Coláiste na Tríonóide, Baile Átha Cliath
Trinity College Dublin
Ollscoil Átha Cliath | The University of Dublin

Faculty of Engineering, Mathematics and Science

School of Chemistry

Moderatorship Examination   **Trinity Term 2017**

Nanoscience, Physics & Chemistry of Advanced Materials

Senior Sophister

SS NPCAM

(Comprehension and Problem Solving Paper)

Paper Coordinator: Prof Gun'ko

Friday 5ᵗʰ May   **SNIAM PHYS**   **09.30 – 12.30**

The Chemistry Examination Board

Complete the comprehension exercise in **Section A** and **answer 6** questions from **Section B**

Section A accounts for 40% and **Section B** accounts for 60%

Please use separate answer books for each question

Tables of physical constants and relative atomic masses and a periodic table are attached.

Spectroscopic tables, character tables, Mathematical Tables (Old) or Formulae and Tables (New) and graph paper are available from the invigilators, if required.

Non-programmable calculators are permitted for this examination – please indicate the make and model of your calculator on each answer book used.

Section A

Read the short paper from [*NATURE MATERIALS, 2016, 824 - 825*] on the next two pages and then perform the following exercises (a)-(d) below.

(a)   Prepare a brief summary of the article (1 page max)

[10 marks]

(b)   Explain the main chemical and physical principles behind the plasmon-driven synthesis of Au nanoprisms and write equations of the relevant chemical reactions.

[10 marks]

(c)   Explain the role of PVP layers on the Au nanoparticles in growing of Au nanoprisms. Draw the (111) plane for a face-centered cubic (FCC) lattice. Determines how many distinct sets of (111) planes (not parallel to each other) are present in the FCC gold lattice.

[10 marks]

(d)   Calculate the ratio between growth rates in light and dark conditions for a spherical gold nanoparticle with the initial diameter of 7 nm, if after 2 hours under light its diameter was 105 nm, while after 2 hours in the darkness the corresponding nanoparticle diameter was only 14 nm.

[10 marks]

PLASMON-DRIVEN CHEMICAL SYNTHESIS

Growing gold nanoprisms with light

An adsorbed polymer directs the photochemical growth of colloidal Au single-crystal nanoprisms following visible metal excitation.

Louis Brus

Noble metal Au nanocrystals are industrial catalysts and are also widely employed in medicinal assays and therapeutics, for example in spherical DNA diagnostic nanostructures[1]. In medicine, the intrinsic chemical stability of Au is an asset, yet this inertness makes it difficult to synthesize single-crystal nanostructures of designed size and shape. Photochemical synthetic processes often show exquisite specificity and control as compared with thermal reactions. However, metals do not have the type of molecular or semiconductor excited states that lead to normal photochemistry. Nevertheless, writing in *Nature Materials*, David Wei and co-workers[2] now show that Au nanocrystals can be grown with controlled shape by optical excitation

of the metal itself. An organic polymer adsorbed on specific surface facets directs photo-excited electrons as they reduce aqueous $AuCl_4^-$ ions to add metal to the growing nanocrystal.

It is rather amazing that visible-light excitation of a metal can cause any sort of photochemistry. Optical excitation creates excited ('hot') electrons and holes that decay back to the ground electronic state within a few femtoseconds, heating the nanocrystal. This ultrashort lifetime contrasts with the much longer lifetimes resulting from excitation of molecules (or semiconductors) having significant highest occupied molecular oribital (HOMO)–lowest unoccupied molecular orbital (LUMO) bandgaps. Typically, the larger the gap, the slower the excited-state

relaxation time — and thus the greater the possibility of chemical reaction.

Metals have no bandgap, and so relaxation is ultrafast. Nevertheless, a photochemical growth process was discovered by accident more than a decade ago[3,4]. An aqueous colloid of small Ag nanocrystal seeds was left under standard indoor lighting for some weeks. Afterwards, many small seeds had converted to a few larger nanocrystals. This is a type of light-induced ripening, reducing the overall metal surface energy by creating nanoparticles with a larger size. The nanoparticles were stabilized by a dense layer of adsorbed citrate anions. It was suggested that an electron from citrate tunnelled into the hot hole of the optically excited Ag metal, creating a chemically unstable oxidized citrate that immediately decomposed to liberate CO_2 (refs 5,6). The extra electron inside Ag (from filling the hot hole) was thus trapped and effectively charged the nanocrystal, creating a steady-state photovoltage under weak continuous-wave irradiation. Nanocrystals with larger photovoltage preferentially reduced aqueous Ag^+, leading to growth.

However, all efforts to extend these ideas to the far more important Au aqueous plasmonic nanocrystal system failed. Now, in a remarkably detailed study at the single-nanocrystal level, Wei and colleagues have discovered that plasmon-driven growth of Au nanoprisms can be obtained by mixing polyvinylpyrrolidone (PVP) in an aqueous solution containing Au nanocrystal seeds and tetrachloroauric acid ($HAuCl_4$). They observed that the positively charged polymer PVP preferentially adsorbs on twin plane defects along the perimeter
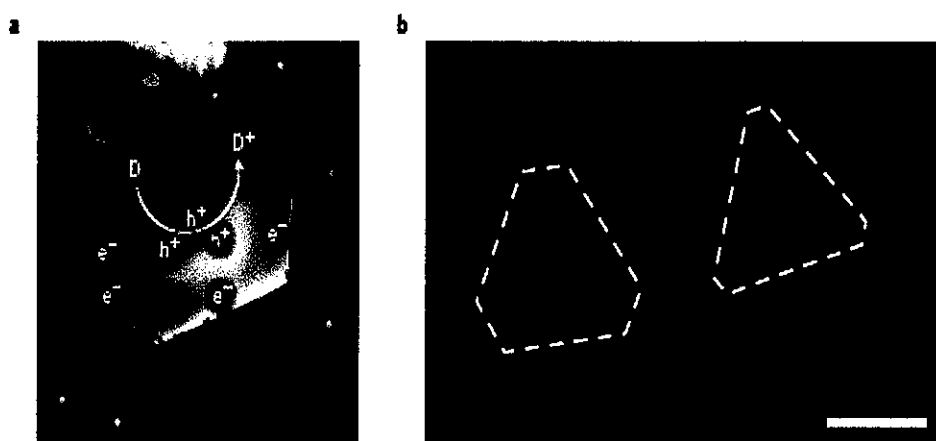

Figure 1 | Plasmon-driven synthesis of Au nanoprisms. a, A sketch of the photochemical growth of a Au nanoprism. Hot electrons (e^-) generated in the Au nanoprism (yellow) by visible-light irradiation participate in the reduction of Au precursors (red arrow) with the assistance of PVP polymer adsorbed along the perimeter of the nanoprism, whereas hot holes (h^+) are scavenged by the donor (D) methanol (white arrow). **b,** A scanning electron microscopy image of Au nanoprisms after irradiation. The white dashed lines represent the initial dimensions of the nanoprisms. Scale bar, 100 nm. Part **a** courtesy of Brendan Sweeny, Yueming Zhai, Joseph DuChene, Jingjing Qiu and Wei David Wei, and part **b** adapted from ref. 2, Nature Publishing Group.

of the Au nanocrystals, whereas it was previously thought that PVP would form blocking layers on the top and bottom (111) facets. This system was irradiated in the Au visible plasmon bands, and added methanol served as a sacrificial donor, reacting with Au hot holes to form formaldehyde. During colloidal growth, positive PVP appears to both concentrate aqueous $AuCl_4^-$ from solution along the prism perimeter, and to attract the extra electrons from methanol oxidation. The reaction between $AuCl_4^-$ and the extra electrons leads to reduction of the Au precursors and growth of the nanoprisms (Fig. 1). The microscopic details of how exactly the polymer facilitates $AuCl_4^-$ reduction are not yet clear, however, but will surely be the subject of much future research.

Several factors combine to make hot carrier surface reactions — such as those observed by Wei and colleagues — significant in nanocrystals. The plasmon phenomenon creates an especially large optical cross-section for nanocrystal light adsorption, increasing the rate of photogenerated charge carriers with respect to bulk metal. Also, due to the small size of the metal nanoparticles, hot carriers are necessarily created within a few nanometres of the surface; as a result, despite their femtosecond lifetime, they reach the surface before losing energy. The adsorbed species involved in the photochemical reaction has two key properties: first, it must have an affinity level at the energy of the hot carrier; second, carrier capture must trigger a femtosecond irreversible process, able to extract energy before the carrier returns into the metal.

Several examples of chemical reactions initiated by electrons excited in metal nanocrystals have been reported[7,8]. Visible excitation of Ag nanocrystals photocatalyses selective oxidation of organics by molecular O_2, including the industrially important conversion of ethylene to ethylene oxide[9,10]. A hot electron is captured by adsorbed O_2 to make adsorbed superoxide O_2^-, which evolves for a few femtoseconds towards its longer natural bond length. The electron then jumps back into the metal, leaving a vibrationally excited 'activated' neutral O_2 that reacts faster. Similarly, adsorbed H_2 (and deuterated D_2) can be activated in this way on Au nanocrystals, increasing the rate of the exchange reaction between H_2 and D_2 (ref. 11). Another type of irreversible process occurs when a nanocrystal is adsorbed on a transparent semiconductor such as TiO_2. Optically excited hot electrons are injected into the semiconductor conduction band; their return is prevented by the strong interfacial electric field of a Schottky junction. Such systems are being explored for use in photochemical water splitting[12] and photovoltaic devices[13].

The demonstration that the addition of polymers such as PVP enables unprecedented Au hot carrier photochemical growth suggests new possibilities. With controlled growth, Au nanocrystals with very specific sizes and shapes — and therefore a tailored plasmon absorption spectrum — can potentially be created to optimize medical applications. In fact, the local heating created by Au excitation is already being explored for cancer photodynamic therapy; in the future, specific hot carrier reactions beneficial for therapeutics may also be found. Hot carrier reactions might also be used to controllably modify the biological surface ligands that determine function in medical applications.   ◻

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References
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Section B

Question 1
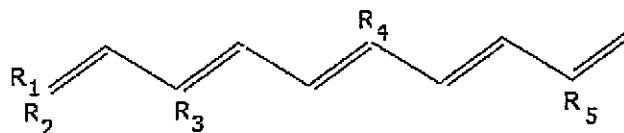
This diagram shows the structure of a conjugated polyene dye. (The side groups R do not form part of the conjugation and merely ensure the molecule's solubility.) The π electrons are treated by 'particle on a line' theory and are capable of exploring a one-dimensional space of length L. The resulting quantized energy levels are given by the theory as

$$E_n = n^2 \frac{h^2}{8mL^2} \qquad (n = 1, 2, 3, \ldots)$$

where m is the mass of the electron. Given that each C—C bond length is 1.4×10^{-10} m and that there is one π electron from each of the 10 carbon atoms of the main chain calculate the wavelength of the molecule's lowest-energy electronic transition.

[10 marks]

Question 2

(a) A two-dimensional crystal has a rectangular lattice with lattice constants a and b ($a > b$). Sketch the first Brillouin zone for the crystal, indicating the values of k_x and k_y at the Brillouin Zone boundaries.

[4 marks]

(b) Assuming that the two-dimensional crystal is monovalent, and that the potential due to the ions can be neglected (i.e. free electron model), use the fact that the Fermi energy in a 2D system is $E_F = n\pi\hbar^2/m$ to derive an expression for the 2D Fermi wavevector. Hence show that the Fermi surface will extend beyond the first Brillouin zone if $a > \pi b/2$. Note that n is the number of electrons **per unit area.**

[6 marks]

Question 3

Carbon dioxide can react with water according to the equation below.

$$CO_2(g) + 2H_2O\ (l) \rightleftharpoons HCO_3^-(aq) + H_3O^+(aq)$$
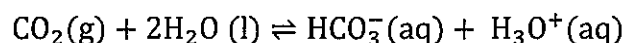
The equilibrium constant for the above reaction is $K_{eq} = 4.5 \times 10^{-7}$ at 15 °C.

Assuming that atmospheric pressure varies with altitude according to the expression below,

$$P = (-1.0 \times 10^{-4})A + 1$$

where P is atmospheric pressure in units of atm and A is altitude in meters, and further assuming that the temperature (15 °C) and composition (21% O_2, 78% N_2, 0.96% other and 0.04% CO_2) of air remain constant for the range of altitudes considered here, answer the following questions.

(a)   Do you expect the pH of water to be higher at higher altitude or lower altitude? Explain your answer.

[3 marks]

(b)   What is the pH of unpolluted rain water in equilibrium with clean air

      (i)   at an altitude of 5000 meters above sea level?

      (ii)   at sea level?

Henry's gas constant for CO_2 in water is 0.033 mol L^{-1} atm^{-1}.

[4 marks]

(c)   If aquatic life needs a minimum of 2×10^{-4} mol/L dissolved O_2 to live and Henry's gas constant for O_2 in water is 1.28×10^{-3} mol L^{-1} atm^{-1} at 15 °C, what is the maximum altitude at which aquatic life can survive?

[3 marks]

Question 4

An electron is moving freely inside a one-dimensional infinite potential box with walls at $x = 0$ and $x = a$. If the electron is initially in the ground state ($n = 1$) of the box and if we *suddenly* quadruple the size of the box (i.e., the right-hand side wall is moved instantaneously from $x = a$ to $x = 4a$), calculate the probability of finding the electron in:

    (a)   the ground state of the new box and

    (b)   the first excited state of the new box.

Use sketches if necessary and where appropriate.

[10 marks]

Question 5

When heated above 120 °C barium titanate, $BaTiO_3$, undergoes a phase transition from tetragonal to the cubic perovskite structure, space group $Pm\bar{3}m$, with a lattice parameter, a, of 4.012 Å.
Sketch the perovskite structure.

Calculate the d-spacings of the first four reflections of the powder diffraction pattern.

Strontium titanate, $SrTiO_3$, also adopts the cubic perovskite structure. Would you expect its d-spacings to be larger or smaller than those of $BaTiO_3$? Why?

[10 marks]

Question 6

The period d of a transmission diffraction grating is 100 nm with each slit (actually a slot) being of width a =75nm, in a 10nm thick foil. A beam of phthalocyanine molecules (diameter 1.5 nm and of molar mass 514.5) impinges one at a time at normal incidence onto the grating. Over time a diffraction pattern builds up on a screen or detector 500 mm after the grating where the 1st order matter diffraction peak is observed to be at a distance of 30μm from the central 0th order matter peak. How fast are the phthalocyanine molecules moving?

[10 marks]

Question 7

(i)   Calculate the change in entropy of 1.0 L of water at 0 °C when it absorbs 235 J of energy from a heater.

(ii)   If the 1.0 L of water is at 99 °C, what is its entropy change?

(iii)   Explain any difference in entropy change.

[10 marks]

Question 8

It is possible to levitate a frog in a strong magnetic field with a vertical field gradient. Beginning with the potential energy, U, of a magnetic moment, μ, in a magnetic field, **B**,

$$U = -\,\mu \cdot \mathbf{B}$$

explain how a frog can be levitated in this way. The diamagnetic susceptibility of water is -10^{-5} and its mass density is 10^3 kg m^{-3}.

Estimate the minimum value of $B_z \frac{dB_z}{dz}$ needed to levitate the frog.

[10 marks]

Question 9

The selective hydrogenation of alkynes is a very important reaction in the purification of rich olefin streams in oil refineries and large-scale production of commodity chemicals. Using the enthalpy of combustion and heat capacity data at 298K shown below, calculate:

(a)   Enthalpy ($\Delta_r H^0$) and internal energy ($\Delta_r U^0$) of reaction at 298K for the hydrogenation of acetylene (C_2H_2) to ethylene (C_2H_4).

(b) $\Delta_r H^0$ for the same reaction at 427K. Assume all heat capacities to be constant over the temperature range involved.

$\Delta_c H^0$ (H_2) = -285.83 kJ mol^{-1} ; $\Delta_c H^0$ (C_2H_2) = -1300 kJ mol^{-1} ; $\Delta_c H^0$ (C_2H_4) = -1411 kJ mol^{-1}. $C^0_{p,m}$ (H_2) = 28.82 kJ K^{-1} mol^{-1} ; $C^0_{p,m}$ (C_2H_2) = 43.93 kJ K^{-1} mol^{-1} ; $C^0_{p,m}$ (C_2H_4) = 43.56 kJ K^{-1} mol^{-1}.
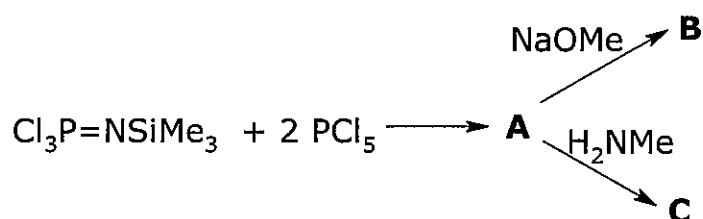
[10 marks]

Question 10

After sunset the air temperature above a pond drops to -10 °C. A pond, which is initially at 4 °C, begins to freeze at the surface. Estimate the thickness of the ice after 10 hours. The latent heat of melting of ice is 333 kJ kg^{-1} K^{-1}, thermal conductivity is 2.3 W $m^{-1}K^{-1}$ and density is 920 kg m^{-3}. Neglect the heat released by the liquid water as it cools from 4 °C to 0 °C.

[10 marks]

Question 11

Predict the polymeric products **A**, **B** and **C** in the reaction scheme below. Write down the mechanism of the formation for product **A**.

$$Cl_3P=NSiMe_3 \ + \ 2 \ PCl_5 \longrightarrow A \begin{matrix} \nearrow \text{NaOMe} \rightarrow \textbf{B} \\ \rightarrow H_2NMe \\ \searrow \textbf{C} \end{matrix}$$

[10 marks]

Question 12

A superconducting solenoid is immersed in a bath of liquid helium at 4K. It carries a current which generates a field of 10 Tesla in a bore of diameter 0.1 m and length 0.1 m. It suddenly transits to the normal state, and a large amount of liquid helium boils off. What volume of liquid helium boils off?

For liquid helium:

Specific latent heat of vapourisation = 21 kJ kg^{-1} and density = 125 kg m^{-3}

[10 marks]

School of Chemistry

Use the following values for fundamental constants, conversion factors, relative atomic masses and other data for examination purposes:

Constants

Gas Constant	$R = 8.314$ J K^{-1} mol^{-1}
Boltzmann's Constant	$k_B = 1.381 \times 10^{-23}$ J K^{-1}
Planck's Constant	$h = 6.626 \times 10^{-34}$ J s
	$\hbar = h/2\pi = 1.055 \times 10^{-34}$ J s
Velocity of light	$c = 2.998 \times 10^{8}$ m s^{-1}
Charge of electron	$e = -1.602 \times 10^{-19}$ C
Rest mass of electron	$m_e = 9.109 \times 10^{-31}$ kg
Rest mass of proton	$m_p = 1.673 \times 10^{-27}$ kg
Atomic Mass Unit	$1\ u = 1.66054 \times 10^{-27}$ kg
Vacuum permittivity	$\varepsilon_0 = 8.854 \times 10^{-12}$ J^{-1} C^2 m^{-1}
Vacuum permeability	$\mu_0 = 4\pi \times 10^{-7}$ J s^2 C^{-2} m^{-1}
Bohr magneton	$\beta = 9.274 \times 10^{-24}$ J T^{-1}
Nuclear magneton	$\beta_n = 5.051 \times 10^{-27}$ J T^{-1}
Rydberg constant	$R_\infty = 2.178 \times 10^{-18}$ J $= 1.097 \times 10^{5}$ cm^{-1}
Avogadro's constant	$N_A = 6.022 \times 10^{23}$ mol^{-1}
Faraday constant	$F = N_A e = 9.648 \times 10^{4}$ C mol^{-1}
Bohr radius	$a_0 = 5.292 \times 10^{-11}$ m
Molar volume at S.T.P.	$M_v = 22.414$ dm^3 mol^{-1}
Proton magnetic moment	$\mu_p \qquad\qquad 2.79\ \mu_N$

Conversion factors etc.

$1\ eV = 1.602 \times 10^{-19}$ J $= 9.648 \times 10^{4}$ J mol^{-1}

$1\ cm^{-1} = 1.986 \times 10^{-23}$ J $= 11.96$ J mol$^{-1} = 1.240 \times 10^{-4}$ eV

$1\ \text{Å} = 0.1$ nm $= 10^{-10}$ m

1 standard atmosphere $= 1.013 \times 10^{5}$ N m^{-2} (Pa) $= 760$ torr $= 1013$ mbar

$1\ L = 1$ dm$^3 = 10^{-3}$ m^3

1 Debye (D) $= 3.336 \times 10^{-30}$ C m

1 calorie $= 4.184$ J

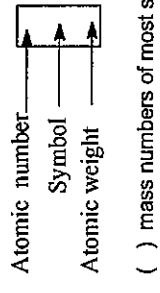$R = 8.314$ J K^{-1} mol$^{-1} = 8.206 \times 10^{-2}$ L atm K^{-1} mol^{-1}

Approximate values of relative atomic masses

(Values in brackets are mass numbers of longest lived isotopes).

Ac	(227)		Ge	72.6		Po	(209)
Ag	107.9		H	1.0		Pr	140.9
Al	27.0		He	4.0		Pt	195.1
Am	(243)		Hf	178.5		Pu	(244)
Ar	40.0		Hg	200.6		Ra	(226)
As	74.9		Ho	164.9		Rb	85.5
At	(210)		I	126.9		Re	186.2
Au	197.0		In	114.8		Rh	102.9
B	10.8		Ir	192.2		Rn	(222)
Ba	137.3		K	39.1		Ru	101.1
Be	9.0		Kr	83.8		S	32.0
Bi	209.0		La	138.9		Sb	121.8
Bk	(247)		Li	6.9		Sc	45.0
Br	79.9		Lu	175.0		Se	79.0
C	12.0		Lw	(257)		Si	28.1
Ca	40.1		Md	(258.1)		Sm	150.4
Cd	112.4		Mg	24.3		Sn	118.7
Ce	140.1		Mn	54.9		Sr	87.6
Cf	(242.1)		Mo	95.9		Ta	180.9
Cl	35.5		N	14.0		Tb	158.9
Cm	(247)		Na	23.0		Tc	(97.9)
Co	58.9		Nb	92.9		Te	127.6
Cr	52.0		Nd	144.2		Tl	204.4
Cs	132.9		Ne	20.2		Th	232.0
Cu	63.5		Ni	58.7		Ti	47.9
Dy	162.5		No	(259.1)		Tm	168.9
Er	167.3		Np	(237)		U	238.0
Es	(252.1)		O	16.0		V	50.9
Eu	152.0		Os	190.2		W	183.9
F	19.0		P	31.0		Xe	131.3
Fe	55.9		Pa	(231)		Y	88.9
Fm	(257)		Pb	207.2		Yb	173.0
Fr	(223)		Pd	106.4		Zn	65.4
Ga	69.7		Pm	(145)		Zr	91.2
Gd	157.3						

The Periodic Table of the Elements

Key

Atomic number →
Symbol →
Atomic weight →

( ) mass numbers of most stable isotope

1	2	3 (3d, 4d, 5d)	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1 **H** 1.0079																	2 **He** 4.0026
3 **Li** 6.941	4 **Be** 9.01218											5 **B** 10.81	6 **C** 12.011	7 **N** 14.0067	8 **O** 15.9994	9 **F** 18.9984	10 **Ne** 20.179
11 **Na** 22.98977	12 **Mg** 24.305											13 **Al** 26.9815	14 **Si** 28.0855	15 **P** 30.9738	16 **S** 32.06	17 **Cl** 35.453	18 **Ar** 39.948
19 **K** 39.0983	20 **Ca** 40.08	21 **Sc** 44.9559	22 **Ti** 47.88	23 **V** 50.9415	24 **Cr** 51.996	25 **Mn** 54.938	26 **Fe** 55.847	27 **Co** 58.9332	28 **Ni** 58.69	29 **Cu** 63.546	30 **Zn** 65.38	31 **Ga** 69.72	32 **Ge** 72.59	33 **As** 74.9216	34 **Se** 78.96	35 **Br** 79.904	36 **Kr** 83.80
37 **Rb** 85.4678	38 **Sr** 87.62	39 **Y** 88.9059	40 **Zr** 91.22	41 **Nb** 92.9064	42 **Mo** 95.94	43 **Tc** (97.9)	44 **Ru** 101.07	45 **Rh** 102.9055	46 **Pd** 106.42	47 **Ag** 107.868	48 **Cd** 112.41	49 **In** 114.82	50 **Sn** 118.69	51 **Sb** 121.75	52 **Te** 127.60	53 **I** 126.9045	54 **Xe** 131.29
55 **Cs** 132.9054	56 **Ba** 137.33	57 **#La** 138.9055	72 **Hf** 178.49	73 **Ta** 180.9479	74 **W** 183.85	75 **Re** 186.207	76 **Os** 190.2	77 **Ir** 192.22	78 **Pt** 195.08	79 **Au** 196.9665	80 **Hg** 200.59	81 **Tl** 204.383	82 **Pb** 207.2	83 **Bi** 208.9804	84 **Po** (209)	85 **At** (210)	86 **Rn** (222)
87 **Fr** (223)	88 **Ra** 226.0254	89 ***Ac** 227.0278															

LANTHANUM SERIES

58	59	60	61	62	63	64	65	66	67	68	69	70	71
Ce 140.12	**Pr** 140.9077	**Nd** 144.24	**Pm** (145)	**Sm** 150.36	**Eu** 151.96	**Gd** 157.25	**Tb** 158.9254	**Dy** 162.50	**Ho** 164.9304	**Er** 167.26	**Tm** 168.9342	**Yb** 173.04	**Lu** 174.967

* ACTINIUM SERIES

90	91	92	93	94	95	96	97	98	99	100	101	102	103
Th 232.0381	**Pa** 231.0359	**U** 238.0389	**Np** 237.0482	**Pu** (244)	**Am** (243)	**Cm** (247)	**Bk** (247)	**Cf** (242.1)	**Es** (254)	**Fm** (257)	**Md** (258.1)	**No** (259.1)	**Lr** (260)