Green Analytical Chemistry

Protocols' guidelines for authors

GREE(N)AC is the first research journal in the field of analytical chemistry which publishes (next to regular research articles, reviews, short communications, letters and perspectives) articles in the *Protocol* format. *Protocols* are detailed descriptions of previously developed analytical methods in the form of a ready-to-use recipe, along with key information facilitating its implementation and comparison against other competing methods.

The main objective of *Protocols* is to describe the procedures already developed by analytical chemists, which are generally appreciated and applied in the analytical community. The authors should have sufficiently extensive and documented experience in using the described method. It is not necessary that the authors of the *Protocol* were also the authors of the original method. In addition to analysts working in academia, *Protocols* can be written by researchers using the method of interest as a routine procedure in their daily laboratory work in the commercial and industrial sectors.

Presented methods can incorporate minor modifications and improvements over the primary methods, for example highlighting the green aspects of the method, however without affecting the physicochemical fundamentals. Every method described as a *Protocol* should be appropriately validated, even after minor modifications.

Publication in the format of a *Protocol* is especially recommended to authors/users of high-quality analytical methods consistent with the concept of sustainable development, and interested in its popularization and promotion of methodological/instrumental innovations used. The described methods can be used in laboratories with different specificity, not necessarily analytical, e.g. for the analysis of the composition of chemical synthesis products. Proposals from different geographical regions, e.g. from economically less developed countries, showing standard methods that are in use in the reality of the limited availability of laboratory infrastructure, are also very welcome.

This new format of articles published in the Green Analytical Chemistry journal will be especially useful for researchers interested in quick and smooth implementation of well verified methods addressing specific analytical problems, and their factual comparisons to other alternative methods.

Protocols will be published by invitation of the Editor(s), but also unsolicited authors' proposals sent to Editorial office prior to submission will be considered. There is no words limit for *Protocols*, but it is required that each of them include the following sections:

1. Introduction:

a. primary objective of the method (type of analysis, detailed description of the analytes, the analytical techniques used, type and nature of the sample and its matrix)

b. theoretical basis of the method

c. calibration methodology, calibration/validation data, measures for assuring data quality

d. modifications and improvements compared to the primary method with the supporting data

2. Material and Human Resources:

a. a list of all chemicals with a detailed description (IUPAC name, manufacturer, purity, molecular weight, CAS number, estimated volume/mass of a given reagent used for analyzing a considered number of samples, related risk factors/hazard pictograms)

b. research instruments (name of the device, exact type, manufacturer, media used, estimated consumption of utilities, e.g. electricity for analyzing a considered number of samples)

c. list of other materials and small laboratory equipment (name, manufacturer, quantity for analyzing a considered number of samples)

d. human resources (the estimated number of working hours of qualified personnel for analyzing a considered number of samples, the required qualifications and the estimated number of training hours of new personnel required for the correct and independent application of the method)

3. Protocol

The exact description of all the stages of the analysis step by step, with the numbering of all the activities, along with a detailed description of the preparation of instruments and reagents, calibration, data analysis, procedures for the finishing of work and rinsing the equipment. Some steps should be emphasized by placing a verbal colored mark in the following cases: particularly dangerous for the staff or physically burdensome (**ATTENTION!**), particularly difficult manually or requiring a lot of experience (**TRICKY!**), or which, due to their long duration, do not require the constant assistance of an analyst (**PAUSE!**). Moreover, the duration of each step should be defined with the objective range indicated by the symbol: **TIME: A-B min**. Here is an example:

Step 1. **TIME: 10 - 15 min.** *A detailed description of the implementation of step 1*

Step 2. **TIME: 5 - 7 min. ATTENTION!** *A detailed description of the implementation of step 2*

Step 3. **TIME: 20 - 25 min.** *A detailed description of the implementation of step 3*

Step 4. **TIME: 10 - 15 min. TRICKY!** *A detailed description of the implementation of step 4*

Step 5. **TIME: 30 - 40 min. PAUSE!** *A detailed description of the implementation of step 5*

…

Step X. **TIME: 15 - 20 min .** *A detailed description of the implementation of step X*

4. Problem solving: description of the most common problems encountered in the application of the procedure, indicating the stage at which they occur, their nature, probable cause and recommended solutions for their elimination.

5. Alternative methods: brief description of the most frequently used alternative methods in relation to the method described in *Protocol* (instrumental techniques used, the most important differences from the presented method of practical importance)

6. Protocol evaluation

As input for the assessment, in addition to the standard validation data, the following should be considered: cost of analysis (expressed by the actual use of individual resources, without the need to provide market prices – because they are variable and dependent on time and place), analysis time, sample consumption, reagent consumption/waste production, reagent toxicity, capability for recycling residues, environmental hazards, estimated energy consumption or resulting carbon footprint, degree of miniaturization, automation and portability. These parameters should be assessed carefully and objectively, using the data provided in the previous sections. All calculations of the above parameters should be provided together with a description of the assumptions made. In the case of parameters that are difficult to quantify, a qualitative assessment is possible, e.g. by comparing it with another popular technique. It may be helpful to use the following template tables:

Table 1. An example of how to calculate the total analysis time and the amount of waste generated.

Step	**Duration**	**Volume of waste generated**
1. Preparation of reagents: conducted 1x	10-15 min	100-150 mL
2. Preparation of instruments: conducted 1x	5-10 min	-
3. Calibration: conducted 1x	20-25 min	200-300 mL
4. Sample analysis: conducted 60x*	3 min x 60* = 180 min	10 mL x 60* = 600 mL
5. Data analysis: conducted 1x	30-40 min	-
…	…	…
X. Finishing of work: conducted 1x	10-20 min	100-200 mL
Totally:	**255-290 min***	**1000-1250 mL***

* for analyzing 20 samples, each determination carried out in triplicate (60 runs)

Table 2. An example of how to present data facilitating estimation of the total cost and indication of the most expensive components.

Component of the cost estimate	**Real consumption***	**Approximated percentage of the total cost******
Reagent 1	0.1 L	10%
Reagent 2	0.5 L	5%
Material 1	20 pieces	5%
Material 2	1 meter	20%
Instrument 1	Depreciation rate: 0.010%** 1 working hour needed	40%
Instrument 2	Depreciation rate: 0.001%** 2 working hours needed	15%
Electricity	100 kWh	5%
Personnel	not applicable***	0%

* for analyzing 20 samples, each determination carried out in triplicate (60 runs); ** the adopted level of depreciation (in the case of research equipment, it is reasonable to assume the rate from 0.010% (rapid loss of value) to 0.001% (slow loss of value) for 1 hour of continuous operation of the device; *** estimation of the cost related to the employment of qualified personnel may be relevant to commercial and service laboratories; **** relative values, depending on the time, place and particular application, given to identify the most expensive components of the described procedure

Table 3. An example of how to assess safety/environmental hazards related to the given analytical method.

Hazard type	**Exposition**
High temperature	small (1/3)
High voltage	none (0/3)
Noise/ultrasounds	medium (2/3)
Toxic vapors	large (3/3)
Injury/sharp objects	none (0/3)
Biohazard	none (0/3)
Radiation	none (0/3)
Others	none (0/3)
Summary	**6/24 = 75% safety level**

The method of estimating the above parameters is not defined explicitly, although it should always ensure the greatest possible objectivity. In the absence of required estimates or doubts related to their calculation, the authors may be asked to make appropriate corrections/clarifications at the stage of the peer-review process.

The comprehensive evaluation should be the basis for an in-depth discussion of the method in comparison to the alternative methods described, its strengths and weaknesses, the resulting most preferred applications, and future directions for the further development of the methodology. It should be demonstrated to what extent the method is consistent with the idea of Green Analytical Chemistry, and secondly, what is its sustainability expressed in compromising greenness with actual functionality (compliance with the idea of White Analytical Chemistry). It is recommended, but not required, to use special tools for method assessing: Eco-Scale, AGREE, GAPI, RGB 12, HEXAGON, MCDA, etc:

A. Gałuszka, Z.M. Migaszewski, P. Konieczka, J. Namieśnik, Analytical Eco-Scale for assessing the greenness of analytical procedures, TrAC-Trends Anal. Chem. 37 (2012) 61-72

F. Pena-Pereira, W. Wojnowski, M. Tobiszewski, AGREE—analytical GREEnness metric approach and software, Anal. Chem., 92 (2020) 9210076-9210082

J. Płotka-Wasylka, A new tool for the evaluation of the analytical procedure: green analytical procedure index, Talanta, 181 (2018) 204-209

P.M. Nowak, R. Wietecha-Posłuszny, J. Pawliszyn, White Analytical Chemistry: An approach to reconcile the principles of Green Analytical Chemistry and functionality, TrAC-Trends Anal. Chem. 138 (2021) 116223

A. Ballester-Caudet, P. Campíns-Falcó, B. Pérez, R. Sancho, M. Lorente, G. Sastre, C. González, A new tool for evaluating and/or selecting analytical methods: summarizing the information in a hexagon, TrAC-Trends Anal. Chem. 118 (2019) 538-547

P.M. Nowak, P. Kościelniak, M. Tobiszewski, A. Ballester-Caudet, P. Campíns-Falcó, Overview of the three multicriteria approaches applied to a global assessment of analytical methods, TrAC-Trends Anal. Chem. 133 (2020) 116065

7. Summary and conclusions

A clear conclusion on the preferred use of the described method and its largest advantages / limitations, based on the evaluation result including the green and functionality criteria.

8. Literature

9. Information about the authors (not obligatory)

A profile picture and short description (max. 150 words) of the authors' scientific and research profile: short CV, positions held, scientific achievements, scientific and other interests.

10. Supplementary material (not obligatory)

In addition to the above, it is possible to add other supplementary sections. Visualizations of the selected crucial steps of the protocol in the form of photos, videos or detailed schemas are particularly welcome.