



Life Sciences

## Validation Guide

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USTR 2174

### Supplement for Pall<sup>®</sup> Kleenpak<sup>™</sup> Nova NP7 Filter Capsules

This supplement should be read in conjunction with Publications “Validation Guide for Pall Kleenpak Nova Filter Capsules” (VG KLN NOV; USTR 2099)

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# 1. Validation Supplement Overview

## 1.1 Introduction

This supplement contains data applicable to **Pall Kleenpak** Nova NP7 filter capsules. These filter capsules have integrally moulded inlet and outlet connections and precision moulded vent and drain fittings. For full details on test procedures and tabulated data on **Pall Kleenpak** NP6 filter capsules please refer to Pall Publication VG KLN NOV or USTR 2099. The purpose of this supplement is to document the additional testing that has been performed to demonstrate the strength and suitability of the NP7 capsules for use in pharmaceutical filtration applications.

**Kleenpak** Nova filter capsules incorporate standard 25 cm/10" (NP6) and 50 cm/20" (NP7) **Pall** filter cartridges and are available with a number of different membrane types. Information about the validation of the different **Pall** filter membranes is available in separate validation guides.

**Kleenpak** Nova filter capsules with a 'G' designation in the part number are provided non-sterile. Depending on the filter membrane incorporated in the filter capsules, they are suitable for sterilization either by autoclaving or gamma-irradiation. Further details about the tolerance of different **Pall** membranes to autoclaving and gamma-irradiation are available from your local Pall sales company or distributor.

## 1.2 Summary of Conclusions

### Burst Testing

Sixty-five **Kleenpak** Nova NP7 capsules were burst pressure tested. This testing included both capsules with sanitary flange compatible inlet and outlet connections (P-1 option) and hosebarb inlet and outlet connections (P-6 option, suitable for ½ inch tubing). The testing employed untreated samples, samples that had been exposed to repeated autoclave cycles at 125°C and samples that had been exposed to gamma-irradiation at doses of approximately 50 kGy. All capsules had burst pressures in excess of 204 psig (14.1 barg) giving an acceptable safety factor over the maximum recommended operating pressure of 43.5 psig (3 barg). Based on mean data the **Kleenpak** Nova NP7 capsule's burst strength appears to drop by approximately 100 psig (6.9 barg) after gamma irradiation at 50kGy.

This testing indicated that gamma irradiation had the greatest effect on the materials and structure of the capsule.

### Creep Rupture Testing

**Kleenpak** Nova NP7 filter capsules have been designed to be capable of operating at up to 43.5 psig (3 barg) for 168 hours (1 week) in continuous use. In order to allow for integrity testing with short excursions at higher test pressures the capsules have also been designed to be capable of operating at up to 90 psig (6.2 barg) for a maximum accumulated total time of 10 hours. The creep rupture data presented using typical **Kleenpak** Nova capsules demonstrate the very large safety margins that have been incorporated into these pressure claims. Additional data demonstrates that **Kleenpak** Nova NP7 capsules have satisfactory safety margins after gamma irradiation at 50kGy.

### **Pressure Fatigue Testing**

Kleenpak Nova NP7 filter capsules can withstand repeated pressurisation cycles, as demonstrated by fatigue testing where typical capsules were exposed to 100,000 pressure cycles from 0 to 43.5 psig (3 barg) and back to 0 psig again.

### **Water Flow Characteristics**

The water flow/pressure drop data presented for empty Kleenpak Nova filter capsules can be used in conjunction with the pressure drop characteristics of standard 50cm (20") Pall filter cartridges to form the basis for sizing filter systems employing Kleenpak Nova filter capsules.

### **Extractables Testing**

The level of aqueous and ethanol extractables for empty Kleenpak Nova filter capsules, irradiated with doses of up to 50.0 kGy were found to be extremely low. Out of 6 extraction tests performed on NP7 the non-volatile residue extracted was < 1 mg when water was used as the extraction fluid and < 15 mg when 96% ethanol was used as the extraction fluid.

Actual service life may impose different conditions, such as different exposure times, temperature, liquid purity etc. Evaluation under actual process conditions is therefore also recommended.

### **Shelf Life Studies**

Burst pressure tests performed on irradiated Kleenpak Nova NP7 filter capsules that had been stored for one year demonstrated that all capsules had burst pressures in excess of 180 psig (12.4 barg) giving an acceptable safety factor over the maximum recommended operating pressure of 43.5 psig (3 barg).

### **Biological Reactivity Tests**

The materials of construction of empty Kleenpak Nova filter capsules were found to meet the requirements of the current United States Pharmacopoeia for Class VI-121°C Plastics. (see data in Pall Publication VG KLN NOV or USTR 2099; the materials of construction used for all connector options and all capsule sizes are identical)

## 2. Burst Testing

### 2.1 Introduction

The purpose of these tests was to demonstrate that **Kleenpak** Nova NP7 capsules withstand the maximum pressure rating of 43.5 psig (3 barg) with an appropriate safety margin. Burst tests were performed on empty untreated **Kleenpak** Nova NP7 capsules and also on samples that had either been autoclaved or gamma-irradiated at a dose of approximately 50 kGy. Methods are as stated in Pall Publication VG KLN NOV or USTR 2099

### 2.2 Results

The results of the burst pressure tests are shown in Tables 2-1 to 2-4. The average burst pressure of the non-treated **Kleenpak** Nova NP7 capsules was found to be 343 psig (23.6 barg) for P-1 style and 315 psig (21.7 barg) for P-6 style. After exposure to six one-hour autoclave cycles at 125°C the average burst pressure for NP7 capsules P-1 style was 352 psig (24.3 bar) and for P-6 style 348 psig (24.0 barg). Exposure to gamma-irradiation was found to reduce the burst pressures of the **Kleenpak** Nova NP7 filter capsules. The average burst pressure of samples exposed to approximately 50 kGy was 250 psig (17.2 barg) for P-1 style and 239 psig (16.5 barg) for P-6 style.

**Table 2-1 Burst Pressures of Non-Sterilized Kleenpak Nova NP7 Capsules**

**Non-Sterilized Kleenpak Nova NP7 P-1 Style Capsules**

<b>Pall Kleenpak Nova NP7 Capsule Serial Number</b>	<b>Burst Pressure</b>
<b>PB733086</b>	315 psig/21.7 barg
<b>PB733087</b>	355 psig/24.5 barg
<b>PB733088</b>	260 psig/17.9 barg
<b>PB733089</b>	325 psig/22.4 barg
<b>PB733090</b>	355 psig/24.5 barg
<b>PB735011</b>	295 psig/20.3 barg
<b>PB735012</b>	305 psig/21.0 barg
<b>PB735013</b>	310 psig/21.4 barg
<b>PB735014</b>	310 psig/21.4 barg
<b>PB735015</b>	300 psig/20.7 barg
<b>PB737024</b>	450 psig/31.0 barg
<b>PB737064</b>	420 psig/29.0 barg
<b>PB737066</b>	400 psig/27.6 barg
<b>PB737074</b>	400 psig/27.6 barg
<b>PB737087</b>	400 psig/27.6 barg
<b>PB737114</b>	290 psig/20.0 barg
<b>PB737115</b>	260 psig/17.9 barg
<b>PB737116</b>	355 psig/24.5 barg
<b>PB765009</b>	420 psig/29.0 barg
<b>PB770003</b>	325 psig/22.4 barg
<b>Average burst pressure</b>	<b>343 psig / 23.6 barg</b>

**Non-Sterilized Kleenpak Nova NP7 P-6 Capsules**

<b>Pall Kleenpak Nova NP7 Capsule Serial Number</b>	<b>Burst Pressure</b>
PB733091	325 psig/22.4 barg
PB733092	290 psig/20.0 barg
PB733093	320 psig/22.1 barg
PB733094	325 psig/22.4 barg
PB733095	315 psig/21.7 barg
<b>Average burst pressure</b>	<b>315 psig/21.7 barg</b>

**Table 2-2 Burst Pressures of Kleenpak Nova NP7 Capsules Autoclaved  
for 6 One Hour Cycles at 125°C**

**Autoclaved Kleenpak Nova NP7 P-1 Capsules**

<b>Pall Kleenpak Nova NP7 Capsule Serial Number</b>	<b>Burst Pressure</b>
PB733021	300 psig/20.7 barg
PB733032	360 psig/24.8 barg
PB733034	335 psig/23.1 barg
PB733047	360 psig/24.8 barg
PB733048	360 psig/24.8 barg
PB734001	380 psig/26.2 barg
PB734002	345 psig/23.8 barg
PB734003	380 psig/26.2 barg
PB734004	380 psig/26.2 barg
PB734005	345 psig/23.8 barg
PB735001	370 psig/25.3 barg
PB735002	370 psig/25.3 barg
PB735003	355 psig/24.5 barg
PB735004	280 psig/19.3 barg
PB735005	360 psig/24.8 barg
<b>Average burst pressure</b>	<b>352 psig/24.3 barg</b>

### Autoclaved Kleenpak Nova NP7 P-6 Capsules

Pall Kleenpak Nova NP7 Capsule Serial Number	Burst Pressure
PB733001	345 psig/23.8 barg
PB733003	335 psig/23.1 barg
PB733005	370 psig/25.3 barg
PB733009	380 psig/26.2 barg
PB733010	340 psig/23.4 barg
PB734006	380 psig/26.2 barg
PB734007	335 psig/23.1 barg
PB734008	365 psig/25.2 barg
PB734009	340 psig/23.4 barg
PB734010	355 psig/24.5 barg
PB735006	370 psig/25.3 barg
PB735007	340 psig/23.4 barg
PB735008	260 psig/17.9 barg
PB735009	340 psig/23.4 barg
PB735010	360 psig/24.8 barg
<b>Average burst pressure</b>	<b>348 psig/24.0 bar</b>

**Table 2-3 Burst Pressures of Kleenpak Nova NP7 P-1 Capsules  
Gamma Irradiated with a Dose of 50 kGy**

Pall Kleenpak Nova NP7 Capsule Serial Number	Burst Pressure
PB764065	260 psig/17.9 barg
PB764070	250 psig/17.2 barg
PB764076	250 psig/17.2 barg
PB764077	240 psig/16.5 barg
<b>Average burst pressure</b>	<b>250 psig/17.2 barg</b>

**Table 2-4 Burst Pressures of Kleenpak Nova NP7 P-6 Capsules  
Gamma Irradiated with a Dose of 50 kGy**

<b>Pall Kleenpak Nova NP7 Capsule Serial Number</b>	<b>Burst Pressure</b>
<b>PB764005A</b>	240 psig/16.6 barg
<b>PB764006A</b>	260 psig/17.9 barg
<b>PB764016A</b>	265 psig/18.2 barg
<b>PB764020A</b>	225 psig/15.5 barg
<b>PB764023A</b>	205 psig/14.1 barg
<b>Average burst pressure</b>	<b>239 psig/16.5 barg</b>

### **2.3 Conclusions**

Sixty-five **Kleenpak** Nova NP7 capsules were burst pressure tested. This testing included both P-1 and P-6 style capsules and employed untreated samples, samples that had been exposed to repeated autoclave cycles at 125°C and samples that had been exposed to gamma-irradiation at doses of approximately 50 kGy. All capsules had burst pressures in excess of 205 psig (14.1 barg) giving an acceptable safety factor over the maximum recommended operating pressure of 43.5 psig (3 barg). Based on mean data the **Kleenpak** Nova NP7 capsule's burst strength appears to drop by approximately 100 psig (6.9 barg) after gamma irradiation at 50kGy.

### 3. Creep Rupture Testing

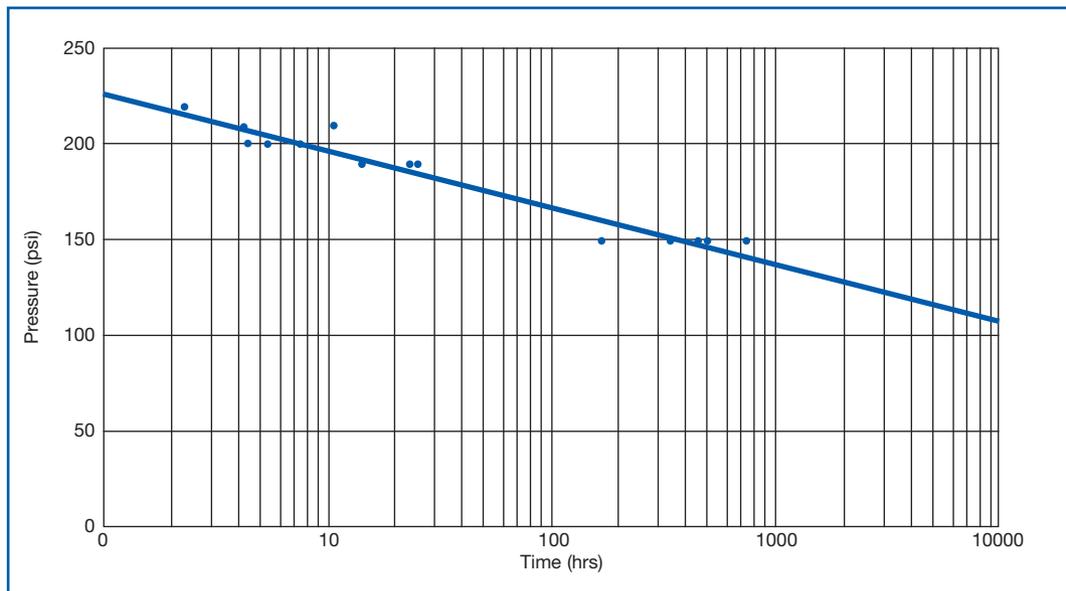
#### 3.1 Introduction

Creep rupture testing was performed in order to demonstrate the strength and stability of **Kleenpak** Nova capsule bodies over extended periods of time whilst under pressure. Methods are as stated in Pall Publication VG KLN NOV or USTR 2099.

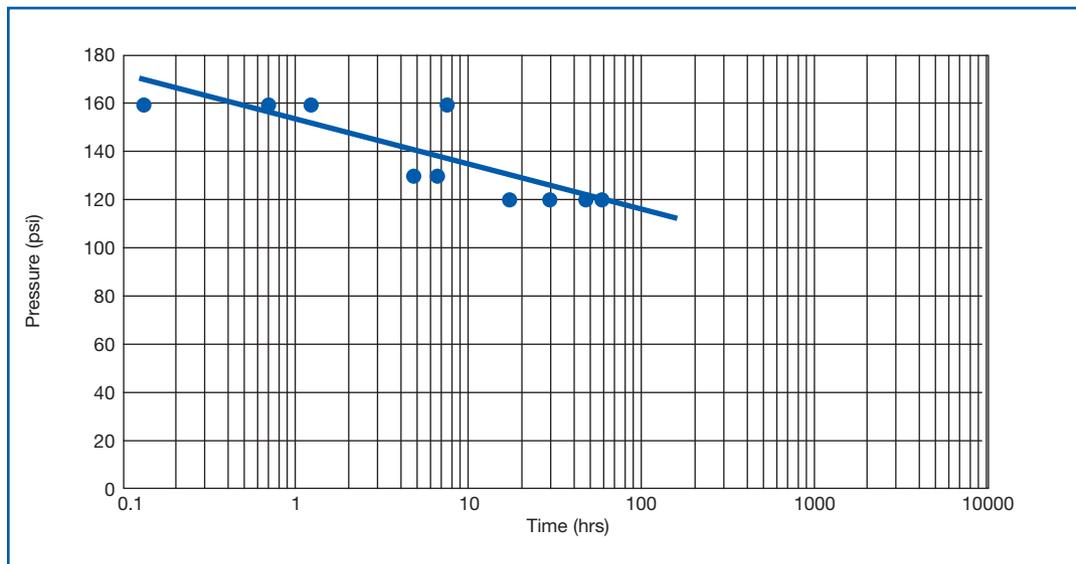
#### 3.2 Results

The results are shown in Figure 3-1. The graph represents the values obtained from testing empty **Kleenpak** Nova NP7 filter capsules (autoclaved at 125°C for 6 one hour cycles) at pressures between 150 psig (10.3 barg) and 220 psig (15.2 barg). The data has been extrapolated to predict the creep-rupture of samples maintained at constant pressure for up to 7,000 hours (292 days). Based on this extrapolation, it can be expected that **Kleenpak** Nova filter capsules will withstand approximately 700 hours (29 days) at 140 psig (9.7 barg) and 7,000 hours (292 days) at 110 psig (7.6 barg). Additional data on **Kleenpak** Nova NP7 filter capsules gamma-irradiated to 50kGy is shown in figure 3.2. This shows that irradiated **Kleenpak** Nova NP7 filter capsules will withstand approximately 100 psig (6.9 barg) for 500 hours.

**Figure 3-1 Mean Creep Rupture Pressures for Empty Kleenpak Nova NP7G Filter Capsules**



**Figure 3-2 Mean Creep Rupture Pressures for Empty Kleenpak Nova NP7S Filter Capsules (Gamma Irradiated to 50kGy)**



### 3.3 Conclusions

Kleenpak Nova NP7 filter capsules have been designed to be capable of operating at up to 43.5 psig (3 barg) for 168 hours (1 week) in continuous use. In order to allow for integrity testing with short excursions at higher test pressures the capsules have also been designed to be capable of operating at up to 90 psig (6.2 barg) for a maximum accumulated total of 10 hours. The creep rupture data presented using typical Kleenpak Nova capsules demonstrates the very large safety margins that have been incorporated into these pressure claims.

## 4 Pressure Fatigue Testing

### 4.1 Introduction

The purpose of these tests was to demonstrate that **Kleenpak** Nova NP7 filter capsules can withstand repeated cycles of pressurisation up to the maximum recommended operating pressure of 43.5 psig (3 barg). For these tests five samples with sanitary flange inlet and outlet connections were used (P-1 style) and five samples with hosetail inlet and outlet connections were used (P-6 style). Methods are as stated in Pall Publication VG KLN NOV or USTR 2099.

### 4.2 Results

The fatigue test results are shown in Table 4-1. Ten **Kleenpak** Nova NP7 capsules (autoclaved at 125°C for 6 one hour cycles) were tested and none of them showed any visible signs of leak paths or damage after 100,000 4 second pressure cycles of 0 to 43.5 psig (3 barg).

**Table 4-1 Results of Fatigue Testing for Kleenpak Nova Capsules**

<b>Inlet/Outlet Connection Option</b>	<b>Pall Kleenpak Nova Capsule Serial Number</b>	<b>Signs of Leak Paths/Damage following Exposure to 100,000 4 Second Pressure Cycles from 0 to 43.5 psig (3 barg)</b>
Sanitary flange inlet/outlet connections (P1) style)	PB733018	None
	PB733023	None
	PB733026	None
	PB733038	None
	PB557045	None
Hosetail inlet/outlet connections	PB733002	None
	PB733004	None
	PB733006	None
	PB733007	None
	PB733008	None

### 4.3 Conclusions

**Kleenpak** Nova NP7 filter capsules can withstand repeated pressurisation cycles, as demonstrated by fatigue testing where typical capsules were exposed to 100,000 4 second pressure cycles from 0 to 43.5 psig (3 barg).

## 5. Determination of Water Flow Characteristics

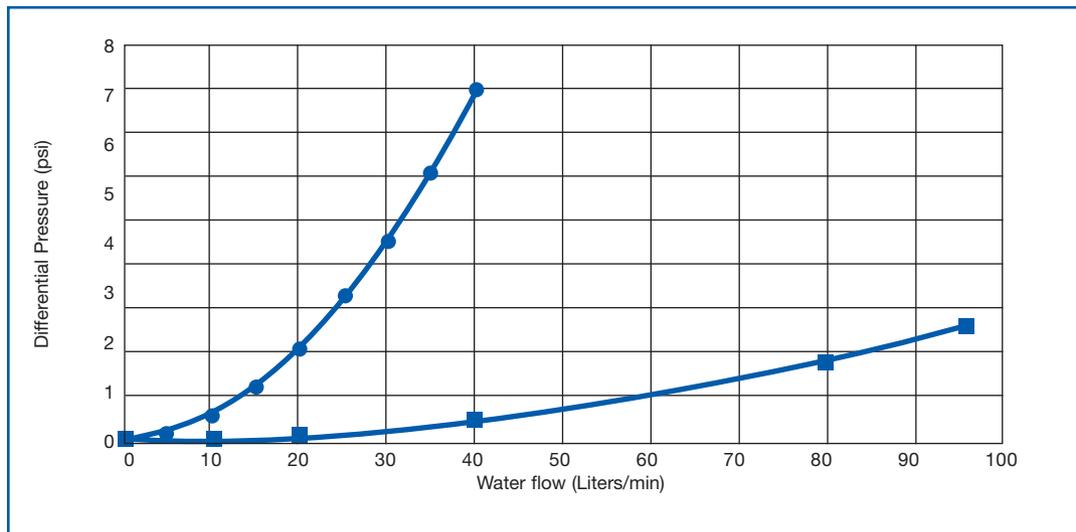
### 5.1 Introduction

The purpose of these tests was to determine the pressure differential characteristics of **Kleenpak** Nova NP7 filter capsules when subjected to different inlet water flow rates. Methods are as stated in Pall Publication VG KLN NOV or USTR 2099.

### 5.2 Results

The water flow/differential pressure characteristics of empty **Kleenpak** Nova NP7 filter capsules are shown in Figure 5-1. **Kleenpak** Nova NP7 filter capsules are available with a range of different membrane types, incorporating standard Pall 50 cm (20") filter cartridges. The water flow/differential pressure data presented here can be used to provide guidance on filter sizing.

**Figure 5.1 Water Flow/Differential Pressure Characteristics for Empty Kleenpak Nova NP P6, P9 and P1 Filter Capsules**



### 5.3 Conclusions

The water flow/pressure drop data presented for empty **Kleenpak** Nova NP7 filter capsules can be used in conjunction with the pressure drop characteristics of standard 50cm (20") Pall filter cartridges to form the basis for sizing filter systems employing **Kleenpak** Nova NP7 filter capsules.

## 6. Extractables Testing

### 6.1 Introduction

The purpose of these tests was to determine the amount of material that can be extracted from irradiated typical empty **Kleenpak** Nova filter capsules by water and ethanol at ambient temperature by re-circulating 4 liters of test fluid for 4 hours. Methods are as stated in Pall Publication VG KLN NOV or USTR 2099.

### 6.2 Results

The results (Table 8-1) show the typical quantities of non-volatile residue extracted per **Kleenpak** Nova capsule using deionized water and 96% ethanol at ambient temperature. The results reported are typical for standard production capsules.

**Table 6-1 Aqueous and Ethanol Extractables for  
Kleenpak Nova NP7 Filter Capsules**

Extraction Fluid	Gamma-Irradiation	Pall Kleenpak Nova Capsule Serial Number	Non-volatile Residue (mg)
Water	50kGy	PB76417	1
		PB76464	1
		PB76479	1
96% Ethanol	50kGy	PB76458	15
		PB76474	14
		PB76480	15

### 6.3 Conclusions

The level of aqueous and ethanol extractables for empty **Kleenpak** Nova filter capsules, irradiated with doses of up to 50 kGy are extremely low. Out of 6 extraction tests performed the non-volatile residue extracted was < 1 mg when water was used as the extraction fluid and < 15 mg when 96% ethanol was used as the extraction fluid.

Actual service life may impose different conditions, such as different exposure times, temperature, liquid purity etc. Evaluation under actual process conditions is therefore also recommended.

## 7. Shelf Life Studies

### 7.1 Introduction

These tests were performed to demonstrate that an adequate safety margin is maintained for the burst pressures of gamma-irradiated **Kleenpak** Nova NP7 filter capsules following storage at room temperature for one year.

### 7.2 Results

**Table 7-1 Burst Pressures of Kleenpak Nova NP7 P-1 Capsules Gamma-Irradiated with a Dose of 50 kGy and then Stored for One Year at Room Temperature**

<b>Pall Kleenpak Nova NP7 Capsule Serial Number</b>	<b>Burst Pressure</b>
<b>PB764010</b>	190 psig/13.1 barg
<b>PB764034</b>	200 psig/13.8 barg
<b>PB764047</b>	180 psig/12.4 barg
<b>PB764057</b>	190 psig/13.1 barg
<b>PB764060</b>	200 psig/13.8 barg
<b>Average burst pressure</b>	<b>192 psig/13.2 barg</b>

### 7.3 Conclusions

Burst pressure tests performed on irradiated **Kleenpak** Nova NP7 filter capsules that had been stored for one year at room temperature demonstrated that all capsules had burst pressures in excess of 180 psig (12.4 barg) giving an acceptable safety factor over the maximum recommended operating pressure of 43.5 psig (3 barg).



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