Procedure for determining strontium-90 in fish

G-Sr-90-FISCH-01

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1 Scope

The procedure described here is suitable for determining the specific Sr-90 activity in samples of fish (fish meat and whole fish) that are to be investigated according to the Precautionary Radiation Protection Act (IMIS routine programme). It may also be employed for monitoring Sr-90 in fish meat according to the Guideline for the Monitoring of Emissions and Immissions of Nuclear Installations in Regular Operation.

During this procedure Y-90 is extracted from the sample as representative for Sr-90, which requires that Sr-90 and Y-90 are in the state of radioactive equilibrium.

2 Sampling

For sampling fish, it is referred to procedure G-γ-SPEKT-FISCH-01.

The sample volume is determined by whether fish flesh or whole fish is to be analysed. If the sample does not consist exclusively of deboned fillet, but also contains skin and bones, a Sr-90 content must be expected that is larger by at least one order of magnitude, because strontium is accumulated mainly in bones and skin. For a Sr-90 analysis of fish flesh, an original sample comprising 2 kg to 5 kg in fresh mass should be available. If whole fish is to be analysed, a sample of 1 kg in fresh mass may suffice.

3 Analysis

3.1 Principle of the procedure

Following the ashing of the sample material and after a radioactive equilibrium between Sr-90 and its daughter nuclide Y-90 has been reached, an extraction procedure is applied to separate the Y-90 content, which is then transformed to an yttrium oxalate precipitate that will be suitable for beta counting. Determining the chemical yield of yttrium then makes use of complexometric titration. Measuring the Y-90 activity, of which will be assumed that its activity is equal to that of Sr-90, is effected with the aid of a low-level beta anticoincidence measuring instrument.

3.2 Sample preparation

For preparing samples including their ashing, see procedure G-γ-SPEKT-FISCH-01.

3.3 Radiochemical separation

The separation procedure described in the following is designed for processing a maximum of 100 g of ash, and can be applied to substantially smaller original volumes without changes.

3.3.1 The fish ash is dried at 110 °C for one hour and stored in exsiccate awaiting the analysis to commence.

3.3.2 50 g of whole-fish ash or 100 g of fish-flesh ash, as may be the case, are weighed in in a 1000 ml-glass beaker (tall shape) and moistened with distilled water.

3.3.3 The sample is then fortified with the following carrier solutions (for their preparation, see section 7.1):

5,0 ml of yttrium carrier solution (50 mg Y^{3+})
1,5 ml of strontium carrier solution (60 mg Sr^{2+})
2,5 ml of barium carrier solution (50 mg Ba^{2+})
2,0 ml of caesium carrier solution (60 mg Cs^{+})

Strontium, caesium and barium serve as retention carriers.

3.3.4 The moistened ash is fortified with 1 ml of hydrochloric acid (12,1 $mol \cdot l^{-1}$) per g of ash, heated to boiling for 30 minutes and cooled afterwards.

3.3.5 Together with distilled water (1 ml per g of ash), the reaction mixture is filled into a suitably large centrifuge tube and centrifuged. The clear solution is collected in a 1000 ml-glass beaker.

3.3.6 The residue suspended by adding 1 ml of hydrochloric acid (6,1 $mol \cdot l^{-1}$) per g of residue and centrifuged. This step is repeated. Each time, the supernatant solution is added to the solution obtained in step 3.3.5.

3.3.7 The combined solutions are adjusted to a pH of 1 to 1,5 with the aid of a solution of caustic soda. Here it must be ensured that no salts are precipitated. A slight degree of turbidity is acceptable.

3.3.8 The cooled solution (pH 1 to 1,5) is transferred to a sufficiently large separation funnel (typically 1000 ml) and mixed with 100 ml of bis-(2-ethylhexyl)-hydrogen phosphate (HDEHP, 0,45 $mol \cdot l^{-1}$) in n-heptane.

3.3.9 Date and time are recorded (reference point of time for Y-90), and the separation funnel is shaken for about 10 minutes.

3.3.10 The aqueous solution (lower phase) is separated and discarded. The organic phase is purified five times with 100 ml of hydrochloric acid (1 $mol \cdot l^{-1}$) each for 1 minute. The purifying solution is discarded.

3.3.11 The yttrium is re-extracted by liquid-liquid-extraction five times with 50 ml of hydrochloric acid (9 $mol \cdot l^{-1}$) each. The five hydrochlorated extracts are collected in a 500 ml-separation funnel. The organic phase that has been liberated of its yttrium content is purified with 50 ml of distilled water or a diluted solution of caustic soda (ca. 1 $mol \cdot l^{-1}$) each in the sequence water, caustic soda solution, water and then transferred to a collection container for organic waste solutions.

3.3.12 The hydrochloric acid solution is purified with 250 ml of trioctyl methyl ammonium chloride (ADOGEN 464) in toluol for 10 minutes. The aqueous phase should be colourless thereafter.

3.3.13 The aqueous (lower) phase is then transferred to a 1 l-glass beaker prefilled with 200 ml of distilled water and heated afterwards.

3.3.14 Using ammonia solution (13,4 $mol \cdot l^{-1}$), it is neutralised and adjusted to a pH of about 9. The yttrium hydroxide contained will cause some turbidity. It is heated until it starts boiling and cooled in an ice bath.

3.3.15 Once cooled, the largest portion of the solution is decanted and the remaining solution with the yttrium hydroxide precipitate is transferred to a centrifuge glass.

3.3.16 Following the centrifuging and discarding of the supernatant solution, the precipitate is dissolved in a few drops of nitric acid (7,2 $mol \cdot l^{-1}$). About 20 ml of distilled water are added, and the yttrium is precipitated as oxalate by adding 25 drops (about 3 ml) of saturated oxalic acid solution under heating.

3.3.17 To verify that the precipitation process has been complete, a few drops of ammonia solution (13,4 $mol \cdot l^{-1}$) are added until a pH of 1,5 is reached.

3.3.18 The solution is left to cool, while the precipitate sedimentates, and is then filtered by means of a Hahn's suction apparatus on a blue-band filter (weight has been noted before!). A little distilled water and methanol are used for flushing the glass beaker.

3.3.19 The precipitate is dried at 110 °C for 30 minutes, weighed, covered with Mylar foil, and prepared for measuring on the counting source carrier. The determined weight is used to calculate the area fill density in $mg \cdot cm^{-2}$.

3.3.20 Subsequent to measuring, the chemical yield of the yttrium separation needs to be determined. Gravimetrically determining the yttrium oxalate will be too imprecise, as oxalic acid will usually be co-precipitated with the precipitate. The yttrium yield is therefore determined by complexometric means. This requires that the yttrium oxalate precipitate is dissolved. Therefore, the precipitate together with Mylar foil and filter paper are dissolved in a warm solution in a 300 ml-Erlenmeyer flask with 10 ml of titriplex-III solution (0,1 $mol \cdot l^{-1}$) and 20 ml of borate buffer (0,1 $mol \cdot l^{-1}$).

3.3.21 A spatula's tip of eriochrome black-T indicator (rubbed with NaCl 1:99) is added and titrated with zinc sulphate solution (0,1 $mol \cdot l^{-1}$) to the transition point from blue to violet.

3.3.22 For comparison, 50 mg of yttrium from the carrier solution are fortified with 10 ml of titriplex-III solution (0,1 $mol \cdot l^{-1}$), 20 ml of borate buffer (0,1 $mol \cdot l^{-1}$), and a spatula's tip of indicator, and titrated with zinc sulphate solution (0,1 $mol \cdot l^{-1}$).

3.3.23 The chemical yield of yttrium is calculated according to a rule of three from the consumption of titriplex-III solution (0,1 $mol \cdot l^{-1}$) in the counting source with its known yttrium content (step 3.3.22) and in the counting source with its unknown yttrium content (step 3.3.21); see section 5.

4 Measuring the activity

The description of the measuring procedure provided here in sections 4 and 5 is more extensive that would actually be necessary for fish meat alone so that it can also be applied in the procedures G-Sr-90-SCHAL-01 and G-Sr-90-KRUST-01. Interferences by co-precipitated radionuclides must be expected in particular in the analysis of flesh of the common mussel (G-Sr-90-SCHAL-01), whereas these will typically play only a less important role in the case of fish meat.

Measuring the Y-90 oxalate precipitate makes use of a low-level beta anticoincidence counter whose central detector is run in anticoincidence mode with a guard detector. For further reducing the background effect, the detector system is surrounded all around with 20 cm of steel so that count rates of the background effect of less than $8 \cdot 10^{-3}$ s^{-1} are achieved.

Measuring the Y-90 counting source may be disturbed by co-extracted radionuclides (e. g., Ac-228, cerium isotopes, radon products). In particular, the first measuring following the preparation of the counting source may be influenced by co-precipitated Ac-228 (half-life 6,13 hours) or radon products from the ambient air that might have accumulated in the counting source during the filtering process by a Hahn's suction apparatus (see step 3.3.18 in section 3.3).

If cerium isotopes have been co-precipitated, these may now cause an increased net count rate that can be identified with certainty as an external contribution only if the counting source is re-measured a few days later.

The following procedure is therefore suggested:

The Y-90 counting source is measured three or four times at intervals of about 1 to 3 days, applying measuring periods of 30000 s or 36000 s each. Recording such a decay gradient serves mainly the following goals:

1. In the case of an imperfectly homogeneous and symmetrical (ideally circular) precipitate, repeatedly mounting the counting source in the detector will cause any position-dependent count rate to be averaged. The average count rate measured will therefore approximate that of a perfect counting source more closely than a single measurement.
2. If the decrease in the count rate does not occur as expected may be indicative of the presence of interfering impurity activity, as has been mentioned above.

The recorded decay gradient (e. g., illustrated graphically) serves to assess the presence of disturbances. If assessing the graphical illustration or a linear regression does not provide evidence for such a disturbance, the net count rate of the first measurement can be used for calculating the Sr-90 activity. If an inflated net count rate is found to be exclusive to the first measurement, the Sr-90 activity can be specified from the net count rate of the second measurement (the first measurement is then discarded). If, on the other hand, the measured count rate decreases significantly slower throughout all three or four measurements than could be expected according to the half-life of Y-90 (64,10 h), an impact of longer-lived radionuclides has been established. The contribution of the Y-90 contained therein can then be quantified only, if it is not too small relative to that of the disturbance, by means of linear regression, which will require employing software with the necessary capabilities (1). If such software is not available, it is recommended to repeat the radiochemical analysis.

For calibrating the measuring instrument for the oxalate precipitates, an Y-90 calibration source is produced with the aid of a Sr-90 solution that is in radioactive equilibrium and whose exact activity needs to be known. This requires that the Y-90 be separated by means of hydroxide precipitation, upon which the hydroxide is re-precipitated for purification. Finally, the Y-90 is precipitated as oxalate and measured.

The fill density per area of the calibration source must correspond to those of routine samples. In order to reduce the impact of random errors (chemical yield, preparation of the precipitates, variation in efficiency), several counting source are to be used for calibration. The chemical yield of Y-90, $\eta_{\text{Y-90}}$, is determined by complexometric means after measurement.

For monitoring the detector efficiency in the long run, a calibration source is employed that contains Sr-90 and Y-90 in radioactive equilibrium.

The count rate of the background effect is derived from a larger number of measurements taken from various inactive yttrium oxalate precipitates.

5 Calculation of the results

Supposing that ($R_i(t_i) = R_i$; $i = 1,2,3$ or 4) are the *net count rates* of in total 3 or 4 individual measurements taken of the Y-90 decay gradient, the regression equation reads as follows:

$$R_i = \omega_1 + \omega_2 \cdot f(t_i) \tag{1}$$

and the decay factor, $f(t_i)$, valid for measurement i, is calculated according to equation 2:

$$f(t_i) = f(t_i, t_\text{m}, \lambda_{\text{Y-90}}) = \frac{e^{-\lambda_{\text{Y-90}} \cdot t_i} \cdot \left(1 - e^{-\lambda_{\text{Y-90}} \cdot t_\text{m}}\right)}{\lambda_{\text{Y-90}} \cdot t_\text{m}} \tag{2}$$

in which:

t_i	time period between Y-90 precipitation and commencement of the measuring process i, in s;
t_m	duration of counting source measurement, in s;
$\lambda_{\text{Y-90}}$	decay constant for Y-90: $3{,}00 \cdot 10^{-6}\ \text{s}^{-1}$;
ω_1	determined constant background contribution (s^{-1}) that stems from long-lived isotopes (e. g., Ce-isotopes);
ω_2	Y-90-net count rate (s^{-1}) relative to the point of time of the Y-90 precipitation.

The regression parameters ω_1 and ω_2 are obtained from applying a linear regression according to equation (1), regarding the $f(t_i)$ values as independent x values and $R_i(t_i)$ as dependent y values. It should be noted here that the half-life for the decay (of Y-90) is a fixed parameter. In a graphic illustration (y versus x), the 3 or 4 results should well align along a straight line that extends from $x = 0$ [i. e., $f(t) = 0$] through $y = 0$ if there were no disturbances. The graphic will indicate whether the first y-value, $R_1(t_1)$, exhibits a surplus (interference by Ac-228 or radon products) or the straight line extrapolated down to $f(t) = 0$ does not pass through the origin and so indicates a background contribution (e. g., by Cerium isotopes) that is significantly larger than zero.

If simple computation software for linear regression according to equation (1) is available, e. g., in the shape of a pocket calculator with simple statistics functions, the calculated parameter ω_2 can be used as Y-90 net count rate, $R_{\text{Y-90}}$, at the point of time of the Y-90 precipitation.

If the decay gradient does not suggest any interferences and linear regression calculations are not an option, the first measured value, R_1, can be used to obtain the Y-90 net count rate, $R_{\text{Y-90}}$, at the point of time of the Y-90 precipitation:

$$R_{\text{Y-90}} = \frac{R_1(t_1)}{f(t_1)} \tag{3}$$

The standard deviation, $s(R_{\text{Y-90}})$, of the Y-90 net count rate, $R_{\text{Y-90}}$, at the point of time of the Y-90 precipitation is obtained according to:

$$s(R_{\text{Y-90}}) = \frac{s(R_1)}{f(t_1)} \tag{4a}$$

and

$$s(R_i) = \sqrt{\frac{R_i}{t_\text{m}} + R_0 \cdot \left(\frac{1}{t_0} + \frac{1}{t_\text{m}}\right)} \tag{4b}$$

in which:

t_0 duration of background measurement, in s;

R_0 background count rate, in s^{-1}.

If only the first net count rate of a decay curve is excessive (presence of Ac-228 or radon products), the parameters of the second measurement (with index 2) are to be used in equations (4a) and (4b).

Equations (4a) and (4b) are strictly limited to the case where $R_{\text{Y-90}}$ has been obtained from a singular measurement. If, on the other hand, $R_{\text{Y-90}}$ has been derived by applying a linear regression, the standard deviation to be applied (which cannot be detailed here due to the complex error calculations involved) will generally be narrower than the one calculated according to equations (4a) and (4b).

Calculating the specific Sr-90 activity of the fish sample, $a_{\text{Sr-90}}$, relative to the fresh mass (FM) and the date of sampling, follows equation (5):

$$a_{\text{Sr-90}} = \frac{R_{\text{Y-90}}}{\varepsilon_{\text{Y-90}} \cdot \eta_{\text{Y-90}} \cdot m_\text{A} \cdot q_\text{F} \cdot f_0} \tag{5}$$

in which:

$R_{\text{Y-90}}$	Y-90 net count rate at the point of time of the Y-90 precipitation, in s^{-1};
$\varepsilon_{\text{Y-90}}$	Y-90 detection efficiency for the oxalate counting source, depending on the area fill density of the counting source;
$\eta_{\text{Y-90}}$	chemical yield of yttrium; see equation (6);
m_A	mass of ash used for analysis, in kg;
q_F	ratio of fresh mass/ash mass;
t_A	time elapsed from sampling to Y-90 precipitation, in s;
$\lambda_{\text{Y-90}}$	decay constant of Sr-90: $7{,}65 \cdot 10^{-10}\ \text{s}^{-1}$;
f_0	Sr-90 decay factor: $\text{e}^{(-\lambda_{\text{Sr-90}} \cdot t_\text{A})}$.

For the chemical yttrium yield determined by means of complexometric titration (see step 23 in section 3.3), the following applies:

$$\eta_{\mathrm{Y\text{-}90}} = \frac{10\ \mathrm{ml} - V_{\mathrm{Pr}}}{10\ \mathrm{ml} - V_{\mathrm{Tr}}} \tag{6}$$

wherein:

V_{Pr} consumption of zinc sulphate solution (0,1 $\mathrm{mol \cdot l^{-1}}$) for a sample with an unknown yttrium content, in ml;

V_{Tr} consumption of zinc sulphate solution (0,1 $\mathrm{mol \cdot l^{-1}}$) for the sample with the known yttrium content, in ml.

The standard deviation, $s(a_{\mathrm{Sr\text{-}90}})$, of the specific activity, $a_{\mathrm{Sr\text{-}90}}$, reads:

$$s(a_{\mathrm{Sr\text{-}90}}) = \frac{s(R_{\mathrm{Y\text{-}90}}) \cdot a_{\mathrm{Sr\text{-}90}}}{R_{\mathrm{Y\text{-}90}}} \tag{7}$$

with $s(R_{\mathrm{Y\text{-}90}})$ according to equations (4a) and (4b).

5.1 Worked example

The following variables are given as:

Date of precipitation:	30.Sep.1981, 8.50 h
Background count rate:	R_0 = 0,0042 $\mathrm{s^{-1}}$;
Duration of background measurement:	t_0 = 36000 s;
Decay constant for Y-90:	$\lambda_{\mathrm{Y\text{-}90}}$ = $3{,}00 \cdot 10^{-6}\ \mathrm{s^{-1}}$.

Table 1 provides the following details for each measurement *i*: Date and time of the commencement of measuring; the gross count number and the period of measuring *i*; the period of time elapsed, t_i, between precipitation and commencement of measuring; the calculated net count rate, R_i, and its standard deviation, $s(R_i)$, as calculated according to equation (4b).

Tab. 1:

i	Date	Time	gross count number	measuring period (s)	time difference t_i (h)	net count rate R_i ($\mathrm{s^{-1}}$)	standard deviation $s(R_i)$ ($\mathrm{s^{-1}}$)
1	30.Sep.81	16:45	553	36000	7,92	0,01120	0,000737
2	01.Oct.81	07:25	375	28500	22,58	0,00899	0,000760
3	05.Oct.81	08:10	170	24000	119,33	0,00292	0,000642
4	06.Oct.81	17:20	275	36000	152,50	0,00347	0,000573

Table 2 summarizes the data required for applying a linear regression: The decay factor, $f(t_i)$, for each measurement i as computed from measuring period, time difference, and Y-90 decay constant (see equation 2), and the net count rate, R_i.

Tab. 2:

i	Decay factor $f(t_i)$ x-variable	Net count rate $R_i(t_i)$ (s^{-1}) y-variable
1	0,8702	0,01120
2	0,7510	0,00899
3	0,2659	0,00292
4	0,1826	0,00347

Calculating the linear regression according to equation (1) produces:

$$y = 5{,}79 \cdot 10^{-4} + 0{,}0117\ x$$

The first coefficient, ω_1, $5{,}79 \cdot 10^{-4}\ s^{-1}$, which represents the possible contribution by an interfering longer-lived radionuclide, will amount to only 13 % of the value of the background count rate, R_0, so that it does not suggest the presence of such a disturbance. The second coefficient, ω_2, $0{,}0117\ s^{-1}$, represents the Y-90 net count rate at the point of time of the Y-90 precipitation:

$$R_{\text{Y-90}} = 0{,}0117\ s^{-1}$$

Using the data provided in Tables 1 and 2, the standard deviation of $R_{\text{Y-90}}$ results from applying equations (3), (4a) and (4b) as follows:

$$s(R_{\text{Y-90}}) = \frac{0{,}000737}{0{,}8702}\ s^{-1} = 0{,}000847\ s^{-1}$$

Using the additional data:

$$\eta_{\text{Y-90}} = 0{,}911;\ \varepsilon_{\text{Y-90}} = 0{,}306;\ m_{\text{A}} = 0{,}0261\ \text{kg};\ q_{\text{F}} = 74{,}76;\ f_0 = 0{,}996$$

the specific Sr-90 activity is quantified as follows:

$$a_{\text{Sr-90}} = \frac{0{,}0117}{0{,}306 \cdot 0{,}911 \cdot 0{,}0261 \cdot 74{,}76 \cdot 0{,}996}\ \text{Bq} \cdot \text{kg}^{-1}(\text{FM}) = 0{,}0216\ \text{Bq} \cdot \text{kg}^{-1}(\text{FM})$$

and applying equation (7) shows the standard deviation of the specific Sr-90 activity as being:

$$s(a_{\text{Sr-90}}) = \frac{0{,}000847 \cdot 0{,}0216}{0{,}0117}\ \text{Bq} \cdot \text{kg}^{-1}(\text{FM}) = 0{,}0016\ \text{Bq} \cdot \text{kg}^{-1}(\text{FM})$$

5.2 Consideration of uncertainties

The calculated standard deviation considers only the contribution from counting statistics. The uncertainties originating from determining the detection efficiency and the chemical yield, which may amount to as much as 5 % each. A few percent of uncertainty from determining the sample masses and the ratio between fresh mass and ash mass are also added (other errors can be neglected) and need to be added to the uncertainty from counting statistics according to the rules outlined in chapter IV.5 of this procedures manual, section 4.9. Experience shows that the total uncertainty will amount to a value of about 10 % to 15 %.

6 Characteristic limits of the procedure

The detection limit is determined in a simplified manner by taking into consideration only the first measurement in the decay gradient. Analogous to equation (5), the detection limit for the specific Sr-90 activity, $g_{\mathrm{Y\text{-}90}}$, relative to the fresh mass (FM) and date of sampling is obtained according to equation (8):

$$g_{\mathrm{Sr\text{-}90}} = \frac{G_{\mathrm{Y\text{-}90}}}{\varepsilon_{\mathrm{Y\text{-}90}} \cdot \eta_{\mathrm{Y\text{-}90}} \cdot m_{\mathrm{A}} \cdot q_{\mathrm{F}} \cdot f_0 \cdot f(t_1)} \tag{8}$$

in which, next to the variables already specified:

$G_{\mathrm{Y\text{-}90}}$ detection limit of the Y-90 net count rate, $R_{\mathrm{Y\text{-}90}}$, at the point of time of measurement, in s^{-1};

$f(t_1)$ decay factor for the first measurement of the Y-90 content; see equation (2).

For the detection limit $G_{\mathrm{Y\text{-}90}}$, which is calculated according to section 2.1 of chapter IV.5 of this procedures manual, using a background count rate of R_0 = 0,005 s^{-1} and measuring periods of $t_m = t_0$ = 36000 s will produce a result of $G_{\mathrm{Y\text{-}90}}$ = 2,75·10^{-3} s^{-1}.

Supposing an Y-90 decay factor, $f(t_1)$, of 0,74 for the first measurement, an Y-90-detection efficiency of $\varepsilon_{\mathrm{Y\text{-}90}}$ = 0,33, as well as a chemical Y-90 yield of $\eta_{\mathrm{Y\text{-}90}}$ = 0,85, the detection limits for a specific Sr-90 activity relative to the point of time of measuring vary with the original mass, $m_{\mathrm{A}} \cdot q_{\mathrm{F}}$ (in kg fresh mass), as indicated in Table 3.

Tab. 3:

Original mass kg (FM)	$g_{\mathrm{Y\text{-}90}}$ Bq·kg^{-1} (FM)
1	0,0133
2	0,0066
3	0,0044
4	0,0033
5	0,0026

7 Catalogue of chemicals and equipment

7.1 Chemicals

All chemicals used should be of the minimum purity grade '*pro analysi*'.

- Yttrium carrier solution: 10 mg·ml^{-1} Y^{3+}. 8,54 g of $YCl_3·6H_2O$ are dissolved in distilled water and replenished to a total volume of 250 ml (the concentration is determined complexometrically); prepared in a measuring flask.
- Strontium carrier solution: 40 mg·ml^{-1} Sr^{2+}. 24,154 g of $Sr(NO_3)_2$ are dissolved in distilled water and replenished to a total volume of 250 ml; prepared in a measuring flask.
- Caesium carrier solution: 30 mg·ml^{-1} Cs^{+}. 9,501 g of CsCl are dissolved in distilled water and replenished to a total volume of 250 ml; prepared in a measuring flask.
- Barium-carrier solution: 20 mg·ml^{-1} Ba^{2+}. 9,514 g of $Ba(NO_3)_2$ are dissolved in distilled water and replenished to a total volume of 250 ml; prepared in a measuring flask.
- HDEHP in n-Heptane: 0,45 mol·l^{-1}. 150 ml of bis-(2-ethyl-hexyl)-hydrogen phosphate are replenished with n-Heptane to a total volume of 1000 ml.
- Adogen in Toluol: 300 ml of Adogen 464 (trioctylmethyl ammonium chloride) are replenished with toluol to a total volume of 1000 ml.
- Borate buffer: 0,1 mol·l^{-1}. 38,1 g of $Na_2B_4O_7·10\ H_2O$ and 6,18 g H_3BO_3 are dissolved in distilled water and replenished to a total volume of 1000 ml.
- Zinc sulphate solution (0,1 mol·l^{-1})
- Titriplex-III solution (0,1 mol·l^{-1})
- Eriochrome-black T (indicator for metal titrations): 99 parts of NaCl and one part of the indicator are mixed thoroughly in a mortar.
- Nitric acid (14,4 mol·l^{-1})
- Hydrochloric acid (12,1 mol·l^{-1})
- Ammoniac solution (13,4 mol·l^{-1})
- Solution of caustic soda (400 g of NaOH per litre)
- Hydrochloric acid (9 mol·l^{-1}): 744 ml of hydrochloric acid (12,1 mol·l^{-1}) are replenished with distilled water to a total volume of 1000 ml.
- Hydrochloric acid (1 mol·l^{-1}): 83 ml of hydrochloric acid (12,1 mol·l^{-1}) are replenished with distilled water to a total volume of 1000 ml.
- Saturated oxalic acid solution (solubility: 102 g $(COOH)_2·2\ H_2O$ per litre of H_2O at 20 °C).

7.2 Equipment

For equipment for sampling and preparing samples including ashing, see procedure G-γ-SPEKT-FISCH-01.

- Exsiccator;
- Measuring beakers (1000 ml; tall and wide shape, respectively);
- Centrifuge, centrifuge tubes (200 ml and 400 ml, respectively);
- Separation funnel (1 l, 500 ml);
- Hahn's suction apparatus, 20 mm in diameter;

- Drying cabinet;
- Mylar foil;
- Precipitate carrier, "ring+disc", 1 inch in diameter;
- Analysis scale;
- Burette;
- Erlenmeyer flask (300 ml);
- Anticoincidence beta-measuring device.

References

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