

## MT 36.4 EMULSION CHARACTERISTICS AND RE-EMULSIFICATION PROPERTIES

### SCOPE

The method is intended for determining the emulsification characteristics and re-emulsification properties of emulsion concentrates (EC) and other products forming emulsions after dilution in water at rates of 0.1% to 5%.

### REASON FOR REVISION

The revision is intended to give better indications on the evaluation of initial emulsification and emulsion stability and is harmonised with similar methods regarding the stability of formulations in water.

The temperature is changed from  $30 \pm 2$  °C to  $25 \pm 5$  °C.

Where the same conditions are used, results obtained with MT 36.4 are equivalent to those obtained with MT 36.3. MT 36.4 supersedes MT 36.3.

### OUTLINE OF METHOD

An emulsion in Standard Water is prepared. The ease of emulsification is assessed, as well as the stability of this emulsion in terms of amounts of separated matter during standing undisturbed for 2 h. Where initial emulsification and/or emulsion stability is not evident, the ability of the system to re-emulsify at the end of a 24 h period is determined.

### REAGENTS

*Standard Water D*, MT 18.1 unless otherwise specified

### APPARATUS

*Measuring cylinders*, glass, 100 ml with stopper. The distance between the 100 ml graduation mark and the bottom of the stopper should be not more than 7 cm and not less than 3 cm.

*Pipette* or

*Balance* with an accuracy of at least 0.01 g

*Lamp*

### PROCEDURE

All operations are performed at ambient temperature ( $25 \pm 5$  °C).

#### (a) *Initial emulsification*

Fill the measuring cylinder approximately to the 95 ml mark with Standard Water. Add the required amount of the test sample gently onto the surface of the water and fill up to 100 ml with Standard Water (Note 1). The test sample may either be added volumetrically or gravimetrically using the determined density. Insert the stopper and invert the cylinder ten times (Note 2).

Allow the cylinder to stand for approximately 30 s. Observe whether the mixture has emulsified completely, giving 100 ml of an emulsion which appears, on visual examination (Note 3), to be uniform and complete (Note 4). Carefully observe whether any separation is found (Note 5). The emulsion must be looked at from different angles to allow accurate assessment of uniformity/separation. This includes

- observation of sediment at the bottom of the cylinder during the last inversion,
- observation of oil, cream, particles, or separate liquid phase (see figure 1 to 3).
- observation of separate liquid phase at the bottom edge of the cylinder by positioning the cylinder in an approximate 45° angle and waiting for a few seconds (see figure 4 to 5).

In case the initial emulsification is not complete, record the observations (Note 5).

**(b) *Emulsion stability on standing***

Allow the cylinder to stand undisturbed. 2 h after initial emulsification (Note 6) observe visually (Note 3) whether any separation is found. Position the cylinder in an approximate 45° angle, wait for a few seconds and observe any separated liquid phase at the bottom edge of the cylinder. Invert the cylinder once, to evaluate sediment on the bottom of the cylinder. Return the cylinder into an upright position and record the observations.

No further testing is required if no separation is observed.

Otherwise allow the cylinder and its contents to stand undisturbed for further 22 h. Repeat the procedure as described for the evaluation after 2 h. Record the observations.

**(c) *Re-emulsification 24 h after initial emulsification***

At the end of the 24 h period and after having finished procedure (b) *Emulsion stability on standing* invert the cylinder 10 times and follow the evaluation procedure described in (a). In case the re-emulsification is not complete, record the observations (Note 5, 7).

If there is a need to determine a small volume of a separated phase at the bottom accurately, emulsion tubes can be used (Note 8).

## REPORTING

Report the emulsion characteristics and re-emulsification properties by specifying the following parameters:

- Test concentration of the emulsion.
- Standard Water used if it is not Standard Water D.
- Initial emulsification: whether complete and emulsion uniform or not. In case the initial emulsification is not complete, report the observations and volumes (Note 9).

- Volume (Note 9), position and type of separation after 2 h (cream, oil, sediment, or others).
- Volume (Note 9), position and type of separation after 24 h, if performed (cream, oil, sediment, or others).
- Re-emulsification (if performed): whether complete and emulsion uniform or not. In case the re-emulsification is not complete, report the observations and volumes (Note 9).
- Use of emulsion tubes, if relevant.

Note 1: Unless otherwise specified the test should be performed at the highest and lowest recommended use rates if within the scope of the method. Where the recommended concentrations are below 0.1% or higher than 5%, the test must be performed at concentrations of 0.1% and 5%, respectively. If performed at concentrations above 5% the visual observation may not be possible.

Note 2: The expression 'invert the cylinder' implies that the stoppered cylinder is tipped by hand 180 °, and is then brought back to its original position, the whole operation being completed in approximately 2 s.

Note 3: Illuminate the cylinder with the lamp. Adjust the position (transmitted or reflected) and the angle of the light for optimal viewing of the phase boundary, if present. In some cases, the use of lamps with different colour temperatures (e.g., more white or yellow light) may be appropriate.

Note 4: Completeness means absence of cream, oil, separate liquid phase, sediment, particles, and other material that is not dissolved or emulsified. No neat product shall be left on the inner surface of the cylinder. Traces should be interpreted as incomplete emulsification. Foam is not considered as criterion against completeness of emulsification and uniformity of emulsion.

Note 5: Separation includes 'oil', 'cream', any other liquid phase, 'sediment', or other particles, which cannot be considered as part of a homogeneous emulsion.

Note 6: Results after standing for 30 min can be added, optionally.

Note 7: Results after standing for 24 h and 30 min can be added, optionally.

Note 8: Using emulsion tubes for the determination of separation on bottom: *Emulsion tubes*, ASTM centrifuge tubes, borosilicate glass with conical bottom, 15 cm (6 inch), graduated to 100 ml (ASTM D 91 and D 96) *Rubber stopper*, fitting in the emulsion tube and provided with a 80 mm glass ventilation tube (external diameter 4.5 mm; internal diameter: 2.5 mm)

### Procedure

Immediately after forming of the emulsion transfer the 100 ml to an emulsion tube. Stopper and allow to stand in an upright position for a specified time. Observe visually (Note 3) whether any separation is present.

Note 9 Separated volumes of less than 1 ml should be reported as < 1 ml.

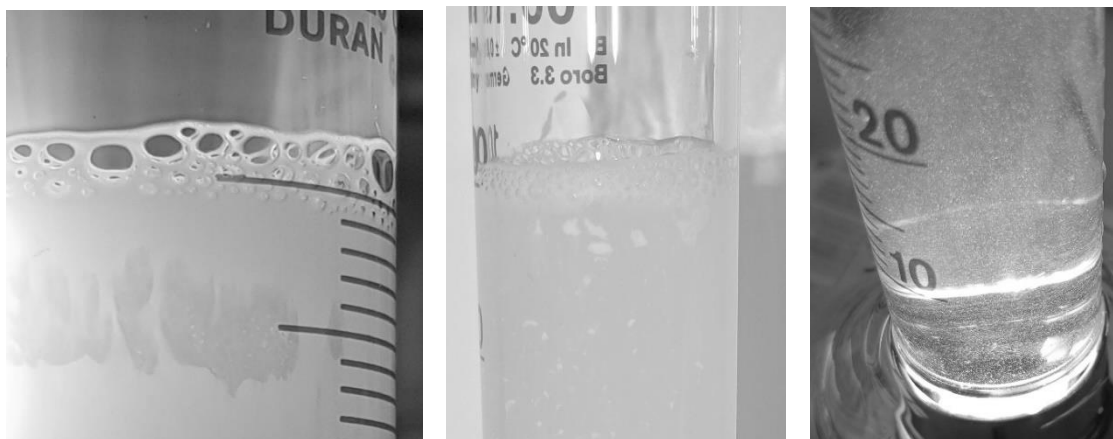


Fig. 1: Incomplete emulsification (from left to right: material at glass surface, particles in an emulsion, particles without emulsion)

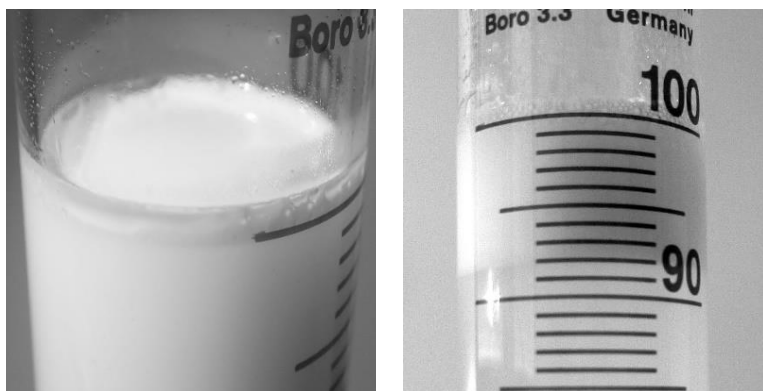


Fig. 2: Cream < 1 ml (left); Cream 8 ml (right)



Fig. 3: Oil 6 ml (with some cream)

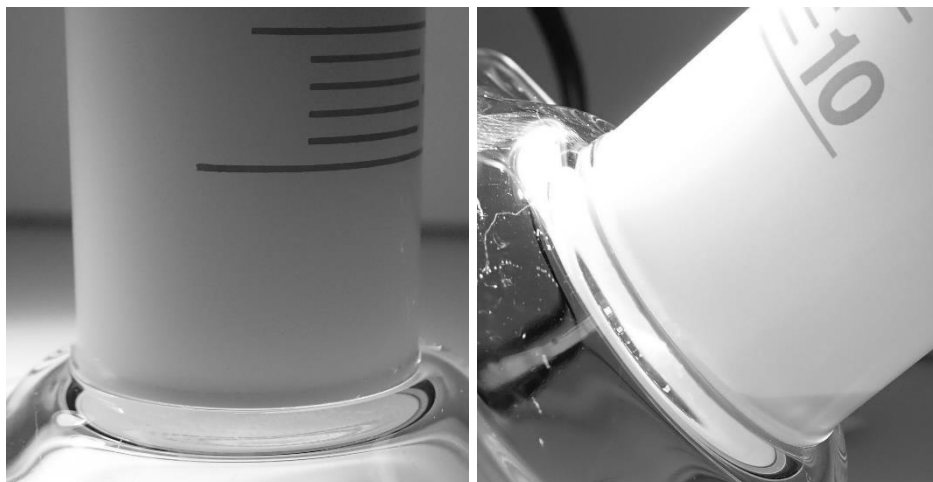


Fig. 4: Separated phase at bottom, approximately 1 ml



Fig. 5: No separated phase visible