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Complexometric determination of Nickel (II) in its synthetic alloys using selected Hydroxytriazene as Metallochromic indicator

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Abstract

A simple, rapid and reasonable selective Complexometric technique for nickel (II) determination using some selected hydroxytriazene as a metallochromic indicator is reported in the present study. The colour change at the end point was from greenish-yellow/yellow to colourless with sharp end point. The pH ranges were 9.3-9.7, 9.0-9.5, 8.5-9.0, 8.0-8.5 while temperature ranges were 25-60, 25-60, 25-60, 25-50 and 25-50 ^{0}C for reagent (i), (ii), (iii), (iv), and (v) respectively. Nickel(II) was determined accurately up to concentration as low as 3.0x10^{-3}M for reagents (ii), (iv), and (v)) while for reagents (i) and (iii) the concentration range could be even lowered to 1.0x10^{-3}M for the determination of nickel (II). The ions such as Cl^-, Br^-, CH_3COO^-, CO_3^{2-}, PO_4^{3-}, SO_4^{2-}, $C_2O_4^{2-}$, $S_2O_3^{2-}$, NO^{2-}, SO_3^{2-}, S^{2-}, HPO_4^{2-}, F^-, NO^3, WO_4^{2-}, $MO_7O_{24}^{6-}$, I^-, NH_4^+, Na^+, K^+ did not show any interference in the determination of nickel (II) even when they were present in tenfold excess. Ba^{2+}, Mg^{2+}, Ca^{2+}, were tolerated up to five-fold excess. However Mn^{2+}, Pb^{2+}, Hg^{2+}, Sn^{2+}, Th^{4+}, Cd^{2+}, Co^{2+}, Cu^{2+},Zn^{2+}, interfered even at equivalent amount. The method was used to determine nickel in its synthetic alloy with maximum relative error of 0.78 when using secondary masking agent.

Keywords: Hydroxytriazenes; Nickel; Complexometric Techniques; Synthetic Nickel Alloy; Secondary Masking Agent; Foreign Ions; Colour Change

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1. Introduction

Nickel is a silver-white metal which occurs in nature as a mixture of five stable isotopes (Ombaka, 2018). Pure Nickel is used as a protective coating for a number of elements and industrial equipment in order to enhance the value, utility and lifespan of these facilities due to its resistant to corrosion in water or air (Alan, 1992; Nigam et al., 2015). It's used in the chemical and food processing industries to prevent iron from being contaminated. Furthermore, nickel properties can be controlled and varied over broad ranges. Nickel plating finds numerous applications in industry.

Nickel (II) aqueous solution without strong complexing agents consist of green hexaaquanickel (II) ions. Nickel (II) forms 4, 5 and 6 coordination numbers with a number of ligands and the structural types includes: octahedral, trigonal-bipyramidal, square pyramidal, tetrahedral and square. Nickel (II) complexes are temperature dependent, sometimes concentration dependent and often exist between the above structural types and this complicates their equilibria (Albert and Geoffrey, 1980).

Nickel based alloys find broad applications due to their unique properties. For example, the high resistivity and heat resistance of nickel-chromium alloys makes them to be used in electric resistance heating elements. The nickel-iron alloys have soft magnetic properties which allow them to be utilized in electronic devices and for electromagnetic shielding of computers and communication equipment. Low expansion behaviour of iron-nickel alloys caused by a balance between thermal expansion and magnetostrictive changes with temperature allows them to be used as lead frames in packaging electronic chips and as the shadow-masks in colour television tubes. The requirement of the thermal expansion during storage and transportation tanks for the growing liquid natural gas industry can be solved by using iron-nickel alloys. The combination of good mechanical properties, chemical resistivity and biocompatibility allows nickel based alloys to be used in the fields of dentistry and implantology (Rader, 2012). The other properties which makes nickel alloys to be popular comprises of shape memory characteristics of equiatomic nickel-titanium alloys which makes them to be used as actuators, hydraulic connectors and eyeglass frames. The high strength at high temperatures and resistance to stress relaxation which contributes wrought nickel-beryllium-titanium to be employed for demanding electrical/electronic applications. The high conductivity and near resistance make cast nickel-beryllium-carbon alloys to be utilized for tooling in glass forming operations (John, 1990).

The nonferrous alloys which nickel constitutes either as a major or minor percentage can be classified as given below: High Nickel alloys {Nickels(nickel 200, nickel 201, nickel 204, nickel 205, nickel 211, nickel 220, nickel 230, nickel 270, Durannickel alloy 301, permanickel alloy 300), nickel-chromium alloys, nickel-chromium-iron alloys, Hastelloy alloys, superalloys, nickel-copper alloys}; Low nickel alloys {copper-nickel alloys, copper-nickel-zinc alloys (nickel-brasses) and miscellaneous alloys i.e. those which do not fall in any of the categories above which include, Ferrous alloys {wrought steels (low-alloys steels, ultra high strength structural steels maraging steels, special steels), cast steels and irons{cast steels, cast iron}, stainless steels {martenistic stainless steels, austenitic stainless steels, precipitation-hardening stainless steels, thermal expansion and constant modulus alloys, magnetic alloys {magnetically soft materials}, permanent magnets (Samuel, 1968).

Karpet et al. (2015) studied the effect of the nickel concentration on the phase composition, microstructure and hardness of VCrMnFeCoNi (x=0-3) high entropy alloys and the results revealed that an increase of the nickel concentration decreases the volume fraction of the beta-phase. Mathew et al. (2017) investigated effect of Ni-content on phase transformation behaviour of NiTi-20 at % Zr high temperature shape memory alloy and the findings indicated that, variations in Ni-content contributed to vastly different transformation temperatures and responds in a drastically different manner to aging treatment at 550 and 600^{o}C. This shows that, monitoring the levels of nickel in various materials which have applications in the different fields is necessary.

A large number of classical methods are available for the quantitative determination of nickel. These methods suffer from interference from ions which are commonly encountered in various materials containing nickel, which makes them to be inferior to the Complexometric methods (Abraham and Narayana, 1991). Some of the Complexometric techniques involves the use of masking agents, solvent extraction, demasking, classical separation, pH adjustment, heating, kinetic masking and removal of anions which makes the procedure not cost effective and time consuming. Furthermore, metal ion indicators like murexide, solochrome black T and bromophrogallol red which are commonly used for the determination of nickel are expensive and not easily synthesized in the normal laboratory. Jingying (2016) reported the use of a new class of chelators and indicators based on highly selective ionophores embedded in ion-selective nanosphere emulsions with the emulsion with view of achieving titrations in situ by complete instrumental control, thin layer electrochemistry. This technique has a weakness pertaining increase of the cost hence a number of developing countries might not be able to put this technique into practice. In view of the above there is serious need for continuous search for a technique which is simple, cost effective, less time consuming and can be implemented in developing countries.

Hydroxytriazenes are a class of metallochromic indicator which coordinate with the number of transition and non-transition metals via N-OH and –N-N=N- group (Ombaka et al., 2013).

The utility of hydroxytriazene as a metallochromic indicator was demonstrated by the studies carried out by Krishna et al. (2001) and Ombaka et al. (2013). These studies showed that, metal ions like chromium, iron can be determined successfully in presence of varying quantities of foreign ions (cations and anions). However, literature reveals that no attempt has been made to use hydroxytriazene as metallochromic indicator for determination of Nickel (II) in various material despite its advantages like easy to synthesize and high yield. In view if this, the research has explored possibility of utilizing hydroxytriazene as metallochromic indicator in determination of Nickel (II) using EDTA as a titrant in the presence of foreign ions and its synthetic alloys.

2. Material and methods

2.1. Apparatus, chemicals and reagents

pH meter (HI 2211 pH/ OR meter) HANNA Instruments, ultra-pure water equipment (Model MILLI-Q equipped with Q-POD), analytical grade chemicals, ultra pure water.

2.2. Preparations of solutions

2.2.1. Preparation of standard solution of 0.01m Zn2+

Appropriate amount of AR zinc pellets was accurately weighed and then transferred into a 50ml volumetric flask, dissolved in a minimum amount of concentrated hydrochloric acid. This was followed by addition of a few drops of concentrated nitric acid in order to speed up the rate of dissolution. The solution was then diluted to the mark using ultra-pure water (Vogel, 1961; Mendham, 2000).

2.2.2. Preparation of indicator solutions

Murexide indicator solution was prepared by suspending 0.5g of powdered murexide indicator in ultra-pure water followed by thorough shaking. The undissolved solid was allowed to settle. The saturated supernatant liquid was used for titration. Xylenol orange indicator solution was prepared by dissolving 0.005g of xylenol orange in 10ml of ultra-pure water. 0.1M hydroxytriazene indicators solutions were prepared by dissolving each hydroxytriazene in the required quantity of ethanol (AR) (Vogel, 1961; Mendham, 2000).

2.2.3. Preparation of 0.01M EDTA

1000ml of EDTA stock solution was prepared by dissolving the required quantity of AR disodium dihyrogen ethylenediaaminetetraacetate dehydrate which was initially dried at 80^0C for 2 hours in ultra pure water. This was followed by taking 25ml of the zinc ion solution into a conical flask and diluted with ultra pure water to about 75ml and 3 drops of xylenol orange indicator solution was added. Then powdered hexamine was added, with agitation, to the resulting yellow solution until it acquired intense red colour. This solution was titrated using EDTA solution as the titrant at pH range 6-7. Weaker solutions were prepared by proper dilution of the stock solution using ultra-pure water (Vogel, 1961; Mendham, 2000).

2.2.4. Preparation of a stock solution of 0.01M Ni2+ and 1M ammonium chloride solution

A 1000ml of 0.01M Ni^{2+} solution was prepared by taking the required amount of nickel chloride hexahydrate of B.D.H, AR grade in ultra pure water. A few drops of concentrated hydrochloric acid were added to prevent hydrolysis. 25ml of this solution was transferred into a conical flask and diluted to 100ml with ultra pure water. 5-6 drops of freshly prepared murexide indicator was added, followed by 10ml of 1M ammonium chloride solution. Thereafter, concentrated ammonia solution was added drop wise until the pH was about 7 (until the solution changes to yellow). The solution was titrated using a standard solution of 0.01M EDTA until the end point was approached and then, the solution was rendered strongly alkaline with about 10ml of concentrated ammonia solution and the titration continued until the colour changed from yellow to bluish-violet. The EDTA solution was added drop wise near the end point since nickel complexes react rather slowly with EDTA. 20ml of 1M NH_4Cl was prepared by dissolving appropriate amount of AR NH_4Cl in ultra-pure water (Vogel, 1961; Mendham, 2000).

2.2.5. Preparation of 1% tris-buffer and 1.0% perchloric acid solution

1% tris – buffer solution was prepared by dissolving 1.0g of tris-buffer in minimum quantity of ultra-pure water followed by dilution to 100ml using AR ethanol. 1.0% perchloric acid solution was prepared by adding 1.0ml of perchloric acid to 100ml of AR ethanol (Vogel, 1961; Mendham, 2000).

2.3. Procedures

2.3.1. Effect of pH variation

The titration of nickel (II) was carried out by performing titration at various pH using 1.0% tris-buffer solution and 1.0% perchloric acid solution. For each of the hydroxytriazene, the optimum pH range was determined where the colour change at the end point was sharp and most perceptible

2.3.2. Effect of temperature

The effect of temperature was studied by titration of Nickel (II) in the temperature range of 25-60^{0}C. The optimum temperature where the end point was perceptible and sharp for hydroxytriazene was noted.

2.3.3. Optimum concentration range of Nickel (II)

The optimum concentration range for each indicator was determined by titrating 0.001M to 0.01M Ni^{2+} solution against equimolar solution of EDTA so as to ascertain the minimum concentration of Nickel(II) which can be determined using hydroxytriazene as indicator.

2.3.4. Interference studies

Interference of 34 diverse ions in various amounts has been studied for Complexometric determination of nickel (II) with each hydroxytriazene as indicators. For this, titrations of nickel (II) solution containing 2.9355mgs were performed against equimolar EDTA solutions under optimum conditions using hydroxytriazene as indicator. The same titration was repeated in presence of equivalent amounts of the interfering species. The ions which did not interfere at equivalent amount were studied for their interference at five fold excess. Further tenfold excess of these species for those ions only which did not interfere up to five fold excess. No interference studies were performed beyond tenfold excess. For the preparation of sodium, potassium and ammonium salts of respective anions used. For the preparation of cation solutions respective chloride, nitrate salts were used.

2.3.5. Titration procedure

A 10ml of nickel (II) solution was diluted to 30ml with ethanol-water mixture. The corresponding pH was adjusted with 1.0% tris-buffer solution and 1.0% perchloric acid solution. 5-6 drops of hydroxytriazene were added which resulted in instantaneous development of greenish-yellow [(reagent number (i) and (ii) or yellow (reagent number (iii), (iv) and (v)]. The solution was very slowly titrated at room temperature with equimolar solution of EDTA. Close to the end point, one or two drops of indicator were further added to this solution to enhance the perceptibility of the end point. The end point where colour sharply changed to

colourless was recorded as the end point in all the cases. These results were compared with the results obtained using murexide as indicator.

3. Results and discussion

Results of the effects of concentration under optimum condition are shown in table 1.

Table 1. Determination of Nickel (II) at different concentrations

Concentration of Nickel(II)ions: Concentration of EDTA					$1.0x10^{-3}M$ $1.0x10^{-3}M$	$5.0x10^{-3}M$ $5.0x10^{-3}M$	$3.0x10^{-3}M$ $3.0x10^{-3}M$
S/no	Indicator	Colour change at the end point	pH range	Temperature range ^{o}C	Titre value in ml		
(i)	3-Hydroxy-3-0-Tolyl-1-0-carboxyphenyltriazene	Greenish yellow to colourless	9.3-9.7	25.0-60.0	10.0	10.0	10.0
(ii)	3-Hydroxy-3-m-Tolyl-1-0-carboxy-phenyltriazene	Greenish yellow to colourless	9.0-9.5	25.0-60.0	10.0	10.0	9.7
(iii)	3-hydroxy-3-m-tolyl-1-p-sulphanato (sodium salt) phenyltriazene	Yellow to colourless	9.0-9.5	25.0-60.0	10.0	10.0	9.9
(iv)	3-Hydroxy-3-o-Tolyl-1-m-hydroxyphenyltriazene	Yellow to colourless	8.50-9.0	25.0-50.0	10.0	10.0	8.8
(v)	3-Hydroxy-3-phenyl-1-m-hydroxyphenyltriazene	Yellow to colourless	8.0-8.5	25.0-50.0	10.0	10.0	9.3

Titration of Nickel (II) solution=10.0ml; Titer value for n=3 obtained using murexide as indicator=10.0ml

The colour change at the end point of reagent (i) and (ii) was greenish-yellow to colourless while for reagents (iii) and (iv), and reagent (v) was yellow to colourless. The change in colour for reagents (i), (ii) and (iv) was very sharp whereas for reagents (iii) and (v) the change in colour at the end point was found to be sharp. The pH range of titration for reagent (i), (ii), (iii), (iv), and (v) were 9.3-9.7, 9.0-9.5, 8.5-9.0, 8.0-8.5 respectively. The results revealed that Nickel (II) can be determined with high accuracy in the temperature

range of 20-60^{0}C for reagents (i), (ii) and (iii) while for reagents (iv) and (v) requires a temperature range of 25-50^{0}C above which colour with indicator faded and therefore clarity at end point diminished.

The minimum concentration of Nickel (II) in the solution which could be determined using each of these hydroxytriazenes as indicators are summarized in table 1. The results show that Nickel (II) can be determined accurately using these hydroxytriazenes up to concentrations as low as 3.0x10^{-3}M. In case of reagents (i) and (iii), concentration range could be even lowered to 1.0x10^{-3}M for the determination of Nickel (II) while for the remaining three hydroxytriazene end points were not very clear below 3.0 x10^{-3}M. A close examination of the data presented in table 1, it can be deduced that reagents (i) and (iii) can be used as indicators for Complexometric determination of Nickel (II) at low concentration hence they have advantages over other the three hydroxytriazenes used in the present investigation. Further, wide temperatures range, fairly reasonable pH range and very sharp end point are some of the characteristic features of these hydroxytriazenes as indicators. The pH range when reagent (iii) is used as indicator is slightly greater than that of reagent (i), making it to have an extra advantage.

3.1. Effect of foreign ions

The effect of various cations and anions in the determination of Nickel (II) was studied in order to establish the suitability of the proposed method for quantitative determination of Nickel (II) in nickel alloy. These were performed by adding different amounts of diverse ions to a solution containing 2.9355mgs of Nickel (II) and then determined quantitatively using hydroxytriazene as indicator. The tolerance levels of the various foreign ions studied are show in table 2.

Table 2. Effect of diverse ions in Nickel (II) determination

Diverse ions	**Indicator (i)**	**Indicator (ii)**	**Indicator (iii)**	**Indicator (iv)**	**Indicator (v)**
	pH range 9.3-9.7	**pH range 9.0-9.5**	**pH range 9.5-9.0**	**pH range 98.5-9.0**	**pH range 8.0-8.5**
	Mole ratio Diverse ion: Nickel(II)	**Mole ratio Diverse ion: Nickel(II)**	**Mole ratio Diverse ion: Nickel(II)**	**Mole ratio Diverse ion: Nickel(II)**	**Mole ratio Diverse ion: Nickel(II)**
Cl^-	10	10	10	10	10
Br^-	10	10	10	10	10
CH_3COO^-	10	10	10	10	10
CO_3^{2-}	10	10	10	10	10
PO_4^{3-}	10	10	10	10	10
SO_4^{2-}	10	10	10	10	10
$C_20_4^{2-}$	10	10	10	10	10
I^-	10	10	10	10	10
$S_20_3^2$	10	10	10	10	10
NO_2^-	10	10	10	10	10
SO_3^{2-}	10	10	10	10	10

S^{2-}	10	10	10	10	10
HPO_4^{2--}	10	10	10	10	10
F^-	10	10	10	10	10
NO_3^-	10	10	10	10	10
WO_4^{2-}	10	10	10	10	10
$MO_70_{24}^{6-}$	10	10	10	10	10
NH_4^+	10	10	10	10	10
Na^+	10	10	10	10	10
K^+	10	10	10	10	10
U^{6+}	1	1	1	1	1
Mn^{2+}	10	10	10	10	10
Ba^{2+}	5	5	5	5	5
Pb^{2+}	-	-	-	-	-
Hg^{2+}	-	-	-	-	-
Sn^{2+}	-	-	-	-	-
Th^{4+}	-	-	-	-	-
Cd^{2+}	-	-	-	-	-
Mg^{2+}	5	5	5	5	5
Ca^{2+}	5	5	5	5	5
Zr^{4+}	1	1	1	1	1
CO^{2+}	-	-	-	-	-
Cu^{2+}	-	-	-	-	-
Zn^{2+}	-	-	-	-	-

- Indicates the ion interfered; Concentration of nickel (II) ions 5.0x10^{-3}M; Concentration of EDTA 5.0x10^{-3}M; Temperature of titration 25-30oC; Titration volume of Nickel (II) ions 10ml

An error of less than or equal to +/- 0.1ml in the recovery was considered to be tolerable. The results in this table suggest that, the pattern of interference for all hydroxytriazenes was identical. The results further indicates that ions such as Cl^-, Br^-, CH_3COO^-, CO_3^{2-}, PO_4^{3-}, SO_4^{2-}, $C_2O_4^{2-}$, $S_2O_3^{2-}$, NO^{2-}, SO_3^{2-}, S^{2-}, HPO_4^{2-}, F^-, NO^{3-}, WO_4^{2-}, $MO_7O_{24}^{6-}$, I^-, NH_4^+, Na^+, K^+ do not show any interference in the determination of nickel(II) even when they were present in tenfold excess. Ba^{2+}, Mg^{2+}, Ca^{2+}, were tolerated up to five fold excess. However, Mn^{2+}, Pb^{2+}, Hg^{2+}, Sn^{2+}, Th^{4+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Zn^{2+}, interfered even at equivalent amount. Some of these interference can be avoided by using appropriate secondary masking agent like guanidine, sodium oxalate, citric acid, fluoride and cysteine for Hg2+, Pd2+, Mn2+, (sN4+ and Fe3+) and Cu2+ respectively. The interference due to Fe3+, Al3+ and Cr3+ can be eliminated by use of citrate and tartrate. The results of interference demonstrate that the method can be employed in Complexometric determination of nickel (II) in various nickel alloys using appropriate secondary masking agent.

3.2. Determination of Nickel (II) in synthetic alloy using 3-hydroxy-3-m-tolyl-1-p-sulphanato (sodium salt) phenyltriazene

Various synthetic mixtures of nickel (II) with manganese, iron, copper, chromium or aluminium were prepared according to their compositions. The amount of Nickel (II) in the mixture was determined

complexometrically by help of appropriate masking agent using reagent (iii) as indicator. The results of these analyses are summarized in table 3 below.

Table 3. Determination of Nickel (II) in synthetic mixture of metal ions (n=3)

mixture	Ni present %	Ni found %	Relative error
Ni+Mn+Fe+Cu	66.50	66.67	+0.26
Ni+Mn+Fe+Cu+Cr	76.00	76.31	+0.41
Ni+Mn+Fe+Cu+Cr+Al	60.50	60.79	+0.48
Ni+Mn+Fe+Cu+Cr	36.00	36.28	+0.78

The results of table 3 show that the maximum relative error of the method was 0.78%. The tabulated for six degree of freedom (N_1+N_2-2) at the 95% confidence level was 2.447 and tabulated value was found to be 0.0217. From this it can be deduced that, there is no statistical difference between these results and therefore the proposed method can be used to provide reliable results.

4. Conclusion

Five new metallochromic indicators for direct Complexometric determination of Nickel (II) have been introduced. The results obtained by these five hydroxytriazenes are comparable to murexide as indicator. The interference studies clearly revealed that, direct Complexometric determination of Nickel (II) can be carried out in the presence of a large number of anions and cations using these five hydroxytriazenes with fair accuracy. Easy preparation, better yield, wide temperature range, fairly reasonable pH range and very sharp end point are some of the advantages of these five hydroxytriazenes over other metallochromic indicators. The proposed method can be utilized for the determination of Nickel (II) in its alloys with help of the secondary masking agent. 3-hydroxy-3-m-tolyl-1-o-carboxyphenyltriazene and 3-hydroxy-3-m-tolyl-1-p-sulphanato (sodium salt) phenyltriazene can find applications in those alloys which consist of low amount of nickel. The proposed method does not require heating, readjustment of pH or demasking agent. The proposed method is simple, rapid and reasonable selective and attribute to the method being attractive to a wide range of applications.

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