# Reliable and simple determination of stickies applying NIR measurement – establishing a new standard for the paper industry

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## INTRODUCTION:

Stickies are tacky contaminants within recycled pulp that impact the papermaking process negatively, causing web breaks at the paper machine and resulting in production losses. Today's laboratory sticky measurements are mainly conducted according to ISO 15360-2 [1], within which the measuring procedures according to TAPPI T277 [2] and INGEDE 4 [3] are summarized. These methods are well established in the paper industry, require an extensive sample preparation and a variety of manual working steps.

Determining the sticky content of stock samples using the near-infrared (NIR) macro sticky measurement method provides several advantages compared to the laboratory methods established in the paper industry. The accuracy of this measurement technique is significantly higher, and the effort to prepare and evaluate samples is noticeably lower compared to today's measuring techniques. To standardize sample preparation and the measuring procedure, the technical rule DIN SPEC 6745 [4]: "Paper, board and pulps - Recycling - Determination of stickies and non-tacky contaminants in recycled pulps and papers applying near-infrared measurement" was elaborated and published in March 2020. Due to its technological and technical advantages, as well as the availability of a technical rule to prepare samples and perform measurements using the NIR measurement technique, this new approach to measuring macro stickies has the potential to supersede existing measurement techniques in the future as a new standard for the paper industry.

This paper presents Voith Paper's experiences in performing sticky measurements according to DIN SPEC 6745, applying the NIR measurement technique. The results of measurements made using a PTS NIR macro sticky measurement device, which was installed in June 2020 at Voith Paper's laboratory, will be discussed and compared to results of established measurement techniques.

### State of the art – measuring stickies according to ISO 15 360-2

Measuring stickies according to ISO 15360-2 "Estimation of Stickies and Plastics - Part 2: Image analysis method" [1] is well established and proven within the paper industry. Two main procedures for measuring stickies are described in ISO 15 360-2, following the measuring procedure described in:

1. TAPPI T277 – "Macro stickies content in pulp: the "pick-up" method" [2]
2. INGEDE 4 – "Analysis of macro stickies in pulps" [3]

The measuring procedures described in these norms are generally similar; but, single process steps and equipment to be used may vary such that measuring results will differ depending on the procedure chosen.

Since sticky samples are evaluated at Voith Paper according to TAPPI's "pick-up method," the sticky measuring procedure according to ISO 15 360 - 2/ TAPPI T277 is shown in Figure 1. In total, eight steps are needed to prepare the sticky sample and to measure the white-colored stickies using a conventional scanner. Typically, a laboratory technician needs 100 to 120 minutes to evaluate a single stock sample.

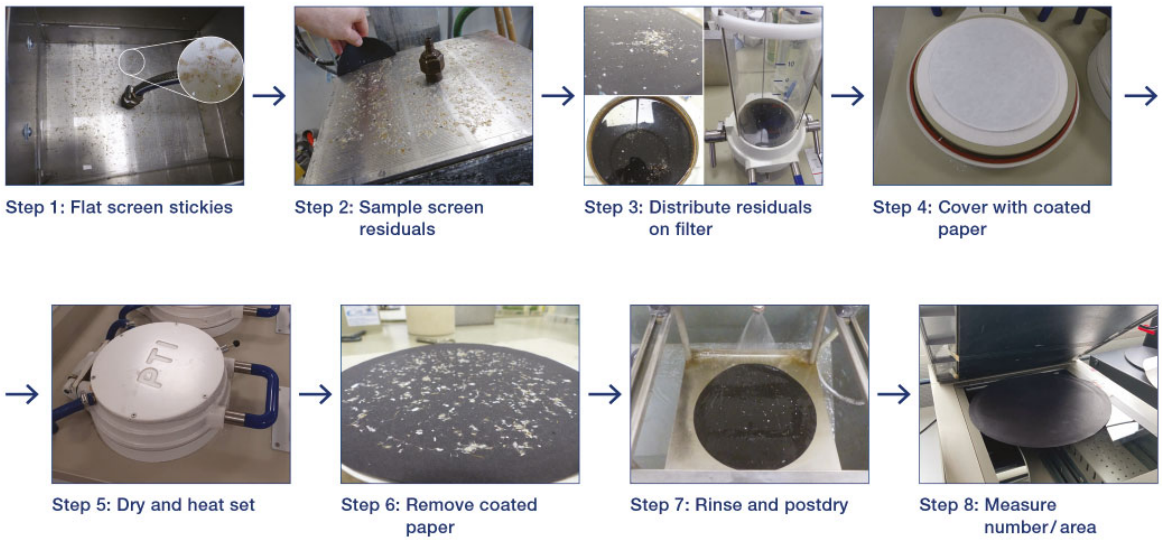


*Figure 1: Procedure to measure stickies according to ISO 15 360 - 2/ TAPPI T277*

To assess the results of interlaboratory studies of sticky measurements according to DIN SPEC 6745, the procedure described in [5, 6] are applied. Two main parameters are considered for this purpose:

- **Repeatability** is defined as "an estimated limit below which the difference between two test results is expected to fall 95% of the time, when the results are obtained at a single laboratory. Repeatability reflects the variability inherent in the experimental system used to determine its value(s)." [5]
- **Reproducibility** is defined "an estimated limit below which the difference between two test results is expected to fall 95% of the time, when the results are obtained in different qualified laboratories for the same homogeneous source of material." [5]

Due to the high number of manual working steps for sticky measurements according to ISO 15 360 - 2/ TAPPI T277, the repeatability and reproducibility were defined as "the maximum expected difference between two test results" and are summarized as follows [7]:

- **Repeatability** within a laboratory: 28% maximum deviation between two results if performed by the same lab technician.
  - **Note:** Within a recent Voith Paper study for three different sample series (different sticky loads), the complete sample preparation process was conducted three times each by different lab technicians and the repeatability was 39%. The calculation for the repeatability was done as described in [5,6].
- **Reproducibility** (between laboratories): 101% maximum deviation between two results.

To increase the statistical reliability of sticky measurement results according to ISO 15 360 - 2/ TAPPI T277, especially to prove technological performance of machinery sold, the number of measurements is increased and for guarantee runs within Voith Paper, typically 12 measurements are performed for every sampling position. Furthermore, it is important that all measurements in this context are performed by one laboratory only. Due to aging effects of stock samples, the effort to organize logistics such that samples from overseas can be evaluated in time is high.

### Exploring a new standard – measuring stickies according to DIN SPEC 6745 using NIR

Measuring stickies according to DIN SPEC 6745: "Paper, board and pulps - Recycling - Determination of stickies and non-tacky contaminants in recycled pulps and papers applying near-infrared measurement" [4] was introduced in 2020 and thus is relatively new for the paper industry.

The sticky measuring procedure according to DIN SPEC 6745 is shown in Figure 2. This procedure is very straight forward, with the only steps to prepare the stock samples for NIR measurement being hand sheet forming and subsequent drying. The hand sheets can be analyzed by the NIR measurement device, as briefly described in the following:

- The sheets are placed under the NIR camera on the x-y table of the NIR sticky measurement device.
- The sheets are scanned in strips under homogeneous lightning conditions and using a NIR camera. For every pixel, the spectral information for wave lengths of 1,320 nm to 1,900 nm is recorded.
- Using a NIR imaging approach [8], neighboring pixels that cannot be assigned to fibers or fillers are summarized as contaminants.
- A further classification is made, and these contaminants are classified and assigned to the sticky and non-tacky contaminant groups according to the spectral information measured by the NIR camera. For example, PVAc, acrylates and EVA are chemical substances assigned to the sticky contaminants, whereby, PE and PS are chemical substances assigned to the non-tacky contaminants.

Typically, four hand sheets are formed for each stock sample and analyzed sequentially using the NIR measurement device. A laboratory technician needs 40 to 50 minutes to evaluate a single stock sample according to DIN SPEC 6745; that measuring time is almost half of the time required to use the ISO 15 360 - 2/ TAPPI T277 approach. Since the ability to make hand sheets is widespread within the paper industry, it thus becomes possible to prepare hand sheets anywhere in the world in a timely manner after sample-taking and sending the dried hand sheets to a laboratory that is able to perform NIR macro sticky measurements. This provides several advantages in the daily business of a paper producer and a supplier within the paper industry.

Due to the minimized number of manual working steps, the accuracy of results improved using the NIR measuring procedure according to DIN SPEC 6745 compared to ISO 15 360 - 2/ TAPPI T277. The following preliminary results for repeatability, defined as "the maximum expected difference between two test results" [7] for the NIR method according to DIN SPEC 6745 are for this purpose introduced:


*Figure 2: Procedure to measure stickies according to DIN SPEC 6745*

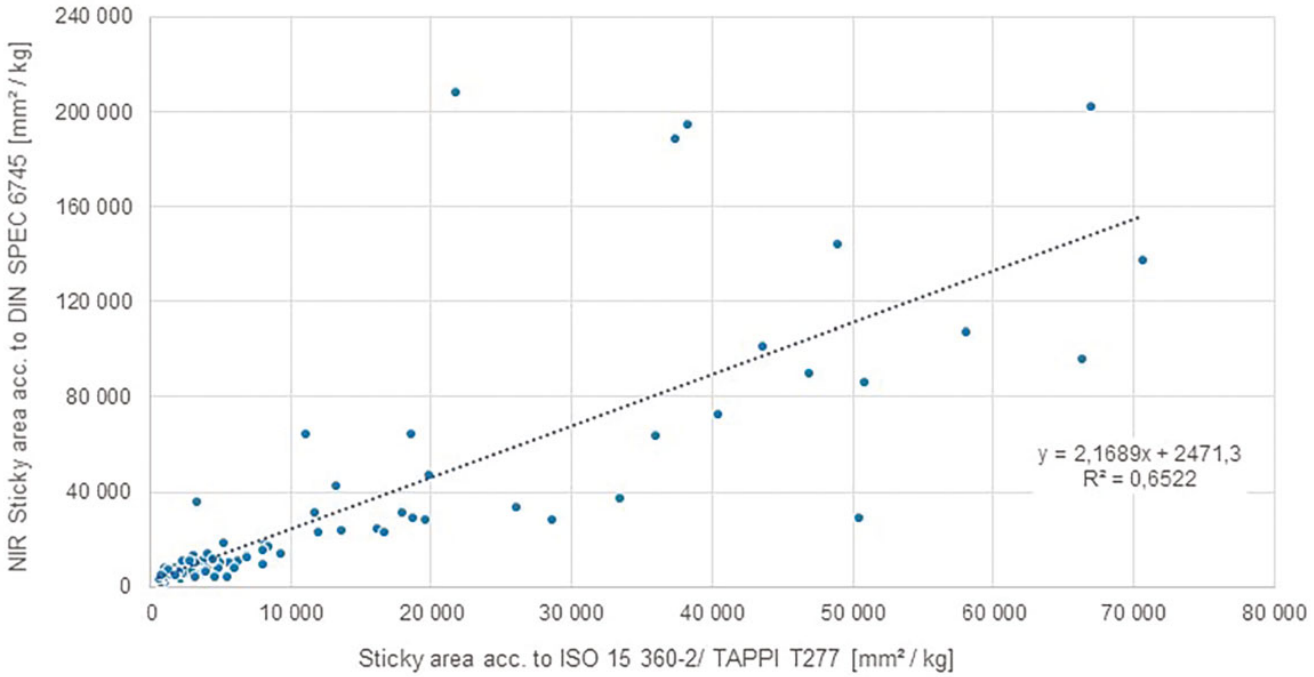


***Figure 3: Comparing sticky measurements according to ISO 15 360 - 2/ TAPPI T277 to DIN SPEC 6745 for OCC samples – where macro stickies > 150 µm are plotted for both measuring principles***

- **Repeatability** to determine sticky loads from a stock sample (within a laboratory): 18.0% maximum deviation between two results.
    - **Note:** For three different samples series (different sticky loads) that were also used for the study in Chapter 2, the complete sample preparation process was conducted three times each by different lab technicians. The calculations for the repeatability was followed as described in [5, 6].
- **Reproducibility** (between laboratories) will be elaborated in the near future in a joint project between PTS Heidenau and Voith Paper.

Due to the increased accuracy of the NIR sticky measurement according to DIN SPEC 6745, the number of measurements to be performed, e.g., during a guarantee run, can be reduced in the future. When conservatively halving the number of repetitive measurements from 12 to six samples for NIR sticky measurements according to DIN SPEC 6745, the total laboratory effort is reduced from 22 hours to five hours only at increased accuracy and reliability of the sticky measurement results.

**Comparison between ISO 15 360 - 2/ TAPPI T277 and DIN SPEC 6745 sticky measurements**

Since sticky measurements results according to ISO 15 360 - 2/ TAPPI T277 are well established in the paper industry, comparing the results of sticky measurements that are made according to the new DIN SPEC 6745 to the established ISO 15360 - 2/ TAPPI T277 may help the paper industry to better classify the new measurement results. For this purpose, more than 70 comparing measurements were conducted, where for the same stock samples, sticky values according to ISO 15 360 - 2/ TAPPI T277 and DIN SPEC 6745 were determined in parallel.

The results summarized in Figure 3, where the sticky measurement results according to DIN SPEC 6745 are plotted over the sticky measurement results according to ISO 15 360 - 2/ TAPPI T277, revealed that the sticky measurement results according to DIN SPEC 6745 were higher than results according to ISO 15 360 - 2/ TAPPI T277 for samples taken from OCC plants in Europe. Due to the high scatter of the sticky measurement results according to ISO 15 360 - 2/ TAPPI T277, the regressions coefficient of determination, $R^2$, was low as expected. The findings summarized above, impact the definition of sticky reductions as follows:


***Figure 4: Sticky reduction for same process technology due to different measuring techniques – example for OCC samples***

- Assuming a constant sticky inlet load to coarse screening of approximately 21,000 mm²/kg measured according to ISO 15 360 - 2/ TAPPI T277, and assuming that the accept quality was 970 mm²/kg for the short fiber (SF) fraction of an OCC stock preparation plant and 3700 mm²/kg for the long fiber (LF) fraction, both measured according to ISO 15 360 - 2/ TAPPI T277, the sticky reduction was 91.4% at a mass split in fractionation of 30% LF/ 70% SF, see Figure 4.
- Transferring these measurement results to DIN SPEC 6745 results using the approximation function given in Figure 3, the sticky reduction drops to 84.9%, although the process technology did not change.

Since the paper industry is striving for increased sticky reductions, the results shown in Figure 3 may require a paradigm shift to accepting lower sticky reductions due to the sticky measurements performed according to DIN SPEC 6745 compared those performed according to ISO 15 360 – 2/ TAPPI T277 for the same OCC samples.

**Summary**

In this paper, the experiences made by Voith Paper performing sticky measurements according to DIN SPEC 6745, applying the NIR measurement technique, were presented and discussed in detail. Today's common method of performing sticky measurements according to ISO 15 360-2 is well established and proven within the paper industry. However, due to the high number of manual working steps, the sticky measurement technique described in ISO 15 360-2 is prone to error. Performing sticky measurements according to DIN SPEC 6745 has several benefits that are summarized as follows:

- Reduced time and labor effort to prepare samples with increased accuracy and reliability of sticky measurement results
- Easy sample preparation by making hand sheets on globally available equipment

Since sticky measurements according to DIN SPEC 6745 differ from those values known to papermakers using ISO 15 360-2, the paper industry needs to get used to these new values and their impact on sticky reduction values on unchanged process technology. Certainly, this will require a change process; but, the benefits summarized above due to the application of the sticky measurements performed according to DIN SPEC 6745 will outweigh for the paper industry in the long term.

Besides the benefits summarized in this paper, applying the NIR sticky measurement technique according to DIN SPEC 6745 is possible for the applications described as follows:

- Determining the sticky load of final paper samples
- Studying and evaluating the recyclability of packaging papers in the future using the information about non-tacky components and stickies determined according to DIN SPEC 6745

Due to the availability of the technical rule, DIN SPEC 6745 "Paper, board and pulps - Recycling - Determination of stickies and non-tacky contaminants in recycled pulps and papers applying near-infrared measurement" and commercially available measurement equipment, measuring stickies using the NIR measurement technique has the potential to set a new standard within the paper industry.

References

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