



SCHIFF BASE-MODIFIED MAGNETIC NANOSORBENTS FOR SELECTIVE PRECONCENTRATION OF TRACE METAL IONS: A REVIEW OF DONOR-ATOM DESIGN, SELECTIVITY MECHANISMS AND ANALYTICAL PERFORMANCE

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ABSTRACT

Trace metal ions in water, foods and biological samples often are below the detection limits of atomic spectrometric detectors. They come with a hefty matrix. For a ligand specific to the sorbent surface, magnetic solid-phase extraction provides a straightforward response. Schiff bases are formed in a single condensation step and can coordinate the azomethine nitrogen with O, N or S donors, which makes them interesting for this purpose. This review discusses Schiff base modified magnetic nanosorbents from the standpoint of donor atoms. We detail the assembly of the magnetic core, the protective shell and the anchoring imine, and we classify the ligands into N,O, N,N, N,S, N₂O₂ and N,N,S families. Their behaviour is connected to hard-soft acid-base principles, chelate ring size, stiffness and substituent effects. Selectivity is then discussed in terms of non-specific background uptake, the pH window, ion imprinting and competition from matrix ions. Twenty verified studies are compared in a table of analytical performance. Reported detection limits run from below 1 ng L⁻¹ with ICP-MS, through 0.05 to 0.24 µg L⁻¹ with flame AAS, to 10 µg L⁻¹ with spectrophotometry, and enrichment or preconcentration factors from 1 to 1000. The comparison also shows recurring weak points: hydrolysis of the imine in acidic eluents, loosely held ligands, mixed use of enrichment and preconcentration factors, limited multi-element selectivity tests and infrequent use of certified reference materials. A minimum reporting set is proposed so that future methods can be compared on equal terms.

Keywords: Schiff base; magnetic solid-phase extraction; Fe₃O₄ nanoparticles; donor atoms; HSAB; trace metal preconcentration; ion-imprinted polymer.

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1. INTRODUCTION

Toxic metal ions such as Pb(II), Cd(II) and Hg(II) still exist in drinking water, food and biological fluids, usually in the quantities that are difficult to detect directly [1]. The analytical problem is an ancient one. The analyte is at the level of µg L⁻¹ or even ng L⁻¹, whereas sodium, calcium, magnesium and dissolved organic matter are at much higher quantities and interfere with the measurement [2]. Thus, a separation and preconcentration step is added in most procedures based on flame or electrothermal atomic absorption, ICP-OES and, fairly frequently, ICP-MS. Solid-phase

extraction became the usual answer because it is simple, uses little solvent and couples easily with these detectors [2]. Magnetic solid-phase extraction (MSPE) added one more convenience. The sorbent is dispersed in the sample and then pulled out with a magnet, so no column, filtration or centrifugation is needed [3-6]. Bare iron oxide, however, has little selectivity of its own. Its surface has to carry a ligand that does the choosing. Schiff bases is a natural choice. They are prepared in a single condensation step between an aldehyde or ketone and a primary amine [7] and the azomethine nitrogen can be linked with phenolic oxygen, pyridine nitrogen, thiophene or thione sulphur and other donor groups in nearly any arrangement [8,9]. This flexibility is the reason they are so frequently observed on magnetic supports. It also explains why the literature has become difficult to read. In the literature various ligands, supports and figures of merit are reported and few examine why a certain donor set should favour a given ion. This review starts from that question of design. We first describe how Schiff base-modified magnetic sorbents

are built. We then sort the anchored ligands by their donor atoms and relate them to hard-soft acid-base behaviour and chelate ring size [10,11]. Selectivity mechanisms, the analytical workflow and the reported performance follow, with a table of previous studies. We close with the gaps that, in our reading, still hold the field back.

2. BUILDING THE SORBENT

2.1. The magnetic core and its shell

Most sorbents in this field start from magnetite. Fe_3O_4 nanoparticles are usually made by co-precipitating Fe(II) and Fe(III) salts with ammonia or sodium hydroxide, a route that goes back to Massart [12]. Thermal decomposition and solvothermal methods give narrower size distributions, but they cost more and leave surfactants that must be removed [13,14]. Small magnetite particles behave as single magnetic domains and are superparamagnetic at room temperature. They respond strongly to an external magnet, yet keep no remanent magnetisation once it is taken away, so they disperse again easily after each step [14].

Naked magnetite is a poor platform for trace analysis, though. It aggregates, it oxidises slowly in air and it dissolves in the acids used for elution [14]. A shell solves most of these problems. Silica is the common choice. Stöber-type hydrolysis of tetraethyl orthosilicate in ethanol, water and ammonia [15] deposits a dense silica layer, and a templated second layer can add ordered mesopores and more surface area [16]. Silica also brings silanol groups, and that is what matters for the next step. Polydopamine is a useful alternative. It deposits on almost any surface from a mildly alkaline dopamine solution and carries catechol and amine groups, and the coating can react further with amine and thiol nucleophiles [17]. Chitosan [18,19], graphene oxide [20,21], agarose [22] and mesoporous MCM-41 silica [23] play a similar role through their own amino, oxygen-containing or silanol groups. An imine-linked covalent organic framework has even been grown on aminated Fe_3O_4 and used as the shell itself [24].

2.2. Anchoring the imine

There are two main ways to place the Schiff base on the shell. In the stepwise route, the silica surface is first treated with 3-aminopropyltriethoxysilane (APTES), and the aldehyde is then condensed with the surface amine, usually in refluxing ethanol or methanol. This is the route drawn in Figure 01, and in our reading it is the most common one. In the pre-formed route, the ligand is condensed with an aminosilane in solution, purified and only then grafted. This costs one more step but gives a defined ligand that is easier to characterise. A third option reverses the roles. An aldehyde is placed on the surface first, for example by activating an amine-rich layer with glutaraldehyde, and an amine-bearing ligand is then condensed onto it. Glutaraldehyde also serves to tie pre-formed imines to chitosan, as with a methionine-glutaraldehyde Schiff base on magnetic chitosan [18]. Some ligands are not

bound covalently at all. They are coated onto the support [25,26], which is quick but leaves the ligand free to leach during elution.

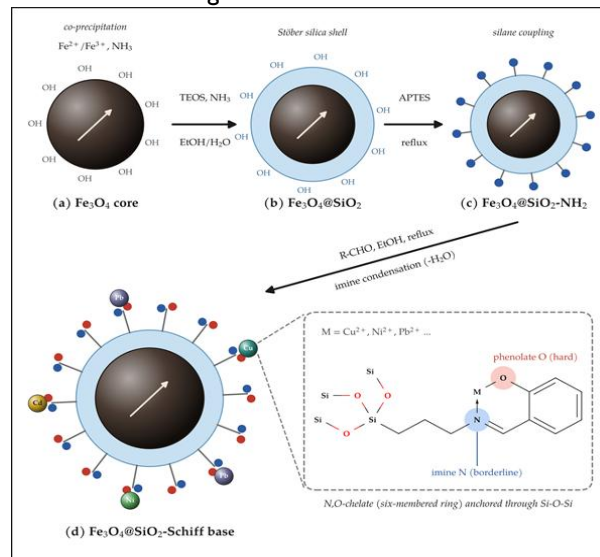


Figure 01: Stepwise construction of a Schiff base-modified magnetic nanosorbent: (a) co-precipitated Fe_3O_4 core, (b) Stöber silica shell, (c) amine layer from APTES and (d) imine formed by condensation with an aldehyde, here salicylaldehyde. The inset shows the anchored N,O-chelate, with M the bound metal ion.

Sizes and arm densities are not to scale.

All the routes give some unreacted amine. This is not always a fault, as a free amine is a poor donor itself, and changes the surface charge with pH. It does make the binding sites less consistent and compromises any stringent assertion about the donor set. Ligand loading is seldom measured with care. The standard estimates are nitrogen elemental analysis and thermogravimetric mass loss. The $\text{C}=\text{N}$ stretching band in the infrared spectrum shows that the imine is there but not how much of it.

One imine bond property that demands attention from the start. Formation of imines is a reversible process and the $\text{C}=\text{N}$ bond hydrolyses to aldehyde and amine in aqueous acid [27,28]. These conditions are employed to take the metal off during elution. Reduction of the imine to a secondary amine, as occurs in reductive amination [29], removes the weakness but also modifies the donor and its shape. We come back to this trade-off in Section 7.

3. DONOR-ATOM DESIGN

3.1. Donor sets found on magnetic supports

Every Schiff base carries the azomethine nitrogen. It is an sp^2 nitrogen with its lone pair in the molecular plane, a borderline donor in Pearson's terms [10], and on its own it binds rather weakly. What turns a Schiff base into a good chelator is a second donor placed two or three bonds away, so that a five- or six-membered chelate ring closes around the metal. Figure 02 shows the anchored ligand types that recur in the literature, and Table 01 collects their main features.

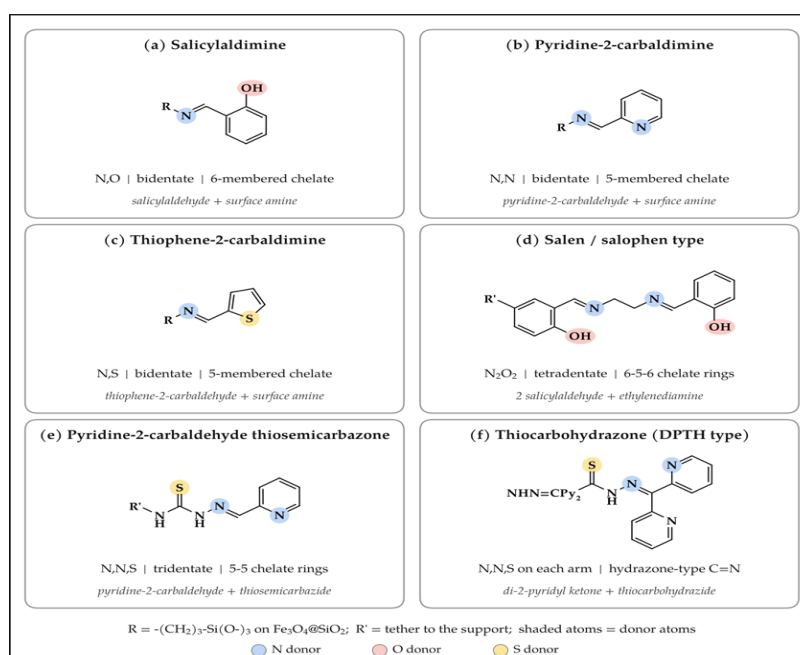


Figure 02: Representative Schiff base units found on magnetic supports, grouped by donor set. Shaded atoms are the donor atoms. R is the propylsilyl tether on Fe₃O₄@SiO₂ and R' a tether to the support; for the thiocarbohydrazone in (f) only one of the two identical arms is drawn.

The salicylaldimine unit (N,O) is the workhorse. Salicylaldehyde reacts cleanly with surface amines, and its phenolate oxygen adds a hard, negatively charged donor that forms a six-membered ring with the imine nitrogen. Pyridine-2-carbaldehyde and thiophene-2-carbaldehyde give N,N and N,S bidentate units with five-membered rings. Salen-type ligands, built from two salicylaldehyde units and a diamine, offer a planar N₂O₂ pocket that holds many divalent ions and also the uranyl ion [8,9]. Thiosemicarbazones bring a thione sulfur, and with a pyridyl or ortho-hydroxy group on the aldehyde they become tridentate N,N,S or O,N,S donors [30]. Thiocarbohydrazones such as DPTH, made from di-2-pyridyl ketone and thiocarbohydrazide, carry an N,N,S set on each arm. Strictly they are hydrazone-type imines, not classical Schiff bases, but they bind in the same way and have been used on magnetic particles for Hg and the noble metals [21,31,32].

Table 01: Main donor-atom families of Schiff bases anchored on magnetic supports, with the preference expected from HSAB and chelate ring-size reasoning [9-11,30].

Ligand family	Typical precursors	Donor set (denticity)	Chelate ring(s)	Character of the pocket	Expected preference	Examples in Table 2
Salicylaldimine	salicylaldehyde or a substituted salicylaldehyde + surface amine (APTES)	N,O (2)	one 6-membered	borderline N + hard phenolate O	borderline and harder ions: Cu ²⁺ , Ni ²⁺ , Zn ²⁺ , Pb ²⁺ , UO ₂ ²⁺	[23,26,33,34]
Pyridine-2-carbaldimine	pyridine-2-carbaldehyde + surface amine	N,N (2)	one 5-membered	two borderline N	borderline ions; the 5-membered ring favours larger ions such as Pb ²⁺ and Cd ²⁺	pyridyl N within the thio units below [20,31]
Thiophene-2-carbaldimine	thiophene-2-carbaldehyde + surface amine	N,S (2)	one 5-membered	borderline N + soft S	soft and borderline ions: Hg ²⁺ , Ag ⁺ , Pd ²⁺ , Cd ²⁺	thiophene on an imine COF [24]
Salen and salophen	two salicylaldehyde units + ethylenediamine or o-phenylenediamine, tethered through a ring substituent	N ₂ O ₂ (4)	6-5-6	planar, rigid in salophen	Cu ²⁺ , Ni ²⁺ , Co ²⁺ and UO ₂ ²⁺ ; the rigid bridge sharpens size selection	[22,25,35,36]

Thiosemicarbazone	aldehyde + thiosemicarbazide	N,S (2); N,N,S or O,N,S (3) with a pyridyl or o- OH group	5-5 or 6-5	soft thione S + borderline N	Hg ²⁺ , Ag ⁺ , Pd ²⁺ , Pt ²⁺ , Cu ²⁺	[20]
Thiocarbohydrazone (hydrazone type)	di-2-pyridyl ketone (DPTH) or 2-pyridyl 3-sulfophenyl ketone (PSTH) + thiocarbohydrazide	N,N,S on each arm (3)	5-5	soft S + borderline N	Hg ²⁺ and the noble metals (Pd, Pt, Au, Ag, Ir, Os)	[21,31,32,37]
Other imines	vanillin, isatin, thiazolyl imines, amino acid-glutaraldehyde imines	often unclear	not always possible	mixed	set by the partner donor; some lack a chelating partner	[18,38-40]

Denticity matters for two reasons. More donor atoms raise stability through the chelate effect. They also add geometric demands, so a tetradentate pocket tends to reject ions that cannot adopt the required arrangement. A surface rarely offers the clean geometry of a free ligand in solution, however. Neighbouring arms may share one metal ion and some arms lie bent towards the silica. The donor sets quoted in papers, and in Table I, are best read as the intended ones.

3.2. Hard, soft and borderline donors

Pearson's principle of hard and soft acids and bases is still the most useful single guide [10]. Hard ions such as Fe(III), Al(III), Th(IV) and the uranyl ion prefer hard oxygen donors. Soft ions such as Hg(II), Ag(I), Pd(II), Pt(II) and Cd(II) prefer soft sulfur donors. Cu(II), Ni(II), Co(II), Zn(II) and Pb(II) are borderline and bind well to nitrogen. For a Schiff base this gives a simple design rule (Fig. 3a). The imine nitrogen is the constant part, and the partner donor sets the preference. A phenolate oxygen pulls the pocket towards harder ions, a pyridine nitrogen keeps it borderline, and a thiophene, thioether or thione sulfur pulls it towards soft ions.

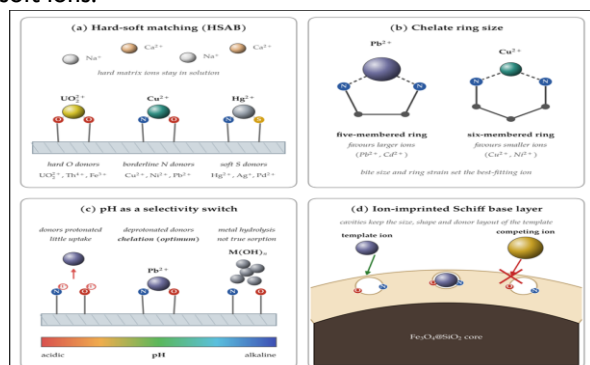


Figure 03: Four levers that set selectivity on Schiff base magnetic sorbents: (a) hard-soft matching between donor and ion, (b) chelate ring size, (c) the pH window between donor protonation and metal hydrolysis and (d) ion-imprinted cavities.

The rule has a second, less obvious benefit. The major ions of natural waters, Na⁺, K⁺, Ca²⁺ and Mg²⁺, are all

hard. A pocket made only of nitrogen and sulfur therefore largely ignores them, while an oxygen-rich pocket may not. On a thiophene-modified imine framework, for example, Na⁺ and K⁺ up to 9000 mg L⁻¹ and Ca²⁺ and Mg²⁺ up to 6000 mg L⁻¹ did not interfere with the extraction of Hg(II), Pb(II) and Bi(III) [24].

Within the borderline group the Irving-Williams order (Mn < Fe < Co < Ni < Cu > Zn) predicts that Cu(II) forms the most stable complex with almost any nitrogen or oxygen donor set [41]. It helps explain why Cu(II) tends to be a strong competitor in multi-element tests, and why selectivity among borderline ions is harder to reach than selectivity between classes.

3.3. Ring size, rigidity and substituents

Hancock and Martell showed that chelate ring size is a selectivity tool in its own right [11,42]. Five-membered chelate rings suit large metal ions best, while six-membered rings suit small ones. For a ligand designer this means that a pyridine-2-carbaldimine, which closes a five-membered ring, should lean towards Pb(II) or Cd(II) when compared with a salicylaldehyde, whose six-membered ring should favour Cu(II) and Ni(II) (Fig. 3b). The rule describes relative preference, not absolute binding strength, and on a surface it is easily masked by heterogeneous sites. Even so, few papers in this area seem to use it on purpose.

Rigidity is a second handle. A phenylene bridge (salophen) holds the N₂O₂ donors in a more fixed position than the flexible ethylene bridge of salen. A preorganised pocket loses less entropy on binding and discriminates better by size [11], although binding can become slower.

The imine itself can be tuned as well. Imines from aromatic aldehydes are conjugated with the ring and are more stable towards hydrolysis than those from aliphatic aldehydes [28], which is one practical reason why aromatic aldehydes such as salicylaldehyde are used so widely. Ketimines, such as those from di-2-pyridyl ketone, place a second substituent on the azomethine carbon and make the pocket more crowded.

Substituents on the aromatic ring tune donor strength. Electron-withdrawing groups such as nitro or bromo

make the phenol more acidic, so the ligand deprotonates and binds at lower pH. Electron-donating groups such as methoxy do the opposite [43]. In practice this shifts the working pH window more than it changes the order of selectivity. It is a cheap and underused way to move the optimum pH away from the range where the target ion begins to hydrolyse.

4. SELECTIVITY MECHANISMS

4.1. Chelation and the non-specific background

Uptake on these sorbents is rarely one single process. The intended route is inner-sphere chelation by the imine pocket. Alongside it, residual silanol groups, unreacted amines and any exposed iron oxide bind cations through electrostatic attraction and ion exchange. This background is weak and unselective, but it is not always small. Silanol groups lose protons as the pH rises, so the non-specific share grows with pH and with poor ligand coverage, and the uptake of matrix ions grows with it. A ligand-free control, $\text{Fe}_3\text{O}_4@\text{SiO}_2$ or $\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-NH}_2$ tested under the same conditions, is the simplest way to see how much of the signal really comes from the Schiff base. El-Sheikh et al. did exactly this with bare and APTES-coated magnetite and found the capacity order VSB-Mag > 4-HSB-Mag > Sil-Mag > Mag [38]. Their best sorbent was the vanillin imine, whose OH group sits para to the C=N bond and cannot close a chelate ring with it. At their optimum pH of 9, then, something more than chelation was probably at work.

4.2. pH as a switch

pH controls nearly everything in these systems (Fig. 3c). At low pH the imine nitrogen and the phenolate or pyridine donors are protonated, and protons compete with the metal, so uptake is poor. As pH increases, donors are released and chelation starts. At higher pH the metal hydrolyses and may precipitate as a hydroxide on or near the particles which might be confused with sorption. The window between these boundaries is tight. The studies in Table 2 which give an optimum are in the range between pH 3.0 and 9, with five of the eight values lying between pH 4 and 7. Because every ion has its own hydrolysis behaviour and its own affinity for the donor set, pH is also a separation tool in its own right. An ion that binds strongly at low pH can be collected while a weaker one stays in solution, and a mild eluent can strip one metal but leave another on the sorbent. García-Mesa et al., for instance, collected noble metals, Sb and Hg on a thiocarbohydrazone sorbent at pH 3.0, where the transition metals present in a certified water caused no interference at $\mu\text{g L}^{-1}$ levels [21].

4.3. Imprinted cavities

Ion imprinting adds a memory of shape to the donor set (Fig. 3d). The Schiff base ligand is first complexed with the target ion and then built into a cross-linked layer around the magnetic core, after which the template is washed out [44-46]. The cavities that remain keep the size, the coordination geometry and the spacing of the donors. A uranyl-salophen surface-

imprinted magnetic sorbent, for example, gave a selectivity coefficient for UO_2^{2+} over Th^{4+} of 4.232, against 1.002 for its non-imprinted twin [36], and a salen-based Ag(I)-imprinted polymer reached an enhancement factor of 293 [35]. The gain is real, but it costs capacity, because only part of the layer forms well-defined sites, and it needs thorough template removal. Incomplete removal leaves traces of the very analyte the method is meant to measure, which shows up as a high blank.

4.4. Competing ions and the evidence for binding

Selectivity is generally tested by adding foreign ions one by one at increasing ratios to the analyte and seeing if recovery is still satisfactory. This is helpful but hopeful. In real samples there are numerous competitors at the same time, perhaps Fe(III) or Cu(II) in great excess, and their impacts are not cumulative.

Direct proof of the binding mode is not as widespread as it should be. The main indications are a shift in the C=N stretching band upon metal uptake, the appearance of new metal-nitrogen and metal-oxygen bands at low wavenumber and changes in the N 1s, O 1s or S 2p binding energies in XPS. Such evidence is infrequently mentioned in the abstracts prepared for Table 2, but it may be found in certain full texts. Such facts change a donor set from an assumption into an observation and should accompany any assertion of selectivity.

5. THE ANALYTICAL WORKFLOW

5.1. From sample to signal

A typical MSPE run consists of six phases (Fig. 4). The sample is adjusted to the ideal pH, the sorbent is disseminated in the sample by vortex mixing or ultrasonic and the metal is chelated rapidly because of the limited diffusion routes. The particles are then pulled to the side of the vial by a magnet and the supernatant removed. The metal is stripped using a tiny amount of eluent, often dilute HNO_3 or HCl . For soft ions, a sulphur compound is often added, thiourea [21,35] or cysteamine [24], because it competes for the metal through its own sulphur. EDTA has also been utilised [18]. The eluate is then read. Flame AAS is the most widely used technique while ICP-OES or ICP-MS are used when lower detection limits or multi-element determinations are required (Table 02).

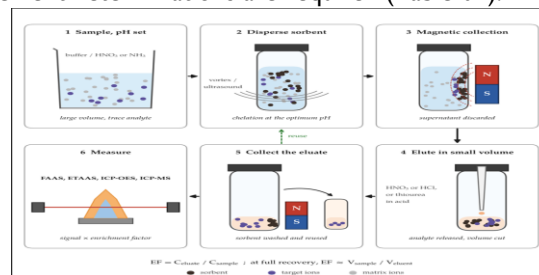


Figure 04: The magnetic solid-phase extraction cycle used with Schiff base sorbents, from pH adjustment to measurement, with the relation between enrichment factor and volumes at full recovery.

The eluent must be compatible with both the detector and the sorbent. -FAAS is tolerant to quite strong acid and moderate volumes, which is one of the reasons why it goes so well along with MSPE. ETAAS and ICP-MS reward small and clean eluates, but are less merciful to high acid, salt or organic loads, which affect the furnace program or the plasma [2]. Thiourea eluents perform well for soft ions, but contribute carbon and sulphur to the solution entering the instrument. A high EF is only beneficial when the eluate still fits the working requirements of the detector .

For a long time optimisation was done one variable at a time. Response surface designs are currently prevalent, notably central composite and Box-Behnken designs, as the pH, sorbent mass, contact time and eluent strength interact [47,48]. For instance, central composite designs were utilised to optimise the Hg(II) approach of Keramat and Zare-Dorabei [20] and the uranyl method of Nazari Serenjah et al. [22].

5.2. Figures of merit that need care

The enrichment factor (EF) and the preconcentration factor (PF) are commonly used interchangeably however they are not the same. PF is a volume ratio, sample volume divided by the final eluent volume. EF is a ratio of concentrations, usually defined as the ratio of calibration slopes with and without preconcentration. They only agree at full recuperation. For a procedure with PF 200 and recovery 90% the EF is closer to 180 and the difference increases for weaker recovery.

Detection limits should be according to IUPAC practice [49] with the blank taken through the complete method. Validation should involve trueness against certified reference materials and precision under stated conditions [50]. Capacity poses a comparable question. The maximal sorption capacity obtained from a Langmuir fit is the saturation value for a batch test. The relevant capacity for analysis is the amount of analyte that can be loaded with quantitative recovery, which is normally significantly lower and is rarely mentioned.

6. ANALYTICAL PERFORMANCE IN PREVIOUS STUDIES

Table 2 summarises twenty research (2012–2025), in which a Schiff base or a closely related hydrazone-type imine was immobilised on a magnetic support and the latter was utilised for analytical preconcentration. We only included papers with a verifiable DOI and those reported useful figures of merit in the abstract or full text. One of the most recognised 2012 studies of a Schiff base/silica/magnetite sorbent for heavy metals has subsequently been rejected and is not included [51]. Values are as printed and n.a. indicates data not available in the text we were able to read. The picture is patchy. Atomic absorption was the most common detector used, in seven experiments, five of which were specifically in flame mode. ICP-OES is reported in five investigations, ICP-MS, cold-vapour ETAAS, UV-Vis spectrophotometry and fluorescence appear once or twice. Three abstracts do not even refer to the detector[38–40]. N,O units of the salicylaldimine family and related imines, together with N₂O₂ salen or salophen pockets, account for nine studies. They were applied to borderline ions, to Cd(II) and Ag(I), and in three cases to uranium. The sulfur-bearing units, thiosemicarbazones and thiocarbohydrazones, serve mostly Hg(II), the noble metals and other soft or vapour-forming elements, much as HSAB reasoning would predict (Section 3.2). Four of these come from one research group, which built on-line flow systems around DPTH and PSTH sorbents, coupled them with vapour generation and ICP-OES, and reached throughputs of about 13 to 17 samples per hour [21,31,32,37].

Fig. 5 plots the detection limit against the reported EF or PF for the studies that give both. Three points stand out. First, the detector sets the floor more than the sorbent does. The ICP-MS values for Hg(II), Pb(II) and Bi(III) sit below 1 ng L⁻¹ [24], whereas the flame AAS points stay between 0.05 and 0.241 µg L⁻¹ even when EF approaches or passes 100 [18,34,35]. Second, a large factor is not needed when the detector is sensitive. Cold-vapour ETAAS reached 7.8 ng L⁻¹ for Hg with a PF of only 5.4 [31], about the same as a thiosemicarbazone sorbent with an EF of 193 measured by ICP-OES [20]. Third, the factors themselves spread from 1 for some elements on a multi-element PSTH sorbent [37] to a PF of 1000 for uranium [33]. Much of that spread reflects the sample-to-eluent volume ratio the authors chose, not a property of the ligand.

Table 02. Schiff base-modified magnetic sorbents used for the preconcentration of trace metal ions: sorbent, donor set, conditions and figures of merit as reported.

Sorbent (Schiff base unit)	Dono r set	Analyt e(s)	Sample	Techni que	p H	Eluent	EF or PF	LOD	q _{max} (mg g ⁻¹)	Other report ed data	Re f.
N,O donor sets (salicylaldimine and related units)											
Fe ₃ O ₄ functionalized with salicylaldehyde groups (Sal- APS-FMNPs)	N,O	U(VI)	natural waters	ICP- OES	7	1 mL (type n.a.)	PF 1000	0.24 ng m L ⁻¹	49	RSD 1.5%	[3 3]
Iron oxide NPs, APTES + salicylaldehyde	N,O	Cd, Pb, Ni, Co	environm ental waters	FAAS	n. a.	acidifie d methan	EF 79.1- 95.7	0.183 - 0.241	n.a.	certified referenc e	[3 4]

(capillary microextraction)						ol		$\mu\text{g L}^{-1}$		waters	
MCM-41-coated magnetite + N-(4-methoxysalicylidene)-4,5-dinitro-1,2-phenylenediamine	N ₂ O	Cu, Ni, Co	natural waters, plant reference samples	AAS	n.a.	n.a.	n.a.	0.03-0.04 ng mL^{-1}	n.a.	linear 1.0-300 $\mu\text{g L}^{-1}$	[23]
Magnetite + three silane Schiff bases (VSB, 4-HSB, 2-HSB)	N ₂ O ^a	Cd, Pb	tap and well water	n.a.	9	10 mL 0.1 M HNO ₃	EF 8.4-10.1 (Cd), 7.3-11.4 (Pb)	1.5 (Cd), 17 (Pb) $\mu\text{g L}^{-1}$	n.a.	recovery in real waters 58.1-72.2%	[38]
Magnetite coated with salicylaldazine	O ₂ N, N ₂ O	Cu	tap, river and lake water	AAS	n.a.	n.a.	n.a.	1.6 $\mu\text{g mL}^{-1}$ ^b	n.a.	linear 2-40 $\mu\text{g mL}^{-1}$	[26]
N₂O₂ donor sets (salen and salophen units)											
Agarose-coated MNPs + N,N'-bis(4-hydroxysalicylidene)-1,2-phenylenediamine	N ₂ O ₂	UO ₂ ²⁺	spiked waters	UV-Vis (Arsenazo III)	n.a.	HCl	n.a.	10 $\mu\text{g L}^{-1}$	n.a.	RSD 3.4%; CCD optimisation	[22]
Fe ₃ O ₄ -Mn ₂ O ₃ composite coated with salen (BSED)	N ₂ O ₂	Cd	fruits	FAAS	4.0	1.5 mL 2 M HNO ₃	EF 36; PF 33	320 $\mu\text{g L}^{-1}$ _c	n.a.	RSD 1.1%	[25]
Magnetic Ag(I)-imprinted polymer (salen + 4-vinylpyridine)	N ₂ O ₂	Ag(I)	waters	FAAS	n.a.	thiourea (pH about 3)	EF 293	50 ng L^{-1}	28.2	CRMs BCR 288 and CPB-I	[35]
Fe ₃ O ₄ core with a uranyl-salophen surface-imprinted layer	N ₂ O ₂	UO ₂ ²⁺	pond, river and polluted water	fluorescence	n.a.	none (sorbent used directly)	n.a.	0.15 nmol L^{-1}	n.a. ^d	k(U/Th) 4.232 vs 1.002 (non-imprinted); 6 cycles	[36]
N,N,S and S donor sets (thiosemicarbazones, thiocarbohydrazones and a thiophene-modified imine framework)											
Fe ₃ O ₄ @GO + pyridine-2-carboxaldehyde thiosemicarbazone	N,N,S	Hg(II)	foods and waters	ICP-OES	n.a.	0.5 mL 0.1 M HCl	EF 193	0.0079 $\mu\text{g L}^{-1}$	n.a.	RSD 1.63%; CCD optimisation	[20]
Silica-coated MNPs + DPTH ^e	N,N,S	Hg	biological and sea water	FI CV-ETAAS	n.a.	n.a.	PF 5.4	7.8 ng L^{-1}	n.a.	three CRMs; on-line	[31]
Silica-coated MNPs + PSTH	N,N,S	14 element	sea, estuarine,	on-line CVG	n.a.	n.a.	EF 1-385.5	0.01-11.30	n.a.	CRMs SLRS-4,	[37]

e		s incl. Hg, Pd, Pt, Bi, Cd, Cu	lake, river and well water	ICP- OES				$\mu\text{g L}^{-1}$		TMDA 54.4, SW2, SRM 1643e	
Silica-coated MNPs + DPTH ^e	N,N,S	Pt, Pd, Os, Ir, Rh, Ag, Au	sea and tap water	on-line CVG ICP- OES	n. a.	n.a.	n.a.	0.03 (Ag, Pt) to 100 (Rh) $\mu\text{g L}^{-1}$	n.a.	CRMs NIST 2557, TMDA 54.4, SRM 1643e	[3 2]
DPTH on magnetic graphene oxide (DPTH@MG O) ^e	N,N,S	Pt, Pd, Ir, Os, Ag, Au, Sb, Hg	sea, mineral and tap water	on-line ICP- OES	3. 0	thiourea 2.1% in HNO_3 6.5%	EF 3 (Hg) to 33 (Os)	0.05 (Hg) to 2.6 (Pd) $\mu\text{g L}^{-1}$	4.50- 9.72	CRM TMDA 54.5; reactor refilled after 200 cycles	[2 1]
Imine COF (TPB + DMTA) on Fe_3O_4 , thiophene- modified	S, N	Hg(II), Pb(II), Bi(III)	lake and river water	ICP-MS	6	0.8 mL 0.1 M HNO_3 + 8% cysteine HCl	EF 188	0.41- 0.97 ng L^{-1}	n.a.	three CRMs; 20 cycles	[2 4]
Other or unspecified imine units											
Magnetic chitosan + methionine- glutaraldehyde Schiff base	N,O,S _g	Cu, Pb, Cd	industrial wastewater	FAAS	5. 5	2 mL 0.1 M EDTA	PF 50- 60	0.10- 0.24 $\mu\text{g L}^{-1}$	45- 46.5	at least 4 cycles	[1 8]
Chitosan-Schiff base grafted GO, magnetic	n.a.	Pb	whole blood	micro- sampling FAAS	6. 3	1.0 mL 0.8 M HNO_3	PF 20 (absolute recovery 40%)	0.06 ng mL^{-1}	n.a.	RSD 1.8% (intra- day)	[1 9]
Fe_3O_4 -APTES + 4-bromo-3- (thiazol-2- ylimino)methyl phenol	N,S,O _g	Pb	food and environmental samples	n.a.	6. 5- 8. 0	n.a.	n.a.	n.a.	n.a.	relative selectivity factor 150- 1500; 8 cycles	[4 0]
Fe_3O_4 @ SiO_2 functionalized with isatin groups ^f	n.a.	Cd, Ni, Pb, Zn	waters and agricultural products	n.a.	n. a.	n.a.	n.a.	0.11- 0.47 ng mL^{-1}	100- 120	RSD 2.5-3.1%	[3 9]
Schiff base- functionalized SiO_2 @ Fe_3O_4 (SB not specified)	n.a.	Cd	water and wastewater	UV-Vis	n. a.	n.a.	n.a.	0.741 $\mu\text{g L}^{-1}$	n.a.	n.a.	[5 2]

Values as printed in the source (mostly the abstract); n.a., not available in the text consulted. EF, enrichment factor; PF, preconcentration factor; CCD, central composite design; CVG, chemical vapour generation; CV, cold vapour; FI, flow injection; GO, graphene oxide; MGO, magnetic GO; COF, covalent organic framework; (M)NPs, (magnetic) nanoparticles. ^a VSB is a vanillin imine whose OH group is para to C=N; 4-HSB and 2-HSB are not expanded in the abstract. ^b As printed. ^c As printed; this lies above the stated linear range (10-120 $\mu\text{g L}^{-1}$). ^d Scatchard analysis gave 9.26

and $82.9 \mu\text{mol g}^{-1}$. ^e Thiocarbohydrazone (hydrazone-type C=N). ^f Imine linkage not stated in the abstract. ^g Donor atoms present in the ligand; the binding set is not stated.

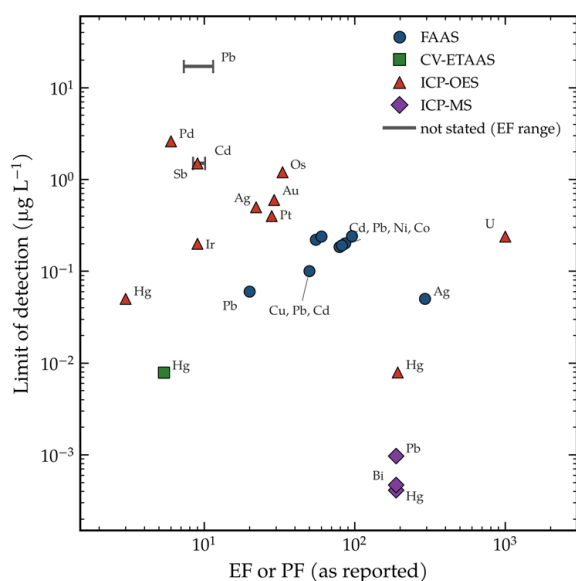


Figure 05: Detection limit against the enrichment or preconcentration factor for the Table 2 studies that report both, plotted as printed (LOD in $\mu\text{g L}^{-1}$). Bars show EF ranges where only a range was given. The two flame AAS clusters are Cd, Pb, Ni and Co [34] and Cu, Pb and Cd [18]. One method was left out because its printed LOD lies above its own linear range [25], and one because only unpaired ranges were given [37].

Capacities, where reported, run from $4.50\text{--}9.72 \text{ mg g}^{-1}$ for the elements held on the on-line DPTH@MGO sorbent [21] up to 120 mg g^{-1} for Cd(II) on an isatin-modified $\text{Fe}_3\text{O}_4\text{@SiO}_2$ [39]. The low-capacity DPTH@MGO system was nevertheless among the best validated, which is a useful reminder that capacity and analytical quality are different things. Selectivity data are thinner. In the texts we could read, apart from the imprinted uranyl sorbent discussed in Section 4.3 [36], only a thiazolyl imine sorbent for Pb(II) quantified selectivity, with relative selectivity factors of 150 to 1500 [40]. Most other studies report tolerance ratios only.

The studies also differ in format more than a table can show. Most work in batch, with a vial and a hand-held magnet. Shah et al. used salicylaldimine-decorated iron oxide in a capillary microextraction format together with a surfactant and an ionic liquid [34]. Keramat and Zare-Dorabei dispersed their sorbent with ultrasound [20], and Seidi et al. read a 1 mL eluate by microsampling FAAS, which suits small volumes [19]. At the other extreme, the imprinted uranyl sorbent went straight into a fluorescence reaction without any elution [36]. Each choice changes the effective factor, the time per sample and the risk of losses, so comparisons across formats need care.

Recovery deserves a closer look. A magnetic chitosan-Schiff base sorbent for Pb(II) in whole blood reported a PF of 20 together with an absolute recovery of 40% [19], which puts the effective enrichment close to 8. El-

Sheikh et al. obtained recoveries of 58.1 to 72.2% from tap and well water [38]. These are honest figures, and they show why EF, PF and recovery belong together in every report.

7. GAPS AND PRACTICAL RECOMMENDATIONS

Looking at the research side by side, five gaps stick out. The first is chemical stability. The imine bond is reversible in acid [27,28] nevertheless acids are the typical eluents. Reuse is documented in just five out of the twenty studies in Table 2. The claims vary between at least four cycles [18] and eight cycles [40] and 20 cycles [24] and one on-line reactor was replenished only after 200 loading and elution cycles [21]. The uranyl imprinted sorbent lost 4.58% of its capacity after 6 cycles [36]. Only a few articles describe ligand loss, iron leaching or blank drift across those cycles. A simple check like nitrogen content of the sorbent or iron in the eluate after every few cycles would answer the dispute. Reduced amine analogues and ligands with two anchoring points need a direct comparison with their imine progenitors. The second is the technique in which selectivity is tested. Single interferent tolerance ratios offer little about a genuine sample. Competition with numerous ions at realistic ratios and selectivity coefficients assessed against a non-imprinted or ligand-free control should become standard. Third is reporting. EF and PF are still confused, and maximal capacity is often stated as if it were analytical capacity (Section 5.2). Nor are there any easy cross-checks. One abstract reports the detection limit ($320 \mu\text{g L}^{-1}$) above the top end of the indicated linear range [25], which is probably a typing error but indicates how rarely these data are read against each other. And the fourth one is validation. Only seven of the twenty research in Table 2 use certified reference materials. Some of the other approaches depend on spike recovery in one or two matrices which is not a complete alternative. Fifth is the logic of design. Many ligands appear to be chosen because the aldehyde is cheap or familiar, rather than because the donor set is appropriate for the target. Immobilisation itself is not necessarily an advantage either. Shtaiwi et al. showed that the addition of a vanillin Schiff base to the extraction medium gave a larger working pH range for Pb(II) than when anchored on magnetite, with better results in real waters [53]. The HSAB class, chelate ring size and substituent effects already provide a simple framework (Sections 3.2 and 3.3) and quantum-chemical computations could help rank potential ligands before any synthesis. Greenness and practicality may now be rated using tools such as AGREE and the white analytical chemistry model [54–56], which allows comparison of methods on more than detection limit alone. Automation is the logical next step. Well-prepared Schiff base sorbents can deal with routine

jobs, as on-line magnetic systems are already running at 13 to 17 samples/h using certified materials as checks [21,32,37]. The minimum information we recommend that every new article should report is listed in Table 3.

Table 03: A minimum reporting set suggested for new Schiff base magnetic preconcentration methods.

Item	Why it matters	Suggested way to report it
Ligand loading	links capacity and selectivity to the number of sites	mmol of ligand per g of sorbent from N analysis or TGA, with the calculation shown
Donor set and binding evidence	turns the intended donor set into an observed one	FTIR shift of the C=N band and XPS N 1s, O 1s or S 2p before and after metal uptake
EF and PF, kept apart	prevents inflated performance claims	PF as $V(\text{sample})/V(\text{eluent})$; EF as the slope ratio, always with the recovery
Analytical capacity	defines the working range of the method	largest analyte mass that still gives quantitative recovery, next to the Langmuir maximum
Multi-element selectivity	reflects real samples	recoveries with several competitors at realistic ratios; selectivity coefficients against a non-imprinted or ligand-free control
Stability on reuse	imine hydrolysis and iron leaching	recovery, ligand content and Fe in the eluate checked every few cycles
Blank and LOD	IUPAC-consistent detection limits	blank carried through the full procedure; number of blanks and the factor used stated [49]
Trueness	method validation	certified reference material with a matrix close to the samples [50]
Greenness and practicality	comparison beyond detection limit	AGREE or white analytical chemistry score [55,56]

8. CONCLUSION

Schiff base-modified magnetic nanosorbents are a marriage of two basic ideas: a ligand that can be prepared in one condensation step and a support that can be collected with a magnet. They combine to produce quick, column-free preconcentration that takes flame AAS into the trace range and pushes ICP techniques still lower. Most of the behaviour is

determined by the donor atoms. Phenolate oxygen, pyridine nitrogen and sulphur partners adjust the imine pocket to hard, borderline or soft ions, while chelate ring size and stiffness provide a second level of selectivity. pH and ion imprinting round out the picture. The weak points are just as obvious. Both the imine bond and the iron core are exposed to the acids used for elution, selectivity is sometimes assessed too lightly, and figures of merit are not provided in a uniform way. Future studies should select ligands by design not habit, honestly test stability over many cycles, maintain EF and PF separate and validate against certified materials. These processes can advance these sorbents from many single demonstrations to reliable routine methods.

9. CONFLICT OF INTEREST

The authors declare no conflicts of interest.

11. ETHICAL APPROVAL AND INFORM COPNSENT

Not applicable.

12. ACKNOWLEDGEMNET

Not Declared.

13. FUNDING

Nil

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