



Data Article

Dataset of compounds in glyphosate-free herbicides

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ABSTRACT

We analysed 14 recently marketed pesticides for consumers, available in France, Germany, and Poland. They were supposed to be glyphosate-free herbicides; glyphosate was banned for sale to the public in 2019. Measurements of 36 metals, 16 polycyclic aromatic hydrocarbons, 6 essential minerals, and glyphosate plus aminomethyl phosphonic acid, were performed in a laboratory accredited for regulatory purposes. The technologies used were respectively inductively coupled plasma mass spectrometry, gas-chromatography coupled with mass spectrometry, and high-performance liquid chromatography. These data can be used by scientists, the public, and regulatory bodies.

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Specifications Table

| | |
|--------------------------------|--|
| Subject | Agricultural sciences; biological sciences, environmental science, pollution, health toxicology and mutagenesis, health and medical sciences, public health and health policy |
| Specific subject area | Study of the undeclared composition of formulants of new glyphosate-free herbicides, especially by mass spectrometry |
| Type of data | 2 Tables in Supplementary material: 1 – Detailed description of samples analysed. Table 1 is adapted from Seralini and Jungers [3], with the addition of 3 columns, detailing the providers of the samples, the lot numbers, and the dates of production. 2 – Detailed description of the quantification of metals, salts, polycyclic aromatic hydrocarbons, and glyphosate and its metabolite in the same samples. |
| How data were acquired | The technologies used were respectively inductively coupled plasma mass spectrometry (ICP-MS), gas-chromatography coupled with mass spectrometry (GC-MS), and high-performance liquid chromatography (HPLC) with further fluorescence detection. |
| Data format | Raw Analysed |
| Parameters for data collection | The samples were collected in supermarkets, stored at room temperature as indicated, and run in the machines according to the protocols described above. |
| Description of data collection | The raw data were collected through the technologies indicated and classified by date when a replication of the measure was performed (in these cases the arithmetic means were indicated as M). The values were specified only when they were above the limits of detection and quantification. In other cases, samples scoring positive but below the level of quantification were recorded as above the level of detection. The samples A-N are described in Table 1. The units and products measured are in Table 2. |
| Data source location | Institution: University of Caen Normandy City/Town/Region: Caen, Normandy Country: France |
| Data accessibility | With the article Repository name: Mendeley Data Direct URL to data: http://dx.doi.org/10.17632/jsy2zsktcf.1 |
| Related research article | G.E. Seralini, G. Jungers [3] Toxic compounds in herbicides without glyphosate, Food and Chemical Toxicology. https://doi.org/10.1016/j.fct.2020.111770 |

Value of the Data

- Today, only the declared active principle of a pesticide is available on the labels of pesticide containers, or through the manufacturer's information. However, this declared chemical represents in the products analysed in this study only between 1.25 and 71.7% of the final composition, depending on the pesticide (Table 1). There are many other compounds in the formulations, which are always mixtures. Their exact compositions are not known to the scientific community. However, we have previously demonstrated that these can be more toxic than the declared active principles [1,2]. These newly available mixtures are thus analysed for some toxic compounds, essentially petroleum residues, and heavy metals. We also checked for the presence of glyphosate and its main metabolite aminomethylphosphonic acid (AMPA), in order to better understand the process of synthesis of these new herbicides, declared as glyphosate-free. We did not perform an exhaustive analysis of all possible chemicals contained in these pesticides, which are generally undeclared. It is useful and important to access these data for risk assessment.
- Regulatory bodies, risk assessors, scientists in this area of research, and public for general knowledge may use these data.
- These data might be used/reused for further insights and/or development of experiments, to compare different batches of the same pesticide formulation and to compare one pesticide formulation with other products, as well as to check if certain compounds were declared in market authorizations, for legal purposes.

1. Data Description

Table 1 corresponds to the description of the herbicides studied in this work A-N, with 8 columns including the herbicide brand name, the nature of the declared active principle, its percentage in the formulation, the authorization number for marketing, the company marketing the product, the provider and the batch authorization number, and the date of production.

Table 2 presents the raw data for all the elements measured in the samples described in Table 1. Metals measured are indicated in the first column, beginning with arsenic and mercury, because they are measured in $\mu\text{g/L}$, while all the others are in mg/L . Then a line corresponds to the total for metals found in each sample per mg/L . The 6 life salts or essential minerals in mg/L (copper is in metals) are indicated, followed also by their total. Finally, polycyclic aromatic hydrocarbons (PAHs in $\mu\text{g/L}$) are indicated, finishing with their total per sample. The column finishes with the detection of undeclared molecules of the pesticides glyphosate and AMPA in $\mu\text{g/L}$, followed by the total. On the right, all the results are indicated per sample, according to the date of measurement or by batch number. Means (M) are in highlighted in blue.

Both tables include their legends.

2. Experimental Design, Materials and Methods

To ensure the accuracy and reproducibility of the data, for adequate replications, standard deviations and coefficients of variations, all measurements were performed in laboratories accredited by COFRAC, the French accreditation body.

2.1. ICP-MS for essential minerals and metals

ICP-MS was used according to the norm NF EN ISO 17294-2. The sample is first mineralized, then nebulized. Regarding equipment, the 7700 Series for ICP-MS (Agilent Technologies) can be used. For nebulization, a modified Lichte with a cyclonic chamber can be used. For essential minerals, calcium, magnesium, phosphorus, potassium, and sodium were detected; for metals or metalloids, other minerals, aluminum, antimony, arsenic, barium, beryllium, bismuth, cadmium, chromium, cobalt, copper, iron, gallium, germanium, hafnium, indium, lithium, lead, manganese, molybdenum, nickel, niobium, palladium, rhenium, selenium, silicon, silver, strontium, tantalum, tellurium, thallium, tin, titanium, tungsten, vanadium, zinc, and zirconium were assayed. The MO176 ICPAES method was also used. Mineralization of water was adapted from the norm NF EN ISO 15587-2; the mineralization was performed by evaporation with addition of nitric acid. This part of ISO 15587 specifies a method for extracting trace elements from a water sample, using nitric acid as a digestion agent. The method is applicable to all types of water with a suspended solids concentration of less than 20 g/l and a total organic carbon (TOC) concentration, expressed as carbon, of less than 5 g/l. The analysis of a blank and the calibration range were accomplished; this was controlled by an independent standard with the acceptance criterion range of $\pm 10\%$. Other independent standards were analyzed every 10-15 samples with an acceptance criterion of $\pm 20\%$; at the end of the measurements, low-end standards were analyzed. The aerosol in plasma after nebulization allows an ionization of atoms. Arsenic was measured with the adapted method MO309 or adapted MO309-NF EN ISO 17294-2.

2.2. GC-MS for PAHs

The international normalized method DIN 38407-39 was applied for PAHs measures. First, PAHs in water were extracted with cyclohexane. The extract was concentrated by evaporation. The PAHs were then separated by gas chromatography (GC) on capillary

columns with suitable stationary separation phases and identified and quantified by MS. The products assayed were acenaphthene, acenaphthylene, anthracene, benzo(A)anthracene, benzo(A)pyrene, benzo(B)fluoranthene, benzo(G,H,I)perylene, benzo(K)fluoranthene, chrysene, dibenzo(A,H)anthracene, fluoranthene, fluorene, indeno(1,2,3-C,D)pyrene, naphthalene, phenanthrene, and pyrene.

In detail, after adding a mixture of isotope-labelled standards to the samples, the extracts obtained by liquid/liquid extraction were dried with anhydrous sodium sulphate and concentrated prior to analysis by gas chromatography with mass spectrometer (GC/MS). Chromatographic separation was performed on a Varian VF-Xms column. Quantification was done by isotope dilution.

2.3. HPLC and fluorescence detections for glyphosate and AMPA

HPLC followed by final stack derivatization and fluorescence detection (F22, all method DIN 38407-22) was performed for G and its main metabolite aminomethyl phosphonic acid (AMPA). In principle, the sample is acidified and passed through a cation exchanger to obtain an eluate with higher concentrations of glyphosate and AMPA. After acidification with hydrochloric acid, the eluate is purified by filtering it through an anion exchanger. The filtrate is evaporated to dryness and the residue is taken up in a buffer solution for the HPLC analysis. Glyphosate and AMPA are separated by isocratic HPLC using a cation exchanger and immediately subjected to a two-stage postcolumn derivatization in which glyphosate is converted to glycine by oxidation with sodium hypochlorite in the first step.

Ethics Statement

Data are presented accurately and made publicly available for this research from a public institution.

Credit Author Statement

Professor Seralini was the main investigator and supervised the work; Gerald Jungers helped with technical assistance and in making the tables.

Declaration of Competing Interest

The authors declare that they have no competing financial interests, patents, or personal relationships that could have influenced the work reported in this paper.

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Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:[10.1016/j.dib.2020.106564](https://doi.org/10.1016/j.dib.2020.106564).

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