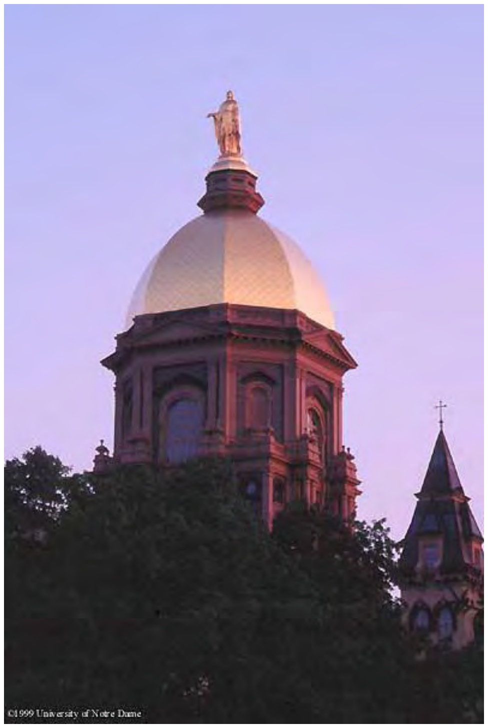
Energy Applications of Ionic Liquids



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cSEND
CENTER FOR SUSTAINABLE ENERGY AT NOTRE DAME

Summer School on Green Chemistry and Sustainable Energy
July 24, 2013

Outline

- **What are ILs?**
  - **Properties**
  - **Are they green?**
- **Applications**
  - **CO_2 capture**
  - **Biomass processing**
  - **ILs as electrolytes**
  - **Absorption refrigeration**

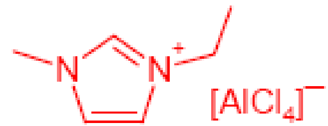
What are Ionic Liquids?

- Organic salts that are liquid at temperatures around ambient
- Liquid over a wide range of temperatures; hence, can be used as solvents
- Demonstrated successes as reaction solvents (olefin dimerization, metathesis, isomerizations, Diels-Alder, Friedel-Crafts alkylations and acylations, hydrogenations, C-C coupling)
- Ionic liquids have vanishingly low vapor pressures
  - fugitive emissions not a problem
  - worker exposure less likely
  - flammability danger decreased

Ionic Liquids-Evolution

- 1980s: Chloroaluminate Ionic Liquids

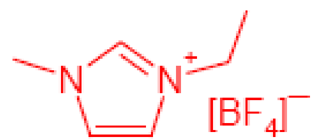
  1st generation



J.S. Wilkes, J.A. Levisky, R.A. Wilson and C.L. Hussey, *Inorg. Chem. 21* (**1982**) 1263-1264.

- 1990s: Air- and moisture-stable Ionic Liquids

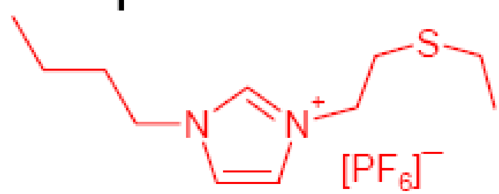
  2nd generation



J.S. Wilkes and M.J. Zaworotko, *J. Chem. Soc. Chem. Commun* (**1992**) 965-966.

- 2000s: First examples of „Task Specific Ionic Liquids"

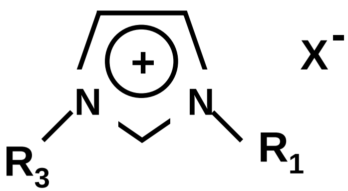
  3rd generation



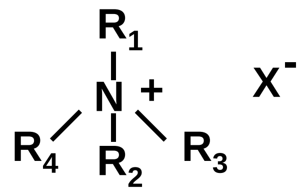
A.E. Visser, R.P. Swatloski, W.M. Reichert, R. Mayton, S. Sheff, A. Wierzbicki, J.H. Davis, Jr. and R.D. Rogers, *Chem. Commun.* (**2001**) 135-136.

W. Pitner – Merck KGaA

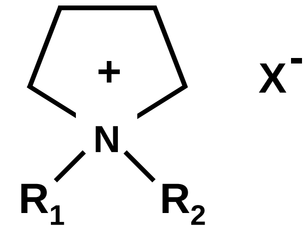
Typical Ionic Liquids



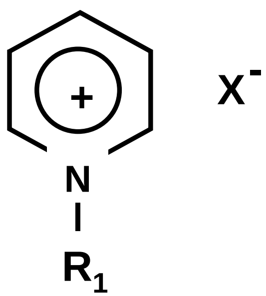
imidazolium



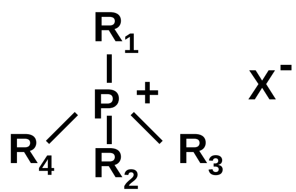
tetra alkylammonium



pyrrolidinium



pyridinium

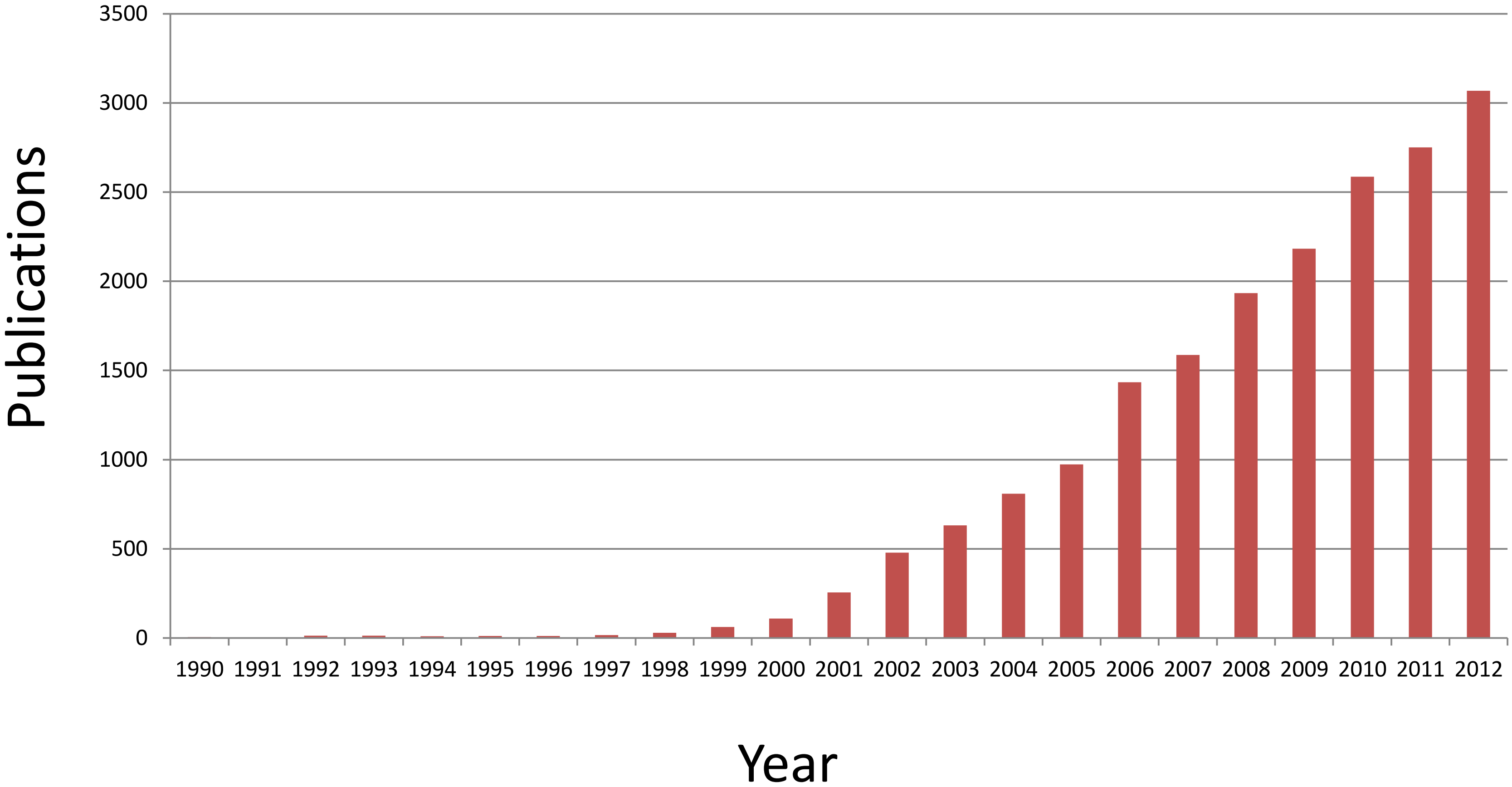


tetra alkylphosphonium

X = Cl, NO_3, CH_3CO_2, CF_3CO_2, BF_4, CF_3SO_3, PF_6, $(CF_3SO_2)_2N$

1-hexyl-3-methylimidazolium $(CF_3SO_2)_2N$ = [hmim][Tf_2N]

Growth in Publications



Properties – Liquid Range

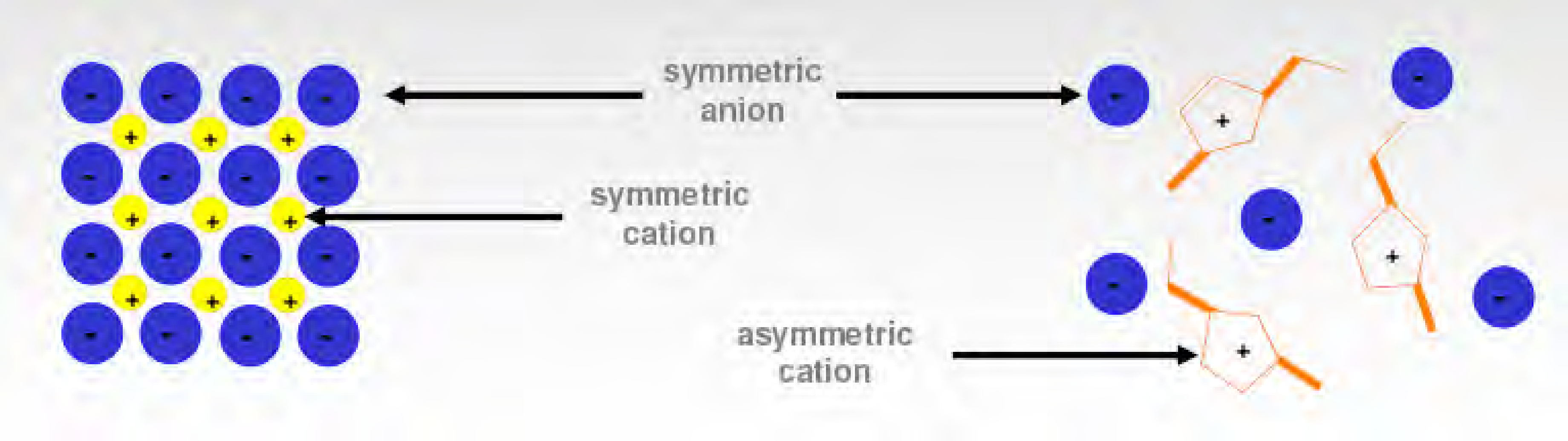
- Liquid over a wide range of temperatures (e.g. -70 ºC – 400 ºC)

Compound	T_m or T_g (ºC)	T_{decomp} (ºC)
[emim][Tf_2N]	-17	
[emmim][Tf_2N]	25	
[pmmim][Tf_2N]	-82	462
[bmim][[Cl]	-69	264
[bmim][[Br]	-50	273
[bmim][[BF_4]	-85	361
[bmim][[PF_6]	11	
[bmim][Tf_2N]	-2	422
[bmim][triflate]	13	392
[bmim][methide]	-65	413
[bmim][dca]	-6	300
[bmmim][[BF_4]	37	380
[bmmim][[PF_6]	-58	373

Fredlake et al., J. Chem. Eng. Data, 49, 2004, 954-964

Lowering Melting Points

- Asymmetry
- Branching
- Poorly coordinating anions



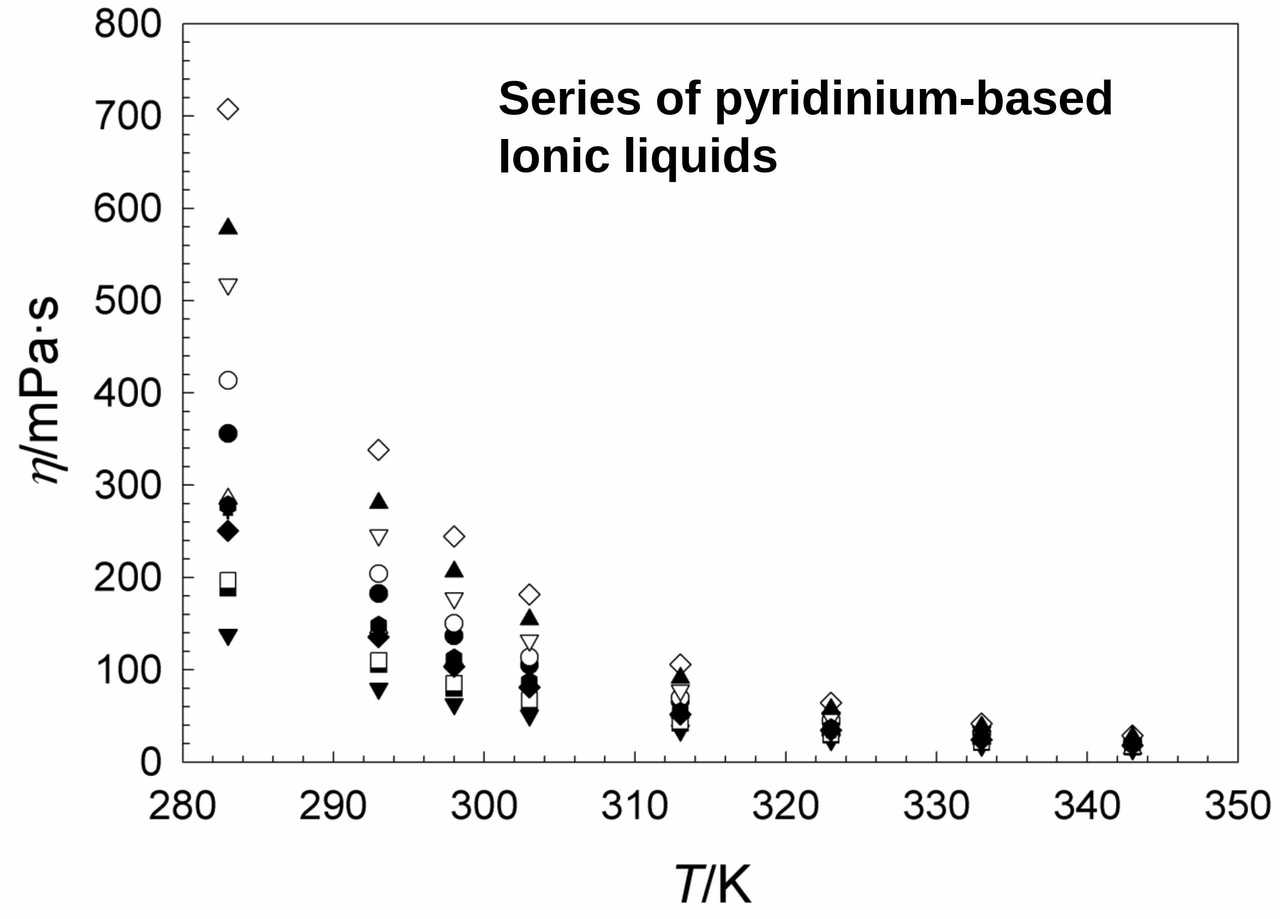
T. Shubert - Iolitec

Viscosities

Ionic Liquid	Viscosity / mPa s
Diethyl ether	0.22
Water	1.00
Ethylene glycol	16
1-ethyl-3-methylimidazolium dicyanamide	21
1-ethyl-3-methylimidazolium tetracyanoborate	22
1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide	27
1-ethyl-3-methylimidazolium thiocyanate	38
1-ethyl-3-methylimidazolium tetrafluoroborate	38
1,2-propylene glycol	40
1-ethyl-3-methylimidazolium trifluoromethylsulfonate	43
1-ethyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate	44
1-ethyl-3-methylimidazolium methylsulfate	93
1-butyl-3-methylimidazolium tetrafluoroborate	154
1-hexyl-3-methylimidazolium tetrafluoroborate	224
4-methyl-*N*-butylpyridinium tetrafluoroborate	291
1-butyl-3-methylimidazolium hexafluorophosphate	371
1-octyl-3-methylimidazolium tetrafluoroborate	468
1-butyl-2,3-dimethylimidazolium tetrafluoroborate	932
Glycerol	934

W. Pitner – Merck KGaA

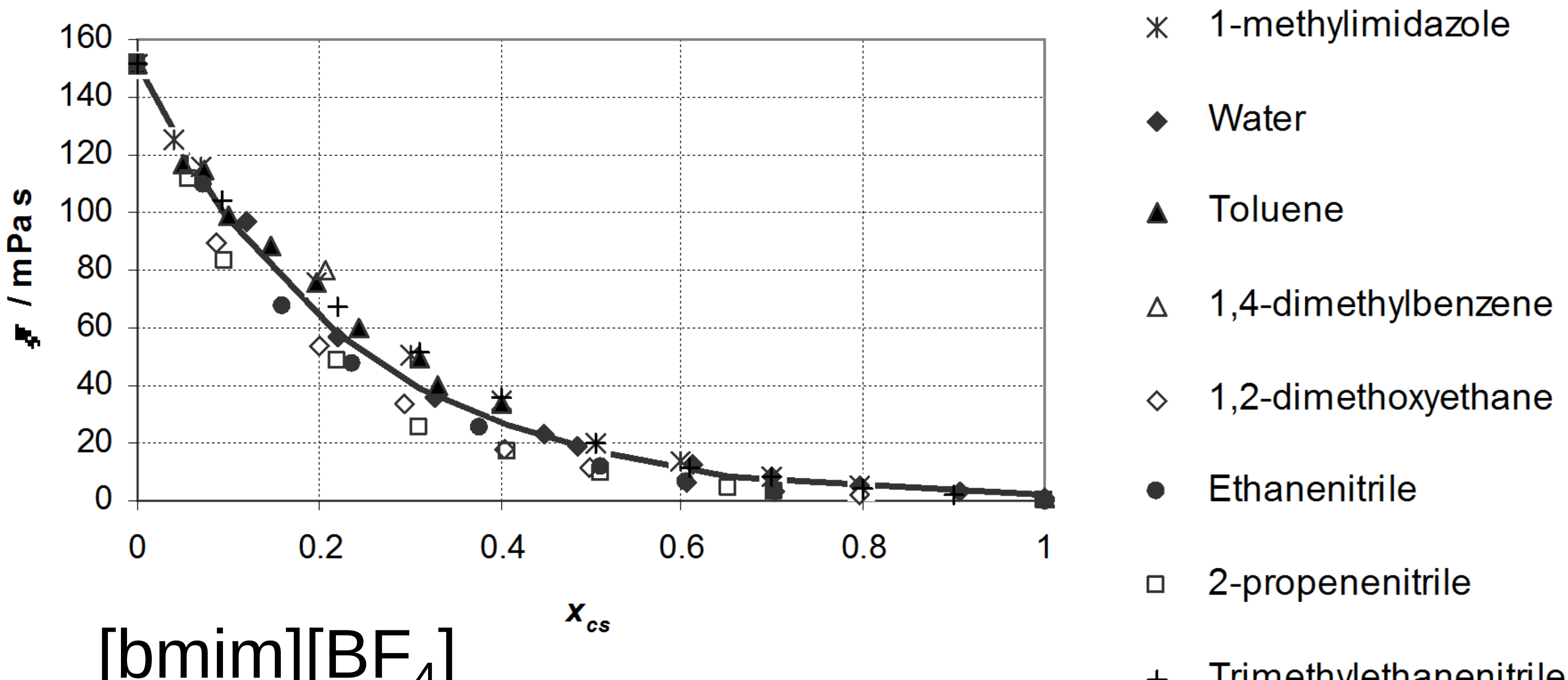
Viscosities



Crosthwaite et al., J. Chem. Thermodynamics, 37, 2005, 559-568

Properties Sensitive to Impurities!

- Presence of water, organics, etc. reduces viscosity, density, etc.

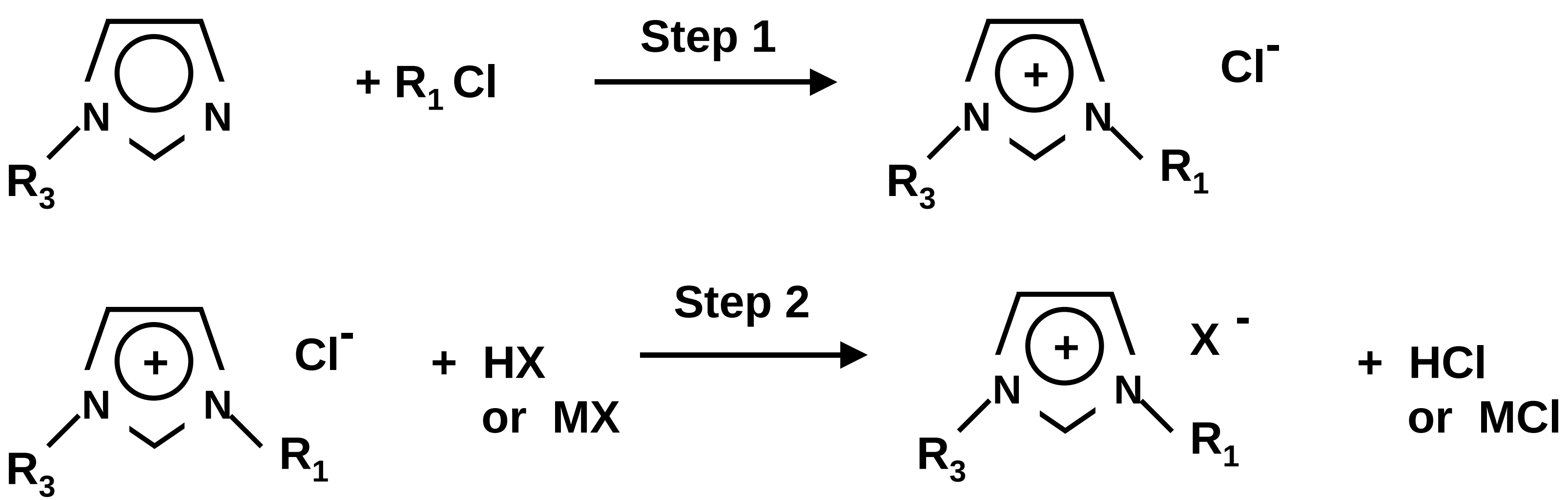


[bmim][BF_4]

Seddon et al., Pure Appl. Chem, 2000

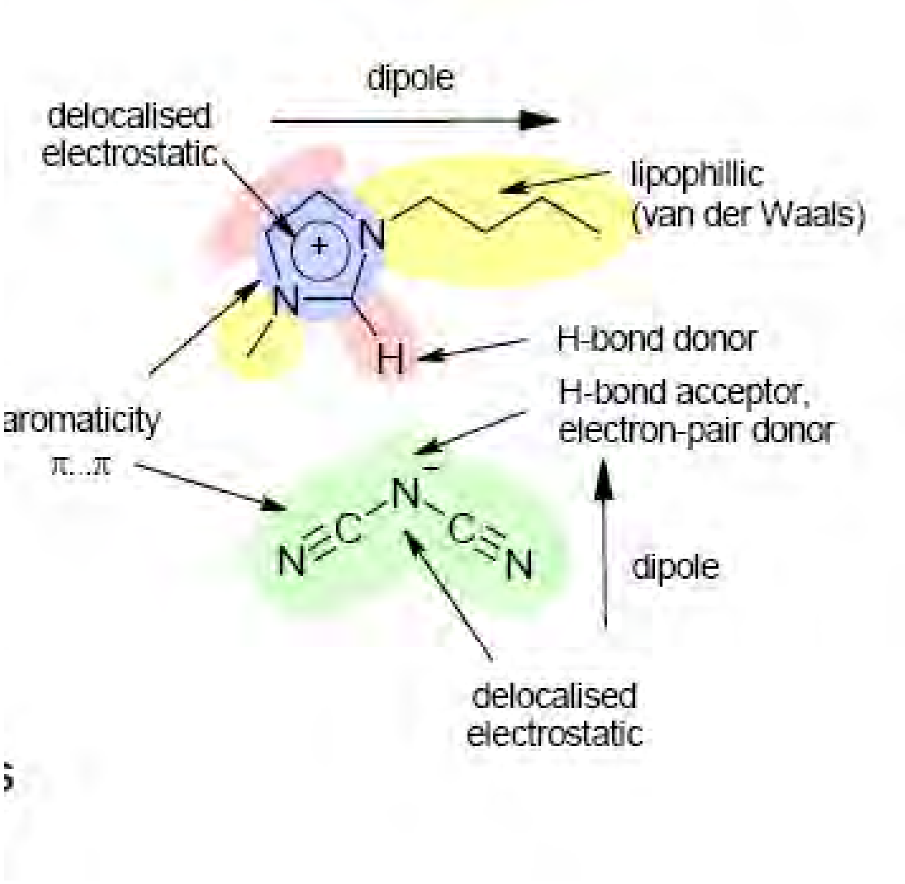
What Impurities?

- Water – all ILs very hygroscopic
- Added solutes (reactants, products, catalysts)
- By-products from making ILs (e.g., halides)
- Decomposition products from IL synthesis
- Hydrolysis products (e.g., HF) – don't use PF_6 or BF_4 ILs!!!
- $EtSO_4$ hydrolyzes too



Solvation Properties

- Ability to dissolve polar, nonpolar and aromatic compounds
- ‘Tunable’ solubility



W. Pitner – Merck KGaA

Are ILs Green?

- Nonvolatile, will not cause air pollution
- Dissolve in water; what is toxicity to aquatic organisms?
- Biodegradability?
- Bioaccumulation? Generally, NO; very small K_{OW}
- Non-mutagenic

Toxicity

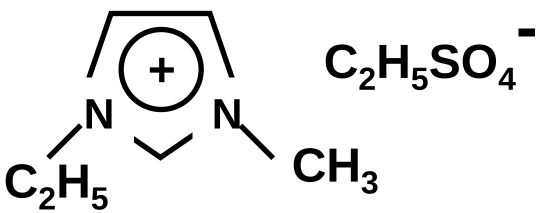
- Can make extremely toxic or NOT
- Can't classify ILs as a group
- [emim][$EtSO_4$] approved in Europe under REACH
  – Hydrolyzes to [emim][HSO_4] + ethanol

	BMIM Cl[1)]	EMIM $EtOSO_3$[2)]	MTEOA $MeOSO_3$[3)]
Acute oral toxicity	toxic	not harmful	not harmful
Skin irritation	irritant	non-irritant	non-irritant
Eye irritation	irritant	non-irritant	non-irritant
Sensitization	non-sensitizing	non-sensitizing	non-sensitizing
Mutagenicity	non-mutagenic	non-mutagenic	non-mutagenic
Biological degradability	not readily degradable	not readily degradable	readily biodegradable
Toxicity to daphniae	acute toxic	acutely not harmful	acutely not harmful
Toxicity to fish	acutely not harmful	-	acutely not harmful

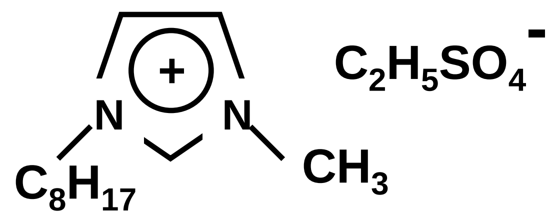
[1)] BMIM Cl = 1-Butyl-3-methylimidazolium chloride
[2)] EMIM EtOSO3 = 1-Ethyl-3-methylimidazolium ethylsulfate
[3)] MTEOA MeOSO3 = Tris-(2-hydroxyethyl)-methylammonium methylsulfate

M. Maase - BASF

Toxicity and Biodegradability



-Low toxicity to aquatic organisms
-Cation NOT biodegradable



-High toxicity to aquatic organisms
-Cation NOT biodegradable

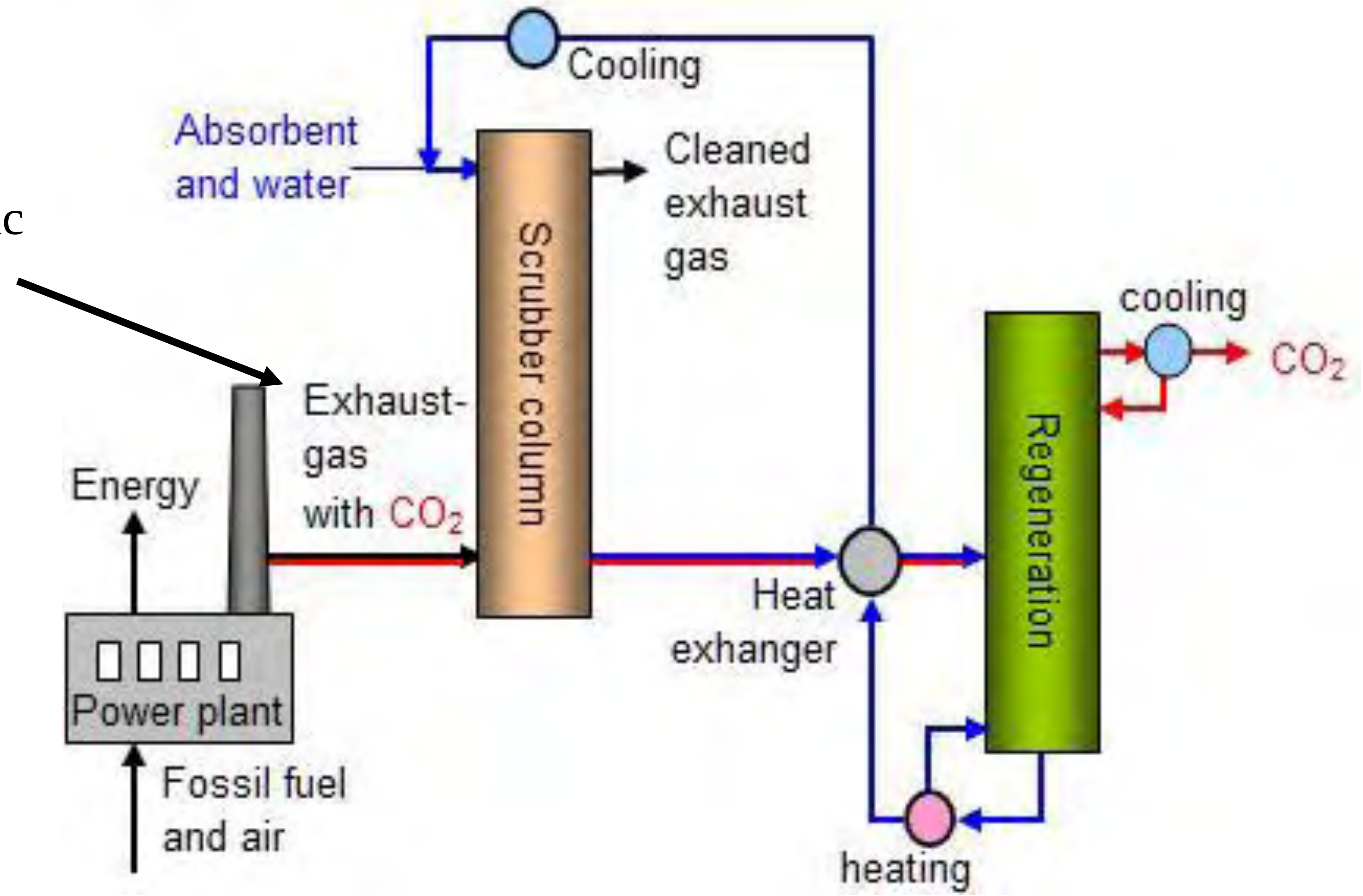
Quaternary ammonium and pyridinium cations can be biodegradable; phosphonium and imidazoliums generally NOT

CO_2 Capture - McKinsey Conclusions

- Lots of opportunities for GHG reductions from efficiency improvements
- Energy efficiency SAVES $$$, even considering the capital investment
- Can't meet target (reduce current CO_2 emissions by ½) without Carbon Capture and Sequestration (CCS)

Post-Combustion Capture Conventional PC Power Plant

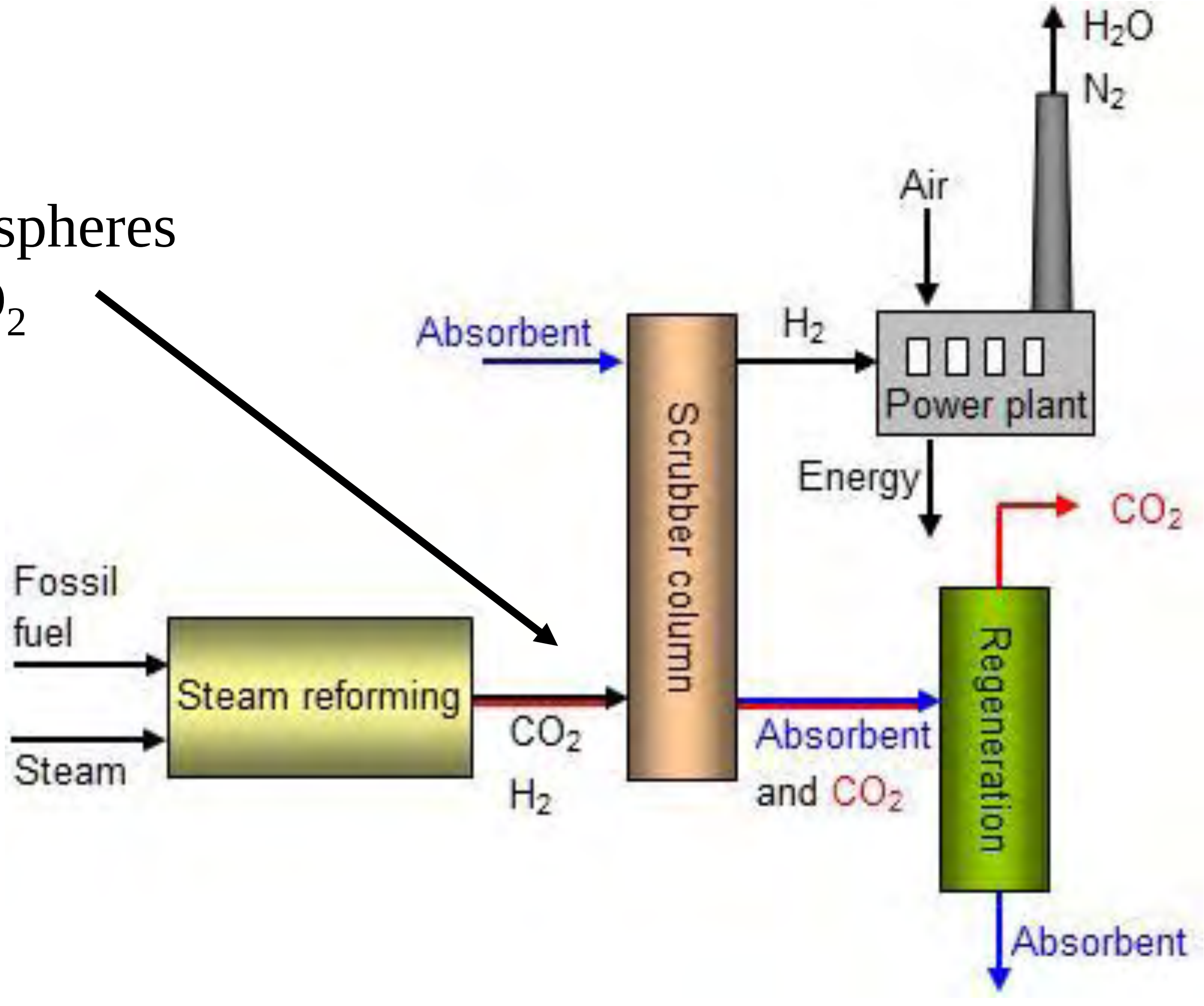
Atmospheric pressure
~12% CO_2



http://www.bellona.org/factsheets/1191913555.13

Pre-Combustion Capture - IGCC

>20 atmospheres
>30% CO_2



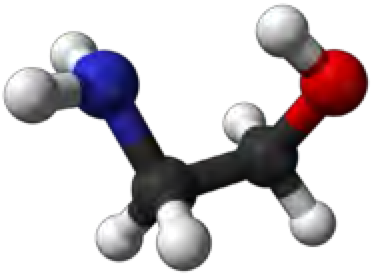
http://www.bellona.org/factsheets/1191913555.13

US Dept. of Energy CCS Targets

- Separation and Capture
  - >50% of electricity from conventional coal in U.S.
  - Need low energy and cost post-combustion capture technology
  - Target is ~35% increase in cost of electricity
- Sequestration/Storage
- Monitoring, Mitigation and Verification

Carbon Capture & Sequestration

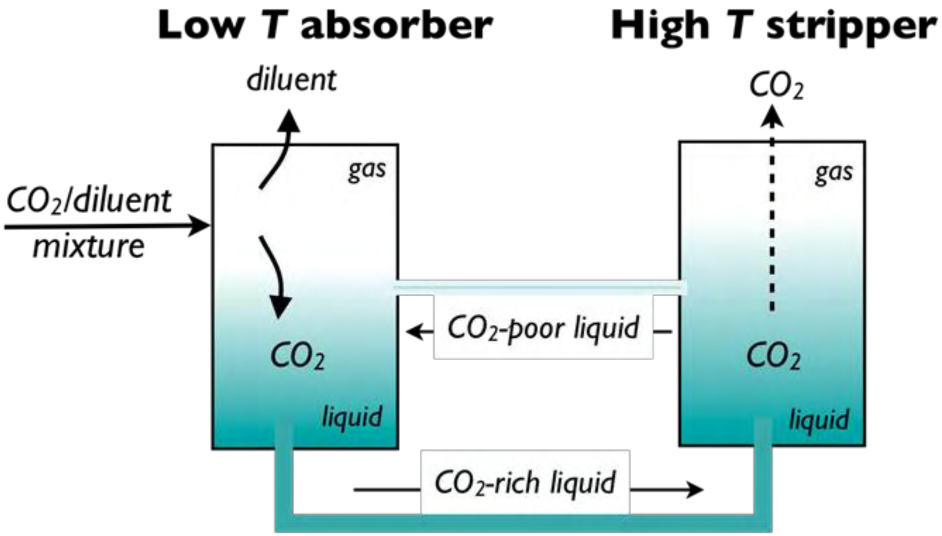
- Existing technology (dilute aqueous amines) impractical
  - Monoethanolamine (MEA)
  - High parasitic energy load (~28%; 80% increase in COE)
    - Large heat of reaction
    - Energy lost in evaporation of water
  - Corrosive, side reactions
  - Degrades at low temperatures
- Alternative – Ionic Liquids

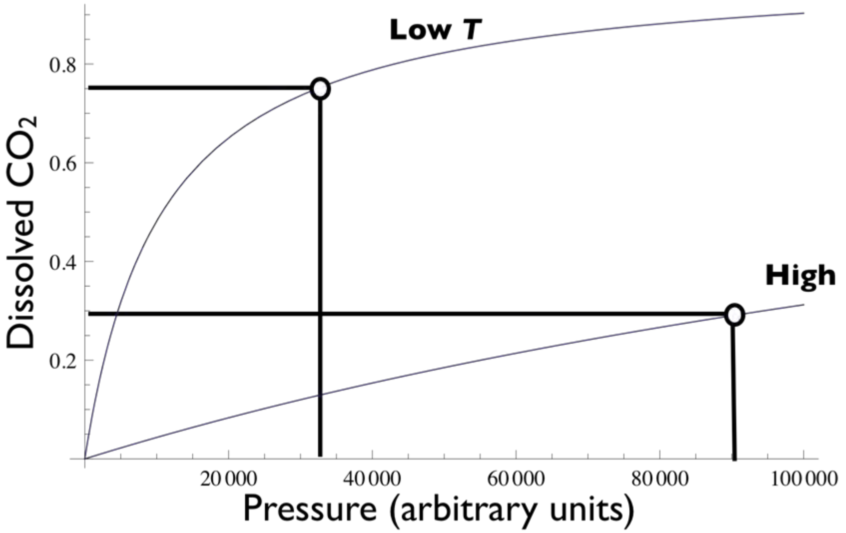


Key Properties for CO_2 Capture

- High CO_2 solubility
- High CO_2 selectivity
- Ease of regeneration
  - Low enthalpy of solution
  - Low solubility with water
  - Low heat capacity
- Stability
  - Thermal
  - Other gases (e.g., SO_2)
- Low viscosity
- Inexpensive

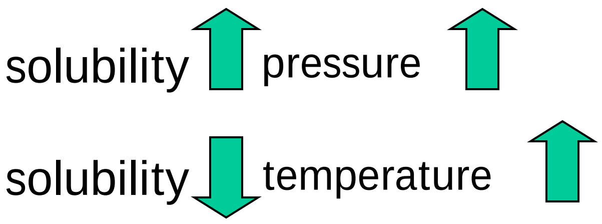
Process Configuration



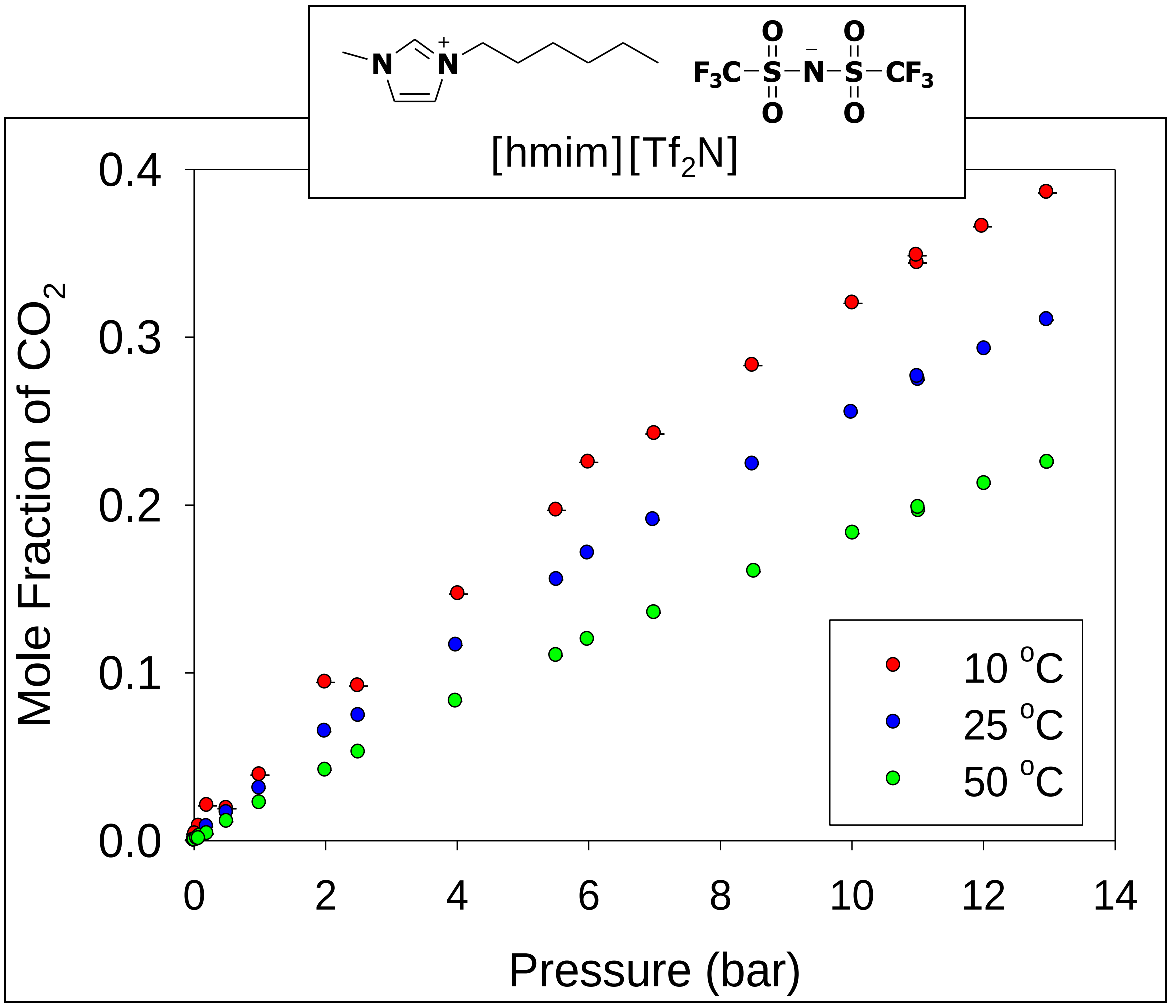


Pure Gas Solubility - CO_2

- Gas solubility



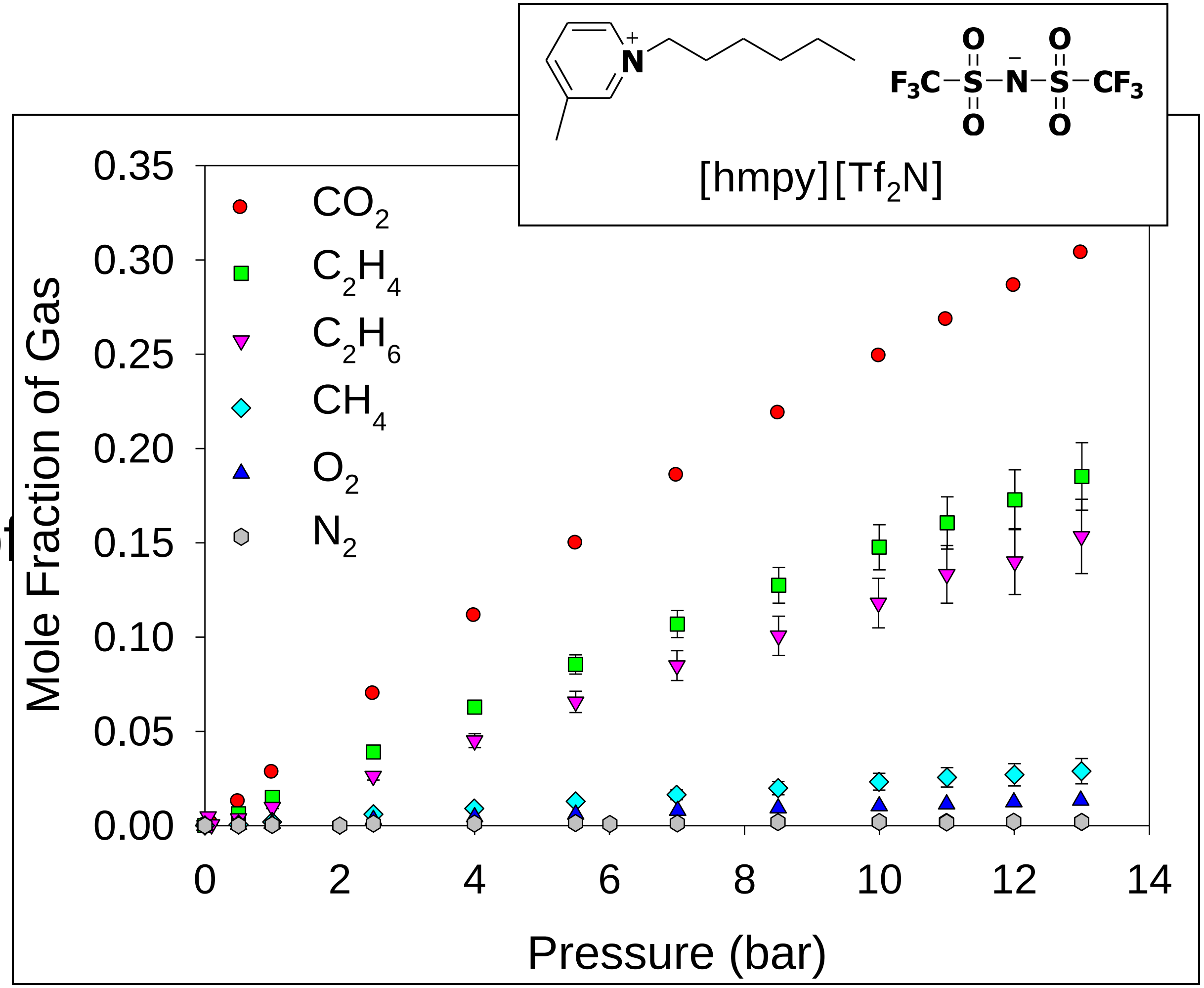
- Important for reusability of ILs
  - Absorb at low T
  - Remove at high T
- Trend seen for CO_2 solubility in all ILs measured



Muldoon, et al., JPC B, 2007, 111, 9001-9009

Pure Gas Solubility – Other gases

- Gas solubility measured in [hmpy][Tf$_2$N]
  - Similar trends are seen with other ILs
- CO_2 has the highest solubility of the gases measured
- Good selectivity!



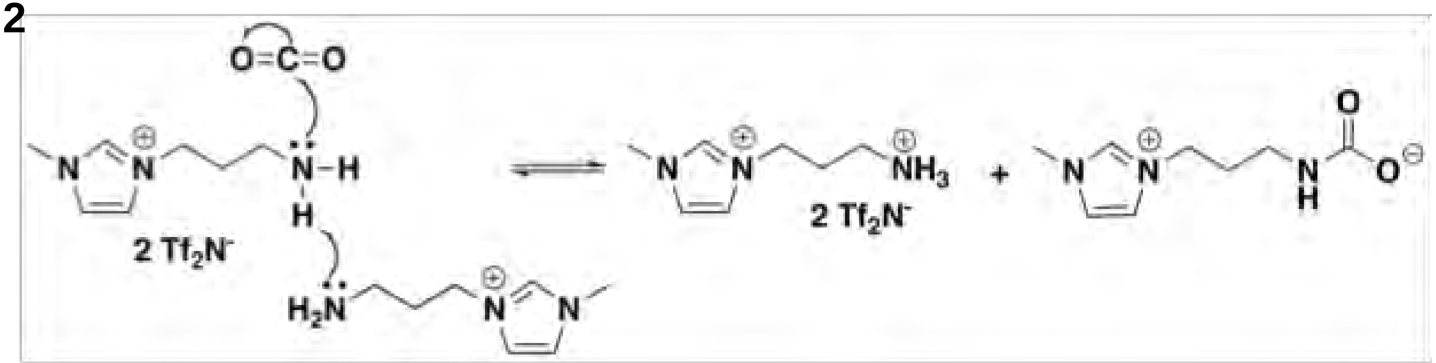
Anderson, et al., ACR, 2007, 40, 1208-1216

Limitations of Physical Absorption

- **Physical solubility**
  - **Low heat of absorption**
  - **~12 kJ/mol by T dependence of isotherms and direct calorimetric measurements**
  - **Low regeneration energy**
  - **Large IL circulation rates**
  - **Desorption at low P increases compression costs**
  - **Would need ~10x increase in solubility to beat aqueous MEA**
- **Chemical complexation**
  - **Strong enough to increase capacity and decrease IL circulation rates**
  - **Weak enough to keep regeneration energies (and temperatures) down**

Chemical Complexation with ILs

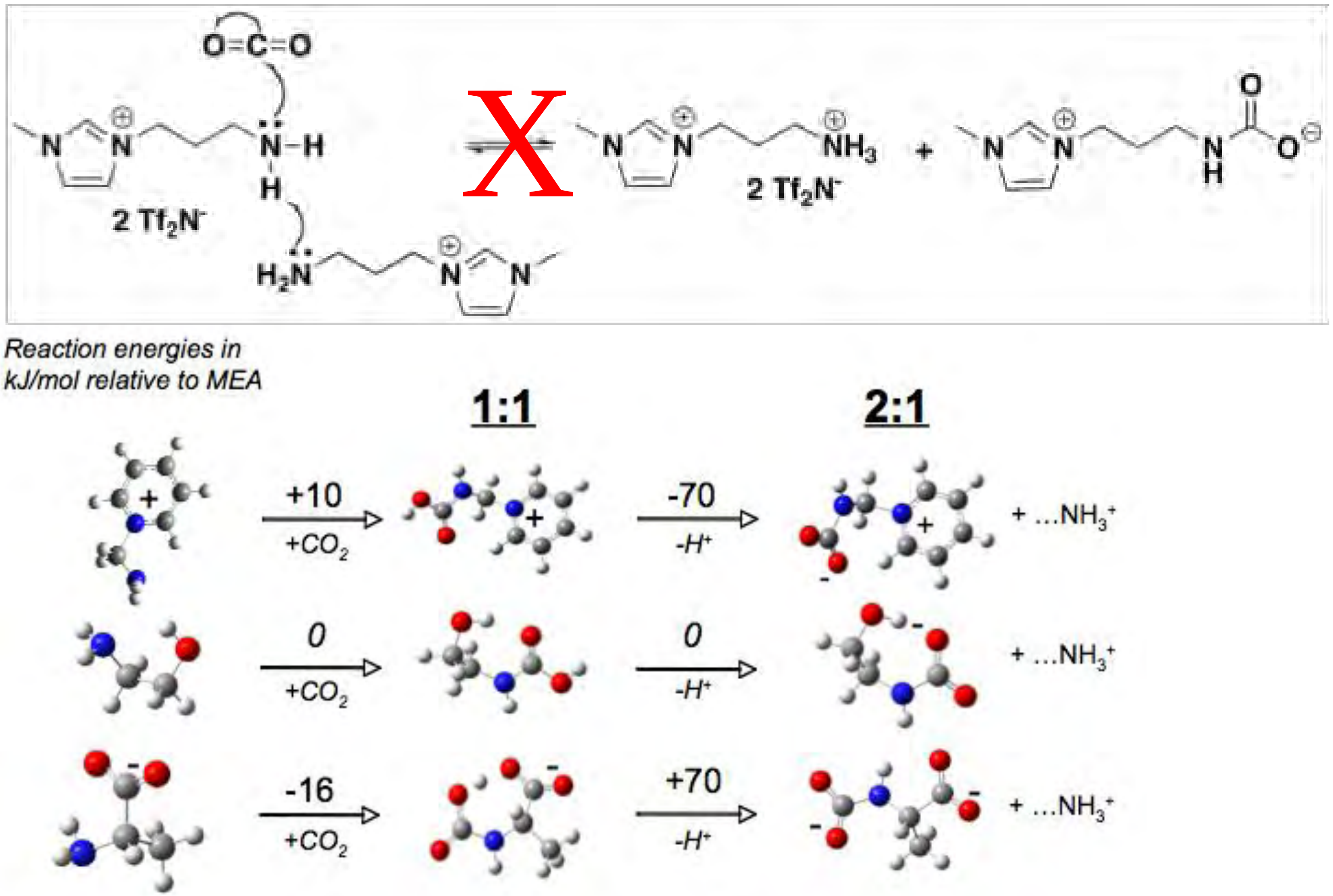
- Known chemistry – add amine to IL to react with CO_2



- "Task Specific Ionic Liquids"
- Much higher capacity for CO_2
- Same mechanism as amines
- CO_2 binds with two ILs; 1:2 mechanism

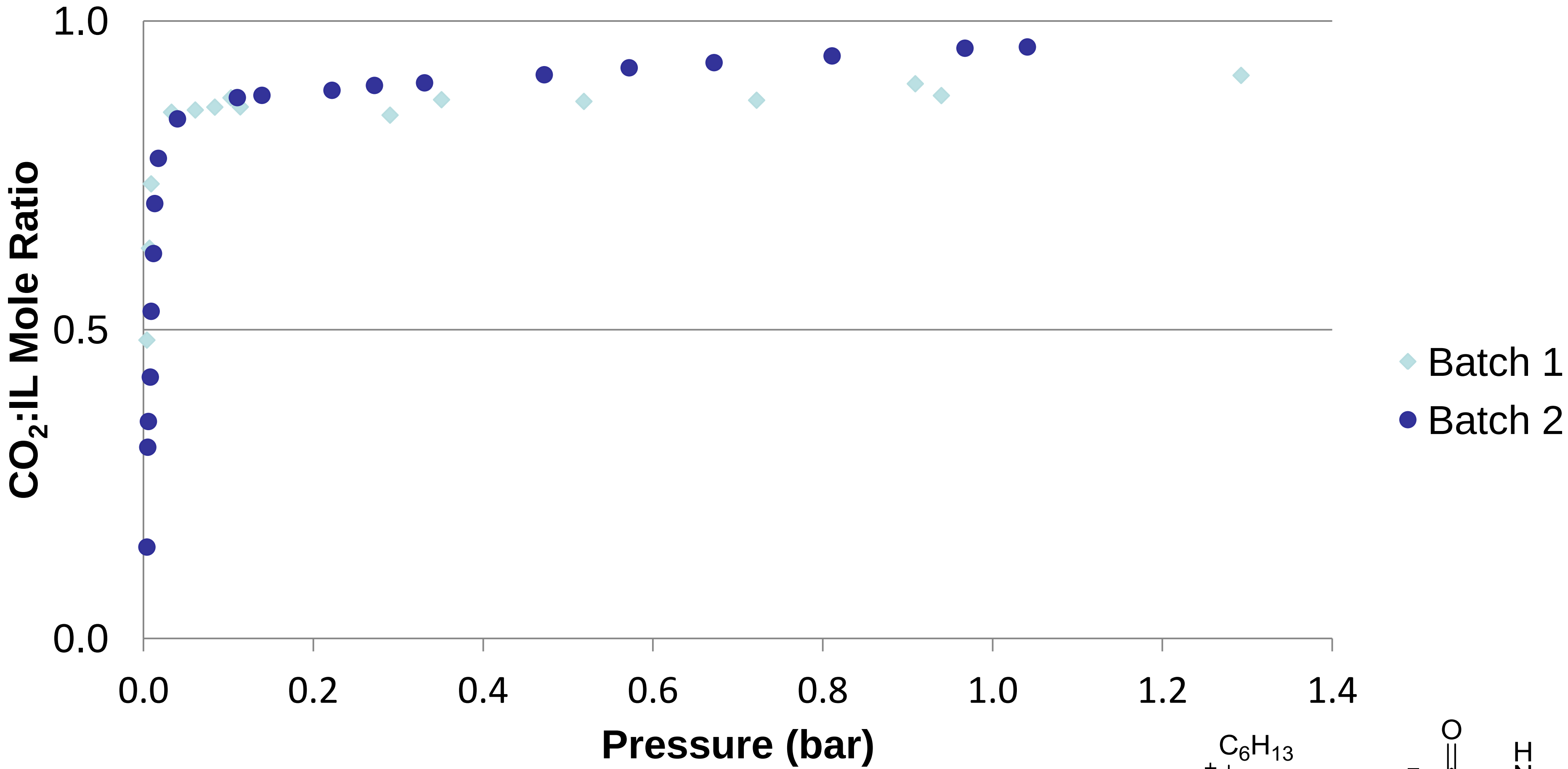
Bates, E. D.; Mayton, R. D.; Ntai, I.; Davis, J. H., *J. Am. Chem.*, **2002**, *124*, 926.

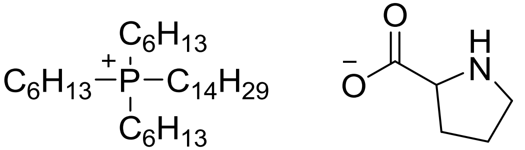
Designing ILs for 1:1 binding



Mindrup and Schneider, *ACS Symp. Series* **2010**

Confirmation of ILs with 1:1 binding

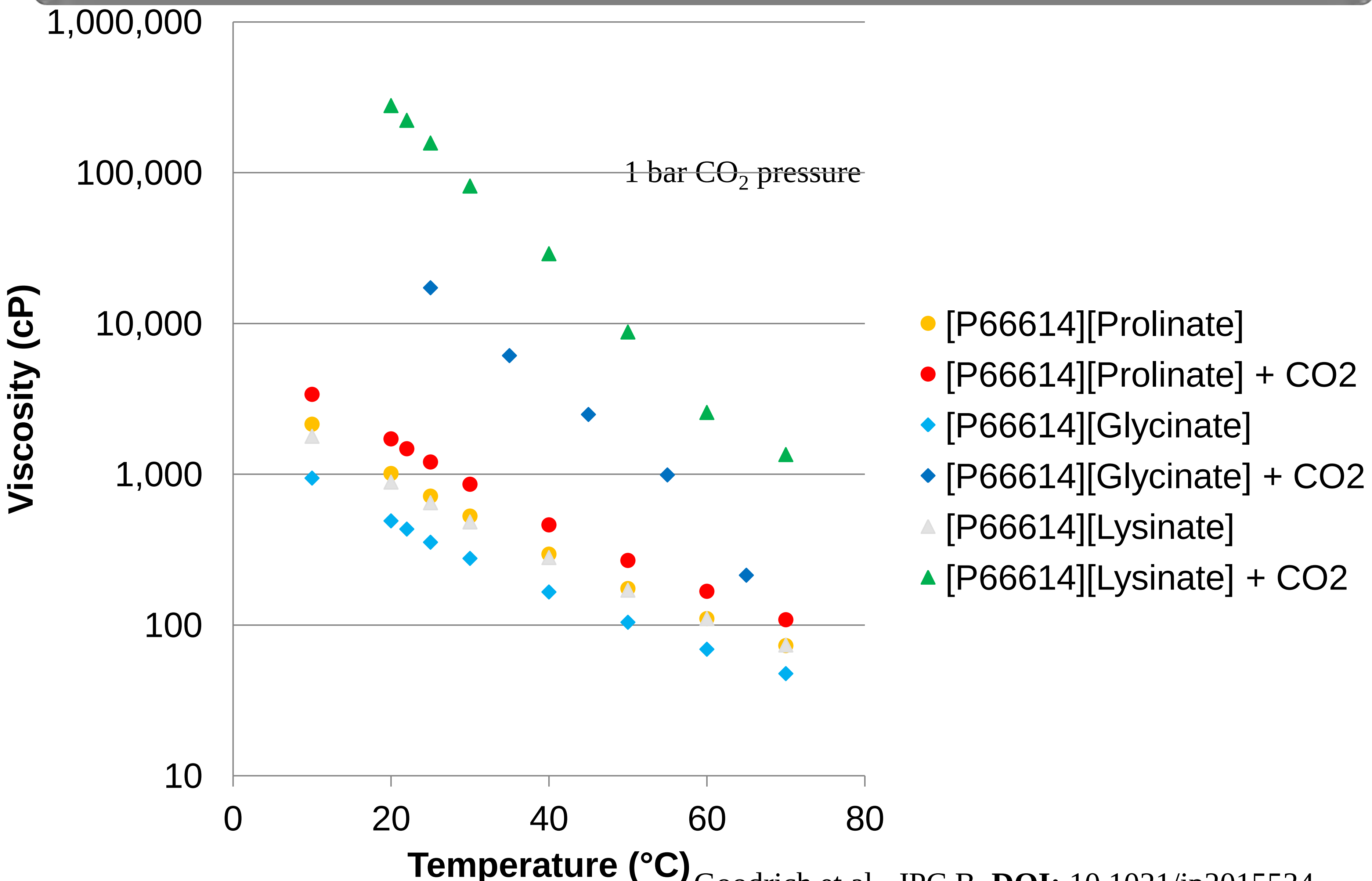




- Close to 1 mole CO_2/mole IL absorbed
- Curves level out at the same point between different batches
- Increased noise at higher pressures

Goodrich et al., JPC B, **DOI:** 10.1021/jp2015534

Effect of CO_2 on Viscosities



Goodrich et al., JPC B, **DOI:** 10.1021/jp2015534

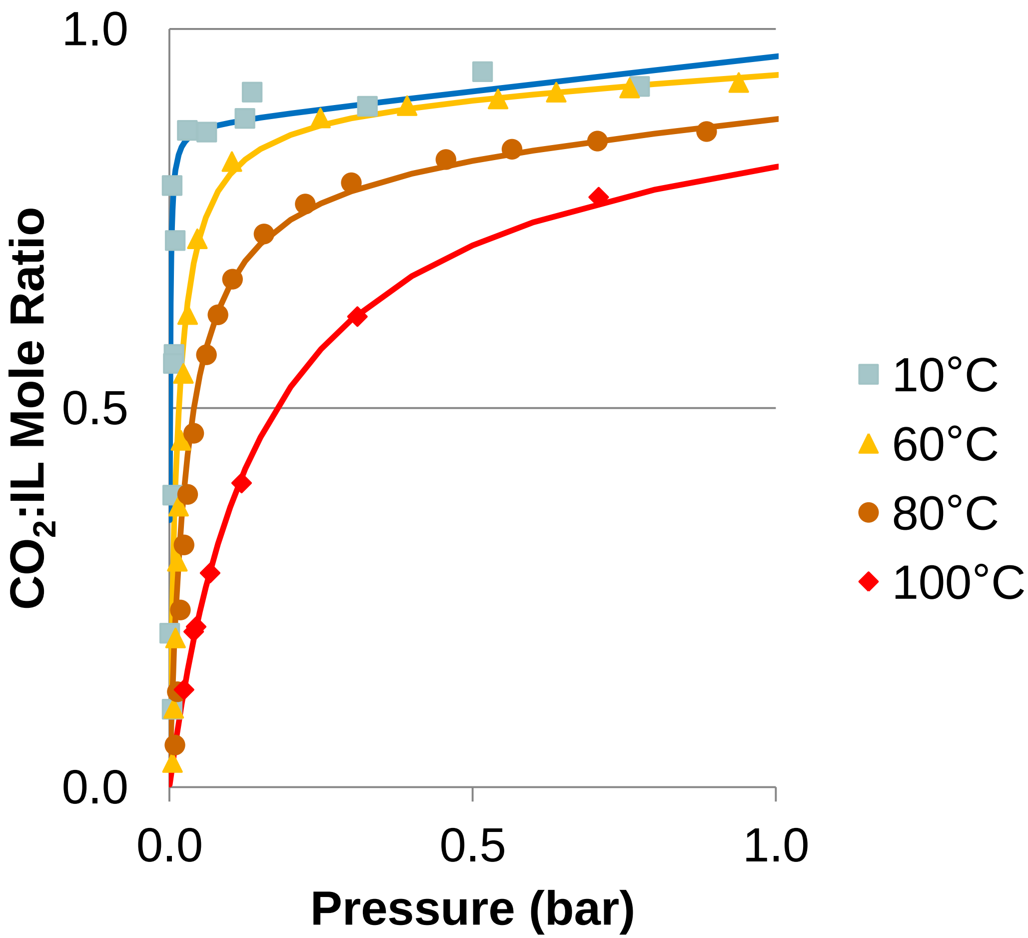
Modeling of CO_2 Uptake

$$CO_{2(g)} \rightleftharpoons CO_{2(phys)}$$

$$CO_{2(phys)} + IL \rightleftharpoons IL\text{-}CO_2$$

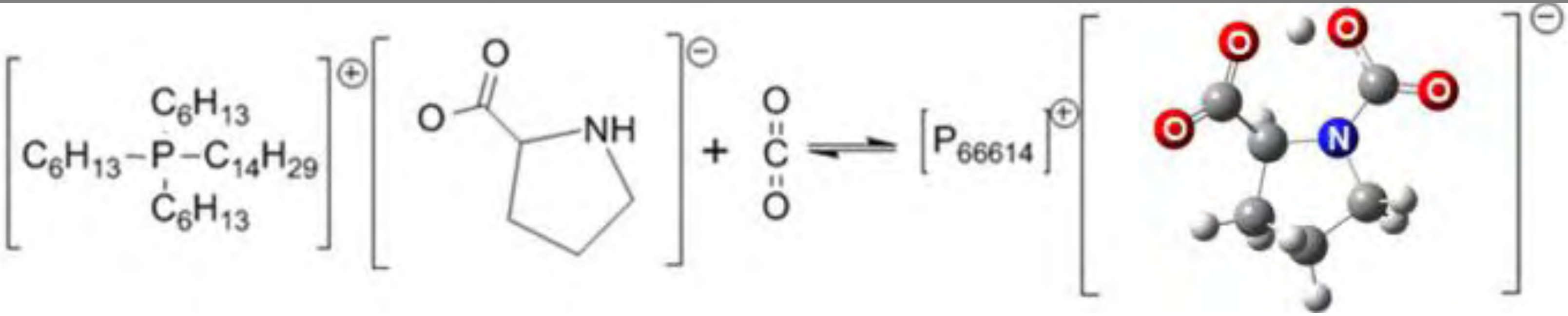
$$z = \frac{P}{h} + \frac{K*C*P}{1+K*P}$$

- <u>Variables</u>
  - z = Mole Ratio,
    moles CO_2 / moles IL
  - P = Pressure
- <u>Parameters</u>
  - Henry's Law describes Physical Absorption
  - K = Equilibrium Constant for Chemical Absorption
  - C = activation sites / moles IL

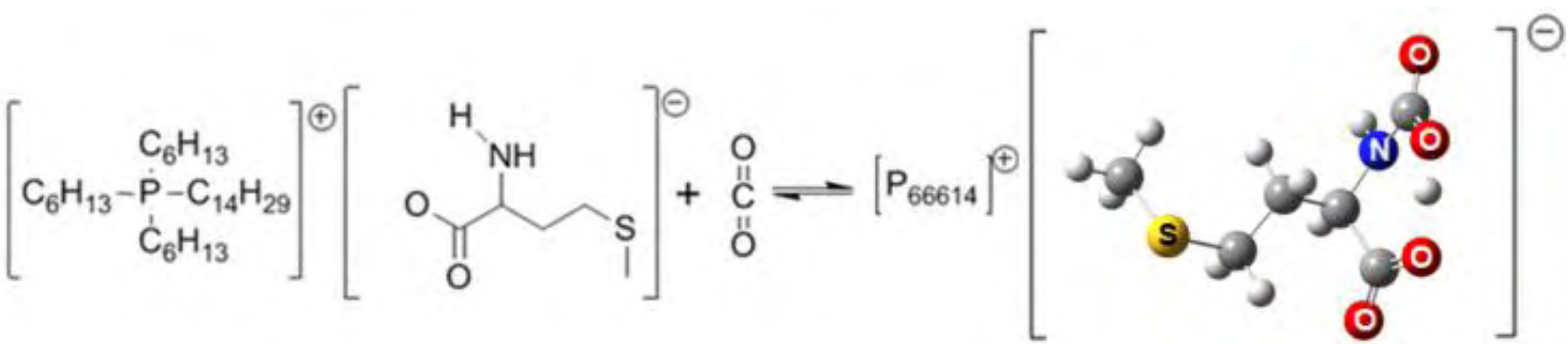


Goodrich et al., JPC B, **DOI:** 10.1021/jp2015534

Heat of Absorption



Ionic Liquid	ΔH, kJ/mol_{CO_2}
$[P_{66614}]$[Prolinate]	-80
$[P_{66614}]$[Methioninate]	-64

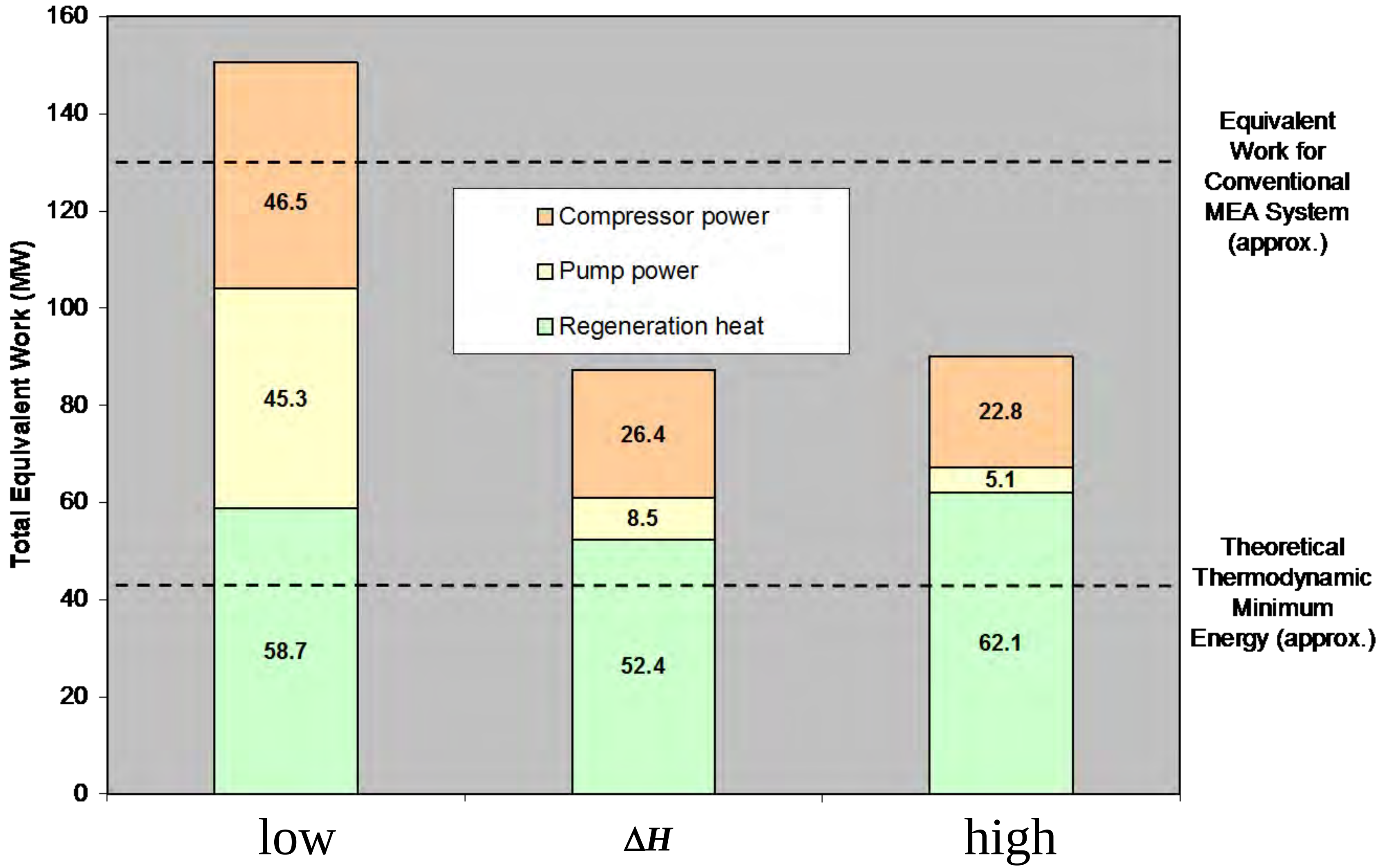


Gurkan, B., et. al., *J. Am. Chem. Soc.*, 2010, 132, 2116-2117

Optimization and Process Analysis

- Process simulation used to evaluate the sensitivity of a representative 500 MW coal plant CO_2 capture process to ionic liquid properties
- Results will be used to guide the development of next-generation ionic liquids
- Requirements
  - Set flue gas inlet T, P and composition
  - 90% CO_2 removal
  - Adjust regenerator T and P to minimize energy needs
- Sensitivity variables
  - Stoichiometry
  - Enthalpy of reaction
  - Loading (Keq)
  - Water miscibility

Total Equivalent Work

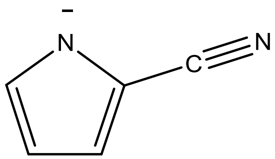


Trimeric

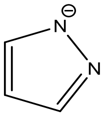
Different Aprotic Heterocyclic Anions

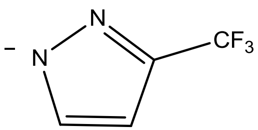
- Retain amine in ring structure but further reduce free hydrogens

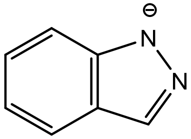
pyrrolides

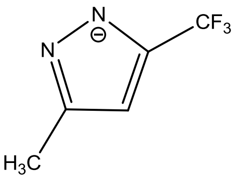


pyrazolides

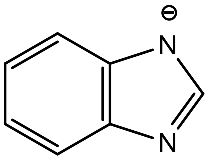


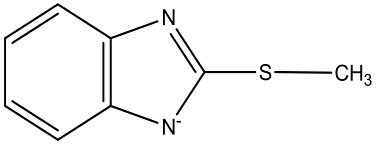


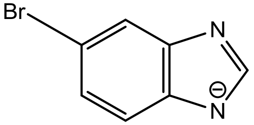




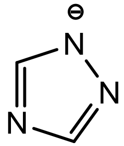
imidazolides

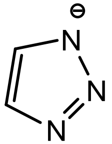






triazolides

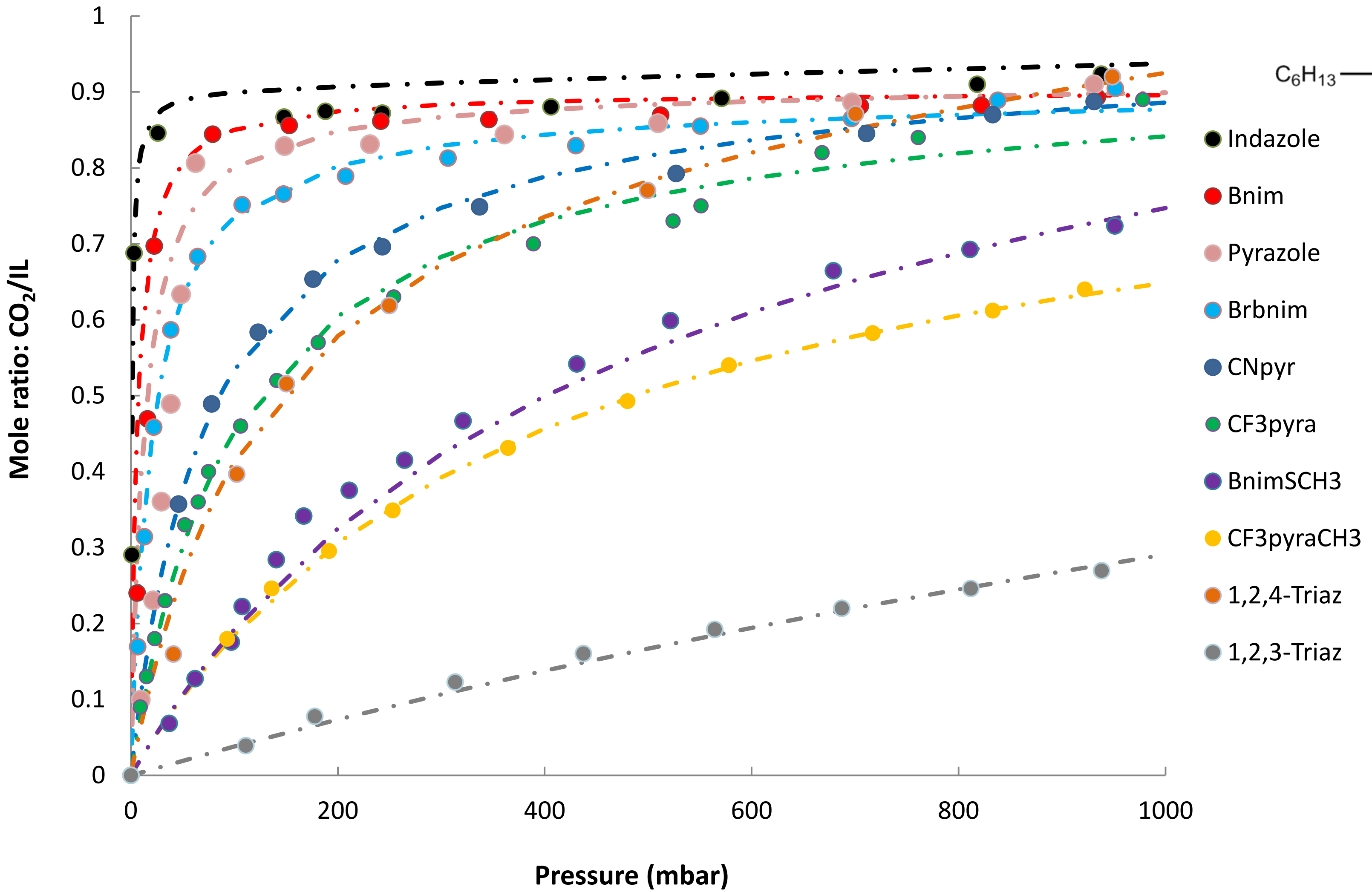


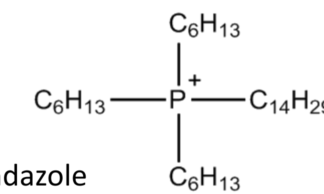


$-80^{o}C < T_g < -65^{o}C$

$260^{o}C < T_{decomp} < 330^{o}C$

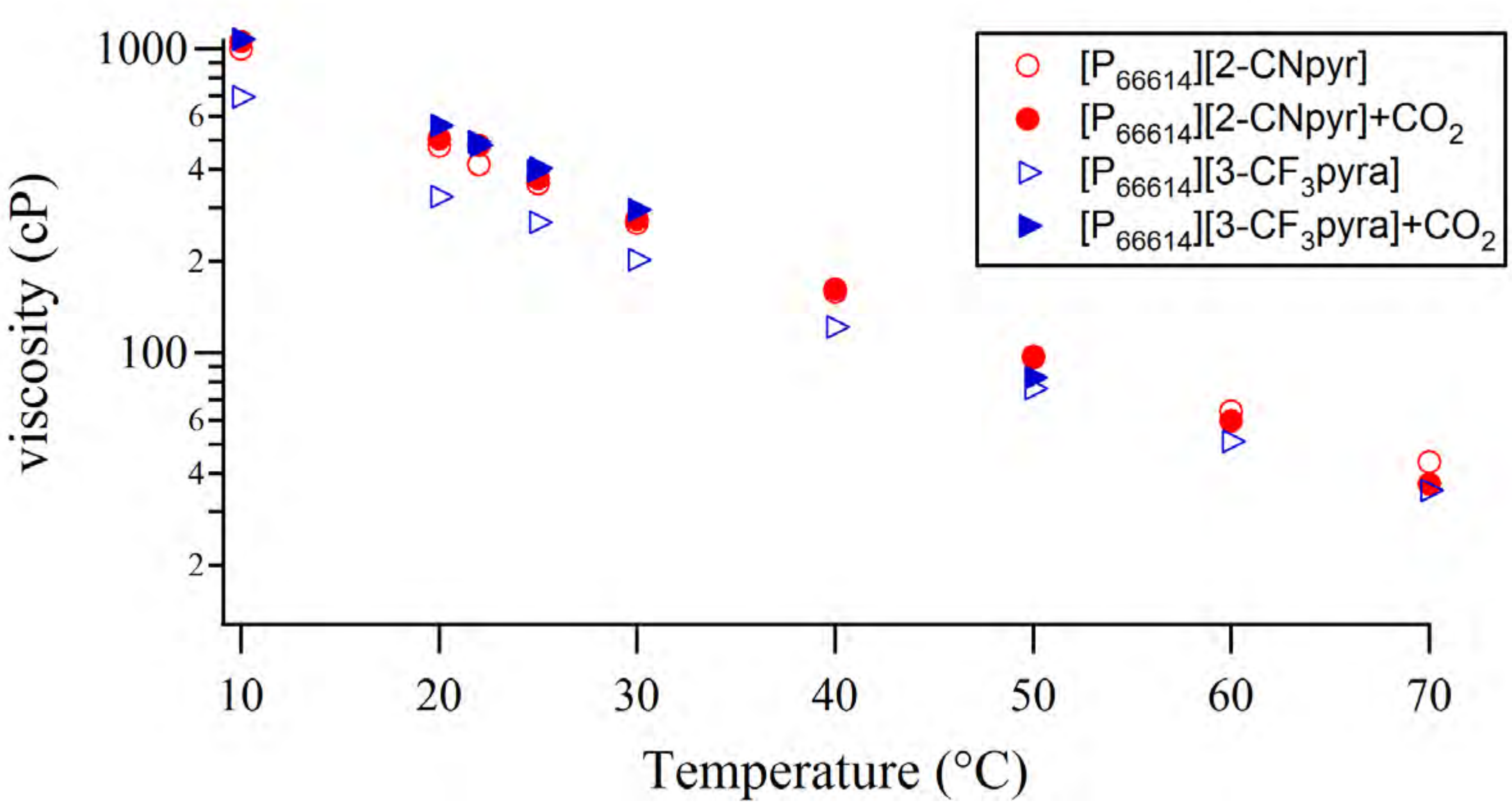
AHA CO_2 Uptake





AHA Viscosities

Very small viscosity increase when saturated with CO_2 at 1 bar and 22 °C



Gurkan et al., JPC Lett, 2010

Path Forward for CO_2 Capture with ILs

- Tested in lab-scale unit (4 in. columns) both at Notre Dame and Babcocks & Wilcox
- Using $[P_{66614}]$[2-cyanopyrrolide]
- 25 liters from Koei Chemicals
-ΔH_{chem} lower than optimal (-43 kJ/mole)
- Continue development and testing of new AHA ILs

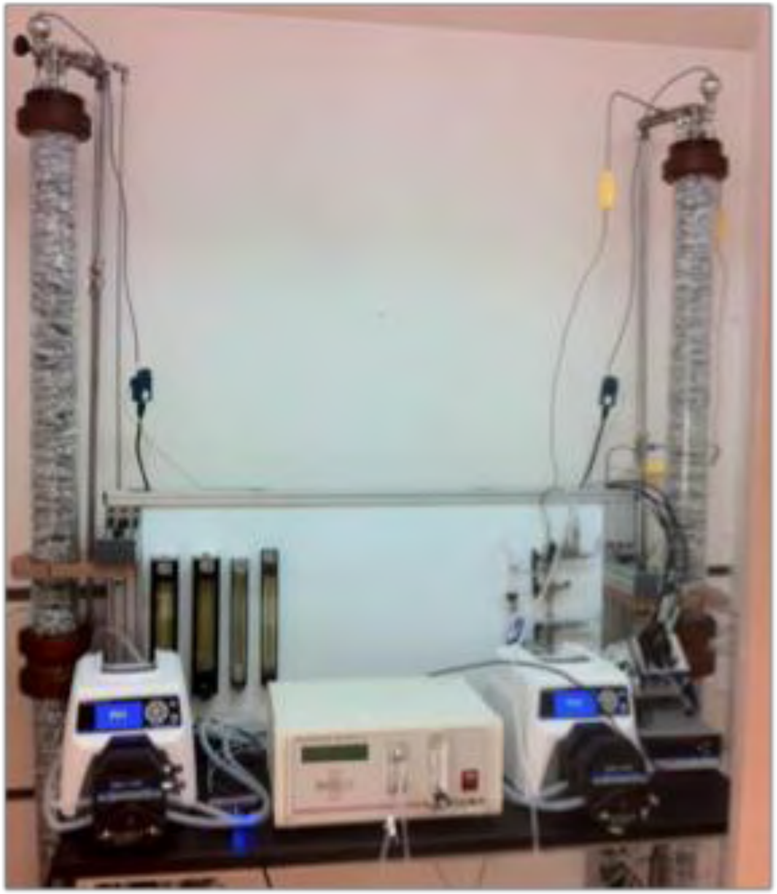
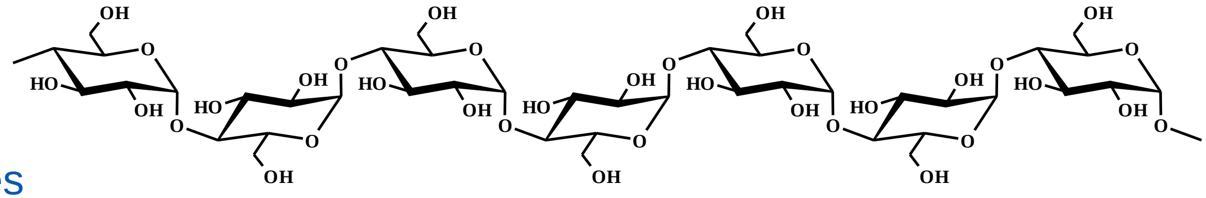


Figure 1: Image of the lab-scale unit constructed for Task 18.

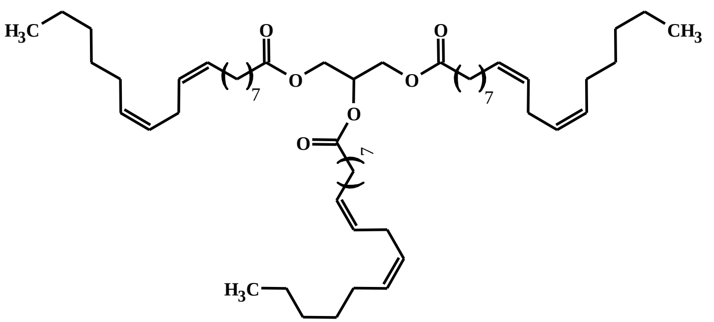
Key Non-Cellulosic Constituents of Biomass

Starch: 70-75% (Corn)



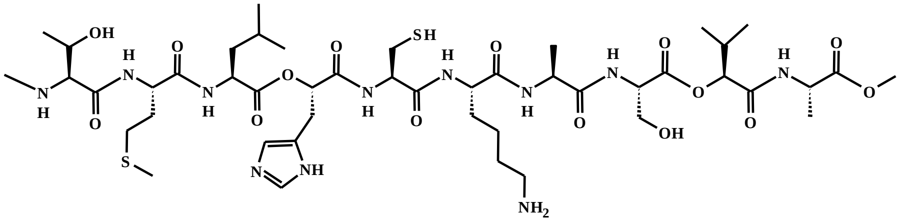
- Readily hydrolysable
- Basis for existing biorefineries
- Easily separable and fermentable fuels & chemicals

Oil: 4-7% (Corn) 18-20% (Soybeans)



- Readily separable from feedstock
- Starting material for clean Biodiesel
- Readily converted via chemical routes

Protein: 20-25% (Corn) 80% (Soybean Meal)



- Mostly used as a feed
- Underutilized as a polymer building block
- Potential feedstock for chemicals and resins

Biomass Chemistry

Lignin: 15-25%

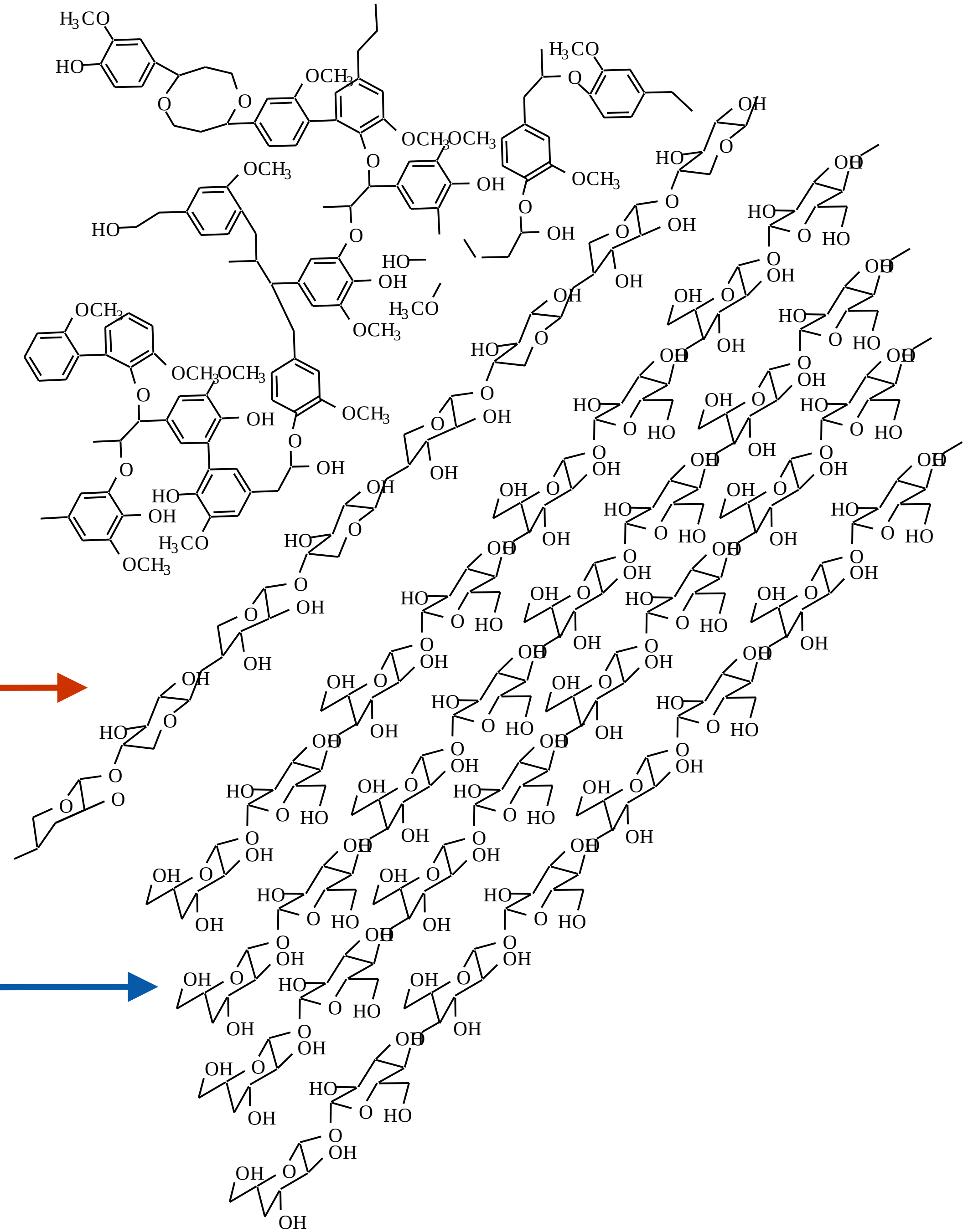
- Complex aromatic structure
- Resists biochemical conversion
- Requires high temperatures to convert

Hemicellulose: 23-32%

- Polymer of 5- and 6-carbon sugars
- Easily depolymerization
- 5-carbon sugars hard to metabolize

Cellulose: 38-50%

- Polymer of glucose
- Susceptible to enzymatic attack
- Glucose easy to metabolize



Use of Biomass

- Use starch, oil and protein for food
- Burn lignan to generate heat and power
- Use cellulose (and maybe hemi-cellulose) for chemicals and fuel
- Need to separate the components
- Use enzymes to convert the cellulose

Processing of Biomass with Ionic Liquids

- Cellulose doesn't dissolve in water or common organic solvents
- Cellulases must attack solid matrix
- Mass transfer limitations
- Solvents to dissolve
  - dimethylacetamide (DMAC)/LiCl solvents
  - N-methylmorpholine-N-oxide (NMNO) and phosphoric acid
  - Ionic liquids

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COMMUNICATIONS

Published on Web 04/17/2002

Dissolution of Cellose with Ionic Liquids

Richard P. Swatloski, Scott K. Spear, John D. Holbrey, and Robin D. Rogers*

Center for Green Manufacturing and Department of Chemistry, The University of Alabama, Tuscaloosa, Alabama 35487

Received February 1, 2002

Table 1. Solubility of Dissolving Pulp Cellulose in Ionic Liquids

ionic liquid	method	solubility (wt %)
$[C_4mim]Cl$	heat (100 °C)	10%
	(70 °C)	3%
$[C_4mim]Cl$	heat (80 °C) + sonication	5%
$[C_4mim]Cl$	microwave heating 3−5-s pulses	25%, clear viscous solution
$[C_4mim]Br$	microwave	5−7%
$[C_4mim]SCN$	microwave	5−7%
$[C_4mim][BF_4]$	microwave	insoluble
$[C_4mim][PF_6]$	microwave	insoluble
$[C_6mim]Cl$	heat (100 °C)	5%
$[C_8mim]Cl$	heat (100 °C)	slightly soluble

Processing of Biomass in Ionic Liquids

- IL/biomass solutions VERY viscous
- Most cellulases inactive in ILs that dissolve cellulose
- Can reconstitute cellulose by adding antisolvents (e.g., water, acetone)
- Pretreatment methods
  - Acids, ammonia, hot water, lime
  - Explosive decompression, ammonia fiber explosion
  - Ionic liquids

Processing of Biomass with Ionic Liquids

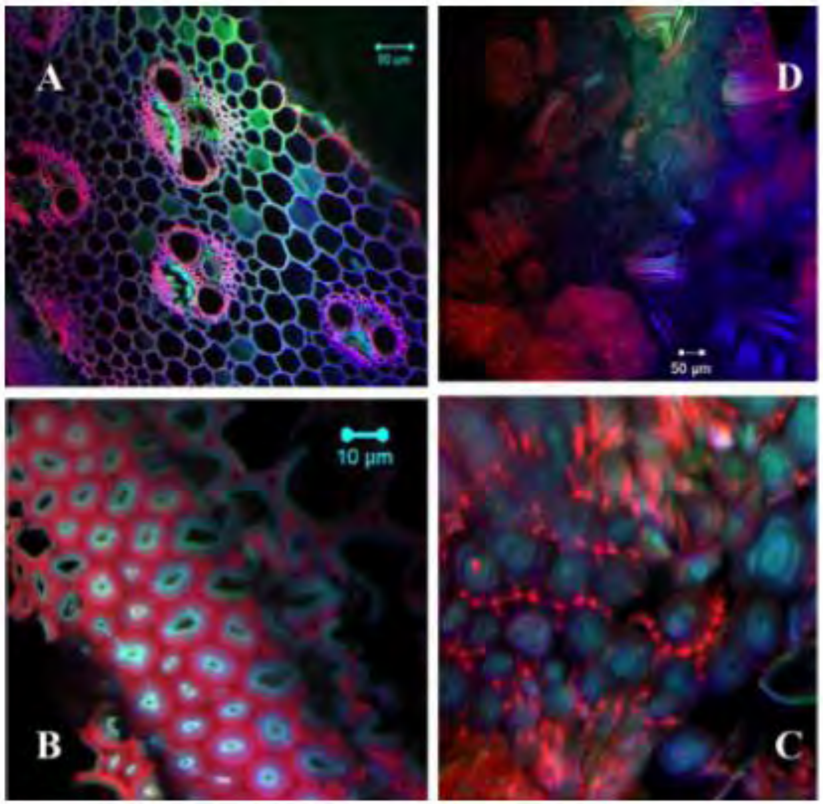


Figure 4. In situ dynamic study of switchgrass dissolution in ethyl methyl imidazolium acetate. Confocal fluorescence images of switchgrass stem section before pretreatment (**a**), and after 20 (**b**) and 50 (**c**) min of pretreatment. Complete breakdown of organized plant cell wall structure (**d**) is observed after 2 h.

ARTICLE

BIOTECHNOLOGY and BIOENGINEERING

2009, 104(1), 68-75

Visualization of Biomass Solubilization and Cellulose Regeneration During Ionic Liquid Pretreatment of Switchgrass

Seema Singh,[1,2] Blake A. Simmons,[1,2] Kenneth P. Vogel[3]

-[emim][acetate] disrupts/solubilized plant cell wall

- breaks up hydrogen bonding

- regenerate cellulose by adding water as an anti-solvent

- regenerated cellulose easily processed by cellulases (15x kinetics vs. untreated)

ILs as Electrolytes
Batteries, Supercapacitors, Fuel Cells

- Wide electrochemical window (up to 4-6 V)
- Can get larger voltages than you can get with an aqueous solution
- Need high conductivity

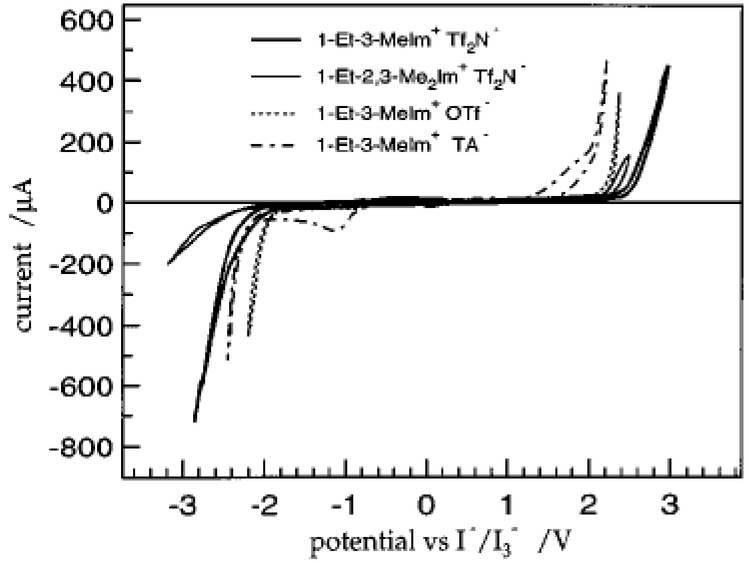


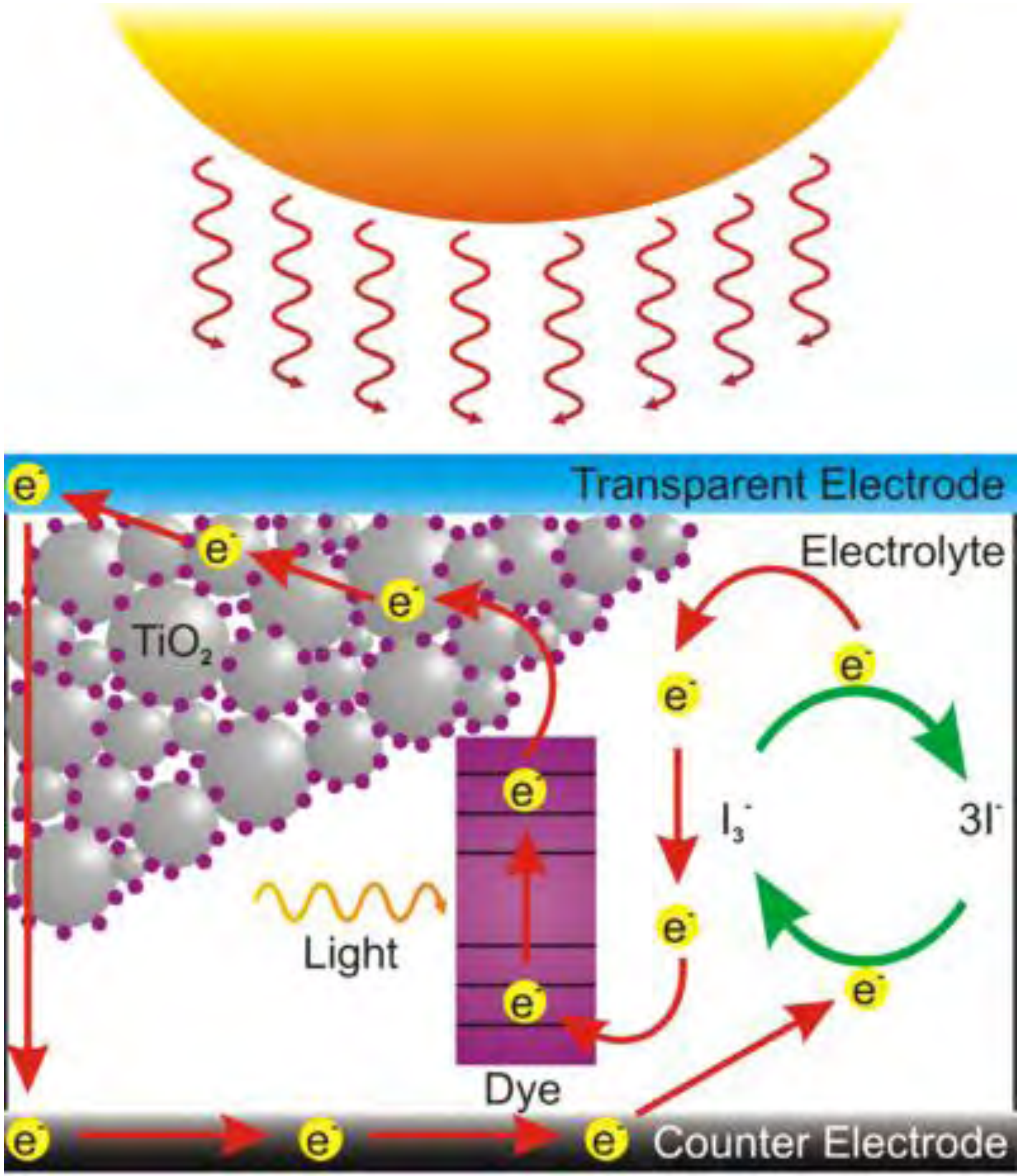
Figure 7. Cyclic voltammograms for a stationary Pt electrode (0.78 mm^2) in various ionic liquids at 23 °C. Sweep rate: 50mV/s. The I^-/I_3^- couple has a measured potentiel of −0.195 V vs ferrocene/ferrocenium in $EtMeIm^+Tf_2N^-$. The reduction current observed in the vicinity of −1.0 V in $EtMeIm^+TA^-$ appears to result from the reduction of dissolved H_2O. Effective drying of this salt could not be performed because of the low decomposition temperature (see Figure 3).

Electrolysis of water

Anode (oxidation): $2\ H_2O(l) \rightarrow O_2(g) + 4\ H^+(aq) + 4e^-$ $E_o^{ox} = -1.23$ V

Cathode (reduction): $2\ H^+(aq) + 2e^- \rightarrow H_2(g)$ $E_o^{red} = 0.00$ V

Dye Sensitized Solar Cells



-Dye sensitized solar cells
-Developed by Michael Gratzel in 1991
-Molecular dye on TiO_2 absorbs sunlight
-Electrons flow through TiO_2 to electrode
-Electrons power external load
-Electrons reintroduced to cell through counter electrode
-Electrons flow through electrolyte back to dye, through iodide/tri-iodide couple

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Review

Dye-sensitized solar cells

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Received 3 June 2003; received in revised form 1 July 2003; accepted 1 July 2003

Abstract

The dye-sensitized solar cells (DSC) provides a technically and economically credible alternative concept to present day p–n junction photovoltaic devices. In contrast to the conventional systems where the semiconductor assume both the task of light absorption and charge carrier transport the two functions are separated here. Light is absorbed by a sensitizer, which is anchored to the surface of a wide band semiconductor. Charge separation takes place at the interface via photo-induced electron injection from the dye into the conduction band of the solid. Carriers are transported in the conduction band of the semiconductor to the charge collector. The use of sensitizers having a broad absorption band in conjunction with oxide films of nanocrstalline morphology permits to harvest a large fraction of sunlight. Nearly quantitative conversion of incident photon into electric current is achieved over a large spectral range extending from the UV to the near IR region. Overall solar (standard AM 1.5) to current conversion efficiencies (IPCE) over 10% have been reached. There are good prospects to produce these cells at lower cost than conventional devices. Here we present the current state of the field, discuss new concepts of the dye-sensitized nanocrystalline solar cell (DSC) including heterojunction variants and analyze the perspectives for the future development of the technology.

Keywords: Solar light energy conversion; Dye-sensitized solar cells; Nanocrystalline oxide semiconductor films; Organic hole conductors; Ionic liquids; Ruthenium charge transfer sensitizers

Inorg. Chem. **1996,** *35,* 1168−1178

Hydrophobic, Highly Conductive Ambient-Temperature Molten Salts†

Pierre Bonhôte,* Ana-Paula Dias, Nicholas Papageorgiou, Kuppuswamy Kalyanasundaram, and Michael Grätzel*

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Received August 15, 1995⊗

New, hydrophobic ionic liquids with low melting points (<−30 °C to ambient temperature) have been synthesized and investigated, based on 1,3-dialkyl imidazolium cations and hydrophobic anions. Other imidazolium molten salts with hydrophilic anions and thus water-soluble are also described. The molten salts were characterized by NMR and elemental analysis. Their density, melting point, viscosity, conductivity, refractive index, electrochemical window, thermal stability, and miscibility with water and organic solvents were determined. The influence of the alkyl substituents in 1, 2, 3, and 4(5)-positions on these properties was scrutinized. Viscosities as low as 35 cP (for 1-ethyl-3-methylimidazolium bis((trifluoromethyl)sulfonyl)amide (bis(triflyl)amide) and trifluoroacetate) and conductivities as high as 9.6 mS/cm were obtained. Photophysical probe studies were carried out to establish more precisely the solvent properties of 1-ethyl-3-methylimidazolium bis((trifluoromethyl)sulfonyl)amide). The hydrophobic molten salts are promising solvents for electrochemical, photovoltaic, and synthetic applications.

Ionic Liquids as absorbent in chillers

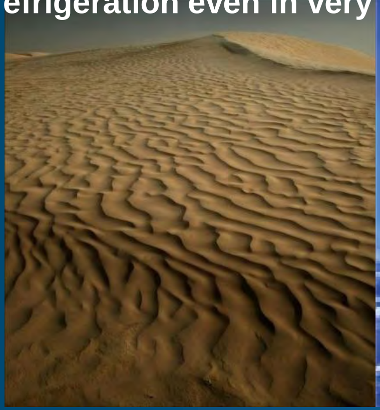
degussa.
creating essentials

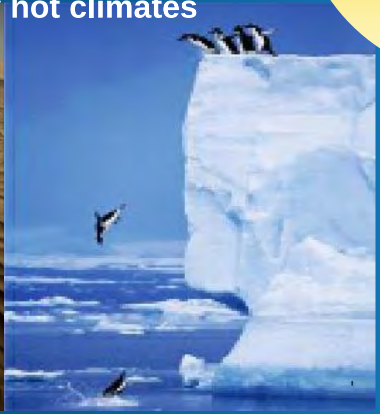
1

Absorption chillers with cooling capacities of over 30MW need over 100t of absorbent

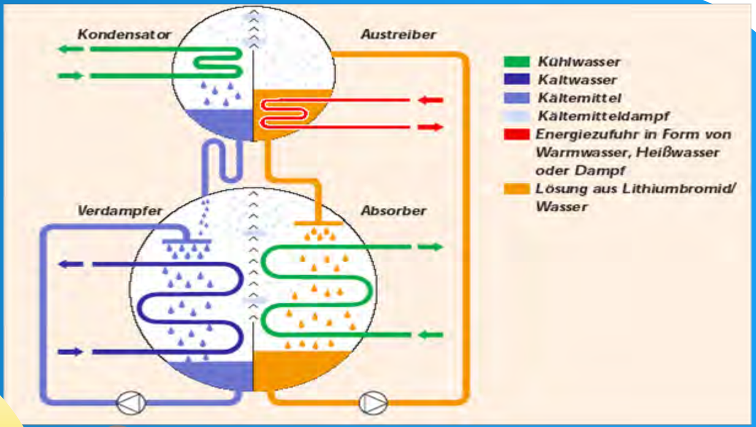
It might be possible to convert heat directly into refrigeration even in very hot climates

4



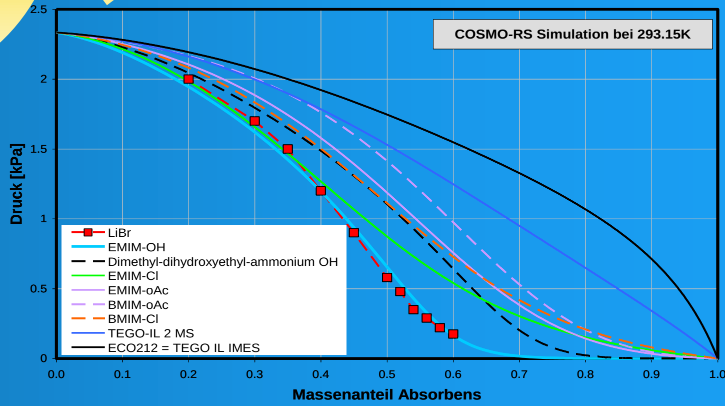


Ionic Liquids

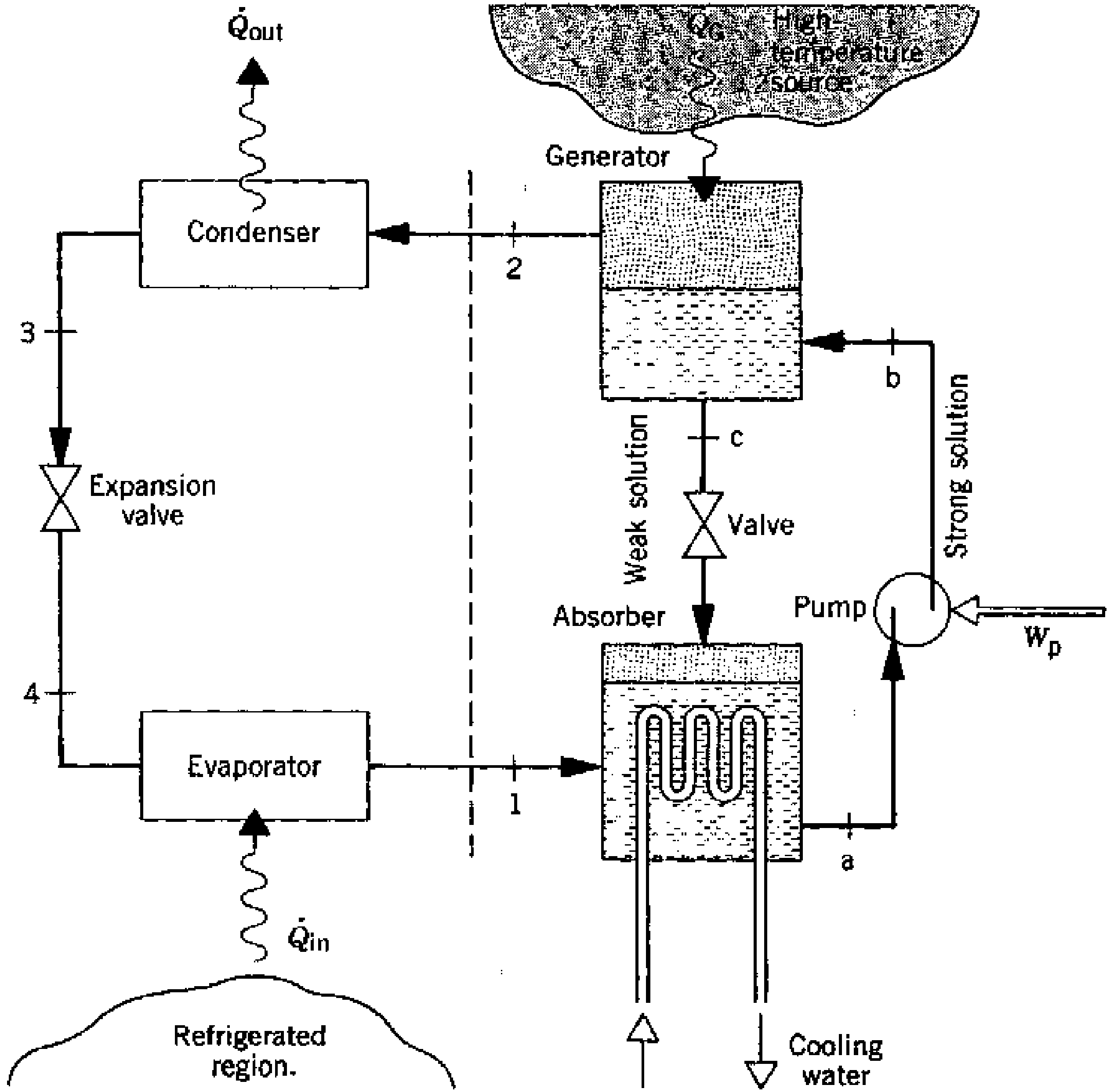


2 **Today LiBr is used as absorbent, but it has serious technical limitations**

3 **ILs show similar thermodynamic behaviour and could resolve tech problems**



P. Schwab - Degussa



$\dot{Q}_{out}$
Q_G
High-temperature source
Generator
Condenser
2
3
b
c
Expansion valve
Weak solution
Valve
Strong solution
Pump
W_p
Absorber
4
Evaporator
1
a
$\dot{Q}_{in}$
Refrigerated region.
Cooling water

Absorption Refrigeration

- Replace electrical energy with low value heat energy
- Problems with conventional systems
  - LiBr/water is corrosive, solidification problems
  - Water/ammonia is toxic, odor nuisance
- Replace with IL/water, IL/CO_2, IL/HFC systems

Coefficient of Performance

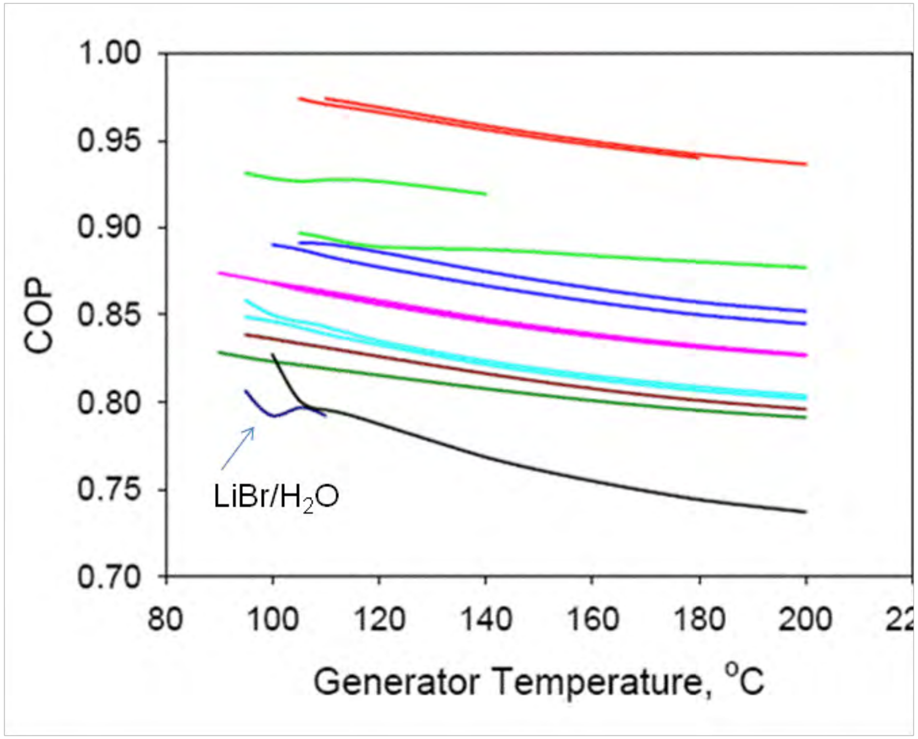
- COP = chilling capacity/(work+heat in)
- COPs much lower for absorption refrigeration systems than vapor compression systems but lower grade energy (heat instead of electricity)
- COPs depend on enthalpies at each place in cycle
- H as function of T, P and composition
- Measure C_p of pure ILs, ΔH_{mixing} of IL/water, VLE

$$P = x_i \gamma_i P_i^{sat}$$

Absorption Refrigeration

- Excellent coefficients of performance, especially for generation temperatures < 150 °C

T_{evap} = 5 ºC
T_{cond} = 50 ºC
T_{abs} = 40 ºC



Summary

- ILs finding many uses in diverse energy applications
- CO_2 capture
- Biomass processing
  - Dissolution/pretreatment of cellulose
- ILs as electrolytes
  - Batteries, Supercapacitors, Fuel Cells, DSSC
  - Heat transfer fluid for solar thermal
- Absorption refrigeration
- Tunable nature is what is important – not one, but many different solvents