Extraction-Spectrophotometric Determination of Niobium in Steels using Sulphochlorophenol S and High-Molecular Amines

N. Ivanov¹, R. Borissova¹, and E. Veselinova²

¹ Dept. of Anal. Chem., Higher Inst. of Chem. Technol., Sofia

² Inst. of Metallurgy, Sofia-Botunez, Bulgaria

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Extraktionsphotometrische Bestimmung von Niob in Stahl unter Verwendung von Sulfochlorphenol S und hochmolekularen Aminen

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A procedure is recommended for the spectrophotometric determination of niobium in steels using sulphochlorophenol S as a reagent. For the decomposition of the sample a new method has been worked out making use of solid tartaric acid after slow treatment with HCl and H₂O₂. For the extraction of the complex formed several high-molecular amines have been studied, employing a concentration of 5–10% in butanol and a volume relation between aqueous and organic phases of 3:1. Best results were obtained with laurylamine and cetylamine, the

latter being however more expensive. For 15 µg of Nb 2 ml of the reagent solution (0.03%) are required. Maximum permissible concentration of steel is 1–1.2 mg/ml. The standard deviation has been found to be 3%. Beer's law is obeyed up to 15 µg of Nb in the aqueous phase. Mn, Cr, Ni, Co, W, Ti, Si and other foreign ions do not interfere. Optimum conditions are incorporated in the following working direction.

Procedure. Dissolve 0.25 g of the steel sample with 5 ml of conc. HCl, heat carefully on a sand bath adding every 2–3 min 1–2 drops of H₂O₂ until complete decomposition of the carbides, concentrate to about 3 ml, add 3 g of solid tartaric acid, 7 ml of conc. HCl and boil softly for 5 min. Cool, dilute to 50 ml, transfer a 5 ml aliquot to a 25 ml volumetric flask, add 1 ml of 5% tartaric acid solution, 0.5 ml of 10 M HClO₄ and 2 ml of 25% NH₂OH · HCl solution together with 3 ml of water. Heat carefully on a sand bath until the solution becomes colourless. Then add 4 ml of 5% tartaric acid solution, 0.5 ml of 10 M HClO₄, 2 ml of 0.03% sulphochlorophenol S solution and 1 ml of water. Cool (after 10 min), add 6.00 ml of 2% laurylamine solution in n-butanol, shake for 2–3 min, transfer after 15–20 min a part of the organic phase into a 1 cm wet cell and measure the absorbance at 660 nm against a blank prepared in the same way. Molar absorptivity is 18000 l · mol⁻¹ · cm⁻¹.

Dr. N. Ivanov, Dept. of Anal. Chemistry, Higher Inst. of Chem. Technology, Sofia-56, Bulgaria