

WATER EXCHANGE OF POLYAMIDE MATERIALS AND LINK TO MECHANICAL PROPERTIES

Summary

This white paper is intended as background information for the Moisture Diffusion Tool found on Envalior's online tool page.

Moisture diffusion in polyamides depends on the surrounding humidity and temperature. Polyamides absorb a certain amount of moisture and this affects the material's glass transition temperature, as well as the diffusion speed. Moisture uptake and release measurements validate that our online model can describe both the absorption and desorption speed accurately.

Eventually, these processes are reflected in the mechanical properties of the materials. The practical examples found in this white paper show how you can use our online tools to your benefit.



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Polyamides & Moisture

Polyamide materials have a semi-crystalline nature under application conditions—they consist of a crystalline phase and an amorphous phase. The crystalline phase is not able to take up water and is considered an obstacle for water migrating through the polyamide sample. The amorphous phase can absorb water and the migration of water occurs in this amorphous phase. The relative amount of crystalline phase depends on the type of polyamide and on the way the polyamide is processed. In shaping processes such as injection or blow molding, fast cooling from the melt to ambient conditions will typically lead to lower levels of crystallinity compared to slower cooling. Post-process thermal annealing may trigger additional crystallization leading to higher levels of crystallinity. Since the crystalline phase does not absorb water, typically higher levels of crystallinity will lead to lower equilibrium water contents. The moisture diffusion model allows you to make predictions of two extreme situations:



- A situation for a polymer sample directly after its shaping process (relative low crystallinity level and hence a relative high equilibrium water uptake).
- A situation for a polymer sample that was exposed to an additional thermal treatment triggering additional post crystallization (relative high crystallinity level and hence a relative low equilibrium water uptake).

EQUILIBRIUM WATER SOLUBILITY

The equilibrium water solubility varies when different polyamides are compared with each other. The general trend is that the equilibrium water solubility increases with the polarity of the polyamide. PA410 is less polar compared to PA6 and PA66 while these two polyamides are less polar compared to PA46. Along this series we see an increase of equilibrium water solubility: PA410 < PA6/PA66 < PA46.

For a given polyamide material, its equilibrium water solubility is mainly determined by the relative humidity of the surrounding atmosphere. The equilibrium water solubility of a polyamide material exposed to a complete dry atmosphere (0% relative humidity (RH)) is zero; the water solubility of a polyamide material exposed to a complete wet atmosphere (100%RH) is maximum. Intermediate relative humidity values will lead to intermediate solubility values. The water uptake and water release processes are reversible. Switching from an atmosphere with a low RH with a corresponding low water solubility to an atmosphere with a high RH will lead to a higher water solubility. Switching back to an atmosphere with the low RH will lead again to the same low water solubility. In Figure 1, examples are shown of water solubility versus RH of different polyamides at room temperature. Equilibrium water uptake for polyamides is only slightly dependent on temperature. An increase of temperature will lead to a slightly lower equilibrium solubility. Expressions dealing with the equilibrium moisture as a function of relative humidity and temperature are part of the moisture diffusion tool.

Figure 1

a) Equilibrium moisture content at 23°C as function of humidity for Akulon® K224–HG6 (PA6), Stanyl® TW200F6 (PA46) and Akulon® S223– HG6 (PA66), all containing 30wt% glass fiber reinforcement and b) equilibrium moisture concentration of Akulon® S223–HG6 (PA66) as function of humidity for 23°C and 70°C.



EFFECT OF FILLERS LIKE GLASS FIBERS

Commercial polyamide grades often contain glass fibers to improve the mechanical performance of the materials. These glass fibers act as impermeable objects for water, which has two consequences. First of all, the equilibrium water uptake will be reduced compared to a sample without glass fibers simply because glass cannot absorb water. Secondly, the diffusion speed of the water will be reduced due to the presence of glass fiber. Since glass fibers are impermeable, diffusing water has to migrate around the glass fiber obstacles leading to a slowing down of the diffusion process. These effects are accounted for by the model at hand.

GLASS TRANSITION TEMPERATURE

The glass transition temperature (Tg), which marks the transition between the glassy and rubbery state, depends on the amount of water that is present in the polyamide material. In Figure 2, some examples are given for the Tg of PPA polyamides. For dry MX51 material the Tg is 155°C.

However, the same material exposed to a wet atmosphere may contain 3.4 wt% water, leading to a much lower Tg of 65°C.

Figure 2

Example of the resulting Tg values for ForTii® MX1, ForTii® MX3, ForTii® MX51 and ForTii® MX53, all PPA grades.

Tg (DMTA) verses moisture content



DIFFUSION Behavior

DIFFUSION SPEED

In the above section, water equilibrium solubility values are discussed that are reached for extremely long exposure times. You can imagine that when switching from a low to a high RH atmosphere, it takes a while before this new equilibrium value is reached. Water migrates in the sample by a diffusion process. The diffusion process can be slow or fast, leading to different times for reaching the new equilibrium water solubility value. The higher the temperature, the faster the diffusion process will be. It is easy to imagine that sample thickness also affects this time. For thicker samples, the water diffusion process will take longer for water to reach all parts of the sample, leading to a longer time to reach this equilibrium value for the complete sample.

In the example below, a dry polyamide plaque of ForTii[®] MX51 glass fiber filled polyphtalamide (PPA) material with a thickness of 3 mm is exposed to a completely wet atmosphere (RH=100%) at T=100°C. The water uptake versus the square root of time is displayed in Figure 3a. It takes approximately 100 hrs (SQRT(time)=10 hrs^{3/2}) before the equilibrium water solubility of 3.8 wt% is reached. Subsequently, the same sample was again exposed to a completely dry atmosphere (RH=0%) at the same

Figure 3a/b

Moisture uptake (left) and moisture release (right) as function of time for ForTii® MX51 (PPA GF30) at 100°C.





MX51, desorption at 100°C, 0% RH

temperature. The water exchange process is reversible as can be observed in Figure 3b.

However, the remarkable observation is that drying out the sample takes much longer than the water uptake process. The drying process takes more than 1000 hours (SQRT $(time) > 31.6 hours^{\frac{1}{2}})$ to complete and hence is more than a factor 10 slower.

The explanation is related to the state of the polyamide, which can be either glassy or rubbery. Below the line in Figure 2, the polyamide material is in its glassy state, for which the diffusion process of water is relatively slow. Above the line in the graph, the material is in the rubbery state with a relatively fast diffusion process.

In Figure 4, the 3 mm thick polyamide sample is displayed schematically. The first image is representative for an intermediate time during the water uptake process at T=100°C. Water has started to diffuse into the sample leading to the green intermediate water solubility line.

Figure 4

Schematic representation of the glassy (dark blue) and rubbery (yellow) regions inside a polyamide plate during (left) moisture absorption and (right) moisture desorption. The green profile indicates the amount of moisture present as function of the plate's width. Bottom: photograph of testing plates with various colors.



The water has not yet reached the central part of the sample, but the outer parts of the sample contain already certain amounts of water. This leads to a situation for which the outer parts of the material are rubbery (high water solubility and fast diffusion) at the given temperature while the central part of the material (zero or low water solubility) is still glassy. Water diffusion in the outer rubbery parts is a fast process and will "eat away" the glassy central layer over time.

The second image represents the situation at an intermediate time in the drying process at the same temperature. The outer layers have already been dried out to a certain extent while the central part still contains relatively high amounts of water. Consequently, the outer layers have become glassy while the central part is still rubbery. This results in a relatively slow release of the water still present in the central part since to reach the outside dry atmosphere, it must diffuse through the glassy outer layers. Diffusion in these outer layers will be slow due to the glassy character. The water in the central part is encapsulated between glassy layers preventing fast release to the outside atmosphere leading to a much slower drying out process compared to the water uptake process.

These specific diffusion features are captured in an advanced non-Fickian diffusion model that is part of the "under-the-hood" machinery of the moisture diffusion tool.

WATER EXCHANGE MODEL



As described in the above paragraphs, water exchange in polyamide parts relates to:

- The equilibrium water solubility value that strongly links to the relative humidity of the external atmosphere. Upon changing the external atmospheric conditions to more wet or dry conditions, this equilibrium water solubility value is the end value that is reached after a certain time.
- The water diffusion speed determines the time to reach the equilibrium water solubility. The diffusion process will lead to certain water concentration profiles over the thickness of the part.

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A model has been created to predict the water exchange process as a function of time for polyamide parts that have a plate—like geometry with a certain thickness. In this model, the factors governing the equilibrium water solubility value and the diffusion speed are incorporated. The model allows calculation of the water uptake during a wetting— or drying treatment. Overall water solubility of the part can be predicted as well as water concentration profiles during different time intervals during the process. The model is also capable of dealing with a plate that has two different atmospheres with different relative humidities on both sides of the plate-like sample. Figure 5 shows an example with 100% humidity on the left side and 0% on the right side. The blue line is the steady-state situation. Please note that the concentration profile under these conditions is not linear since the water diffusion coefficient is a function of the local water concentration.

Figure 5

Moisture concentration profile as function of plate thickness for an asymmetric boundary condition (K224–HG6).



ACCELERATED CONDITIONING



In most datasheets stress–strain curves for two conditions are provided:

- Dry as moulded: < 0.2wt% water
- Conditioned: the amount of water that is reached by accelerated conditioning by storing the sample in an environment of 70°C, and 62%RH until equilibrium is reached (acc. ISO1110).

Note that the conditions of 70°C and 62%RH are used to achieve equilibrium in a reasonable time frame. For a 4 mm thick tensile bar of 30 wt% glass fiber filled PA6 material Akulon® K224–HG6, this procedure will take 4–5 weeks, at these conditions. The modelled water uptake under these conditions is presented in Figure 6 by the dark blue line. At a temperature of 23°C and 50%RH (standard laboratory conditions), the speed of diffusion of moisture in polyamides is relatively slow, and the process to reach equilibrium will take many months (green line in Fig.6). In the end the equilibrium amount of moisture reached in the accelerated conditioning will be in a similar range (between 2 and 2.5 wt%) compared to the one achieved at 23°C and 50%RH but at practical timescales. Hereafter we will refer to this moisture state as 'conditioned'.



Figure 6

Modelled moisture concentration for K224–HG6 (right) as function of time for two different conditioning scenarios and (left) a photograph of one of the conditioning chambers.



MOISTURE ABSORPTION MEASUREMENTS

During the conditioning (acc. ISO1110) the weight of the tensile bars (type ISO527-1A) of a PA6 material is measured over time (see Figure 7a). The increase in weight over time reflects the diffusion uptake of water in the polyamide. After 816 hours the weight is constant, and the parts were stored in a sealed bag at room temperature until testing. The equilibrium uptake of moisture is 3.2wt%. In addition, we have calculated this case with the diffusion model explained in the first section of this paper. As the ISO527-1A specimens cannot be treated as an infinite plate, we correct the calculation by considering the volume and surface area of the tensile bar. Based on these a representative thickness can be calculated and is used in the simulation. As stated in the introduction the simulation considers two states regarding the crystallinity of the specimen. As only the amorphous part of the polyamide

can take up moisture, the level of crystallinity influences the amount of absorbed water. The first state considers a relative low degree of crystallinity (curve labeled 'asmoulded in Figure 7a), in the second state we assume a higher degree of crystallinity, which can occur when a part during its life is exposed to temperatures above its Tg for longer periods of time (curve labeled 'annealed' in Figure 7a).

The transient response during the first 150 hours matches quite well. The equilibrium value is overpredicted by 7% for the 'as-moulded' result and by 2% for the 'annealed' result.

Figure 7a/b

Moisture uptake in wt% for an ISO5271A tensile bar of Akulon® K222–D (left). At the start of the process the bar contains 0% moisture and is stored in a conditioning chamber with a temperature of 70°C, and a relative humidity of 62%. The diffusion model predictions are shown for comparison (for both 'as moulded' and 'annealed' states. Moisture in wt% for an ISO527–1A tensile bar of Stanyl® TW200F6 (PA46 with 30wt% glass fiber) (right). At the start of the process the bar contains 0% moisture and is stored in water at a temperature of 70°C. The diffusion model predictions are shown for comparison ('as moulded' state).







Figure 7b shows the uptake of moisture for ISO527–1A specimens of PA46–GF30, held in water of 70°C. Here we also added the curve calculated with our moisture diffusion model (in 'as moulded' state). The overall correlation between measurement and prediction is very good. From

this curve it is also clear that the specimens were not able to reach the equilibrium condition. After the conditioning to each moisture level, the tensile bars were packed in sealed bags until the tensile tests were conducted. We will consider the tensile results in section 10.

Akulon® K222–D; Conditioning @ 70°C / 62 % RH (acc. ISO 1110)

APPLICATION EXAMPLES OF MOISTURE DIFFUSION TOOL

The moisture diffusion tool can be used to predict water exchange of polyamide parts under application conditions. For the application examples given below, the moisture exchange modelling predictions are performed for an infinite plaque of a certain thickness.

The first example deals with the water uptake and water release of a plaque of ForTii® MX51 glass fiber filled polyphtalamide (PPA) material with a thickness of 3 mm. The experimental results and the interpretation of these results have already been discussed in paragraph 5. In this section, the modelling predictions are presented. The plaque, that was initially dry, is exposed at T=100°C to a wet atmosphere and water uptake is predicted. After the water uptake has reached a constant value, the plaque is dried out at T=100°C against a dry atmosphere. The modeling results are presented in Figure 8 with water content displayed against uptake and drying time. The model is capable of predicting the fact that water uptake

is much faster compared to water release, which is attributed to the fact that diffusion of water in the glassy amorphous state is significantly slower compared to the rubbery amorphous state. This effect is accounted for in the moisture diffusion modelling tool. For a detailed explanation of this effect for this application example, see paragraph 5.

Figure 8

Moisture uptake (in dark blue) and moisture release (in green) over time at T=100°C for a 3mm thick plaque of MX51 $^{\circ}$ (glass filled PPA) material.



Moisture uptake and release for a plaque of MX51 material

The next example deals with an automotive application of a polymer part that is present under the hood close to the internal combustion engine. The part is 1 mm thick and consists of ForTii® Ace MX53B material (PPA material with 50wt% glass fiber) that is initially dry. The example represents a situation that during a day (24 hours) the car is driving from 9am to 10am and the rest of the day the car is on the parking lot. During driving conditions, the polymer part is exposed to a temperature of 100°C and 0% RH and during parking conditions the part is exposed to a temperature of 23°C and 100% RH. These daily conditions are applied in a repetitive cyclic daily regime for 150 days (3600 hours).

In Figure 9a, the water concentration profile is plotted over the thickness of the part at the end of each day (cycle) for specific days. After the first day, the two outer slabs of the polymer part already contain moisture while the inner part of the sample is still dry. With an increase of the number of days, the situation in the outer slabs is not really changed. For the inner part however, it is observed that the moisture content is gradually increasing over the number of cycles and has reached an almost constant value after 125 days.

Figure 9a/b



Moisture profile over thickness for a ForTii Ace MX53B (glass filled PPA) part after 1, 8, 27, 64, 125 and 150 days exposition to cyclic conditions (left). Overall moisture content development versus time during one day for the indicated cycle (right). One cycle represents one day. Cyclic conditions are specified in the text.



Figure 9b shows the overall average moisture content in the part is displayed during the daily rhythm for various days. The drying out effect of the outer slabs each day during driving conditions is observed as well as the water uptake during parking conditions. This daily rhythm of water exchange is maintained during the total period of 150 days, and what is clearly observed is that the overall water content is increased with the number of days and is hardly changing any more for long periods of time (125 and 150 days). This overall water content in the polymer part is displayed as a function of time in Figure 10. The daily rhythm is visible as well as the water contents levelling off after approximately 100 days to an end value in the range 1.0-1.3wt%. It should be noted that this end value is not equal to one of the equilibrium values belonging to parking or driving conditions, but it is a value somewhere in between these two equilibrium values. This end value and its distribution over thickness is relevant since it will affect the mechanical performance of the part.

Figure 10

Overall moisture content versus time for a ForTii® Ace MX53B (glass filled PPA) part exposed to cyclic conditions. One cycle represents one day. Cyclic conditions are specified in the text.



Time (days)

This moisture diffusion tool offers the possibility to expose a polyamide part to a wide variety of non-cyclic or cyclic conditions that are relevant for specific application conditions. The last application example shows that the tool is rather unique in the sense that one can mimic complex application conditions affecting the moisture uptake and hence the performance of the polymer part. Another unique feature of the model is that the water diffusion is described in a non-Fickian manner, accounting for the plasticization effect of water in these polyamide materials manifesting itself in e.g. large differences in water uptake times compared to water release times.

MECHANICAL Properties

EFFECT ON MECHANICAL PROPERTIES

In previous sections (pages 8–10) we have given some details on the preparation of tensile specimens that are tested for stress–strain at different moisture contents. In the following sections, we will consider the effects of moisture content on the mechanical properties of polyamide materials.

Figure 11 shows the storage modulus E' versus temperature (at 1 Hz) measured on a bar of PA6 material, from which the Tg can be deduced from the drop of modulus upon the increase of temperature over a small range. The graph shows curves for dry material (dark blue curve, 0%RH) and for a material in the conditioned state (green curve, acc. ISO1110). Please note that the small 'bump' in the green curve at around 90°C is a result of post–crystallization. The approximate Tg value for the dry material is 67°C. In general, it is observed that the Tg shifts to lower temperatures with increasing water content. For the green curve the value of Tg is 10°C.

Figure 11

Storage modulus of PA6 at dry and conditioned state (acc. ISO1110) from a DMTA measurement.



The decrease of modulus between the glassy and rubbery state is considerable. Well below Tg, for example at a temperature of 23°C, the modulus of dry, unfilled PA6 is about 3 GPa. Well above Tg at 120°C, it has dropped to 0.5 GPa. In the region between Tg and the melting temperature (Tm), the modulus is determined by the crystalline structure.

Moisture shifts the Tg transition to a lower temperature. This means that for a constant temperature, above room temperature, the modulus will decrease with increasing moisture content. However, for temperatures lower than 0°C, we notice that the effect of moisture on the Young's modulus reverses. In the low temperature regime, the glassy modulus is higher for a PA containing (more) water.



Figure 12 shows the dry and conditioned stress-strain curves measured at 23°C for one of our PA6 grades. In the accompanying table the most relevant mechanical properties are given. The absorbed water (3.2wt% in this example) acts as a plasticizer, decreasing Young's modulus and strength considerably as well as increasing ductility (strain at break).

Figure 12

Stress-strain curves of injection moulded ISO5271A Akulon® K222–D (unfilled PA6), measured at 23°C. Sample 1 is measured in the dry state and sample 2 is measured in the conditioned state. The right image is a close-up of the left graph.



23°C	DRY	CONDITIONED
Young's Modulus	3157 MPa	968 MPa
Yield Stress	87 MPa	44 MPa
Strain at Yield	4%	23%

Figure 13 shows stress—strain curves for the PA46—GF30 material for (ISO5271A) tensile specimens that contain different amounts of water, all measured at 23°C. The method of conditioning in this case was to store the dry specimens for several hours in water of 70°C. The uptake of moisture was measured by evaluating the weight of the bars over time (results see Figure 7b).

From the curves in Figure 13, we observe that this glassfilled material shows a decreasing Young's modulus and strength for increasing amounts of absorbed water. In addition, the strain-at-break increases. None of these phenomena behave linear, which makes simple interpolation of datasheet values inaccurate.

Figure 13

Stress-strain curves of injection moulded ISO527–1A Stanyl® TW200F6 (PA46 with 30wt% glass fiber), containing different amounts of moisture, all measured at 23°C.



EFFECT ON BENDING STIFFNESS

In the tensile experiment the applied strain is constant over the complete cross-section. For simplicity, when measuring or calculating the moisture effects on tensile performance, it is common to take the average of the moisture fraction over the cross-section rather than considering the inhomogeneous moisture distribution. That may be enough for tensile stiffness and strength. However, if the moisture is not evenly distributed, this may make a big difference for the bending stiffness and strength. In a bending experiment the highest strains occur in the outer layers. Bending stiffness of a part depends on the inhomogeneous distribution of the Young's modulus over the part thickness rather than the average value of the modulus. Imagine a plaque with thickness h and width b exposed to asymmetric humidity conditions of 50%RH on one side and 100%RH on the other side at 23°C (see Figure 14a). The bending stiffness of the plaque is given by:

$$D_{bend} = b \int_0^h \frac{E(z)(z-z_0)^2}{1-\nu^2} dz$$

in which E(z) is the function of Young's modulus with thickness position z as variable, z0 the neutral axis and v is the Poisson's ratio. Taking 50% GF filled PA6 plaque with 1 mm thickness as an example, we can calculate its bending stiffness based on the inhomogeneous distribution of the Young's modulus as well as that based on the average Young's modulus over the cross–section. As Figure 14b





Environmental temperature: 23°C

Figure 14

a) Diagram of a plaque exposed to asymmetric moisture boundary conditions and b) Bending stiffness of Akulon® K224–HG0 in asymmetric humidity conditions.



Moisture Diffusion

DIMENSIONAL Change

Another effect of moisture absorption is that parts dimensions change. The amount of dimensional change depends on material, filler, processing conditions, and service conditions.

Here we present results for injection moulded plates (80x80x3 mm). We consider an unfilled PA6 material



The table in Figure 15 shows the relative weight uptake for both materials, under these conditions. For each material and moisture state the average length, width, and thickness of 5 specimens was determined. All results of dimensional changes due to moisture are shown in Figure 15. (Akulon® K222–D), and one that is filled with 30wt% of glass fibers (Akulon® K224–HG6). The specimens have been prepared in three different moisture equilibrium states:

- Samples containing no moisture
- Conditioning until equilibrium: first at 70°C/62%RH, followed by storage at 23°C/50%RH
- Conditioning until equilibrium: first at 70°C/92%RH, followed by storage at 23°C/80%RH

For the unfilled PA6 plates (Figure 15a) the length and width increase are about equal. The relative increase in thickness direction is much higher than the in-plane directions. This anisotropy is linked to the crystalline microstructure due to injection molding.

Grade Name	Condition	Moisture Uptake
Akulon® K222-D	23°C / 50%RH	2.98 wt%
Akulon® K222–D	23°C / 80%RH	5.76 wt%
Akulon® K224–HG6	23°C / 50%RH	2.18 wt%
Akulon® K224–HG6	23°C / 80%RH	4.32 wt%

For the PA6–GF30 specimens, we observe the lowest relative expansion in length (or parallel) direction, the intermediate one in the width (or perpendicular) direction and the highest one in the thickness direction. This is linked to the effect of the orientation of the glass fibers. The fiber orientation is highest in the length direction and suppresses the expansion in this direction the most. The anisotropy in expansion is highly linked to the glass fiber orientation.

At this point in our modelling approach, we have only considered the diffusion of moisture over time. In our observations concerning the change of part dimensions due to moisture absorption, we observe that the microstructure (with or without fillers) has a distinctive effect. For the future we intend to couple moisture diffusion modelling with modelling of the mechanical performance of injection moulded parts. This will require us to consider the expansion of the material due to moisture absorption, as well as the stress–strain response due to moisture absorption.

Figure 15

Water uptake (table) and resulting dimensional changes of PA6 specimens for two equilibrium moisture conditions at 23°C. The left graph refers to Akulon® K222–D (unfilled PA6) plates (80x80x3 mm), the right graph to the Akulon® K224–HG6 (PA6–30wt% glass fiber filled) plates.



In our observations concerning the change of part dimensions due to moisture absorption, we observe that the microstructure, with or without fillers, has a distinctive effect.

CONCLUSION

Most polyamides are semi-crystalline materials that absorb moisture, depending primarily on their degree of crystallinity, amount of reinforcement, the surrounding humidity, and temperature. Moisture lowers the materials glass transition temperature and has an effect on the mechanical properties.

Experiments revealed that, counterintuitively, the water absorption speed is different from the desorption speed. This phenomenon is related to the glass transition temperature and the state of the polyamide, as the rubbery state absorbs moisture faster compared to the glassy state. Our model can describe this phenomenon well, as the examples of accelerated conditioning and moisture absorption experiments clearly show. Moreover, the model can be used to apply multiple cyclic boundary conditions. The material's moisture content lowers its modulus and strength while increasing its strain at break. Because these are important parameters, accurate prediction of these properties is key for any engineer. However, also for the estimation of bending stiffness, understanding of the underlying phenomena is important. Dimensional change due to moisture absorption is an anisotropic phenomenon that depends on the fiber and crystal orientation. Our experts can advise you on any of these topics.

Many of the aspects discussed in this white paper can be modelled using our online tools. This helps you make smarter material choices and create lighter and stronger parts.

To learn more, contact us Envalior.com.



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