Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.


# Impact of residence time distributions in reacting magnesium packed-beds on Grignard reagent formation – Pump-induced flow behaviour in non-reacting magnesium beds (part 1)

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## Electronic Supplementary Information (ESI)

## Content

## 1 Performing residence time distribution experiments

Exemplary cumulative distribution functions obtained from the normalized measurement data as well as by fitting the dispersion model to the measurement data are presented. The cumulative distribution functions shown represent the results being close to the mean Bodenstein number Bo mentioned or displayed within the publication, unless otherwise specified. The dimensionless residence time distribution functions presented are obtained from the model cumulative distribution functions.

Specific Bodenstein number and hydrodynamic residence time τ are noted.

Pumps used for residence time distribution experiments corresponding to the presented distribution functions are listed in table 1.

Table 1: Details of pumps used during residence time measurements.

| **pump** | **pump type** | **pump name** | **manufacturer** |
|---|---|---|---|
| **A** | syringe pump | PN1610 syringe dosing system | Postnova Analytics GmbH |
| **B** | syringe pump | LA-120 | Landgraf Laborsysteme HLL GmbH |
| **C** | micro annular gear pump | mzr-7205 | HNP Mikrosysteme GmbH |
| **D** | valveless rotary piston pump | Reglo-CPF Digital with ISM321 drive and RH1CKC pump head | Ismatec, Cole-Parmer Instrument Company LTD |

# 2 Residence time measurements in beds of metal turnings

## 2.1 Coarse magnesium turnings

### 2.1.1 Pump A

**Bo = 83**

**τ = 584 s**

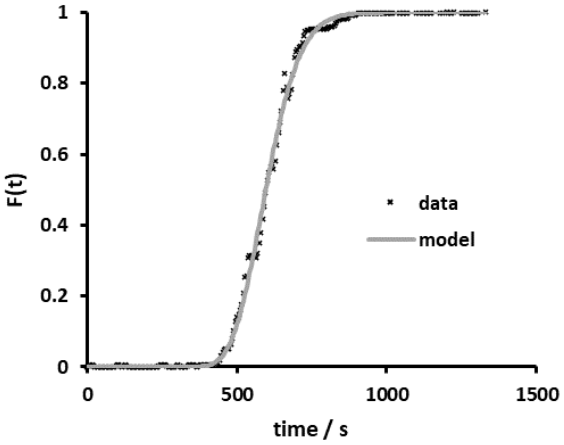


Figure 1: Cumulative distribution function, pump A, coarse magnesium turnings, displacement of tracer.

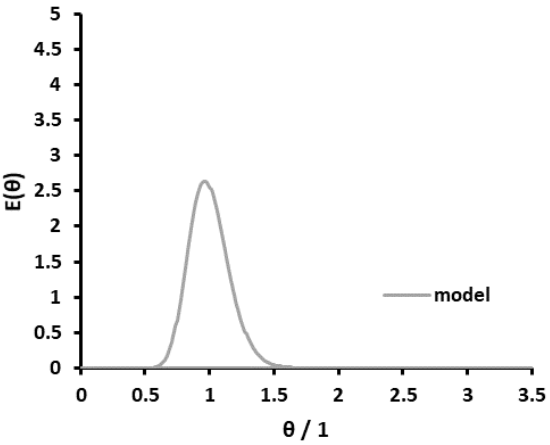


Figure 2: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, displacement of tracer.

**Bo = 68**

**τ = 602 s**

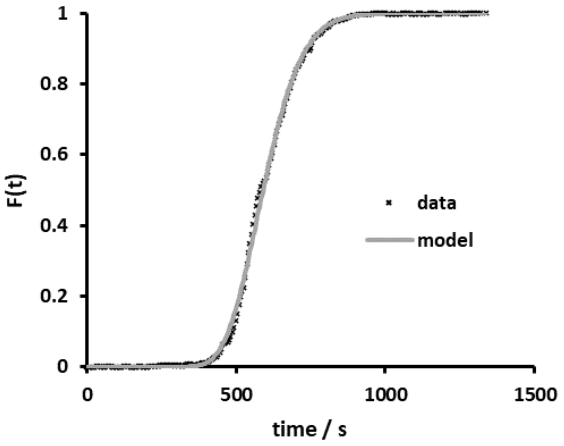


Figure 3: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer.

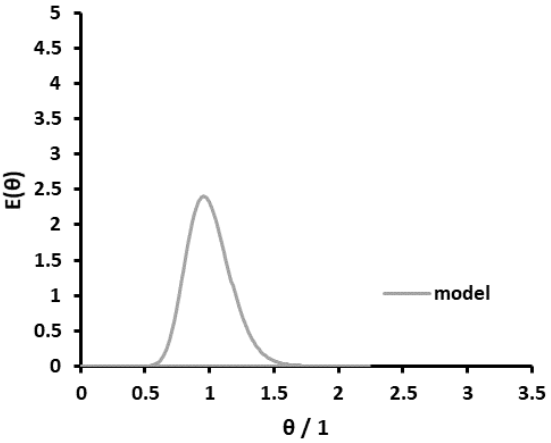


Figure 4: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer.

### 2.1.2 Pump C

**Bo = 83**

**τ = 615 s**

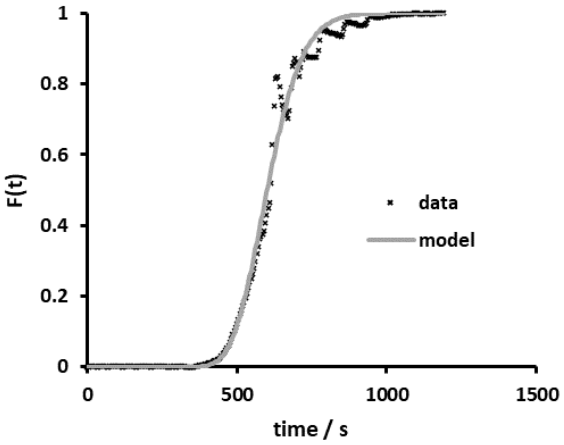


Figure 5: Cumulative distribution function, pump C, coarse magnesium turnings, displacement of tracer.

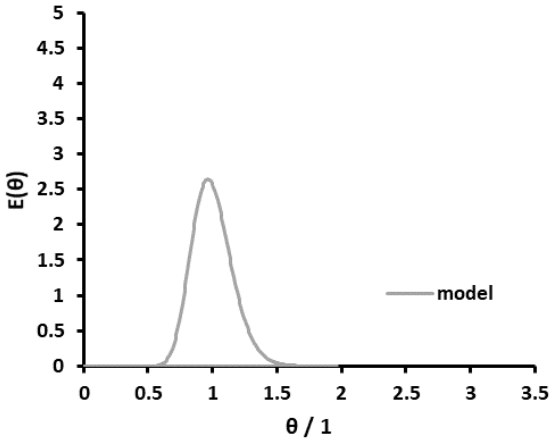


Figure 6: Dimensionless residence time distribution function, pump C, coarse magnesium turnings, displacement of tracer.

**Bo = 47**

**τ = 596 s**

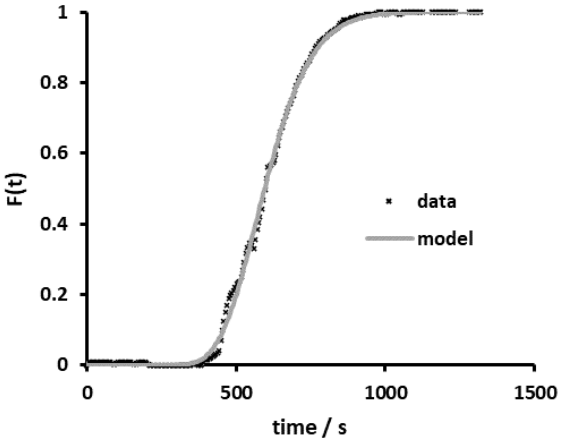


Figure 7: Cumulative distribution function, pump C, coarse magnesium turnings, introduction of tracer.

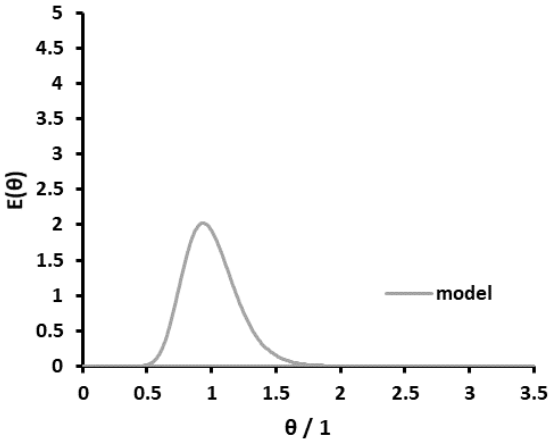


Figure 8: Dimensionless residence time distribution function, pump C, coarse magnesium turnings, introduction of tracer.

### 2.1.3 Pump D

**Bo = 93**

**τ = 689 s**

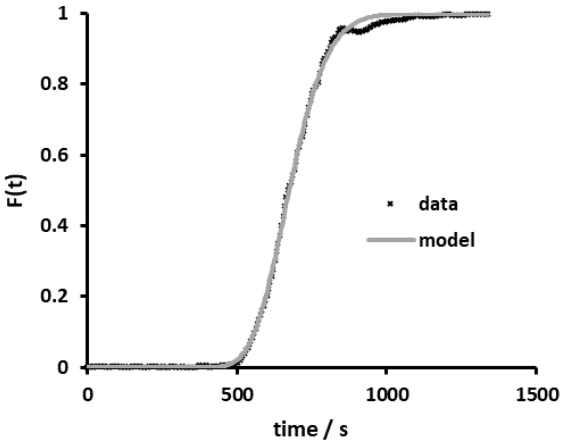


Figure 9: Cumulative distribution function, pump D, coarse magnesium turnings, displacement of tracer.

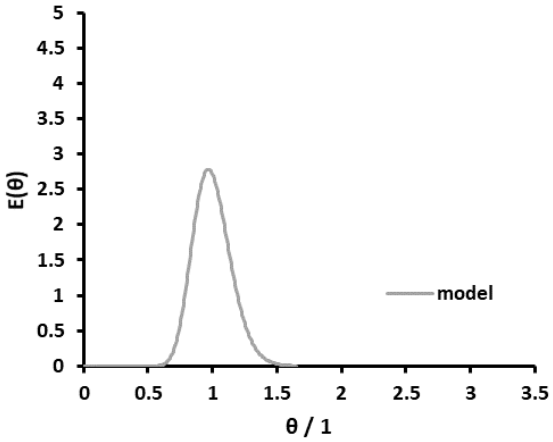


Figure 10: Dimensionless residence time distribution function, pump D, coarse magnesium turnings, displacement of tracer.

**Bo = 53**

**τ = 588 s**

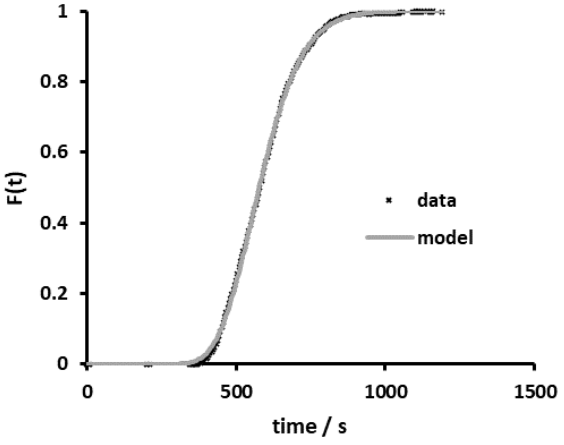


Figure 11: Cumulative distribution function, pump D, coarse magnesium turnings, introduction of tracer.

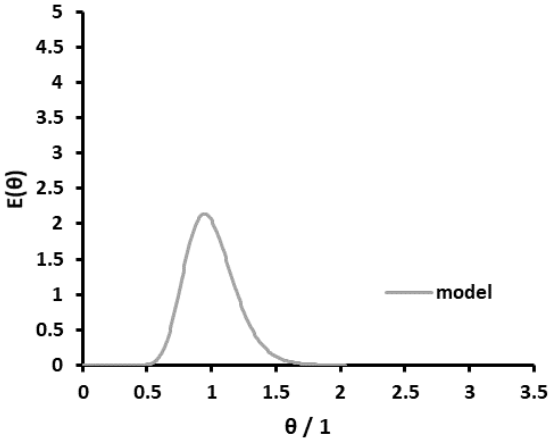


Figure 12: Dimensions less residence time distribution function, pump D, coarse magnesium turnings, introduction of tracer.

## 2.2 Fine magnesium turnings

### 2.2.1 Pump A

**Bo =141**

**τ = 522 s**

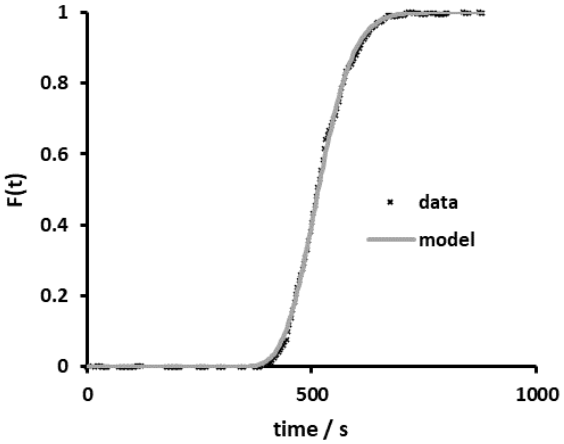


Figure 13: Cumulative distribution function, pump A, fine magnesium turnings, displacement of tracer.

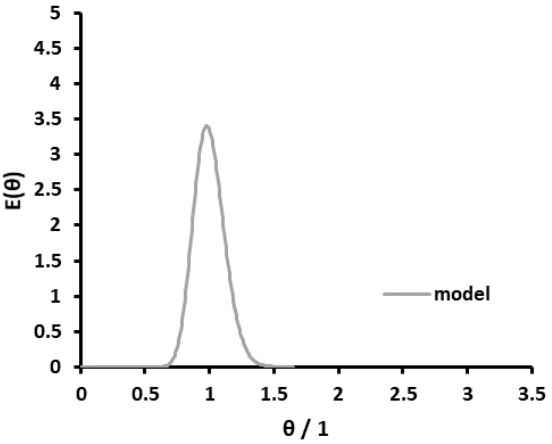


Figure 14: Dimensionless residence time distribution function, pump A, fine magnesium turnings, displacement of tracer.

**Bo = 169**

**τ = 539 s**

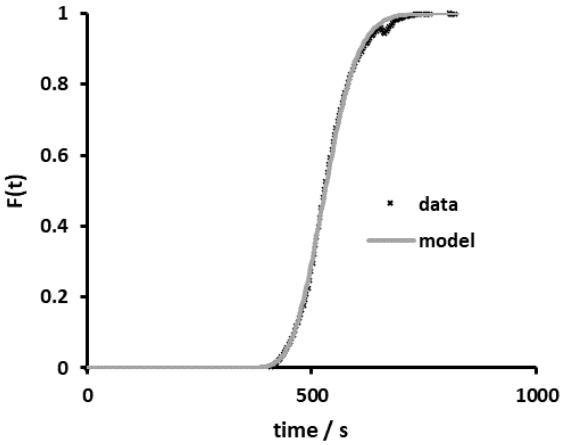


Figure 15: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer.

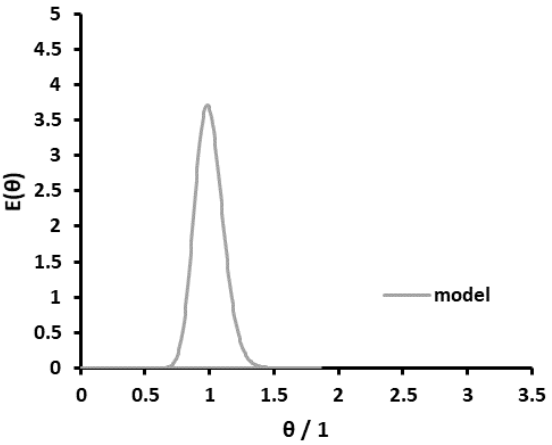


Figure 16: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer.

### 2.2.2 Pump C

**Bo = 196**

**τ = 544 s**

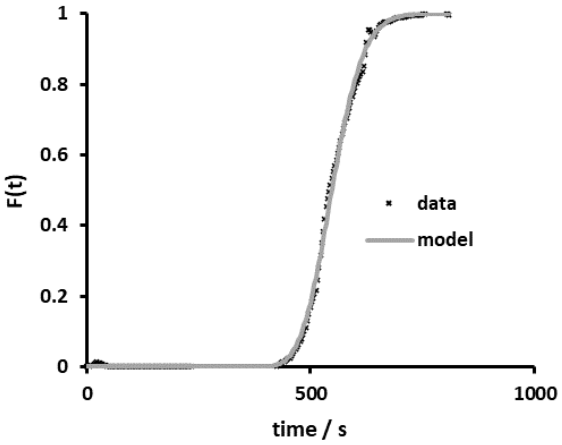


Figure 17: Cumulative distribution function, pump C, fine magnesium turnings, displacement of tracer.

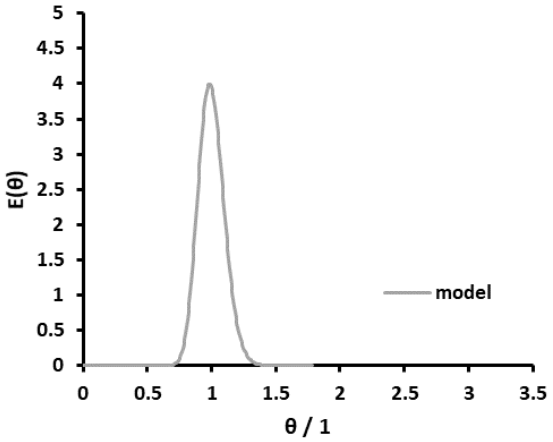


Figure 18: Dimensionless residence time distribution function, pump C, fine magnesium turnings, displacement of tracer.

**Bo = 195**

**τ = 508 s**

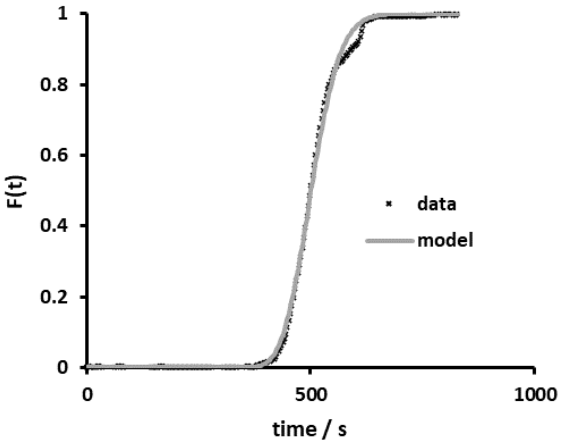


Figure 19: Cumulative distribution function, pump D, fine magnesium turnings, introduction of tracer.

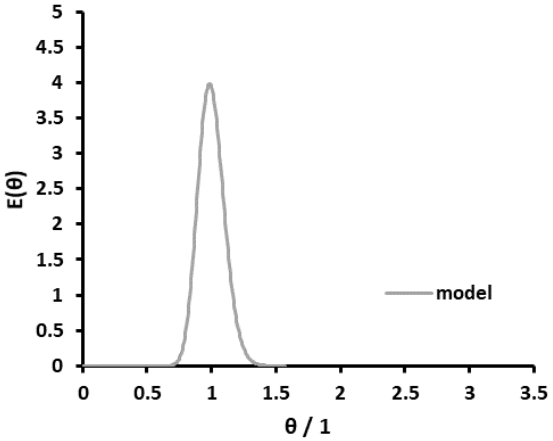


Figure 20: Dimensionless residence time distribution function, pump D, fine magnesium turnings, introduction of tracer.

### 2.2.3 Pump D

**Bo = 229**

**τ = 607 s**

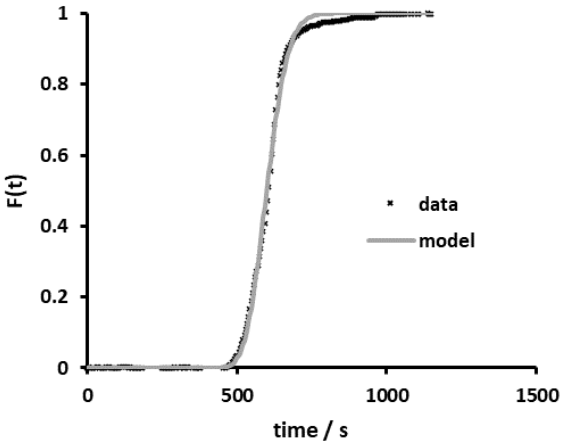


Figure 21: Cumulative distribution function, pump D, fine magnesium turnings, displacement of tracer.

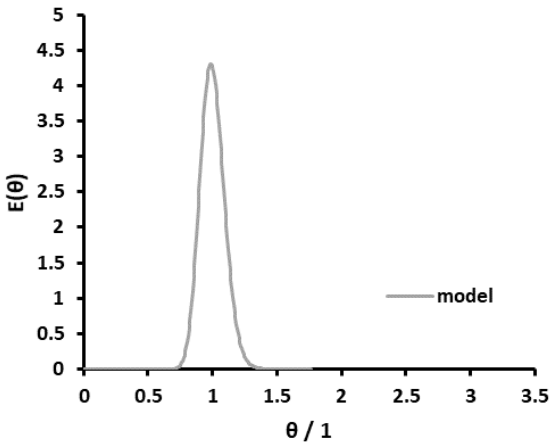


Figure 22: Dimensionless residence time distribution function, pump D, fine magnesium turnings, displacement of tracer.

**Bo = 179**

**τ = 491 s**

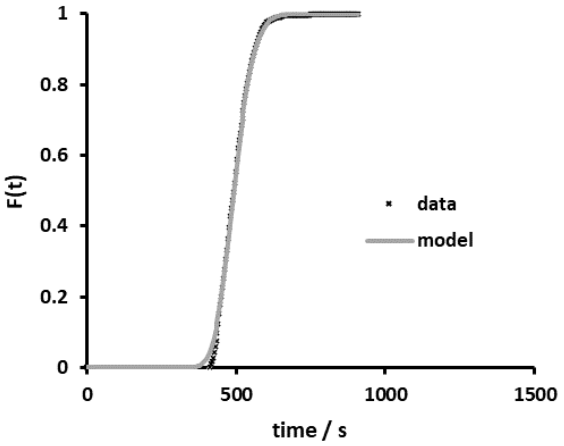


Figure 23: Cumulative distribution function, pump D, fine magnesium turnings, introduction of tracer.

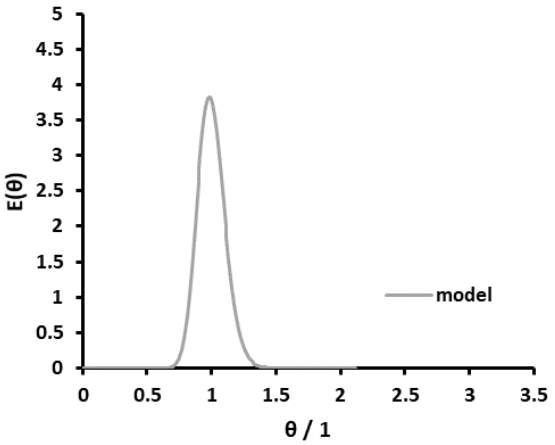


Figure 24: Dimensionless residence time distribution function, pump D, fine magnesium turnings, introduction of tracer.

## 2.3 Mixed magnesium turnings

### 2.3.1 Pump A

**Bo = 147**

**τ = 593 s**

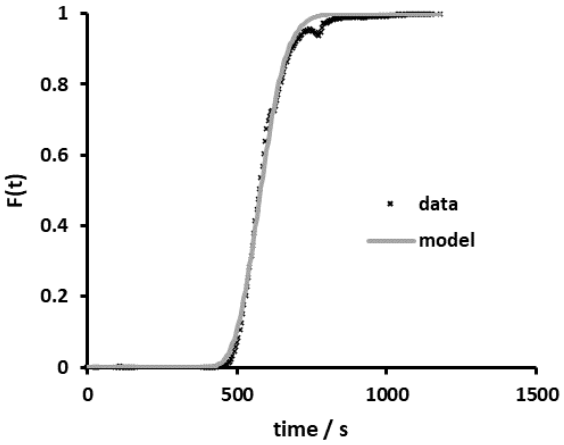


Figure 25: Cumulative distribution function, pump A, mixed magnesium turnings, displacement of tracer.

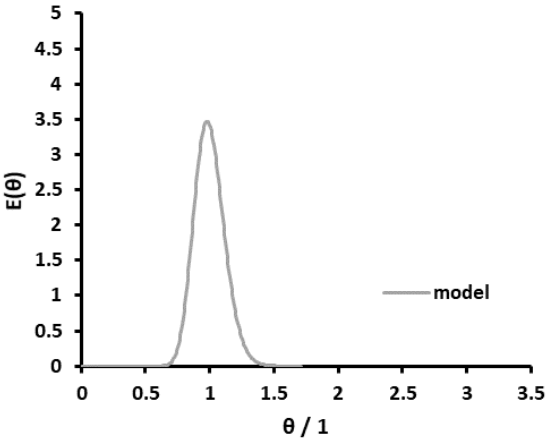


Figure 26: Dimensionless residence time distribution function, pump A, mixed magnesium turnings, displacement of tracer.

**Bo = 61**

**τ = 528 s**

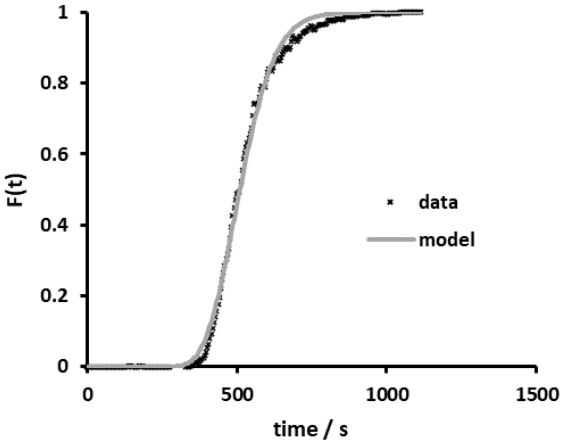


Figure 27: Cumulative distribution function, pump A, mixed magnesium turnings, introduction of tracer.

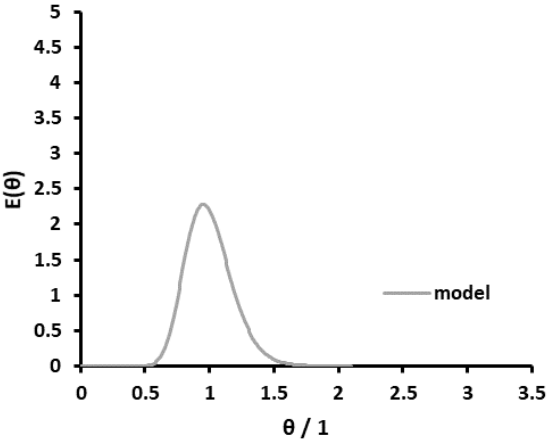


Figure 28: Dimensionless residence time distribution function, pump A, mixed magnesium turnings, introduction of tracer.

### 2.3.2 Pump D

**Bo = 121**

**τ = 624 s**

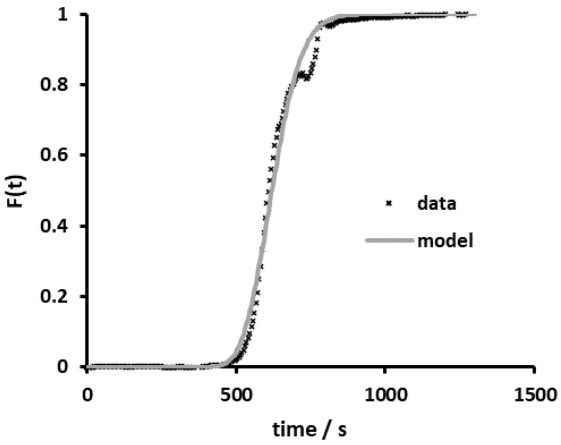


Figure 29: Cumulative distribution function, pump D, mixed magnesium turnings, displacement of tracer.

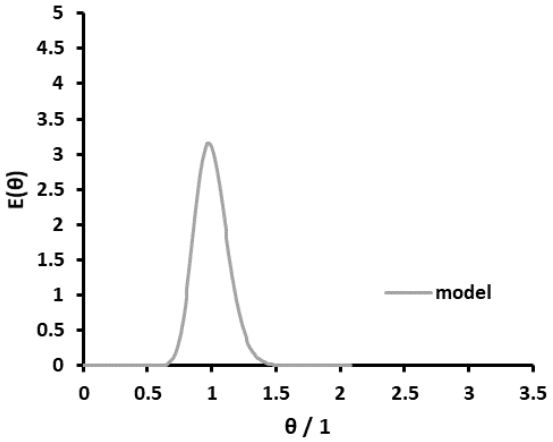


Figure 30: Dimensionless residence time distribution function, pump D, mixed magnesium turnings, displacement of tracer.

**Bo = 49**

**τ = 598 s**

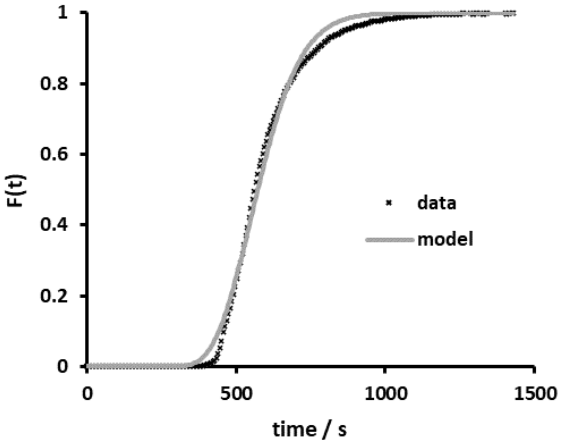


Figure 31: Cumulative distribution function, pump D, mixed magnesium turnings, introduction of tracer.

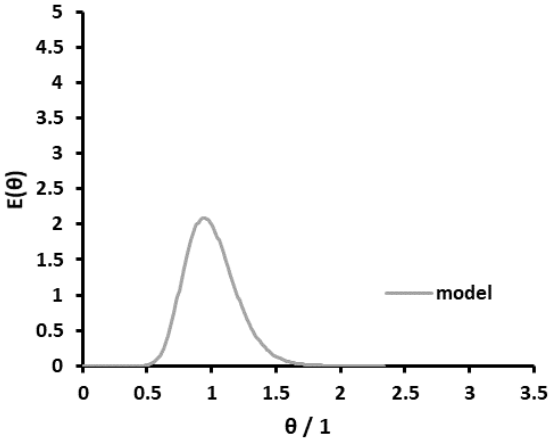


Figure 32: Dimensionless residence time distribution function, pump D, mixed magnesium turnings, introduction of tracer.

# 3 Influence of jogging motor

## 3.1 Coarse magnesium turnings

### 3.1.1 Jogging motor only for filling

**Bo = 26**

**τ = 1064 s**

$\dot{V} = 1$ **mL/min**

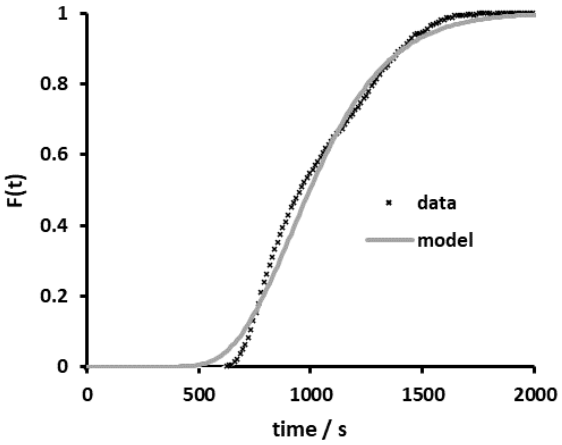


Figure 33: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =1 mL/min, jogging motor only for filling.

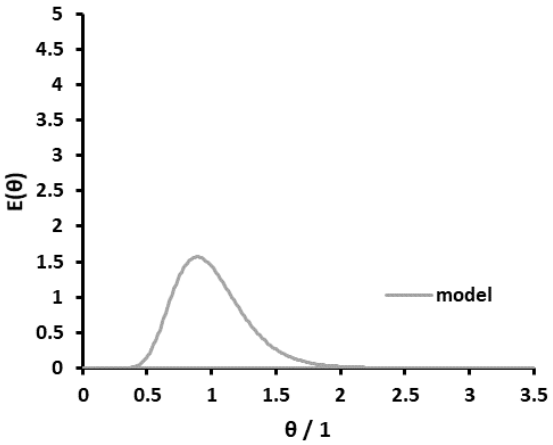


Figure 34: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 1 mL/min, jogging motor only for filling.

**Bo = 32**

**τ = 472 s**

$\dot{V} = 2$ **mL/min**

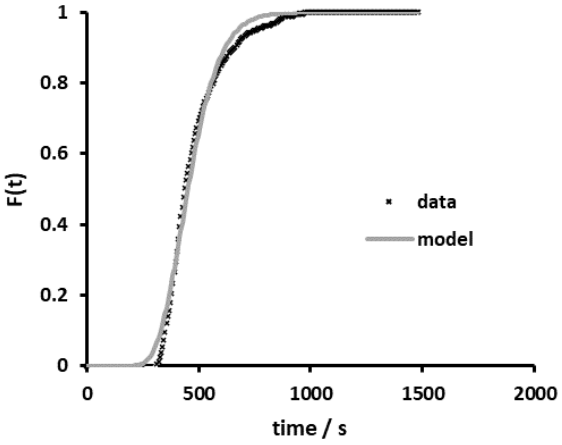


Figure 35: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =2 mL/min, jogging motor only for filling.

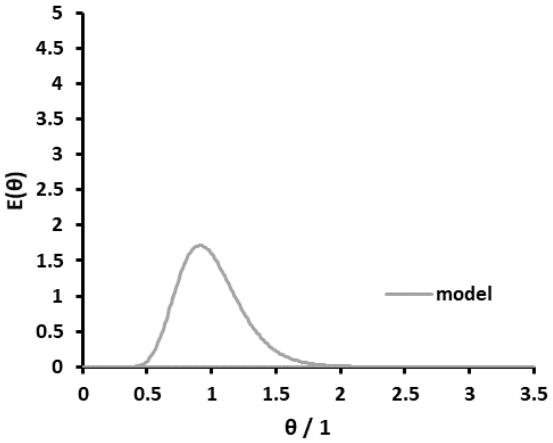


Figure 36: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 2 mL/min, jogging motor only for filling.

**Bo = 45**

**τ = 369 s**

$\dot{V} = 3$ **mL/min**

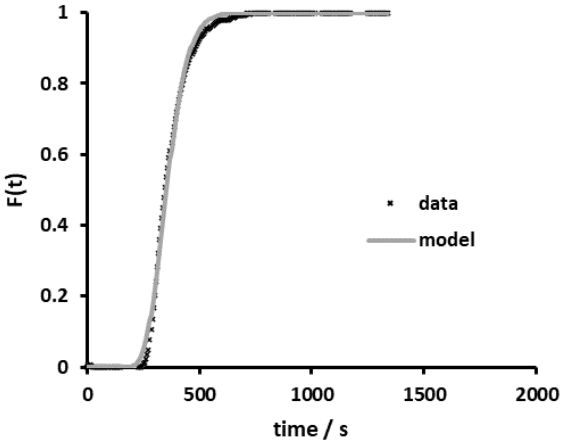


Figure 37: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =3 mL/min, jogging motor only for filling.

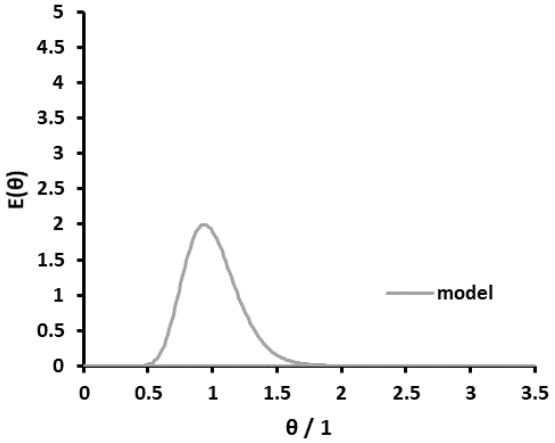


Figure 38: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 3 mL/min, jogging motor only for filling.

**Bo = 38**

**τ = 256 s**

$\dot{V} = 4$ **mL/min**

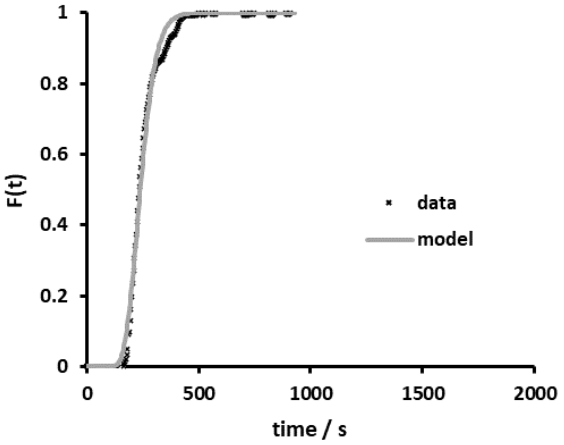


Figure 39: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =4 mL/min, jogging motor only for filling.

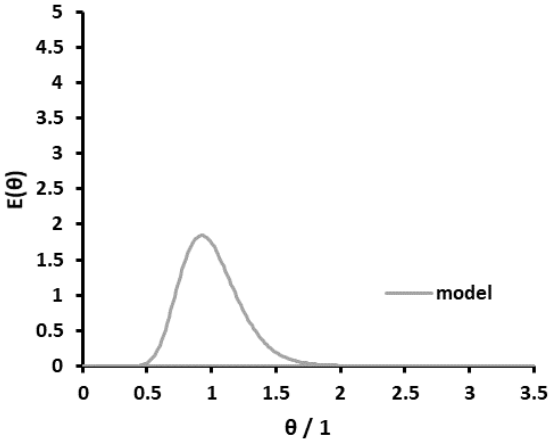


Figure 40: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 4 mL/min, jogging motor only for filling.

### 3.1.2 Jogging motor for filling and RTD measurement

**Bo = 55**

**τ = 1141 s**

$\dot{V} = 1$ **mL/min**

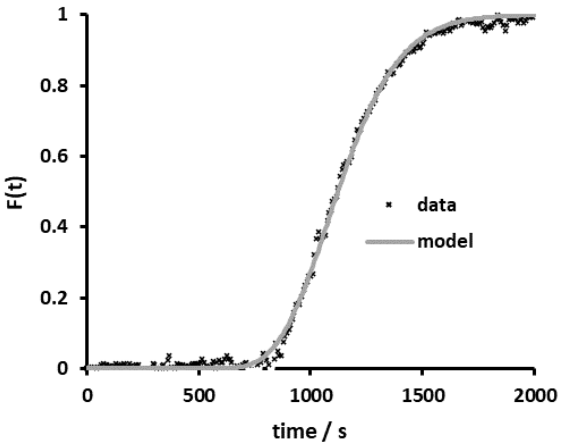


Figure 41: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =1 mL/min, jogging motor for filling and RTD measurement.

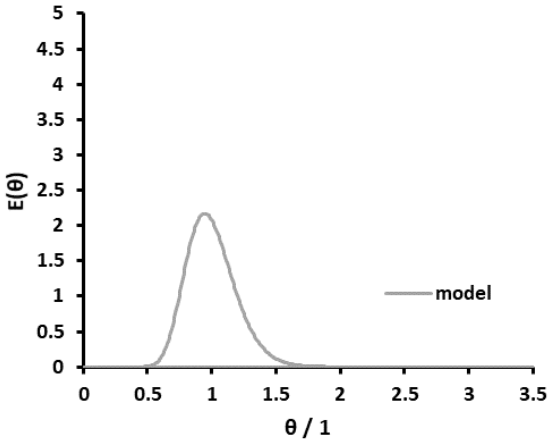


Figure 42: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 1 mL/min, jogging motor for filling and RTD measurement.

**Bo = 75**

**τ = 625 s**

$\dot{V} = 2$ **mL/min**

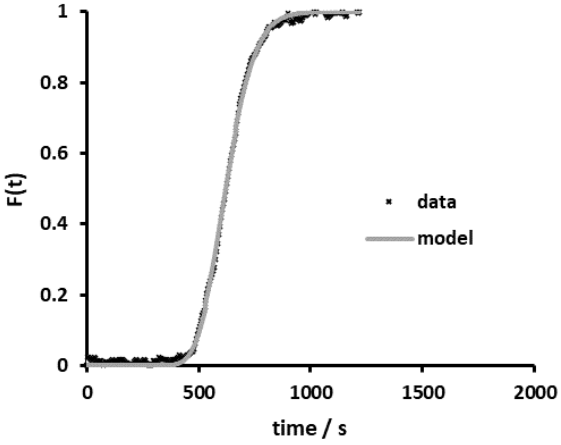


Figure 43: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =2 mL/min, jogging motor for filling and RTD measurement.

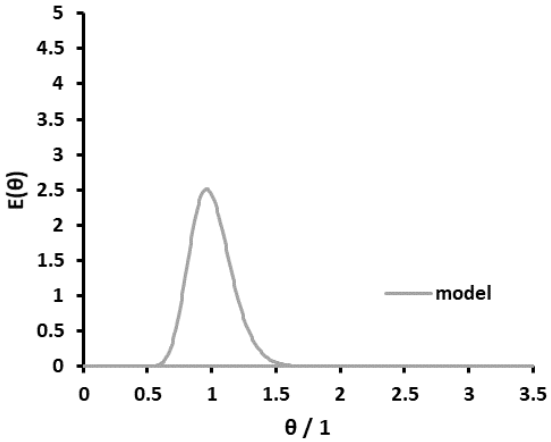


Figure 44: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 2 mL/min, jogging motor for filling and RTD measurement.

**Bo = 86**

**τ = 382 s**

**$\dot{V} = 3$ mL/min**

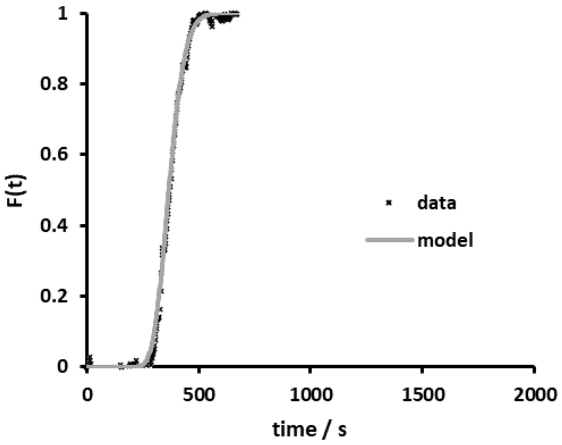


Figure 45: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =3 mL/min, jogging motor for filling and RTD measurement.

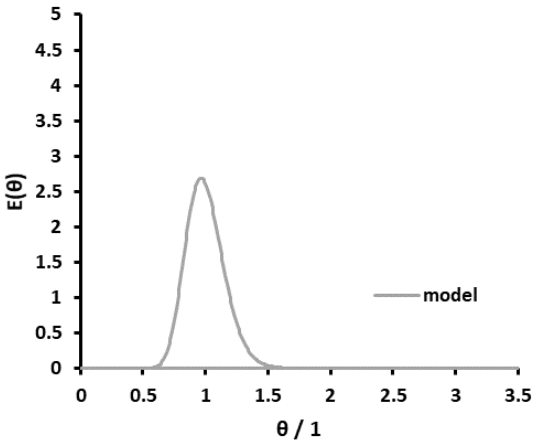


Figure 46: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 3 mL/min, jogging motor for filling and RTD measurement.

**Bo = 86**

**τ = 322 s**

**$\dot{V} = 4$ mL/min**

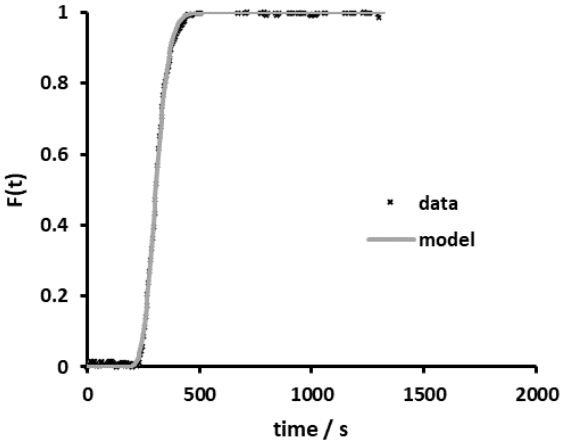


Figure 47: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =4 mL/min, jogging motor for filling and RTD measurement.

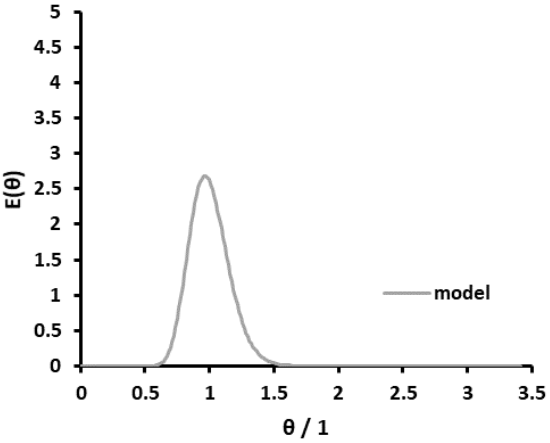


Figure 48: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 4 mL/min, jogging motor for filling and RTD measurement.

### 3.1.3 Jogging motor only for RTD measurement

**Bo = 30**

**τ = 1023 s**

**$\dot{V} = 1$ mL/min**

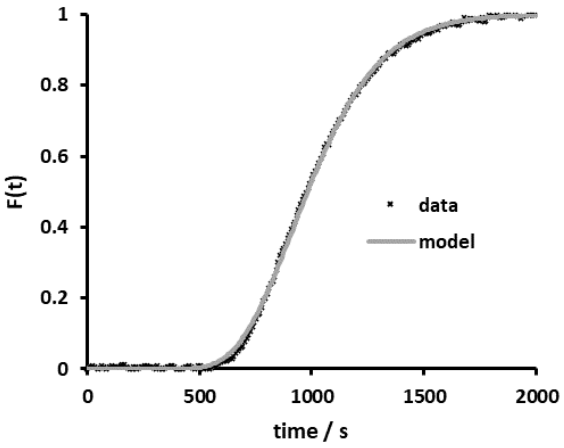


Figure 49: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =1 mL/min, jogging motor only for RTD measurement.

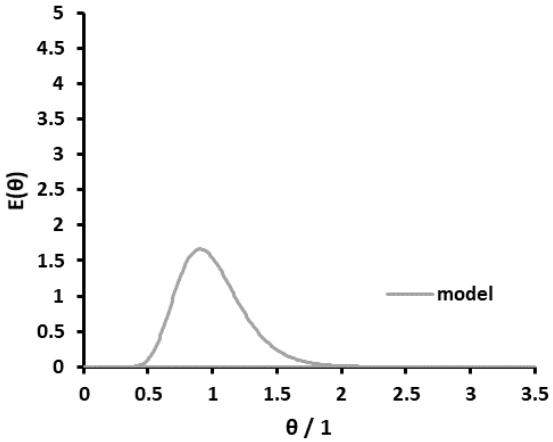


Figure 50: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 1 mL/min, jogging motor only for RTD measurement.

**Bo = 49**

**τ = 565 s**

**$\dot{V} = 2$ mL/min**

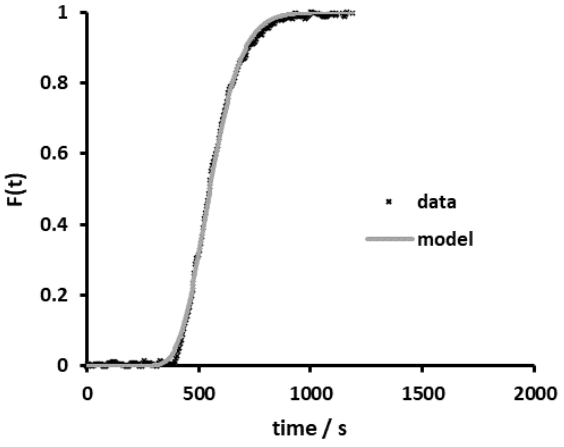


Figure 51: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =2 mL/min, jogging motor only for RTD measurement.

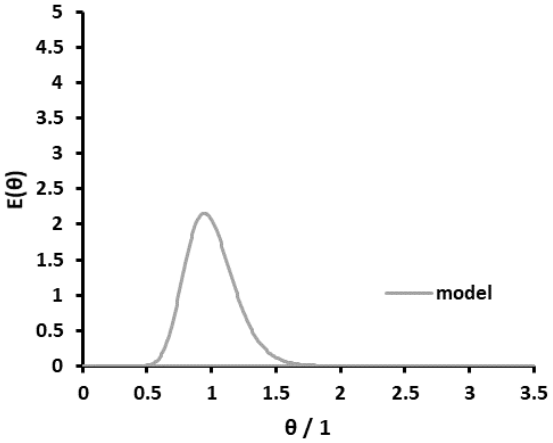


Figure 52: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 2 mL/min, jogging motor only for RTD measurement.

**Bo = 54**

**τ = 379 s**

$\dot{V} = 3$ **mL/min**

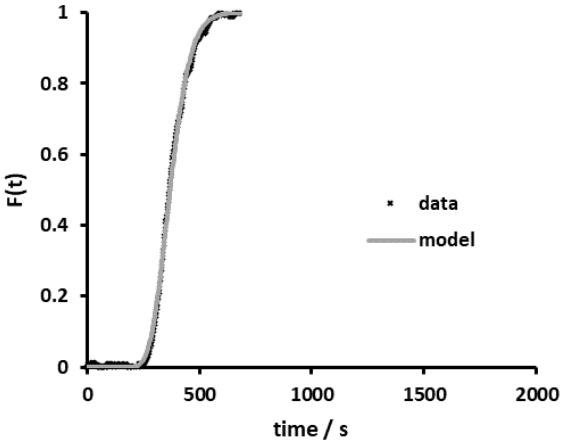


Figure 53: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =3 mL/min, jogging motor only for RTD measurement.

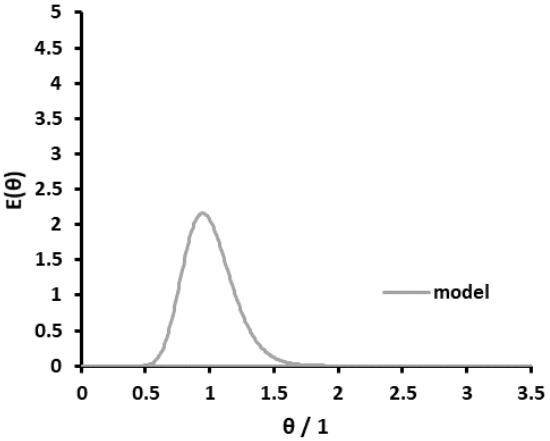


Figure 54: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 3 mL/min, jogging motor only for RTD measurement.

**Bo = 64**

**τ = 282 s**

$\dot{V} = 4$ **mL/min**

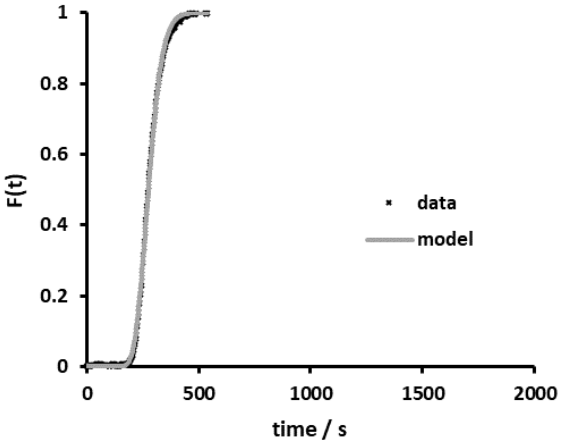


Figure 55: Cumulative distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate =4 mL/min, jogging motor only for RTD measurement.

**118**

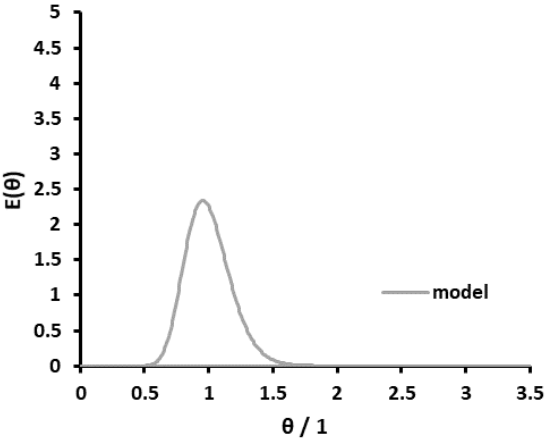


Figure 56: Dimensionless residence time distribution function, pump A, coarse magnesium turnings, introduction of tracer, flow rate = 4 mL/min, jogging motor only for RTD measurement.

## 3.2 Fine magnesium turnings

### 3.2.1 Jogging motor only for filling

**Bo = 119**

**τ = 1093 s**

**$\dot{V} = 1$ mL/min**

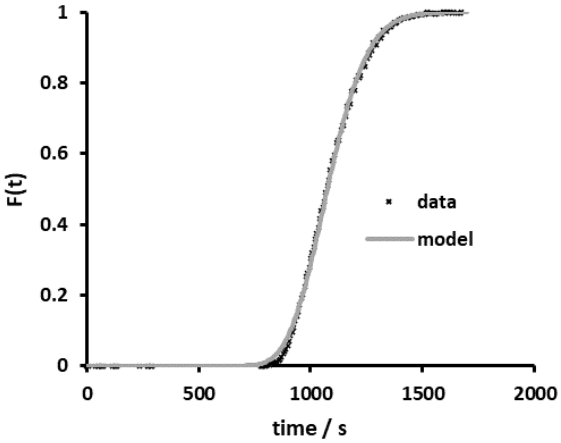


Figure 57: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =1 mL/min, jogging motor only for filling.

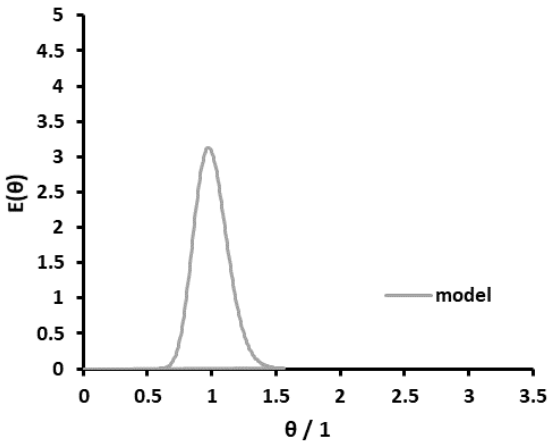


Figure 58: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 1 mL/min, jogging motor only for filling.

**Bo = 160**

**τ = 533 s**

**$\dot{V} = 2$ mL/min**

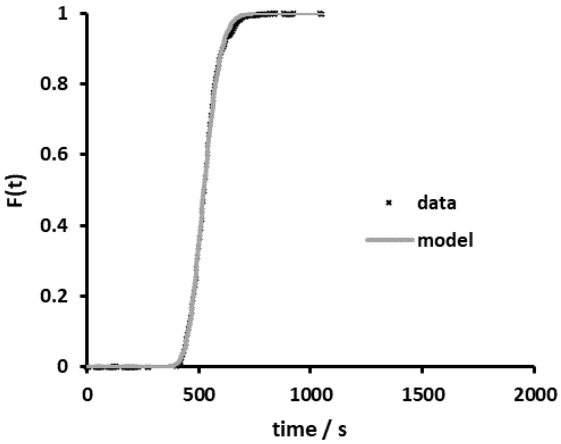


Figure 59: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =2 mL/min, jogging motor only for filling.

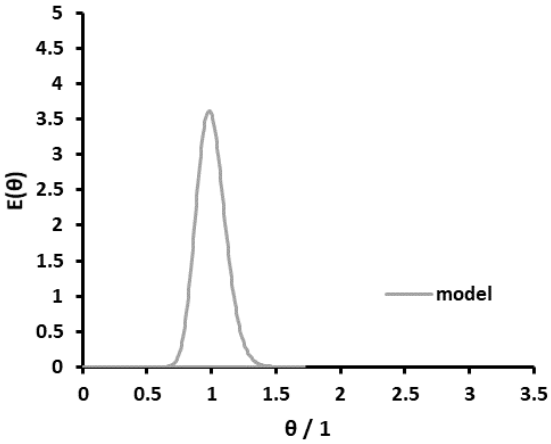


Figure 60: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 2 mL/min, jogging motor only for filling.

**Bo = 167**

**τ = 356 s**

**$\dot{V}$ = 3 mL/min**

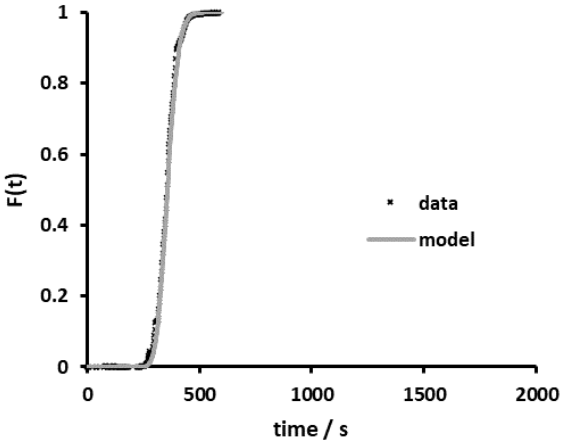


Figure 61: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =3 mL/min, jogging motor only for filling.

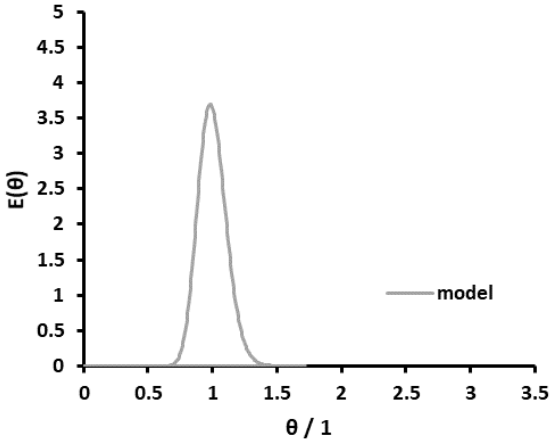


Figure 62: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 3 mL/min, jogging motor only for filling.

**Bo = 166**

**τ = 271 s**

**$\dot{V}$ = 4 mL/min**

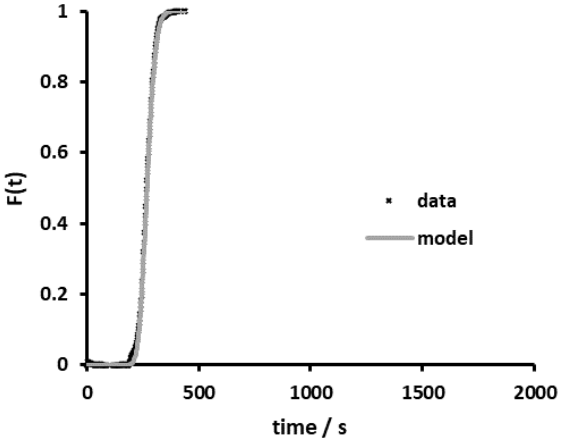


Figure 63: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =4 mL/min, jogging motor only for filling.

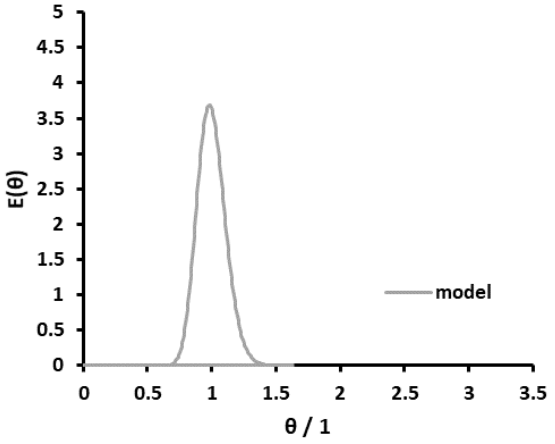


Figure 64: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 4 mL/min, jogging motor only for filling.

### 3.2.2 Jogging motor for filling and RTD measurement

**Bo = 78**

**τ = 1024 s**

$\dot{V} = 1$ **mL/min**

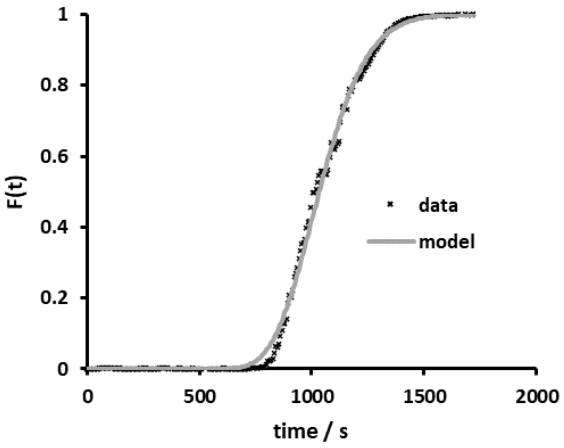


Figure 65: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =1 mL/min, jogging motor for filling and RTD measurement.

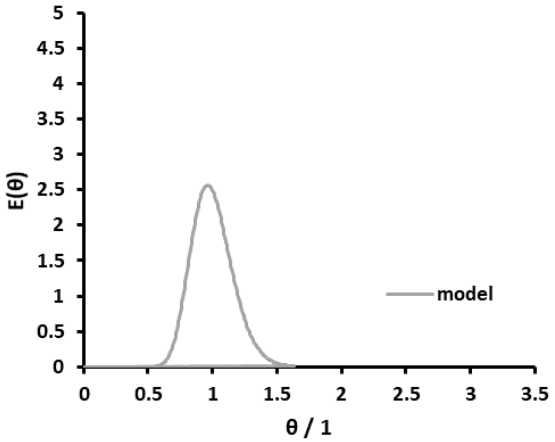


Figure 66: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 1 mL/min, jogging motor for filling and RTD measurement.

**Bo = 169**

**τ = 539 s**

$\dot{V} = 2$ **mL/min**

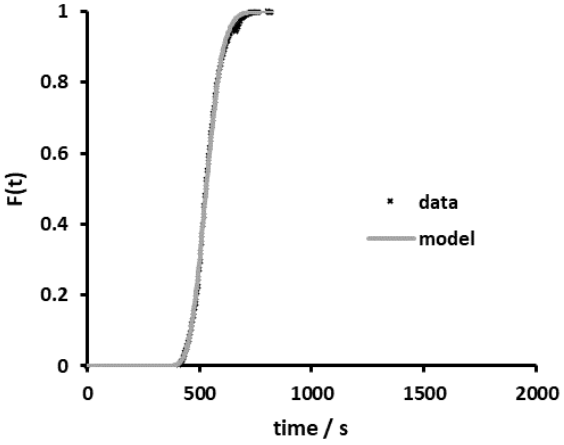


Figure 67: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =2 mL/min, jogging motor for filling and RTD measurement.

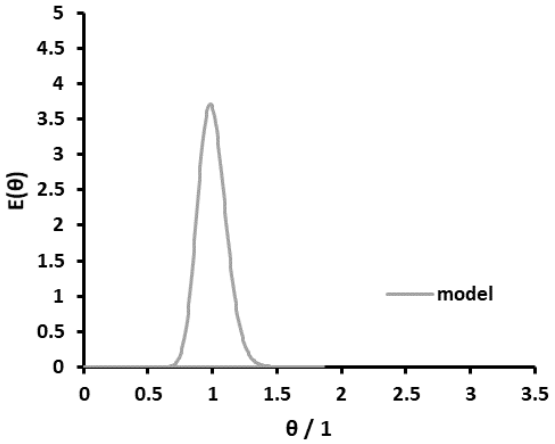


Figure 68: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 2 mL/min, jogging motor for filling and RTD measurement.

**Bo = 166**

**τ = 368 s**

**$\dot{V} = 3$ mL/min**

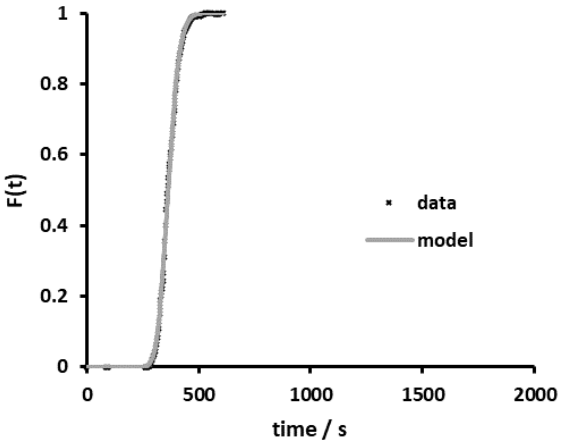


Figure 69: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =3 mL/min, jogging motor for filling and RTD measurement.

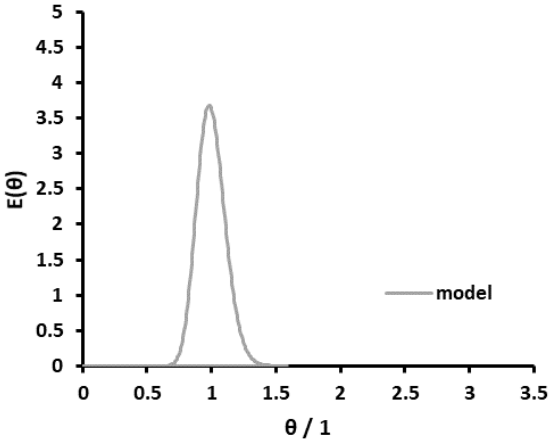


Figure 70: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 3 mL/min, jogging motor for filling and RTD measurement.

**Bo = 131**

**τ = 274 s**

**$\dot{V} = 4$ mL/min**

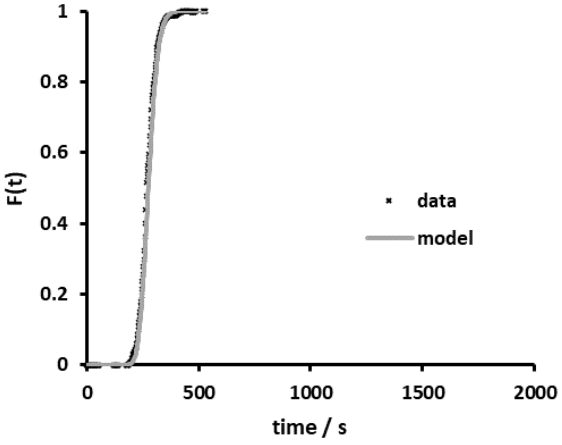


Figure 71: Cumulative distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate =4 mL/min, jogging motor for filling and RTD measurement.

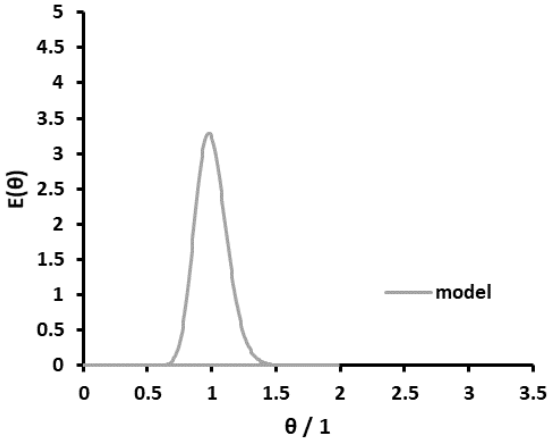


Figure 72: Dimensionless residence time distribution function, pump A, fine magnesium turnings, introduction of tracer, flow rate = 4 mL/min, jogging motor for filling and RTD measurement.

# 4 Oscillating flow conditions in packed magnesium beds

## 4.1 Coarse magnesium turnings

The corresponding cumulative distribution functions and the obtained dimensionless residence time distributions to the data points marked in red in figure 73 are displayed in this chapter as exemplary distribution functions.

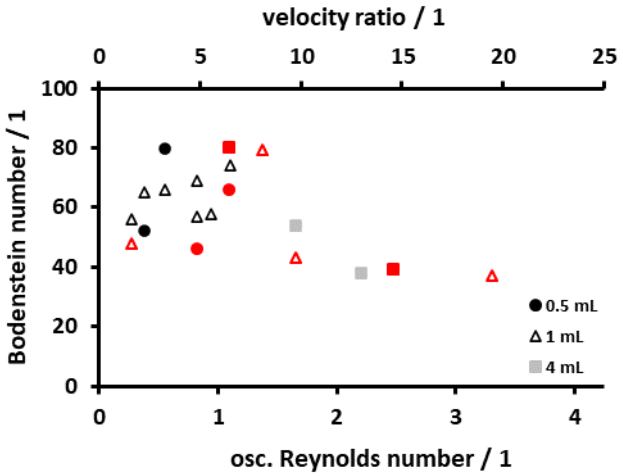


Figure 73: Bodenstein numbers as a function of oscillatory Reynolds number and velocity ratio for the use of coarse magnesium turnings and different stroke volumes.

### 4.1.1 Stroke volume = 0.5 mL

**Bo = 66**

**τ = 570 s**

**stroke volume = 0.5 mL**

**frequency = 0.0067 $s^{-1}$**

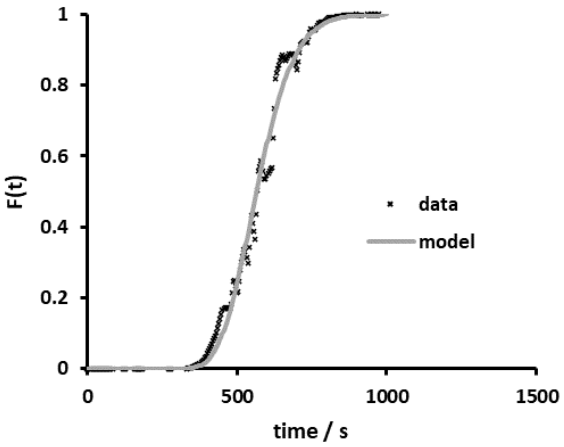


Figure 74: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

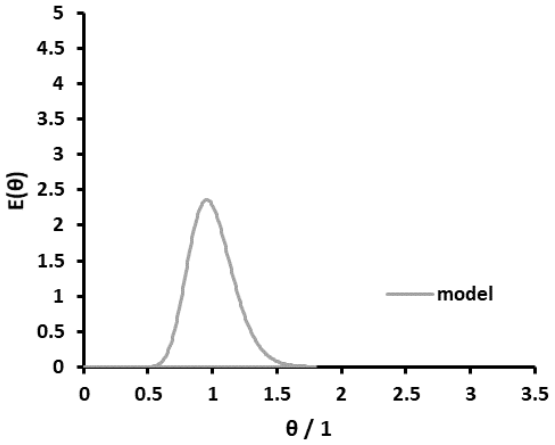


Figure 75: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

**Bo = 46**

**τ = 625 s**

**stroke volume = 0.5 mL**

**frequency = 0.05 $s^{-1}$**

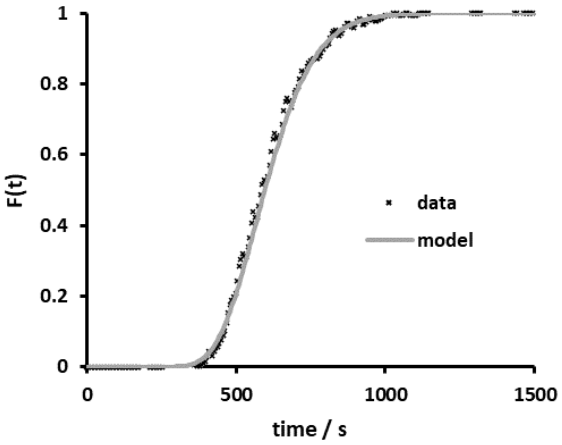


Figure 76: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

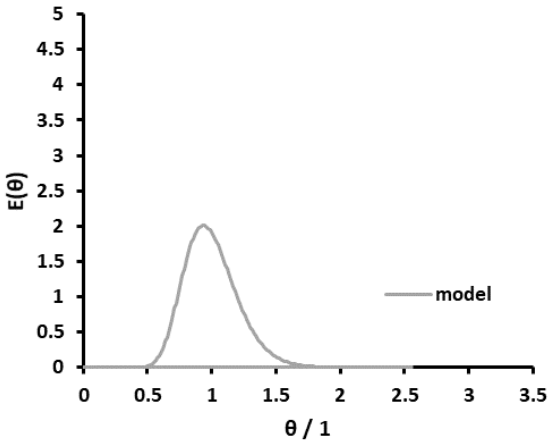


Figure 77: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

### 4.1.2 Stroke volume = 1 mL

**Bo = 48**

**τ = 623 s**

**stroke volume = 1 mL**

**frequency = 0.008 $s^{-1}$**

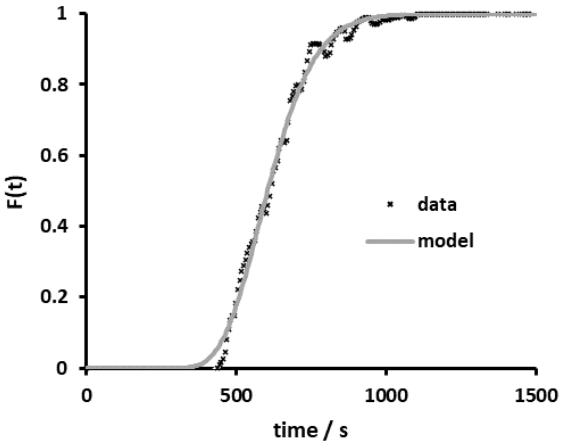


Figure 78: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

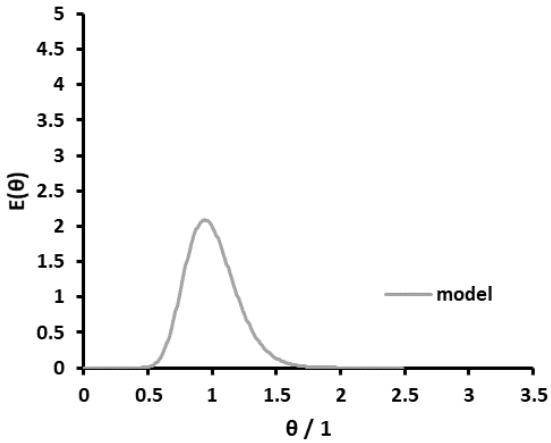


Figure 79: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

**Bo = 79**

**τ = 615 s**

**stroke volume = 1 mL**

**frequency = 0.042 $s^{-1}$**

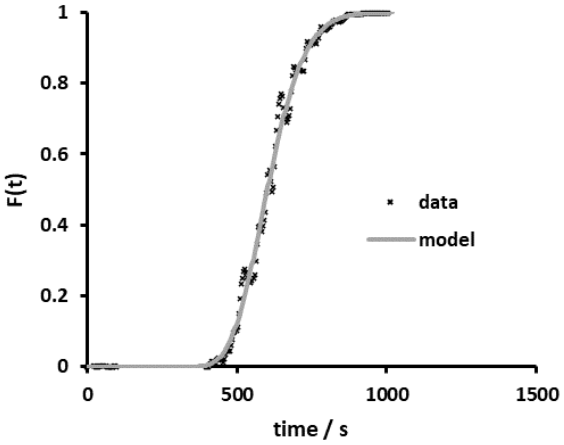


Figure 80: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

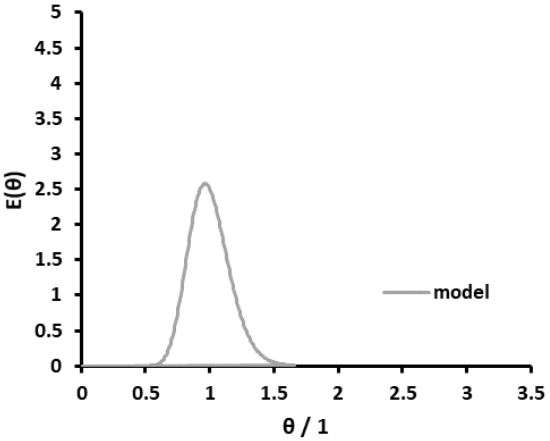


Figure 81: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

**Bo = 43**

**τ = 673 s**

**stroke volume = 1 mL**

**frequency = 0.05 $s^{-1}$**

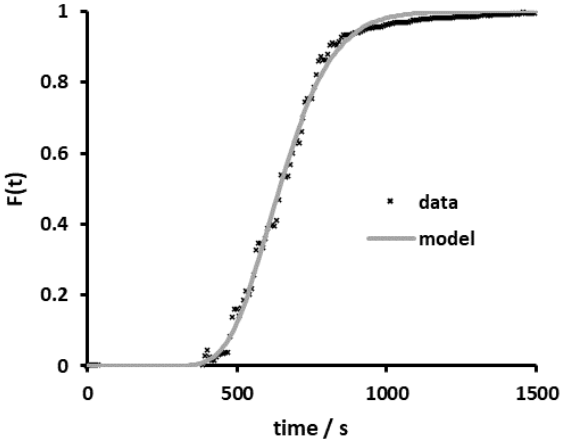


Figure 82: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

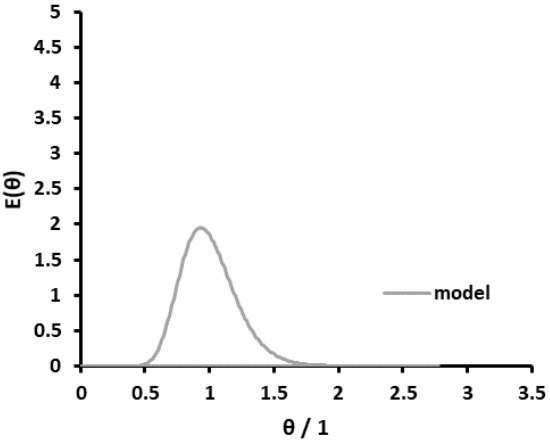


Figure 83: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

**Bo = 37**

**τ = 662 s**

**stroke volume = 1 mL**

**frequency = 0.1 $s^{-1}$**

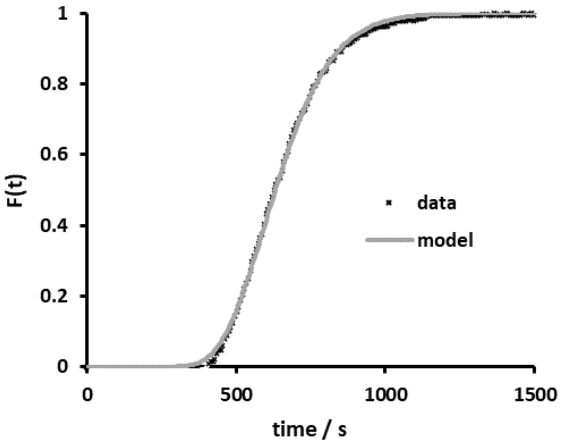


Figure 84: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

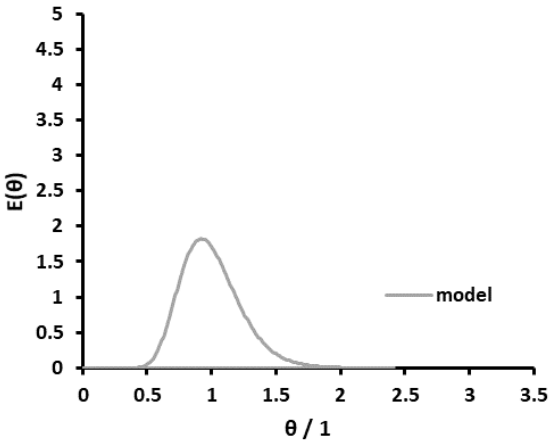


Figure 85: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

### 4.1.3 Stroke volume = 4 mL

**Bo = 80**

**τ = 672 s**

**stroke volume = 4 mL**

**frequency = 0.008 $s^{-1}$**

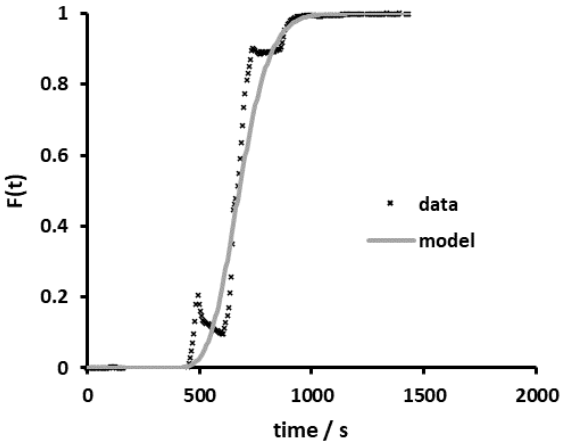


Figure 86: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

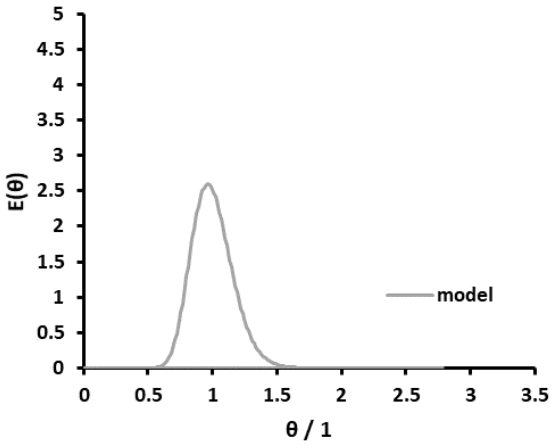


Figure 87: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

**Bo = 39**

**τ = 650 s**

**stroke volume = 4 mL**

**frequency = 0.019 $s^{-1}$**

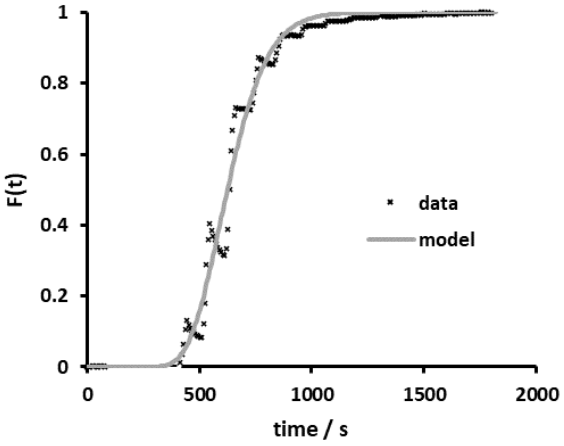


Figure 88: Cumulative distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

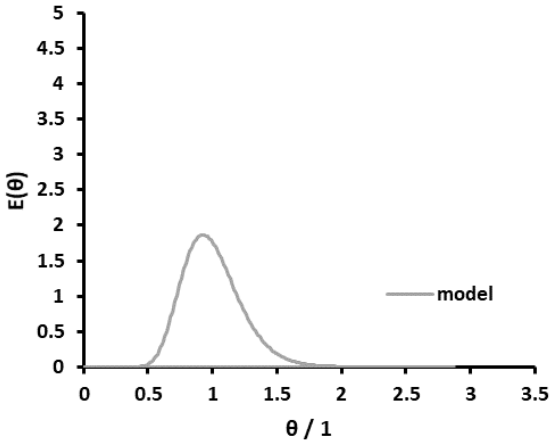


Figure 89: Dimensionless residence time distribution function, pump C + pump B, coarse magnesium turnings, introduction of tracer.

### 4.2 Fine magnesium turnings

The corresponding cumulative distribution functions and the obtained dimensionless residence time distributions to the data points marked in red in figure 90 are displayed in this chapter as exemplary distribution functions.

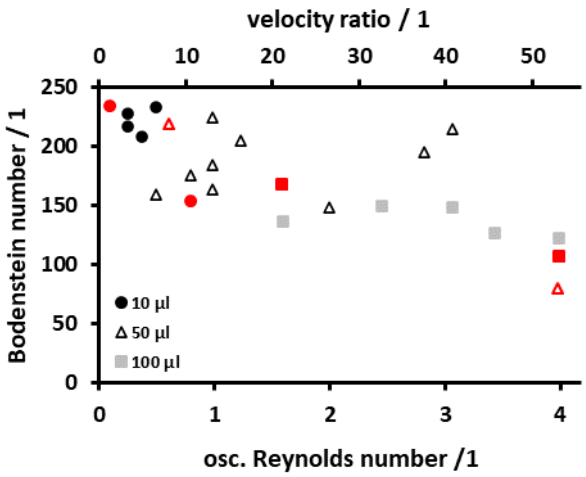


Figure 90: Bodenstein numbers as a function of oscillatory Reynolds number and velocity ratio for the use of fine magnesium turnings and different stroke volumes.

### 4.2.1 Stroke volume = 10 µL

**Bo = 234**

**τ = 538 s**

**stroke volume = 10 µL**

**frequency = 0.67 $s^{-1}$**

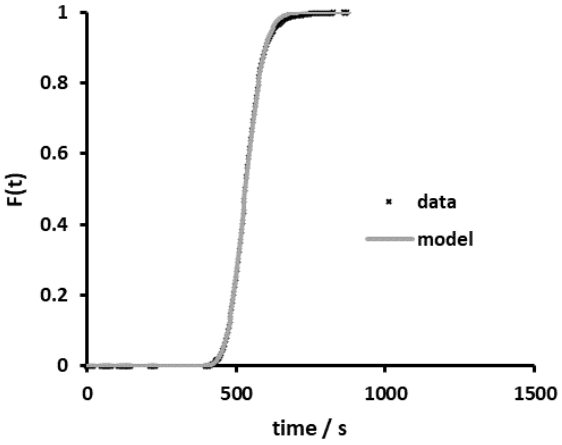


Figure 91: Cumulative distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

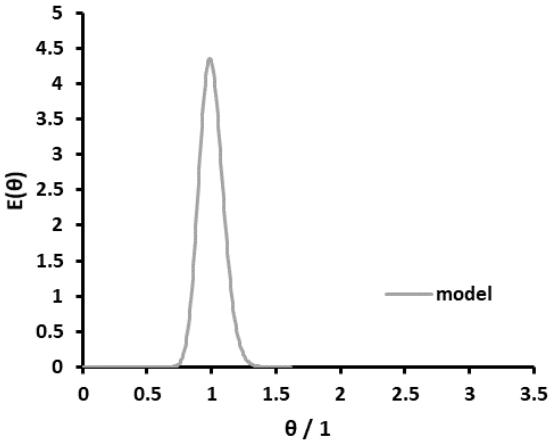


Figure 92: Dimensionless residence time distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

**Bo = 153**

**τ = 502 s**

**stroke volume = 10 µL**

**frequency = 5.42 $s^{-1}$**

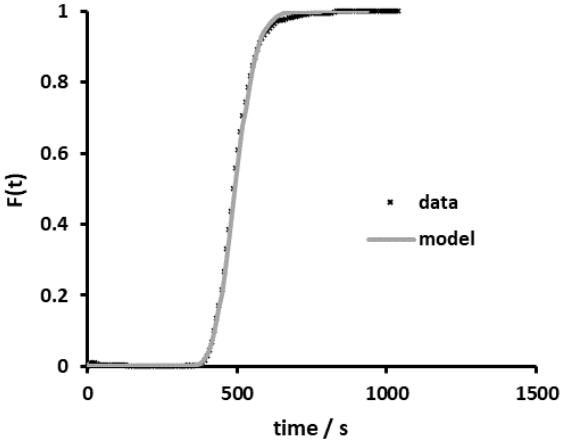


Figure 93: Cumulative distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

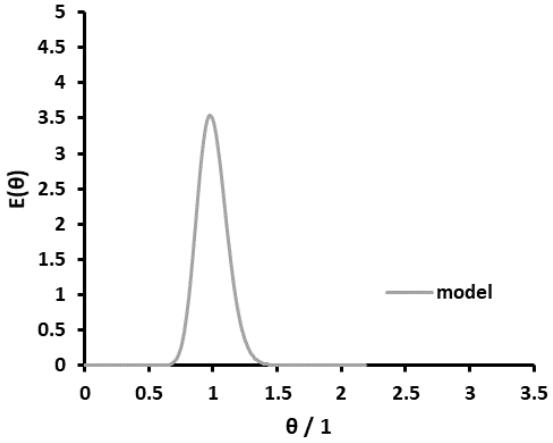


Figure 94: Dimensionless residence time distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

### 4.2.2 Stroke volume = 50 µL

**Bo = 219**

**τ = 543 s**

**stroke volume = 50 µL**

**frequency = 0.83 $s^{-1}$**

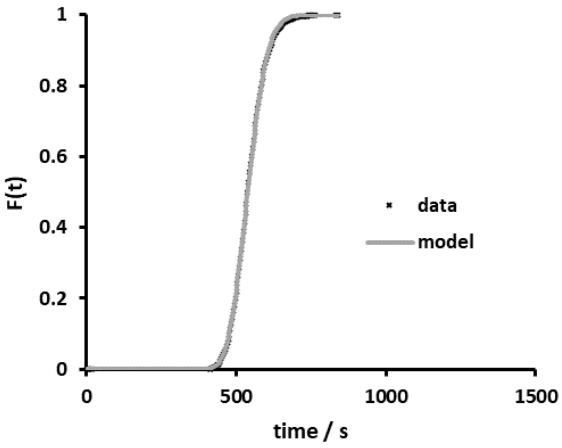


Figure 95: Cumulative distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

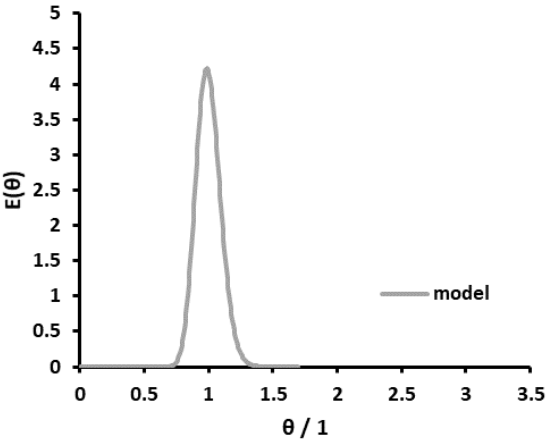


Figure 96: Dimensionless residence time distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

**Bo = 80**

**τ = 507 s**

**stroke volume = 50 µL**

**frequency = 5.42 $s^{-1}$**

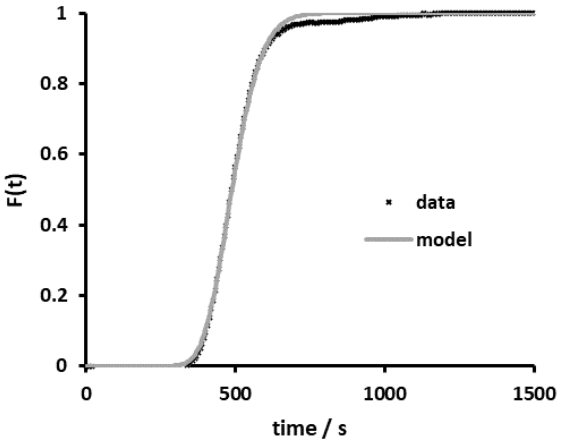


Figure 97: Cumulative distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

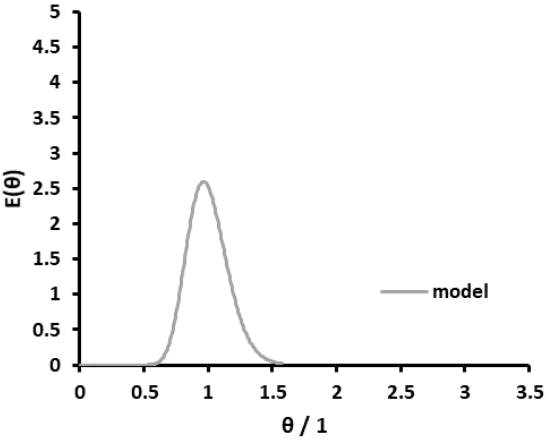


Figure 98: Dimensionless residence time distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

### 4.2.3 Stroke volume = 100 µL

**Bo = 168**

**τ = 525 s**

**stroke volume = 100 µL**

**frequency = 1.08 $s^{-1}$**

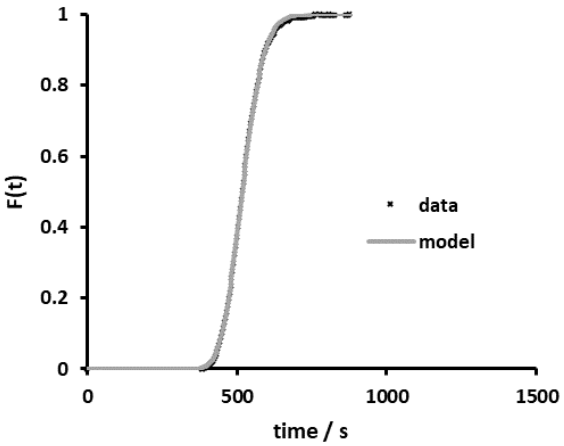


Figure 99: Cumulative distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

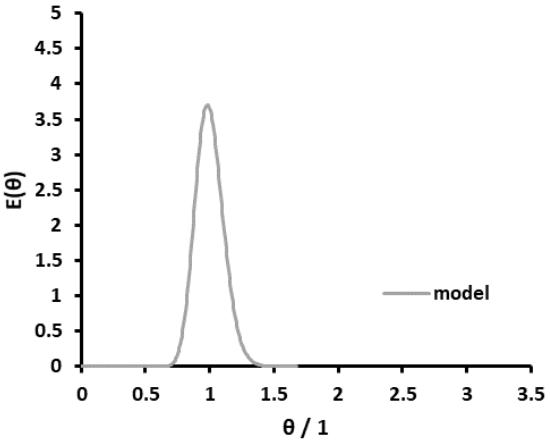


Figure 100: Dimensionless residence time distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

**Bo = 107**

**τ = 500 s**

**stroke volume = 100 µL**

**frequency = 2.72 $s^{-1}$**

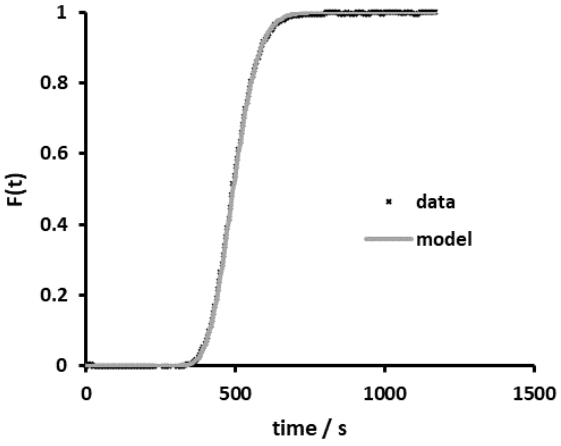


Figure 101: Cumulative distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.

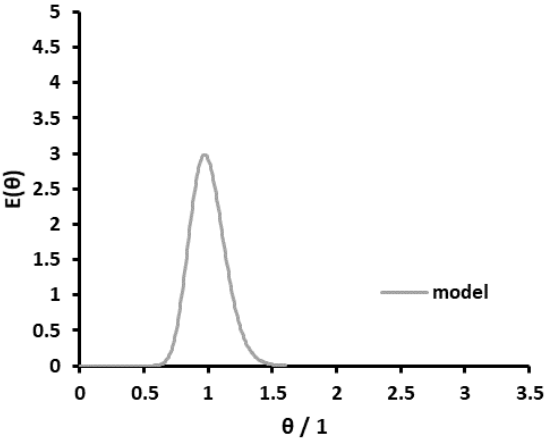


Figure 102: Dimensionless residence time distribution function, pump C + pump D, fine magnesium turnings, introduction of tracer.