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# Nitrogen, phosphorus, potassium, calcium and magnesium release from two compressed fertilizers: column experiments

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wetting was completed, the soils were weighed to determine the water content at saturation. Then, distilled water started to flow through the columns by gravity. The flow rate and the pH and electrical conductivity of the leachates were measured for 18 days. By this time, the electrical conductivity was stabilized at around  $9 \mu\text{S cm}^{-1}$ , and one fertilizer tablet was placed in each column (excepting controls), introduced in the upper part of the soil (within the first centimeters). The water flow was resumed and, on average, six water samples were collected daily from each column for 15 days, preserving it at  $4^\circ\text{C}$ . pH and electrical conductivity were measured in freshly collected samples; when values for these parameters were very similar in successive samples, the sampling frequency was reduced to once a day. At the end of the columns experiment, the flow of distilled water was stopped, the samples corresponding to each day were mixed and homogenized and an aliquot reserved for analysis. The whole period of water flow was 80 days and the total water flow was 56.15 L. At the end of the experiment, the remaining of each tablet was collected and analyzed.

## 2.4 Chemical analysis

The following determinations were performed in leachates: electrical conductivity and pH (potentiometric methods), concentrations of  $\text{NH}_4^+$  and  $\text{NO}_3^-$  (by steam distillation), P (by visible spectrophotometry; Olsen and Sommers, 1982), Ca, Mg, Na, K and Al (by atomic absorption or emission spectrometry).

Soil samples before and at the end of the experiment were subjected to the following determinations: pH in water (soil:water ratio 1 : 10), total carbon and nitrogen (using a LECO 2000 auto-analyzer), exchange Ca, Mg, Na, K, Al (extracted by  $1 \text{ M NH}_4\text{Cl}$  and measured by a Perkin-Elmer AAnalyst 200 atomic absorption spectrometer), available phosphorus (Olsen and Sommers, 1982). The effective cation exchange capacity was calculated as the sum of Ca, Mg, Na, K and Al, extracted by  $1 \text{ M NH}_4\text{Cl}$ .  $\text{NO}_3^-$  and  $\text{NH}_4^+$  were extracted by  $2 \text{ M KCl}$  and determined by steam distillation (Keeney and Nelson, 1982).

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controls. The cumulative loss was considerably higher in treatment 11-18-11 compared to treatment 8-8-16, as expected from the higher P content in that treatment (Table 1).

### 3.1.3 Alkaline and alkaline-earth cations

Similarly to other species, a strong potassium release was observed in the first 5.97 L of leachate in both fertilized treatments. From that moment on, the release of K went down to levels similar to control (Fig. 5). The cumulative losses of K in both fertilizer treatments were markedly superior to those in controls during the whole period of experiment (Fig. 5), and significantly higher ( $p < 0.001$ ) in treatment 8-8-16 than in 11-18-11.

Calcium was also strongly released in treatment 11-18-11 at the beginning of the experiment (Fig. 6). Contrary to other elements, after an initial decrease, calcium concentrations in leachates from this treatment increased again from the seventh day and remained higher than those in controls throughout the trial. Despite the calcium contents in tablet 8-8-16 being not much lower than in 11-18-11 (Table 1), calcium concentrations in leachates in treatment 8-8-16 were higher than in controls only in the first two days of leaching; from then on, the values were similar to those of the control columns and significantly lower than in treatment 11-8-11. This means that, at the end of the experiment, even after the flowing of 56 L water, the 8-8-16 tablet still had high Ca content. At the end of the experiment, the calcium accumulated in leachates was about 20 times higher in treatment 11-18-11 compared to 8-8-16.

Magnesium leaching was similar in both fertilizer treatments at the beginning of the experiment (Fig. 7). As was the case for other elements, the greatest loss corresponded to a leachate volume of 5.97 L ( $1426 \text{ L m}^{-2}$ ). From the tenth day, magnesium leaching was negligible in treatment 11-18-11, but continued until the end of the experiment in treatment 8-8-16 (Fig. 7), in agreement with the greater Mg content of this tablet (Table 1).

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The differences between the two treatments regarding the amount and type of the elements that have been leached may be related to the quantity contributed by each treatment, as well as the different solubility of the compounds that form the tablets.

### 3.2 Change of soil parameters after percolation

5 The soil used in the experiment presented a very low initial pH, which ~~had~~ increased after the experiment, particularly in the fertilized columns (Table 2). In fertilized columns, cations contributed by fertilizers may replace acid exchange cations, which would result in soil alkalization. This seems to be particularly remarkable in treatment 8-8-16, which is richer in K and Mg; also leachates from this treatment, excepting the initial  
10 period, had higher pH values than those from treatment 11-18-11 (Fig. 1). In control columns, the pH increase might result from alkalizing reactions occurring in reducing conditions. The carbon concentration in soil ~~had~~ decreased slightly after the experiment in all columns (Table 2). **This fact can result from leaching of soluble organic substances.**

15 Ammonium concentrations in soil at the final stage were higher in the fertilized columns, particularly in treatment 8-8-16, compared to the initial stage and also to control columns, but the differences were not significant. These results were comparable to ammonium concentrations in leachates. Apparently, treatment 8-8-16 ~~contributed~~ more ammonium than treatment 11-18-11. With regard to nitrate, no significant differences were observed between treatments or with respect to the initial stage. Nitrogen  
20 contributed by fertilizers may have been leached, ~~either as nitrate (although nitrate leaching was very similar from fertilized and unfertilized columns, Fig. 3) or as ammonium, or, more likely,~~ lost through de-nitrification processes or immobilized in microbial biomass.

25 Unlike nitrogen, final available phosphorus concentrations in fertilized soil columns were notably higher than in the initial stage or in control columns, particularly in treatment 11-18-11, which provides more P (Table 2). These results are in agreement with the limited measured P leaching and may be related to the recognized low mobility

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of this element in soils, and particularly in acid soils (Gil-Sotres et al., 1982; Garcia-Rodeja and Gil-Sotres, 1997).

As for the exchange cations, the concentrations of calcium, magnesium and potassium increased in the fertilized columns. Calcium was significantly higher in treatment 11-18-11, while K and Mg were higher in treatment 8-8-16 (Table 2). The relative increases of Ca, Mg and K in both fertilized treatments were in agreement with their respective contributions (more K and Mg in 8-8-16, more Ca in 11-18-11). After a water flow equivalent to 13 years rainfall, and despite leaching losses, particularly of potassium, both fertilized soils were significantly enriched in these exchangeable cations. The remarkable decline of exchange aluminum in both fertilized treatments, compared to either initial stage or control columns, is related to the pH increase (Table 2) and the input of other cations with fertilizers.

The effective cation exchange capacity (eCEC) was very low both at the initial stage and in control soils at the final stage. In fertilized columns, the soil effective CEC at the final stage had significantly increased (Table 2), being moderately low (between 4 and 9 cmol(+) kg<sup>-1</sup>), according to Buol et al. (1975). The increase of eCEC is related to the pH increase, given the variable-charge nature of the soils used in the experiment.

### 3.3 Nutrient balances during the experiment

The percentages of nutrients released from the tablets were calculated from the nutrient amounts contained initially in the fertilizer tablets and the amounts remaining at the end of the experiment (Table 3). Similarly, the percentages of leaching losses were calculated by comparing the accumulated leaching losses with the amounts of nutrients released (Table 3).

In general, the percentages of nutrients released at the end of the trial were very high, excepting Ca and Mg from tablet 8-8-16. However, the percentage of leaching loss was extremely low for nitrogen (< 4%). The nitrogen contributed by the tablets may be retained by soil in different ways: immobilized in microbial biomass, fixed by hydroxyaluminium vermiculites, etc. Part of the nitrogen may be lost from soil, either

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by leaching or through de-nitrification processes. De-nitrification is expected to play an important role in the reducing conditions prevailing during the experiment. Also Paramasivam and Alva (1997) reported low recovery of the applied N in the leachate (from 5 % to 28 %) in experiments with different urea-based controlled-release formulations (Meister, Osmocote, and Poly-S) added to soil columns, attributing it to the combination of loss of N through  $\text{NH}_3$  volatilization, microbial assimilation of the applied N and de-nitrification processes. Phosphorus was leached at low rates (Table 3), as expected from its well-known low mobility and in agreement with the increases in soil available P. By contrast, potassium leaching was relatively high (more than 60 % of the total released). The potassium not leached can remain in the soil either as exchange cation or fixed by hydroxyaluminium vermiculites that are very common in these granitic acidic soils. Núñez-Delgado et al. (1997) also reported a nearly total P retention in soil and low  $\text{NH}_4^+$  and K leaching in column experiments carried out with Galician soils after the addition of cattle slurry. In another study also using laboratory columns and different slow release fertilizers, but with a lower total water flow (21 L), Broschat and Moore (2007) obtained a P leaching between 47 and 80 %, lower than that of N and K (> 80 %). These percentages are clearly higher than those found in our study, probably because Broschat and Moore (2007) filled up their columns with washed sand, with much lower retention capacity for elements and compounds. Calcium and magnesium leaching, similarly to Ca and Mg release, were relatively high in treatment 11-18-11 and low in treatment 8-8-16 (Table 3). Besides the increase of exchange Ca and Mg (Table 2), immobilization by the microbial biomass and precipitation of insoluble Ca and Mg compounds may explain the differences in the percentage leached.

## 4 Conclusions

At the end of the trial, after the percolation of an amount of water equivalent to 13 years rainfall in the area, releases from fertilizer tablets were more than 80 % for most nutrients. Under the conditions of this study, Ca and Mg were usually released at lower

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rates, especially in the treatment 8-8-16 (less than 60%), while more than 99% of N was released from both tablets. Despite this, the amounts leached are generally low when compared with the total released. Most leaching occurred at the beginning of the experiment, within an interval of flow equivalent to 1.5 years rainfall. From that moment on, an increase of pH and a sharp decrease of nutrient concentrations were observed in leachates. **The overall results indicate that most of the elements contained in the fertilizers remained in the soil or were lost by volatilization.** At the end of the percolating study, the concentrations of available Ca, Mg, K and P had increased significantly in the soils into the fertilized columns, along with pH and effective CEC, showing at the same time a decrease of exchangeable Al. This means that, under the conditions of this study, the fertilizer treatments maintained their effects in these soils even after the passage of a water flow equivalent to 13 years rainfall. In these conditions, the formulation 8-8-16 underwent a lower overall nutrient loss, then being more suitable for crops having a nutrient demand sustained over time, also implying lower risks of water pollution, while the formulation 11-18-11 would be more suitable for crops with a strong initial demand.

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**Table 1.** Initial tablet weights (grams) and N, P, K, Mg and Ca amounts (grams) applied to each column with the treatments.

| Treatment | Initial weight | N    | P    | K    | Mg   | Ca   |
|-----------|----------------|------|------|------|------|------|
| 8-8-16    | 38.03          | 5.04 | 1.74 | 4.49 | 2.89 | 1.27 |
| 11-18-11  | 30.83          | 5.29 | 2.68 | 2.50 | 0.29 | 1.79 |





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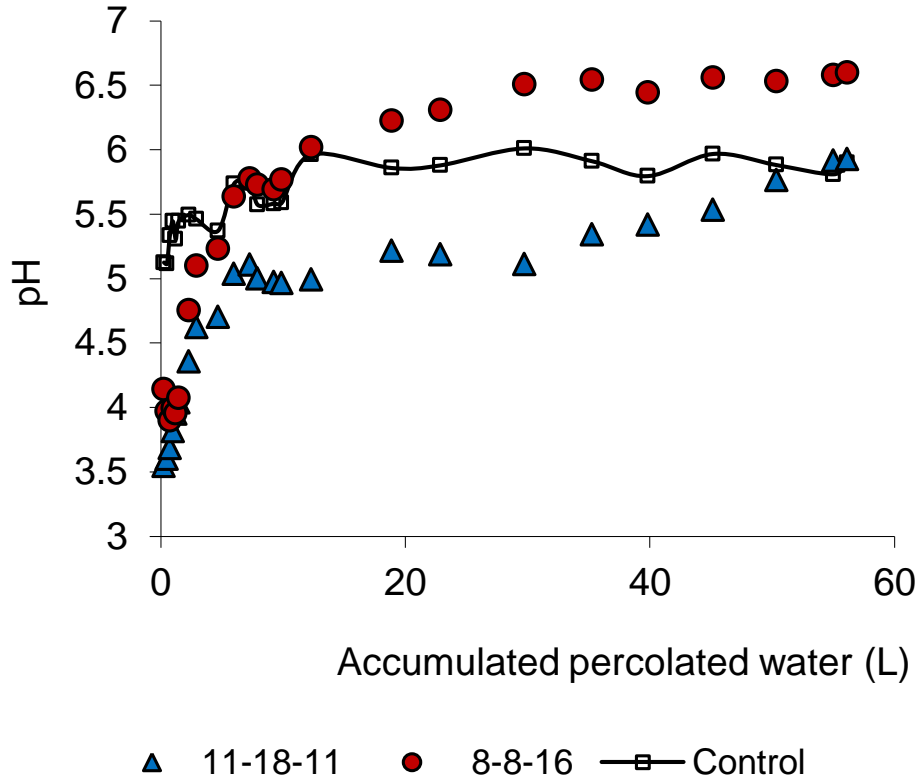
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**Table 3.** Percentages of nutrients released and leached from the tablets at the end of the experiment.

| Treatment |             | N     | P     | K     | Ca    | Mg    |
|-----------|-------------|-------|-------|-------|-------|-------|
| 11-18-11  | Released, % | 99.87 | 81.09 | 98.57 | 94.31 | 81.40 |
|           | Leached, %  | 2.82  | 28.76 | 70.61 | 53.53 | 78.11 |
| 8-8-16    | Released, % | 99.78 | 99.99 | 98.95 | 18.51 | 58.37 |
|           | Leached, %  | 3.86  | 8.76  | 63.96 | 19.61 | 18.85 |

Released: amount lost from the fertilizer tablet referred to the initial amount.

Leached: accumulated leaching loss referred to nutrient release.



**Figure 1.** pH of leachates from fertilized and control columns as a function of the volume of percolated water.

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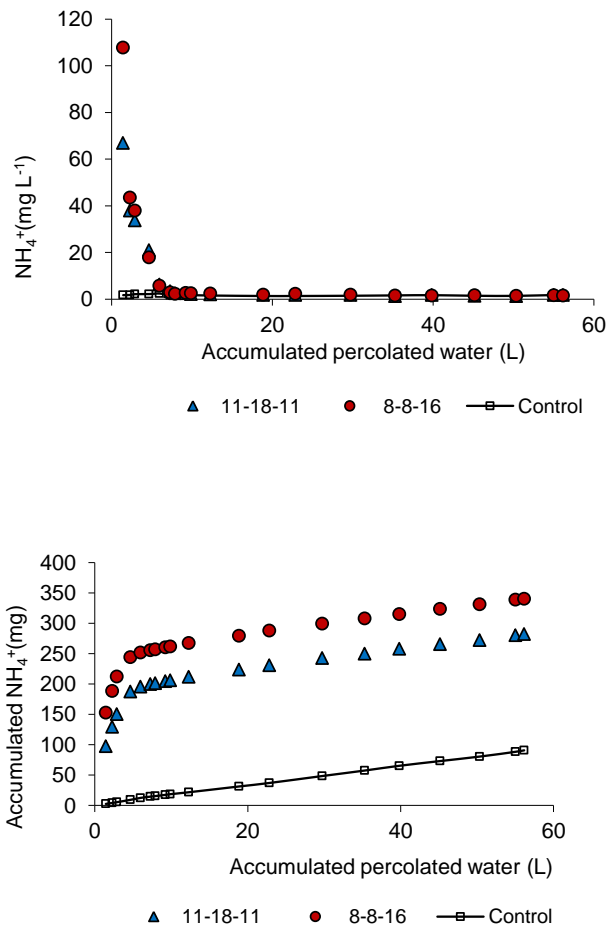
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**Figure 2.** Ammonium concentrations in leachates and accumulated NH<sub>4</sub><sup>+</sup> losses from fertilized and control columns along the experiment.

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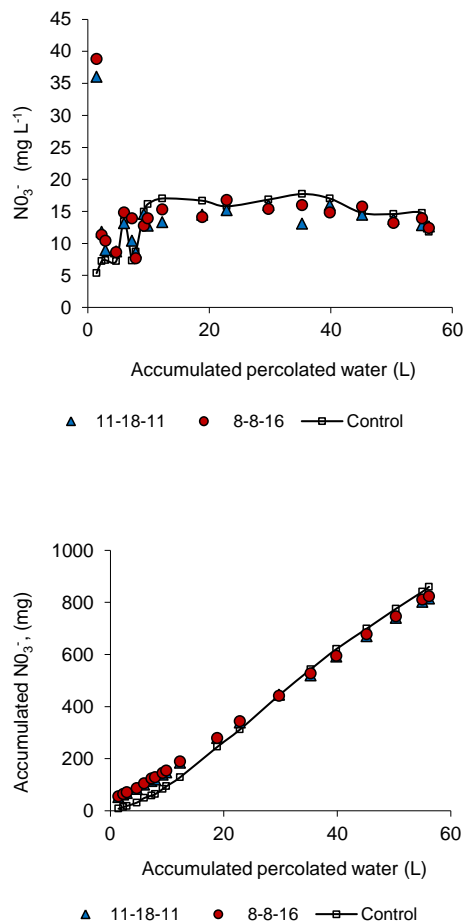
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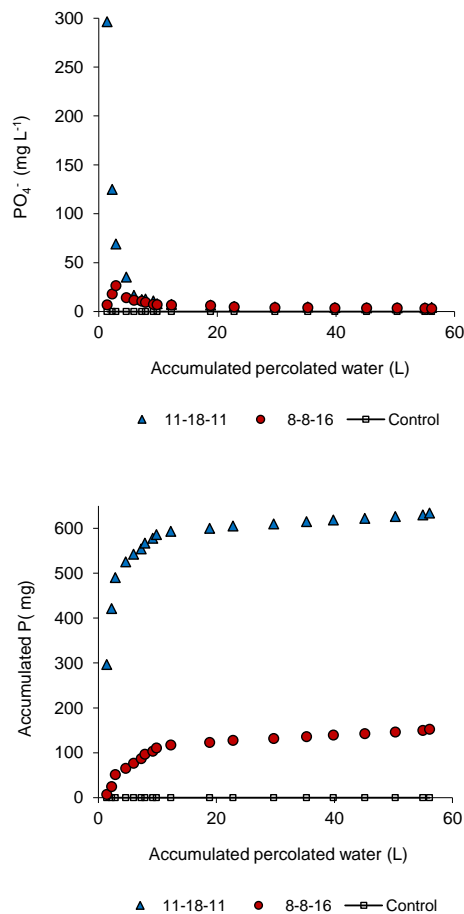
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**Figure 3.** Nitrate concentrations in leachates and accumulated  $\text{NO}_3^-$  losses from fertilized and control columns along the experiment.

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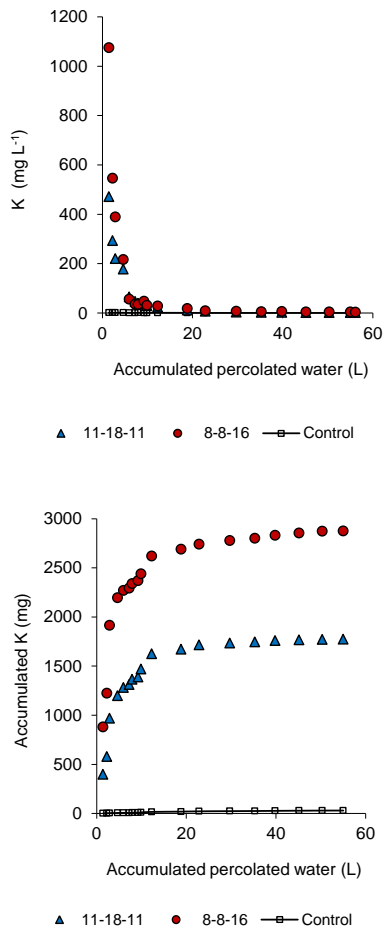
**Figure 4.** Phosphorus concentrations in leachates and accumulated P losses from fertilized and control columns along the experiment.

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**Figure 5.** Potassium concentrations in leachates and accumulated K losses from fertilized and control columns along the experiment.

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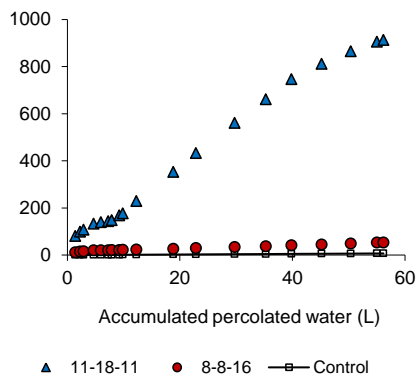
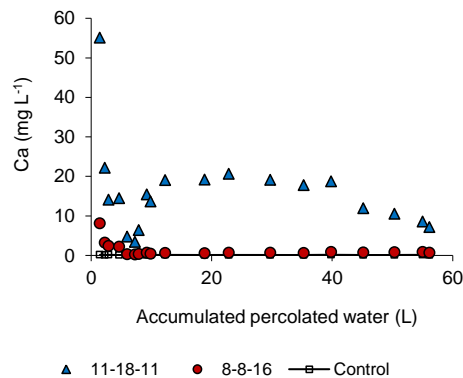
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**Figure 6.** Calcium concentrations in leachates and accumulated Ca losses from fertilized and control columns along the experiment.

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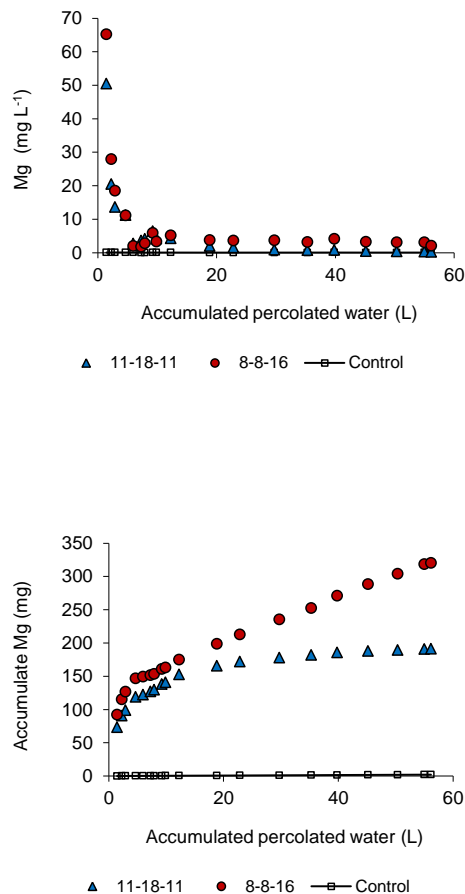
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**Figure 7.** Magnesium concentrations in leachates and accumulated Mg losses from fertilized and control columns along the experiment.

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