

# Aging of a $\text{Li}_2\text{B}_4\text{O}_7$ -glass - 5 years of analysis

Dr. Elke Thisted  
Elkem Solar AS Research



# How did the idea start?



## Användarmöte Röntgenfluorescens

Stockholm 19.-20.09.2005

### Impurities from the tube

- possible elements Ni, Zn, Cu, Fe,
- use glasstablet
- analyse everyday



### Stability of a boratebased glass tablet

- "no stable Na-analysis"
- "change in Cl and S over time"

# Where was it for the last 5 years?



- placed on the X-tray of our PW2404
- supposed to be analysed daily



# Parameters

- Channelsettings

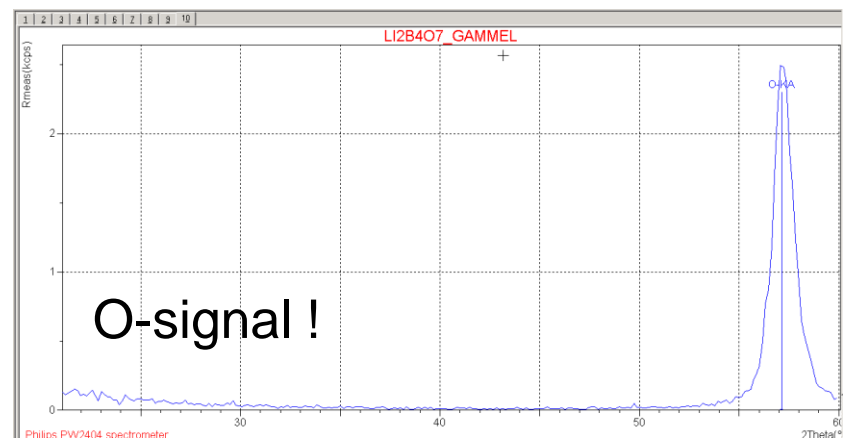
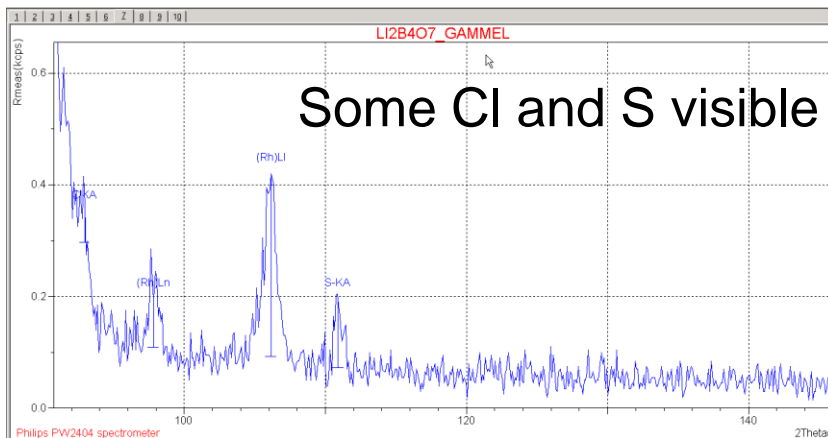
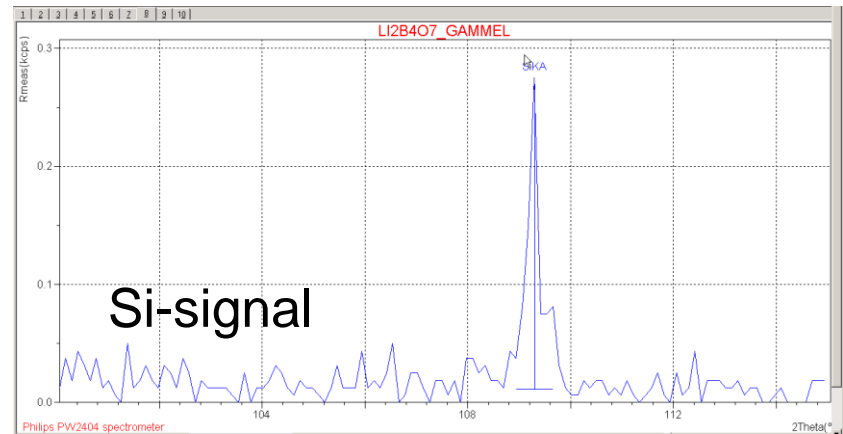
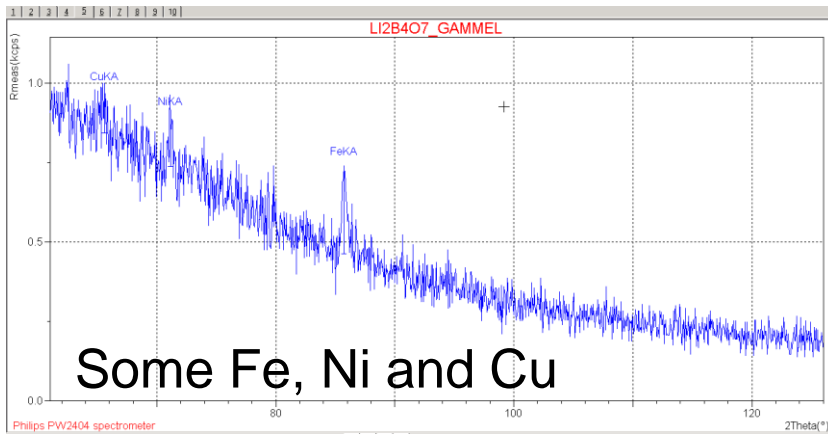
Channel	Type	Line	X-tal	Collimator	Detector	Tube filter	kV	mA	Angle (°2T)	Offset Bg1 (°2T)	Offset Bg2 (°2T)
Fe	Gonio	KA	LiF 200	300 µm	Flow	None	60	66	57.4960	-0.6532	
Ni	Gonio	KA	LiF 200	150 µm	Flow	None	60	66	48.6792	-0.4030	
Cu	Gonio	KA	LiF 200	150 µm	Flow	None	60	66	45.0250	0.6006	
Zn	Gonio	KA	LiF 200	300 µm	Scint.	None	60	66	40.0150	0.8260	2.5602
W	Gonio	LA	LiF 200	300 µm	Flow	None	60	66	43.0026	-0.3074	
Na	Gonio	KA	PX1	700 µm	Flow	None	32	125	23.0928		
S	Gonio	KA	Ge 111-C	300 µm	Flow	None	32	125	110.6850		
Cl	Gonio	KA	Ge 111-C	300 µm	Flow	None	32	125	92.7672		
Rh1	Gonio	KA	LiF 200	300 µm	Scint.	None	60	66	17.5636		
Rh2	Gonio	LA	Ge 111-C	300 µm	Flow	None	60	66	89.4982		

Channel	PHD1 LL	PHD1 UL
Fe	15	60
Ni	18	59
Cu	20	58
Zn	15	71
W	23	61
Na	16	78
S	27	78
Cl	28	77
Rh1	27	78
Rh2	29	76

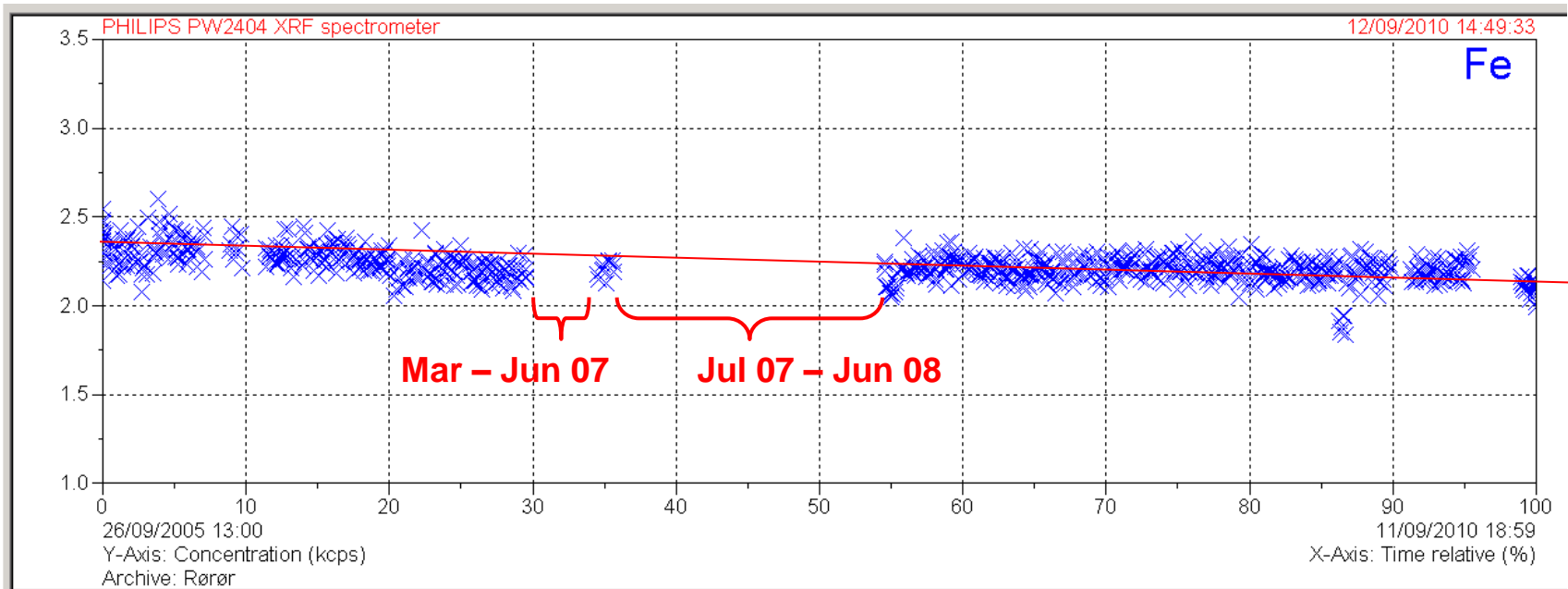
- checked angles + PHD – proper preparation of settings for Fe, Ni, Cu, Zn and W – started 26.09.2005
- quick & dirty setup: Na – started 17.01.2006; Cl and S – started 13.06.2006

- Instrument – most likely the same Rh-tube since 3.10.2003
- Tablet not cleaned with Ethanol before 11.09.10!

# How does the $\text{Li}_2\text{B}_4\text{O}_7$ -tablet look when scanned in Sept 05?

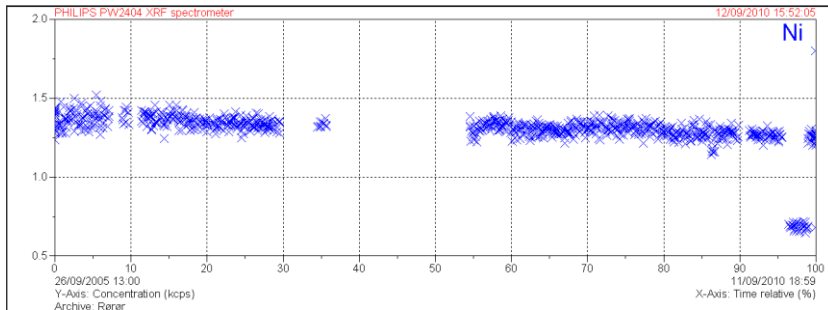


# Running the tablet for 5 years:

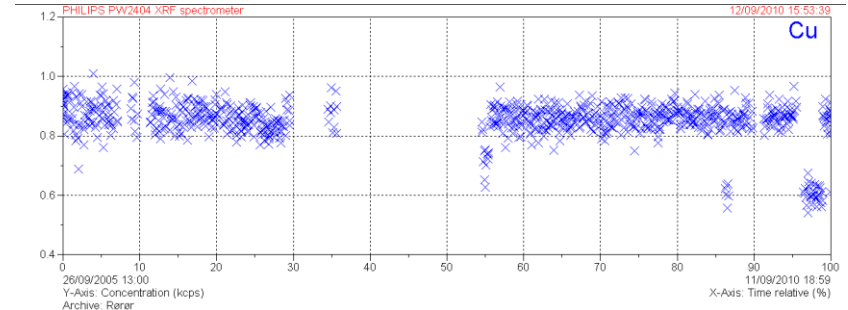


- Some time periods no results (sample erased from sample list or wrong sample measured)
- trend over time – slowly decreasing; reason: several possibilities, but nothing definite

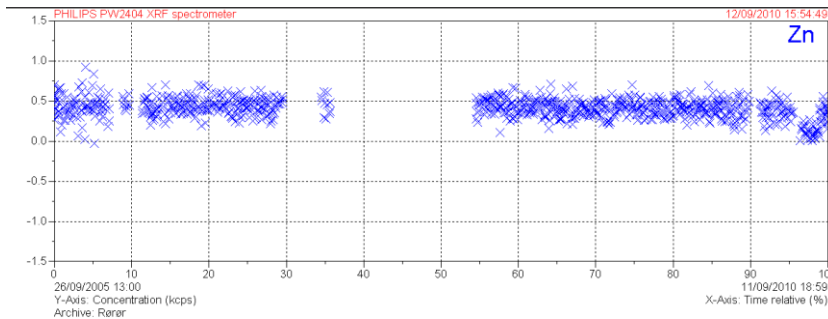
# Running the tablet for 5 years (2):



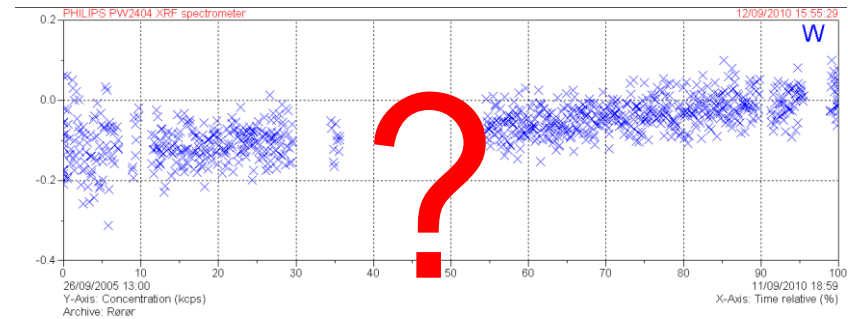
Ni: also slowly decreasing



Cu: seems rather stable

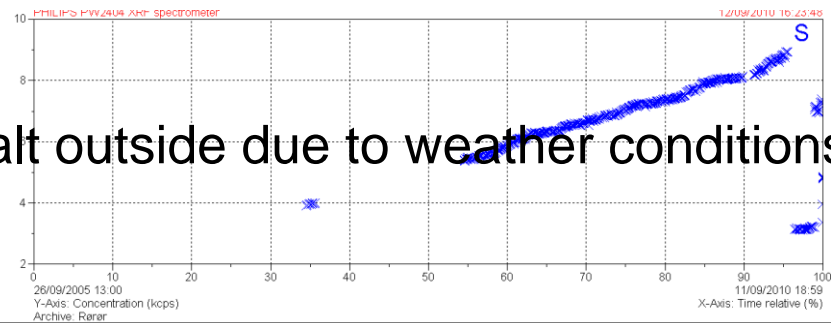
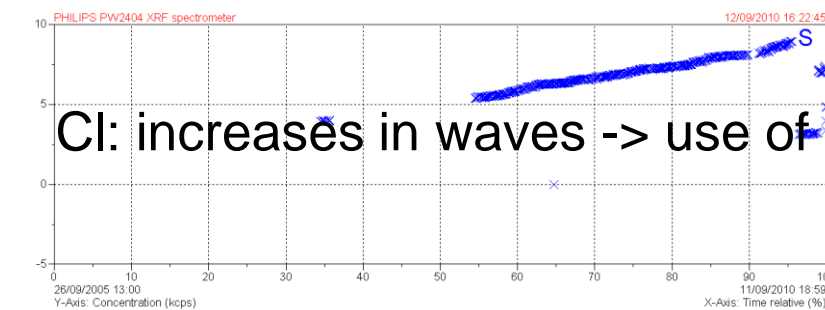
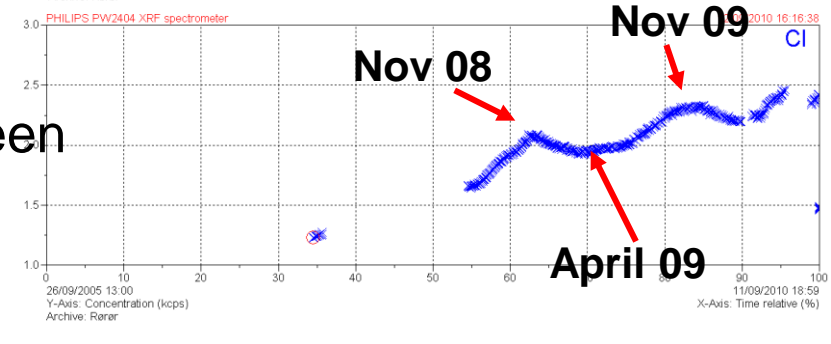
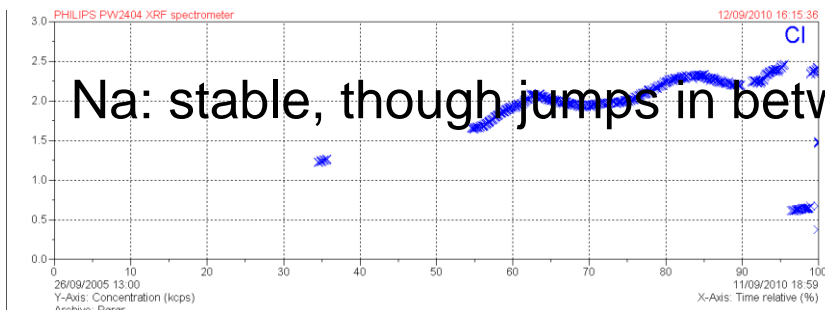
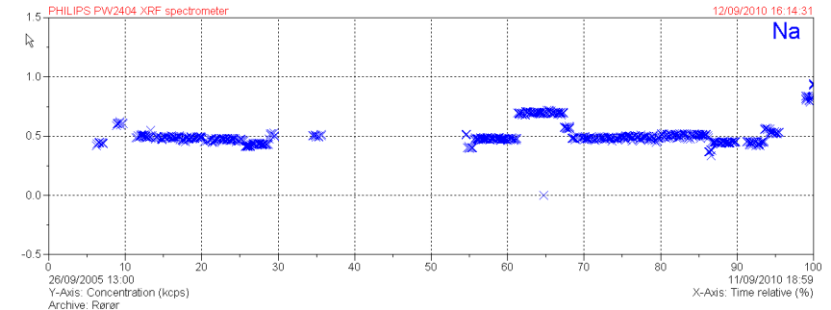
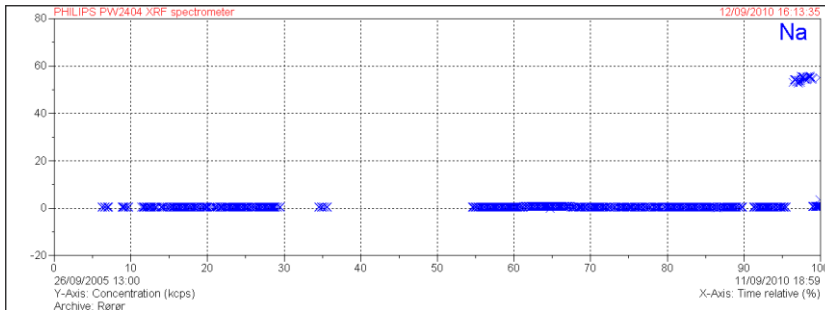


Zn: seems rather stable



W: increasing? Nettosignal yes, but due to faster decrease in signal in the bk- than in the top-position

# Running the tablet for 5 years (3):



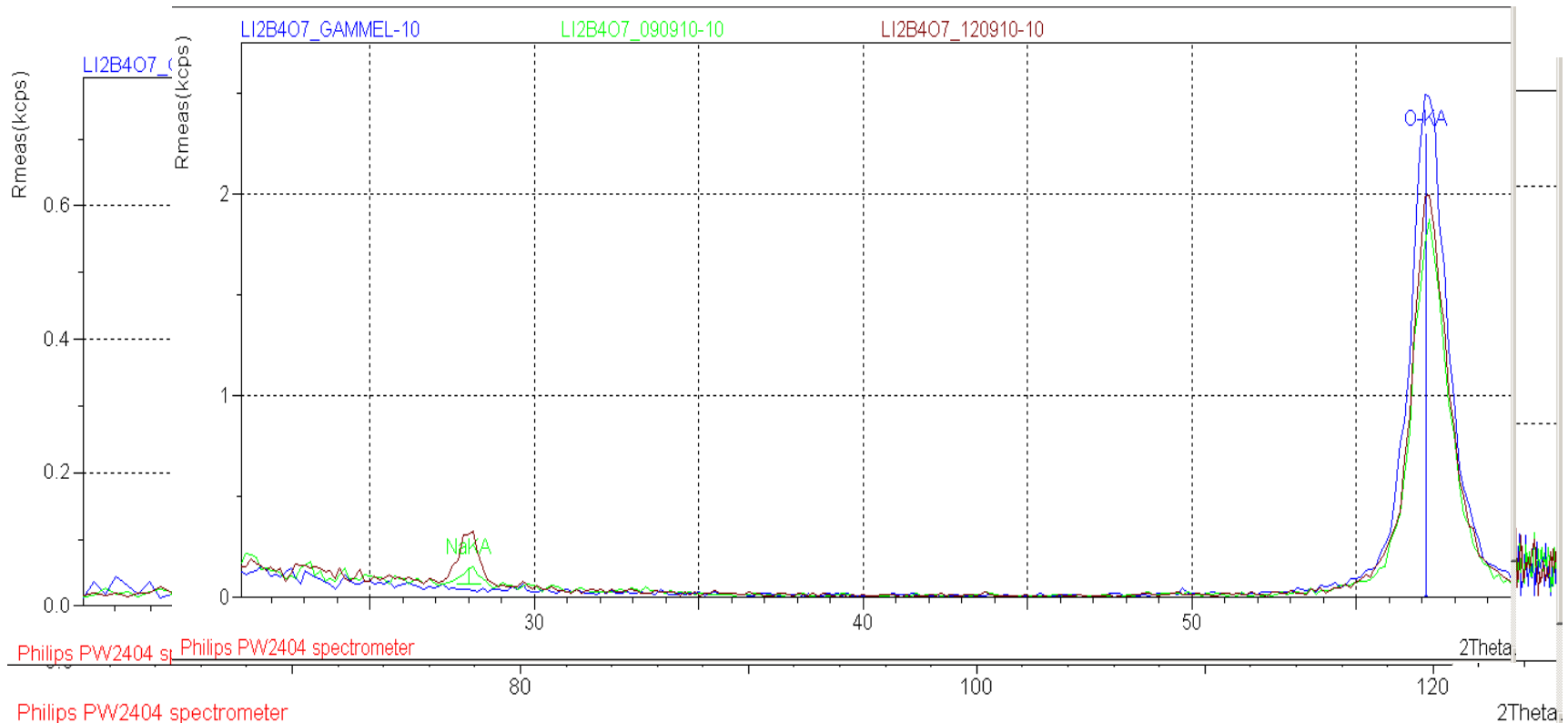
Na: stable, though jumps in between

Cl: increases in waves -> use of salt outside due to weather conditions

S: steady increase over the past 5 years

# How does the $\text{Li}_2\text{B}_4\text{O}_7$ -tablet look when scanned now?

Na appears in 5 years, cleaned over 50% by Ethanol  
Si increases in 5 years, drops to original signal after cleaning  
Stable Fe, Ni and Cu



# Possible explanations/speculations:

- Part of the Cl-increase from salt used when winter comes ( $\text{NaCl}$ ,  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ) – but constant  $\text{Na}^+$  ?
- Only effects lighter elements due to analysis volume – but why is Na "stable"
- Does  $\text{Na}^+$  go into the borate network and  $\text{Cl}^-$  stays outside due to ionic radius?



– Picture from <http://www.chemguide.co.uk/atoms/properties/atradius.html>

- Where does the S comes from?

# Conclusion and further work

---

- No permanent control samples from borate at our lab until further investigations are done (at least not for Cl and S)
- Make one new or a new set of borate tablets to run for the next 5 years including more elements, proper setup for all elements
- Use also Ca – more input from you ??
- Any suggestion is welcome – literatur advice?

**Thank you for your attention!**