



Process for the Purification of Alpha-omega- diiodoperfluorinated Compounds

Speaker

Giuseppe Resnati

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NFMLab, Dept. Chemistry and Materials “Giulio Natta”, Politecnico di Milano, Italy



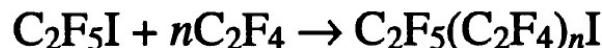
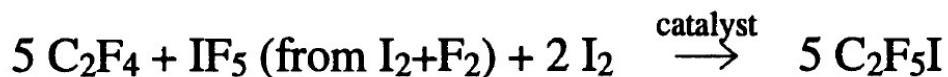


Perfluoroalkyl iodides



Perfluoroalkyl iodides are key intermediates in fluoroorganic chemistry

- **Synthesis:**



- **Reactions:**

- **radical reactions:**

- addition to double and triple bonds
 - addition to isonitriles
 - fluoroalkylation of aromatics

- reactions with nucleophiles (carbon, phosphorous, sulfur, selenium, ...)
 - formation of organometallic derivatives (Li, Na, Mg, Cu, Zn, Cd, ...)
 - electrochemical transformations

- **Uses:**

- surfactants
 - textile, leather, paper finishes



Halogen bonding: A paradigm in supramolecular chemistry



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AT555073 (T)

Bibliographic data

Description
Claims
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Bibliographic data: AT555073 (T) — 2012-05-15

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PROCESS FOR THE PURIFICATION OF ALPHA,OMEGA-DIIODOPERFLUORINATED COMPOUNDS

Page bookmark AT555073 (T) - PROCESS FOR THE PURIFICATION OF ALPHA,OMEGA-DIIODOPERFLUORINATED COMPOUNDS

Inventor(s): METRANGOLO PIERANGELO [IT]; RESNATI GIUSEPPE [IT]; CARCENAC YVAN [IT] ± (METRANGOLO, PIERANGELO, ; RESNATI, GIUSEPPE, ; CARCENAC, YVAN)

Applicant(s): MILANO POLITECNICO [IT] ±

Classification: - international: C07C17/389; C07C17/392; C07C19/16; C07C211/63
- cooperative: C07C17/38; C07C17/389; C07C17/392; C07C17/395; C07C19/16; C07C211/63 → more

Application number: AT20080774705T 20080703

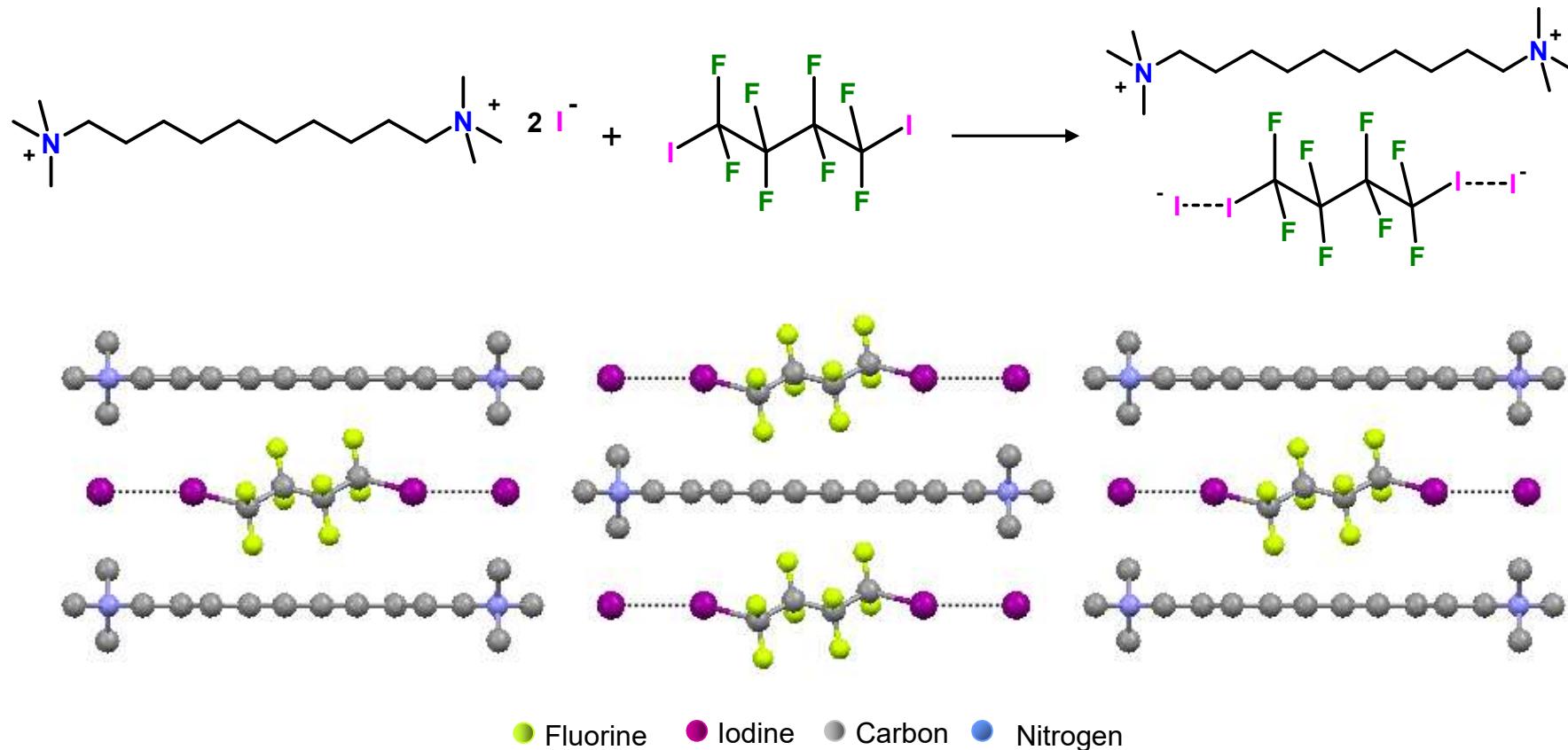
Priority number(s): WO2008EP58588 20080703 ; IT2007MI01384 20070711

Also published as: □ WO2009007302 (A2) □ WO2009007302 (A3) □ US2010197967 (A1) □ JP2010533136 (A) → ITMI20071384 (A1) □ EP2181082 (A2) □ EP2181082 (B1) → less

Abstract not available for AT555073 (T)
Abstract of corresponding document: WO2009007302 (A2)

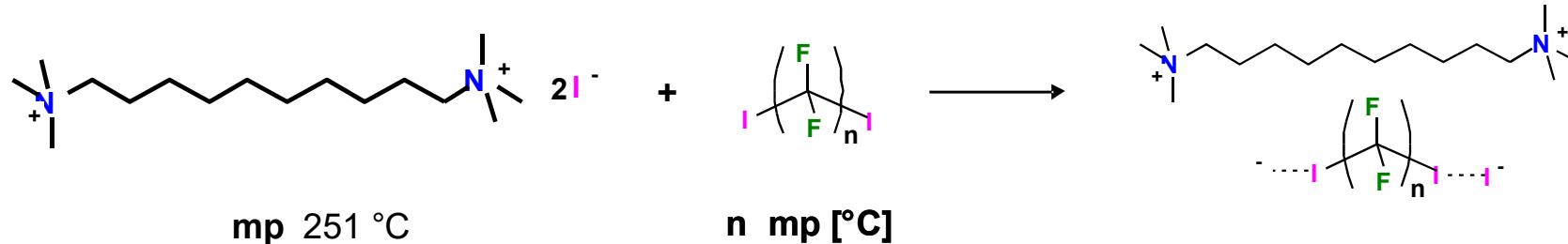
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The invention concerns a process for purifying α,β -diiodoperfluorinated compounds of formula I-(CF₂CF₂Y)_m-I comprising contacting an impure α,β -diiodoperfluorinated compound with a suitable sequestering agent, with formation of a reversible adduct and separating the α,β -diiodoperfluorinated compound from the adduct itself. The invention also concerns α,β -diiodoperfluorinated compounds of formula I-(CF₂CF₂Y)_m-I wherein m is 5 to 10 and having 100% purity grades.



- I···I bond distance: 3.452 Å
- I···I-(CF₂)₄-I···I distance: 14.483 Å
- C-I···I⁻ angle: 166.02°
- ⁺N-(CH₂)₁₀-N⁺ distance: 13.628 Å
- $\Delta = 0.85 \text{ \AA}$

Science 2009, 323, 1461 (highlighted on Chem. Eng. News 2009, 87(March 16), 49)



n mp [°C]

2	-28
4	-9
6	25
8	77

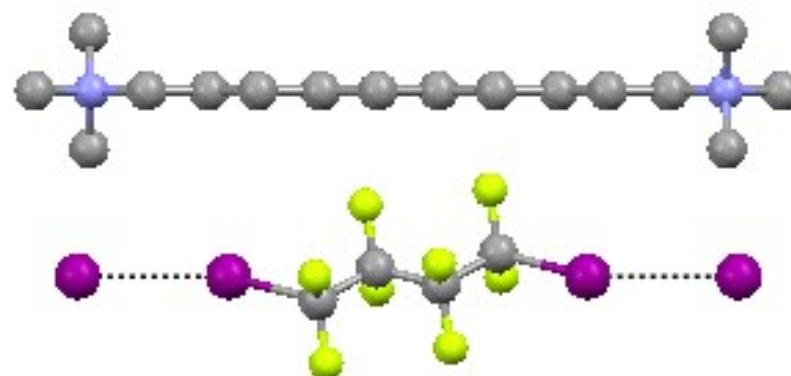
n	Mp [°C]	mp [°C]	Δ mp [°C]	Solubility mean values [mg/mL] ^a
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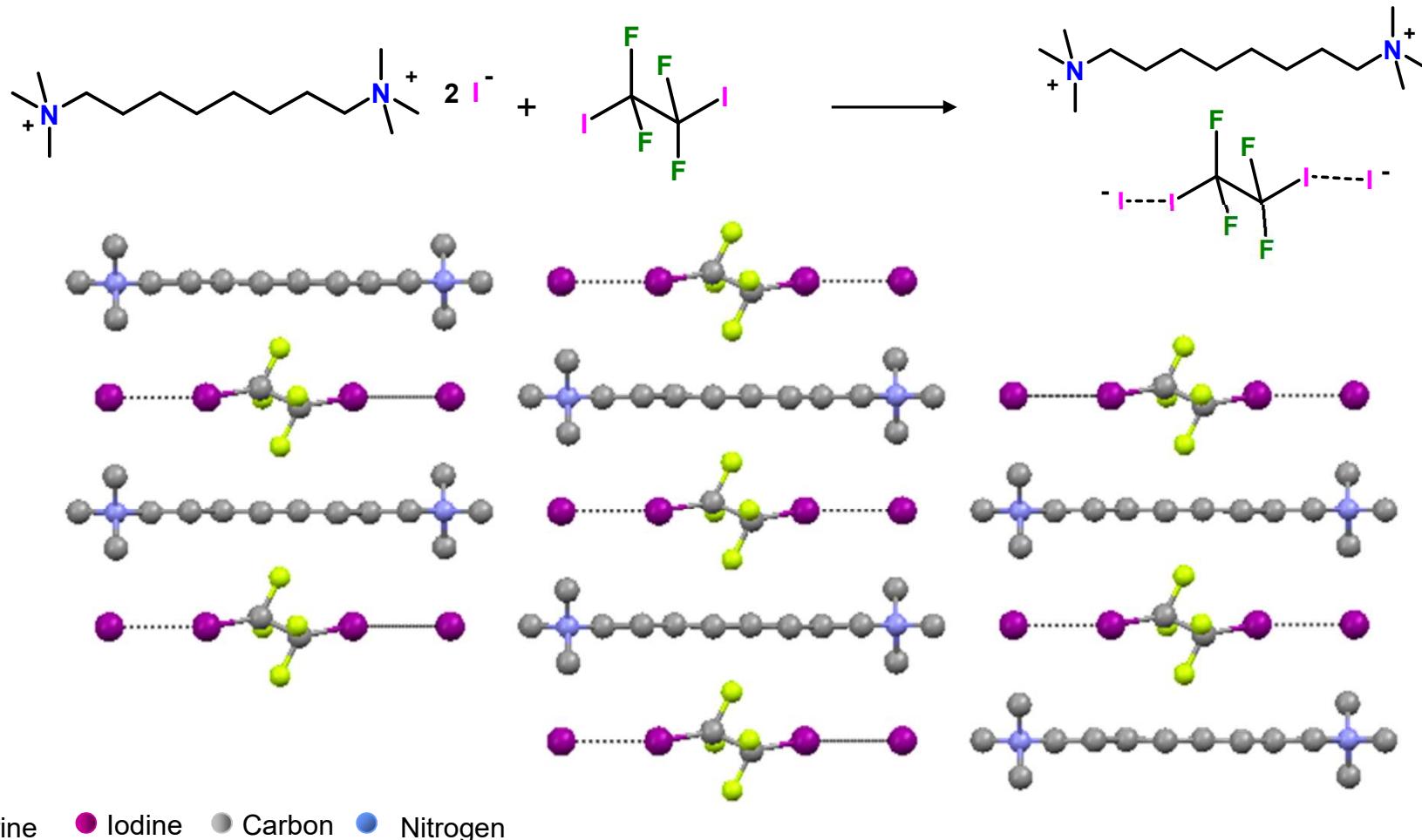
2	140	111	29	38
4	230	121	109	<1
6	191	138	53	15
8	132	164	-32	47

^a (CH₂Cl₂/MeOH 9/1)

Decamethonium iodide invariably precipitates I-(CF₂)_n-I out of I-(CF₂)_n-I mixtures

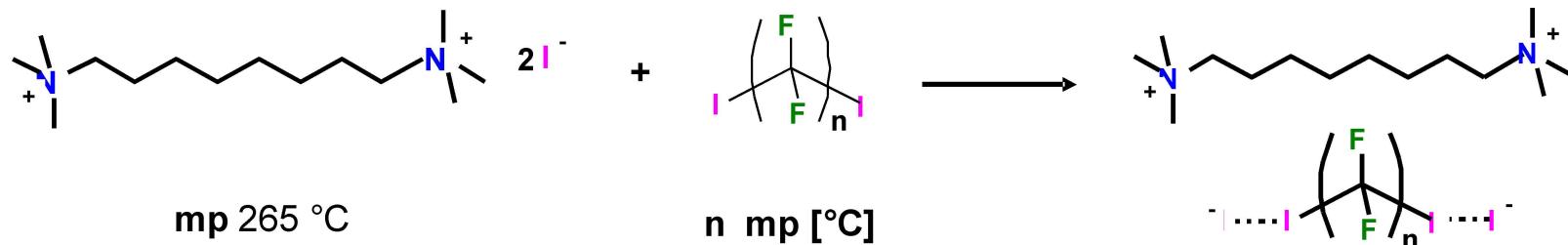
n	2	4	6	8
2	/	0/100	5/95	28/72
4	/	/	100/0	100/0
6		/	64/36	
8			/	





- $\text{I} \cdots \text{I}$ bond distance: 3.502 Å
- $\text{I} \cdots \text{I}-(\text{CF}_2)_2-\text{I} \cdots \text{I}^-$ distance: 12.142 Å
- C-I \cdots I $^-$ angle: 169.16°
- $^{+}\text{N}-(\text{CH}_2)_8-\text{N}^+$ distance: 11.343 Å

• $\Delta = 0.80 \text{ \AA}$



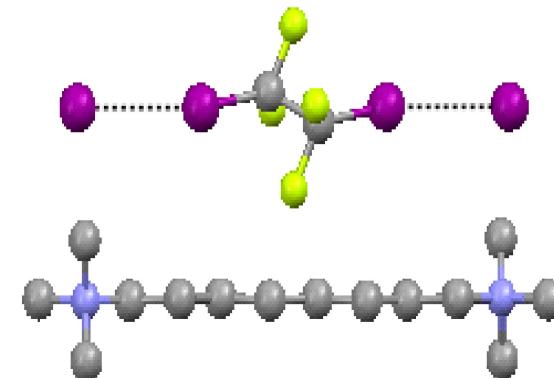
n mp [°C]

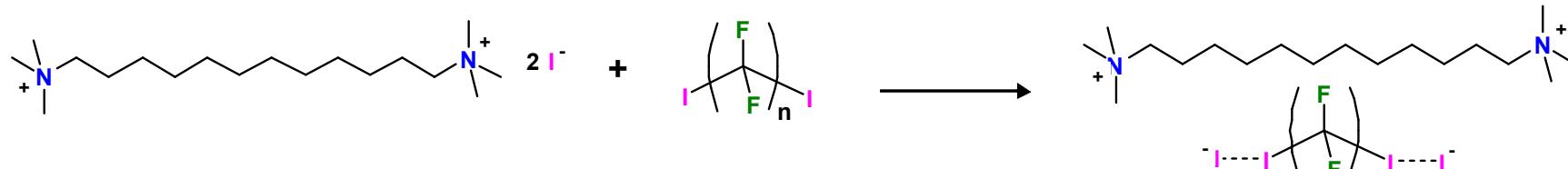
2	-28
4	-9
6	25
8	77

n	mp [°C]	mp [°C]	Δ mp mean values [°C]
2	201	118	83
4	188	128	60
6	182	135	37
8	180	171	9

Octamethonium iodide invariably precipitates I-(CF₂)₂-I out of I-(CF₂)_n-I mixtures

n	2	4	6	8
2	/	100/0	100/0	100/0
4	/	71/29	55/45	
6	/		62/38	
8				/





mp 222°C

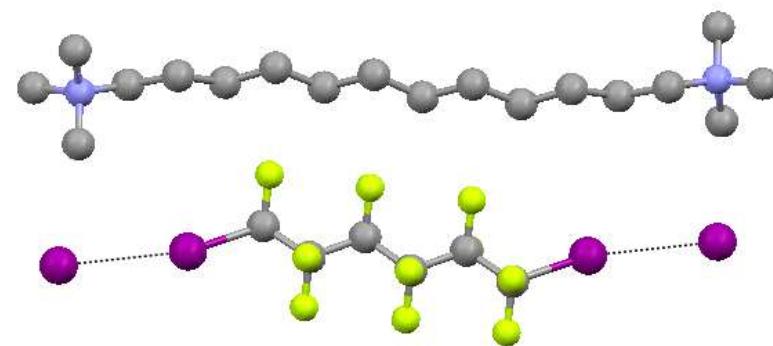
n mp [°C]

2	-28
4	-9
6	25
8	77

n	mp [°C]
2	134
4	218
6	226
8	191

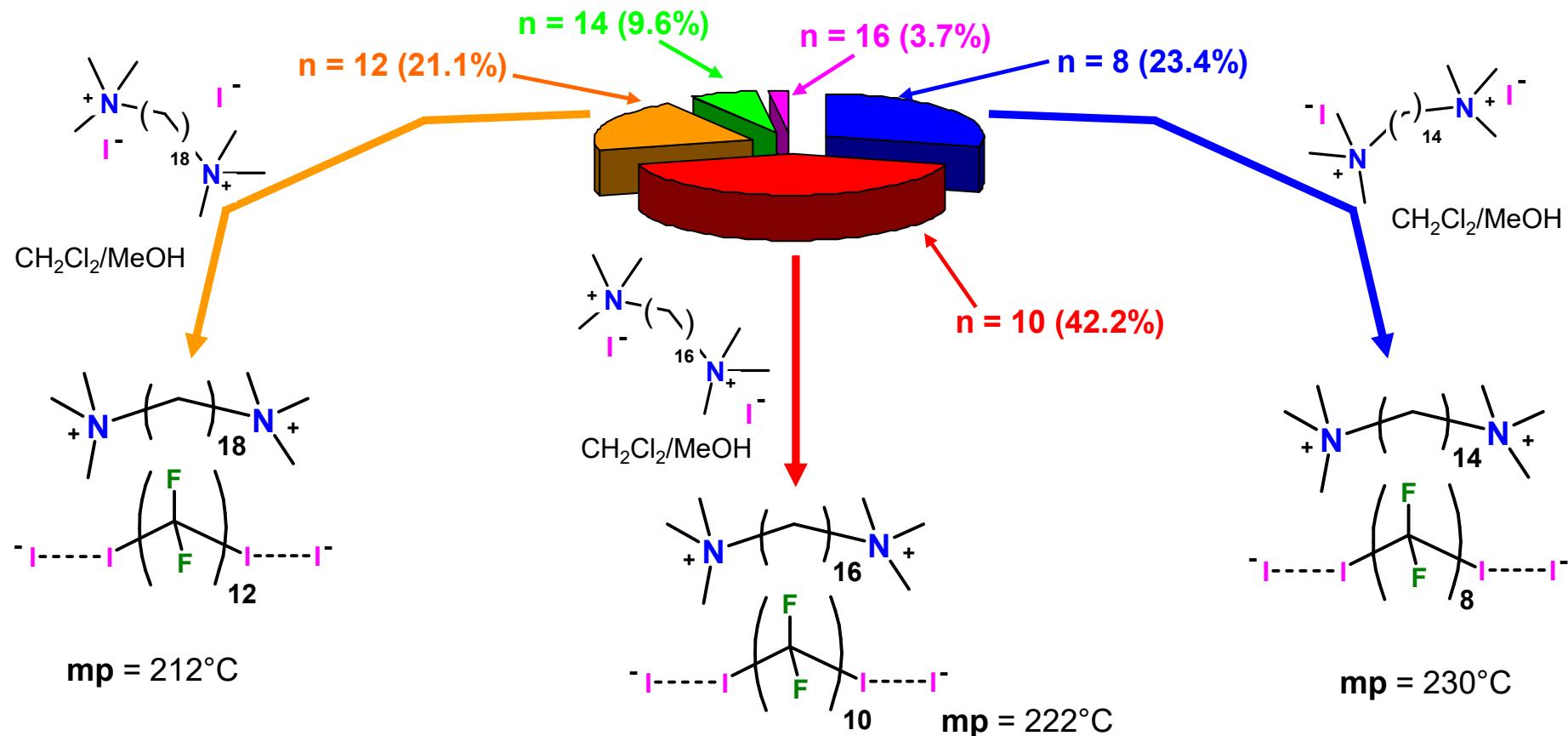
Dodecamethonium iodide invariably precipitates I-(CF₂)₆-I out of I-(CF₂)_n-I mixtures

n	2	4	6	8
2	/	15/85	0/100	3/97
4	/	/	0/100	22/78
6			/	100/0
8				/



Science 2009, 323, 1461 (highlighted on Chem. Eng. News 2009, 87(March 16), 49)

Separation of I-(CF₂)_n-I mixtures



Patent application PCT/EP2008/ 058588 (2008)

- Quantitative recovery of pure diiodoalkanes by their vacuum distillation from the cocrystals
- Recovery of the pure onium salt for recycle
- Exploitation of byproducts
- Easy scalability of the process
- Scrambling of telomers during purification is avoided
- Convenient handling/storage/transport

- Possible extension of the technology to other α,ω -dihaloperfluorocarbons (change of the number and nature of the heavy halogen, of the size and nature of the carbon spacer)

... questions?



Related impact



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