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CHEMISTRY OF FOSSIL FUEL

Homogeneous Pyrolysis of 1,4-Dimethoxynaphthalene

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Abstract—Homogeneous pyrolysis of 1,4-dimethoxynaphthalene was studied. The yield and group composition of liquid products and the component compositions of the gaseous products and hydrocarbons were determined; the asphaltenes and neutral oxygen-containing compounds were characterized. The kinetic parameters were determined, and possible mechanisms suggested, for decomposition of 1,4-dimethoxynaphthalene with the formation of CH_4, H_2, CO, and CO_2.

The need to study high-temperature transformations of compounds of strictly definite classes and, especially, of individual components corresponding to fragments of the organic matter of various caustobioliths and making the major contribution to formation of liquid products was convincingly substantiated in [1–16].

The goal of this study was to analyze the features of homogeneous pyrolysis of 1,4-dimethoxynaphthalene (DMN), determine the group composition of liquid products and the component compositions of the gaseous products and hydrocarbons, characterize asphaltenes and neutral oxygen-containing compounds (NOCs), and calculate the kinetic parameters of decomposition of 1,4-DMN and formation of CH_4, H_2, CO, and CO_2.

Pyrolysis of 1,4-DMN was performed on an installation described in [17]; the group composition of the liquid products was determined according to [18].

Data on the material balance of 1,4-DMN pyrolysis (Table 1) show that the thermal stability of this compound is relatively low. As the pyrolysis temperature was increased from 700 to 800°C and the pyrolysis time, from 1.5 to 4.5 s, the yield of liquid products decreased from 53.4 to 17.3 wt %, yield of gaseous products increased from 24.7 to 37.4 wt %, and yield of pyrocarbon increased from 21.2 to 44.3 wt % (based on the feed). The content of “crude benzene” was low and did not exceed 2.6 wt % (800°C, 3 s), which is apparently due to the lack of a simple route from a system of two fused aromatic rings to mononuclear aromatic hydrocarbons.

It should be noted that variation of temperature at fixed pyrolysis time affects the product yields less strongly than variation of the pyrolysis time. For example, as the pyrolysis temperature is raised from 750 to 800°C, at fixed pyrolysis times of 1.5 and 4.5 s, the yield of liquid products decreases by 8.5 and 8.8 wt %, respectively, whereas an increase in the pyrolysis time from 1.5 to 4.5 s causes a decrease in the yield of liquid products by 19.9 and 20.2 wt %, respectively.

Table 1 shows that, in the initial steps of 1,4-DMN pyrolysis (1.5 s), CO makes a half of the gaseous pyrolysis products (54.0 vol % at 700°C and 50.1 vol % at 750°C). Methane and CO_2 are formed in comparable amounts. As the pyrolysis temperature and time are increased, the content of CO decreases from 54.0 to 36.4 vol %, with the yield of hydrogen growing by a factor of 5 (from 4.6 to 23.2 vol %). Hence, with increasing temperature and time of pyrolysis, the relative contribution of condensation processes, occurring in parallel with the degradation to CO, CO_2, and CH_4, increases. The condensation processes can be monitored by formation of hydrogen and pyrocarbon. The yield of ethene decreases from 11.2 (700°C, 1.5 s) to 0.9 vol % (800°C, 4.5 s). This is apparently due to its participation in condensations by the Diels–Alder mechanism, more active under more severe conditions.

The tendency of 1,4-DMN to undergo condensations is confirmed by the high yield of asphaltenes, varying from 33.5 to 61.6 wt % based on the liquid products. The asphaletene structures are insufficiently

Table 1. Material balance of 1,4-DMN pyrolysis

Pyrolysis product	Yield at indicated pyrolysis temperature, °C								
	700			750			800		
	and time, s								
	1.5	3.0	4.5	1.5	3.0	4.5	1.5	3.0	4.5
Liquid products, wt % based on 1,4-DMN	53.4	41.5	35.0	46.0	35.0	26.1	37.5	24.9	17.3
Groups of compounds, wt % based on liquid products:									
phenols	20.5	16.3	2.8	14.2	3.0	1.8	3.0	2.4	1.3
hydrocarbons	5.5	13.5	22.7	11.2	25.2	36.4	16.2	33.7	42.2
neutral oxygen-containing compounds	9.5	6.2	3.7	6.6	4.7	2.2	5.2	3.9	1.4
asphaltenes	33.5	43.1	58.4	44.5	48.4	51.2	61.6	54.5	52.1
resinous substances	28.0	20.9	12.4	23.5	18.7	8.4	14.0	5.5	3.0
Pyrocarbon, wt % based on 1,4-DMN	21.2	26.5	30.0	24.8	30.5	36.7	28.1	37.2	44.3
Natural gas, wt % based on 1,4-DMN	24.7	31.0	33.5	28.2	33.2	35.5	3.1	35.3	37.4
Components of pyrolysis gas, vol %:									
H_2	4.6	7.2	9.4	11.2	16.0	18.9	15.0	17.3	23.2
CH_4	17.0	20.1	21.8	16.4	18.6	19.3	16.5	18.5	19.1
CO	54.0	50.1	49.2	50.1	44.3	42.8	44.1	40.6	36.4
CO_2	11.8	14.5	13.8	15.8	17.5	16.0	19.4	20.9	18.2
C_2H_4	11.2	6.6	5.2	6.0	2.4	1.3	2.7	1.7	0.9
Crude benzene, wt % based on 1,4-DMN	0.7	1.0	1.5	1.0	1.3	1.7	1.3	2.6	1.0

stable: With the pyrolysis time increasing from 1.5 to 4.5 s at 700–750°C, asphaltenes tend to accumulate, whereas at 800°C their relative content decreases from 61.6 to 52.1 wt %, with the yield of pyrocarbon growing simultaneously (Table 1).

The second most abundant group of liquid products is that of hydrocarbons. Their amount increases from 5.5 (700°C, 1.5 s) to 42.2 wt % (800°C, 4.5 s).

The yield of phenols is substantial only at 700 (1.5–3.0 s) and 750°C (1.5 s), amounting to 20.5, 16.3, and 14.2 wt %, respectively, but it decreases dramatically (to 1.3 wt %) at 800°C (4.5 s).

The content of NOCs, the main of which is unchanged 1,4-DMN, in liquid pyrolysis products is lower: Their yield decreases from 9.5 (700°C, 1.5 s) to 1.4 wt % (800°C, 4.5 s).

When studying thermochemical transformations of various compounds, it is important to obtain detailed data on the qualitative and quantitative composition of the products, in order to elucidate the main pyrolysis pathways, reveal its features, and find the best conditions for obtaining useful products in the highest yields.

The hydrocarbon fraction of liquid products of 1,4-DMN pyrolysis was studied by GLC. The yields of its components are listed in Table 2.

The major components of the hydrocarbon fraction are naphthalene, diphenyl, fluorene, and phenanthrene. The content of 2- and 1-methylnaphthalenes, pyrene, and chrysene is also high. A characteristic feature of the pyrolysis of 1,4-DMN is that the hydrocarbon fraction contains no anthracene.

The content of naphthalene, methylnaphthalenes, dimethylnaphthalenes, and acenaphthene decreases as the pyrolysis conditions are made more severe. At the same time, the content of more condensed components, such as phenanthrene, fluoranthene, pyrene, and chrysene, increases.

Table 2. Yields of components of the hydrocarbon fraction of liquid products formed in pyrolysis of 1,4-DMN

Hydrocarbon	Yield $c \times 10^2$ (wt %) based on the feed, at indicated pyrolysis temperature, °C								
	750			800			850		
	and time, s								
	1.5	3.0	4.5	1.5	3.0	4.5	1.5	3.0	4.5
Naphthalene	48.46	117.66	150.96	104.07	151.70	106.94	78.98	93.98	53.30
2-Methylnaphthalene	8.81	14.57	17.48	10.30	12.35	12.30	7.90	9.23	5.84
1-Methylnaphthalene	20.85	36.42	45.29	22.67	26.46	22.71	16.40	15.10	5.84
Diphenyl	21.15	34.18	42.90	44.82	56.45	56.78	39.49	60.42	69.36
2,6- and 2,7-Dimethylnaphthalenes	3.82	5.60	5.56	3.09	7.06	5.68	3.65	4.20	3.65
2,3- and 1,5-Dimethylnaphthalenes	4.11	6.16	5.56	3.09	8.82	8.52	6.08	7.55	5.11
Acenaphthene	2.06	2.80	2.38	2.06	2.65	1.89	1.82	2.52	1.46
Fluorene	9.40	21.85	37.75	20.01	44.98	54.89	30.38	47.00	45.27
Phenanthrene	9.40	28.02	68.33	48.43	106.72	159.00	84.44	146.84	157.70
Fluoranthene	1.47	4.48	7.15	4.64	17.64	24.61	16.40	25.17	23.36
Pyrene	3.82	5.53	16.69	7.73	18.52	30.29	13.97	26.01	28.47
Chrysene	3.27	8.97	25.42	7.73	26.46	4.48	22.48	41.12	39.43

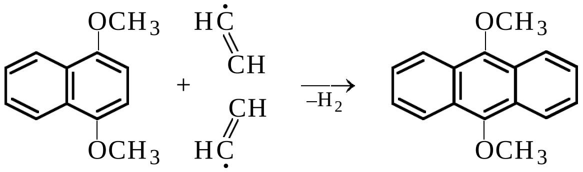
Table 3. Parameters of asphaltenes and neutral oxygen-containing compounds formed in pyrolysis of 1,4-DMN

T, °C	τ, s	Parameter
		Asphaltenes
700	4.5	*M* 226, C 91.60, H 5.69, O 2.71, DA 0.784, $C_{20.30}H_{12.16}O_{0.45}$, QG 0.73, AG 0.44, O_c 0.32, IN 0.21
750	1.5	*M* 250, C 92.20, H 5.65, O 2.15, DA 0.791, $C_{19.21}H_{14.12}O_{0.34}$, QG 1.07, AG 0.03, O_c 0.44, IN 0.13
	3.0	*M* 263, C 92.50, H 5.60, O 1.90, DA 0.797, $C_{20.27}H_{14.73}O_{0.31}$, QG 0.83, AG 0.24, O_c 0.27, IN 0.28
	4.5	*M* 291, C 92.90, H 5.54, O 1.56, DA 0.800, $C_{22.53}H_{16.12}O_{0.28}$, QG 0.47, AG 0.72, O_c 0.56, IN 0.14
800	1.5	*M* 271, C 93.00, H 5.50, O 1.50, DA 0.810, $C_{22.00}H_{19.91}O_{0.25}$, QG 0.61, AG 1.43, O_c 0.55, IN 0.29
	3.0	*M* 305, C 93.30, H 5.48, O 1.22, DA 0.810, $C_{23.71}H_{16.71}O_{0.23}$, QG 0.72, AG 0.56, O_c 0.23, IN 0.16
	4.5	*M* 326, C 94.20, H 5.44, O 0.36, DA 0.817, $C_{25.59}H_{17.72}O_{0.07}$, QG 0.33, AG 0.63, O_c 0.85, IN 0.42
		Neutral oxygen-containing compounds
700	4.5	*M* 181, QG 0.37, AG 0.33, O_c 0.33, IN 0.33
750	1.5	*M* 175, PG 0.07, QG 0.69, AG 0.69, O_c 0.25, IN 0.13
	3.0	*M* 200, PG 0.25, QG 0.43, AG 0.71, O_c 0.70, IN 0.17
	4.5	*M* 214, PG 0.14, QG 0.05, AG 0.85, O_c 0.36, IN 0.07

Note: (*M*) molecular weight; (C, H, O) content of carbon, hydrogen, and oxygen, respectively, wt % daf; (DA) degree of aromaticity, (PG) phenolic groups; (QG) quinoid groups; (AG) alkoxy groups; (O_c) oxygen in heterocycles; and (IN) iodine number, g-equiv mol^{-1}.

For asphaltenes, the molecular weight and elemental and quantitative functional compositions were determined (Table 3).

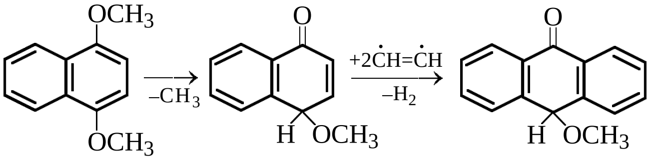
An increase in the molecular weight of asphaltenes from 250 to 326 in the examined range of parameters of 1,4-DMN pyrolysis indicates intensification of condensation processes. The high content of carbon (91.6–94.2%) and low content of hydrogen (5.44–5.65%) and oxygen (0.36–2.71%), in combination with the high molecular weight, point to the predominantly aromatic character and high degree of condensation of asphaltenes and to the relatively low content of oxygen-containing functional groups in them. The major functional groups of asphaltenes are alkoxy, quinoid, and oxygen-containing heterocyclic groups. The content of methoxy groups increases from 0.03 to 0.72 g-equiv mol^{-1} as the time of pyrolysis at 750°C is increased from 1.5 to 4.5 s. At 800°C and pyrolysis time of 3 s, their content reaches a maximum (1.56 g-equiv mol^{-1}) and then decreases to 0.63 g-equiv mol^{-1} at pyrolysis time of 1.5 s. This may be due to attack on the 1,4-DMN molecule by radical fragments generated in the initial stage of degradation, which results in incorporation of methoxy groups in asphaltene structures:



As the residence time of the feed in the heated zone is increased, methoxy groups have time to decompose not only in 1,4-DMN, but also in asphaltenes, and their content decreases.

The content of quinoid groups tends to gradually decrease, which is due to their very high lability. It is important that quinoid groups, identified previously in asphaltenes formed by pyrolysis of a number of phenols [1–9] were also detected in asphaltenes formed by pyrolysis of 1,4-DMN, with the phenolic hydroxy groups being absent.

In view of this fact, we can suggest the following scheme of accumulation of quionid groups in asphaltenes in 1,4-DMN pyrolysis:



Heterocyclic oxygen can be formed as follows:

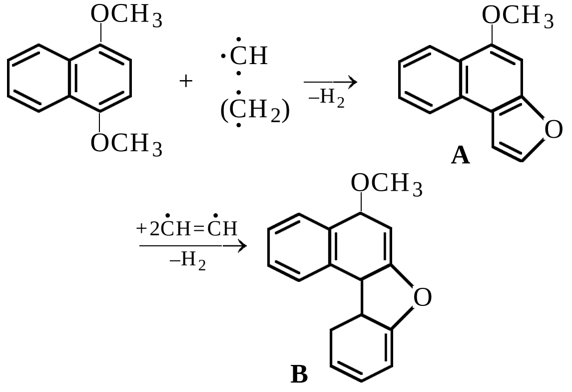


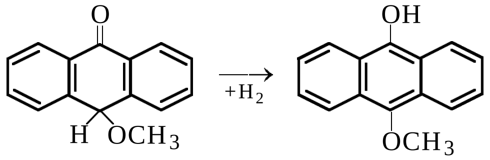
Table 4. Kinetic equations of overall decomposition of 1,4-DMN and formation of particular gaseous pyrolysis products*

Process	K
Overall decomposition of 1,4-DMN	$1.10 \times 10^7 \exp(-123\,186/RT)$
Formation of:	
H_2	$2.63 \times 10^4 \exp(-79\,191/RT)$
CH_4	$3.31 \times 10^6 \exp(-52\,794/RT)$
CO	$2.58 \times 10^7 \exp(-159\,639/RT)$
CO_2	$2.94 \times 10^7 \exp(-162\,153/RT)$
C_2H_4	$2.36 \times 10^7 \exp(-27\,654/RT)$

* Reaction order 1.5, except the formation of CO_2 (reaction order 2.0).

The minimum in the content of heterocyclic oxygen, attained in 3 s and followed by its growth (4.5 s), is apparently due to easy transformation of unstable heterocycles of type **A** into more condensed and more stable structures of type **B**. Naturally, this multistage process must take certain time; therefore, the highest content of heterocyclic oxygen is reached in 4.5 s.

As compared to asphaltenes, NOCs have lower molecular weight (175–236). Their characteristic feature is the presence of small amounts of phenolic hydroxy groups (0.07–0.25 g-equiv mol^{-1}), reaching a maximum in 3 s. This is apparently due to their formation by reduction of the quinoid groups under the conditions when the concentration of these groups becomes high; in addition, increased concentrations of hydrogen are detected:



The content of the other functional groups is, on the whole, two times lower than that in asphaltenes.

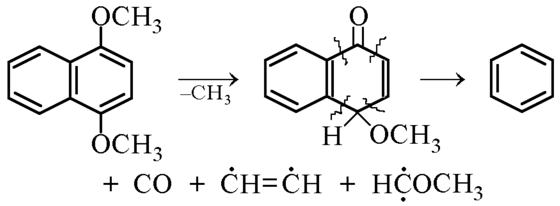
The data on material balance, group composition, and component composition of the gaseous and liquid pyrolysis products were used to calculate the kinetic characteristics of the process (Table 4).

The overall decomposition of 1,4-DMN is described by an equation of the order of 1.5, with the activation energy E_a = 123.35 kJ mol^{-1}. The relatively low value of E_a of the overall decomposition process is apparently due to a significant contribution of radical reactions occurring with low energy barrier. The activation energy is considerably lower than that of pyrolysis of naphthalene (278.64 kJ mol^{-1} [19]), α-naphthol (233.80 kJ mol^{-1} [7]), and 1,4-dimethylnaphthalene (159.09 kJ mol^{-1} [20]). Thus, methoxy groups destabilize the naphthalene core to a greater extent than phenolic hydroxyls and methyl groups do. The destabilizing effect of two methoxy groups is considerably stronger than that of a single methoxy group, so that E_a of 1,4-DMN decomposition is closer to that of 1,4-dimethylnaphthalene decomposition than to that of decomposition of compounds with a single functional group. The effect of oxygen atoms is stronger than that of methyl groups.

To elucidate the reaction mechanism and deduce the kinetic equations allowing control over formation of individual compounds with the use of an appropriate mathematical model of the process, it is of large interest to calculate the kinetic characteristics of particular pathways of the complex pyrolysis process.

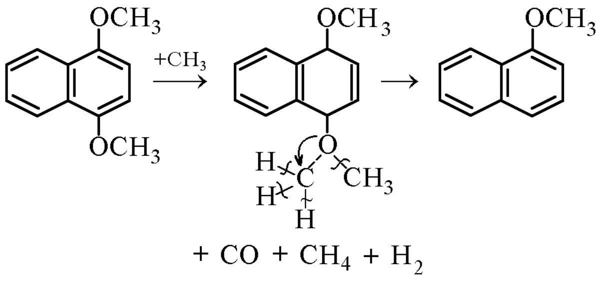
Accumulation of CO follows a kinetic equation of order 1.5; E_a of this process is 159.64 kJ mol^{-1}. This activation barrier is two times lower compared to formation of CO from α-naphthol, which is probably due to a greater contribution of radical reactions to elimination of CO.

Transformation of 1,4-DMN into quinoid compounds must involve initiating demethylation. In this connection, we suggest the following scheme of CO formation. The first stage is initiation:



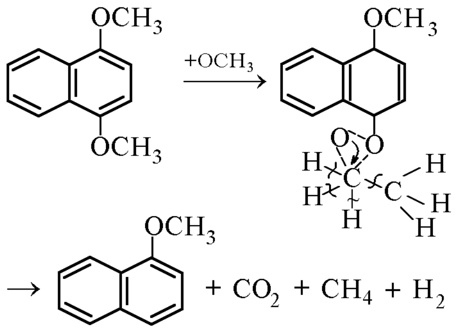
This process generates a large number of radical products intensifying the cleavage of the 1,4-DMN molecule. The intermediate formation of the quinoid structure is confirmed by identification of quinoid groups in pyrolysis products, e.g., in asphaltenes and NOCs.

Second stage:



A calculation of the theoretical E_a, assuming additivity of the bond energies in the activated complex [21], gave a value of 155.03 kJ mol^{-1}, in good agreement with the experimental value of 159.64 kJ mol^{-1}. This confirms the validity of the suggested scheme of the process. An additional evidence in favor of the presence of a second bimolecular initiating stage in the formation of CO is the reaction order of 1.5, since in the monomolecular cleavage of the ring in phenolic compounds it is equal to 1.0.

CO_2 formation is characterized by a similar activation energy (162.15 kJ mol^{-1}, Table 4), which suggest a kinetic relationship between these two processes, or similar mechanisms of formation of CO and CO_2. In this connection, we assume the following scheme: The first stage is initiation, similar to that in CO formation; the second stage is



The calculated activation energy of CO_2 formation is 159.22 kJ mol^{-1}, which is nicely consistent with the experimental value of 162.15 kJ mol^{-1}; this fact supports the mechanism suggested. For anisole, E_a of CO_2 formation is 206.15 kJ mol^{-1}, i.e., it is higher than that for 1,4-DMN [8]. This is a manifestation of the destabilizing effect of two methoxy groups.

The relatively low energy barrier of methane formation (E_a 52.79 kJ mol^{-1}) is most probably due to the radical recombination mechanism of its formation, from both demethylation products and radical fragments of cleaved aromatic rings. This process is described by a kinetic equation of order 1.5.

The above schemes of CO and CO_2 formation involve formation of methane as a by-product. A calculation of the effective activation energy of methane formation by the method of additivity of the bond energies in the activated complex gave a value of 56.57 kJ mol^{-1} (with account taken of the demethylation stage); the experimental value is 52.79 kJ mol^{-1}. This indicates that the actual mechanism of methane formation is similar to that suggested here, with the only difference that the contribution of the less energy-consuming demethylation process is somewhat higher (in calculations, it was assumed to be equal to the contribution of the ring cleavage).

In the case of naphthalene and α-naphthol, the activation energy of methane formation is higher by almost a factor of 5 (E_a 239.67 and 256.85 kJ mol^{-1}, respectively), which reflects the fact that, for these compounds, the only pathway of methane formation is energy-consuming cleavage of the aromatic ring.

Ethene is formed with a still lower activation energy (E_a 27.65 kJ mol^{-1}); the reaction order is 1.5. This is facilitated by the presence of C_2 radical fragments (e.g., $\dot{\mathrm{C}}\mathrm{H}{=}\dot{\mathrm{C}}\mathrm{H}$) in the stage of initiation of ring cleavage with the formation of CO. The C_2 radical fragments recombine with hydrogen present in the reaction zone to give ethene:

$$\dot{\mathrm{C}}\mathrm{H}{=}\dot{\mathrm{C}}\mathrm{H} + \mathrm{H_2(2H)} \longrightarrow \mathrm{CH_2{=}CH_2}$$

Such radical recombination reactions have zero activation energy, which is consistent with the low activation energy of the overall process of ethene accumulation in the pyrolysis zone. The very low preexponential term, equal to 2.36, supports the presumed formation of ethene from radical fragments like $\dot{\mathrm{C}}\mathrm{H}{=}\dot{\mathrm{C}}\mathrm{H}$ or by recombination of methyl radicals, followed by dehydrogenation, since the probability of radical recombination under conditions of dilution with an inert gas (1 : 25 by volume, neglecting additional dilution with gaseous and vaporous pyrolysis products) is very low. As a result, the concentration of ethene is low in most cases, although, in principle, it could be high.

Hydrogen can be formed by a number of pathways, including condensation of the initial 1,4-DMN molecules and radical fragments from degradation processes. The hydrogen formation is satisfactorily described by a kinetic equation of order 1.5, with $E_a = 79.19$ kJ mol^{-1} (Table 4). For 1,4-dimethylnaphthalene, this value is higher by almost a factor of 2 (159.22 kJ mol^{-1}), and for α-naphthol [7] and naphthalene [19] it is still higher (269.84 and 349.87 kJ mol^{-1}, respectively), which is indicative of the large contribution of molecular condensation reactions for these compounds.

The activation energy of hydrogen formation from anisole, 98.88 kJ mol^{-1} [8], proved to be the closest to that of hydrogen formation from 1,4-DMN.

CONCLUSIONS

(1) The thermal stability of 1,4-dimethoxynaphthalene is relatively low; its noticeable decomposition starts at 700°C. The major liquid products are asphaltenes and hydrocarbons. The major gaseous product is CO.

(2) The hydrocarbon fraction consists of naphthalene, methyl- and dimethylnaphthalenes, phenanthrene, fluoranthene, pyrene, and chrysene. The molecular weight of asphaltenes varies within 250–326, and that of neutral oxygen-containing compounds, within 175–236. The main functional groups are quinoid, alkoxy, and oxygen-containing heterocyclic groups.

(3) The kinetic parameters of overall decomposition of 1,4-dimethylnaphthalene and formation of particular gaseous products were calculated; the mechanisms of formation of these products were suggested.

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