

**ZELLCHEMING**ASSOCIATION OF CHEMICAL PULP AND PAPER
CHEMISTS AND ENGINEERSTechnical Committee for Recovered Paper Utilization (RECO)
Technical Sub-Committee for Recycling Technology (RECO 1)**1 Introduction**

Representative pulp samples taken from recovered paper treatment plants cannot always be analysed immediately after sampling. Sampling process and analysis can be several hours or even days apart, depending on whether the samples are analysed by in-house laboratories or externally. In particular recycled pulp suspension samples can undergo changes over time due to microbial activity, which can affect the results of their analysis.

The present recommendation for storage and preservation of recycled pulp samples is based on a research project funded by the INFOR Research Committee of the German Pulp and Paper Association /1-4/. The storage periods investigated in this project amounted to up to seven days for samples kept at room temperature and in cold storage, and to up to eight months for frozen samples. Experiences with the use of preservatives are based on maximum storage times of seven days at room temperature. At the time of testing, room temperatures were in the range of +22 °C ±2 °C in most cases. All experiences are based on tests done with process waters or recycled pulp suspension samples taken at approx. 1 % and approx. 4 % stock consistency from recovered paper treatment plants processing mixed recovered paper grades and sorted graphic paper for deinking for the production of corrugating base as well as graphic paper.

2 Purpose and scope

The method makes recommendations for the storage of recycled pulp suspension samples and process waters in case their physical and chemical characteristics cannot be measured directly after sampling. As a matter of principle, measurements should be taken immediately after sampling to avoid storage of the samples. In case it is still necessary to store recycled pulp samples, the method makes recommendations for storage and preservation, depending on the analyses to be performed.

3 Definitions

DIN 6735 defines recycled pulps as „pulp obtained from waste paper treatment for new paper manufacture“ /5/. Within the context of this leaflet, the term recycled pulp sample is understood to mean not only the finished pulp, but any samples of recycled pulp suspensions occurring in the treatment process as well as any type of process water occurring within the recovered paper treatment plant.

4 Principle

This technical leaflet makes recommendations for the storage and preservation of recycled pulp samples by means of thermal and chemical measures. As a matter of principle, recycled pulp samples intended for testing should be representative samples. It is therefore advisable to take samples according to the ZELLCHEMING Technical Leaflet RECO 1/2007: „Representative sampling of recycled pulp suspensions in paper mills “/6/.

If the samples cannot be measured within approx. four hours after sampling, the procedure defined by this technical leaflet shall apply.

5 Apparatus and aids

Suitable for the storage of recycled pulp samples at low temperatures are refrigerators or cold rooms as well as upright / chest freezers or freezing chambers capable of maintaining the target temperatures specified.

Chemical additives suitable for the preservation of recycled pulp samples are sodium azide, formaldehyde or commercial biocides.

6 Preparations

Any technical aids or devices required for storage must be made available and must have reached their operating temperatures before putting the samples into storage. If preservatives are used, keep the respective chemical additives ready. When using automatic sampling to obtain 24 hour mixed samples, any device-specific recommendations including cooling must be met. As a matter of principle, the storage time starts as soon as the samples are available.

7 Procedure – general aspects

If possible, avoid unnecessary extra storage times at elevated temperatures or without preservative addition.

Recycled pulp samples should be put into cold storage in suitable containers and as fast as possible. When using a freezer, activate the quick-freeze mode if available, to prevent other samples from defrosting or starting to thaw.

Preservatives must be stirred homogeneously into the recycled pulp sample, complying with all relevant safety regulations.

8 Guidelines for the storage and preservation of pulp samples

As a matter of principle, measurements should be taken immediately after sampling, if possible. If this is not possible, cold storage is sufficient in many cases to prevent major changes in physical and chemical characteristics during the first seven days after sampling. If longer storage is necessary, samples intended for COD and TOC measurements should be deep-frozen. Samples intended for organic acid detection should be kept in cold storage or mixed with a preservative (preferably sodium azide or commercial biocide). Make sure to add enough preservative, depending on the pre-loading of sample material. Samples intended for suspension and sheet property

measurements should not be frozen - this could lead to irreversible fibre changes. Storage or preservation should generally be avoided when measuring the pH, redox potential, charge distribution, precipitation potential of tacky contaminants, hydrogen sulphide content and bacterial count of recycled pulp samples – these measurements should be taken immediately after sampling, if possible. **Table 1** gives an overview of possible and preferable storage options, and of those to be avoided.

9 Sampling report

The sampling report should state the type and duration of storage and/or amount of preservatives added (see Chapter 8 in the bibliographical reference /6/), as well as any deviations from this leaflet.

10 Bibliography

- 1) Putz, H.-J. und Strauß, J.: Einfluss der Probenlagerung von Altpapierstoff auf Messergebnisse physikalischer und chemischer Kenngrößen. Final report of INFOR project 90, Darmstadt, February 2008
- 2) Putz, H.-J. und Strauß, J.: Einfluss der Lagerung von Altpapierstoffproben auf physikalische und chemische Eigenschaften. Preprint PTS Deinking Symposium, Leipzig, 2008, pp. 4-1 – 4-16
- 3) Putz, H.-J. und Strauß, J.: Einfluss der Lagerung von Altpapierstoffproben auf physikalische und chemische Eigenschaften. IPW, no. 6, 2008, pp. 87 – 97
- 4) Putz, H.-J. und Strauß, J.: Impact of the storage of recycled fibre pulp samples on physical and chemical properties. Progress in Paper Recycling, vol. 18, no. 1, November 2008, pp 16 - 23
- 5) N. N.: Papier, Pappe und Halbstoffe: Übersicht von Begriffen. DIN 6735: 2000-06. In DIN-Taschenbuch 118, Beuth Verlag GmbH, Berlin, Wien, Zürich, 6th edition, 2003
- 6) N.N.: Repräsentative Probenahme von Altpapierstoff-Suspensionsproben in Papierfabriken. ZELLCHEMING-Arbeitsblatt 1/2007 des Fachausschusses RECO vom 29.12.2007. (Representative sampling of recycled pulp suspensions in paper mills. ZELLCHEMING Technical Leaflet 1/2007 issued by the RECO Technical Sub-committee on 29 December 2007)
http://www.zellcheming.de/service/content_merkblaetter_fa16.php

Table 1: Recommendations for storage
 (if samples cannot be measured immediately)
 X = recommended storage condition
 XX = highly recommended storage condition
 --- = inadvisable storage condition
 n. d. = not done

Parameter	Storage at room temperature	Cold storage	Frozen storage	Preservative use
Temperature	20 °C to 22 °C	1 °C to 6 °C	≤ -18 °C	1 °C to 22 °C
Duration	7 days max.	7 days max.	8 months max.	7 days max.
Stock consistency	0.2 % – 5 %	0.2 % – 5 %	1 % – 5 %	4 % – 5 %
SUSPENSION PROPERTIES				
SR, WRV, flakes	X	XX	---	X
Fibre length	X	XX	---	n. d.
Macrostickies	X	XX	---	n. d.
SHEET PROPERTIES				
Tensile, tear, SCT, RCT, CMT	X	XX	---	X
OPTICAL PROPERTIES				
Brightness, luminosity	X	XX	X	n. d.
Dirt specks	---	X	X	n. d.
PROCESS WATER CHARACTERISTICS				
COD, TOC	---	X	XX	n. d.
pH, redox potential, PCD, precipitation potential	---	---	---	---
Organic acids	---	XX	X	XX
H ₂ S, bacterial count	---	---	---	---