

FD-Application FD-LS-118

Catchwords: Life Science, peptides, synthesis, sample purification by HPLC, organic solvents, ethanol-water, storage, trifluoroacetic acid, wide neck filter bottles

Lyophilization of peptides after synthesis and HPLC purification with ethanol as eluent

Application:

Service laboratory methods, Service for the Institute: Lyophilization of peptides for storage, 1. crude product after synthesis
2. after HPLC purification

Process technology (summary):

• Product designation	Peptides
• Type of solvent, ca. percentage of dry matter	Aqueous, aqueous-ethanol (up to 70 %)-TFA, proportion of dry matter max. 0,5 %
• Type of vessel, number of samples, volume per sample	Wide neck filter bottles V= 100 ml- 500 ml, on average 3 samples/run
• Type of machine / configuration	ALPHA 2-4 LSC with acrylic glass chamber and heated shelves
• Freezing (place, range of temperature, freezing point)	In dry ice/isopropanol, freezing point not determined
• Process flask-drying /inside /outside /Epsilon*	outside
• Vacuum main-drying (final vacuum or controlled)	30 mbar mbar, controlled
• Temperature of shelf, program mode?	26 °C
• Time duration of main drying (T _{5F} /t)	Overnight
• Final-drying? Vacuum?	Yes, 4 h, 14 mbar

Result and comments:

In the methods the harmful acetonitrile could be replaced as HPLC mobile phase by ethanol. The acquisition of a new FD system with heated shelves simultaneously enables the relatively rapid removal of the eluent in a single operation in the freeze dryer

There was a larger vacuum pump (20 m³/h) used to prevent the thawing of the ethanol samples at the beginning of the process in the phase of the vacuum assembly.

*explanation

Process inside	(Freezing and) drying inside the ice condenser chamber
Process outside	Freezing separately (e.g. freezer), drying outside the ice condenser, e.g. with acrylic chamber
EPSILON	Type of machine with rectangular product chamber, front loader