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The used pretreatment method has an influence on the participant results and, the participant performance evaluation in the PTs. Therefore, the influence has to be taken into account when evaluating the PT results. That enables participants to compare their results with the others using similar pretreatment method.

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Influence of sample pretreatment of PT samples for metal analyses

Various kinds of sample pretreatment methods are used in laboratories based on the purpose of the field of analysis or the requirements of international standards or authorities. The sample pretreatment could have an impact on the result especially in metal analyses.

Various pretreatment methods

For metal analyses of solid (e.g. soil, fly ash) and industrial wastewater samples proficiency test (PT) provider Proftest Syke at Finnish Environment Institute provides a possibility to report the results according to the used pretreatment method. The results are grouped and evaluated separately according to the pretreatment method.

The PT participants can use different pretreatment methods as there are no national requirements for the used pretreatment method in Finland.

In Proftest Syke PTs for solid samples statistically significant differences have been observed between nitric acid and aqua regia digestion pretreatment methods.

The participants mostly use nitric acid (HNO_3) or aqua regia (1:3 or 3:1 HNO_3:HCl) digestion for the metal analyses of solid samples.

For the industrial wastewater samples the participants report results of metal analyses in two groups: without sample pretreatment and with acid digestion pretreatment.

The results from different sample pretreatment methods for metal analyses are evaluated separately, and the reported results are statistically tested for differences between the used sample pretreatment methods.

Influence to PT evaluation

• For fly ash sample, the participants using nitric acid digestion pretreatment method reported significantly higher results for V and Zn than the participants using aqua regia digestion pretreatment method (Figure 1) [1].

• For soil sample, the participants using nitric acid digestion pretreatment method reported lower results for Al, Ba, Ca, Cr, Fe, K, Mg, Mn, and V than the participants using aqua regia digestion pretreatment method (Figure 2) [2].

• For industrial wastewater samples, the participants using acid digestion as pretreatment method reported significantly higher results for Al, Ca, B, Cr, Fe, Mo, Ti, and Zn than the participants who reported their results without sample pretreatment (Figure 3) [1, 2].

Conclusion

The used acids and their amount ratio, the sample weight or volume in relation to the acid volume, as well as the digestion temperature and hold times, can highly influence the recoveries.

Thus, the used pretreatment method could have an influence on the participant results and, further, the participant performance evaluation.
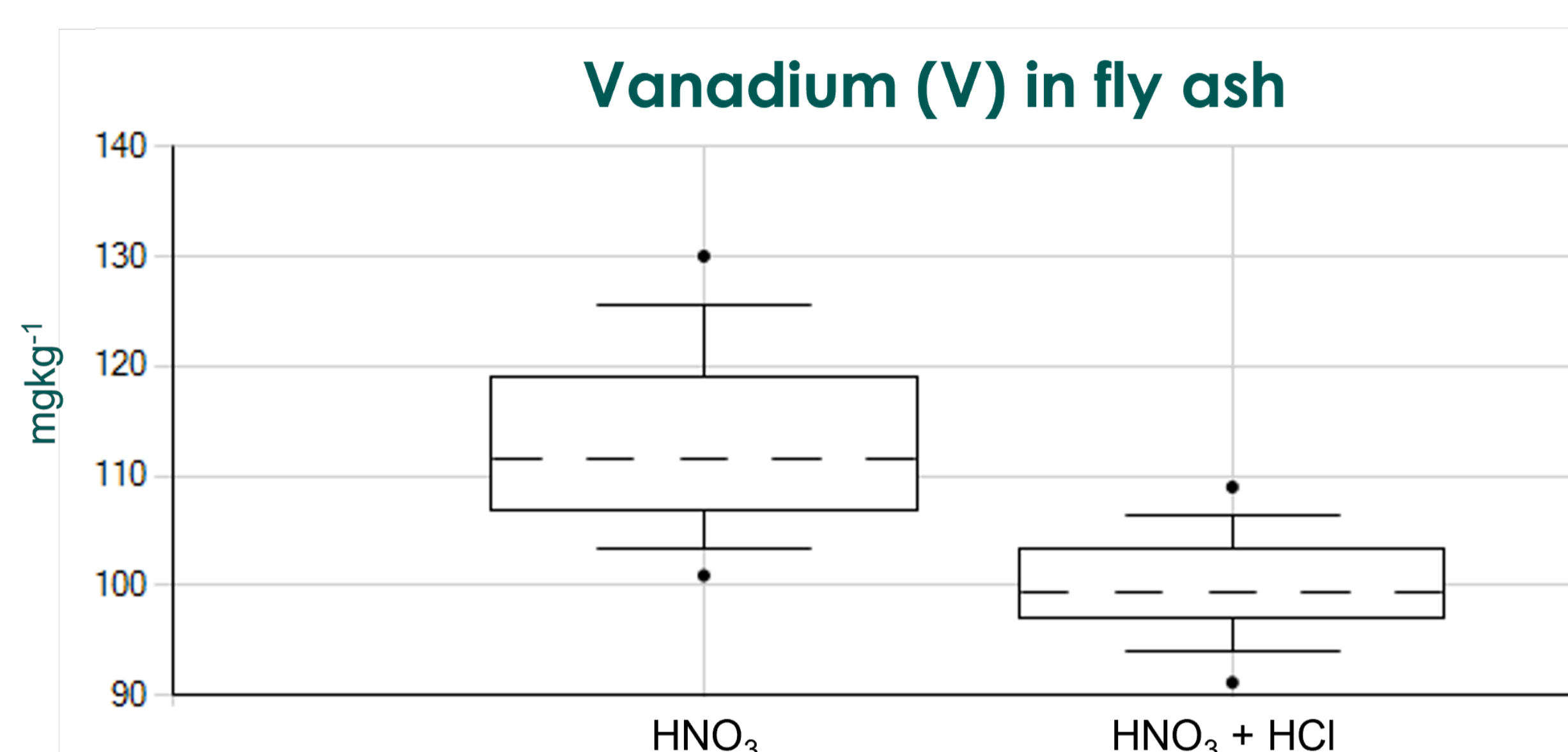

Figure 1. An example of statistically significant difference for vanadium in fly ash sample after different pretreatment methods [1].*
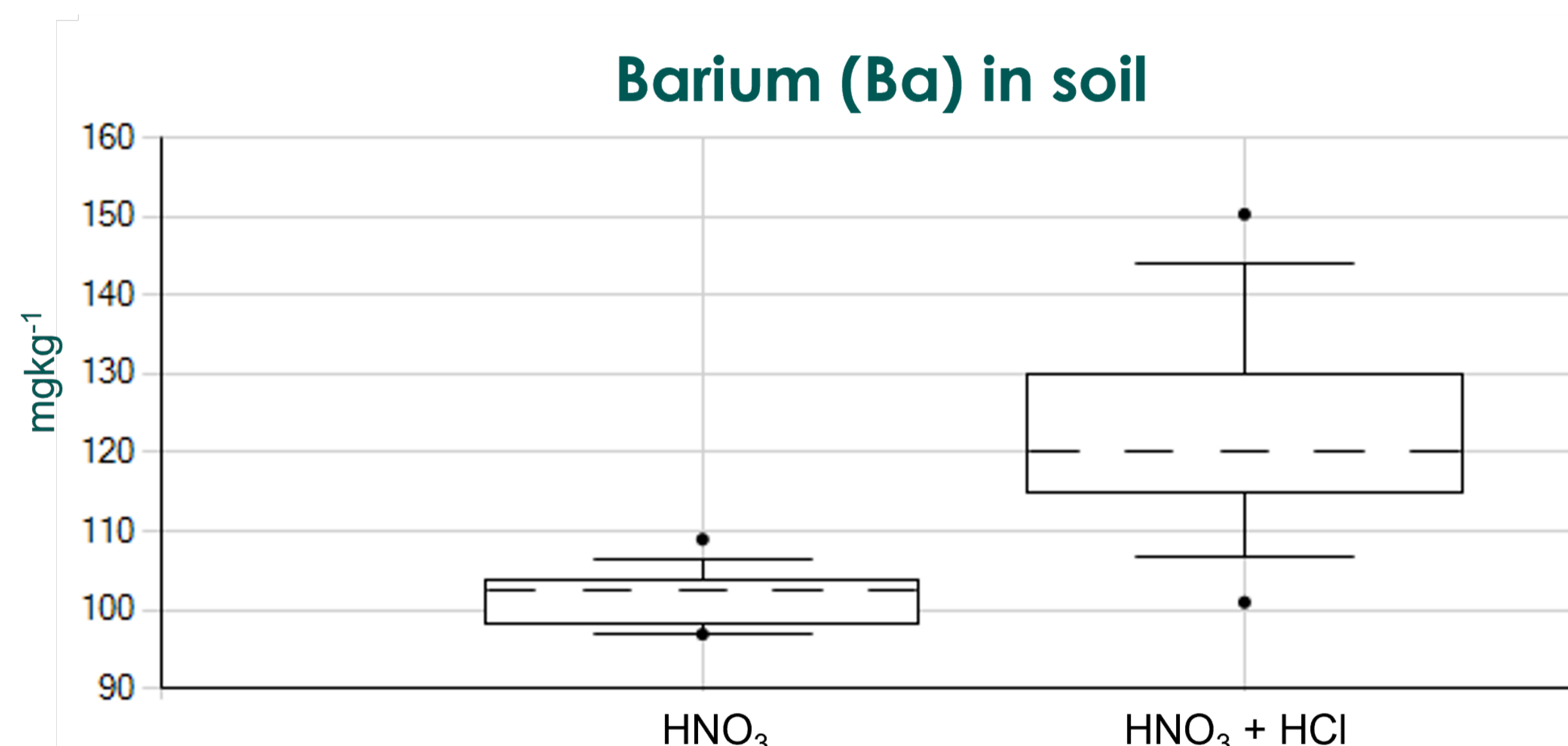

Figure 2. An example of statistically significant difference for barium in soil sample after different pretreatment methods [2].*
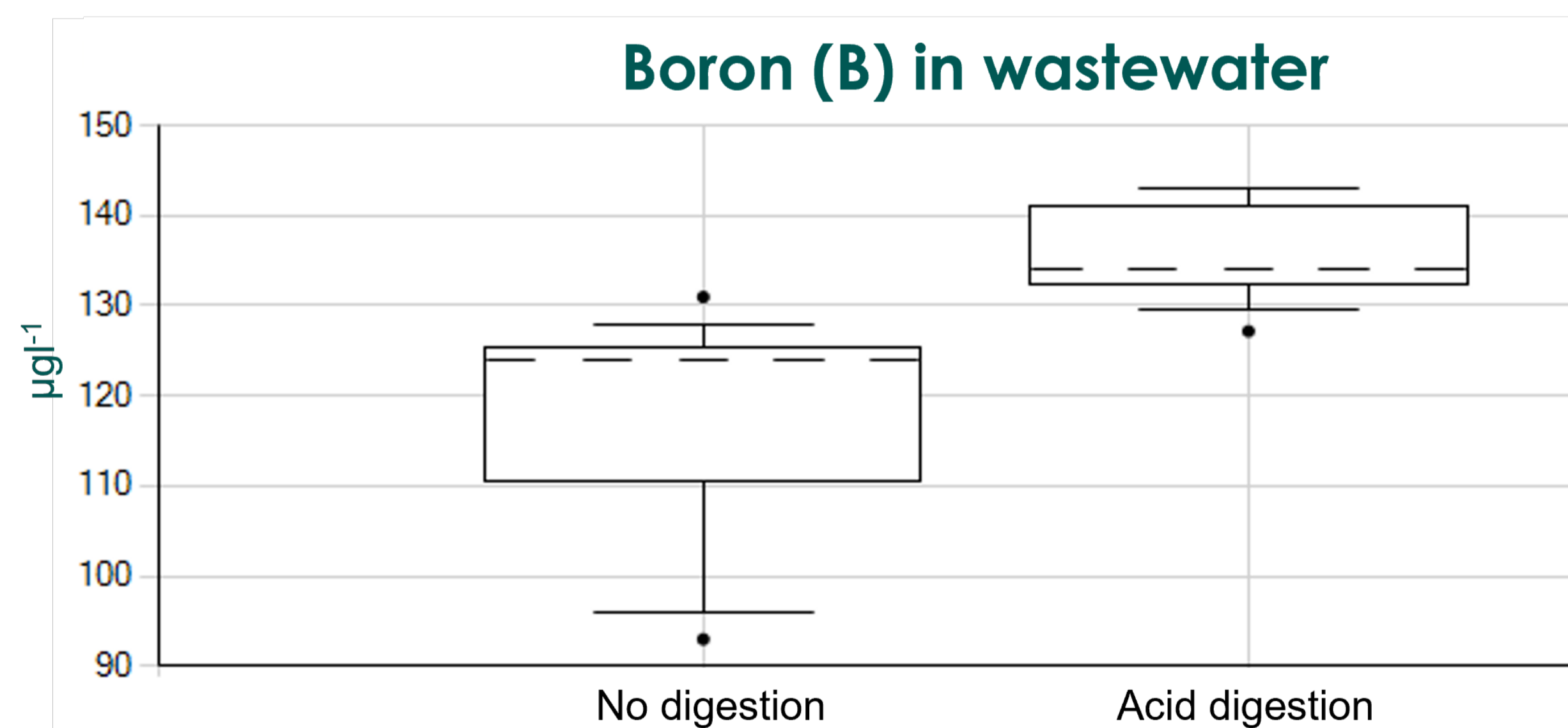

Figure 3. An example of statistically significant difference for boron in wastewater sample after pretreatment methods [2].*

*In the boxplot figures 1-3:
• In the box the upper and lower limit included 50 % of the results.
• The dashed vertical line in the middle of the box is the median of the results.
• The vertical lines above and under the box describe the limits of 80 % of the results.
• The black dots describe the highest and smallest results with-in the center 90 % of the results.

References:

[1] Leivuori M. et al. 2023, Proficiency test 12/2022 Wastewaters and fly ash metal analyses, Reports of the Finnish Environment Institute 10/2023 (http://hdl.handle.net/10138/357229).

[2] Leivuori, M. et al. 2022, Interlaboratory proficiency test 10/2021, Wastewater and soil metal analyses, Reports of the Finnish Environment Institute 16/2022 (http://hdl.handle.net/10138/343009).

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