GRIPHITE, A HYDROPHOSPHATE GARNETOID

Duncan McConnell*

Abstract

Griphite is cubic and its structure is similar to that of garnets. The unit cell contains 
$8[(Na, Al, Ca, Fe)_3Mn_5(PO_4)_2(OH)_6]$ and this formula represents a complex example of 
$X_3Y_5(ZO_4)_{3-n}(OH)_n$. The latter is a variation of $X_3Y_5(ZO_4)_3$ which is necessary to 
accommodate the hydrogarnets and plazolite. An alternate formula is suggested and cannot 
be eliminated as a possibility, namely, $8[(Na, Ca, Fe, Mn)_3(Al, Mn)_5(PO_4)_2(0H)_6]$. The 
space group could not be determined but appears to have lower symmetry than that of 
garnet ($O_h^{19} = Ia3d$). Griphite and other substances with structures similar to garnet are 
classified as garnetoids.

Introduction

The name griphite was proposed in 1891 by W. P. Headden (1) for a 
hydrated phosphate of manganese, aluminum, sodium, calcium, and 
iron. Griphite (γρῦθος = an enigma) seemed an appropriate name because 
of the chemical complexity of this mineral and the apparent absence of a 
simple relation to any other known mineral.

The present investigation will demonstrate that the chemical composi-
tion, physical properties and x-ray diffraction data for griphite permit its 
classification as a garnetoid. Several interesting implications are to be 
attached to the fact that a sodium-calcium-manganese-aluminum hydro-
phosphate with garnet-like structure exists. Its existence suggests the 
possible replacement of calcium and silicon by sodium and phosphorus in 
garnets and garnetoids. Of particular interest is the possible entry of 
these constituents into the lattice of cubic tricalcium aluminate hexa-
hydrate, one of the well established hydration products of portland 
cement, because both sodium and phosphorus are considered objection-
able constituents in cement clinker, if they exceed certain limits.

Ionic substitutions involving sodium and phosphorus are relatively un-
common among garnets, or their occurrence has been overlooked as only 
a few analyses report Na$_2$O (about 2 per cent being the maximum 
amount), and the analyses that indicate P$_2$O$_5$ are extremely rare (the 
amount of P$_2$O$_5$ being less than 1 per cent). Although the writer (2) has 
demonstrated in ellestadite almost complete replacement of PO$_4$-groups 
by SO$_4$- and SiO$_2$-groups in the structure of apatite, nothing conversely 
similar has been previously shown among the garnets. Thus the rather

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1 The term "garnetoid" is introduced to designate those substances which are not 
primarily silicates but have structures similar to that of true garnets. Examples are the 
various hydrogarnets, berzeliite, plazolite, and griphite.
complete substitution of PO₄-groups for SiO₄-groups encountered in
grphite is noteworthy.

Cubic tricalcium aluminate hexahydrate has recently been investigated
thoroughly by E. P. Flint, H. F. McMurdie, and L. S. Wells (3). These in-
vestigators likewise demonstrated the existence of a hydrogarnet corre-
sponding to andradite and having the composition 3CaO·Fe₂O₃·6H₂O.
The structural data presented by these authors, the data obtained for
berzeliiite by W. Bubeck and F. Machatschki (4), and the complete struc-
tural investigation of plazolite presented by A. Pabst (5), are invaluable
in the establishment of the relationship of griphite to other garnetoids.

**Physical Properties of Griphite**

Headden (1) described griphite as “amorphous” because it is isotropic
on microscopic examination and no crystal forms were observed. How-
ever, the relationships among the properties of griphite, plazolite,² and
grossularite are quite apparent when tabulated.

<table>
<thead>
<tr>
<th>Property</th>
<th>Griphite</th>
<th>Plazolite</th>
<th>Grossularite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness</td>
<td>5½</td>
<td>6½(?)</td>
<td>6</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>3.40</td>
<td>3.13</td>
<td>3.58 ca.</td>
</tr>
<tr>
<td>Refractive index</td>
<td>1.64–1.66</td>
<td>1.675</td>
<td>1.735</td>
</tr>
<tr>
<td>Fusibility</td>
<td>Easily fused</td>
<td>Fusible</td>
<td>3</td>
</tr>
</tbody>
</table>

Griphite apparently lacks the ability to form euhedral crystals, differ-
ing from garnets in this respect. However, it is not amorphous but ex-
hibits discontinuous vectorial properties, as is demonstrated by the x-ray
diffraction pattern which it produces. Both griphite and plazolite are
soluble in HCl, whereas true garnets (except schorlomite) are difficultly
soluble or insoluble in ordinary acids. Absence of cleavage is character-
istic of the garnetoids as well as the garnets.

**X-Ray Diffraction Data**

Powder diffraction patterns were obtained for griphite (near Harney
City, Pennington County, South Dakota; U.S.N.M. No. R-5315, obtained
from Headden), grossularite (6), schorlomite (Magnet Cove, Arkansas),
Uvarovite (Washington, Nevada County, California) and andradite (near
Randsburg, California) through the use of unfiltered iron radiation and
precision cameras (M. J. Buerger’s model) with r = 57.3 mm. The cell
dimensions of these are as follows, omitting andradite:

- **Griphite**: a₀ = 12.26 (all ± 0.01 Å)
- **Schorlomite**: a₀ = 12.09
- **Uvarovite**: a₀ = 11.97
- **Grossularite**: a₀ = 11.87

² D. S. Belyankin and V. P. Petrov (Am. Mineral., 26, 450–453, 1941) have recently
shown through reexamination of hibschite that this substance is similar to plazolite.
Table 1. Comparison of Powder Diffraction Diagrams
(Unfiltered Fe Radiation—$r = 57.3$ mm.)

<table>
<thead>
<tr>
<th>No.</th>
<th>Indices*</th>
<th>Schorlomite**</th>
<th>Graphite</th>
<th>Hausmannite†</th>
<th>Mn-bergellite‡</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$d_{\text{exp}}$</td>
<td>$I$</td>
<td>$d_{\text{calc.}}$</td>
<td>$I$</td>
<td>$d$</td>
</tr>
<tr>
<td>1</td>
<td>220</td>
<td>4.31</td>
<td>&lt;1</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td>320</td>
<td>—</td>
<td>3.401</td>
<td>&lt;1</td>
<td>3.39</td>
</tr>
<tr>
<td>3</td>
<td>$\beta400$</td>
<td>3.34</td>
<td>&lt;1</td>
<td>(3.37)</td>
<td>3.277</td>
</tr>
<tr>
<td>4</td>
<td>321</td>
<td>3.026</td>
<td>5</td>
<td>3.066</td>
<td>3.066</td>
</tr>
<tr>
<td>5</td>
<td>400</td>
<td>2.975</td>
<td>2</td>
<td>(3.01)</td>
<td>2.974</td>
</tr>
<tr>
<td>6</td>
<td>$\beta420$</td>
<td>410, 322</td>
<td>8</td>
<td>2.742</td>
<td>2.743</td>
</tr>
<tr>
<td>7</td>
<td>420</td>
<td>2.702</td>
<td>8</td>
<td>2.742</td>
<td>2.743</td>
</tr>
<tr>
<td>8</td>
<td>431</td>
<td>2.366</td>
<td>1</td>
<td>2.404</td>
<td>2.408</td>
</tr>
<tr>
<td>9</td>
<td>520, 432</td>
<td>521</td>
<td>2.297</td>
<td>2.281</td>
<td>2.281</td>
</tr>
<tr>
<td>10</td>
<td>610</td>
<td>2.015</td>
<td>3</td>
<td>2.015</td>
<td>2.018</td>
</tr>
<tr>
<td>11</td>
<td>611, 532</td>
<td>1.964</td>
<td>2</td>
<td>1.828</td>
<td>1.832</td>
</tr>
<tr>
<td>12</td>
<td>620</td>
<td>1.909</td>
<td>&lt;1</td>
<td>1.789</td>
<td>1.832</td>
</tr>
<tr>
<td>13</td>
<td>$\beta640$</td>
<td>541, 1.845</td>
<td>&lt;1</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>14</td>
<td>630, 542</td>
<td>1.781</td>
<td>2</td>
<td>1.781</td>
<td>1.832</td>
</tr>
<tr>
<td>15</td>
<td>444</td>
<td>1.743</td>
<td>1</td>
<td>1.770</td>
<td>1.770</td>
</tr>
<tr>
<td>16</td>
<td>640</td>
<td>1.678</td>
<td>7</td>
<td>1.700</td>
<td>1.700</td>
</tr>
<tr>
<td>17</td>
<td>642</td>
<td>1.614</td>
<td>&gt;10</td>
<td>1.638</td>
<td>1.637</td>
</tr>
<tr>
<td>18</td>
<td>730</td>
<td>1.601</td>
<td>—</td>
<td>1.610</td>
<td>1.604</td>
</tr>
<tr>
<td>19</td>
<td>732, 651</td>
<td>1.557</td>
<td>1</td>
<td>1.574</td>
<td>1.563</td>
</tr>
<tr>
<td>20</td>
<td>800</td>
<td>1.512</td>
<td>3</td>
<td>1.533</td>
<td>1.536</td>
</tr>
<tr>
<td>21</td>
<td>820, 644</td>
<td>1.487</td>
<td>2</td>
<td>1.487</td>
<td>1.478</td>
</tr>
<tr>
<td>22</td>
<td>822, 660</td>
<td>1.424</td>
<td>&lt;1</td>
<td>1.437</td>
<td>1.437</td>
</tr>
<tr>
<td>23</td>
<td>840</td>
<td>1.351</td>
<td>5</td>
<td>1.338</td>
<td>1.344</td>
</tr>
<tr>
<td>24</td>
<td>842</td>
<td>1.319</td>
<td>5</td>
<td>1.322</td>
<td>1.325</td>
</tr>
<tr>
<td>25</td>
<td>921, 761, 655</td>
<td>1.290</td>
<td>4</td>
<td>1.307</td>
<td>1.303</td>
</tr>
</tbody>
</table>

* The indices do not pertain to hausmannite.
** Several of the weaker $\beta$-lines are omitted.
† Only the more intense lines are listed. Data from G. Aminoff: Zeits. Krist., 64, 475 (1926).
‡ Data from W. Bubeck and F. Machatschki: op. cit. Reflection from 211 was not observed by these authors although the calculated intensity is 6. Planes 653 and 752 gave observed intensities 2 and 1, respectively.
The cell edges of schorlomite and uvarovite are given, although analyses of these specimens are not available, because published measurements for these garnets are very rare in the literature. In addition it was observed that the diffraction pattern of schorlomite is not significantly different from those of other garnets. Therefore schorlomite is more closely related to the garnets than the garnetoids.

The similarities of the powder diffraction patterns of schorlomite and griphite are shown in Fig. 1 and the measurements obtained from these are given in Table 1. With one exception (line No. 13, Table 1) all of the lines obtained from the sample of griphite yield cubic indices. This line, presumably extraneous, may have been contributed by hausmannite. It was not feasible to completely eliminate one of the contaminating substances, a mineral which appears deep red-brown to opaque in thin section and resembles hausmannite in other respects.

![Fig. 1. Powder diffraction patterns of schorlomite (above) and griphite. Unfiltered Fe radiation.](image)

If hausmannite is to be assumed as a contaminating substance, the diffraction pattern must be interpreted through consideration of all of the intense lines of this mineral, and these are shown in Table 1 for comparison. However, this assumption does not explain the occurrence of reflections from 320, 411, 730, 630 or 542, and 520 or 432, although the first two of these are similar in spacing to $\beta$-lines. Of significance are the similarities in intensities between the corresponding lines of griphite and Mn-berzeliite. The experimental and calculated intensities for Mn-berzeliite obtained by Bubeck and Machatschki (4) are given for comparison (Table 1).

Notwithstanding the similarities, there are certain differences among the patterns of schorlomite, griphite, and Mn-berzeliite, the most noticeable being the occurrence in the griphite pattern of reflections prohibited by the space group $O_h^{10}$. Otherwise, it is quite apparent that the more intense lines are those characteristic of garnet. X-ray data must be
considered with caution when powder diffraction methods are employed to the exclusion of other methods and, particularly, when contamination of the sample is probable. Thus it is not possible to decide the space group of graphite but there is every indication that the structure closely simulates one based upon a body-centered cubic lattice. The true symmetry appears to be lower than that of garnet (O_h^{10} = Ia3d).

Were it not for several additional factors the conclusion that graphite is a garnetoid might seem unjustifiable. These factors are:

1. The composition indicated by Headden’s analysis conforms with the type formula of garnets and garnetoids.
2. The calculated density is virtually identical with the experimental value.
3. The refractive index is similar to what would be expected for a substance with the structure of garnet and the chemical composition of graphite.

Chemical Data

The mean of Headden’s analyses is used as a basis for calculating the cell contents (Table 2), assuming that all of the cations present enter and completely fill the 64 cationic positions of normal garnet and that the lattice contains 96 oxygens. This permits the correlation of graphite and the type formula \(X_3Y_2(ZO_4)_{3m}\), or the derived formula \(X_3Y_2(ZO_4)_{3-m}(OH)_{2m}\), in one or the other or a combination of the following ways:

\[
\begin{align*}
& (Na,Al,Fe)_{3}Mn_{2}(PO_4)_{3}(OH)_2 \\
& (Na,Mn,Ca,Fe)_{3}(Al,Mn)_{3}(PO_4)_{3}(OH)_2
\end{align*}
\]

where \(Na \approx 1; Ca + Fe \approx 1; Al \approx 1\)

Of these possibilities, the former seems more probable, but nothing definite can be decided without a detailed structural investigation. The x-ray and chemical data available at present are not sufficiently complete for this purpose and several factors impede more accurate determinations.

The density calculated from the molecular weight \((8 \times 474.5)\) and the cell edge \((12.26 \text{ Å})\) is 3.399. This compares most favorably with the density determined by Headden \(3.401\) which was confirmed by the writer as 3.40.

The calculations of the cell contents and density have been based on the assumption that all cationic positions of the lattice are filled, but the simultaneous assumption of 96 oxygen positions yields a slight discrepancy between the positive and negative charges. This is not serious, however, and probably is to be explained by the presence of one or more of the impurities mentioned below. An alternate explanation can be obtained by assuming that there are vacancies in the lattice. If it is assumed
Table 2. Chemical Analysis and Cell Contents of Grifite

<table>
<thead>
<tr>
<th>Oxides*</th>
<th>Weight per cents</th>
<th>Molecular ratios</th>
<th>Ionic ratios</th>
<th>Contents of ( \frac{1}{8} ) unit cell</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Positive ions</td>
</tr>
<tr>
<td>Li₂O</td>
<td>trace</td>
<td>—</td>
<td>—</td>
<td>.86</td>
</tr>
<tr>
<td>Na₂O</td>
<td>5.52</td>
<td>.089</td>
<td>.178</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>0.30</td>
<td>.003</td>
<td>.006</td>
<td>.03</td>
</tr>
<tr>
<td>CaO</td>
<td>7.47</td>
<td>.133</td>
<td>.133</td>
<td>.64</td>
</tr>
<tr>
<td>MgO</td>
<td>0.15</td>
<td>.004</td>
<td>.004</td>
<td>.02</td>
</tr>
<tr>
<td>FeO</td>
<td>4.00</td>
<td>.056</td>
<td>.056</td>
<td>.27</td>
</tr>
<tr>
<td>MnO</td>
<td>29.64</td>
<td>.418</td>
<td>.418</td>
<td>2.02</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>10.13</td>
<td>.099</td>
<td>.198</td>
<td>.96</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>38.52</td>
<td>.271</td>
<td>.542</td>
<td>2.62</td>
</tr>
<tr>
<td>Cl</td>
<td>0.11</td>
<td>.003</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>trace</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td>H₂O</td>
<td>4.29</td>
<td>.238</td>
<td>( 4 \times .119 )</td>
<td>4×0.58†</td>
</tr>
<tr>
<td>Insol.</td>
<td>0.16</td>
<td>—</td>
<td>—</td>
<td>12 oxygens</td>
</tr>
</tbody>
</table>

* W. P. Headden, analyst. Mean of 2 complete and 3 partial analyses.
† Since SiO₂ \( \leftrightarrow 2H₂O \), the sum of all positive ions should be 8 when 4H are considered equivalent to Si, provided no vacancies occur.

that \( \frac{1}{8} \) unit cell has 24 negative charges (12 oxygens) and the cationic charges are balanced in their proper ratios, 7.64 cations are indicated rather than 8. There is no good reason for assuming vacancies in the lattice, however, and it is fully as reasonable to believe that the ratios of the cations would be slightly modified by deduction of impurities, if these could be determined quantitatively.

Spectroscopic observations failed to indicate the presence of prominent lines of Si, As or V in the range 2250–3600 Å, indicating that these elements are not more abundant than traces. With the possible exception of CC₂, Headden’s analysis seems to be complete and little improvement could be expected in terms of a new analysis because it is not feasible to separate the minor impurities mentioned below.

Associated Minerals

Grifite resembles most garnets in the number and diversity of the included minerals. The following substances were observed as inclusions:

Hausmannite (?)—Deep red-brown to opaque in thin section. Some of the diffraction lines (Table 1) correspond with the more intense lines of hausmannite.
Pseudowavellite—Colorless; uniaxial (+); $\epsilon > n$ of graphite, $\omega = n$ of graphite; medium-low birefringence. Identification made certain by microchemical tests and comparison of x-ray pattern made from concentrated material with patterns of pseudowavellite from Fairfield, Utah, and from Bavaria (U.S.N.M. No. R4271).

Apatite or mangan-apatite (?)—Colorless; low relief in graphite; low birefringence; uniaxial (−). Similar material occurs as spherulites. Possibly accounts for F and/or Cl of Headden's analysis. Identified by optical methods only.

Quartz (?)—Colorless; high negative relief in graphite; low birefringence; uniaxial (+). Identified by optical methods only.

Amblygonite or montebrasite (?)—Colorless; low negative relief in graphite; moderate birefringence; occasionally with polysynthetic twinning. Possibly accounts for Li$_2$O reported in Headden's analysis. Identified by optical methods only.

All of these minerals are present in very small amounts except hausmannite which is frequently abundant. Two other substances occur as traces but these could not be identified. Diffraction lines of the identified substances, except hausmannite, were not found on the patterns prepared from selected samples of graphite.

In addition to the minerals occurring as inclusions, two specimens furnished by Dr. John C. Haff show considerable amounts of albite, quartz, and a white mica in association with the graphite. One of the unidentified substances occurring as inclusions resembles mica although its identification could not be confirmed.

It is noticeable that three of the contaminating substances are phosphates of one or more of the principal cations of graphite, and this suggests that the cationic ratios of Headden's analysis cannot be strictly representative of graphite. However, there is every reason to believe that they very closely approach the true ratios because the impurities apparently occur in very small amounts, if considerable care is taken to exclude the dark-colored material containing hausmannite. Headden's samples probably did not contain more than a few per cent of this material because they were carefully selected in this respect.

**Discussion**

The x-ray, chemical, physical, and optical data for graphite are all contributory to the conclusion that this mineral has a structure similar to that of garnets. There seem to be no theoretical objections to this and, consequently, graphite has been classified as one of the garnetoids. The properties of the garnetoids are summarized in Table 3.

The occurrence of isomorphous mixtures of the Ca-Fe- and Ca-Al-hydrogarnets with corresponding true garnets has been shown (3); plazolite (7) represents such an intermediate isomorphous compound. Manganese- and magnesium-berzelliite probably form a complete series of isomorphous mixtures but these "molecules" are not known to occur
### Table 3. Summary of the Properties of Garnetoids

<table>
<thead>
<tr>
<th>Composition</th>
<th>n</th>
<th>G</th>
<th>H</th>
<th>F</th>
<th>Solubility</th>
<th>Cell edge</th>
</tr>
</thead>
<tbody>
<tr>
<td>Griphite (Na, Al, Ca, Fe)₃Mn₅(PO₄)₂·₅(OH)₂</td>
<td>1.64–1.66</td>
<td>3.40</td>
<td>5½⁺</td>
<td>easy⁴⁺</td>
<td>sol. HCl</td>
<td>12.26 Å</td>
</tr>
<tr>
<td>Plazolite, Ca₃Al₂(SiO₄)₂(OH)₄</td>
<td>1.67⁵⁺</td>
<td>3.13⁺</td>
<td>64⁺</td>
<td>fusible</td>
<td>sol. HCl</td>
<td>12.14⁵⁺</td>
</tr>
<tr>
<td>Ca-Al-hydrogarnet*</td>
<td>1.60⁶⁺</td>
<td>2.52⁺</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>12.56⁶⁺</td>
</tr>
<tr>
<td>Sr-Al-hydrogarnet*</td>
<td>—</td>
<td>3.12⁺</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>13.02⁷⁺</td>
</tr>
<tr>
<td>Ca-Fe-hydrogarnet*</td>
<td>—</td>
<td>2.78⁺</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>12.74⁷⁺</td>
</tr>
<tr>
<td>Mn-berzeliite (Ca₂Na)Mn₂(AsO₄)₅</td>
<td>1.78¹⁺</td>
<td>4.45¹⁺</td>
<td>5¹⁺</td>
<td>3¹⁺</td>
<td>sol. acid</td>
<td>12.46¹⁺</td>
</tr>
<tr>
<td>Mg-berzeliite (Ca₂Na)Mg₂(AsO₄)₅</td>
<td>1.71¹⁺</td>
<td>3.95¹⁺</td>
<td>5¹⁺</td>
<td>3¹⁺</td>
<td>sol. acid</td>
<td>12.35¹⁺</td>
</tr>
</tbody>
</table>

* Compounds produced by synthesis, not known to occur naturally.

isomorphously mixed with any other garnetoid or garnet. Nothing is known regarding the miscibility of griphite with garnets and other garnetoids. However, analyses of garnets occasionally indicate the presence of small amounts of Na₂O and, rarely, very small amounts of P₂O₅.

A similar compound, originally described as triplite but later referred to griphite, from near Rapid City, South Dakota, was analyzed by L. G. Eakins (8). The analysis shows a slightly higher value for P₂O₅, slightly less water, considerably more fluorine and 2.36 per cent of Fe₂O₃. In general, however, this analysis is conformable with Headden's results and confirms the composition assigned to griphite.

Eakins' analysis reports about 1/4 of 1 per cent of CO₂. The fact that specimens studied in the present investigation were observed to liberate gas when treated with acid suggests that a small amount of CO₂ was overlooked by Headden.

Carbon dioxide is known to enter the lattice of apatite (9) and there is no theoretical reason which prohibits its occurrence in the structure of griphite. In the present investigation observations made with the microscope revealed the liberation of gas during treatment with dilute H₂SO₄ from portions of the griphite that were completely isotropic. It can be said with certainty that neither calcite nor aragonite was present.

**Summary**

Griphite is cubic and its structure is similar to that of garnets, i.e., it closely simulates a structure based on a body-centered cubic lattice, although the true symmetry is probably lower than that of garnets.

The structural formula of griphite is \(8[(Na,Al,Fe)₃Mn₂(PO₄)₃·5(OH)]\), which correlates with the type formula \(X₃Y₂(ZO₄)₃·m(OH)\₄\), the latter being a variation of the formula \(X₃Y₂(ZO₄)₃\).

The properties of the garnetoids (substances other than simple silicates having a structure similar to garnets) have been assembled and compared. Pseudowavellite is reported from a second American locality. Its identification was confirmed by x-ray and microchemical methods.

Schorlomite is classified as a true garnet rather than a garnetoid because the substitution encountered here does not produce any significant changes in the powder diffraction pattern. Lattice dimensions are given for schorlomite (12.09 Å) and for uvarovite (11.97 Å) in addition to griphite (12.26 Å).

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