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# A comparative study of some nanomaterials used in the consolidation and treatment of glass photographic negatives

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#### Abstract- :

The negatives are records and documents of monuments, social life and heritage to serve as witnesses and to tell the history of a time period for the present and the near and distant future .With the great development in the science of conservation and restoration of monuments and cultural heritage. Making the choice of different nanomaterials proposed to reinforce photographic negatives, they used nanomaterials from the same components of the photographic layer and compared their results with the best nanomaterials that have proven successful with glass antiquities (Silver Nano Nitrat - Nano Gelatin MatrialNano-Emulsion of Silver Nitrate with Gelatin -Nano Primal Composite with Nano Zinc).

The evaluation is done by characterizing the physical and mechanical properties by measuring hardness, density, and dynamic light scattering (DLS). As well as analyzing the difference in surface area and pore size, as well as analyzing the angle of surface contact and knowing the resistance of these materials to moisture, As well as examining and analyzing through TEM,and Raman Spectroscopy. Through these examinations and analysis, it was found that silver nitrate gave the best consolidation results, followed by silver nitrate emulsion with gelatin, and in last place came the distortion of the external and internal appearance of the photographic layer Nanopremial with zinc oxide.

The experiments proved that the best material to use is silver nanonitrate with a concentration of 3%. The process was applied to the historical negative B0641 from the collections of the Bella Photography Studio, which has a separation of the photographic layer from the glass holder in several places that were treated and strengthened with silver nanonitrate material.

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**Keywords:** - photographic negatives - glass support - detachment – restoration - straightening - reinforcement - Nano-material

#### **Introduction: -**

Since glass plate historical photographic negatives consist of a glass plate and a photographic layer that is very sensitive to deterioration due to the binder (gelatin) and silver halides (bromide, iodide and silver nitrate), the glass plate is very susceptible to damage.

Consolidation is the process of repairing the mechanical strength of photographic glass negatives that have become weakened as a result of exposure to unfavorable environmental conditions in the preservation environment in which they were found, Strengthening materials increase the strength of the cohesion between the weak parts of the negative and is a necessary process in case of damage caused by aging and the environmental conditions in which the negative is found, Or when it is desired to give the passive more resistance, the consolidation process should be done gradually. Research on the materials used with photographic negatives in international museums and the German mission in the Ministry of Antiquities, which is responsible for the restoration of these negatives, found that they use pure gelatin at a concentration of 3: 7 per cent and then nanomaterials were applied to the main components of the photographic layer and the results were compared with one of the nanomaterials which has been shown to be successful with glass artifacts and for which no solvents have been used to damage the gelatin bonding material of the glass photographic film.(Silver Nano Nitrat - Nano Gelatin Matri - Nano-Emulsion of Silver Nitrate With Gelatin - Nano Primal Composite With Nano Zinc)

The experiments proved that the best material to use is silver nanonitrate at a concentration of 3%, and the process was applied to the historic B0641 negative from the collections of the Bella Photography Studio.

#### <u>Materials and Methods</u> <u>1- Materials :</u>

• Photographic glass negatives for experimental study consisting of (glass slides cm3\*cm3\*cm3\*3mm - photographic sensitive layer consisting of gelatin, halogenated alkali (potassium iodide + potassium bromide), silver nitrate, gold salt and Ammonia.

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• Nanomaterials from the same components of the photographic layer were used and their results were compared with the best nanomaterials that have been proven to work well with glass monuments, namely

- Silver Nano Nitrat
- Nano Gelatin Matrial
- Nano-Emulsion of Silver Nitrate with Gelatin
- Nano Primal Composite with Nano Zinc

• The process was applied to a historic B0641 negative from the collection of the Bella Photography Studio.

## 2- Methods-:

#### 2-1- Documentation: -

The oldest photographic studios in Egypt were searched to have glass negatives that are actually applied and contacted the Bella Studio, managed by Mr Ashraf Bella, a photographer - fig. 1, Bella's supervisor still works with the old camera that Bella used in 1890, the German Lehnhoff camera, which is a wooden box mounted on a fourwheel stand - Fig. 2, The photographs and negatives held by Mr Ashraf are characterised by the presence of visible silver nitrate on them as a result of the acidification method as well as the knowledge and visibility of the storage location -Photo 3, The damage has become a clear picture of the most important factors that have affected it, as well as the manifestations of these factors in the form of clay calcification - Fig. 4, Wrinkling of the gelatinous layer - Fig. 5 - and separation and peeling of the photographic layer - Fig. 6

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Fig. 1 Bella Studios in the old downtown area	Fig.2: Lehnhoff's old German camera	Fig. 3 shows the warehouse where the photographic glass negatives are stored.
Fig. 4 shows B 0641 clay calcifications illustrate the glass negative	Fig. 5 shows Demonstrates the wrinkling phenomenon of glass negative B 0641	Fig. 6 shows The appearance of flaking and detachment of the photographic film from the glass plate is shown for glass negative B 0641

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The negative was lifted after its initial cleaning because its details were not visible and therefore it was photographed to put the different manifestations of damage were drawn 1: 1, then using AutoCAD and Photoshop to produce a figure (7).



Figure (7) shows the authentication of signed programmes with glass negative damage manifestations B0641

## 2-1-1 Diagnostic process

This is the first step in the diagnosis of the historical photographic negative on glass holder No. B 0641 from inside the storage room of Bella Studio on the roof of the property and the previous memory is preserved inside the storage room without a carton on the floor under a wall topped by a roof with a hole through which rain, dust and mud calcifications fall, and a visual inspection has been made to make a preliminary study of the damage aspects of the historical photographic negative on glass holder No. B 0641 subject of the study. A magnifying glass was used to identify clay calcification, detachment of the gelatin layer, wrinkling of the sensitive photographic layer and other damage that cannot be seen with the naked eye. In this case, after it was documented, it was preliminarily cleaned to visualise the damage caused by the impact of calcified layers of dust and mud as a result of the aforementioned improper storage.

The historical photographic negative on glass holder B 0641 was found to be the most significant form of deterioration: -

**Dirty Surface Appearance:-** It is the result of the accumulation of dust, dirt and clay calcifications - Fig. 4 - resulting from the wrong storage and not realising the importance of these negatives, which were found on the floor of the unpaved warehouse as well as the dilapidated roof, which works to leak rainwater loaded with accumulated dust on top of the surface of the warehouse.

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**Phenomena Speckle** :- The yellow stains on the negative - Fig. 4 - are due to insufficient fixing time or the fixing material is corrupted, thus forming silver salts that are not completely dissolved and are affected by light and coloured yellow. **Wrinkling :-** This negative shows the phenomenon of wrinkling - fig. 5. as this phenomenon is associated with resins with low viscosity such as gelatin. The addition of desiccants in large quantities during preparation and the application of gelatin as a protective layer in a thick layer have also been found to cause this phenomenon in addition to the high relative humidity, which increases the dissolution of the gelatinous layer from one place to another within the negative. **Flaking**:- This may be due to the thickness of the sensitive gelatinous layer, the long drying time, poor preparation of the glass holder, poor adhesion of the gelatinous layer, relative humidity and temperature variations (Fig. 6).

#### 2-2 Experimental: -2-2-1 Experimental Samples and Artificial Aging: -

The experimental aspect included the fabrication and preparation of experimental samples of photographic negatives on a glass substrate. The sensitive photographic layer was deposited on a glass substrate at a temperature of 49 °C with a layer thickness of 1. 200 microns in the glass panels: 200 microns in glass plates-Fig. 8 - , After pouring the solution, we have manufactured samples of photographic negatives that have been exposed to light to give the same effect as light on photographic film - Fig. 9



# 2-2-2 Preparation and production of selected nanomaterials

Nano-materials are nanoscale chemical substances that are used with high quality and are prepared by several methods, all of which have in common their dependence on the atomic scale, i.e. one atom towards another atom. The smaller the size scale of the mass of the substance, the different the chemical potency, meaning the smaller the scale, the greater the chemical potency of the substance <sup>(1)</sup>. Nanomaterials were used for the main components of the photographic layer

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and the results were compared with one of the nanomaterials which proved to be successful with glass monuments and for which no solvents were used to damage the gelatin binder in the sensitive glass photographic film.

- Silver Nano Nitrat
- Nano Gelatin Matrial
- Nano-Emulsion of Silver Nitrate with Gelatin
- Nano Primal Composite with Nano Zinc

The materials were tested using TEM to determine the highest resolution of the material on the nanoscale and to confirm the size of the nanoparticles for each selected material.

#### 2-2-2-1 Transmission Electron Microscope (TEM): -

The materials were examined by morphology through a high-resolution transmission electron microscope (HRTEM, JEOL TEM-2100, Japan) with a voltage of 250 kV and a magnification of 20 X at the Graduate School of Nanotechnology laboratories of Cairo University, Samples were prepared prior to measurement by sonication-assisted nanoparticle extraction at a maximum temperature of 85% Fahrenheit for 30 minutes. Finally, 50 microns were added to the SEM and air dried for 5 hours. Visualisation of materials at three levels of nanoscale miniaturisation 50NM,<sup>1</sup> this is illustrated in Table No.1

The processor is placed 0.1 cm wide in the process meter with tests for one hour at 1500 rpm, the results are shown in the figures for each material - see Table no.1



Table 1 shows the shape, size and dispersion of nanoparticles using TEM

<sup>&</sup>lt;sup>1</sup> (Youssef F, Mohamed G, Ismail S, et al. (2021) Synthesis, characterization and in vitro antimicrobial activity of florfenicol-chitosan nanocomposite. Egyptian journal of chemistry 64(2): 941–948.).

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#### <u>2-2-3 Accelerated aging processes on laboratory-manufactured samples</u> (before and after treatment) :-

#### 2-2-3-1 Aging by humidity and thermal ageing

The negative samples manufactured before and after consolidation with the selected nanomaterials were exposed inside the aging oven at 105°C and 90% relative humidity for 120 hours. The manufactured samples were then placed inside the desiccator. A saturated solution of sodium hydroxide was placed inside the desiccator to provide humid conditions and then the desiccator was placed inside the convection oven, and the desiccator was placed inside the convection oven. This can be seen in Fig. 10.

#### 2-2-3-2 Chemical ageing by acids and alkalis of manufactured samples

The manufactured negative samples were subjected to acid aging using 10% HCL and 10% NaOH solution for a period of one month before and after reinforcement with the selected nanomaterials, as shown in fig. 11.

#### 2-2-3-3 Optical ageing

The fabricated samples were exposed for 120 hours continuously to UV light using a UV Lamp, 600 W, 400 nm and then exposed again after being treated with the selected nanomaterials - fig. 12

	- Ple	
Fig. 10 shows the samples inside the dicyclotron for thermal and moisture aging.	Fig. 11 shows aging by acids and alkalis	Fig. 12 illustrating UV light aging

#### 2-2-4 Results and discussion: -

2-2-4-1 Optical, physical and mechanical characterisation of experimental samples

### 2-2-4-1-1 Hardness :

One of the most important tests of mechanical properties is the measurement of hardness, i.e. the extent of the material's ability to scratch. A Micro Hardness machine was used to measure hardness at the Faculty of Science, Cairo University, and by measuring the hardness of the manufactured samples before and after reinforcement with the selected nanomaterials, the following results were given in Table (2).

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Sample type	Hardness values for samples		Average	
	Sample 1	Sample 2	Sample 3	hardness Kg/mm2
Negativity is manufactured before aging.	340	368	365	357.6
Manufactured negativity after aging	274	200	337	270.3
Negativity manufactured after consolidation nano AgNO3	430.1	425.6	432.3	429.3
Negativity manufactured after consolidation nano gelaten	395	399.6	398.6	408.9
Negativity manufactured after consolidation nano AgNO3+ gelaten	380	376	379.6	378.5
Negativity manufactured after consolidation nano ZnO + AC33	354.6	384	372	370.2

Table2 shows the hardness values of the hardened experimental samples

#### 2-2-4-1-2 Density

One of the most important physical properties of negative samples manufactured after aging processes and also after strengthening with selected nanomaterials. Density expresses the relationship between the weight of the material and its volume and is estimated in g / cm3 Density was measured at the laboratory of the Faculty of Science of Cairo University and was as follows Table (3)

Sample type	Density	Density values for samples		
	Sample 1	Sample 2	Sample 3	density g/cm3
Negativity is manufactured before aging.	2.50	2.52	2.52	2.51
Manufactured negativity after aging	2.41	2.46	2.44	2.43
Negativity manufactured after consolidation nano AgNO3	2.62	2.60	2.59	2.60
Negativity manufactured after consolidation nano gelaten	2.55	2.58	2.57	2.56
Negativity manufactured after consolidation nano AgNO3+ gelaten	2.54	2.56	2.56	2.55
Negativity manufactured after consolidation nano ZnO + AC33	2.53	2.52	2.51	2.52

Table (3) shows the density values of the hardened experimental samples

# 2-2-4-1-3 DLS dynamic light scattering analysis

A non-destructive and fast method for determining the size of particles in the range of a few nanometres to microns. This characterisation is based on changes in the pattern of dots that are darkened or lightened with dark and light dots. Determining the intensity of scattering of light rays leads to the measurement of particles <sup>(1)</sup>. By comparing the results through the DLS spectra in Figure 13, we find that the nanopremium material with zinc oxide nanoparticles added to it gave the highest light scattering measurement, Silver nitrate is followed by silver nitrate

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emulsion with gelatin, then gelatin, and finally silver nitrate, indicating silver nitrate's high ability to absorb light and change colour.



Fig. (13) shows the comparison of the DLS spectral pattern results for the selected reinforcement materials

## 2-2-4-2 Analysing the contact angle

The contact angle is defined as the angle at which a liquid forms at the boundary of the intersection with a liquid, gas or solid. A contact angle of 90 degrees is usually considered a boundary value, If the angle of contact with water is less than 90 degrees, the surface is considered hydrophilic and if the angle of contact with water is greater than 90 degrees, the surface is considered hydrophilic.

It is clear from the comparison shown in Figure (14,15,16,17,18) that silver nitrate gave high moisture resistance followed by nano gelatin, while nano primal with zinc oxide gave very poor moisture resistance, so it is preferable to exclude them in the consolidation of photographic negatives.<sup>2</sup>



<sup>&</sup>lt;sup>2</sup> (1)El-Wakeel, S. T., Abdel-Karim, A., Ismail, S. H., & Mohamed, G. G. (2022). Development of Ag-dendrites @Cu nanostructure for removal of selenium (IV) from aqueous solution. Water Environment Research, 94(4), e10713. https://doi.org/10.1002/wer.10713

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#### 2-2-4-4 Raman Spectroscopy

Raman spectroscopy is a type of spectroscopy that specialises in the study of low-frequency molecular vibration modes in a system, This relies on the phenomenon of the inelastic scattering of light on molecules, known as Raman scattering. This technique is used in the study of the properties of materials.

A comparison of the results in Figure (19) shows that silver nanonitrate with gelatin has the highest light scattering scores according to the red-coloured Raman spectra, Gelatin has the lowest values in light scattering, followed by silver nanonitrate (blue colour) and gelatin has the lowest values in light scattering (green colour), indicating that it significantly absorbs light, thus changing the colour to negative and opaque.



Fig. 19 shows the pattern of Raman spectra of the sample surface after strengthening the experimental samples with the selected strengthening materials

Through the previous tests of the reinforcing materials selected by the researcher, it became clear that silver nitrate nano-nano gave the best results in reinforcement in terms of resistance to moisture, hardness and discolouration, followed by silver nitrate with gelatin and then gelatin .It should be excluded in the strengthening of photographic glass negatives because it has given poor results and distortion in the external and internal appearance of samples, as well as its inability to resist moisture and colour change, as well as its poor hardness and lack of resistance to scratches.

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#### 2-3: Examining and analyzing the components of (negative B0641)

#### 2-3-1 Transmission Electron Microscope (TEM):-

It was examined through an electron microscope at three degrees of miniaturisation at nano size 25NM, 100MN and 50NM, which showed the extent of damage to silver halide granules and silver folding as well as gelatin damage and wrinkling on the glass crystals, as shown in Fig. 20,21,22



## 2-3-2 Infra-Red (IR) Examination: -

The sample was examined and compared with several standard samples of varnish, gelatin and gelatin to find that the fluorescence pattern of the historical sample of photographic negativity corresponds to the standard sample of gelatin to confirm that the type of negativity is photographic glassy-gelatinous negativity and this appears through a figure no.23 showing the fluorescence patterns of both the negative sample in red, the standard sample of gelatin in blue, the varnish sample in brown and the gelatin sample in black.



Fig. (23) shows the comparison of fluorescence patterns for both the negative sample and the standard sample for gelatin, varnish and gelcoat

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#### 2-3-3 SEM with EDX inspection and analysis: -

The first sample was examined in the laboratory of the Graduate School of Nanotechnology at Cairo University, and the analysis of the first sample in Figure (24) showed that the resulting elements in Table (5) through the form of the pattern

EDX analysis and SEM imaging show clusters that take the form of rosettes, which is the characteristic form of silver sulfide compounds and is indicative of sulfide gas Hydrogen from any of the surrounding sources, perhaps from the atmosphere or the coating of the wooden box in which the negatives are stored. This is evidenced by the presence of silver at 80. 10 per cent and 0.39 per cent sulphur, as well as some other salts of silver halides such as chlorine (10.8 per cent) and bromide (1.16 per cent), these crystals appear on the surface of the negative in the form of shiny black spots, known as the Silver Tamish phenomenon, which causes darkening and reduced efficiency of the negative.



Fig. 25 shows the position of a decomposition pattern for region A, from which the photographic layer is missing, at a magnification of 15,000 X

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After reviewing the signs of damage through documentation, analysis and inspection, the following treatment plan was initiated: -

# 2-4 Application: -

# 2-4-1 Cleaning Processes

The mechanical cleaning process started with soft brushes to get rid of the dust and dirt stuck on the sensitive surface. A separation was evident in the sensitive photographic layer, which made it difficult to work on the negative, so the mechanical cleaning was stopped and the chemical cleaning started. Figures No. 26, The cleaning was limited to iodine solution in alcohol. The immersion process was carried out in a basin for no more than 5 minutes with very careful stirring and changing the solution to make sure that the negative is cleaned Figures 27 and the parts were dried immediately after cleaning using indirect air. Figures 28



# 2-4-2 Separating and stabilising the sensitive gelatinous layer

The individualisation process is a very important step and is the only solution in complex cases where a negative number B0641 has been exposed as a result of poor storage, in addition to the amount of clay calcification that was on top of the negative, As a result of repeated exposure to moisture, there are also manufacturing defects evident in the lack of proper application of the delicate gelatin layer on the glass plate. The crumpled pieces were stabilised by using gelatin with the individual in the direction of the air to put the crumpled gelatin back in place, as can be seen in Figures29 and 30

As for the detached and wrapped parts, the wrapped parts have been very carefully sorted where they are developed and individualised using Klucel-G (GHP cellulose is a material used as a reinforcing material and adhesive and has been proven to work well for archaeological materials). This material works on the flexibility and ease of handling, rearranging and assembling the photographic

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layer on the glass plate. The gelatin is then used in combination with chlorosilane to recoat and stabilise the layer on the glass plate Figures 31.



# 2-4-3 Labelling process

The negative numbering was done from the side of the glass support from the bottom at the far left side using Japanese paper saturated with Paraloid B72, then added to the surface using brushes, and then the number was written (Figures 32).

# 2-4-5 Consolidation process

The consolidation and insulation process was carried out in two stages by spraying and then applying soft brushes with 3% silver nitrate nanocomposite to consolidate the previously separated areas, which were straightened and stabilised as shown in Figures 33 and 34.

	5						The second
Fig. (32)	shows	the	Fig. 33 shows the first	Fig.	34	sho	wing
numbering	of	the	stage of consolidation	consolid	ation	with	soft
negative	numbe	ering	by spraying	mattress	ses		
according	to	the					
numbering	followed						

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# 2-4-6 Packaging Processes

This process was carried out after the completion of the restoration work and full documentation of the negative and was done using acid-free cardboard (free acide board) and a reinforcing method for the negative to preserve it during the storage process to prevent shaking or breakage. This method was achieved as it is suitable for display and storage at the same time as the dimensions of the negative were raised after the completion of the restoration - Fig. (35). 3 cm was added on each side and a depth with a name to prepare the preservation box, then acid-free paper was placed - Fig. 36, then Ethafome material was divided to be around and around the negative in all directions to support it and prevent shaking, and a moisture absorbent material of silica gel was placed - Fig. 37, and the negative was covered with acid-free transparent paper - then all data was written with the registration form - Fig. 38, and a picture of the negative after restoration - Fig. 39. The negative was converted into a positive photograph to know the shape of the final image and become a complete record of the negative to prevent it from being circulated frequently in order to preserve it - Fig. (40)

Fig. 35 shows the	Fig.36 shows the lining of	Fig. (37) shows the final
dimension of the packing	the box with acid-free	shape of the negative after
box.	paper	treatment and encapsulation
$ \begin{array}{c} \label{eq:second} \\ \hline \begin{tabular}{lllllllllllllllllllllllllllllllllll$		
Fig. (38) shows the form of	Fig. (39) shows the shape	Fig. (40) shows the printed
the attached registration	of the negative after	positive image of negativity
form	restoration, photographed	and a clear view of the streets
	with Silverfast scanning software	of the centre of Cairo

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## **General results and discussion**

Manufactured glass photographic negative samples were treated with four nanomaterials at a concentration of 3% and 7%, namely silver nitrate nanomaterial, nano-gelatin, silver nitrate emulsion with gelatin, silver nitrate emulsion with gelatin, silver nitrate emulsion with zinc oxide. The physicochemical and mechanical tests on the manufactured and archaeological samples treated with these nanomaterials led to the following results: -

-Silver nitrate nanomaterials were successful in preserving the overall appearance of the glass samples treated with them, without affecting the colour of the treated surfaces, while the use of nanopremium with zinc resulted in a thick, white, negatively distorted coating.

-The treated 3% silver nitrate nanomaterials gave lower colour change values than the permissible limit, which fulfils one of the most important requirements for a protective material.

- Silver nitrate nanocomposite at 3% concentration is considered the best material in terms of its ability to repel water as it achieved the highest values in the Water Contact Angle test Nanopremium composite with zinc oxide achieved the lowest value for the water contact angle and therefore is not favoured for use in the protection of photographic surfaces due to its inability to repel water.

- The 3% silver nanonitrate compound is considered the best material and compounds used in this study, as it has a high water repellent ability, has a self-cleaning property and has a great ability to resist various industrial weathering processes, as well as not affecting the colour of the treated samples.

# The Recommendation

- Treatment methods vary from case to case depending on the state of vitreous photographic negativity and the restorer's vision
- It is recommended to use 3% silver nitrate nano-composite in the process of chemical strengthening and insulation as it improves the mechanical and physical properties of the treated surface and insulation from high humidity due to its water-repellent property.
- The need to assess and avoid any risks arising from the use of nanomaterials during their preparation to reach optimal technical specifications for application.
- Photographic negatives executed on glass mounts and stored in warehouses should be protected by creating suitable preservation conditions of temperature and relative humidity, which should not exceed 42%, and the warehouse atmosphere should be

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characterised by purity and constant ventilation, with regular maintenance of these objects.

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