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APPENDICES

APPENDIX A

CALCULATION METHODS

A.1 CALCULATION OF ACTIVE AREA AND SPECIFIC AREA OF CATHODE

A.1.1 Stainless steel cathode with spherical hole. It has the following dimension

Width (l)		0.1	m
Length (L)		0.08	m
Thickness (t)		0.001	m
Length of reactor (L_R)		0.09	m
Hole	- Hole number (n)	741	hole
	- Diameter (f)	0.002	m

The active area of cathode

$$= [(2 \times l \times L) + (2 \times l \times t) + (2 \times L \times t)] - (2 \times \frac{\pi}{4} \times f^2 \times n) + (\pi \times f \times t \times n)$$

$$= [(2 \times 0.1 \times 0.08) + (2 \times 0.1 \times 0.001) + (2 \times 0.08 \times 0.001)] - (2 \times \frac{\pi}{4} \times 0.002 \times 0.002 \times 741) + (\pi \times 0.002 \times 0.001 \times 741)$$

$$= 163.6 \times 10^{-4} \quad m^2$$

The specific area

$$= \frac{(L \times l) - (\frac{\pi}{4} \times f^2 \times n) + (\pi \times f \times t \times n)}{L \times L_R \times l}$$

$$= \frac{(0.08 \times 0.1) - (\frac{\pi}{4} \times 0.002 \times 0.002 \times 741) + (\pi \times 0.002 \times 0.001 \times 741)}{0.08 \times 0.09 \times 0.1}$$

$$= 14.3 \quad m^{-1}$$

A.1.2 Zinc plate cathode. It has the following dimension

Width (l)	0.1	M
Length (L)	0.082	m

The active area of cathode

$$= l \times L$$

$$= 0.1 \times 0.082$$

$$= 82 \times 10^{-4} \quad m^2$$

A.1.3 Stainless cathode with square hole. It has the following dimension

Width (l)	0.088	m
Length (L)	0.088	m
Thickness (t)	0.001	m
Hole - Hole number (n)	49	hole
- Dimension of square hole (f)	0.008	m

The active area of cathode $= [(2 \times l \times L) + (2 \times l \times t) + (2 \times L \times t)] - (2 \times f^2 \times n) + (4 \times f \times t \times n)$

$= [(2 \times 0.088 \times 0.088) + (2 \times 0.088 \times 0.001) + (2 \times 0.088 \times 0.001)] - (2 \times 0.008 \times 0.008 \times 49) + (4 \times 0.008 \times 0.001 \times 49) = 111.5 \times 10^{-4}$ m^2

A.2 Calculation of current density, recovery percentage and current efficiency

Given			
	Applied current (i)	0.16	A
	Active area (A_e)	0.016	m^2
	Initial metal weight (W_i)	0.971	g
	Metal weight at time t (W_t)	0.164	g
	Number of electron (n)	2	equivalent / mol
	Faraday constant (F)	26.8	A-h/equivalent
	Molecular weight of metal (M_W)	63.5	g/mol
	Electrolysis time (t)	6	hr

Current density (j) $= \dfrac{i}{A_e}$

$$= \frac{0.16}{0.0016} = 10 \quad A/m^2$$

Recovery percentage ($\% R$) $= \left[\dfrac{W_i - W_t}{W_t}\right] \times 100$

$$= \left[\frac{0.971 - 0.164}{0.971}\right] \times 100 = 83.11$$

Current efficiency (ϕ) $= \dfrac{n \times F \times W_i \times (\%R)}{100 \times M_W \times i \times t} \times 100$

At 83.11 % recovery

Current efficiency $$= \frac{2 \times 26.8 \times 0.971 \times 83.11}{100 \times 63.5 \times 0.16 \times 6} \times 100$$

$$= \frac{4325.51}{6096} \times 100 \qquad = 70.96\ \%$$

A.3 Calculation of mass transfer coefficient, k_L

The mass transfer coefficient is calculated by a trial & error method as shown in Chapter III. For example,

Given	Electrolyte flow rate (*Q*)	3	l/min
	Current density (*j*)	10	A/m^2
	Number of electron (*n*)	2	equivalent / mol
	Faraday constant (*F*)	96500	A-s / equivalent
	Specific area (a_e')	14.3	m^{-1}
	Molecular weight of metal (M_W)	63.5	g/mol
	Initial concentration (C_0)	16.21	mol/l
	Electrolysis time (*t*)		

and suppose k_L is 2.03×10^{-5} m/s, then

$$C^* = \frac{j}{k_L \times n \times F}$$

$$= \frac{10}{2.03 \times 10^{-5} \times 2 \times 96500} = 2.55 \quad \text{mol/l}$$

$$t^* = \frac{(C_0 - C^*) \times n \times F}{j \times a_e'}$$

$$= \frac{(16.21 - 2.55) \times 2 \times 96500}{10 \times 14.3 \times 60} = 307.3 \quad \text{min}$$

$$C_{av} = \frac{C_{exp} + C_1}{2}$$

At $t < t^*$: $$C_1 = C_0 - \frac{(j \times a_e' \times t)}{n \times F}$$

At $t > t^*$: $$= C^* \exp[-k_L \times a_e' \times (t - t^*)]$$

At $t = 0$ min: $$C_l = 16.21 - \frac{(10 \times 14.3 \times 0 \times 60)}{2 \times 96500} = 16.21$$

$$C_{av} = \frac{16.21 + 16.21}{2} = 16.21$$

$$\left(\frac{C_l - C_{exp}}{C_{av}}\right)^2 = \left(\frac{16.21 - 16.21}{16.21}\right)^2 = 0$$

The example calculation is expressed below

Time (min)	C_{exp} (mol/l)	t^* (min)	k_L (m/s)	C_l (mol/l)	C_{av} (mol/l)	$\left(\frac{C_l - C_{exp}}{C_{av}}\right)^2$
0	16.21	307.3	2.03×10^{-5}	16.21	16.21	0.00000
30	14.81	307.3	2.03×10^{-5}	14.88	14.85	0.00002
60	13.57	307.3	2.03×10^{-5}	13.55	13.56	0.00000
90	12.52	307.3	2.03×10^{-5}	12.21	12.36	0.00060
120	10.90	307.3	2.03×10^{-5}	10.88	10.89	0.00000
180	8.60	307.3	2.03×10^{-5}	8.21	8.41	0.00217
240	6.19	307.3	2.03×10^{-5}	5.54	5.87	0.01218
300	3.34	307.3	2.03×10^{-5}	2.88	3.11	0.02248
360	1.13	307.3	2.03×10^{-5}	1.02	1.07	0.01049
420	0.34	307.3	2.03×10^{-5}	0.36	0.35	0.00255
						$\sum = 0.0549$

$$S = \sqrt{\sum\left(\frac{C_l - C_{exp}}{C_{av}}\right)^2} = \sqrt{0.0549}$$

$$= 0.22471$$

Changing the value of k_L until the minimum S is obtained.

A.4 Calculation of dimensionless number

A.4.1 Dimensionless group

Given	Gap inter electrode (h)	0.052	m
	Width (l)	0.1	m
	Cross sectional area of reactor (A_R)	8×10^{-3}	m^2
	Diffusion coefficient (D_a)	5.89×10^{-10}	m^2/s
	Electrolyte density (ρ)	1005.6	kg/m^3
	Electrolyte viscosity (μ)	9.29×10^{-4}	kg/(m·s)
	Electrolyte flow rate (Q)	0.934	l/min
	Mass transfer coefficient (k_L)	1.25×10^{-5}	m/s

Hydraulic diameter (d_h)

$$= \frac{2 \times l \times h}{h + l}$$

$$= \frac{2 \times 0.1 \times 0.052}{0.052 + 0.1} = 0.0684 \quad \text{m}$$

Electrolyte velocity (U)

$$= \frac{Q}{A_R}$$

$$= \frac{0.934}{1000 \times 8 \times 10^{-3}} = 0.117 \quad \text{m/min}$$

Reynolds number (Re)

$$= \frac{\rho \times d_h \times U}{\mu}$$

$$= \frac{1005.6 \times 0.0684 \times 0.117}{9.29 \times 10^{-4} \times 60} = 144$$

Sherwood number (Sh)

$$= \frac{k_L \times d_h}{D_a}$$

$$= \frac{1.25 \times 10^{-5} \times 0.0684}{5.89 \times 10^{-10}} = 1452$$

Schmidt number (Sc)

$$= \frac{\mu}{\rho \times D_a}$$

$$= \frac{9.29 \times 10^{-4}}{1005.6 \times 5.89 \times 10^{-4}} = = 1568$$

A.4.2 Dimensionless equation

The dimensionless equation can be expressed in terms of Sherwood number, Reynolds number and Schmidt number.

$$Sh \quad = \quad a\,Re^{b}\,Sc^{c} \quad = \quad a\,Re^{b}\,Sc^{1/3}$$

When Sc is 1568. If we plot between log ($Sh / Sc^{1/3}$) and log Re, the slope is b and intercept is log a. The following table presents the example calculation of dimensionless equation.

Q (l/min)	U (m/min)	k_L (m/s)	Re	Sh	Log (Re)	$\frac{Sh}{Sc^{1/3}}$	$\mathrm{Log}(\frac{Sh}{Sc^{1/3}})$
0.93	0.117	1.25×10^{-5}	144	1452	2.16	128.	2.11
1.54	0.192	1.34×10^{-5}	237	1556	2.38	137	2.14
1.94	0.242	1.39×10^{-5}	299	1614	2.48	142	2.15

The plot between log ($Sh / Sc^{1/3}$) versus log Re give the following data.

Slope = 0.145 = b

Intercept = 1.795 = log (a)

Then b = 0.145

a = 62

So Sh = $62\ \mathrm{Re}^{0.145}\ \mathrm{Sc}^{1/3}$

A.5 Calculation of developed model error

This model error is determined by the difference of actual specific area and those calculated from the developed model.

Given	Current density (j)	10	A/m^2
	Number of electron (n)	2	equivalent/mol
	Faraday constant (F)	96500	A-s/equivalent
	Actual specific area (a_e')	14.3	m^{-1}

From $C_t = C_0 - \dfrac{(j \times a_e' \times t)}{n \times F}$

The plot of (C_t - C_O) versus t gives

$$\text{slope} = -\frac{j \times a_e'}{n \times F}$$

Example

Time (min)	C (mol/m^3)	C_t - C_O
0	15.84	0
30	14.15	1.69
60	12.68	3.15
90	11.14	4.69
120	9.97	5.87
180	7.30	8.54
240	4.84	11.00
300	2.37	13.47

$$\text{Slope} = 0.0443 = -\frac{j \times a_e'}{n \times F}$$

Then

$$a_e' = \frac{n \times F}{j} \times \text{Slope}$$

$$= \frac{2 \times 96500}{10 \times 60} \times 0.0443$$

$$= 14.25 \quad m^{-1}$$

A.6 Material balance between cathodic and anodic compartment

Given			
	Applied current	1	A
	Volume in cathodic (anodic) part (V)	0.5	liter
	Number of electron (n)	1	equivalent / mol
	Faraday constant (F)	96500	A-s / equivalent
	Experimental pH (pH_{exp})	1	
	Electrolysis time (t)	0	min
	Area of membrane (A)	0.0077	m^2

A.6.1 Material balance in cathodic compartment

This balance was performed by proton balance at time t. The flux of proton is the amount of proton that should be reacted with hydroxide ion in this compartment.

Mole of proton measured during experiment (n_{H+exp}) = $V \times 10^{-pH_{exp}}$

Mole of proton consumption ($n_{H+consumed}$) = $\dfrac{i \times t}{n \times F}$

Mole of proton difference in time interval ($n_{H+dif\,int}$) = $n_{H+consumed\ at\ t1} - n_{H+consumed\ at\ t}$

Mole of theory proton ($n_{H+theory}$) = $n_{H+exp\ at\ t} - n_{H+dif\,int\ at\ t+1}$

Mole of proton difference (n_{H+diff}) = $n_{H+theory} - n_{H+exp}$

Time difference (t_{diff}) = $t_1 - t$

Loss flux of proton (N_{H+}) = $\dfrac{n_{H+diff}}{t_{diff} \times A}$

The following table shows the example calculation of pH and loss flux of proton in cathodic compartment.

Time (min)	pHexp	n_{H+exp} (mol)	$n_{H+consumed}$ (mol)	$n_{H+dif\,int}$ (mol)	$n_{H+theory}$ (mol)	n_{H+diff} (mol)	t_{diff} (min)	N_{H+} (mol/m^{2}s)
0	1	0.05	0		0.05	0	0	
60	1.23	0.0294	0.0373	0.0373	0.0127	-0.0167	60	-6.01×10^{-4}
120	1.67	0.0107	0.0746	0.0373	-0.0079	-0.0186	60	-6.66×10^{-4}
150	2.25	0.0028	0.0933	0.0187	-0.0080	-0.0108	30	-7.73×10^{-4}
180	2.90	0.0006	0.1119	0.0187	-0.0158	-0.0165	30	-1.18×10^{-3}
195	3.21	3.08×10^{-4}	0.1212	0.0093	-0.0087	-0.0090	15	-1.29×10^{-3}
210	6.35	2.23×10^{-7}	0.1306	0.0093	-0.0090	-0.0090	15	-1.29×10^{-3}
225	11.72	9.53×10^{-13}	0.1399	0.0093	-0.0093	-0.0093	15	-1.34×10^{-3}
								-1.02×10^{-3}

A.6.2 Material balance in anodic compartment

The material balance was performed by proton balance at time t. From this balance, the flux of proton indicates the loss of proton from anodic compartment which is corresponding to the amount of hydroxide ion coming from cathodic compartment to this compartment.

Mole of proton measured during experiment (n_{H+exp}) = $V \times 10^{-pH_{exp}}$

Mole of proton production ($n_{H+produced}$) = $\dfrac{i \times t}{n \times F}$

Mole of proton difference in time interval ($n_{H+dif\,int}$) = $n_{H+produced\ at\ t1} - n_{H+produced\ at\ t}$

Mole of theory proton ($n_{H+theory}$) = $n_{H+exp\ at\ t} + n_{H+dif\,int\ at\ t+1}$

Mole of proton difference (n_{H+diff}) = $n_{H+theory} - n_{H+exp}$

Time difference (t_{diff}) = $t_1 - t$

Loss flux of proton (N_{H+}) = $\dfrac{n_{H+diff}}{t_{diff} \times A}$

Time (min)	pH_{exp}	n_{H+exp} (mol)	$n_{H+produced}$ (mol)	$n_{H+dif\,int}$ (mol)	$n_{H+theory}$ (mol)	n_{H+diff} (mol)	t_{diff} (min)	N_{H+} (mol/m^{2}s)
0	1	0.05	0		0.05	0	0	
60	0.98	0.0524	0.0373	0.0373	0.0873	0.0349	60	1.25×10^{-3}
120	0.89	0.0644	0.0746	0.0373	0.0897	0.0252	60	9.06×10^{-4}
150	0.83	0.0740	0.0933	0.0187	0.0831	0.0091	30	6.54×10^{-4}
180	0.78	0.0830	0.1119	0.0187	0.0926	0.0096	30	6.91×10^{-4}
195	0.77	0.0849	0.1212	0.0093	0.0923	0.0074	15	1.06×10^{-3}
210	0.74	0.0910	0.1306	0.0093	0.0942	0.0033	15	4.67×10^{-4}
225	0.70	0.0998	0.1399	0.0093	0.1003	0.0005	15	7.87×10^{-5}
								0.73×10^{-3}

A.7 Derivation of pH evolution modelling

Example: Derivation of the second model of chromium recovery

Given	initial pH	pHi
	Current	i
	Electrolysis time	t
	Latent time	t_1
	Precipitation time	t_2
	Number of electron	n
	Faraday constant	F
	Volume of solution	l
	Initial chromium concentration	$[Cr]_i$

Proton production in the system, $[H^+] \quad = \quad \dfrac{i \times t}{n \times F \times V}$

1st zone model:

From the basic equation: $pH \quad = \quad -\log [H^+]$

The increasing of pH in the system is calculated by

$$pH_{m2} \quad = \quad -\log \text{[initial proton - proton production in the system]}$$

$$pH_{m2} \quad = \quad -\log \left[10^{-pH_i} - \frac{i \times t}{n \times F \times V} \right]$$

This equation is sufficient when $t < t_1$. If $t = t_1$, it indicates that the amount of proton disappearance from the system equals to the amount of initial proton.

$$10^{-pHi} \quad = \quad \frac{i \times t_1}{n \times F \times V}$$

$$t_1 \quad = \quad 10^{-pH_i} \frac{n \times F \times V}{i}$$

2nd zone model

From $\quad Cr^{3+} + 3OH^{-}- \longrightarrow Cr(OH)_3$

Mole of chromium ion consumption $\quad = \quad (\frac{1}{3}) \times$ mole OH^-

$$\text{Chromium consumption (mole/l)} \quad = \quad \left(\frac{1}{3}\right) \times \frac{i \times t}{n \times F \times V}$$

$$\text{Chromium consumption (g/l)} \quad = \quad \left(\frac{52}{3}\right) \times \frac{i \times t}{n \times F \times V}$$

From experimental data

$$pH_{exp} \quad = \quad \frac{[Cr] - 4.01}{-0.67} \qquad \frac{4.01 - [Cr]}{0.67}$$

$$pH_{m2\text{-}exp} \quad = \quad (4.01 - [\text{initial chromium - chromium consumption}]) \, / \, 0.67$$

$$pH_{m2\text{-}exp} \quad = \quad \left[4.01 - [Cr]_i + \frac{52 \times i \times (t - t_1)}{3 \times n \times F \times V}\right] / \, 0.67$$

This equation is not sufficient when chromium consumption by equation equals to initial chromium concentration or $t = t_2$

$$[Cr]_i \quad = \quad \frac{52 \times i \times (t_2 - t_1)}{3 \times n \times F \times V}$$

$$t_2 \quad = \quad t_1 + \frac{3 \times n \times F \times V}{52 \times i}$$

3^{rd} zone model

This zone is corresponding to the increasing of pH by OH^- which is produced by water reduction reaction.

From

$$pH_{m2} \quad = \quad -\log [H^+]$$

$$= \quad -\log \frac{10^{-14}}{[OH-]}$$

$$= \quad -\log \frac{10^{-14}}{\left[\frac{i \times (t-t_2)}{n \times F \times V}\right]}$$

$$pH_{m2} \quad = \quad -\log 10^{-14} \left[\frac{n \times F \times V}{i \times (t - t_2)}\right]$$

This equation is used to predict the pH evolution at $t > t_2$

A.8 Calculation of total operating cost

Example: Data from Plant II solution

Given			
	Electricity cost	2.863	Baht/kW-hr
	Total solution volume	0.5	liter
	Cost of copper (1 kgCu = 1.366 US$)	59.42	Baht/kg Cu
	Land fill cost	0.874	Baht/kg

For copper recovery

- Current intensity (*i*)	0.11	A
- Voltage (*V*)	3	V
- Electrolysis time (*t*)	12	hr
- Recovery weight (*Rw*)	0.929	kg/m^3

For chromium and nickel

- Current intensity (*i*)	1.0	A
- Voltage (*V*)	15.5	V
- Electrolysis time (*t*)	3	hr
- Sludge weight of chromium and nickel (*Ws*)	2.647	kg/m^3

Total electricity consumption = $[(i \times V \times t)_{Cu} + (i \times V \times t)]_{Cr,Ni}$

= $[(0.11 \times 3 \times 12) + (1.0 \times 15.5 \times 3)]/1000$ kW-hr

= 0.05046 kW-hr

= $(0.05046 \times 1000)/0.5$ $kW\text{-}hr/m^3$

= 100.92 $kW\text{-}hr/m^3$

Total electricity cost = Total electricity consumption × Electricity cost

= $100.92 \times 2.863 = 288.93$ $Baht/m^3$

Recovery cost (*Rc*) = $Rc_{Cu} = Rw \times \text{Cost}$

= $0.929 \times 59.42 = 55.20$ $Baht/m^3$

Total land fill cost of sludge = *Ws* × Land fill cost

= $2.647 \times 0.874 = 2.313$ $Baht/m^3$

Total operating cost = Electricity cost - Recovery cost + Total land fill cost

= $288.93 - 55.20 + 2.313$

= 236.04 $Baht/m^3$

= 5.43 $US\$/m^3$

APPENDIX B

ANALYTICAL METHODS

B.1 Metal concentration measurement by Atomic Absorption Spectroscopy (AA) [69, 70]

The technique of Atomic Absorption developed by Sir Alan Walsh of C.S.I.R.O. in mid 1950's has become the preferred method of elemental analysis. Walsh discovered that the majority of free atoms in the commonly used flames were in the ground state. A light source emitting a narrow spectral line of the characteristics energy is used to excite the free atoms formed in the flame. The decrease in energy (absorption) is then measured. The absorption is proportional to the concentration of free atoms in the flame, given by Lambert - Beer law

$$\text{Absorption} = \log \frac{i_0}{i_t} = k.c.L.$$

where	i_0	intensity of incident radiation emitted by the light source
	i_t	intensity of transmitted radiation (amount not absorbed)
	c	concentration of sample (free atom)
	k	constant (can be determined experimentally)
	L	path length

B.1.1 Atomic absorption instrument

-Hollow cathode lamps: It is used as the radiation source to excite the free atoms in the flame. The hollow cathode lamp produces a narrow spectral line characteristics of the material of the cathode and the fill gas (neon or argon).

-Flame atomization: The flame atomization consists of three major components, a nebulizer, a spray chamber and a burner.

a. *Nebulizer* used in atomic absorption is the pneumatic type in which the flow of gasses through the orifice creates a vacuum drawing the sample into the capillary.

b. *Spray chamber* acts as a classifier to remove large particles and supply a constant feed of fine particles to the burner.

c. *Burners* is a laminar flow premix burner with long path length.

B.1.2 Type of flame

a. Air - acetylene flame: This is the most widely used flame in atomic absorption spectroscopy. Four distinguishable flame are used

- Oxidizing flame (lean) - a very stiff flame with small inner blue cone
- Stoichiometric flame - a stiff flame with bigger blue cone and an almost luminous appearance
- Luminous flame - a luminous flame with prominent blue cone.
- Reducing flame (rich) - luminous flame almost sooty at the tope.

b. Nitrous oxide-acetylene flame: This is a much hotter (approximately 2,900°C) and faster burning flame and should be used with a nitrous oxide - acetylene burner. Four types of flame are distinguished by

- Oxidizing flame (lean) - a very stiff flame in which the red feather is 1 cm or less
- Stoichiometric flame - a clear flame with evidence of slight yellow at the junction of the read feather with is 1 - 2 cm.
- Reducing flame (rich) - a yellow flame with the read feather just visible.
- Very reducing flame - a flame that is extremely luminous with almost the formation of soot.

B.1.3 Analytical procedure

a. Calibration method: This method has no interferent effect. Usually at least three standards and blank are used to cover the range 0.1 to 0.8 Abs. The calibration is performed by using the blank solution to zero the instrument.

The standard is analysed with the lowest concentration first, and the blank run between standards, to ensure the baseline (zero point) has not changed. Sample are analysed and their absorbances are recorded.

b. Ultimate precision method: Two standard solutions are made by giving absorption very similar to the sample, in the range of 0.4 to 0.6 Abs. (one slightly below and one slightly above the sample absorbance). Zero is set using the low standard and 1.000 g/l concentration with the height standard. The difference between the standard is small (say 0.10 Abs.), a very accurate reading can be made and a precision of 0.1 % to 0.2 % is possible.

c. Standard method addition: The method relies on the addition of a small known amount of standard to an aliquot of sample.

B.1.4 Analytical data of the elements

Copper

Reagents for standard preparation

Aqueous: -Copper metal foil or wire (99.99%)

Non aqueous: - Copper 4-cyclohexylbutyrate

Preparation of 1,000 mg/l standard: Dissolve 1.0000 g of copper in 50 ml of 6N nitric acid and dilute to 1 litre

Atomic absorbance

Lamp current: - 3.0 mA

Flame type: - Air-acetylene (oxidizing)

Optimum condition

Wave length (nm)	Slit width (nm)	Working range (mg/l)	Sensitivity (mg/l)
324.7	0.5	1 - 5	0.025
327.4	0.5	2.5 - 10	0.050
217.9	0.2	7.5 - 30	0.16
222.6	1.0	45 - 180	1.0
249.2	0.5	180 - 730	4.0
244.2	1.0	400 - 1,700	9.0

Interferences

Few interferences have been reported for copper

Flame emission

Wave length: -327.4 nm

Slit width: - 0.2 nm

Flame type: - Nitrous oxide - acetylene

Chromium

Reagents for standards preparation

Aqueous: -Chromium metal foil or wire (99.99%)

Non aqueous: - Tris (1-phenyl-1,3-butanediono) chromium (iii)

Preparation of 1,000 mg/l standard: Dissolve 1.0000 g of chromium metal in 50 ml of conc hydrochloric acid and dilute to 1 litre

Atomic absorbance

Lamp current: - 6.0 mA

Flame type: - Air-acetylene (highly reducing)

Optimum condition

Wave length (nm)	Slit width (nm)	Working range (mg/l)	Sensitivity (mg/l)
357.9	0.2	2 - 15	0.05
359.3	0.2	4 - 20	0.09
360.5	0.2	5 - 30	0.10
425.4	0.2	7 - 40	0.17
428.9	0.2	15 - 60	0.35

Interferences

Iron, cobalt, nickel, barium, aluminium and sodium cause interference with chromium absorbance. This effect are removed by the use of nitrous oxide - acetylene flame.

Flame emission

Wave length: -425.4 nm

Slit width: - 0.2 nm

Flame type: - Nitrous oxide-acetylene

Nickel

Reagents for standard preparation

Aqueous: -Nickel metal foil or wire (99.99%)

Non aqueous: - Nickel 4 - cyclohexylbutyrate

Preparation of 1.000 mg/l standard: Dissolve 1.0000 g of nickel metal in 50 ml of 6N nitric acid and dilute to 1 litre

Atomic absorbance

Lamp current: - 4.0 mA

Flame type: - Air-acetylene (oxidizing)

Optimum condition

Wave length (nm)	Slit width (nm)	Working range (mg/l)	Sensitivity (mg/l)
232.0	0.2	1.8 - 8	0.04
341.5	0.2	6 - 25	0.12
352.4	0.5	6 - 28	0.14
351.5	0.5	20 - 80	0.4

Interferences

Two lines of wave length at 341.5 nm and 352.4 nm are preferred for routine analysis over the 232.0 nm line due to excessive curvature caused by an unresolved non-resonance line at 232.14 nm.

Flame emission

Wave length: -341.5 nm

Slit width: - 0.2 nm

Flame type: - Nitrous oxide - acetylene

Zinc

Reagents for standard preparation

Aqueous: -Zinc metal foil or wire (99.99%)

Non aqueous: - Zinc 4-cyclohexylbutyrate

Preparation of 1,000 mg/l standard: Dissolve 1.0000 g of zinc metal in 40 ml of 5N hydrochloric acid and dilute to 1 litre

Atomic absorbance

Lamp current: - 5.0 mA

Flame type: - Air-acetylene (oxidizing)

Optimum condition

Wave length (nm)	Slit width (nm)	Working range (mg/l)	Sensitivity (mg/l)
213.9	0.5	0.4 - 1.5	0.008
307.6	0.5	3,000 - 12,000	66

Interferences

Non - atomic absorption should be corrected at 213.9 by use of a continum lamp.

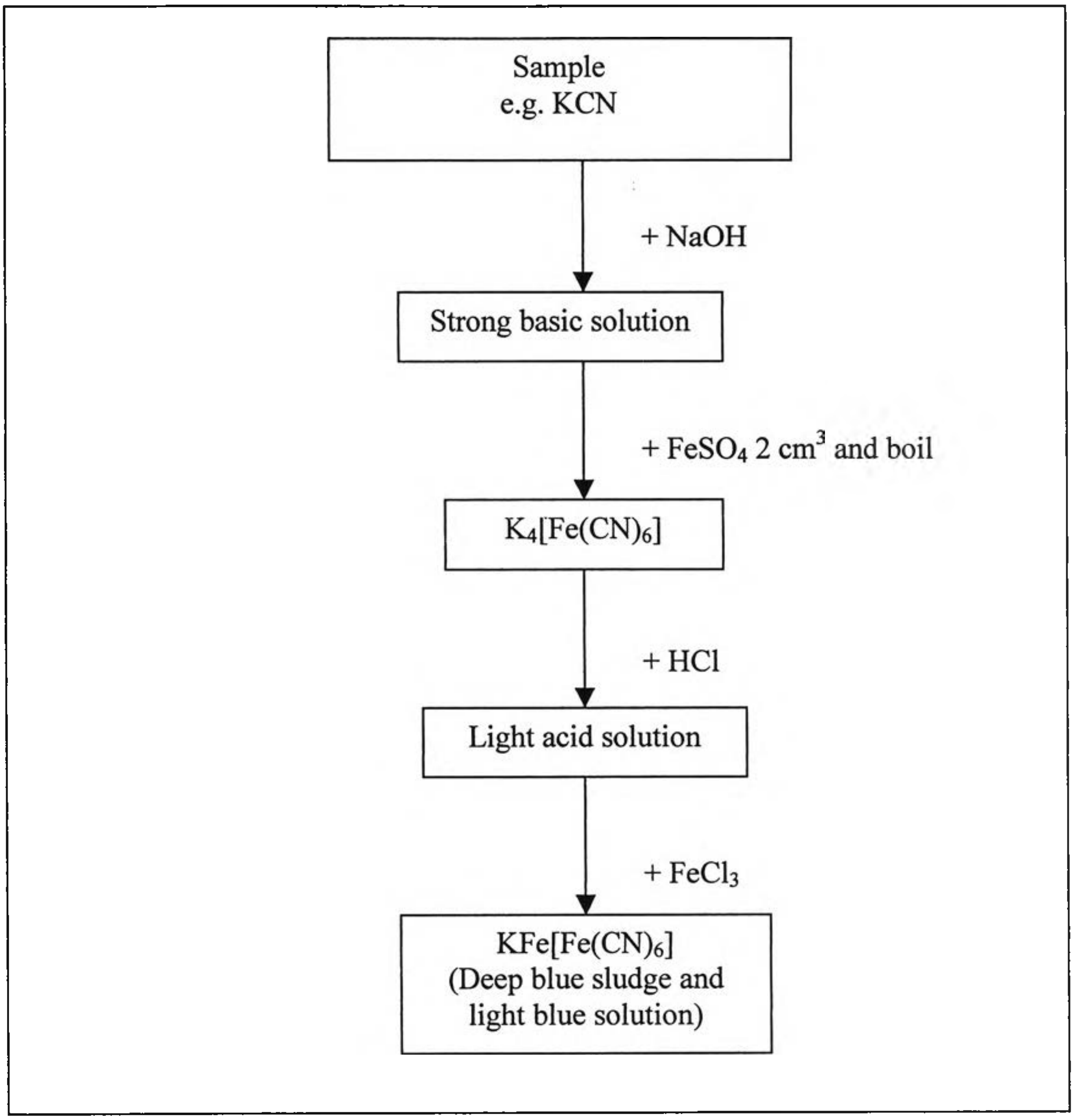
Flame emission

Wave length: -213.9 nm

Slit width: - 0.2 nm

Flame type: - Nitrous oxide-acetylene

B.2 Cyanide testing "Prussian Blue Test" [71]



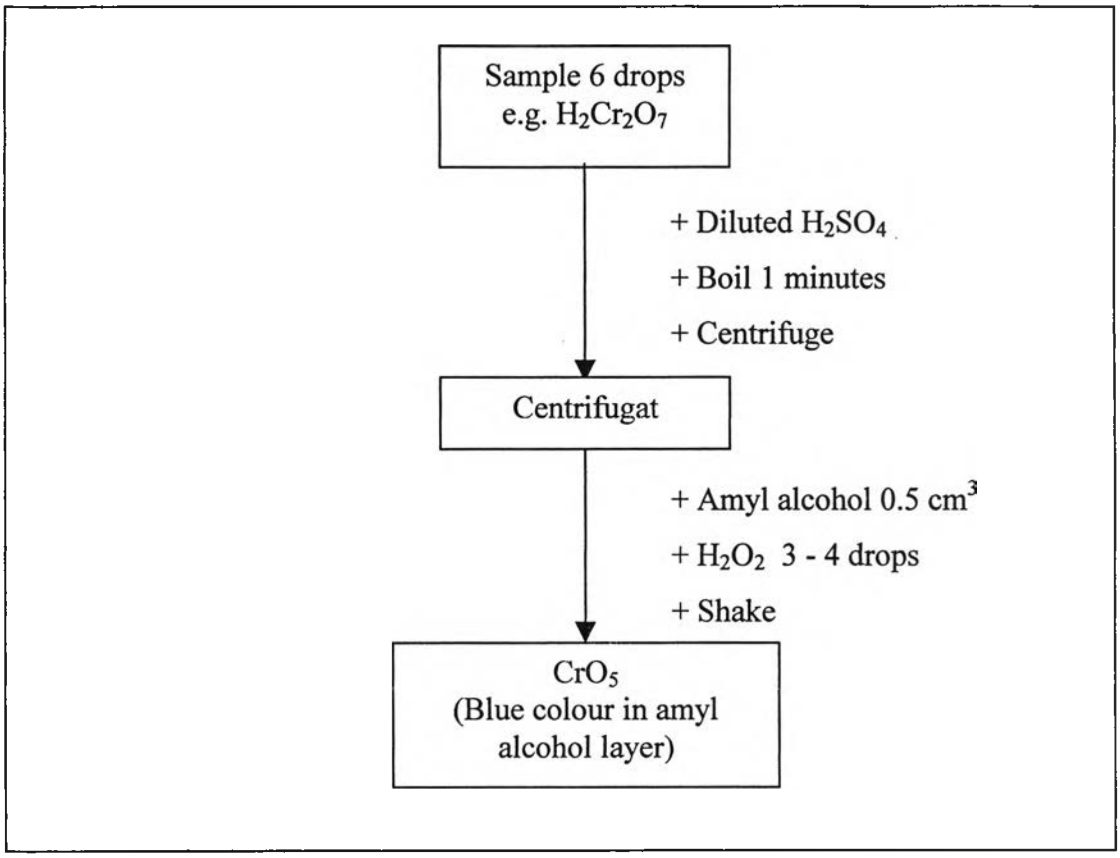
Reactions

$$6KCN + 2NaOH + FeSO_4 \longrightarrow Na_2SO_4 + 2KOH + K_4[Fe(CN)_6]$$

$$K_4[Fe(CN)_6] + FeCl_3 \longrightarrow KFe[Fe(CN)_6] + 3KCl$$

The positive test shows the deep blue sludge and light solution of $KFe[Fe(CN)_6]$.

B.3 Chromate testing [72]



Reaction

$$H_2Cr_2O_7 + 4H_2O_2 \longrightarrow 2CrO_5 + 5H_2O$$

The positive test shows the blue colour of CrO_5 in amyl alcohol layer and this colour is not stable. It is oxidized with air to green colour of chromic acid and releases oxygen.

APPENDIX C

DISCHARGED WASTE WATER STANDARD FOR INDUSTRY IN THAILAND

Physical and chemical property	Limited value
pH	5 - 9
Permanganate value	< 60 mg/l
Dissolved solids	< 2,000 mg/l
Sulphide (as H_2S)	< 1 mg/l
Cyanide (as HCN)	< 0.2 mg/l
Heavy metal	
Zinc	< 5 mg/l
Chromium	< 0.5 mg/l
Arsenic	< 0.25 mg/l
Copper	< 1 mg/l
Mercury	< 0.05 mg/l
Cadmium	< 0.03 mg/l
Barium	< 1 mg/l
Selenium	< 0.02 mg/l
Lead	< 0.2 mg/l
Nickel	< 0.2 mg/l
Manganese	< 5 mg/l
Tar	No
Oil & Grease	< 5 mg/l < 15 mg/l (For refinery industry)
Formaldehyde	< 1 mg/l
Phenols & Cresols	< 1 mg/l
Insecticide & Radioactive material	No
BOD	< 20 mg/l
Temperature	< 40 ^0C

APPENDIX D

TOXICITY SOURCES FROM ELECTROPLATING PROCESS [73]

Category	Substance	Source	Treatment process
Oil	Fat	Specimen preparation	Fat separation
Solid	Sand and polishing material	Surface polishing	Precipitation
Acid	Sulphuric acid, Hydrochloric acid, Nitric acid, Citric acid, Phosphoric acid	Chemical polishing	Neutralization
Base	Sodium hydroxide, Sodium carbonate, Sodium silicate	Fat cleaning, Neutralization process	Neutralization
Cyanide	Cyanide compound	Electro - cleaning or plating bath	Chlorine oxidation
Heavy metal ions	Copper, Nickel, Iron, Cadmium, Zins, Aluminium, Manganese	Plating bath and preparation	Basic treatment
Chromic acid	Chromic acid	Plating solution and chromate process	Reduction and precipitation
Complex ions	Complex ions of copper and nickel	Plating solution	Neutralization and precipitation

APPENDIX E

GENERAL PROPERTIES OF METALS

The property of copper, chromium, nickel and zinc is presented by following data. [61, 74]

1. Copper

Copper (symbol Cu, atomic weight 63.546, atomic number 29, cubic structure) is reddish orange metal. The origin name is *cyprium* (Latin word). The number of proton per electron is 29 and number of neutron is 35. It has a density of 8.96 g/cm^3 at 293 K, a boiling point of 2,840.15 K and a melting point of 1,356.15 K, relatively high thermal (94 m/ohm.mm^2) and electrical conductivities (59.3 m/ohm.mm^2), a specific heat of 0.39 J/g °C. It is used in electrical conductor, jewelry, coin, plumbing. The next table shows several states, oxidation number, metal form, colour, and crystallization system of copper.

State	Oxidation number	Metal form	Colour and Crystallization system
Solid	0	Cu	Copper, red, cubic
	+0.5	Cu_4O	Sub - oxide, green
	+1	Cu_2O	Copper oxide, red, cubic
		Cu_2O hydr	Oxide of copper hydrate or hydroxide $Cu(OH)_2$, yellow
	+2	CuO	Copper oxide, black, cubic
	+3	Cu_2O_3	Sesquioxide, red
	+4	CuO_2	Peroxide, bluish-black
Liquid	+1	Cu^+	
	+2	Cu^{2+}	Copper, light blue
		$HCuO_2^-$	Dicuprite ion, blue
	+3	Cu^{3+}	Copper (III) ion
		CuO_2^-	Cuprate ion, read or blue

2. Chromium

Chromium (symbol Cr, atomic weight 51.9961, atomic number 24, cubic structure) is gray metal discovered in 1797 by Louis Vauquelin. The origin name is *chroma* (Greek word). The number of proton per electron is 24 and number of neutron is 28. It has density of 7.19 g/cm^3 at 293 K, boiling point of 2,945.15 K and a melting point of 2,130.15 K, relatively low thermal (16 m/ohm.mm^2) and electrical conductivities (6.7 m/ohm.mm^2), specific heat of 0.46 J/g °C. It is used as stainless steel. The next table shows several states, oxidation number, metal form, colour, and crystallization system of chromium.

State	Oxidation number	Metal form	Colour and Crystallization system
Solid	0	Cr	Chromium, white, cubic
	+2	CrO	Black, hexagonal
	+2	CrO hydr	Yellow
	+2.67	Cr_3O_4	Oxide of chromo - chromic, brown
	+3	Cr_2O_3 hydr	Green, hexagonal
	+3.6	Cr_5O_9	Violet, orthorhombic
	+4	CrO_2 hydr	Brown
	+6	CrO_3	Deep red, orthorhombic
	+8	CrO_4	Tetroxide of chromium
	+10	CrO_5	Pentoxide of chromium, blue
Liquid	+2	Cr^{2+}	Chromium ion, bluish - green
	+3	Cr^{3+}	Chromic ion, green
		$Cr(OH)^{2+}$	Chromyl ion, green or violet
		$Cr(OH)_2^{+}$	Chromyl ion
		CrO_2^{-}	Chromite ion, green
		CrO_3^{3-}	Chromite ion, green
	+6	H_2CrO_4	Chromic acid, reddish - orange
		$HCrO_4^{-}$	Acid chromate ion, orange
		CrO_4^{2-}	Chromate ion, yellow
		$Cr_2O_7^{2-}$	Dichromate ion, orange

3. Nickel

Nickel (symbol Ni, atomic weight 58.71, atomic number 28, cubic structure) is a lustrous, silvery-white metal discovered in 1751 by Alex Cronstedt. The origin name is *kupfernickel* (German word). The number of proton per electron is 28 and number of neutron is 31. It has density of 8.9 g/cm^3 at 293 K, boiling point of 3,005.15 K and a melting point of 1,726.15 K, relatively low thermal (22 m/ohm.mm^2) and electrical conductivities (14.7 m/ohm.mm^2), specific heat of 0.45 J/g °C. It has high resistance to corrosion and oxidation, excellent strength and toughness at elevated temperatures, and is capable of being magnetized. It is attractive and very durable as a pure metal, and alloys readily with many other metals. It is used in electroplating metal alloys, nickel-cadmium batteries. The next table shows several states, oxidation number, metal form, colour, and crystallization system of nickel.

State	Oxidation number	Metal form	Colour and Crystallization system
Solid	0	Ni	Nickel, white, Cubic
	+2	NiO hydr	Oxide of nickel hydrate or hydroxide $Ni(OH)_2$, green
	+2.67	Ni_3O_4 hydr	Oxide of nickelo - nickelic bihydrate $Ni_3O_4.2H_2O$
	+3	Ni_2O_3 hydr	Oxide of nickel monohydrate $Ni_2O_3.H_2O$, black
	+4	NiO_2 hydr	Peroxide of nickel bihydrate $NiO_2.2H_2O$, black
Liquid	+2	Ni^{2+} $NiOH^+$ NiO^{2-} NiO_2^{2-} Ni^{3+} NiO_4^{2-}	Nickel ion, green ? Binickelite ion Nickelite ion Nickelic ion Nickelate ion

4. Zinc

Zinc (symbol Zn, atomic weight 65.37, atomic number 30, hexagonal structure) is bluish metal discovered in 1746 by Andreas Marggraf. The origin name is *zin* (German word). The number of proton per electron is 30 and number of neutron is 35. It has density of 7.133 g/cm^3 at 293 K, boiling point of 1,180.15 K and a melting point of 692.73 K, relatively low thermal (27 m/ohm.mm^2) and electrical conductivities (16.9 m/ohm.mm^2), specific heat of 0.39 J/g °C. It is used as metal coating, rust protection, brass, bronze and nickel. The next table shows several states, oxidation number, metal form, colour, and crystallization system of zinc.

State	Oxidation number	Metal form	Colour and Crystallization system
Solid	0	Zn	Zinc, white, hexagonal
	+2	ZnO hydr	White, orthorhombic
	+3	Zn_2O_3 hydr	Zinc hydrate sesquioxide, $Zn_2O3.H_2O$
	+3.33	Zn3O5 hydr	?
	+4	ZnO_2	Zinc peroxide
		ZnO_2 hydr	Zinc hydrate peroxide $ZnO_2.H_2O$
Liquid	+2	Zn^{2+}	Zinc ion
		$ZnOH^+$	Zincyle ion
		$HznO_2^-$	Dizincate ion
		ZnO_2^{2-}	Zincate ion

BIOGRAPHY

Ms. Mali Hunsom was born on December 20, 1975 in Samutsakhon, Thailand. After, she finished her Bachelor degree at the Department of Chemical Technology, Faculty of Science, Chulalongkorn University in 1998, she has further persuade her study in a doctorate degree at the same department since 1999.