

NATIONAL UNIVERSITY OF SINGAPORE

CM2142 – Analytical Chemistry 1

SEMESTER II : AY2014/15

TIME ALLOWED: 2 HOURS

INSTRUCTIONS TO STUDENTS

1. This assessment paper contains **FOUR (4)** questions and comprises **THIRTEEN (13)** printed pages.
2. Students are required to answer **ALL** questions.
3. Students should write the answers for each question on a new page.
4. **2 A4, double sided sheets of notes** are allowed.
5. Non-programmable calculators are allowed.
6. Formula Sheet and Periodic Table are attached at the back.

Question 1

- (a) The mass (in g) of a sample was determined by two different methods, gravimetric and volumetric. The values are given below:

	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7
Gravimetric	2.310	2.309	2.308	2.310	2.311	2.309	2.307
Volumetric	2.301	2.298	2.299	2.301	2.299	2.299	2.297

Are the methods equivalent at 95% confidence level? Confirm by performing an appropriate significance test. Explain why the Paired t-test is *not* an appropriate test.

(7 marks)

- (b) In a binary resin mixture, 20% of the particles have a 30% by weight coating (analyte) and a density of 0.48 g/cm^3 , whereas the remaining have 1% by weight coating (analyte) and a density of 0.24 g/cm^3 . Assume that the average particle in the batch being sampled is about 1 mm in diameter.

(i) What should the mass of the gross sample be if the sampling relative uncertainty is 0.5%?

(7 marks)

(ii) If the maximum sample loading is 1 g, determine the final diameter in mm of the resin before analysis.

(4 marks)

(iii) The results below were obtained:

Run	Laboratory Sample 1	Laboratory Sample 2	Laboratory Sample 3	Laboratory Sample 4	Laboratory Sample 5
1	10.3	9.5	12.1	9.6	11.6
2	9.8	8.6	13.0	8.3	12.5
3	11.4	8.9	12.4	8.2	11.4

If the relative uncertainty of 15% is required, what is the appropriate number of laboratory samples that should be analysed, at the 95% confidence level?

(7 marks)

Question 2

(a) Describe, with diagrams where appropriate, a suitable extraction technique for:

(i) limonene, which smells like lemon/oranges, from dish washing liquid

(ii) caffeine from coffee beans

(7 marks)

(b) A sample that is soluble in hexane is to be separated using HPLC. Describe the specific instrument parameters, including values where appropriate, and a common detector that you would recommend.

(10 marks)

- (c) Describe an appropriate HPLC method that would separate sodium and calcium ions, given that the method uses a conductivity detector. In your description, include a schematic of the column set up and also list the factors that determine the order of elution.

(8 marks)

Question 3

- (a) A 1.0 g sample of zinc metal is dissolved in 50 mL of 6.0 M HCl solution and diluted to the mark in a 250 mL volumetric flask. A 25 mL portion is transferred to a polarographic cell and oxygen is flushed out. A polarogram recorded in the range 0 to -1 V vs. SCE has $E_{1/2} = -0.65$ V and $i_d = 32.0$ μ A. A 5 mL portion of 5.0×10^{-4} M CdCl₂ solution is added directly to the polarography cell which already contains the Zn solution, oxygen is again flushed out, and a second polarogram is recorded. The polarogram shows the same $E_{1/2}$ but the $i_d = 77.5$ μ A. What is the % weight of Cd impurity in the metal? Sketch the polarograms that would be obtained, labelling the axes, $E_{1/2}$ and i_d .

(12 marks)

- (b) For the silver/silver chloride RE vs. SHE, what is the chloride ion concentration in the filling solution if the potential measured is 0.20 V, given that $K_{sp}(\text{AgCl}) = 1.8 \times 10^{-10}$ and $E^\circ_{\text{Ag}/\text{Ag}^+} = 0.80$ V. Explain the role of the RE in a 3-electrode voltammetry set up.

(6 marks)

- (c) Describe the working principle of the scanning electron microscopy (SEM) in secondary electron imaging mode. Include a schematic diagram in your description.

(7 marks)

Question 4

- (a) Acetate buffer is a common buffer. From its K_a expression, determine the Buffer equation. Write a procedure for the preparation of 250 mL of pH 5.0 acetate buffer using 1 M acetic acid and solid sodium acetate, given that $K_a = 1.8 \times 10^{-5}$.

(8 marks)

- (b) Determine the concentration of calcium in a solution of calcium sulphate that has been buffered to pH 1. Given that $K_{sp}(\text{CaSO}_4) = 2.4 \times 10^{-5}$; Sulphuric acid $K_{a1} = \text{large}$ and $K_{a2} = 1.02 \times 10^{-2}$.

(10 marks)

- (c) Calculate the concentration of Ca^{2+} in a solution of 0.01 M CaY^{2-} at pH 6 ($\alpha_4 = 2.3 \times 10^{-5}$) given that $K_{\text{CaY}^{2-}} = 5.0 \times 10^{10}$. Define and write an expression for α_4 .

(7 marks)

END OF PAPER

CM2142 Formula Sheet

Statistical Analysis

$$\mu = \frac{\sum_{i=1}^N x_i}{N}$$

$$\bar{x} = \frac{\sum_{i=1}^N x_i}{N}$$

$$\sigma = \sqrt{\frac{\sum_{i=1}^N (x_i - \mu)^2}{N}} \quad \sigma = \sqrt{\frac{\sum_{i=1}^N x_i^2 - \frac{\left(\sum_{i=1}^N x_i\right)^2}{N}}{N}}$$

$$s = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N-1}} \quad s = \sqrt{\frac{\sum_{i=1}^N x_i^2 - \frac{\left(\sum_{i=1}^N x_i\right)^2}{N}}{N-1}}$$

$$s^2 = \sigma^2 = \frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N-1}$$

$$RSD = \frac{s}{\bar{x}}$$

$$CV = \frac{s}{\bar{x}} \times 100\%$$

$$z = \frac{x - \mu}{\sigma}$$

$$t = \frac{x - \mu}{s}$$

$$s_m = \frac{s}{\sqrt{N}}$$

$$\mu = \bar{x} \pm \frac{z\sigma}{\sqrt{N}}$$

$$\mu = \bar{x} \pm \frac{ts}{\sqrt{N}}$$

Significance Tests

$$CL = (1 - \alpha) \times 100\%$$

$$z = (\bar{x} - \mu_0) \frac{\sqrt{N}}{\sigma}$$

$$t = (\bar{x} - \mu_0) \frac{\sqrt{N}}{s}$$

$$s_{\text{pooled}} = \sqrt{\frac{(N_1 - 1)s_1^2 + (N_2 - 1)s_2^2}{N_1 + N_2 - 2}}$$

$$t = \frac{\bar{x}_1 - \bar{x}_2}{s_{\text{pooled}} \sqrt{\left(\frac{N_1 + N_2}{N_1 N_2}\right)}}$$

$$t = (\bar{d} - \Delta_0) \frac{\sqrt{N}}{s_d}$$

$$F = \frac{s_1^2}{s_2^2}$$

$$Q = \frac{|x_q - x_n|}{(\text{highest value} - \text{lowest value})} = \frac{|x_q - x_n|}{w}$$

$$\bar{x} = \left(\frac{N_1}{N}\right)\bar{x}_1 + \left(\frac{N_2}{N}\right)\bar{x}_2 + \left(\frac{N_3}{N}\right)\bar{x}_3 + \dots + \left(\frac{N_i}{N}\right)\bar{x}_i$$

$$SSF = N_1(\bar{x}_1 - \bar{x})^2 + N_2(\bar{x}_2 - \bar{x})^2 + N_3(\bar{x}_3 - \bar{x})^2 + \dots + N_i(\bar{x}_i - \bar{x})^2$$

$$SSE = (N_1 - 1)s_1^2 + (N_2 - 1)s_2^2 + (N_3 - 1)s_3^2 + \dots + (N_i - 1)s_i^2$$

$$SST = SSF + SSE = (N - 1)s^2$$

where s^2 is the sample variance of all data points.

$$SST = SSF + SSE$$

$$(N - 1) = (i - 1) + (N - i)$$

$$MSF = \frac{SSF}{i - 1}$$

$$MSE = \frac{SSE}{N - i}$$

$$F = \frac{MSF}{MSE}$$

Sampling and Calibration

$$N = \frac{(1-p)}{p\sigma_r^2}$$

$$LOD = \bar{y}_B + 3s_B$$

$$LOQ = \bar{y}_B + 10s_B$$

$$N = p(1-p) \left(\frac{d_A d_B}{d^2}\right)^2 \left(\frac{X_A - X_B}{\sigma_r X}\right)^2$$

$$\sqrt{N} = \frac{ts_s}{\bar{x}\sigma_r}$$

$$SS_{\text{resid}} = \sum_{i=1}^N (y_i - \hat{y})^2 = \sum_{i=1}^N [y_i - (mx_i + c)]^2$$

$$R^2 = 1 - \frac{SS_{\text{resid}}}{SS_{\text{total}}}$$

$$SS_{\text{total}} = \sum_{i=1}^N (y_i - \bar{y})^2 \text{ where } \bar{y} = \frac{\sum_{i=1}^N y_i}{N}$$

$$s_c = \frac{\sqrt{\frac{\sum_{i=1}^N (y_i - \bar{y})^2 - m^2 \sum_{i=1}^N (x_i - \bar{x})^2}{N-2}}}{m} \sqrt{\frac{1}{U} + \frac{1}{N} + \frac{(\bar{y}_U - \bar{y})^2}{m^2 \sum_{i=1}^N (x_i - \bar{x})^2}}$$

TABLE 6-4

Error Propagation in Arithmetic Calculations		
Type of Calculation	Example*	Standard Deviation of y^\dagger
Addition or subtraction	$y = a + b - c$	$s_y = \sqrt{s_a^2 + s_b^2 + s_c^2}$ (1)
Multiplication or division	$y = a \times b/c$	$\frac{s_y}{y} = \sqrt{\left(\frac{s_a}{a}\right)^2 + \left(\frac{s_b}{b}\right)^2 + \left(\frac{s_c}{c}\right)^2}$ (2)
Exponentiation	$y = a^x$	$\frac{s_y}{y} = x \left(\frac{s_a}{a}\right)$ (3)
Logarithm	$y = \log_{10} a$	$s_y = 0.434 \frac{s_a}{a}$ (4)
Antilogarithm	$y = \text{antilog}_{10} a$	$\frac{s_y}{y} = 2.303 s_a$ (5)

* a , b , and c are experimental variables with standard deviations of s_a , s_b , and s_c , respectively
 \dagger These relationships are derived in Appendix 9. The values for s_y/y are absolute values if y is a negative number.

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TABLE 7-1

Confidence Levels for Various Values of z	
Confidence Level, %	z
50	0.67
68	1.00
80	1.28
90	1.64
95	1.96
95.4	2.00
99	2.58
99.7	3.00
99.9	3.29

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*

TABLE 7-3

Values of t for Various Levels of Probability					
Degrees of Freedom	80%	90%	95%	99%	99.9%
1	3.08	6.31	12.7	63.7	637
2	1.89	2.92	4.30	9.92	31.6
3	1.64	2.35	3.18	5.84	12.9
4	1.53	2.13	2.78	4.60	8.61
5	1.48	2.02	2.57	4.03	6.87
6	1.44	1.94	2.45	3.71	5.96
7	1.42	1.90	2.36	3.50	5.41
8	1.40	1.86	2.31	3.36	5.04
9	1.38	1.83	2.26	3.25	4.78
10	1.37	1.81	2.23	3.17	4.59
15	1.34	1.75	2.13	2.95	4.07
20	1.32	1.73	2.09	2.84	3.85
40	1.30	1.68	2.02	2.70	3.55
60	1.30	1.67	2.00	2.62	3.46
∞	1.28	1.64	1.96	2.58	3.29

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TABLE 7-4

Critical Values of F at the 5% Probability Level (95% confidence level)									
Degrees of Freedom (Denominator)	Degrees of Freedom (Numerator)								
	2	3	4	5	6	10	12	20	∞
2	19.00	19.16	19.25	19.30	19.33	19.40	19.41	19.45	19.50
3	9.55	9.28	9.12	9.01	8.94	8.79	8.74	8.66	8.53
4	6.94	6.59	6.39	6.26	6.16	5.96	5.91	5.80	5.63
5	5.79	5.41	5.19	5.05	4.95	4.74	4.68	4.56	4.36
6	5.14	4.76	4.53	4.39	4.28	4.06	4.00	3.87	3.67
10	4.10	3.71	3.48	3.33	3.22	2.98	2.91	2.77	2.54
12	3.89	3.49	3.26	3.11	3.00	2.75	2.69	2.54	2.30
20	3.49	3.10	2.87	2.71	2.60	2.35	2.28	2.12	1.84
∞	3.00	2.60	2.37	2.21	2.10	1.83	1.75	1.57	1.00

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TABLE 7-5

Critical Values for the Rejection Quotient, Q^*

Number of Observations	Q_{crit} (Reject if $Q > Q_{crit}$)		
	90% Confidence	95% Confidence	99% Confidence
3	0.941	0.970	0.994
4	0.765	0.829	0.926
5	0.642	0.710	0.821
6	0.560	0.625	0.740
7	0.507	0.568	0.680
8	0.468	0.526	0.634
9	0.437	0.493	0.598
10	0.412	0.466	0.568

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* Adapted from *Fundamentals of Analytical Chemistry (8th Edition)* Douglas A. Skoog, et. al. Thomson Brooks / Cole

Extraction Techniques

$$[A]_i = \left(\frac{V_{aq}}{K_D V_{org} + V_{aq}} \right)^i [A]_0 \quad \text{or} \quad [A]_i^T = \left(\frac{V_{aq}}{D V_{org} + V_{aq}} \right)^i [A]_0^T$$

$$F_A = \frac{K_D R_v}{K_D R_v + 1} \quad \text{or} \quad \frac{D_A R_v}{D_A R_v + 1}$$

where $R_v = \text{phase - volume ratio}, \frac{V_{org}}{V_{aq}}$

$$G_A = 1 - F_A$$

$$D = \frac{[HA]_{org}^T}{[HA]_{aq}^T} = \frac{K_D [H^+]}{[H^+] + K_a} = K_D \alpha_{HA}$$

$$D = \frac{[B]_{org}^T}{[B]_{aq}^T} = \frac{K_D [OH^-]}{[OH^-] + K_b} = K_D \alpha_B$$

$$D_M = K_{DM} K_{ML} \left(\frac{K_a}{K_{DL}} \right)^n \left(\frac{[HL]_{org}}{[H^+]_{aq}} \right)^n = K'' \left(\frac{[HL]_{org}}{[H^+]_{aq}} \right)^n$$

$$\text{Masking Factor} = -\log \frac{\alpha_{M_2}}{\alpha_{M_1}} \quad \text{where} \quad \alpha_M = \frac{[M^{n+}]_{aq}}{[M^{n+}]_{aq}^T}$$

Chromatography

$$t_R = t_S + t_M$$

$$\bar{v} = \frac{L}{t_R}$$

$$u = \frac{L}{t_M}$$

$$F = \frac{V_M}{t_M}$$

$$\text{Open column:} \quad F = u_o A = u_o \times \pi r^2$$

$$\text{Packed column:} \quad F = \pi r^2 u_o \epsilon$$

$$\bar{v} = u \times \frac{1}{1 + K \frac{V_S}{V_M}}$$

$$k = \frac{t_R - t_M}{t_M} = \frac{t_S}{t_M}$$

$$\alpha = \frac{(t_R)_B - t_M}{(t_R)_A - t_M} = \frac{(t_S)_B}{(t_S)_A}$$

$$N = \frac{L}{H}$$

$$H = \frac{\sigma^2}{L}$$

$$N = 16 \frac{t_R^2}{W^2} \quad N = 5.54 \frac{t_R^2}{W_{1/2}^2}$$

$$H = A + \frac{B}{u} + C_s u$$

$$H = \frac{B}{u} + C_s u + C_M u$$

$$A = 2 \lambda d_p$$

$$B = 2 \gamma D_M$$

$$C_s = \frac{2k}{3(k+1)^2} \frac{d^2}{D_s}$$

$$C_M = \frac{1+6k+11k^2}{96(k+1)^2} \frac{d^2}{D_M}$$

$$R_s = \frac{2[(t_R)_B - (t_R)_A]}{W_A + W_B}$$

$$R_s = 1.175 \frac{[(t_R)_B - (t_R)_A]}{[W_{1/2A} + W_{1/2B}]}$$

$$R_s = \left(\frac{\sqrt{N}}{4} \right) \left(\frac{\alpha - 1}{\alpha} \right) \left(\frac{k_B}{1 + k_B} \right)$$

Gas Chromatography

$$I = 100 \left[n + (N-n) \frac{\log t_{R(\text{unk})}' - \log t_{R(n)}'}{\log t_{R(N)}' - \log t_{R(n)}'} \right]$$

Electrophoresis

$$u_{ep} = \frac{Q}{f} E = \mu_{ep} E$$

$$f = 6\pi r \eta$$

$$E = \frac{V}{L}$$

$$u_{eo} = \mu_{eo} E$$

$$\mu_{eo} = \frac{u_{eo, \text{neutral}}}{E} = \frac{L_d / t_{\text{neutral}}}{V/L}$$

$$\mu_{app} = \mu_{ep} + \mu_{eo} = \frac{L_d}{t}$$

$$N = \frac{L_d^2}{2Dt} = \frac{\mu_{app} V L_d}{2D L}$$

$$R_s = \frac{2[t_B - t_A]}{W_A + W_B}$$

$$R_s = \left(\frac{\sqrt{N}}{4} \right) (\alpha - 1)$$

Electrochemistry

$$A = C / s$$

$$V = J / C$$

$$V = i R$$

$$Q = n F = i t$$

$$w = n F E = Q E$$

$$P = w / t = i E$$

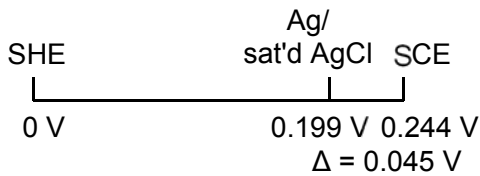
$$\Delta G = -n F E = -w$$

$$E_{\text{cell}} = E^{\circ}_{\text{cell}} - \frac{RT}{nF} \ln Q$$

$$E_{\text{cell}} = E^{\circ}_{\text{cell}} - \frac{0.059}{n} \log Q$$

Potentiometry

$$E_{\text{cell}} = E_{\text{ind}} - E_{\text{ref}} + E_j$$



$$E_{\text{outside}} = \frac{\Delta G_{\text{solvation}}}{nF} - \left(\frac{RT}{nF} \right) \ln \left(\frac{a_m}{a_o} \right)$$

$$E_{\text{cell}} = k + \left(\frac{RT}{nF} \right) \ln a_o = k + \frac{0.059}{n} \log a_{\text{sample}} \quad \text{at 298 K}$$

$$\text{where } k = \frac{\Delta G_{\text{solvation}}}{nF} - \left(\frac{RT}{nF} \right) \ln a_m - E_{\text{inside}}$$

$$E_{\text{cell}} = k - 0.059 \text{ pH at 298 K}$$

$$(V_o + V_s) 10^{\frac{E_{\text{cell}}}{Y}} = 10^{\frac{k}{Y}} c_a V_o + 10^{\frac{k}{Y}} c_s V_s$$

$$\text{where } Y = \beta \left(\frac{RT}{nF} \ln 10 \right)$$

Voltammetry

$$i = \frac{n F A D ([\text{analyte}]_{\text{bulk}} - [\text{analyte}]_{x=0})}{\delta}$$

$$\frac{Q}{E} = C$$

$$i_p = 2.686 \times 10^5 n^{3/2} A c D^{1/2} v^{1/2}$$

$$\Gamma = Q / n F A$$

$$(i_d)_{\text{max}} = 708 n D^{1/2} m^{2/3} t^{1/6} c$$

Chemical Equilibrium

$$\Delta G = \Delta H - T\Delta S$$

$$\Delta G = RT \ln Q/K = RT \ln Q - RT \ln K$$

$$\Delta G^\circ = -RT \ln K$$

$$\Delta G = \Delta G^\circ + RT \ln Q$$

$$\ln \frac{K_2}{K_1} = \left(\frac{-\Delta H^\circ}{R} \right) \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$$

$$K_p = (RT)^{\Delta n} K_c$$

Acid Base Equilibrium

$$K_a \times K_b = K_w$$

$$\text{pH} = \text{p}K_a + \log_{10} \frac{[\text{base}]}{[\text{acid}]}$$

$$\mu = \frac{1}{2} ([A]Z_A^2 + [B]Z_B^2 + [C]Z_C^2 + \dots)$$

$$a_x = [X] \gamma_x$$

$$-\log \gamma_x = \frac{0.51 Z_x^2 \sqrt{\mu}}{1 + 3.3 \alpha_x \sqrt{\mu}}$$

TABLE 14-1**Some Important Acid/Base Indicators**

Common Name	Transition Range, pH	pK _a [*]	Color Change [†]	Indicator Type [‡]
Thymol blue	1.2–2.8	1.65§	R–Y	1
	8.0–9.6	8.96§	Y–B	
Methyl yellow	2.9–4.0		R–Y	2
Methyl orange	3.1–4.4	3.46§	R–O	2
Bromocresol green	3.8–5.4	4.66§	Y–B	1
Methyl red	4.2–6.3	5.00§	R–Y	2
Bromocresol purple	5.2–6.8	6.12§	Y–P	1
Bromothymol blue	6.2–7.6	7.10§	Y–B	1
Phenol red	6.8–8.4	7.81§	Y–R	1
Cresol purple	7.6–9.2		Y–P	1
Phenolphthalein	8.3–10.0		C–R	1
Thymolphthalein	9.3–10.5		C–B	1
Alizarin yellow GG	10–12		C–Y	2

*At ionic strength of 0.1.

†B = blue; C = colorless; O = orange; P = purple; R = red; Y = yellow.

‡(1) Acid type: $\text{HIn} + \text{H}_2\text{O} \rightleftharpoons \text{H}_3\text{O}^+ + \text{In}^-$; (2) Base type: $\text{In} + \text{H}_2\text{O} \rightleftharpoons \text{InH}^+ + \text{OH}^-$.

§For the reaction $\text{InH}^+ + \text{H}_2\text{O} \rightleftharpoons \text{H}_3\text{O}^+ + \text{In}$.

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* Adapted from *Fundamentals of Analytical Chemistry (8th Edition)* Douglas A. Skoog, et. al. Thomson Brooks / Cole

Periodic Table of the Elements

		Main groups																	
		Transition metals																	
1A ^a	1	2A											3A	4A	5A	6A	7A	8A	18
1	1	2											13	14	15	16	17	18	2
	H	He											B	C	N	O	F	Ne	
	1.00794	4.002602											10.811	12.0107	14.0067	15.9994	18.998403	20.1797	
2	3	4											13	14	15	16	17	18	10
	Li	Be											Al	Si	P	S	Cl	Ar	
	6.941	9.012182											26.981538	28.0855	30.973761	32.065	35.453	39.948	
3	11	12											31	32	33	34	35	36	10
	Na	Mg											Ga	Ge	As	Se	Br	Kr	
	22.989770	24.3050											69.723	72.64	74.92160	78.96	79.904	83.80	
4	19	20											49	50	51	52	53	54	10
	K	Ca											In	Sn	Sb	Te	I	Xe	
	39.0983	40.078											114.818	118.710	121.760	127.60	126.90447	131.293	
5	37	38											81	82	83	84	85	86	10
	Rb	Sr											Tl	Pb	Bi	Po	At	Rn	
	85.4678	87.62											204.3833	207.2	208.98038	[208.98]	[209.99]	[222.02]	
6	55	56											114	114	114	116	116	116	10
	Cs	Ba											[285]	[285]	[285]	[289]	[289]	[289]	
	132.90545	137.327																	
7	87	88																	
	Fr	Ra																	
	[223.02]	[226.03]																	
		Main groups																	
	57	58	59	60	61	62	63	64	65	66	67	68	69	70	70	70	70		
	*La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Yb	Yb	Yb		
	138.9055	140.116	140.90765	144.24	[145]	150.36	151.964	157.25	158.92534	162.50	164.93032	167.259	168.93421	173.04	173.04	173.04	173.04		
	89	90	91	92	93	94	95	96	97	98	99	100	101	102	102	102	102		
	†Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	No	No	No		
	[227.03]	232.0381	231.03588	238.02891	[237.05]	[244.06]	[243.06]	[247.07]	[247.07]	[251.08]	[252.08]	[257.10]	[258.10]	[259.10]	[259.10]	[259.10]	[259.10]		