Piezoelectric Droplet Generator for the Calibration of Particle-Sizing Instruments

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Using a piezoceramic tube and a continuous glass capillary, droplets in a diameter range between 10 µm and 100 µm can be generated. This corresponds to a volume of up to 0.6 pl. The velocity of the generated droplets depends on droplet size but is constant for each diameter. The liquid can be dosed as single droplet, as an accumulation of droplets or as a chain of droplets in a frequency range between 1 Hz up to 3 kHz. The lifetime of the droplets depends on droplet size and the chemical and physical properties of the dispersed liquid. In addition, for water droplets the humidity of the air near the droplet trajectory influences the lifetime of the droplets.

1 Introduction

Work involving the optical measurement of particles, as well as research into aerosol technology and the development of different types of measuring equipment, makes use of different methods to produce and to examine single particles and particle collectives in the lower micrometer range. By means of various levitation devices the very fine particles in this range can then be held in the measuring volumes of optical laser processes. Some of the methods used are, for example, electrodynamic levitation [1] (particle size from 1 µm–100 µm), magnetic levitation (particle size from 150 µm–10^4 µm) or ultrasonic levitation (particle size dependent on material).

For measuring instruments in this range it is important to obtain spherical particles which are homogeneous and with surfaces as smooth as possible. This can be most easily achieved with liquid droplets, which in the range below 100 µm are usually perfectly spherical.

It is helpful for the calibration of such optical particle-sizing instruments to make use of, i.e. to generate, monodisperse droplets of uniform diameter. However, the capability to measure the exact reproduction of particle-size distribution with such instruments has until now been frustrated by the lack of so-called calibration distributions. Any attempt in the lower micrometer range to prepare these by using extremely fine solid particles is negated by the forces exerted in this size range: Since particles would continually be lost and the original particle-size distribution would not be reproducible, such a "norm" would not be generally applicable.

Instead, for this purpose it is useful to use multiple droplet generators which can, for instance (and ideally), produce droplets of 10, 20, 30, 40, ... µm diameter. The number of droplets produced per generator must be programmed and monitored. Any number of droplets can be programmed per size category, so that this number per category can then be scanned and registered by the particle-sizing instrument. In this way any type of distribution can be programmed and the registration checked by the sizing instrument. This method is reproducible. Moreover, it becomes of secondary importance whether the exact diameter is produced as indicated by the decade or whether there is a degree of variance.

Electrostatic generators are capable of generating uniform droplets [2]. Other generators work without the use of any auxiliary equipment, the only means needed is a stimulation to produce droplets of equal size, for example:

- work in the overpressure range (droplet diameter from 50 µm–300 µm) or
- function without pressure (droplet diameter from 10 µm–100 µm).

2 Producing Droplets by Means of Piezo Droplet Generators

2.1 Droplet Generator in the Overpressure Range

When a liquid is forced through a nozzle by means of high pressure, the jet breaks up at high velocity in a wave shape. In order to produce droplets of equal size, the liquid is subjected to a (dominant) parasitic frequency. This is achieved, for instance, by stimulating the rhythmic contraction [3] of a concentric piezoceramic tube placed around a capillary in which the liquid is contained. Thereby the static drip-off leads to a diameter relation as follows (droplet d to a capillary orifice D), which is determined by the density ρ and the surface tension σ of the liquid, as well as the acceleration due to gravity g:

$$d = \sqrt[3]{(6\sigma D / \rho g)} \qquad (1)$$

Using the findings of Rayleigh and Weber it is possible to calculate the optimal parasitic frequency, whereby the droplet diameter bears a relation [4] to the nozzle diameter, which simplified (for water) can be shown as:

$$d \cong 1,9 \cdot D \qquad (2)$$

The result is a continual generation of droplets having the same diameter (droplet chain).

The continuous droplet generators can also be operated with excitation frequencies outside the optimal parasitic frequency. The results thus obtained, for example using water and a nozzle of 36 µm (diameter), produced droplets with a diameter ranging

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0930-7516/01/0303-0265 $ 17.50+.50/0

between 114 µm and 70 µm in the frequency range from 15 kHz to 75 kHz. During a (separate) extended experiment of 6 hours the phase-Doppler anemometer used to monitor the droplets recorded a standard deviation of 1.8 µm for the droplet diameter and a standard deviation of 0.09 m/s for the droplet velocity of 7.8 m/s measured in the vicinity of the nozzle orifice.

2.2 Droplet-on-Demand/Non-Pressure Generator

The droplet-on-demand generator has been developed from ink-jet computer printer technology. By means of a piezoceramic, a pressure wave is produced in a capillary system filled with liquid, causing a droplet to be ejected from a nozzle at high velocity[5]. The droplet diameter corresponds to the diameter of the nozzle and is thus smaller than in the case of generators working with the overpressure principle [10].

Since generators in the overpressure range are continually producing droplets at high frequencies, they use relatively large test amounts. Non-pressure generators can produce either single droplets, or sequences and droplet chains in the kHz range, whichever is required. Test amounts can therefore be reduced to less than a picoliter – which results in delivering an extremely precise dispensing of the liquid. Whereas the reproducibility of droplet size is very good, there is on the other hand the risk when using open systems of a blockage of the nozzle orifice and a build-up of algae, unless precautionary measures are taken.

Which type of generator will be more suitable depends on the application. For the production of droplets with a diameter of < 50 µm, as well as for requirements of single droplets or continual dispensing, the non-pressure system offers the most advantages.

Our efforts were subject to the requirement of developing a non-pressure droplet generator capable of producing droplets of constant diameter (with great precision) over a time period of several hours for tests with respect to phase-Doppler anemometry (PDA). Furthermore, ten classes of diameter were to serve for comparison tests. The generator had to be easy to maintain and no time should be lost in obtaining the desired droplet diameter. Costs were to be kept at a minimum. Data was already available from experience gained in the development of continually operating droplet generators in the overpressure range using piezoceramic tubes.

The following contains a description of the steps involved in producing single droplets and droplet chains in the kHz range by means of the drop-on-demand technique.

3 Description of the Components

3.1 Piezoceramic Tube

Almost the entire inner and outer surface of the piezoceramic tube[6] has an electrode. A voltage between the inner and outer electrode causes the tube to contract in a longitudinal direction thereby reducing its diameter.

First, an electronic signal generator delivers a pulse to the piezocrystal. The droplet sequence is determined by the pulse repetition frequency, or pulse rate. Since the piezocrystal is not operated in resonance, the pulse rate has to be low enough to allow the oscillation being caused by the pulse to die out. A ceiling is thus placed on the upper limit of repetition frequency. The pulse energy must be adapted to the droplet volume and be transmitted onto the liquid from the piezoceramic tube via the capillary. The mechanical coupling of the piezoceramic to the capillary is therefore of paramount importance. The piezoceramic tube used has a mechanical coupling factor of 0.6 for the dimensions: length = 20 mm, overall diameter = 2.2 mm, inner diameter = 1 mm. Fig. 1 shows a sectional view of the piezoceramic tube with imbedded capillary.

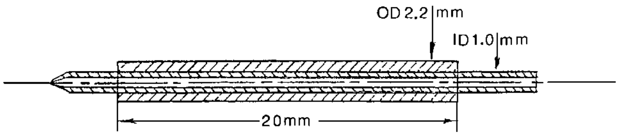

Figure 1. Piezoceramic tube with imbedded capillary.

3.2 Capillaries

Capillaries[7] made of borosilicate glass are used. Their outer diameter is one millimeter and predetermined by the inner diameter of the piezoceramic tube. The inner diameter is variable and can be freely selected within any manufacturing limits. We have chosen 0.6 mm as the guidance value for the inner diameter of the capillary.

3.2.1 Finishing of Capillary

The diameter of the droplet produced by the electromechanical pulse is approximately equivalent to the diameter of the capillary orifice. By varying the pulse amplitude it is possible to alter this value by approximately 10 %. Basically, there are two ways to taper the guidance value of 0.6 mm to a nozzle of 50 µm:

- pulling
- melting.

The pulling method is used to manufacture the micropipettes used in biology. The capillary is held at both ends, heated by means of a filament in the middle and then pulling pressure is applied at the ends. Depending on the heat and pulling force applied, the capillary is thus tapered in the middle until it breaks at the thinnest point. The fracture point is then ground to size the capillary orifice. After polishing the point of break the orifice is ready to use.

Switzer [8] describes an alternative technique. The capillary is held vertically over a weak propane gas flame. The heated end of the capillary contracts slowly in a longitudinal direction, narrowing the inner diameter whilst the outer diameter remains virtually unaffected. In this way it is then possible under the microscope to produce the nozzle orifice desired. The precise shape and a sharp break-off edge at the end face of the nozzle are crucial for ensuring droplet stability and reproducibility.

3.2.2 Bonding the Piezoceramic Tube to the Capillary

It is important to ensure the best possible bonding of the capillary to the piezoceramic tube. Optimal bonding entails:
- optimal mechanical coupling
- optimal thermal rating
- long-term stability

Suitable bonding is achieved using an epoxy two-component adhesive [9], e.g., Araldit AY103 and hardener HY991 supplied by Ciba-Geigy, or EPO-TEK 353ND from Polytek. Fig. 2 shows a wired-up piezoceramic tube with capillary (PTK), the PTFE protective tubing and the electrical wiring. The PTFE protective tubing provides electrical insulation as well as protection from mechanical damage.

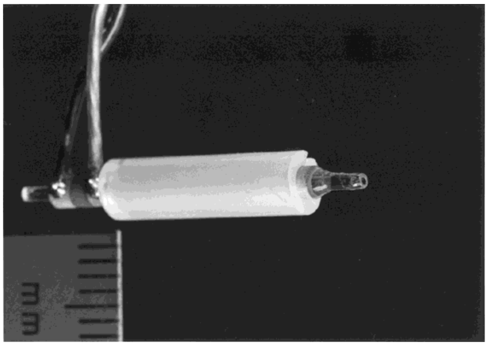

Figure 2. Piezoceramic tube with capillary and PTFE protective tubing.

3.3 Electronic Control Device

The electronics essentially constitute a pulse generator. The adjustable variables are:
- pulse width
- pulse amplitude
- pulse rate

In order to ensure stable functioning, there must be no temporal drift of these three parameters. This is particularly important with regard to pulse width and pulse amplitude. The following values have been determined for adjustment of the pulse width: droplet diameter in µm corresponds to the pulse width in µs. The pulse amplitude is dependent on the quality of the mechanical pulse transmission. From past experience we are also able to say: droplet diameter in µm corresponds to the amplitude in volts.

3.4 Storage Tank and Mounting of Head Unit

Since there is no mechanical coupling to any particular material mass, the user is relatively free to design the mechanical construction to suit the desired purpose. One simply has to ensure that the tip of the PTK corresponds to the level of the liquid.

4 Functional Tests and Results

Functional tests were carried out using bidistilled water (first passed through a 0.2 µm filter).

Using a hypodermic syringe, slight overpressure is built up in the storage tank until liquid begins to emerge from the tip of the device. The liquid should be held in the tip of the capillary with a miniscus.

After the apparatus has been set up the preselected pulse rate can then be started. Using a halogen lamp it is then possible to observe whether the droplets are formed. The easiest method for checking is to set up a halogen lamp with no more facilities behind the capillary and to visualize the droplet as a shadow image.

An LED stroboscope can also be used for continual monitoring and to check the droplet generator when put into operation for the first time. By means of making slight adjustments to delay the flash opposite the pulse on the piezoceramic tube, it thus becomes possible under the microscope to observe how pulse and amplitude influence droplet formation. The droplet velocity can be adjusted to within limits by means of the amplitude. If the amplitude is raised too high, satellite droplets are formed.

Using a phase-Doppler anemometer it was possible to measure the velocity distributions as well as the diameter size distributions of a 22 µm, 51 µm and 86 µm diameter droplet chain (each 10.000 runs at 50 Hz droplet sequence). Fig. 3 shows the velocity distribution. The narrow width of velocity distribution is evidence for the stable operation of the

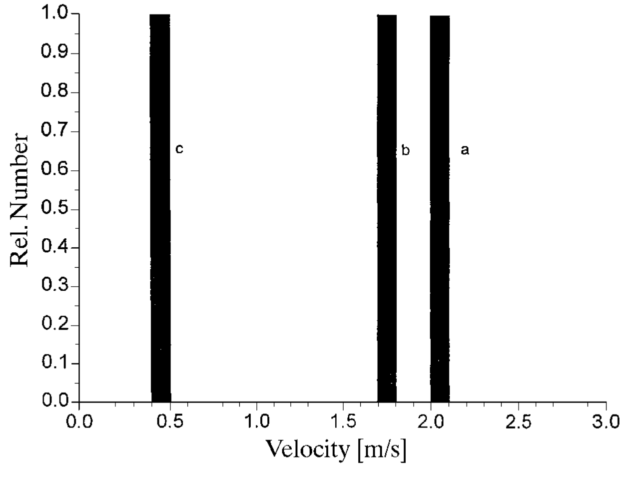

Figure 3. Velocity distribution of droplet chains: a) 22 µm, b) 51 µm, c) 86 µm.

generator. For many applications, e.g., as a reference for calibration or for dispensing, the standard deviation of droplet diameter is the most crucial value. This is verified by the results of the size distribution of droplets shown in Fig. 4. The diagrams reveal only a very slight standard deviation: Diagram a) 0.7 µm, diagram b) 0.4 µm, c) 0.8 µm. One can thus speak of a very precise reference procedure.

Bidistilled water was used for all the experimental runs. The open construction of the device permits easy changeover to a different droplet diameter as well as ease of maintenance, e.g., removing blockages of the head unit. In addition to water, other liquids with different viscosity and surface tension values were also used to produce droplets, e.g., glycerine (up to 86 % w/w), DMSO, nitro.

The objectives described in the introduction, i.e., to be able to operate ten droplet generators, to create different distributions, were achieved. The computer-controlled system has been tested. Fig. 5 shows the multiple head (tenfold) with pulse motor control.

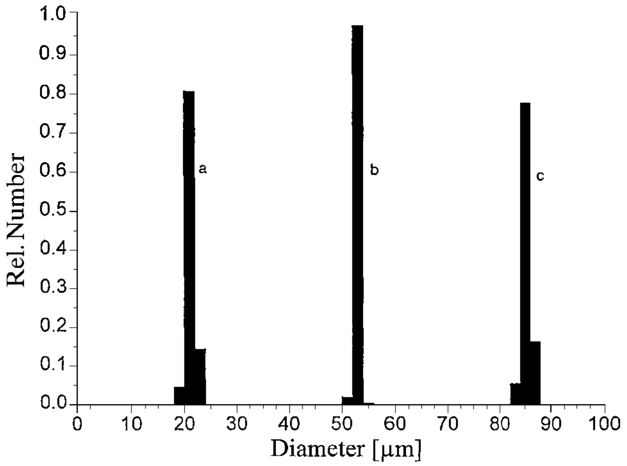

Figure 4. Diameter distribution of different droplet chains: a) 22 µm, b) 51 µm, c) 86 µm.

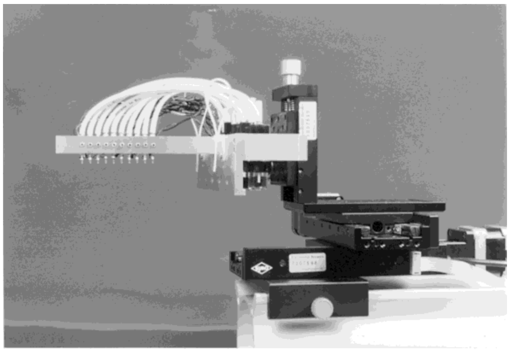

Figure 5. Multiple-head droplet generator with pulse motor control.

4 Conclusion

A droplet generator which works without pressure was successfully developed and tested. Its characteristics are:

- droplet diameter range from 10 µm to 100 µm
- droplet sequence from single droplet to droplet chain of 3 kHz
- good long-term stability of droplet diameter and droplet velocity

The device can easily be changed to provide the droplet diameter desired. Cleaning of the capillary is a simple operation, resulting in high availability factor and low running costs.

Received: April 20, 2000 [CET 1230]

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