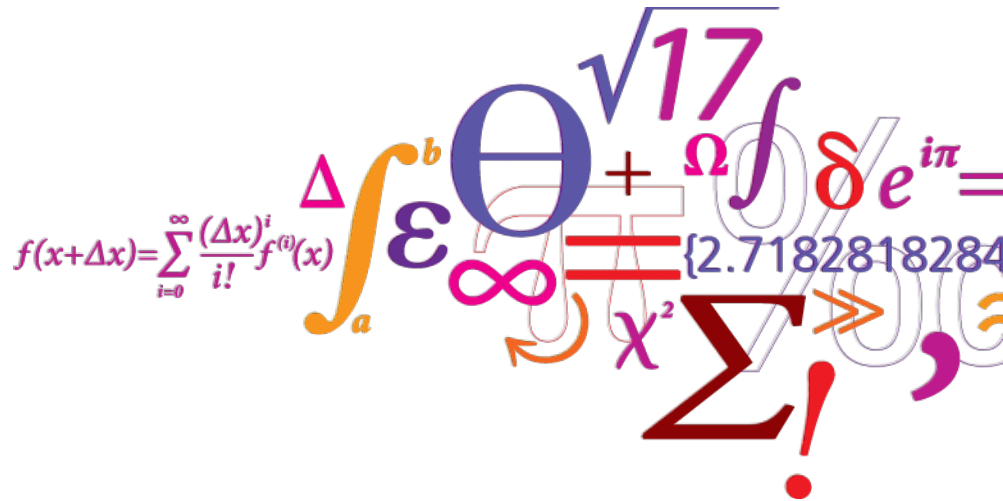


Application of Rapid and Automated Techniques in Radiochemical Analysis

---Inspirations from NKS-B Rapid-Tech Project

Jixin Qiao



NKS-B Rapid-tech project [AFT/B(14)7]

- Funded by Nordic Nuclear Safety Research (NKS) for 2014-2016

• Partners:

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Project objectives

- To explore the application of different rapid techniques in determination of radionuclides, thus to improve the analytical efficiency of present radioanalytical methods in the areas of E, W and R.
- Specific tasks:
- Identification of current needs and problems in methodology development for rapid determination of ^{90}Sr and actinides.
- Identification of individual processes wherein rapid techniques can be potentially applied to improve the analytical efficiency.

Our focus

No.	Rapid techniques
1	Flow injection (FI)/sequential injection (SI)
2	High performance liquid chromatography (HPLC)
3	Vacuum box system
4	On-line detection (FI-ICPMS, HPLC-ICPMS)
5	other effective sample treatment approaches (microwave assisted digestion, alkaline fusion, co-precipitation)

Rapid radiochemical analysis

Flow/sequential injection chromatographic separation:

Flow injection (FI) system:

- Peristaltic pump
- Continuous flow

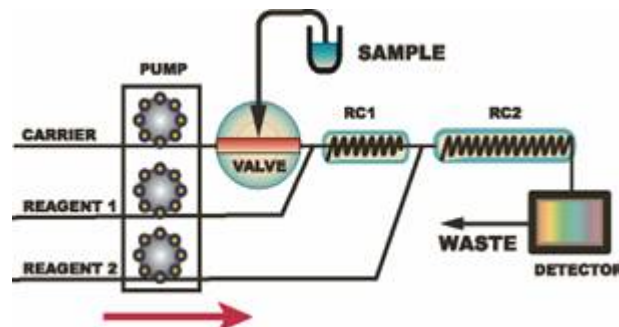


Fig. Scheme of a FI system

Sequential injection (SI) system:

- Syringe pump
- Selection valve

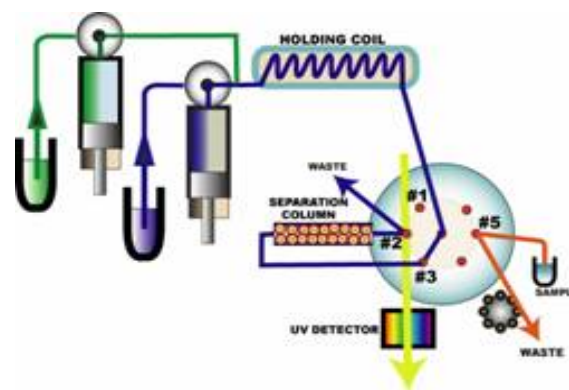


Fig. Scheme of a SI system

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Rapid radiochemical analysis

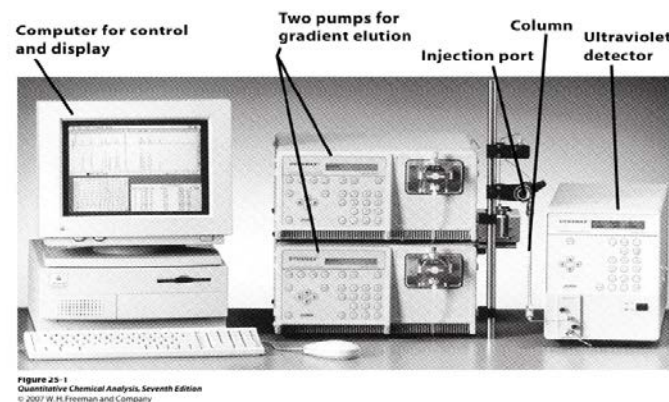
High performance liquid chromatography (HPLC):

Advantages:

- Fully automated
- Can be connected directly with MS

Disadvantages:

- Only handle small samples
- Single sample processing
- High cost



HPLC system

Our focus

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Rapid radiochemical analysis

Vaccum box:

Advantages:

- Multi-sample processing
- Easy operation
- Low cost
- Flexible

Disadvantages:

- Need human attention



Eichrom vacuum box

Our focus

No.	Rapid techniques
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Rapid radiochemical analysis

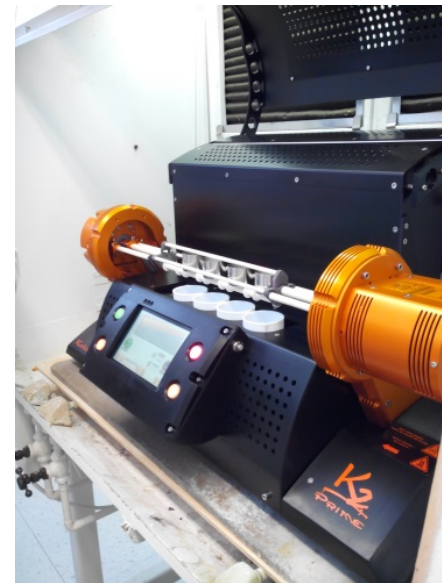
Alkaline fusion --- Rapid sample pre-treatment techniques for solid samples:

Fluxes can be used for alkaline fusion:

- NaOH
- Na_2O_2
- NaCO_3
- LiBO_2
- Others



Busen burner

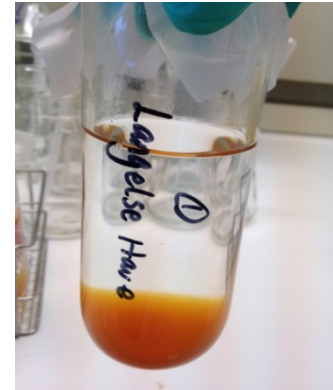


Katanax automatic electric fluxer

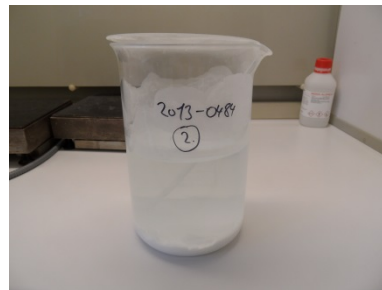
Rapid radiochemical analysis

Co-precipitation --- Rapid sample pre-treatment for liquid samples:

- Carbonates (e.g., CaCO_3)
- Oxalates (e.g., CaC_2O_4)
- Hydroxides (e.g., $\text{Fe}(\text{OH})_3$)
- Oxides (e.g., MnO_2)
- Phosphates (e.g., $\text{Ca}_3(\text{PO}_4)_2$, BiPO_4)
- Others (e.g., AMP for Cs)



$\text{Fe}(\text{OH})_3$ co-precipitation



CaCO_3 co-precipitation

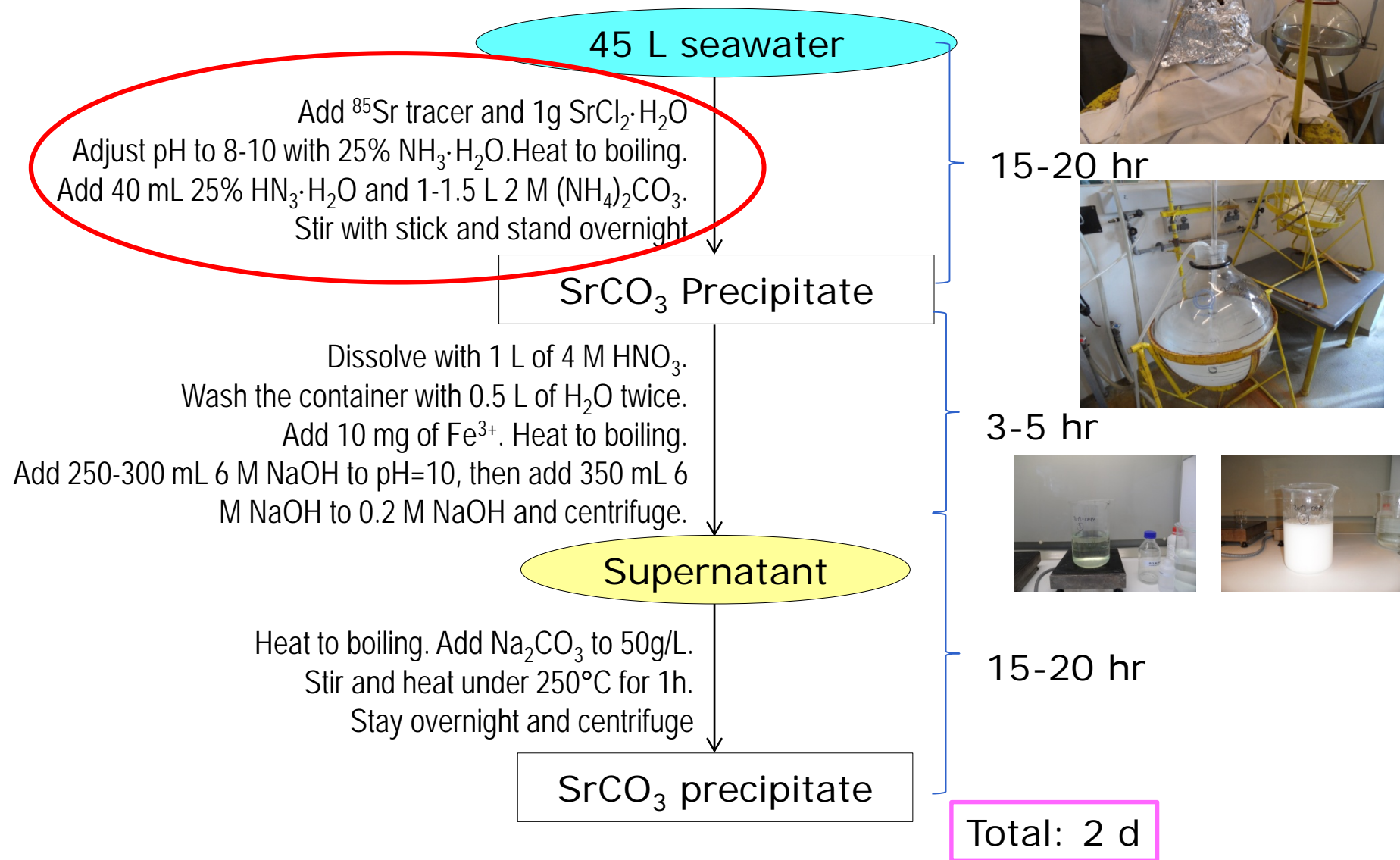


CaC_2O_4 co-precipitation

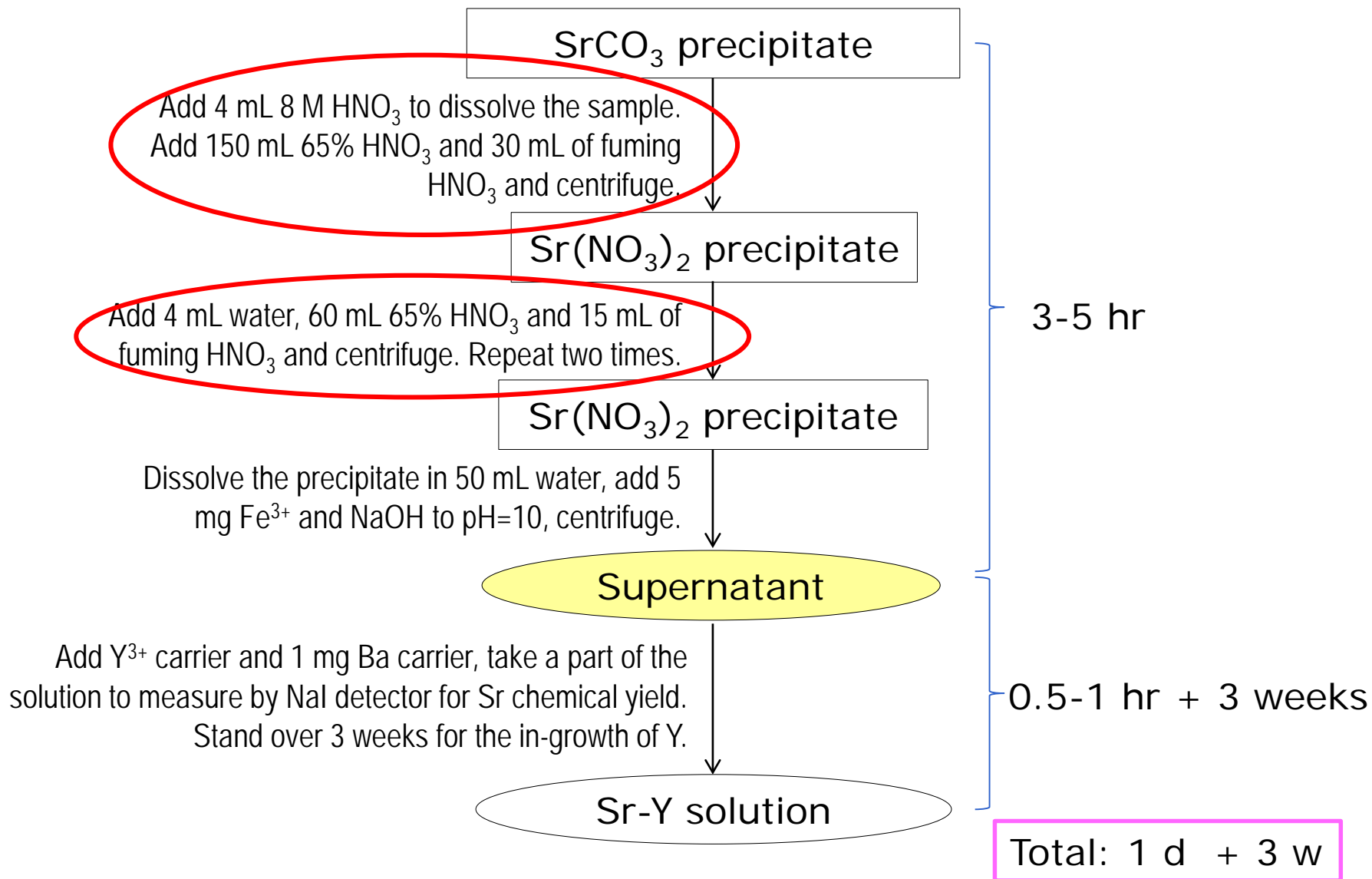
NKS-B Rapid-tech project 2014-2015

- Current analytical methods for Sr and actinides were screened among participating institutes.
- Conclusions:
 - 1) Current application of novel automated techniques in Nordic countries is very limited.
 - 2) There is a need for end users to become more aware of the advantages of improved techniques for radiochemical assays.
- **Example: ^{90}Sr determination for 45 L seawater (DTU-Nutech routine analysis)**

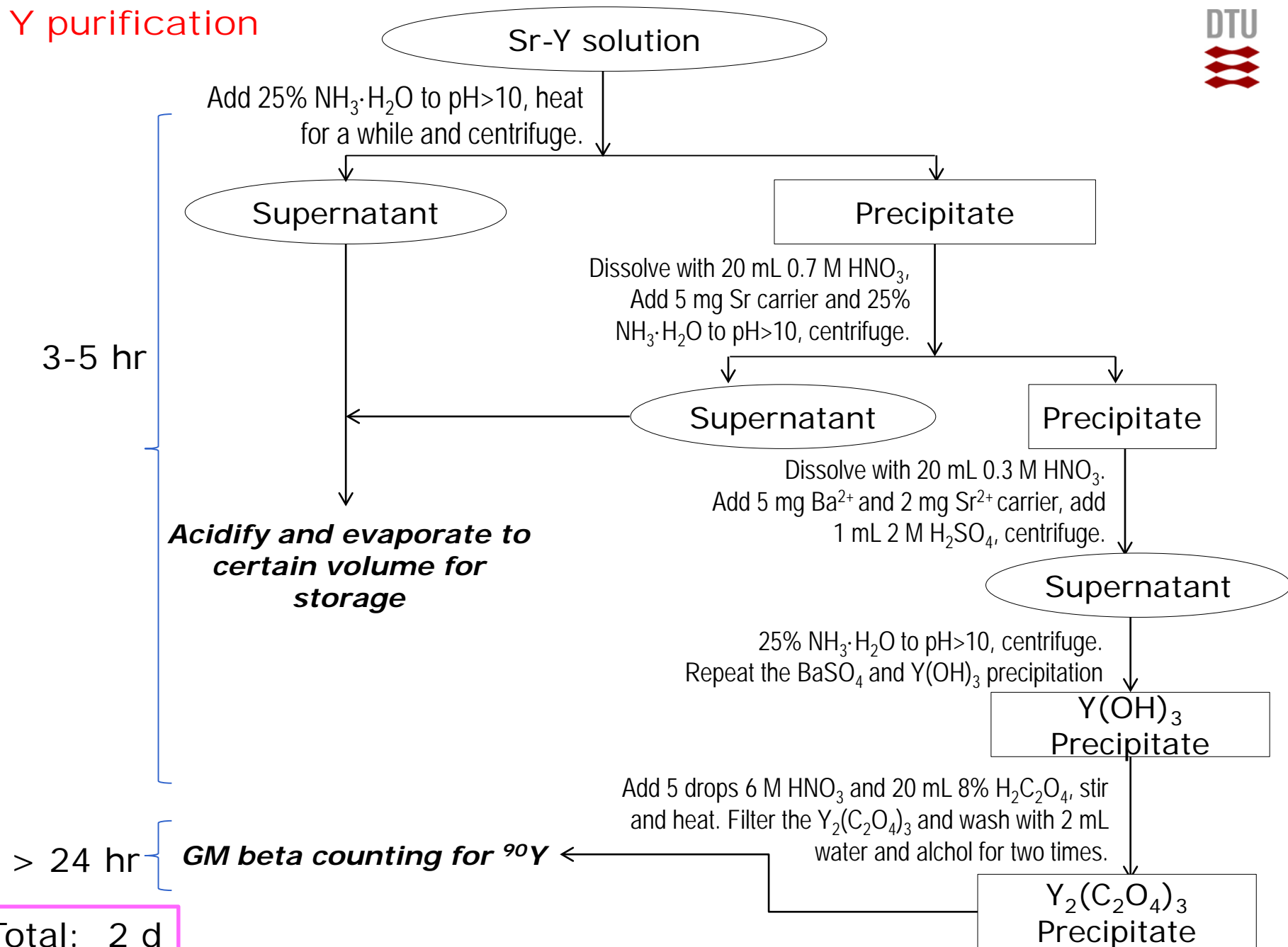
Pre-concentration and preliminary Ca removal



Further Ca removal and purification



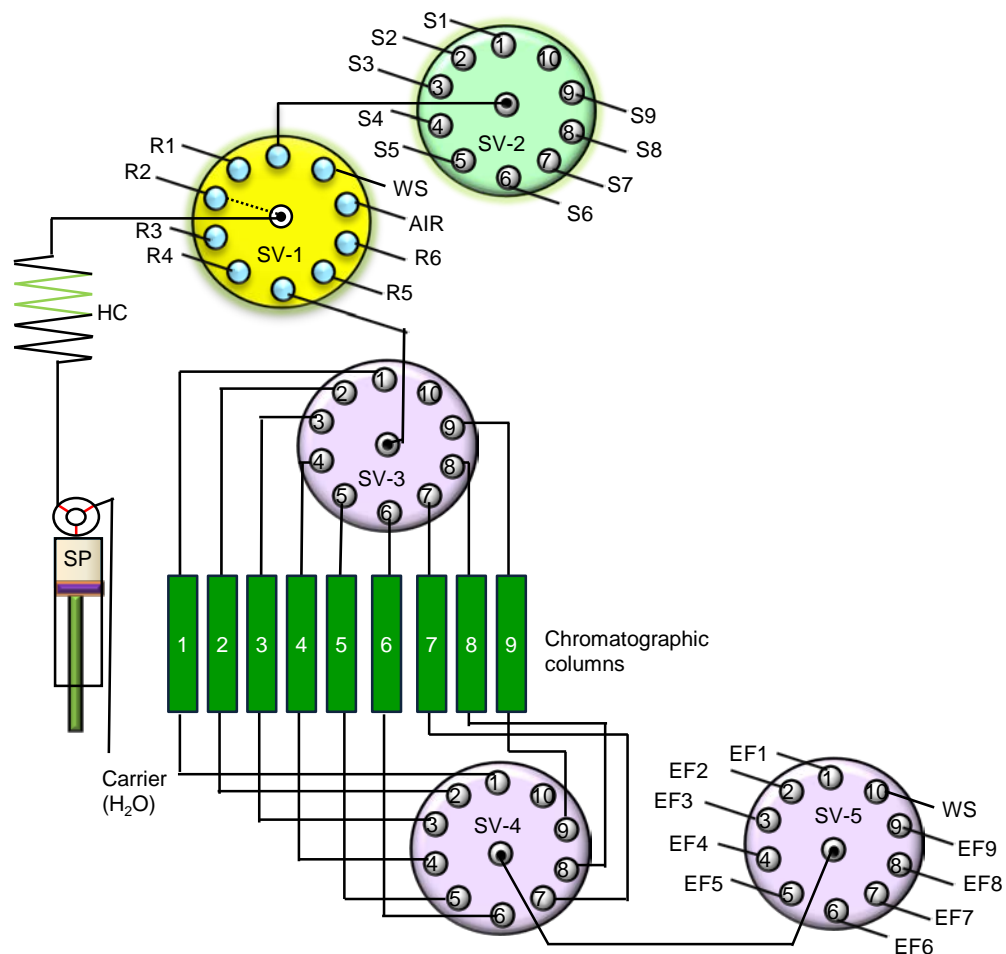
Y purification



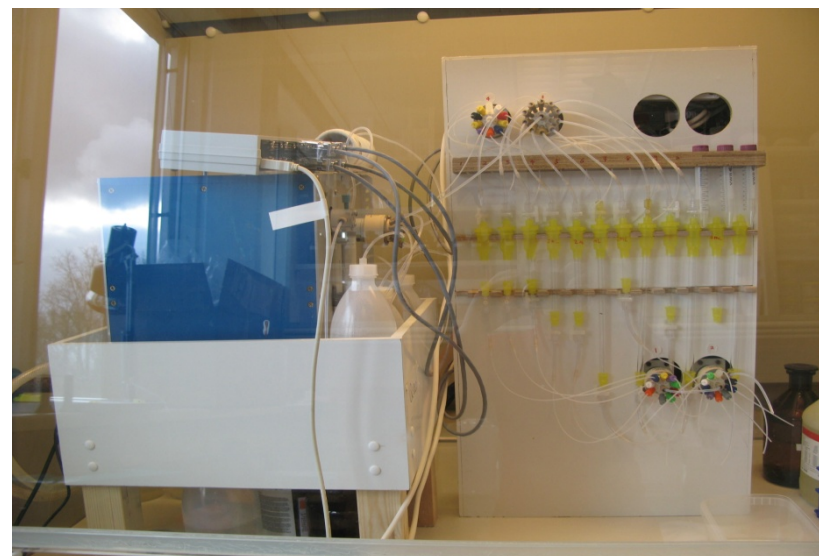
Performance evaluation and potential improvement

Advantages		
Robust, high applicability, high precision and accuracy, relatively low LOD (5 mBq)		
Disadvantage		
Item	Detailed problem	Possible solution
Low safety factor	highly toxic chemical $(\text{NH}_4)_2\text{CO}_3$	CaC_2O_4 co-precipitation, room temp.
	noxious and corrosive fuming nitric	Repeated application of NaOH for removal of Ca
Low efficiency (Analytical turnover >5 days excluding 3-week ingrowth of ^{90}Y)	Repeated $\text{Sr}(\text{NO}_3)_2$ precipitation	Application of Sr resin
	Long waiting time for ^{90}Y ingrowth	Direct counting of ^{90}Sr by LSC
High labor intensity	Manual operation	Flow/sequential injection

Sequential injection unit

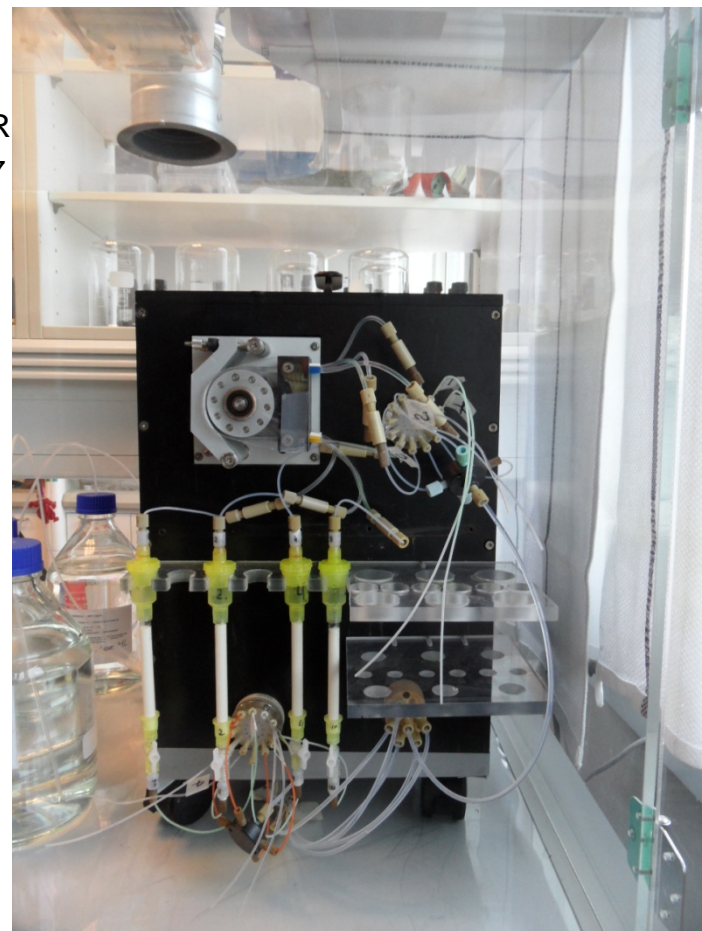
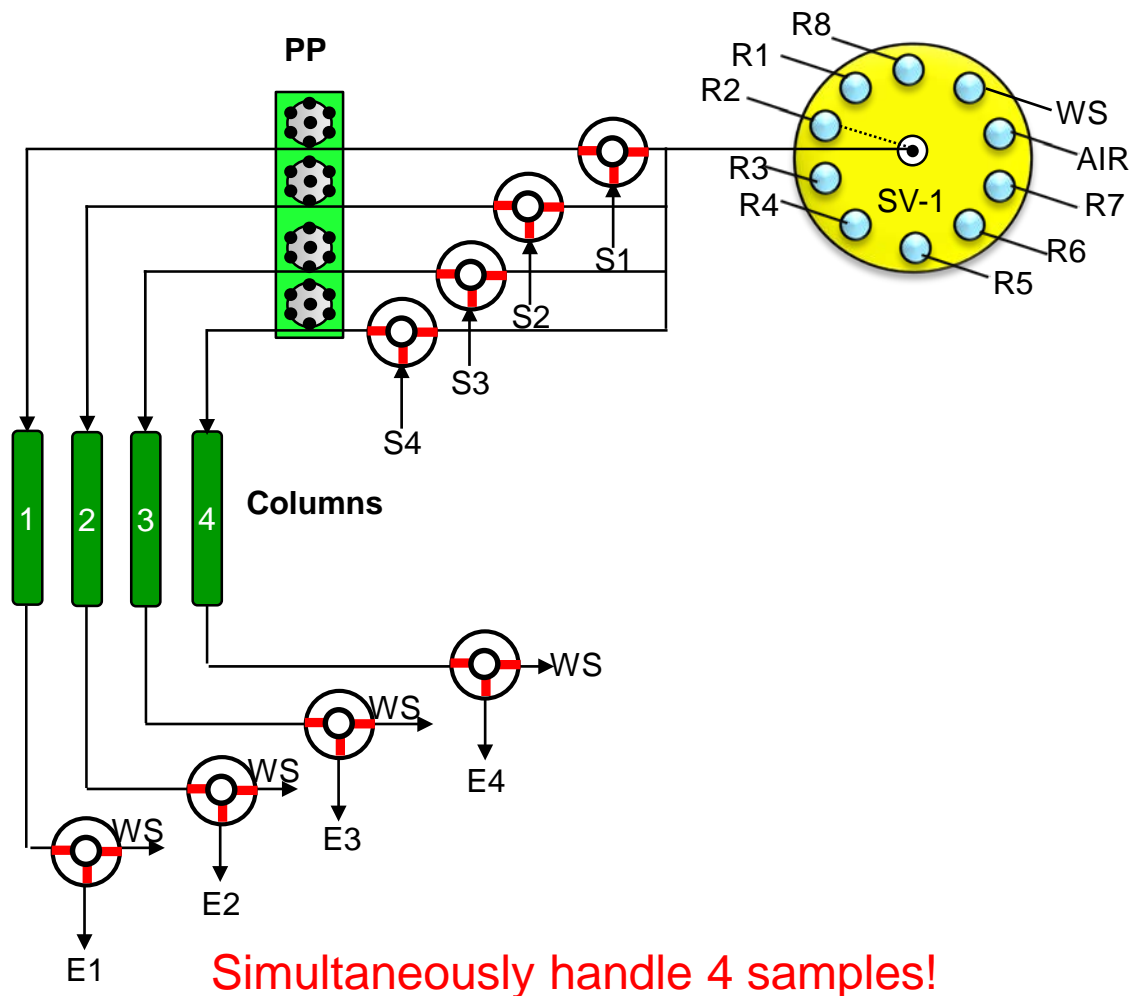


Automatically handle 9 samples!
Work overnight !



Qiao, J. X., Hou, X. L., Roos, P., Miró, M. *Analytica Chimica Acta*. 2011.

Flow injection unit



Qiao, J. X., Shi, K. L., Hou, X. L., Nielsen, S., Roos, P. Environmental Science & Technology. 2013.

Conclusions

- More efforts are needed to improve the application of automated and rapid techniques in radiochemical analysis
- More support from NKS or other foundations for future projects
- More communication and collaboration among Nordic and international labs

Thank you!