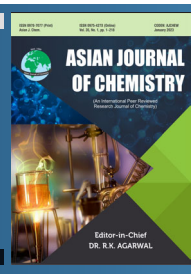
Asian Journal of Chemistry; Vol. 35, No. 11 (2023), 2726-2732

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2023.28553



Modification of QuPPe-PO-Method for the Determination of Glyphosate and Aminomethylphosphonic Acid using High Strength Silica Chromatographic Column

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Received: 10 August 2023; *Accepted*: 22 September 2023; *Published online*: 31 October 2023; AJC-21428

Glyphosate (Gly) is a popular herbicide often used for crop desiccation. The use of glyphosate leads to the contamination of the agricultural products as well as soil, surface and groundwater. For food safety control and environment monitoring, a method developed based on liquid chromatography tandem mass spectrometry, which allows quantitative determination of glyphosate and aminomethylphosphonic acid (AMPA), without derivatization. The LOQ of Gly and AMPA in raw materials of plant origin is 0.1 mg/kg; LOQ of Gly in water is 0.001 mg/L, AMPA is 0.002 mg/L; LOQ of Gly in soil is 0.02 mg/kg, AMPA is 0.04 mg/kg. Extraction from raw materials of plant origin was performed by acetic acid solution of methanol in water, in the presence of EDTA-Na_2 and dichloromethane. Extraction from water was carried out in the presence of EDTA-Na_2 and acetic acid. From soil, Gly and AMPA were extracted with 0.1% of ammonia solution. The extracts are purified on OASIS HLB; proteins were precipitated with acetonitrile, an aliquot of the extract was concentrated. The resulting calibration dependence equations were linear and the correlation values (R) were ≥ 0.99. Relative standard deviation values were in the range of 1.77-19.5%, while the recoveries were from 80.5 to 108%.

Keywords: Glyphosate, Sample preparation, aminomethylphosphonic acid, HSS T3, QuPPe-PO method.

INTRODUCTION

Quantitative direct determination of glyphosate (Gly) and aminomethylphosphonic acid (AMPA) by ESI-MS/MS is usually performed using zwitterionic, ion exchange and others specific columns and the sample preparation stage is simplified in compared with derivatization based methods [1,2]. The detailed approach based on direct determination of Gly and AMPA in raw materials of plant origin is described by QuPPe-PO method [3]. The method (for cereals, pulses, nuts and oily seeds) implies an extraction by sample soaking in 9 mL of deionized water, with extraction step by 10 mL of methanol containing 1% of formic acid (with extra 0.1 mL of formic acid). A 1 mL of 10% solution of tetrasodium salt of ethylenediaminetetraacetic acid (EDTA-Na_4) was used for the signal improvement. Refrigerated high-speed centrifugation at -10 ºC or a long freeze-out stage at -20-80 ºC was used after the extraction step. For proteins and lipids removal, 2 mL of raw extract is transferred into a tube with 2 mL of acetonitrile (with 0.1 g of C_{18}) for shaking and centrifugation. The final step of the sample preparation is ultrafiltration through a 5 kDa cut-off filter. The usage of high methanol content (50%) at the extraction stage is the one of disadvantages.

Even though methanol allow to precipitate of some proteins, peptides and suspended particles of the sample, at the same time, the extraction completeness of Gly and AMPA is decreased and the extract became saturated with lipids (especially when analyzing oilseeds). Moreover, extracts with a high percentage of lipids are not suitable for analysis on ion exchange columns. Another disadvantage is the usage of dispersive purification with C_{18} sorbent after reaching extract composition 25/75% (when 75% is the mixture of methanol and acetonitrile). It is doubtful that purification efficiency will be enough if the extraction was made with 50% methanol. Many peptides and lipids have masses below 5 kDa, therefore, the feasibility of using an ultrafiltration device is also questionable. For decreasing lipids content in the extracts, it is useful to decrease methanol content and to add a non-polar solvent at the extraction stage,

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e.g. hexane or dichloromethane. Also for complex matrices, it is an adequate decision to use a fast cleanup protocol on Oasis HLB [4] or C_{18} sorbent, but before the protein precipitation stage with acetonitrile. The acetonitrile/extract ratio of 1/1, probably, is not enough for optimum proteins precipitation. There is no information regarding analysis of soils and water in the method.

The aim of this study was the development of a robust and simple approach to the analysis of Gly and AMPA in raw materials of the plant origin, water and soil using HSS-like columns without derivatization.

EXPERIMENTAL

Methanol 99% (CAS 67-56-1), acetonitrile 99% (CAS 75-05-8), formic acid 99% (CAS 64-18-6), dichloromethane (CAS 75-09-2), hexane (CAS 110-54-3), ammonium acetate 99% (CAS 631-61-8), ammonium formate 99% (CAS 540-69-2), 2-aqueous EDTA-Na_2 99% (CAS No. 6381-92- 6), glyphosate (CAS 1071-83-6), AMPA (CAS 1066-51-9), glyphosate-2-^{13}C,^{15}N,-IS (CAS 285978-24-7), hydrochloric acid 37% (CAS 7647-01-0), ammonium hydroxide 25% (CAS 1336-21-6), acetic acid 99% (CAS 64-19-7), Bondesil C_{18} 40 µm sorbent (Agilent, USA), ultrafiltration device Spin-X UF 10000 MWCO (Corning, USA) were purchased from Merck (Germany). Oasis HLB SPE cartridges with 60 mg of sorbent, 3 mL vol. were purchased from Waters (USA). Deionized water was obtained from a Millipore water purification system (Merck, Germany). Extraction and mixing were carried out on Reax 2 and Reax control shakers (Heidolph, Germany). Extracts were concentrated on a Pierce Reacti-Therm III module (Thermo, USA). For determination, the Shimadzu 8060 mass-spectrometer with Nexera X2 chromatograph (Shimadzu, Japan) was used. The following chromatography columns were used in this study: ACQUITY UPLC HSS T3 (1.8 µm, 2.1 × 100 mm (Waters, USA)), Hypersil Gold aQ (3 µm, 2.1 × 100 mm (Thermo, USA)), luna phenyl-hexyl (5 µm, 2 mm × 150 mm (Phenomenex, USA)). Stock solutions of analytes (0.5 mg/mL) were prepared in deionized water. Working mixtures were prepared in concentrations of 0.1, 0.01 and 0.001 mg/mL. The working solution of IS was prepared in deionized water to a concentration of 0.1 mg/mL. For extraction of Gly and AMPA from plant materials, a mixture of deionized water, methanol and acetic acid was used in a percentage ratio of 79/20/1. The EDTA-Na_2 solution was prepared by dissolving 5 g of 2-aqueous salt in 45 mL of deionized water in an ultrasonic bath. HPLC separation was carried out on ACQUITY UPLC HSS T3 column operated at 35 ºC. Phase A - methanol and acetonitrile mixture at 70/30 relation; Phase B - 5% methanol in deionized water. The separation program was as follows: from 0 to 3 min - 2% B, up to 3.5 min gradient to 100% B, from 3.5 to 8.0 min gradient to - 5% A, in 8.1 min - 2% B and to 13 min. The flow rate was set at 0.45 mL/min. The retention times were as follows: Gly (Gly IS) - 4.2 min, AMPA - 4.3 min. The detection was performed in negative ionization mode (Table-1).

Sample preparation for cereals, pulses, nuts, oily seeds and tea: A sample (2 g) with aliquots of standards and IS was mixed with 19 mL of the extraction solution and 1 mL of EDTA-Na_2 solution. The sample was treated on Heidolph Reax 2 shaker for about 20 min. Dichloromethane (5 mL) were added for defatting (do not use for tea analysis) and the extraction was continued for 10-15 min and centrifuged at 4750 rpm and 20 ºC. The extract was applied for the SPE step (Oasis HLB), as described below: cartridge was first pre-conditioned with 2 mL of methanol, 2 mL of water and 0.8 mL of extract (all to waste); then 1 mL of extract was applied on the cartridge and collected into a new tube. 2 mL of acetonitrile was added into the tube with precleaned extract for protein precipitation. The tube contend was mixed on the shaker and centrifuged at 4750 rpm and 4 ºC in 10-15 min. The extract (2 mL) was evaporated till 1 mL (in case of reconstitution the acetonitrile was used) at 45-50 ºC and centrifuged at 15000 rpm and 4 ºC in 10-15 min.

Sample preparation for surface and groundwater: In a polypropylene tube, 5 mL of sample and aliquots of standards (and IS), 0.05 mL of acetic acid and 0.2 mL of EDTA-Na_2 solution were added. The content of the tube were mixed on a Reax control shaker and then the tube was centrifuged at 4750 rpm and 4 ºC for 15-20 min. Next, the Oasis HLB cleanup was applied and subsequent manipulations were carried out as described above. For clear/transparent samples SPE cleanup is not used.

Sample preparation for soil: A 2 g of sample with aliquots of standards and internal standard was mixed with 20 mL of 0.1% NH_4OH solution. The sample was treated on Heidolph Reax 2 shaker for about 20 min and centrifuged at 4750 rpm and 20 ºC. Next, the Oasis HLB cleanup was applied and subsequent manipulations were carried out as described above. Final sample should be stored in a polypropylene inserts (or in a polypropylene vials) before analysis.

RESULTS AND DISCUSSION

Detection: Specifically, glyphosate was identified using daughter ions with masses of 63.05 and 80.9 *m/z*. However it is possible to analyze Gly using 150 and 124 *m/z*, but the intensity was adequate from second/third calibration point. There are several articles reported about the determination of Gly and AMPA on ACQUITY UPLC HSS T3 column [5-8]. But in those of them that relate to the direct determination of Gly, a separation program was chosen incorrectly. This leads to analytes elution before the first minute. Instead of gradient

TABLE-1
DETECTION PARAMETERS

Analyte	Parent ion (*m/z*)	Daughter ions (*m/z*)	Q1 (V)	CE (V)	Q3 (V)
Glyphosate	168.2	63.05/80.9/150	21/18/15	24/14/15	14/17/30
AMPA	110.2	79.15/63.15	25/25	27/22	29/13
Glyphosate IS	171.05	62.95	25	22	13

elution starting immediately after sample injection, it is advisable to enter into the gradient program an isocratic region formed by an organic solvent with a low eluting capacity for analytes. Their retention time will be delayed until water content in the mobile phase become high enough. This can help to obtain Gly and AMPA peaks in more preferable time region. Up to this point, it is recommended to direct the flow from the column, bypassing the source of the mass spectrometer to the drain. There are several chromatographic columns were tested for Gly and AMPA analysis: ACQUITY UPLC HSS T3, Hypersil Gold aQ and Luna phenyl-hexyl. The first time, the best shape of the chromatographic peaks was achieved on the Hypersil Gold aQ column, however, after the sample preparation scheme and gradient were refined, the final choice was made on the ACQUITY UPLC HSS T3 column (Fig. 1). It was found that for HSS T3 column, using of an additives for negative ionization mode in the mobile phases (ammonia solution, formic acid and ammonium acetate/formate) leads to decreasing of the analytical signal.

Extraction: The raw materials of plant origin such as cereals, pulses, nuts, oily seeds and tea are complicated objects for research. To reduce the negative effects of the sample components extracted together with Gly and AMPA, it was necessary to choose the optimal extraction solvent and extract purification method. When developing the extraction method, attention was paid to two aspects: the composition of the main extraction solvent and the additives used to enhance the extraction. In total, six experiments (from A to F) were carried out on a soya beans sample (2 g) contaminated with Gly and AMPA at LOQ level (Fig. 2).

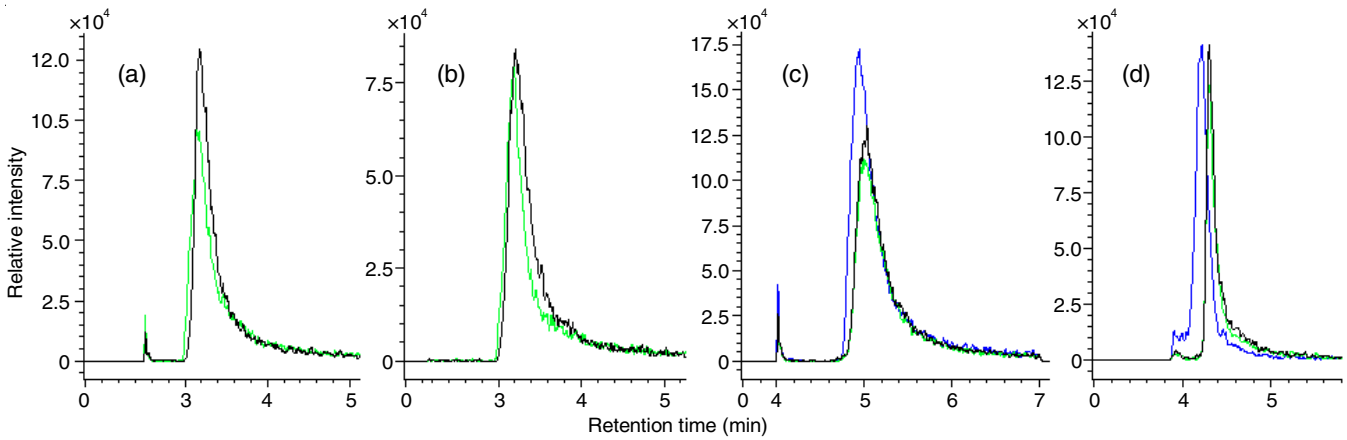

Fig. 1. Mass-chromatograms of Gly and AMPA at 1 mg/kg obtained on various chromatographic columns, (a) Hypersil Gold aQ; (b) luna phenyl-hexyl, (c) ACQUITY UPLC HSS T3, (d) ACQUITY UPLC HSS T3, after gradient optimization

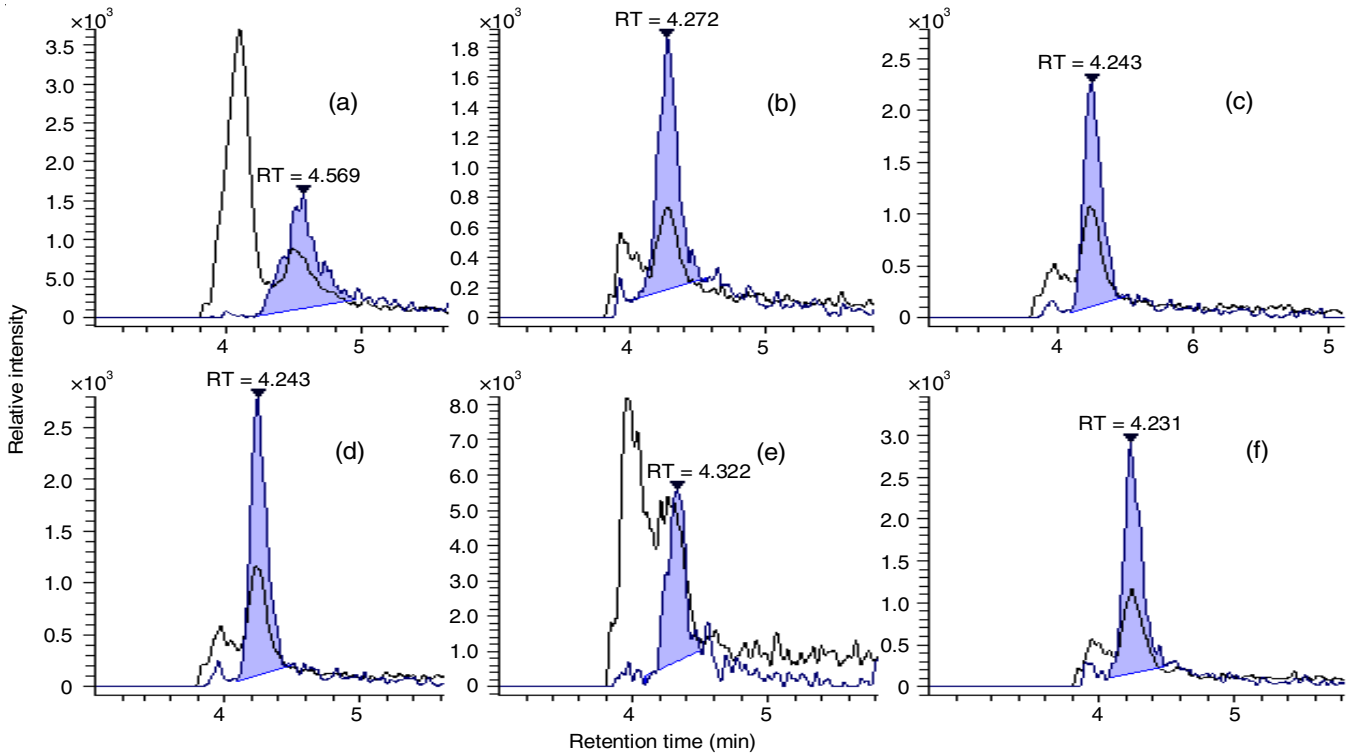

Fig. 2. Mass-chromatograms of Gly product ions in comparison of different extraction ways at 0.1 mg/kg

Sample A: Extracted with 20 mL of 0.08 M HCl solution (addition of 5 mL of dichloromethane for defatting).

Sample B: Extracted with 19 mL of 1% acetic acid solution, 1 mL of EDTA-Na_2 solution and 5 mL of dichloromethane.

Sample C: Extracted with 19 mL of 10% methanol and acetic acid (up to 1%), in the presence of 1 mL of EDTA-Na_2 solution and 5 mL of dichloromethane.

Sample D: Extracted with 19 mL of 20% methanol with acetic acid (up to 1%), in the presence of 1 mL of EDTA-Na_2 and 5 mL of dichloromethane.

Sample E: Extracted with 20 mL of 20% methanol with acetic acid (up to 1%), without EDTA-Na_2, with 5 mL of dichloromethane.

Sample F: Extracted with a mixture of 20% methanol with addition of acetic acid (up to 1%), in the presence of 1 mL of EDTA-Na_2 solution, without dichloromethane.

The almost comparable results were obtained in samples C, D and F, however, the purest extract was in sample D. The presence of dichloromethane during extraction seems to reduce the solubility of Gly, but its usage is helpful for the elimination of lipids and better phase separation when working with complex objects and oilseeds. The optimal amount of acetonitrile was also selected. A 2 mL of acetonitrile/1 mL of extract demonstrated minimal losses of Gly and AMPA and allowed to precipitate proteins and peptides from the extract.

For water samples: Three experiments were made with 5 mL sample aliquots, contaminated by Gly and AMPA. In first experiment, the sample was directly applied to Oasis HLB - "A". Further sample preparation was carried out as described above, with full protocol. In second experiment, the ammonia solution was added up to 0.1% v/v, before Oasis HLB cleanup - "B". In third experiment, in 5 mL of the sample 0.05 mL of acetic acid and 0.2 mL of EDTA-Na_2 solution were added - "C". For soil samples, three experiments were made: extraction with 20 mL of 1% acetic acid - "A"; extraction with 19 mL of 1% acetic acid and 1 mL of EDTA-Na_2 solution - "B"; extraction with 20 mL of 0.1% ammonia solution - "C". The next procedure for the experiments was as described above, with full protocol. The best variant of extraction of Gly and AMPA from water and soil samples is found as "C" (Fig. 3).

Cleanup: The SPE cleanup stage was checked on the Oasis HLB cartridge and on Bondesil C_{18} sorbent (0.5 g per cartridge). As a result of the experiment, it was found that the use of an Oasis HLB cartridge allows us to obtain the purest extracts with the maximum response of Gly and AMPA. In absence of Oasis HLB cartridges, cleanup stage can also be carried out on Bondesil C_{18} sorbent. The difference between the results is negligible. Losses in case of refusal of SPE stage depend on the type of matrix, but not more than 10-12%. It should be noted that in SPE-purified extracts, the peak shape of the analytes was better and the values of the signal-to-noise (S/N) ratios for the second daughter ion were higher. In general, the use of SPE stage prolongs the chromatographic column life time and is advisable for mass-spectrometers with low-performance ion sources, as well as when analyzing such research objects as tea (all types), soil, contaminated surface water and feed based on raw materials of plant origin.

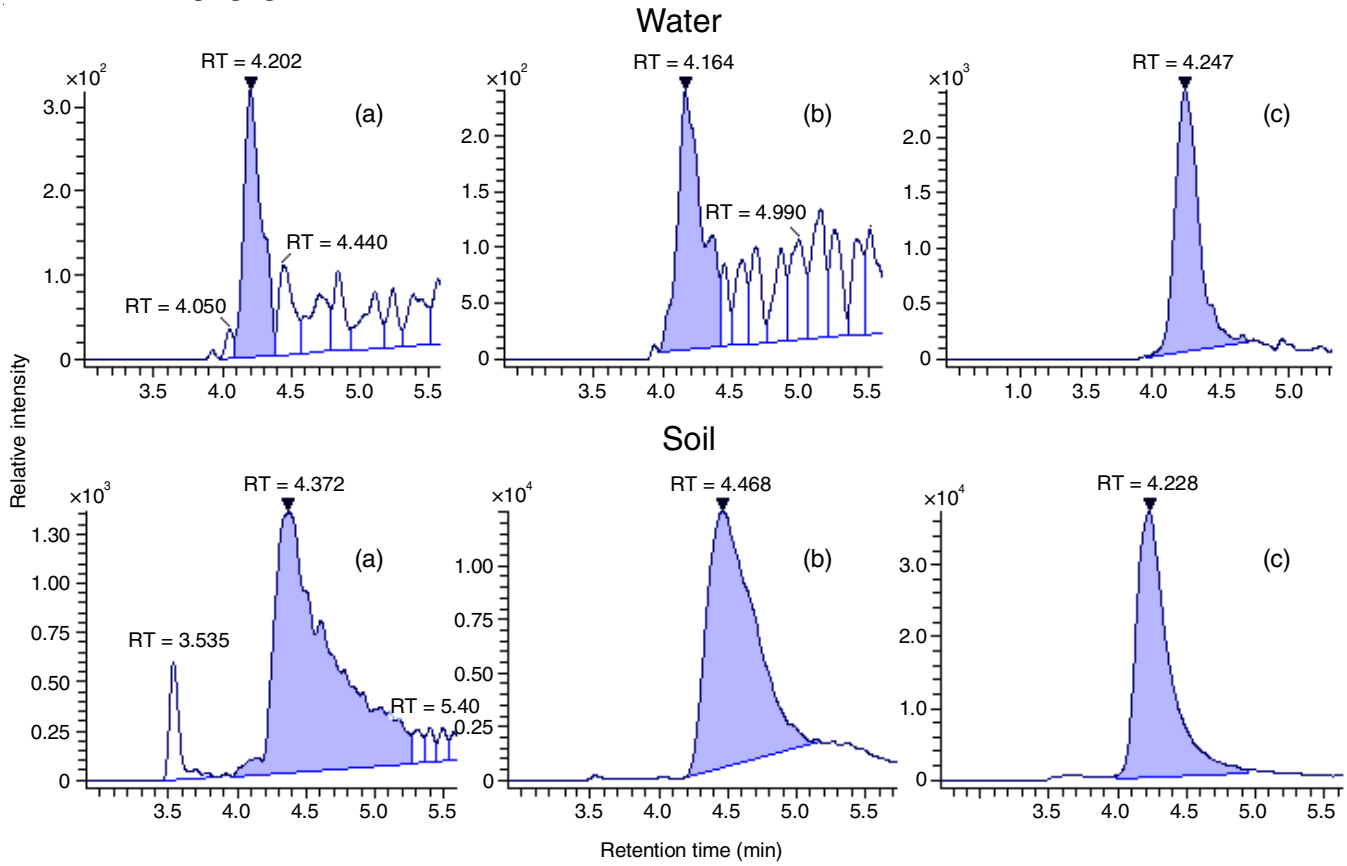

Fig. 3. Mass-chromatograms of Gly in water and soil samples with different extraction approaches

Comparison with QuPPe-PO method: For the methodologies comparison, two samples of soya beans were contaminated by Gly and AMPA at the LOQ level with following sample preparation and analysis. One of the samples (2 g) was treated as described above (HSS T3 column protocol) and the second (5 g) was treated regarding the QuPPe-PO method protocol (with slight modification). The modification consisted of the usage of EDTA-Na_2 instead of EDTA-Na_4; the usage of the 10 kDa ultrafiltration device instead of 5 kDa; the final extract was treated with acetonitrile (1/1, v/v) for extra peptides elimination. During the experiment, it was found that acetonitrile mixed with QuPPe-PO method extract gave an active formation of suspended particles (precipitation of peptides and lipids). These indicate that the analysis of the extract without an extra acetonitrile cleanup step can lead to contamination or blockage of both the valve rotor and the chromatographic column (due to precipitation of peptides inside of an analytical instrument). Even taking into account the final dilution with acetonitrile, the difference between the analytical signals is about 10 times in favour of sample preparation regarding HSS T3 column protocol (Fig. 4).

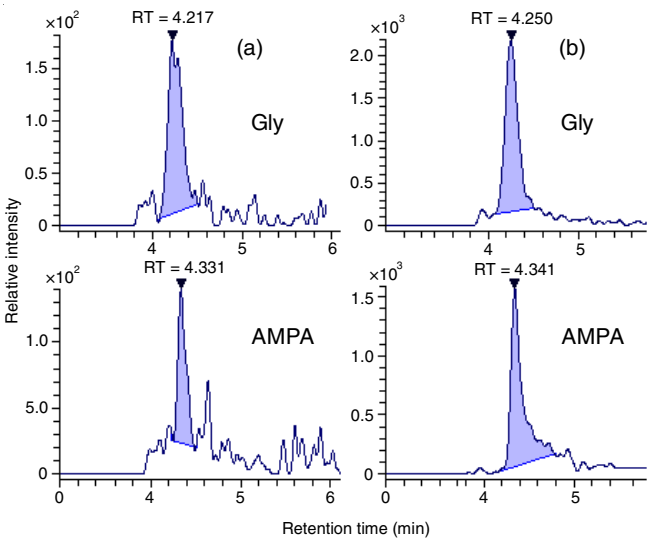

Fig. 4. Comparison of mass-chromatograms obtained with different sample preparation, (a) QuPPe-PO method with extra acetonitrile for protein precipitation; (b) complete ACQUITY UPLC HSS T3 column protocol described above

Approbation of the method: The LOQ of Gly and AMPA, written in the abstract were confirmed for the next matrices: soya beans (*Glycine max*) and soybean meal, pea seeds (*Pisum sativum*), flax seeds (*Linum usitatissimum*), oat (*Avena sativa*), rice (*Oryza sativa*), wheat (*Triticum aestivum*), barley (*Hordeum vulgare*), maize (*Zea mays*), buckwheat (*Fagopyrum esculentum*), lentils (*Lens culinaris*), amaranth seeds (*Amaranthus* spp.), niger seeds (*Guizotia abyssinica*), sesame seeds (*Sesamum indicum*), safflower seeds (*Carthamus tinctorium*), sunflower seeds (*Helianthus annuus*), chia seeds (*Salvia hispanica*), quinoa seeds (*Chenopodium quinoa*), tea (*Camellia sinensis*), pond water (taken during the period of active snowmelt), black soil and podzols soils. The method makes it possible to analyze a complex granulated animal feeds without mineral additives and raw milk (at LOQ - 0.05 mg/L both AMPA and Gly). The method is not suitable for the analysis of glufosinate or work with bottom sediments such as sapropel. The initial sample preparation protocol for raw milk is as follows: 2 mL of sample was extracted by 7 mL of extraction solution for plant materials with 1 mL of EDTA-Na_2 solution. At the half of extraction time, 1 mL of dichloromethane was added and extraction was continues. After centrifugation the Oasis HLB cleanup was applied and subsequent manipulations should be carried out as described above. The examples of some mass-chromatograms are shown in Fig. 5.

The stability of prepared extracts was tested during 5 days period with storage at 15 ºC in the autosampler. For this one soil and one soya beans sample were contaminated by Gly and AMPA at the LOQ level and sample preparation was made as described above. The final extracts were separated into two parts for storage in plastic inserts and glass vials. For soya beans extracts stored in a glass vial, the following change in the content of Gly (as a percentage of the initial value, 100 on 1st day) were: 88 (3rd day) > 79 (4th day) > 73 (5th day); and 89 (3rd day) > 83 (4th day) > 82 (5th day) for the extract stored in plastic. Almost the same was for AMPA. There is an increase of baseline was recorded for analytes in the extract stored in glass vial (more on about 10-15% in comparison with extract stored in plastic insert). The slight deterioration of chromatographic peaks shape was observed for AMPA in this sample. Taking into account the data above, the usage of plastic vials or inserts it is not mandatory for extracts included acetic acid and EDTA-Na_2 (given that analysis is performed no later than 3 days after sample preparation). For soil extracts stored in a glass vial the following change in the content of Gly, (as a percentage of the initial value, 100 on 1st day) were: 82 (3rd day) > 60 (4th day) > 31 (5th day); and 85 (3rd day) > 66 (4th day) > 45 (5th day) for the extract stored in plastic container. In the extract stored in a glass vial, an increasing deterioration in peaks shape was observed starting from the third day of storage. Significant impairments were observed for AMPA. Thus usage of plastic vials or inserts is highly recommended for extracts based on ammonia hydroxide solutions.

Pre-validation experiments were made for assessments of relative standard deviation (RSD), mean (Mean), standard deviation (SD) and recovery. For this three calibrations were made (based on soya beans, soil and water) and eight samples per level of the first point, middle and high points were analyzed. The sample preparation protocol was described above in the materials and method part. The results are shown in Table-2, which concluded that the developed sample preparation protocol is reliable enough and the methodology based on it can be recommended for full validation and future use in routine laboratory practice.

Conclusion

A sensitive method was developed for direct glyphosate (Gly) and aminomethylphosphonic acid (AMPA) determination in agricultural products, soil, surface and groundwater. The method is economical and fast in comparison with derivatization based methods and not dramatically more complex

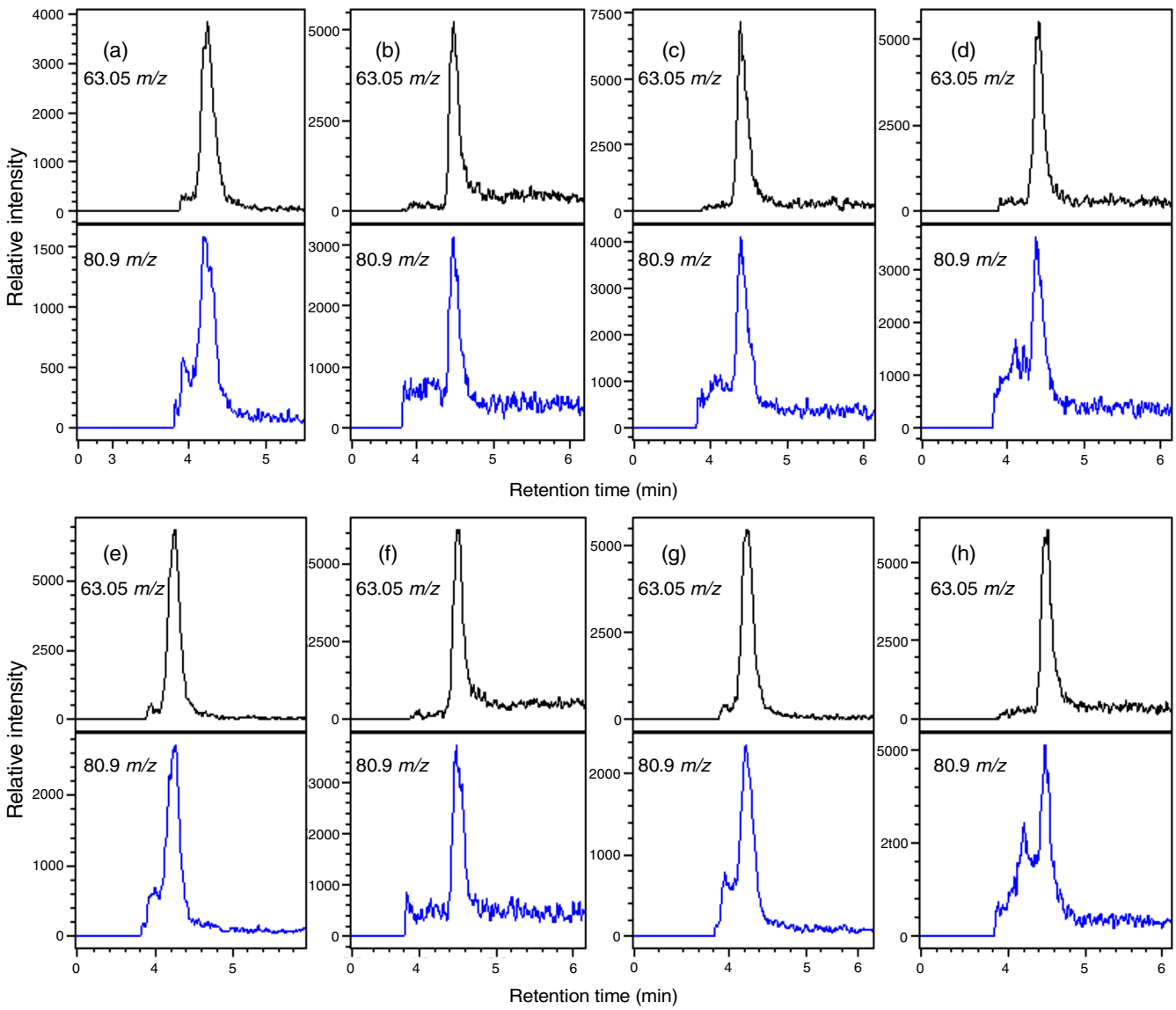

Fig. 5. Examples of Gly mass-chromatograms obtained at LOQ in various matrices, (a) barley; (b) flax seeds; (c) sunflower seeds; (d) sesame seeds; (e) rice; (f) milk; (g) wheat; (h) amaranth seeds

TABLE-2
PRE-VALIDATION EXPERIMENT RESULTS (n = 8)

Soya beans									
Gly (mg/kg)	Mean (mg/kg)	SD (mg/kg)	RSD (%)	Recovery (%)	AMPA (mg/kg)	Mean (mg/kg)	SD (mg/kg)	RSD (%)	Recovery (%)
0.1	0.106	0.01162	10.96	106	0.1	0.080	0.00602	7.48	80.5
1.0	1.010	0.02663	2.63	101.1	1.0	1.006	0.04077	4.05	100.6
5.0	5.092	0.12209	2.39	101.8	5.0	5.168	0.09184	1.77	103.3
Soil									
Gly (mg/kg)	Mean (mg/kg)	SD (mg/kg)	RSD (%)	Recovery (%)	AMPA (mg/kg)	Mean (mg/kg)	SD (mg/kg)	RSD (%)	Recovery (%)
0.02	0.0196	0.00323	1.49	98	0.04	0.038	0.00755	19.49	96.8
0.2	0.199	0.02045	10.28	99.5	0.2	0.203	0.03305	16.27	101.5
0.8	0.782	0.06453	8.24	97.8	0.8	0.865	0.06211	7.17	108.1
Water									
Gly (mg/kg)	Mean (mg/kg)	SD (mg/kg)	RSD (%)	Recovery (%)	AMPA (mg/kg)	Mean (mg/kg)	SD (mg/kg)	RSD (%)	Recovery (%)
0.001	0.00094	0.000113	12.12	93.5	0.002	0.0021	0.00034	16.09	105
0.01	0.01018	0.001124	11.05	101.7	0.01	0.0096	0.00134	13.87	96.7
0.05	0.05113	0.003758	7.35	102.2	0.05	0.0511	0.00536	10.47	102.2

than the original QuPPe-PO method sample preparation protocol. The pre-validation results exhibit good recoveries of analytes within the range of 80-108% and RSD below 20% for intraday repeatability. An equation of calibration dependences based on soya beans, soil and water were linear, with the correlation coefficient values (R) ≥ 0.99. The practical LOQ of Gly and AMPA was set at 0.1 mg/kg for raw materials of plant origin (linearity 0.1-5 mg/kg); 0.001 mg/L for Gly in water, AMPA-0.002 mg/L (linearity 0.001/0.002-0.05 mg/L); 0.02 mg/kg for Gly in soil, AMPA-0.04 mg/kg (linearity 0.02/0.04-0.8 mg/kg). The study results can act as an addition to the QuPPe-PO method in terms of usage HSS T3 chromatographic column.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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