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Scale-up from the Büchi Mini Spray Dryer B-290 to the Niro MOBILE MINOR™

Scale-up from bench-top research to laboratory production

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Introduction

The two leading suppliers of spray drying systems in their respective sectors, Büchi Labortechnik AG in the laboratory scale and GEA Niro A/S in the industrial scale have teamed up to offer their combined comprehensive knowledge of spray drying applications to their customers. After first trials with a Büchi Mini Spray Dryer B-290, the next task for most users is to go to a bigger scale.
This leaflet describes a hands-on approach to perform a scale-up from initial trials in the research lab with the Mini Spray Dryer B-290 to laboratory production with the Niro MOBILE MINOR™ spray dryer. The MOBILE MINOR™ is itself a natural next step up after the Mini Spray Dryer B-290, as well as being an excellent first trial plant when slightly larger feed quantities are available.
The objectives are to maintain the most important process conditions constant during scale-up, such as to obtain same particle sizes and residual humidity in the produced powders. Guidelines to adjust the droplet size of the spray and the relationships between water evaporation rate, inlet and outlet temperature are explained.

Spray drying technology

Spray drying is a widely applied process technology in the chemical, food and pharmaceutical industries. It is a single unit operation rapidly transforming liquids (solutions, emulsions, slurries, pastes or even melts) into powders in one simple and robust continuous process. A spray of fine droplets generated by an atomizing device (e.g. nozzles) is mixed with a stream of hot air whereby the water evaporates from the droplet while suspended in the drying air.
The evaporation process uses heat from the drying air. Because of the water evaporation from the product, the droplet and powder temperature remain at or below the spray dryer outlet drying air temperature during the entire drying process. Initially the droplet temperature is approaching the wet-bulb temperature and as the particle dries the temperature increases towards the spray dryer outlet drying air temperature.
Drying takes place within seconds due to the very large liquid surface area of the spray and the high heat transfer coefficients. Hence, it is possible to dry at moderate temperatures, which enables even heat sensitive materials to remain active after spray drying.
The rapid drying and consequently fast stabilization of solids in the feed makes spray drying ideal for particle engineering e.g. encapsulation, production of dry emulsions or amorphous material, as well as the ability to adjust powder characteristics such as particle size, density, flowability and dispersability.
The range of product applications continuously expands, so that today spray drying has connections with many things in daily life: milk powder, baby food, instant coffee, pharmaceuticals, detergents or dyes.

The key to success is reproducible drying conditions when the feed droplets convert to particles. The droplet has to dry to a non-sticking particle, while being suspended in the drying chamber. If not, the wet or sticky particle hits the wall and either starts coating the drying chamber or slightly agglomerates with other particles.
The equilibrium of the water in the vapour and the water content in the final product normally depends on temperature and drying air vapour concentration. A drier product is reached with higher outlet temperature and/or lower spray dryer outlet drying air vapour concentrations.
Decreasing the liquid feed rate, whilst maintaining a constant outlet temperature by reducing the inlet temperature, results in a decrease in the drying air vapour concentration.
The drying time of a particle increases with spray chamber size setting up the upper limit of the maximum achievable particle size in a given spray dryer. This limit especially depends on product characteristics and also process parameters. It may be impossible to obtain a usable powder in a small drying chamber, whereas using a larger chamber a robust process can be designed. When spray drying is evaluated as a feasible new technology for new product development, one has to be aware of "false negatives" if only a small drying chamber is used. Contact Büchi's and Niro's experts at the customer test centres for feasibility testing of your product!

Some materials dried in spray dryers exhibit thermoplastic and/or hygroscopic behaviour. Such materials require extra attention. Hygroscopic materials require a higher drying temperature and/or a lower drying air vapour concentration to achieve a desired residual moisture level. Hygroscopic materials have a strong affinity to the used solvents and need to be handled with care because of the easy re-absorption of vapours from the air phase if the temperature drops or the vapour concentration rises. Typical reabsorption places are cold surfaces in the dryer (e.g. non-insulated parts of the dryer), final product discharge containers and, subsequently, during

Current market trends in spray drying:

- Improved bioavailability of drugs
- Drug delivery by inhalable particles
- Biodegradable polymers as matrix materials for drug encapsulation
- Biotech products like yeasts and cell cultures
- Dry powders of agglomerated nanoparticles
- Fuel cell batteries in the automotive industry
- Microencapsulation and masking of fragrances and aromas
- Natural products for traditional Chinese medicine

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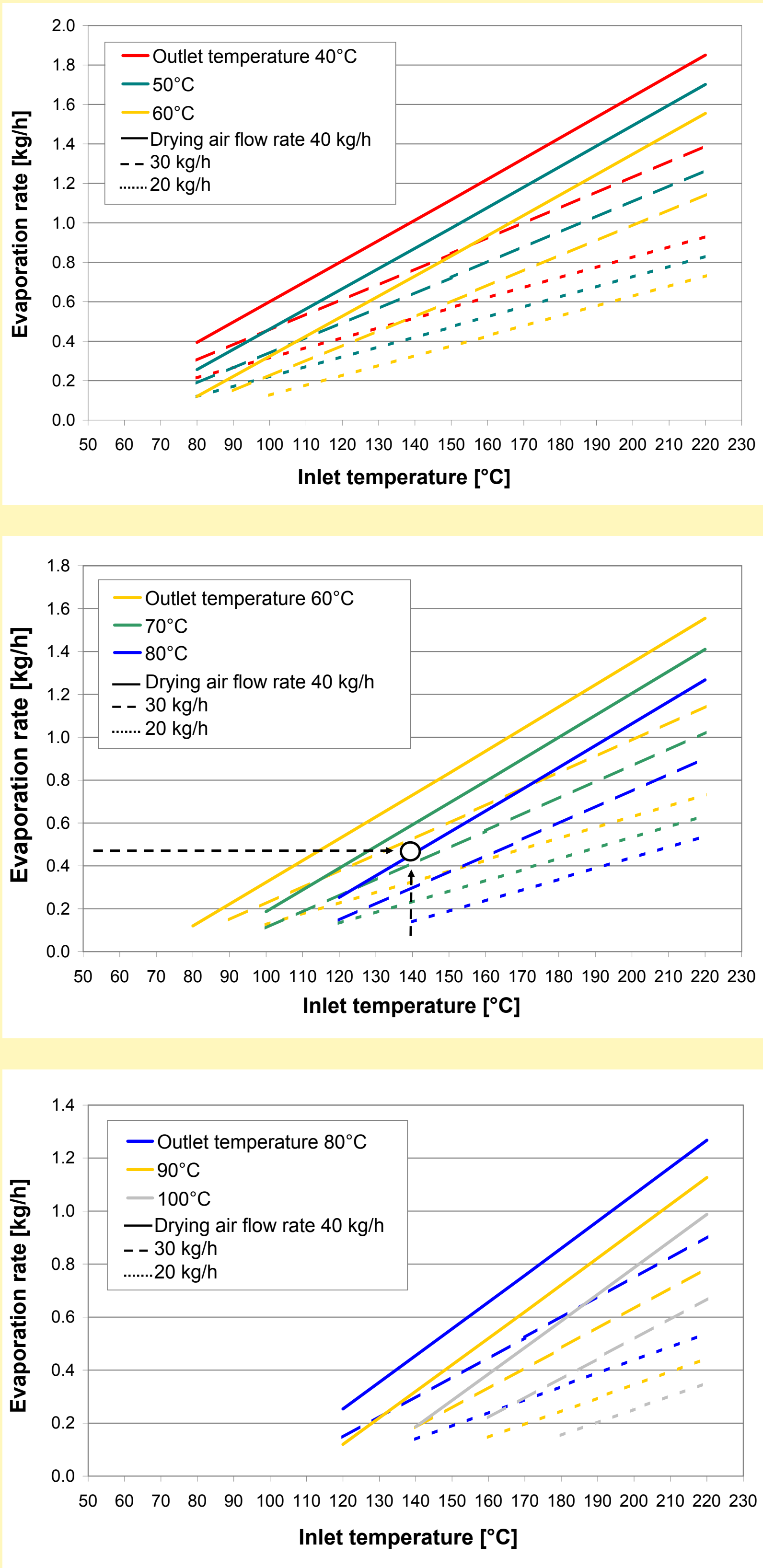

Figure 1: Drying air flow rates in the Mini Spray Dryer B-290 as a function of inlet temperature and water evaporation rate at different outlet temperatures.

laboratory analysis of the powder. For materials exhibiting thermoplastic behaviour, e.g. compounds with low glass transition temperature, the temperature range for drying is limited by two contradicting factors. Such a product becomes sticky at higher temperatures but at the same time the temperature needs to be high enough to dry the product. Moreover, the residual solvent in the particles acts as a plasticizer. This reduces the allowable particle temperature before becoming sticky compared to what may be measured in the laboratory on the dry material.

Scale-up procedure

The starting point of the scale-up procedure is the situation, where good process conditions were found with the Mini Spray Dryer B-290. The task is now to get a powder produced on the MOBILE MINOR™ with similar residual moisture content and particle size, but at a higher production rate.

The target must be to keep the most important process parameters constant and adjust the ones that have the least process impact. In a spray dryer the key process parameters in order of importance are the outlet air temperature, the droplet size and the outlet vapour concentration. The outlet temperature is measured directly and is kept constant during the scale-up. The humidity in the final powder is related to the humidity in the air leaving the drying chamber. To find this condition for the Mini Spray Dryer B-290, the water[1] evaporation rate and the total air flow rate (drying air and atomization air) have to be known.

Firstly, the water evaporation rate is

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[1] *Although air, water, moisture and humidity are used in the text, the descriptions equally cover processes using nitrogen as drying and atomizing gas with solvent vapour and feed liquids based on solvents (e.g. ethanol, acetone etc.). Feed is used to describe any solution or suspension or. The term droplet is used for atomized feed or solvent before turning into particles*

calculated from a simple mass balance with the following parameters: feed flow rate, total solids content in the feed and the residual water content in powder. The feed flow rate can accurately be found by weighing the feed container before and after an experimental run.

$$\mathbf{EVR} = \mathbf{FR} \cdot (1 - \mathbf{TS}) - \mathbf{FR} \cdot \mathbf{TS} \cdot \frac{\mathbf{RW}}{1 - \mathbf{RW}}$$

(Formula 1)

EVR = evaporation rate [kg/h]
FR = feed flow rate [kg/h]
TS = total solids in the feed [kg solids/kg feed]
RW = residual water in the powder [kg water/kg wet powder]

The drying air flow rate in the Mini Spray Dryer B-290 is set by the aspirator setting, which is an indicative reading. However, this is not sufficiently accurate for scale-up calculations as the flow rate changes with the pressure drop across the plant e.g. due to powder build up in the filter. Instead the accurate inlet and outlet air temperature readings are used. Applying the heat and mass balance equation across the spray dryer the evaporation capacity diagrams can be drawn (Figures 1).
Based on the evaporation rate, the inlet and outlet temperatures, the total drying air flow rate (in kg/h) can be estimated. The error variations due to two-fluid nozzle atomization air flow rates are negligible (error < 5%).

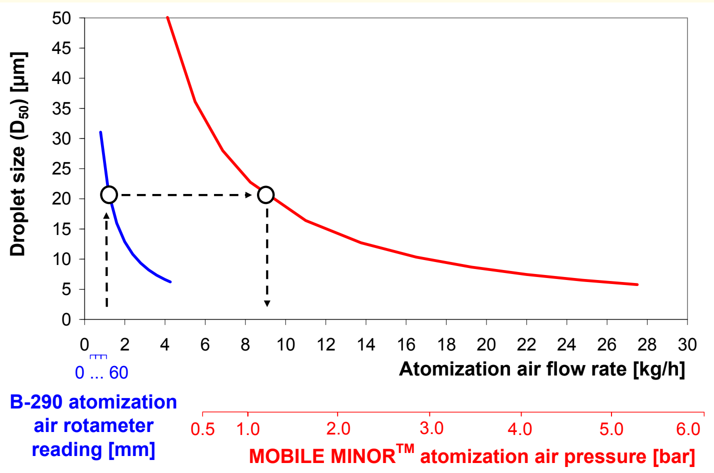

Figure 2: Water droplet size (D_{50}) as a function of atomization air flow rate for the MOBILE MINOR™ and the Mini Spray Dryer B-290 two-fluid nozzles.

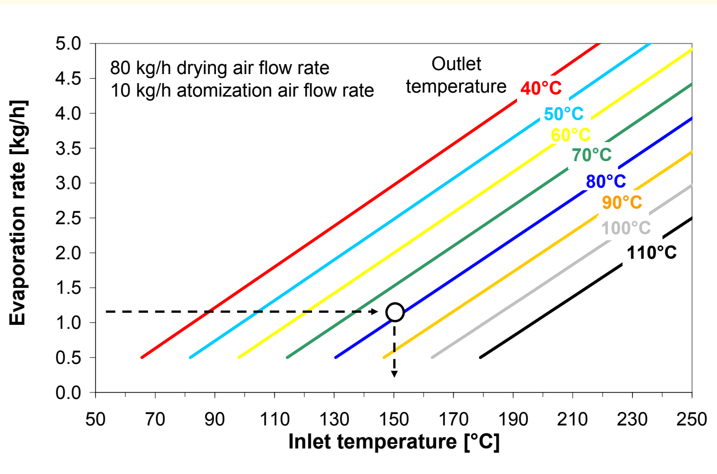

Figure 3: MOBILE MINOR™ inlet drying air temperature as a function of water evaporation rate and outlet drying air temperature.

Now, the outlet drying air vapour concentration can be calculated from a simple mass balance across the spray dryer.

$$\mathbf{Y_{OUT}} = \frac{\mathbf{G_{IN}} \cdot \frac{\mathbf{Y_{IN}}}{1 + \mathbf{Y_{IN}}} + \mathbf{G_{TFN}} \cdot \frac{\mathbf{Y_{TFN}}}{1 + \mathbf{Y_{TFN}}} + \mathbf{EVR}}{\frac{\mathbf{G_{IN}}}{1 + \mathbf{Y_{IN}}} + \frac{\mathbf{G_{TFN}}}{1 + \mathbf{Y_{TFN}}}}$$

(Formula 2)

Y_{OUT} = outlet drying air vapour concentration [kg vapour/kg dry air]
Y_{IN} = inlet drying air vapour concentration [kg vapour/kg dry air]
Y_{TFN} = two-fluid nozzle atomization air vapour concentration [kg vapour/kg dry air]
G_{IN} = drying air flow rate (including water vapour) [kg/h]
G_{TFN} = two-fluid nozzle atomization air flow rate (including water vapour) [kg/h]

If the drying gas is ambient air the water concentration varies significantly from day to day; typically this is in the range of 2 to 20 g water per kg dry air. Humidity data can be supplied from local weather stations. To decrease the relative humidity in the inlet air of the Mini Spray Dryer B-290 the Dehumidifier B-296 can be used. This additional dehumidification step may improve the drying capacity and allows the removal of more water per unit time - especially for hygroscopic materials.
On the other hand, the compressed air normally used for the two-fluid nozzle is very dry and quite consistent - typically in the range of 2 to 3 g water per kg dry air.
If the drying gas is fresh nitrogen the vapour concentration is zero. In a closed loop configuration with a condenser such as the Inert Loop B-295, the vapour concentration can be calculated from the condenser outlet gas temperature at 100% saturation using the Antoine equation for the relevant solvent.

The next part in scaling up is to get the same particle size. In general, the droplet size distribution emerging from a two-fluid nozzle depends upon liquid viscosity, surface tension and the mass rate of atomization air and liquid feed rate.

The droplet size is difficult to estimate or measure. A simple approach is to operate the MOBILE MINOR™ nozzle at conditions providing similar droplet sizes as in the Büchi nozzle for water. This way, the droplet size is similar for the first run on feed, however slight adjustments must be expected.

Figure 2 shows the mean droplet size for water obtained with the two-fluid nozzles for the Mini Spray Dryer B-290 and the MOBILE MINOR™ as a function of the atomization air flow rate. The effects of liquid feed rate are minor within the relevant flow range and can be disregarded in this approximate method.

The flow rate of atomization air is the major driver in determining the particle size of the final powder. A higher relative velocity between the air and the liquid increases the shear forces and provides a higher atomizing energy. This reduces the mean droplet size. The curves in Figure 2 can be used to estimate the required atomization air flow rate (or supplied pressure) to achieve a similar droplet size in the MOBILE MINOR™ as was achieved in the Mini Spray Dryer B-290. The droplet (and particle) size is limited in the Mini Spray Dryer B-290 due to the relatively small drying chamber. If a larger particle size is required the MOBILE MINOR™ offers a larger drying chamber with longer drying times and the possibility to dry larger droplets to solid particles. Naturally, this requires a reduction of the atomization air flow rate and typically also need an adjustment of the operating temperatures to maintain the same residual moisture content in the final powder.

Beside the two-fluid nozzle, the MOBILE MINOR™ offers the possibility to change the atomization technique to a rotary atomizer or a fountain mode two-fluid nozzle. The fountain mode configuration provides longer residence times than the co-current two-fluid nozzle in the drying chamber and is therefore more suitable to dry larger droplets of heavy and heat resistant materials.

The next step is to calculate the evaporation rate for the MOBILE MINOR™ required to achieve the same vapour concentration in the outlet air as in the Mini Spray Dryer B-290 (Formula 3). The feed rate is then determined with Formula 4.

$$\mathbf{EVR} = \frac{Y_{OUT} \cdot \left(\frac{G_{IN}}{1+Y_{IN}} + \frac{G_{TFN}}{1+Y_{TFN}} \right) - G_{IN} \cdot \frac{Y_{IN}}{1+Y_{IN}} - G_{TFN} \cdot \frac{Y_{TFN}}{1+Y_{TFN}}}{}$$

(Formula 3)

$$\mathbf{FR} = \mathbf{EVR}\,\frac{1-\mathbf{RW}}{1-\mathbf{RW}-\mathbf{TS}}$$

(Formula 4)

Example with Mini Spray Dryer B-290 data:		
Inlet drying air temperature:		140 °C
Outlet drying air temperature:		75°C
Feed flow rate:		0,60 kg/h
Total solids in the feed:		0,25 kg solids/kg feed
Residual water in powder:		0,05 kg water/ kg wet powder
Atomization air flow rate:		0.5 kg/h
Inlet drying air vapour conc.:		0,010 kg vapour/kg dry air
Atomization air vapour conc.:		0,002 kg vapour/kg dry air
Step 1: Formula 1	→	$EVR_{B\text{-}290}$ = 0.44 kg/h
Step 2: Figure 1	→	$G_{IN\ B\text{-}290}$ = 40 kg/h
Step 3: Formula 2	→	$Y_{OUT\ B\text{-}290}$ = 0.021 kg vapour /kg dry air
Step 4: Figure 2	→	D_{50} = 20 micron, $G_{TFN\ MM}$ = 9 kg/h
Step 5: Formula 3	→	EVR_{MM} = 1.04 kg/h
Step 6: Figure 3	→	$T_{IN\ MM}$ = 150°C

The final results from the example:

	Mini Spray Dryer B-290	**MOBILE MINOR™**
Equivalent droplet size	**20 micron**	
Outlet drying air vapour concentration	**0.021 kg vapour / kg dry air**	
Inlet drying air temperature	140 °C	**150 °C**
Outlet drying air temperature	80 °C	
Feed flow rate	0.60 kg/h	**1.41 kg/h**
Total solids in the feed	0.25 kg solids/kg feed	
Residual moisture in the final powder	0.05 kg water/kg powder	
Atomization air flow rate	0.5kg/h	**9 kg/h**
Inlet drying air vapour concentration	0.010 kg vapour/kg dry air	
TFN atomization air vapour concentration	0.002 kg vapour/kg dry air	

Solvent	Factor
Methanol	0.50
Ethanol	0.38
Isopropyl alcohol	0.30
Acetone	0.22
Toluene	0.18
Ethyl acetate	0.17
Dichloromethane	0.15
Chloroform	0.11

Table 1: Approximate factors to convert obtained solvent evaporation rates to equivalent water evaporation rate.

Finally, the inlet temperature of the drying air is estimated from Figure 3. In most spray drying plants, the drying air flow rate is kept within a limited range to maintain correct loading and velocity in the air disperser, powder transporting ducts, cyclone and filters. Spray dryers are optimised for a specific drying air flow rate. Typically, 80 kg/h are used in the MOBILE MINOR^{TM} as drying air flow rate.

It is recommended to bring the plant to steady temperature conditions by atomizing pure water. During this starting period, the estimated inlet temperature from Figure 3 can be adjusted for a perfect match with the evaporation rate (e.g. correction for errors due to variations of the atomization air flow rate, which are neglected in Figure 3).

When steady conditions are reached the pump is switched to the real liquid feed. An adjustment of the feed rate is probably needed as the liquid now contains solids.

If the solvent of choice is not water, then Figures 1 cannot be used directly. In this case, a conversion factor is required to convert the obtained solvent evaporation rate to the equivalent water evaporation rate in Figure 1 and Figure 3.

Table 1 contains approximate conversion factors for common solvents in spray drying for quick reference. The conversion factors are dependent mainly on the heat of evaporation, but also the solvent heat capacity, feed and drying temperatures and the inlet drying gas vapour content. The conversion factors are most accurate for solvents with a high heat of evaporation (larger conversion factor) and low inlet drying gas vapour content (e.g. single-pass nitrogen). Note that most organic solvents are highly flammable and if sprayed into air they constitute an explosion risk!

Conclusions

Compared to other unit operations, spray drying is relative simple to scale up. However, spray drying still remains a unit operation with some uncertainties and difficulties to scale up. The main reasons are the big influence of material properties and the drying behaviour of the product. With this leaflet, Büchi Labortechnik AG and Niro A/S demonstrate a practical procedure to scale up a spray drying process from the Mini Spray Dryer B-290 to the MOBILE MINOR^{TM}.

Based on decades of experience and thousands of installations worldwide, Büchi Labortechnik AG and Niro A/S offer a consistent spray drying solution for any scale of operation.

Spray drying appears to be the method of choice in preparing powders for new application fields in biopharmaceuticals and controlled drug delivery systems. It is a one step method, which allows fast processing of small batches at reasonable yields and where microparticles with the desired size and shape can be obtained.

The most important technical features of the Mini Spray Dryer B-290 and the MOBILE MINOR^{TM} are listed in Table 2.

Büchi Labortechnik AG

As world leader in laboratory scale spray drying, Büchi Labortechnik AG offers the perfect product solution for first trial processing, feasibility studies, process development and regular product research. The Mini Spray Dryer models are - with over 2'700 sold units since 1979 - the most often used spray drying laboratory instruments in the world for quick and gentle drying of aqueous or organic products to powder.
Its impressive features include efficient performance with short set-up times, an effective integrated nozzle cleaning mechanism and a high degree of flexibility with the glassware. Büchi offers product solutions and transfers application know-how for its customers' benefit.
Visit Büchi's online new Spray Dryer Application Database with over 350 products together with recommended process conditions (www.buchi.com). The database gives an excellent overview of research regarding the preparation of microparticles using different materials and substances. It is structured by product category, type of application and industrial sector. The application range provides search functions from spray drying, micro encapsulation, spray agglomeration and spray chilling.

Mini Spray Dryer B-290

The Mini Spray Dryer B-290 from Büchi Labortechnik AG is a laboratory scale instrument to perform spray drying processes down to 30 mL batch volume and up to 1 litre of water or organic solvent per hour. Thanks to the glassware, the complete drying process from the two-fluid nozzle down to the powder collection vessel is visible. The Mini Spray Dryer B-290 typically has the capability of generating particles from 2 to 25 microns. Fine particles are produced because of the short residence time in such a compact spray dryer. The residence time of the drying air within the spray chamber is about 1.5 seconds.
The powder collection is provided by a glass-made cyclone separator, which is internally coated with a thin antistatic film to educe powder adhesion to the glass wall. The separation works by centrifugal forces by virtue of inertia of the solid particles. To improve powder recovery a smaller cyclone with a narrower cyclone inlet is used. This smaller High Performance cyclone provides better efficiency in collecting fine particles.
By means of the Inert Loop B-295, the application range is extended to spray dry organic solvents safely. The potential use of the Mini Spray Dryer B-290 and its forerunner models B-191 and B-190, is steadily reported in scientific literature by a huge number of fundamental investigations. The bench-top Mini Spray Dryer B-290 is the ideal instrument to perform feasibility studies with a certain product to spray dry!

GEA Niro A/S

Niro's expertise in converting liquids (solutions or suspensions) into powders with customer defined characteristics is recognised worldwide. Whether the plant produces 16 tonnes of milk powder per hour or just 100 g of a precious catalyst, the initial steps in developing the industrial product are often made in Niro's test station in Copenhagen (Denmark). Experience has been gained over more than 70 years. A reference list of some 10'000 industrial plants across the globe and 30'000 test reports from Niro's test centres were established.
Niro offers a large range of different scales of spray dryers (www.niro.com). Beside the MOBILE MINOR™ as a work horse and widely used instrument, the SDMICRO™ covers the need to spray dry small quantities under GMP regulations and for clinical trials. A few examples from the pharmaceutical industry are fine particles for inhalation, solid dispersion to increase bioavailability, and encapsulation for controlled drug release or taste masking.

MOBILE MINOR™

The MOBILE MINOR™ is a flexible, easy-to-handle laboratory spray dryer. Since the introduction in 1948, more than 2'100 units have been sold worldwide. Today's versions meet the increasing demand for safety, sanitary and flexibility. Due to the different nozzle techniques, the larger chamber diameter and the longer residence time, the MOBILE MINOR™ is a very versatile instrument to further investigate the spray drying process or to make production batches. Achievable particle size depends on nozzle selection and the total solids content of the liquid feed. The MOBILE MINOR™ may produce particles in the 2 to 80 micron range and the drying air residence time is between 15 and 25 seconds.
The MOBILE MINOR™ model with a pneumatically operated chamber roof can be equipped with three alternative atomizing systems, ensuring an option is available that is best suited to the task. With the two-fluid nozzle in the centre of the chamber roof, atomization is created by compressed air. The second option is to place a rotary atomizer in the centre of the chamber roof where an air turbine supplies energy to the atomizer wheel by means of compressed air. Finally, there is the fountain mode in which the two-fluid nozzle sprays upwards and atomization is created by compressed air. For operation with feeds based on flammable solvents or powders subject to explosion risk, special designs are available.

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Technical features	Mini Spray Dryer B-290	MOBILE MINOR™
Water evaporation capacity	1.0 kg/h, higher for organic solvents	0.5-6.0 kg/h, higher for organic solvents
Sample volume	30 mL - 1 L	100 mL - 10 L
Drying air flow rate	up to 40 kg/h	80 kg/h at 200°C
Atomization flow rate for two-fluid nozzle	0.1 - 1.0 kg/h at 5 – 8 bar	4 - 25 kg/h at 0.5 - 6.0 bar
Heating power	2.3 kW	9 kW
Maximum inlet temperature	220 °C	350 °C
Spray chamber size (diameter/cylindrical height)	165 mm/600 mm	800 mm/620 mm (optional 860 mm)
Space requirements (LxWxH)	600 x 500 x 1100 mm	2500 x 2000 x 2300 mm
Weight	48 kg	250 kg
Nozzle types	Two-fluid nozzle with nozzle cleaning function	Rotary atomizer, two-fluid nozzle in co-current or fountain mode
Operating conditions	Open, optional closed cycle with Inert-Loop B-295	Open, optional closed cycle Inert Loop
Achieved particle size	2 - 25 µm	2 - 80 µm

Table 2: Technical features of the Mini Spray Dryer B-290 and the MOBILE MINOR™.

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