

Study of reproducibility in scientific publications based on the statistical evaluation of validation results and uncertainty estimation in analytical chemistry.

Bruna Ferreira¹, Vitor Paccès², Igor Olivares³, Emanuel Carrilho⁴

¹ University of São Paulo, Chemistry Institute (IQSC-USP); brunadrielen@iqsc.usp.br

² University of São Paulo, Chemistry Institute (IQSC-USP); vitorusp@iqsc.usp.br

³ University of São Paulo, Chemistry Institute (IQSC-USP); igorolivares@iqsc.usp.br

⁴ University of São Paulo, Chemistry Institute (IQSC-USP); emanuel@iqsc.usp.br

Keywords: reproducibility, validation, software, uncertainty, statistics.

Introduction

The data presented in 2016 by the article “1500 scientists lift the lid on reproducibility” by Monya Baker², brought up a discussion about the potential and significant crisis of reproducibility in scientific research. In the same article, the knowledge causes were raised according to the experience of the consulted researchers, and the most prominent points refer to the low power and application of statistical tools and a better application of intra-laboratory validation. Within this context, in this work, the research was developed based on verification of this information in Chemistry (area whose researchers reported the most difficulty in reproducing results).

Objective

Check whether the perception of the existence of a reproducibility crisis could be confirmed and quantified with the application of the performance criteria for the validation of analytical methods presented in scientific documents as mentioned in that article.

Development

It was used validated software aimed at testing laboratories, called ConfLab Validation and ConfLab Uncertainty (www.ConfLab.com.br) to obtain data and reference values, were used, which were designed to attend the main Quality Management Systems related to these requirements. The discussions took place by comparing the results obtained as they were presented in the original documents with the results obtained by the software. The results presented provide evidence that justifies the possible existence of problems related to the lack of knowledge about the correct use of tests and statistical tools, which promotes the misapplication of performance parameters and, consequently, an inappropriate analysis of the data.



+



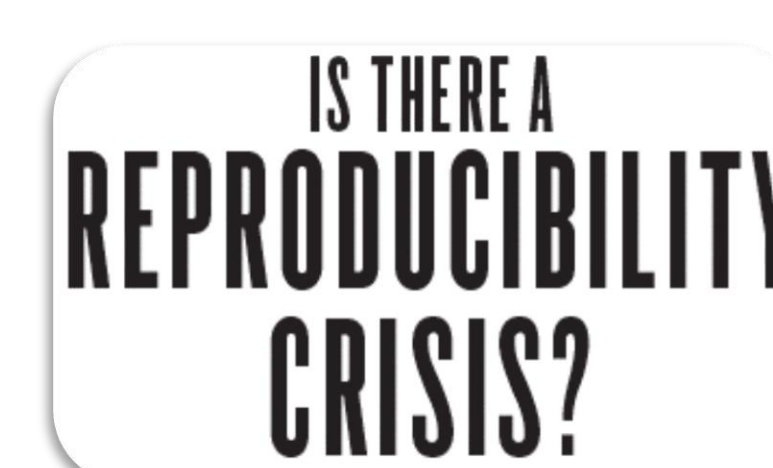
ConfLab

+



Criteria of acceptance

=

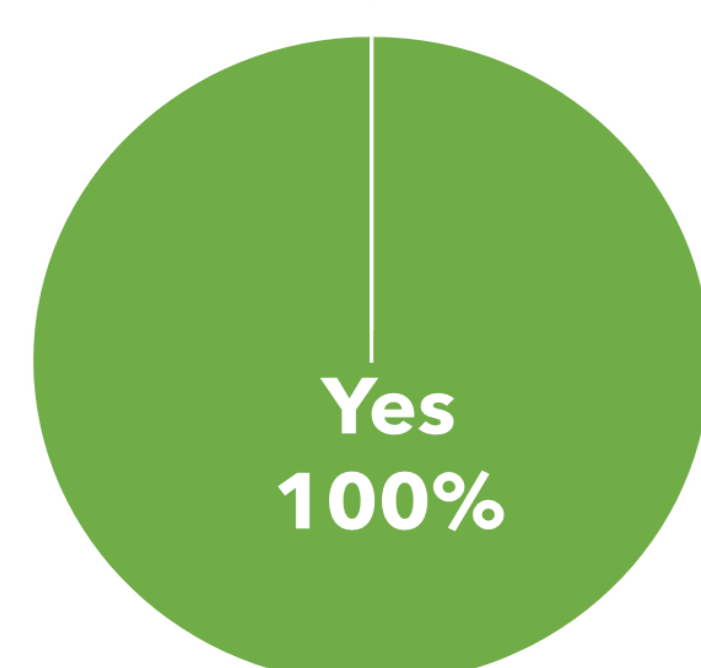


Results

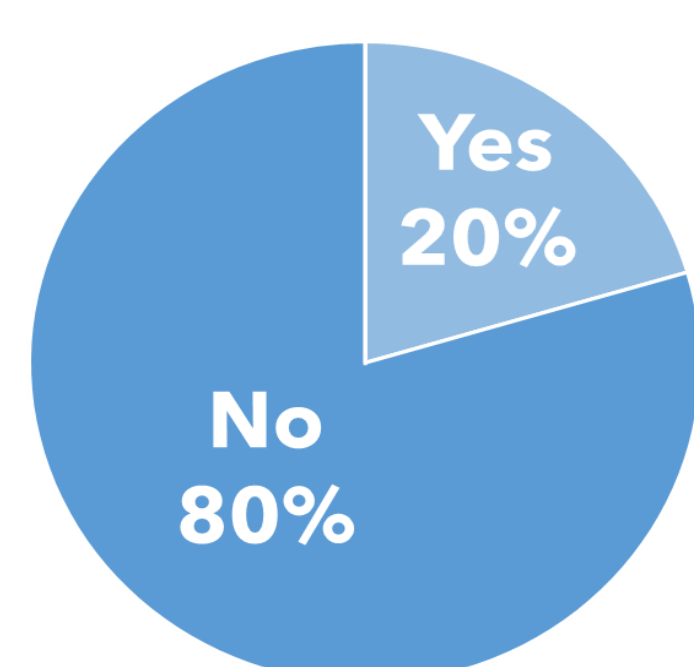
Figure 1: Characterization about the application of the protocols a) frequency that the authors understood the contexts of application of their method b) of the performance parameters that were performed, the author correctly understood the form of application of the test in all of them.

Characterization according to the protocol used

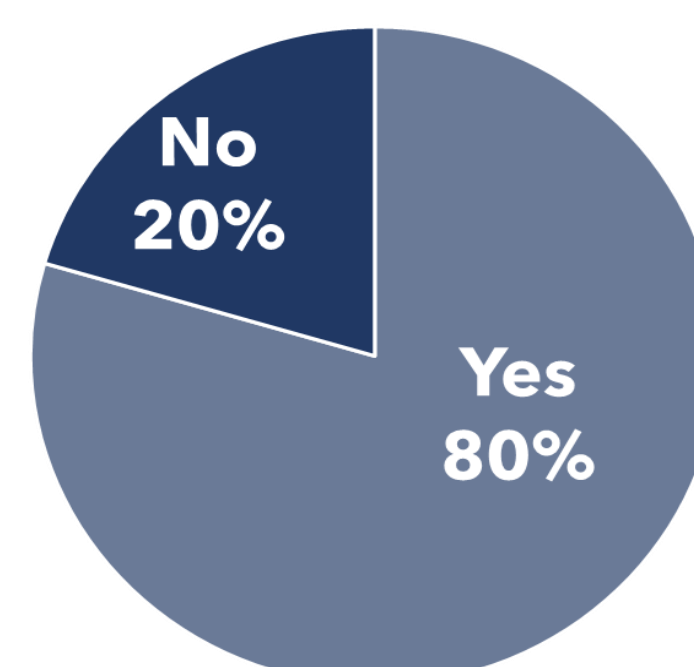
Mention of the use of any protocol



Use of the correct protocol



Use of more than one protocol



Accomplishing all the parameters required by the standard

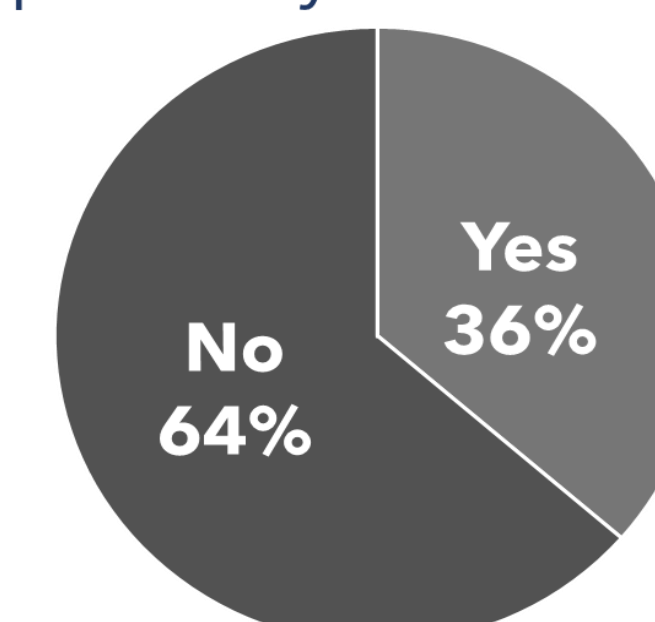
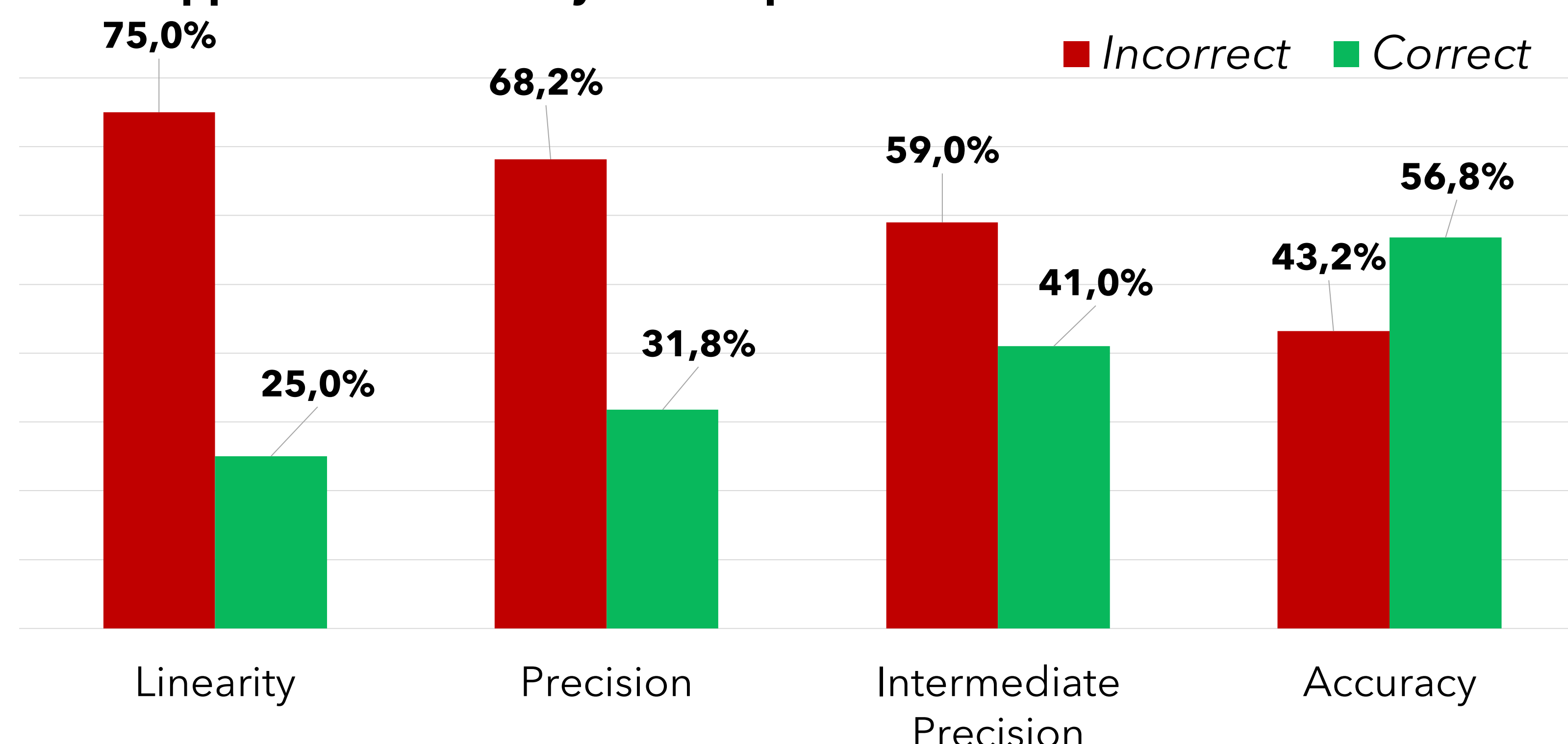


Figure 2 - Comparison between the frequencies that the test and the study of the performance parameter were performed correctly with incorrect ones.

Application and study of each parameter - Correct vs Incorrect



Conclusion

With the data obtained so far, it is possible to be close to establishing a confirmation of the lack of knowledge about the correct application of method validation and uncertainty calculations as statistical tools, which can result in the misuse of performance parameters. This information increases the likelihood of admitting the significant negative influence of this confirmation on the reproduction of published methods, as stated in that article (BAKER, 2016). However, for a more precise conclusion, studies based on uncertainty estimation calculations will still be carried out to relate these conclusions to the existence of a crisis of reproducibility of analytical methods. The next step is discussions based on calculations of estimation of measurement uncertainty will be added to assess their potential contribution to the effects of the reproducibility of analytical methods.

Acknowledgments

This research was funded by CAPES, CNPq, IQSC/USP and Softwares ConfLab.

References

1. ABNT NBR ISO / IEC 17025: 2017. Third ed. Rio de Janeiro: ABNT ISO / IEC, 2017.
2. B. M. 1,500 scientists lift the lid on reproducibility. Nature, 2016, Volume 533, n. 7604, p. 452–454.
3. B. V. Studies for the validation of planning and reporting methods: Supplement to the Eurachem Guide on fitness for the purpose of analytical methods. 2019, p. 32.
4. EURACHEM / CITAC. Use of uncertainty information in conformity assessment. First UK edition: EURACHEM and CITAC Working Group, 2007.
5. EURACHEM; CITAC. CG Guide 4. Third ed. Sussex, United Kingdom: Eurchem and CITAC Working Group, 2012.
6. JCGM 200:2012 International vocabulary of metrology – Basic and general concepts and associated terms (VIM) - 3rd edition, 2012.
7. PACCES, V.H.P. et al. Practical Application of the Concepts of Guarantee the Validity of Results with Focus on ISO / IEC 17025: 2017. 1. ed. São Paulo-SP: REMESP, 2020.