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Lead quantitation challenge

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1 Meet the challenge

The discovery of lead belongs to the accounts of ancient history. This heavy metal was known to Egyptians, Hebrews, Phoenicians, Greeks, and Romans which used it and already had some understanding of its toxicity [1–3]. Lead poisoning has been related to several symptoms which include neurological and behavioral effects, abdominal pain, anemia, arthritis, and damages to kidneys [1, 2]. Across history, numerous examples of widespread plumbism have been documented. In the Roman Empire, lead cookware and utensils were common and lead acetate was used as a sweetener for food and wine. As a result, the average daily intake of lead for Roman aristocrats was well-above those of lower class [1]. In fact, the widespread occurrence of saturnine gout in Roman aristocrats has led some scholars to suggest that lead poisoning has played an important role in the fall of the Roman Empire [1]. Another epidemic of gout affected England during the 18th century and is related to lead content in wines [1, 3]. Closer to our days, the use of lead paints in housing and tetraethyl lead in gasoline has contributed to large-scale pediatric lead poisoning for most of the 20th century [2]. The severity of lead poisoning has gained the attention of many regulatory bodies and led to establishment of safe exposure limits in order to prevent toxic effects of lead [2]. As an example, lead content in drinking water is now widely regulated worldwide and Canadian standards allow a Maximum Acceptable Concentration of 5 μg/L [4].

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2 The challenge

At the very low concentration of 5 μg/L, robust quantitation of lead is generally achieved by inductively coupled plasma mass spectrometry (ICPMS). As shown in Fig. 1, lead consists of four stable isotopes. As the most abundant isotopes, lead-206, lead-207, and lead-208 are usually employed for sensitive quantitation of lead by ICPMS.

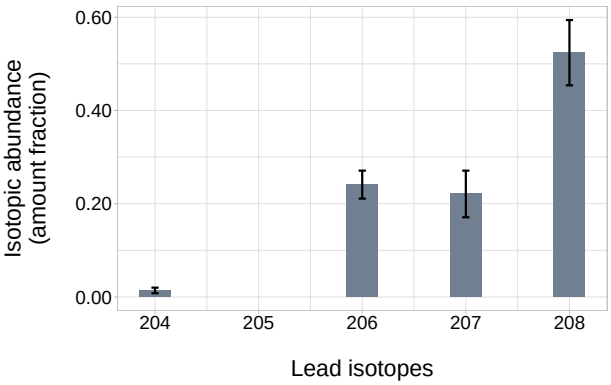

Fig. 1: Isotopic composition of lead: amount fraction of its isotopes and their natural variations in common lead [5]

As part of a quality assurance program, a synthetic drinking water sample (A) was sent for lead analysis to two laboratories. In both cases, three lead isotope signals (lead-206, lead-207, and lead-208) were used for quantitation and quality control purposes. The calibration curve was prepared by analysis of available aqueous lead standard solutions and was used to determine the mass fraction of lead in the drinking water sample A, $w(\mathrm{Pb})_\mathrm{A}$. Table 1 contains all measurement data reported by the two testing laboratories.

Table 1: Determination of lead in a synthetic drinking water sample by two laboratories[a]

Lab. 1 *w*(Pb)	^{206}Pb	^{207}Pb	^{208}Pb	Lab. 2 *w*(Pb)	^{206}Pb	^{207}Pb	^{208}Pb
0.00	0	0	0	**0.00**	0	0	0
0.95	378	326	796	**1.20**	455	342	861
2.50	994	857	2094	**3.00**	1138	855	2153
3.40	1352	1165	2848	**5.20**	1973	1482	3733
5.90	2346	2022	4942	**8.30**	3149	2365	5958
7.20	2863	2468	6031	**11.0**	4173	3134	7896
Sample	1861	1748	4117	**Sample**	1629	1530	3603

[a] *w*(Pb) is the mass fraction of lead in the calibration standards (μg/L). ^{206}Pb, ^{207}Pb, ^{208}Pb are the ICPMS signals measured at m/z of 206, 207, 208 Da.

Laboratory 1 reported the following results:

$$\begin{aligned} &^{206}\text{Pb: } w(\text{Pb})_{\text{A}} = 4.7\ \mu\text{g/L} \\ &^{207}\text{Pb: } w(\text{Pb})_{\text{A}} = 5.1\ \mu\text{g/L} \\ &^{208}\text{Pb: } w(\text{Pb})_{\text{A}} = 4.9\ \mu\text{g/L} \end{aligned} \tag{1}$$

Laboratory 2 reported the following results:

$$\begin{aligned} &^{206}\text{Pb: } w(\text{Pb})_{\text{A}} = 4.3\ \mu\text{g/L} \\ &^{207}\text{Pb: } w(\text{Pb})_{\text{A}} = 5.4\ \mu\text{g/L} \\ &^{208}\text{Pb: } w(\text{Pb})_{\text{A}} = 5.0\ \mu\text{g/L} \end{aligned} \tag{2}$$

Despite the absence of outliers or spectral interferences, a large disagreement between the results was observed with values ranging from 4.3 μg/L Pb to 5.4 μg/L Pb. Notably, neither of the laboratories were able to determine if the lead levels in the water sample are above or below 5 μg/L Pb [4].

Can you provide reasons for the disagreement between the lead concentrations reported by the these laboratories?

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