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Hazenite: a new secondary phosphorus, potassium and magnesium fertiliser

CONOR WATSON^{1*}, JOACHIM CLEMENS², FLORIAN WICHERN¹

¹Life Sciences Faculty, Rhine-Waal University of Applied Sciences, Kleve, Germany

²SF-SoepenberGmbH, Huenxe, Germany

*Corresponding author: conor.watson@hsrw.eu

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Abstract: Secondary fertilisers are becoming an important alternative to conventional mined fertilisers. For the first time, the struvite "relative" hazenite ($\text{KNaMg}_2(\text{PO}_4)_2 \cdot 14 \text{H}_2\text{O}$) has been artificially synthesised. A pot trial assessed whether hazenite-fertilised ryegrass had comparable potassium (K), magnesium (Mg), or phosphorus (P) uptake and shoot yields with treatments receiving conventional K (muriate of potash), Mg (kieserite) or P (triple superphosphate, TSP) fertilisers. Ryegrass shoot biomass production and K/Mg uptake in replicates receiving hazenite were as good as or superior to those amended with conventional fertilisers. Phosphorus uptake of plants whose P source was TSP was significantly higher than that of the hazenite-amended replicates without significantly higher shoot biomass, indicating luxury P uptake. Hazenite's constituent sodium (Na) makes it a potentially useful soil amendment for forage grasses or natrophilic crops such as sugar beet. Its component Mg would also be desirable in forage grasses to pre-empt ruminant hypomagnesemia or in crops with a relatively high Mg demand, for example, maize. Furthermore, hazenite represents a good alternative to KCl for chlorophobic crops such as potatoes. However, given its unusual ratio of Mg, P, and K, the ideal application of hazenite would probably be in combination with other fertilisers.

Keywords: fertilisation; macronutrient; waste stream; nutrient recycling

Secondary fertilisers are becoming an increasingly important alternative to conventional mined fertilisers. They represent an innovation in which beneficial products can be won from waste streams and whose prime asset is improved resource use efficiency (Oenema et al. 2012), contributing to nutrient recycling on a local or regional level which is especially important in areas with large amounts of excess nutrients (van der Wiel et al. 2019). Hazenite, $\text{KNaMg}_2(\text{PO}_4)_2 \cdot 14 \text{H}_2\text{O}$, was first identified as a biologically synthesised mineral in degraded cyanobacteria of the highly alkaline and saline Lake Mono by Yang et al. (2011). They surmised the water supplied the potassium (K), sodium (Na), and magnesium (Mg) while membrane phospholipids were the main source of phosphorus (P). They reported structural comparability of hazenite to struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6 \text{H}_2\text{O}$), a mineral which can be precipitated from

nutrient-rich wastewaters. As with struvite, hazenite is acid-soluble and poorly soluble in water.

The hazenite "relative" struvite has shown promise as a slow-release fertiliser of good phosphorus effectiveness (Weissengruber et al. 2018). Given that maximum yields are not achieved due to insufficient P on more than 40% of global arable land (Zhu et al. 2018) as well as projected rising fertiliser cost and geopolitical considerations affecting the availability of rock phosphate ore deposits (Egle et al. 2015), struvite has attracted interest primarily as a potential P source.

Numerous studies have proven struvite to perform similarly or favourably with reference fertilisers in terms of P uptake or yield of wheat (Massey et al. 2009), ryegrass (Bonvin et al. 2015), maize (Antonini et al. 2012), tomato (Uysal et al. 2014), sorghum and forage rye (Vogel et al. 2017). Hazenite's P content (11.2%)

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is comparable to that of struvite's (12.6%), but, as yet, its effectiveness as a P fertiliser has been untested.

Given struvite's considerable Mg content (9.9% by mass) and the declining Mg uptake of many crops in recent decades, illustrated, for example, in cereal grain Mg contents (Vogel et al. 2017), its suitability as a magnesium fertiliser has been much overlooked. Relatively few studies have considered this in comparison to work ascertaining struvite's effects as a P fertiliser. Vogel et al. (2017) reported the superior field growth of struvite-fertilised forage rye and sorghum to plants receiving triple superphosphate (TSP) and attributed this to a positive magnesium effect. Watson et al. (2019) demonstrated struvite to effect a superior Mg uptake in ryegrass when compared with a commercially available magnesium sulphate fertiliser. Kataki et al. (2016) noted Mg concentrations in soils might rise following repeated struvite amendment; this was acknowledged as potentially disruptive to Ca nutrition but beneficial to P uptake. As for P, hazenite's Mg content (8.8%) is only marginally lower than that of struvite's, and so is another potential provider of this macronutrient.

Besides the macronutrients P and Mg, hazenite also contains K (7.1% of its composition) and the non-essential element Na (4.2%). There is no essential plant K concentration with regard to its role in vacuole osmosis as Na can partially replace it (Barraclough and Leigh 1993). Some plants, termed natrophiles, demonstrate a yield benefit from the uptake of Na. While some natrophiles have evolved a Na demand to realise an optimal yield (Edmeades and O'Connor 2003), most only demonstrate an improved growth through Na if K is lacking (Chiy and Phillips 1996). Natrophobes, on the other hand, do not respond to Na as they cannot transport root Na to shoots; therefore, even if the soil has insufficient K, there is little scope for Na to replace K (Edmeades and O'Connor 2003).

Excessive Na will inhibit plant uptake of K (Gorham 2007), whereas yields from soils with sufficient K are not detrimentally affected by moderate Na application (Edmeades and O'Connor 2003). For animal production, 0.1% Na in herbage dry matter is considered vital (ibid), therefore hazenite represents a potentially useful soil amendment for grass used to feed dairy cows.

To our knowledge, this is the first time hazenite has been artificially synthesised and tested as a secondary fertiliser. The aim of this experiment was to ascertain whether hazenite performs as well as commercially-available P, K, and Mg fertilisers in terms of macronutrient provision and shoot biomass yield.

MATERIAL AND METHODS

Substrate, hazenite, and fertilisers used. The substrate used in the pot trial comprised a 1:1 mixture of quartz sand (0.1–0.5 mm, Reinstedter Kieswerk GmbH, Falkenstein, Germany) and air-dried (sieved < 4 mm) soil. The soil used was the B horizon of a stagnic luvisol used as grassland (textural class silt loam with 10% sand, 80% silt and 10% clay, pH 5.5 in 0.01 mol/L CaCl₂, 0.29% total organic carbon, < 0.05% nitrogen (N)) from Neulouisendorf, Germany. The soil contained average Mehlich-3 extractable concentrations of 15.4 mg P, 109 mg K and 71.7 mg Mg/kg. When these respective values were further reduced by a 1:1 dilution of the soil with quartz sand, the resulting substrate was categorised as having "low" P (Havlin et al. 2014) and "low" K and Mg (Hanlon 2007) concentrations.

The hazenite used originated from an industrial wastewater treatment plant that treats wastewater (2100 m³ per day) from the milk industry. A pilot plant was set up to demonstrate the effectiveness of P-removal *via* hazenite production in two smaller wastewater streams (around 250 m³ per day). The pilot plant was a one-third-filled 17 m³ batch reactor. The two streams were wastewater from reverse osmosis and the regeneration of the anion exchanger. The magnesium source was its chloride salt. The precipitated hazenite had a molar ratio of 1 (P):1.05 (Mg):0.50 (Na):0.35 (K), which was close to the expected ratio of 1:1:0.5:0.5. Quantitative analysis *via* X-Ray diffractometry detected 100% hazenite.

The commercially available fertilisers used were triple superphosphate (Ca(H₂PO₄)₂, ICL Fertilisers, Ludwigshafen, Germany), kieserite (MgSO₄·H₂O) and muriate of potash (KCl, K + S KALI GmbH, Kassel, Germany).

Pot trial design. The goal of the pot trial was to compare the effectiveness of hazenite with conventional fertilisers as a source of K, Mg or P. Therefore, the following treatments were set up (each with seven replicates):

- (1) triple superphosphate as P source (TSP);
- (2) hazenite as P source (Haz-P);
- (3) kieserite as Mg source (Kies);
- (4) hazenite as Mg source (Haz-Mg);
- (5) muriate of potash as K source (MoP);
- (6) hazenite as K source (Haz-K).

Each pot contained 4 kg sand-soil mixture and was sown with 0.75 g seeds of *Lolium multiflorum italicum* L. cv. Fabio. Hazenite or conventional fertiliser amend-

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ments were mixed thoroughly with the substrate in a sealed barrel before the soil-sand mix was transferred to a pot. The amendments were added to the soil-sand mix to provide 27 mg P/kg, 22 mg Mg/kg, or 101 mg K/kg. A solution (adjusted to pH 6.0 ± 0.1 with 1 mol/L NaOH), ensuring that equal quantities of all other nutrients were applied to each treatment "pair" (namely hazenite *versus* TSP as a P source, *versus* kieserite as an Mg source and *versus* muriate of potash as a K source), was added at the beginning of the experiment to bring all pots to 70% of the substrate's water-holding capacity (WHC). Thereafter pots were twice-weekly gravimetrically maintained at 70% WHC using demineralized water.

The amount of $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$ was reduced in pots receiving TSP (treatment 1) to account for Ca applied with that mineral; the shortfall of N was accounted for with increased NH_4NO_3 application. Where P supply was investigated (treatments 1 and 2), the Mg and K applied in hazenite was matched with Mg and K applied in solution (additionally added as $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ and K_2SO_4). The nutrient solution for treatments 1 and 2, therefore provided:

- 0.65 mg/kg H_3BO_3 , $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4 \text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$;
- 2.5 mg/kg $\text{MnSO}_4 \cdot \text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$ and $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$;
- 12.5 mg/kg $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$;
- $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$: 100 mg/kg (treatment 1) or 205 mg/kg (treatment 2);
- NH_4NO_3 : 230 mg/kg (treatment 1) or 195 mg/kg (treatment 2);
- K_2SO_4 : 265 mg/kg (treatment 1) or 215 mg/kg (treatment 2);
- $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$: 440 mg/kg (treatment 1) or 225 mg/kg (treatment 2).

Where Mg supply was investigated (treatments 3 and 4), the P and K applied in hazenite was matched with P and K applied in solution (applied as KH_2PO_4 , entailing a reduced amount of K_2SO_4). The nutrient solution for treatments 3 and 4, therefore provided:

- 0.65 mg/kg H_3BO_3 , $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4 \text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$;
- 2.5 mg/kg $\text{MnSO}_4 \cdot \text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$ and $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$;
- 12.5 mg/kg $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$;
- 205 mg/kg $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$;
- 195 mg/kg NH_4NO_3 ;
- K_2SO_4 : 105 mg/kg (treatment 3) or 145 mg/kg (treatment 4);
- KH_2PO_4 : 250 mg/kg (treatment 3) or 125 mg/kg (treatment 4).

Where K supply was investigated (treatments 5 and 6), the P and Mg applied in hazenite was matched with

P and Mg applied in solution (additionally added as $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ and $(\text{NH}_4)_2\text{HPO}_4$). The nutrient solution for treatments 5 and 6, therefore provided:

- 0.65 mg/kg H_3BO_3 , $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4 \text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$;
- 2.5 mg/kg $\text{MnSO}_4 \cdot \text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$ and $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$;
- 12.5 mg/kg $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$;
- 205 mg/kg $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$;
- 410 mg/kg NH_4NO_3 (treatment 6);
- $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ 2540 mg/kg (treatment 5) or 1270 mg/kg (treatment 6);
- $(\text{NH}_4)_2\text{HPO}_4$: 1360 mg/kg (treatment 5) or 680 mg/kg (treatment 6).

The pot trial was conducted over 77 days under controlled conditions in a greenhouse providing respective night and day temperatures of 16–21 °C and 20–23 °C and a photoperiod of 16 h. All pots were arranged in a randomised block design. Shoots from all pots were harvested 2 cm above the soil surface after days 21, 35, 49, 63 (treatments 5 and 6 only), and 77. Following each harvest, shoot weights (after drying overnight at 70 °C), and P, K, and Mg concentrations were determined (*via* an Optima 8000 ICP-OES, Perkin Elmer, Baesweiler, Germany) after concentrated sulphuric acid/hydrogen peroxide digestion. After the final harvest, roots were separated from the soil-sand mix *via* sieving and rinsed thoroughly in tap water, followed by distilled water prior to being dried to a constant mass at 70 °C.

Calculations and statistical analyses. The figures presented in the tables show arithmetic means with standard errors, and are reported on an oven-dry basis. Prior to mean comparison, all data were tested for normal distribution and homoscedasticity. Normal distribution was checked visually by a normal quantile-quantile plot. If data were not normally distributed, the box-cox transformation was conducted. All statistical calculations were performed using the computing environment R. To test for statistically significant differences between means of treatment groups, one-way analysis of variance (ANOVA) was performed with block effect accounted for. Where a significant difference ($P < 0.05$) was observed between treatments, a post hoc Tukey honestly significant difference (*HSD*)-test was conducted.

Relative fertiliser efficiencies were calculated according to the following equation:

$$(\text{hazenite parameter})/(\text{fertiliser parameter}) \times 100$$

with fertiliser signifying TSP, kieserite or muriate of potash, and parameter denoting total dry shoot biomass (g) or total K/Mg/P uptake (mg).

RESULTS

No significant differences were observed in dry shoot weights of each individual harvest, therefore only cumulated dry shoot weight data are portrayed in Tables 1 to 3. P uptake of plants whose P source was hazenite was significantly lower than when the P source was triple superphosphate (Table 1); this possibly led to the considerably higher root biomass in the hazenite-amended replicates. The higher P uptake in TSP-amended replicates did not induce significantly higher cumulated shoot biomass, however, indicating luxury P uptake in these replicates. The cumulated shoot biomass, Mg uptake and root biomass of plants utilising hazenite as an Mg source were not significantly lower than in replicates that received a commercially-available kieserite Mg fertiliser (Table 2). Replicates utilising hazenite as a K source had significantly higher shoot biomass than replicates receiving muriate of potash (Table 3). However, there were no significant differences between these treatments in terms of the cumulated K uptake or root biomass.

Where K fertilisation was investigated (treatments 5 and 6), the nutrient solutions had markedly higher concentrations of N, S, Mg, and P than in the solutions applied to the other treatments. This would explain the considerably lower investment of the plants in root biomass in these treatments (Table 3) than where the supply of P (Table 1) or Mg (Table 2) was investigated.

The relative fertiliser efficiencies (RFEs) were between 94.9% and 126% for the cumulated shoot weight determinant (Tables 1 to 3). With reference to nutrient uptake, the RFEs were between 81.8% (P) and 99.7% (K).

Table 1. Mean values of the dry root weight, the cumulated dry shoot weight after 4 harvests, the cumulated phosphorus (P) uptake of each treatment, and the relative fertiliser efficiency (RFE)

	Hazenite	Triple superphosphate
Shoot weight (g)	4.07 (0.09) ^a	4.29 (0.07) ^a
RFE (%)	95	100
P uptake (mg)	10.41 (0.51) ^b	12.72 (0.38) ^a
RFE (%)	82	100
Root weight (g)	5.62 (0.90) ^a	4.26 (0.72) ^a

Values show arithmetic means and are followed by standard errors in brackets ($n = 7$). In each row, different letters indicate significant differences

DISCUSSION

Given its unusual ratio of Mg, P, and K, hazenite's ideal application would probably be in combination with other fertilisers, as has been reported for the structurally similar struvite (Kataki et al. 2016). A secondary fertiliser containing three macronutrients and beneficial elements such as the hazenite used in our experiment must be considered in terms of the nutrients' bioavailability, and of the influence, one element may have on another's uptake by plants. The retention of magnesium, potassium, and sodium are largely controlled by ion exchange processes in soil, meaning they are of greater susceptibility to leaching but become more rapidly bioavailable than phosphate. The latter, whose retention and release in the soil is dominated by chemisorption and precipitation-dissolution processes, can, therefore, be less bioavailable in conditions of unfavourable pH. The relatively acidic pH of the soil component of the substrate used in this experiment was favourable to hazenite dissolution: Gérard (2016) reported that acidification should cause P solubility to rise in P-deficient soils rich in clays or iron (Fe) oxides. Degryse et al. (2017) stated that lower than pH 8, the solubility of the hazenite "relative" struvite rises with falling pH. Barrow (2017) reported that, as pH falls from 6 to 4, root phosphate absorption and phosphate desorption from soil rises. He postulated that these two phenomena are likely to outweigh an increased amount of phosphate adsorbed by soil.

Sludge-derived struvites, with their high P effectiveness, low water solubility, and low toxic element content, have been reported by Weissengruber et al. (2018) to be potentially less contaminating than composts, manures, and rock phosphate. The structurally similar hazenite,

Table 2. Mean values of the root weight, the cumulated dry shoot weight after 4 harvests, the cumulated magnesium (Mg) uptake of each treatment, and the relative fertiliser efficiency (RFE)

	Hazenite	Kieserite
Shoot weight (g)	3.98 (0.07) ^a	4.19 (0.09) ^a
RFE (%)	95	100
Mg uptake (mg)	10.41 (0.89) ^a	10.88 (0.89) ^a
RFE (%)	96	100
Root weight (g)	5.52 (1.18) ^a	6.28 (1.39) ^a

Values show arithmetic means and are followed by standard errors in brackets ($n = 7$). In each row, different letters indicate significant differences

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Table 3. Mean values of the dry root weight, the cumulated dry shoot weight after 5 harvests, the cumulated potassium (K) uptake of each treatment, and the relative fertiliser efficiency (RFE)

	Hazenite	Muriate of potash
Shoot weight (g)	8.05 (0.90) ^a	6.39 (0.89) ^b
RFE (%)	126	100
K uptake (mg)	479 (48) ^a	480 (52) ^a
RFE (%)	99.7	100
Root weight (g)	1.49 (0.29) ^a	0.99 (0.23) ^a

Values show arithmetic means and are followed by standard errors in brackets ($n = 7$). In each row, different letters indicate significant differences

derived from milk industry wastewaters as in this study, may also be considered to be so.

Seedlings are sensitive to P shortfall and may not fully recover from temporary inadequate P supply (Sanchez 2007). A critical concentration of 2.5 mg P/g dry matter has been reported for ryegrass (Bonvin et al. 2015). Based on accumulated shoot weights and P uptake (Table 1), the replicates grown in our hazenite-amended, nutrient-poor substrate achieved a concentration of 2.56 mg/g. In a pot trial using a similarly P-deficient substrate and a struvite application rate (25 mg P/kg substrate) similar to our hazenite application rate, Antonini et al. (2012) reported struvite to have met more than half of the ryegrass P demand and all of that of Mg.

Phosphates have a synergistic effect on Mg uptake in plants and *vice versa* due to stimulated root growth following P application and the ionic balance of cation/anion uptake (Merhaut 2007). Low Mg concentrations in forage grasses are highly undesirable as it can lead to hypomagnesemia in the blood of grazing animals (Havlin et al. 2014). The sufficiency of Mg in ryegrass was reported to lie in the range of 0.16% to 0.32% (Merhaut 2007), based on the cumulated shoot weights and Mg uptake (Table 2), replicates grown in our hazenite-amended, nutrient-poor substrate achieved a concentration of 0.26%.

Magnesium has a function in stress physiology; therefore, crop demand for it may increase in unfavourable conditions (Gransee and Führs 2013). Relatively high extractable Mg concentrations can potentially detrimentally impact Ca nutrition of some crops, for example, tomato and woody ornamentals (Merhaut 2007). Therefore hazenite should be considered as an Mg source for crops with a relatively high Mg demand, such as maize and pulses.

Potassium is reported to affect Mg uptake; it possibly inhibits root to shoot transport of Mg (Gransee and Führs

2013). For example, higher K application rates applied to field-grown grass by Chiy et al. (1998) tended to reduce Mg concentrations. The opposite effect of Mg suppressing K uptake is not particularly marked; it is probable that K transporters are highly specific, whereas some Mg transporters can also permit K uptake (Gransee and Führs 2013). Uysal et al. (2014) reported K uptake by maize and tomato to decrease with increasing struvite application, but it is not clear whether the Mg in the struvite could be attributed to this suppression.

Barracough and Leigh (1993) reported K concentrations in sap to be independent of P. Regarding substitution of K; they stated Na to be more efficacious than Mg, possibly due to divalents being less easily absorbed and internally translocated. Within the plant, K and Na compete for absorption sites; the binding sites on root cell plasma membranes have a high affinity for K and a low one for Na (Chiy et al. 1998). Fertiliser Na can raise both plant Na and K concentrations (Edmeades and O'Connor 2003) as some plant transporters may use Na⁺ gradients to enhance K⁺ translocation (Mäser et al. 2002).

Sodium applied to pasture naturally high in Na can negatively impact macronutrient uptake (Chiy and Phillips 1996) whereas Edmeades and O'Connor (2003) reported Na application in soils with sufficient K to have no detrimental effects; indeed, Na fertiliser can increase pasture yields where K is deficient due to the partial substitution of Na for K. Chiy et al. (1998) reported increased grass leaf K concentrations following application of a Na fertiliser and a combined Na/K fertiliser. Among other crops, K can be substituted by Na in edible parts to varying degrees, for example, from 1% in wheat and rice to up to 90% in red beet (Gorham 2007).

KCl is generally used more often than the sulphate salt as a fertiliser as it is cheaper, and most crops are not chlorophobic (Mengel 2007). In addition to K losses by leaching from sandy soils being more likely to be greater if it is applied in a chloride rather than phosphate form (Havlin et al. 2014), the hazenite used in this experiment (effectively a K fertiliser with Na) would be suitable for natrophilic crops such as sugar beet and cabbage, and represents a good alternative to KCl for chlorophobic crops such as potatoes, onions and fruit trees (Mengel and Kirkby 2001).

The hazenite "relative" struvite is viewed as a slow-release fertiliser due to its low solubility compared to conventional acidified fertilisers (Degryse et al. 2017). It must be acknowledged that the effectiveness of the hazenite used in this experiment as P, Mg, and K sources may have been exaggeratedly high as it was not in a granular form, whereas TSP, kieserite, and muriate of potash were.

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Degryse et al. (2017) reported granular struvite to dissolve at a rate of up to 0.43 mg/day in acidic soil; the "powdery" fine hazenite used in this experiment could, therefore, be assumed to have an even higher dissolution rate.

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