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CHEMICAL EDUCATION

Microscale experiments for general chemistry

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Microscale experiments offer several attractive features for the beginning chemistry course: a reduced consumption of chemicals, low cost materials, improved safety in the laboratory classes, and a non-existent waste disposal problem. The microscale approach were applied to five general chemistry experiments: Stoichiometry of a Salt Formation, Concentration Effects on Reaction Rates, Reversible Reactions, Electromotive Force of Galvanic Cells and Electrolysis. The microscale experiments generated results which were similar to the macroscale analogues.

Keywords: microscale chemistry, laboratory experiments, general chemistry

MICROSCALE METHODS HAVE INTRODUCED A NEW EXCITEMENT IN the chemistry laboratory courses. The dimensions of the materials employed in the laboratory experiments are being changed radically. Many experiments have been demonstrated to work well at a reduced scale, introducing possibilities for greater savings and improved safety in the laboratory classes.

Microscale experiments involve drop-size amounts of reagents and therefore reduce chemical consumption. As a result, less safety hazards are encountered and waste disposal problems are almost eliminated. These experiments can be completed within a shorter period of time, compared to the larger scale analogue, allowing the student more time to concentrate on the chemical principle involved. This reduction in the scale of the experiment has been shown not to affect the development of the desired skills in the student [1].

This paper presents five microscale experiments on important concepts in the beginning chemistry course. These experiments use common reagents and low cost instruments.

MICROSCALE EQUIPMENT

One important apparatus in microscale chemistry is the micro-well plate. It was originally intended for use in biomedical laboratories but has found utility in several other applications. It is compact, often with dimensions of 12 cm by 8 cm, and provides multiple containers for small volumes of solutions (Fig. 1). It is made of an acrylic polymer material which can be easily rinsed and reused. It, however, dissolves in some organic solvents and can be used only at temperatures below 70°C [2]. It is relatively inexpensive (ca P100.00) and is easily available from biomedical suppliers.

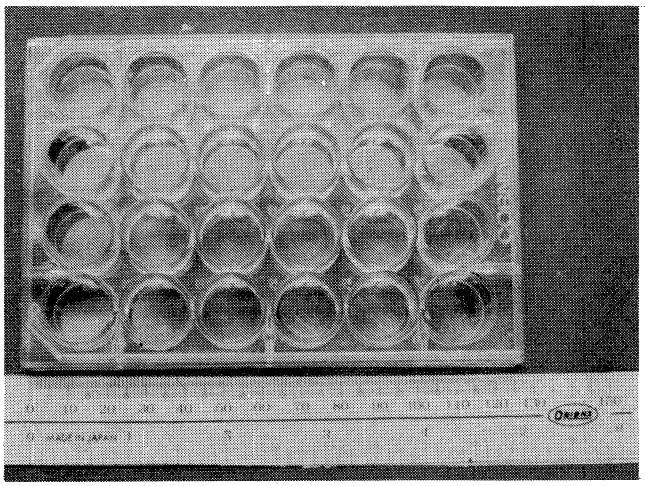

Figure 1. A Micro-well plate

Another important microscale equipment is the 1-mL syringe which is particularly useful for the measuring and dispensing of small quantities of reagents. It is made of a polymer material which is compatible with most solutions used in the general chemistry laboratory. It can be readily purchased at a low cost (ca P10.00) from the drugstores.

EXPERIMENT 1: STOICHIOMETRY OF A SALT FORMATION

A metal ion combines with anions in a definite ratio. The combining ratio of a cation and an anion to form an insoluble salt can be determined by mixing varying amounts of solutions containing the reactant ions. The amount of product formed can be estimated visually through the turbidity of the mixture. The greatest turbidity will be observed when the ions are mixed at the correct stoichiometric ratio.

In the experiment, different volumes of a 0.01 M solution of $AgNO_3$ and a 0.01 M solution of K_2CrO_4, as given in Table 1, are mixed in a microwell. The amount of the precipitate formed can be estimated by viewing each well vertically. The greatest amount of precipitate is observed in Well A4, wherein the mole ratio of the Ag^+ to the $CrO_4^=$ ion is 2:1. This result is consistent with the balanced chemical equation from the formation of silver chromate:

$$2\ Ag^+_{(aq)} + CrO_4^{=}{}_{(aq)} \rightarrow Ag_2CrO_{4(s)} \qquad (1)$$

Table 1. Volume of reagents to be mixed in the stoichiometry experiment

Well number	A1	A2	A3	A4	A5
Volume (mL) of 0.01 M $AgNO_3$ solution	0.2	0.4	0.6	0.8	1.0
Volume (mL) of 0.01 M K_2CrO_4 solution	1.0	0.8	0.6	0.4	0.2

EXPERIMENT 2: CONCENTRATION EFFECTS ON REACTION RATES

The effect of concentration on reaction rates can be studied through the iodine clock reaction. This reaction involves the iodate ions and hydrogen sulfite ions:

$$IO_3^-{}_{(aq)} + 3\ HSO_3^-{}_{(aq)} \rightarrow I^-{}_{(aq)} + 3\ SO_4^{2-}{}_{(aq)} + 3\ H^+_{(aq)} \qquad (2)$$

In the reaction mixture, an excess of iodate ions is mixed with hydrogen sulfite ions in aqueous solutions. When all of the hydrogen sulfite ions have reacted, the iodide ions react with the excess iodate ions to form iodine.

$$5\ I^-_{(aq)} + IO_3^-{}_{(aq)} + 6\ H^+_{(aq)} \rightarrow 3\ I_{2(aq)} + 3\ H_2O \qquad (3)$$

The reaction rate is determined by noting the time needed for a blue color to appear in the presence of a starch indicator.

In the experiment, varying concentrations of hydrogen sulfite ions ($\leq$ 0.025M) with added starch are prepared and mixed in microwells with an equal volume of a solution containing iodate ions (0.01M). The time for the appearance of the blue color is noted for each mixture and its reciprocal is plotted against the concentration of the hydrogen sulfite ions. Fig. 2 shows a typical plot obtained from this experiment.

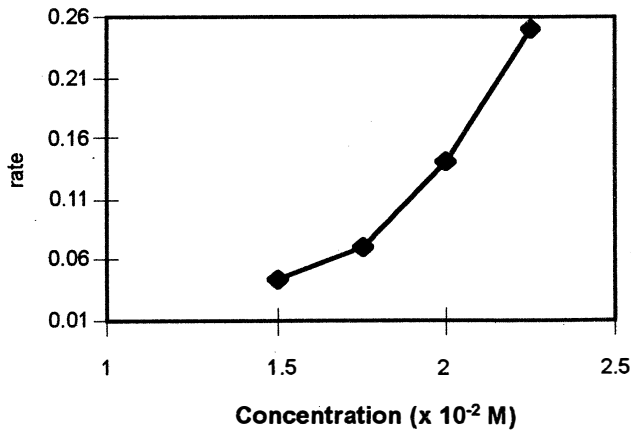

Figure 2. Plot of rate against concentration of hydrogen sulfite ions

EXPERIMENT 3: REVERSIBLE REACTIONS

Most chemical reactions can be considered to be reversible. The products of a reaction can react with each other to form the original reactants:

$$A + B = C + D \qquad (4)$$

When the rate of the forward and reverse reactions are equal, a state of dynamic equilibrium exists. The factors affecting the direction of the favored reaction in a reversible reaction can be studied in the Iron (III) - Thiocyanate system. The presence of the reactants and products are indicated through the color of the species.

In the experiment, varying amounts of the following solutions: 0.01 M $FeCl_3$, 0.01 M KSCN, and 0.01M NaOH, as given in Table 2, are mixed in a microwell. The solutions in wells A1 and A2 are control solutions, their colors corresponding to those of the reactants and products, respectively. The intensity of the color of the solutions of the other wells (wells A3 to A5) are compared with the control solutions.

Table 2. Volume of reagents to be mixed in the reversible reaction experiment

Well number	A1	A2	A3	A4	A5
Volume (mL) of 0.01 M $FeCl_3$ solution	0.5	0.5	1.0	0.5	0.5
Volume (mL) of 0.01 M KSCN solution	-	0.5	0.5	1.0	0.5
Volume (mL) of distilled H_2O	1.0	0.5	-	-	-
Volume (mL) of 0.01 M NaOH solution	-	-	-	-	0.5

Increasing the volume of one of the reactants (as in wells A3 and A4) favors the forward reaction, thus, increasing the concentration of the products. Lowering the concentration of one of the reactants, by the addition of a precipitating agent (as in well A5), favors the reverse reaction, decreasing the concentration of the products.

EXPERIMENT 4: ELECTROMOTIVE FORCE OF GALVANIC CELLS

The electromotive force of a galvanic cell is the voltage of a cell when measured under high impedance conditions. This voltage is equal to the difference in the reduction potential of the electrodes comprising the cell.

Half-cells can be assembled on a microwell and coupled to each other by means of a simple salt-bridge. The electromotive force of the cell is measured using a digital multimeter. The half-cell is constructed using a metal strip or wire, such as copper, lead and zinc, immersed in a 0.1 M solution of its ion. The salt bridge is prepared by wetting a piece of filter paper with a saturated solution of potassium chloride.

The experimental values obtained from this experiment are not significantly different from the values calculated from the Nernst equation: $E_{cell} = E_{cathode} - E_{anode}$, where $E_{cathode}$ and E_{anode} refer to the half cell potentials of the cathode and anode, respectively. A typical set of results is given in Table 3.

Table 3. Electromotive force of galvanic cells

Cell reaction	Theoretical value	Experimental value
$Zn\|Zn^{2+}(0.1M)\|\|Cu^{2+}(0.1M)\|Cu$	1.100	1.09
$Zn\|Zn^{2+}(0.1M)\|\|Pb^{2+}(0.1M)\|Pb$	0.637	0.62
$Pb\|Pb^{2+}(0.1M)\|\|Cu^{2+}(0.1M)\|Cu$	0.463	0.46

EXPERIMENT 5: ELECTROLYSIS

Electrolysis is the process in which electrical energy is used to produce a chemical change in a cell. The electrolytic cell consists of an anode and a cathode where oxidation and reduction reactions occur, respectively.

A simple electrolytic cell can be constructed using two adjacent microwells containing a 0.1 M solution of sodium sulfate mixed with a small amount of bromthymol blue indicator. The two wells are coupled by means of a filter paper wetted with the sodium sulfate solution. A piece of pencil lead is immersed in each of the wells and connected to the terminals of a 9-V battery.

Color changes are observed in each well within two minutes. The solution in one well turns yellow because of the formation of hydrogen ions from the electrolysis reaction:

$$2\,H_2O_{(l)} \rightarrow 4\,H^+_{(aq)} + O_2(g) + 4\,e^-_{(aq)} \qquad (5)$$

The well in which this reaction takes place is the anode. The solution in the other well turns blue because of the production of OH^- ions from the following reduction reaction:

$$4\,H_2O_{(l)} + 4\,e^-_{(aq)} \rightarrow 4\,OH^-_{(aq)} + 2\,H_{2(g)} \qquad (6)$$

The well in which this change occurs acts as the cathode in this cell.

A similar set-up can also be used to monitor the electrolysis of potassium iodide. In this case, the electrolyte employed is a solution of 0.1 M potassium iodide containing a small amount of phenolphthalein and starch indicator. The electrolysis products are the hydroxide ion, which is indicated by the pink color it gives with phenolphthalein, and iodine, which is shown by the blue-black color it produces with starch.

CONCLUSION

Microscaling of experiments attempts to provide an inexpensive, safe and time-effective means to keep Chemistry an investigative science. The five experiments cited in this paper use small amounts of reagents and therefore, reduce chemical consumption. Each of the experiments can be performed within a two hour laboratory period with ample time for the students to focus on the chemical principles involved. This reduction in the scale has been shown to give similar results as the macroscale analogues.

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