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Application to the Determination of Cobalt in Aluminum
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ABSTRACT

High-absorbancy spectrophotometry was applied to the precise determination of about 0.15 per cent cobalt in aluminum by the 1-(2-pyridylazo)-2-naphthol (PAN) method. The absorbancies of sample aliquots were measured versus a standard containing 50 μ g of cobalt. The relative concentration error for a single measurement, assuming a 1-per cent transmittancy error, is 0.95 per cent and the coefficient of variation of 5 cobalt determinations is 0.5 per cent.

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HIGH-PRECISION DETERMINATIONS BY MEANS OF THE HIGH-ABSORBANCY SPECTROPHOTOMETRIC METHOD.

Application to the Determination of Cobalt in Aluminum

Gerald Goldstein

A method which would have a precision of less than 1 per cent was required for the determination of about 0.15 per cent of cobalt in high-purity aluminum. Since the gravimetric methods for the determination of small amounts of cobalt are not sufficiently precise and accurate, other methods of determining cobalt were considered.

High-absorbancy spectrophotometry⁽¹⁾ is commonly utilized to increase the range of concentration of an absorbing species which can be determined spectrophotometrically. The relative concentration error associated with the absorbancy measurement is, at the same time, decreased by use of the high-absorbancy method. In a normal spectrophotometric method in which the absorbancy measurements are made versus a reagent blank, the relationship between the concentration and absorbance of the absorbing species is represented by

$$C = \frac{-\log T}{ab} \quad (1)$$

where C = concentration of unknown
 T = transmittancy of unknown measured vs reagent blank
 a = molar absorbancy index
 b = cell thickness, cm

The relative concentration error, $\frac{dC}{C}$, which is associated with a given transmittancy error, dT , is evaluated by a differentiation of equation (1):

$$\frac{dC}{C} = \frac{-0.434}{T \log T} dT \quad (2)$$

For a one per cent error in the measurement of transmittancy, the relative concentration error, $\frac{dC}{C}$, has a minimum value of 2.72 per cent at a transmittancy measurement of $T = 0.368$.

In the high-absorbancy or differential spectrophotometric method, absorbancy measurements are made versus a reference standard which contains a known concentration of the absorbing species, C_S , then

$$C = C_S - \frac{\log T'}{ab} \quad (3)$$

where C = concentration of unknown
 C_S = concentration of reference standard
 T' = transmittancy of unknown measured versus the reference standard

The relative concentration error, $\frac{dC}{C}$, is now found upon differentiation of equation (3) to be

$$\frac{dC}{C} = \frac{-0.434 dT}{T(abCs - \log T)} = \frac{-0.434 dT}{T(A_{\text{std vs blank}} + A_{\text{sample vs reference standard}})}$$

where $A = -\log T$, so that $\frac{dC}{C}$ decreases as the absorbance of the reference standard, C_B , increases and as the absorbance of the sample or unknown versus the reference standard approaches zero.

It is possible, therefore, to utilize the high-absorbancy method for the purpose of increasing the precision of spectrophotometric determinations, and at the same time, retain the rapidity and ease of operation which is characteristic of spectrophotometry. The PAN spectrophotometric method⁽²⁾ was applied to the determination of 0.15 per cent of Co in aluminum in the following manner:

A 0.9085-g sample was dissolved in 10 ml of aqua regia, and the solution was diluted to 100 ml in a volumetric flask. Five 5-ml aliquots were taken, and 2 ml of 0.1 per cent citric acid, 2 ml of 50 per cent ammonium acetate, 1 ml of 0.1 per cent PAN in ethanol were added; then the solutions were diluted to 25 ml. The solutions were then extracted with two 10-ml portions of CHCl_3 . The extracts were transferred to 25-ml volumetric flasks and diluted to volume with CHCl_3 . Standard cobalt solutions containing 50, 60, 70, 80, and 90 μg of cobalt were treated in the same manner. The absorbance of the 50- μg standard was measured vs a reagent blank, and the other solutions were measured vs the cobalt standard. The results are given in Table I.

Table I

High-Absorbancy Spectrophotometric Determination of Cobalt in Aluminum

Conditions:	Volume, ml	25
	PAN, 0.1 per cent in ethanol, ml	1
	Wavelength, m μ	640
	Cells, cm	1
<u>Solution, Co, μg</u>	<u>Absorbancy vs Co Standard, 50 μg</u>	
50	0.570(a)	
60	.134	
70	.263	
80	.382	
90	.460	
Samples	.238, .240, .241, .239, .238; Avg. .239	

(a) vs reagent blank

From the calibration curve obtained by plotting the absorbancy of the standards vs their concentration, it was determined that the sample aliquots contained 68.1 μg of cobalt, and the sample therefore contained 0.150 per cent cobalt. The relative concentration error of a single measurement, assuming a 1 per cent T error, is

$$\frac{dC}{C} = \frac{0.434}{0.578 (0.570 + 0.239)} = 0.93 \text{ per cent}$$

and the coefficient of variation of the 5 cobalt determinations is 0.5 per cent.

References

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