

SEPPIC

LERITE™

Glass frosting decoration



Ingredients that inspire

LERITE™

Glass satin, etching or frosting is a widely used decoration technique for liquor bottles, cosmetic bottles, tableware, lampshades and decorative objects to change their appearance and feel from clear and brilliant to an opaque or velvety frost.



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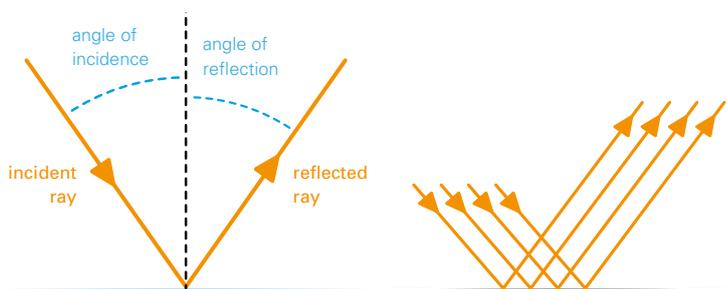
GLASS FROSTING DECORATION

For more than 30 years SEPPIC has manufactured a range of glass frosting agents sold world-wide under the trade name of LERITE.

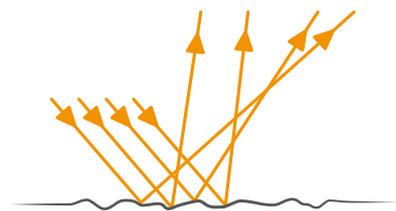
The different grades of the LERITE range allow some variation of satin appearance, from matte to shiny.

LERITE grades are white free flowing dry powders which, when mixed with an acid solution, produce a bath suitable for frosting glass. Glass items are immersed in the etching solution and then rinsed thoroughly with water. The satin effect is achieved as the silica in the glass reacts chemically with the etching solution, microscopically altering the glass surface. The following pictures show the difference between a normal glass surface and an etched one. The frosted appearance is the result of the different way that the light reflects off from the pyramids formed by the chemical attack.

Normal glass specular reflection



Frosted glass diffuse reflection



SEPPIC recommends the exclusive use of hydrochloric acid (HCl) when satin etching with **LERITE**. The use of hydrofluoric acid (HF) can be avoided with **LERITE** leading to a significant improvement in the safety of the frosting operation, in addition to a reduction in the fluoride content of the waste water.

GLASS ACID ETCHING

Soda-lime-silica glass, window glass and lead-oxide glass, crystal glass, all contain silica SiO_2 and sodium oxide Na_2O . During the glass etching, the SiO_3^{2-} and Na^+ on the glass surface, react with the hydrofluoric acid, HF , that is generated by the reaction between the ammonium bifluoride, $(\text{NH}_4)(\text{HF}_2)$ in the **LERITE**,

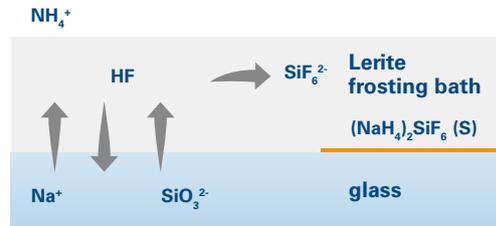
and the hydrochloric acid, **HCl**.

The points where the reaction is initiated are rapidly covered in crystals of ammonium fluorosilicate $(\text{NH}_4)_2\text{SiF}_6$. The areas that have not yet been covered with crystals continue to react with the etching bath creating microscopic pyramids that provide the glass frosted effect.

LERITE grades enable a quick reaction.

They also create a homogeneous satin effect as there is a consistent repetition of the starting attack points resulting in a uniform repartition of the pyramids and an optimal frosted effect.

The mechanism of the etching reaction is as follows:

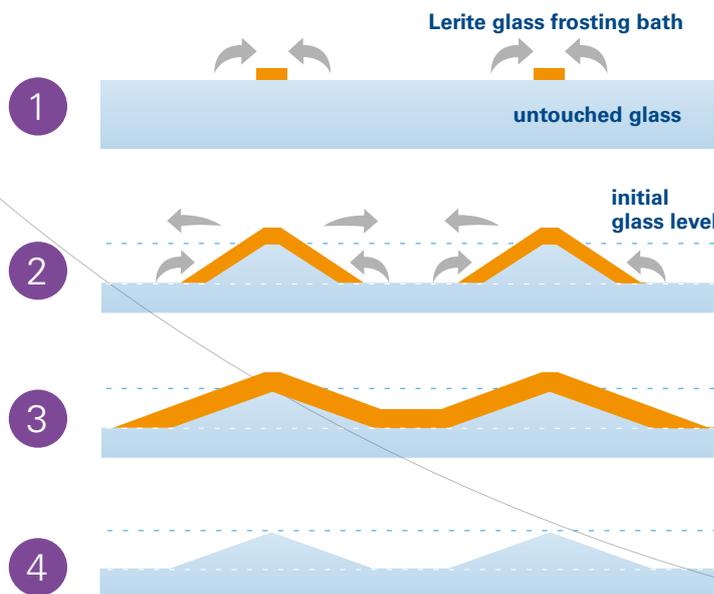


The global reaction is: $\text{SiO}_2 + 6 \text{HF} \longrightarrow \text{H}_2\text{SiF}_6 + 2 \text{H}_2\text{O}$

H_2SiF_6 is a strong acid ($\text{pK}_a < 0$) that disassociates: $\text{H}_2\text{SiF}_6 \longrightarrow 2 \text{H}^+ + \text{SiF}_6^{2-}$

$2 \text{NH}_4^+ + \text{SiF}_6^{2-} \longrightarrow (\text{NH}_4)_2\text{SiF}_6(\text{s})$

Ammonium fluorosilicate $(\text{NH}_4)_2\text{SiF}_6(\text{s})$ is the main component of the layer that covers the glass at the end of the etching process. Ca_2SiF_6 and Na_2SiF_6 are also formed in this layer but in a lesser quantity.



Points of reaction

Nucleation of $(\text{NH}_4)_2\text{SiF}_6$

Lateral growth of $(\text{NH}_4)_2\text{SiF}_6$

Microscopic pyramids are formed on the glass surface which end up covered by a thin layer of $(\text{NH}_4)_2\text{SiF}_6$

After rinsing with water, 100% glass is left, the surface has been altered and the frosted effect won't be lost over time. This is an advantage over coatings.

BENEFITS OF LERITE

- ✓ Easy to use and to handle.
- ✓ Reduced frosting time.
- ✓ Uniform and quick frosting for consistent productivity and quality.
- ✓ Use of hydrochloric acid, less dangerous than hydrofluoric acid.
- ✓ Reduced fluoride level in the frosting bath (around 50% less compared to a mixture 50/50 ammonium bifluoride and HF 55%).
- ✓ The **LERITE** bath should be regularly refilled so the satin-etching efficiency of the bath is maintained and the waste is reduced.
- ✓ The **LERITE** glass frosting bath has an almost unlimited lifetime.
It should be topped off from time to time with fresh **LERITE** to replace extracted material that clings to the frosted glass.
- ✓ The finished glass is 100% pure glass, there is no additional color or ink added therefore the satin decoration is not altered though time and there are no potential problems for recycling; glass treated with **LERITE** is fully recyclable and does not contain leachable coatings.
- ✓ Convenient packaging: plastic bags inside boxes of 25 Kg.
- ✓ **LERITE** industrial formulations are produced on the SEPPIC site based in Castres, France, which is qualified ISO14001, ISO9001, OHSAS 18001.



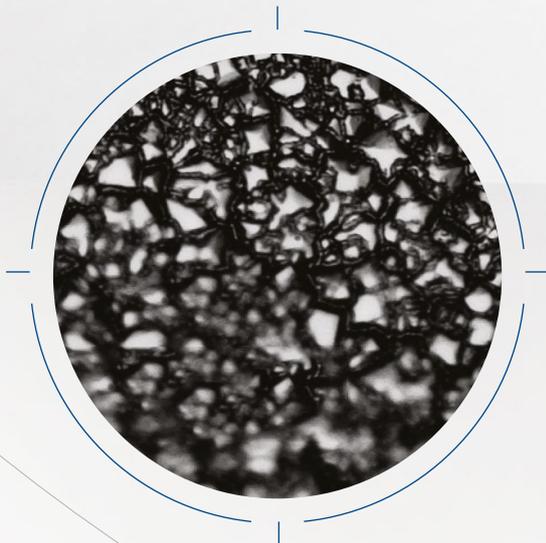
LERITE RANGE

LERITE is appropriate for satin-etching clear soda lime glass, colored glass, opal glass and crystal glass. However sodium-borosilicate glass cannot be etched with a standard frosting bath.

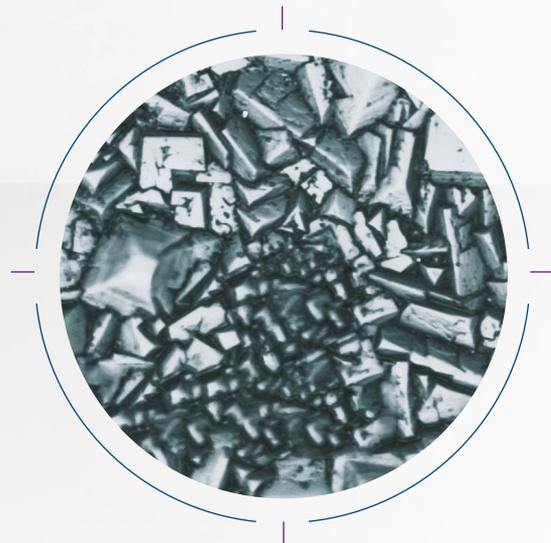
GRADE		ASPECT
LERITE SX 13	→	Matte finish, pronounced white effect
LERITE SX 20	→	Silky, fine and gleaming effect
LERITE SX 28	→	Very shiny and fine effect
LERITE SX 55	→	Shiny
LERITE SX 61	→	Standard matte finish
LERITE SX 86	→	Matte and opaque



When looked at through a microscope, the difference between a matte finish versus a shiny and transparent finish is the size of the pyramids created at the surface.



Matte finish :
the pyramids are smaller, **10 micron**



Shiny and transparent :
the pyramids are larger, **20 micron**

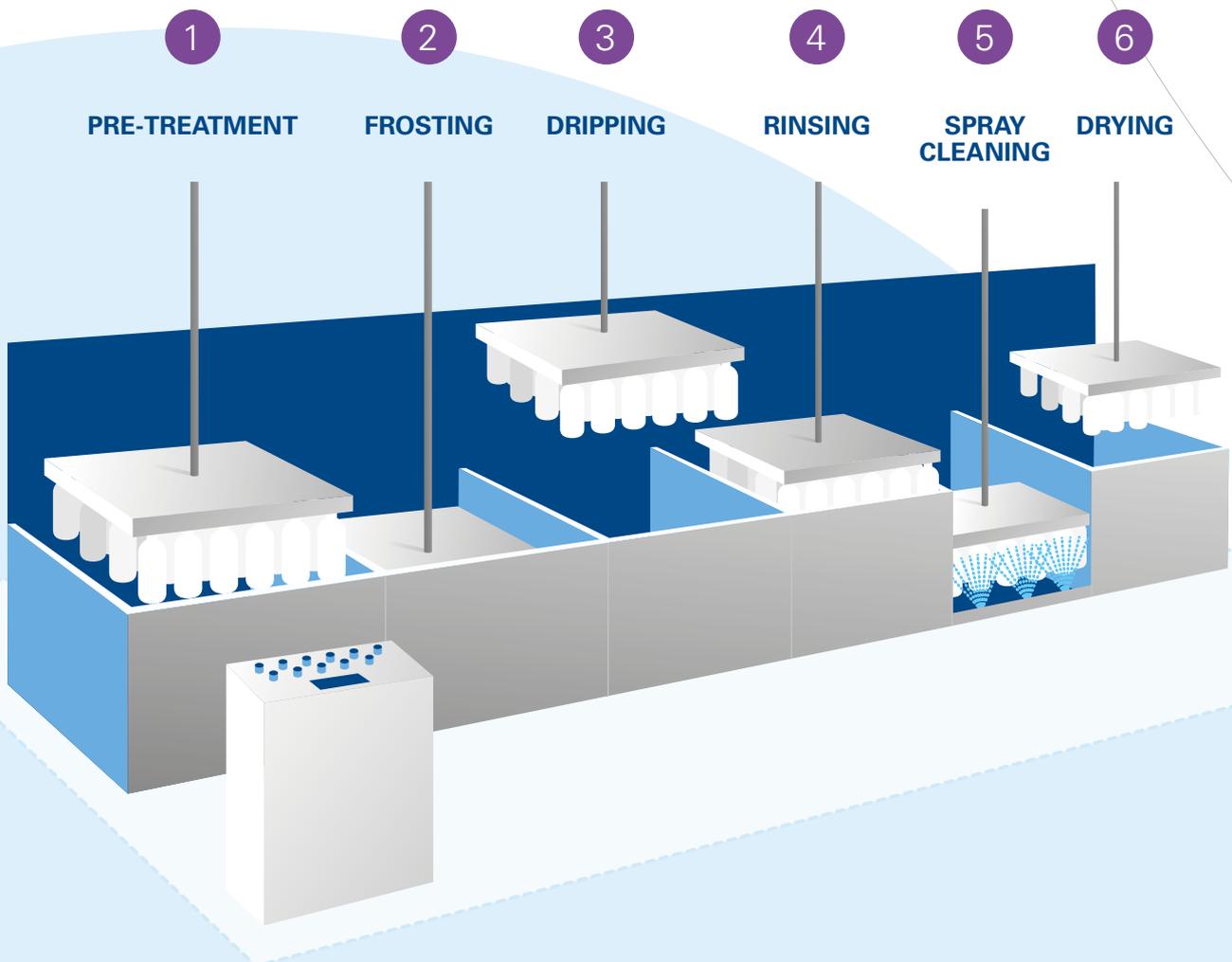
GLASS FROSTING PROCESS

It is possible to perform the frosting process using automated equipment like the one in the diagram or a manual process.



In any case, always use only PVC or PP equipment and wear rubber gloves (Viton or Nitrile), protective goggles and boots.

Always obey local safety regulations.



1. PRE-TREATMENT

For optimal results, before frosting, SEPPIC strongly suggests to perform an acid pre-treatment in order to get rid of residues on the surface such as:

- Mould release agents.
- Hot end treatment:
 - To mitigate the defects related to glass forming (sharp edges, scissors marks, mold gaskets), two techniques are used to improve the appearance and quality of the final glass products through localized reflow glass: reburn oxygas and polishing with oxyhydrogen flame.
 - In order to provide mechanical resistance organo-metallic salts $TiCl_4$ or $SnCl_4$ are applied on the glass surface.
 - The glass to be frosted should not have had a hot end treatment. Otherwise, the pre-treatment here proposed won't be fully effective.
- Cold end treatment: to avoid scratches, polyethylene waxes or oleic acid are sprayed at the surface of the glass as lubricants.
- Transportation and handling residues: dust or fingerprints.

1.1 Pre-treatment bath preparation

Hydrochloric acid (HCl):

- 32-34% concentration
- 20°-22°C / 68°-72°F temperature
- 1.17 g/cm³ density at 20°C / 68°F

LERITE

- 20 to 40 grams of **LERITE** per liter of hydrochloric acid
- The addition of **LERITE** enhances glass wettability and prepares the surface for frosting

1.2 Pre-treatment process

Dipping time: 15 to 20 seconds to reduce HCl transfer into the frosting bath. Over 20 seconds could cause mottling on the surface, affecting final satin quality.

1.3 Pre-treatment bath lifetime

It depends on the cleanliness of the glass and the production rate. It has to be changed regularly. Observing the color change from pale yellow to brown of the pre-treatment bath, is a reliable way to know when it's necessary to renew the pre-treatment bath.

1.4 Importance of the pre-treatment

If there is a poor pre-treatment, it is possible to find defects such as gaps, mottling, stains, fingerprints or lack of frosting.



2. GLASS FROSTING

2.1 Glass frosting bath preparation:

Hydrochloric acid (HCl):

- 40 liters / 10,6 gallons
- 32- 34% concentration
- Density 1.159 Kg HCl/L at 20°C / 68°F

LERITE

- 100 Kg / 220 pounds / 4 boxes.
- Pour 50 Kg / 110 pounds of **LERITE** in a tank.
- Add progressively 20 liters of acid (HCl)
- Mix slowly by manual paddle or mechanical propeller. At first it makes a heavy paste, after thorough mixing, it becomes a viscous liquid.
- When the blend is homogeneous, add another 50 Kg (110 pounds) of **LERITE** and 20 liters (10,6 gallons) of acid.
- The total volume of the frosting bath will vary between 93 and 111 liters depending on the **LERITE** grade.
- The newly prepared bath is rather thick and the temperature drops to approximately -15°C (5°F).
- It is necessary to let the bath mature for at least 24 hours so that the temperature returns to room temperature and viscosity decreases.

2.2 Glass satin etching process

The bottle necks are inserted into rubber fixtures on plastic boards. The boards should have holes to allow a good circulation of the bath during dipping.

There are various types of rubber fixtures, the objective is that they perfectly fit the neck of the bottle to prevent any infiltration of the frosting bath or water.

Dipping time: 30 to 60 seconds.

Dripping time: 10 to 15 seconds to reduce **LERITE** transfer into rinsing bath. The drip off step conserves **LERITE**, and reduces the amount and concentration of waste water to be treated.

2.3 Working conditions

Temperature: the suggested temperature of the bath frosting ranges from 18°C (64°F) to 25°C (77°F). 20°C (68°F) is the standard temperature. It is better to keep the glass item and the frosting bath at same temperature.

Bath homogeneity: a careful attention has to be paid to bath homogeneity. Agitation with recirculation or paddles is recommended. **LERITE** formulas include agents that delay the decantation and maintain the optimal viscosity of the bath.

Glass age: it is better if the glass has been recently produced, if possible less than three months since date of manufacture.

Glass dryness: glass must be dried before the satin etching. Ribbon air blow is recommended. The exception is when a pre-dip treatment is used, as suggested before. In this case the glass should not be dripping, but have a thin layer of the predip bath as it enters the frosting bath.

Density: the control of the **LERITE** bath density enables the decorator to optimize quality and material consumption at it is related to the quality of the satin result.

Density (ρ) is the ratio of weight/volume. To measure it is necessary to weigh a given volume of frosting-bath-sample with a plastic graduated bottle. Ex: 850 ml weighs 1210 g. Bath density is $1210 / 850 = 1.42$ g/ml.



GRADE		DENSITY RANGE OF THE FROSTING BATH
LERITE SX 13	—————>	1.52 < ρ < 1.57
LERITE SX 20	—————>	1.32 < ρ < 1.37
LERITE SX 28	—————>	1.36 < ρ < 1.41
LERITE SX 55	—————>	1.41 < ρ < 1.46
LERITE SX 61	—————>	1.38 < ρ < 1.43
LERITE SX 86	—————>	1.52 < ρ < 1.57

**When taking a sample of to measure the density, it is vital to ensure that the frosting bath is well agitated and perfectly homogeneous.*

2.4 Refill

LERITE glass frosting bath has an almost unlimited lifetime as it is not thrown away but recharged regularly with a fresh mixture of hydrochloric acid and **LERITE**. The composition of the **LERITE** refill bath and frequency of replenishment can affect quality and productivity. The bath should be refilled mid-shift and at shift end as experience dictates. Anytime the glass is not entirely covered, more refill should be added, to prevent quality issues on the finished glass.

Composition of the refill:

The composition of the refill supply depends on the presence of pre-treatment or not. After the pre-treatment bath, the glassware carries along some acid from pre-treatment bath.

REFILL	WITHOUT PRE-TREATMENT	WITH PRE-TREATMENT
LERITE	100 Kg / 220 pounds	100 Kg / 220 pounds
HYDROCHLORIC ACID 32-34%	37 L / 9.8 gallon	Up to 20 L / 5.3 gallon

Frequency

We suggest refilling the frosting bath once every 4 hours, or as needed to maintain level of the bath.

3. RINSING AND DRYING

TYPES OF RINSING

- 1 Dipping into water. This bath should be changed once every 2 hours.
- 2 Water spray.
- 3 Dipping into acidulated water (20% HCl - 80% water).
- 4 Dipping into demineralized water (for colored glass in particularly) or hot water.

Defects due to poor rinsing:

- Frosting quality can be affected by inappropriate rinsing.
- Powder residue, especially on the shoulders and the bottom of the bottle.
- Mottling.

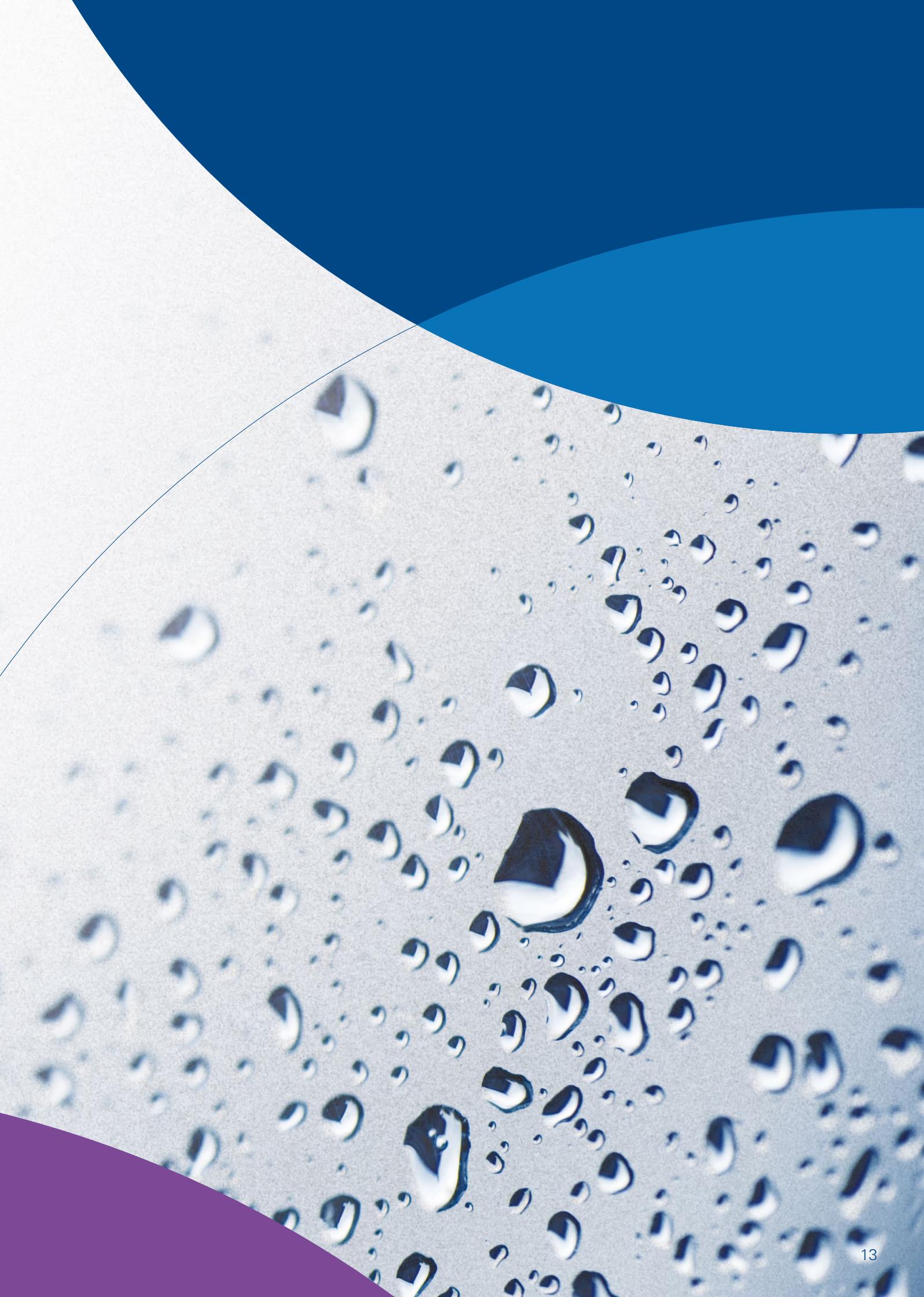
Rinse effluents:

- Contain fluorine ions F⁻ 5000 mg/L and ammonium ions NH₄⁺ 2500 mg/L
- Treatment of fluoride: lime precipitation
- Treatment of ammonium: difficult due to their high solubility
 - Biological treatment
 - Stripping
 - Evaporation under reduced pressure

For more information request the brochure about treatment of effluents and most importantly check with local environmental authorities the requirements for compliance with the local disposal regulation.

DRYING

Infra red equipment or hot air circulation (air knife) are the most appropriate dryers.



HANDLING, PACKAGING & STORAGE

PACKAGING

LERITE is packaged in **25 Kg (55 lb) card-board boxes** with an internal polyethylene bag.

Dimensions of the box: **45 x 29 x 32 cm (18 x 11 x 13 in)**

These boxes are palletized into: **900 Kg (1980 lb) net weight**
and approximate **954 Kg (2103 lb) gross weight** of the pallet.

Dimensions of the pallet: **100 x 120 x 140 cm (39 x 47 x 55 in)**



STORAGE

Store indoors at room temperature with adequate ventilation and away from leaks or water sources.

HANDLING

As **LERITE** contains ammonium bifluoride, special precautions related to toxic chemicals must be taken (see Safety Data Sheet).

Wear rubber gloves (Viton or Nitrile), protective goggles and boots.

Use only polyvinyl chloride (PVC) or polypropylene (PP) equipment.

An appropriate exhaust system placed at surface of pre-treatment and frosting bath is necessary, as fumes are heavier than air.

If operators have to lean above frosting bath, a dome hood system placed overhead is proscribed.

In case of contact with eyes, flush immediately with plenty of water and consult a physician.

Toxic if swallowed: make sure that bottles are perfectly sealed before frosting and check that no leakage has occurred during the whole process. The neck of any bottles to be frosted must be well sealed to prevent interior contamination of the bottles.



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