



Simultaneous Spectrometric Determination of Co^{2+} and Ni^{2+} in Polimineral Drug Using Multivariate Calibration

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Abstract

The application of the method for the simultaneous spectrophotometric determination of the divalent ions of cobalt and nickel for analysis of a pharmaceutical formulation of polimineral drug is reported. This method uses 5-[(Hexahydroxy-2,4,6,-trioxo-5-pyrimidinyl) imino]-2,4,6, (1H, 3H, 5H)-pyrimidinetrione, monoammonium salt (Murexide) as the complexing agent and several chemometric approaches. A comparative study of the results obtained by using principal component regression (PCR) and partial least-squares regression (PLS) for absorbance and first-derivative data is presented. In general, the best recovery values are obtained by the PLS method for absorbance data.

Introduction

Among the most widely used analytical methods for determination of Co^{2+} and Ni^{2+} ions in different samples are those based on the UV-Visible spectrophotometry techniques, due to both the resulting experimental rapidity and simplicity and the wide application. However, the simultaneous determination of these ions by the use of the traditional spectrophotometry techniques is difficult because, generally, the absorption spectra overlap in a visible region.

Quantitative spectrophotometry has been greatly improved by the use of a variety of

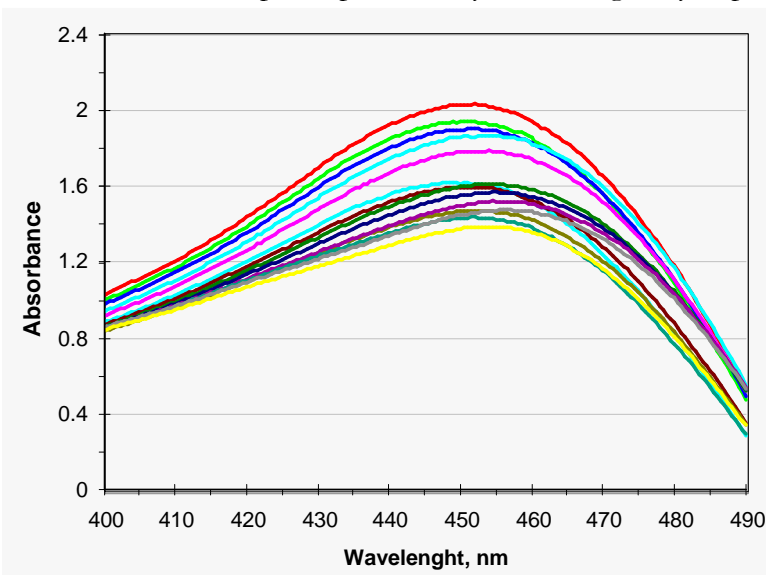


Fig. 1; Absorbance spectra of 14 calibration samples of cobalt and nickel mixtures.

multivariate statistical method, particularly principal component regression (PCR) and partial least squares regression (PLS). In this work, PCR and PLS algorithms are applied to the simultaneous determination of cobalt and nickel in synthetic mixtures and adapted to simultaneous determination of the cited ions in real samples of a polimineral medicine in the visible region. The results obtained by the two methods are compared and discussed.

Materials & Methods

All absorbance measurements digitized spectra were collected on a Shimadzu UV 2102 PC double beam UV-Vis spectrophotometer, using a 10 mm quartz cell. The recorded spectra were digitized with an interval of 0.5nm between consecutive points. Therefore 181 data points were used to represent a spectrum in the range of 400-490nm. The computations

All absorbance measurements digitized spectra were collected on a

were made on a Pentium IV computer. All the programs were written in MATLAB (Mathworks, Version 6.1) by the authors.

The samples were first dried and mineralized using concentrated nitric acid, and the mixture was heated under reflux up to the disappearance of nitrous fumes. Then pH of the solutions was adjusted to 8.0 with concentrated NaOH. The reagents were added in the following order: buffer solution (pH 8.0) - metal solution - murexide. Finally, the samples were diluted to 25 ml with doubly distilled water in a calibrated flask. The final concentration of these solutions varied in their respective linear determination ranges 0.354 - 5.893 and 0.352 - 5.869 $\mu\text{g ml}^{-1}$ for cobalt and nickel, respectively.

We have used 21 synthetic samples: 14 for calibration and 7 for prediction. Fig.1 shows absorbance spectra for calibration set. The models obtained were applied for 3 polimineral drug.

Results

The complexes Co(II) and Ni(II) - murexide are highly absorbing substances in the visible region of the spectrum and have maximum absorption at $\lambda = 456$ and 451.5 nm, respectively. The recovery values (%) are obtained by PCR and PLS methods for absorbance and first derivative data. The results are summarized in Table 1 where S1, S2 and S3 are Oligogranul Ni-Co (in tablet form),

Tab. 1, Recoveries (%) of Co and Ni by the calibration methods

		PCR		PLS	
		A	D1	A	D1
S1	Co	83.31	113.77	110.26	111.63
	Ni	71.45	87.55	96.64	95.02
S2	Co	105.20	123.08	104.11	108.32
	Ni	86.22	76.27	99.01	93.78
S3	Co	130.53	143.46	97.56	89.39
	Ni	111.68	68.84	98.65	104.22

Suboligo Ni-Co (in tablet form) and Oligosol Ni-Co (ampoule to be taken orally) respectively. A, D1 are Absorbance and First-Derivative respectively.

Conclusions

The cobalt-nickel mixture is an extremely difficult complex system due to the high spectral overlapping observed between the absorption spectra for these components. The PLS model was successfully applied to the simultaneous

determination of Co(II) and Ni(II) in polimineral medicine. The best recovery values are obtained by the PLS method for absorbance data.

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