

On the comparison of electrorheological measurements with different generation of an electric field

P Peer^{1,4}, M Stenicka², P Filip¹ and V Pavlinek^{2,3}

¹Institute of Hydrodynamics, Academy of Sciences of the Czech Republic,
Pod Patankou 5, 166 12 Prague 6, Czech Republic

²Centre of Polymer Systems, University Institute, Tomas Bata University in Zlin,
Nad Ovcirnou 3685, 760 01 Zlin, Czech Republic

³Polymer Centre, Faculty of Technology, Tomas Bata University in Zlin,
namesti T. G. Masaryka 275, 762 72 Zlin, Czech Republic

E-mail: peer@ih.cas.cz

Abstract. The rheological measurements of PANI powders mixed (10 wt.%) in silicone oil were compared using two electrorheological (ER) apparatuses different in generation of an electric field: (i) a rotational rheometer Physica MCR 501 (Anton Paar Co.) equipped with the electrorheological cell, (ii) a rotational rheometer Bohlin Gemini CVOR 150 (Malvern Instruments) modified for ER experiments. It was shown that the data obtained for a plate-plate arrangement using the Physica MCR 501 provide the highest values exceeding both the plate-plate data for the Gemini 150 and the, mutually comparable, concentric cylinders data for both rheometers.

1. Introduction

Most of commercial available rheometers can be equipped with electrorheological (ER) devices fully using the functionality of the basic instruments. However, generation of an electric field in the individual devices substantially differs. The aim of this contribution is to compare the experimental data obtained from the following rheometers: a rotational rheometer Physica MCR 501 (Anton Paar Co.) and a rotational rheometer Bohlin Gemini CVOR 150 (Malvern Instruments). For both instruments two geometrical arrangements were used: parallel plates and concentric cylinders. Each system is covered with a hood ensuring safety regulations and temperature stability controlled by Peltier elements (Physica) or fluid circulator (Bohlin). The bottom plate for the parallel plate device and the cup in the concentric cylinder setup are grounded. Opening the hood automatically switches the power supply off. The rheometers themselves are fully isolated from an electric current. The substantial difference between the two electrorheologically adapted rheometers consists in completing an electric circuit through an upper shaft.

In the case of the MCR 501 a spring wire adhering to an upper shaft by an application of a spring completes a circuit loop, see figure 1. Intensity of an electric field is varied through an external DC high voltage power supply unit HCP 14-12500 (F.U.G. Elektronik GmbH, Rosenheim, Germany). It provides an electric field strength up to 12.5 kV/mm at the condition that electric current does not exceed 1 mA (otherwise a voltage is proportionally reduced). Dimensions of the measuring systems

⁴ Corresponding author.

used are as follows: P-PTD200/E parallel plates of a diameter 50 mm, C-PTD200/E a bob and cup arrangement with the inner and outer diameter 16.6 mm and 18 mm, respectively.

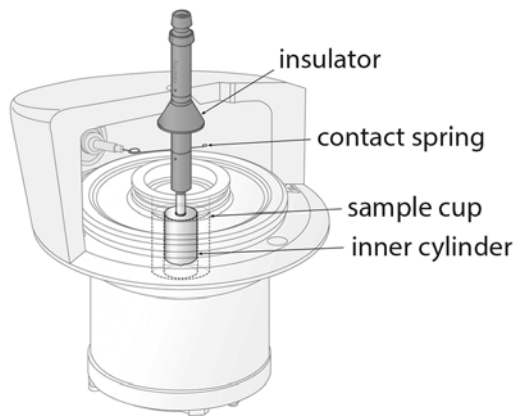


Figure 1. Geometrical position of a spring wire (contact spring) in the Physica MCR 501 equipped with the C-PTD200/E device (a bob and cup arrangement).

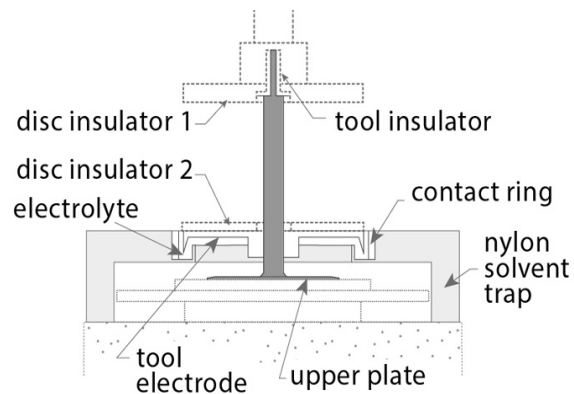


Figure 2. Geometrical location of electrolyte in the Gemini 150 device.

On the other hand the circuit loop in the Gemini 150 device is completed through an electrolyte housed in the circumferential gutter, see figure 2. Intensity of an electric field, influenced also by a density of electrolyte, is regulated by a DC high-voltage source TREK (TREK 668B, USA) providing the electric field strength up to 3 kV/mm with a maximum electric current 5mA. Dimensions of the measuring systems used are as follows: the diameter of parallel plates attains 40 mm and in the concentric arrangement the inner and outer diameter is 25 mm and 27.5 mm, respectively.

Prior to the ER experiments using either rheometer there is a necessity to carry out the measurements if mere air is applied. This enables to measure a friction between a spring wire and an upper shaft, or between a contact ring and electrolyte. Then these magnitudes have to be subtracted from the data obtained for ER materials.

2. Experimental

2.1. Material

Electrorheological suspension was prepared by mixing of PANI powder (Sigma Aldrich, USA, base, 50,000 g/mol) with silicone oil (Lukosiol M200, Chemical Works Kolin, Czech Republic) in 10 wt.% concentration. PANI powder was ground, sieved to obtain particle sizes smaller than 45 μm , and dried at 80°C in vacuum oven to a constant weight.

Electrorheological properties (shear viscosity, storage and loss moduli) of polyaniline particles suspended in silicone oil were measured using both devices and both geometrical arrangements. Each point in steady shear flow measurements as well as in small-strain oscillatory tests (dynamic amplitude sweep and frequency sweep) was measured at least three times.

2.2. Geometrical arrangements

The following gaps were used for the individual ER devices:
Physica MCR 501: PP 50 - gap 0.3 mm, CC 17 - gap 0.7 mm;
Gemini 150: PP 40 - gap 0.3 mm, CC 25 - gap 1.25 mm.

2.3. Temperature

The temperature was kept at 20°C during experiments.

2.4. Conductivity measurement

The DC conductivity of PANI base pressed into pellets (13 mm in diameter, 1 mm in thickness) was determined by a four-point method direct conductivity measurement in a Van der Pauw setup. The conductivity of PANI samples attained 7.72×10^{-9} S/cm.

2.5. Scanning electron microscope

Figure 3 illustrates morphology of PANI particles using a scanning electron microscope (SEM, Tescan, Czech Republic) with the accelerating voltage of 30 kV.

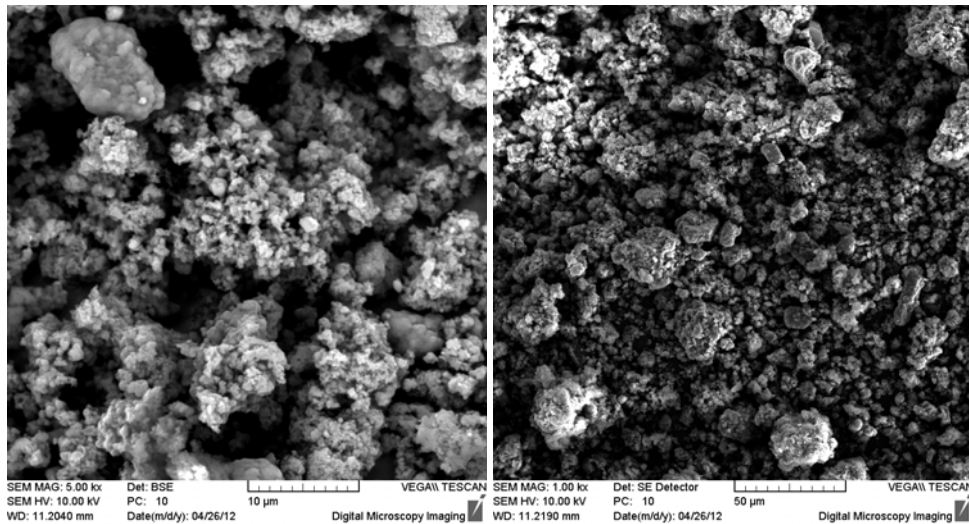


Figure 3. SEM pictures of PANI particles.

3. Experimental intercomparison of both generations of an electric field

First, flow curves of a carrier fluid (silicone oil) were obtained followed by ER measurements of the prepared ER suspension. The comparisons were always made for all four ER devices (two in either rheometer).

3.1. Measurements of silicone oil without electric field

Basic introductory comparison was carried out with a carrier silicone oil. This measurement is documented in figure 4. As can be seen, the flow curves for shear rate in the range $1-100 \text{ s}^{-1}$ are identical.

3.2. Measurements of ER suspension

The samples (10 wt.% concentration of PANI powder in silicone oil) were stirred mechanically and then placed in an ultrasonic bath for 30 s before each measurement. Consequently, the intercomparisons were made for three different modes. The courses of measurements (time intervals, electric field strength (xxx V stands for the individual values of voltage as indicated in the figures), range of measured points) are summarised in table 1.

For each mode, figures 5-10 compare behaviour of flow curves consequently for the same geometry (plate-plate (PP) or concentric cylinders (CC)) of both rheometers, and both geometries of individual rheometers. The experimental data in these figures are always reduced by the values obtained for the corresponding measurements with mere air.

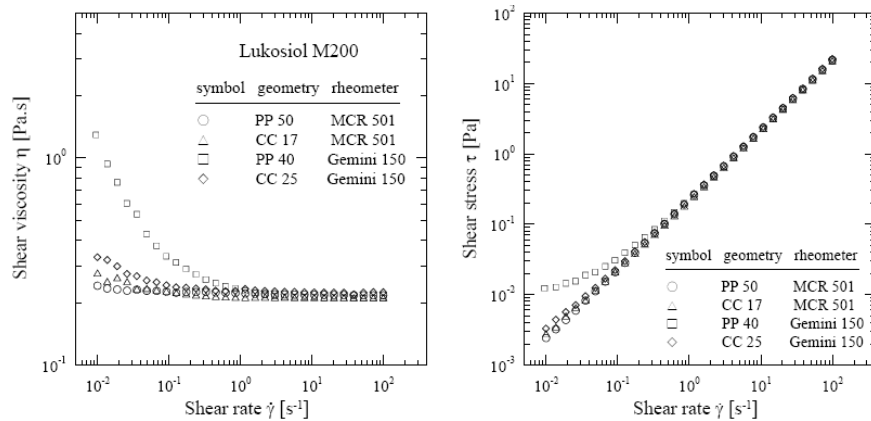


Figure 4. Flow curves of silicone oil ($E = 0$ kV/mm).

Table 1. Regimes of measurements for the individual modes.

$T=20^\circ\text{C}$	Mixing	Pause	Pause	Measurement
Steady shear	10 s^{-1}	0 s^{-1}	0 s^{-1}	$0.01 - 100\text{ s}^{-1}$
	30 s	30 s	60 s	
	0 V	0 V	xxx V	xxx V
Amplitude sweep	10 s^{-1}	0 s^{-1}	0 s^{-1}	$0.0001 - 1 / 0.01[-]$
	30 s	30 s	60 s	5 Hz / 3 Hz
	0 V	0 V	xxx V	xxx V
Frequency sweep	10 s^{-1}	0 s^{-1}	0 s^{-1}	$0.001 / 0.0006 [-]$
	30 s	30 s	60 s	0.1 - 10 Hz
	0 V	0 V	xxx V	xxx V

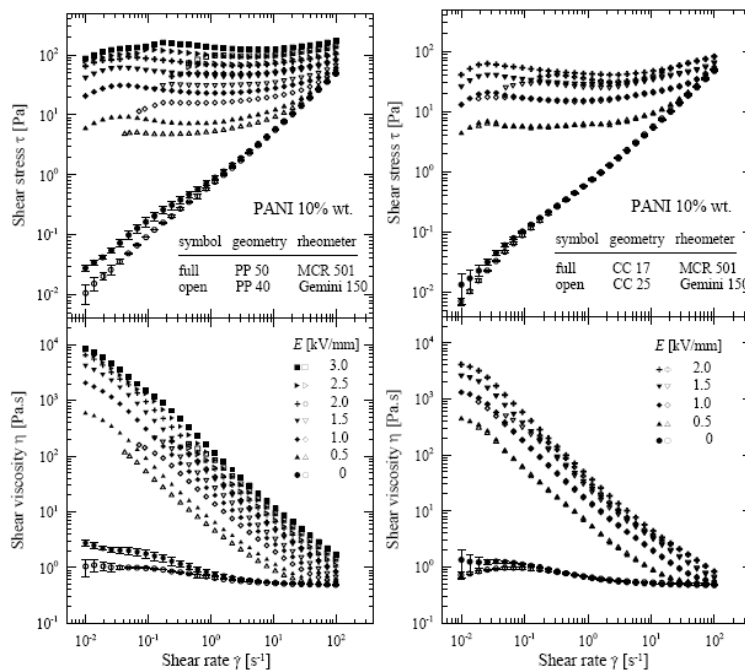


Figure 5. Shear viscosity and shear stress in dependence on shear rate, same geometrical arrangement (PP or CC), different rheometers.

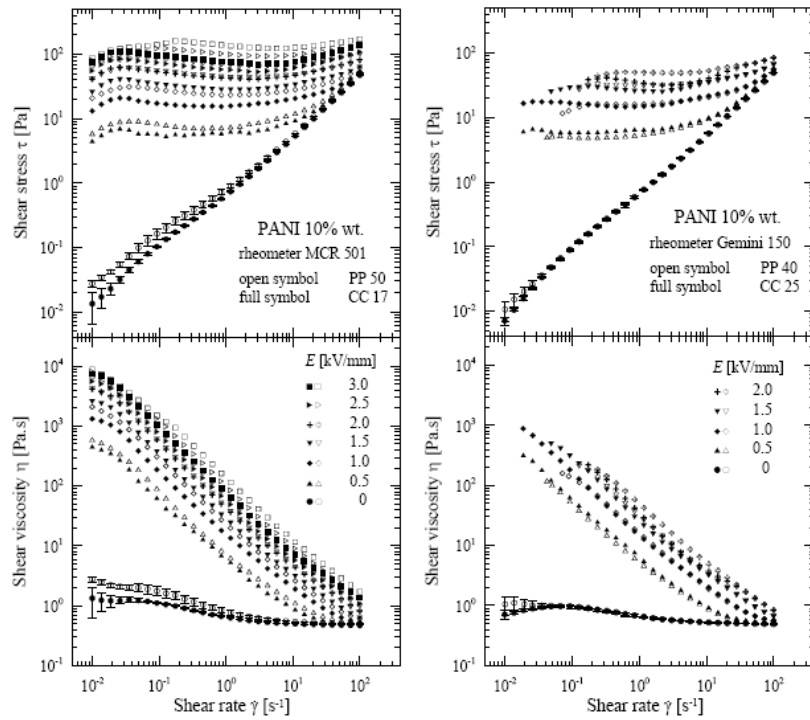


Figure 6. Shear viscosity and shear stress in dependence on shear rate, different geometrical arrangements, same rheometer.

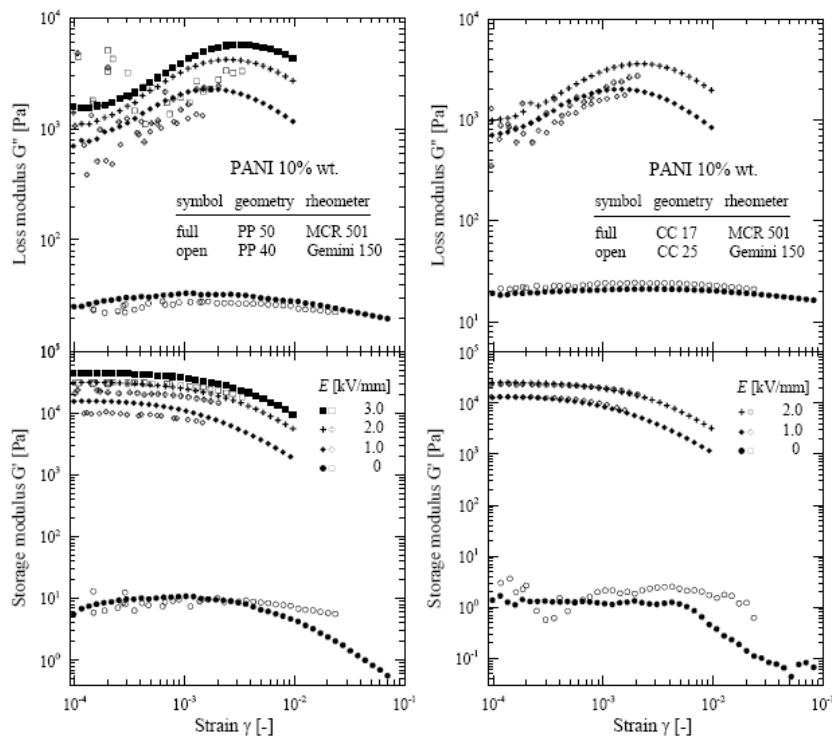


Figure 7. Storage and loss moduli in dependence on strain, same geometrical arrangement (PP or CC), different rheometers.

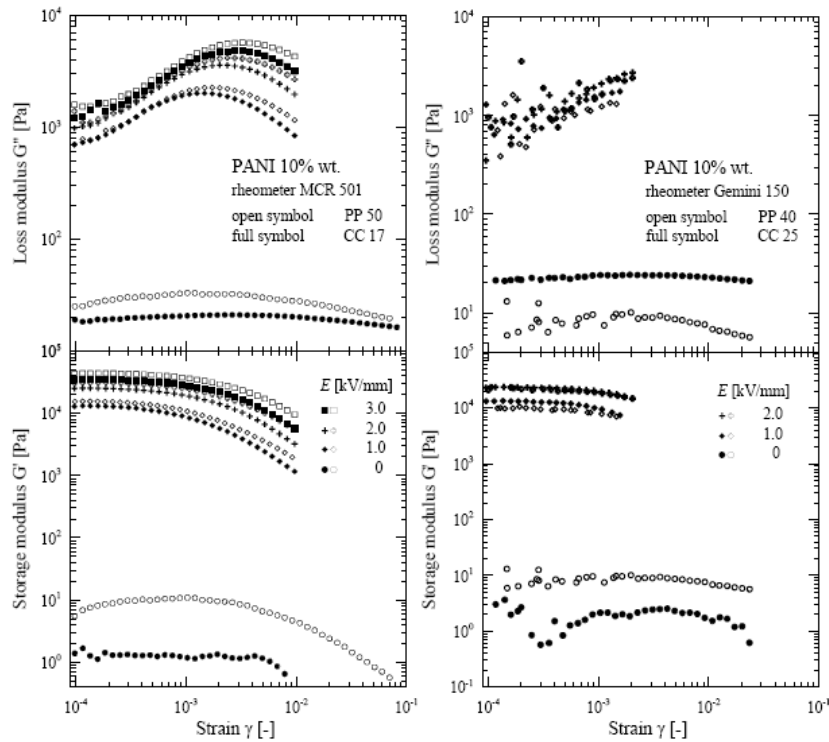


Figure 8. Storage and loss moduli in dependence on strain, different geometrical arrangements, same rheometer.

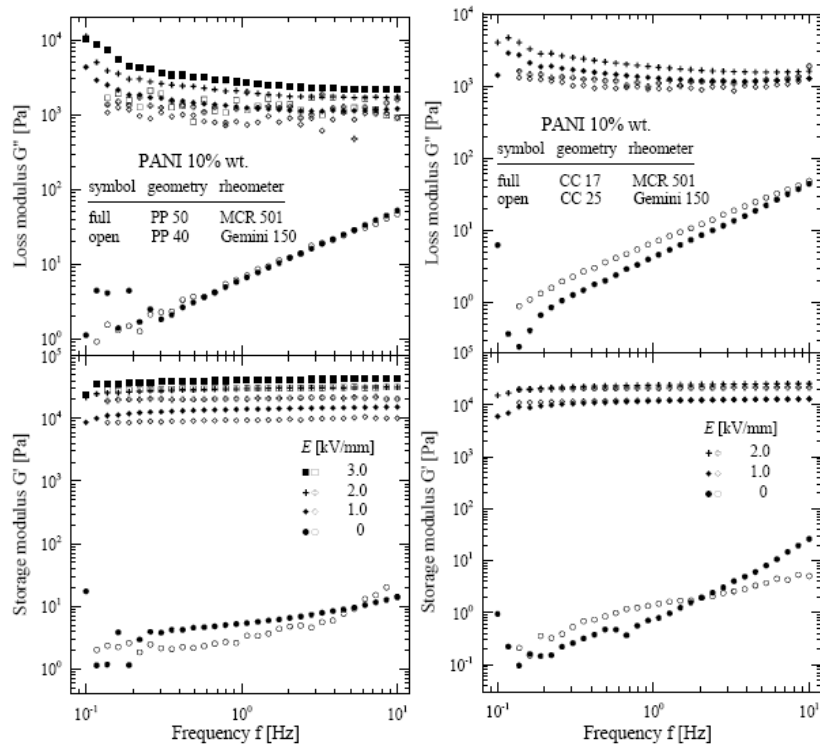


Figure 9. Storage and loss moduli in dependence on frequency, same geometrical arrangement (PP or CC), different rheometers.

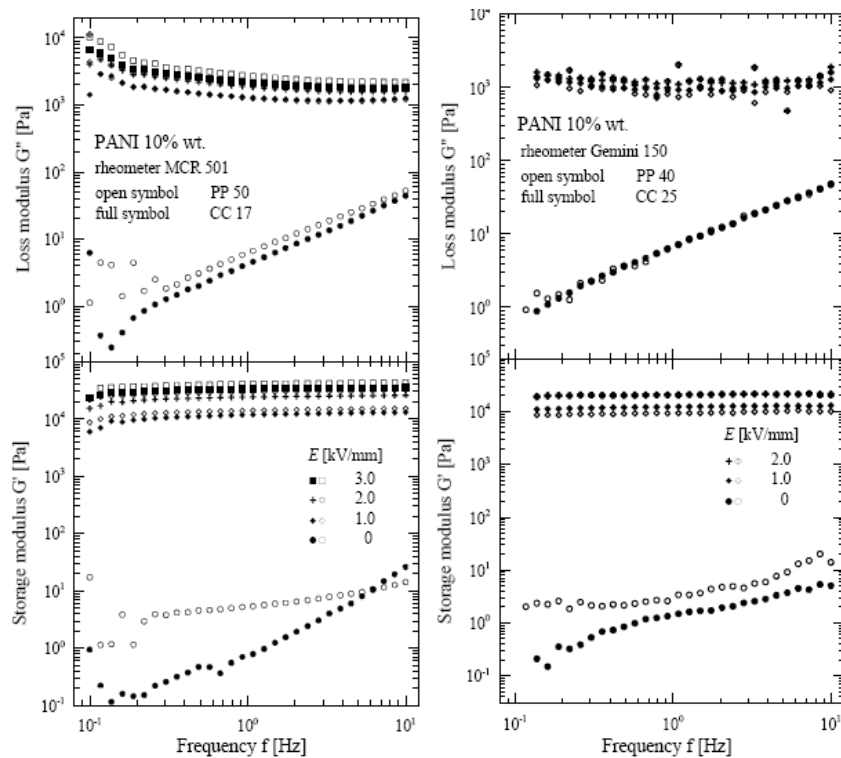


Figure 10. Storage and loss moduli in dependence on frequency, different geometrical arrangements, same rheometer.

4. Discussion and conclusions

The experimental data obtained for different geometrical arrangements and different rheometers exhibit non-negligible discrepancies. This fact is not surprising, cf. e.g. Rides *et al.* [1] who compared shear viscosity results obtained at high shear rates using extrusion rheometers (the same manufacturer) including an instrumented injection moulding machine.

There is a series of reasons causing this ambiguity: differences in construction of both rheometers (nevertheless in both cases all the parts in contact with the materials are made of stainless steel (1.4571 -MCR 501, 316 - CVOR 150)), anti-shock balancing in the laboratories (significant for lower values of independent variables), generation of an electric field, diameters of corresponding geometrical arrangements (e.g. 40 vs. 50 mm).

Based on figures 5-10 it is possible to come to the conclusions that

- the effect of completion of an electric circuit is different, but loosely speaking for mere air in both cases comparable e.g. with viscosity of water, however, when ER fluids are applied this value is not significant;
- the data from the Physica MCR 501 are smoother and behaviour of the corresponding curves is more continuous (at this point an age difference of the individual devices is also projected); in the case of the Gemini 150 the experimental data are closer for both geometries used;
- in the Physica MCR 501 usage of parallel plate geometry provides - on average - higher values in comparison with concentric cylinder geometry;
- when using parallel plate geometry, the data obtained by the Physica MCR 501 provide higher values (shear viscosity, storage and loss moduli vs. strain or frequency) than those obtained by the Gemini 150;
- when using concentric cylinder geometry, the data obtained by the Physica MCR 501 and the Gemini 150 are comparable in spite of the difference in corresponding gaps.

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References

- [1] Rides M, Kelly A L and Allen C R G 2011 *Polym. Testing* **30** 916