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Labs taught in Introductory University Chemistry (Chem 200) at Red Deer College

Expt #	Title of Experiment (brief details of experiment)
1	Buret and Scale Operation
2	Hydrates (Determination of the number of waters of hydration of an inorganic compound.)
3	Determination of the Ideal Gas Constant (Determination of the gas constant, R, using the equation PV = nRT.)
4	Determination of the Molarity of an HCL solution
5	Determination of % Fe in unknown sample. (Determination of Fe using an oxidation –reduction reaction between Fe (II) and permanganate.)
6	Complex Nickel Compounds (Preparation of a coordination compound.)
7	Complex Nickel Compounds (Determination of the formula for the coordination compound.)
9 / 10	Redox Reactions: Oxidation states of Vanadium (Reduction of vanadium (V) to vanadium (IV) with sulfite, titrate with permanganate.) (Reduction of vanadium (V) to vanadium (II) with Zn, stabilize to vanadium (IV), titrate with permanganate.)
11	Enthalpy of Reaction (Molar enthalpy of neutralization for HCl/NaOH, HOAc/NaOH; Molar enthalpy of formation of Mg using Mg/HCl, MgO/HCl.)
12	Rates of Chemical Reactions: The kinetics of oxidation of Iodide by peroxydisulfate (Effect of concentration and temperature on rate, determine the overall order of the reaction, calculate the rate constant.)
13	Chemical Equilibrium, (iodine-triiodide equilibrium constant) (Determination of the equilibrium constant for $I^{-1} + I_2 \rightarrow I_3^{-1}$ in water.)
15	Titration Curves (HCL titrated with NaOH, HOAc titrated with NaOH; determine equivalence point, pKa.)
16	Titration Curves (NH_3 titrated with HCL, H_3PO_4 titrated with NaOH; determine equivalence point, pKa)
17	Qualitative Analysis of Anions
18 / 19	Qualitative Analysis of Cations
20	Electrolysis (Determination of the Faraday constant and atomic weight of Cu)

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Expt #1:   Buret and Scale Operation

- recording of volume and mass as an assessment of the proper recording of data

Expt #2:   Hydrates

- repeated heating of a hydrate and mass determination of the anhydrous form
- determination of the percentage mass of water
- determination of anion and cation of hydrate
- determination of the empirical formula of the hydrate

Expt #3:   Determination of the Ideal Gas Constant

- generate N_2 gas from reaction of sulfamic acid and sodium nitrite
- collect gas over water
- calculate pressure of N_2 gas, accounting for vapour pressure of water and pressure due to the height of the water in the buret:
  where $P_{nitrogen}$ is derived from: $P_{atm} = P_{nitrogen} + P_{water\ vapour} + P_{hydrostatic\ (i.e.,\ water\ column)}$
- compensate for the volume difference due to the differential temperature $P_1V_1/T_1 = P_2V_2/T_2$
- calculate gas constant R from $PV = nRT$

Expt #4:   Determination of the Molarity of an HCl Solution

- preparation of a primary standard, oxalic acid
- standardization of a NaOH solution
- titrate unknown HCl solution with NaOH, using an indicator to determine endpoint
- calculation of Molarity of unknown HCl solution
- calculation of Relative Standard Deviation

Expt #5:   Determination of % Fe using a Redox Titation

- general redox equation: $Fe^{+2} + MnO_4^{-1} \rightarrow Mn^{+2} + Fe^{+3}$
- standardization of a potassium permanganate solution using ferrous ammonium sulfate (FAS, Ammonium iron (II) sulfate), $Fe(NH_4)_2(SO_4)_2$, as the primary standard
  - titrate potassium permanganate against known amount of FAS
  - calculate molarity of permanganate based on balanced equation:
    $5Fe^{+2} + MnO_4^{-1} + 8H^{+1} \rightarrow Mn^{+2} + 5Fe^{+3} + 4H_2O$
- titrate Fe sample with permanganate solution
- determine moles of Fe, determine mass of Fe (moles Fe x 55.8 g/mole Fe)
- determine mass % Fe: mass Fe/mass of sample

Expt #6:   Complex Nickel Compounds
- two complex compounds were prepared:
  $Ni(NH_3)_4(NO_2)_2$ and
  $Ni(NH_3)_x(Cl)_x$

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Expt #7:   Complex Nickel Compounds

- determination of formula for coordination compound $Ni(NH_3)_x(Cl)_x$
- determination of %Cl using the Volhard titration: precipitate Cl^{-1} using excess Ag^+, titrate excess Ag^+ with thiocyanate (forms AgSCN which is insoluble), indicator for end point is $FeSCN^{+2}$, utilizing a small amount of Fe (as ferric nitrate)
- determination of %Ni using EDTA (1Ni:1EDTA) titration using murexide indicator
- %NH_3 determined by difference from total Ni and Cl
- 3 possibilities: $[Ni(NH_3)_6]Cl_2$; $[Ni(NH_3)_6Cl]Cl$; $[Ni(NH_3)_6Cl_2]$

Expt #9:   Redox Reactions: Oxidation states of Vanadium

Oxidation States of vanadium

Oxidation	Ionic form	Name	Color
+ 5	VO_3^{-1}	metavanadate	none
+ 5	VO_2^{+1}	pervanadyl (dioxovanadium)	yellow
+ 5	VO_4^{-3}	orthovanadate	none
+ 4	VO^{+2}	vanadyl	blue
+ 3	V^{+3}	vanadium III	Green
+ 2	V^{+2}	vanadium II	violet

- preparation of metavanadate from ammonium metavanadate
    - dissolve NH_4VO_3 in NaOH
- preparation of the starting solution, pervanadyl
    - add sulfuric acid to the metavanadate (solution turns yellow)
    - $VO_4^{-3} + 4H^+ \rightarrow VO_2^{+1} + 2H_2O$
- preparation of vanadyl (using the reducing agent sulfite, in the form SO_2)
    - add sodium sulfite to the pervanadyl (solution turns blue)
    - $2H^+ + 2VO_2^{+1} + SO_3^{-2} \rightarrow H_2O + 2VO^{+2} + SO_4^{-2}$
- titrate vanadyl with permanganate
    - $5VO^{+2} + MnO_4^{-1} + H_2O \rightarrow 5VO_2^{+1} + 2H^+ + Mn^{+2}$

Expt #10:   Redox Reactions: Oxidation states of Vanadium

- reduction of pervanadyl with zinc (solution turns violet)
    - $8H^+ + 2VO_2^{+1} + 3Zn \rightarrow 4H_2O + 2V^{+2} + 3Zn^{+2}$
- stabilize by adding pervanadyl (convert to stable form, because V^{+2} and V^{+3} is air oxidized; VO^{+2} - not air oxidized)
    - $V^{+2} + 2VO_2^{+1} + 2H^+ \rightarrow 3VO^{+2} + H_2O$
- titrate vanadyl with permanganate

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Expt #11:   Enthalpy of Reaction

- enthalpy of neutralization for reactions of HCl/NaOH and HOAc/NaOH
    - monitor temperature for neutralization reaction
    - calculate heat of neutralization: $q = ms\Delta T = (mL \times g/mL)(J/^{\circ}C\cdot mole)(^{\circ}C)$
    - enthalpy change $= \Delta H = q$
    - calculate molar enthalpy $= \Delta H/n$
- enthalpy of formation of MgO
    - determine heat of reaction for Mg + HCl, determine molar enthalpy change
    - determine heat of reaction for MgO + HCl, determine molar enthalpy change
    - molar enthalpy change for $Mg^{+2} + H_2O \rightarrow MgO + 2H^+$, inverse sign
    - using Hess' law

      $Mg + 2H^+ \rightarrow Mg^{+2} + H_2$ $\qquad\qquad \Delta H_1 = ?$
      $Mg^{+2} + H_2O \rightarrow MgO + 2H^+$ $\qquad \Delta H_2 = ?$
      $H_2 + \frac{1}{2} O_2 \rightarrow H_2O$ $\qquad\qquad\qquad \Delta H_2 = -285 \text{ KJ/mole}$

      _____

      $Mg + \frac{1}{2} O_2 \rightarrow MgO$ $\qquad\qquad\quad \Delta H_f = ?$

Expt #12:   Rates of Chemical Reactions

- determination of the effect of the concentration on the rate of a reaction
    - monitor rate of reaction between peroxdisulfate ($S_2O_8^{-2}$) and iodide (I^-):
      (i) $S_2O_8^{-2} + 2I^- \rightarrow 2SO_4^{-2} + I_2$
    - follow rate of iodine formation by a secondary reaction, where [thiosulfate, $S_2O_3^{-2}$] is small and constant, (ii) is fast compared to (i), starch as indicator:
      (ii) $2S_2O_3^{-2} + I_2 \rightarrow S_4O_6^{-2} + 2I^-$
    - monitor time of reaction for varying concentration of $[S_2O_8^{-2}]$ & $[I^-]$
    - determine rate of reaction: dividing iodine formed by reaction time $\Delta[I_2]/\Delta t$
    - determine the order of each reactant, n and m, using two rate equations where one concentration is held constant

  $Rate_1/ Rate_2 = (k[\text{constant-}S_2O_8^{-2}]^m [\text{variable-}I^-]^n) / (k[\text{constant-}S_2O_8^{-2}]^m [\text{variable-}I^-]^n)$
  $Rate_1/ Rate_2 = ([\text{variable-}I^-]^n) / ([\text{variable-}I^-]^n)$

    - determine the overall order of the reaction, sum n and m
    - calculate the rate constant for each of the experimental runs

- determination of the effect of temperature on the rate of a reaction
    - monitor rate of reaction between peroxdisulfate ($S_2O_8^{-2}$) and iodide (I^-) by monitoring rate at different temperatures
    - monitor time of reaction for varying temperatures: 1, 10, 20, 40 °C
    - calculate the rate constant, k, for each temperature
    - plot log k *versus* inverse temperature (1/T)
    - determine the activation energy E_a

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Expt #13:   Chemical Equilibrium (iodine-triiodide equilibrium constant)

- determination of the equilibrium constant for the iodine-triiodide in water
  $I^{-1} + I_2 \rightarrow I_3^{-1}$; chemical equilibrium
- prepare aqueous solution of iodine – triiodide
- extract aqueous layer with CCl_4 and separate the two phases
- titrate CCl_4 layer with thiosulfate, which contains I_2
- titrate aqueous layer with thiosulfate = total iodine in form of I_2 and I_3^{-1}
- determine I_2 in aqueous layer knowing K for I_2 distribution between aqueous and CCl_4 layer; K = (I_2 org)/ (I_2 aqueous) = 90.5
- titration of aqueous layer gives I_2 aqueous and I_3^{-1} aqueous = Total
- Total - I_2 aqueous = I_3^{-1} aqueous
- amount of I^{-1} at equilibrium must be equal to initial $[I^{-1}] – [I_3^{-1}$ aqueous], because for every I_3^{-1} formed, one I^{-1} was consumed
- calculate K for chemical equilibrium: $K_{aq} = [I_3^{-1}]/[I_2] [I^{-1}]$

Expt #15:   Titration Curves (strong base/strong acid; strong base/weak acid)

- titrate NaOH with HCl (unknown concentration), plot pH *versus* volume NaOH
  determine equivalence point
  determine concentration of unknown [HCl], using $(M_{NaOH})(V_{NaOH}) = (M_{HCl})(V_{HCl})$
- titrate NaOH with HOAc (unknown concentration), plot pH *versus* volume NaOH
  determine equivalence point
  determine concentration of unknown [HOAc], $(M_{NaOH})(V_{NaOH}) = (M_{HOAc})(V_{HOAc})$
  determine the pKa for HOAc (pH at which acid is ½ titrated)

Expt #16   Titration Curves(weak base/strong acid; polyprotic acid)

- titrate NH_3 with HCl, plot pH *versus* volume HCl
  determine equivalence point
  determine the pKa for NH_4^+
- titrate H_3PO_4 with NaOH, plot pH *versus* volume NaOH
  determine the pK_{a1} and pK_{a2}

Expt #17:   Qualitative Analysis of Anions

- qualitative analysis of anions based on the formation of precipitates of, e.g., Ag^+, Ba^{+2} and brown ring test for nitrate (add reagents H_2SO_4 & $FeSO_4$, that ultimately form an addition compound $FeNOSO_4$) and addition of ammonia (for formation of ammonia complex).

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Expt #18 &19: Qualitative Analysis of Cations

- qualitative analysis performed for a series of cations from 5 different groups
- unknowns contained 4 different cations
- general determination of cations present based on solubility of corresponding chlorides, sulfides, e.g. AgCl, CuS
- use of temperature to effect separation due to differing solubility products
- additionally, precipitation used to isolate ions from mixture, to aid in subsequent identification of remaining cations
- identification based on differential solubility of hydroxides
- identification based on formation of ammonia complexes
- Additional identification based on the color of aqueous solution subjected to a flame test.

Expt #20:   Electrolysis

- set up electrolytic cell where Cu is the anode (+) and nichrome is the cathode (-)
- anode: $Cu \rightarrow Cu^{+2} + 2e^-$
- cathode: $2H^+ + 2e^- \rightarrow H_2$
- perform electrolysis and record data on vol H_2 collected, current (I), weight Cu lost, time (sec)
- calculate #moles of H_2 produced, using $PV = nRT$,
    where $P_{hydrogen}$ is derived from: $P_{atm} = P_{hydrogen} + P_{water\ vapour} + P_{water}$
- calculate the number of moles of e^- consumed:
    moles of $e^- = 2$(#moles of H_2 produced)
- from the current, calculate the #Coulombs of charge transferred:
    1amp = 1 Coulomb/sec
    Coulomb = amps•sec
- calculate the Faraday constant (F) = #Coulombs/ moles of e^-
- calculate the atomic weight {atomic mass} for Cu
    using the definition: For redox reactions involving metals, the gram equivalent weight (GEW) of a metal is the weight of metal which reacts with or <u>releases one mole of electrons</u>. Therefore, the GEW is the mass of metal per mole of electrons transferred.

    GEW = #grams Cu/ moles of e^- = gm Cu/ mole of e^-
    Atomic weight = (gm Cu/ mole of e^-)(2 mole of e^- /mole Cu)

    e.g.   GEW = 0.0803 gm/2.5 x 10^{-3} moles e^- = 32.1 gm/ mole of e^-
    Atomic weight = (32.1 gm Cu/ mole of e^-)(2 mole of e^- /mole Cu) = 64.2
    or   GEW = #grams Cu/ moles of e^- = gm Cu/ equivalent
    Atomic weight = (gm Cu/equivalent)(2 equivalents Cu/mole Cu)

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APPENDIX

(1) Experiment #9/10:
   1. $2H^+ + SO_3^{-2} \rightarrow H_2O + SO_2$ {reducing agent}
          {sulfite in acid is degraded into SO_2, which can be air oxidized to sulfate, SO_4^{-2}}
   2. $2H_2O + SO_2 \rightarrow SO_4^{-2} + 4H^+ + 2e^-$ ref: Chem lab manual
   3. $1e^- + VO_2^+ + 2H^+ \rightarrow VO^{+2} + H_2O$

   Overall: $2H^+ + 2VO_2^{+1} + SO_3^{-2} \rightarrow H_2O + 2VO^{+2} + SO_4^{-2}$

(2) Experiment #9/10,12:
   SO_2 sulfur dioxide, {IV}
   SO_3^{-2} sulfite, {sulfate (IV)}
   SO_4^{-2} sulfate, {sulfate (VI)}
   $S_2O_3^{-2}$ thiosulfate
   $S_2O_8^{-2}$ peroxydisulfate, {VI)}
   $S_4O_6^{-2}$ tetrathionate

(3) Experiment #17:
   -        Ag^+: $AgCl, AgBr, Ag_2SO_4, AgI$
   -        Ba^{+2}: $BaSO_4$
   -        CO_3^{-2} {carbonates} in HCl $\rightarrow CO_2$ gas
   -        nitrates - brown ring test
   -        ammonia for cation complexes

(4) Experiment #18/19:
   Group I: ppt when Cl^- added $\rightarrow PbCl_2, Hg_2Cl_2, AgCl$
   Group II: ppt when thioacetamide added $\rightarrow Bi_2S_3, CuS, CdS$
   Group III: insoluble in strong NaOH $\rightarrow Fe(OH)_3, Mn(OH)_2, Co(OH)_2, Ni(OH)_2$
   Group IV: soluble in strong NaOH $\rightarrow Al(OH)_4, Cr(OH)_4, Zn(OH)_4$
   Group V: did not react to this point $\rightarrow Ca, NH_4$

(5) Electrochemistry basics:
   1.   VANO – Voltaic cell/Anode/Negative/Oxidation
   2.   Voltaic Cell: voltage of cell
                  $$E_{cell} = E_{cathode} - E_{anode}$$
          − If E_{cell} is positive, then the cell is spontaneous.
          − For a spontaneous cell, the anode must always be a smaller number, i.e., a smaller
            positive or more negative.
          − Metal(s) │ M(aq) ││ M(aq) │ Metal(s);   Anode ││ Cathode
          − SOAP: Strong Oxidizing Agent is more Positive redox potential
          − SRAN: Strong ReducingAgent is more Negative redox potential
   3.   EAPO – Electrolytic cell/Anode/Positive/Oxidation
          − to determine the minimum voltage needed to get electrolysis to work, use
                  $$E_{cell} = E_{anode} - E_{cathode}$$, as written for the electrolytic cell