



Pilot and laboratory drier

for use in potentially explosive atmospheres

by Dietmar Segmehl



Figure 1: Explosion protected series ML pilot drier with explosion protected control system in stainless steel cabinet

The name BOLZ-SUMMIX® from Wangen in Allgaeu Germany is synonymous with the supply of drying and mixing systems for the pharmaceutical, fine chemicals, food and metal industries. BOLZ-SUMMIX®, a member of the MPE Group, has been producing cone-screw dryers and mixers for many years. Cone-screw dryers are vacuum contact dryers that, with their fully enclosed design and indirect heating, are well suited to processing hazardous or toxic powders wet with solvents or water. In these agitated dryers residual moisture levels of a few ppm can be easily achieved.

Along with units for production, special laboratory and pilot units are needed for the development of new products, for scale-up trials for production line design, and for ongoing quality monitoring in production; these special pieces of equipment are designed for the related small amounts of material used and other special requirements.

So-called pilot equipment, developed during many years of collaboration with companies in the pharmaceutical and chemical industry, are already used in many areas. These units need to be continuously adapted to meet increased requirements and be further developed. In particular, the desire to also use these machines in hazardous areas zones 1 and 2 or zones 21 and 22 has been addressed.

During the development of these devices, comprehensive know-how on large cone screw driers, and their design as used in production, were transferred to this laboratory and pilot drier.

Its design is as compact as possible to ensure the necessary mobility so that transport from one location to another through doors, along corridors, and in lifts is possible without problems (Figure 1).

An increasing trend that can be seen is toward so-called »kilo labs«. The requirements of these small, flexible and mobile devices are the same as those on the larger driers. Careful planning for usage in the laboratory or in small batch production is imperative. These handy devices can be used for any method required: drying under vacuum conditions, mixing, moistening with special injection systems; and yes, even coating, granulating and many other work steps are possible. Anything with high accuracy, that does not harm the product, and with a low energy requirement.

The results of the tests are used for »scale-up« production. Existing or new methods are further developed, verified or optimised. A requirement on the control system is that it be able to read in and read out data in the form of trend graphs in hazardous areas.

Of course the equipment must meet the latest regulations and standards, as well as the ATEX directive 94/9/EC. The pilot units can be supplied in accordance with PED, ASME, ÖNORM, Stoomwezen or other regulations.

Explosion protection

In collaboration with the company R.STAHL, a bespoke explosion protected control system was developed for the pilot drier that has demonstrated its ease of use on numerous occasions during trials, and that can be used in hazardous areas zones 1 and 2 or in atmospheres containing dust in zones 21 and 22.

The Programmable Logic Controller (PLC) and other power equipment that have a source of ignition in operation are installed in a flameproof Cubex enclosure of type 8264. The connections are made via an enclosure in type of protection increased safety »e«. The parameters are entered and the results displayed via the explosion protected HMI Terminal ET 416 manufactured by R.STAHL HMI (Figure 2). The terminal, the Cubex enclosure, a flameproof emergency



Figure 2: Explosion protected operator terminal ET 416

stop button, and a display for indicating operating states are all installed in a stainless steel enclosure on which no special protection requirements are placed, as all the devices installed are already explosion-protected.

The HMI Terminal, based on PC functions, is of modular design and the individual function modules are optimized in relation to type of protection applied: flameproof, intrinsically safe, in increased safety or encapsulated. Volume and weight are therefore significantly reduced compared to other solutions. The connections are designed in type of protection increased safety »e« or intrinsic safety »i«.

Data is entered directly on the 10.4" monitor using a touch screen. The »Windows XP embedded« operating system and the application »Winn CC flexible« ensure reliable operation of the system. A high level of safety is achieved by the use of a flash disc with no fan or other rotating parts. Data backup or recovery and output of recorded trend curve files is possible even in a hazardous area using an intrinsically safe USB port with an intrinsically safe USB stick. The advantages of the terminal ET 416 in the varying locations where the drier is used are the large viewing angle, the high luminance and the very bright display with good legibility even in direct sunlight.

The other electrical components such as the motors for driving the orbit arm mixer screw system and for the vacuum pump, as well as the sensors, are purchased from specialized manufacturers, explosion protected in corresponding types of protection such as »flameproof enclosures«, »increased safety« and »intrinsic safety«, and approved as per the conformity assessment procedure according to the ATEX directive 94/9/EC. Mechanical equipment such as the vacuum pump and the stepless gearbox are specified and purchased with EC manufacturers' declarations with confirmation of their suitability for usage in potentially explosive atmospheres – the ATEX declaration of conformity. →

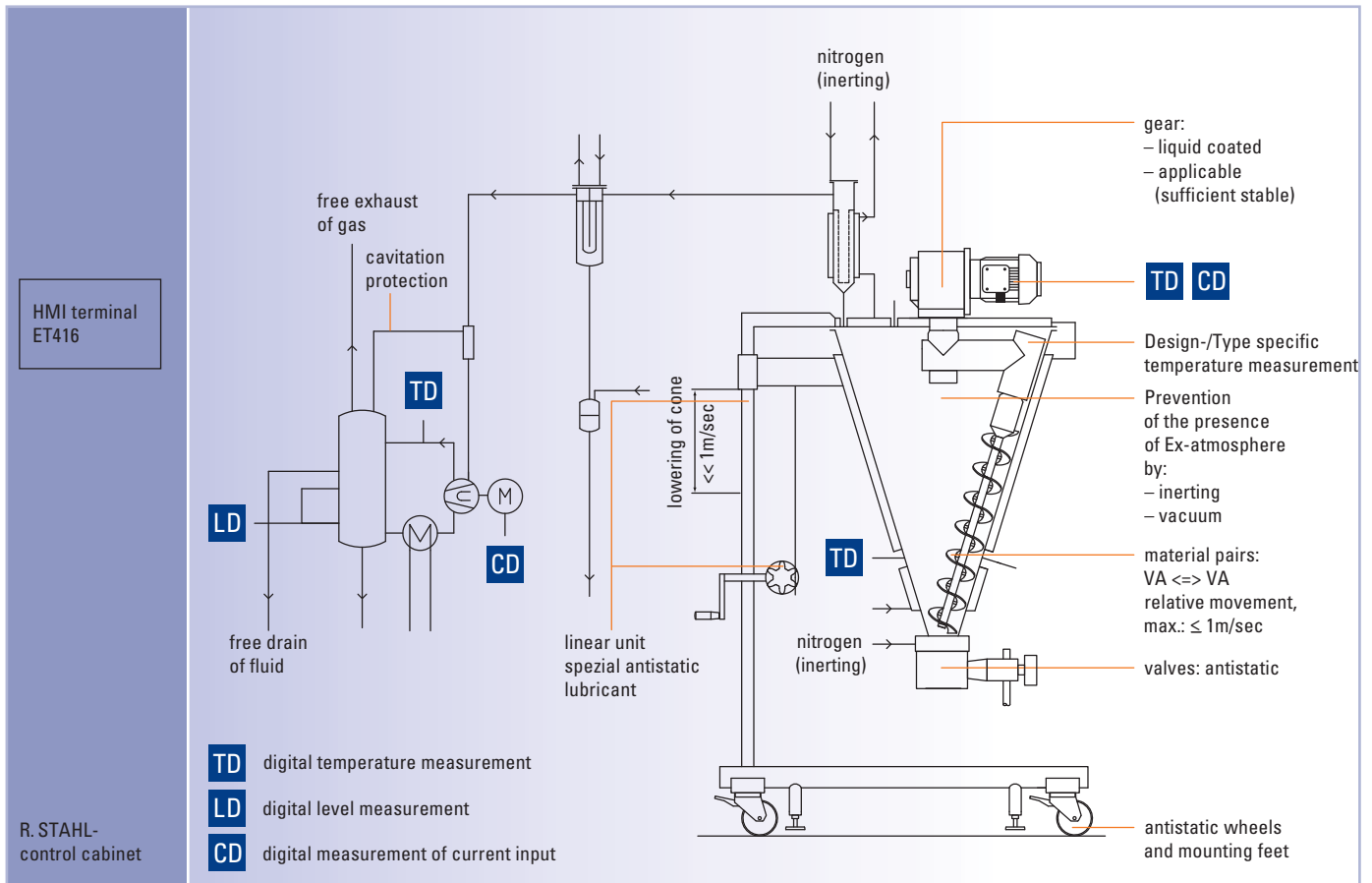


Figure 3: Schematic diagram of the pilot drier ML 05

For components manufactured in-house (the mixing elements and transfer gear) an ignition hazard assessment in accordance with EN 13463-1 must be performed. For this purpose, among other aspects, the calculated maximum surface temperatures were checked and verified by a series of measurements in operation under continuous full load, which then permit classification in temperature class T4. The material pairs have been selected such that mechanical sparks capable of causing ignition are not to be expected. The plastic parts, in particular, parts moved mechanically, are anti-static so that no hazardous electrostatic charging can occur. The inside of the drying vessel, the space containing the product, is inertized during operation by an inert gas (Figure 3). Safe operation is monitored by the control system which also ensures incorrect operation is prevented.

The main operating conditions are stated on the type plate; other operating instructions necessary for explosion protection are described in the section covering explosion protection in the operating instructions; the instructions only authorize skilled persons to operate the explosion-protected drier.

Fully equipped

The equipment in the pilot plants includes:

- › Continuously adjustable drive for the orbit arm mixer screw agitator,
- › Dust filter with filter cleaning feature,
- › Closed heating system,
- › Heated mixing screw,
- › Vacuum system with combined solvent recovery,
- › Injection devices,
- › Cleaning in process (CIP-system),
- › Sampler, luminaires,
- › Measuring equipment, switches and operator controls, safety devices,
- › Ex control system for semi-automatic operation with measured value/trend curve storage for process analysis.



Figure 4: **BS-miniDRY®** for very small quantities of product

The pilot driers/mixers are not tied to a fixed location; they are intended for mobile use and for this purpose are ready to plug in. The patented conical vessel can be lowered and makes visual inspection easy; thorough cleaning is also possible due to an ergonomic working position. With a total height of 1.850 mm and a width of 1.400 mm in the transport position, a 30 litre pilot drier can be moved from laboratory to laboratory without problems.

With a process volume of 10 to 175 litres, the pilot machines are ideally suited to innovation tests, even with long series of trials, for quality assurance, for the production of smaller quantities in pharmaceutical companies and pharmacies, as well as for numerous other tasks in the pharmaceutical and chemical industry.

The closed drying system is the design answer to increasingly strict requirements on the production departments and the protection of the environment. The pilot machines are also built in accordance with this advanced principle. They are designed as pressure vessels and do not allow any dangerous polluting substances to escape. The heating system supplies the process heat, the vacuum system draws off the vapour, which is then recovered in a condenser. This means that the process takes place in a completely closed circuit. The device is therefore the first and most effective protective measure for the environment and for the operator, as the protection is inherent in the design.

Small – modular – mobile – flexible

During research and development for product development there are generally only a few grams of a new substance available. Even the kilo labs units available on the market today are too big for these small quantities, meaning that reliable trials or simulations cannot be performed. To ensure, however, representative results, for example for drying processes, laboratory devices are necessary that have a useful volume adjusted to small quantities, that are very small and can be used as flexibly as possible for the processes to be simulated. To address these requirements, a new laboratory drier compliant with GMP and suitable for the smallest product quantities with a flexible useful volume of 150 to 1.500 millilitres was developed in cooperation with a leading pharmaceutical company in Switzerland (Figure 4). The experience of the pharmaceutical company in the laboratory sector combined with the know-how available at BOLZ-SUMMIX® on the design and manufacture of contact driers made it possible to develop a suitable laboratory unit.

This laboratory vacuum conical mixer drier, which can be used in almost any standard laboratory fume cupboard due the variable overall height and variable working height, ensures work in the laboratory is ergonomic. As a result numerous processes, e.g. drying, mixing, homogenisation, de-gassing, moistening etc. can be simulated.

Components such as vacuum unit, solvent recovery, and a heating/cooling system, complete this laboratory drier to form an autonomous laboratory unit.

A control system adapted to laboratory requirements ensures that all data can be acquired, evaluated and processed, and are available for scale-up calculations.

In addition to the equipment found on the pilot unit, special PAT laboratory measurement equipment such as NIR-probe, mass spectrometer, moisture measuring device, and a camera for process monitoring with PC are integrated.

Bespoke control system programs can simulate the process in a production machine and therefore provide important findings for the production process even during development. The control and measuring equipment provided make it possible to program a defined drying process based on pressure, temperature and moisture curves.

If the laboratory drier **BS-miniDRY®** is to be used in a potentially explosive atmosphere, e.g. for monitoring quality or very small quantity production, the explosion protected variant is used. The protection measures are similar to those on larger pilot units.

The new laboratory drier **BS-miniDRY®** is the ideal and logical development of the existing very successful ML series pilot drier, and is very well suited to product development, very small quantity production and scale-up in the laboratory or in hazardous areas in production.