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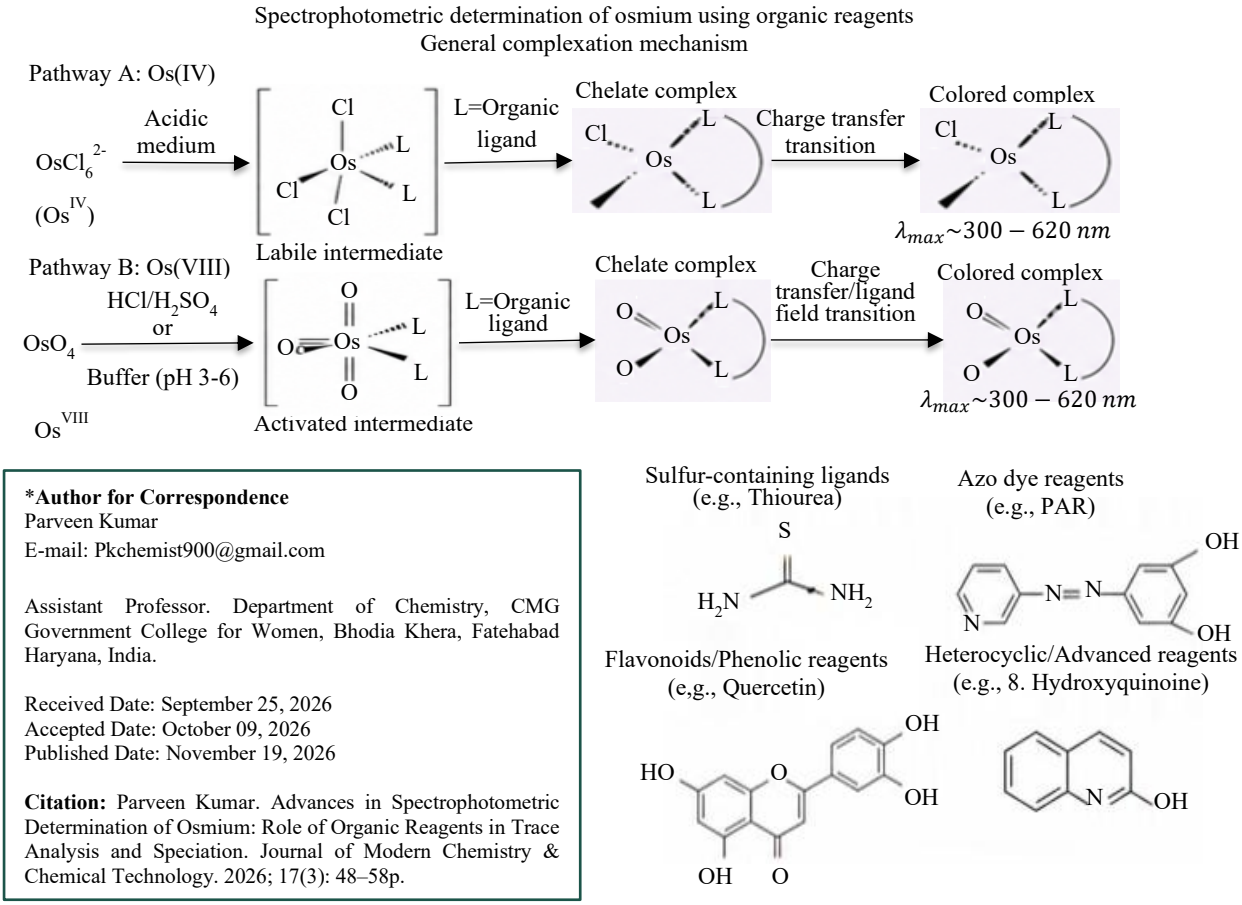
Advances in Spectrophotometric Determination of Osmium: Role of Organic Reagents in Trace Analysis and Speciation

Parveen Kumar*

Abstract

Osmium, a rare and highly toxic platinum-group metal, requires accurate and sensitive analytical determination due to its industrial importance and environmental hazards, particularly in the form of osmium tetroxide (OsO_4). Spectrophotometric techniques remain among the most versatile and cost-effective approaches for osmium analysis. This review provides a comprehensive evaluation of spectrophotometric methods employing a wide range of organic reagents, including sulfur-containing ligands, azo dyes, flavonoids, nitroso compounds, phenolics, and advanced catalytic systems. A total of 50 reagents are critically discussed with respect to their structural characteristics, coordination mechanisms, analytical performance, and applicability. Special emphasis is placed on sensitivity enhancement, selectivity, and recent developments such as derivative and catalytic spectrophotometry. The review also outlines current challenges and future directions in osmium analysis.

Graphical Abstract



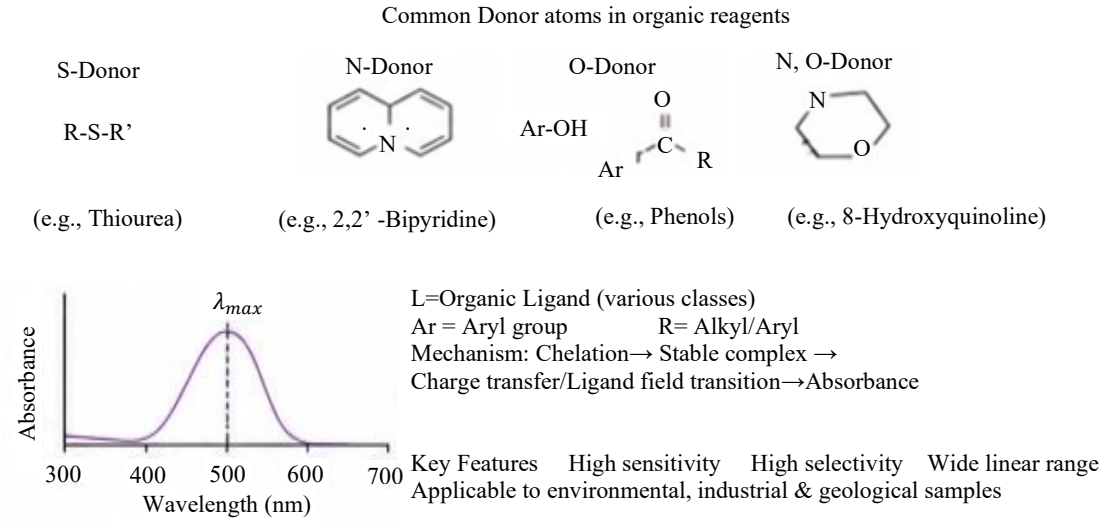
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Keywords: Osmium, spectrophotometry, azo dyes, thiosemicarbazones, catalytic methods, metal-organic complexes.

INTRODUCTION

Osmium (Os), a member of the platinum-group metals (PGMs), is extensively utilized in catalysis, electronic devices, and alloy manufacturing due to its exceptional physicochemical properties [1-3]. Despite its industrial significance, the analytical determination of osmium remains challenging, primarily due to its ability to exist in multiple oxidation states and the extreme toxicity associated with its highest oxidation state, osmium tetroxide (OsO_4) [4-5]. OsO_4 is highly volatile and a powerful oxidizing agent, necessitating the development of precise, sensitive, and reliable analytical techniques for its detection and quantification, particularly at trace levels [6].

Among the available analytical methods, spectrophotometry has emerged as a preferred approach owing to its simplicity, cost-effectiveness, and adaptability for trace analysis [7, 8]. The technique relies on the formation of colored complexes between osmium species and suitable organic reagents, which can be quantitatively measured in the UV–visible region [9]. Over time, a wide range of organic ligands has been developed, each offering specific advantages in terms of sensitivity, selectivity, and operational convenience [10]. These ligands not only enhance the detectability of osmium but also enable its determination in complex matrices.

From a chemical perspective, osmium exhibits a wide range of oxidation states, varying from −2 to +8, with Os(IV) and Os(VIII) being the most relevant in analytical applications [11]. In aqueous and acidic environments, osmium commonly exists as OsO_4 or as chloro-complexes such as $OsCl_6^{2-}$, along with other oxo-species [12]. The reactivity of osmium toward organic ligands is governed by several key factors, including its oxidation state, the pH of the medium, the nature of donor atoms present in the ligand (such as sulfur, nitrogen, and oxygen), and the redox characteristics of the ligand system [13].

A thorough understanding of these chemical parameters is essential for the rational design and selection of reagents in spectrophotometric methods. This review, therefore, systematically examines the role of various organic reagents in the spectrophotometric determination of osmium, with particular emphasis on their structural features, coordination behavior, and analytical performance.

EXPERIMENTAL METHODOLOGY

Preparation of Stock Solutions

A standard osmium stock solution (1000 μg mL^{-1}) was prepared from potassium osmate or osmium tetroxide using double-distilled water under controlled conditions. Owing to the high toxicity and

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volatility of OsO_4, all solution preparations were carried out in a well-ventilated fume hood. Working standard solutions were obtained by appropriate serial dilution of the stock solution.

Stock solutions of organic reagents (1×10^{-3}–1×10^{-2} M), including azo dyes, thiourea derivatives, flavonoids, and phenolic compounds, were prepared in distilled water, ethanol, or other suitable solvents depending on their solubility. Buffer solutions of the desired pH were prepared according to standard procedures.

INSTRUMENTATION

Absorbance measurements were carried out using a UV–Visible spectrophotometer (e.g., Shimadzu UV-1800) equipped with 1 cm matched quartz cells. The pH of the solutions was measured using a calibrated digital pH meter. All measurements were performed at the respective maximum absorption wavelength (λ^{max}) of the osmium–reagent complexes.

General Procedure

An appropriate aliquot of osmium solution was transferred into a calibrated volumetric flask, followed by the addition of a suitable organic reagent and buffer solution to maintain the required pH. The mixture was diluted to the mark with distilled water and allowed to stand for complete color development. The absorbance of the resulting solution was measured against a reagent blank at the corresponding λ^{max}. Quantitative determination was carried out using calibration curves constructed under identical experimental conditions.

SPECTROPHOTOMETRIC ANALYSIS OF OSMIUM USING ORGANIC REAGENTS

A wide range of organic ligands have been employed for the spectrophotometric determination of osmium, owing to their ability to form intensely colored and stable metal–ligand complexes.

Sulfur-Containing Reagents

Sulfur-containing ligands, particularly thiourea and thiosemicarbazone derivatives, are widely employed for the spectrophotometric determination of osmium due to the strong affinity of sulfur donor atoms toward soft metal centers such as Os(IV) and Os(VIII). These ligands typically coordinate through sulfur and nitrogen donor atoms, forming stable chelate complexes that produce intense coloration in the visible region via ligand-to-metal charge transfer transitions as shown in Figure 1.

- *Ethylene Thiourea (ETU):* Ethylene thiourea reacts rapidly with Os(VIII) in a strongly acidic medium (pH ≈ 1.0) to form a stable purple-colored complex exhibiting a maximum absorbance (λmax) at approximately 490 nm. The reaction proceeds instantaneously without the need for heating, making it highly suitable for rapid analytical applications. The complex formation is attributed to coordination through sulfur donor atoms, leading to charge-transfer transitions responsible for the observed color intensity. The method demonstrates excellent selectivity, with negligible interference from Fe^{3+} and other platinum-group metals [14].
- *Allyl Thiourea:* Allyl thiourea forms a moderately stable complex with Os (VIII) in acidic medium (pH 1–2), with λmax observed near 300 nm in the ultraviolet region. The coordination involves sulfur donation along with partial π-interaction from the allyl group, which enhances complex stability. However, due to relatively low molar absorptivity, the method exhibits limited sensitivity and is primarily employed for mechanistic and comparative studies rather than trace-level quantification [15].
- *Phenyl Thiourea:* Phenyl thiourea reacts with Os(IV) in acidic medium to produce a colored complex with λmax around 310 nm. The presence of the phenyl substituent enhances π-conjugation, thereby improving molar absorptivity compared to unsubstituted thiourea. The complexation mechanism involves coordination through sulfur atoms, with additional stabilization arising from π-electron delocalization within the aromatic ring system [16].
- *o-Methoxyphenyl Thiourea (OMePT):* o-Methoxyphenyl thiourea forms a stable complex with Os(IV) in 0.8 M HCl, exhibiting λmax at 518 nm. The methoxy group acts as an electron-

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donating substituent, enhancing electron density on the ligand and improving both complex stability and color intensity. This reagent is particularly suitable for extraction-spectrophotometric methods and demonstrates moderate molar absorptivity with good reproducibility [17].

- *Thiosemicarbazide:* Thiosemicarbazide reacts with Os(VIII) in acidic medium to form a colored complex with λmax around 360 nm. The ligand coordinates through both nitrogen and sulfur atoms, forming a stable five-membered chelate ring. Although the method provides only moderate sensitivity, it is widely used as a precursor for synthesizing more advanced thiosemicarbazone derivatives with enhanced analytical performance [18].
- *Benzaldehyde Thiosemicarbazone:* Benzaldehyde thiosemicarbazone forms a stable complex with Os(IV) in acidic medium, exhibiting λmax near 420 nm. The aromatic ring system enhances conjugation, resulting in increased molar absorptivity. Coordination occurs via azomethine nitrogen and sulfur atoms, producing a stable chelate structure with improved analytical sensitivity [19].
- *Salicylaldehyde Thiosemicarbazone:* Salicylaldehyde thiosemicarbazone forms a highly stable complex with Os(IV) in acidic medium, with λmax ≈ 430 nm. The presence of both hydroxyl and azomethine functional groups significantly enhances coordination strength through chelation. This reagent provides high sensitivity and is widely applied in trace-level determination of osmium [20].
- *Acetone Thiosemicarbazone:* Acetone thiosemicarbazone reacts with Os(VIII) to form a colored complex with λmax around 400 nm. The ligand exhibits moderate molar absorptivity and is suitable for routine spectrophotometric analysis where ultra-high sensitivity is not required [21].
- *2-Acetylpyridine Thiosemicarbazone:* This reagent forms a highly stable complex with Os(IV) in acidic medium, showing λmax near 440 nm. The pyridine ring introduces an additional nitrogen donor site, enhancing coordination strength, selectivity, and analytical sensitivity [22].
- *Thiourea:* Thiourea forms a simple complex with Os(VIII) in acidic medium, exhibiting λmax around 330 nm. Although it shows lower sensitivity compared to its substituted derivatives, its simplicity, availability, and ease of use make it a widely employed reagent in spectrophotometric analysis [23].

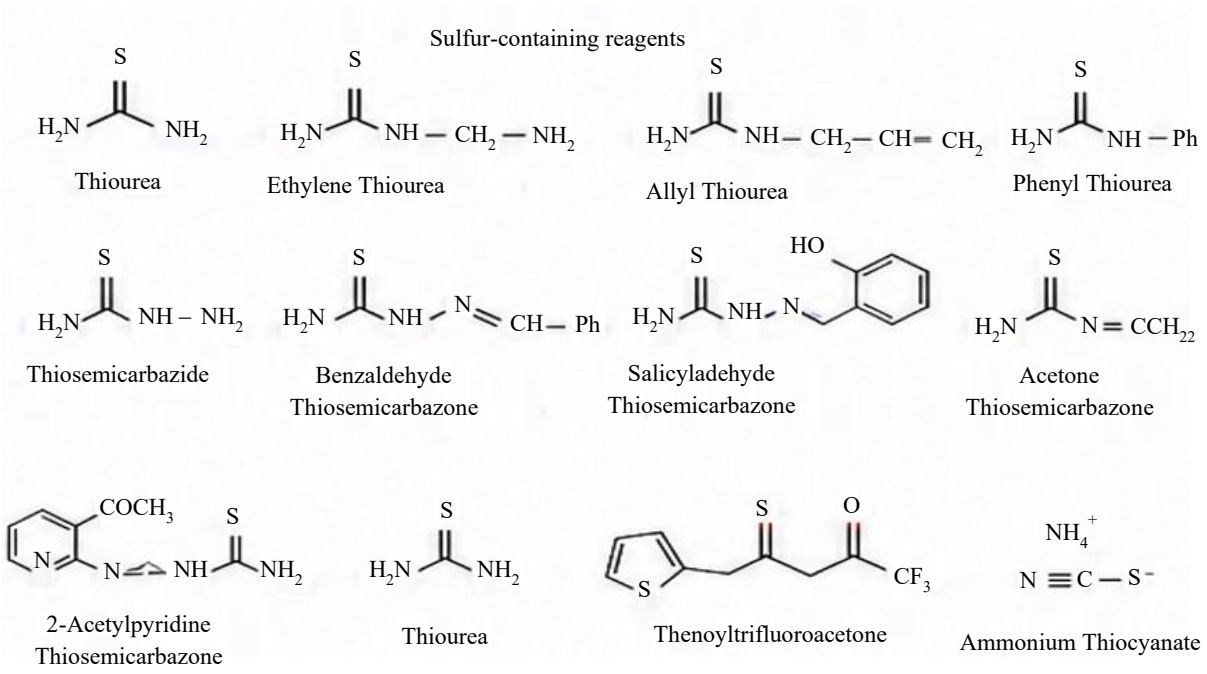


Figure 1. Sulfur-containing reagents.

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Azo Dye Reagents

Azo dyes represent one of the most important classes of chromogenic reagents for osmium determination due to their high molar absorptivity and intense coloration arising from extended conjugation as depicted in Figure 2.

- *4-(2-Pyridylazo)resorcinol (PAR):* PAR forms a stable chelate complex with Os(IV) in slightly acidic to near-neutral medium (pH ≈ 5.0), exhibiting λmax at ~520 nm. The coordination involves azo nitrogen and phenolic oxygen atoms, forming a five-membered chelate ring. The complex exhibits high molar absorptivity (~10^4 L mol^{-1} cm^{-1}), enabling sensitive trace-level determination with minimal interference [24].
- *1-(2-Pyridylazo)-2-naphthol (PAN):* PAN reacts with Os(IV) in acidic medium to form an intensely colored complex with λmax around 540 nm. The extended conjugation of the naphthol ring significantly enhances molar absorptivity. The reagent is particularly useful in extraction-spectrophotometric methods due to the formation of neutral complexes soluble in organic solvents [25].
- *Eriochrome Black T (EBT):* Eriochrome Black T forms a moderately stable complex with Os(IV) in acidic medium with λmax at ~540 nm. Although less sensitive than PAR and PAN, it offers rapid color development and operational simplicity, making it suitable for routine analysis [26].
- *Tropaeolin Dyes (O, OOO-I, OOO-II):* These dyes react with Os(VIII) in strongly acidic media, producing complexes with λmax values ranging from 480 to 510 nm. The presence of sulfonate groups enhances solubility, while increased conjugation improves analytical sensitivity [27-29].
- *Xylenol Orange:* Xylenol Orange forms a highly stable complex with Os(IV), exhibiting λmax at ~560 nm due to coordination through multiple donor sites, resulting in high stability and sensitivity [30].
- *Arsenazo III:* Arsenazo III produces intensely colored complexes with Os(IV), exhibiting λmax at ~600 nm. The presence of multiple azo and sulfonic groups ensures strong binding and exceptional sensitivity [31].
- *Methyl Orange:* Methyl Orange forms a weak complex with Os (VIII), exhibiting λmax at ~470 nm. Due to low molar absorptivity, it is mainly used for qualitative or preliminary studies [32].
- *Congo Red:* Congo Red forms a moderately stable complex with Os (VIII), exhibiting λmax around 500 nm. While it shows moderate sensitivity, it is useful in comparative analytical studies [33].

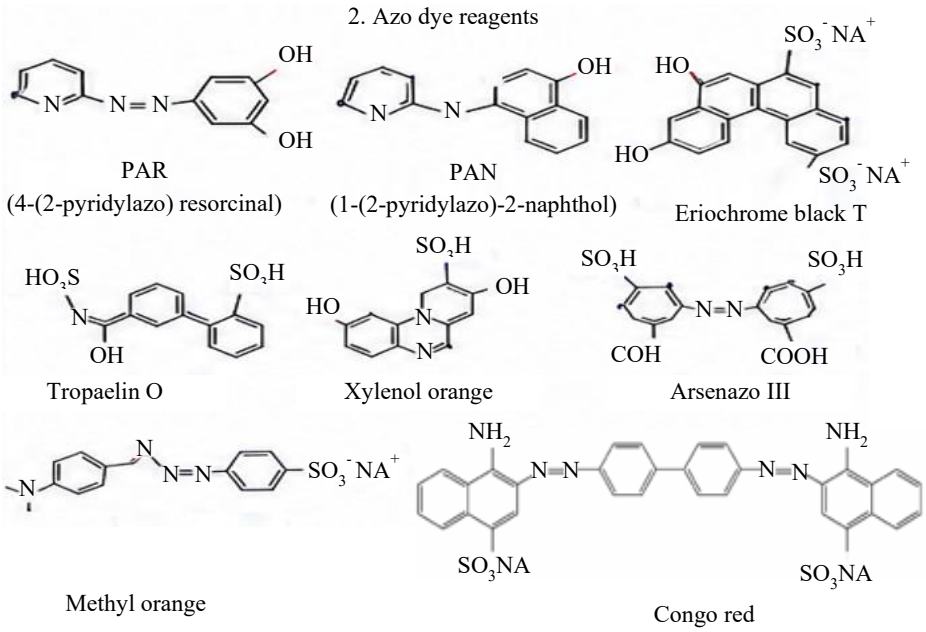


Figure 2. Azo dye reagents.

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Flavonoids, Nitroso, and Phenolic Reagents

Flavonoids and phenolic compounds act as oxygen-donor ligands, forming stable chelates through hydroxyl and carbonyl groups as shown in Figure 3.

- *Quercetin:* Quercetin forms a selective complex with Os (VIII) in acidic medium (0.05 M HCl), exhibiting λmax at ~370 nm. It enables speciation analysis with detection limits as low as 0.01 μg mL^{-1} [34].
- *Flavonoids (Morin, Rutin, Catechin, Luteolin):* These compounds exhibit λmax values between 300–410 nm, with luteolin showing comparatively higher sensitivity due to multiple hydroxyl groups [35].
- *Nitroso R Salt:* Nitroso R salt forms a highly stable complex with Os (VIII) at pH ≈ 4.8, showing λmax around 510 nm and excellent analytical reliability.
- *Phenolic/Nitroso Compounds:* ***Nitroso***-naphthol, o-aminophenol-p-sulphonic acid, resorcinol, and catechol exhibit λmax values between 310–520 nm, depending on ligand structure and coordination mode.

Heterocyclic, Advanced, and Catalytic Reagents

Reagents such as pyrogallol, diphenylcarbohydrazide, m-carboxyphenylfluorone, dithizone, and 8-hydroxyquinoline form stable complexes with osmium, with λmax values ranging from 350 to 575 nm. Among these, m-carboxyphenylfluorone provides exceptionally low detection limits as shown in Figure 4 [36]**.**

Catalytic spectrophotometric methods significantly enhance sensitivity through osmium-catalyzed oxidation of dyes. Rhodamine B, crystal violet, malachite green, carminic acid, safranin, and fluorescein enable ultra-trace detection in the ng mL^{-1} range, with carminic acid-based systems showing the highest sensitivity as shown in table 1.

COMPARISON AND FUTURE SCOPE

Comparative Evaluation of Organic Reagents

A comparison of different classes of organic reagents used for the spectrophotometric determination of osmium shows clear differences in their sensitivity, selectivity, and practical usefulness.

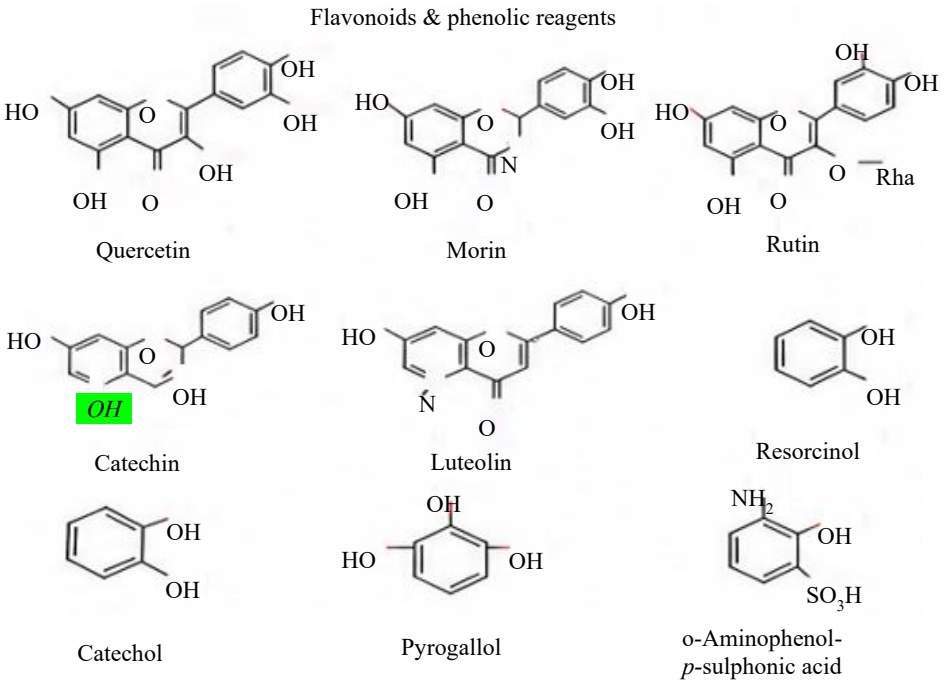


Figure 3. Flavonoids, nitroso, and phenolic reagents.

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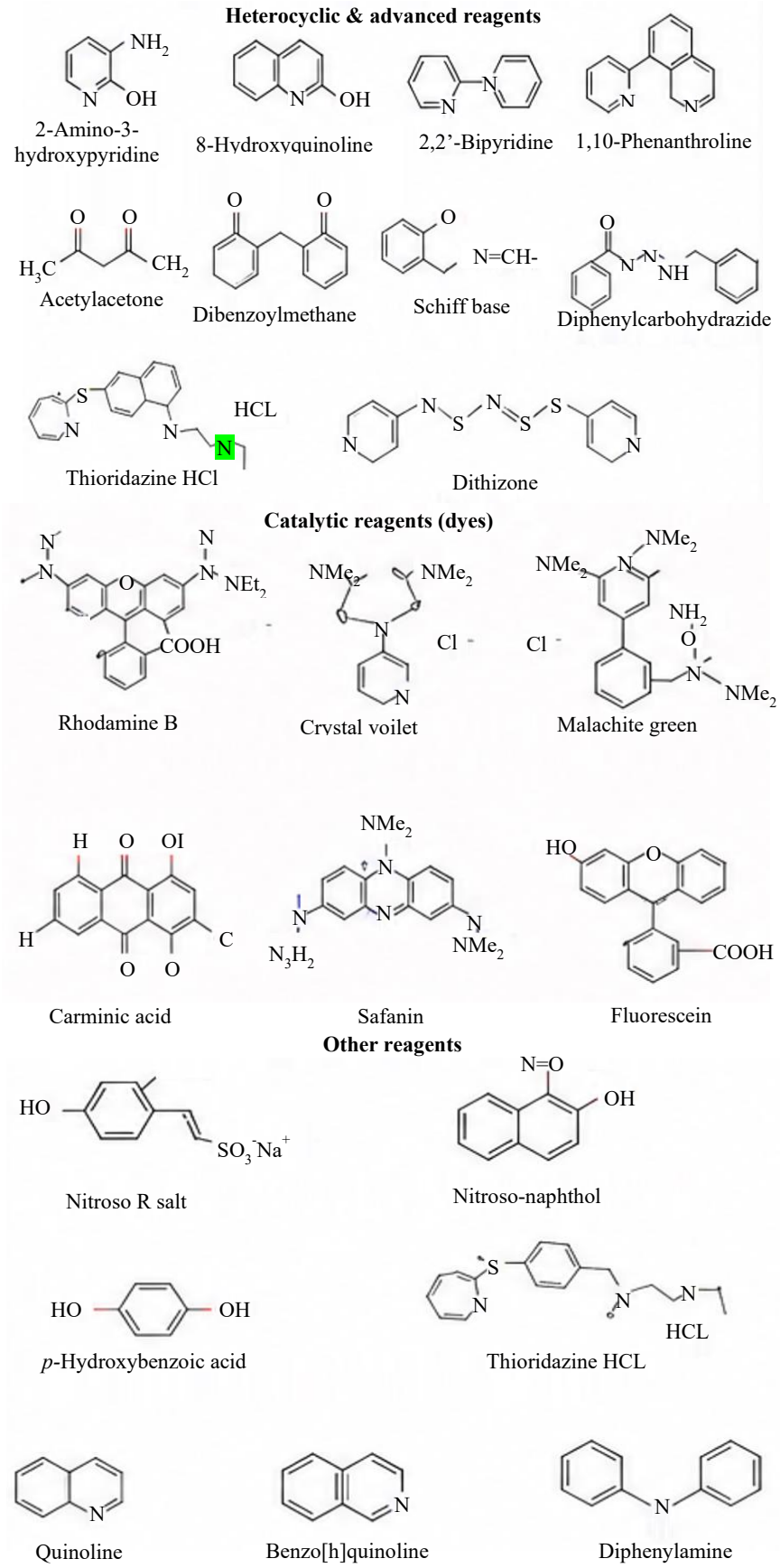


Figure 4. Hetrocyclic and catalytic reagents.

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Table 1. Comparison of different organic reagent for the spectrophotometric analysis of Osmium.

Sr. No.	Organic Reagent	Oxidation State	λ^{max} (nm)	ε (L mol^{-1} cm^{-1})	LOD (µg mL^{-1})	Medium / pH
1	Ethylene Thiourea	Os (VIII)	490	1.5×10^4	0.02	pH 1.0
2	Allyl Thiourea	Os (VIII)	298	0.9×10^4	0.05	Acidic
3	Phenyl Thiourea	Os (IV)	310	1.2×10^4	0.03	Acidic
4	o-Methoxyphenyl Thiourea	Os (IV)	518	2.12×10^3	0.089	0.8 M HCl
5	Thiosemicarbazide	Os (VIII)	360	1.0×10^4	0.04	Acidic
6	Benzaldehyde Thiosemicarbazone	Os (IV)	420	1.8×10^4	0.02	Acidic
7	Salicylaldehyde Thiosemicarbazone	Os (IV)	430	2.0×10^4	0.01	Acidic
8	Acetone Thiosemicarbazone	Os (VIII)	400	1.3×10^4	0.03	Acidic
9	2-Acetylpyridine Thiosemicarbazone	Os (IV)	440	2.1×10^4	0.01	Acidic
10	Thiourea	Os (VIII)	330	0.8×10^4	0.06	Acidic
11	PAR (4-(2-pyridylazo)resorcinol)	Os (IV)	520	3.5×10^4	0.005	pH 5
12	PAN (1-(2-pyridylazo)-2-naphthol)	Os (IV)	540	4.0×10^4	0.004	Acidic
13	Eriochrome Black T	Os (IV)	540	2.5×10^4	0.01	Acidic
14	Tropaeolin O	Os (VIII)	480	2.8×10^4	0.008	Acidic
15	Tropaeolin OOO-I	Os (VIII)	500	3.0×10^4	0.007	Acidic
16	Tropaeolin OOO-II	Os (VIII)	510	3.2×10^4	0.006	Acidic
17	Xylenol Orange	Os (IV)	560	3.8×10^4	0.005	Acidic
18	Arsenazo III	Os (IV)	600	4.5×10^4	0.003	Acidic
19	Methyl Orange	Os (VIII)	470	1.5×10^4	0.02	Acidic
20	Congo Red	Os (VIII)	500	2.2×10^4	0.01	Acidic
21	Quercetin	Os (VIII)	370	3.0×10^4	0.01	0.05 M HCl
22	Morin	Os (VIII)	380	2.5×10^4	0.02	Acidic
23	Rutin	Os (VIII)	360	2.0×10^4	0.03	Acidic
24	Catechin	Os (IV)	300	1.2×10^4	0.05	Acidic
25	Luteolin	Os (VIII)	410	2.8×10^4	0.02	Acidic
26	Nitroso R Salt	Os(VIII)	510	3.5×10^4	0.01	pH 4.8
27	Nitroso-naphthol	Os (VIII)	520	3.2×10^4	0.01	Acidic
28	o-Aminophenol-p-sulphonic acid	Os (VIII)	440	2.0×10^4	0.02	pH 3
29	Resorcinol	Os (IV)	310	1.0×10^4	0.05	Acidic
30	Catechol	Os (IV)	320	1.5×10^4	0.04	Acidic
31	Pyrogallol	Os (VIII)	420	2.2×10^4	0.02	Acidic
32	2-Amino-3-hydroxypyridine	Os (IV)	520	2.8×10^4	0.01	Acidic
33	Diphenylcarbohydrazide	Os (VI)	550	4.5×10^4	0.003	Acidic
34	m-Carboxyphenylfluorone	Os (VIII)	575	5.0×10^4	0.002	Acidic
35	Thenoyltrifluoroacetone	Os (IV)	360	2.5×10^4	0.02	Organic phase
36	Ammonium thiocyanate	Os (IV)	460	2.0×10^4	0.03	Acidic
37	Dithizone	Os (IV)	500	3.5×10^4	0.005	Organic
38	Thioridazine HCl	Os (VIII)	520	3.0×10^4	0.01	Acidic
39	8-Hydroxyquinoline	Os (IV)	370	2.2×10^4	0.02	Neutral
40	2,2′-Bipyridine	Os (II/IV)	520	2.8×10^4	0.02	Neutral
41	1,10-Phenanthroline	Os (II/IV)	510	3.2×10^4	0.01	Neutral
42	Schiff base (Salicylaldehyde–aniline)	Os (IV)	430	2.5×10^4	0.02	Acidic
43	Acetylacetone	Os (IV)	350	1.8×10^4	0.03	Organic
44	Dibenzoylmethane	Os (IV)	420	2.6×10^4	0.02	Organic
45	Rhodamine B	Os (VIII)	555	5.5×10^4	0.001	Catalytic
46	Crystal Violet	Os (VIII)	590	4.8×10^4	0.002	Catalytic
47	Malachite Green	Os (VIII)	620	5.0×10^4	0.001	Catalytic
48	Carminic Acid	Os (VIII)	495	5.8×10^4	0.00002	Catalytic
49	Safranin	Os (VIII)	520	3.5×10^4	0.005	Catalytic
50	Fluorescein	Os (VIII)	490	6.0×10^4	0.0005	Catalytic

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Sulfur-containing reagents, such as thiourea and thiosemicarbazone derivatives, interact strongly with osmium due to the presence of sulfur donor atoms. These reagents generally form stable complexes in acidic medium and offer good selectivity. However, their sensitivity is moderate, and their molar absorptivity is lower than that of azo dyes and catalytic systems.

Azo dye reagents are among the most effective for osmium determination. Their extended conjugation and strong chromophoric nature result in intense (color) development in the visible region. Reagents like PAR, PAN, and Arsenazo III show high molar absorptivity and low detection limits, making them suitable for trace analysis. However, some interference from other metal ions may occur in complex samples.

Flavonoids and phenolic reagents provide a greener and more environmentally friendly option. These compounds coordinate with osmium through oxygen donor atoms and show moderate sensitivity with reasonable selectivity. Some reagents, such as quercetin, are also useful for speciation studies. However, their overall performance is generally lower compared to azo dyes and catalytic methods.

Heterocyclic and advanced reagents offer flexible coordination behavior and moderate to high sensitivity. Among all categories, catalytic spectrophotometric methods show the highest sensitivity, with detection limits reaching the ng mL^{-1} level. These methods are based on the catalytic effect of osmium in dye oxidation reactions, which significantly enhances the analytical signal. However, they require careful control of experimental conditions and may sometimes lack selectivity [37].

Future Scope

Although significant progress has been made, there is still considerable scope for improvement in the spectrophotometric determination of osmium.

Future work should focus on developing new organic reagents with higher sensitivity and better selectivity. Special emphasis should be given to reagents that can selectively detect different oxidation states of osmium, which is important for speciation analysis in complex systems.

There is also a growing need to develop environmentally friendly methods. The use of green reagents, aqueous media, and solvent-free techniques should be encouraged to reduce environmental impact and improve safety.

The integration of modern technologies offers promising opportunities. The use of nanomaterials and advanced materials such as metal–organic frameworks (MOFs) can enhance sensitivity and selectivity by improving signal response and adsorption properties.

In addition, automation and miniaturization techniques, such as flow injection analysis and microfluidic systems, can make these methods faster, more reliable, and suitable for routine analysis.

Finally, more studies should focus on real sample analysis, including environmental, biological, and industrial samples, to demonstrate the practical applicability of these methods.

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