and precision, the whole operation does not require more than 15 min for a single determination. It can therefore be recommended for the routine analysis of plutonium.

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Rapid Method for the Determination of Lead in Lead Styphnate

Schnellmethode für die Bestimmung von Blei in Bleistypnate

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In a previous communication [1] a rapid method for the dissolution and determination of lead in lead styphnate was described, using isobutylmethyl ketone for the extraction of styphnic acid and titration of lead in the separated aqueous phase with EDTA.

When replacing isobutylmethyl ketone by a water-miscible solvent, viz. ethanol, adding an excess of EDTA to the solution and back-titrating with lead nitrate, the time required for the whole determination could be further reduced to about 5 min. The standard deviation, calculated from 5 determinations, was 0.11%.

The same principle can be applied also to the salts of other organic acids which are sufficiently soluble in alcohol, like salicylic or resorcylic acid, but not to stearates.

Procedure. To an exactly weighed amount of less than 0.5 mMol (200–220 mg) of lead styphnate, add 20 ml of ethanol and 5 ml of nitric acid (1 + 9) and swirl for a few seconds to dissolve all lead styphnate. Add exactly 25 ml of 0.02 M EDTA, about 250 ml of water, and 10–15 drops of 0.2% Xylenol Orange indicator solution. Neutralize with 1 M

sodium hydroxide to the first reddish tinge and add 1–2 drops of nitric acid (1 + 9) until the colour changes to a bright yellow. Add 5 ml of buffer solution (100 ml of 1 M acetic acid added to 1 litre of 1 M sodium acetate) and titrate back the excess EDTA with 0.02 M standard lead nitrate solution to the first reddish tinge. Run a blank.

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Rapid Method for the Determination of Fe, Co, and Ni in Kovar and Similar Sealing Alloys Using Solvent Extraction

Schnellmethode für die Bestimmung von Fe, Co und Ni in Kovar und ähnlichen Einschmelzlegierungen unter Verwendung der Lösungsmittelextraktion

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A rapid method for the determination of Fe, Co, and Ni in Kovar alloy is given, using two consecutive extractions into isobutylmethyl ketone. First, Fe is extracted from hydrochloric acid solution [2] and then Co, after addition of ammonium thiocyanate [1] to the remaining water phase.

Each of the three metals is determined by back-titration of an excess of EDTA with lead nitrate at pH 5.3 to 5.5 using Xylenol Orange indicator: Fe after re-extraction into water, using an aliquot; Co in presence of the organic phase, and Ni in the remaining water phase. Mn, representing only 0.3% of the alloy, is neglected.

The mean of 5 determinations (at a room temperature of 30°C) of Kovar alloy of the nominal composition:

Fe 53.7%, Co 17%, Ni 29% (and Mn 0.3%) was 52.97, 16.69, 29.09% with standard deviations of 0.15, 0.24, 0.08%, respectively.

The whole procedure, after dissolution of the metal, takes about one hour.

Experimental

Reagents

Isobutylmethyl ketone, solvent grade, of which a fraction distilling between 114 and 116°C is used.

Ethanol, 96%.