

Freeze-Drying

Introduction

Freeze-drying, or lyophilization, is a dehydration process that is often used to preserve a perishable food or material. It is also frequently used as a way to remove water from goods, making them lighter and easier to transport. Lyophilization is widely recognized as an important technique in many industries. It is used, for example, in the pharmaceutical industry for the preservation of antibiotics, in the manufacturing of semiconductor ceramics, and by preservation initiatives, such as in the restoration of water-damaged documents. And of course, the process is widely used in the food industry, to preserve tasty snacks that can last up to 30 years. The wet substance is frozen and ice (or some other frozen solvent) is removed through sublimation in the presence of a high vacuum.

Freeze-drying relies on sublimation, where a frozen liquid undergoes phase change directly from a frozen state and into a gaseous state. As can be shown in a phase diagram, at very low pressures and temperatures, a solid can pass directly into the gaseous stage without passing through the intermediary liquid phase.

This model demonstrates the process of ice sublimation in a vial under vacuum-chamber conditions, a test case for many freeze-drying setups.

Model Definition

In this example, you model the process of sublimation of ice through the pores of a porous medium in a vial. Figure 1 depicts the model geometry cut in half.



Figure 1: Vial model geometry.

Since the geometry and boundary conditions are axisymmetric, a 2D axisymmetric model would be sufficient. However, for demonstration purposes and to make the model more general, a good compromise considering the computation time is to solve the model in 3D with a symmetry on the y = 0 plane.

MASS TRANSFER

The frozen product (here skim milk) initially fills 98% of the vial. The remaining gap contains the dried product and the vapor generated by sublimation, and expands during the sublimation process. The inert gas is neglected in the simulation.

The vapor flow rate $Q_{\rm m}$ through the pores of the dried product is defined by the stationary Darcy's law:

$$\nabla \cdot \left(-\frac{\rho_{\rm v} \kappa}{\mu_{\rm v}} \nabla p_{\rm v} \right) = Q_{\rm m} \tag{1}$$

where ρ_v is the vapor density, μ_v is the vapor dynamic viscosity, p_v is the vapor partial pressure, and κ is the permeability of the dried product.

The top of the vial is close to vacuum, maintained at a vapor pressure p_0 of 24 Pa.

At the sublimation interface, the mass flux N_0 is:

$$N_0 = \varepsilon \rho_{\rm ice} V_{\rm s} \tag{2}$$

where ρ_{ice} is the ice density, ε is the porosity of the product, and V_s is the sublimation interface velocity.

HEAT TRANSFER

You solve the heat transfer equation for porous media, without convection in the frozen domain,

$$(\rho C_p)_{\rm eff,\,fr} \frac{\partial T}{\partial t} + \nabla \cdot (-k_{\rm eff,\,fr} \nabla T) = 0 \tag{3}$$

and with convection in the dried domain

$$(\rho C_p)_{\text{eff, dr}} \frac{\partial T}{\partial t} + \rho_v C_{p, v} \mathbf{u} \cdot \nabla T + \nabla \cdot (-k_{\text{eff, dr}} \nabla T) = 0$$
(4)

Here, $(\rho C_p)_{\text{eff,dr}}$ and $k_{\text{eff,dr}}$ refer to the equivalent volumetric heat capacity and thermal conductivity of the dried region, $(\rho C_p)_{\text{eff,fr}}$ and $k_{\text{eff,fr}}$ refer to the equivalent volumetric heat capacity and thermal conductivity of the frozen region, $C_{p,v}$ is the specific heat capacity of vapor, and **u** is the velocity obtained from Darcy's law.

In the vapor domain, the density is calculated using the molar mass of vapor, M_v , and the ideal gas law:

$$\rho_{\rm v} = \frac{M_{\rm v} p_{\rm v}}{RT} \tag{5}$$

where R is the universal gas constant.

Convective exchanges with the surrounding air occur all around the vial. At the bottom of the vial, convective exchanges with the shelf occur as well, and the heat transfer coefficient is lower on the center than on the exterior of the vial due to an air gap.

ICE-VAPOR INTERFACE

Assuming thermodynamic equilibrium at the phase change interface, the sublimation front temperature T_s is defined from the vapor pressure p_v at this boundary, using the Clausius–Clapeyron relation:

$$T_{\rm s} = 2.19 \times 10^{-3} \frac{L_s}{(28.89 - \log(p_{\rm v}))} \tag{6}$$

where L_{s} is the sublimation latent heat.

Simultaneous heat and mass balances at the sublimation interface lead to the Stefan condition for the interface velocity V_s :

$$V_{\rm s} = \frac{Q_{\rm s}}{\rho_{\rm ice} L_{\rm s}} \tag{7}$$

where $Q_{\rm s}$ is the jump in the normal heat flux at the interface.

Results

The process of primary drying takes about 10 hours to transform most of the initial amount of ice. The frozen product has the proportions shown in Figure 2.

Time=10.032 h Left: Final interface position; right: temperature and total heat fluxes



Figure 2: Temperature and heat flux at the end of the drying time period.

You can see from this visualization that the sublimation front has a concave curved shape that is lower around the outside edges of the vial wall. As the ice becomes a vapor, the mass of the solid decreases and therefore the mesh in the frozen domain squeezes as well.

Notes About the COMSOL Implementation

To set up the application in COMSOL Multiphysics, use the Deformed Geometry interface to track the ice surface, and then compute coupled mass and heat balances on the moving mesh. Figure 3 shows the initial mesh and Figure 4 shows the deformed mesh at the end of the simulation.



Figure 3: Initial swept mesh.



Figure 4: Deformed mesh at the end of the simulation.

In Equation 7, Q_s corresponds to the jump in the normal heat flux at the interface. This quantity can be precisely evaluated through the Lagrange multiplier for temperature, T_1m. This variable is computed in the **Phase Change Interface** feature, which sets a fixed temperature weak constraint on the sublimation front.

References

1. A. Alonso and others, "Time-scale modeling and optimal control of freeze-drying," *Journal of Food Engineering*, vol. 111, pp. 655–666, 2012.

2. W.J. Mascarenhas, H.U. Akay, and M.J. Pikal, "A computational model for finite element analysis of the freeze-drying process," *Computer Methods on Applied Mechanics and Engineering*, vol. 148, pp. 105–124, 1997.

3. Hriberšek and others, "Lyophilization model of mannitol water solution in a laboratory scale lyophilizer", *Journal of Drug Delivery Science and Technology*, vol. 45, pp. 28–38, 2018.

Application Library path: Heat_Transfer_Module/Phase_Change/freeze_drying

Modeling Instructions

From the File menu, choose New.

NEW

In the New window, click 🙆 Model Wizard.

MODEL WIZARD

- I In the Model Wizard window, click 间 3D.
- 2 In the Select Physics tree, select Fluid Flow>Porous Media and Subsurface Flow> Darcy's Law (dl).
- 3 Click Add.
- 4 In the Select Physics tree, select Heat Transfer>Porous Media> Heat Transfer in Porous Media (ht).
- 5 Click Add.
- 6 In the Select Physics tree, select Mathematics>Deformed Mesh>Legacy Deformed Mesh> Deformed Geometry (dg).
- 7 Click Add.
- 8 Click 🔿 Study.
- 9 In the Select Study tree, select General Studies>Time Dependent.
- 10 Click 🗹 Done.

For this model, some parameters are needed. Start by loading all of them from a text file.

GLOBAL DEFINITIONS

Parameters 1

- I In the Model Builder window, under Global Definitions click Parameters I.
- 2 In the Settings window for Parameters, locate the Parameters section.
- **3** Click **b** Load from File.
- 4 Browse to the model's Application Libraries folder and double-click the file freeze_drying_parameters.txt.

Add empty materials that will be linked later to the porous materials.

Vapor

- I In the Model Builder window, under Global Definitions right-click Materials and choose Blank Material.
- 2 In the Settings window for Material, type Vapor in the Label text field.

Ice

- I Right-click Materials and choose Blank Material.
- 2 In the Settings window for Material, type Ice in the Label text field.

Product (Skim Milk)

- I Right-click Materials and choose Blank Material.
- 2 In the Settings window for Material, type Product (Skim Milk) in the Label text field.

GEOMETRY I

- I In the Model Builder window, under Component I (compl) click Geometry I.
- 2 In the Settings window for Geometry, locate the Units section.
- 3 From the Length unit list, choose mm.

Cylinder I (cyl1)

- I In the **Geometry** toolbar, click 💭 **Cylinder**.
- 2 In the Settings window for Cylinder, locate the Size and Shape section.
- 3 In the Radius text field, type R0.
- **4** In the **Height** text field, type Z0.
- 5 Click to expand the Layers section. In the table, enter the following settings:

Layer name	Thickness (mm)
Layer 1	Zi

- 6 Clear the Layers on side check box.
- 7 Select the Layers on top check box.

Cylinder 2 (cyl2)

- I In the Geometry toolbar, click 问 Cylinder.
- 2 In the Settings window for Cylinder, locate the Size and Shape section.
- 3 In the Radius text field, type R0.
- 4 In the **Height** text field, type Z0.

5 Locate the Layers section. In the table, enter the following settings:

Layer name	Thickness (mm)
Layer 1	R0/2

Block I (blk1)

- I In the **Geometry** toolbar, click **[]** Block.
- 2 In the Settings window for Block, locate the Size and Shape section.
- 3 In the Width text field, type R0*5.
- 4 In the **Depth** text field, type R0*2.
- 5 In the **Height** text field, type Z0.
- 6 Locate the Position section. In the x text field, type -R0*2.
- 7 In the y text field, type -R0*2.

Difference I (dif I)

- I In the Geometry toolbar, click i Booleans and Partitions and choose Difference.
- 2 In the Settings window for Difference, locate the Difference section.
- 3 Click **Paste Selection**.
- 4 In the Paste Selection dialog box, type cyl1 cyl2 in the Selection text field.
- 5 Click OK.
- 6 In the Settings window for Difference, locate the Difference section.
- **7** Find the **Objects to subtract** subsection. Click to select the **Delta Activate Selection** toggle button.
- 8 Select the object **blk1** only.
- 9 Click 🟢 Build All Objects.
- **IO** Click the \longleftrightarrow **Zoom Extents** button in the **Graphics** toolbar.

These steps create a geometry similar to that in Figure 1.

DEFINITIONS

Interface

- I In the Model Builder window, expand the Component I (compl)>Definitions node.
- 2 Right-click **Definitions** and choose **Variables**.

Define the sublimation temperature at the phase change interface according to the Clausius-Clapeyron equation.

3 In the Settings window for Variables, type Interface in the Label text field.

4 Locate the Variables section. In the table, enter the following settings:

Name	Expression	Unit	Description
T_s	2.19e-3*DelHs*1[kg/J]/ (28.89-log(p*1[1/Pa]))* 1[K]	К	Interface temperature

Integration 1 (intop1)

- I In the Definitions toolbar, click 🖉 Nonlocal Couplings and choose Integration.
- 2 In the Settings window for Integration, locate the Source Selection section.
- **3** Click **Paste Selection**.
- 4 In the Paste Selection dialog box, type 1 3 5 in the Selection text field.
- 5 Click OK.

Minimum I (minop I)

- I In the Definitions toolbar, click 🖉 Nonlocal Couplings and choose Minimum.
- 2 In the Settings window for Minimum, locate the Source Selection section.
- **3** From the Geometric entity level list, choose Boundary.
- 4 Click **Paste Selection**.
- 5 In the Paste Selection dialog box, type 6 13 20 in the Selection text field.
- 6 Click OK.

Add porous materials to the materials node, but do not define their properties at this point. After setting up the physics interface, COMSOL Multiphysics automatically detects which material properties are needed.

MATERIALS

Porous Material I (pmat1)

In the Model Builder window, under Component I (compl) right-click Materials and choose More Materials>Porous Material.

Porous Material 2 (pmat2)

- I Right-click Materials and choose More Materials>Porous Material.
- **2** Select Domains 1, 3, and 5 only.

Now, set up the physics interfaces. Start with the Darcy's law interface, that simulates the vapor flow through the pores of the dried layer.

- **3** Click the **5** Show More Options button in the Model Builder toolbar.
- 4 In the Show More Options dialog box, in the tree, select the check box for the node Physics>Advanced Physics Options.
- 5 Click OK.

DARCY'S LAW (DL)

- I In the Model Builder window, under Component I (compl) click Darcy's Law (dl).
- 2 Select Domains 2, 4, and 6 only.
- 3 In the Settings window for Darcy's Law, click to expand the Equation section.
- 4 From the Equation form list, choose Stationary.
- **5** Locate the **Physical Model** section. In the p_{ref} text field, type **0**.
- **6** Click to expand the **Discretization** section. Select the **Compute boundary fluxes** check box.
- 7 Select Domains 2, 4, and 6 only.

Fluid I

- In the Model Builder window, under Component I (compl)>Darcy's Law (dl)> Porous Medium I click Fluid I.
- 2 In the Settings window for Fluid, locate the Fluid Properties section.
- 3 From the Fluid type list, choose Ideal gas.
- 4 From the Gas constant type list, choose Mean molar mass.

Initial Values 1

- I In the Model Builder window, under Component I (compl)>Darcy's Law (dl) click Initial Values I.
- 2 In the Settings window for Initial Values, locate the Initial Values section.
- **3** In the *p* text field, type Pc.

Pressure 1

- I In the Physics toolbar, click 📄 Boundaries and choose Pressure.
- 2 Select Boundaries 7, 14, and 21 only.
- 3 In the Settings window for Pressure, locate the Pressure section.
- **4** In the p_0 text field, type Pc.

Mass Flux 1

I In the Physics toolbar, click 📄 Boundaries and choose Mass Flux.

- 2 Select Boundaries 6, 13, and 20 only.
- 3 In the Settings window for Mass Flux, locate the Mass Flux section.
- **4** In the N_0 text field, type -ht.pci1.vn*rho_ice*por_p.

Symmetry I

- I In the Physics toolbar, click 🔚 Boundaries and choose Symmetry.
- **2** Select Boundaries 4, 12, and 25 only.

HEAT TRANSFER IN POROUS MEDIA (HT)

Now, set up the Heat Transfer in Porous Media interface, in both dried and frozen layers.

Dried Layer

I In the Model Builder window, under Component I (compl)>

Heat Transfer in Porous Media (ht) right-click Porous Medium I and choose Rename.

- 2 In the Rename Porous Medium dialog box, type Dried Layer in the New label text field.
- 3 Click OK.

Fluid I

- I In the Model Builder window, click Fluid I.
- 2 In the Settings window for Fluid, locate the Heat Convection section.
- **3** From the **u** list, choose **Darcy's velocity field (dl/porous I)**.
- 4 Locate the Thermodynamics, Fluid section. From the Fluid type list, choose Ideal gas.
- 5 From the Gas constant type list, choose Mean molar mass.

Porous Matrix I

- I In the Model Builder window, click Porous Matrix I.
- 2 In the Settings window for Porous Matrix, locate the Matrix Properties section.
- 3 From the Define list, choose Solid phase properties.

Initial Values 1

- I In the Model Builder window, under Component I (compl)> Heat Transfer in Porous Media (ht) click Initial Values I.
- 2 In the Settings window for Initial Values, locate the Initial Values section.
- **3** In the *T* text field, type Ti.

Frozen Layer

- I In the Physics toolbar, click 🔚 Domains and choose Porous Medium.
- 2 In the Settings window for Porous Medium, type Frozen Layer in the Label text field.

3 Select Domains 1, 3, and 5 only.

Porous Matrix I

- I In the Model Builder window, click Porous Matrix I.
- 2 In the Settings window for Porous Matrix, locate the Matrix Properties section.
- **3** From the **Define** list, choose **Solid phase properties**.

Ambient Heat Flux

- I In the Physics toolbar, click 🔚 Boundaries and choose Heat Flux.
- 2 In the Settings window for Heat Flux, type Ambient Heat Flux in the Label text field.
- 3 Locate the Heat Flux section. From the Flux type list, choose Convective heat flux.
- 4 In the *h* text field, type 3.6.
- **5** In the T_{ext} text field, type Ta.
- 6 Locate the Boundary Selection section. Click 📄 Paste Selection.
- 7 In the Paste Selection dialog box, type 2 5 7 14 21 22 23 in the Selection text field.
- 8 Click OK.

Surface-to-Ambient Radiation I

- I In the Physics toolbar, click 🔚 Boundaries and choose Surface-to-Ambient Radiation.
- **2** Select Boundaries 7, 14, and 21 only.
- 3 In the Settings window for Surface-to-Ambient Radiation, locate the Surface-to-Ambient Radiation section.
- **4** From the ε list, choose **User defined**. In the associated text field, type **0.9**.
- **5** In the T_{amb} text field, type Ta.

Shelf Heat Flux (Center)

- I In the Physics toolbar, click 🔚 Boundaries and choose Heat Flux.
- 2 In the Settings window for Heat Flux, type Shelf Heat Flux (Center) in the Label text field.
- **3** Locate the Boundary Selection section. Click Paste Selection.
- 4 In the Paste Selection dialog box, type 10 in the Selection text field.
- 5 Click OK.
- 6 In the Settings window for Heat Flux, locate the Heat Flux section.
- 7 From the Flux type list, choose Convective heat flux.
- **8** In the h text field, type 11.

9 In the T_{ext} text field, type Ts.

Shelf Heat Flux (Exterior)

- I In the Physics toolbar, click 🔚 Boundaries and choose Heat Flux.
- 2 In the Settings window for Heat Flux, type Shelf Heat Flux (Exterior) in the Label text field.
- 3 Locate the Boundary Selection section. Click 🗂 Paste Selection.
- 4 In the Paste Selection dialog box, type 3 17 in the Selection text field.
- 5 Click OK.
- 6 In the Settings window for Heat Flux, locate the Heat Flux section.
- 7 From the Flux type list, choose Convective heat flux.
- **8** In the h text field, type 62.3.
- **9** In the T_{ext} text field, type Ts.

Symmetry I

- I In the Physics toolbar, click 🔚 Boundaries and choose Symmetry.
- **2** Select Boundaries 1, 4, 9, 12, 24, and 25 only.

Phase Change Interface 1

- I In the Physics toolbar, click 📄 Boundaries and choose Phase Change Interface.
- 2 In the Settings window for Phase Change Interface, locate the Boundary Selection section.
- **3** Click **Paste Selection**.
- 4 In the Paste Selection dialog box, type 6 13 20 in the Selection text field.
- 5 Click OK.
- **6** In the **Settings** window for **Phase Change Interface**, locate the **Phase Change Interface** section.
- 7 In the $T_{\rm pc}$ text field, type T_s.
- 8 In the $L_{s \to f}$ text field, type DelHs.
- 9 From the Solid side list, choose Downside.

DEFORMED GEOMETRY (DG)

Finish with the Deformed Geometry interface under the Definitions node. Roller boundaries are set up along the vial and the symmetry axis. The **Phase Change Interface** feature in the Heat transfer interface already specifies a prescribed normal mesh velocity.

I In the Model Builder window, under Component I (compl) click Deformed Geometry (dg).

- 2 In the Settings window for Deformed Geometry, locate the Frame Settings section.
- 3 From the Geometry shape function list, choose I.
- **4** Locate the **Free Deformation Settings** section. From the **Mesh smoothing type** list, choose **Hyperelastic**.

Free Deformation I

- I In the Physics toolbar, click 🔚 Domains and choose Free Deformation.
- 2 In the Settings window for Free Deformation, locate the Domain Selection section.
- **3** From the Selection list, choose All domains.

Prescribed Mesh Displacement 2

- I In the Physics toolbar, click 📄 Boundaries and choose Prescribed Mesh Displacement.
- **2** In the **Settings** window for **Prescribed Mesh Displacement**, locate the **Boundary Selection** section.
- 3 Click Clear Selection.
- 4 Select Boundaries 1, 2, 4, 5, 9, 12, and 22–25 only.
- **5** Locate the **Prescribed Mesh Displacement** section. Clear the **Prescribed Z displacement** check box.

MATERIALS

The material properties can now be specified.

Porous Material I (pmat1)

- I In the Model Builder window, under Component I (compl)>Materials click Porous Material I (pmatl).
- 2 In the Settings window for Porous Material, locate the Homogenized Properties section.
- **3** In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Permeability	kappa_iso ; kappaii = kappa_iso, kappaij = 0	kappa_p	m²	Basic

4 Locate the Phase-Specific Properties section. Click **H** Add Required Phase Nodes.

Solid I (pmat1.solid1)

I In the Model Builder window, click Solid I (pmat1.solid1).

- 2 In the Settings window for Solid, locate the Solid Properties section.
- 3 From the Material list, choose Product (Skim Milk) (mat3).
- **4** In the θ_s text field, type 1-por_p.

Porous Material 2 (pmat2)

- I In the Model Builder window, under Component I (compl)>Materials click Porous Material 2 (pmat2).
- 2 In the Settings window for Porous Material, locate the Phase-Specific Properties section.
- **3** Click **Add Required Phase Nodes**.

Fluid I (pmat2.fluid1)

- I In the Model Builder window, click Fluid I (pmat2.fluidI).
- 2 In the Settings window for Fluid, locate the Fluid Properties section.
- 3 From the Material list, choose Ice (mat2).

Solid I (pmat2.solid I)

- I In the Model Builder window, click Solid I (pmat2.solidI).
- 2 In the Settings window for Solid, locate the Solid Properties section.
- 3 From the Material list, choose Product (Skim Milk) (mat3).
- **4** In the θ_s text field, type 1-por_p.

GLOBAL DEFINITIONS

Vapor (mat1)

- I In the Model Builder window, under Global Definitions>Materials click Vapor (mat1).
- 2 In the Settings window for Material, locate the Material Contents section.
- **3** In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Mean molar mass	Mn	M_v	kg/mol	Basic
Dynamic viscosity	mu	mu_v	Pa·s	Basic
Heat capacity at constant pressure	Ср	Cp_v	J/(kg·K)	Basic
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	k_v	W/(m·K)	Basic

Ice (mat2)

- I In the Model Builder window, click lce (mat2).
- 2 In the Settings window for Material, locate the Material Contents section.
- **3** In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Heat capacity at constant pressure	Ср	Cp_ice	J/(kg·K)	Basic
Density	rho	rho_ice	kg/m³	Basic
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	k_ice	W/(m·K)	Basic

Product (Skim Milk) (mat3)

- I In the Model Builder window, click Product (Skim Milk) (mat3).
- 2 In the Settings window for Material, locate the Material Contents section.
- **3** In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Heat capacity at constant pressure	Ср	Ср_р	J/(kg·K)	Basic
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	k_p	W/(m·K)	Basic
Density	rho	rho_p	kg/m³	Basic

MESH I

Set up a uniform mesh in the vertical direction in order to ease the mesh displacement and to avoid remeshing during the computation.

- I In the Model Builder window, under Component I (compl) click Mesh I.
- 2 In the Settings window for Mesh, locate the Sequence Type section.
- 3 From the list, choose User-controlled mesh.

Size

- I In the Model Builder window, under Component I (compl)>Mesh I click Size.
- 2 In the Settings window for Size, locate the Element Size section.
- 3 From the Predefined list, choose Finer.

Free Tetrahedral I

In the Model Builder window, right-click Free Tetrahedral I and choose Disable.

Free Triangular 1

- I In the Mesh toolbar, click A Boundary and choose Free Triangular.
- 2 Select Boundaries 7, 14, and 21 only.

Size 1

- I Right-click Free Triangular I and choose Size.
- 2 In the Settings window for Size, locate the Element Size section.
- 3 From the Predefined list, choose Extra fine.
- **4** Locate the **Geometric Entity Selection** section. From the **Geometric entity level** list, choose **Edge**.
- 5 Select Edges 8 and 29 only.

Boundary Layers 1

- I In the Mesh toolbar, click Boundary Layers.
- 2 In the Settings window for Boundary Layers, locate the Geometric Entity Selection section.
- 3 From the Geometric entity level list, choose Boundary.
- 4 Select Boundaries 7 and 21 only.

Boundary Layer Properties

- I In the Model Builder window, click Boundary Layer Properties.
- 2 Select Edges 8 and 29 only.
- 3 In the Settings window for Boundary Layer Properties, locate the Layers section.
- 4 In the Number of layers text field, type 4.

Swept I

- I In the Mesh toolbar, click A Swept.
- 2 In the Settings window for Swept, click to expand the Source Faces section.
- **3** Select Boundaries 7, 14, and 21 only.
- 4 Click to expand the Destination Faces section. Select Boundaries 3, 10, and 17 only.

Distribution I

- I Right-click Swept I and choose Distribution.
- 2 In the Settings window for Distribution, locate the Domain Selection section.
- 3 Click Clear Selection.

4 Select Domains 2, 4, and 6 only.

5 Locate the Distribution section. In the Number of elements text field, type 16.

Distribution 2

- I In the Model Builder window, right-click Swept I and choose Distribution.
- 2 In the Settings window for Distribution, locate the Domain Selection section.
- 3 Click Clear Selection.
- 4 Select Domains 1, 3, and 5 only.
- 5 Locate the Distribution section. In the Number of elements text field, type 8.

STUDY I

The necessary time to dry the product is unknown. An initial guess of less than 24 hours is made.

Step 1: Time Dependent

- I In the Model Builder window, under Study I click Step I: Time Dependent.
- 2 In the Settings window for Time Dependent, locate the Study Settings section.
- **3** From the **Time unit** list, choose **h**.
- 4 In the **Output times** text field, type range(0,0.1,0.5) range(0.5,0.5,24).

Solution 1 (soll)

- I In the Study toolbar, click **Show Default Solver**.
- 2 In the Model Builder window, expand the Solution I (soll) node.

Add a stop condition to stop the simulation when the interface is close to the bottom of the vial.

- 3 In the Model Builder window, expand the Study I>Solver Configurations> Solution I (soll)>Time-Dependent Solver I node.
- 4 Right-click Study I>Solver Configurations>Solution I (soll)>Time-Dependent Solver I and choose Stop Condition.
- 5 In the Settings window for Stop Condition, locate the Stop Expressions section.
- 6 Click + Add.
- 7 In the table, enter the following settings:

Stop expression	Stop if	Active	Description
<pre>comp1.minop1(z)/Z0- 0.03</pre>	Negative (<0)	\checkmark	Interface close to the vial bottom

- 8 Locate the Output at Stop section. From the Add solution list, choose Step after stop.
- 9 Clear the Add warning check box.

Switch from segregated solver to a fully coupled one to ease the convergence.

- IO Right-click Time-Dependent Solver I and choose Fully Coupled.
- **II** In the **Settings** window for **Fully Coupled**, click to expand the **Method and Termination** section.
- **12** In the **Damping factor** text field, type 0.9.
- **I3** From the **Jacobian update** list, choose **Once per time step**.
- 14 From the Stabilization and acceleration list, choose Anderson acceleration.
- **I5** In the **Study** toolbar, click **= Compute**.

RESULTS

Only half of the vial geometry has been built for the calculations. In the next steps, mirror plots are defined to visualize the entire geometry in postprocessing plots.

Mirror 3D I

- I In the Model Builder window, expand the Results>Datasets node.
- 2 Right-click Results>Datasets and choose More 3D Datasets>Mirror 3D.
- 3 In the Settings window for Mirror 3D, locate the Plane Data section.
- 4 From the Plane list, choose XZ-planes.

Cut Point 3D 1

- I In the **Results** toolbar, click **Cut Point 3D**.
- 2 In the Settings window for Cut Point 3D, locate the Point Data section.
- **3** In the **X** text field, type **0**.
- **4** In the **Y** text field, type **0**.
- 5 In the Z text field, type range (Z0, -Z0/6,0).

6 From the Snapping list, choose Snap to closest boundary.

To create the plots shown in Figure 3 and Figure 4, reproduce the following steps.

Mesh

- I In the Results toolbar, click 间 3D Plot Group.
- 2 In the Settings window for 3D Plot Group, type Mesh in the Label text field.

Mesh I

I Right-click Mesh and choose Mesh.

- 2 In the Settings window for Mesh, locate the Coloring and Style section.
- 3 Click Change Color Table.
- 4 In the Color Table dialog box, select Aurora>AuroraBorealis in the tree.
- 5 Click OK.

Selection 1

- I Right-click Mesh I and choose Selection.
- 2 Select Boundaries 1-3, 6, 9, 10, 13, 17, 20, 22, and 24 only.
- 3 In the Mesh toolbar, click 💿 Plot.
- **4** Click the 4 **Zoom Extents** button in the **Graphics** toolbar.

Initial Mesh

- I In the Model Builder window, right-click Mesh and choose Duplicate.
- 2 In the Settings window for 3D Plot Group, type Initial Mesh in the Label text field.
- 3 Locate the Data section. From the Time (h) list, choose 0.
- **4** In the **Initial Mesh** toolbar, click **I** Plot.
- **5** Click the |+| **Zoom Extents** button in the **Graphics** toolbar.

Sublimation Interface

To create the plot shown in Figure 2, follow these steps.

- I In the Home toolbar, click 🚛 Add Plot Group and choose 3D Plot Group.
- 2 In the Settings window for 3D Plot Group, type Sublimation Interface in the Label text field.
- 3 Locate the Data section. From the Dataset list, choose Mirror 3D I.
- 4 Click to expand the Title section. From the Title type list, choose Manual.
- **5** In the **Title** text area, type Left: Final interface position; right: temperature and total heat fluxes.

Slice 1

- I Right-click Sublimation Interface and choose Slice.
- 2 In the Settings window for Slice, locate the Expression section.
- **3** In the **Expression** text field, type T.
- 4 Locate the Plane Data section. In the Planes text field, type 1.
- 5 Locate the Coloring and Style section. Click Change Color Table.
- 6 In the Color Table dialog box, select Thermal>HeatCameraLight in the tree.

7 Click OK.

Isosurface I

- I In the Model Builder window, right-click Sublimation Interface and choose Isosurface.
- 2 In the Settings window for Isosurface, locate the Expression section.
- **3** In the **Expression** text field, type Zg.
- 4 Locate the Levels section. From the Entry method list, choose Levels.
- **5** In the **Levels** text field, type Z0-Zi.
- 6 Locate the Coloring and Style section. Click Change Color Table.
- 7 In the Color Table dialog box, select Aurora>JupiterAuroraBorealis in the tree.
- 8 Click OK.
- 9 In the Settings window for Isosurface, locate the Coloring and Style section.
- **IO** Clear the **Color legend** check box.
- II In the Sublimation Interface toolbar, click 💿 Plot.

Streamline 1

- I Right-click Sublimation Interface and choose Streamline.
- In the Settings window for Streamline, click Replace Expression in the upper-right corner of the Expression section. From the menu, choose Component I (compl)>
 Heat Transfer in Porous Media>Domain fluxes>ht.tfluxx,...,ht.tfluxz Total heat flux (spatial and material frames).
- **3** Locate the **Streamline Positioning** section. From the **Positioning** list, choose **Magnitude controlled**.
- **4** Locate the **Coloring and Style** section. Find the **Line style** subsection. From the **Type** list, choose **Tube**.

Color Expression 1

- I Right-click Streamline I and choose Color Expression.
- 2 In the Settings window for Color Expression, locate the Expression section.
- **3** In the **Expression** text field, type T.
- 4 Locate the Coloring and Style section. Clear the Color legend check box.
- 5 Click Change Color Table.
- 6 In the Color Table dialog box, select Thermal>HeatCameraLight in the tree.
- 7 Click OK.

Line I

- I In the Model Builder window, right-click Sublimation Interface and choose Line.
- 2 In the Settings window for Line, locate the Expression section.
- **3** In the **Expression** text field, type **1**.
- 4 Locate the Coloring and Style section. From the Coloring list, choose Uniform.
- 5 From the Color list, choose Gray.
- 6 In the Sublimation Interface toolbar, click 💽 Plot.

Volume 1

- I Right-click Sublimation Interface and choose Volume.
- 2 In the Settings window for Volume, locate the Data section.
- 3 From the Dataset list, choose Study I/Solution I (soll).
- 4 Locate the Expression section. In the Expression text field, type dom==1 | dom==3 | dom==5.
- 5 Locate the Coloring and Style section. Click Change Color Table.
- 6 In the Color Table dialog box, select Aurora>AuroraBorealis in the tree.
- 7 Click OK.
- 8 In the Settings window for Volume, locate the Coloring and Style section.
- 9 Clear the Color legend check box.

Translation 1

- I Right-click Volume I and choose Translation.
- 2 In the Settings window for Translation, locate the Translation section.
- 3 In the x text field, type -2.5*R0.
- 4 Clear the Apply to dataset edges check box.
- **5** In the Sublimation Interface toolbar, click **I** Plot.
- 6 Click the **Zoom Extents** button in the **Graphics** toolbar.

Temperature history

Plot the temperature variation during the primary drying at seven locations equally spread along the vial.

- I In the Home toolbar, click 🚛 Add Plot Group and choose ID Plot Group.
- 2 In the Settings window for ID Plot Group, type Temperature history in the Label text field.
- 3 Locate the Data section. From the Dataset list, choose None.

- 4 Click to expand the Title section. From the Title type list, choose Manual.
- 5 In the Title text area, type Temperature history at seven different heights at the center of the vial.
- 6 Locate the Plot Settings section.
- 7 Select the x-axis label check box. In the associated text field, type Time (h).
- 8 Select the y-axis label check box. In the associated text field, type Temperature (K).
- 9 Locate the Legend section. From the Position list, choose Upper left.

Point Graph 1

- I Right-click Temperature history and choose Point Graph.
- 2 In the Settings window for Point Graph, locate the Data section.
- 3 From the Dataset list, choose Cut Point 3D I.
- 4 Locate the y-Axis Data section. In the Expression text field, type T.
- 5 In the **Temperature history** toolbar, click **I** Plot.
- 6 Click to expand the **Quality** section. Click to expand the **Coloring and Style** section. From the **Width** list, choose **2**.
- 7 Click to expand the Legends section. Select the Show legends check box.
- 8 From the Legends list, choose Evaluated.
- **9** In the Legend text field, type z/Z0=eval(z/Z0).

Ice mass

Then, plot the ice mass decreasing during the primary drying.

- I In the Home toolbar, click 📠 Add Plot Group and choose ID Plot Group.
- 2 In the Settings window for ID Plot Group, type Ice mass in the Label text field.
- **3** Locate the **Title** section. From the **Title type** list, choose **Manual**.
- 4 In the Title text area, type Ice mass relative to initial amount.
- 5 Locate the **Plot Settings** section.
- 6 Select the x-axis label check box. In the associated text field, type Time (h).
- 7 Select the y-axis label check box. In the associated text field, type (%).
- 8 Locate the Legend section. Clear the Show legends check box.

Global I

- I Right-click Ice mass and choose Global.
- 2 In the Settings window for Global, locate the y-Axis Data section.

3 In the table, enter the following settings:

Expression	Unit	Description
comp1.intop1(1)*2/(pi*R0^2*(Z0-Zi))	1	

- **4** In the **Ice mass** toolbar, click **I Plot**.
- 5 Click to expand the Coloring and Style section. From the Width list, choose 2.

Initial Ice Mass

Finally, check the mass conservation: the difference between the initial and the final ice mass should be equal to the amount of vapor that has left the vial.

- I In the **Results** toolbar, click (8.5) **Global Evaluation**.
- 2 In the Settings window for Global Evaluation, type Initial Ice Mass in the Label text field.
- **3** Locate the **Expressions** section. In the table, enter the following settings:

Expression	Unit	Description
<pre>comp1.intop1(rho_ice)*2*por_p</pre>	mg	Ice mass

- 4 Locate the Data Series Operation section. From the Transformation list, choose Maximum.
- 5 Click **= Evaluate**.

Final Ice Mass

- I Right-click Initial Ice Mass and choose Duplicate.
- 2 In the Settings window for Global Evaluation, type Final Ice Mass in the Label text field.
- **3** Locate the **Data Series Operation** section. From the **Transformation** list, choose **Minimum**.
- 4 Click **=** Evaluate.

Vapor Flux

- I In the Results toolbar, click ^{8,85}_{e-12} More Derived Values and choose Integration> Surface Integration.
- 2 In the Settings window for Surface Integration, type Vapor Flux in the Label text field.
- **3** Select Boundaries 7, 14, and 21 only.
- **4** Locate the **Expressions** section. In the table, enter the following settings:

Expression	Unit	Description
dl.bndflux*2	mg/h	Vapor flux leaving the vial

- **5** Locate the **Integration Settings** section. Select the **Integration order** check box.
- 6 Locate the Data Series Operation section. From the Transformation list, choose Integral.
- 7 Click **= Evaluate**.