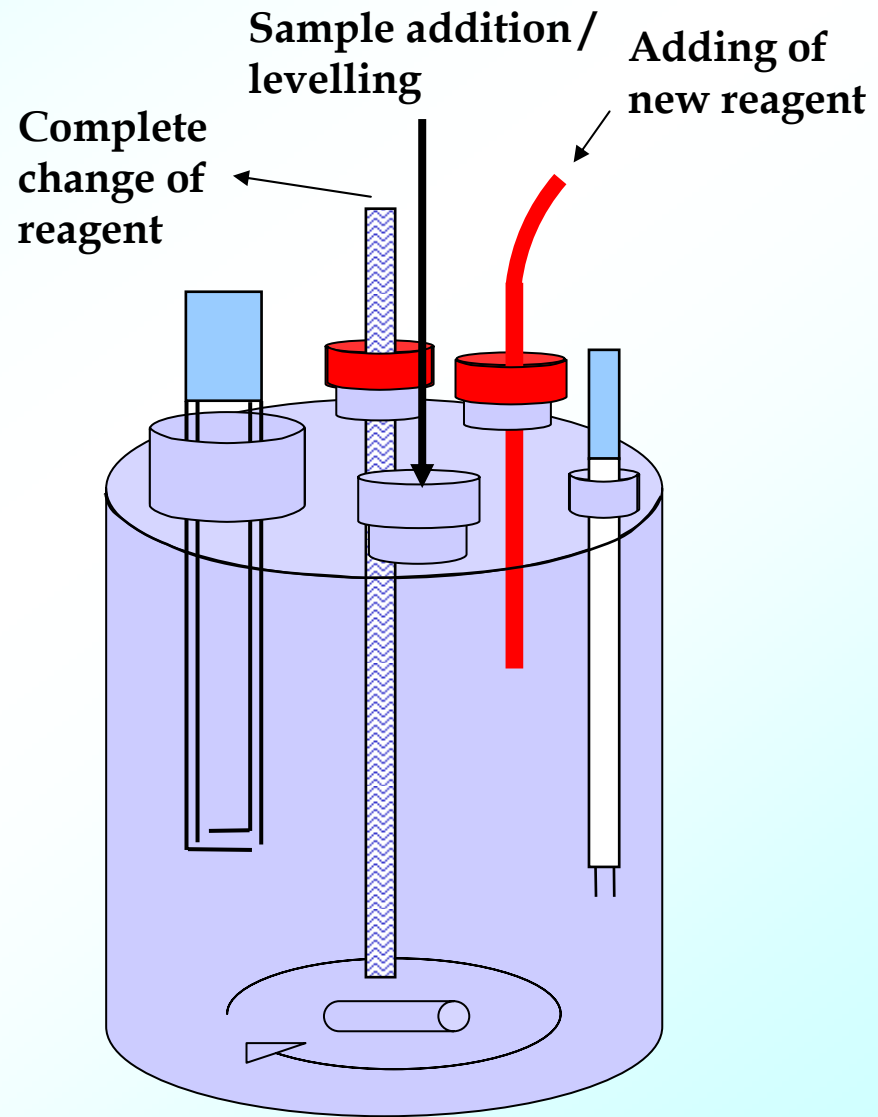
Automation of Karl Fischer Titration

- Automated pipetting, transferring, dispensing and dosing of samples and auxiliary liquids
- Operating sequences consist of different methods like blank values, water equivalent measuring and different sample treatments
- Improvement of reproducibility and accuracy



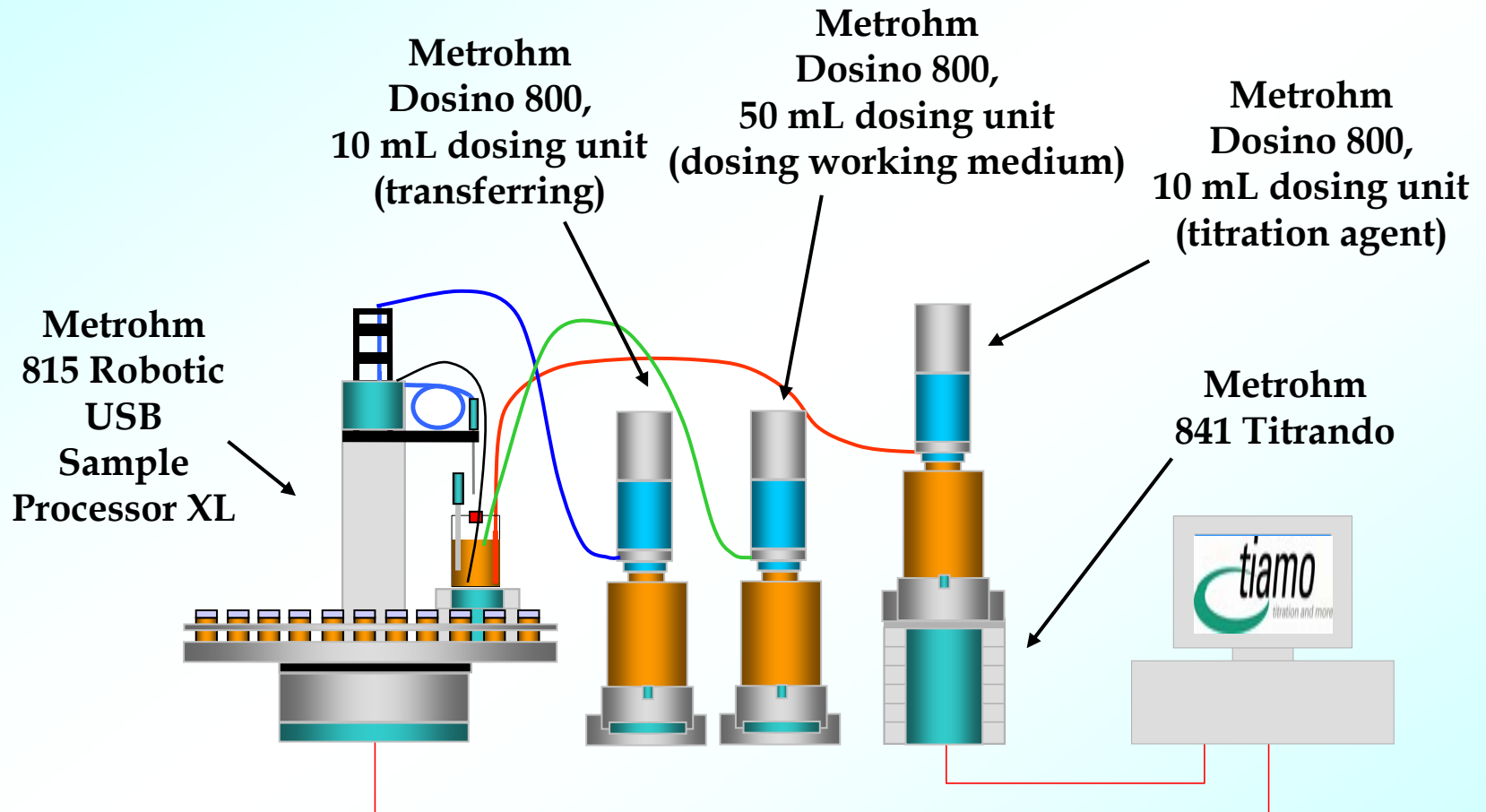


Volumetric cell

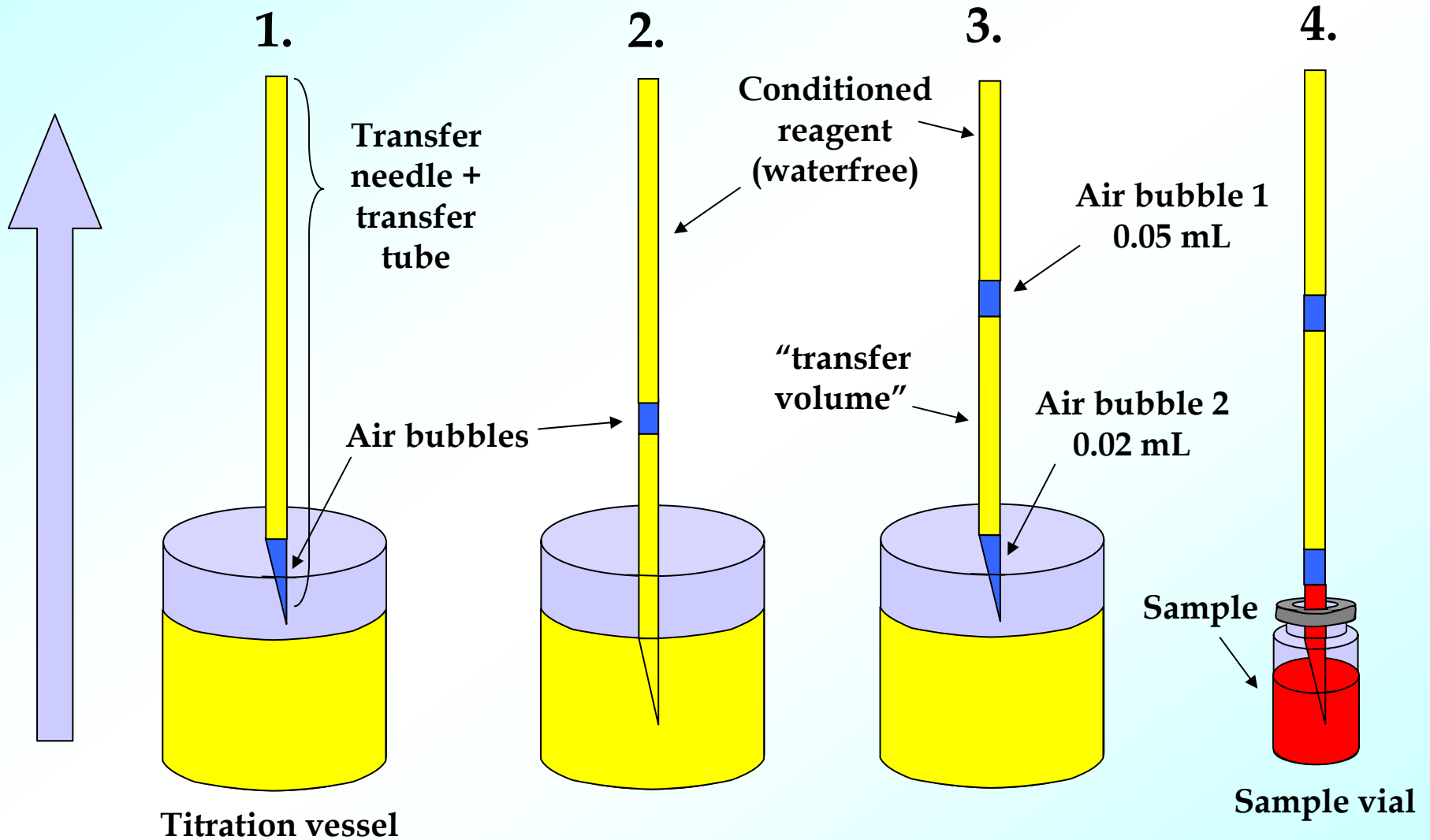


Coulometric cell

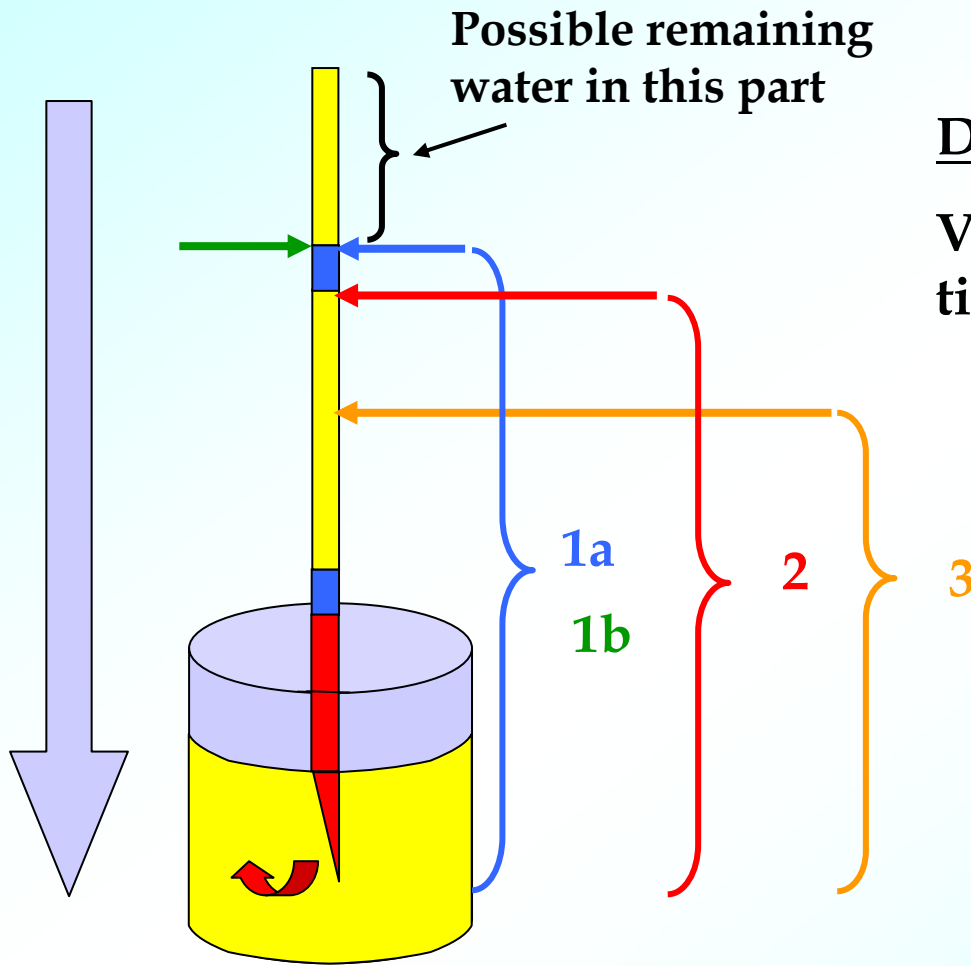
Schematic setup for automated volumetric titrations



Filling of the transfer tube / steps of aspiration



Transfer of liquids to titration vessel



Difference between the methods:

Varying injection volumes into the titration vessel

Aspiration and
transfer speed:

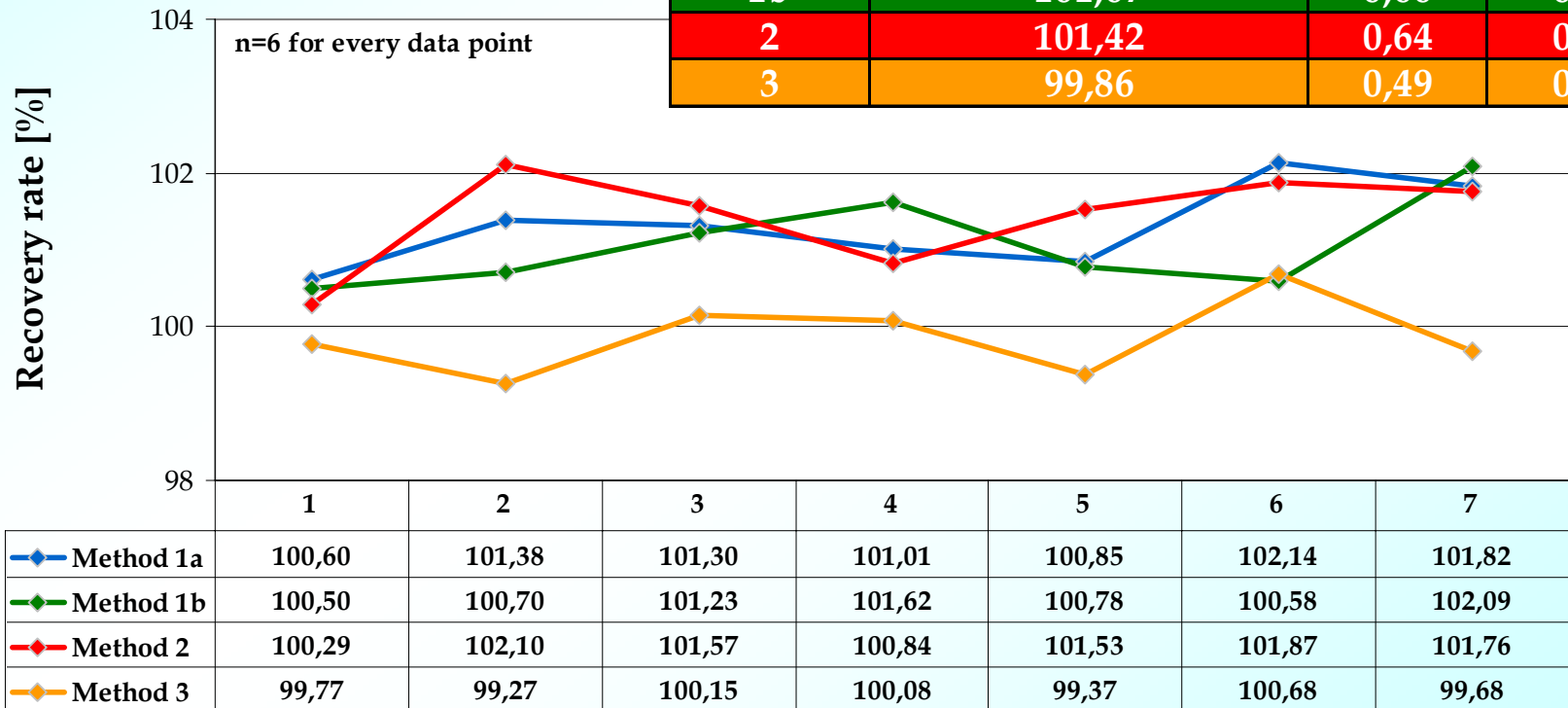
5 mL / min

Results of volumetric determinations / Standards

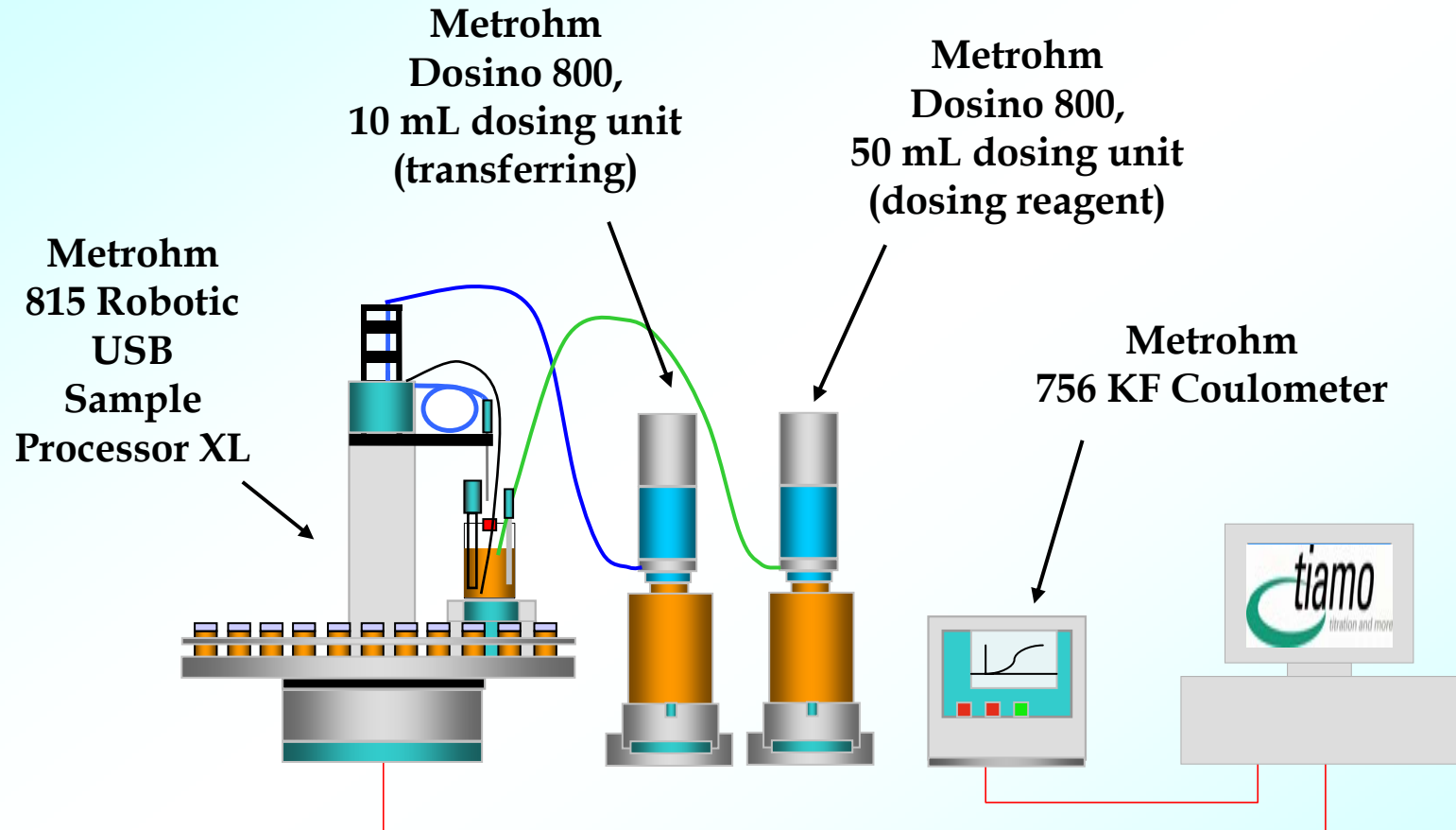
Hydranal Methanol Water Standard 5.00

Sample Volume 0.5 mL

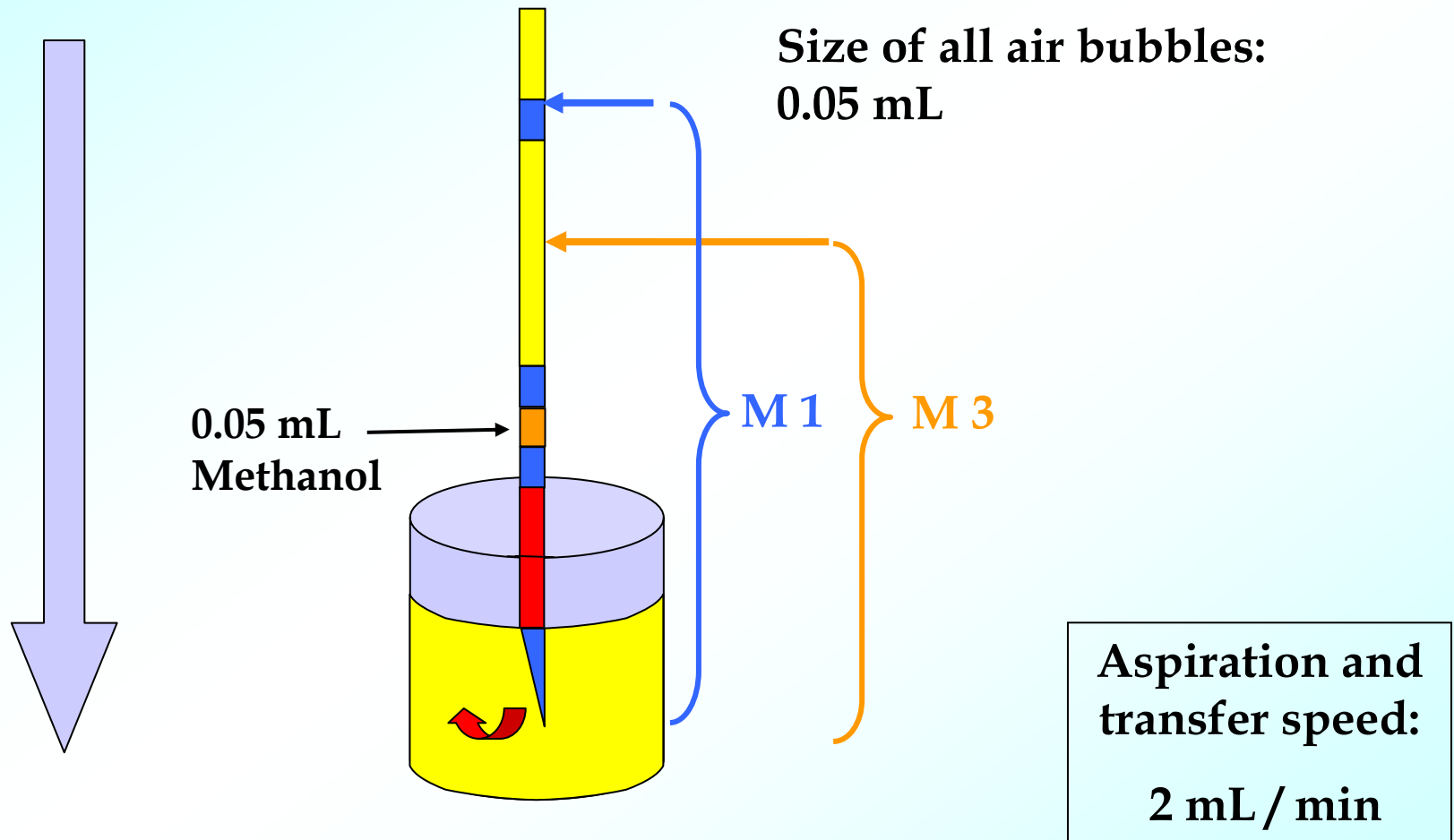
Method	Average Recovery rate	S (%)	RSD (%)
1a	101,30	0,54	0,5
1b	101,07	0,60	0,6
2	101,42	0,64	0,6
3	99,86	0,49	0,5



Schematic setup for automated coulometric titrations



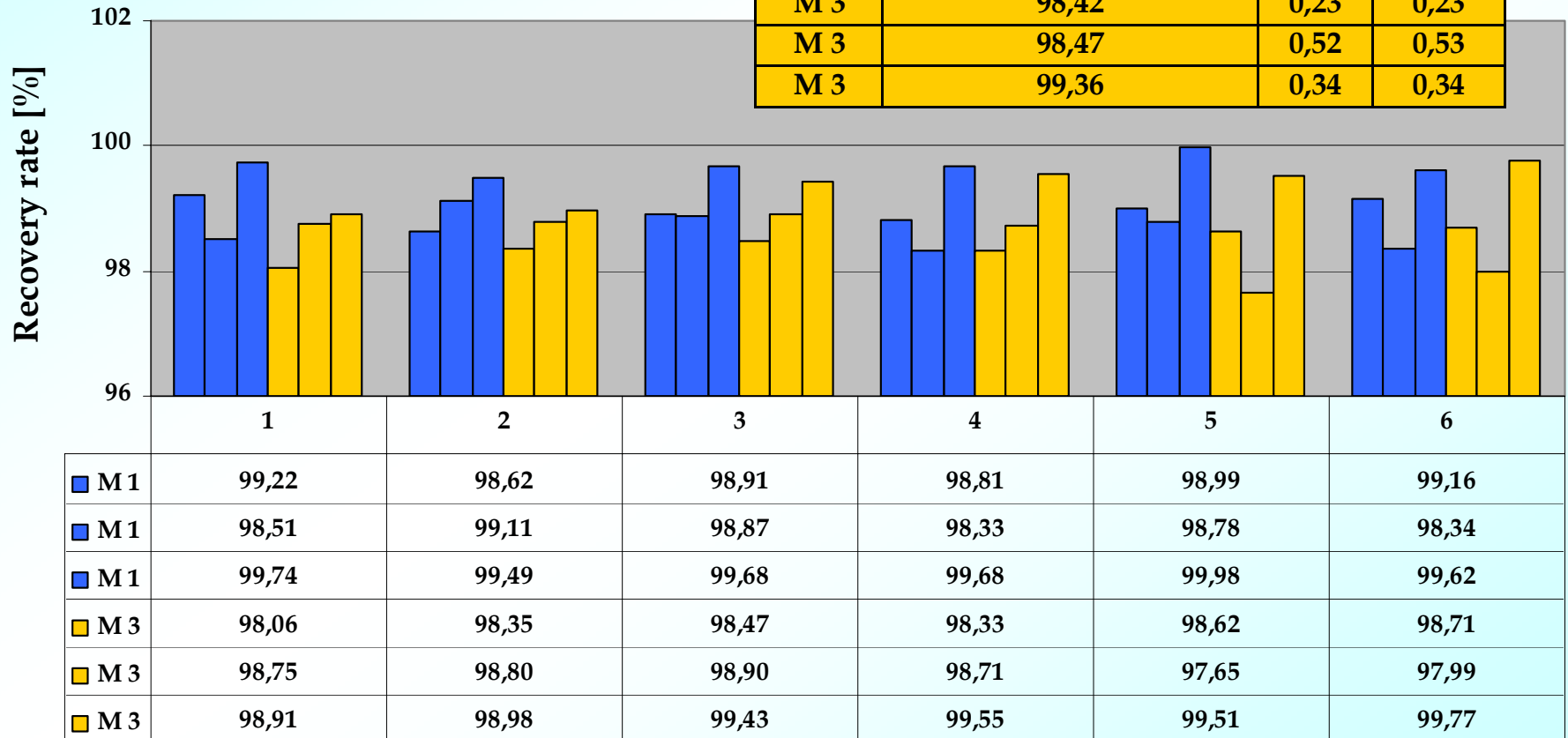
Transfer of liquids to titration vessel



Results of coulometric determinations / Standards

Hydranal Water Standard 1.00
Sample Volume 1 mL

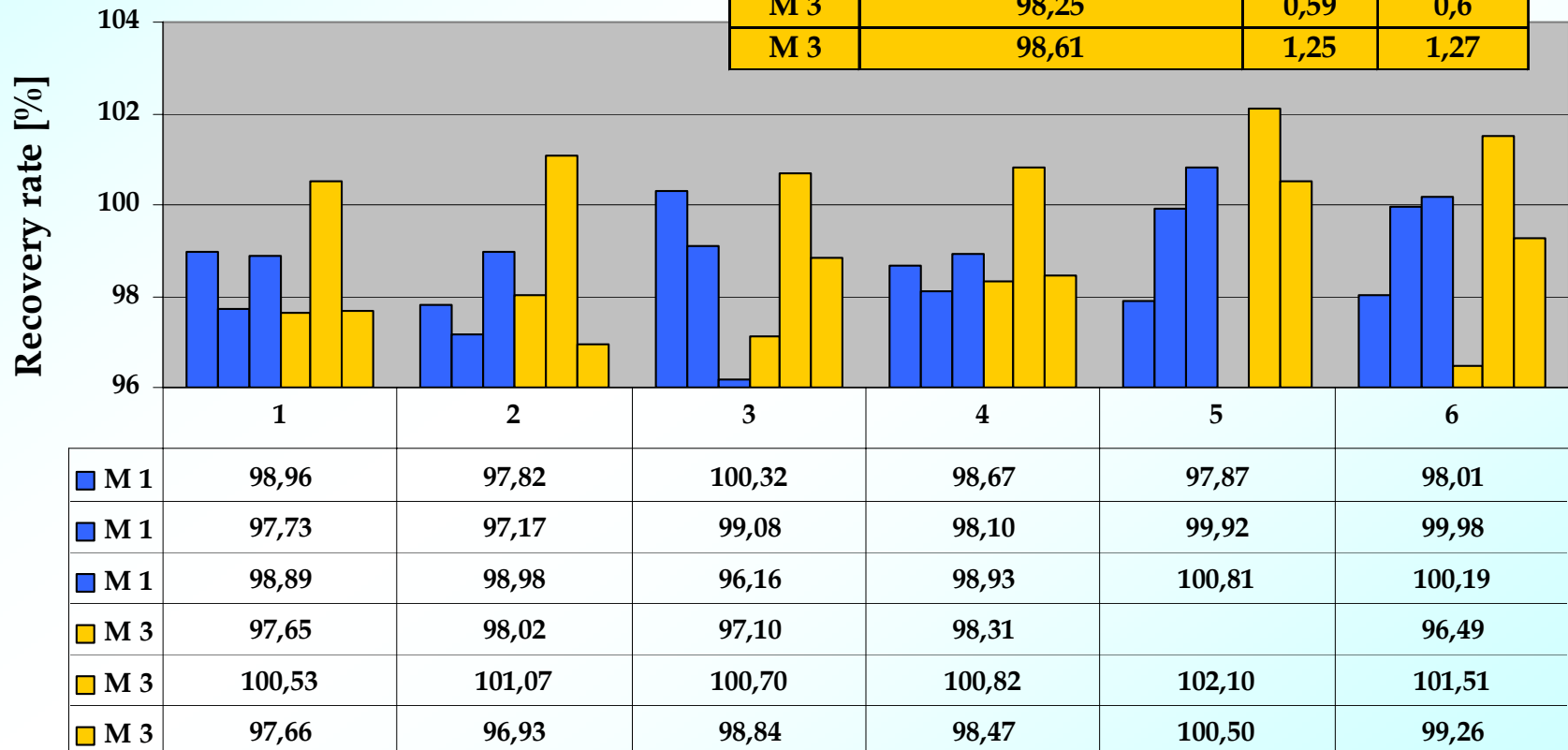
Method	Average Recovery rate (%)	S (%)	RSD (%)
M 1	98,95	0,22	0,22
M 1	98,66	0,32	0,32
M 1	99,70	0,16	0,16
M 3	98,42	0,23	0,23
M 3	98,47	0,52	0,53
M 3	99,36	0,34	0,34



Results of coulometric determinations / Standards

Hydranal Water Standard 0.10
Sample Volume 2 mL

Method	Average Recovery rate (%)	S (%)	RSD (%)
M 1	98,61	0,96	0,97
M 1	98,66	1,18	1,19
M 1	98,99	1,60	1,61
M 3	97,09	1,22	1,26
M 3	98,25	0,59	0,6
M 3	98,61	1,25	1,27



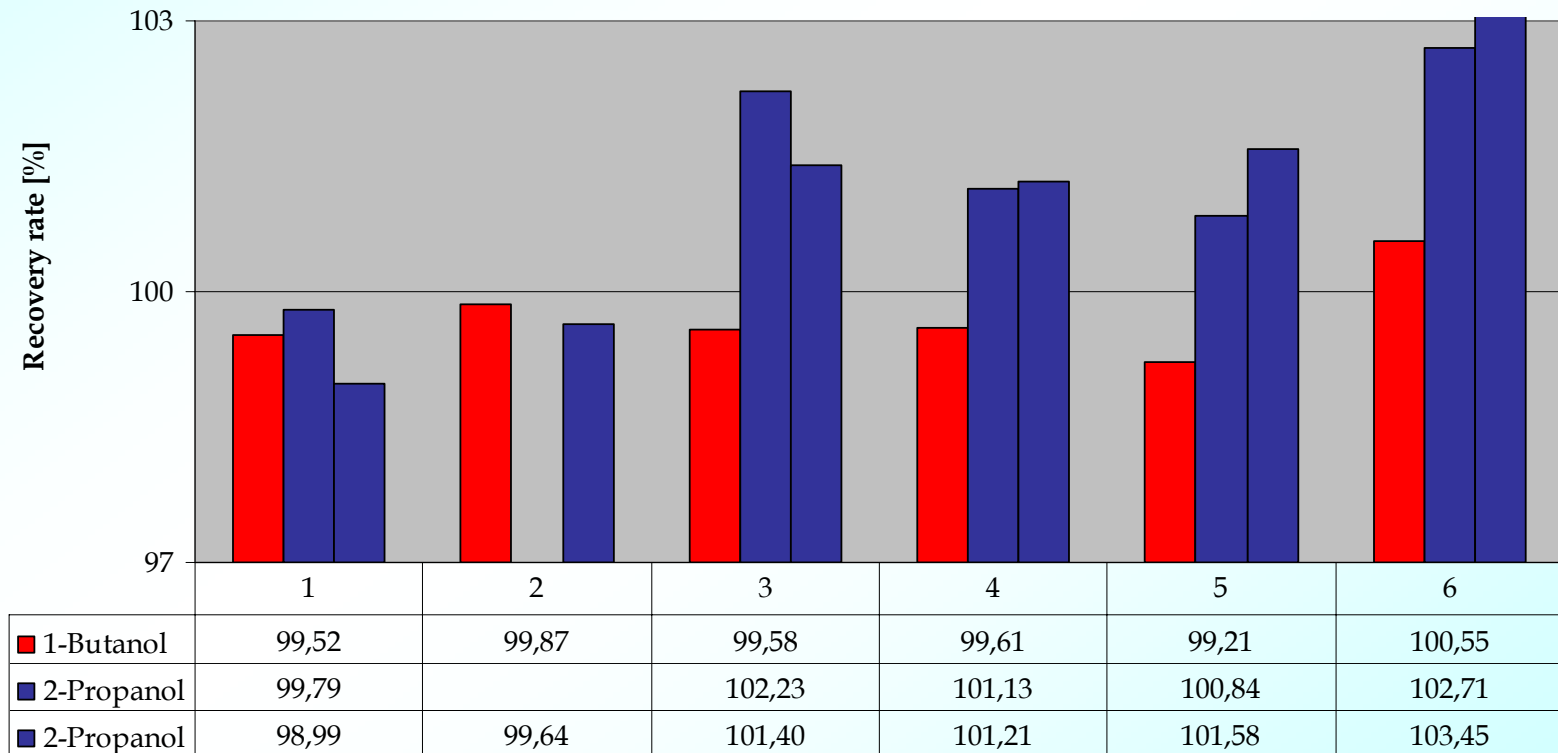
Results of coulometric determinations / Solvents

Sample Volumes:

1-Butanol: 2 mL (approx. 300 µg water)

2-Propanol: 0,5 mL (approx. 150 µg water)

Solvent	Average Recovery rate (%)	S (%)	RSD (%)
1-Butanol	99,73	0,46	0,46
2-Propanol	102,15	2,23	2,18
2-Propanol	101,05	1,58	1,56



Thank you for your attention !

