DTA + IR + BET Characterisation of the ALBUM samples

Stephan Kaufhold & Reiner Dohrmann

Analyses performed

XRF,
XRD (powder),
CEC (Cu-Triene),
DTA,
IR,
BET,
LECO

Missing:
XRD coarse fractions
XRD clay fraction (texture slides)
XRD Rietveld calculation (AQ®)
CEC (AgTu, $BaCl_2$)
LCD (alkylammonium)

XRD powder

	JNB Album ´Kunigel V1´ Bentonite *Japan*	CAN Album ´Deponit Ca N´ Bentonite *Milos?*	ROK Album ´Rokle´ Bentonite *CzechRepl.*	CAL Album ´Calcigel´ Bentonite *Bavaria?*	COB Album ´Callovo Oxf.´ Clay *Bure, F*	IBE Album ´IBECO Seal´ Bentonite *??*	LOT Album ´MX 80´ Bentonite *Wyoming, US*	ASH Album ´Asha 505´ Bentonite *India*	FRI Album ´Friedland´ Clay *Germany*	FEB Album ´Febex´ Bentonite *Almeria, Sp.*	IKO Alb ´Ikoson Benton *Milos,*
tmorillonite	+	+	+	+	?	+	+	+		+	+
ed layers					?				+-		
c/Illite			-	+-	+-	-	-		+-		
rtz	+-		-	+-	+	-	+-		+		-
spar				-	-						-
optilolite	-										
ite	-	-		-	+	-				-	-
mite		-		-	-						
gonite					-						
inite			--		-			+-	+-		
rite				-	-						
tobalite							-				-
sum							--		-		
thite								-			
rite									-		
e/Anatase			-					--			
ite			--								
te	-	-			-		--		--		

Some minerals questionable…

What might help ?

Of course: LECO, IR, DTA

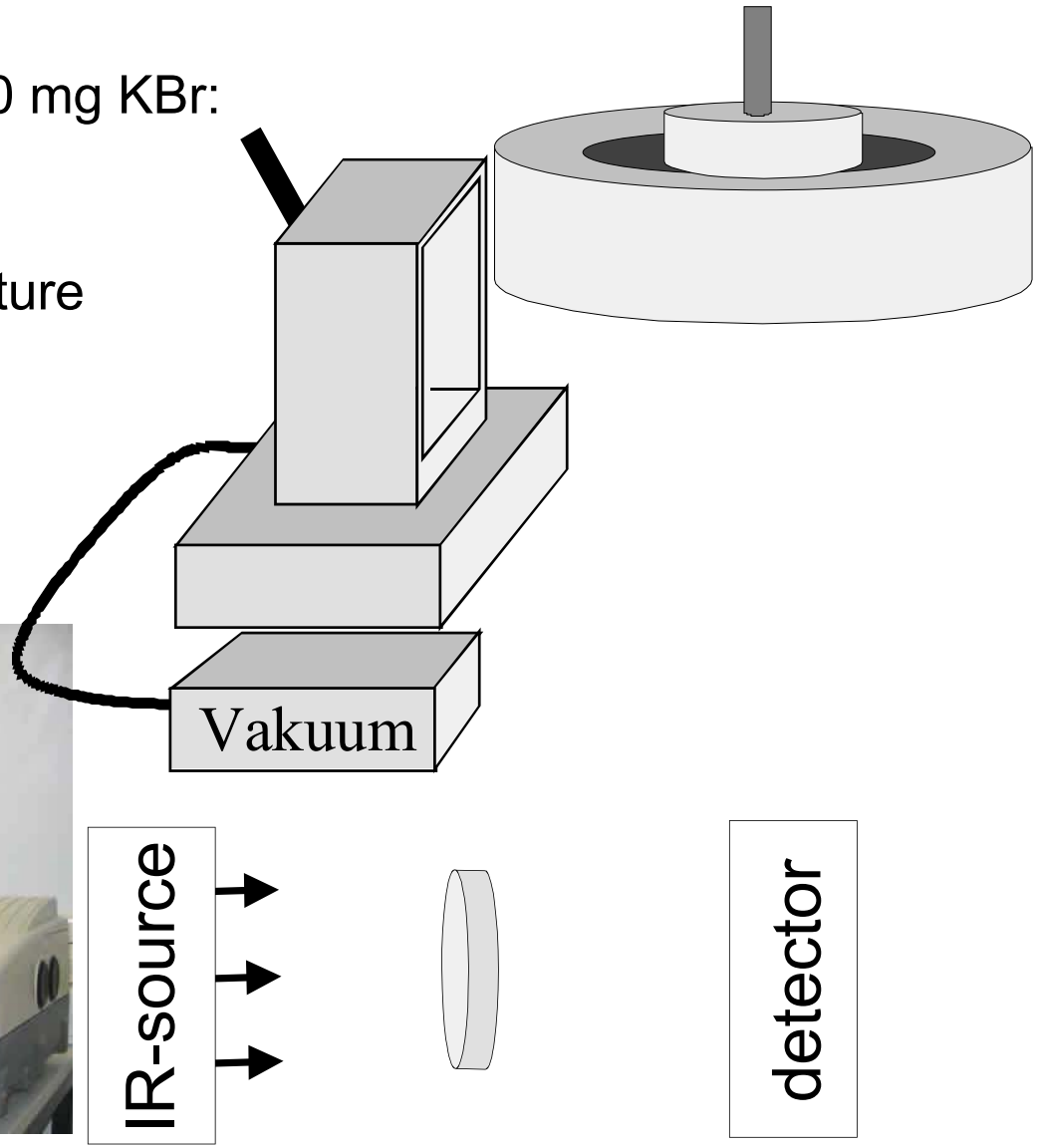
Infrared-spectroscopy

Weigh 1 mg sample and 200 mg KBr:
Mixing by mortar

Press a pellet out of the mixture

Measure background
(200 mg KBr only)

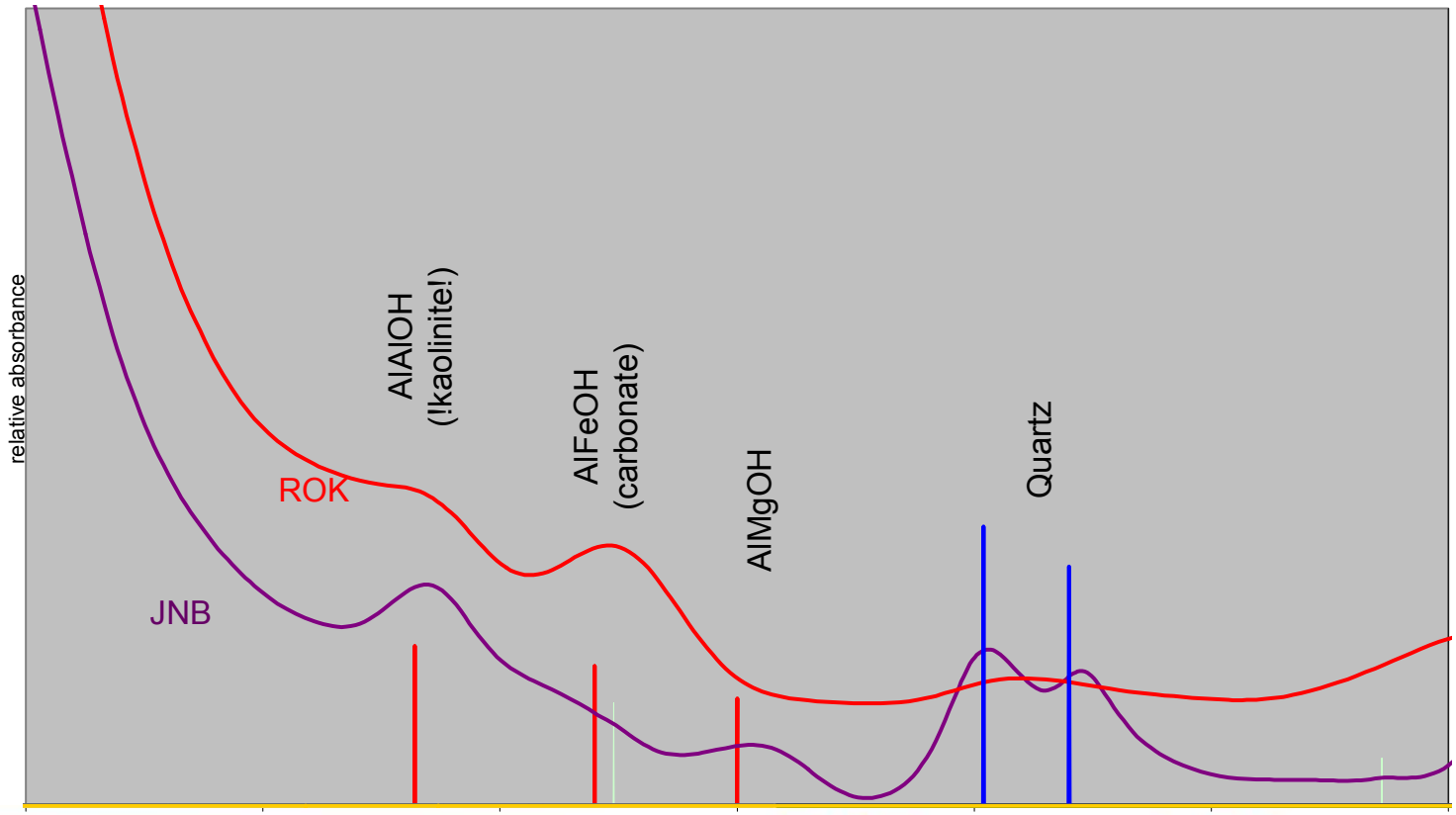
Measure sample



Infrared-spectroscopy

Advantage 1
low detection limit
kaolinite, organic matter, carbonates, quartz, (sulphates)

Advantage 2
octahedral layer composition of smectites can be estimated (e.g. Fe-rich or not)



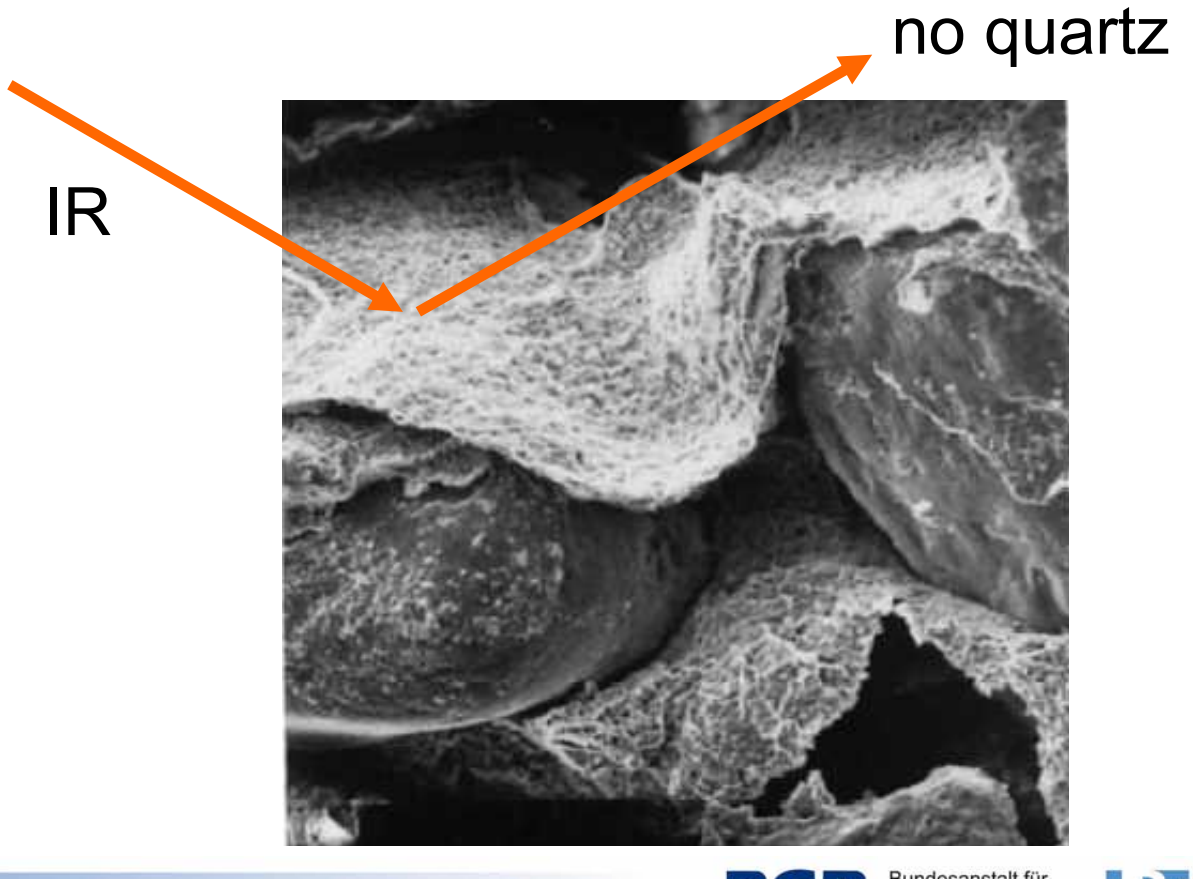
Infrared-spectroscopy

Disadvantage 1

a lot of minerals cannot be detected being present as minor component

Disadvantage 2

quantitative information can be obscured by microstructure



Differential thermal analysis

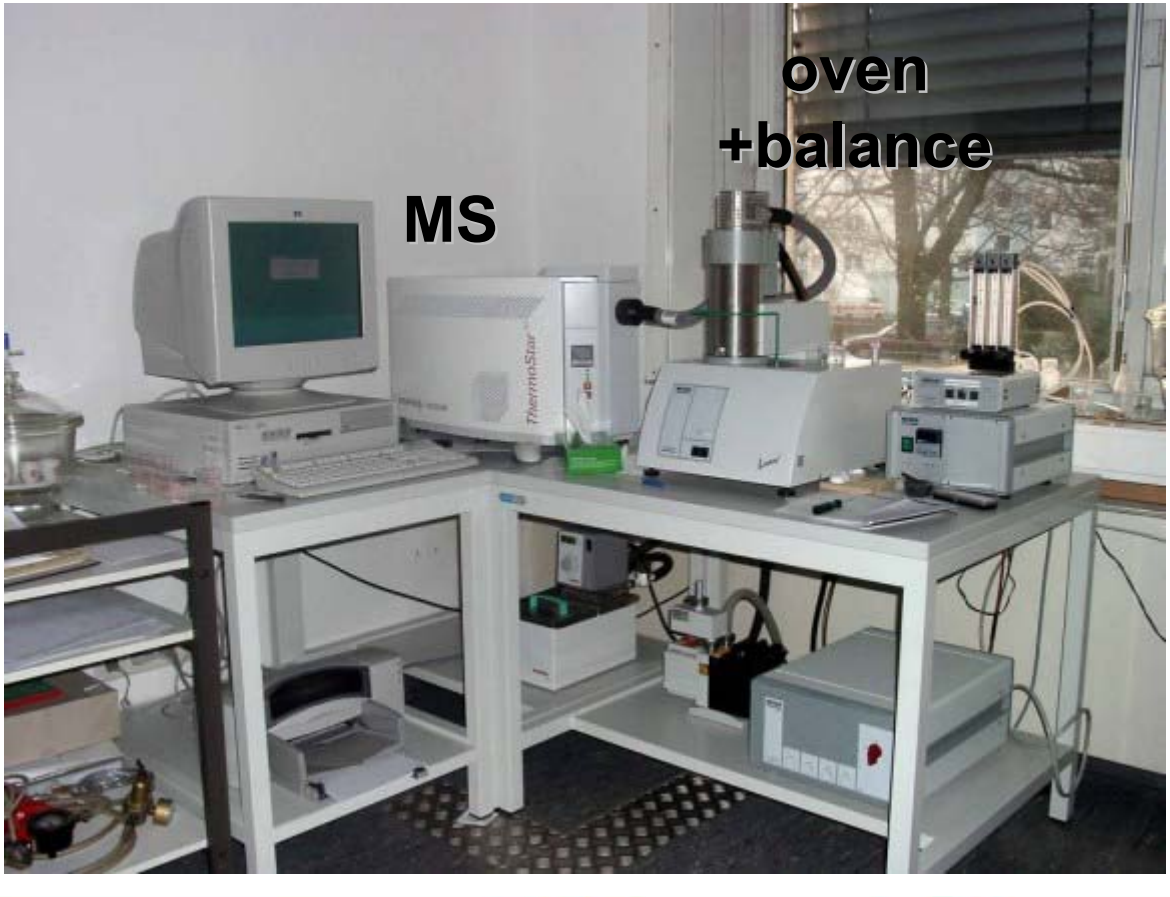
Weigh appr. 200 mg in Pt-cruciple

sample is heated and investigated with respect to

weight loss

energy (exo-, endo-thermal reaction)

CO_2, H_2O, SO_2 by mass spectrometry



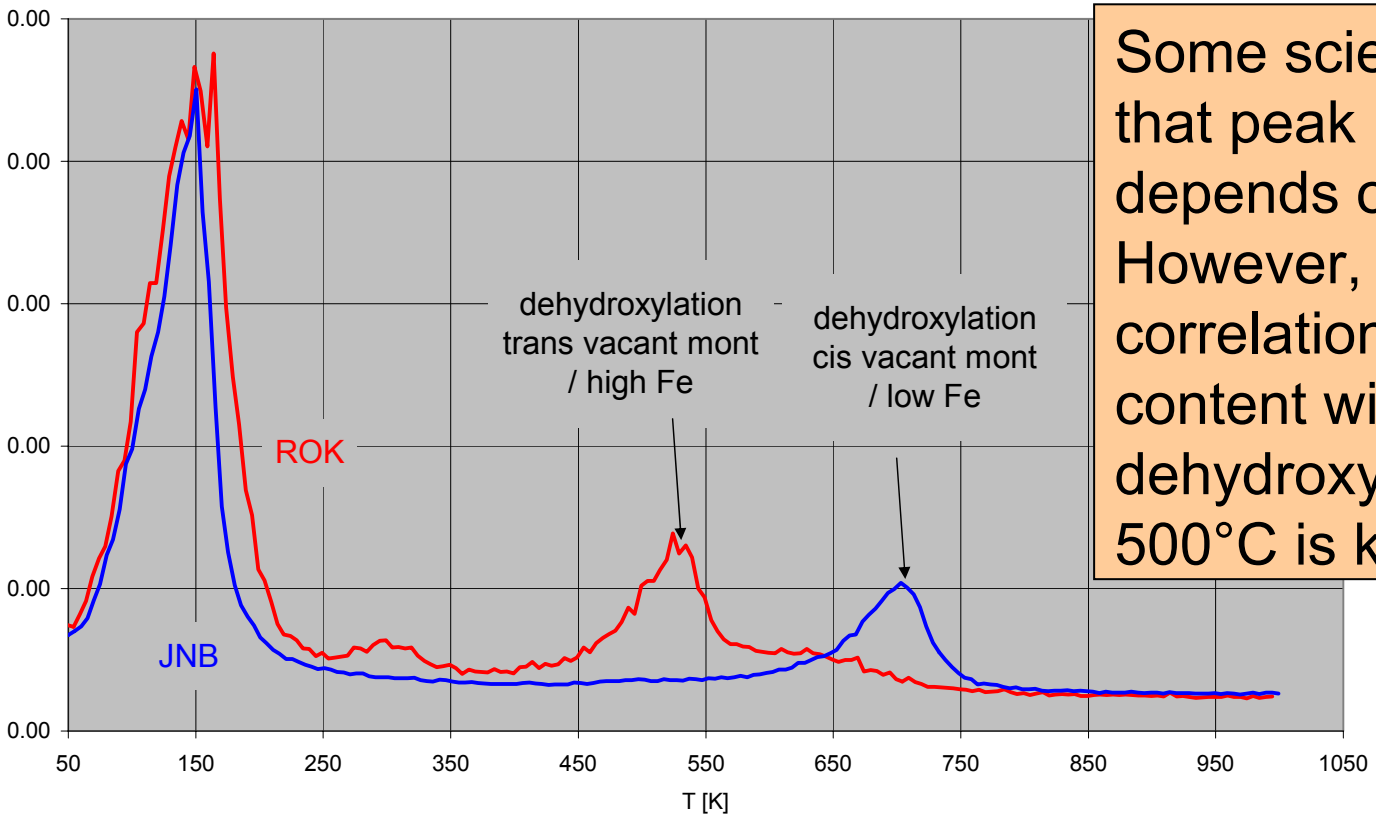
Differential thermal analysis

Advantage 1

low detection limit sulphides, sulphates, organic matter, carbonates

Advantage 2

information about octahedral layer vacancy and/or Fe content can be gained



Some scientists claim that peak position depends on vacancy. However, a strong correlation of Fe^{VI}-content with dehydroxylation at 500°C is known !!

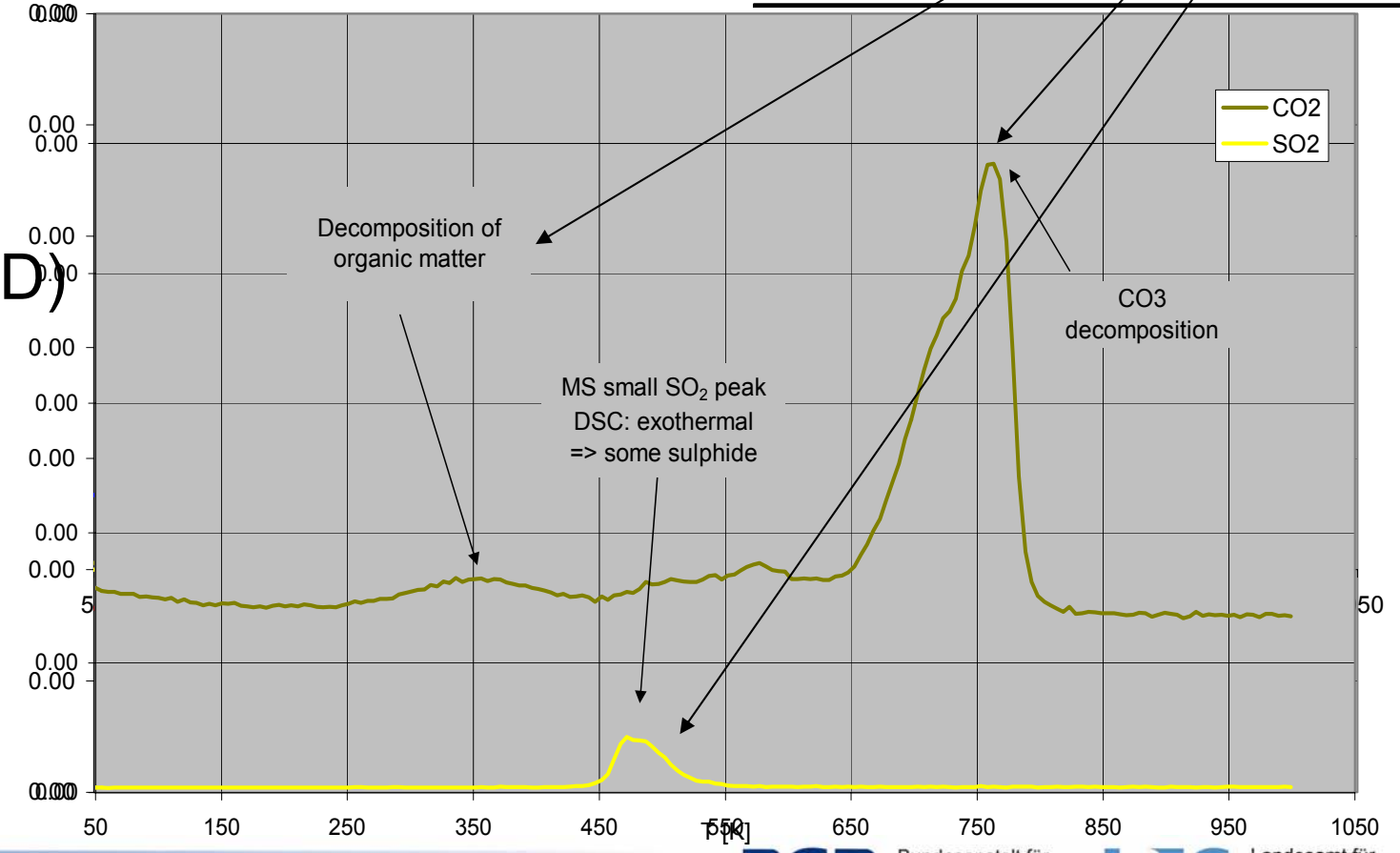
JNB

XRD: montm., quartz, clinopt., calcite

Where is S ?

pyrite
0.5-1 wt.%
(difficult to
see with XRD)

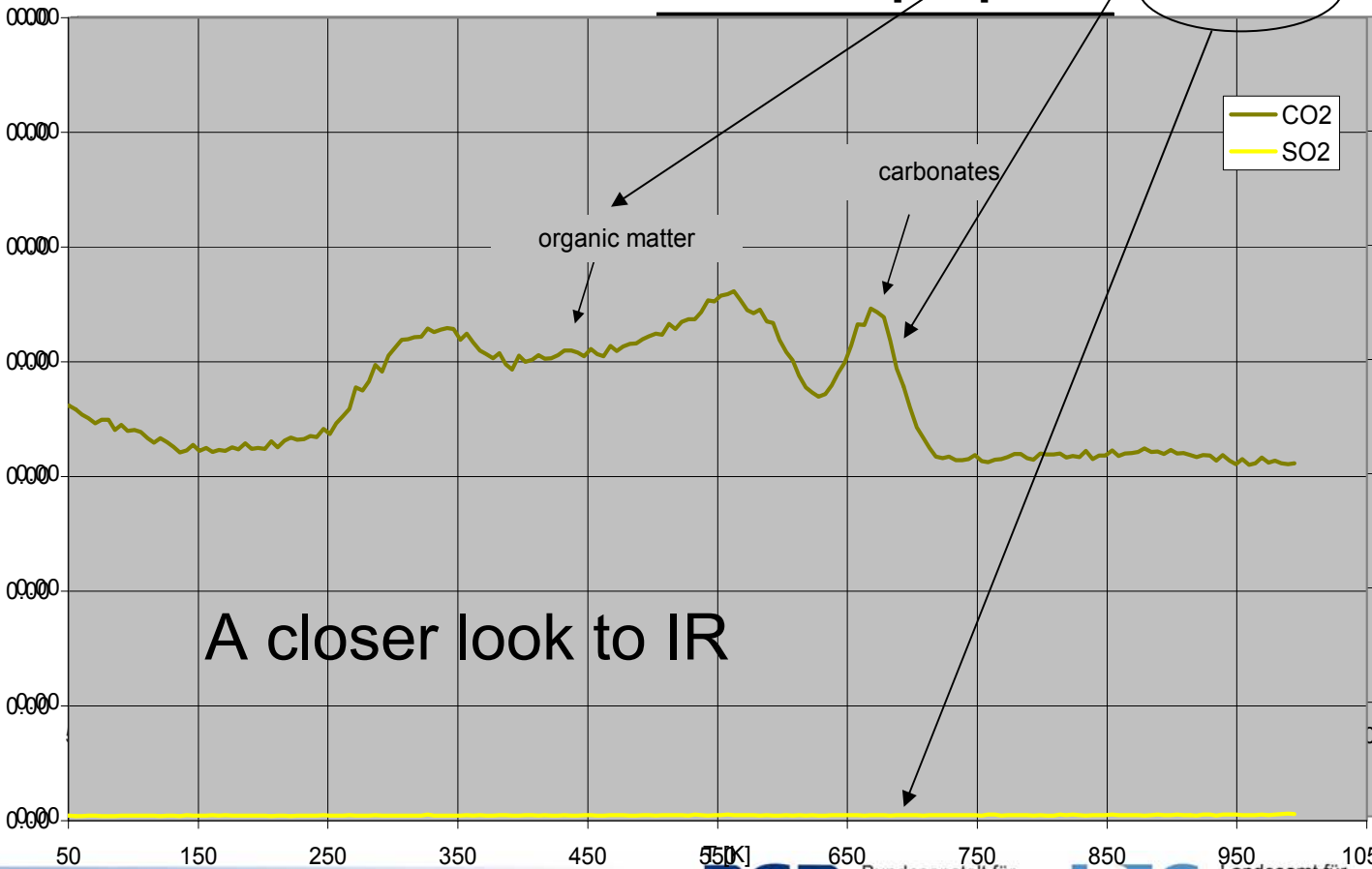
LECO		
C_{total}	[wt.%]	0.43
C_{org}	[wt.%]	0.07
C_{inorg}	[wt.%]	0.36
S_{total}	[wt.%]	0.34



ROK

XRD: montm., muscov., quartz, rutile/anatase?, apatite?

LECO		
C_{total}	[wt.%]	0.27
C_{org}	[wt.%]	0.17
C_{inorg}	[wt.%]	0.1
S_{total}	[wt.%]	0.02



ROK

XRD: montm., muscov., quartz, rutile/anatase?, apatite?

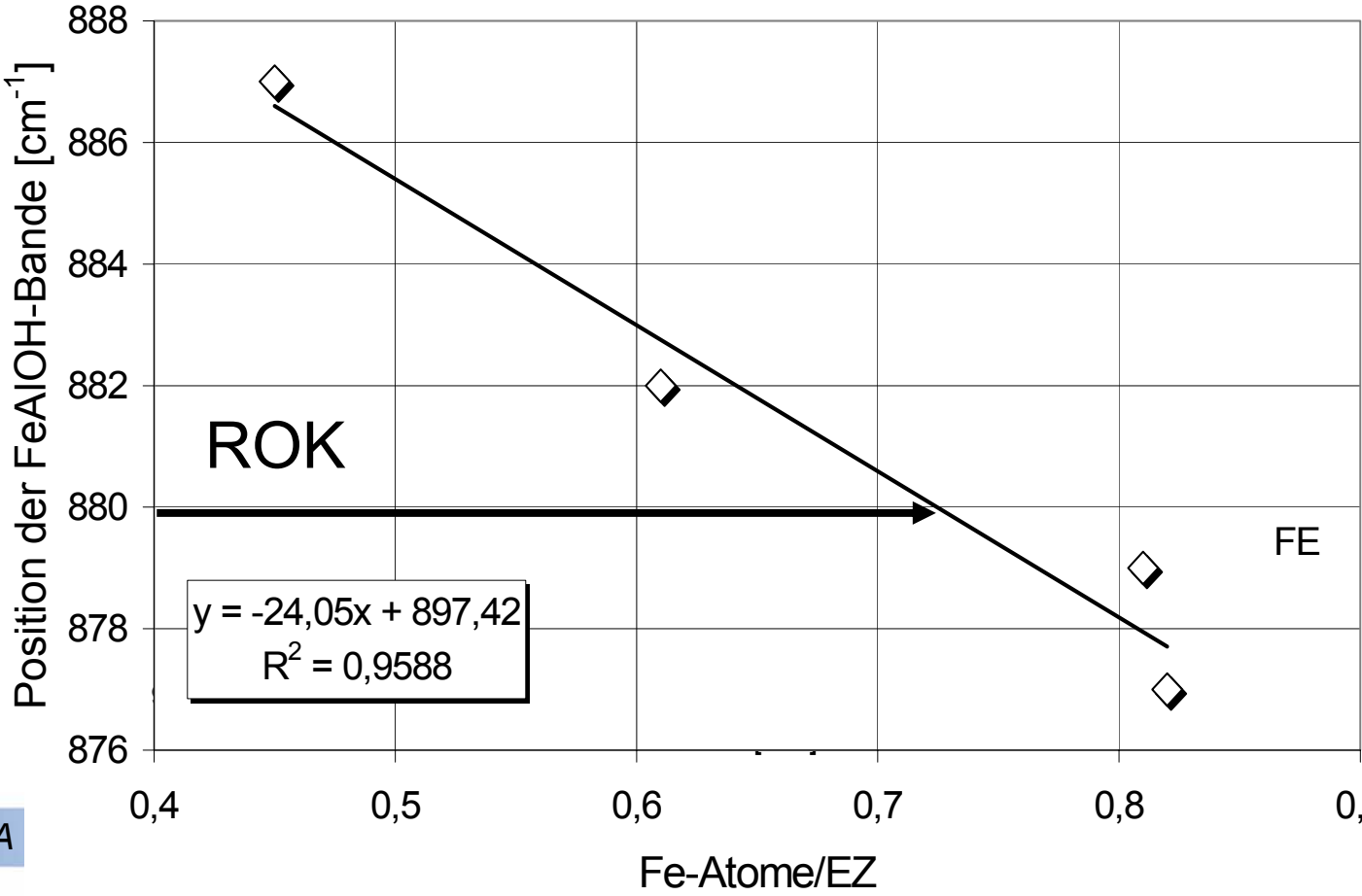
IR: trace kaolinite

carbonate at detection limit

F-rich mont. (0.7 Fe^{VI}/EC)

muscovite questionable

LECO		
C_{total}	[wt.%]	0.27
C_{org}	[wt.%]	0.17
C_{inorg}	[wt.%]	0.1
S_{total}	[wt.%]	0.02



aufhold & Dohrmann: DTA

Conclusions

DTA and IR complement XRD results

DTA and IR are consistent with LECO analysis
(no discrepancies between IR, DTA, LECO detected)

LECO provides accurate figures related to the abundance of important minerals as sulphides & sulphates, carbonates – even small changes (before and after) can be detected !

Detection limits (ca.)

	C_{inorg}	Sulphides	Sulphates	Kaolinite
Infrared	0.2 wt.%	/	> 1 wt.%	0.1 wt.%
DTA-MS	0.1 wt.%	0.1 wt.%	0.2 wt.%	> 1 wt.%

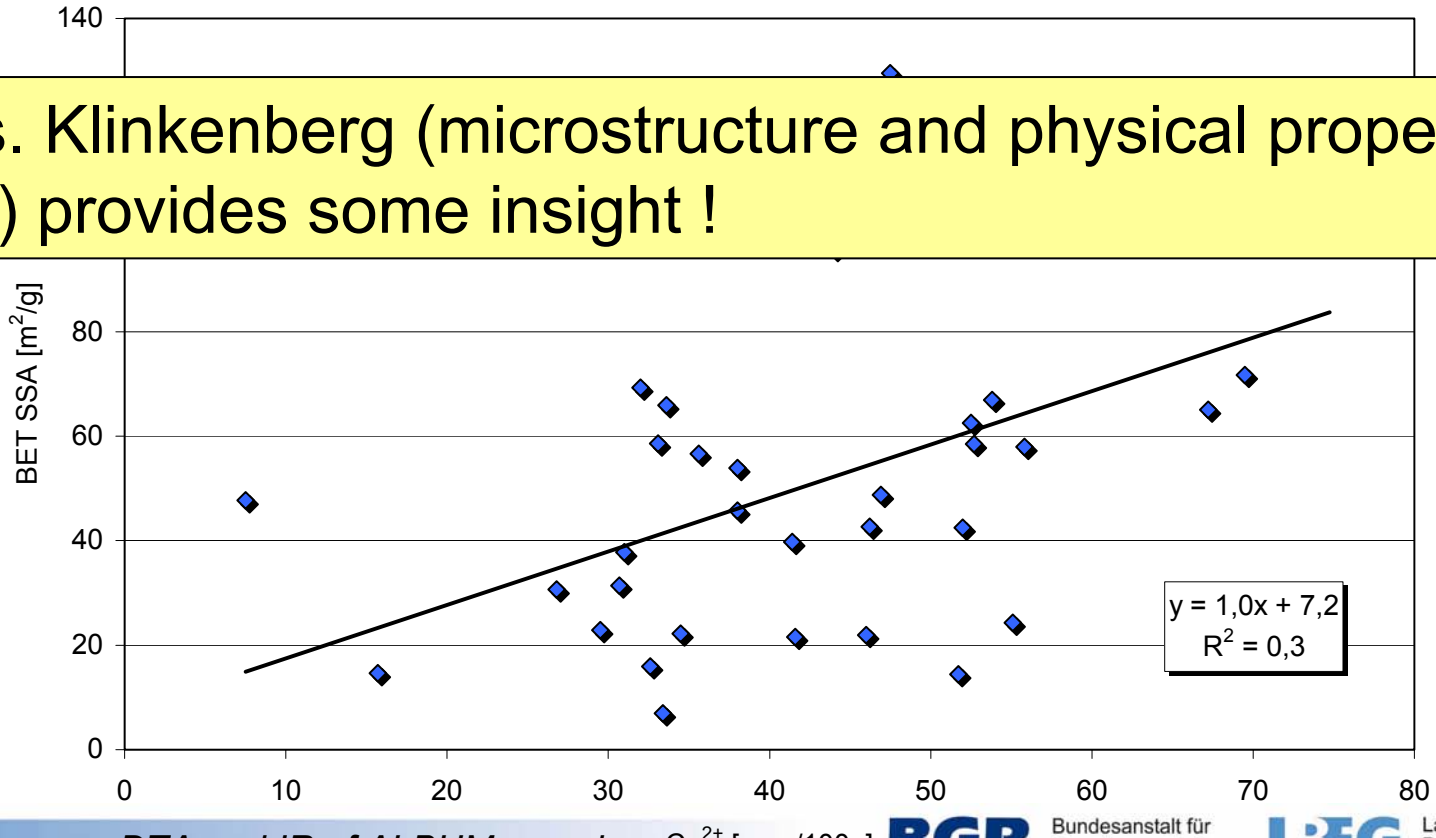
What do we learn from BET ?

N_2 adsorption ⇒ microporosity rather than real surface area

Leaching creates micropores ⇒ changes can be observed

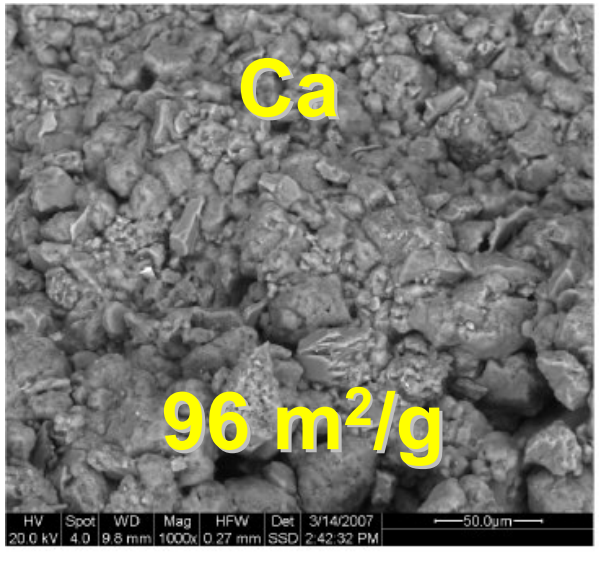
BET SSA depends on Ca / Na occupation… why ?

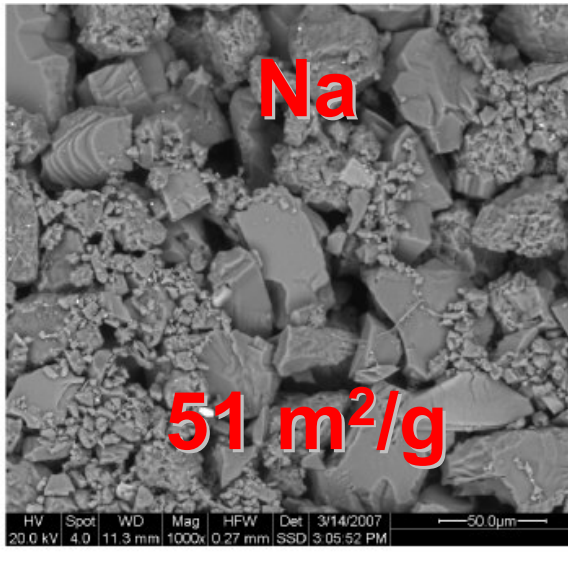
PhD Ms. Klinkenberg (microstructure and physical properties of clays) provides some insight !

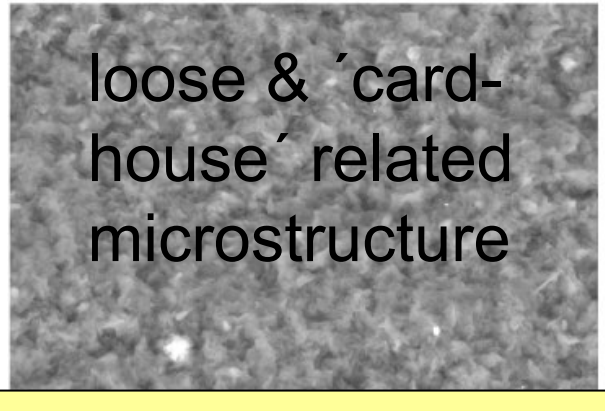


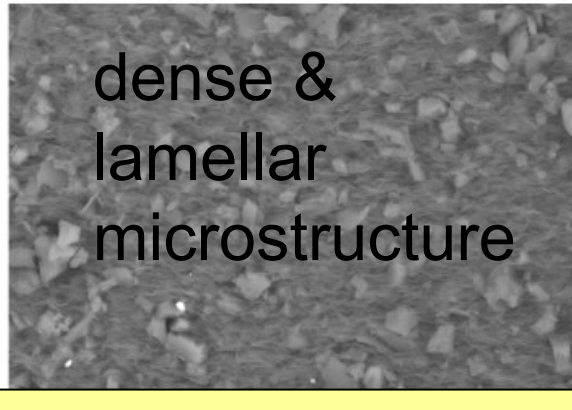
What do we learn from BET ?

Bentonite morocco
Ca and Na saturated
dispersed & dried





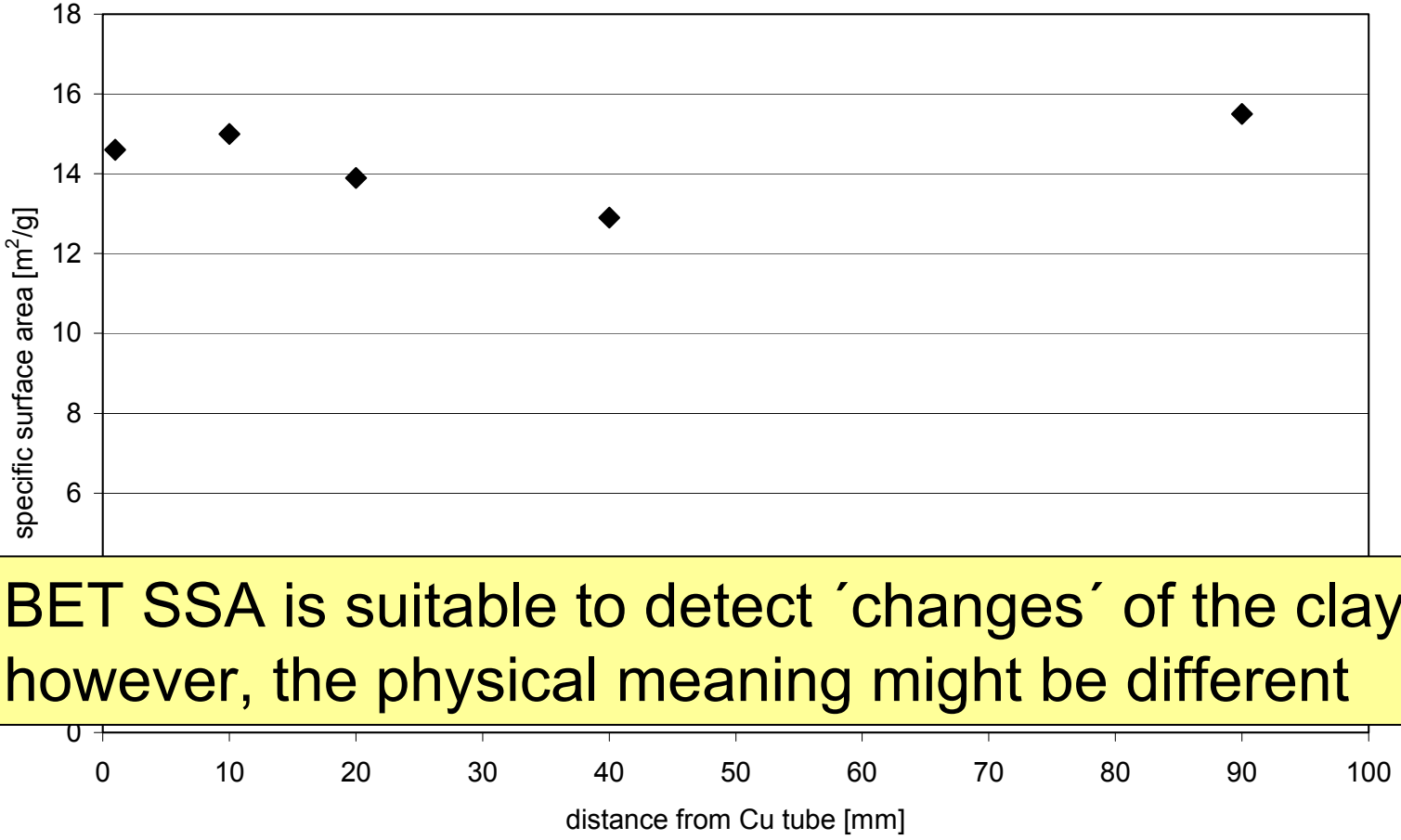




BET SSA depends on micro- and mesoporosity and hence on microstructure (arrangement of minerals to each other)

What do we learn from BET ?

In case of LOT A2 experiment BET reflects gypsum precipitation (in pores)



BET SSA is suitable to detect ´changes´ of the clay, however, the physical meaning might be different

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COB

XRD: montm/ML, muscov., quartz, fsp., calcite/dolomite, kaolinite, chlorite, pyrite?

LECO		
C_{total}	[wt.%]	3.94
C_{org}	[wt.%]	0.65
C_{inorg}	[wt.%]	3.29
S_{total}	[wt.%]	0.68