

Generation of Monodisperse Micron-Sized Droplets using Free Adjustable Signals

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Abstract

This paper describes the generation of small water droplets in the size range down to a few microns. Commercially available inkjet printing devices are not suitable for producing such droplets since they produce satellite droplets. Furthermore, standard drop-on-demand devices are normally restricted to the generation of droplets with the same size as the orifice diameter. Using a new and more sophisticated computer-based signal generation system, smaller-sized droplets can be generated from the same orifice. A key feature of the design is the

generation of freely definable pulses. This enables the generation of acoustic modes within the fluid of the droplet generator, which leads to the generation of droplets without satellites. Only very few pulse forms enable the generation of suitable acoustic modes. Therefore, it is necessary to look for the specific pulse corresponding to the chosen droplet generator. Flexible pulse form generation appears to be more suitable than simple pulse forms for the generation of such droplets.

Keywords: drop-on-demand, micron-sized droplet diameter, piezoelectric droplet generator

1 Introduction

In many laboratories small, monodisperse droplets with a constant ejection speed are required. Their applications range from pure science, e.g., fluid dynamics and physical optics, to technological applications, e.g., DNA synthesis and droplet-based manufacturing. A recent overview is given by Lee in a monograph [1]. In addition to the many applications described by Lee [1], droplet generators are used for the development of particle sizing instruments [2]. Mitschke et al. [3] and Riefler et al. [4] describe methods to measure the size of inhomogeneous droplets. Small droplets are also subject to investi-

gate inclusions within droplets [4]. A further application of micron-sized monodisperse droplets is the calibration of optical measurement systems [5]. Other important fields of application for droplet generators are their use as micropumps [6, 7] and as dispensing devices for DNA microarrays [8]. The collision of droplets, produced by a droplet generator, with a surface has been investigated both for general purposes [9] and for the investigation of combustion processes [10]. Studies of the dynamic behavior of droplets for fire suppression make use of droplet generators to measure the cooling capacity of the water aerosols [11]. In rapid prototyping applications, DNA-sensors, LEDs, electronic devices and even refractive microlenses have been produced with drop-on-demand droplet generator systems [12].

Kung et al. [13] reported on a measuring device for single molecules using a droplet generator. This device is capable of producing droplets with a diameter of 4 μm next to the tip. The authors used a tip with a very small orifice measuring just 1 μm in diameter. After a few millimeters, the droplets are reduced from 4 to 1 μm in size due to evaporation.

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In this paper, attention is focused on piezoelectric drop-on-demand systems. In the 1970's, the first drop-on-demand designs were patented and some years later, the first numerical simulations were performed (see Kyser et al. [14] and literature cited therein). Nowadays, many new droplet generator designs are based on silicon-based piezosystems [15], which provide the possibility of integrating many droplet generators into a single device. A comparison of different designs and systems can be found in Burgold et al. [16].

Commercial inkjet print heads are commonly used for research applications where small droplets are required. The most frequently used working principles are bubble jet and piezoelectric inkjet systems. However, these devices have some drawbacks. The size of the droplets generated is still relatively large ($d_{drop} \geq 50 \mu\text{m}$) and, furthermore, satellite droplets are produced. In bubble jet or thermal inkjet print heads, a heating pulse is used to generate an expanding vapor bubble. This bubble forces liquid out of the nozzle, which results in the generation of a droplet. Stable operation of thermal inkjet print heads is highly dependent on the chemical composition of the fluid. This restricts research applications where diverse liquids commonly have to be dispersed. Moreover, non heat-resistant liquids cannot be dispersed.

Consequently, there is a recurrent interest in simple piezoelectric drop-on-demand devices that can be used with a wide range of different liquids. The nozzles are formed at the end of a glass capillary. In this paper, it is demonstrated that with such simple cylindrical drop-on-demand devices, improved operation can be achieved using sophisticated driving signals. Most simple piezoelectric droplet generators are made by hand. Therefore, the exact mechanical dimensions of the droplet generator and especially the nozzle will vary from device to device. An improved electronic system will help to find the best suitable driving signal to achieve stable droplet generation using individual droplet generators. Furthermore, the use of nozzles with a smaller diameter improves the stable generation of smaller droplets ($d_{drop} \leq 20 \mu\text{m}$).

The aim of this paper is to show how to generate highly stable, yet very small droplets with a very narrow size distribution over long time periods. In the first section, the simple piezoelectric droplet generator used for the application will be described. Following this, the generation of the driving signal and the signal amplification will be explained. A presentation of the optical visualisation system used then follows. Finally, some sample results of small droplets are presented.

2 Design

2.1 Droplet Generator

A description of the piezoelectrical droplet generator that was used can be found elsewhere [2, 17]. Since the basic design is similar, only a short description is provided here. A glass capillary is surrounded by a piezoceramic tube. After applying a voltage pulse, the contraction of the piezoceramic accelerates the fluid and leads to droplet ejection. A glass capillary (borosilicate) with an outside diameter of $d_o = 1 \text{ mm}$ and an internal diameter of $d_i = 0.58 \text{ mm}$ or 0.72 mm is provided with a nozzle and two types of piezoceramic tubes, i.e., a short one (14 mm) and a longer one (20 mm). The nozzle diameters of the droplet generators vary between 20 and $60 \mu\text{m}$, and in the case of continuous jet applications, sometimes up to $150 \mu\text{m}$.

2.2 Generation of Driving Signal

In the design presented by Ulmke et al. [2, 17] the driving signals were produced using specially designed electronic hardware. As such, only a limited number of the driving signal parameters could be varied. These included pulse width, pulse height and exponential decay of the pulse edge.

The present new design can be represented by the block diagram shown in Figure 1. The piezoelectric driving unit is split into a software part (Labview) and an electronic part (Amplifier). This separation allows one to generate completely arbitrary signals, dependent only on the capabilities of the digital-analog converter (DAC). A conventional PC is used to generate the signals. The software-based signal generation system was developed using Labview (National Instruments).

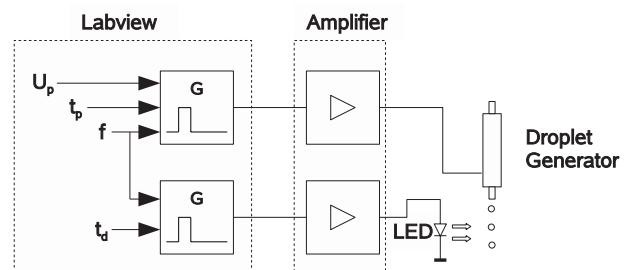


Fig. 1: Block diagram of the electronic driver; the left part corresponds to the signal generation within the PC using a Labview program; the signal is transferred via a RG-58 coaxial cable to the amplifier; U_p and t_p each consist of six individually adjustable voltages with a corresponding signal time; f_{drop} is the frequency of the generated droplets and t_d is the delay time of the LED stroboscope driver.

A screenshot of the front panel of the instrument can be seen in Figure 2. The DAC is a PCI board from the same manufacturer with a sampling rate of 1 MS/s, and thus, the shortest pulse duration is 1 μ s. The voltage output U_{out} of this PCI board is freely adjustable in the range $-10 \text{ V} \leq U_{out} \leq 10 \text{ V}$.

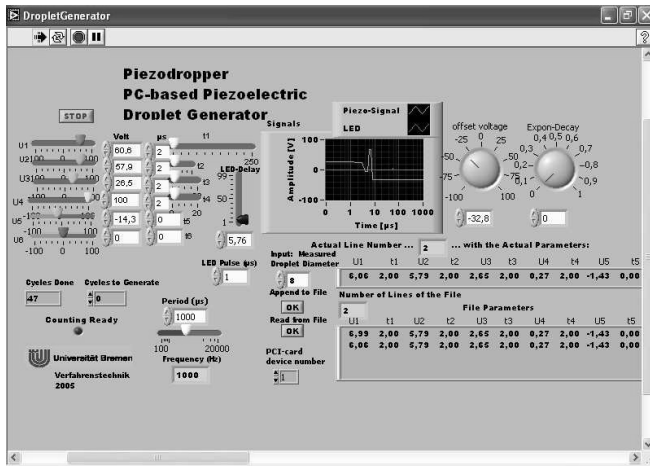


Fig. 2: Screenshot of the control software programmed with Labview. In the center is an illustration of a software oscilloscope. The voltages and their corresponding time duration in μ s can be adjusted on the left. The resulting pulse form is shown in a logarithmic time scale for better resolution. The LED delay is also illustrated as a short spike. To the right of the oscilloscope are the knobs for adjusting the offset voltage and the exponential drop. Further down are entry fields for the storage of useful parameters.

Using this software, it is possible to generate arbitrary pulse forms. At the moment, the pulse form consists of six voltage levels U_1, \dots, U_6 , each with an individual adjustable time duration, t_1, \dots, t_6 . The pulse generation flowchart diagram of the Labview program is shown in Figure 3. The exponential decay of the pulse edge can be adjusted for the last voltage stage, U_6 . An offset voltage U_{off} (referred to as 0 V) applied to the piezoceramic tube results in mechanical pre-stressing of the glass tube. This improves the droplet generation process.

In addition, the software generates a delayed pulse signal for the LED stroboscope driver. The delay time is adjustable and this enables the generated droplets to be observed and monitored at different time steps.

The implementation of a counter routine allows the generation of a defined number of droplets. The number of droplets and all other signal parameters can be saved in a text file onto the computer hard disk. Each line of these files contains one parameter set for generating a particular droplet size. After reading a previously saved text file, the system generates an exact reproduction of a former droplet configuration.

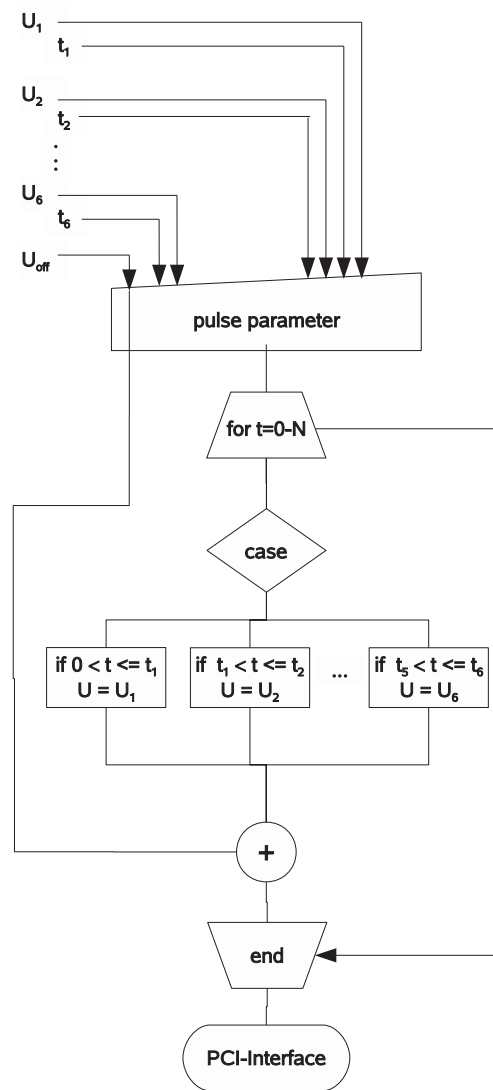


Fig. 3: Block diagram of the pulse generation algorithm; each voltage U_i and pulse time t_i pair generates a voltage U_i during $t_i \mu$ s; the offset voltage U_{off} is added after pulse generation; N corresponds to the pulse period in μ s.

2.3 Amplifier

Further development of the electronic device led to a new design for the high voltage amplifier. A dual polarity regulated power supply delivers a fast high voltage operational amplifier, manufactured by Apex Microtechnology. A circuit diagram of the linear, non-inverting drive amplifier is shown in Lee [1]. The pulse signal from the DAC is amplified by ca. a factor of 10. This amplified pulse signal drives the piezoceramic tube. An example of a driving pulse used to generate the droplets is given in Figure 4.

In order to observe the droplets at different U time t_i stages, an amplifier was also developed to drive the LED stroboscope. The driving signal with an adjustable delay is

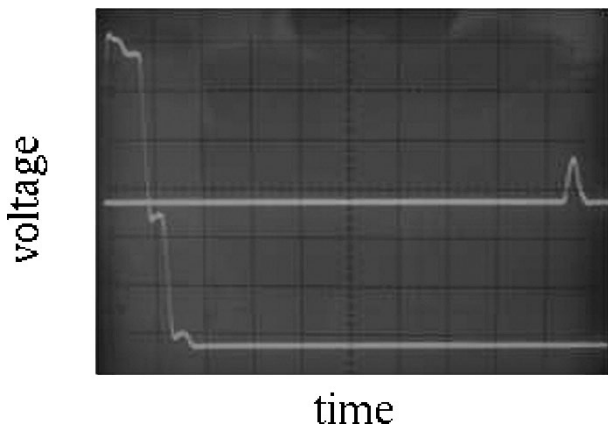


Fig. 4: Photograph of the oscilloscope screen; the staircase signal is the pulse used to generate droplets with a diameter of $d_{drop} = 8 \mu\text{m}$ (one square corresponds to 10 V and 10 μs); the small right peak is the driving signal of the LED stroboscope (one square corresponds to 1 V and 10 μs).

generated by the DAC on the PCI board. If the droplet velocity is high, the stroboscope flash time should be in the time range of $t_f = 1 \mu\text{s}$. Such a short exposure times require high illumination intensities. Therefore, it was decided to drive the stroboscope LED with a MOSFET stage to generate high current pulses with an overload of the LED by a factor of 10 to 20.

LEDs were chosen instead of laser diodes for the stroboscope since monochromatic light would produce a high level of interference in the generated droplets. Laser diodes may be useful in typical light scattering measurements where, in particular, the interference patterns are the desired response of the object. However, in the current work, it was intended to observe the droplets optically to gather immediate information about the system response to changes in the driving pulse. Furthermore, continuous light is required for making adjustments to the optical setup. Both of these demands are best satisfied with LEDs.

3 Optical Setup

The optical setup is illustrated in Figure 5. The droplet generator consists of the piezoceramic tube and produces the droplets. The droplet generator surrounds are observed using two video microscopes. The main component of the first imaging optical part is a long distance microscope (Qestar *QM100*) with a CCD camera, *CCD 1*. The focus point of the *QM100* is adjusted to the shortest value of ca. 150 mm with a resolution of 1.1 μm (0.024 arc s). This leads, together with a doubling lens between

QM100 and *CCD1*, to a maximum magnification of ca. 880. The droplets are illuminated by a Köhler setup with *LED1* as the light source. The collector *Col* maps the lightening chip area of *LED1* onto the iris diaphragm, *Id1*, in the vicinity of the condenser *Con*. *Id1* is also referred to as the aperture diaphragm. The second iris diaphragm, *Id2*, is the luminous-field diaphragm, which serves to adjust the diameter of the illuminated light spot.

The high magnification of the *QM100* is responsible for difficulties in detecting the droplets, in particular when the pulse signal for the droplet generator is being adjusted. Therefore, a second video microscope was set up with a lower magnification of 130. These two monitoring devices enable the easy adjustment of the droplet parameters.

With this setup, one is also able to measure the velocity of the generated droplets. The velocity is determined simply by measuring the distance on the video monitors divided by the time difference. The distance measurement should be performed after the droplets have become spherical in order to fulfill the conditions for constant droplet velocity [18].

In order to observe very small droplets, the limit of resolution for the optical system has to be in the size range of several microns. Thwar and Velegol [19] reported that the limit of resolution with an ordinary microscope is in the range of up to a hundred nanometers. They used an immersion objective which was very close to the objects. In contrast, the droplets in the current setup are far away from the *QM100* objective and, therefore, one cannot expect a resolution as high as reported by Thwar and Velegol [19].

4 Experimental Results

Each droplet generator has its own individual parameter set. The most important parameters are amplitude and

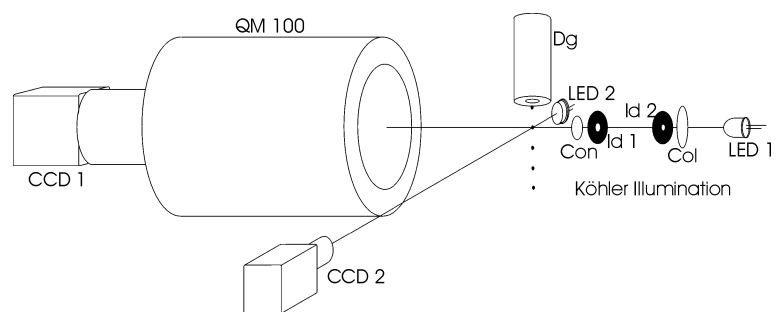


Fig. 5: Block diagram of the experimental apparatus. The droplets from the droplet generator DG are illuminated perpendicular from LED1 and from LED2.

pulse duration. The determination of the appropriate parameter values has attracted considerable attention. Satellite droplets may appear if the amplitude of the pulse is too large. An amplitude that is significantly too large produces arbitrary droplets like a spray. The other important parameter is pulse duration. As a rule of thumb, the shorter the pulse duration, the smaller are the droplets.

The current investigation results in the introduction of a third important parameter. The usual pulse shape in drop-on-demand droplet generators is a rectangular pulse. However, Dong et al. [20] investigated single- and double-peak waveforms. They found that the waveform should be specially designed to avoid satellite droplets. The current investigation confirms this observation and arbitrary waveforms are adjustable with the new electronic design. It is found that the generation of a staircase pulse leads to the stable generation of smaller droplets compared with those generated by a rectangular pulse or pulses with rounded shape, e.g., exponentially damped, rising or falling edges.

Figure 6 shows a small droplet from a captured video stream. Distilled water at room temperature (dyn. viscosity, $\eta = 0.001 \text{ Ns/m}^2$, surface tension, $\sigma = 0.072 \text{ N/m}$) was used as the droplet substrate. The generation rate of the droplets is $f_{drop} = 200 \text{ Hz}$, but f_{drop} may vary between single droplet generation and up to $f_{drop} \approx 1 \text{ kHz}$. The frame rate of the CCD camera is $f_f = 25 \text{ Hz}$. Therefore, the image in Figure 6 is a shot that consists of four droplets. It is important to emphasize that the droplet generation process is highly stable. Thousands of droplets are generated over periods of at least some minutes. A previous investigation using a phase-Doppler anemometer showed a very low standard deviation of $\sigma \approx 0.4\text{--}0.8$ for 10000 generated droplets [2]. In addition, each droplet was illuminated with a very short light flash of $t_f = 1 \mu\text{s}$. For these reasons, the droplet generation

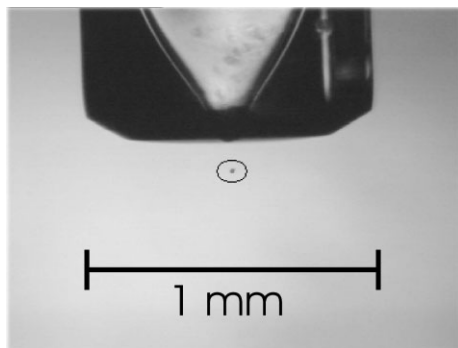


Fig. 6: Image of a slightly contrast-enhanced $8 \mu\text{m}$ droplet within the circle, together with the tip of the droplet generator; nozzle diameter is $40 \mu\text{m}$.

process is not a single event that needs to be observed with high speed cameras, and hence, one can observe an average number of droplets. The frame in Figure 6 shows the tip of the droplet generator. One can see the taper in the bright interior, which leads to a nozzle with a diameter of $40 \mu\text{m}$. The image of the droplet consists of only a few pixels. The contrast of the pixels was slightly increased and the droplet was marked with a circle.

The use of the *QM100* leads to better resolution, shown in Figure 7. The frame in Figure 7 shows a droplet with a diameter of $d_{drop} = 8.4 \mu\text{m}$. The droplet is from the same process as shown in Figure 6. With the higher resolution of the *QM100* instrument, the process of droplet formation and separation can easily be studied. Some video frames have been captured where the different modes generated after applying the pulse shown in Figure 4 can be seen. The velocity of the droplets shown in Figure 7 is ca. $v = 5 \text{ m/s}$. This is about twice the velocity of larger droplets ($v \approx 2 \text{ m/s}$). This relationship between increasing droplet velocity and decreasing droplet diameter is also stated by Temple [21]. Two arrows are inserted in the image shown in Figure 7 and they indicate that the fluid is wetting the nozzle surface. The nozzle itself with a diameter $d = 40 \mu\text{m}$ is invisible between the arrows.

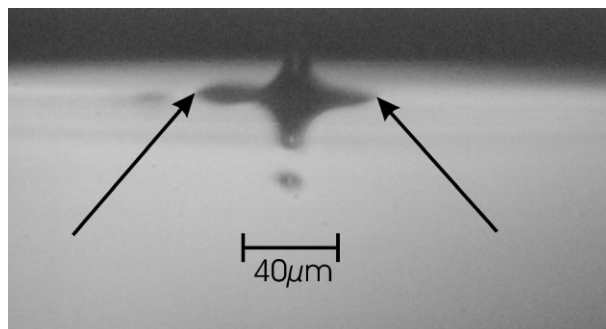


Fig. 7: Image of an $8 \mu\text{m}$ droplet shortly after separation from the bulk fluid; the size of the image is a 748×384 pixel; the arrows indicate the wetting fluid during a droplet generation process.

5 Discussion

The operating principle of the current piezoelectric drop-on-demand droplet generator is the compression of the fluid within a cylindrical vessel, which, in the present case, is a glass capillary with an internal diameter of $d_i = 580\text{--}720 \mu\text{m}$. This compression creates a complex acoustic wave within the fluid chamber that leads to a pressure gradient field. As a result, liquid is pushed out of the nozzle at the nozzle fluid-air interface.

The avoidance of satellite droplets is a main objective for droplet generators in laboratories. In the case of large droplets, the generated satellite droplets are some-

times large enough to fly stably in the same direction as the main droplet, which means that they may not disturb the investigation. However in most cases satellite droplets are undesired. To avoid satellite droplets, one has to keep the ejected liquid column as short as possible. The surface tension of the fluid is then strong enough to absorb the fluid back to the fluid chamber. This can be achieved by carefully adjusting the electrical driving signal, in particular the voltage amplitude, while observing the generated droplets with a monitoring device, e.g., a video microscope. An example of a short liquid column is shown in Figure 7. Lee [1] and Furbank and Morris [22] presented photographs showing long liquid columns and the resultant formation of a satellite droplet consisting of these liquid columns.

As a rule of thumb, one can state for the current piezoelectrical droplet generator, that pulse durations in the range of 60–100 μs generate droplets with diameters of $d_{drop} \approx 40\text{--}100 \mu\text{m}$, while pulses shorter than 10–20 μs generate small droplets with diameters of $d_{drop} \approx 20\text{--}40 \mu\text{m}$. Pulse durations between 1–10 μs result in even smaller droplets, with diameters of $d_{drop} \leq 10 \mu\text{m}$. The study by Temple [21] deals with the modeling of acoustic waves of the fluid in a droplet generator. The author concludes that a special configuration of interacting pressure pulses is responsible for the generation of droplets much smaller than the nozzle diameter. Droplets from typical drop-on-demand generators normally have the same diameter as the nozzle. However, short driving pulses generate droplets with a quarter of the diameter of the nozzle [21]. At the same time, the velocity of these droplets is twice as the usual droplets. This dependence of increasing droplet velocity upon decreasing droplet diameter is confirmed by the current results (see section 4 Experimental Results). The early observations made by Shield et al. [18] are also confirmed using single rectangular pulses. They stated that the pulse length is a critical parameter for proper drop ejection and that each droplet generator is related to one individual fundamental pulse length. However, it has been found that each droplet generator possesses more than one fundamental pulse length. In combination with specially adjusted pulse voltages, each droplet generator has a stable mode of operation for different specific combinations of pulse length and pulse voltage.

Arbitrary shaped pulse as well as bipolar pulse excitation enables the generation of smaller droplets compared with single monopolar pulses. This result is in line with that of Chen and Basaran [23].

Finally, although only water was reported as the substrate in this study, other fluids such as dimethyl sulfoxide or 20% glycerol-water mixture have been successfully applied to generate slightly larger droplets.

6 Conclusions

A freely adjustable driving pulse enhances the droplet generating process. Such pulses enable the generation of smaller droplets compared with single monopolar pulses. As a result, the droplet diameters may vary in the range $d_{drop} = 8\text{--}70 \mu\text{m}$ for a droplet generator with an orifice of 40 μm diameter.

The detection of droplets generated in the size range smaller than 10 μm is fairly complicated. In contrast to the use of common microscopes, in order to image these small droplets, long-distance microscopes together with Köhler illumination, adapted for long distances, are required.

The droplet generator device presented in this paper is based on the use of one PCI-card with 4 independent analog output channels. This allows the simultaneous generation of 4 independent droplets with an appropriately adapted software panel. Droplet collision experiments can be investigated with the use of two amplifiers and two droplet generators.

In addition, a predefined number of droplets can be generated, which may be used for dosage of small fluid quantities down to 0.5 pL for specialized applications, e.g., a batch micro-reactor.

7 Nomenclature

Symbols

d_{drop}	droplet diameter
d_o	outside diameter of the glass capillary
d_i	inside diameter of the glass capillary
U_p	pulse voltage
t_p	pulse duration
t_d	delay time of light flash
t_f	flash time of stroboscope
f_{drop}	droplet generation frequency
U_{off}	offset voltage
v	velocity
η	dynamic viscosity
σ	surface tension

Abbreviations

<i>DAC</i>	Digital-Analog-Converter
<i>Id</i>	Iris diaphragm
<i>Con</i>	Condenser lens
<i>Col</i>	Collector lens
<i>Dg</i>	Droplet generator

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