1 Introduction

Both national laws and an increasing number of supranational recommendations are prescribing emission limits for waste-water treatment plants (WWTP). In order to be able to control maximum run-off concentrations, for instance, frequent sampling of influent and effluent at WWTP is still usual.

Furthermore, the pollution of waste water at many stages of the treatment process must be determined for purposes of influencing operation and/or control and thus optimising treatment performance, and thereby minimising costs and the use of resources, and also optimising the protection of our environment.

For this purpose, it is widely accepted today that substances and substance groups in waste waters should be monitored
1. continuously and
2. without delay (in “real time”)

This is the only way allowing quick reaction to changes in waste-water composition or operational conditions.

Online measurement in waste water using photometric instruments for individual wavelengths, a state-of-the-art technique for some years now, has proved its value – however, only following sophisticated on-site calibration, just for single parameters so far, and often prone to cross-sensitivities.

A multitude of laboratory tests has proved that even more accurate and, above all, more selective measurement can be done based on a continuous optical spectrum reaching from low Ultraviolet to visible light. However, compact, robust submersible UV/VIS spectrometers were not available for use in field and outdoor applications before now.

The brand-new s::can analysers can be directly immersed into the medium, making it possible for the first time to combine the advantages of in-situ measurement with those of spectrometry, eliminating such well-known drawbacks as sampling errors, biochemical or physical degradation, etc. The measurement quality level thus arrived at, opens up completely new vistas.

2 Parameters

2.1 Basic Information on Calibration

The s::can calibration methods, developed during many years of co-operative work with university institutes and several chemistry laboratories, make use of the most up-to-date statistical and mathematical procedures.

With respect to application, s::can can offer two different calibration strategies, viz.
1) Polynomial adaptation to known spectra.
Used successively for individual substances and sum parameters, i.e. Nitrate, Nitrite, Benzene, etc. The parameters released are characterised by their particular selectivity, and are largely free from cross-sensitivities. Thus, they are instantly and universally applicable without local calibration.

2) Should the spectral information available be insufficient, the device immediately accesses the “global” calibrations stored, based on advanced statistical analysis of s::can’s large data bases. Although they are just as precise, they are not as selective as with the first method. Comparable selectivity can be achieved, however, with following precise local calibration and reference lab analysis.

2.2 Organic Carbon Compounds

The parameters for assessment of organic carbon loads (COD, BOD5, TOC, solids, etc.) constitute sum parameters whose measurement procedures evince various drawbacks: reproducibility, comparability, measurement ranges, cross-sensitivities, use of chemicals, etc. They cannot be considered as absolutely “true” parameters. For this reason, increased interest has been shown in the past few years – by privates and authorities as well – in substitute measurement methods, if they provide the great advantage of time-continuity.

In terms of its methodical approach, spectrometry features the advantage of not indirectly measuring oxygen demand, but rather of directly measurement the concentration of oxidable organically bound carbon.

Standard deviation of COD concentrations from spectral measurement show substantially less dispersion than those measured in the laboratory; the uncertainties pertaining to the COD (above all in the lower concentration region) are well known – they are even intensified by the usual application of standardised batch tests at WWTPs. Thanks to the lower dispersion of measurement values (when determined via UV/VIS spectrometry), a lower detection limit can be achieved.
Figure 1: COD – Comparison of spectro::lyser\textsuperscript{TM} with standard laboratory measurement.

Independent laboratories determined the correlation between laboratory COD (total) and the instrument measurement values over a measuring range of 20 to several 1000 mg/l at different measuring points of so far 16 WWTPs. Depending on the local calibration, the correlation coefficient $r^2$ was always between 0.78 and 0.98 (cf. chapter 8, and following example pages).

Similarly, the calibration to filtered samples always yields a good and independent correlation.

The estimation of biodegradable carbon (BOD\textsubscript{5}) using spectrometry is a particularly suitable method. Often a specific spectral range can be related to small-molecular, easily degradable substances.

### 2.3 Solids, Turbidity

The linear regression between solids concentration and the spectral parameter yielded good results ($r^2 = 0.85 - 0.99$). This good correlation was confirmed at all plants tested so far.

Figure 2: Comparison of solids\_eq with lab analysis

### 2.4 Nitrate

Regressions between nitrate concentrations and spectral extinction parameters similarly yield high correlation coefficients.

The spectral Nitrate measurement is extremely robust. Opposite to single or dual wavelength photometry, it works well under most conditions, including the aeration tank, and is much less cross-sensitive to organic carbon and turbidity.

Figure 3: Continuous measurement in the aeration tank

Figure 4: Comparison with laboratory values
2.5 UV/VIS Absorbance Spectrum

The continuous UV/VIS absorbance spectrum features two substantial advantages:

1. Nitrate, turbidity and organic substances can be measured simultaneously
2. Qualitative assessment of waste-water composition is possible

Figure 5: Absorbance spectra of waste-water samples at various measurement points

Figure 5 shows the absorbance spectra in the influent of a WWTP, which is strongly influenced by an industrial paper mill. The peak at the wavelengths around 280 nm reflects degradable organic matter (BOD). Due to biological degradation, it decreases successively from the influent (blue) over the intermediate treatment (green) to the effluent of the after-treatment (red). In this instance, measurement of the SAC 254 alone is inadequate for determining the concentration of BOD compounds since, at 254 nm, absorbance correlates almost exclusively with solid matter (turbidity), but there is almost no selective relationship with BOD at 254 nm.

Furthermore, dissolved carbon compounds can only be measured safely after spectral compensation of turbidity. The traditional way of compensation by measuring only one additional wavelength in the visible range is very often not suitable for this purpose because of the changing character of particles and color.

Using local calibration, various carbon groups can be distinguished by specific features in the fingerprint, in particular as regards their degradability, allowing conclusions to be drawn with respect to the treatment process. Depending on the properties of the waste water, correlation coefficients to the BOD of 0.75 to 0.9 can be anticipated. On-site calibration covering the waste water under consideration leads to correlations of \( r^2 = 0.8 \) to 0.9. Evidently, simple photometric devices are not capable of identifying characteristics of this type.

3 Advantages of Process Spectrometry in Comparison with Simple Process Photometry

Spectral determination of water-quality parameters features the following advantages:

- **Cost efficient - multiparametric**
  Even at minimum specification, the continuous UV/VIS spectrum enables simultaneous measurement of the parameters of organic carbon, nitrate and turbidity, for which only one s::can spectrometer is required, instead of merely three simple process photometers.

- **Lower cross-sensitivity on turbidity, coloration, surface growth, etc.**
  Potential interference, not detectable by dual-wavelength measurement, can nearly always be compensated with the help of spectral information.

- **Greater precision**
  Since cross-sensitivity is substantially reduced by this means, heterodyning of measurement signals due to interference/noise is significantly less than with simple photometer devices.

- **Higher selectivity**
  Many individual substances and/or substance groups can be allocated to significant spectral features.

- **Greater reproducibility**
  These advantages result in very high reproducibility as well as the benefit of universal applicability, without the absolute necessity of local calibration.

- **Calibration matching other substances and substance groups**
  Aside from the parameters usually applied in waste-water engineering (COD, BOD, DOC, nitrate, turbidity), s::can offers individual parameter calibrations using proprietary advanced chemometric tools.

- **Qualitative evaluation**
  In addition to the calibrated parameters, the qualitative spectral information (“fingerprint”) can be directly applied for alarm and control systems, etc. Qualitative and quantitative differentiation of carbon fractions (for more detailed assessment of the waste water, e.g. from industrial charges) is possible.
4 Special Advantages of the s::can Measuring Instruments

- Measurement dynamics
A special feature of s::can spectrometers is their extreme dynamic range (1:1000 or (0) 1 to 1000 mg/l for COD_eq, up to 1: 5.000 for Nitrate). With the same configuration, measurements can be taken in the sewer, influent, intermediate / after-treatment and effluent, as well as in water bodies. Only when measuring nitrate in the influent and aeration tank a smaller path length is required.

- Long-term stability
Dual-beam measurement enables all changes in the measurement system to be captured and compensated. Together with spectral compensation of biological growth on windows and/or turbidity fluctuations, the dissolved substances can be measured with exceptional long-term stability, enabling continuous monitoring of waste-water quality and the operation/control of the treatment process.

In cases of high solid-matter content, the (hydraulic) instrument-cleaning unit is connected via an external pressure line. With the help of this extremely effective device, measurements of many months without ever removing the probe from the water are normal.

- No maintenance necessary
Maintenance is not even possible because there are no serviceable parts on the hermetically sealed instrument. When no pressure cleaning unit is in use, deposits or biological growth can build up on the windows, although the high-energy UV radiation prevents most organisms to grow up. Windows need merely be cleaned from time to time, enabling the user to establish the intervals between cleanings himself.

- Field and Outdoor Use
UV/VIS spectrometry technology to this date has never been available in such a compact design, enabling the use of the spectro::lyser™ even in tight spatial situations. Owing to the lightweight, robust design (all surfaces are anodised), and the absence of moving components in contact with the medium, as well as due to the 12-volt supply and explosion shielding, the instrument is also suitable for use under rough or hazardous environmental conditions.

- Flexible measurement process
Using intelligent algorithms, microprocessors adapt the hardware (exposure time, oversampling, etc.) and the measurement parameters to changing conditions.

5 Application for the use of s::can spectrometer in waste water

5.1 Assessments and parameters

- Measurement of UV/VIS spectra over a wavelength range of approximately 200 – 750 nm
  - compensation of turbidity
  - simultaneous determination of various substances
  - differentiation between various carbon fractions
  - detection of irregular charges

- Calibration for the following parameters:
  - organic substances (calibration for T(D)OC, COD, BOD₅ and/or SAC 254, SAC280, SAC330, SAC500)
  - solids
  - nitrate

- Ease of use
The user remains unaffected by complexity of the measurement process, i.e. he doesn’t need to manipulate the process and he merely receives the definitive measurement figures.
5.2 Application and Installation

- **Selection of measuring path length** depending on matrix and substance concentration
  - 2 mm or 5 mm in the influent
  - 1 mm in the aeration tank
  - 5 or 35 mm in the effluent

- **Evaluation** if automatically hydraulic window-cleaning is necessary, using pressurised water or air
  - applications with need of very long (several months) maintenance intervals
  - in cases of increased biological activity in the waste water, or higher fat contents
  - also operational in the sewer system when used in combination with compressed-air or CO2 bottles

- **Recommended measurement points**
  - behind the rake unit and/or sand tank
  - before the return sludge feed
  - before discharge

- **Installation**

  For purposes of installation in treatment plants, we recommend our simple Aluminum or PVC installation pipes w. brackets.

  The floating pontoon of carbon-reinforced glass-fibre material is an advanced option, the practical hydraulic design ensures that no clogging or the like can form.
6 Additional applications and features of s::can measuring systems

6.1 System integration
1. Our interface terminal con::stat allows total system integration and most advanced visualisation and provides analogue and digital interfaces to all kind of other sensors and control systems. Complete monitoring stations can be programmed to completely meet customer’s needs concerning visualisation, process control and data exchange.

2. The instrument can be operated and read with any commercial notebook/pc computer via the USB-interface and our proprietary software.

3. The instrument is also operable in data-logger mode without any additional periphery. However, no continuous data access is possible in this mode.

4. In addition, s::can offers a waterproof field-case (accumulator) for stand-alone operation in the field.

6.2 Operation and data transfer
- Measured values can be integrated into automated processes (operation, control, alarm) via various standard interfaces or databases.

6.3 Simultaneous water-level measurement
- Sensor for pressure-measurement integrated in the analyser and the software.

- Estimation of flow-through if level-flow relation is known

6.4 Simultaneous temperature measurement
- Sensor for temperature measurement integrated in the instrument and the software

6.5 Integration of other sensors
- s::can offers sensors for pH, redox and conductivity that are completely integrated into our measuring systems.

- Sensors for O2 and Ammonium can also be integrated in co-operation with other suppliers.

6.6 Measurement in sewer-systems
- indirect charge control

- identification and monitoring of charges: the continuous spectrum enables distinction to be made among waters based on differences in their composition, even following irregularities in the overall influent spectrum. References available upon request.

- Exceeding of limit values via charge alarm

- Control of waste-water load during rainwater run-off prior to release into the discharge

- Load-dependent sewer management

6.7 Networking of multiple devices
Multiple measurement points can be united wireless to a centrally operable network. Together with its associates, s::can supplies the complete equipment.
7 Some Reference Measurements

(References from other plants are available upon request)

The spectro::lyser™ has been running for 2 months so far without any maintenance, without ever touching the instrument, and no trend or drift can be seen so far. The daily and weekly periods, and also a weak long term trend in most parameters can be seen and was proven by lab measurements. The parameters were checked frequently by laboratory measurements: the correlations \( r^2 \) were better than 0.85 for all parameters without any local calibration. After local calibration, all correlations \( r^2 \) are between 0.9 and 0.98. This shows that the parameters Nitrate, COD_homogenised, COD_filtrated, and Total Solids can be measured in the influence of a WWTP with one instrument only and stable over many months.

Figure 6: Time series in the influent of a large WWTP.

Figure 7: Zooming into one week of this period, the fine resolution of measurement and the independence of the parameters can be evaluated. The clear daily period as well as a weaker weekly period can be distinguished.
The time series of the suspended solids and COD equivalents in the influent (cf. Figure 8) show distinct daily maximums during the late afternoon, which are barely prominent with the filtered COD-filt_eq. The measurement sequence shows that the majority of the COD load is fed into the plant in solid form.

![Figure 8: Time series in the aeration tank influent, WWTP A](image)

In the aeration tank influent (Figure 8), the maxima are not visible until night-time, due to the delayed effect of the pre-treatment. The distinct decrease of the COD_eq., solids content and SAC254 on September 17, 2000 is due to the weekend in between. As a measure of the dissolved organic substance, the SAC254 once again shows a significantly lower dynamic behaviour.

![Fig. 9: Time series of turbidity in the influent of the aeration tank of municipal WWTP A, in comparison of 3 instruments](image)

The time series were measured within the extended testing programme of a large treatment plant which aimed at the evaluation of all different kinds of online instruments before the complete re-build of the plant. The spectro::lyser™ convinced the test staff not only in case of solids, but also for organic carbon and Nitrate measurements. More details available upon request.
In figure 10, a series of COD_eq is printed against time, as well as some laboratory reference measurements.

The correlation was better than expected, because the specro::lyser is situated directly behind the screen, and the disturbance from the screen was high. There has been no drift over the measuring period so far, and the correlation to COD is linear over the whole range which is typical for regular municipal waste water.

When studying the daily sequences more closely in figure 11, the multifarious occurrences (not perceptible using conventional measurement methods and/or measurement solutions) can be observed. The peaks and curves visible here are entirely interpretable and explicable.
The time series depicted in Figure 12 show that dynamic behaviour also occurs in the concentrations of both nitrate and COD_eq. in a WWTP effluent. Thus, continuous monitoring of loads would be advantageous in terms of logging limit exceeds and minimising loads.

In figure 13 / 14, the Nitrate series in the effluent of WWTP A are printed on a weekly and daily basis, this time compared to the results of a batch cabinet analyser (colorimetric).

The much higher measuring resolution, but also less frequent irregular peaks at the spectro::lyser™ data are clearly evident.
8 Parameter ranges for municipal waste waters

Global calibrations of s::can G::series (nitro::lyser, carbo::lyser & multi::lyser)

### Global calibrations of nitro::lyser

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**Näherungswerte wegen kleinen Messwege

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*Basierend auf den gesammelten Wassermatrizen der gegenständlichen "Globalen Kalibrierung".

*Based on the collected water matrices of the described "global calibration".

Prazision gleich der zugehOgeren Untergrenze des Bereichs (z.B.: Anwendung Belebung, Parameter NO3-Neq Prazision +/- 0,1 mg/l)

Precision equals corresponding lower limit of monitoring range (e.g.: application aeration, parameter NO3-Neq precision +/- 0,1 mg/l)

Global calibrations" are standard spectral algorithms that facilitate the easy local calibration on site under the conditions of typical applications. We can provide or develop additional "Global calibrations" for s::can spectro::lyser™, i.e. for industrial applications, and periodically release new ones. Please ask our engineers if we can provide another standard solution for your measuring task if the listed data does not meet your application!

Cross-sensitivity, measuring range & precision always depend on the character of the matrix. Local Calibrations will change cross-sensitivity, measuring range and precision. Precision can be improved by oversampling with n°0,5 measurements.
9 Some selected references

s::can references Q2/2005

List not complete - selection of s::can systems sold to customers; no trials listed;

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*customer data available