# The Application of Process Tomography for the In-line Measurement of Rheology

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## ABSTRACT

Tomographic measurements uncover information about the nature and distribution of components within a sensing domain from the acquisition of signals obtained from sensors located on the periphery of the subject i.e. a pipe, or vessel[1]. This study utilises a multi-scale Electrical Resistance Tomography (ERT) technique to extract a real-time, in-line velocity profile and ultimately, when coupled with the measurement of differential pressure, elucidate rheology. Not only may this be utilised as a fast and accurate process control and optimisation tool, but also facilitate enhanced product quality[2].

To experimentally validate this methodology, varying concentrations of the fluids Glycerol, Xanthan Gum and Carbopol 940 have been utilised. Such a technique has demonstrated the effective, in-line measurement of both viscosity and rheology with an accuracy of approximately 98% and a 95% confidence interval (CI) of ±1%.

## INTRODUCTION

An understanding of rheological properties is vital in the operation and optimisation of performance of unit operations. Typically, rheometry is conducted off-line with a sample taken and has been applied to processes with varying success; however, the development of a novel, in-line rheology measurement affords rapid and accurate process control and enhanced product quality[3]. Hitherto, numerous on-line rheometers have been developed, but all of which are based upon intrusive techniques and are unable to handle commercial non-Newtonian fluid systems[3].

Electrical Resistance Tomography (ERT) is an established technique which exploits the distribution of electrical impedance properties of a process domain[4]. Acquired signals are able to uncover information about the nature and distribution of components that are located within the sensing zone with tomography proven to be a powerful tool for the mapping of concentration distributions[5]. A typical ERT system involves the injection of a current or the application of a voltage to the sensing domain and the subsequent measurement of the returned voltage, or current, according to pre-defined measurement protocols[1]. Unlike optical methods, ERT possesses the capability to interrogate the behaviour of opaque systems.

Conventionally, ERT utilises the aforementioned impedance distribution to afford the qualification and quantification of phenomena such as mixing; however, the recent innovation of multi-scale sensors and use of cross-correlation algorithms has permitted ERT to systematically measure flow profiles within a pipe[5]. This velocity profiling may then be coupled with a differential pressure measurement to extract the rheological properties of a fluid.

This study outlines the effective use of the aforementioned technique in the real-time measurement of rheology from the laminar flow of model fluids within a pipe.

The fluids utilised were varying concentrations of:

- Glycerol (Newtonian Fluid)
- Xanthan Gum (Power Law Fluid)
- Carbopol 940, pH 4.4 (Herschel-Bulkley model was selected)

Not only does this measurement technique facilitate in-line and real-time measurement, the use of a velocity profile enables extremely low shear rates to be accessed and hence greater rheological information may be extracted. Moreover, with appropriate automation, conventional ERT parameters may be established such as: homogeneity and phase concentration.

Additional ERT velocimetry techniques have been theorised which are suitable for application within vessels and provide good correlation with viscosity under experimentation. Further experimental validation and the development of more complex models are required to relate such measurements to rheology.

## EXPERIMENTAL METHODS, SETUP AND INSTRUMENTATION

### Materials

In order to assess the functionality of the sensor, it is required to be experimentally validated. To do so, the following materials utilised to produce solutions of varying concentrations:

- Glycerol, supplied by Darrant Chemicals
- Xanthan Gum *from Xanthomonas Campestris*, supplied by Sigma-Aldrich
- Carbopol 940, supplied by Sigma-Aldrich

All materials were prepared in batches of 15 L, with the desired volume of material and tap water (≈1.0 mS $cm^{-1}$) measured. This was then subsequently agitated using a 4-blade, 45° pitched blade turbine at 300 rpm to ensure homogeneity of the solution.

In order to produce the desired gel structure of Carbopol 940, its pH was adjusted to 4.4 with the use of 1.0 M Sodium Hydroxide, supplied by Sigma-Aldrich.

### Off-line Rotational Rheometry

The rheological properties of the aforementioned materials were analysed with the use of an AG R2 rotational rheometer, supplied by TA instruments. A 2° stainless steel cone and plate geometry of diameter 40 mm was applied. The sample was held at 25°C with a logarithmic shear rate ramp applied across a shear rate range of 1-200 $s^{-1}$, over a duration of 10 minutes. This is analogous to the shear rates observed at experimental flow rates within the flow loop. These results formed the basis from which the ERT in-line rheometer would be validated. The results for Glycerol and Xanthan Gum are represented in Tables 1 and 2, respectively.

Table 1. Glycerol Rheological Parameters

| Concentration/ wt% | Viscosity/ mPa.s |
|---|---|
| 25 | 6.9 |
| 50 | 9.9 |
| 75 | 22.6 |

Table 2. Xanthan Gum Rheological Parameters, where k is consistency index and n is power index

| Concentration/ wt% | k/ Pa.$s^n$ | n |
|---|---|---|
| 0.1 | 0.071 | 0.723 |
| 0.5 | 2.062 | 0.268 |
| 1 | 12.64 | 0.108 |

Hitherto, a single Carbopol 940 system, of 0.2 wt%, has been tested upon and employing the Herschel-Bulkley constitutive equation the rheological parameters are: a yield stress of 25.2 Pa;

consistency index of 6.7 Pa $s^n$ and power index of 0.42.

ERT In-line Rheometry Methodology

This technique is able to afford the non-invasive characterisation of rheology within a process situation. To do so, a multi-scale sensing technique was utilised alongside the cross-correlation algorithm. This permits the tagging of fluid motion from one plane to another generating a delay signal and ultimately its velocity[6]. Multi-scale sensors have been employed to specifically target sensitivity to regions of interest and encompass both near-wall and central velocity measurements. The measurement of velocity enables the shear rate at each radial position to be yielded. The acquired tomogram may then be segmented into layers with a dual-scale sensor affording the development of a velocity profile which possesses 18 radial measurements. A dual-scale sensor is demonstrated in Figure 1.

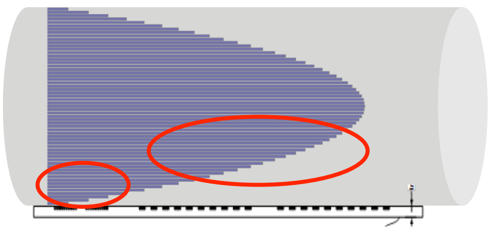

Figure 1. Dual-scale sensor.

The extracted velocity profile may then be coupled with the measurement of differential pressure which may be used to extract the shear stress profile, via Eq 1. This equation is obtained from a force balance upon a cylindrical element under steady state, incompressible, laminar flow[7].

$$\tau(r) = \frac{\Delta P}{2L} r \quad [1]$$

where $\tau$ is the shear stress, $\Delta P$ is the differential pressure measurement, L is the length over which the differential pressure is measured and r is radius.

The computation of rheology may be conducted utilising two methodologies. This study extracts shear viscosities and rheological model parameters using a non-linear parametric fitting to the theoretical laminar velocity profile of specific constitutive equations, Method 1. Such examples of this include Newtonian, power law and Bingham fluid models. In order to overcome an ill-conditioned fitting problem, a Gauss-Newton algorithm was additionally employed. It is envisioned that this technique will operate in either a quality or process control tool role.

A secondary technique, Method 2, utilises the differentiation of a $4^{th}$ order polynomial fitted to the velocity profile and can be utilised to extract the rheology of a fluid without prior knowledge of its behaviour. Consequently, this can be used with a fluid of unknown rheological parameters to act as an investigative tool.

In the presence of a yield stress, both systems may be used; however, the radial position at which differential of the fitted velocity profile is equal to zero is considered to be the plug flow region. This may then subsequently be related to yield stress via Eq 2.

$$\tau_y = \frac{\Delta P}{2L} R_0 \quad [2]$$

where $\tau_y$ is the yield stress, and $R_0$ is the plug flow radius.

All of the described analysis code and outputted graphs have been generated using the ITS v5r software and MATLAB.

Furthermore to the measurement of rheology, conventional tomography parameters may be extracted including homogeneity and phase concentration. The measurement of rheology, accompanied by ERT acting as a visualisation technique, possesses the capabilities to determine an understanding of shear-induced particle migration[8].

Experimental Equipment

To test such a sensor, a flow loop was built, with a simplified diagram outlined in Figure 2. This is fabricated from polypropylene pipe with an internal pipe diameter of 0.022 m. It consists of a reservoir, to load the test fluid, which is subsequently circulated using a flexible impeller pump, supplied by Liverani. During experimentation, the flow rate was varied within a range of 5 - 11.2 L $min^{-1}$; this allows the demonstration that the methodology is uninfluenced by changes in flow rate. This range was selected as to ensure that hydrodynamic entrance effects may be neglected, hence the velocity profile is fully developed, with flow within the laminar regime.

The ERT multi-scale sensor is coupled with a differential pressure sensor, supplied by Endress+Hauser, and operated using the ITS v5r system and software at a frame rate of 315 frames per second. Such a system is located next to a tracer generation methodology to provide a conductivity contrast. Once the fluid is being circulated, the rheology measurement system is able to operate at a temporal resolution of one measurement per 30 seconds; this is expected to be further improved via automation. When compared to a conventional off-line rheometer, which requires appropriate sampling and is extremely time consuming, this affords a vast competitive edge. The flow rate, temperature and conductivity are also monitored. A large number of experiments, across numerous days, were conducted in order to demonstrate the repeatability of this sensing technique.

## RESULTS AND DISCUSSION

Glycerol

In this study, ERT in-line rheometry has been effectively employed in the real-time measurement of viscosity of 75 wt% Glycerol. A Glycerol solution may be described as Newtonian.

An example of the obtained results, both velocity profile and resultant viscosity measurements are displayed in Figure 3. It can be seen that the measured velocity profile represents the traditional parabolic nature expected for a Newtonian fluid within the laminar flow regime.

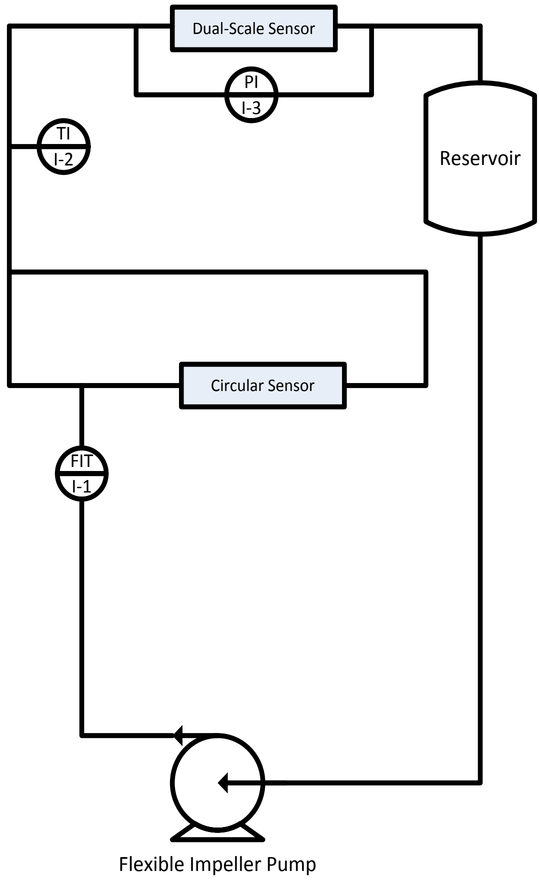


Figure 2. Simplified diagram of flow loop

Such results demonstrate that across over 4,500 combinatorial data points the obtained viscosity possesses an average accuracy of 98% and a maximum variability of only ±5%. The predominant deviations may additionally be attributed to variations in the flow rate outputted by the pump with the temporal resolution of the flow meter greater than that of the differential pressure gauge. Moreover, the level of fitting to the

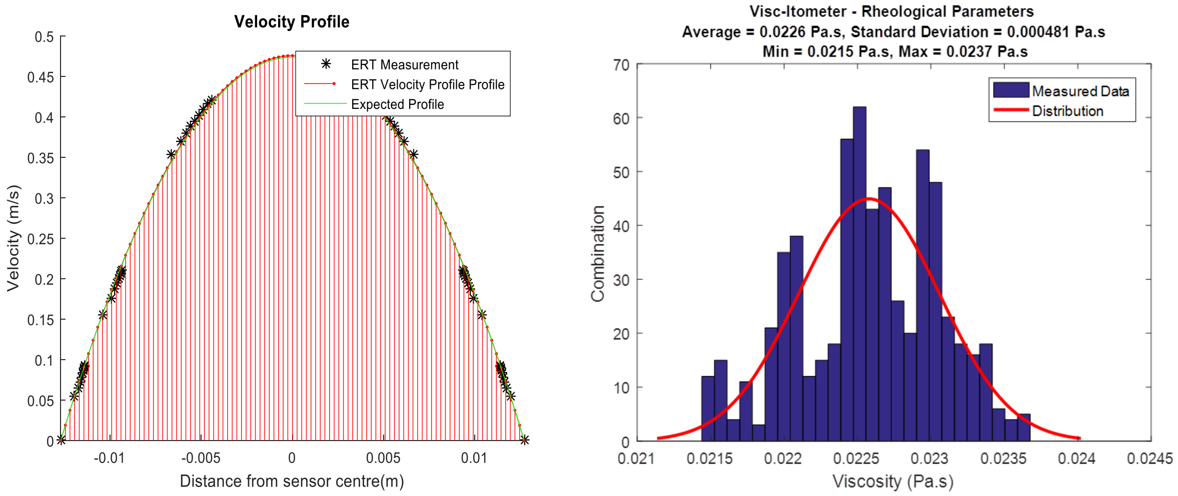


Figure 3. (left) Acquired velocity profile, (right) histogram of resultant viscosity data from a single experimental set.

Newtonian velocity profile is seen to be minimal with a 95% CI of $\pm 2.25 \times 10^{-4}$ Pa s (≈1%) and an average correlation coefficient, $R^2$, equal to 0.991. This fitting behaviour may be further enforced by Figure 4.

Figure 4 displays the expected and obtained rheograms from the two analysis methods outlined in the ERT in-line methodology section. When employing the Method 2 analysis technique, represented in the Figure by 'Polynomial Fitting', it assumes no prior knowledge of the rheological behaviour and yet is able to map the expected rheological behaviour.

Consequently, the ERT methodology can be considered to be an extremely powerful tool in the real-time, in-line measurement of viscosity as the output can be said to be analogous that of a conventional rotational rheometer.

Xanthan Gum

Despite being accurately able to extract viscosity, numerous on-line viscometers exist within the market, making the ability to elucidate rheology vital. To interrogate this, the rheological properties of three concentrations of Xanthan Gum solutions, 0.1, 0.5 and 1.0 wt%, were monitored.

From experimentation using the latter concentration, an example of the acquired velocity profile is displayed in Figure 5 and is evidently distinguishable from that obtained for Glycerol.

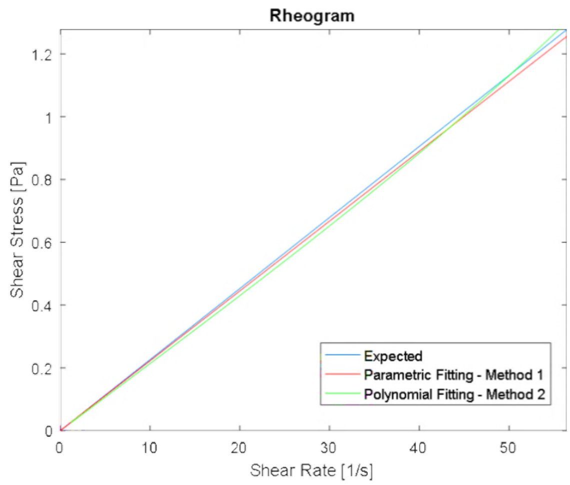


Figure 4. An example rheogram.

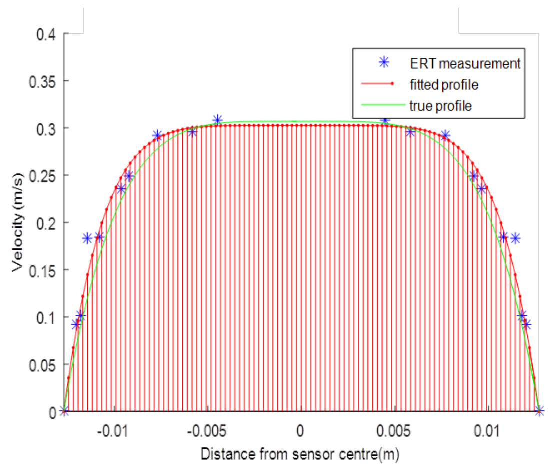


Figure 5. Obtained velocity profile for 1.0 wt% Xanthan Gum.

Similarly to Glycerol, an extensive number of datasets were taken for each of the aforementioned Xanthan gum concentrations. Due to an ill-conditioned problem arising within the parametric fitting, poor stability in rheological parameters was initially problematic with large deviations observed. However, the inclusion of a variation upon the Gauss-Newton algorithm into the rheology calculation has ensured that a maximum variation of ±5% of each parameter is observed, akin to that observed during the aforementioned Glycerol experiments.

The effectiveness of this algorithm is highlighted in Table 3; this data outlines the measurement of rheological parameters at varying flow rates across different days. The consistency index, k, and power index, n, display a maximum change of only 1%; this is despite an increase in flow rate of 53%. Furthermore, the correlation coefficients, when fitting to the specific constitutive equation, and accuracies are analogous to those observed when interrogating Glycerol.

Table 3. Difference in power law parametric fitting with flow rate.

| | Flow rate/ L $min^{-1}$ | | |
|---|---|---|---|
| Parameter | 5.3 | 8.1 | % Change |
| k/ Pa $s^n$ | 13.241 | 13.322 | 0.61 |
| n | 0.0926 | 0.0937 | 1.07 |

To affirm that the repeatability of the instrument is as a consequence of appropriate performance and not a lack of sensitivity, all of the aforementioned Xanthan Gum concentrations were examined. The differing rheological behaviour of each solution can be represented in a rheogram, or flow curve, and a viscosity profile. Figure 6 displays such graphs obtained from the ERT rheometry technique and demonstrates that the rheological behaviour of each concentration is detected. This performance is addtionally observed to be repeatable across over 3,000 combinatorial experiments for each concentration.

As a result of this analysis, the ERT methodology may not only be termed an in-line viscometer but moreover a rheometer.

Carbopol 940

In the rheology measurement of Carbopol 940, its pH was amended to 4.4 using 1.0 M Sodium Hydroxide. This facilitates the formation a gel structure and hence a yield stress.

Using the technique described within the experimental methods section, a yield stress has been able to be detected with good agreement to that which is obtained from an off-line rotational rheometer. The average obtained yield stress from ERT measurements is 23.7 Pa, which is comparable to the yield stress of 25.2 Pa of the AR G2 rheometer. This equates to a dissimilarity of just 6.2%.

## SENSOR OPPORTUNITIES

### ERT In-line Rheometer

It is evident that ERT in-line rheometry is an extremely powerful tool which is capable of enhancing process operability and efficiency; it additionally presents opportunities in product quality improvements.

Moreover, elucidating rheology from a velocity profile enables access to lower shear rates than conventional rheometry; typically the low shear rate range is the area of most interest in the study of complex fluids. The measurement of rheology at such shear rates may provide an advancement in the understanding of complex rheological behaviour.

A similar opportunity arises when combining rheological measurements with conventional tomography outputs, namely radial concentration profiles, to extend the

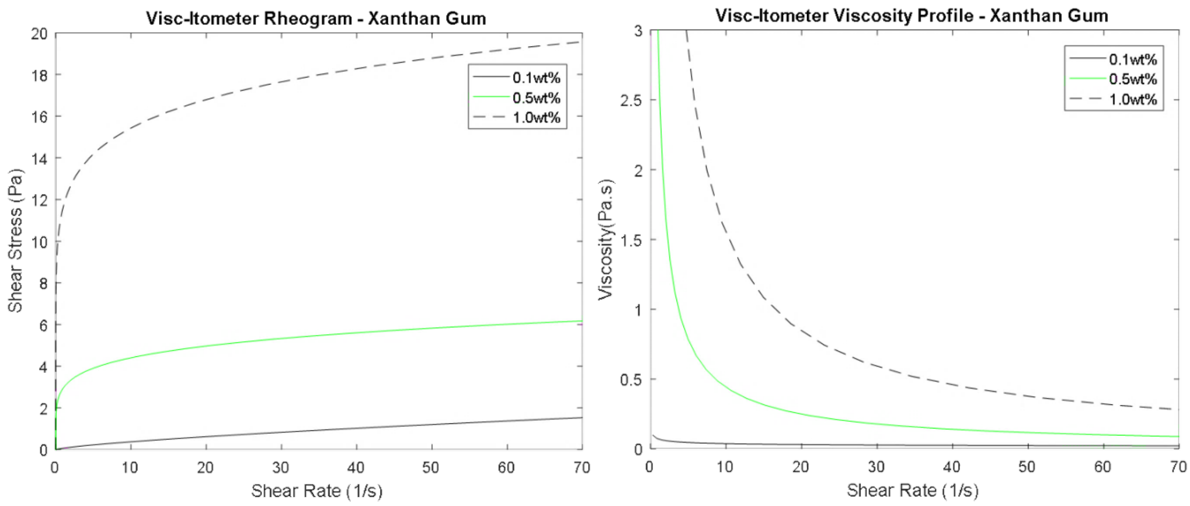

Figure 6. Xanthan Gum Rheology Analysis (left) rheogram, (right) viscosity profile

knowledge of the complex phenomenon of shear-induced migration of suspensions[8].

Additional applications for this sensor include the monitoring of liquid displacement. Through observation of the velocity profile, differences in the mixing zone can be elucidated when changing from one fluid to another; this is typically when an operation mode is altered i.e. start-up, shutdown.

Vessel-based Rheology

The application of multi-scale velocimeters, with the use of differing configurations, may be extended to use within vessels. Within a vessel, certain flow fields are able to be ascertained, whether it be within a storage tank or agitated mixing vessels. Such flow fields may subsequently correlated to the viscosity of the fluid if other physical properties are known.

Inside a stagnant fluid, a small bubble may be generated with its motion monitored using ERT velocimetry. Due to this setup, relatively simple flow fields arise and with the development of more complex models to solve equations of motion for specific constitutive equations, rheology may potentially be able to be elucidated. It must be noted that technqiue has only been conceptualised with simple feasibility studies conducted. The measurement of rheology in a vessel would generate an extremely potent control and optmisation tool for mixing. It may additionally be utilised in the quality control of incoming raw materials and other stored process fluids.

## CONCLUSION

In summary, this paper outlines a novel methodology for the in-line measurement of rheology using multi-scale Electrical Resistance Tomography coupled with a differential pressure measurement. This permits the rapid, accurate measurement of rheology to act as a process control, optimisation and quality control tool. In order to experimentally validate this technique, three material solutions were chosen: Glycerol, Xanthan Gum and Carbopol. All of which possess varying rheological properties with the acquired parameters and flow curves in extremely good agreement, 98% accuracy, with conventional rotational rheometry techniques. Figure 7, represents the ERT rheogram, for the aforementioned fluids to demonstrate the ability to detect varying rheological behaviours.

Moreover, the ERT system has been deemed to be reliable with a 95% CI of ±1% for Glycerol.

The subsequent phase of the project will entail the study of industrial suspensions upon pilot-scale to ensure that scale-up of the system is feasible. However, major problems are not foreseen as classically ERT is known as an extremely scalable technology. Further sensor and analysis algorithm development will additionally be conducted to enhance the sensors capabilities further.

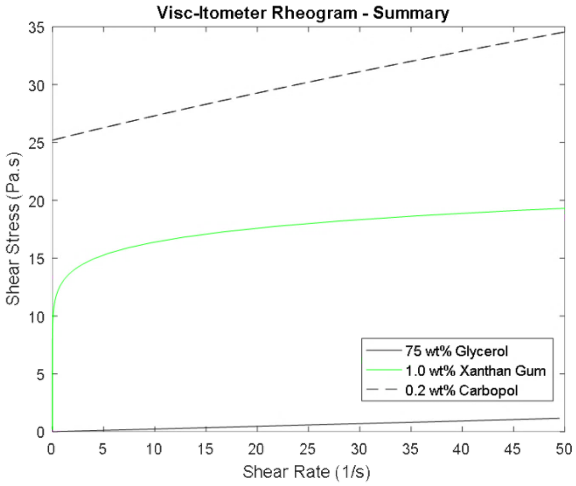


Figure 7. A rheogram summarising the obtained experimental data from this study

Such algorithms will be incorporated to account for more complex rheological models to account for phenomena such as wall-slip. This concept utilises multi-scale tomography sensors to act as a velocimeter whose application may be then extended to vessels and mixing, with correlations between viscosity, and potentially rheology, able to be drawn.

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