Particle Counting –
New system to analyze high quantities of samples

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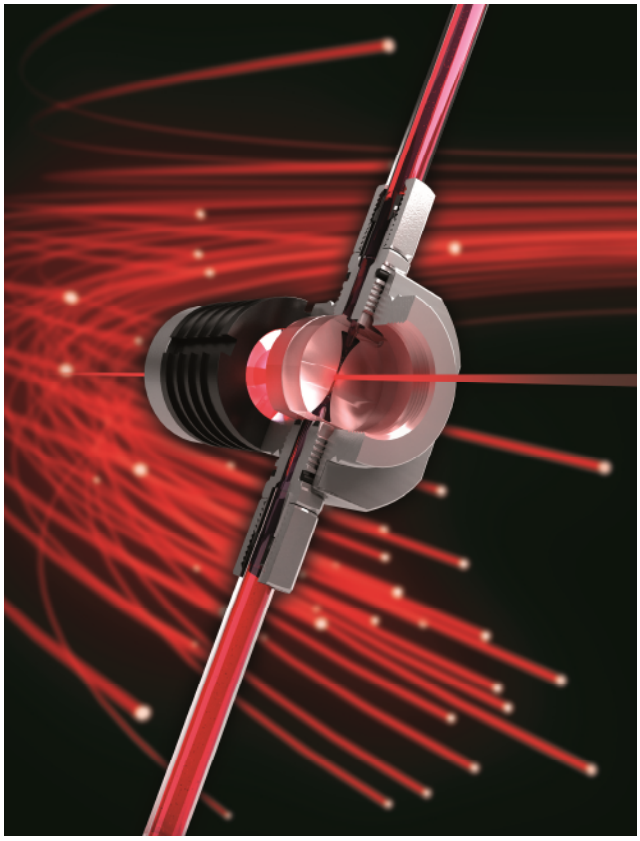
Summary

PAMAS has developed a new system to analyze high quantities of samples. The system is designed to handle unattended sample analysis for several hundred samples per day. The development of this new product was based on the input from users of existing PAMAS autosampler models and from potential customers. Based on these requests for improvement, the new autosampler PAMAS AS3 now includes all requirements that have been brought forward from the users and offers new features regarding reliability, sample preparation, tray handling, flushing and dilution. This presentation introduces into existing PAMAS autosampler models and provides an outlook on the newly developed autosampler PAMAS AS3.

1. Introduction

Particle Counting is a well established technology in many industries including lubrication and tribology. The first particle counting systems were introduced in the 1980s and widely implemented as standard measuring instruments for laboratories during the 1990s. In 1993, PAMAS introduced its first high quantity system – the PAMAS AS1. A second generation autosampler called PAMAS AS2 was developed eight years later, in 2001.

Founded in 1992, PAMAS is the premier independent manufacturer of Automatic Particle Counters and a market leader in many industries including filter testing, fluid power systems, fuel applications, potable water, offshore oil and gas and pharmacy. PAMAS instruments are used as reference systems for many applications. The company is located near Stuttgart in Germany and employs 55 staff members. In 2012, PAMAS had a turnover of 7.5 million Euro.

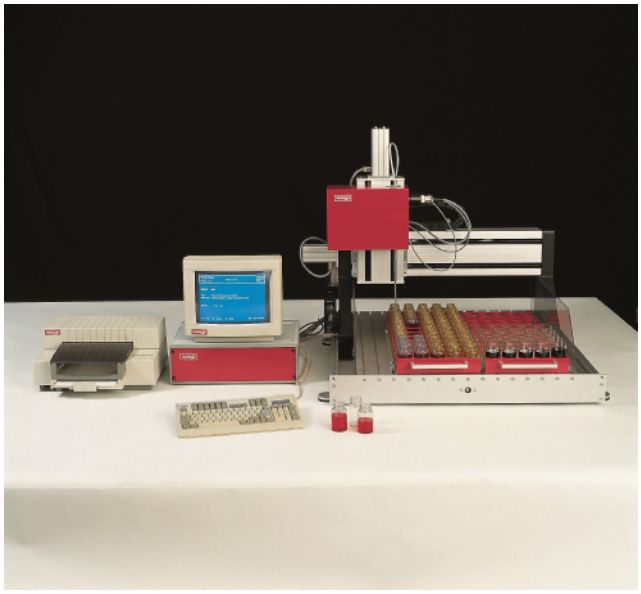


Picture 1: PAMAS particle counters analyze fluid contamination via optical sensor technology. The advanced and highly sophisticated particle sensors (see cross-section of sensor measuring cell in this picture) are developed and manufactured at the PAMAS company plant in Germany. (Picture: PAMAS)

2. Earlier autosampling systems

2.1. PAMAS AS1

The first particle analyzing system for high quantity samples was introduced in 1993. The system was built on a tray for a certain quantity of sample containers. An X-Y coordination system grabbed the sample fluid out of each sample container and led it through the particle counter for analysis. The first autosampler PAMAS AS1 was completely PC controlled and no user interaction was necessary between the samples.



Picture 2: The first autosampling system PAMAS AS1 was introduced in 1993. (Picture: PAMAS)

2.2. PAMAS AS2

In 2001, PAMAS presented a new version of the autosampler. Compared to the earlier PAMAS AS1, the autosampler model PAMAS AS2 offered five new features:

- The particle counter was now flushed between the samples.
- The model PAMAS AS2 was equipped with a conveying belt feeding the sample through the system. With this conveyor, the system perfectly fitted into concatenating system.
- The PAMAS AS2 system was PC controlled and its software could be integrated into existing LIM systems (laboratory information management systems).
- Very flexible parameter settings allowed optimization of precision or speed.
- The system extremely focused on the reliability of measuring results.

Picture 3: The second generation autosampling system PAMAS AS2, which was introduced in 2001, was equipped with a conveyor for concatenating systems. (Picture: PAMAS)

3. Development of a new system to analyze high quantities of samples

3.1. Feedback on user experience with earlier systems

The systems PAMAS AS1 and PAMAS AS2 have been in operation for some ten years now. In collaboration with renowned partners and users of the systems, PAMAS collected feedback and suggestions for improvement. The user experience showed positive and negative aspects of the existing PAMAS autosampling systems.

Among the positive aspects, the system durability, the trouble free operation and the quality of the measuring results were appreciated by the users:

- System durability: The systems are unbreakable. Some units have analysed more than 2.5 millions of samples, which proves the durability of PAMAS systems.
- Trouble free operation: The operators like the units. Trouble free operation is considered as the most important feature for staff operators.
- Quality of measuring results: The users want results on the spot to see correct history. Repeatable and reproducible measuring results are the key requirement in Automatic Particle Counting.

The users also provided valuable feedback on negative aspects of the existing systems and requested to improve the system with regard to sample preparation, sample handling and dilution:

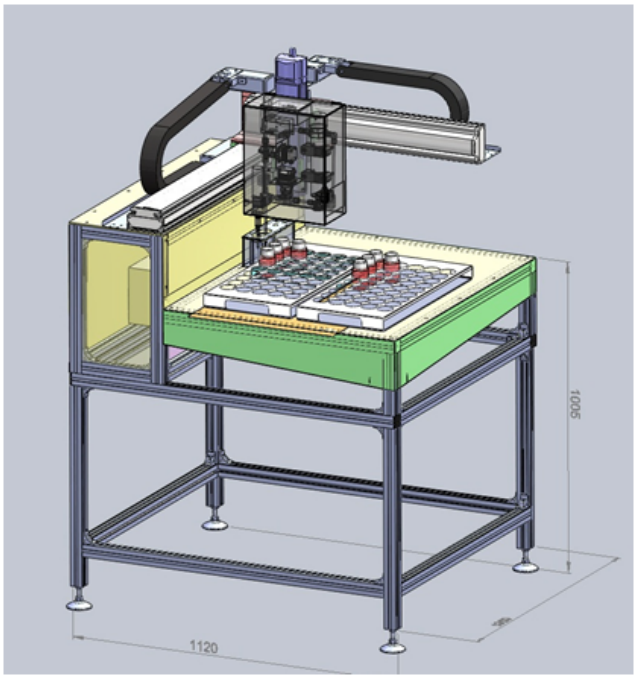
- Sample preparation: When waiting for analysis, the samples sit too long without agitation on the tray or conveyor belt. In order to prevent sedimentation and agglomeration, the samples need to be carefully prepared before measurement.
- Sample handling: The users don't like to put samples from tray to conveyor and again back on tray. The conveyor belt emerged as being impractical for the use in many laboratories.
- Dilution: Some samples are not suitable for automated measurement. Higher viscosity samples and samples with higher concentrations of particles, as well as samples with undissolved additives need to be diluted before measurement. An integrated automated dilution stage would add flexibility and usability.

3.2. Requirements and design considerations for new system

The user feedback clearly proved the high demand for autosampling devices and showed that high quantity analyzing instruments are needed. System reliability is considered to be the key feature for any Automatic Particle Counting system. The use of industrial automation components offers the required reliability. Beside reliability, three additional features were requested by the users: Firstly, a new system requires a sample agitation procedure directly before analysis. Secondly, a new system should use trays rather than a conveyor belt. Finally, a new system needs an automatic dilution system. Against expectation, higher speed was not mentioned as a key requirement for a new autosampling system. It is always welcome, but for the users unattended operation is more important.

4. New Autosampler PAMAS AS3

Based on the user experience, PAMAS is currently developing a new autosampler. The system will be utilizing the most recent and advanced PAMAS technology.



Picture 4: The new autosampling system PAMAS AS3 currently is being developed at the PAMAS company facilities in Rutesheim and will be soon available. (Picture: PAMAS)

Compared to earlier autosampling systems, the new system offers new features with regard to reliability, sample preparation, tray handling, flushing and dilution.

4.1. Reliability

The system is built from high quality automation grade components. All components that may break and wear will be easily available components that are kept on stock. A short delivery time worldwide is essential.

To allow local availability, manufacturers of worldwide available wear parts are preferred. A broken FESTO™ valve could be replaced at a remote place in Australia within 12 hours.

All service personnel that will handle these systems needs training at PAMAS facilities to guarantee competent service worldwide. This will include setup and operation of the units.

4.2. Sample preparation

The new system will include an ultrasonic agitation probe that breaks all agglomerates in the fluid before analysis. The system takes care that the necessary delay between agitation and measurement is kept.

The ultrasonic probe will be sitting on the robot and prepare the n+1 sample while the n sample is analyzed.

The ultrasonic device will be cleaned between the samples to minimise cross contamination.

4.3. Sample handling

The new system will be a XYZ robot that will operate with samples on trays, as these are widely used in high quantity laboratories. RFID (Radio Frequency Identification) or barcode systems can be used to automatically identify the trays.

The system will be linked with an existing LIM system. If the existing LIM system can forward sample IDs based on tray number and x/y position within the tray, no individual sample identification procedure is necessary. If the samples carry RFID or barcode labels, samples can be identified with an attached reader.

Sample identification allows handling individual samples independently. A very close link to the LIM system helps to request information about samples from the LIM system. It is possible to transfer special treatment options at this time. If the LIM system contains for instance information like “necessary dilution factor 9:1” or “average value of the last five results”, the software of the new PAMAS AS3 will handle this information accordingly.

The system can be built to match existing trays. Large systems with several hundreds of samples are possible to allow continuous unattended operation (three shifts within 24 hours).

The system is servo motor driven. This allows a much higher speed and much less noise than stepper motors.

The system table has an integrated sump that collects any spillage. A drainage system can be connected to feed the spillage to a central collection system.

4.4. Flushing

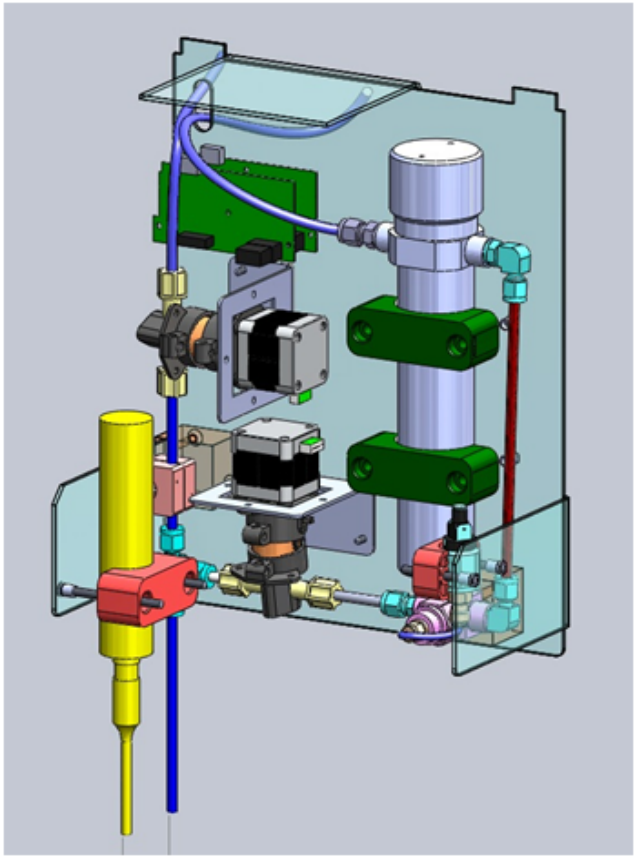
Cross contamination between samples is a critical problem. The new PAMAS AS3 autosampling system will have a flushing device to clean the sampling system between two samples.

If a sequence of samples contains a 22/18/16 sample followed by a 14/9/6 sample, this means that in raw data 4000 000 particles in the first sample are followed by 8000 particles in the next one. But if the sequence is reversed, no flushing is needed and speed can be increased.
The new PAMAS AS3 system can read old values from LIM systems and program an optimized flushing cycle between samples.

Flushing has to include all sample wetted parts including inner tube walls, pumps, outer tube walls of the suction tube and sample preparation devices.

4.5. Dilution System

Some samples may either be too dirty or too viscous or may contain undissolved additives. In this case, sample dilution with low viscous solvents helps to get reliable measuring results. The new system has an automatic dilution system that adds a programmable amount of solvent online to the raw sample. This mixture is fed through a static mixer to achieve good homogeneity of the mixture.



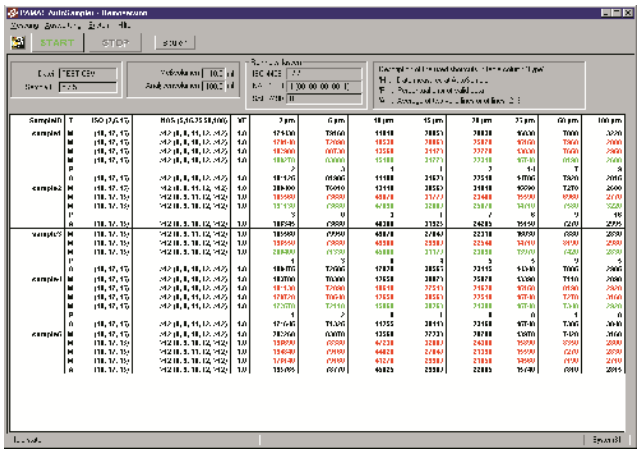
Picture 5: The integrated particle counter includes an ultrasonic agitation probe and a device for automatic dilution. (Picture: PAMAS)

Feeding unmixed fluids with very different vapour points is not easy. If the fluid is pulled through the tubes (vacuum in the tube), the fluid with the lower vapour point may form gas bubbles.

It is essential to design the dilution system so that all deep vacuum zones in the tubing are avoided.

5. Outlook

The new autosampling system PAMAS AS3 is currently being developed at the PAMAS company facilities in Rutesheim. Meanwhile, the mechanical design of the unit is ready and a prototype has been built. Currently, the software of the PAMAS AS3 is being programmed. The software will be optimized and specifically adapted to the customer's needs. For each different LIM system, the software will be individually developed and programmed so that it perfectly fits into the customer's existing laboratory infrastructure. With the aid of the PAMAS autosampling software, measuring results will be reported according to common cleanliness standards including ISO 4406 [1], NAS 1638 [2] and SAE AS 4059 [3].



Picture 6: The PAMAS Autosampler software reports measuring results in compliance with common cleanliness standards including ISO 4406, NAS 1638 and SAE AS 4059. (Picture: PAMAS)

The new PAMAS AS3 will be utilizing the most recent and advanced PAMAS sensor technology. Same as all PAMAS instruments, the integrated particle counter will be calibrated according to the calibration standard ISO 11171 [4]. Calibration according to ISO 4402 [5] can be done on request.

The PAMAS AS3 system will be available in about three months.

6. Conclusion

The market for high quantity systems is small but prestigious. PAMAS has been the major manufacturer for these systems and will carry out this role in the future.

The new system features all requests for improvement that have been brought forward from users.

List of References

[1] ISO 4406:1999: *Hydraulic fluid power – Fluids – Method for coding the level of contamination by solid particles.* Published by: International Organization for Standardization, Geneva, Switzerland. Second edition, December 1999.

[2] NAS 1638:2001: *Cleanliness requirements of parts used in hydraulic systems.* Published by: Aerospace Industries Association of America Inc., Arlington, USA. Approved in January 1964, revised in March 2001. Current status of NAS 1638: This standard has been withdrawn.

[3] SAE AS 4059 E: *Aerospace Fluid Power – Cleanliness Classification for Hydraulic Fluids.* Published by: Society of Automotive Engineers, SAE Aerospace, Warrendale, USA. Revision E, 2005.

[4] ISO 11171:2010: *Hydraulic Fluid Power – Calibration of Automatic Particle Counters for Liquids.* Published by: International Organization for Standardization, Geneva, Switzerland. Second Edition, November 2010.

[5] ISO 4402:1991: *Hydraulic fluid power – Calibration of automatic-count instruments for particles suspended in liquids – Method using classified AC Fine Test Dust contaminant*. Published by: International Organization for Standardization, Geneva, Switzerland. Second edition, October 1991. Current status of ISO 4402: This standard has been withdrawn.