

Iodine speciation in nuclear industry.

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Outline of the talk:

- **Introduction.**
- **Back to basics on the element I.**
- **Speciation of iodine in solution.**
- **Speciation of iodine in the gaseous phase.**
- **Speciation of iodine in the solid phase and imaging.**
- **Conclusions.**

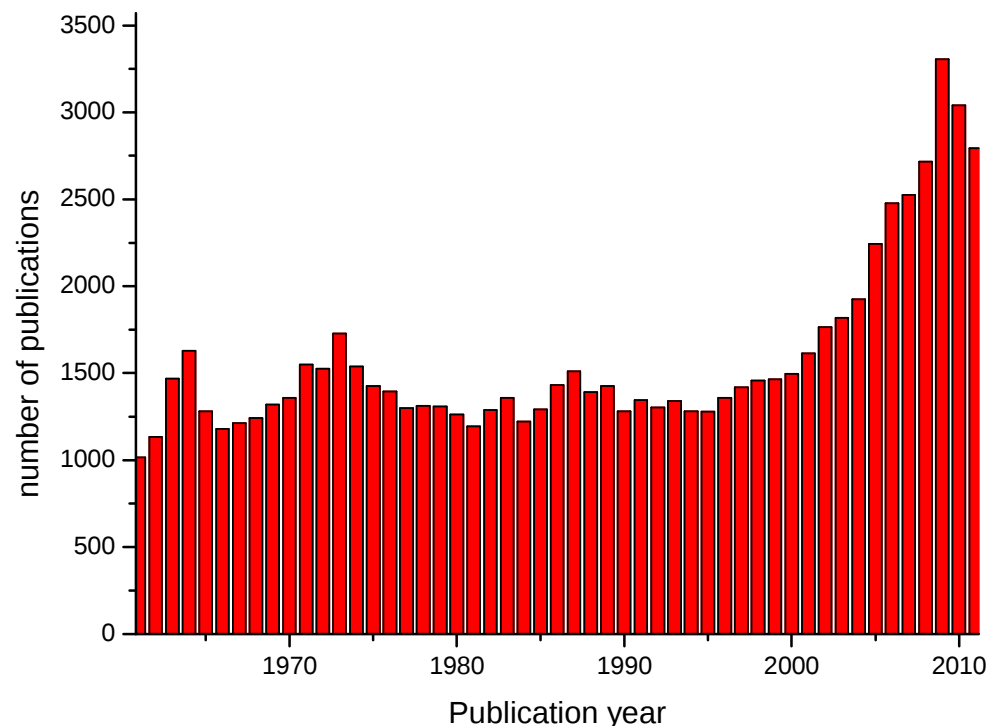
Introduction.

This talk is a review of iodine speciation in the nuclear industry and presents some experimental work done at the Nuclear Energy Division and especially in our laboratory.



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Iodine : 257953 references on SciFinder (3/05/2012) of which 76103 references for stable I-I (CAS number 7553-56-2).



Introduction.

Iodine is a very important element which is present in numerous and various domains of our life.



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- ***Environmental domain:***

From the geosphere to the biosphere : soils, air, sea (450 nM [I₂], brown algae Laminaria Digitata, strongest accumulator of iodine among all living systems).

- ***Medecine and physiology:***

Tissues, urine, colon, thyroïd where it plays an important role in the endocrine system. The body of a healthy adult contains up to 20 mg iodine which 70-80% is in the thyroid. It is used as a disinfectant in aqueous solution (tincture of iodine).

- ***Nuclear industry:***

Iodine is a fission product released in the case of severe nuclear accidents.

It is present under volatile forms (molecular iodine, organic iodides) or under the form of aerosols (metal iodides).

Iodine is released at the dissolution of irradiated nuclear fuels.

Iodine is involved in the massive hydrogen production using iodine sulphur water splitting cycle.

The knowledge of iodine chemistry as well as its speciation are of utmost importance. Analytical techniques for iodine speciation are transverse.

Back to Basics on the element I



Iodine element belongs to the halogen family. Group 17 of the PTable
Discovered by B. Courtois in 1811, from the greek *ιώδης* (violet).

Physical properties of the element:

- 53 electrons ($5s^2 5p^5$)
- numerous isotopes: 108 à 144



The more stable isotopes

Isotope	Period	Decay mode	Energy, MeV
123 I	13.2235 h	α, γ	0.16
125 I	59.4 j	α, γ	0.0355
127 I	stable		
129 I	15.7 Ma	β^-	0.194
131 I	8.0207j	β^-, γ	0.971

17 VIIA
fluor 9 F 18,9984032
chlor 17 Cl 35,4527
brom 35 Br 79,904
iod 53 I 126,90447

← Medical applications
(labeling, curietherapy,...)

↗ Nuclear domain
↘

Similar to the other halogens, the valence electron configuration renders I_2 molecule the stable state of the element with a direct and single I-I bond.

Isotopic abundance of iodine

Table of the Isotopes

11-113



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Elem. or Isot.	Natural Abundance (Atom %)	Atomic Mass or Weight	Half-life/ Resonance Width (MeV)	Decay Mode/ Energy (/MeV)	Particle Energy/ Intensity (MeV/%)	Spin ($h/2\pi$)	Nuclear Magnetic Mom. (nm)	Elect. Quadr. Mom. (b)	γ -Energy / Intensity (MeV/%)
									0.7228/10.3
									1.6910/11.2
									(0.31–1.73)
¹²⁵ I		124.904630	59.4 d	EC/0.1861		5/2+	2.82	-0.89	Te k x-ray
									0.0355
¹²⁶ I		125.905624	13.0 d	EC/		2-	1.44		ann.rad./
				β^+ /2.155	1.13/				Te k x-ray
				β^- /1.258/47	0.87/				0.3887
					1.25/				0.6622
¹²⁷ I	100.	126.904473				5/2+	+2.8133	-0.79	
¹²⁸ I		127.905809	25.00 m	β^- /2.118	2.13/	1+			Te k x-ray
									0.44287
									0.52658
¹²⁹ I		128.904988	1.7×10^7 y	β^- /0.194	0.15/	7/2+	+2.621	-0.55	Xe k x-ray
									0.0396
^{130m} I			9.0 m	1.T./83/0.048		2+			1 k x-ray
				β^- /17/					0.5361
¹³⁰ I		129.906674	12.36 h	β^- /2.949	1.04/	5+	3.35		0.4180
					0.62				0.5361
									0.6685
									0.7395
¹³¹ I		130.906125	8.021 d	β^- /0.971	0.606/	7/2+	+2.742	-0.40	0.08017
									0.28431
									0.36446
									0.63699
^{132m} I			1.39 h	IT		8-			
¹³² I		131.90800	2.28 h	β^- /14/3.58	0.80/	4+	3.09	0.09	1 k x-ray
				1.T./86/	1.03/				0.0980
					1.2/				0.5059
					1.6/				0.52264
					2.16/				0.63019
									0.6506

<http://www.hbcprnetbase.com/>

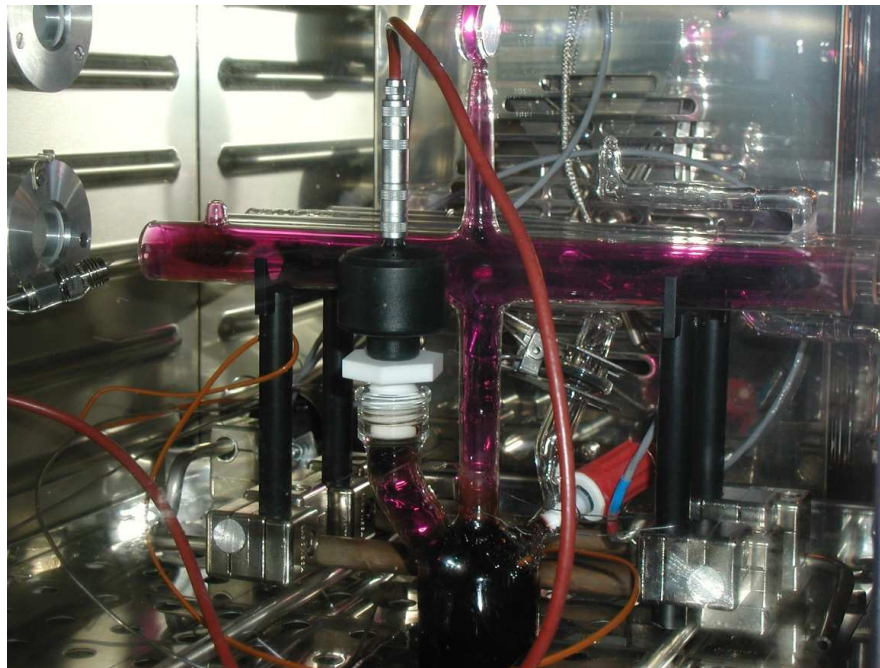
Iodine, a « sticky » molecule.

Black solid with metallic blue grey reflects. (Brown algae have been used as the raw material for iodine production at the origin. Today, the production comes from Chile (2/3) caliche deposits and Japan (1/3) brines from natural gas).

Fusion T° : $113,7^{\circ}\text{C}$

Boiling T° : 184.4°C

It sublimates at the ambient temperature.



Violet vapors of iodine observed around 120°C

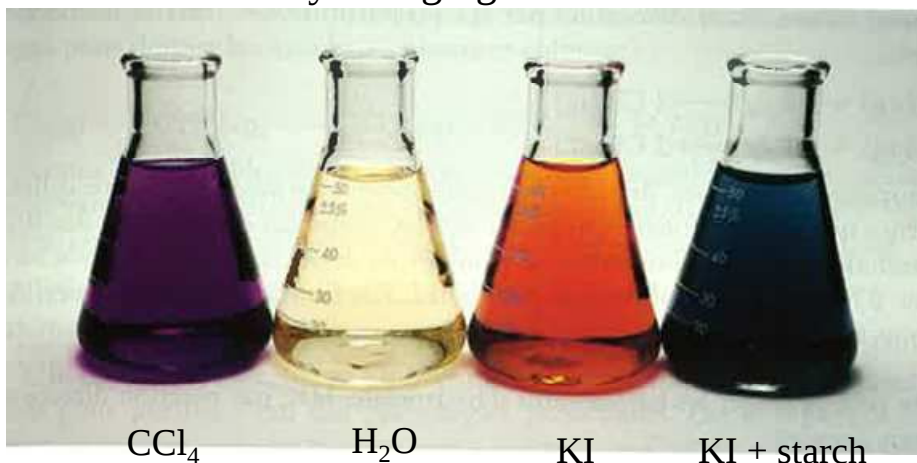
Iodine in solution: an extensive chemistry.

Iodine solubility in water is low: $2.68 \cdot 10^{-3} \text{ mol.L}^{-1}$ at 25°C .

Iodine molecule dissolves in organic solvents and gives different colored solutions. I_2 accepts electrons from the solvent molecule into its LUMO. This lowers the energy of the transition HOMO-LUMO thereby changing the color.



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From Atkins, Jones
Principles of chemistry

Iodine molecule solubility is increased in the presence of I^- ions thanks to the reaction:



Iodine electronegativity is lower than the one of the other halogens.

Oxydant reagent which can also be oxydized.

Iodine has many oxydation states ranging from -1 to +7.

The different oxydation states of iodine in solution



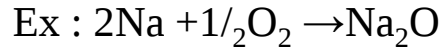
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Thermodynamically
stable valences

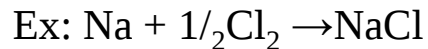
Oxidation state	Species	State	Name
-1	HI	gaseous	hydroiodic acid
	I ⁻	dissolved	iodide anion
	I ₃ ⁻	dissolved	Triiodide anion
	I ₅ ⁻	dissolved	Pentaiodide anion
0	I ₂	solid	Iodine
		dissolved gaseous	
+1	I ⁺		
	HOI	gaseous dissolved	Hypoiodous acid
	H ₂ OI ⁺	dissolved	Hypoiodous acidium cation
	OI ⁻	dissolved	Hypoiodite anion
+3	HIO ₂	dissolved	Iodous acid
	IO ₂ ⁻	dissolved	
+4	I ₂ O ₄	solid	diiodinetetroxide
	I ₄ O ₉	solid	Tetraiodine nonaoxide
+5	HIO ₃	dissolved solid	Iodic acid
	IO ₃ ⁻	dissolved	Iodate anion
	IO ₂ ⁺	dissolved	
	I ₂ O ₅	solid	Diiodine pentoxyde
+7	HIO ₄	dissolved solid	Periodic acid
	IO ₄ ⁻	dissolved	Periodate anion
	HIO ₅ ²⁻	dissolved	
	IO ₅ ³⁻	dissolved	

Some basics on oxydo reduction reactions

Initially, an oxydation reaction is the combination of an element with oxygen (oxydant).



A similar result is obtained with other compounds such as F_2 , Cl_2 , S .

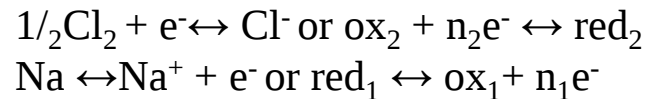


More generally, an oxydation reaction leads to a loss of electrons.

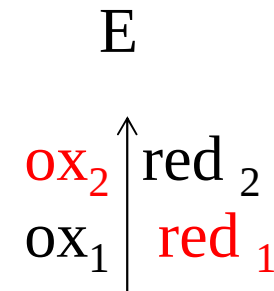
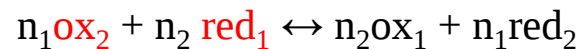
An oxydant is a substance which accepts electrons (O_2 , Cl_2 , ...).

A reductant is a substance which gives electrons (Na , H_2 , C , ...).

The reaction $\text{Na} + 1/2\text{Cl}_2 \rightarrow \text{NaCl}$ can be written:



The final balance is:



Standard Potentials for iodine and its major derivatives at 25°C vs HSE.



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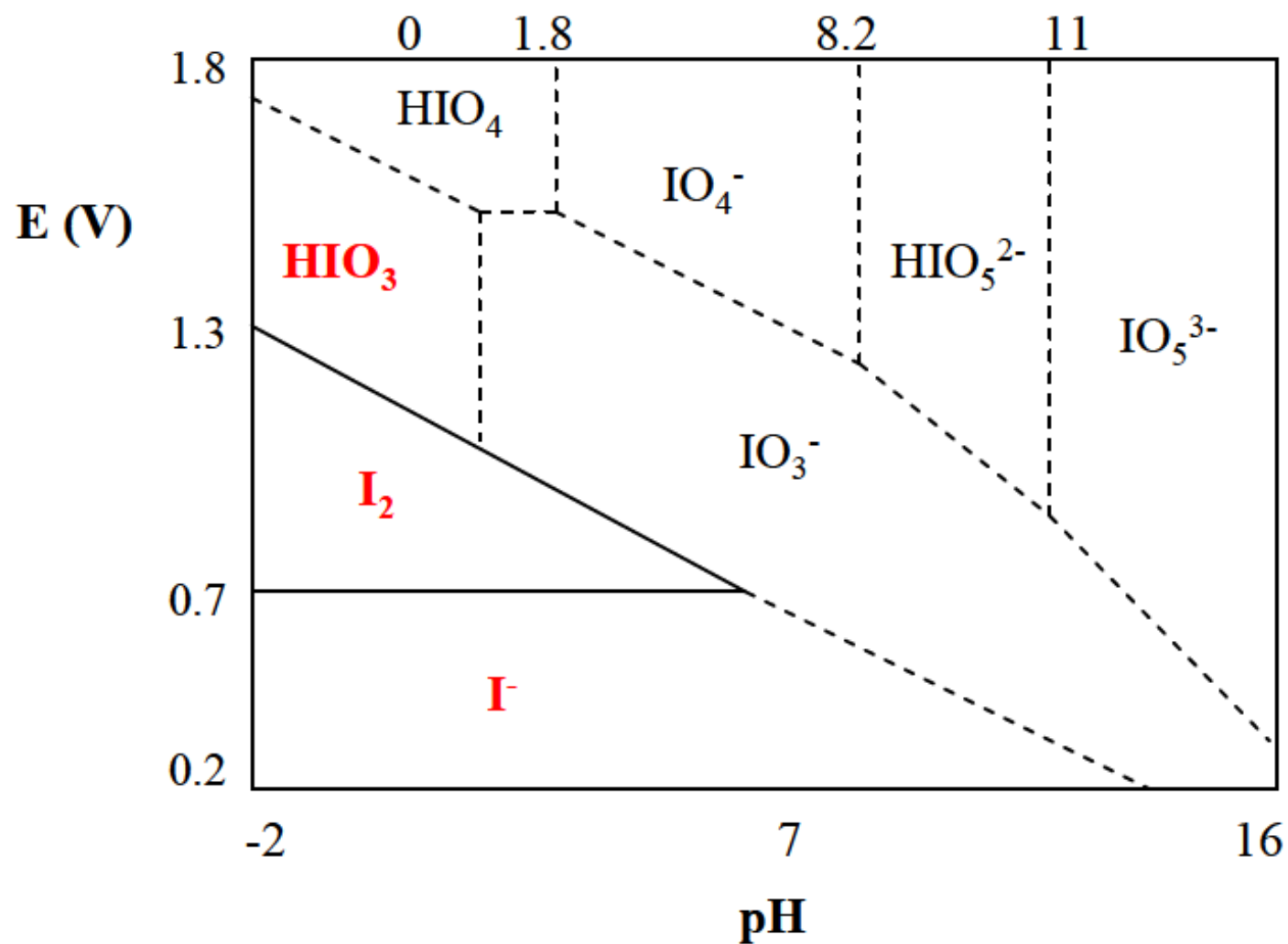
Couple redox	Equation redox	E° (V/ESH)
I_2/I^-	$I_{2(solid)} + 2 e^- \rightarrow 2 I^-$	0,535
	$I_{2(aq)} + 2 e^- \rightarrow 2 I^-$	0,621
I_3^-/I^-	$I_3^- + 2 e^- \rightarrow 3 I^-$	0,536
I_2/I_3^-	$3 I_2 + 2 e^- \rightarrow 2 I_3^-$	0,782
HOI/I^-	$HIO_{(aq)} + H^+ + 2 e^- \rightarrow I^- + H_2O$	0,985
H_2OI^+/I^-	$H_2OI^+ + 2 e^- \rightarrow I^- + H_2O$	0,940
OI^-/I^-	$IO^- + H_2O + 2 e^- \rightarrow I^- + 2 OH^-$	0,472
HOI/I_2	$2 HIO + 2 H^+ + 2 e^- \rightarrow I_2 + 2 H_2O$	1,351
H_2OI^+/I_2	$2 H_2OI^+ + 2 e^- \rightarrow I_2 + 2 H_2O$	1,261
OI^-/I_2	$2 IO^- + 4 H^+ + 2 e^- \rightarrow I_2 + 2 H_2O$	1,354
IO_3^-/I_2	$2 IO_3^- + 12 H^+ + 10 e^- \rightarrow I_{2(aq)} + 6 H_2O$	1,195
	$2 IO_3^- + 12 H^+ + 10 e^- \rightarrow I_{2(solid)} + 6 H_2O$	1,195
IO_3^-/I^-	$IO_3^- + 3 H_2O + 6 e^- \rightarrow I^- + 6 OH^-$	0,257
IO_3^-/OI^-	$IO_3^- + 4 H^+ + 4 e^- \rightarrow IO^- + 2 H_2O$	0,972
IO_3^-/HOI	$IO_3^- + 5 H^+ + 4 e^- \rightarrow HIO + 2 H_2O$	1,134

From C. Cau Dit Coumes, PhD Thesis.

Potentiel pH diagram of iodine



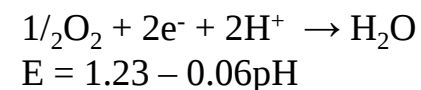
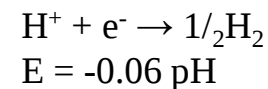
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Couples $\text{H}_2\text{O}/\text{H}_2$ and $\text{O}_2/\text{H}_2\text{O}$



DEN /DANS/DPC/SECR/LRMO

Iodine Species predominance diagram vs pH, in water

Predominant and stable iodine species are: I_2 , I^- , I_3^- , HOI , IO_3^-



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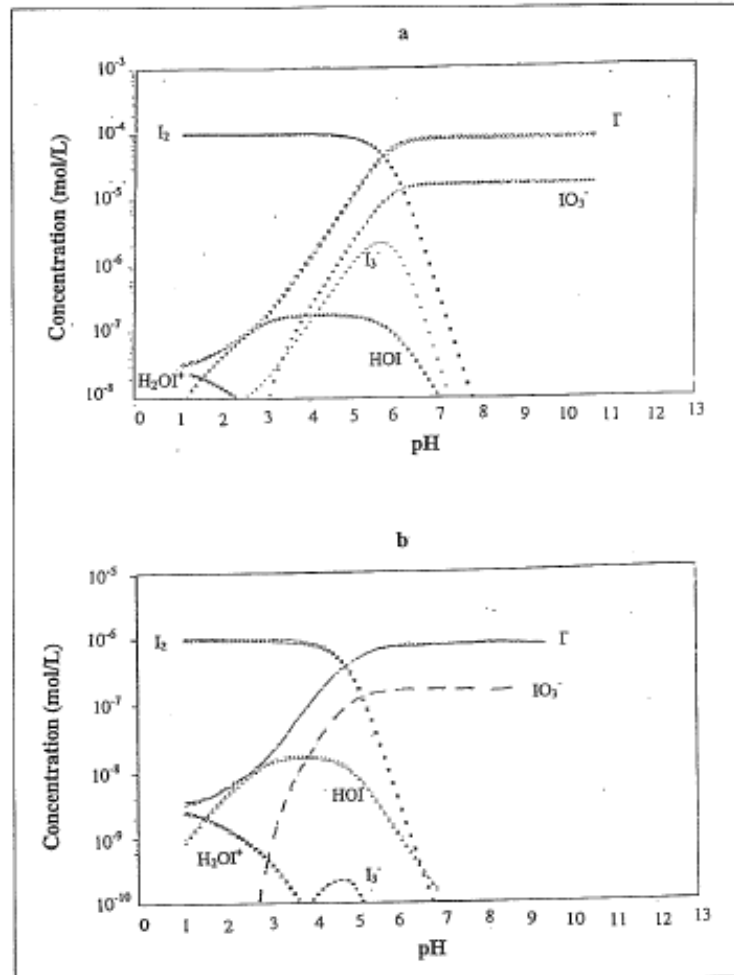


Figure II-5 : Courbes de répartition à 25°C des espèces formées par hydrolyse de l'iode en fonction du pH du milieu.

A : concentration initiale d'iode : 10^{-4} mol.L $^{-1}$

B : concentration initiale d'iode 10^{-5} mol.L $^{-1}$

From C. Cau Dit Coumes, PhD Thesis

Major reactions of iodine and iodates.

**Iodine is only stable in acid slightly oxydant solutions.
When the pH reaches neutrality, iodine hydrolyses.**

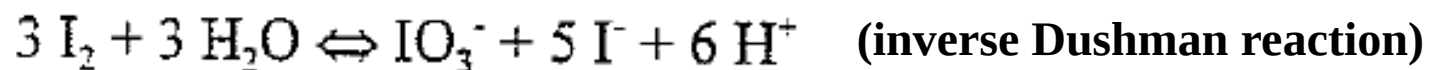
The dismutation (disproportionation, hydrolysis) of iodine in water:



Hypoiodous acid HOI is a non stable thermodynamic amphoter compound:



In alkaline media, (pH>8), iodine dismutates rapidly and totally:



The slow oxydation of I^- by dissolved oxygen:



Analytical techniques for iodine speciation.

Analytical techniques to study iodine species:



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- Classical spectrophotometric techniques
- Precipitation titrations
- Atomic Absorption Spectrometry
- Neutronic Activation
- X ray absorption (XANES, EXAFS) and fluorescence
- ICP mass spectrometry and hyphenated techniques
- Electrochemical and potentiometric techniques
- Scintillation measurements for radiotracers
- Mössbauer spectroscopy (^{129}I)

Recent review: Review of analytical methods for the quantification of iodine in complex matrices, C. P. Shelor, P. K. Dasgupta, *Anal., Chim., Acta*, 702 (2011) 16-36.

Comprehensive Handbook of iodine, Edited by Victor Preedy, Gerard Burrow, Ronald Watson. (2009) .

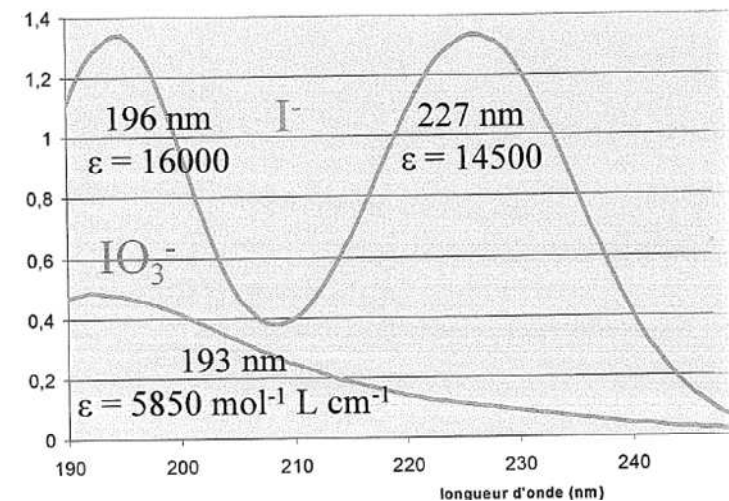
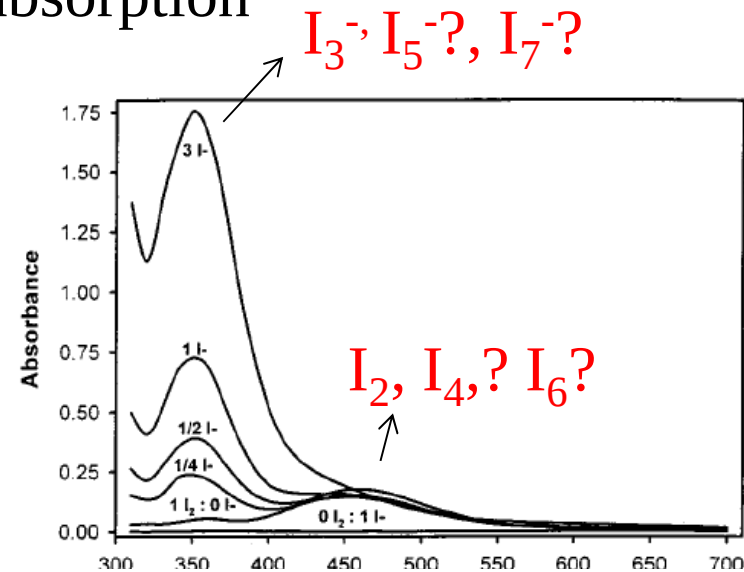
Analytical determination of iodine species in solution.

Optical spectrometry : UV –Visible absorption



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Species	Wavelengths (ϵ , molar extinction coefficient)
I_2	470 nm ($\epsilon=790$) 205 nm ($\epsilon=20000$)
HOI	207,5 nm ($\epsilon= 9680$)
I_3^-	350 nm ($\epsilon= 25700$)
IO_3^-	207,5 nm ($\epsilon=4000$)
I^-	225 nm ($\epsilon= 14400$)



In nitric medium

Analytical determination of iodine species in solution.

Optical spectrometry: Raman diffusion

Table 2. Raman vibrational frequencies observed experimentally for I_3^- , I_5^- and I_2 in various samples

compound	mode designation	sample examined	wavenumber (cm ⁻¹)
I_3^-	v2, bending deformed linear I_3^- [14]	various solutions	70-80
	v1-I stretching (D2'bond) of HI_3^* [8]	HI- I_2 solutions (1:1) in water	84
	v1, symmetric stretching linear I_3^- [14] [23]	various solutions	103-114
	v3, asymmetric stretching linear I_3^- [7]	(dbcr) I_3 in the solid state	125
		(R3S) I_3 solid2	145
		various solutions	143-152
I_5^-	2v2, Fermi Resonance [8]	HI- I_2 solutions (1:1) in water	150
	v I_2 stretching (D1'bond) of HI_3^* [8]	HI- I_2 solutions (1:1) in water	170-172
	v I_2 - I_5 mode in I_5^- (I_3^- disproportion) [7]	(R3S) I_3 liquid	170
	2v1, Fermi Resonance [8]	various solutions	220
I_5^-	v I_3 - I_5 , stretching of I_3^- in L-shaped I_5^- [7]	(dbcr) I_5^1	112
	v I_3 - I_5 , stretching of I_3^- moiety in I_5^- [14]	various samples	104-120
	v I_3 - I_5 , asymmetric stretching of I_3^- in L-shaped I_5^- [7]	(dbcr) I_5^1	143
	v I_5 , bent I_5^- [7]	Polyvinylalcohol thin film	140
	v I_5 , linear I_5^- [7]	iodine doped PVA	155
	v I_5 , linear I_5^- [24]	various solutions	164
	v I_2 - I_5 , stretching of I_2 unit in I_5^-	(R3S) I_5 liquid3	170
		(dbcr) I_5^1	168
		various solutions [16]	160-187
I_2	v I_2 stretching of free I_2 molecule [16]	I_2 in Me2SO	189
		I_2 in CHCl3	207
		I_2 in benzene	209
		pure I_2 solid [25]	180
		pure I_2 molten [21]	194
		pure I_2 vapor [25]	216

1,(dbcr)= dibenzo-18-crown-6; 2,(R3S) I_3 = alkylsulfur triiodide; 3,(R3S) I_5 = alkylsulfur pentaiodide

From Iodine compounds speciation in HI- I_2 aqueous solutions by Raman spectroscopy, Spadoni et al., IJHE, vol37, issue2, (2012), 1326-1334.

Analytical determination of iodine species in solution.

Quantitative Analytical Chemistry:

G. Charlot, D. Bézier, méthodes modernes d'analyse quantitative minérale, Masson éditeurs.

P. Jaulmes, précis de chimie analytique, tome 1, 1965.



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Titration of iodine by volumetric methods: with sodium thiosulfate (with starch, formation of a blue compound) :

1ml of thiosulfate 0.1N is equivalent to 12.69 mg of iodine.

Titration of iodide anions by volumetric methods using silver.

Precipitation of silver salts.

- **Volhard method,**
- **Mohr method,**
- **Fajans method.**

Iodide anions titration by potentiometry:

Titration using a I^- selective electrode.

Determination of total iodine concentration by ICP-MS.



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The use of a plasma torch coupled with a magnetic mass spectrometer (ICP-MS) or optical emission spectrometry (ICP-OES) enables the measurement of iodine traces.

- Importance of the sample preparation.
- The choice of an alkaline medium increases the sensitivity.
- Iodine is difficult to ionize, use of ascorbic acid, reducing agent to study iodide anions (pH Thesis B. Langlois, DEN, 2001).
- Caution to memory effect in the nebulizer

- **Review article:**

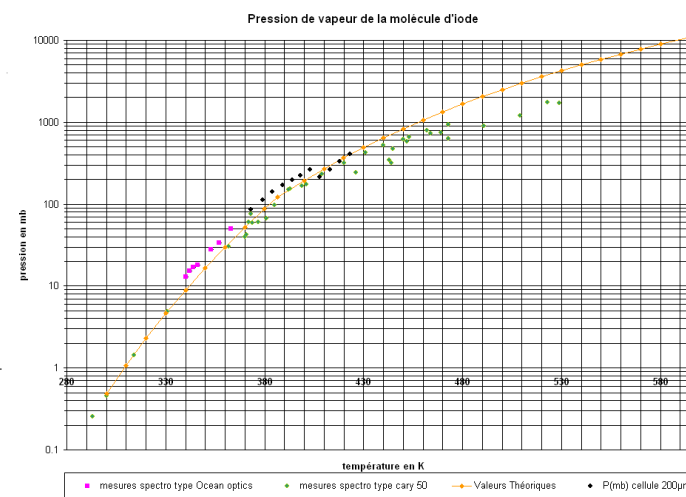
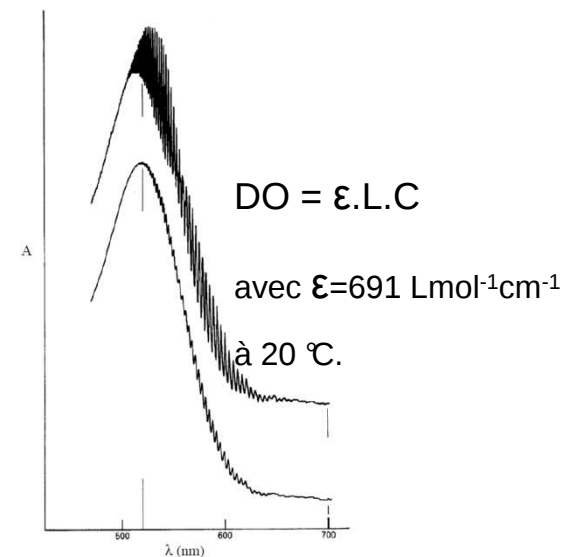
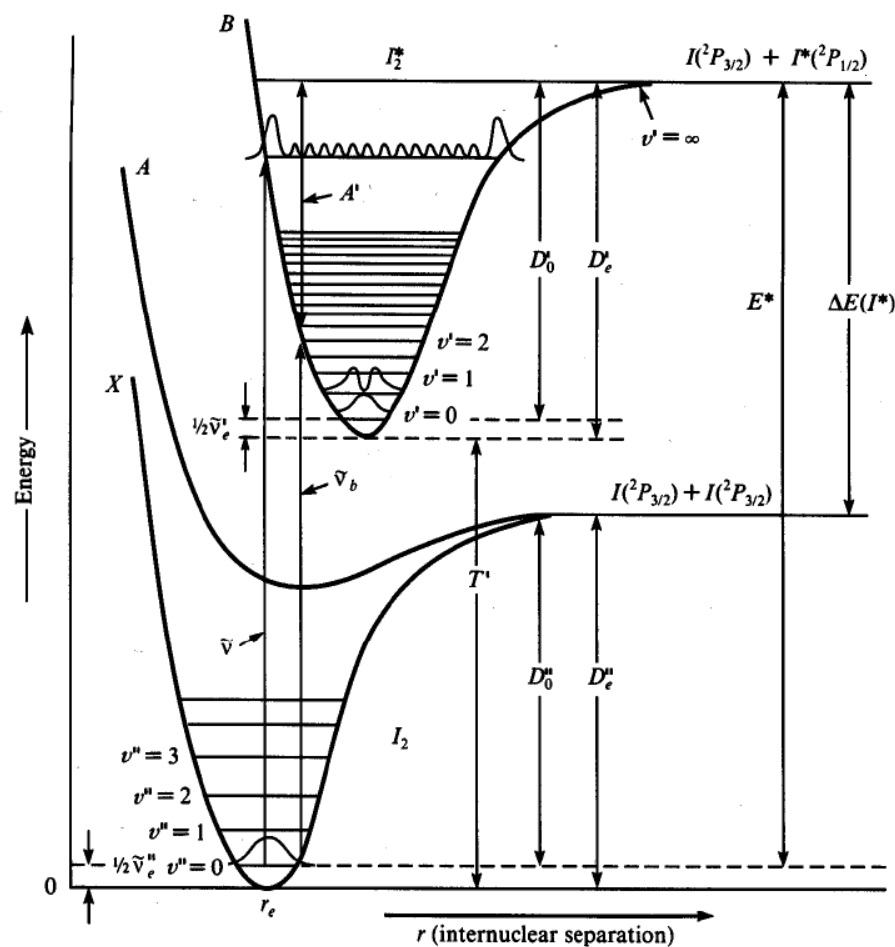
Iodine determination by ICP spectrometry, A.A Oliveira, L. C. Trevizan, J. A. Nobrega, Appl. Spect. Rev., 45:6, (2010), 447-473.

Iodine speciation in the gaseous phase

Violet colored vapor easily detectable in optical absorption spectrometry: UV Visible for example.



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Iodine species in the iodine sulfur thermochemical cycle

Liquid vapour equilibrium measurements are made using optical « online » techniques, to avoid tedious experiments and prevent any vapour composition changes.

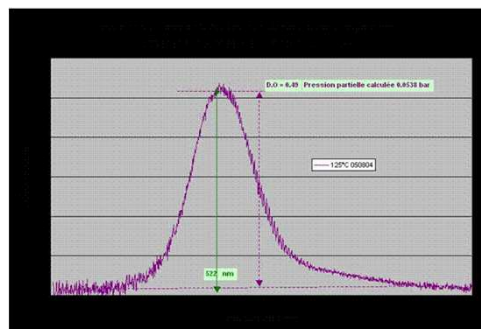
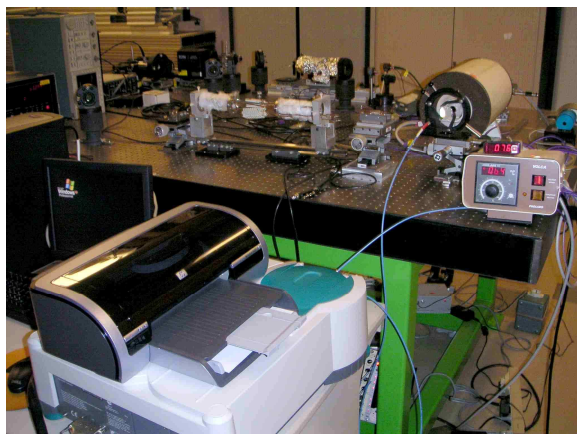


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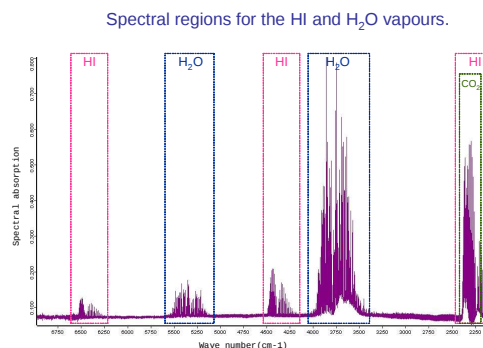
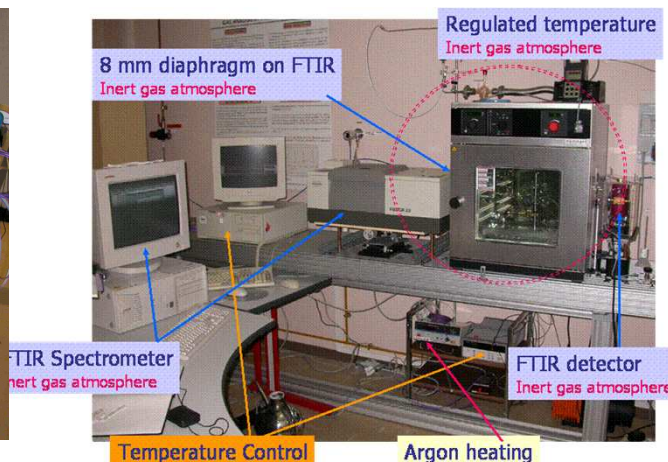
➤ HI (and H₂O): FTIR spectrometry

➤ I₂: UV-Visible spectrometry

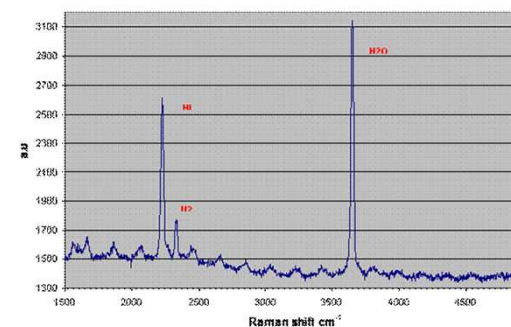
➤ HI, (H₂O and H₂): Evaluation of Raman techniques.



UV/Visible absorption



FTIR spectrometry



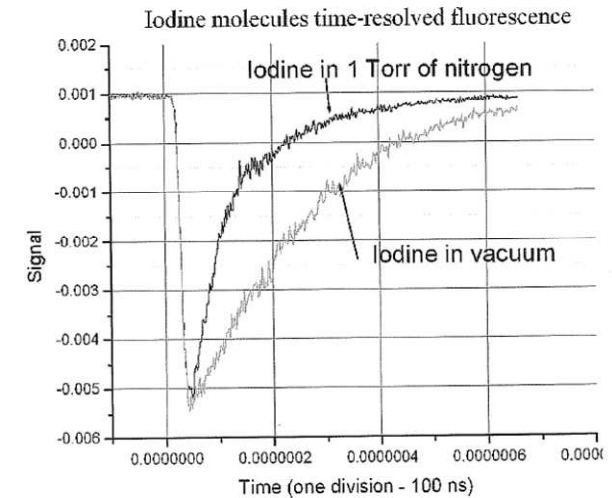
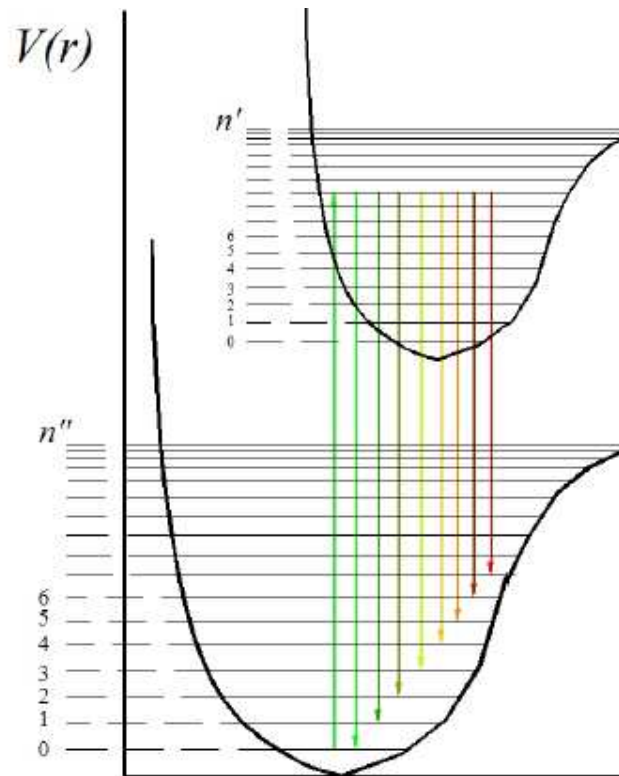
Raman techniques

Iodine speciation in the gaseous phase

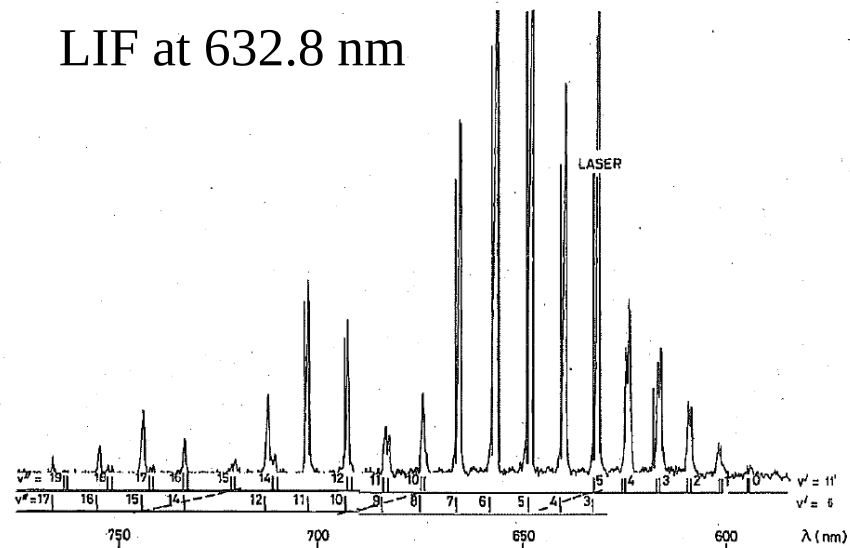
Violet colored vapor detectable in emission: laser induced fluorescence, spontaneous Raman diffusion.
But low concentration only, otherwise fluorescence quenching.



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LIF at 632.8 nm



Gaseous iodine detection by colorimetry

Colorimetric method developped by Iwasaki (1952):

Reduction of iodine in iodide anions after bubbling iodine gas in a 1N NaOH solution.



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A New Microdetermination of Iodide by its Catalytic Reaction

By Iwaji IWASAKI, Satori UTSUMI
and Takejiro OZAWA

(Received August 26, 1952)

The authors found that the orange color of ferric thiocyanate which was formed by dilute potassium thiocyanate and an excess of ferric salt in dilute nitric acid solution, faded very slowly owing to the oxidation of thiocyanate, but rapidly in the presence of minute amounts of iodide.

The catalytic action of iodide on this reaction was studied colorimetrically, and a new microdetermination of iodide was accomplished.

Iodine also catalyzed the fading of ferric thiocyanate in exactly the same way as iodide, but chloride and bromide did not. In preliminary experiments it was found that this catalytic reaction was greatly affected by the presence of small amounts of nitrite. The reaction rate depended not only on the concentrations of iodide present, but also on the concentrations of thiocyanate and nitric acid, and especially on the temperature (Fig. 1).

Therefore these factors must be kept constant for iodide determination.

The determination of iodide could be made by measuring the absorbancy in a definite period of time and using the calibration curves (Fig. 1) previously obtained under the same conditions.

Procedure and Results:

To 10 cc. of the sample solution, 1.0 cc. of mixed reagent (3×10^{-3} or 10^{-3} M in potassium thiocyanate, 3×10^{-4} M in sodium nitrite) and 2.0 cc. of ferric alum reagent (prepared by dissolving 0 g. of ferric alum in 100 cc. of 5.7N nitric acid) were added. The absorbancies were plotted against

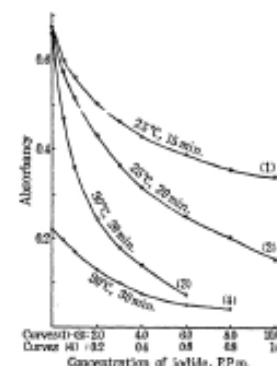


Fig. 1.—Calibration curves of iodide.
Curves (1)–(3): 3×10^{-3} M SCN⁻
Curve (4): 10^{-3} M SCN⁻

the concentrations of iodide as shown in Fig. 1.

The absorbancy was determined by a Beckman Model DU spectrophotometer at 400 m μ using 10 mm. cell. The calibration curves shown in Fig. 1 were reproducible within $\pm 5\%$.

The determination of iodide in concentrations of 0.5–10 p.p.m. could be made under the following conditions: 3×10^{-3} M SCN⁻, 25°C., 20 minutes (Curve 2). The determination of iodide in concentrations of 0.05–0.8 p.p.m. could be made under the following conditions: 10^{-3} M SCN⁻, 30°C., 30 minutes (Curve 4).

It was also possible to determine 0.001–0.05 p.p.m. of iodide under the following conditions: 10^{-3} M SCN⁻, 45°C., 30 minutes.

By this method 0.001–10 p.p.m. of iodide could be determined in the presence of chloride and bromide.

Laboratory of Analytical Chemistry and
Geochemistry, Tokyo Institute of
Technology, Tokyo

(1) I. Iwasaki, S. Utsumi and T. Ozawa, This Bulletin,
25, 224 (1952).

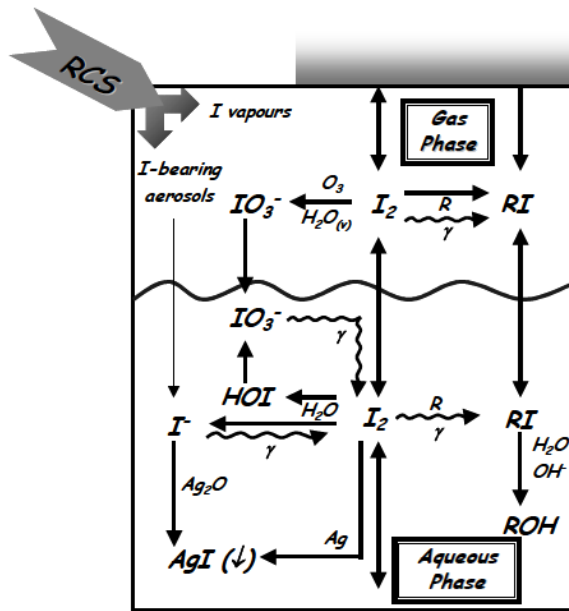
Special attention to organic iodides

Ex : Methyl iodide is very volatile: CH_3I is formed by the interactions between iodine and organic compounds such as oils, paints,...: $T_b = 42.4^\circ\text{C}$.

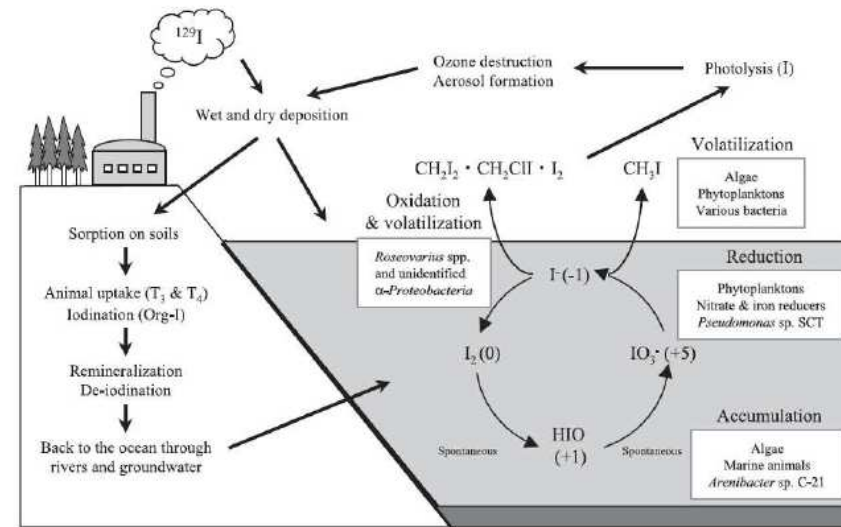


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- Species observed in the gaseous flux released at the dissolution of irradiated fuels,
- Species observed in the measurement of the efficiency of iodine traps,
- Species observed in the containment of a PWR in case of severe accidents,
- Species also observed in marine chemistry.



Simplified diagram of iodine transformations within the containment



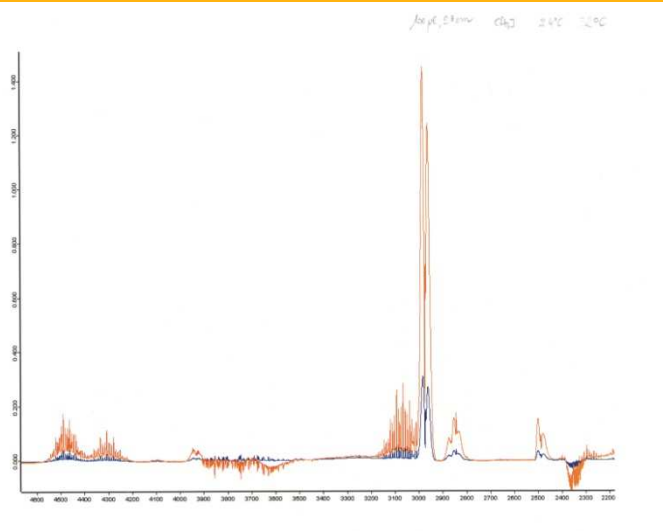
Amachi, S., Microbes Environ. Vol 23, n°4, 269-276 (2008)

Analytical techniques for organic iodine species determination

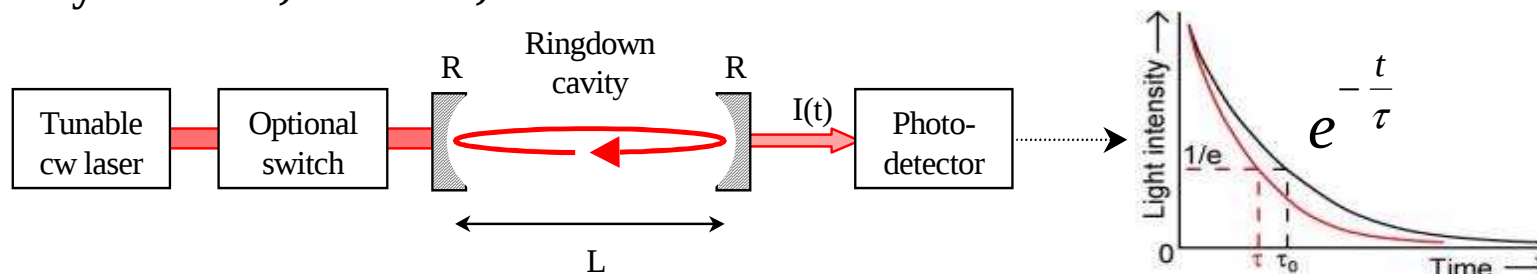


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**FTIR: mid IR range,
LD < ppm.**



**CRDS (Cavity Ring Down Spectroscopy): Optical absorption technique
very selective, sensitive, in the NIR.**



Optical cavity: $L = 1\text{m}$; $L_{\text{optique}} = 10\text{ km}$
Theoretical DL $\sim 10\text{ ppb}$.

$$\alpha(\nu) = \frac{1}{c \tau(\nu)}$$

A. Pailloux, DPC/SECR/LRMO

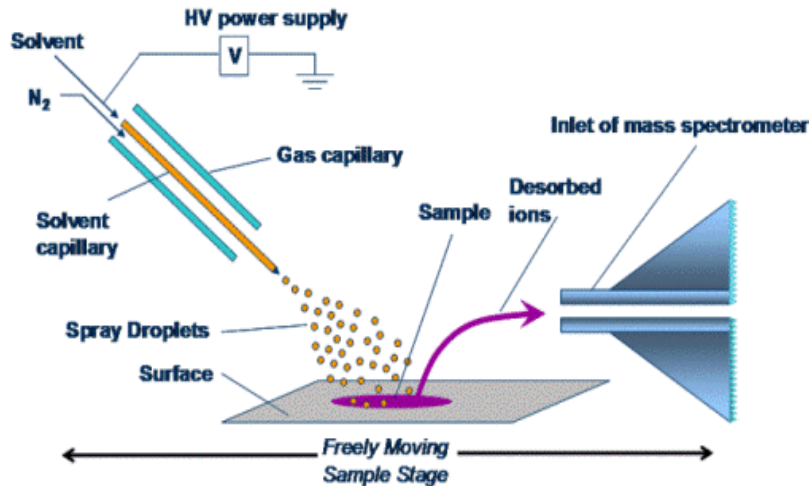
Speciation of solid iodine and imaging.

Evaluation of DESI-TOF technique.

(Desorption – ionisation by electrospray coupled with a time of flight mass spectrometer).



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Principle of DESI technique:

Application of an intense electric potential on capillaries containing the solvent and the vector gas.

- solvent nebulizing
- the droplets desorb the surface molecules

Advantages:

- Direct analyses on the sample without any preparation
- Soft ionization process which produces a lot of molecules and enables speciation studies.
- 2D imaging is possible.

D. Lebeau, DPC/SECR/LRMO

Examples of DESI-TOF potential for solid iodine speciation

Application to waste storage

- Example: 4 mg d'EDTA on 10 g of concrete (400 ppm)

LCT XE Premier (Waters)

Source DESI 2D (Prosolia)

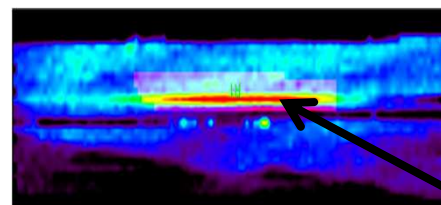


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Image optique



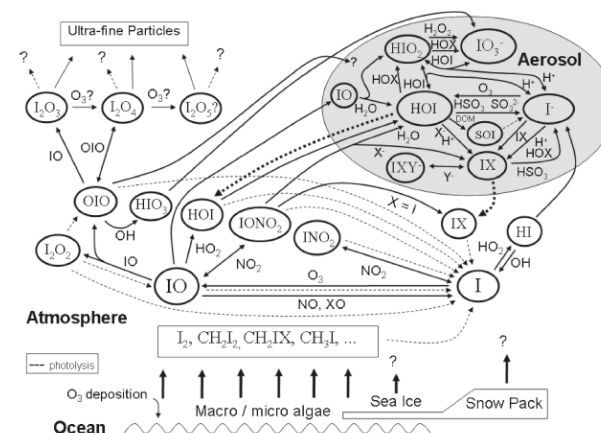
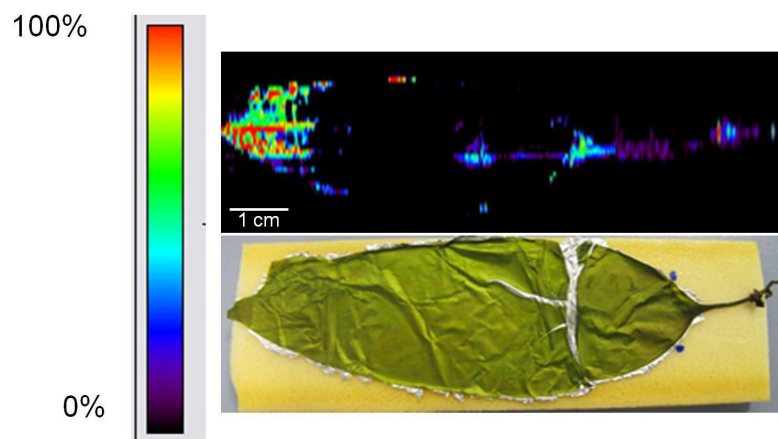
Image ionique
Ion m/z 344



Localisation spécifique de l'ion [EDTA/Fe^{III}]

Application to marine chemistry

- Example: speciation and localization of iodine in *Laminaria digitata*



Examples of DESI-TOF potential for solid iodine speciation

Evaluation of the efficiency of iodine traps:



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Filters with activated charcoal (high specific surface) + KI + TEDA

Isotopic exchange:



In the case of severe accidents, comparative efficiency for I₂ and CH₃I

- **Influence of moisture**
- **Influence of temperature**
- **Influence of NO_x**

Conclusions



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Iodine is a very important element in our life. It appears under numerous and various chemical species which offers an extensive chemistry according to the potential and the pH conditions.

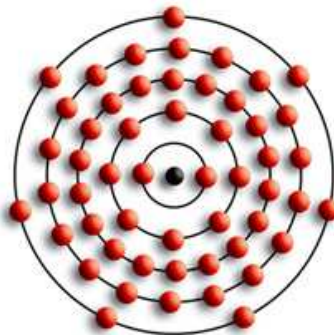
In the nuclear domain, it is a very important fission product especially in the gaseous phase, through the radioactivity of its isotopes. Its detection at low level and its speciation are still the subject of numerous studies.

The continuous evolution of analytical tools enables speciation studies at traces level.

The knowledge of iodine speciation is not well known, particularly in the case of complex matrices.

Acknowledgments and additional bibliography

Acknowledgements to all contributors to iodine speciation knowledges.



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