Nickel in Polyols

Note: *This method is also applicable for determination of nickel in polydextroses.*

Apparatus
Use a suitable atomic absorption spectrometer equipped with a nickel hollow cathode lamp and an air–acetylene flame to measure the absorbance of the Blank solution, the Standard solutions, and the Sample solution as directed under Procedure (below).

Sample solution
Dissolve 20.0 g of the sample in a mixture of equal volumes of dilute acetic acid TS and water and dilute to 100 ml with the same mixture of solvents. Add 2.0 ml of a 1% w/v solution of ammonium pyrrolidinedithiocarbamate and 10 ml of methyl isobutyl ketone. Mix and allow the layers to separate and use the methylisobutyl ketone layer.

Blank solution
Prepare in the same manner as the Sample solution, but omit the sample.

Standard solutions
Prepare three Standard solutions in the same manner as the Sample solution but adding 0.5 ml, 1.0 ml, and 1.5 ml, respectively, of a standard nickel solution containing 10 mg/kg Ni, in addition to the 20.0 g of the sample.

Procedure
Zero the instrument with the Blank solution. Then determine the absorbances at 232.0 nm of each of the Standard solutions and of the Sample solution at least three times each, and record the average of the steady readings for each. Between each measurement, aspirate the Blank solution, and ascertain that the reading returns to its initial blank value.

Prepare a standard curve by plotting the mean absorbances vs concentration for the Standard solutions. Extrapolate the line joining the points on the graph until it meets the concentration axis. Read the concentration of nickel in the Sample solution at the intersection of the standard curve with the concentration axis.