# Methods for Sampling and Analyses at Lake Tegel

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Note: for methods of phytoplankton analyses, see files "phyto LLBB 2007 ff" and "phyto UBA 1987-2006"

## Sampling

**UBA (1984 – 2006):** Sampling was at monthly to weekly intervals (depending on years and additional research programmes) at depth intervals of 0.5 – 1 m using an underwater pump and a plastic tube. After passing a Plexiglass cuvette which held the probes for temperature, oxygen, pH and redox the water gently flowed into the sample bottles: 1 L brown glass bottles for Chlorophyll-a, 250 ml glass bottles for phosphorus and 250 ml glass bottles for nitrogen analysis.

Depth integrated phytoplankton samples were integrated *in situ* and 100 ml filled into a brown glass bottle pre-stocked with Lugol's solution. For species determination and qualitative assessment, 1 L was filtered through a plankton net (10  $\mu$ m mesh size) and the concentrate stored in a 10 ml tube pre-stocked with formaldehyde.

During 1987 - 1990 the depth horizon for integration was determined on the basis of temperature profiles (typically 4-6 m; due to aerator-induced turbulence the metalimnion was often not clear-cut). During 1992 – 1994 samples were taken from 2 m-depth exclusively, assuming this depth to be representative for the epilimnion. During 1995 – 2006 depth-integration of samples was standardized down to 5 m. Exceptions are marked in italics and blue in the phytoplankton file.

**LLBB (since 2007):** Monthly sampling is done with a LIMNOS sampler at discrete depth intervals. For depth-integration of the biological samples these were gently mixed in a bucket and filled into bottles of 100 mL (phytoplankton) and 1-2 L volume (chlorophyll a, phaeopigment a, diatoms) respectively, depending on Secchi depths (if Zs > 1.5 m the sample volume was 2 L for the determination of chlorophyll a and diatoms). Lugol's iodine solution was added to phytoplankton and diatom samples in an amount that achieves a cognac colour of the sample.

The sampled depth-horizon follows the scheme given in Nixdorf et al. (2010) regarding stratification: In phases of vertical mixing, equidistant sampling was down to  $Z_{mean}$  (7.6 m) and during stagnation it was down to the 2.5-fold Secchi depth or down to the metalimnion, respectively, depending on which zone was more pronounced.

### Secchi Disc Transparency

**UBA and LLBB:** measured by lowering a white disc of 20 cm diameter to the point where it is just barely no longer visible, taking care not to wear sunglasses and to take the reading in the shade of the boat to avoid reflection.

#### Temperature, oxygen, pH:

**UBA:** depth profiles were registered at depth intervals of 1 m (sometimes 0.5 m) with an oximeter Oxi 96 (WTW, Germany).

**Oxygen for primary production profiles** was determined with the Winkler titration method.

**LLBB:** depth profiles are measured with a multiparameter probe, e.g. YSI, following standard methods: for temperature DIN 38404 - C4-2 :1976 (LoQ 0.1 °C), for

oxygen DIN ISO 17289-G25:2014 (LoQ 0.1 mg/L); for pH DIN EN ISO 10523-C5:2015 (DIN 38404-C5:2012-04).

# Chlorophyll-a

- **UBA:** ISO 10260 (1992; also DIN 38 412, part 16, 1985) by filtering 1 L of water (less if phytoplankton was very dense) on glass-fibre filters, stored frozen, extracted with simmering ethanol, extracted in the dark for 24 hours and centrifuged at 4000 rpm for 20 minutes. Absorbance was determined with an Elko II, Filter J 67.0 at 667 nm, Carl Zeiss, Germany (successful participation in ring test 1997). Because of occasional negative values for phaeophytin given data were measured as total pigment concentration and not corrected for phaeophytin (multiplying the extinction by the factor 12.2 instead of 29.6 (following Nusch 1975).
- LLBB: DIN 38 412-L16:1985. Water samples were filtered using glass-fibre filters. The filters were immediately frozen and extracted with simmering ethanol (90%) for 6-24h. Extracts were clearified by filtration and absorbance readings were done with a 2-beam spectrophotometer (UNICAM, Evolution ™ 160). For the differentiation of chlorophyll a and phaeopigment a an aliquot oft the extract was acidified according to the standard procedure. LoQ = 2 µg/L

### Nitrate:

- **UBA and LLBB:** DIN EN ISO 10304-1 Water quality Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007); limit of quantification is 0.5 mg/l NO3-N
- **UBA:** For selected samples, we also used commercial test kits (Dr. Lange, Germany) with pre-stocked cuvettes and a Lasa® plus pocket photometer with a reaction based on indophenol blue formation for ammonium (LoQ of 0.015 mg/L) and on 4-nitro-2-6-dimethylpehnol formation for nitrate (LoQ 0.23 mg/L).

# Nitrite

LLBB: DINENISO 10304-1-D20:2009; LoQ 0.01 mg/L

# Ammonia:

**UBA:** Analysis performed by the Berlin Waterworks following the German standard methods for the examination of water, waste water and sludge; cations (group E); determination of ammonia-nitrogen DIN 38406 E 05; Photometer CADAS 100, Dr. Lange; limit of quantification is 0.015 mg/l NH4-N.

LLBB: DIN EN ISO 11732 - E23:2005; LoQ 0.02 mg/L

# Phosphorus (total and soluble reactive fractions)

**UBA:** P was first determined with molybdene blue (after hydrolysis of TP with potassium peroxodisulfate solution and 30 min. of digestion under pressure at > 100 °C) following the method by Koroleff (1983) which later became the ISO 6878 (which in turn is the basis for the DIN EN 1189). Analysis in a 5 cm cuvette and tightly correlating calibration curves enabled a limit of quantification of 1  $\mu$ g/L P.

Some months in 2003 (marked in the respective data files) were an exception: these analyses were with malachite green, following Motomizu. This limit of Quantification of 5-10  $\mu$ g/L was, however, insufficient in face of declining concentrations of TP and very low SRP.

LLBB: DINENISO 15681-1 D45:2005 with digestion following ISO 6878-D11:2004; LoQ 0.01 mg/L

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LLBB: DIN EN 1484-H3:1997; LoQ 0.3 mg/L

#### Silicate

UBA: (only 1994-1999): we followed the IGB's adaptation of the DIN EN 1484 (DEV, H3)

LLBB: DIN ISO 15923-1:2014 (D49), LoQ 0.5 mg/L

#### **UV-extinction**

**UBA:** measured in a photometer at 254 nm

## References

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