you-try-it-03.xlsx
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For use with:
Brian M. Tissue, *Basics of Analytical Chemistry and Chemical Equilibria,* (John Wiley: New York, 2013).
http://www.achem.org

Worksheets in this file

notes	This page with background information.
3.A limiting-reagent	Determining minimum reagent amounts for complete reaction.
3.B gravimetry	Predicting precipitate weights.
3.C titration	Unknown determinations from titration results.
3.D titration-curves	Data plots to determine endpoint.

Background
Refer to Chapter 3 in the text for equations and explanations.
Each worksheet has instructions in the blue shaded box.
For step-by-step help see you-try-it-03guide.pdf.

You-Try-It 3.A Limiting Reagent

Tables 3.A.1 - 3.A.5 list examples of reactions for analytical procedures.
Analytical procedures often use excess reagent.
For each example determine the minimum amount of reagent that is necessary for complete reaction:

1. Determine the minimum concentration of HCl to neutralize the $CaCO_3$.
2. Determine the minimum volume of 0.5 M NaCl for complete reaction to $AgCl_4^{3-}$.
3. Determine the minimum number of moles of succinic acid for complete reaction.
4. Determine the minimum volume of 2 % KI solution necessary for complete reaction of the Cl_2.
5. Determine the minimum number of moles of O_2 for complete combustion.

Table 3.A.1

analyte	reagents	products	reaction type, sample, and procedure
$CaCO_3$	H^+	CO_2 (*g*)	neutralization
	(x M HCl)	Ca^{2+} (aq)	$CaCO_3$ in soil (assume 5 % $CaCO_3$)
			dry soil sample and grind to fine powder
			add 25 mL of HCl solution to beaker and weigh
			add 3.0 g of soil to beaker, swirl, reweigh after 30 min
			$CaCO_3$ is determined from the weight loss using:
			$CaCO_3(s) + 2H^+(aq) \rightarrow Ca^{2+}(aq) + CO_2(g) + H_2O$

Table 3.A.2

analyte	reagents	products	reaction type, sample, and procedure
Ag^+	Cl^-	$AgCl_4^{3-}$ (aq)	complexation
	(0.5 M NaCl)		50.0 mL of 0.001 M $AgNO_3$
			Ag^+ precipitates as AgCl in the presence of Cl^-
			an excess of Cl^- can redissolve the Ag^+ as Ag-Cl complexes

Table 3.A.3

analyte	reagents	products	reaction type, sample, and procedure
Al	$C_2H_4(COOH)_2$	$Al(succ)_2OH$ (*s*)	precipitation of aluminum in antacid tablets
	urea (OH^-)		assume 0.20 g of aluminum hydroxide in a typical sample
			The samples are dissolved with succinic acid and urea
			heating the solution produces ammonia homogeneously
			the slow rise in pH precipitates the product

Table 3.A.4

analyte	reagents	products	reaction type, sample, and procedure
Cl_2	I^-	I_2 (aq)	redox
	(2 % KI)	Cl^- (aq)	100 L of air containing 0.1 mg/m^3 Cl_2.
			bubbling air through 2 % KI solution causes:
			$Cl_2 + 2KI \rightarrow I_2 + 2KCl$
			The I_2 product is determined by titration.

Table 3.A.5

analyte	reagents	products	reaction type, sample, and procedure
C and H	O_2	CO_2 (*g*)	elemental analysis of hydrocarbon
		H_2O (*g*)	100 mg of paraffin wax
			the wax consists of equal weights of:
			$C_{28}H_{58}$, $C_{30}H_{62}$, $C_{32}H_{66}$, and $C_{34}H_{70}$.

References

case 4 adapted from: OSHA method # ID-126SGX
Chlorine and Chlorine Dioxide in Workplace Atmospheres
http://www.osha.gov

You-Try-It 3.B **Gravimetry**

Table 3.B.1 lists several gravimetric analyses.

1. Predict the precipitate weight for each case in Table 3.B.1.

Table 3.B.2 lists a procedure and analytical results for an analysis.

2. Calculate the total suspended solids (TSS) for the water sample.

Table 3.B.1 sample	sample weight	analyte	analyte conc	precipitating agent	precipitate
brass filings	0.1001 g	Cu	60.7%	NH_4SCN	CuSCN
fertilizer	1.55 g	Mg	11.0 %	$(NH_4)_2HPO_4$	$Mg_2P_2O_7$
fertilizer	1.64 g	S	22.0 %	$BaCl_2$	$BaSO_4$
wastewater	250.0 mL	Al^{3+}	122 ppm	succinic acid	Al_2O_3

Procedure to determine total suspended solids (TSS).

Place glass-fiber filter disk in Gooch crucible and apply suction.
Rinse with three 20-mL portions of deionized water and continue suction until dry.
Bring crucible and filter combination to constant weight by:
dry in 104 C oven for 1 hour
cool in dessicator and weigh
repeat drying, cooling, and weighing until weight change is less than 0.5 mg
Use final weight as tared weight. Store in dessicator until use.
Prepare separate crucible/filter combinations for multiple measurements.

Appy suction to tared crucible. Wet filter with a small volume of deionized water to seat it.
Stir water sample with magnetic stirrer. Pipet 100 mL of sample onto the glass-fiber filter.
Wash with three 10-mL portions of deionized water.
Pipet second 100 mL aliquot of sample and repeat washing.
Continue suction until dry, approx 3 min.
Remove crucible/filter/solids combination and bring to constant weight as before.
Repetitive measurements should agree within 5 % of average weight.
Report TSS as mg/L.

Table 3.B.2 crucible/filter/solids to constant weight

trial	tared wt	wt 1	wt 2	wt 3	wt 4
1	31.6476	31.6777	31.6555	31.6552	
2	31.6116	31.6344	31.6195	31.6192	31.6181
3	32.1148	32.1282	32.1224	32.1222	

References

Table 3.B.1 precipitating agents from J. A. Dean, *Analytical Chemistry Handbook* , McGraw-Hill:New York, 1995.

TSS procedure adapted from: Method 2540 D. Total Suspended Solids Dried at 103–105 C.
Standard Methods for the Examination of Water and Wastewater. 20th Ed.,
American Public Health Association: Washington, DC 1999.

You-Try-It 3.C **Titration**

Tables 3.C.1 and 3.C.2 list results of two titration measurements.

The Table 3.C.1 data are results for standardizing a sodium thiosulftate solution with a primary standard.
The titration solution contains the potassium iodate, KIO_3, primary standard and auxiliary reagents.
The overall titration reaction is:

$$IO_3^-(aq) + 6S_2O_3^{2-}(aq) + 6H^+(aq) \rightarrow I^-(aq) + 3S_4O_6^{2-}(aq) + 3H_2O$$

1. Determine the concentration of the sodium thiosulfate titrant.

The Table 3.C.2 data uses the standardized thiosulftate to determine I_2.
The overall titration reaction is:

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

2. Determine the concentration of I_2 in the unknown.

Table 3.C.1

trial	standard weight (g)	titration result (mL)
1	0.1485	38.52
2	0.1514	39.14
3	0.1507	39.07

Table 3.C.2

trial	unknown volume (mL)	titration result (mL)
a	25.00	22.99
b	25.00	22.91
c	25.00	22.72

You-Try-It 3.D **Titration Curves**

A set of titration data is listed in Table 3.D.1 for vinegar, which contains acetic acid.
The analysis procedure is:

Pipet 10.00 mL of the vinegar sample to a 250-mL beaker.
Add 50.0 mL of distilled water and swirl.
Place pH electrode in the solution so that bottom is submerged but not bumping stir bar.
Add titrant stepwise to stirred solution and record stable pH readings.
The titrant is 0.2950 M NaOH.

1. Insert a scatter chart to plot the data as pH versus titrant volume.
2. Generate a Gran plot of the data.
3. Determine the concentration of the acid in the unknown solution.

Table 3.D.1

mL titrant	pH
0.00	3.75
1.02	4.00
1.95	4.15
3.32	4.28
5.11	4.35
6.83	4.45
7.79	4.50
9.93	4.60
11.00	4.65
12.42	4.67
14.07	4.70
15.00	4.77
16.50	4.90
17.35	5.00
18.50	5.10
19.40	5.20
20.45	5.36
21.10	5.50
21.42	5.60
22.20	5.80
22.50	5.90
22.93	6.20
23.32	6.50
23.38	7.00
23.39	7.90
23.40	8.20
23.41	8.70
23.42	9.30
23.44	10.10
23.50	10.50
23.94	11.05
24.91	11.44
26.59	11.77
28.05	11.90
28.98	11.99