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On-line analyte preconcentration with atomic spectrometric detection

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Pre-concentration of analytes, or matrix removal to overcome interferences using mini- or micro-columns of exchange media prior to atomic spectrometric detection is becoming increasingly more common. This paper is a review of some of the more recent applications of chelating, ion exchange and other resins and gels that have been used to accomplish this.

Introduction

Many atomic spectrometric methods of analysis, including electrothermal atomic absorption spectrometry (ETAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES), are prone to matrix interferences. Even inductively coupled plasma mass spectrometry (ICP-MS), which is generally regarded as being relatively free from interferences, suffers from polyatomic ion interferences, especially for the determination of first row transition metals. These interferences are summarized in table 1.

Table 1. Polyatomic ion interferences of importance in food.

| Mass | Element | % Abundance | Interfering ions |
|------|---------|-------------|-----------------|
| 47   | Ti      | 07:32       | HPO<sub>4</sub><sup>2-</sup> |
| 48   | Ti      | 73:98       | 32SO<sub>4</sub><sup>2-</sup>, POH<sup>+</sup> |
| 51   | V       | 99:76       | 35ClO<sub>4</sub><sup>-</sup>, 34SOH<sup>+</sup> |
| 52   | Cr      | 83:76       | 40Ar<sup>26</sup>C<sup>6</sup>+, 35ClO<sub>4</sub><sup>-</sup> |
| 53   | Cr      | 09:51       | 37ClO<sup>+</sup> |
| 54   | Fe      | 05:82       | 40ArN<sup>+</sup>, 37ClO<sub>4</sub> |
| 54   | Cr      | 02:38       | ArN<sup>+</sup>, ClO<sub>4</sub> <sup>+</sup> |
| 55   | Mn      | 100         | 40ArNH<sup>+</sup> |
| 56   | Fe      | 91:66       | 40ArO<sup>+</sup> |
| 63   | Cu      | 69:01       | 40ArNa<sup>+</sup> |
| 64   | Zn      | 48:89       | HPO<sub>4</sub><sup>2-</sup>, 32SO<sub>4</sub><sup>2-</sup>, 37ClO<sub>4</sub><sup>+</sup>, 63CuH<sup>+</sup> |
| 65   | Cu      | 30:09       | H<sup>32</sup>SO<sub>4</sub><sup>+</sup> |
| 69   | Ga      | 60:16       | 37ClO<sub>4</sub><sup>+</sup> |
| 75   | As      | 100         | 40Ar<sup>35</sup>Cl<sup>+</sup> |
| 76   | Se      | 09:02       | 36Ar<sup>75</sup>Ar<sup>+</sup> |
| 77   | Se      | 07:58       | 40Ar<sup>71</sup>Cl<sup>+</sup> |
| 78   | Se      | 23:52       | 40Ar<sup>38</sup>Ar<sup>+</sup> |
| 79   | Br      | 50:54       | 36Ar<sup>79</sup>ArH<sup>+</sup> |
| 80   | Se      | 49:82       | 40Ar<sup>80</sup>Ar<sup>+</sup> |

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The first on-line pre-concentration with atomic spectrometric detection was reported by Olsen et al. [8] who used Chelex-100 to pre-concentrate metals from seawater. One problem with the use of Chelex-100 is the swelling and contracting of the resin associated with changes in its ionic form. Other resins with the same functional group, for example Metpac CC1, have been reported not to suffer this disadvantage [41]. The advantage of chelating resins is that they are fairly selective for transition metals, with alkali and alkaline earth metals being easily eluted with an ammonium acetate buffer. The analyte is then eluted with dilute nitric acid.

Chelation

Many exchange media have been developed, including anion, cation and chelation exchange resins. Chelation exchange is one of the most common forms of sample pretreatment, which can be performed either on-line or off-line. If performed on-line it offers numerous advantages, including removal of the matrix/interferents and pre-concentration with a reduced risk of contamination. The most common chelating resin is Chelex-100, which contains iminodiacetate (IDA) functional groups. These groups readily chelate transition metal ions and other cations, for example lead (II). The IDA group is also present in a number of other chelating resins, for example Muromac-A1, Metpac CC1 and Dowex A1. The first workers to report the use of Chelex-100 are acknowledged to be Riley and Taylor [1]. Several other chelating agents exist, including 8-quinolinol and more specialized resins that chelate specific ions. A list of recent applications of chelating resins is given in table 2.

Chelation may also be used to decrease interferences in ICP-mass spectrometry, for example metal oxides of titanium and molybdenum interfere with copper, zinc and cadmium determinations. By complexing the titanium and molybdenum with N-methylhydroxydimethyl acid they may be retained on a Hamilton PRP-1 column, thereby removing the interference [42].
Table 2. Some applications of chelating resins.

| Analyte | Exchange medium | Matrix | Comments | Reference |
|---------|-----------------|--------|----------|-----------|
| V       | Chelex-100      | Biological matrices | Chemical purification | [2]        |
| Ba, Be, Cd, Co, Cu, Mn, Ni & Pb | Chelex-100 | — | Microcolumns and FI-ICP | [3]        |
| Rare Earths | Chelex-100 | Uranium | Detection by NAA | [4]        |
| Transition metals & Pb | Chelex-100 | Seawater | Detection by GFAAS | [5]        |
| Cd, Cu, Mn, Zn & Pb | Chelex-100 | Water | IC-FAAS online elution with cysteine | [6]        |
| Cd, Cu, Pb & Zn | Chelex-100 | — | FAAS detection | [7]        |
| Cd, Cu, Pb & Zn | Chelex-100 | Seawater | FI-AAS, microcolumn | [8]        |
| Transition metals & Pb | Chelex-100 | Seawater | GFAAS detection | [9]        |
| Cd, Pb, Ni, Cu & Zn | Chelex-100 | Seawater | GFAAS and FAAS | [10]       |
| Cu      | Chelex-100      | — | Development of a valve | [11]       |
| Ce, Co, Eu, Gd, Mn, Y & Zn | Chelex-100 | Brines | γ spectrometry detection | [12]       |
| Transition metals & Pb | Chelex-100 | Seawater | GFAAS & ICP detection | [13]       |
| Transition metals & Pb | Chelex-100 | Seawater | GFAAS detection | [14]       |
| Cd      | Muromac-A1      | Waters, CRMs | Minicolumn, FI-ICP | [15]       |
| Cr, Ti, V, Fe & Al | Muromac-A1 | — | Microcolumn, FI-ICP | [16]       |
| Cd, Zn, Cu, Mn, Pb, Fe & Cr | Muromac-A1 | Biological CRMs | FI-AAS | [17]       |
| Fe, Zn, Cu, Ni & Cr | Muromac-A1 | Water | Online-ICP or ICP-MS detection | [18]       |
| Transition metals & Pb | Metpac CC-1 | Seawater | Online ICP-MS detection | [19]       |
| Al      | 8-quinolinol/EDTA | — | Comaprision of chelating agents immobilised on glass | [20]       |
| Fe      | 8 quinolinol    | — | Minicolumn, chelator on glass. CL detection. | [21]       |
| Cu      | 8 quinolinol    | Waters | Chelator on glass | [22]       |
| Cu      | 8 quinolinol    | Inorganic salts | Chelator on silica gel | [23]       |
| Cu      | 8 quinolinol    | Waters | Chelator on glass | [24]       |
| Cr      | 8 quinolinol    | — | Chelator on glass. FI-ISE detection | [25]       |
| Cu      | 8-quinolinol    | Water | Complexation of analyte followed by adsorption to a macroporous resin | [26]       |
| Cr      | Poly(hydroxamic acid) resin | Seawater | FI-AAS | [27]       |
| Hg      | Picolinic acid amide | River water | Styrene DVB beads impregnated with chelator | [28]       |
| Ag, Au, Cu, Fe, Hg, Ni & Zn | Histidine | Water | Histidine attached to carboxyl of amberlite IRC50 | [29]       |
| Cu, Cd, Pb, Zn, Ag, Co, Fe, In, Ti, V, Bi | Carboxymethylated polyethyleneimine-poly-methylene polypineylene isocyanate | Sea water, bone | No swelling of the resin | [30]       |
| Cu, Cd, Zn | Chelex-100 and AGMP-1 | Waters | FI-AAS | [31]       |
| Various | EDTA-Cellulose/HSO₃ oxine cellulose | — | — | — |
| Hg      | Thiazole & thiazoline groups | Seawater | Elution with HCl and thiourea | [32]       |
| Pb      | Various         | Water | Chelating agents on silica gel | [33]       |
| Mo and W | Kelex-100       | Seawater | Catalytic current polarography detection | [34]       |
| Transition metals | Various | — | Preparation and study of chelators on silica gel | [35]       |
| Ca and Mg | Dowex A1 | Brine | Minicolumn. Spectrophotometric detection | [36]       |
| Various | XAD-4 with 7-dodecyl-8-quinolinol | Seawater | GFAAS and FAAS detection | [37]       |
| Ni, Cu, Pb & Cd | Resin 122 | Waters | Minicolumn, salicylic acid functional group | [38]       |
Cation exchange

Cation exchangers have also been used for pre-concentration and matrix removal prior to atomic spectrometric detection. The most common cation exchange resins are AG 50W and Amberlite 120, both of which contain sulphonic acid functional groups. Recent applications of cation exchange resins are detailed in Table 3. The eluent for cation exchange chromatography ranges from dilute acids to 8M nitric and 6M hydrochloric acids, depending upon the analyte. Further dilution may therefore be required before analysis by atomic spectrometry.

Anion exchange

Anion exchange has also been used to facilitate analyses with atomic spectrometric detection. Anion exchange resins, such as Dowex 1 and Amberlite IRA 400, contain...
Table 5. Other exchange media used.

| Analyte | Exchange medium | Matrix | Comments | Reference |
|---------|-----------------|--------|----------|-----------|
| Pb      | Activated alumina | Water  | FI-AAS microcolumn | [72] |
| Cr      | Activated alumina | Urine  | FI-ICP, minicolumn  | [73] |
| S       | Activated alumina | Water  | FI-ICP, micro-column | [74] |
| Lanthanides | NaAlumino-gallate crystals | — | Na exchange with lanthanides | [75] |
| Cr      | C-18 Rainin column | — | Ion pairing of Cr VI with tetrabutylammonium phosphate | [76] |
| Various | Retardion II A 8 | — | Amphoteric resin | [77] |
| Se      | Sephadex G-25M | Serum  | Column used to de-salt serum ICP-MS detection | [78] |
| Cu, Pb  | C-18            | — | Chelate formed with DDC, 8HQ PAR or PAN | [79] |

quaternary ammonium functional groups. Anion exchange can be used to quantitatively retain complexes of analytes with negatively charged ligands, whilst allowing other interfering cations to elute. Table 4 details some applications of anion exchange chromatography. In addition to retaining complexes of the analytes, there have been other applications. Kamson and Townshend [71] used an Amberlite IRA 400 and a De-acidite FF column to remove the interference effects exerted by phosphate and sulphate on calcium determinations by flame AAS.

Others

Table 5 shows some novel methods of ion exchange. Activated alumina has the advantage of being amphoteric, i.e. in its basic form it may be used to adsorb cations, such as lead [72] and chromium [73]; and in its acidic form it may be used to adsorb anions, such as sulphate [74]. Similarly, Retardion 11A8 is an amphoteric resin with both benzyltrimethylammonium and carboxylic acid exchange groups. Non-polar C-18 (octadecylsilane) columns have also been used for some applications, such as the adsorption of the diethylthiocarbamate complexes of copper and iron [79].

In addition to the papers detailing specific applications of FI and LC, there have been a number of authoritative reviews, for example on FI-ICP [80–83] and FI-atomic spectrometry [84–86]. A very comprehensive review of the literature is the second edition of Ruzicka and Hansen’s book [87].

Several other papers concerned with FI-ICP or FI-ICP-MS also exist [88–95], however many were performed off-line [93–94].

Conclusions

There is a growing trend towards the use of on-line mini- or micro-columns of exchange resins to pre-concentrate the analyte, or remove interfering species from the matrix, prior to atomic spectrometric detection. This may, in part, be due to the simplicity of the apparatus, and to the ease of automation of such systems. The use of on-line columns also leads to a decrease in the chances of contamination associated with the sample handling of off-line batch methods.

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