EXPERIMENT NO.:5

Aim:- To study potentiometric titration of Fe2+ versus Ce4+.

Chemicals:- Ferrous solution 0.1N, Cerium solution 0.1N

Apparatus:- Beaker, Burette, measuring cylinder.

Procedure:- For each part of the experiment, a quick titration should be done in order to establish the approximate volume of titrant required to reach the end point. Then three careful titrations will be performed. Because the redox reaction is a little slow, the titrant must be added relatively slowly, (e.g., 1 mL in 10 seconds when far from the endpoint) even after the approximate endpoint is known. Each student should perform two titrations for the standardization part of the experiment. A 0.05 M Ceric ammonium sulfate will be provided. Students should take 300 mL of the solution per pair. The solution must be standardized, and then the same solution must be used for the iron ore determination.

Preparation of Standard Iron

Accurately weigh four 0.045 - 0.050 g portions of reagent grade Fe powder. Avoid clumps in the powder since they may contain Fe2O3. Transfer each to a 200 mL Erlenmeyer flask and add 15 mL of concentrated HCl. Apply gentle heat in the hood until the powder is completely dissolved. Note that the product of this reaction is FeCl3, and the iron is in the Fe3+ oxidation state. But the titration reaction requires Fe2+ so the iron must be reduced before it can be titrated. This is referred to as pre-reduction. Have everything set up so that the pre-reduction of the iron and the ceric ion standardization or titration can be carried out in rapid succession.

Pre-reduction of Iron

Heat the FeCl3 solution gently and add stannous chloride (0.67 M SnCl2 in 6 M HCl) dropwise until the yellow color of FeCl3 just disappears, then add 2 drops in excess. Add 75 mL of 2 M HCl, mix well, and rapidly add 8 mL of 5 % HgCl2 while stirring the beaker contents. Allow the beaker to stand 5 minutes. (A small amount of white precipitate should form. If no precipitate forms, not enough SnCl2 was added. If a black precipitate forms, too much SnCl2 was added. In either case discard the solution and prepare a new solution.) Proceed with the ceric ion standardization.

Titration with Ce4+ Solution

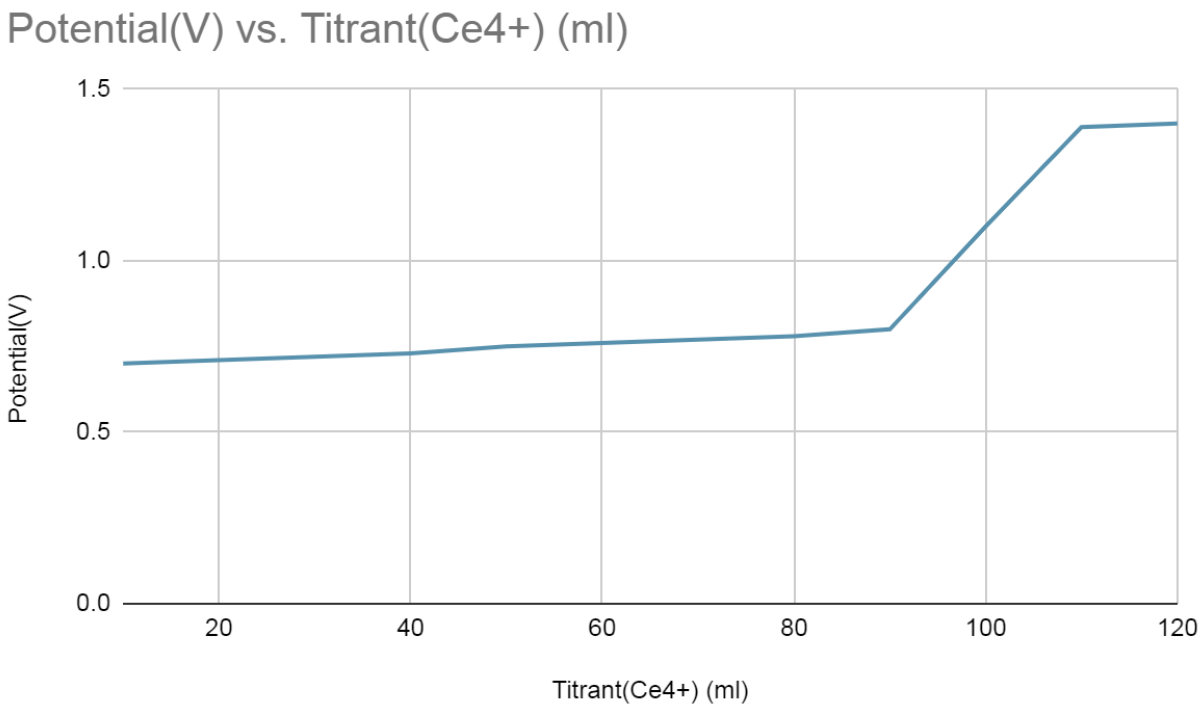
(Be sure to take enough Ce4+ solution to do all the titrations for the entire experiment. Each pair of students should require about 250 mL of this solution.) After allowing the Fe solution to stand for 5 minutes, add 3 - 4 drops of orthophenanthroline indicator solution. Titrate to the disappearance of the red color. Carry a blank solution through the entire analysis, and if necessary, apply a correction for the blank. Determine the concentration of the standard Ce4+ solution.

Analysis of Unknown Iron Sample

A cool, dry ore sample will be provided by the laboratory instructor. Weigh out 0.9 - 1.1 g into clean dry 200 mL Erlenmeyer flask, add 25 mL of concentrated HCl, cover with plastic wrap with a small hole in the wrap (and allow to stand until the following laboratory period - check lab schedule for whether 1 or 2 weeks is allotted for this experiment). Warm the solutions if they have not completely dissolved. When the sample is in solution, transfer it to a 250 mL volumetric flask and mix. Rinse the Erlenmeyer flask 3 times with ~ 25 mL of DI water and transfer the rinse water to the volumetric flask. Dilute the solution to volume. Transfer four 25 mL aliquots of the solution to four 200 mL Erlenmeyer flasks. Warm the solution gently and perform the pre-reduction of the iron as described above. Once the samples have been pre-reduced, you may proceed with the titration of the iron ore samples with Ce4+ as described above. Report the average Fe content of the ore as percent Fe2O3.

Observation table:-

Sr no.	Titrant(Ce4+) (ml)	Potential(V)
1	10	0.70
2	20	0.71
3	30	0.72
4	40	0.73
5	50	0.75
6	60	0.76
7	70	0.77
8	80	0.78
9	90	0.80
10	100	1.10
11	110	1.39
12	120	1.40



Result :-

1	Equivalent Point of titration	90ml
2	Potential at Equivalence point	0.80V