

A potentially underestimated source of CO₂ and other greenhouse gases in agriculture

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SUMMARY

Agriculture is a well-known global contributor to greenhouse gas (GHG) emissions responsible for the global rise in temperatures. A substantial amount of agricultural GHG emissions is linked to the application of synthetic fertilizers. Studies in this field have addressed the related microbiological processes, leaving the purely chemical ones out of focus. Our hypothesis was that some common mineral fertilizers produce environmentally significant GHG effluxes chemically—because of their composition, large quantities of application, and specific conditions frequently present in soil. Therefore, the purpose of our work was to test whether Nitroammofoska (a commercial nitrogen-phosphorus-potassium fertilizer) and other common and readily available mineral fertilizers produce considerable CO₂ effluxes from chemical reactions in water with and without participation of CaCO₃. Based on our results, as much as 20.41% of all CO₂ annually emitted from all activities on land could be produced by (NH₄)₂SO₄ alone, provided all mineral nitrogen fertilizers are used as a mixture of (NH₄)₂SO₄ and K₂HPO₄, and in the presence of CaCO₃. The results also suggested a possibility of considerable economic loss due to intense NH₃ volatilization from ammonium-containing mineral fertilizers, occurring when enough carbonate and/or bicarbonate ions are available.

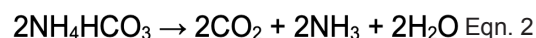
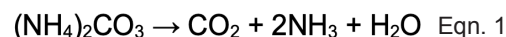
INTRODUCTION

Greenhouse gases (GHGs) are responsible for regulating the atmosphere's temperature and the increase in their quantities has been shown to cause global warming (1, 2). However, the current increases in GHGs, such as carbon dioxide (CO₂), nitrous oxide (N₂O), and methane (CH₄), are taking place at an unprecedented rate in human history. This threatens to overwhelm the environment's mechanisms for GHG sequestration, resulting in severe consequences, some of which are already present: rapid melting of icebergs, greater salinity contrast in different water regions, more extreme temperature fluctuations, unfavorable precipitation changes in different parts of the globe, etc. (2).

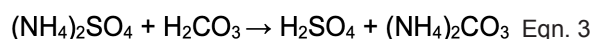
One explanation for this increased rate of change is the contribution of anthropic GHG emissions. A part of this contribution comes from agriculture, which is a well-known global source of GHG emissions and is considered among

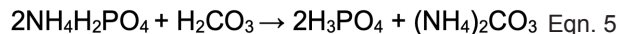
the most significant contributors (3, 4). Within agriculture, fertilizers are responsible for most of these emissions. For instance, most of the emitted agricultural N₂O is derived from synthetic nitrogen (N) fertilizers (5). This emission can take place either via nitrifier denitrification or urea hydrolysis. Nitrifier denitrification is the microbiological process by which some soil autotrophic bacteria convert ammonium cations from mineral N fertilizers into various other forms of N including N₂O (3). Urea hydrolysis is the microbiological process that involves the decomposition of urea (CO(NH₂)₂) into less stable chemical compounds (6). From these destabilized compounds, ammonia (NH₃) is volatilized and subsequently transformed into N₂O (6). Additionally, synthetic N fertilizers, when applied at excessively high rates, may stimulate microbiological mineralization of soil organic matter – the largest stock of organic carbon in terrestrial ecosystems (7, 8). Mineralization of even small portions (less than 1%) of this global stock can produce CO₂ emissions comparable to all anthropogenic CO₂ sources taken together (9).

While these and other microbiologically driven processes attract considerable attention, GHG emissions derived from purely chemical reactions are quite realistic too. The chemical decomposition of the unstable ammonium carbonate ((NH₄)₂CO₃, Eqn. 1) and ammonium bicarbonate (NH₄HCO₃, Eqn. 2) is evidence for that possibility.



Thus, according to the experiments of Song *et al.*, the soil samples with NH₄CO₃ retained significantly fewer ammonia ions than other samples with more stable salts such as NH₄SO₄ (10). Under certain conditions frequently present in soil (e.g., the presence of ammonium ions from various N fertilizers, and of carbonate/bicarbonate ions from rainwater, bedrock material, and/or soil liming) the unstable NH₄CO₃ and NH₄HCO₃ compounds can appear (e.g., Eqn. 3-5) in large quantities even when they are not applied directly as fertilizers. The possibility of considerable GHG emission from these chemical reactions is, therefore, certain.





Although the possibility of GHG emissions resulting from chemical reactions in soil is mentioned in some published research papers (10), so far, no effort has been made to estimate the potential contribution of these reactions to the global warming problem. Therefore, the purpose of our work was to start filling this gap. We hypothesized that NH_4 -containing synthetic fertilizers, used alone or in combination with other common mineral fertilizers, produce environmentally significant GHG effluxes chemically – because of their composition, large quantities of application and because of the frequent presence of carbonates/bicarbonates in soil. To test this hypothesis, Nitroammofoska, a readily available commercial fertilizer containing $\text{NH}_4\text{H}_2\text{PO}_4$, NH_4NO_3 , and KCl, and different chemical compounds that are frequently used as mineral fertilizers (K_2HPO_4 , $(\text{NH}_4)_2\text{SO}_4$, and the mixture of $(\text{NH}_4)_2\text{SO}_4$ and K_2HPO_4) were added to distilled water with and without CaCO_3 . Each sample was monitored periodically for CO_2 efflux. The results demonstrated that some of the tested compounds did produce CO_2 in environmentally significant quantities. Potentially, an equivalent of up to 11.48-20.41% of all CO_2 annually emitted from all activities on land in 2013/14 could have been produced by $(\text{NH}_4)_2\text{SO}_4$ alone, provided all nitrogen-phosphorus-potassium (NPK) fertilizers used during that time contained a mixture of $(\text{NH}_4)_2\text{SO}_4$ and K_2HPO_4 , and the soils they were applied to had CaCO_3 (11). The studied processes also could cause economic losses if the high degree of NH_3 volatilization results in reduced efficiency of mineral NPK fertilizers. Our results demonstrate the need for further research into the related environmental and economic threats and the possibilities of avoiding these threats.

RESULTS

To test whether mineral fertilizers could produce substantial efflux of CO_2 while in water and potentially exposed to CaCO_3 , we added (separately) Nitroammofoska, K_2HPO_4 , $(\text{NH}_4)_2\text{SO}_4$,

and a 1:1 mixture of K_2HPO_4 and $(\text{NH}_4)_2\text{SO}_4$ into flasks with distilled water, with and without CaCO_3 , and then periodically monitored the CO_2 efflux. One flask with distilled water, and one with CaCO_3 in distilled water were used as controls. The changes in the CO_2 concentration in the closed chamber (flask + tubing + analyzer) were measured by an infrared gas analyzer and the obtained data were used to calculate the CO_2 emission rates.

In most experimental conditions, the experimental data were considerably different from the control conditions (see **Figure 1**). Depending on the condition, both CO_2 emission and fixing (i.e., negative emission or decrease of the CO_2 concentration in the chamber air) were observed. The controls, K_2HPO_4 (with and without CaCO_3) and the mixture (without CaCO_3) variants were relatively active in fixing CO_2 from the air in the beginning of the experiment, but this activity tended to decrease or even reverse with time. For example, K_2HPO_4 , both with and without CaCO_3 , most actively fixed CO_2 within the first two days of the experiment, and then, by day six, the process reversed, although the emission rates were relatively low. According to our estimations, and assuming that there were no unregistered peaks in CO_2 emission/fixing rates in between measurements, by the end of the experiment, the overall CO_2 balance became positive for $\text{K}_2\text{HPO}_4 + \text{CaCO}_3$ with 809.07 μg of total carbon emitted as CO_2 (C-CO_2) and remained negative for K_2HPO_4 with 228.40 μg of total C-CO_2 fixed (**Figure 2**). Although K_2HPO_4 with and without $(\text{NH}_4)_2\text{SO}_4$ did not manifest the potential to produce substantial CO_2 efflux, it did significantly slow down the ability of the distilled water to fix CO_2 from the air, as well as considerably increase the CO_2 efflux when used with $(\text{NH}_4)_2\text{SO}_4$ and CaCO_3 (**Figures 1-2**). Thus, the estimated total CO_2 emitted by the mixture (of K_2HPO_4 and $(\text{NH}_4)_2\text{SO}_4$) + CaCO_3 exceeded the total CO_2 emitted by $(\text{NH}_4)_2\text{SO}_4 + \text{CaCO}_3$ by 4.43 times, even though the former variant had two times less of $(\text{NH}_4)_2\text{SO}_4$ than the latter one. The chemical composition of the tested NPK variants also had a significant impact on the rate and

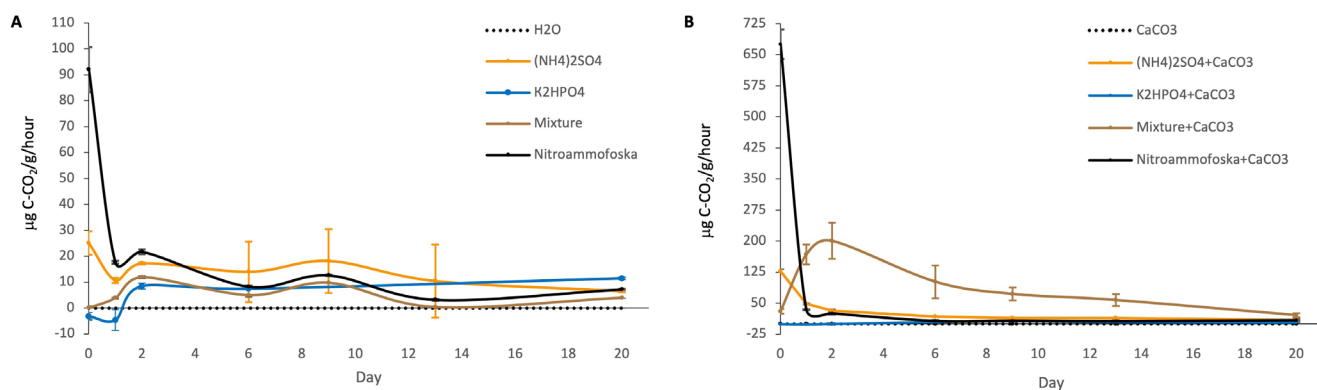


Figure 1: The CO_2 emission rates from different fertilizer variants on different days. Emission rates were normalized to the controls with H_2O (A) and with CaCO_3 (B). We tested each type of fertilizer in flasks with distilled water with and without CaCO_3 . The values represent averages for three replicates. The emission rates for all variants are given in μg of C emitted in the form of CO_2 per hour ($\mu\text{g C-CO}_2/\text{hour}$) from 1 g of the tested substance(s) or, as for the H_2O control, from 30 mL of water. The error bars represent the confidence intervals at $p = 0.05$ and show the presence or absence of statistically significant differences between the measurements.

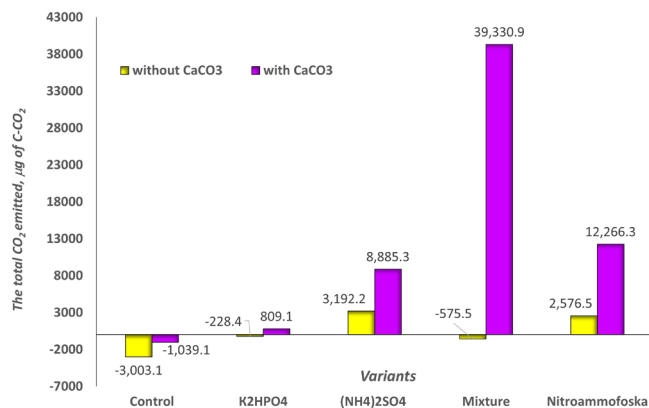


Figure 2: Total CO₂ emitted by different fertilizer variants. Estimated total CO₂ in µg of C in the form of CO₂ (C-CO₂) emitted from the following variants: K₂HPO₄, (NH₄)₂SO₄, the 1:1 mixture of K₂HPO₄ and (NH₄)₂SO₄, and Nitroammofoska. We added each type of fertilizer into flasks with distilled water with and without CaCO₃. To calculate the total C-CO₂ we summed the quantities of C-CO₂ emitted within each variant in between each pair of consecutive measurements throughout the experiment. To estimate C-CO₂ emitted between any two consecutive measurements we multiplied the hours between these measurements by their average CO₂ emission rate. We incubated the variants for 20 days in the dark at room temperature with periodical monitoring of the CO₂ efflux by Li-850 infrared gas analyzer. The tests included three replicates, with only one replicate for each of the controls. See Figure 1 for the data on statistical significance of the CO₂ emission rates measured in different fertilizer variants.

timing of the CO₂ efflux: in the cases of Nitroammofoska and Nitroammofoska+CaCO₃ the bulk of CO₂ emissions was observed in the beginning of the experiment while Mixture+CaCO₃ peaked 2 days later and had much higher values for the total emitted CO₂ (Table 1, Figures 1-2).

There were five fertilizer variants that had positive CO₂ emission rates throughout the entire experiment: (NH₄)₂SO₄, (NH₄)₂SO₄+CaCO₃, Mixture+CaCO₃, Nitroammofoska,

Nitroammofoska+CaCO₃. In all the variants listed, except for Mixture+CaCO₃ the measured emission was the highest immediately after the start of the experiment and then it gradually decreased (Mixture+CaCO₃ peaked two days later). The highest CO₂ emission rates and the highest estimated total CO₂ emitted were observed in the Mixture+CaCO₃ and Nitroammofoska+CaCO₃ variants, with 195.85 and 660.34 µg C-CO₂/g/hour, 39330.88 and 12266.32 µg C-CO₂, respectively (Table 1, Figure 2).

All the variants with constant CO₂ emissions ((NH₄)₂SO₄, (NH₄)₂SO₄+CaCO₃, the mixture+CaCO₃, Nitroammofoska, Nitroammofoska+CaCO₃) included ammonium-containing compounds, and throughout the experiment, the distinct ammonia smell was detectable from the corresponding flasks. Among these variants, the emission was higher in the presence of CaCO₃. Thus, the peak CO₂ emission rates for Nitroammofoska+CaCO₃ and (NH₄)₂SO₄+CaCO₃ was 8.30 and 8.92 times higher than those of Nitroammofoska and (NH₄)₂SO₄, and the estimated total emissions were 4.76 and 2.78 times higher, respectively. In the case of the mixture, the impact was even more radical: the Mixture variant was fixing CO₂ in the absence of CaCO₃, and Mixture+CaCO₃ had the second maximal emission rate and the biggest total CO₂ emitted of all variants (Figure 2).

DISCUSSION

The results demonstrated that commercial mineral fertilizers and chemical compounds that are frequently used as such can indeed produce substantial effluxes of CO₂ while/after dissolving in distilled water, especially when exposed to CaCO₃ – a compound frequently present in or introduced into soil. Thus, the estimated total CO₂ emitted by the most active variant (Mixture+CaCO₃) implies that at least 371.02 g of carbon in the form of CO₂ (C-CO₂) can be released into the atmosphere per every 1 kg of N fertilizer

Fertilizer type \ Day	0	1	2	6	9	13	20
Control 1 (H ₂ O)	-12,45	-8,63	-14,79	-5.60	-9.61	-0.21	-5.39
Control 2 (CaCO ₃)	-14,52	-5,60	-4,79	0.26	-2.46	-1.83	-0.20
K ₂ HPO ₄	-15,73	-13,32	-6,35	1.74	1.77	2.27	0.78
(NH ₄) ₂ SO ₄	12,60	1,96	2,42	8.33	8.52	10.16	1.09
Mixture	-12,43	-4,69	-2,89	-0.63	0.18	0.13	-1.42
Nitroammofoska	79,56	8,94	6,86	2.59	2.92	2.94	1.74
K ₂ HPO ₄ +CaCO ₃	-15,09	-7,14	-4,72	4.9	4.74	2.67	3.32
(NH ₄) ₂ SO ₄ +CaCO ₃	112,35	42,78	26,89	18.01	11.41	12.30	9.54
Mixture+CaCO ₃	15,94	162,0	195,85	102.07	69.70	55.63	20.58
Nitroammofoska+CaCO ₃	660,34	28,75	20,57	7.44	5.17	4.75	8.73

Table 1: The CO₂ emission rates from different fertilizer variants on different days. CO₂ emission rates measured on different days of the experiment for the following fertilizer variants: K₂HPO₄, (NH₄)₂SO₄, the 1:1 mixture of K₂HPO₄+(NH₄)₂SO₄, and Nitroammofoska. We tested each type of fertilizer in the flasks with distilled water with and without CaCO₃. Values represent averages for three replicates. The emission rates for all variants show µg of C emitted in the form of CO₂ per hour (µg C-CO₂/hour) from 1 g of the tested substance(s), or for Control 1 – µg C-CO₂/hour per 30 mL of water, and for Control 2 – µg C-CO₂/hour per 1 g of CaCO₃. See Figure 1 for the statistical significance of the measured emission rates.

applied. This is a considerable number since it suggests that as much as 40.96 teragrams (Tg) of C-CO₂ could have been emitted in this way in 2013-2014, if all 110.40 Tg/year of nitrogen fertilizers applied had contained this same mixture of K₂HPO₄ and (NH₄)₂SO₄, and the soils they were applied to had contained CaCO₃ (11). Keeping in mind that 40.96 Tg of C-CO₂ make 2.56% of all C-CO₂ that was emitted annually from all deliberate human activities on land in the corresponding period (1600.00 Tg of C-CO₂/year), the potential for significant impacts on global warming is evident (12). Moreover, the scale of this impact may increase by many times if the factor of NH₃ volatilization is considered. Judging by the fact that all the variants with high CO₂ emission included ammonium containing fertilizers, that the emissions were considerably stimulated in the presence of CaCO₃, and that a distinct ammonia smell was detectable in each case, it is very plausible that the registered CO₂ efflux resulted from decomposition of unstable (NH₄)₂CO₃ and (NH₄)HCO₃, produced by chemical reactions between the tested (NH₄)₂SO₄/(NH₄)₂HPO₄ and CaCO₃/Ca(HCO₃)₂/H₂CO₃ present in the flask water. If so, depending on the proportion of the produced NH₄HCO₃ and (NH₄)₂CO₃, every unit of emitted C-CO₂ was accompanied by 1.17-2.33 units of emitted N in the form of NH₃ (N-NH₃). Since one unit of volatilized N-NH₃ yields on average 0.01 units of N-N₂O in the atmosphere, 39.33 mg of C-CO₂ produced in the most active variant could well be accompanied by 46.02-91.64 mg of emitted N-NH₃ that could eventually result in 0.46-0.92 mg of N-N₂O. N₂O is 265-298 times more active than CO₂ in heat-trapping (3), meaning that this amount of N₂O would have a heat-trapping effect comparable to that of 137.13-273.09 mg of C-CO₂ and would raise the total amount of GHGs emitted in the most active variant to the level equivalent to 176.46-312.42 mg of C-CO₂. Thus, in 2013-2014 up to 11.48-20.41% of all CO₂ annually emitted from all activities on land could have been produced by (NH₄)₂SO₄ alone, again assuming that all mineral N fertilizers at that time had been used in a mixture of (NH₄)₂SO₄ and K₂HPO₄ in the presence of CaCO₃.

If our suggestion is correct and the measured CO₂ efflux in the most active cases was caused by appearance and decomposition of unstable NH₄HCO₃ and (NH₄)₂CO₃, then simple calculations show that 43.29-86.57% of total N was lost in the Mixture+CaCO₃ variant, depending on the proportion of the produced NH₄HCO₃ and (NH₄)₂CO₃. Thus, besides demonstrating that synthetic NPK fertilizers can produce an environmentally significant amount of GHGs purely from chemical reactions, the presented data also point at a possibility of economic losses if the high degree of NH₃ volatilization results in reduced efficiency of mineral NPK fertilizers.

Our results also indicated possibilities of avoiding or decreasing the potential environmental damage and economic losses from GHG emission and ammonia volatilization under the studied experimental conditions. The tested N fertilizers produced a substantially smaller, sometimes even negative,

CO₂ efflux in the absence of CaCO₃ as well as when tested without K₂HPO₄. For example, the mixture that was most active in producing GHGs in the presence of CaCO₃ (the Mixture+CaCO₃ variant) had a negative total CO₂ efflux when tested without CaCO₃ (-575.49 µg of C-CO₂, **Figure 2**). Also, the same ammonium containing compound ((NH₄)₂SO₄) that produced substantial amounts of CO₂ in the Mixture+CaCO₃ variant, was 4.43 times less active when used without K₂HPO₄ in the (NH₄)₂SO₄+CaCO₃ variant, and 12.32 times less active when used by itself and without CaCO₃ (the (NH₄)₂SO₄ variant, **Figure 2**). Even simple steps like avoiding ammonium-containing mineral fertilizers in soils with high carbonate/bicarbonate content, avoiding simultaneous introduction of the ammonium-based compounds with other mineral fertilizers, or using soil liming can either prevent or radically lessen the negative effects. Also, another possibility for damage prevention could be to substitute the ammonium-based N fertilizers with those based on nitrates. However, additional studies are needed to make sure increases in N₂O effluxes from denitrification and other possible factors do not pose comparable or even greater threats.

Further research is needed to verify our estimations and to clarify the suggested mechanisms. For example, it is possible that in the most active cases of CO₂ efflux, the real emission rate peaks were not immediately after the initiation of the experiments as we assumed but somewhere in between the first two measurements, and that considerably more CO₂ was eliminated between the first two measurements. Also, the suggestion that the CO₂ emissions were coming from the decomposition of (NH₄)₂CO₃ and (NH₄)HCO₃ may not be sufficient to explain the differences in emission rates between the variants that had the same compound of (NH₄)₂SO₄ or (NH₄)₂SO₄+CaCO₃, but differed by the presence/absence of K₂HPO₄. This may also not explain the delay in the peak emission rate in the mixture+CaCO₃ variant as compared to Nitroammofoska+CaCO₃ and the others. Testing the rest of the commonly used mineral fertilizers (and their combinations) and/or monitoring effluxes of the other GHGs may add significant new information regarding the effects of NPKs on total GHG emissions. Finally, it remains to be verified whether the obtained results and estimations can be extrapolated to soil conditions. For example, the total balance of GHGs can be substantially affected by plant growth-related CO₂ fixing (via photosynthesis), that will be stimulated by fertilization. Still, the presented material is sufficient to conclude that the tested potential source of GHGs is real and can be significant in matters of climate change, and that further research into this problem is necessary.

MATERIALS AND METHODS

The commercial fertilizer tested in this study was Nitroammofoska (NH₄H₂PO₄, NH₄NO₃ and KCl), containing N, P₂O₅, and K₂O at 16% each. Besides Nitroammofoska, we also tested K₂HPO₄, (NH₄)₂SO₄, and a 1:1 mixture of both. One gram of each compound or the mixture was added into

100 mL-flasks with 30 mL of distilled water with or without 1 g of CaCO₃ and incubated for 20 days in the dark at room temperature with periodical monitoring of the CO₂ efflux. One flask with 30 mL of distilled water and one with 1 g of CaCO₃ in 30 mL of distilled water were used as Control 1 and Control 2, respectively. The CO₂ efflux in each flask was measured by using the closed dynamic chamber methodology with the help of Li-850 infrared gas analyzer (LI-COR). We measured the rate of increase of the CO₂ concentration in the chamber air, which included the air in the flask, tubing, and the infrared gas analyzer. The obtained data were converted into the emission rate expressed in µg of C-CO₂ from 1 g of the tested substance(s) (or from 30 mL of distilled water in the case of Control 1) per hour. The CO₂ emission rate was calculated according to the following formula (13):

$$R_s = 3600 \cdot \Delta CO_2 \cdot M_C \cdot P / P_i \cdot V / V_i \cdot T_i / T \cdot W_s$$

where R_s is the CO₂ emission rate, 3600 = the number of seconds in 1 hour, ΔCO_2 is the change in CO₂ concentration (micromoles of CO₂ in 1 mole of air) per second in the chamber air, M_C = 12.01 g (the molar mass of C), P = pressure (kPa), P_i = 101.325 kPa (standard pressure), V = air volume (L) in the chamber, V_i = 22.41 L (volume of a mole of air at 0°C), T_i = 273.15 K (standard temperature), T = temperature (K), and W_s = mass (g) of the tested compound(s).

The estimated total CO₂ emitted in different variants was obtained by summing the quantities of CO₂ emitted in between each pair of consecutive measurements. The estimated CO₂ emitted between any two consecutive measurements was calculated by multiplying the hours between these measurements by their average CO₂ emission rate. All tests were carried out in 3 replicates. Based on the replicates, average CO₂ effluxes and their confidence intervals at $p = 0.05$ were calculated for each condition. Overlapping confidence intervals indicated absence of statistically significant differences between average CO₂ effluxes. To obtain the confidence intervals we used the Excel for Microsoft 365 function CONFIDENCE(alpha, standard_dev, size), where alpha is the significance level (0.05), standard_dev is the population standard deviation as calculated using the Excel function STDEV(number1, number 2,...), and size is the sample size.

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