N-(Benzoyl-2-sodium-sulphonate)-N-phenylhydroxylamine as Photometric Reagent for Titanium

N-(Benzoyl-2-natriumsulfonat)-N-phenylhydroxylamin als photometrisches Reagens für Titan

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N-Benzoyl-N-phenylhydroxylamine has already been used for the photometric determination of titanium by several authors [1–3]. We have now modified this reagent by introducing the sulphonic acid group at the 2-position to the carbonyl group, obtaining thereby the advantage of water-solubility of the compound and its complexes. The chelating properties of the original reagent are not altered and there is also no difference in colours and natures of the metal complexes.

Preparation of the Reagent. Slowly add sulphobenzoic anhydride (in dry dioxan) to an equimolar quantity of phenylhydroxylamine (in dry benzene). Stir for about 15 min. Remove the major bulk of the solvent, add carefully alcoholic NaOH solution to pH ~ 8 , precipitate by addition of ether, filter and recrystallise the residue by dissolving in a minimum quantity of water and adding an excess of alcohol. There is no sharp melting point but decomposition occurs at 120°C. Absorption maximum is at 255 nm.

Procedure for Ti Determination. Add 10% HCl and 10% Na acetate solution to the sample (25–250 μg of Ti) to bring the pH to 1.5–2.5, add 5 ml of 5% aqueous reagent solution, make the volume up to 25 ml and measure the absorption at 410 nm using water as a blank.

Most of the common foreign ions do not interfere. Tungstate requires the presence of tartrate ions, iron(III) that of dithionite. Ag(I), Au(III), V(V) and Mo(VI) interfere seriously. The sensitivity according to Sandell is 0.01 $\mu\text{g}/\text{cm}^2$, the molar extinction coefficient 4.32×10^3 .

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Photometric Microdetermination of Phosphorus in Motor Gasoline by Selective Extraction of Molybdophosphoric Acid

Photometrische Mikrobestimmung von Phosphor in Motorenbenzin durch selektive Extraktion als Molybdophosphorsäure

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A method has been worked out for the determination of phosphorus in motor gasoline by combining the sodium carbonate fusion technique [3] with the determination of phosphorus as molybdophosphoric acid [1] and a selective extraction with chloroform-butanol [2,5]. It can also be applied to new lubricating oils and additives containing phosphorus. The absorption spectrum of the complex solution exhibits a maximum at 360 nm. The molar absorptivity at this wave-length is $1900 \text{ l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$. Beer's law is obeyed within the range of 0.4 to 4.0 ppm of phosphorus. The standard deviation is 0.4 ppm. A single determination takes less than 30 min as compared to 6–10 h required by the standard ASTM method. The results obtained are relatively lower than those of the ASTM method, but are within the repeatability limit of the latter. The precision is better than that of the ASTM method. The only interfering element is germanium, which, however, is not commonly present in the petroleum products investigated. Yet, it can be removed (together with As and Si, which otherwise do not interfere) according to the method of Lueck and Boltz [4].

Procedure. Place 3 ± 0.1 g of anhydrous sodium carbonate in a 15 ml platinum crucible, add 2 ml of the sample (1 ml in case of > 50 ppm of P), cover the slurry with an extra amount of 2–3 g of the carbonate and carefully ignite from the top by means of a burner. After the burning has ceased make the crucible red-hot till a clear melt is obtained, cool and dissolve the residue in hydrochloric acid (1:9) till the effervescence has ceased, transfer the contents to a 100 ml volumetric flask and dilute to the mark. Take a suitable aliquot (with 0.01–0.1 mg of P) into a 100 ml beaker, add 20 ml of a mixture of equal parts of dilute hydrochloric acid (1+4) and 10% sodium molybdate solution, adjust the pH to 0.7 ± 0.1 , transfer into a separating funnel, add 10 ml of a mixture of n-butanol and chloroform (1:1), shake for 30 sec and allow the layers to separate (2–3 min). Collect the lower layer in a 25 ml volumetric flask, repeat the extraction with 10 ml of the solvent mix-