Task 1

In this problem you will identify a metal using complexometric titrations. EDTA (the disodium salt of ethylene-diamine-tetraacetic acid) forms stable complexes with most di- and trivalent metal ions.

$M^{2+} + H_2Y^{2-} = MY^{2-} + 2\ H^+$ $M^{3+} + H_2Y^{2-} = MY^{-} + 2\ H^+$

where M is the metal and Y^{4-} is the anion formed from EDTA.
While there is an excess of uncomplexed metal ions present, they bond to the indicator molecules. At the end of the reaction all ions form an EDTA complex and the indicator molecules will be liberated bringing about a change in color. Thus the end point of the reaction is when addition of EDTA does not change the color of the solution. One has to titrate until a steady color is reached. A previously titrated sample can be used for comparison.
First a metal sample is dissolved in nitric acid. After setting the pH of the resulting solution to approximately 5.7 by adding 50 ml of given buffer solution, it is titrated with EDTA. In the second part of the measurement, the oxide of the metal is dissolved and the solution is titrated again with EDTA.
Many solutions used are very acidic, treat them with caution!

Titration of the metal

Weigh accurately 100 to 150 mg of the unknown metal into a beaker. Working under the hood, carefully add 5 cm^3 diluted nitric acid. Heat the sample slightly, use watch glass! Complete dissolution may take 10 minutes. After complete dissolution of the sample, remove the watch glass and reduce the volume of the solution by half with heating. Dilute the solutions to 25 cm^3 with distilled water and transfer the solution to 100 cm^3 volumetric flask. Pipet 10 ml of the solution into a beaker, add 50 cm^3 of buffer solution and add ten drops of xylenol orange indicator. Titrate with 0.02 mol/dm^3 EDTA until a steady yellow color is attained. Repeat as necessary.

Titration of the metal oxide

Weigh accurately about 100 to 150 mg of the oxide, dissolve, dilute and titrate as for the metal in previous step.

a) Identify the metal based on calculations.

b) Give the formula of the oxide.

Remarks: The best marks are not necessarily awarded to results reproducing the theoretically expected values.

Reagent	Concentration	R phrases	S phrases
EDTA disodium salt	~0.02 mol/dm^3[1]	36/38	26-36
Diluted nitric acid	65 %	35	23-26-36-45
Buffer solution	pH 5.7	34-50	26-36/37/39-45-61
Xylenol orange			

[1] Please see the exact concentration from the bottel given to you.

Task 2

Caution: Handle all unknown solutions as if they were toxic and corrosive. Discard them only in the appropriate waste container.

You have seven unknown aqueous solutions. Each solution contains only one compound. The same ion may appear in more than one solution. Every compound formally consists of one type of cation and one type of anion from the following list:

Cations: H^+, NH_4^+, Na^+, Mg^{2+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Cu^{2+}, Ag^+, Ba^{2+}, Pb^{2+}

Anions: OH^-, CO_3^{2-} , CH_3COO^-, NO_3^-, F^-,SO_4^{2-}, S^{2-}, Cl^-, ClO_4^-, MnO_4^-, Br^-, I^-; $Cr_2O_7^{2-}$; CrO_4^{2-}

You have test tubes, distilled water and pH paper.

Identify the compounds in the solutions **1**-**7**. You can use the solubility table for some of the anions on the next page. If you are unable to identify an ion exactly, give the narrowest selection possible.

Remarks:

The unknown solutions may contain minor impurities arising from their exposure to air. The concentration of all solutions is around 5 % by mass so you can expect clearly observable precipitates from the main components. In some cases, precipitation does not occur instantaneously; some substances may remain in an oversaturated solution for a while. Don’t draw negative conclusions too hastily, wait 1-2 minutes where necessary. Always look carefully for all signs of a reaction.

Solubility Table at 25 °C

	NH_4^+	Na^+	Mg^{2+}	Fe^{2+}	Fe^{3+}	Ni^{2+}	Cu^{2+}	Ag^+	Ba^{2+}	Pb^{2+}
CH_3COO^-							HR	1.0		
OH^-				↓ ((Y))	↓ (R)	↓ (B)	↓	-		↓
NO_3^-										
F^-			↓	↓ (W)	↓ (W)	2.6	↓		0.16	↓
SO_4^{2-}								0.84	↓	↓
S^{2-}				↓ (Br)	-	↓ (B)	↓ (B)	↓ (B)		↓ (B)
Cl^-								↓		1.1
ClO_4^-										
MnO_4^-	HR			R				0.91		↓ R
Br^-								↓ ((Y))		0.98
I^-					R		↓ R	↓ ((Y))		↓ (Y)

No entry: Soluble compound ↓: Insoluble compound **R**: Redox reaction at room temperature
HR: Soluble at room temperature. In hot solution a reaction with an observable effect (not necessarily a precipitate) takes place.
Solubilities in g (substance) / 100 g water. Accurately known values between 0.1 and 4 are shown only.
Precipitates whose colour significantly differs from that of their hydrated ions: (**B**) = black, (**Br**) = brown, (**G**) = green, (**R**) = red, (**P**) = purple, (**W**) = white, ((**Y**)) = pale yellow, (**Y**) = yellow.

Task 1 25 % of the total

1b	1d	1e1	1e2	Task 1

a) Metal weighted:

b) Volume of the titrant consumed during metal solution titration:

Average:

c) Metal oxide weighted:

d) Volume of the titrant consumed during oxide titration:

Average:

e) Calculations for the metal and for the oxide:

Calculations for metal:

Calculations for oxide:

Metal identified:

Task 2 15 % of the total

Task 2
108

Only fill out this table when you are ready with all your assignments.

	1	2	3	4	5	6	7
Cation							
Anion							