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ADVANCED
General Certificate of Education
2019

Centre Number

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Candidate Number

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Chemistry

StemUp Model Solution

Assessment Unit A2 3

assessing

Further Practical Chemistry

Practical Booklet B (Theory)



[ACH32]

ACH32

WEDNESDAY 19 JUNE, MORNING

TIME

1 hour 15 minutes.

INSTRUCTIONS TO CANDIDATES

Write your Centre Number and Candidate Number in the spaces provided at the top of this page.

You must answer the questions in the spaces provided.

Do not write outside the boxed area on each page or on blank pages.

Complete in black ink only. **Do not write with a gel pen.**

Answer **all six** questions.

INFORMATION FOR CANDIDATES

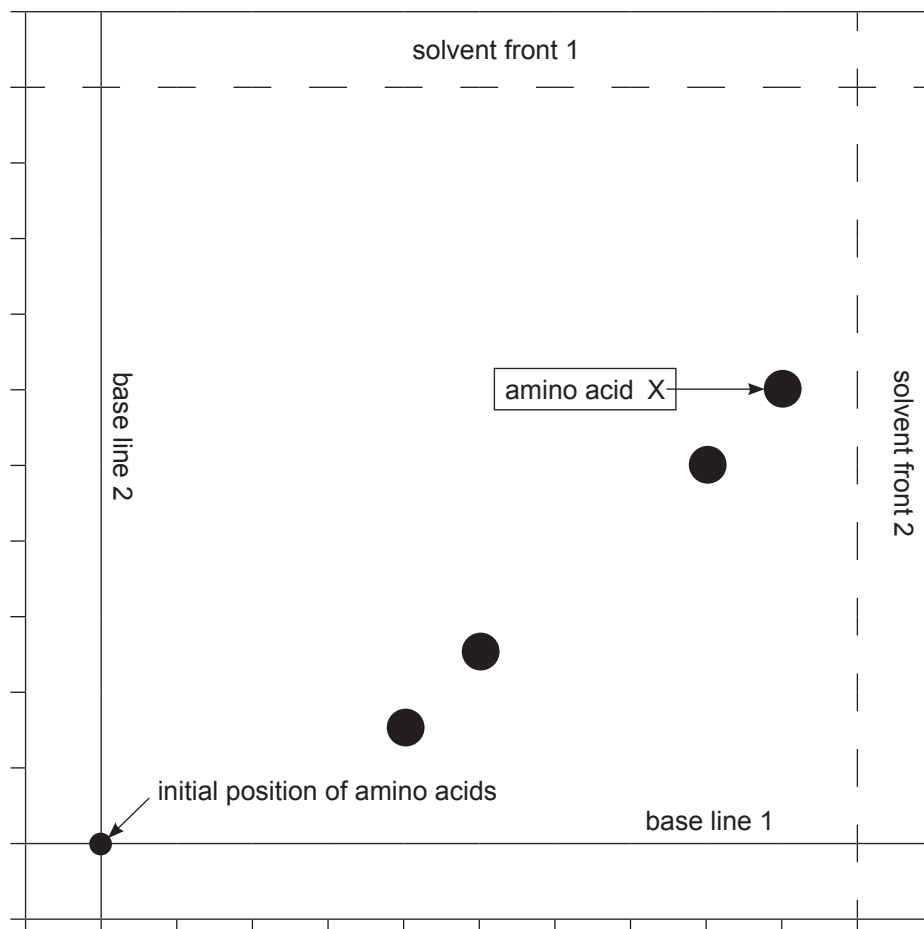
The total mark for this paper is 60.

Figures in brackets printed down the right-hand side of pages indicate the marks awarded to each question or part question.

A Periodic Table of Elements (including some data) is provided.



- 1 A mixture of amino acids was analysed using two-way paper chromatography. The first “solvent” used was a solution of butan-1-ol in ethanoic acid. The second “solvent” was a solution of phenol in water. The chromatogram obtained is shown below.



- (a) A spot of the mixture was placed on base line 1 using a capillary tube. Describe, giving experimental details, how this chromatogram would be obtained.

Run the paper in a covered tank of solvent 1, base line 1 above the solvent level, until the solvent is near the top.

Remove the paper, mark solvent front 1 and allow the paper to dry.

Rotate the paper by 90° (anticlockwise) and run it in solvent 2, then mark the solvent front and dry.

Locate the spots by spraying with ninhydrin and warming (or iodine or UV light).

[4]



- (b) Calculate the R_f values for the amino acid, X, in solvents 1 and 2. Measure to the middle of the spot.

Solvent 1 $R_f = 6 \div 10 = 0.6$

Solvent 2 $R_f = 9 \div 10 = 0.9$ [1]

- (c) Without access to R_f data, how would you show that the amino acid leucine was present and the amino acid serine was absent from the mixture?

Repeat the two-way chromatography under the same conditions using

separate samples of pure leucine and pure serine.

Compare the positions of the spots (R_f values): a spot in the mixture

matches leucine, and none matches serine. [2]

- (d) Why is two-way paper chromatography a better method for separating amino acids than one-way paper chromatography?

It gives greater separation between the spots, so amino acids with

similar R_f values in one solvent are separated. [1]

- (e) GLC MS can be used to identify drugs and determine their purity.

- (i) How would GLC indicate that a drug was pure?

There is only one peak in the gas chromatogram.

[1]

- (ii) Give **two** ways in which the identity of a drug can be established using GLC MS.

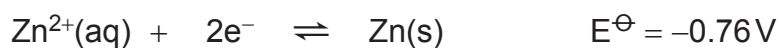
The retention time matches that of the pure drug.

The mass spectrum matches that of the pure drug. [2]

[Turn over]



2 The standard electrode potentials of two half-cells are given below.

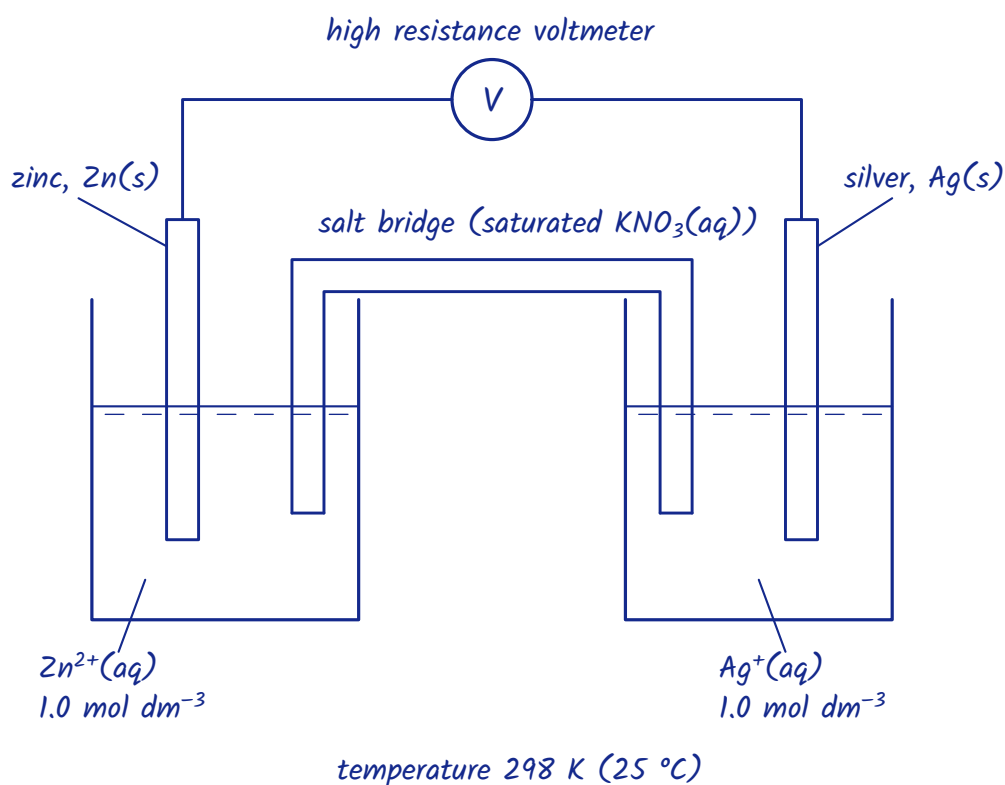


(a) Define the term **standard electrode potential**.

The emf (potential difference) of a half-cell measured when it is connected to the standard hydrogen electrode, under standard conditions (298 K, 100 kPa, 1.0 mol dm⁻³).

[2]

(b) (i) Draw a labelled diagram to show the cell which you would use to measure the potential difference between these two half-cells under standard conditions. Your diagram should include the concentrations of any ions in solution and the temperature at which the measurement would be made.



[4]



(ii) Calculate the emf of this cell.

$$emf = E^{\ominus}(\text{right}) - E^{\ominus}(\text{left}) = +0.80 - (-0.76) = +1.56 \text{ V}$$

[1]

(c) Magnesium is a stronger reducing agent than zinc. The standard electrode potential of the magnesium half-cell can be determined by setting up the following cell:



This cell has an emf of +1.61 V. Calculate the standard electrode potential of the magnesium half-cell.

$$+1.61 = E^{\ominus}(\text{Zn}^{2+}/\text{Zn}) - E^{\ominus}(\text{Mg}^{2+}/\text{Mg}) = -0.76 - E^{\ominus}(\text{Mg}^{2+}/\text{Mg})$$

$$E^{\ominus}(\text{Mg}^{2+}/\text{Mg}) = -2.37 \text{ V}$$

[1]

[Turn over]



3 A series of tests was carried out on two organic liquids, **A** and **B**.

(a) Liquid **A** produced an orange solid when reacted with 2,4-dinitrophenylhydrazine and a silver mirror when heated with Tollens' reagent. The mass spectrum of **A** showed a molecular ion peak at 58.

(i) To which homologous series does **A** belong? Explain how the results of the chemical tests are consistent with your conclusion.

Aldehyde. The orange solid with 2,4-DNPH shows that A

contains a carbonyl group (aldehyde or ketone).

The silver mirror with Tollens' reagent shows A is oxidised, so it is not a ketone.

[3]

(ii) Name **A** and identify the molecular ion.

Propanal; molecular ion is $\text{CH}_3\text{CH}_2\text{CHO}^+$

[1]

(b) Liquid **B** reacted vigorously with phosphorus pentachloride, and an aqueous solution of **B** gave effervescence on addition of sodium carbonate. The high resolution nmr spectrum of **B** contains a singlet, a quartet and a triplet.

(i) To which homologous series does **B** belong? Explain how the results of the chemical tests are consistent with your conclusion.

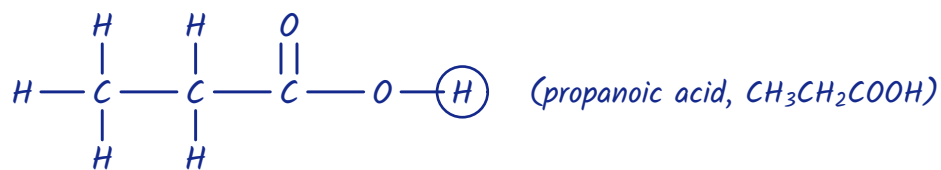
Carboxylic acid. The vigorous reaction with PCl_5 (steamy fumes of HCl) shows B contains an $-\text{OH}$ group.

Effervescence (CO_2) with sodium carbonate shows B is acidic, so a carboxylic acid.

[3]



- (ii) Give the structure of **B** and circle the hydrogen(s) responsible for the singlet. Suggest a chemical shift value for the position of the singlet.



Singlet (COOH) at about 11 ppm (10.0 to 12.0 ppm). [2]

- (iii) Explain how the spin-spin splitting in the nmr spectrum is consistent with the structure given in (ii).

CH₃ is a triplet: there are two H atoms on the adjacent carbon

(n + 1 = 3).

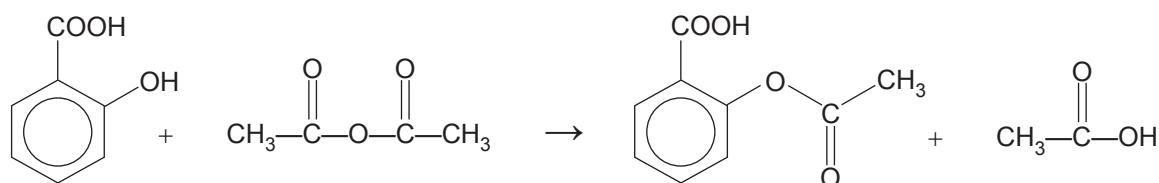
CH₂ is a quartet: there are three H atoms on the adjacent

carbon (n + 1 = 4). [2]

[Turn over



- 4 In the presence of concentrated phosphoric acid, salicylic acid reacts with ethanoic anhydride to form aspirin and ethanoic acid.



Addition of cold water causes the crude product to precipitate as a white solid which is filtered off and recrystallised. The purity of the final product is confirmed by carrying out a melting point determination.

- (a) Assuming a 40% yield, calculate the minimum mass of salicylic acid required to form 18.0 g of aspirin.

$$M_r \text{ aspirin } (C_9H_8O_4) = (9 \times 12) + (8 \times 1) + (4 \times 16) = 180$$

$$\text{moles of aspirin} = 18.0 \div 180 = 0.100 \text{ mol}$$

$$\text{moles needed for 40\% yield} = 0.100 \times 100 \div 40 = 0.250 \text{ mol}$$

$$M_r \text{ salicylic acid } (C_7H_6O_3) = 138$$

$$\text{mass of salicylic acid} = 0.250 \times 138 = 34.5 \text{ g}$$

[3]



- (b) Describe the purification of aspirin by recrystallisation.

Dissolve the crude aspirin in the minimum volume of hot ethanol (or hot water).

Filter the hot solution to remove insoluble impurities.

Allow the filtrate to cool so that crystals form.

Filter off the crystals under suction (Büchner funnel), wash with a little cold solvent and dry.

[4]

- (c) Describe the melting point determination and explain how the result can be used to confirm the purity of the product.

Place a little dry solid in a capillary tube sealed at one end.

Put the tube in a melting point apparatus (or oil bath).

Heat slowly near the melting point.

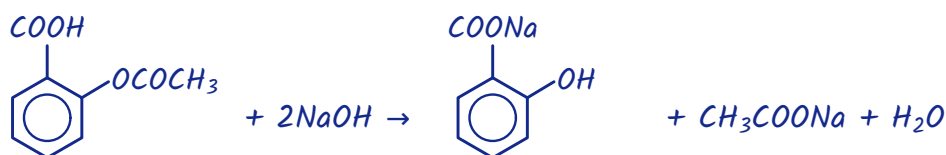
Record the temperatures at which melting begins and ends.

A sharp melting point at the data book value confirms purity.

[5]

- (d) Volumetric analysis of a solution containing aspirin can be carried out by adding an excess of sodium hydroxide solution, warming the mixture to hydrolyse the ester group and then titrating the excess sodium hydroxide with a standard solution of hydrochloric acid.

Write an equation for the reaction of aspirin with excess sodium hydroxide.

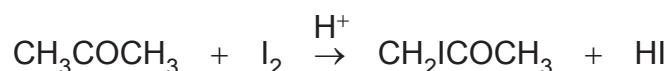


[1]

[Turn over



- 5 Iodine reacts with propanone, in the presence of an acid catalyst, according to the following equation:



The order of reaction with respect to iodine can be determined by mixing iodine with a large excess of both propanone and hydrochloric acid, starting a stopclock and then monitoring how the concentration of iodine changes with respect to time.

- (a) How could you determine the concentration of iodine at regular time intervals using colorimetry?

Plot a calibration curve of absorbance against $[\text{I}_2]$ using solutions of known iodine concentration.

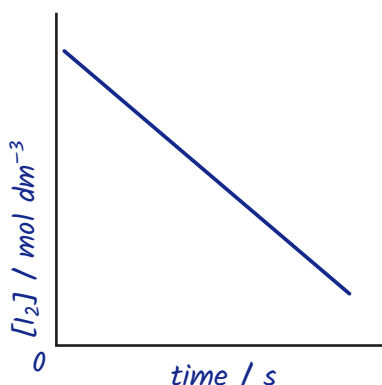
Place the reaction mixture in the colorimeter and record the absorbance at regular time intervals.

Convert each absorbance to $[\text{I}_2]$ using the calibration graph.

[4]

- (b) A graph of iodine concentration against time can be used to show that the reaction is zero order with respect to iodine.

- (i) Sketch the graph and label the axes.



[2]



(ii) How can the graph be used to determine the rate of the reaction?

The rate of reaction is equal to the (negative) gradient of the line.

[1]

(c) Suggest why a large excess of propanone and hydrochloric acid is used.

So the concentrations of propanone and H^+ ions remain effectively constant and only $[I_2]$ changes.

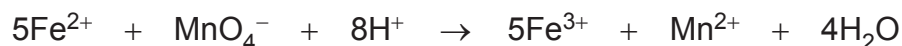
[1]

[Turn over]



- 6 A solution containing iron(II) ions was left exposed to the air for several days. Some of the iron(II) ions were oxidised to iron(III) ions.

- (a) The number of moles of iron(II) ions present in the solution can be determined by titrating with acidified manganate(VII) solution.



- (i) Why is no indicator required in this titration?

*MnO₄⁻ is purple and Mn²⁺ is colourless, so manganate(VII) acts
as its own indicator (self-indicating).*

[1]

- (ii) Give the colour change at the end point.

colourless to pale pink

[2]

- (b) A 25.0 cm³ aliquot of the solution, which had been partially oxidised, was transferred into a conical flask. The solution was acidified and then titrated with 0.020 mol dm⁻³ potassium manganate(VII). 18.0 cm³ was required. Calculate the number of moles of iron(II) ions present in this aliquot.

$$\text{moles MnO}_4^- = 0.020 \times 18.0 \div 1000 = 3.6 \times 10^{-4} \text{ mol}$$

$$\text{moles Fe}^{2+} = 5 \times 3.6 \times 10^{-4} = 1.8 \times 10^{-3} \text{ mol}$$

[2]



- (c) Another portion of the solution, which had been partially oxidised, was treated with an excess of zinc. The unreacted zinc was then removed by filtration and 25.0 cm³ of the filtrate was transferred into a conical flask. The solution was acidified and then titrated with 0.020 mol dm⁻³ potassium manganate(VII) of which 30.0 cm³ was required.

- (i) Use an equation to explain the purpose of the zinc.



Zinc is a reducing agent: it reduces all the iron(III) back to iron(II).

[2]

- (ii) Calculate the percentage of the iron(II) ions which had been oxidised in this solution.

$$\text{Fe}^{2+} \text{ after Zn} = 5 \times 0.020 \times 30.0 \div 1000 = 3.0 \times 10^{-3} \text{ mol}$$

$$\% \text{ oxidised} = (3.0 - 1.8) \div 3.0 \times 100 = 40\%$$

[2]

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12318



16ACH3215

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Question Number	Examiner Mark	Remark
1		
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Total Marks		

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General Information

1 tonne = 10^6 g

1 metre = 10^9 nm

One mole of any gas at 293 K and a pressure of 1 atmosphere (10^5 Pa) occupies a volume of 24 dm^3

Avogadro Constant = $6.02 \times 10^{23} \text{ mol}^{-1}$

Planck Constant = $6.63 \times 10^{-34} \text{ Js}$

Specific Heat Capacity of water = $4.2 \text{ J g}^{-1} \text{ K}^{-1}$

Speed of Light = $3 \times 10^8 \text{ ms}^{-1}$

Characteristic absorptions in IR spectroscopy

Wavenumber/ cm^{-1}	Bond	Compound
550–850	C–X (X = Cl, Br, I)	Haloalkanes
750–1100	C–C	Alkanes, alkyl groups
1000–1300	C–O	Alcohols, esters, carboxylic acids
1450–1650	C=C	Arenes
1600–1700	C=C	Alkenes
1650–1800	C=O	Carboxylic acids, esters, aldehydes, ketones, amides, acyl chlorides
2200–2300	C≡N	Nitriles
2500–3200	O–H	Carboxylic acids
2750–2850	C–H	Aldehydes
2850–3000	C–H	Alkanes, alkyl groups, alkenes, arenes
3200–3600	O–H	Alcohols
3300–3500	N–H	Amines, amides

Proton Chemical Shifts in Nuclear Magnetic Resonance Spectroscopy (relative to TMS)

Chemical Shift	Structure	
0.5–2.0	–CH	Saturated alkanes
0.5–5.5	–OH	Alcohols
1.0–3.0	–NH	Amines
2.0–3.0	–CO–CH	Ketones
	–N–CH	Amines
	$\text{C}_6\text{H}_5\text{–CH}$	Arene (aliphatic on ring)
2.0–4.0	X–CH	X = Cl or Br (3.0–4.0) X = I (2.0–3.0)
4.5–6.0	–C=CH	Alkenes
5.5–8.5	RCONH	Amides
6.0–8.0	– C_6H_5	Arenes (on ring)
9.0–10.0	–CHO	Aldehydes
10.0–12.0	–COOH	Carboxylic acids

These chemical shifts are concentration and temperature dependent and may be outside the ranges indicated above.

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New
Specification



Data Leaflet

Including the Periodic Table of the Elements

For the use of candidates taking
Advanced Subsidiary and
Advanced Level Examinations

Copies must be free from notes or additions of any kind. No other type of data booklet or information sheet is authorised for use in the examinations

gce a/as examinations chemistry

For first teaching from September 2016
For first award of AS Level in Summer 2017
For first award of A Level in Summer 2018
Subject Code: 1110

I II THE PERIODIC TABLE OF ELEMENTS III IV V VI VII 0

Group

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1 H Hydrogen 1																	4 He Helium 2
7 Li Lithium 3	9 Be Beryllium 4											11 B Boron 5	12 C Carbon 6	14 N Nitrogen 7	16 O Oxygen 8	19 F Fluorine 9	20 Ne Neon 10
23 Na Sodium 11	24 Mg Magnesium 12											27 Al Aluminium 13	28 Si Silicon 14	31 P Phosphorus 15	32 S Sulfur 16	35.5 Cl Chlorine 17	40 Ar Argon 18
39 K Potassium 19	40 Ca Calcium 20	45 Sc Scandium 21	48 Ti Titanium 22	51 V Vanadium 23	52 Cr Chromium 24	55 Mn Manganese 25	56 Fe Iron 26	59 Co Cobalt 27	59 Ni Nickel 28	64 Cu Copper 29	65 Zn Zinc 30	70 Ga Gallium 31	73 Ge Germanium 32	75 As Arsenic 33	79 Se Selenium 34	80 Br Bromine 35	84 Kr Krypton 36
85 Rb Rubidium 37	88 Sr Strontium 38	89 Y Yttrium 39	91 Zr Zirconium 40	93 Nb Niobium 41	96 Mo Molybdenum 42	98 Tc Technetium 43	101 Ru Ruthenium 44	103 Rh Rhodium 45	106 Pd Palladium 46	108 Ag Silver 47	112 Cd Cadmium 48	115 In Indium 49	119 Sn Tin 50	122 Sb Antimony 51	128 Te Tellurium 52	127 I Iodine 53	131 Xe Xenon 54
133 Cs Caesium 55	137 Ba Barium 56	139 La [*] Lanthanum 57	178 Hf Hafnium 72	181 Ta Tantalum 73	184 W Tungsten 74	186 Re Rhenium 75	190 Os Osmium 76	192 Ir Iridium 77	195 Pt Platinum 78	197 Au Gold 79	201 Hg Mercury 80	204 Tl Thallium 81	207 Pb Lead 82	209 Bi Bismuth 83	210 Po Polonium 84	210 At Astatine 85	222 Rn Radon 86
223 Fr Francium 87	226 Ra Radium 88	227 Ac [†] Actinium 89	261 Rf Rutherfordium 104	262 Db Dubnium 105	266 Sg Seaborgium 106	264 Bh Bohrium 107	277 Hs Hassium 108	268 Mt Meitnerium 109	271 Ds Darmstadtium 110	272 Rg Roentgenium 111	285 Cn Copernicium 112						

* 58 – 71 Lanthanum series
† 90 – 103 Actinium series

a

X

b

a = relative atomic mass (approx)
x = atomic symbol
b = atomic number

140 Ce Cerium 58	141 Pr Praseodymium 59	144 Nd Neodymium 60	145 Pm Promethium 61	150 Sm Samarium 62	152 Eu Europium 63	157 Gd Gadolinium 64	159 Tb Terbium 65	162 Dy Dysprosium 66	165 Ho Holmium 67	167 Er Erbium 68	169 Tm Thulium 69	173 Yb Ytterbium 70	175 Lu Lutetium 71
232 Th Thorium 90	231 Pa Protactinium 91	238 U Uranium 92	237 Np Neptunium 93	242 Pu Plutonium 94	243 Am Americium 95	247 Cm Curium 96	245 Bk Berkelium 97	251 Cf Californium 98	254 Es Einsteinium 99	253 Fm Fermium 100	256 Md Mendelevium 101	254 No Nobelium 102	257 Lr Lawrencium 103