

II. Water Quality

6. Heavy Metals

6-1. Heavy metals: Cu and Fe (by Kazuho Inaba) * Currently this item is not measured.

1. Measurement methods

(1) Sampling and pretreatment

Monitoring of copper and iron was performed monthly at Sts. 1 and 9 (since April 1989 for copper, since June 1989 for iron), St. 3 (since April 1991), and Sts. 7 and 12 (since April 1994) in Lake Kasumigaura. Data for March 1991 were eliminated because of contamination.

Surface water at each station was collected directly into a 1L stoppered polypropylene bottle, which was rinsed with 6 M nitric acid and ion-exchanged distilled water before use, and brought back to the Institute in an icebox. The samples were filtered with a 0.45- μm membrane filter, ultipor N₆₆ (Pall Trinity Micro Co.), as soon as possible. The filtered solution was placed into 30 mL stoppered polypropylene bottle; the solution was stocked in a refrigerator after being acidified by the addition of 0.5 M analytical grade nitric acid. 500 mL of each sample was filtered in order to minimize the effect of contamination from the apparatus used.

(2) Analysis

The amounts of copper and iron in the sample solutions were determined using an atomic absorption spectrometer using absorption lines at 248.3 nm for iron and 324.8 nm for copper with deuterium background correction. Usually, 10 μL of the sample was injected into the graphite tube, but several tens of μL of the sample was used when the metal concentration was low. The standard temperature-control program recommended by the manufacturer was utilized for the determination. The amounts of copper and iron in the samples were calculated using calibration curves obtained by sets of standard solutions containing up to 10.00 $\mu\text{g/L}$ copper and up to 100.0 $\mu\text{g/L}$ iron. The measurements were conducted at least twice for each sample and if significant errors were detected, the measurement was repeated. Mean values and deviations for each measurement are shown in data.

2. Measuring equipments

Before July 1999: Z-5100A (Perkin-Elmer)

Carbon furnace atomic absorption spectrometry using deuterium light source

Amount of samples injected into carbon furnace was 10 μL .

After August 1999: AAnalyst-600 (Perkin-Elmer)

Carbon furnace atomic absorption spectrometry using Zeeman polarimetry

Amount of samples injected into carbon furnace was 20 μ L.

6-2. ICP (Main Elements)

(by Masataka Nishikawa, Reiko Kumata, Masayo Okawa and Kazuhiro Komatsu)

1. Measurement methods

(1) Sampling and pretreatment

Samples were filtered through glass fiber filters (GF/F, combusted under 400°C before use) immediately after being taken to the laboratory from the sampling site. The filtered samples were stored in a cool and dark room until ICP-AES analysis. This procedure allows measurement of dissolved elements in the surface water of Lake Kasumigaura.

(2) Analysis

The following 25 elements have been measured; Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, Se, Si, Sr, Ti, V and Zn. Only the following elements were quantitatively determined during the measurement period; Al, B, Ba, Ca, Fe, K, Mg, Mn, Na, Si, and Sr. Therefore, we continue monitoring of these 11 elements. The determination limits were calculated as 15 times of the standard deviation of the “zero point” standard, which was obtained by repeat measurements ($n = 10$) of distilled water. The values below the determination limits are shown as 0.0 or 0.00 in the table of the monitoring results, for convenience. In order to compare with the high accuracy measurements of JP-48P, Al and Fe values were determined with the other instruments (before October 1992, and after January 1995) and these are given in parentheses up to the half of nominal determining accuracy.

2. Measuring equipments

ICP-AES

1980 - 1992: Plasma Atomcomp 975 (Jarrell-Ash)

1992 – 1994: JY-48P (Daini Seiko-sha)

Since 1995: ICAP-750 (Japan Jarrell-Ash)

Instrumental and operator errors causing variation in the analytical conditions and outcomes were minimised by utilising in-parallel measurements for six months when an instrument and/or an operator was changed over.