In- and on-line particle size analysis of dry powders in process environment based on laser diffraction including representative sampling

TWISTER & MYTOS

- ★ Realisation
- ★ Instrumentation
- ★ Adaptation
- ★ Conclusion

HST: Andromeda Galaxy

TWISTER & MYTOS – The Concept

Requirements:

★ An efficient in-line PSA consists of

- ✔ Representative sampling
- ✔ Complete dispersion
- ✔ Precise laser diffraction sensor

★ Process stages are usually connected by pipes

- ↳ in-line PSA should be integrated into the pipe
- ↳ Only the information about the PSD should leave the process line, the product remains inside
- ↳ in-line PSA has to perform under process conditions

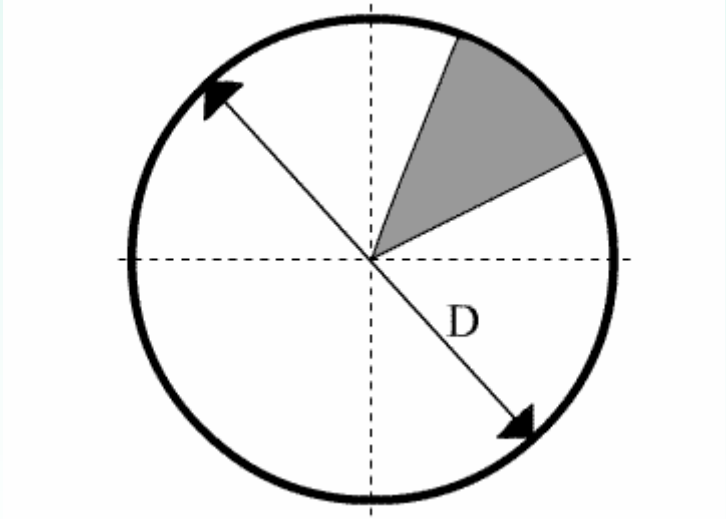
Sampling

- ★ Representative sampling in the process pipe
  - Consideration of the entire pipe cross section
  - All areas in the pipe must be equally weighted
  - Consideration of being iso-kinetic
- ★ Sampling under process conditions
  - Sample mass flow independent of process flow
  - Continuous sampling
  - Interruption of sample mass flow for background measurement

Sampling

★ Continuously working principles

rotating sector field



Realisation: *ROPRON*

✔ Up to *10 t/h*

✔ *2 stages*

✔ *Fall shaft operation*

✔ *Static medium*

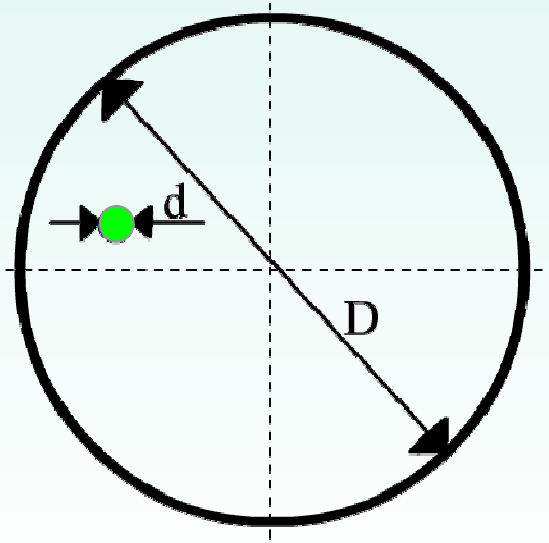
✘ $\dot{m} \approx D^2$ *depends on process !*

“good sampling…”

Sampling

★ Continuously working principles

moving sampling pipe



$$\dot{m} = \frac{\frac{d^2}{4}\pi}{\frac{D^2}{4}\pi}\dot{M} = \frac{d^2}{D^2}\Phi\frac{D^2}{4}\pi = \frac{\pi}{4}\Phi d^2$$

“...better sampling…”

- ✔ *$\dot{m}$ simply adaptable* (d^2 dependence)
- ✔ If flux Φ is constant *$\dot{m}$ is independent on $\dot{M}$*
- ✔ Simple *scale-up*
- ✔ *No need* for primary samplers or splitting stages

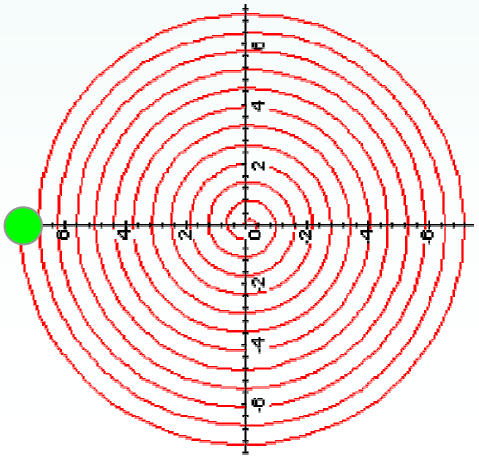
- ✔ $\dot{m} \approx D^2$ i.e. *independent on process*

Sampling

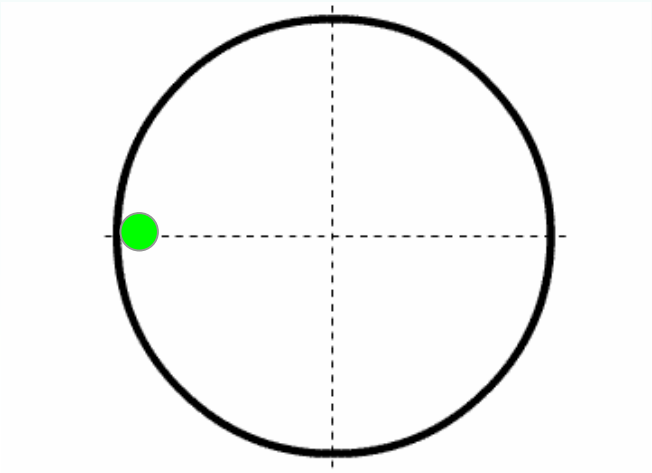
★ Representative:

- ☆ Scan of the *complete cross-section*
- ☆ *Equal areas* must be covered in *equal times*
  - ↳ Velocity *v* of pipe tip depends on (*v (r)*)

(a) linear spiral line



✘ singularity at *r = 0*



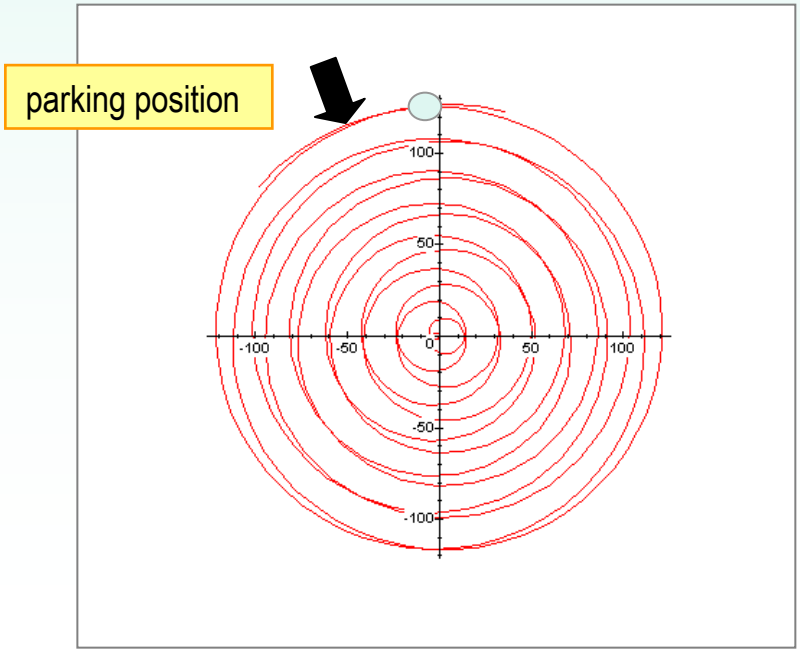
★ Representative sampling:

☆ Scan of the *whole cross-section*

☆ *Equal areas* must be covered in *equal times*

↳ velocity *v* of the pipe tip must depend on r (*v(r)*)

(b) improved spiral line



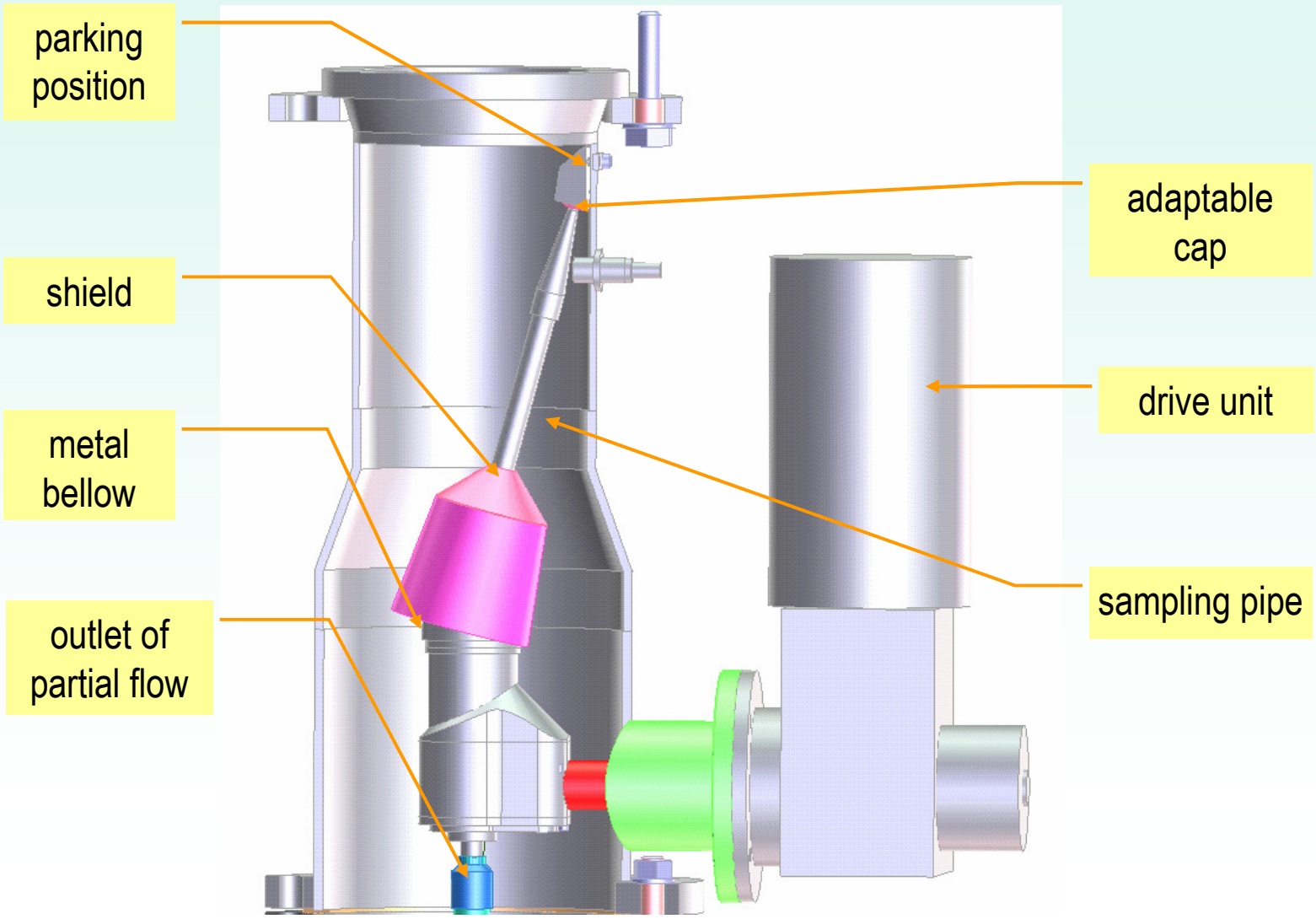
✔ *Smooth v*, avoiding fast accelerations

✔ *Shielded parking position* possible, i.e. $\dot{m}$ = 0 for background measurement

✔ Change of direction i.e. *v = 0 only in the parking position*

Realisation: *TWISTER*

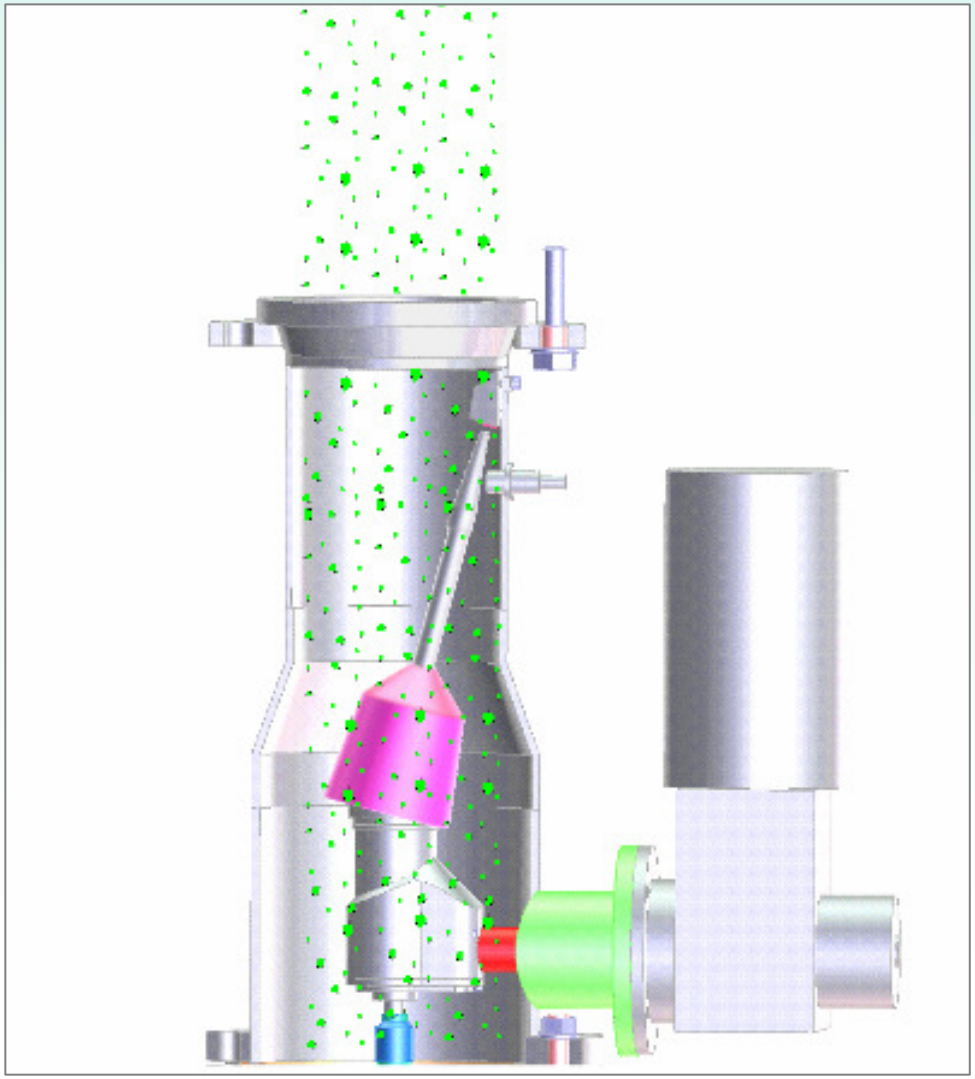
TWISTER



TWISTER

… in operation !

- ✔ Continuous
- ✔ Representative
- ✔ Particle free in parking position



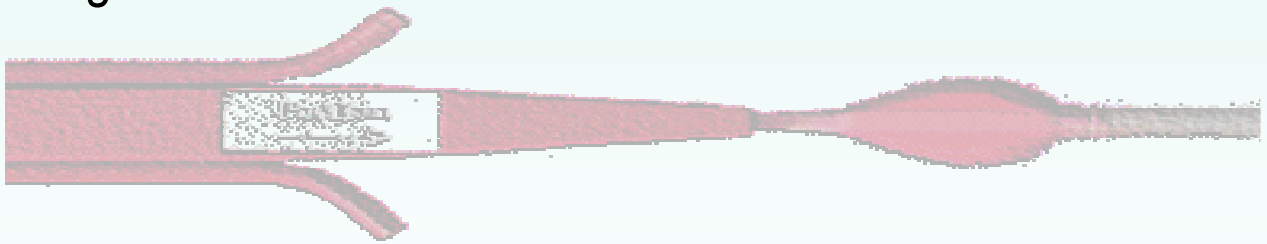
TWISTER

- ★ Quasi iso-kinetic sampling:
  - ☆ Velocity of the particles must remain *unaffected* while entering the sample pipe
  - ☆ *Controllable flow pump* is used for
    - ↳ *Direct feeding* into the subsequent dispersing stage
    - ↳ Pressure control by micro-controller
    - ↳ *Our solution*: Empirical relation $p \rightarrow v_{particle}$ used for
      - ✔ Velocity control
      - ✔ *Robust* design
      - ✔ *Very positive experience*

Dispersion: RODOS Injector

★ *Standard well established dry disperser*

- Particle-to-particle, particle-to-wall collisions, centrifugal forces due to strong velocity gradients
- Dispersion down to 0.1 µm
- Long lifetime



★ *No effect of wear* during standby periods

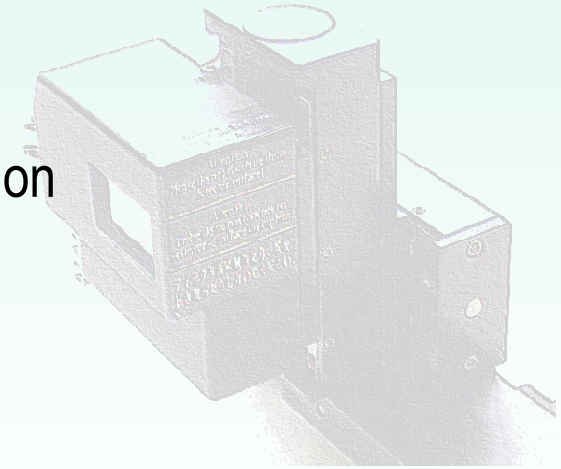
- While sampling pipe is in parking position

★ Cleaning with integrated *flush-back stage*

- Blockages are avoided

Particle Sizing: HELOS Components

- ★ Core components of LB system are identical to *standard off-line LB-PSA* (HELOS)
- ★ Light source, encapsulated, *IP65* or *E(x)*
  - ☆ Rugged *HeNe Laser 5mW*
  - ☆ *Fibre optical cable* und beam expansion
- ★ Detector, encapsulated, *IP65* or *E(x)*
  - ☆ Fourier optics
  - ☆ Multi-element detector with auto-alignment
  - ☆ High speed data acquisition (0.5 ms/PSD)

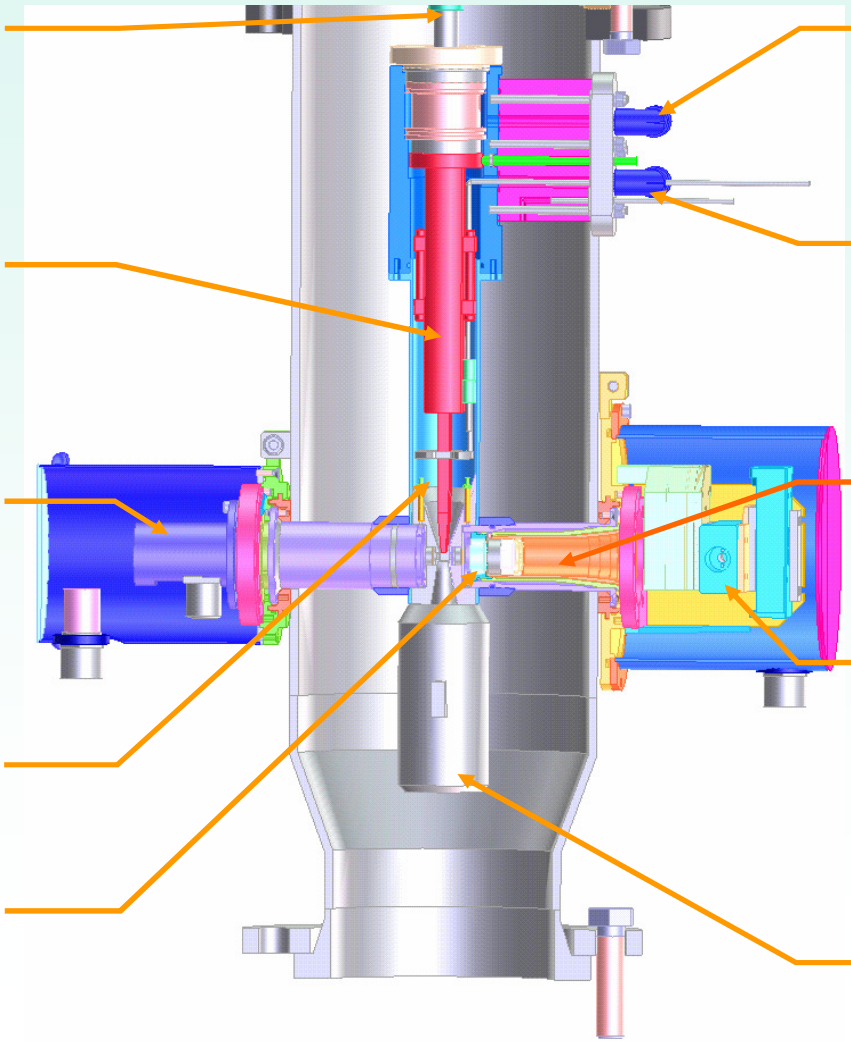


Particle Sizing: MYTOS

- ★ Separate control box, *IP65* or *E(x)*
  - ☆ *Power supply*
  - ☆ *Laser*
  - ☆ Sampler and disperser control unit
  - ☆ Fibre optical link to PC
- ★ PC
  - ☆ PSA information and control system with data base and network capabilities (WINDOX)

MYTOS: ***Rugged in-line combination of approved RODOS dry dispersion and HELOS LD***

MYTOS



inlet for partial flow from sampler

dispersing line

light source and beam expansion

measuring zone

protection window

air supply for dry disperser

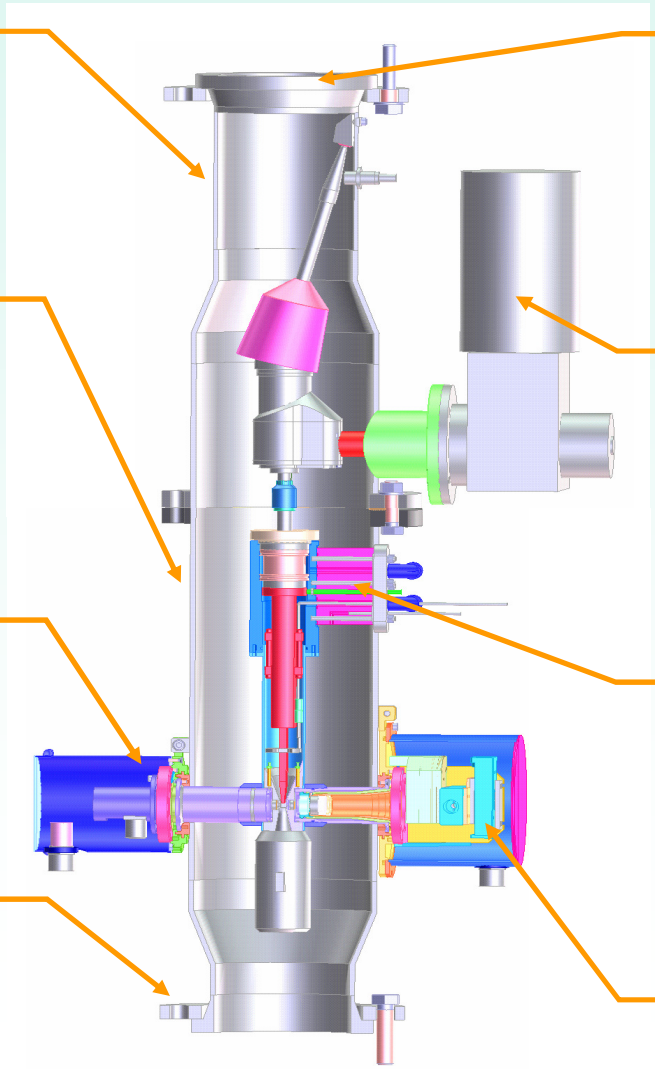
inlet for flow focusing and rinsing

Fourier optics

detector and data acquisition

merging stage

TWISTER & MYTOS



sampling stage

200 mm pipe with built-in dry disperser and PSA

laser and beam expansion unit

150 mm flange to process pipe

150 mm flange to process pipe

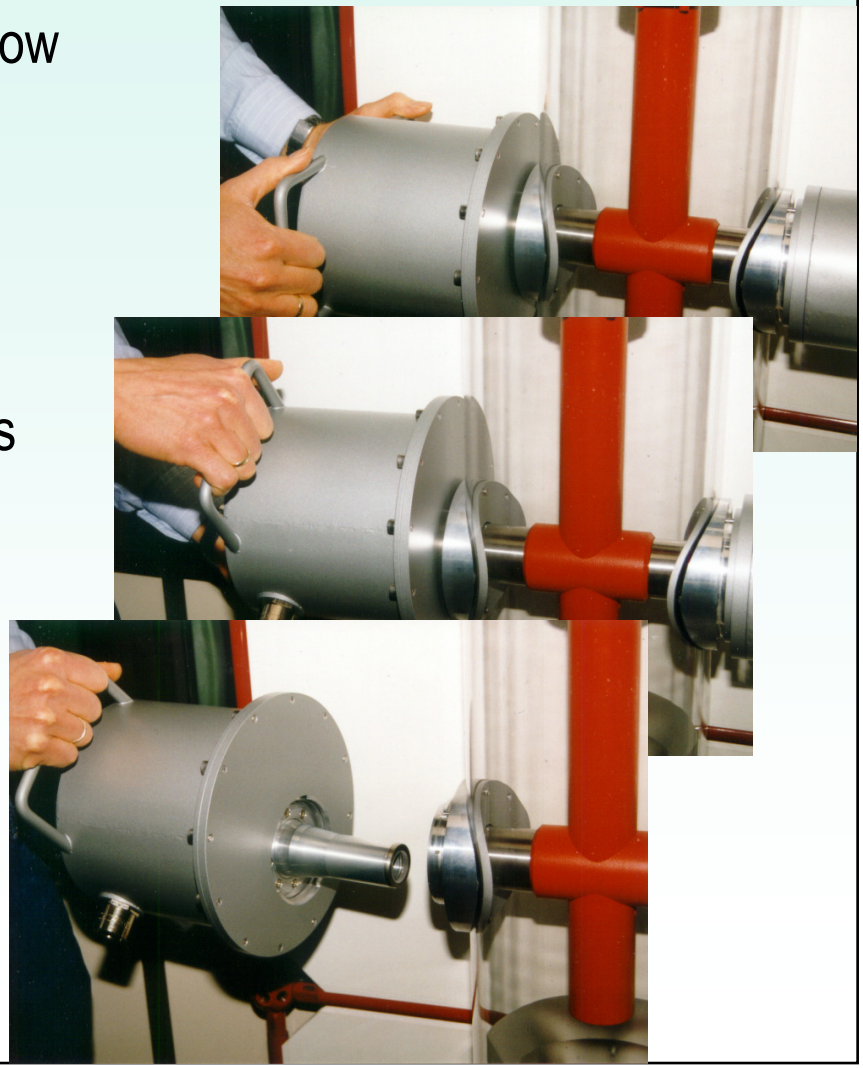
motor and gearbox

pneumatic supply and flush-back unit

detector

Measuring Zone Environment

- ★ *Second injector* with sheath flow used for flow focussing of the aerosol along the centre line
- ★ *Integrated rinsing system* protects built-in dust protection from spray particles
- ★ *Bayonet fastening* of light source and detector unit simplifies serviceability
- ★ *In-situ cleaning* (while process is running)



Realisation

TWISTER & MYTOS

motor and gearbox

pneumatic supply and flush-back unit

detector

150 mm flange to process pipe

150 mm flange to process pipe

sampling stage

200 mm pipe with built-in dry disperser and PSA

mounting plate

laser and beam expansion unit

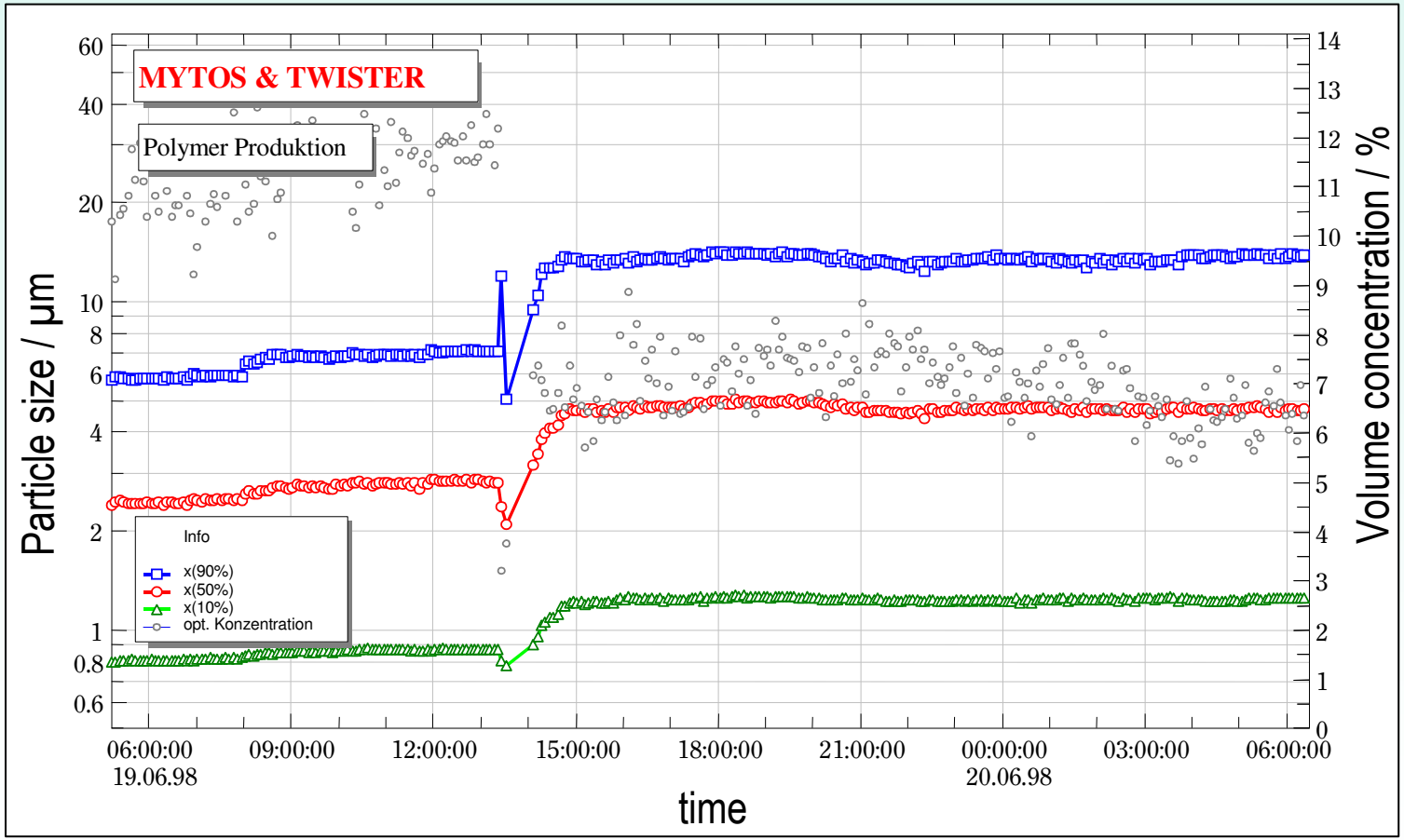
Installation: TWISTER & MYTOS „classic“

- 200 mm process flange
- sampling stage
- control box
- motor and gear box
- pneumatic supply
- 200 mm pipe with built-in dry disperser and PSA
- detector

Installation: TWISTER & MYTOS “up side down”

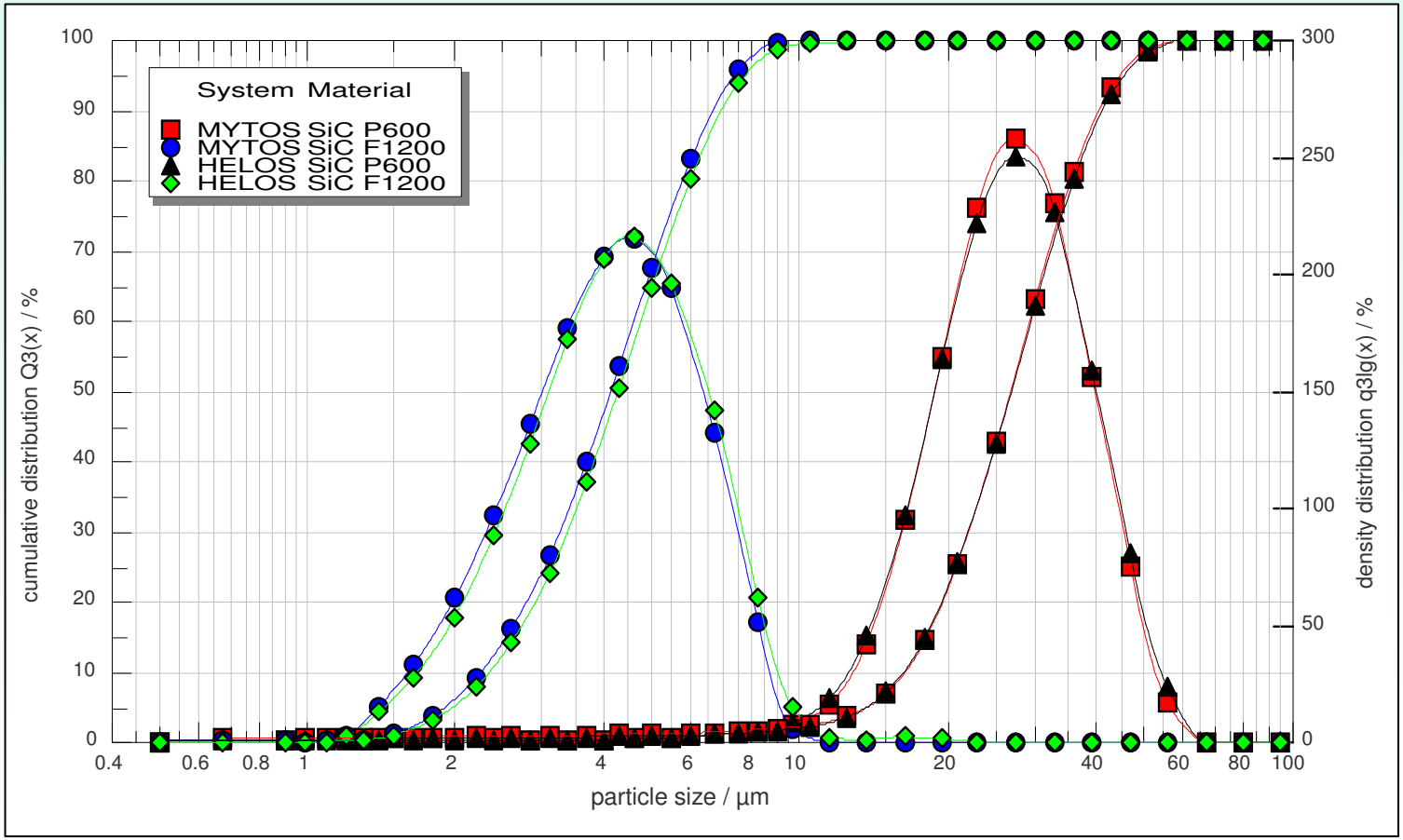
- 100 mm process flange
- 150 mm pipe with built-in dry disperser
- detector
- light source & beam expansion unit
- pneumatic supply
- motor and gearbox
- sampling stage
- 100 mm process flange
- control box

Results of Polymer Production (Excerpt)



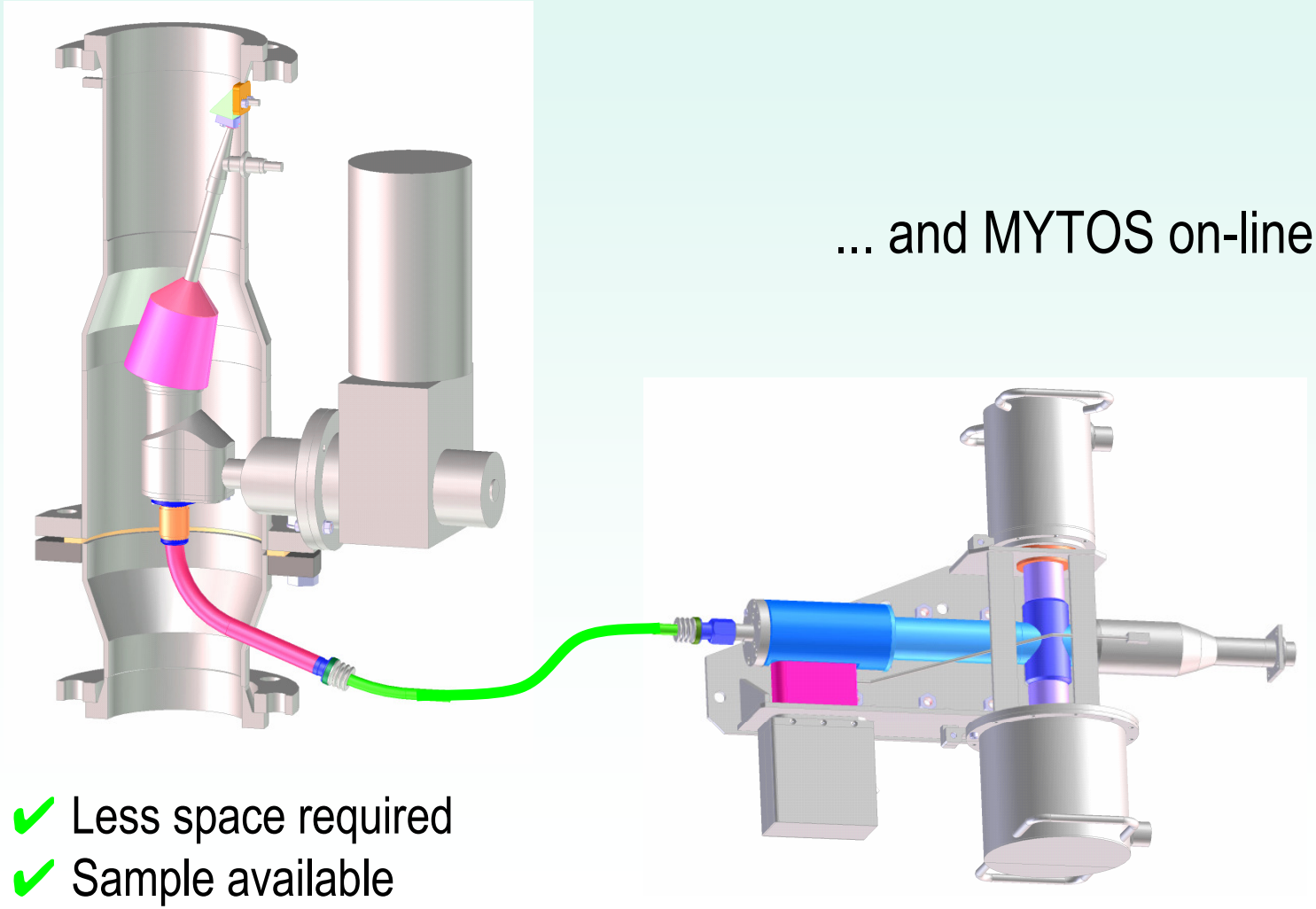
- Long term trend analysis of x_{10}, x_{50}, x_{90} values of several 1000 subsequent measurements with in-line MYTOS & TWISTER

Coherence of MYTOS and HELOS



↳ Comparison of in-line PSA (MYTOS) with off-line PSA (HELOS & RODOS) using two different reference materials

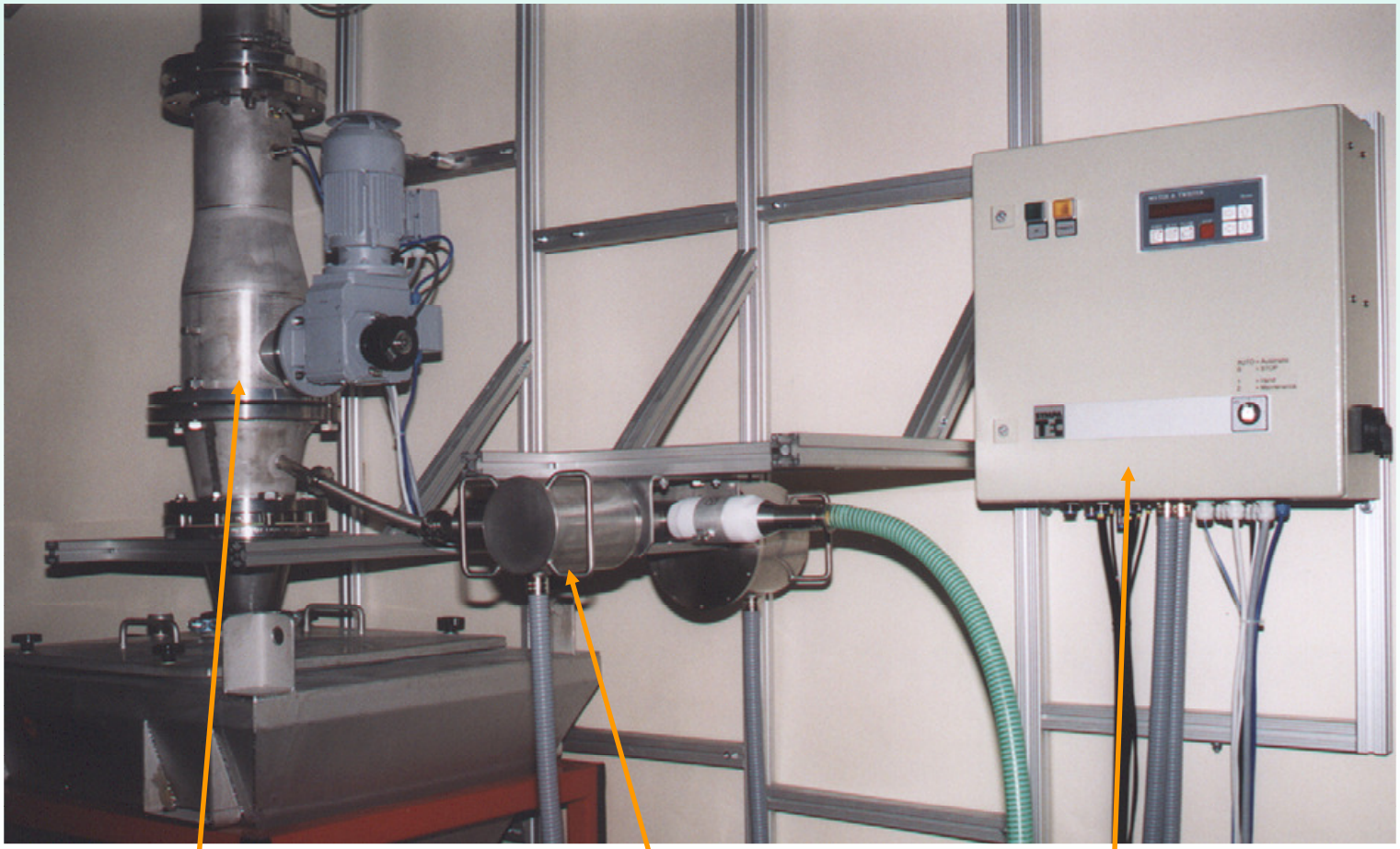
Variation: TWISTER sampling in-line ...



... and MYTOS on-line

- ✔ Less space required
- ✔ Sample available

Installation: TWISTER in-line / MYTOS on-line



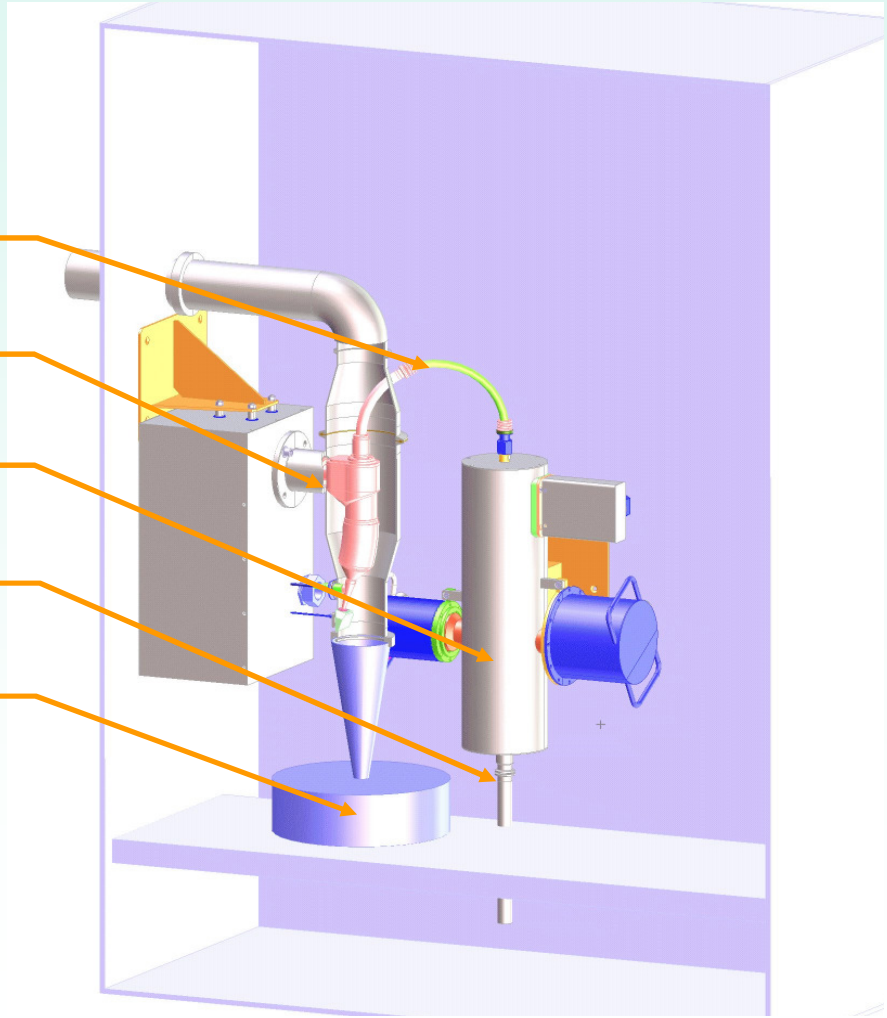
Installation: TWISTER in-line / MYTOS on-line

pipe diameter:
Ø = 400 mm

Installation: TWISTER in-line / MYTOS on-line

pipe diameter:
Ø = 400 mm

Variation: TWISTER & MYTOS as GMP* version



*GMP = Good Manufacturing Practice

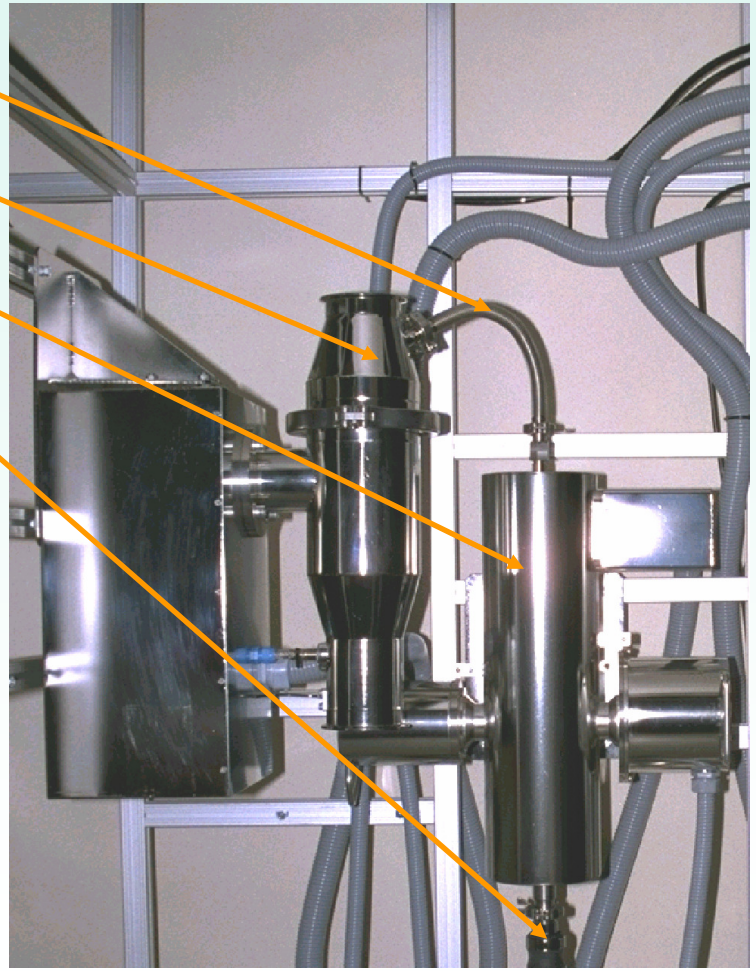
Installation: TWISTER & MYTOS as GMP Version

connection pipe

TWISTER in-line

MYTOS on-line

sample outlet



as GMP-Version with

✔ Electro polished stainless steel surface

TWISTER 50

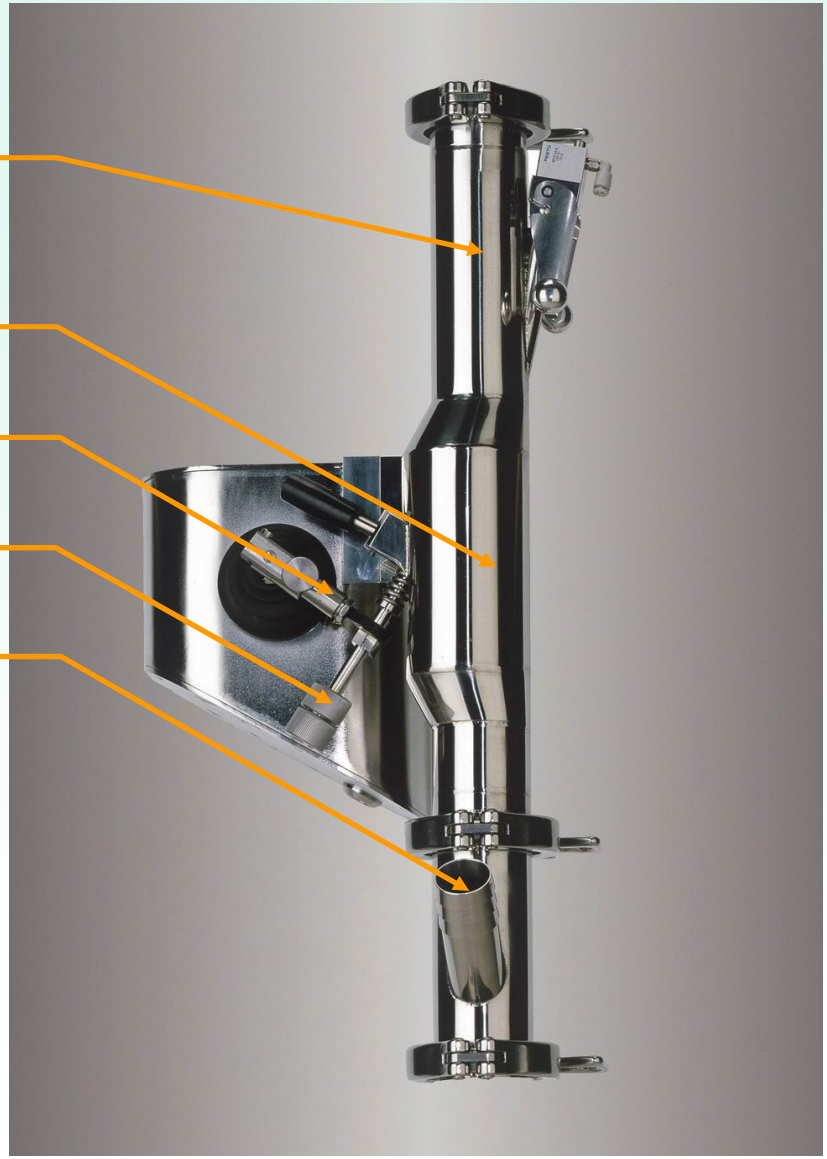
process pipe

TWISTER

TWISTER drive

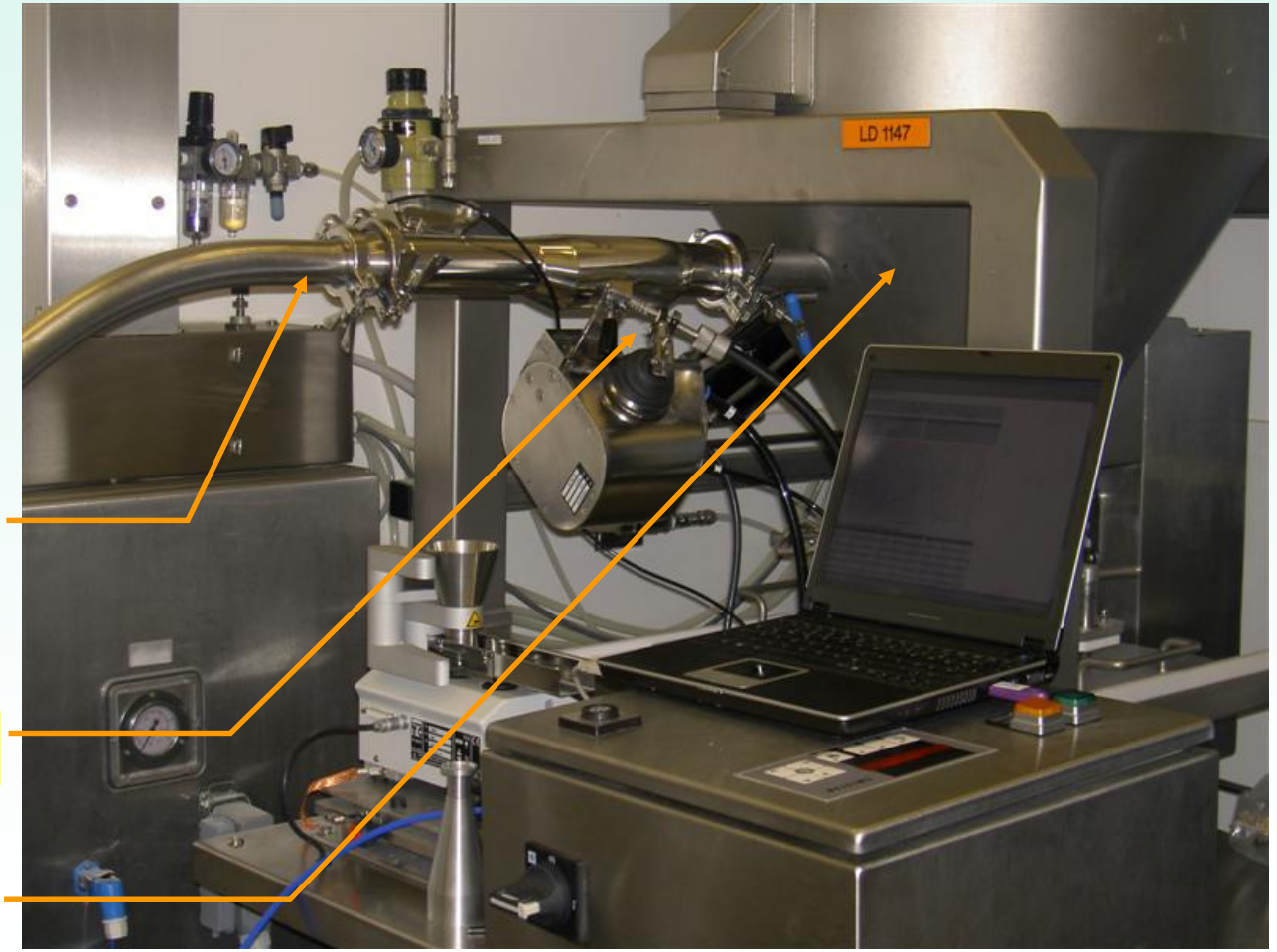
sample outlet

sample feedback



- ✔ GMP compliant
- ✔ For process pipes from 38 – 80 mm

Installation: TWISTER 50

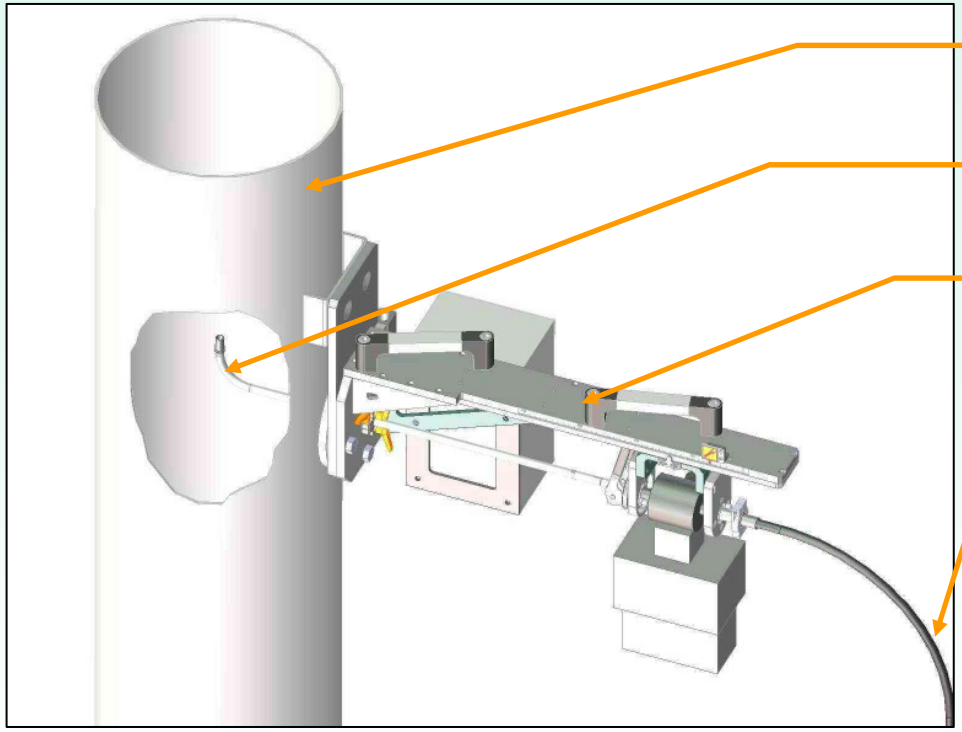


process pipe

TWISTER 50

filter

Variation: MYTOS with static sampler

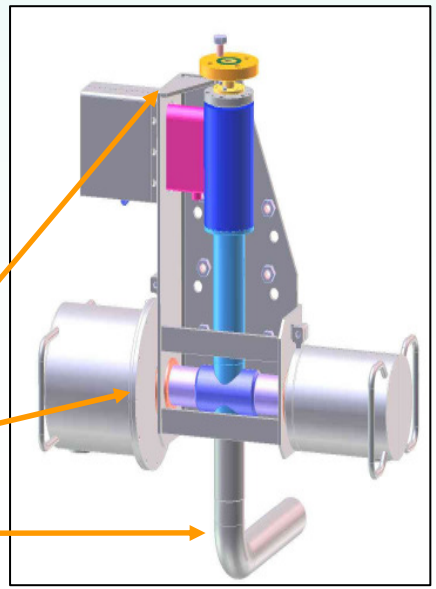


process pipe

goose-neck probe

frame with handles

connector to MYTOS



connector to sampler

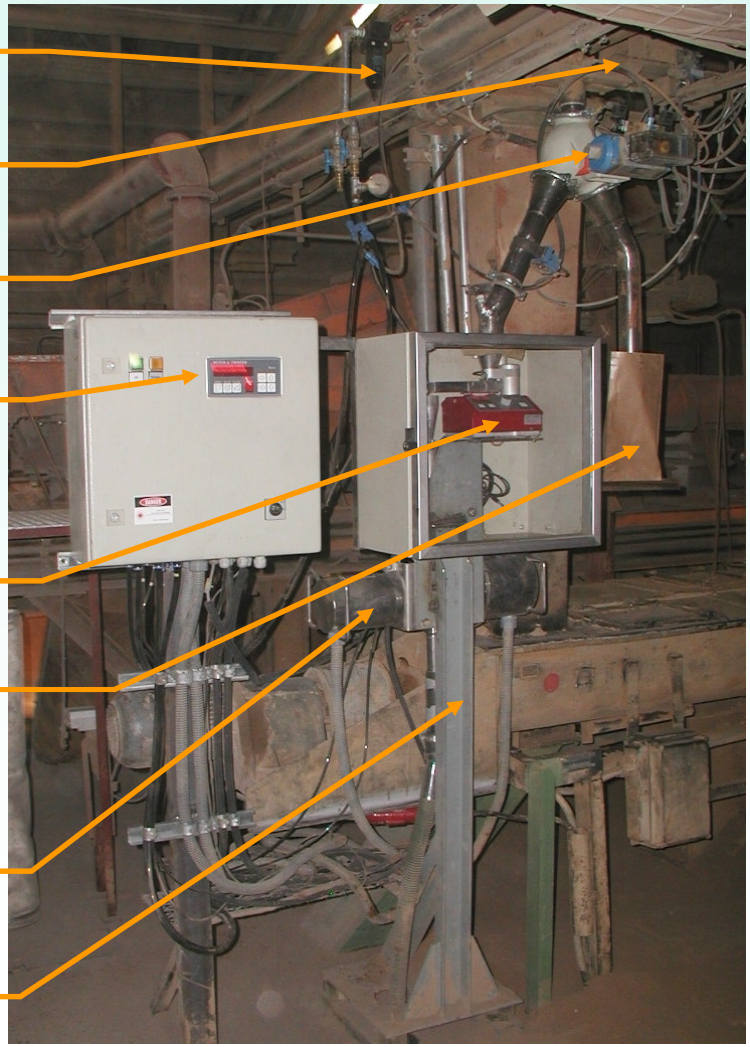
MYTOS on-line

sample feedback

Installation: MYTOS twins on-line

Installation: MYTOS & VIBRI

- screw feeder
- electric flap sampler
- sample switch
- MYTOS & VIBRI control box
- VIBRI
- cumulative samples for off-line analysis
- MYTOS
- mounting rack



Variation: MYTOS & VIBRI - Module

MYTOS and VIBRI as compact solution for automated laboratories

- ✔ Space saving modular design
- ✔ Housing on wheels
- ✔ Integrated cleaning (double hammer add-on and flow controlled particle extraction)
- ✔ Quick connectors for extraction unit and air supply

Installation: MYTOS & VIBRI - Module

sample inlet with fine dust extraction

VIBRI with double hammer add-on

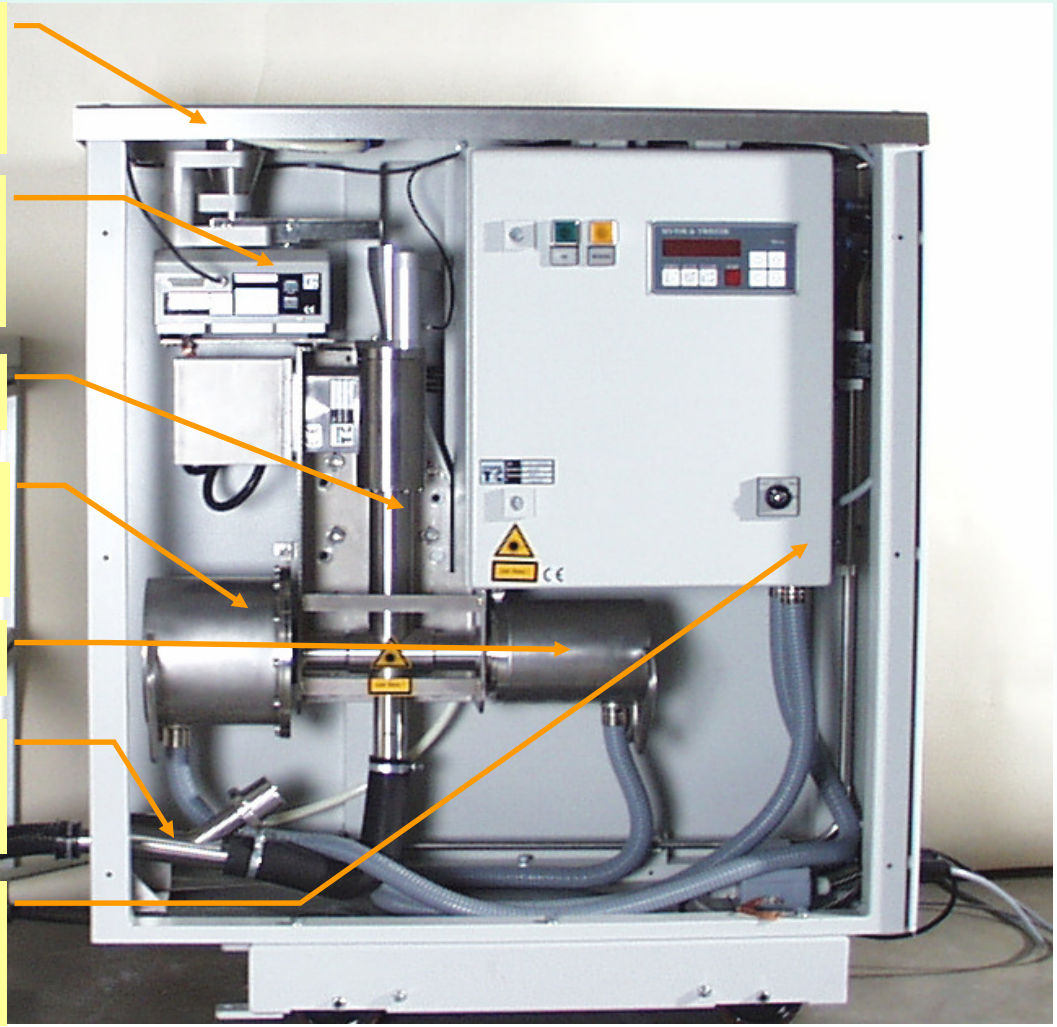
dry disperser

MYTOS detector unit with auto focus

beam expander

connector for extraction unit

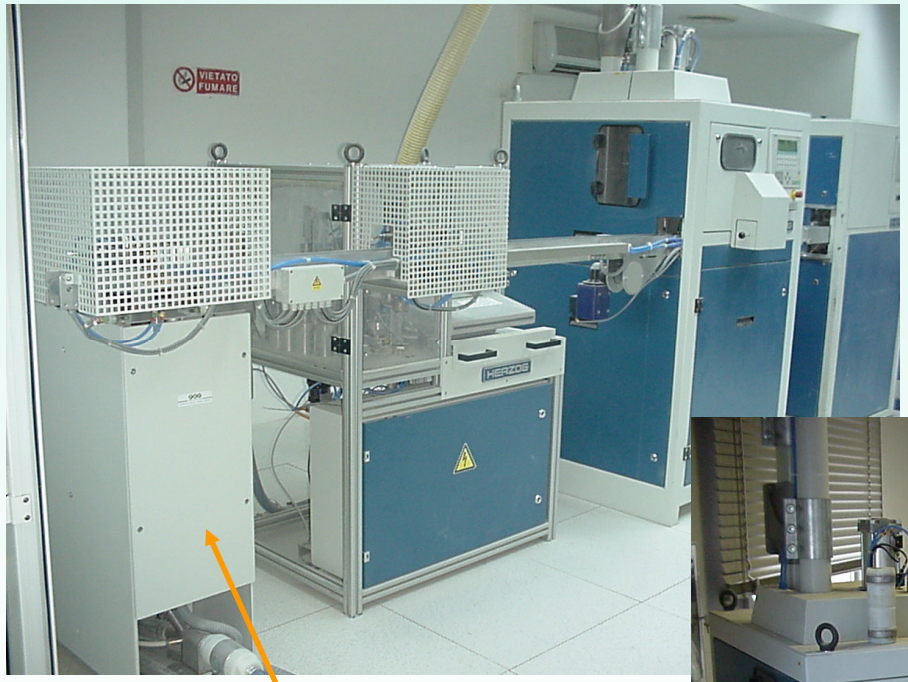
MYTOS & VIBRI controlbox



Installation: MYTOS & VIBRI - Module

MYTOS & VIBRI Module in Polysius Polab© AMT, AOT, etc…

Installation: MYTOS & VIBRI - Module



MYTOS & VIBRI Module
in Herzog automations

MYTOS
PSA

Conclusion

★ MYTOS & TWISTER for in-line laser diffraction combines

- ☆ *Representative sampling*
- ☆ *Dry dispersion*
- ☆ *Laser Diffraction PSA*

Along the *centre line of a production pipe* suitable for:

- ✔ *Dry powders*
- ✔ Production pipes of *any orientation*
- ✔ *Representative* sampling

- ✔ *Small sample quantities* are nearly independent of the primary mass flow
- ✔ The *shielded parking position*
  - ↳ Allows for reference measurements at any time
  - ↳ Reduces wear proportionally to the number of measurements, independent of operation time
- ✔ *Adaptable* to a variety of pipe diameters
- ✔ Simple and *rugged design*
- ✔ All mechanical parts are protected with metal bellows (i.e. *no moving gaskets*) → hazardous areas
- ✔ The results are *coherent* to the well established *off-line* laser diffraction system HELOS & RODOS

Technical Specifications

- ✔ Size range: 0.25 µm to 3500 µm
- ✔ Pressure range: Up to *10 bars*
- ✔ Temperature range: Up to *150°C*
- ✔ Environmental: *IP65*, up to 55°C
  E(x) as option
- ✔ Family for different *diameters* and *size ranges* available:
  - ☆ TWISTER + *in-line* MYTOS: 80 to 200 mm ∅
  - ☆ TWISTER + *on-line* MYTOS: 38 to 660 mm ∅
  - ☆ TWISTER + *on-line* MYTOS (GMP): 38 to 250 mm ∅
- ✔ Combination with other samplers available as well