CO_2 Capture by Novel Supported Ionic Liquid Phase Materials Consisting of Silica Encapsulated Chitosan Ionogels

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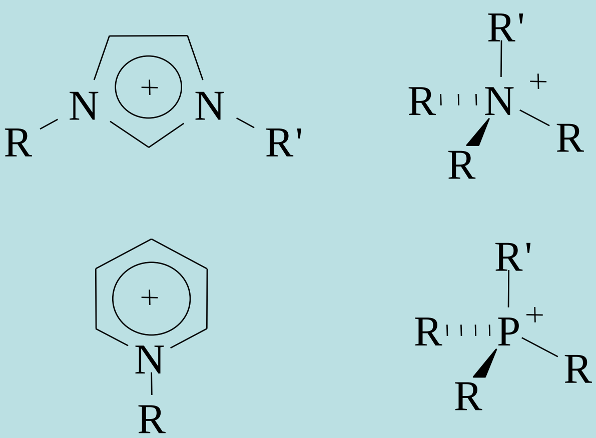
Ionic Liquids

The properties depend on the specific anion/cation combination

cation



anion

Cl^-, Br^-

$[AlCl_4]^-$, $[Al_2Cl_7]^-$

$[PF_6]^-$, $[BF_4]^-$

$[HSO_4]^-$

$[RSO_4]^-$, $[RSO_3]^-$

$[CF_3COO]^-$, $[CF_3SO_3]^-$

$[(CF_3SO_2)_2N]^-$

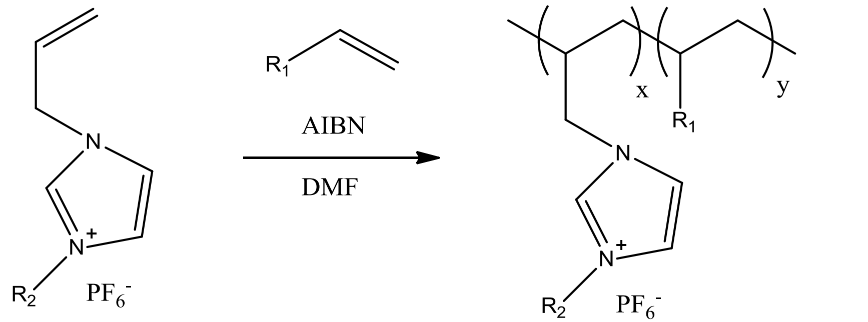
density, acidity, coordination, price

viscosity, therm. capacity, therm. stability, disposal options

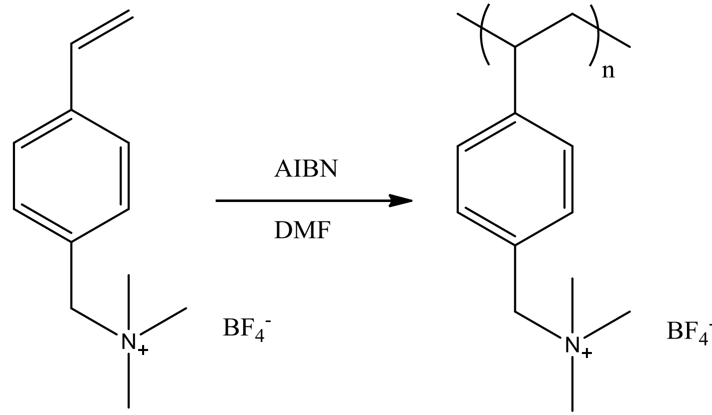
surface tension, electrochem. stability, toxicity

Polymerized Ionic Liquid (PIL)

- Furhter names:
  - *„Poly(ionic liquid)s“*
  - Polyampholytes
  - Polyelectrolyte



R_1 = CN, COOH, N,N-methylenebisacrylamide (MBA)



Guangren Yu, Qingzeng Li, Na Li, Ziwei Man, Chenghao Pu,Charles Asumana, Xiaochun Chen Polymer Engineering & Science, (**2014**) 54, *1*, 59–63,

Zhu, Jiamei; Zhou, Jianhua; He, Kaige; Zhang, Hu Best Journal of Polymer Research (**2011)** 18, *6*, 2011-2015

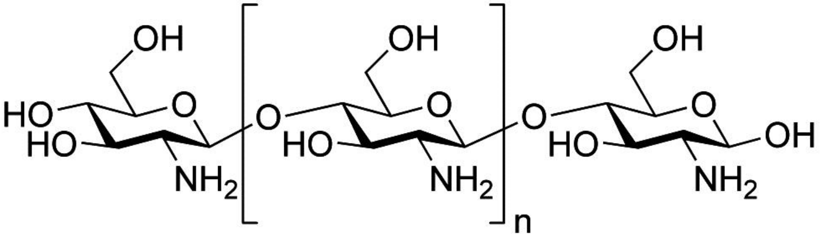
Sebastian Soll; Qiang Zhao; Jens Weber; Jiayin Yuan; *Chem. Mater.* **(2013)** 25, 3003-3010

Using Poly-Ionic Liquid for CO_2 absorption

- Active Material
  - Polymerized ionic liquid
  - PIL and Copolymer
  - Ionogel
- Surface
  - Precipitation
  - Inverse suspension polymerization (seed swelling polymerization)
  - Membrane technology
  - Supported Ionic Liquid Phase SILP
    - hard-templating
  - Inverse SILP
- are favourable due to convenient handling and fast absorption kinetics

Chitosan

- Second most abundant biopolymer after cellulose
  - Renewable and inexpensive
  - Amino groups → chemisorption of CO_2

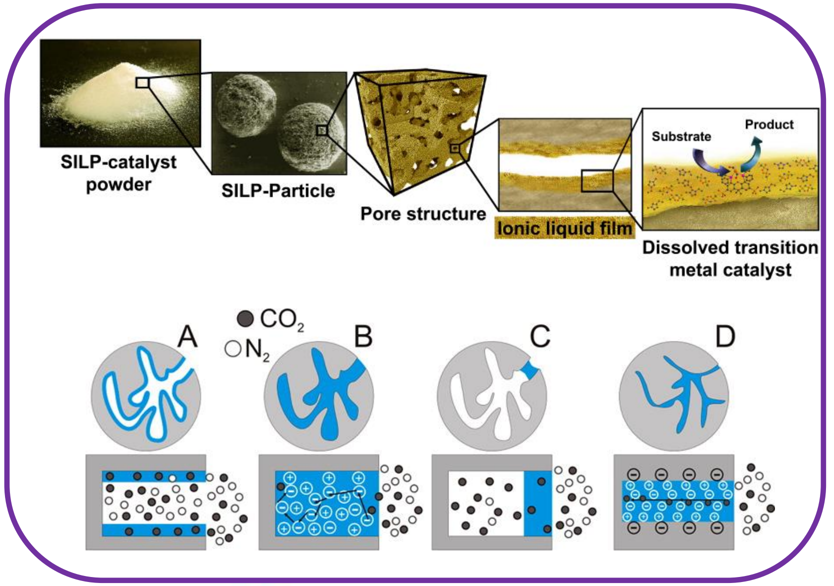


- **<u>Ionogel:</u>** gel like material obtained by dissolving chitosan in IL
  - Suitable ILs have basic anions with high hydrogen donor accepting ability (β>0.8) Cl^-, OAc^-, $RCOO^-$

Inverse SILP

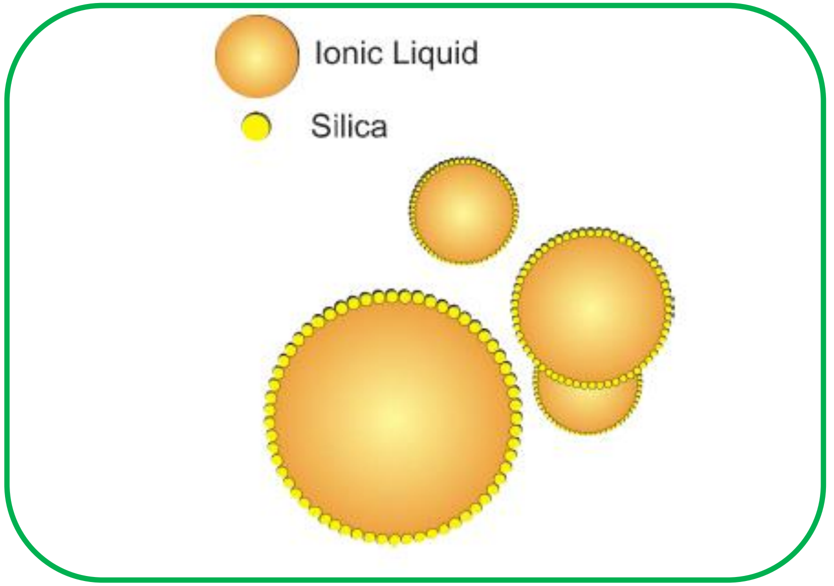
SILP: Supported Ionic Liquid Phase

- Advantage
  - high surface area
  - small amounts of IL needed
  - no diffuision limitation
- Disadvantage
  - low rate $m_{abs} : m_{support}$
  - pore blocking/filling possible



Inverse SILP: drop of IL covered with silica

- Advantages
  - high surface area to volume ratio
  - high rate $m_{abs} : m_{support}$
  - fast kinetics
- Disadvantages
  - not durable under high pressure



Inverse SILPs based on chitosan ionogels

Preparation of inverse SILPs based on chitosan ionogel:

- **I step:** IL+ chitosan → ionogel
- **II step:** ionogel + silica → inverse SILP

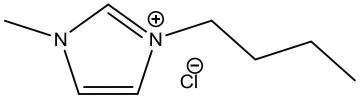
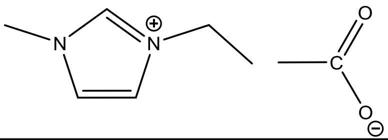
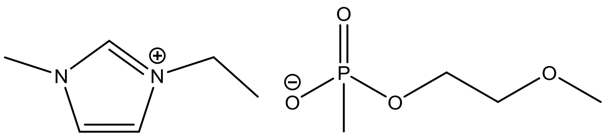
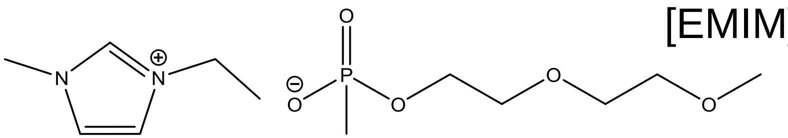
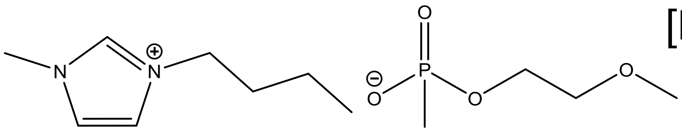
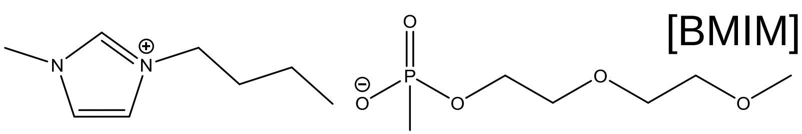
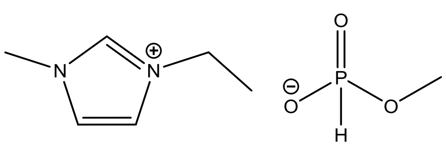
IL + chitosan →

ionogel

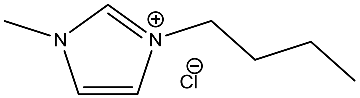
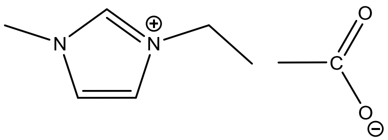
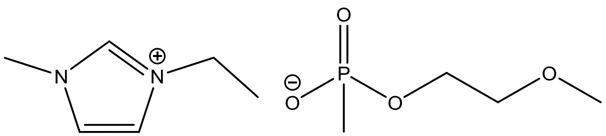
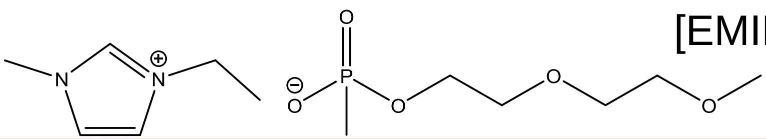
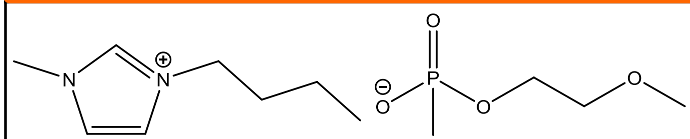
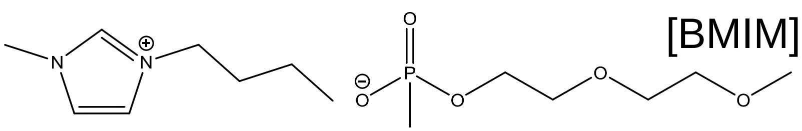
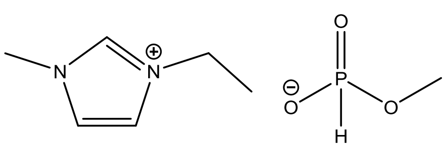
\+ silica →

inverse SILP

Solubility of Chitosan in ILs

IL	Solublity of chitosan
 [BMIM][Cl]	10%
 [EMIM][OAc]	10%
 [EMIM][Me(EG)$_1$(Me)PO$_3$]	7%
 [EMIM][Me(EG)$_2$(Me)PO$_3$]	5%
 [BMIM][Me(EG)$_1$(Me)PO$_3$]	3%
 [BMIM][Me(EG)$_2$(Me)PO$_3$]	3%
 [EMIM][Me(Me)O$_3$]	2%

Solubility of Chitosan in ILs

Ionic liquid	Solublity of chitosan
 [BMIM][Cl]	10%
 [EMIM][OAc]	10%
 [EMIM][Me(EG)$_1$(Me)PO$_3$]	7%
 [EMIM][Me(EG)$_2$(Me)PO$_3$]	5%
 [BMIM][Me(EG)$_1$(Me)PO$_3$]	3%
 [BMIM][Me(EG)$_2$(Me)PO$_3$]	3%
 [EMIM][Me(Me)O$_3$]	2%

Chosen for inverse SILP preparation and CO_2 absorption.

Preparation of Inverse SILP

- **<u>problem:</u>** high viscosity of ionogels

- Stability of cellulose ionogels towards solvents
  - Suitable solvents for homogenous mixing are rather polar (E^N_T>0.3, π*>0.8), weak hydrogen bond donors (α<0.5), moderate hydrogen bond acceptors (β>0.4)
  - Protic solvents: chitosan precipitates
  - DMSO, DMF, DMA: high T_b

Macromol. Mater. Eng. **2011**, 296, 483-493.

- ACN as co-solvent used to decrease the viscosity of chitosan ionogels
  - E^N_T=0.46, π*=0.75, α=0.19, β=0.4
  - Maximum amount ACN without chitosan precipitation: 50wt%

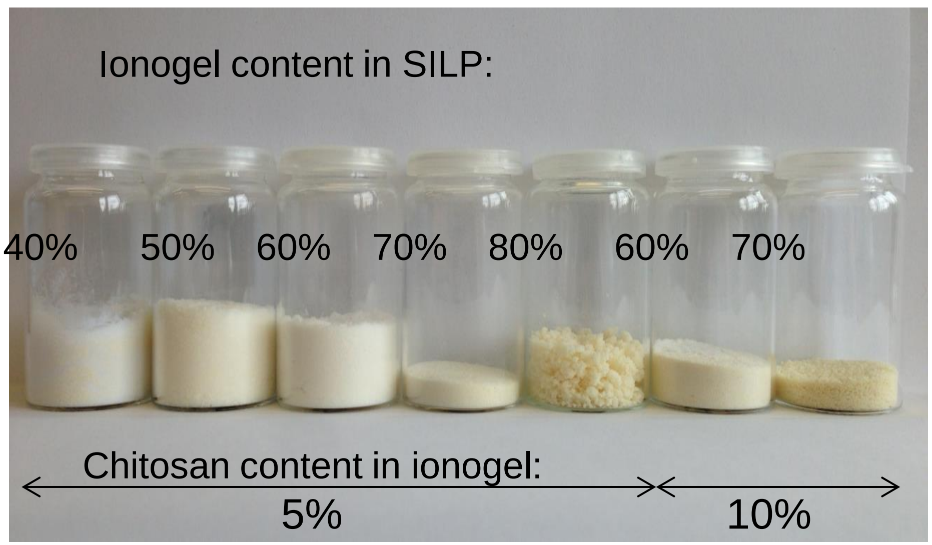
Preparation of Inverse SILP

- **Ionogel (or IL) + ACN + silica**
  mixed by 8-11*10^3 rpm for 15 min.
  - ionogel or IL content in inverse SILP varied 40-80%
  - ACN removed by evaporation

- Characterization: optical microscope

Preparation of Inverse SILP

- Various ionogel: silica ratios
  - Optimal: 40-60% IL or ionogel
  - <40% ionogel: obtained inverse SILP contained a lot of free silica
  - >60% ionogel: obtained inverse SILP not free flowing powder



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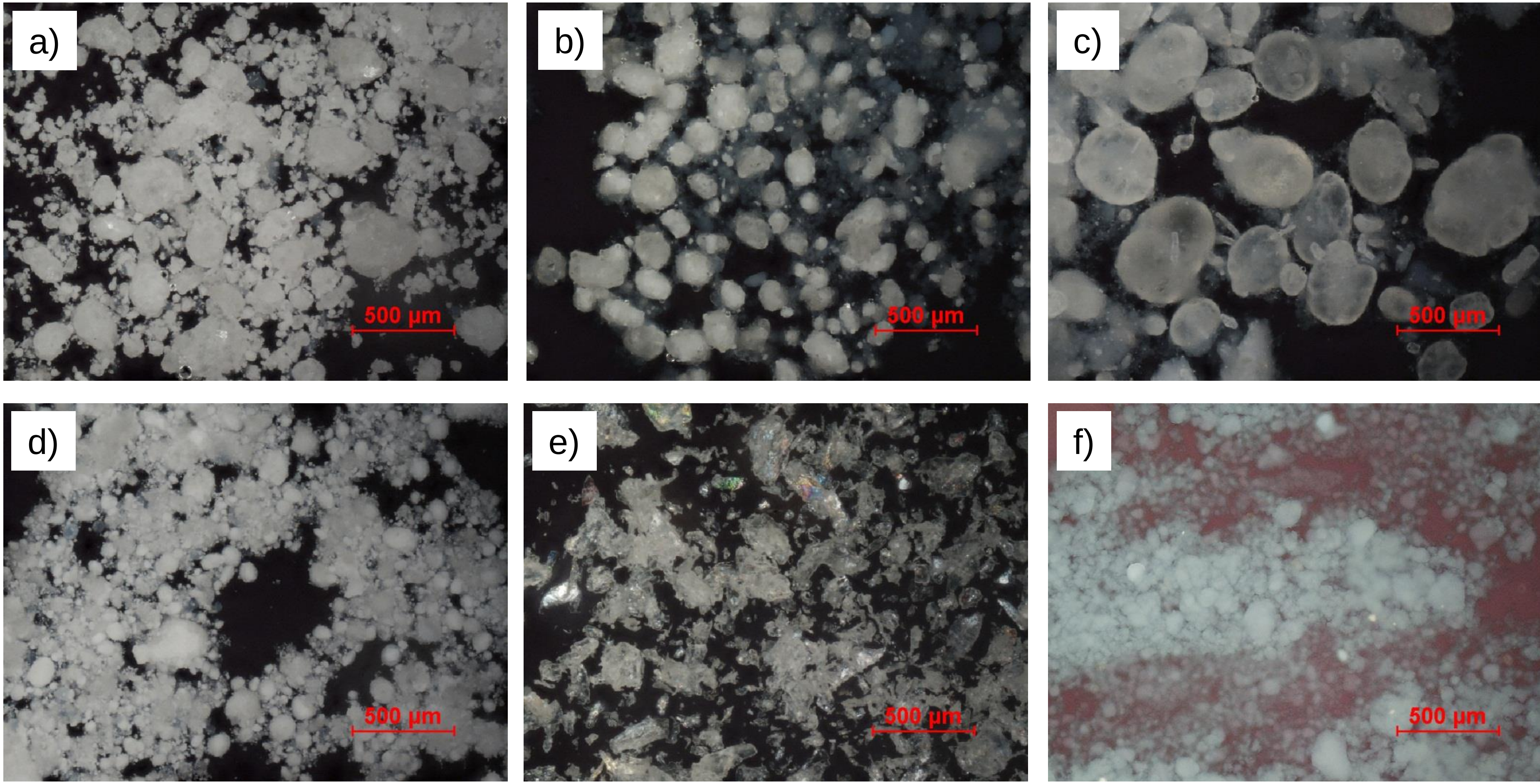


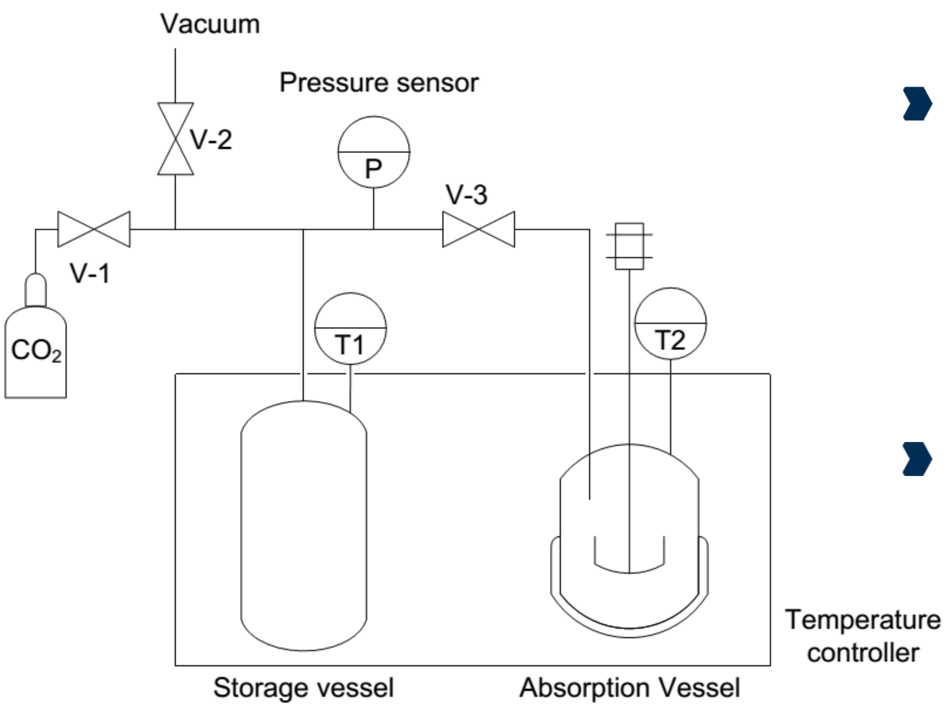
Microscope images (5X magnification): a) [BMIM][Cl], b) [EMIM][OAc], c) [EMIM][Me(EG)$_1$(Me)PO$_3$], d) [EMIM][Me(EG)$_2$(Me)PO$_3$], e) chitosan, f) silica

All inverse SILPs contain 40% ionogel, in ionogel 5% chitosan

Overview of Prepared Inverse SILPs Used for CO_2 Absorption

IL	% silica	% IL or ionogel	% chitosan in ionogel
[EMIM][OAc]	60	40	5
	60	40	-
[EMIM][Me(EG)$_1$(Me)PO$_3$]	60	40	5
	60	40	-
[EMIM][Me(EG)$_2$(Me)PO$_3$]	60	40	5
	60	40	-
[BMIM][Cl]	60	40	5
	60	40	-
	60	40	10
	40	60	5

CO_2 Absorption in Autoclave

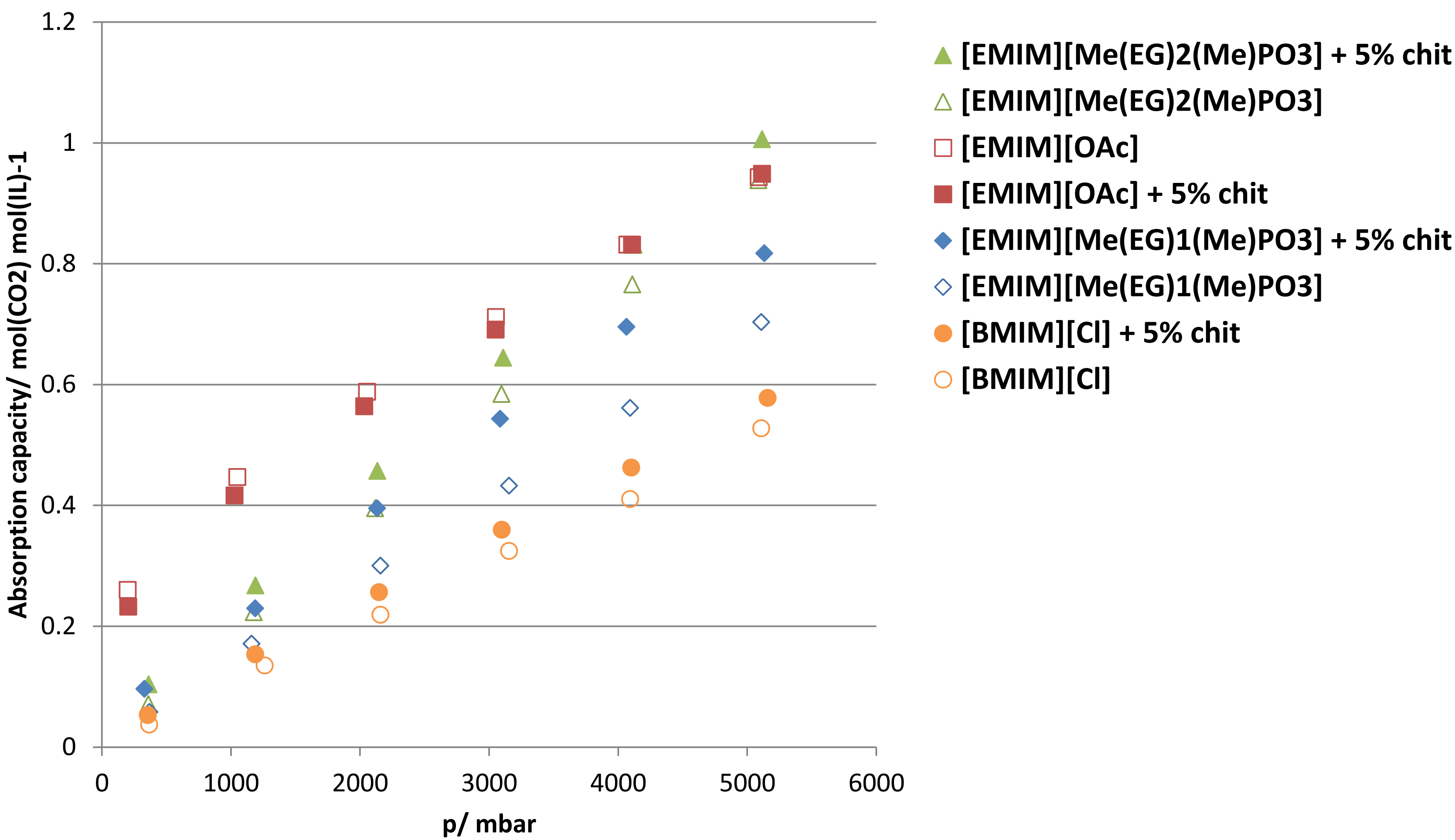


- Absorbed CO_2

$$n_{CO_2} = \frac{P_0 V_S - P(V_S + V_A - V_L) + P_V V_L}{RT}$$

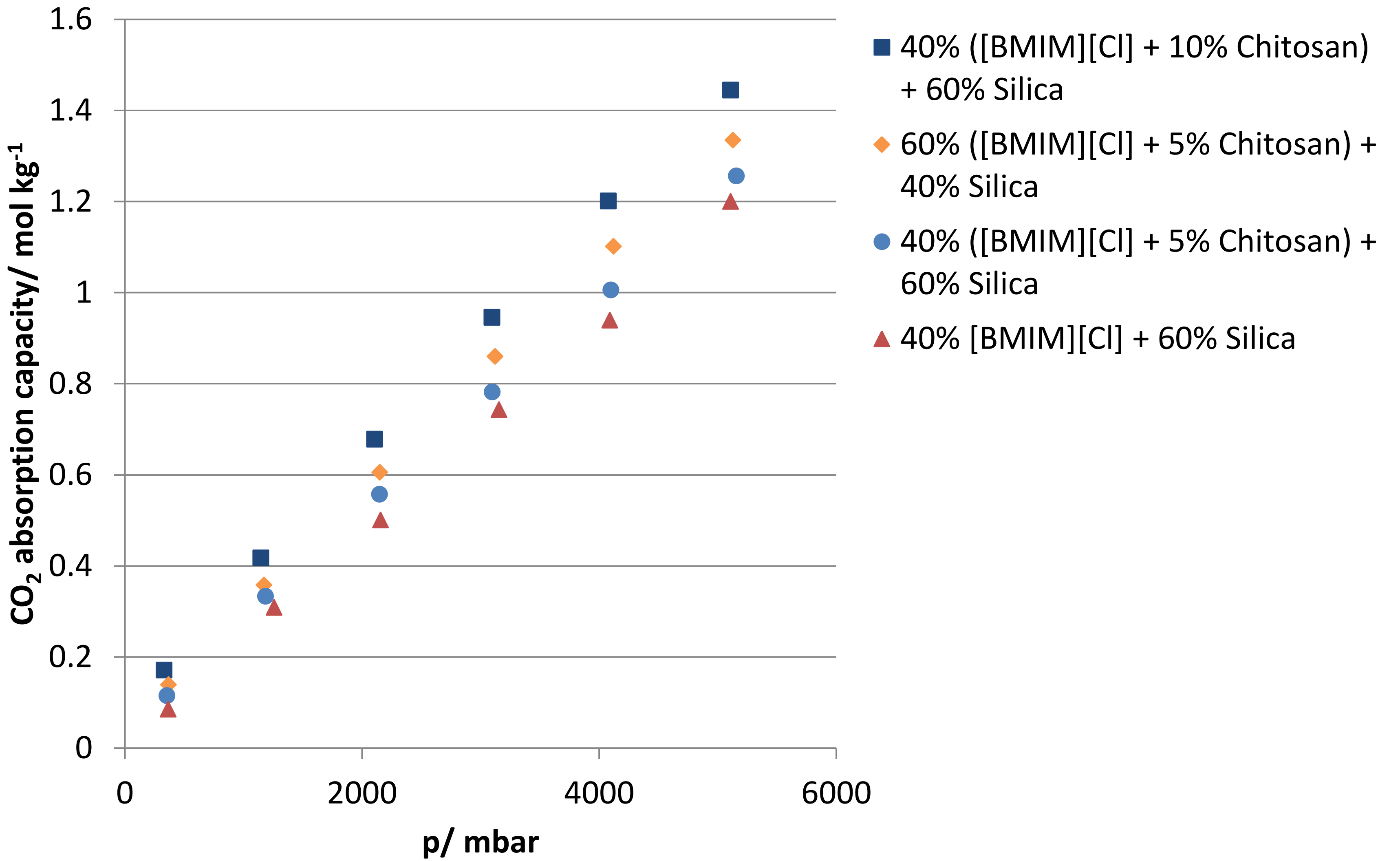
- Advantages
  - Exact measurements
  - Small amounts of sample needed (~5 g)
  - Measurement at different pressures (up 6 bar)
- Disadvantages
  - Long waiting time for equilibrium of high viscous systems

CO_2 absorption in Inverse SILPs



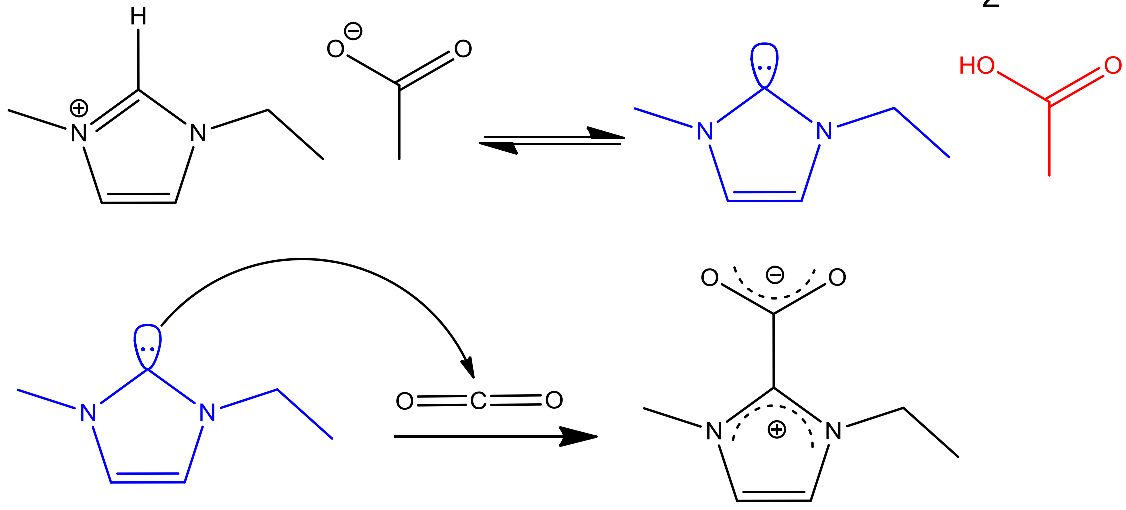
[BMIM][Cl], [EMIM][Me(EG)$_1$(Me)PO$_3$], [EMIM][Me(EG)$_2$(Me)PO$_3$]: with chitosan higher capacity
[EMIM][OAc]: with chitosan lower capacity

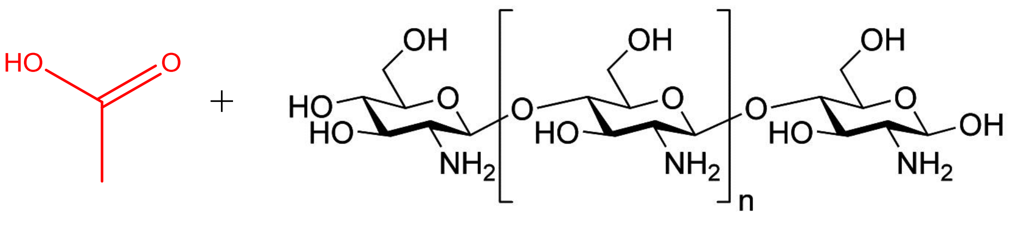
Concentration Variations with [BMIM][Cl] Ionogels



[EMIM][OAc]

- *In situ* carbene formation and reaction with CO_2





Reaction between acetic acid and chitosan very likely

Summary

CO_2 absorption in inverse SILPs based on biopolymer ionogels

- New morphology for IL based poylmers for CO_2 absorption

- Solubility of chitosan tested in various ILs
  - Novel solvents for chitosan: alkyl methylphosphonate ILs

- Method for preparation of inverse SILPs from chitosan ionogels
  - ACN as a co-solvent
  - Variation of ionogel:silica ratio (optimal 40-60% ionogel)

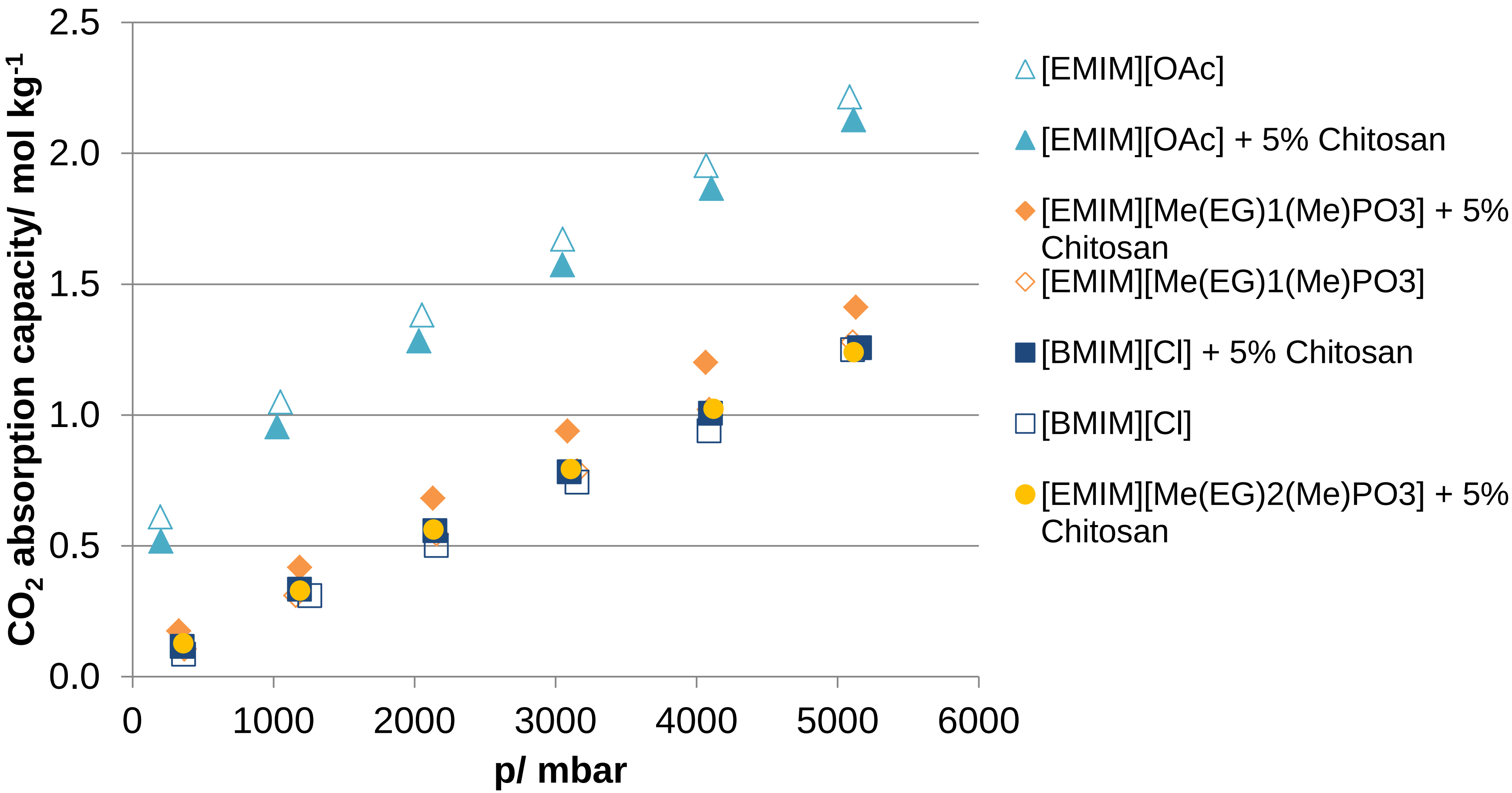
- CO_2 absorption in inverse SILPs based on chitosan ionogels
  - [BMIM][Cl], [EMIM][Me(EG)$_1$(Me)PO$_3$], [EMIM][Me(EG)$_2$(Me)PO$_3$] (physisorption): with chitosan higher capacity
  - [EMIM][OAc] (physisorption + chemisorption): with chitosan lower capacity

Acknowledgements

- *Prof. Peter Wasserscheid*
- *Matthias Bahlmann*
- *Dr. Kaija Pohako-Esko*

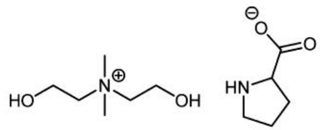
- Novel IOnic LIquid and supported ionic liquid solvents for reversible CAPture of CO_2

CO_2 absorption in inverse SILPs

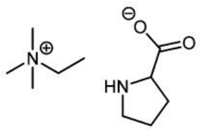


[BMIM][Cl], [EMIM][Me(EG)$_1$(Me)PO$_3$], [EMIM][Me(EG)$_2$(Me)PO$_3$]: with chitosan higher capacity
[EMIM][OAc]: with chitosan lower capacity

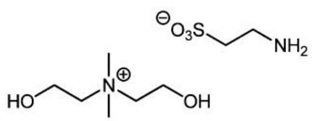
Citosan free inverse SILP materials



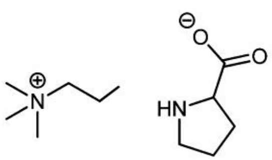
N,N-Dimethyl-N,N-diethanolammonium prolinate



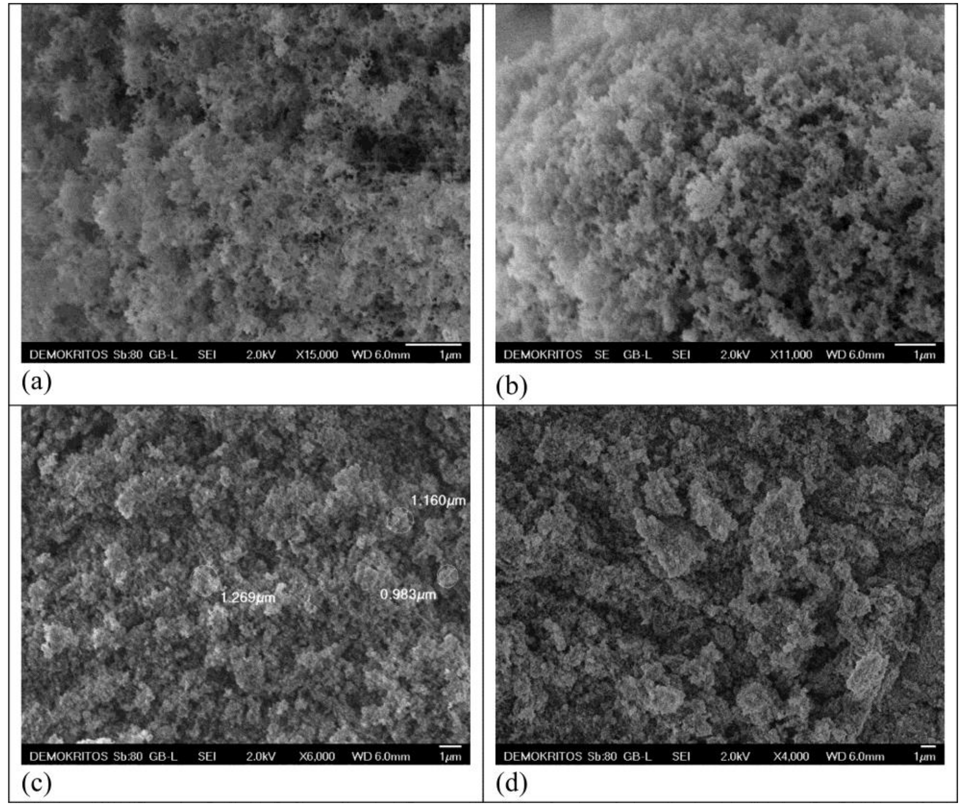
N,N,N-Trimethyl-N-ethylammonium prolinate



N,N-Dimethyl-N,N-diethanolammonium taurinate



N,N,N-Trimethyl-N-propylammonium prolinate



SEM images of (a) hydrophilic silica HDK-T30, (b) hydrophobic silica HDK-H20, (c) 6DD-Taur-H20, and (d) 4TE-Prol-H20.

George Em. Romanos; Peter S. Schulz; Matthias Bahlmann; Peter Wasserscheid; Andreas Sapalidis; Fotios K. Katsaros; Chrysoula P. Athanasekou; Konstantinos Beltsios; N. K. Kanellopoulos; *J. Phys. Chem. C* **2014,** 118, 24437-24451.

Citosan free inverse SILP materials

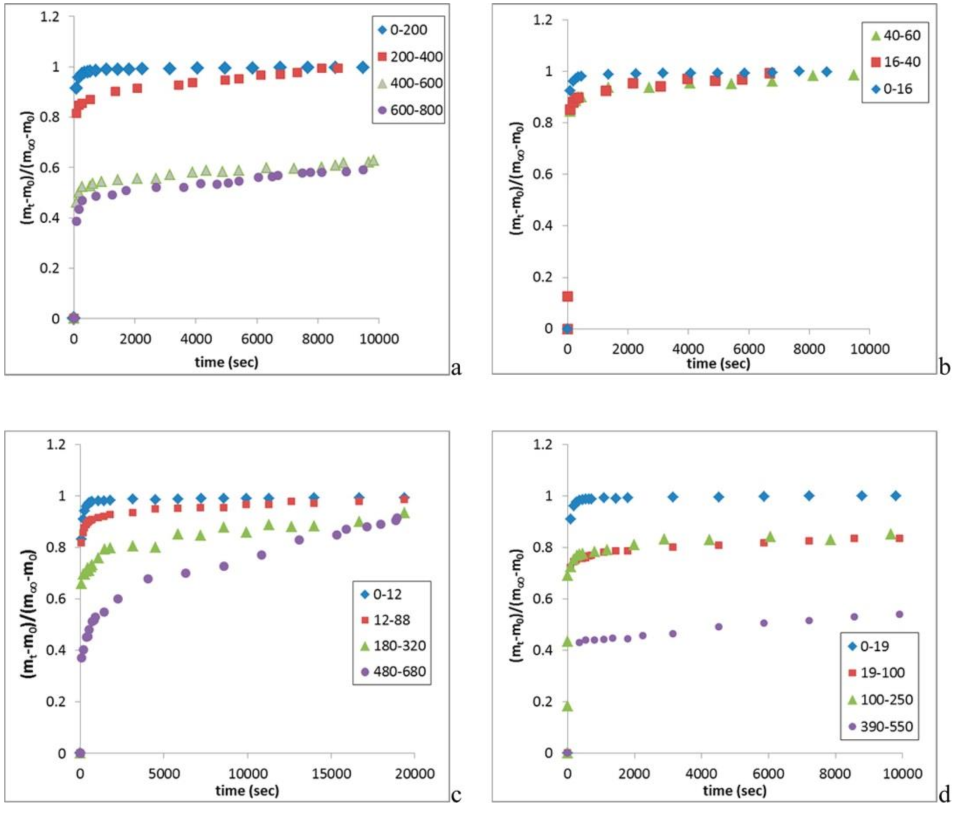
Pore Structure Characteristics as Derived from the LN_2 Porosimetry

	BET (m^2/g)	pore volume (mL/g)	interparticle pore size (nm)[a]	mean pore size BJH (nm)[b]	mean particle size (μm)[c]
1T30	305	0.681	8.9	6.2	0.0267
2H20	195	0.471	9.7	6.2	0.029
3dd-Prol-T30	72	0.457	72	6.2	0.216
4TE-Prol-H20	61	0.27	47	5.6	0.141
5dd-Prol-H20	49	0.363	69.3	6.2	0.21
6DD-Taur-H20	70	0.35	75.5	6.2	0.227
7DD-Taur-T30	63	0.496	61.2	6.2	0.184

[a]Calculated as [4(Pore volume)/BET]. [b]Corresponds to the interparticle space of silica. [c]Calculated as (3× interparticle pore size).

George Em. Romanos; Peter S. Schulz; Matthias Bahlmann; Peter Wasserscheid; Andreas Sapalidis; Fotios K. Katsaros; Chrysoula P. Athanasekou; Konstantinos Beltsios; N. K. Kanellopoulos; *J. Phys. Chem. C* **2014,** 118, 24437-24451.

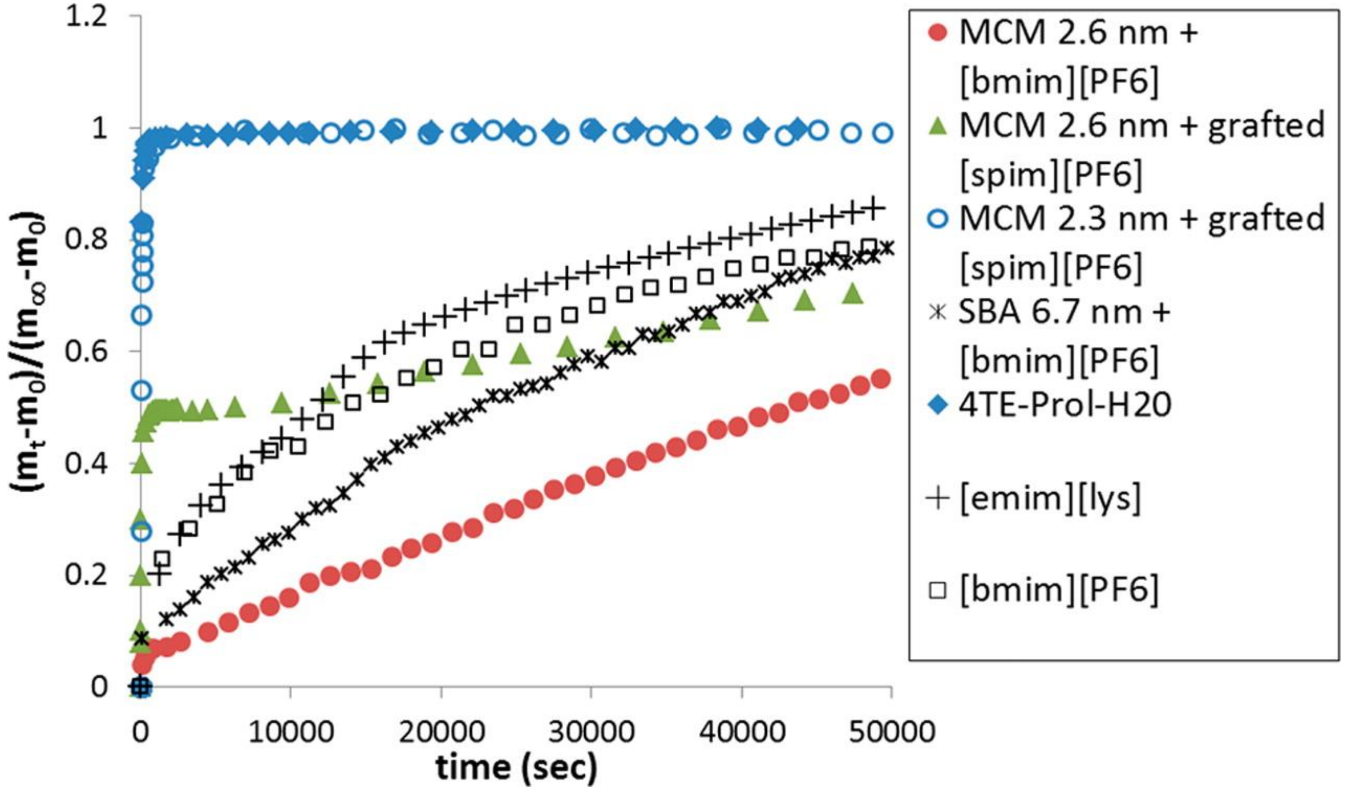
Citosan free inverse SILP materials



Transient curves of CO2 absorption in the “inverse” SILPs at several pressure steps. (a) 3dd-Prol-T30, (b) 3dd-Prol-T30 small pressure steps, (c) 4TE-Prol-H20, and (d) 7DD-Taur-T30. Legends show the respective pressure steps of CO2 (pressure unit = mbar).

George Em. Romanos; Peter S. Schulz; Matthias Bahlmann; Peter Wasserscheid; Andreas Sapalidis; Fotios K. Katsaros; Chrysoula P. Athanasekou; Konstantinos Beltsios; N. K. Kanellopoulos; *J. Phys. Chem. C* **2014,** 118, 24437-24451.

SILP vs. Inverse SILP



CO_2 absorption transients of conventional SILPs, “inverse” SILPs, and the relevant bulk ILs for the first pressure step on a fresh (regenerated) sample.

George Em. Romanos; Peter S. Schulz; Matthias Bahlmann; Peter Wasserscheid; Andreas Sapalidis; Fotios K. Katsaros; Chrysoula P. Athanasekou; Konstantinos Beltsios; N. K. Kanellopoulos; *J. Phys. Chem. C* **2014,** 118, 24437-24451.

- **CO2-facilitated transport performance of poly(ionic liquids) in supported liquid membranes** : J Mater Sci (2015) 50:104–111 DOI 10.1007/s10853-014-8570-z
- . **Efficient fixation of CO2 into cyclic carbonates catalyzed by hydroxyl-functionalized poly(ionic liquids)** DOI: 10.1039/C3RA21872D (Paper) RSC Adv., 2013, **3**, 3726-3732
- **Syntheses and characterization of new poly(ionic liquid)s designed for CO2 capture** DOI: 10.1039/C4RA00071D (Paper) RSC Adv., 2014, **4**, 18164-18170

- **1 mol = 44g = 6.1 cm^3**

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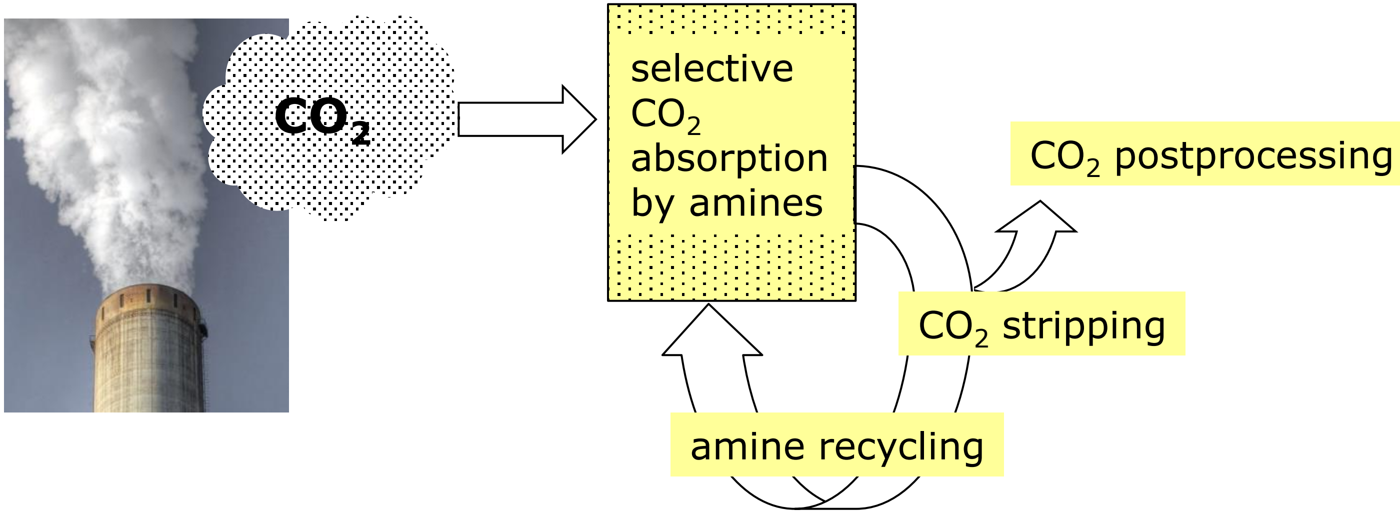
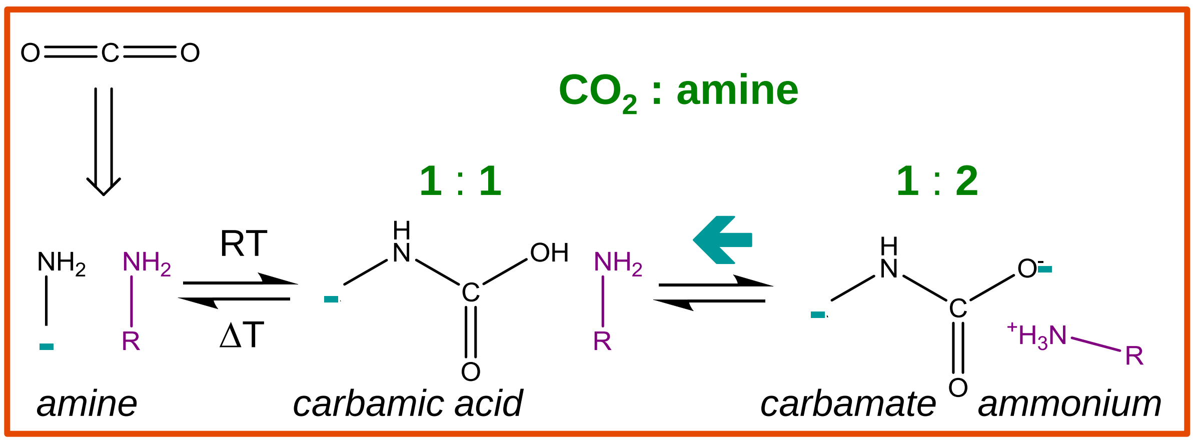
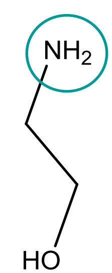
In-situ XPS: amine reaction with CO_2

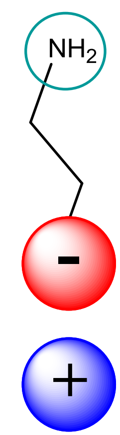




MEA (mono-ethanolamine)



amine-functionalised IL



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Joan Brennecke (Notre Dame)
*→ B. Gurkan et al., J. Am. Chem. Soc. **2010**, 132, 2116–2117*

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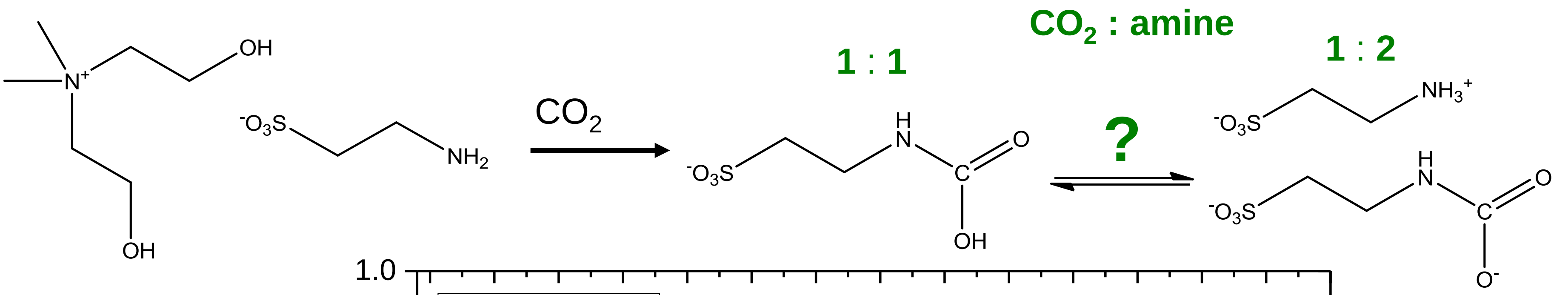
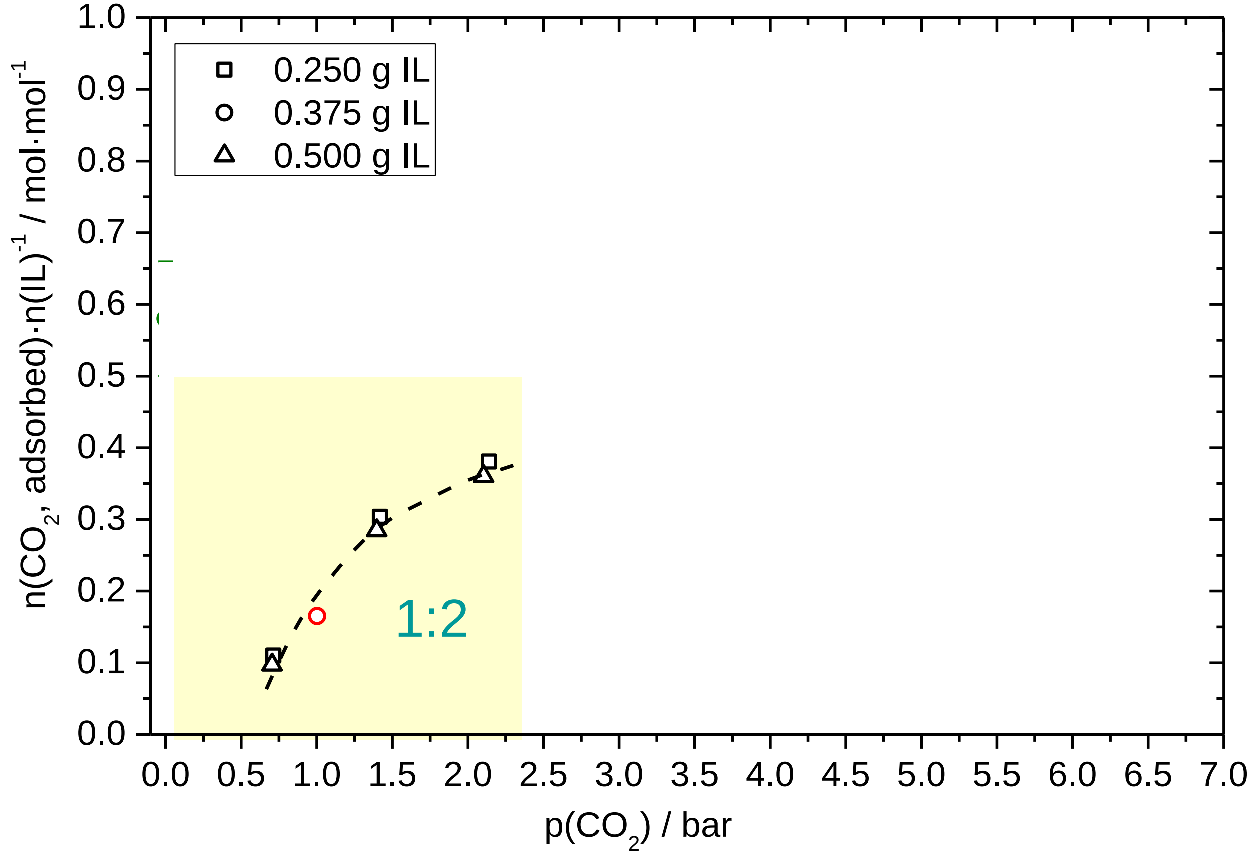
CO_2 equilibrium adsorption in bulk (ex situ)



IL bulk uptake (310 K)



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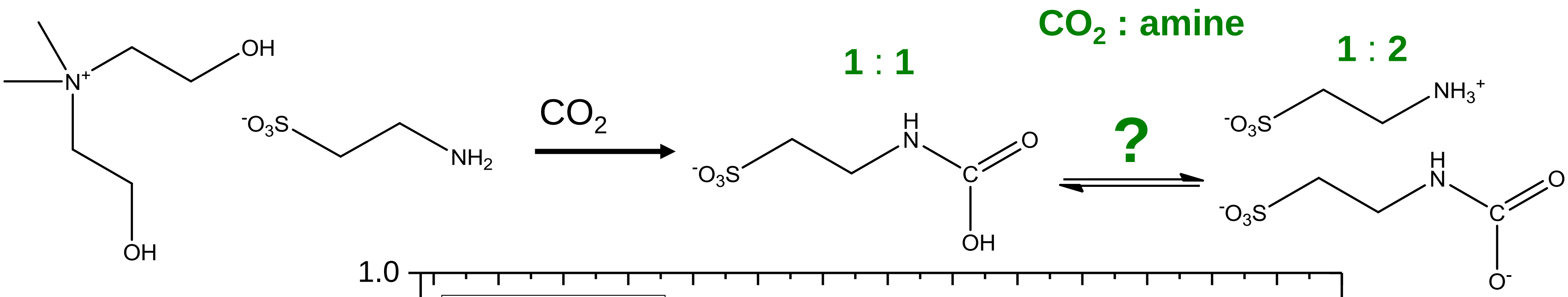
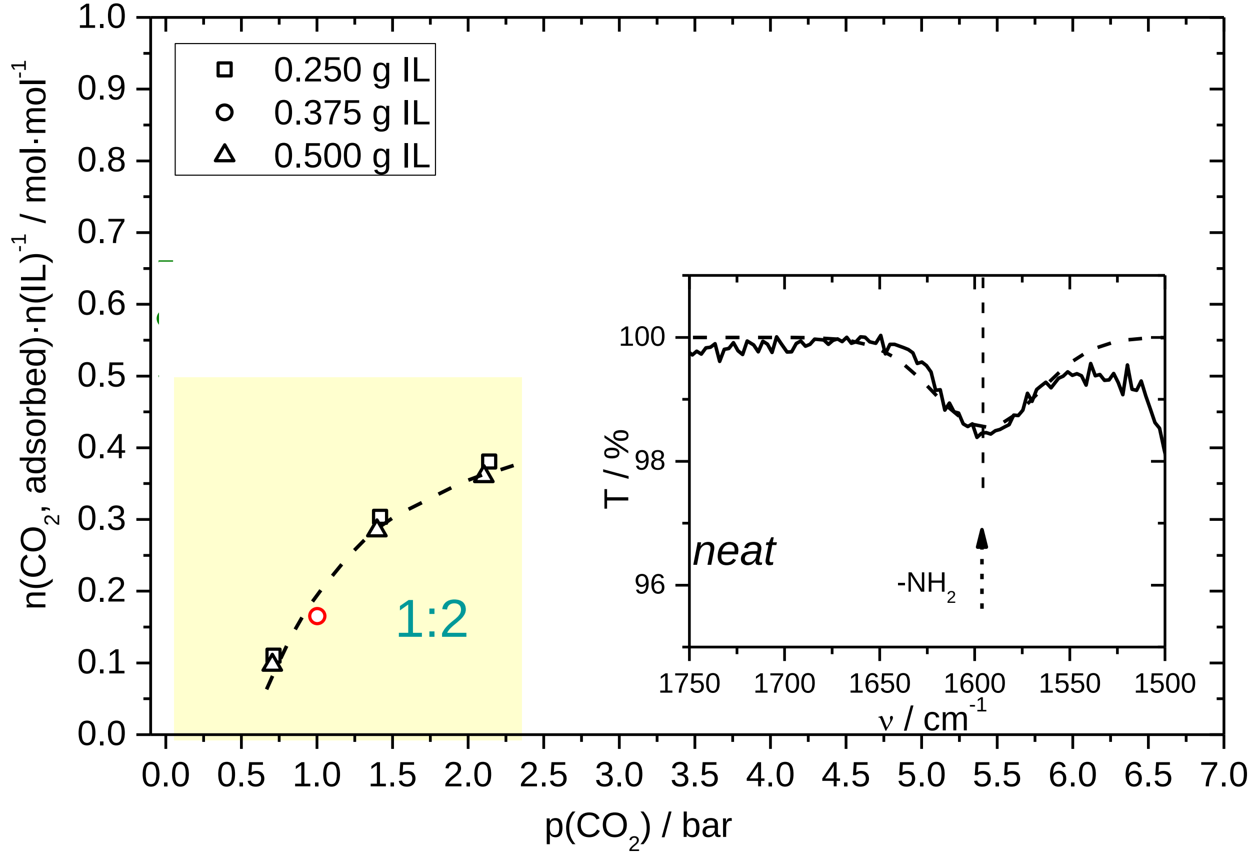
CO_2 equilibrium adsorption in bulk (ex situ)



IL bulk
uptake
(310 K)



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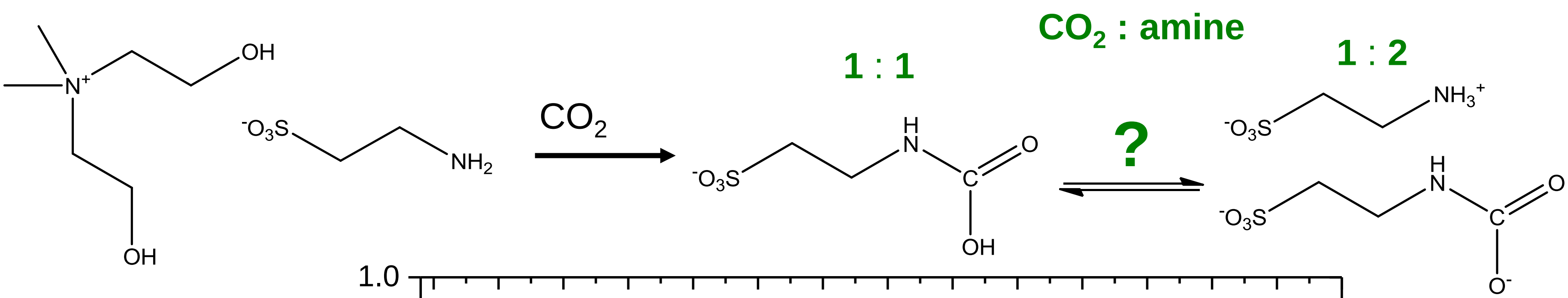

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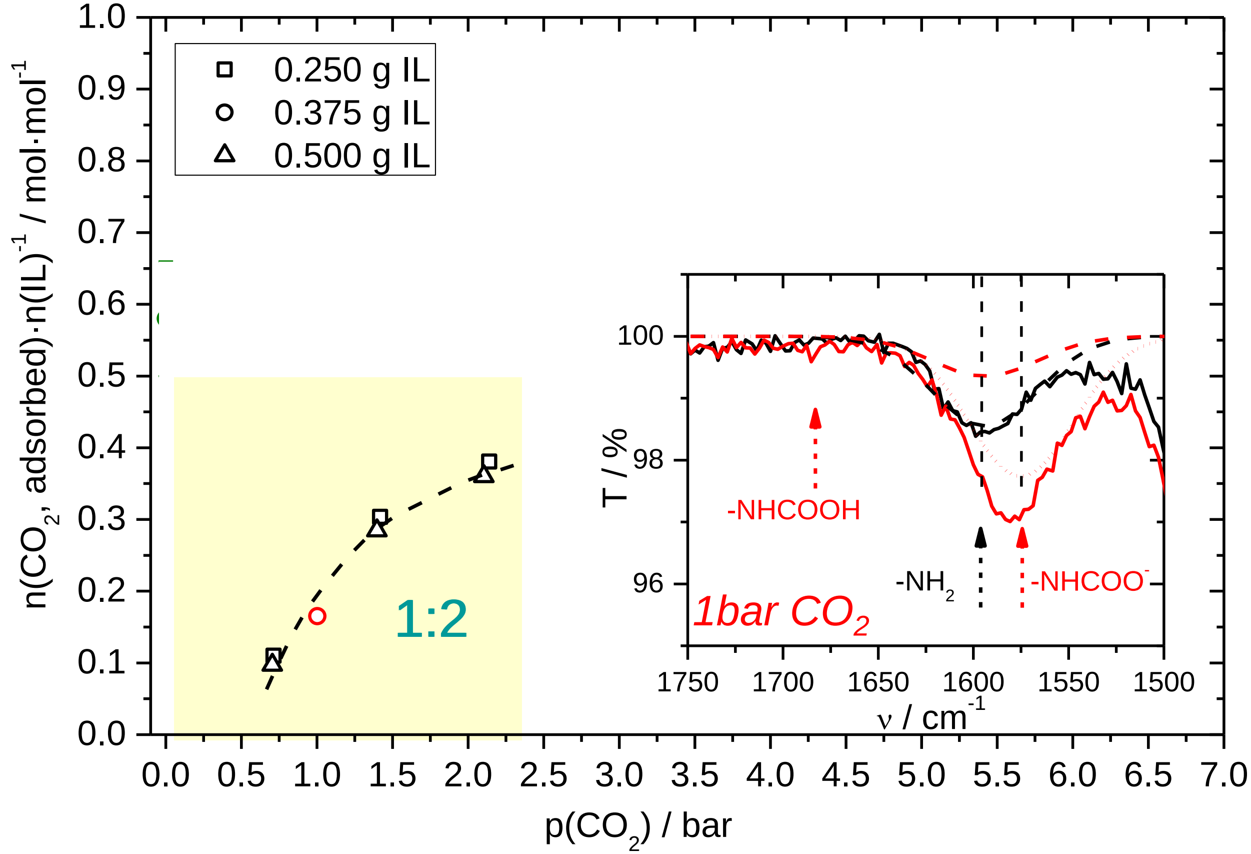

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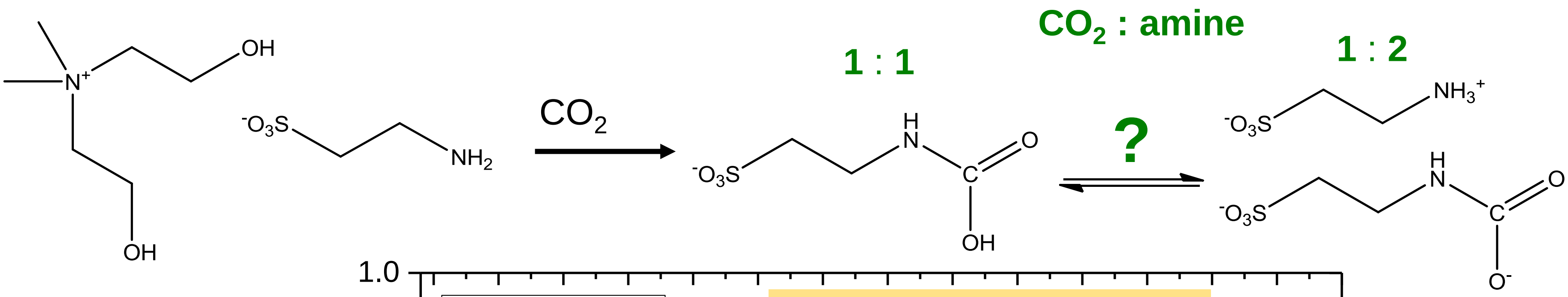

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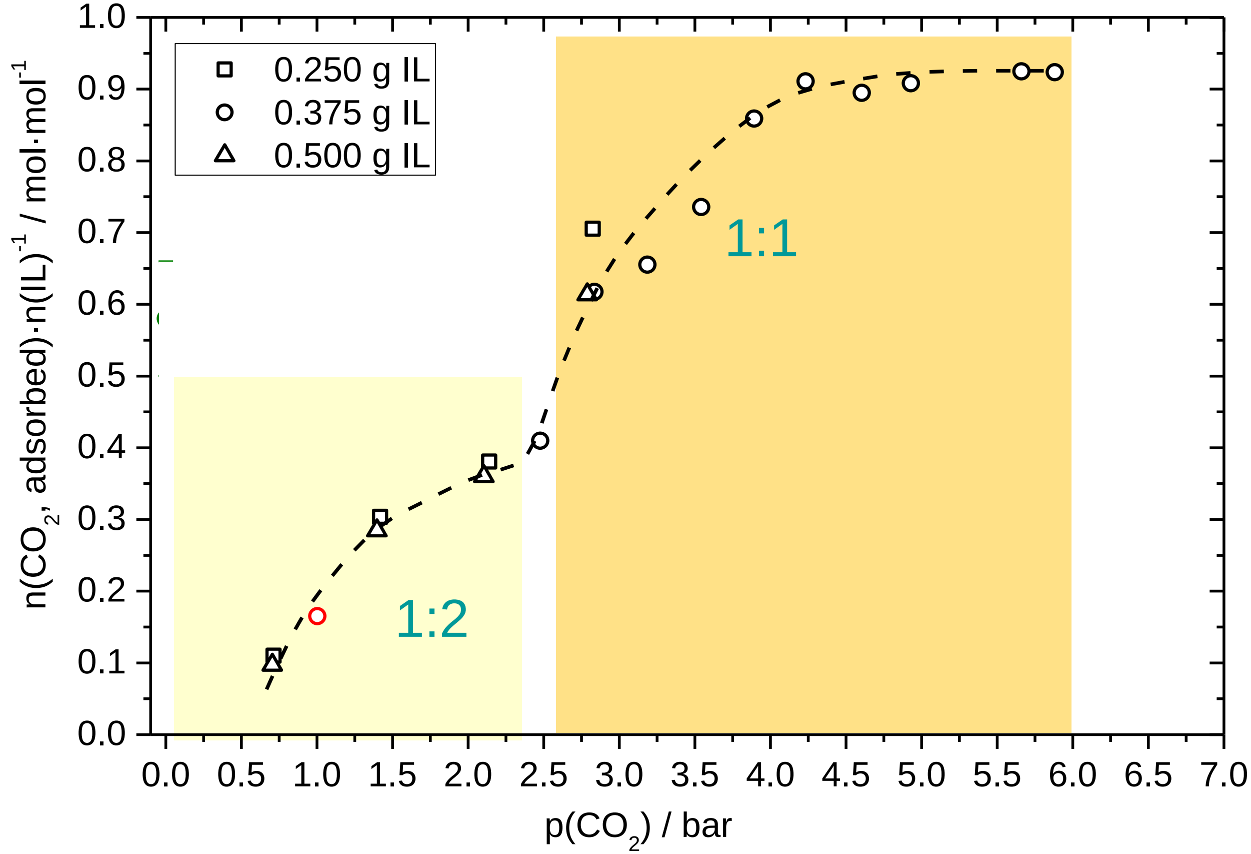
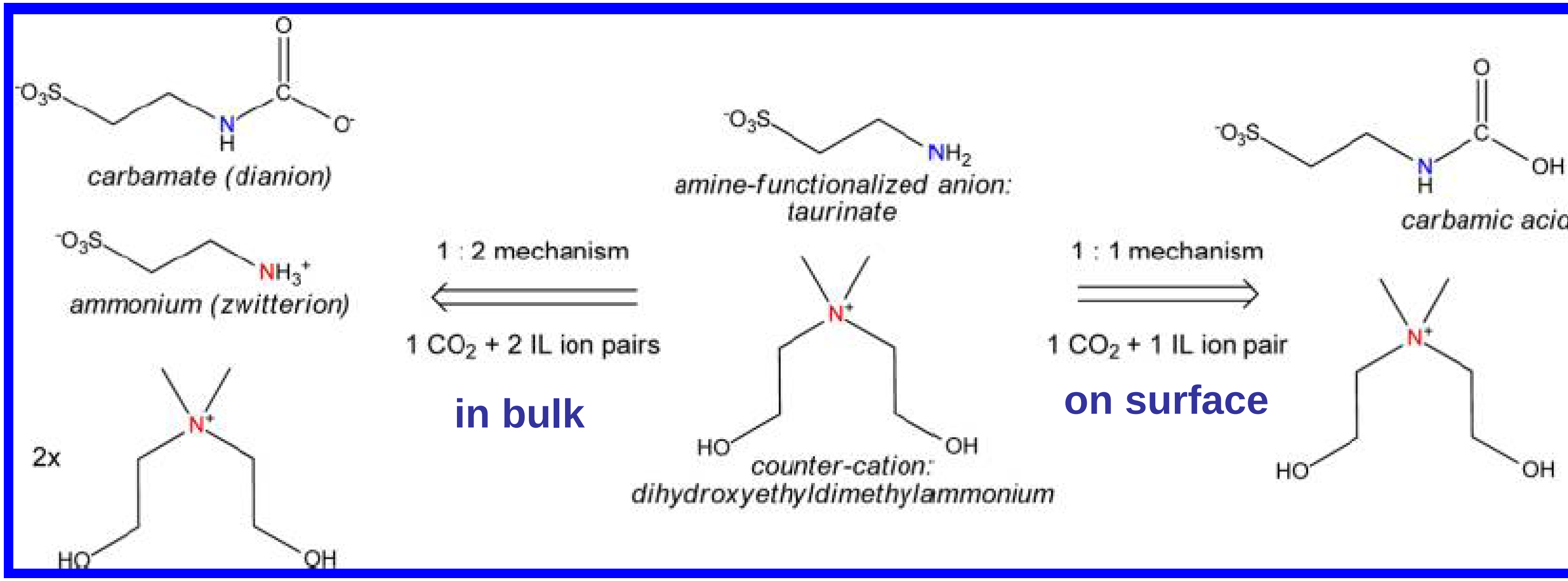


➔ ***p < 2.5 bar: 1 : 2 mechanism in bulk (carbamate)***

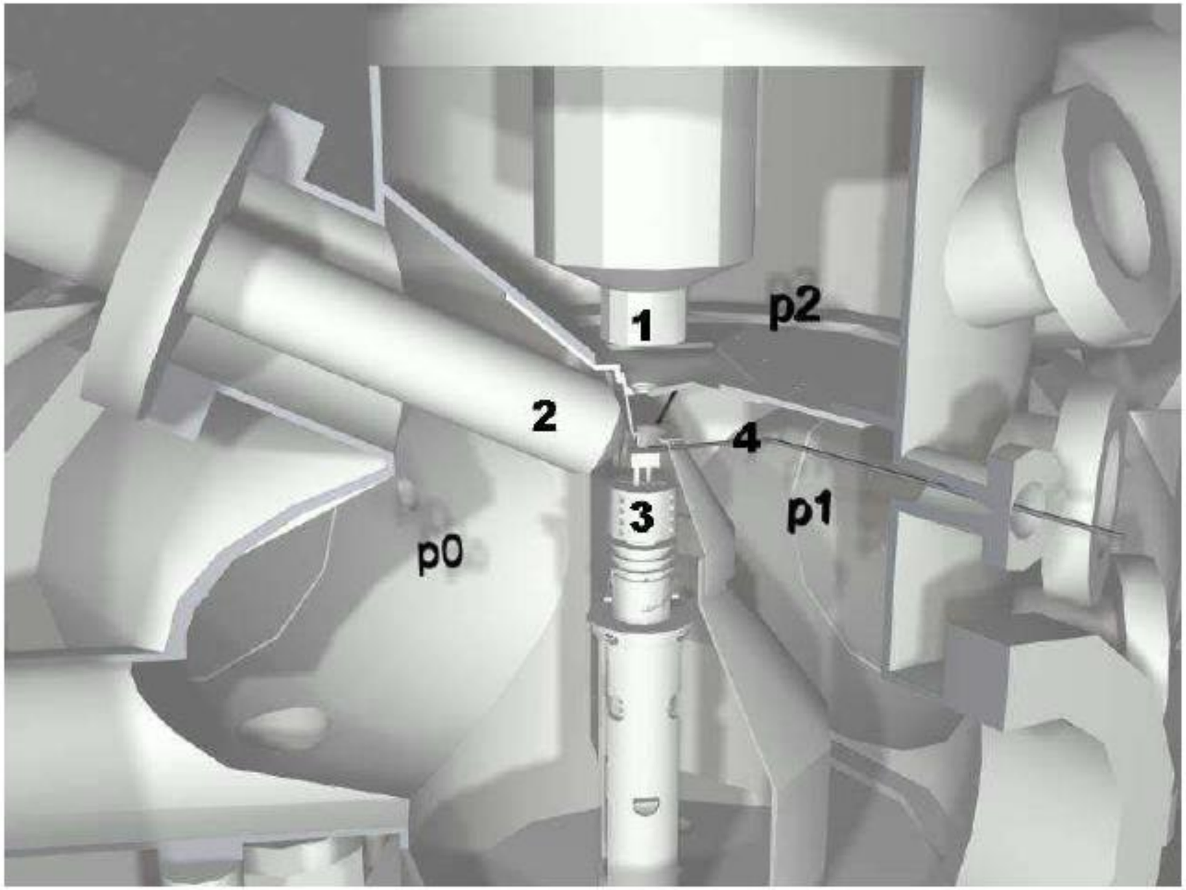
CO_2 Absorption: Bulk vs. Surface

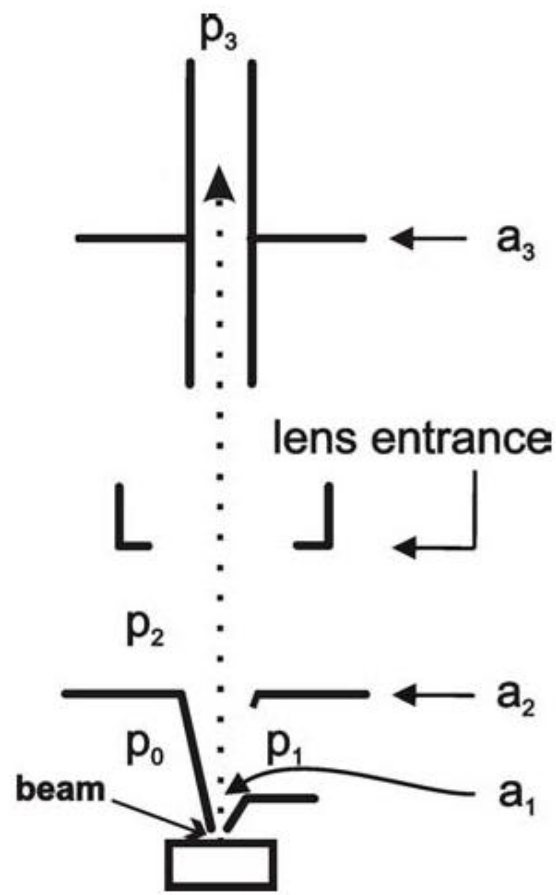


Niedermaier, I.; Bahlmann, M.; Papp, C.; Kolbeck, C.; Wei, W.; Calderón, S. K.; Grabau, M.; Schulz, P. S.; Wasserscheid, P.; Steinrück, H.-P.; Maier, F.
J. Am. Chem. Soc. **2014**, 136, 436−441

In-situ XPS: amine reaction with CO_2

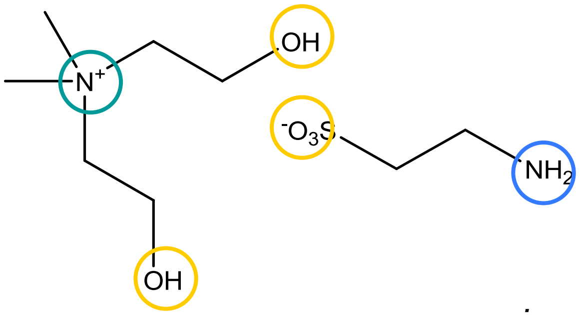
near-ambient pressure XPS: 0.9 mbar CO_2
(atmosphere: ~0.4 mbar partial pressure)





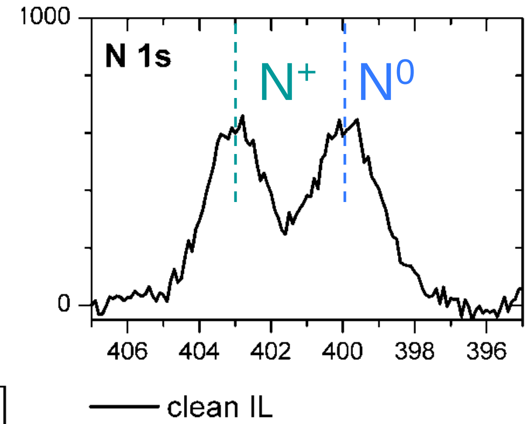
J. Pantförder, *Photoelektronenspektroskopie im „Pressure Gap“ – Aufbau einer neuen Apparatur für Messungen im Druckbereich von 10-10 bis 1 mbar*, PhD thesis, **2004**, Uni Erlangen-Nürnberg

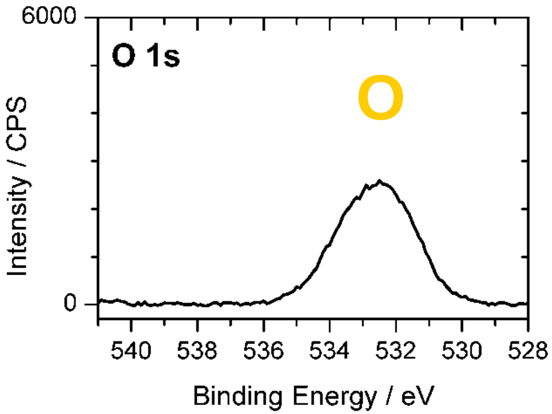
In-situ XPS: amine reaction with CO_2



degassed in UHV at 390K for 2h
measured at RT

amine





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→ *Niedermaier, J. Am. Chem. Soc., 2014, 136, 436*

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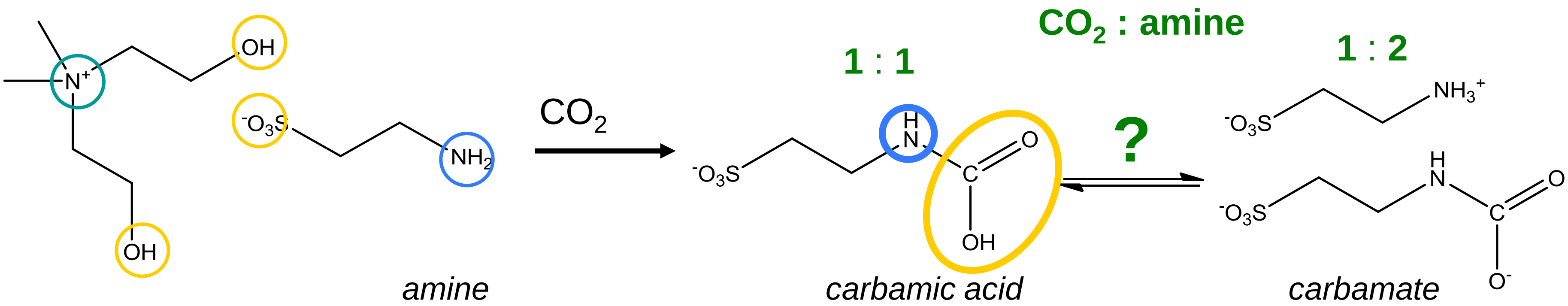
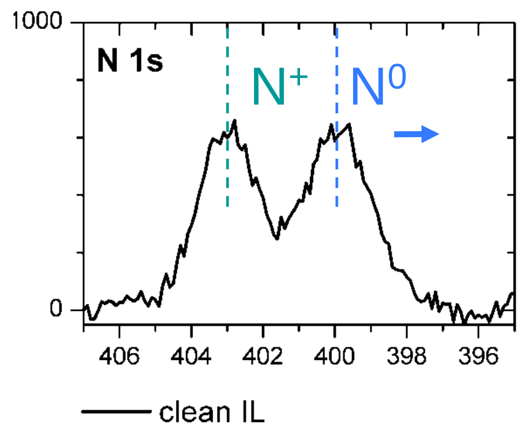
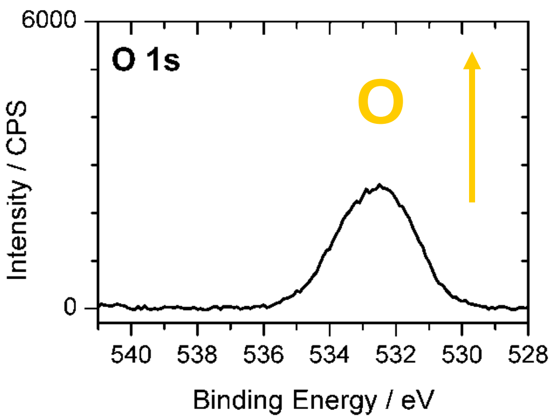
In-situ XPS: amine reaction with CO_2







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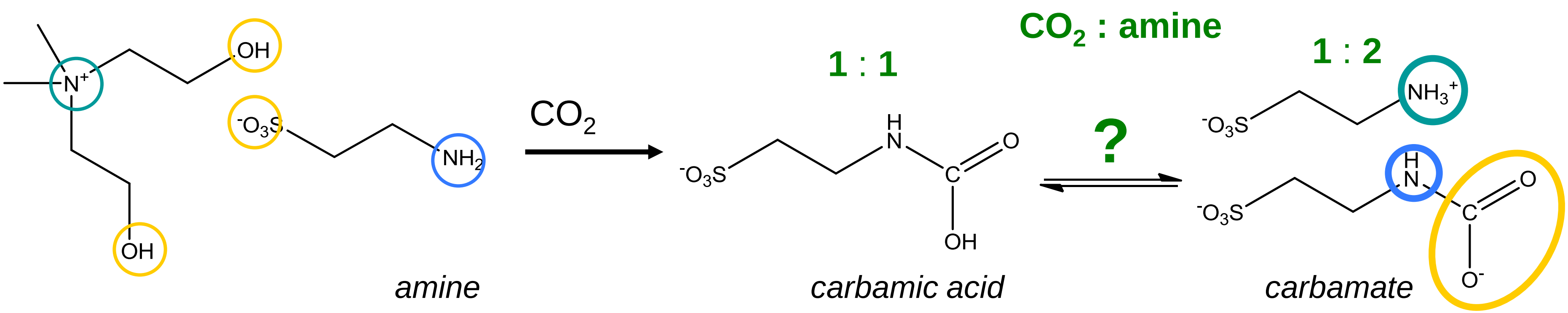

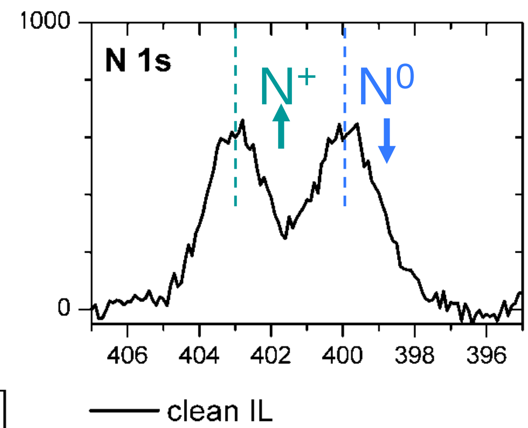

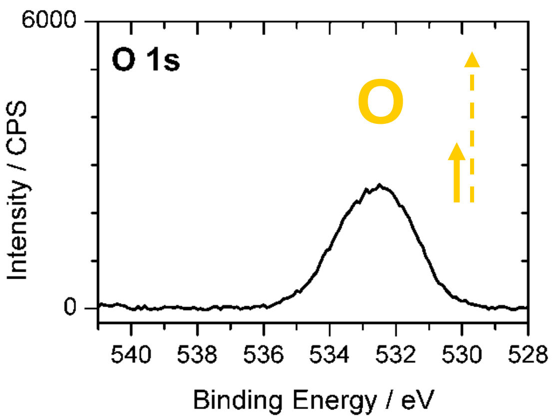

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→ *Niedermaier, J. Am. Chem. Soc., 2014, 136, 436*

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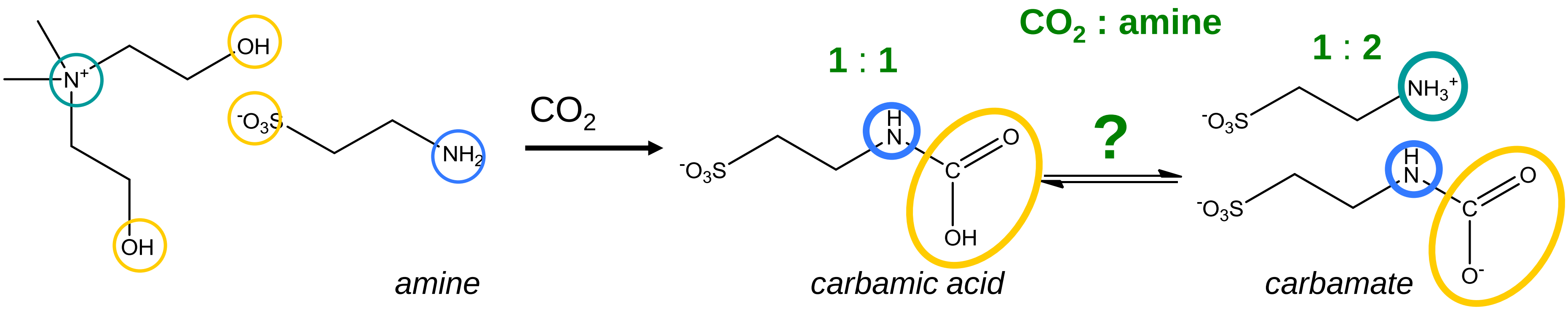
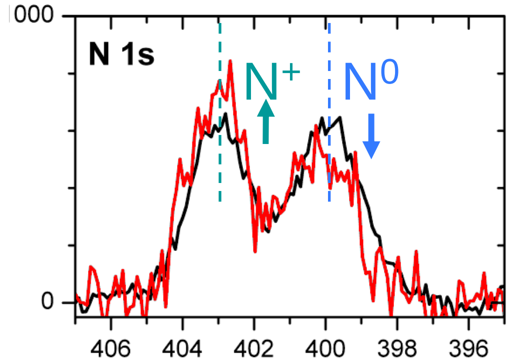
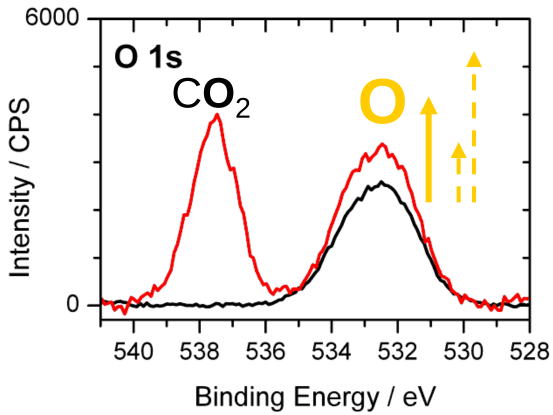




clean IL
during 0.9 mbar CO_2
(corrected for damping)



- O increase → 0.6 CO_2 : 1 IL pair
- N change → 0.15 carbamate + 0.15 ammonium

➔ 0.15 CO_2 carbamate (+0.15 amm.)
0.45 CO_2 carbamic acid
0.25 free amine

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→ *Niedermaier, J. Am. Chem. Soc., 2014, 136, 436*

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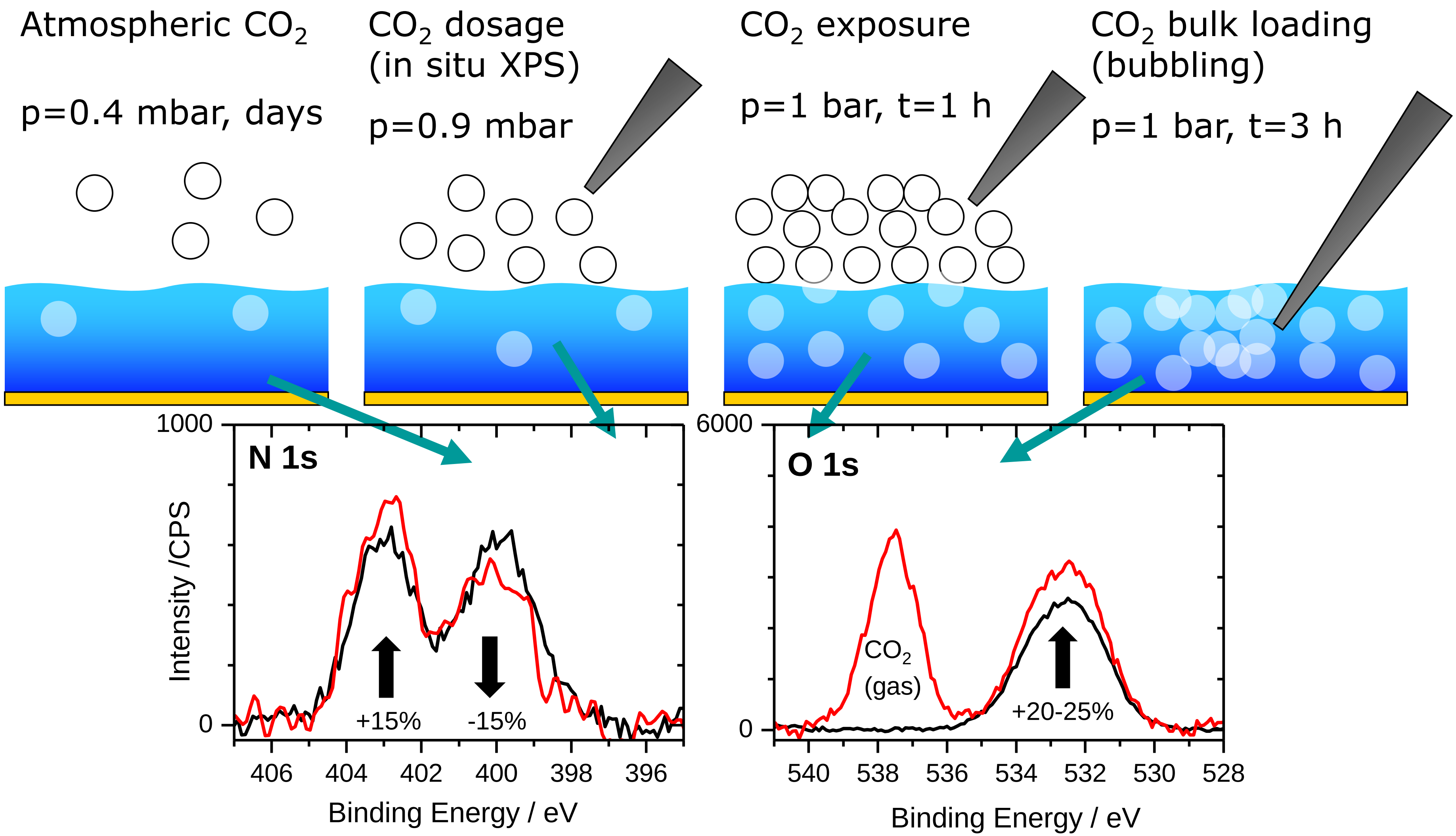
In-situ XPS: amine reaction with CO_2



➔ *at RT equilibrium distribution in near-surface region*

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→ *Niedermaier, J. Am. Chem. Soc., 2014, 136, 436*

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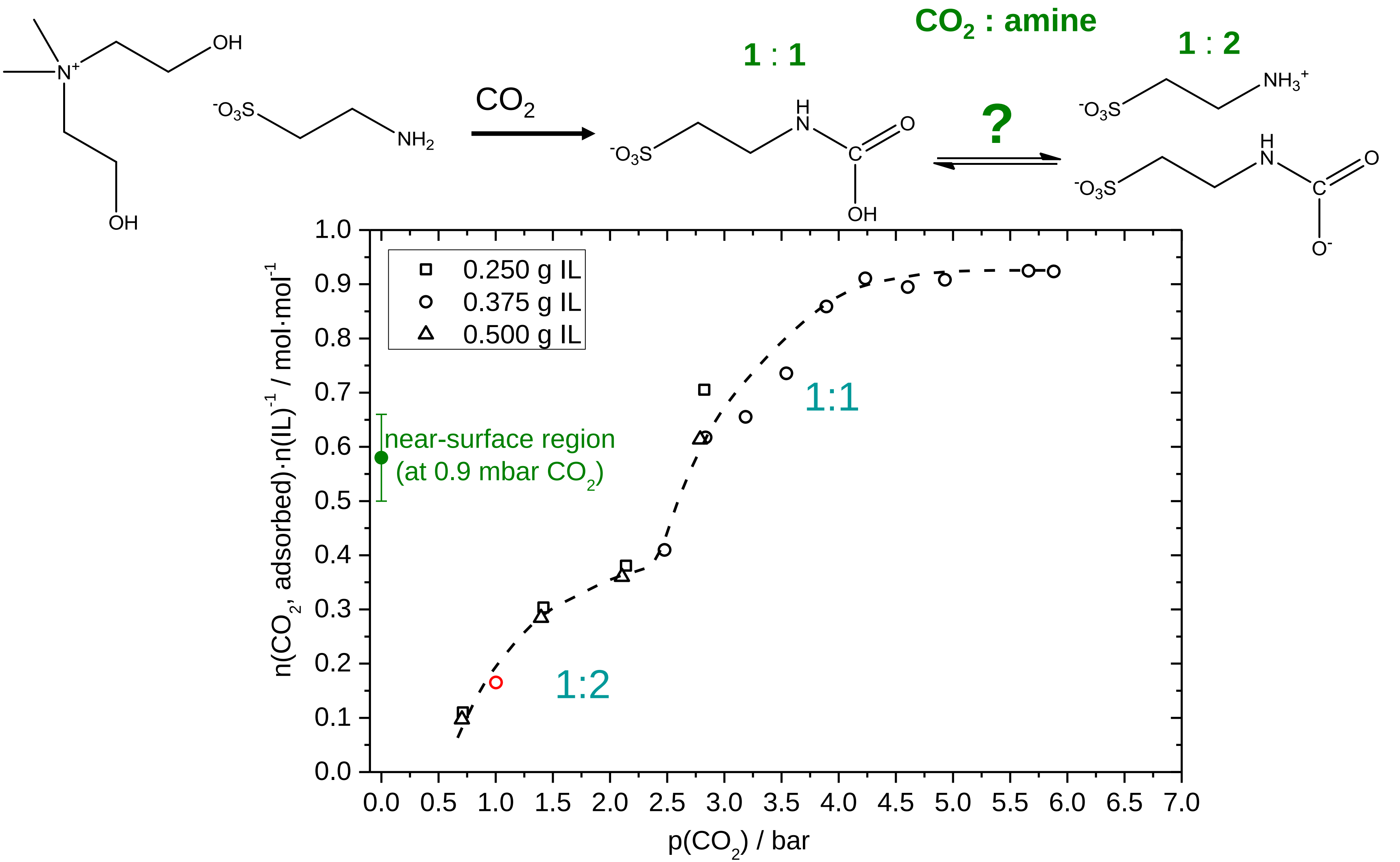
Surface (in situ) vs. bulk (ex situ)



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- ➔ *in bulk at 0.9 mbar , no CO_2 (< 2.5 bar: 1 : 2 uptake, carbamate)*
- ➔ *at surface: high CO_2 (1 : 1, carbamic acid dominating)*

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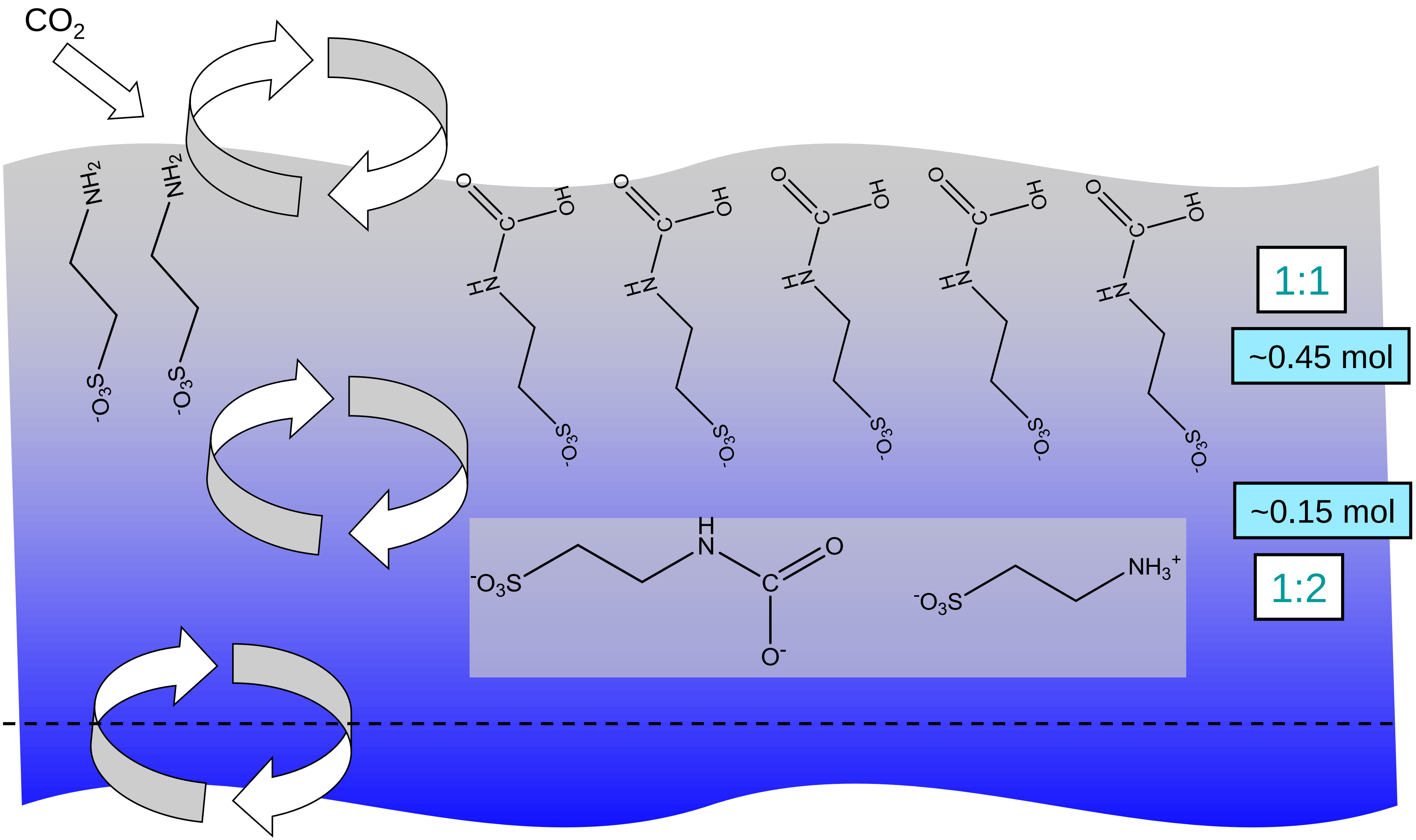
Charge distribution as possible driving force



→ *Niedermaier, J. Am. Chem. Soc., 2014, 136, 436*

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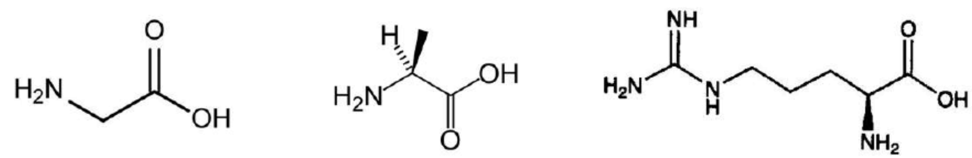
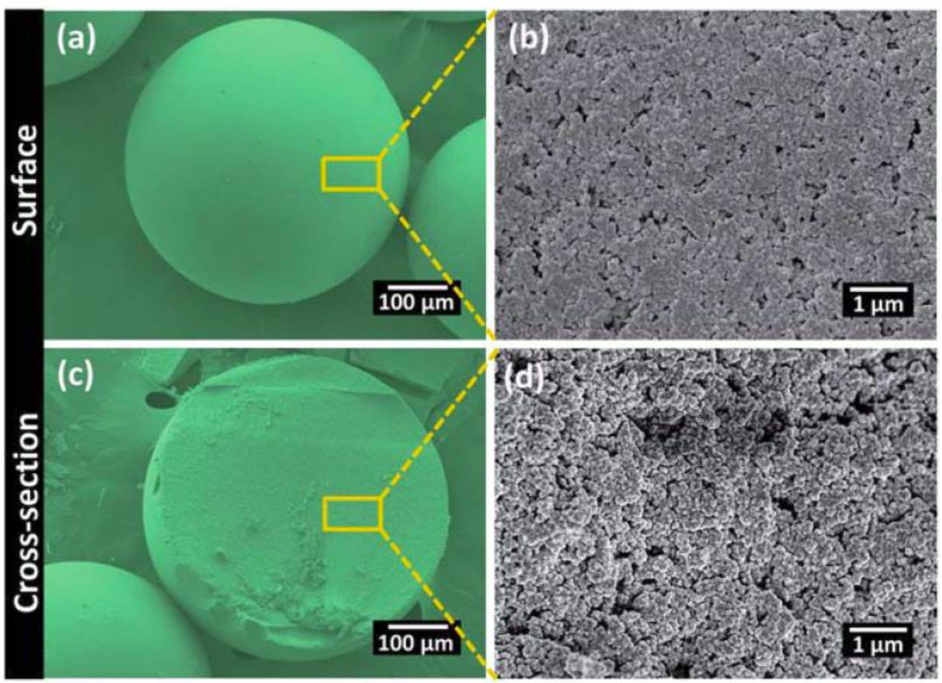
Impact on high surface area sorbents

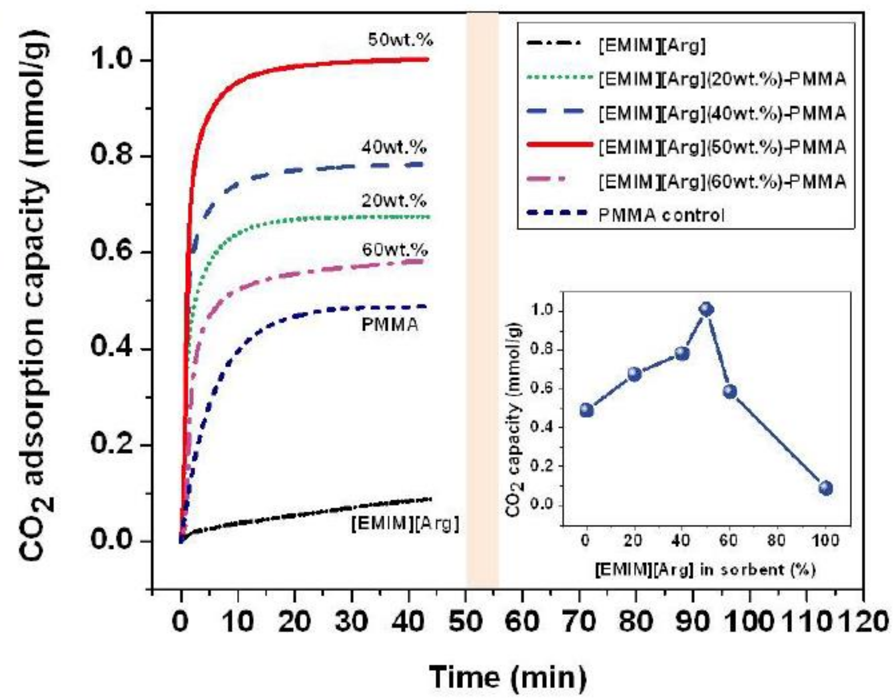
Nanoporous PMMA microspheres impreganted with $[C_2C_1Im][AA]$

$[C_2C_1Im][OH]$ +



Glycine Alanine Arginine





→ *Wang, J. Mat. Chem. A, 2013, 1, 2978*

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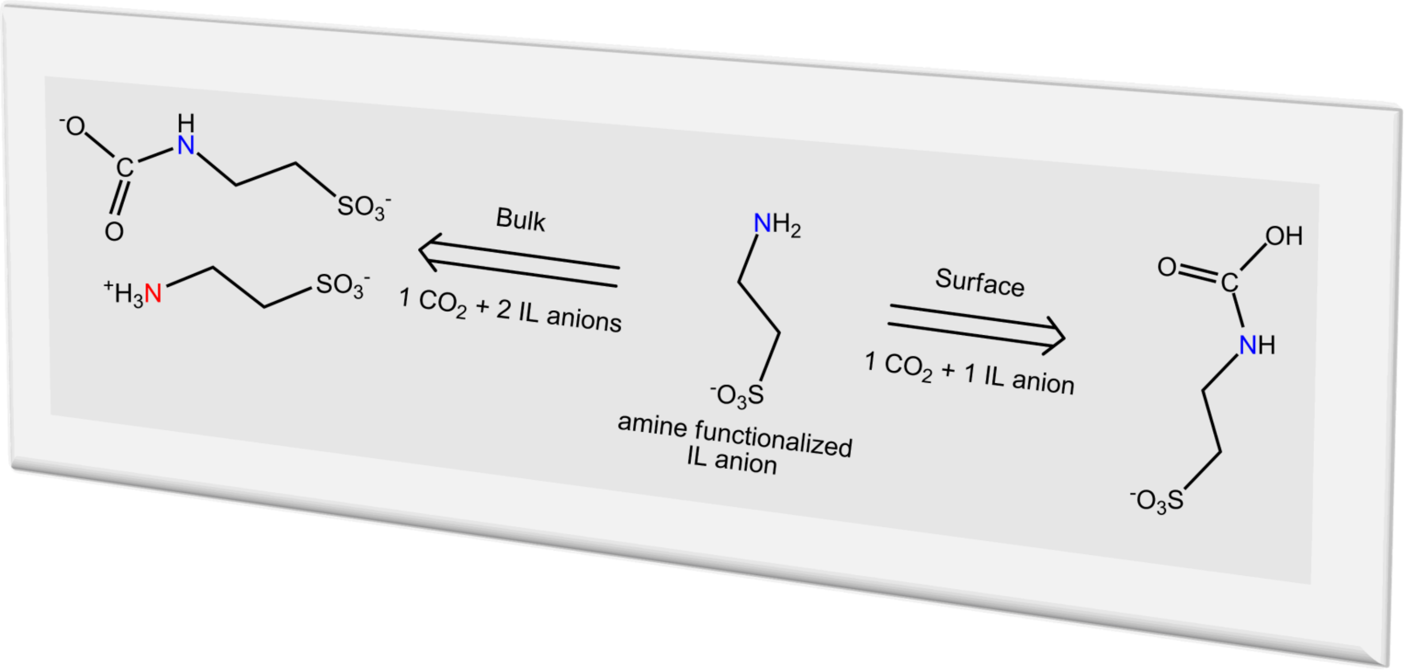
Summary



- *in situ* reaction of amine functionalised IL and CO_2 by near-ambient pressure XPS
- carbamic acid (1 : 1 mechanism) dominating at liquid surface
  → much higher CO_2 content in surface layers even at 1 mbar
- near-surface region: quasi-equilibrium situation
- bulk species depends on p(CO_2), only carbamate at low p

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→ *Niedermaier, J. Am. Chem. Soc., 2014, 136, 436*

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Coauthors and acknowledgements


I. Niedermaier


C. Papp


H.-P. Steinrück


S. Krick


M. Grabau

ENGINEERING OF ADVANCED MATERIALS

IONIC LIQUIDS
DFG – SPP 1191


M. Bahlmann


P.S. Schulz


P. Wasserscheid

Supported by: German Research Foundation (DFG)

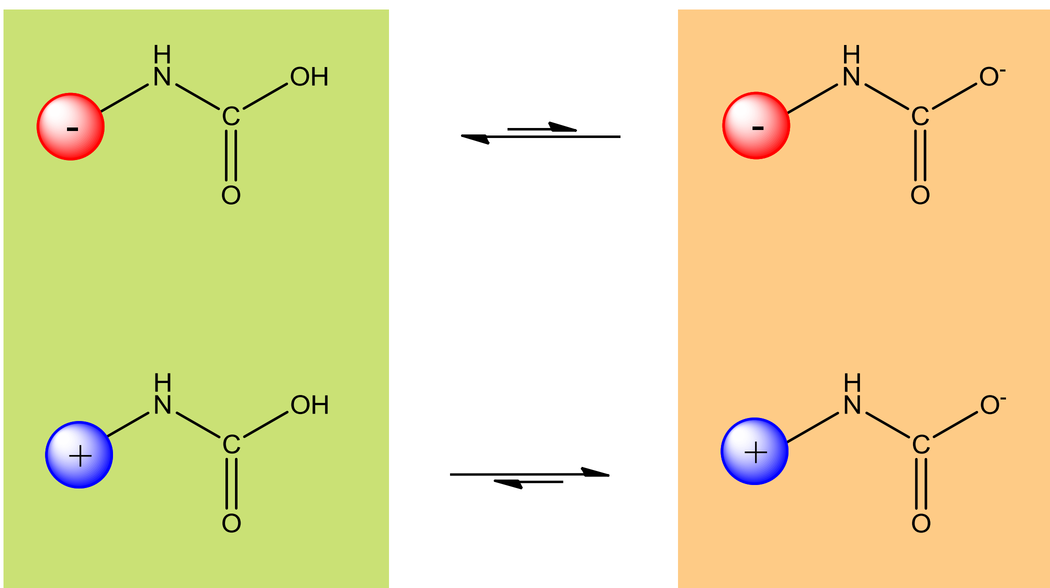
Carbamate vs. carbamic acid

Carbamic acid

$R\text{-}NH_2 + CO_2 \rightarrow$ R-NHCOOH ⇒ 1:1 (1 mol CO_2 per 1 mol IL)

$R\text{-}NHCOOH + R\text{-}NH_2 \rightarrow R\text{-}NHCOO^- + R\text{-}NH_3^+$ ⇒ 1:2 (1 mol CO_2 per 2 mol IL)

Carbamate



1:1 favoured

1:2 favoured

Brennecke group, Notre Dame, Indiana, e.g. B. Gurkan et al., *J. Am. Chem. Soc.* **2010**, 132, 2116–2117

Carbamate vs. carbamic acid

ILs, amine in anion

Soutullo et al., *Chem Mater* **2007**, *19*, 3581

Goodrich et al., *Ind Eng Chem Res* **2011**, *50*, 111

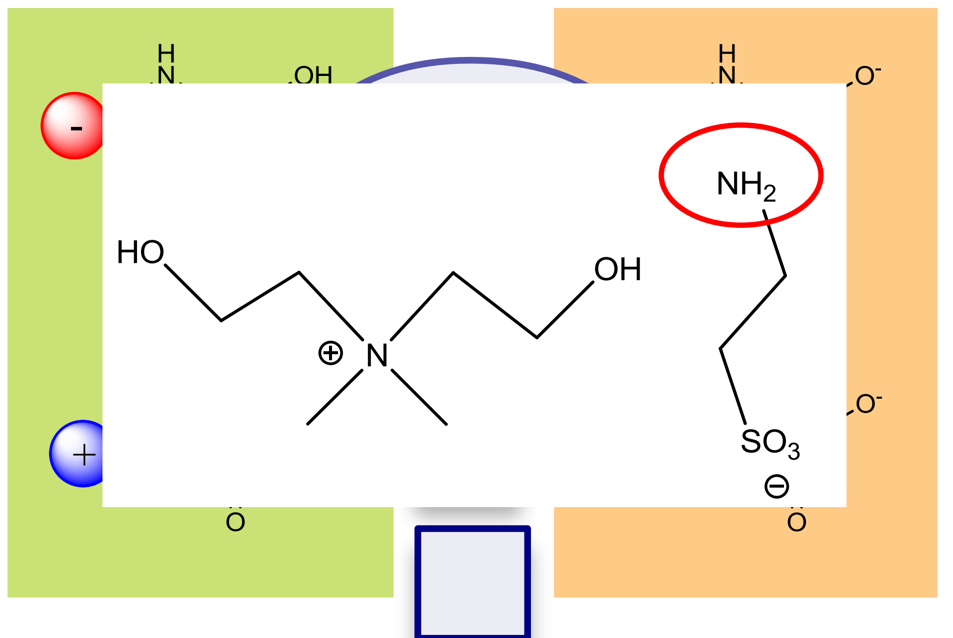
Li et al., *Green Chem* **2008**, *10*, 879

Amine functionalised surfaces:

Knöfel et al., *J Phys Chem C* **2009**, *113*, 21726

Danon et al., *J Phys Chem C* **2011**, *115*, 11540

Bacsik et al., *Langmuir* **2011**, *27*, 11118



Factors determining carbamic acid stability (substituent groups, CO_2 pressure, moisture, and many others …)

e.g. Masuda et al., *Tetrahedron* **2005**, *61*, 213

Goodrich et al., *Ind Eng Chem Res* **2011**, *50*, 111

X-ray photoelectron spectroscopy (XPS)

Experimental:

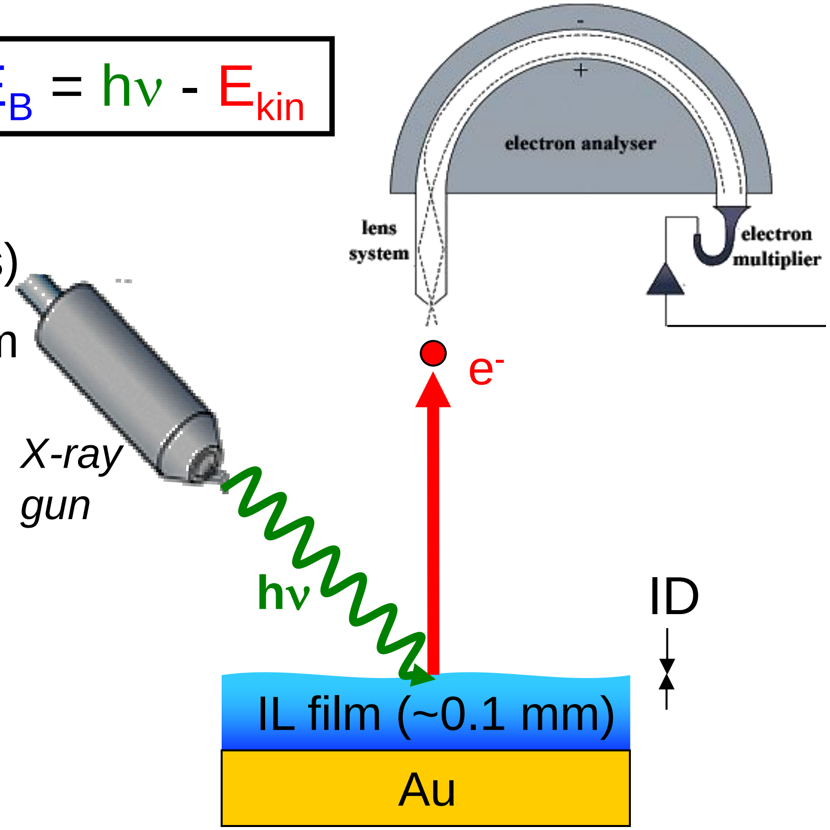
→ VG-ESCALAB 200 (Al K_α, ΔE = 0.9 eV)

→ Sample transfer system

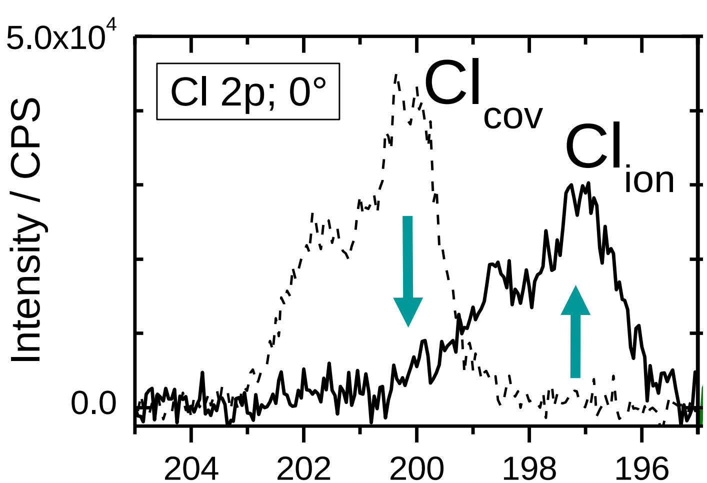
→ Pressure <10^{-8} mbar (clean UHV conditions)

→ Thickness of macroscopic IL-layer: ~ 0.1 mm

$$E_B = h\nu - E_{kin}$$



Cl^0 → Cl^-



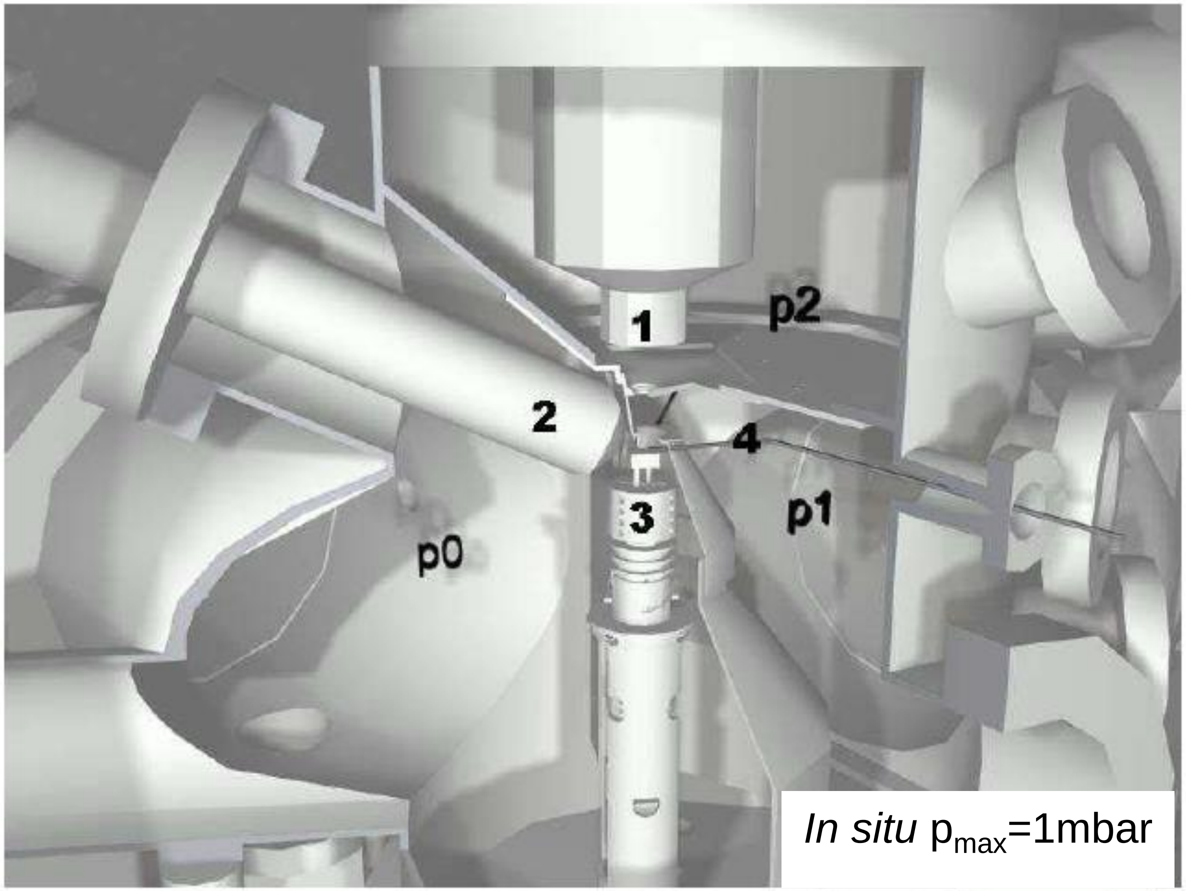
→ Element specific

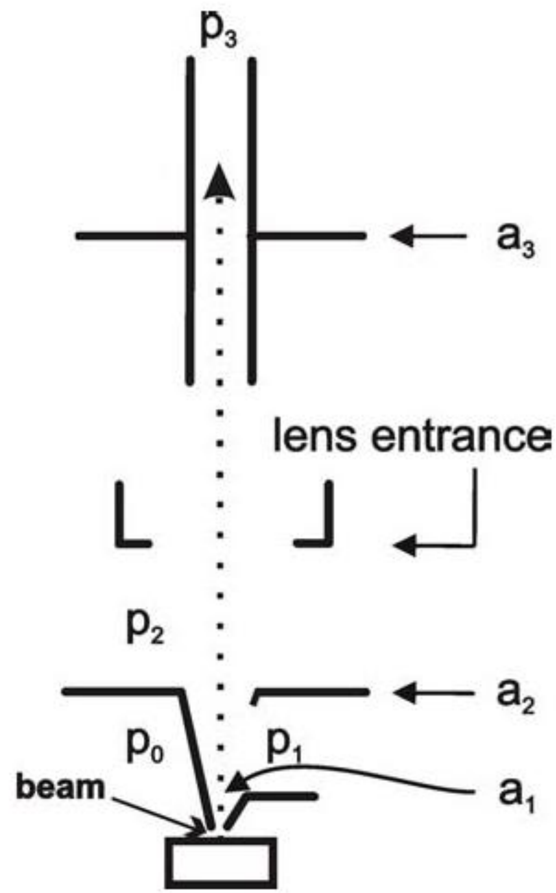
→ Chemical shift

→ Quantitative analysis

→ Surface sensitive (ID_{max} ≈ 7-9nm)

In-situ near ambient pressure XPS



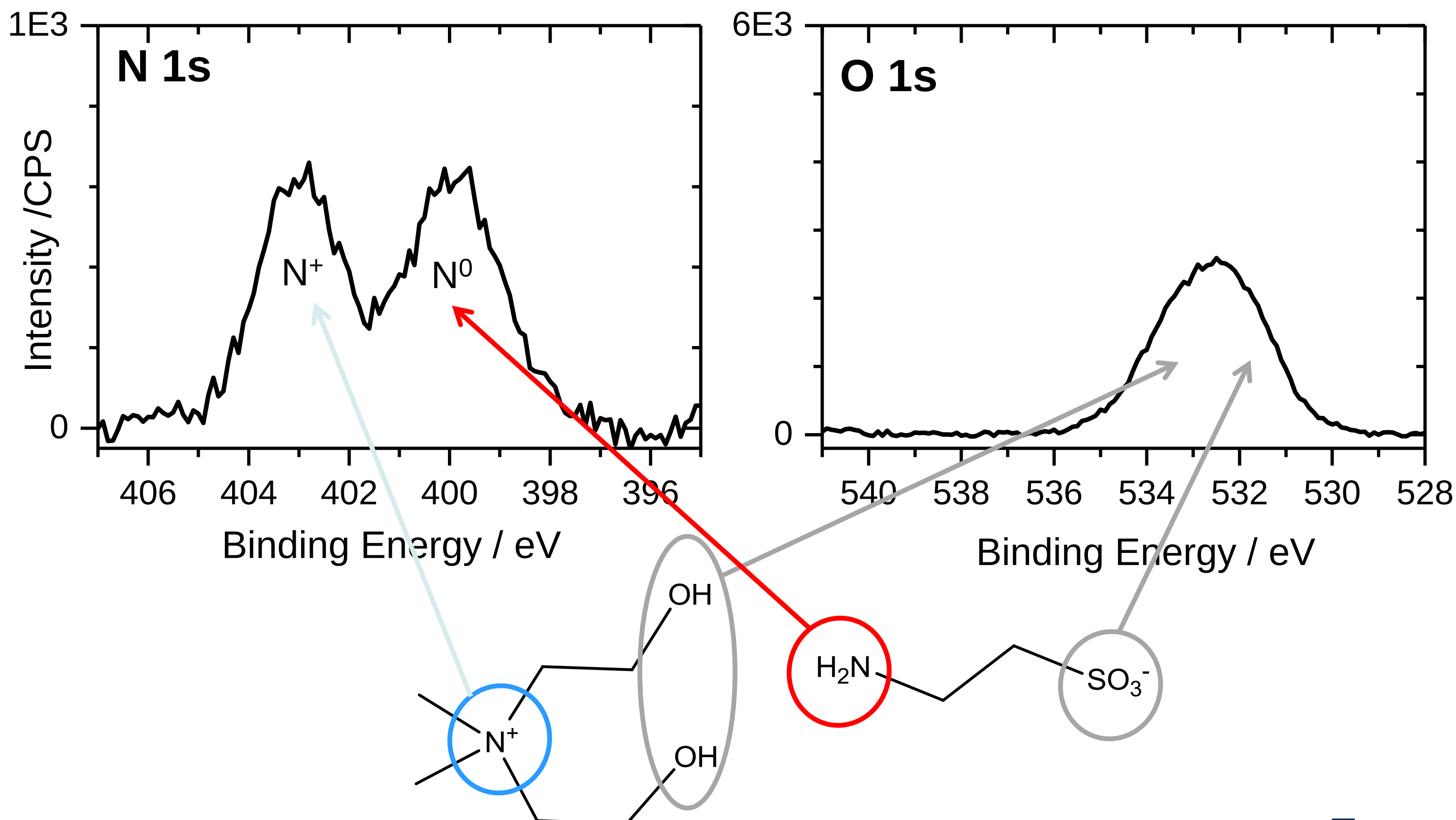


J. Pantförder, **Photoelectron Spectroscopy in the Pressure Gap**

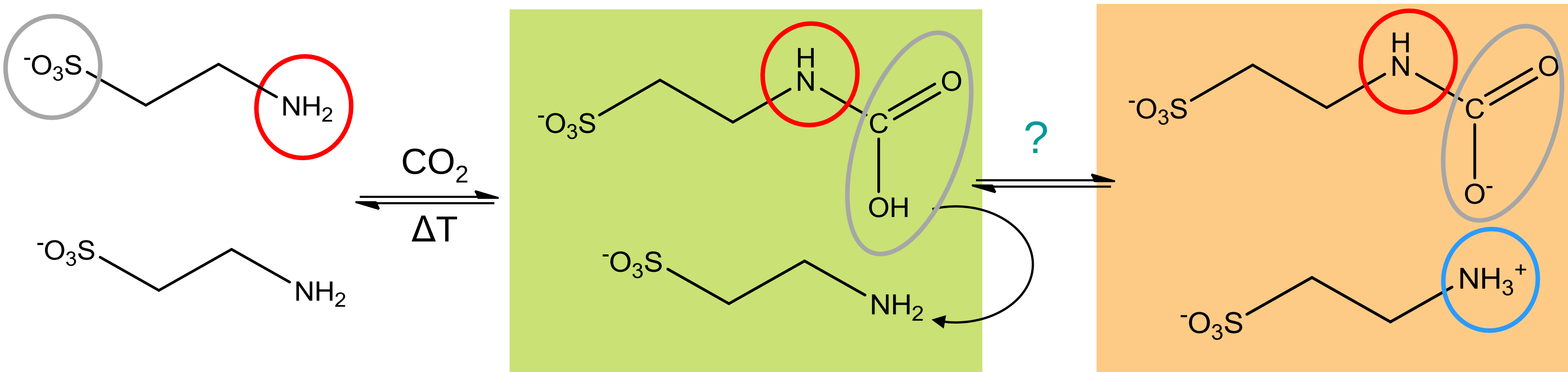
- *PhD thesis,* **2004**, Uni Erlangen-Nürnberg
- *Rev Sci Inst,* **2005**, 76, 014102

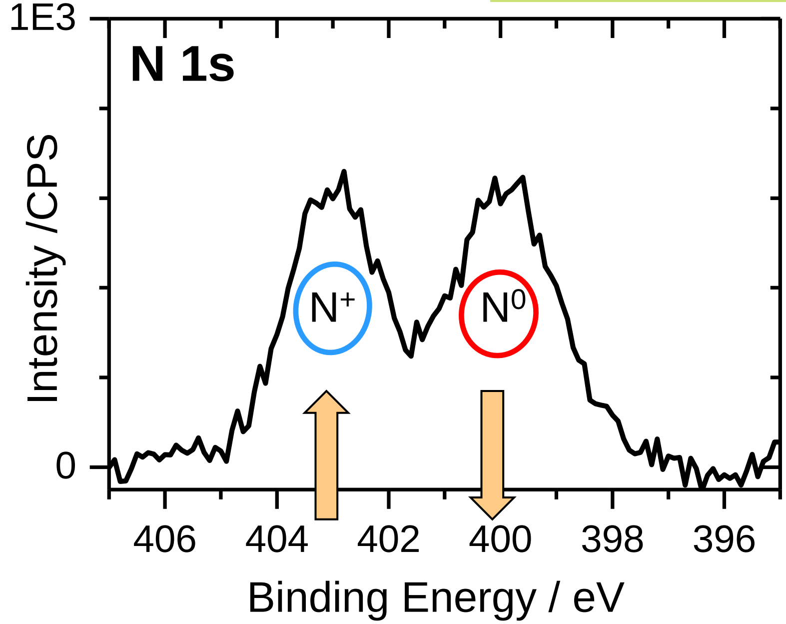
Clean IL after heating for 1h to 390 K

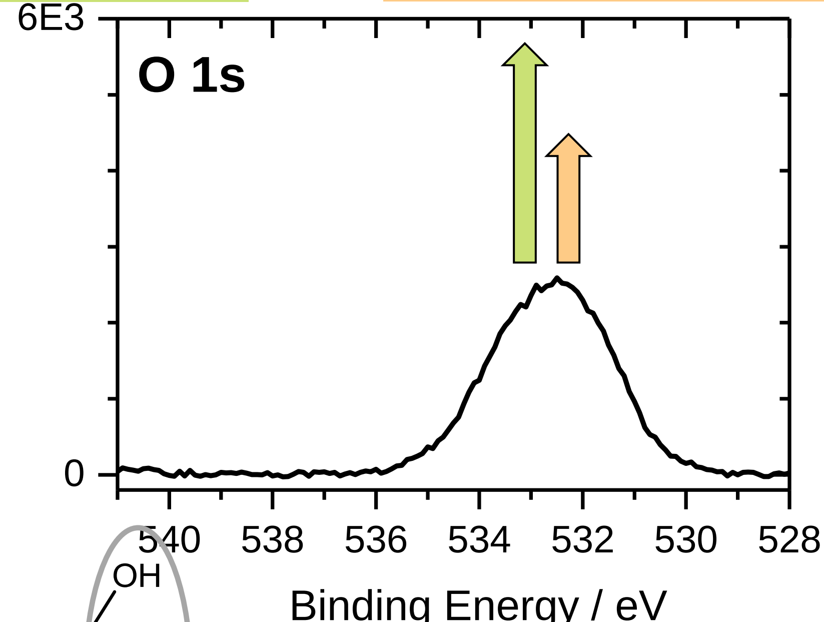
IL surface (ID ~ 7-9 nm)

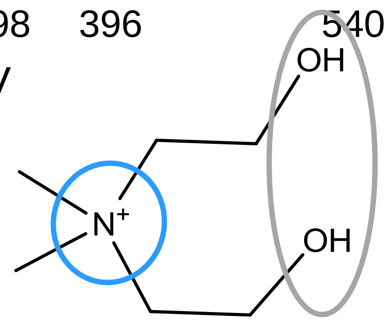


In-situ XPS on amine reaction with CO_2









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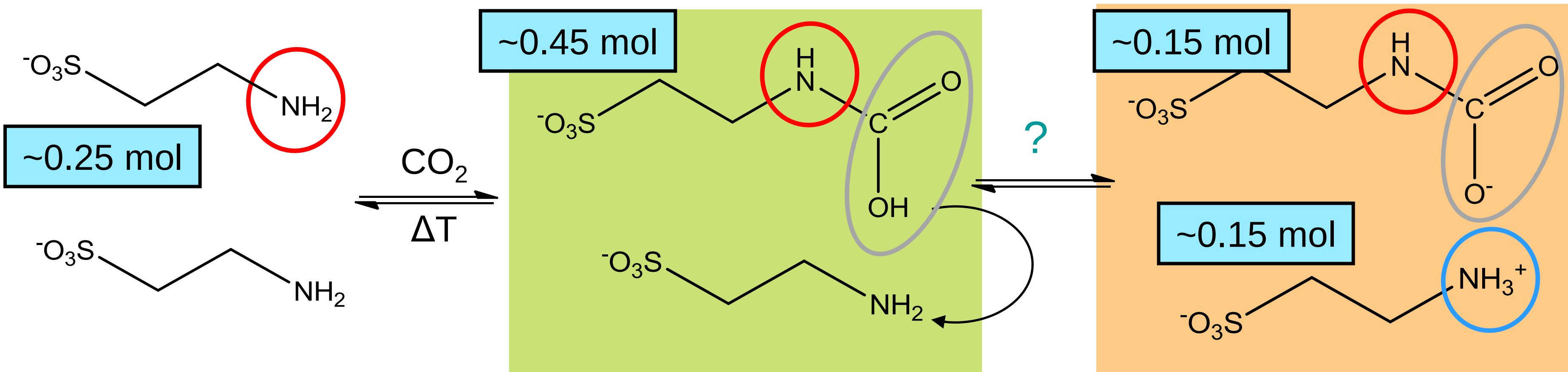
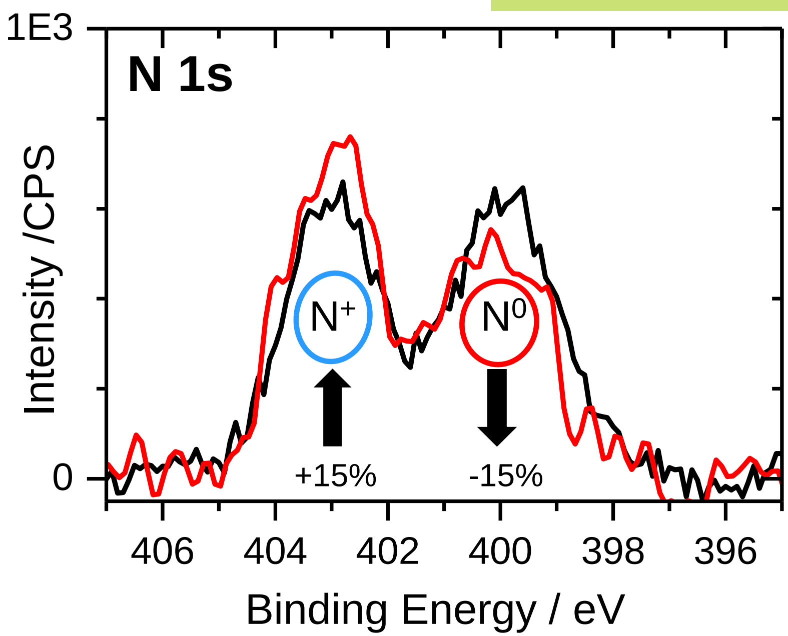
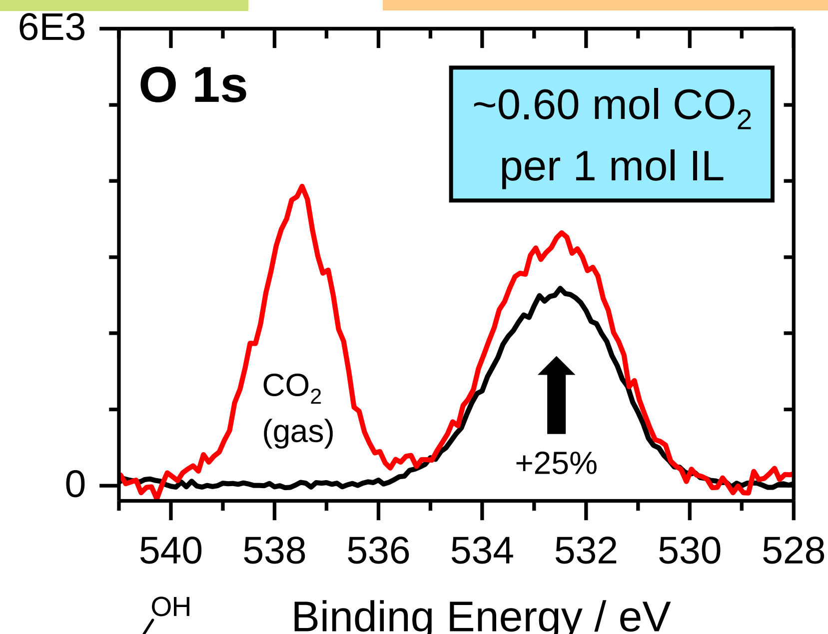
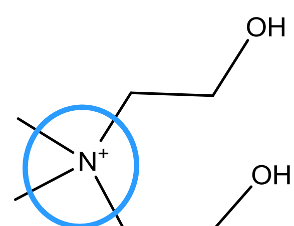




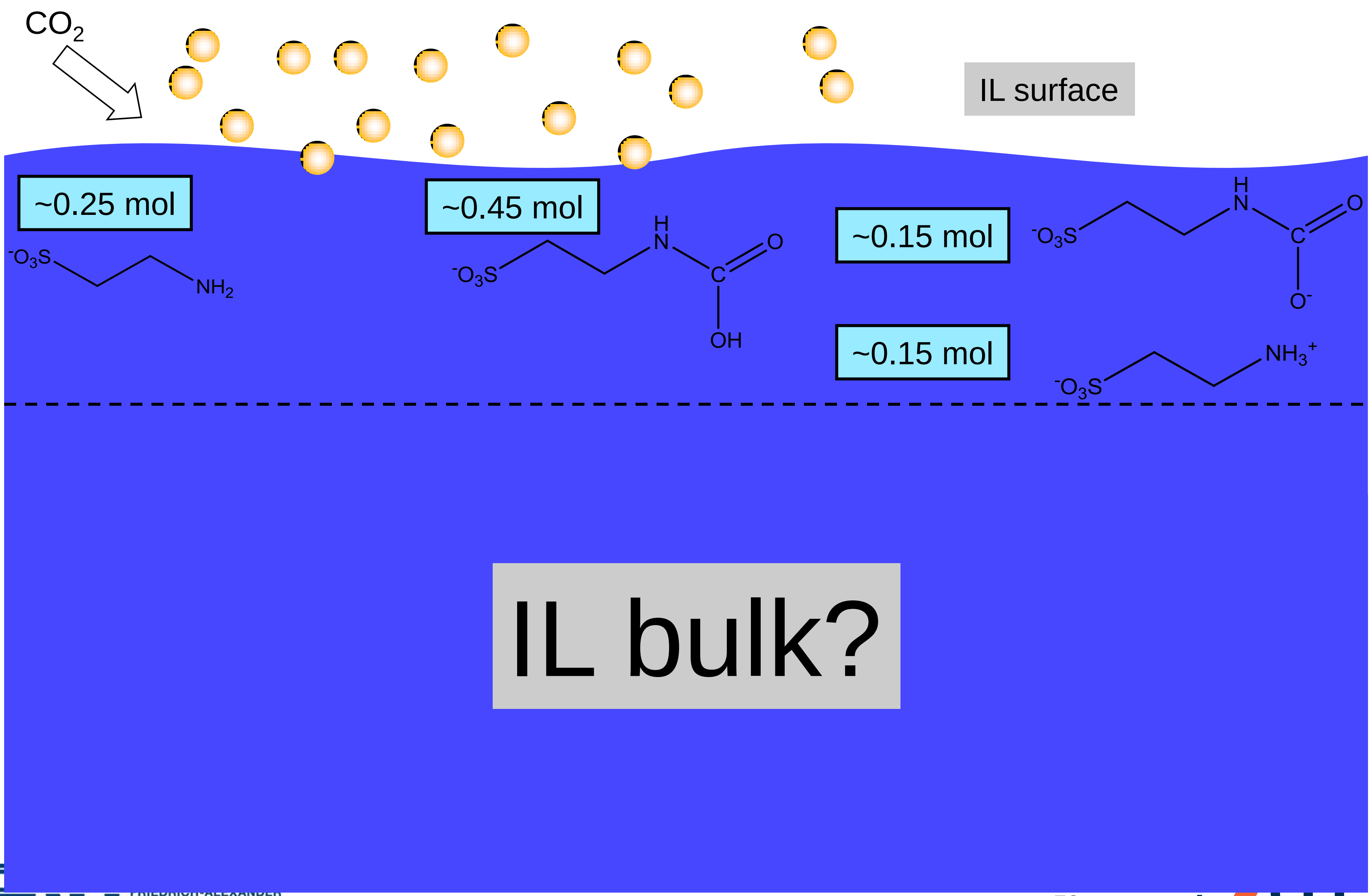


neat IL

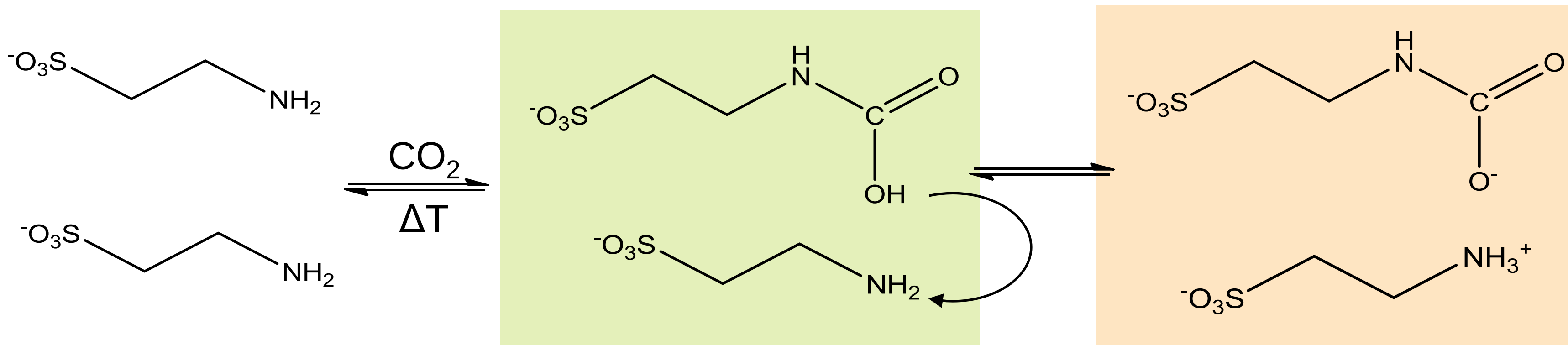
at 0.9 mbar CO_2



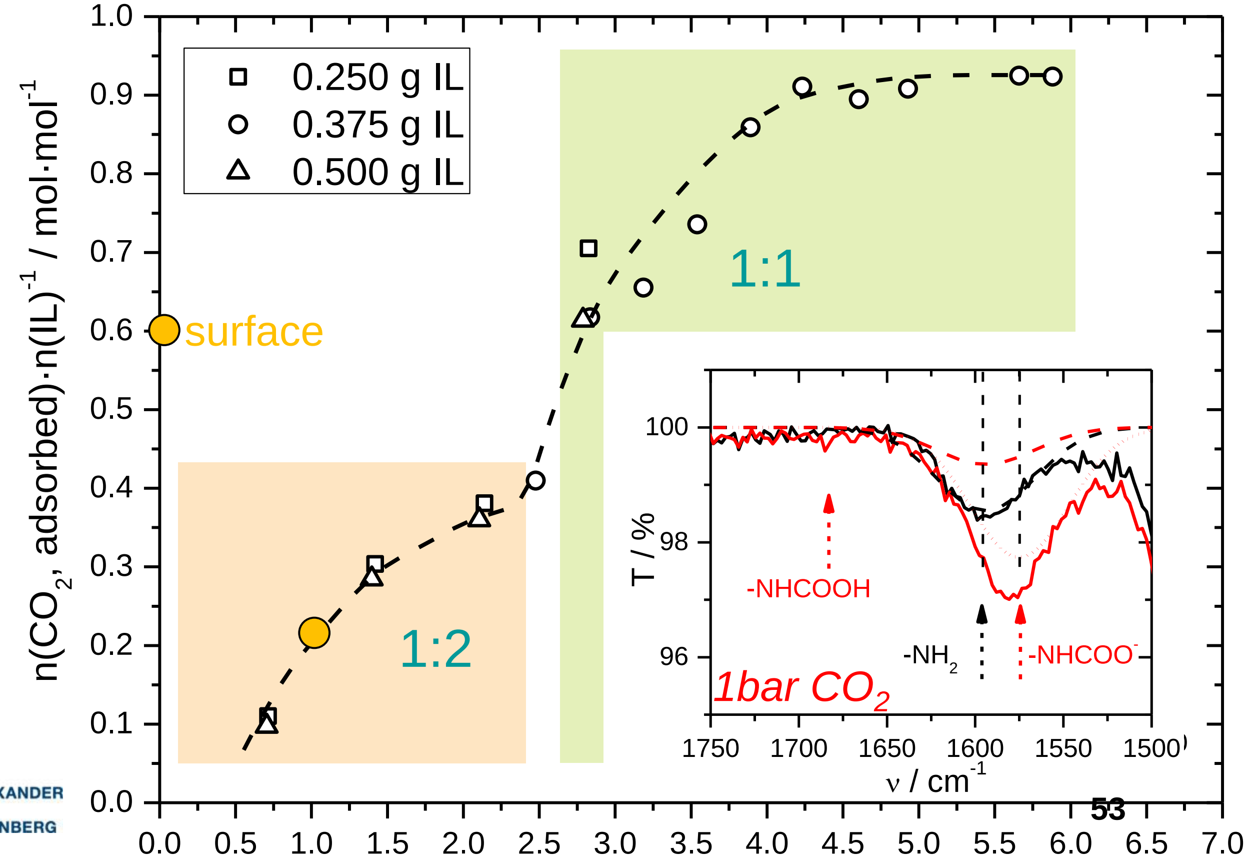
IL surface composition after CO_2 uptake



CO_2 equilibrium adsorption in bulk (ex situ)



IL bulk uptake (310 K)



Matthias Bahlmann

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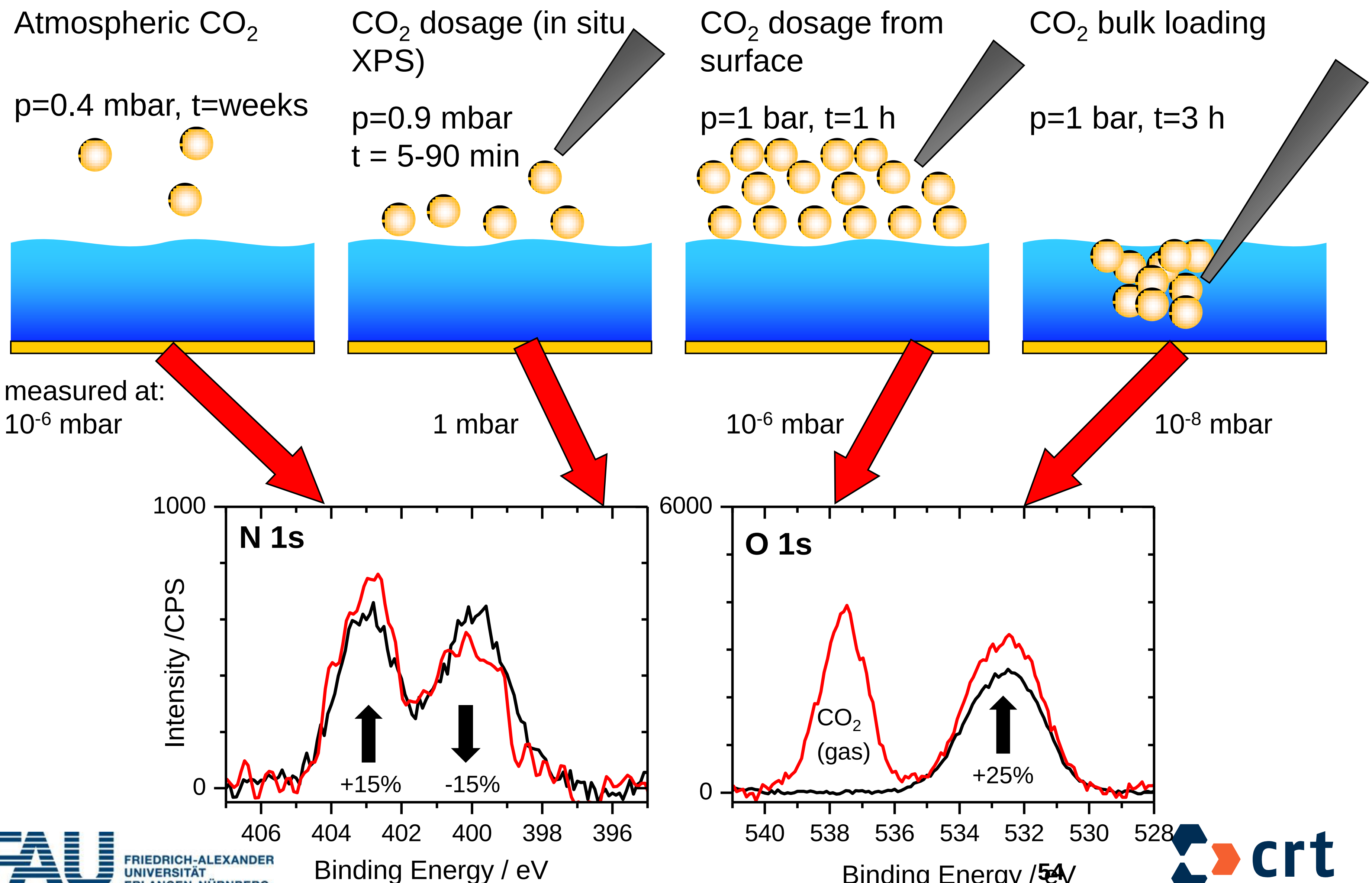

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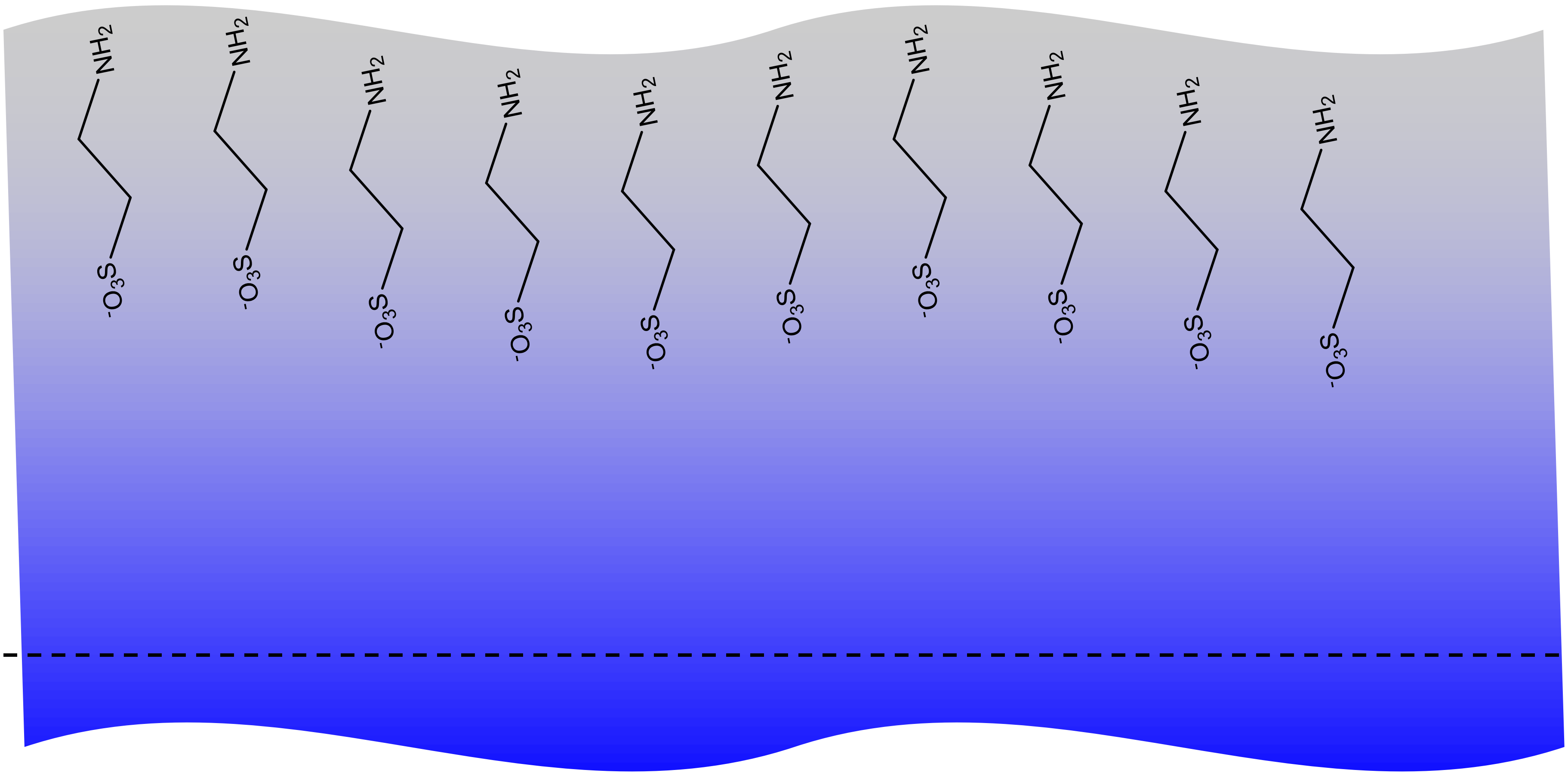
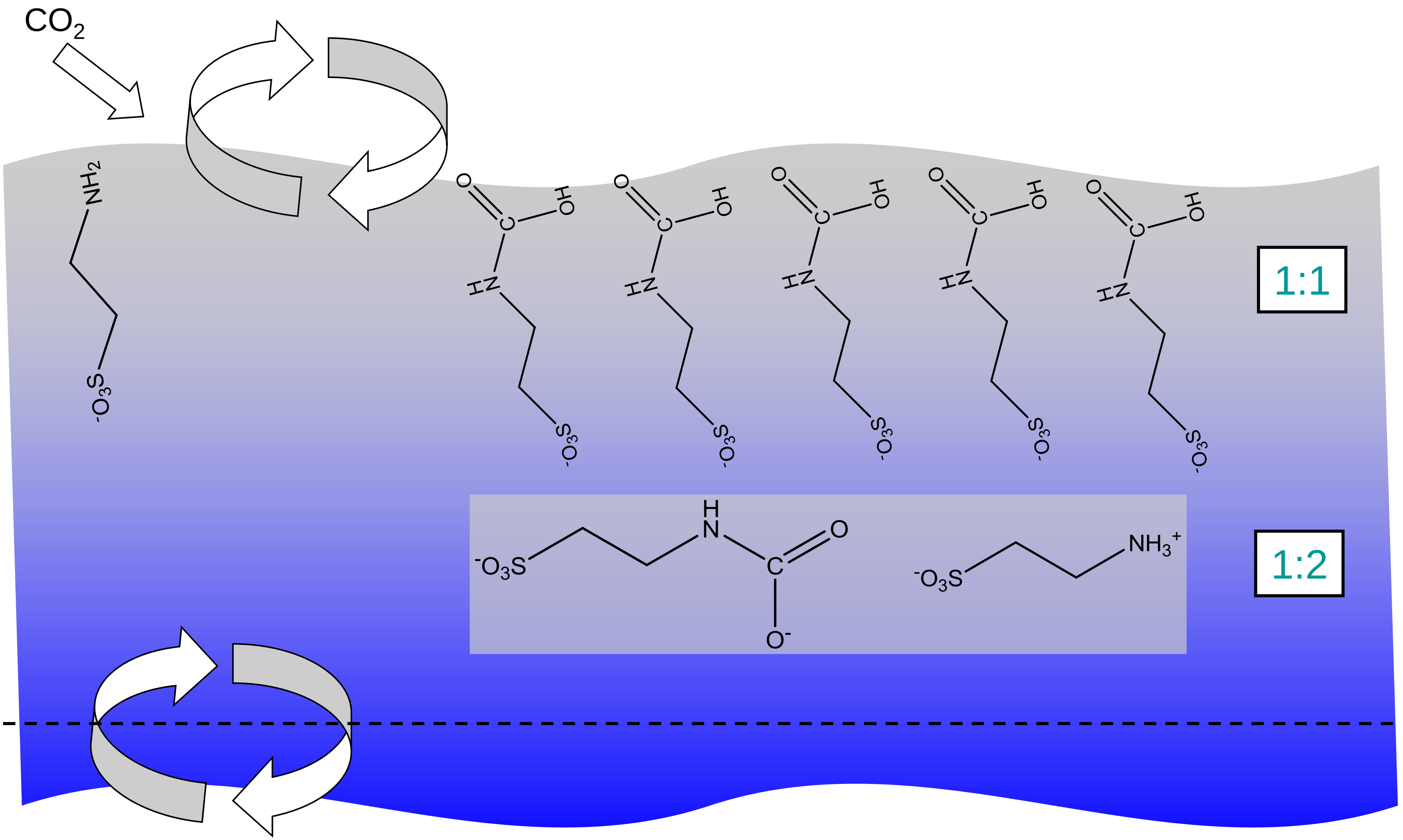
Charge distribution as possible driving force



Niedermaier et al., J. Am. Chem. Soc., 2014, 136, 436

Charge distribution as possible driving force



Niedermaier et al., J. Am. Chem. Soc., 2014, 136, 436

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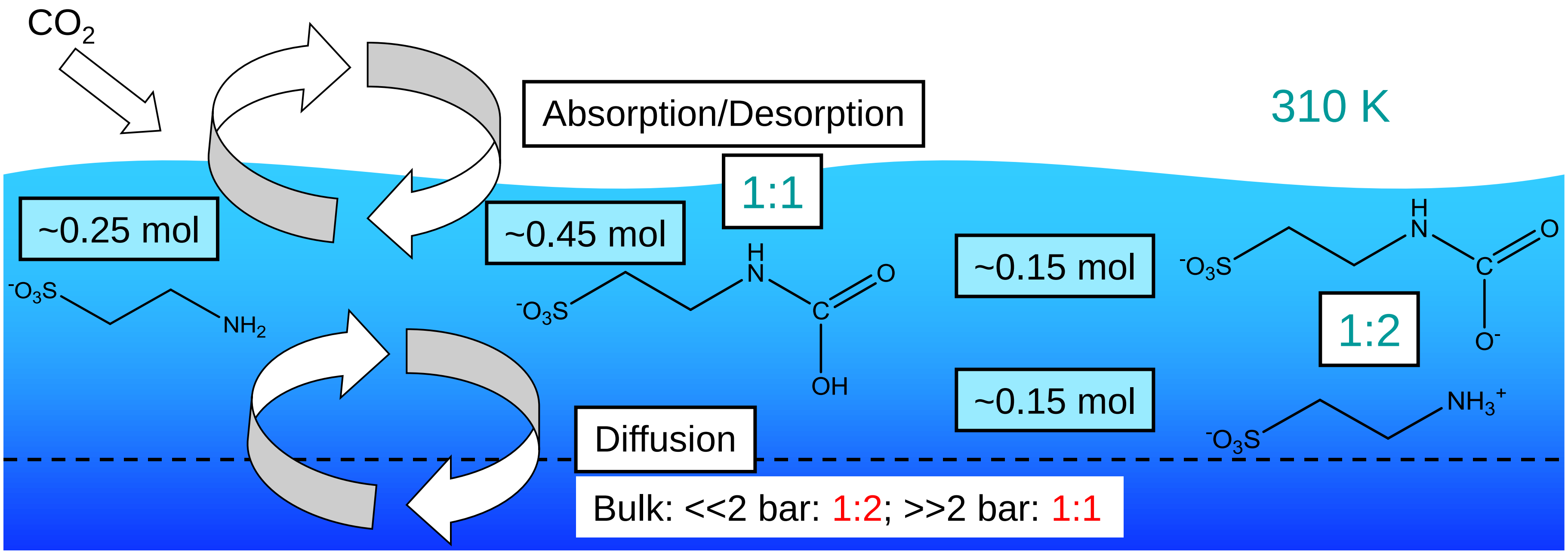
Summary



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Niedermaier et al., J. Am. Chem. Soc., 2014, 136, 436