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## Measuring residence time distribution Protocol

This datasheet is designed to provide industrials with a detailed protocol for measuring Residence Time Distributions. This protocol is based on the experience Tecaliman has gained at animal feed plants. The various protocol phases are described or illustrated using examples.

The suggested method is based on the use of particulate tracers and the pulsed injection tracer incorporation procedure.

### 1. Measurement focus

Define the measurement campaign.

Example: the measurement campaign is designed to qualify meal conditioning times in a soaking vessel prior to pelleting.

### 2. Principle

The distribution of meal residence times in the process (RTD) is estimated using a particulate tracer, which is incorporated by pulsed injection at process input.

The meal samples containing the tracer are recovered at process output.

The dosage of tracer concentration in these samples is used to identify and study residence time distributions at process output (Tecaliman 2004 a).

### 3. Equipment and apparatus

#### 3.1. Animal feed

Note the type of meal and the formula, then identify its particle size and bulk density.

Example: A Dairy Cow feed made to formula 1245 is selected for the test.

The feed's particle size is determined using the methods described in AFNOR standards X-11-501 and ASAE 319-1, in order to select a tracer with a similar particle size.

The feed's median diameter is 624  $\mu\text{m}$ . Sixty-eight percent of particles have an equivalent diameter of between 329 and 1186  $\mu\text{m}$ .

The density of the feed meal is 594 g/litre. This value is measured using AFNOR's referenced method NF 23923-1 of 1993. This datum is used to calculate the volume flow of the treatment procedure.

#### 3.2. Tracer

Several types of particulate tracer can be used (Tecaliman 2004 b). As far as possible, select a tracer with a particle size and bulk density similar to that of the feed.

Once the tracer has been selected, its type, particle size, bulk density and number of particles per gram of tracer have to be noted.

Example: The tracer used is red-dyed, calibrated corn cob particles. Table 1 details its characteristics.

<b>Number of particles per gram</b>	2084
<b>Particle size</b>	100% of particles have an equivalent diameter of between 630 and 800 $\mu\text{m}$
<b>Bulk density (g/litre)</b>	215

Table 1

#### 3.3. Treatment line

Describe the treatment line, present it in an illustration, and specify tracer incorporation locations and sampling points.

Example: The heat treatment line comprises two worm screws D1 and D2, two mixers (MMA and MAL), one soaking vessel and one pelleting press (Figure 1).

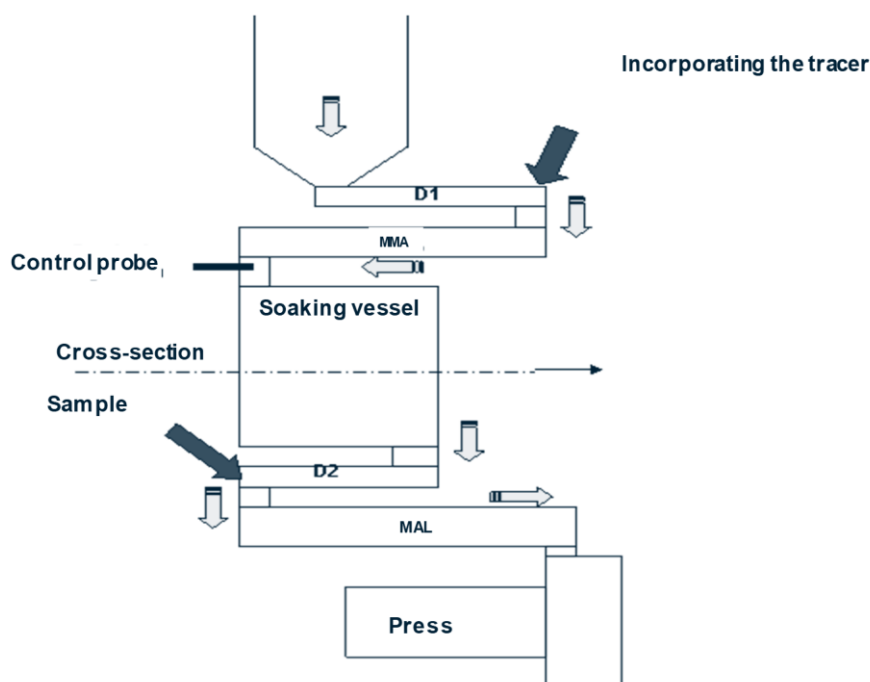


Figure 1

## 4. Methods

### 4.1. Operating the heat treatment line

The plant has to supply the following information: treatment line flow rate, mean treatment time (or throughput time) and, where appropriate, the process fill rate and useable volume.

Example: The mean flow rate of the treatment line is estimated at 10 t/h. and the mean treatment time is between 15 and 20 minutes.

### 4.2. Injecting the tracer

#### 4.2.1. Quantity of tracer to be injected

The precise quantity injected must be known as any overestimation could affect the tracer recovery rate, making the results difficult to interpret.

This quantity is calculated based on:

- maximum pulsed injection time, which should be lower than or equal to  $T_p \times 0.01$  (TECALIMAN 2004a)

- volume flow of the tracer injection in relation to the feed's volume flow ( $< 4\%$ ),
- mean tracer concentration or equivalent concentration "Ceq" that has to be dosed at the process output (assuming that the tracer is evenly distributed throughout the mass of product in the process).

This calculation requires the following data input:

- time taken for the feed to pass through the process ( $T_p$ ), estimated by the plant or calculated from the mass of product in the process and the process flow rate,
- mass flow rate of the feed ( $D_{ma}$ ),
- bulk density of the feed ( $M_{va}$ ),
- bulk density of the tracer ( $M_{vt}$ ),
- number of particles per gram of tracer ( $N_t$ ).

A sample calculation is given in Table 2 and Table 3.

TRACER	Unit	Symbol	Formula
Feed throughput time (plant estimate)	sec.	Tp	
Mass flow rate of the feed (plant estimate)	kg/h	Dma	
Density of the feed (measured)	kg/l	Mva	
Density of the tracer (measured)	kg/l	Mvt	
Useful volume of the process (calculated from the manufacturer's label)	l	Vp	
Number of particles per gram of tracer (measured)	Part./g	Nt	
Number of particles of tracer/gram of feedstuff or equivalent concentration	Part./g	Ceq	
Mass of feedstuff in the process	g	Ma	$Ma = (Dma/3600)*Tp*1000$
Number of particles injected		N	$N = Ma*Ceq$
Quantity of tracer injected	g	Mt	$Mt = Ni/Nt$
Maximum injection time ( $Tp*0.01$ )	sec.	Tmax	$Tmax = Tp*0.1$
Mass flow of tracer during the injection time	kg/h	Dmt	$Dmt = (Mt/1000)/(Tmax*3600)$
Mass flow of tracer as a % of the mass flow of feedstuff	%	$Dmt\%Dma$	$Dmt\%Dma(Dmt/Dma)*100$
Volume flow of feedstuff during the injection time	l/h	Dva	$Dva = Dma/Mva$
Volume flow of tracer during the injection time	l/h	Dvt	$Dvt = Dmt/Mvt$
Volume flow of tracer as a % of the volume flow of feedstuff during the injection time	%	$Dvt\%Dva$	$Dvt\%Dva = (Dvt/Dva)*100$

Table 2

Symbol	Unit	Example
Tp	sec.	1.200
Dma	kg/h	10.000
Mva	kg/l	0.594
Mvt	kg/l	0.215
Vp	l	1.000
Nt	Part./g	2.004
Ceq	Part./g	0.25
Ma	g	3.333.333
Ni		833.333
Mt	g	416
Tmax	sec.	12
Dmt	kg/h	125
$Dmt\%Dma$	%	1.2
Dva	l/h	16.835
Dvt	l/h	580
$Dvt\%Dva$	%	3.4

Table 3: Tracer: dyed corn cob particles

#### 4.2.2. Method and location of tracer injection

At time  $t_0 = 0$  minute, a known mass of tracer is injected rapidly in a single dose (pulsed injection), at the process input. Where possible, the tracer injection should be distributed over the whole cross-sectional area of the entrance to the process under study (Tecaliman 2004 a).

Example: At time  $t_0 = 0$ , a mass of 412 g of tracer is injected rapidly (pulsed injection) at the MMA mixer input (Figure 1).

### 4.3. Sampling procedure

#### 4.3.1. Samples used to characterise the feedstuff

A sample of feedstuff is taken at the mixer output in order to measure the feed particle size.

A sample of feedstuff is taken at the process output in order to identify the feed's bulk density and moisture content.

#### 4.3.2. Samples used to measure RTD

The samples are taken as close as possible to the mixer-reactor (Tecaliman 2004 a).

The samples must be taken over the whole flow, in order to obtain a sample that is truly representative of all the outgoing particles (feed+tracer) at the time of sampling. If this is not possible, the direction in which the samples are taken will have to be alternately reversed; for instance, by taking even-numbered samples from left to right and odd-numbered samples from right to left.

At least 40 samples will have to be taken in order to obtain a complete residence time distribution curve and a tracer recovery rate of around 100%, distributed over a time equal to approximately twice the throughput time.

A stopwatch is activated as the tracer is injected, i.e. at time  $t_0 = 0$  minute.

Each sample must be packed (possibly after being divided up) in a numbered, leaktight plastic pot. This number must match the order of sampling.

Example: Samples are taken at the D2 worm screw output. Given that the process throughput

time is between 15 and 20 minutes, the samples are taken every 30 seconds (sampling time), from time  $t_0 = 0$  minute to time  $t_{43} = 43$  minutes.

In all, 43 samples of at least 100 g are packed in plastic pots identified by a number from 0 to 43, corresponding to the order in which the samples were taken.

#### 4.4. Dosing the tracer

The tracer dosage technique depends on which tracer is used (Tecaliman 2004 b).

To limit the number of analyses, the tracer concentration is firstly dosed on "even-numbered" samples. This dosage is performed over the whole sample, or on a test portion obtained by dividing the sample (use a riffle splitter).

If the amount of data obtained after dosing the tracer in the even-numbered samples is not sufficient to plot residence time distribution curves, additional dosages will be made on odd-numbered samples.

If the samples taken at the plant have dried out during storage, the moisture content of these samples will have to be measured in order to calculate the number of particles per gram of feedstuff at the process output.

Example: Tracer concentration is dosed by counting red-dyed coloured particles in even-numbered samples, which corresponds to a 60-second sampling time and 43 samples. The results are given in Table 4.

Ti = Particle age or residence times (minute)	Sample No.	Sample weight (g)	Number of tracer particles
T0	0	226	0
T1	2	218	0
T2	4	215	0
T14	28	232	0
T15	30	275	1
T16	32	279	1
T42	84	270	8
T43	86	246	5

Table 4

#### 4.5. Means to be deployed

##### 4.5.1. Staff

At the level of the tracer's injection location, one person is needed to carry out this operation.

At the sampling location, at process output: 3 people will have to be present:

- one person to take the samples.
- one person to pass the pots used to pack the samples to the sample taker.
- one timekeeper, tasked with starting and stopping the stopwatch according to the sampling time.

After injecting the tracer, the person tasked with this operation has to help the sampling team carry the samples out of the sampling area, closing the sample pots where necessary.

##### 4.5.2. Equipment and apparatus

Plan for sampling apparatus, numbered pots in which to pack the samples, a stopwatch and the necessary quantity of tracer along with packaging that is suited to the injection location.

## 5. Bibliography

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**Tecaliman 2004b**, Measuring residence time distribution – Tracers – i'Tec\_S12 – november 2004.

**Tecaliman 2004**, Measuring residence time distribution by pulsed injection – Data processing - i'Tec\_S14 – november 2004.