



DEPARTMENT OF CHEMICAL SCIENCES	
MODULE	CEM3B10/CEM01B3 INSTRUMENTAL CHEMICAL ANALYSIS
CAMPUS	APK
EXAM	January 2020

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SESSION:

ASSESSOR:

DR. AA AMBUSHE

EXTERNAL MODERATOR:

PROF. KL MANDIWANA

DURATION: 3 HOURS

MARKS: 100

NUMBER OF PAGES: 13

GENERAL INSTRUCTIONS

1. This paper consists of 13 pages including a Periodic Table and information sheet.
 2. The use of calculators is allowed but mobile phones may not be used.
 3. All answers must be given to correct number of significant figures.
 4. Answer all questions giving detailed explanation wherever required.
 5. Write neatly and legibly.
 6. Use allocated marks to gauge the amount of information to give as answer.
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QUESTION ONE

- 1.1. Write the types of transitions that take place when electromagnetic radiation in each of the following spectral regions interacts with the sample. (2)
- a) Infrared
b) NMR
- 1.2. Either a monochromator or polychromator can be used in a spectrometer as a wavelength selector. Differentiate between these two wavelength selectors. (4)
- 1.3. Draw a fully annotated sketch of a single beam UV-Visible spectrophotometer. (4)
- 1.4. Why are the absorption spectra of ions of the lanthanide and actinide series in aqueous solutions narrower than the absorption spectra of first and second row transition series ions? (3)
- 1.5. Discuss important properties of a solvent that need to be considered during selection of a solvent for UV-VIS absorption experiments. (4)
- 1.6. Ruthenium(II) and osmium(III) complexes can be determined simultaneously by a reaction with methiomerprazine. The absorption maximum of the ruthenium complex occurs at 480 nm, while that of osmium complex occurs at 635 nm. Molar absorptivity data at these wavelengths are as follows:

	Molar absorptivity, ϵ	
	480 nm	635 nm
Ruthenium complex	3.55×10^3	5.64×10^2
Osmium complex	2.96×10^3	1.45×10^4

A 25.0 mL sample was treated with an excess of methiomeprazine and subsequently diluted to 100.0 mL. Calculate the molar concentrations of ruthenium(II), c_{Ru} , and osmium(III), c_{Os} , in the original sample if the diluted solution had an absorbance of 0.533 at 480 nm and 0.590 at 635 nm when measured in a 2 cm cell. (5)

- 1.7. Why is IR spectroscopy less suitable for quantitative analysis than UV/VIS spectroscopy? (3)

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QUESTION TWO

- 2.1. Briefly explain the meaning of each of the following as referred to in atomic spectroscopy:
- a) Atomization (2)
 - b) Nebulization (2)
- 2.2. In flame atomic absorption spectrometry with an acetylene/air flame, the absorbance for iron decreased in the presence of large concentrations of sulfate ion.
- a) Suggest an explanation for this observation. (2)
 - b) Suggest three possible methods of overcoming the potential interference of sulfate in a quantitative determination of iron. (3)
- 2.3. Briefly explain the principle of hydride generation atomic absorption spectrometry. (5)
- 2.4. What are the possible sources of background in AAS? (4)
- 2.5. What are the functions of nebuliser gas flow in ICP-OES and ICP-MS? (3)

2.6. Briefly explain the function of each of the following components of an ICP-OES.

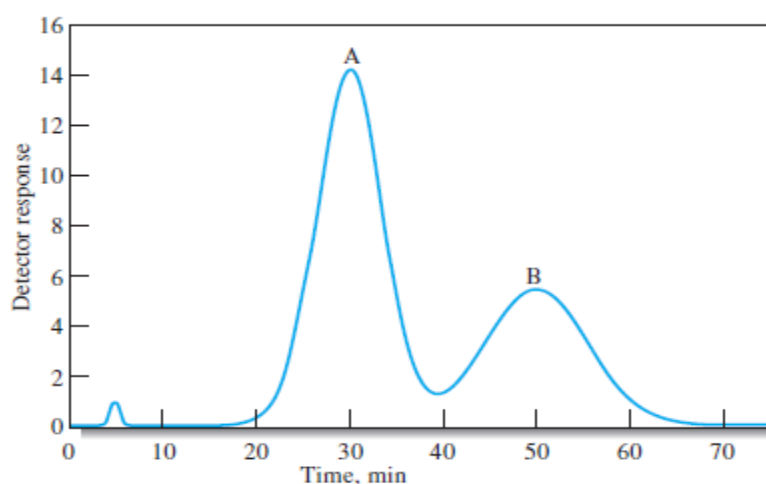
(3)

- a) Peristaltic pump
- b) RF generator
- c) Monochromator

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QUESTION THREE

3.1. A chromatogram of a two-component mixture on a 25-cm packed liquid chromatography column is shown in the figure below. The flow rate was 0.40 mL/min. Consider the peak widths at the base for components A and B are 20 and 25 minutes, respectively.



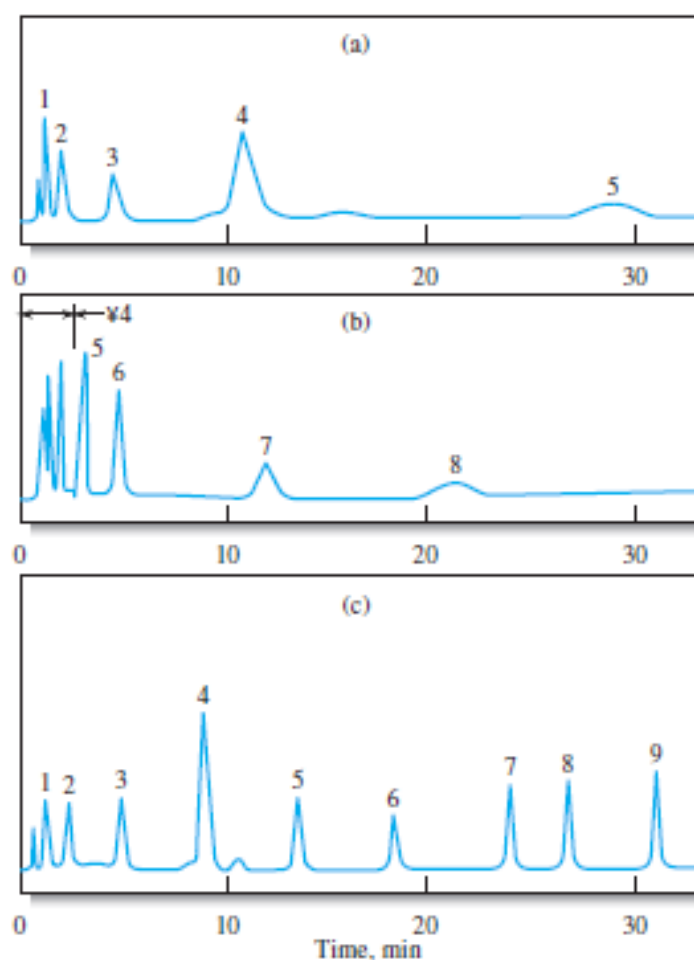
- (a) Find the times that components A and B spent in the stationary phase. (2)
- (b) Determine the retention factors for the two components. (2)
- (c) Calculate the resolution of the two peaks. (2)
- (d) Are these components completely resolved? Give a reason for your answer. (2)
- (e) Calculate the average number of plates for the column. (2)

3.2. Gas chromatography (GC) is used for separation of volatile compounds.

a) Explain how separation occurs in GC when analysing similar compounds. (2)

b) Mention the differences between split and splitless injection. (2)

3.3. In figure below, describe the type of temperature programme that was used for each of the chromatograms shown and its effect on the separation. How was the separation improved? (7)



3.4. On a C_{18} bonded phase, a particular compound moves too slowly in a methanol/acetonitrile (50/50) solvent. What adjustment should be made to the ratio of solvents? (2)

3.5. Clearly distinguish between gel filtration and gel permeation. (2)

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QUESTION FOUR

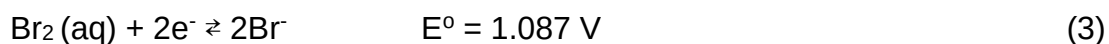
4.1. Describe the alkaline error in the measurement of a pH. (2)

a) Under what circumstances is this error appreciable? (1)

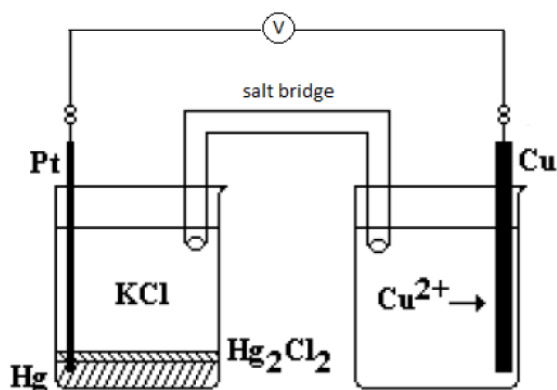
b) How is the pH data affected by the alkaline error? (1)

4.2. Why is it necessary to bubble hydrogen through the electrolyte in a hydrogen electrode? (3)

4.3. Calculate the potential for a platinum electrode immersed in a solution that is 0.0150 M in KBr and 1.00×10^{-3} M in Br_2 .



4.4. The following is an illustration of an electrochemical cell, with a calomel reference electrode.



a) Describe the type of indicator electrode shown above. (1)

b) What are some of the disadvantages associated with using this type of indicator electrode? (2)

c) Define junction potential and comment on where this could possibly arise from for the above set-up. (2)

4.5. Briefly describe asymmetry potential. (2)

4.6. How do concentration polarisation and kinetic polarisation resemble each other and how do they differ? (3)

4.7. Calculate the time needed for a constant current of 0.852 Amperes to deposit 0.250 g of Co(II) as:

(a) Elemental cobalt on the surface of a cathode. (3)

(b) Co_3O_4 on an anode. (3)

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Information sheet

$$[X]_i = \left(\frac{V_{\text{aq}}}{V_{\text{org}}K + V_{\text{aq}}} \right)^i [X]_0$$

$$A_1 = \varepsilon_{M_1} b c_M + \varepsilon_{N_1} b c_N$$

$$A_2 = \varepsilon_{M_2} b c_M + \varepsilon_{N_2} b c_N$$

$$N = 16 \left(\frac{t_R}{W} \right)^2$$

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

$$k_A = \frac{t_R - t_M}{t_M}$$

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

$$N = 5.54 \left(\frac{t_R}{W_{1/2}} \right)^2$$

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

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

$$\text{Faraday} = 96\,485 \text{ C mol}^{-1}$$

Appendix 5

Standard and Formal Electrode Potentials

Half-Reaction	E°, V^*	Formal Potential, $V^\dagger$
Aluminum		
$\text{Al}^{3+} + 3e^- \rightleftharpoons \text{Al}(s)$	-1.662	
Antimony		
$\text{Sb}_2\text{O}_3(s) + 6\text{H}^+ + 4e^- \rightleftharpoons 2\text{SbO}^+ + 3\text{H}_2\text{O}$	+0.581	
Arsenic		
$\text{H}_3\text{AsO}_4 + 2\text{H}^+ + 2e^- \rightleftharpoons \text{H}_3\text{AsO}_3 + \text{H}_2\text{O}$	+0.559	0.577 in 1 M HCl, HClO_4
Barium		
$\text{Ba}^{2+} + 2e^- \rightleftharpoons \text{Ba}(s)$	-2.906	
Bismuth		
$\text{BiO}^+ + 2\text{H}^+ + 3e^- \rightleftharpoons \text{Bi}(s) + \text{H}_2\text{O}$	+0.320	
$\text{BiCl}_4^- + 3e^- \rightleftharpoons \text{Bi}(s) + 4\text{Cl}^-$	+0.16	
Bromine		
$\text{Br}_2(l) + 2e^- \rightleftharpoons 2\text{Br}^-$	+1.065	1.05 in 4 M HCl
$\text{Br}_2(aq) + 2e^- \rightleftharpoons 2\text{Br}^-$	+1.087*	
$\text{BrO}_3^- + 6\text{H}^+ + 5e^- \rightleftharpoons \frac{1}{2}\text{Br}_2(l) + 3\text{H}_2\text{O}$	+1.52	
$\text{BrO}_3^- + 6\text{H}^+ + 6e^- \rightleftharpoons \text{Br}^- + 3\text{H}_2\text{O}$	+1.44	
Cadmium		
$\text{Cd}^{2+} + 2e^- \rightleftharpoons \text{Cd}(s)$	-0.403	
Calcium		
$\text{Ca}^{2+} + 2e^- \rightleftharpoons \text{Ca}(s)$	-2.866	
Carbon		
$\text{C}_6\text{H}_4\text{O}_2$ (quinone) + $2\text{H}^+ + 2e^- \rightleftharpoons \text{C}_6\text{H}_4(\text{OH})_2$	+0.699	0.696 in 1 M HCl, HClO_4 , H_2SO_4
$2\text{CO}_2(g) + 2\text{H}^+ + 2e^- \rightleftharpoons \text{H}_2\text{C}_2\text{O}_4$	-0.49	
Cerium		
$\text{Ce}^{4+} + e^- \rightleftharpoons \text{Ce}^{3+}$		+1.70 in 1 M HClO_4 ; +1.61 in 1 M HNO_3 ; 1.44 in 1 M H_2SO_4
Chlorine		
$\text{Cl}_2(g) + 2e^- \rightleftharpoons 2\text{Cl}^-$	+1.359	
$\text{HClO} + \text{H}^+ + e^- \rightleftharpoons \frac{1}{2}\text{Cl}_2(g) + \text{H}_2\text{O}$	+1.63	
$\text{ClO}_3^- + 6\text{H}^+ + 5e^- \rightleftharpoons \frac{1}{2}\text{Cl}_2(g) + 3\text{H}_2\text{O}$	+1.47	
Chromium		
$\text{Cr}^{3+} + e^- \rightleftharpoons \text{Cr}^{2+}$	-0.408	
$\text{Cr}^{3+} + 3e^- \rightleftharpoons \text{Cr}(s)$	-0.744	
$\text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ + 6e^- \rightleftharpoons 2\text{Cr}^{3+} + 7\text{H}_2\text{O}$	+1.33	
Cobalt		
$\text{Co}^{2+} + 2e^- \rightleftharpoons \text{Co}(s)$	-0.277	
$\text{Co}^{3+} + e^- \rightleftharpoons \text{Co}^{2+}$	+1.808	
Copper		
$\text{Cu}^{2+} + 2e^- \rightleftharpoons \text{Cu}(s)$	+0.337	
$\text{Cu}^{2+} + e^- \rightleftharpoons \text{Cu}^+$	+0.153	
$\text{Cu}^+ + e^- \rightleftharpoons \text{Cu}(s)$	+0.521	
$\text{Cu}^{2+} + \text{I}^- + e^- \rightleftharpoons \text{CuI}(s)$	+0.86	
$\text{CuI}(s) + e^- \rightleftharpoons \text{Cu}(s) + \text{I}^-$	-0.185	

continues

Half-Reaction	E°, V^*	Formal Potential, $V^\dagger$
Fluorine		
$F_2(g) + 2H^+ + 2e^- \rightleftharpoons 2HF(aq)$	+3.06	
Hydrogen		
$2H^+ + 2e^- \rightleftharpoons H_2(g)$	0.000	-0.005 in 1 M HCl, HClO ₄
Iodine		
$I_2(s) + 2e^- \rightleftharpoons 2I^-$	+0.5355	
$I_2(aq) + 2e^- \rightleftharpoons 2I^-$	+0.615 [‡]	
$I_3^- + 2e^- \rightleftharpoons 3I^-$	+0.536	
$ICl_2^- + e^- \rightleftharpoons \frac{1}{2}I_2(s) + 2Cl^-$	+1.056	
$IO_3^- + 6H^+ + 5e^- \rightleftharpoons \frac{1}{2}I_2(s) + 3H_2O$	+1.196	
$IO_3^- + 6H^+ + 5e^- \rightleftharpoons \frac{1}{2}I_2(aq) + 3H_2O$	+1.178 [‡]	
$IO_3^- + 2Cl^- + 6H^+ + 4e^- \rightleftharpoons ICl_2^- + 3H_2O$	+1.24	
$H_5IO_6 + H^+ + 2e^- \rightleftharpoons IO_3^- + 3H_2O$	+1.601	
Iron		
$Fe^{2+} + 2e^- \rightleftharpoons Fe(s)$	-0.440	
$Fe^{3+} + e^- \rightleftharpoons Fe^{2+}$	+0.771	0.700 in 1 M HCl; 0.732 in 1 M HClO ₄ ; 0.68 in 1 M H ₂ SO ₄
$Fe(CN)_6^{3-} + e^- \rightleftharpoons Fe(CN)_6^{4-}$	+0.36	0.71 in 1 M HCl; 0.72 in 1 M HClO ₄ , H ₂ SO ₄
Lead		
$Pb^{2+} + 2e^- \rightleftharpoons Pb(s)$	-0.126	-0.14 in 1 M HClO ₄ ; -0.29 in 1 M H ₂ SO ₄
$PbO_2(s) + 4H^+ + 2e^- \rightleftharpoons Pb^{2+} + 2H_2O$	+1.455	
$PbSO_4(s) + 2e^- \rightleftharpoons Pb(s) + SO_4^{2-}$	-0.350	
Lithium		
$Li^+ + e^- \rightleftharpoons Li(s)$	-3.045	
Magnesium		
$Mg^{2+} + 2e^- \rightleftharpoons Mg(s)$	-2.363	
Manganese		
$Mn^{2+} + 2e^- \rightleftharpoons Mn(s)$	-1.180	
$Mn^{3+} + e^- \rightleftharpoons Mn^{2+}$		1.51 in 7.5 M H ₂ SO ₄
$MnO_2(s) + 4H^+ + 2e^- \rightleftharpoons Mn^{2+} + 2H_2O$	+1.23	
$MnO_4^- + 8H^+ + 5e^- \rightleftharpoons Mn^{2+} + 4H_2O$	+1.51	
$MnO_4^- + 4H^+ + 3e^- \rightleftharpoons MnO_2(s) + 2H_2O$	+1.695	
$MnO_4^- + e^- \rightleftharpoons MnO_4^{2-}$	+0.564	
Mercury		
$Hg_2^{2+} + 2e^- \rightleftharpoons 2Hg(l)$	+0.788	0.274 in 1 M HCl; 0.776 in 1 M HClO ₄ ; 0.674 in 1 M H ₂ SO ₄
$2Hg^{2+} + 2e^- \rightleftharpoons Hg_2^{2+}$	+0.920	0.907 in 1 M HClO ₄
$Hg^{2+} + 2e^- \rightleftharpoons Hg(l)$	+0.854	
$Hg_2Cl_2(s) + 2e^- \rightleftharpoons 2Hg(l) + 2Cl^-$	+0.268	0.244 in sat'd KCl; 0.282 in 1 M KCl; 0.334 in 0.1 M KCl
$Hg_2SO_4(s) + 2e^- \rightleftharpoons 2Hg(l) + SO_4^{2-}$	+0.615	
Nickel		
$Ni^{2+} + 2e^- \rightleftharpoons Ni(s)$	-0.250	
Nitrogen		
$N_2(g) + 5H^+ + 4e^- \rightleftharpoons N_2H_5^+$	-0.23	
$HNO_2 + H^+ + e^- \rightleftharpoons NO(g) + H_2O$	+1.00	
$NO_3^- + 3H^+ + 2e^- \rightleftharpoons HNO_2 + H_2O$	+0.94	0.92 in 1 M HNO ₃
Oxygen		
$H_2O_2 + 2H^+ + 2e^- \rightleftharpoons 2H_2O$	+1.776	
$HO_2^- + H_2O + 2e^- \rightleftharpoons 3OH^-$	+0.88	
$O_2(g) + 4H^+ + 4e^- \rightleftharpoons 2H_2O$	+1.229	
$O_2(g) + 2H^+ + 2e^- \rightleftharpoons H_2O_2$	+0.682	
$O_3(g) + 2H^+ + 2e^- \rightleftharpoons O_2(g) + H_2O$	+2.07	
Palladium		
$Pd^{2+} + 2e^- \rightleftharpoons Pd(s)$	+0.987	

continues

Half-Reaction	E°, V^*	Formal Potential, V^+
Platinum		
$\text{PtCl}_4^{2-} + 2e^- \rightleftharpoons \text{Pt}(s) + 4\text{Cl}^-$	+0.755	
$\text{PtCl}_6^{2-} + 2e^- \rightleftharpoons \text{PtCl}_4^{2-} + 2\text{Cl}^-$	+0.68	
Potassium		
$\text{K}^+ + e^- \rightleftharpoons \text{K}(s)$	-2.925	
Selenium		
$\text{H}_2\text{SeO}_3 + 4\text{H}^+ + 4e^- \rightleftharpoons \text{Se}(s) + 3\text{H}_2\text{O}$	+0.740	
$\text{SeO}_4^{2-} + 4\text{H}^+ + 2e^- \rightleftharpoons \text{H}_2\text{SeO}_3 + \text{H}_2\text{O}$	+1.15	
Silver		
$\text{Ag}^+ + e^- \rightleftharpoons \text{Ag}(s)$	+0.799	0.228 in 1 M HCl; 0.792 in 1 M HClO_4 ; 0.77 in 1 M H_2SO_4
$\text{AgBr}(s) + e^- \rightleftharpoons \text{Ag}(s) + \text{Br}^-$	+0.073	
$\text{AgCl}(s) + e^- \rightleftharpoons \text{Ag}(s) + \text{Cl}^-$	+0.222	0.228 in 1 M KCl
$\text{Ag}(\text{CN})_2^- + e^- \rightleftharpoons \text{Ag}(s) + 2\text{CN}^-$	-0.31	
$\text{Ag}_2\text{CrO}_4(s) + 2e^- \rightleftharpoons 2\text{Ag}(s) + \text{CrO}_4^{2-}$	+0.446	
$\text{AgI}(s) + e^- \rightleftharpoons \text{Ag}(s) + \text{I}^-$	-0.151	
$\text{Ag}(\text{S}_2\text{O}_3)_2^{3-} + e^- \rightleftharpoons \text{Ag}(s) + 2\text{S}_2\text{O}_3^{2-}$	+0.017	
Sodium		
$\text{Na}^+ + e^- \rightleftharpoons \text{Na}(s)$	-2.714	
Sulfur		
$\text{S}(s) + 2\text{H}^+ + 2e^- \rightleftharpoons \text{H}_2\text{S}(g)$	+0.141	
$\text{H}_2\text{SO}_3 + 4\text{H}^+ + 4e^- \rightleftharpoons \text{S}(s) + 3\text{H}_2\text{O}$	+0.450	
$\text{SO}_4^{2-} + 4\text{H}^+ + 2e^- \rightleftharpoons \text{H}_2\text{SO}_3 + \text{H}_2\text{O}$	+0.172	
$\text{S}_4\text{O}_6^{2-} + 2e^- \rightleftharpoons 2\text{S}_2\text{O}_3^{2-}$	+0.08	
$\text{S}_2\text{O}_8^{2-} + 2e^- \rightleftharpoons 2\text{SO}_4^{2-}$	+2.01	
Thallium		
$\text{Tl}^+ + e^- \rightleftharpoons \text{Tl}(s)$	-0.336	-0.551 in 1 M HCl; -0.33 in 1 M HClO_4 , H_2SO_4
$\text{Tl}^{3+} + 2e^- \rightleftharpoons \text{Tl}^+$	+1.25	0.77 in 1 M HCl
Tin		
$\text{Sn}^{2+} + 2e^- \rightleftharpoons \text{Sn}(s)$	-0.136	-0.16 in 1 M HClO_4
$\text{Sn}^{4+} + 2e^- \rightleftharpoons \text{Sn}^{2+}$	+0.154	0.14 in 1 M HCl
Titanium		
$\text{Ti}^{3+} + e^- \rightleftharpoons \text{Ti}^{2+}$	-0.369	
$\text{TiO}^{2+} + 2\text{H}^+ + e^- \rightleftharpoons \text{Ti}^{3+} + \text{H}_2\text{O}$	+0.099	0.04 in 1 M H_2SO_4
Uranium		
$\text{UO}_2^{2+} + 4\text{H}^+ + 2e^- \rightleftharpoons \text{U}^{4+} + 2\text{H}_2\text{O}$	+0.334	
Vanadium		
$\text{V}^{3+} + e^- \rightleftharpoons \text{V}^{2+}$	-0.255	
$\text{VO}^{2+} + 2\text{H}^+ + e^- \rightleftharpoons \text{V}^{3+} + \text{H}_2\text{O}$	+0.337	
$\text{V}(\text{OH})_4^+ + 2\text{H}^+ + e^- \rightleftharpoons \text{VO}^{2+} + 3\text{H}_2\text{O}$	+1.00	1.02 in 1 M HCl, HClO_4
Zinc		
$\text{Zn}^{2+} + 2e^- \rightleftharpoons \text{Zn}(s)$	-0.763	

Appendix 2

Solubility Product Constants at 25°C

Compound	Formula	K_{sp}	Notes
Aluminum hydroxide	$\text{Al}(\text{OH})_3$	3×10^{-34}	
Barium carbonate	BaCO_3	5.0×10^{-9}	
Barium chromate	BaCrO_4	2.1×10^{-10}	
Barium hydroxide	$\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$	3×10^{-4}	
Barium iodate	$\text{Ba}(\text{IO}_3)_2$	1.57×10^{-9}	
Barium oxalate	BaC_2O_4	1×10^{-6}	
Barium sulfate	BaSO_4	1.1×10^{-10}	
Cadmium carbonate	CdCO_3	1.8×10^{-14}	
Cadmium hydroxide	$\text{Cd}(\text{OH})_2$	4.5×10^{-15}	
Cadmium oxalate	CdC_2O_4	9×10^{-8}	
Cadmium sulfide	CdS	1×10^{-27}	
Calcium carbonate	CaCO_3	4.5×10^{-9}	Calcite
	CaCO_3	6.0×10^{-9}	Aragonite
Calcium fluoride	CaF_2	3.9×10^{-11}	
Calcium hydroxide	$\text{Ca}(\text{OH})_2$	6.5×10^{-6}	
Calcium oxalate	$\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$	1.7×10^{-9}	
Calcium sulfate	CaSO_4	2.4×10^{-5}	
Cobalt(II) carbonate	CoCO_3	1.0×10^{-10}	
Cobalt(II) hydroxide	$\text{Co}(\text{OH})_2$	1.3×10^{-15}	
Cobalt(II) sulfide	CoS	5×10^{-22}	α
	CoS	3×10^{-26}	β
Copper(I) bromide	CuBr	5×10^{-9}	
Copper(I) chloride	CuCl	1.9×10^{-7}	
Copper(I) hydroxide*	Cu_2O^*	2×10^{-15}	
Copper(I) iodide	CuI	1×10^{-12}	
Copper(I) thiocyanate	CuSCN	4.0×10^{-14}	
Copper(II) hydroxide	$\text{Cu}(\text{OH})_2$	4.8×10^{-20}	
Copper(II) sulfide	CuS	8×10^{-37}	
Iron(II) carbonate	FeCO_3	2.1×10^{-11}	
Iron(II) hydroxide	$\text{Fe}(\text{OH})_2$	4.1×10^{-15}	
Iron(II) sulfide	FeS	8×10^{-19}	
Iron(III) hydroxide	$\text{Fe}(\text{OH})_3$	2×10^{-39}	
Lanthanum iodate	$\text{La}(\text{IO}_3)_3$	1.0×10^{-11}	
Lead carbonate	PbCO_3	7.4×10^{-14}	
Lead chloride	PbCl_2	1.7×10^{-5}	
Lead chromate	PbCrO_4	3×10^{-13}	
Lead hydroxide	PbOH^+	8×10^{-16}	Yellow
	PbO^+	5×10^{-16}	Red
Lead iodide	PbI_2	7.9×10^{-9}	
Lead oxalate	PbC_2O_4	8.5×10^{-9}	$\mu = 0.05$
Lead sulfate	PbSO_4	1.6×10^{-8}	
Lead sulfide	PbS	3×10^{-28}	
Magnesium ammonium phosphate	MgNH_4PO_4	3×10^{-13}	
Magnesium carbonate	MgCO_3	3.5×10^{-8}	

continues

The Periodic Table

[illegible]