IJARSCT
ISSN (Online) 2581-9429
International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
Impact Factor: 7.301
Volume 3, Issue 1, March 2023

Hydroxytriazenes as Metallochromic Indicators for Palladium (II)

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Abstract: *A close survey of literature reveals that there is a continuous search for good new metallochromic indicators for palladium and that only a few new reagents have been reported since 1976 for complexometric determination of palladium. Further, no hydroxytriazene has so far been used for complexometric determination of palladium. In the present work an attempt has been made to use hydroxytriazenes as metallochromic indicators for palladium.*

Keywords: Complexometric determination metallochromic indicators..

I. EXPERIMENTAL

Standard solution of EDTA:
A stock solution of 0.05M EDTA (ethylene diamine tetra acetic acid disodium salt) was prepared by dissolving the requisite quantity of di-sodium salt of EDTA (BDH, AR) in double distilled water. The solution was standardised by titrating it with standard solution of zinc using xylenol orange as indicator at pH 6 to 7. The phi was adjusted with hexamine. weaker solutions were prepared by its proper dilution with water.

Standard solution of Ferric ammonium sulphate
A 0.05M stock solution of ferric ammonium sulphate was prepared by dissolving the requisite quantity of ferric alum [Fe_2 ($S0_4$).$(NH_2)_2S0_4$ $24H_2O$] of A.R. quality in 25 ml of dilute sulphuric acid and then diluting with distilled water to a definite volume. The ferric alum solution was adjusted to pH of about 2.5 and then standardised by titrating it with standard solution of EDTA using sulfosalicylic acid as indicator. weaker solutions were prepared by its proper dilution with water.

Standard solution of Palladium chloride :
A 0.05M stock solution of palladium (II) chloride (John Baker U.S.A.) was prepared by dissolving the requisite quantity of palladium chloride in the minimum volume of hot conc. HC1 and then diluting with distilled water to a definite volume. The solution is standardized complexo metrically. A known excess of EDTA is added to the known volume of $PdcCl_2$ solution, the pH is adjusted to about 2.5 with ammonium acetate and excess EDTA is then titrated with standard ferric ammonium sulphate solution using sulfosalicylic acid as indicator, weaker solutions were prepared by proper dilution.

Indicator solution:
Solution of each hydroxytriazene 0.1% or 0.05% concentra- tion was prepared by dissolving the requisite quantity of hydroxytriazene in ethanol or water as the case may be.
It was found that none of the ten hydroxytriazenes could be used as an indicator for direct complexometric determination of palladium. Therefore, an attempt has been made to make indirect complexometric determination of palladium by titrating the unconsumed excess EDTA with Fe(III) using hydroxytriazenes as indicators. The hydroxytriazenes used are :
(1) 3-Hydroxy-3-methyl-1-p-sulphonato (sodium salt) phenyl triazene.
(II) 3-Hydroxy-3-propyl-1-p-sulphonato (sodium salt) phenyl triazene.
(III) 3-Hydroxy-3-isopropyl-1-p-sulphonato (sodium salt) phenyl triazene.

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(iv) 3-Hydroxy-3-p-tolyl-1-p-sulphonato (sodium salt) phenyl triazene.
(v) 3-Hydroxy-3-p-chlorophenyl-1-p-sulphonato (sodium salt) phenyl triazene.
(vi) 3-Hydroxy-3-methyl-1-o-carboxy phenyl triazene.
(vii) 3-Hydroxy-3-ethyl-1-o-carboxy phenyl triazene
(viii) 3-Hydroxy-3-phenyl-1-o-carboxy phenyl triazene.
(ix) 3-Hydroxy-3-p-tolyl-1-o- carboxy phenyl triazene.
(x) 3-Hydroxy-3-phenyl-1-p-carboxy phenyl triazene.

The compound Nos. (i), (ii), (iii) and (v) are water soluble and their aqueous solutions were used as indicators. In order to use all the above hydroxytriazenes preliminary investigations have been made. The results have been reported in Table-1V-1. It was found that only seven compounds (i), (ii), (iii), (iv), (v), (vi) and (vii) were found to give sharp colour change at the end point. The remaining hydroxytriazene compound Nos. (viii), (ix) and (x) give a yellow precipitate, which was dissolved by addition of a little alcohol. On addition of Fe(III) solution, the solution becomes black in colour.

On further addition of F in (III) solution a turbidity appears and no change in colour could be observed even on addition of excess of Fe(III) solution. Thus, these three compounds failed to act as metallochromic indicators. It seems that Fe(III) forms more stable a complex with the reagent as compared to EDTA and as such excess EDTA cannot be titrated with Fe(III) solution using these hydroxytriazenes as indicators. The detailed investigation with remaining seven hydroxytriazenes have been worked out and described.

Effect of pH:

The effect of pH was studied and most accurate results were obtained between pH 3.0 to 3.5. The pH was adjusted with ammonium acetate.

Effect of temperature:

Under the optimum conditions of pH all the palladium determinations were made by titrating unconsumed EDTA using seven hydroxytriazenes as indicators at 25^{o}C and 60^{o}C . It is observed that the titre value does not change between 25^{o}C and 60^{o}C and therefore all the titrations were performed at room temperature. The results have been given in Table-IV-2.

Palladium concentration range :

Palladium solution of different concentrations 1x10^{-3}M, 2x10^{-3}M, 5x10^{-3}, 1x10^{-2}M, 2x10^{-2}M and 5x10^{-2}M. were prepared and EDTA solution of corresponding concentrations were also prepared. The excess EDTA was titrated with standard ferric ammonium sulphate solution of corresponding concentrations, using the seven hydroxytriazenes. The results have been given in Table-IV-3 to IV-9 Parallel determinations of excess EDTA were acid made with Fe(III) using sulfosalicylic acid as indicator The results are reported in the same tables and are identical.

General procedure :

For one concentration of Pd(II) details of procedure followed are given :

To a 3 ml of palladium (II) solution, (2x10^{-2}M) , 9 ml of EDTA (2x10^{-2}M) was added and the pH was adjusted to 3 to 3.5 with ammonium acetate. A few drops (5 to 6 drops) of indicator (0.1% solution of hydroxytriazene in ethanol or water as the case may be) were added to it. The excess EDTA was then titrated with standard solution of ferric ammonium sulphate (2x10^{-2}M) . A few drops of indicator were again added near the end point. The colour changes from yellow to bluish green or dark blue at the end point. For the sake of comparison the parallel titrations were carried out using sulfosalicylic acid as indicator.

The same general procedure was repeated for palladium con- centrations 1x10^{-3}M, 2x10^{-3}M, 5x10^{-3}M, 1x10^{-2}M, 1x10^{-2}M, 2x10^{-2}M, 2x10^{-2}M, and 5x10^{-2}M,

The results obtained have been given in Table Nos. IV-3 to iv - 9

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TABLE-IV-1
PRELIMINARY INVESTIGATION

Concentration of Palladium chloride solution	: $2x10^{-2}$M
Concentration of EDTA	: $2x10^{-2}$M
Concentration of Ferric ammonium sulphate	: $2x10^{-2}$M
Volume of Pd(II) solution taken	: 3.0 ml
Volume of EDTA solution added	: 9.0 ml
pH of the titration	: 3.0 -3.5
Colour change at the end point	: Yellow to bluish green/ dark blue

Compound Name of the indicator No.	Volume of ferric ammonium sulphateconsumed (m1)	Remarks
(i) 3-Hydroxy-3-methyl-1-p- sulphonato (sodium salt)	6.0	Good. sharp change in colour phenyl triazene
(ii) 3-Hydroxy-3-propyl-1-p- sulphonato (sodium salt)	6.0	Good. sharp change in colour phenyl triazene
(iii) 3-Hydroxy-3-isopropyl-1-p- sulphonato (sodium salt)	6.0	Good. sharp change in colour phenyl triazene Good. Sharp change in colour
(iv) 3-Hydroxy-3-p-tolyl-1-p-sulphonato (sodium salt)	6.0	Good. sharp change in colour phenyl triazene
(v) 3-Hydroxy-3-p-chlorophenyl-1- p-sulphonato (sodium salt)	6.0	Good. sharp change in colour phenyl triazene
(vi) 3-Hydroxy-3-methyl-1-o- carboxy phenyl triazene	-	Excellent. Sharp change in colour
(vii) 3-Hydroxy-3-ethyl-1-o- carboxy phenyl triazene		
(viii) 3-Hydroxy-3-phenyl-1-o- carboxy phenyl triazene	-	Solution becomes black and turbid on addition of Fe(III). End point not observ- able. do -
(ix) 3-Hydroxy-3-p-tolyl-1-o- carboxy phenyl triazene	-	do-
(x) 3-Hydroxy-3-phenyl-1-p- carboxy phenyl triazene-		-do-

TABLE-IV-2
EFFECT OF TEMPERATURE ON THE ACCURACY OF TITRATION

Concentration of Palladium chloride solution	: $1x10^{-2}$M
Concentration of EDTA	: $1x10^{-2}$M
Concentration of Ferric ammonium sulphate	: $1x10^{-2}$M
Volume of Pd(II) solution taken	: 3.0 ml
Volume of EDTA solution added	: 9.0 ml
pH of the titration	: 3.0 -3.5

Compound	Name of the indicator		Volume of Ferric ammonium sulphate consumed (in ml)
(i)3-Hydroxy-3-methyl-1-p- sulphonato (sodium salt) phenyl triazene		6.0	6.0
(ii) 3-Hydroxy-3-propyl-1-p- sulphonato (sodium salt) phenyl triazene		6.0	6.0
(iii)3-Hydroxy-3-isopropyl-1-p- sulphonato (sodium salt) phenyl triazene		6.0	6.0
(iv) 3-Hydroxy-3-p-tolyl-1-p- sulphonato (sodium salt) phenyl triazene		6.0	6.0
(v) 3-Hydroxy -3 - p - chlorophenyl- 1-p-sulphonato (sodium salt) phenyl triazene		6.0	6.0

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(vi) 3-Hydroxy-3-methyl-1-o- carboxy phenyl triazene 6.0 6.0
(vii) 3-Hydroxy-3-ethyl-1-o- carboxy phenyl triazene 6.0 6.0

TABLE-IV-3
COMPLEXOMETRIC DETERMINATION OF Pd (II) US ING 3-HYDROXY-3-METHYL- 1-p-SULPHONATO (SODIUM SALT) PHENYL TRIAZENE AS INDICATOR

For each concentration volume of Pd (II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxyriazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green
4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

TABLE-IV-4
COMPLEXOMETRIC DETERMINATION OF Pd(II) USING 3-HYDROXY-3-PROPYL- 1-D-SULPHONATO (SODIUM SALT) PHENYL TRIAZENE AS INDICATOR

For each concentration volume of Pd(II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxyltriazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green

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4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

TABLE-IV-5

COMPLEXOMETRIC DETERMINATION OF Pd(II) USING 3-HYDROXY-3-IS CPROPYL- 1-p-SULPHONATO (SODIUM SALT) PHENYL TRIAZENE AS INDICATOR

For each concentration volume of Pd(II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxytriazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green
4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

TABLE-IV-6

COMPLEXOMETRIC DETERMINATION OF Pd(II) USING 3-HYDROXY-3-p-TOLYL-1-p-SULPHONATO (SODIUM SALT) PHENYL TRIAZENE AS INDICATOR

For each concentration volume of Pd(II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxytriazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green

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4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

TABLE-IV-7

COMPLEXOMETRIC DETERMINATION OF Pd(II) USING 3-HYDROXY-3-p- CHLOROPHENYL-1-p-SULPHONATO (SODIUM SALT) PHENYL TRIAZENE AS

For each concentration volume of Pd(II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxytriazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green
4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

TABLE-IV-8

COMPLEXCMETRIC DETERMINATION OF Pd(II) USING 3-HYDROXY-3-METHYL-1-0-CARBOXY PHENYL TRIAZENE AS INDICATOR

For each concentration volume of Pd(II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxytriazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green

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4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

TABLE-1V-9
COMPLEXOMETRIC DETERMINATION OF Pa(II) USING 3-HYDROXY-3-ETHYL-1-0-CA OXY PHENYL TRIAZENE AS INDICATOR

For each concentration volume of Pd(II) solution taken = 3ml = 3.0 ml
Volume of EDTA solution of corresponding concentration added = 9.0 ml
pH of the titration = 3.0-3.5

S.No.	Conc. of Pd(11)	Conc. of solution EDTA Solution	Volume of Ferric ammonium sulphate of corresponding tration consumed (in ml)		Change of colour at the end point using hydroxy triazene as indicator
			Hydroxy- triazene as indi-	Sulfosalicylic acid as indicator	
1	$1x10^{-3}M$	$1x10^{-3}M$	6.0	6.0	Almost colourless to light green
2	$2x10^{-3}M$	$2x10^{-3}M$	6.0	6.0	Light Yellow to green
3	$5x10^{-3}M$	$5x10^{-3}M$	6.0	6.0	Light Yellow to dark green
4	$1x10^{-2}M$	$1x10^{-2}M$	6.0	6.0	Light Yellow to bluish green
5	$2x10^{-2}M$	$2x10^{-2}M$	6.0	6.0	Yellow to bluish green
6	$5x10^{-2}M$	$5x10^{-2}M$	6.0	6.0	Brownish yellow to bluish green

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