Metal Release Analysis of the new 1260 Infinity Bio-inert HPLC System by 7700 ICP-MS

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Abstract

Many analytical applications like the analysis of large bio-molecules or speciation in ultra low-levels of metals with ICP-MS require special analytical systems. A stainless steel free HPLC system is the prerequisite for such applications where inertness is crucial and metal release should be reduced to zero.

The new 1260 Infinity Bio-inert HPLC has a titanium based pump and metal free sample path from autosampler to detector. An active seal wash is also included in the pump. The metal release of this new system was tested with the 7700 ICP-MS system. The values should be compared with an earlier Technical note from Agilent where metal release from an Agilent 1100 HPLC system was measured with ICP-MS as well [1].

The 7700 ICP-MS gives the possibility to cover a wide range of elements with lowest detection limits. The octopole collision mode of the 7700 ICP-MS reduces molecular interferences formed by mobile phase components. Elements of interest were Fe, Cd, Cr, Cu, Au, Zr etc. Mobile phases tested during these experiments were common eluents like 0.1% formic acid, ammonium phosphate buffer and 100 mM NaOH.

Experimental

HPLC Sample Collection:

Due to the fact that the sample path from the Autosampler to the solvent heat exchanger and to the DAD Flow cell of the Bio-inert system is completely metal free, samples were directly taken after the pump.

- Flushing the pump with the mobile phase for 30 min with 1 ml/min flow rate
- The samples were collected over a 30 min. period at 1 ml/min flow rate in fractions of 10 ml with the appropriate mobile phase

Mobile phases:

To test the chemical resistance of the Agilent 1260 Bio-inert HPLC 3 various mobile phases were used. Formic acid and NaOH to illustrate extremes of pH and sodium chloride containing phosphate buffer as a representative solvent in biochromatography.

- 0.1% Formic acid (v/v) pH 2.6
- 100 mM NaOH pH 11.4
- 25 mM Sodium phosphate/250 mM NaCl pH 6.2

ICP-MS Conditions:

A 7700 ICP-MS was used for the analysis of the samples. Most elements were measured without He Collision cell. Elements between mass 40-80 amu were measured in He collision mode, with respect of iron which was measured in Hydrogen.

0.4 ml/min dilution gas were used for very robust plasma conditions.

The calibration standards were prepared in the appropriate mobile phase with respect to the sodium phosphate buffer, due to some contamination issues in the salt. The calibration curve was checked with Nist 1643e, which was diluted 1+9 in the appropriate mobile phase. All recoveries where in between +/- 20%.

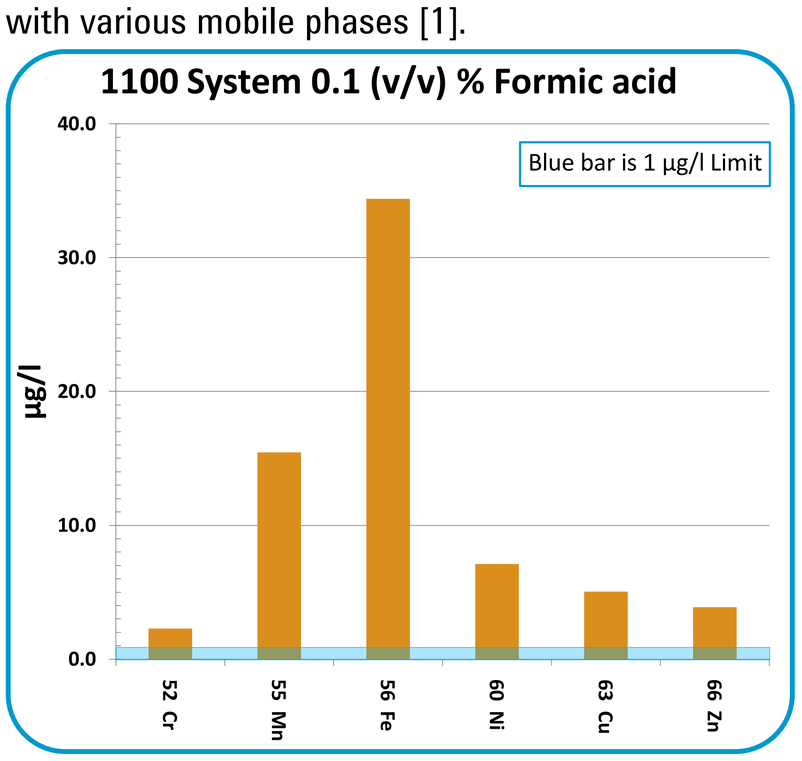
Results and Discussion

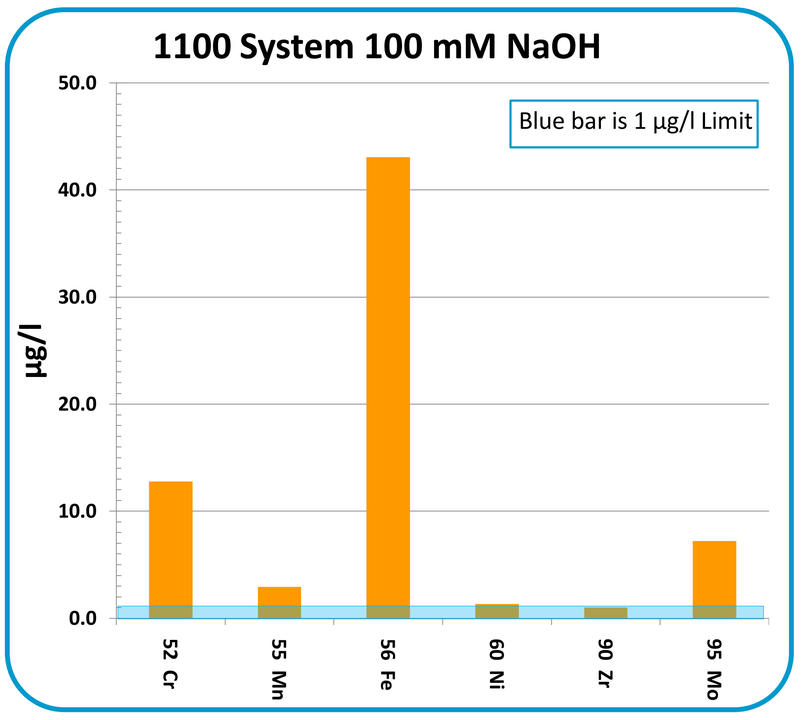
The following table shows a summary of all results during these experiments. All values are given in µg/l. The gas mode and the isotopes are shown in the table as well. The values are average values from 3 taken samples. The respective Standard deviation is given in the table as well. Most elements were below the detection limit in the corresponding run. Detection limit was calculated with three times the standard deviation (s) of the blank signal counts, divided by the sensitivity factor based on analysis of a standard solution.

As already mentioned titanium, platinum, ceramic and inert polymers are involved in the sample path of the Bio-inert LC. The only element which was detected in slightly higher values was titanium in formic acid and in NaOH. But mostly all other values are below 1 µg/l or below the DL and no big influence of various pH was observed.

[ug/l]	Formic acid 0.1% (v/v)	Stdev.	25 mM Sodium phosphate	Stdev.	100 mM NaOH	Stdev.
47 Ti [He]	1.441	0.285	<0.090		13.481	0.214
51 V [He]	<0.031		0.046	0.015	0.005	0.002
52 Cr [He]	0.025	0.005	0.066	0.014	<0.031	
55 Mn [He]	<0.028		<0.009		<0.021	
56 Fe [H2]	<0.041		<0.057		<0.093	
59 Co [He]	0.007	0.0002	<0.003		<0.011	
60 Ni [He]	<0.055		0.202	0.014	<0.299	
63 Cu [He]	<0.021		<0.013		0.040	
66 Zn [He]	<0.058		<0.230		<0.024	
90 Zr [No Gas]	0.211	0.004	<0.006		1.243	
93 Nb [No Gas]	<0.003		<0.003		0.011	0.002
95 Mo [No Gas]	<0.009		<0.007		0.149	0.007
105 Pd [No Gas]	<0.017		<0.004		0.004	0.000
107 Ag [No Gas]	<0.008		0.046	0.006	0.011	
111 Cd [No Gas]	<0.006		<0.008		0.014	0.005
138 Ba [No Gas]	0.041		0.031	0.011	<0.235	
195 Pt [No Gas]	<0.037		<0.006		<0.020	
197 Au [No Gas]	<0.002		<0.006		0.048	0.002
205 Tl [No Gas]	<0.001		<0.006		<0.006	
208 Pb [No Gas]	0.009	0.001	<0.015		0.063	0.010

The following two grafics are values from a technical note. In this note the 1100 HPLC system was tested with various mobile phases [1].





The data we present here show the results of two mobile phases which were tested in the technical note exploring metal release from the Agilent 1100 system.Therefore these values can be used as a comparison to a non-inert standard HPLC system.

The small blue bar at the bottom of the axis reflects 1 µg/l as a threshold value. All values, with respect to titanium, were detected lower than 1 µg/l.

In comparison the metal realease of a standard HPLC system is much higher than from the Agilent Bio-inert HPLC system. Also the effect of basic conditions in 100 mM NaOH were significantly higher with the 1100 system.

The results demonstrate the high corrosion resistance of the 1260 Bio-inert system against various mobile phases. The only higher value was titanium in 100 mM NaOH which is not surprising since the the pump is based on this corrosion resistant material. Particulary elements which normally are released from stainless steel systems like chromium, manganese, iron, nickel, copper and zink were not detected in high values during all these experiments.

Conclusion

The new 1260 Infinity Bio-inert HPLC shows very high chemical resistance against low and high pH solvents and high ionic strength buffers. Most elements could not be detected because the value is below the detection limit. Others like Ti and Zr showed concentrations around 1 µg/l and lower. This is due to the fact that all wetted stainless steel parts of the pump have been replaced by corrosion resistant materials like titanium, platin, ceramic and inert polymers.

Therefore the 1260 Infinity Bio-inert HPLC is not just a solution for typical HPLC Bio-applications, it is also a front-end solution for HPLC-ICP-MS when very low background of metals is essential.

[1] Peter J.W. Stone and Glenn D.Woods, Agilent 1100 HPLC Systems for Ion-Sensitive Analyses and LC-ICP-MS Applications, Technical Note, Agilent Technologies