Practical Handbook on SPECTRAL ANALYSIS

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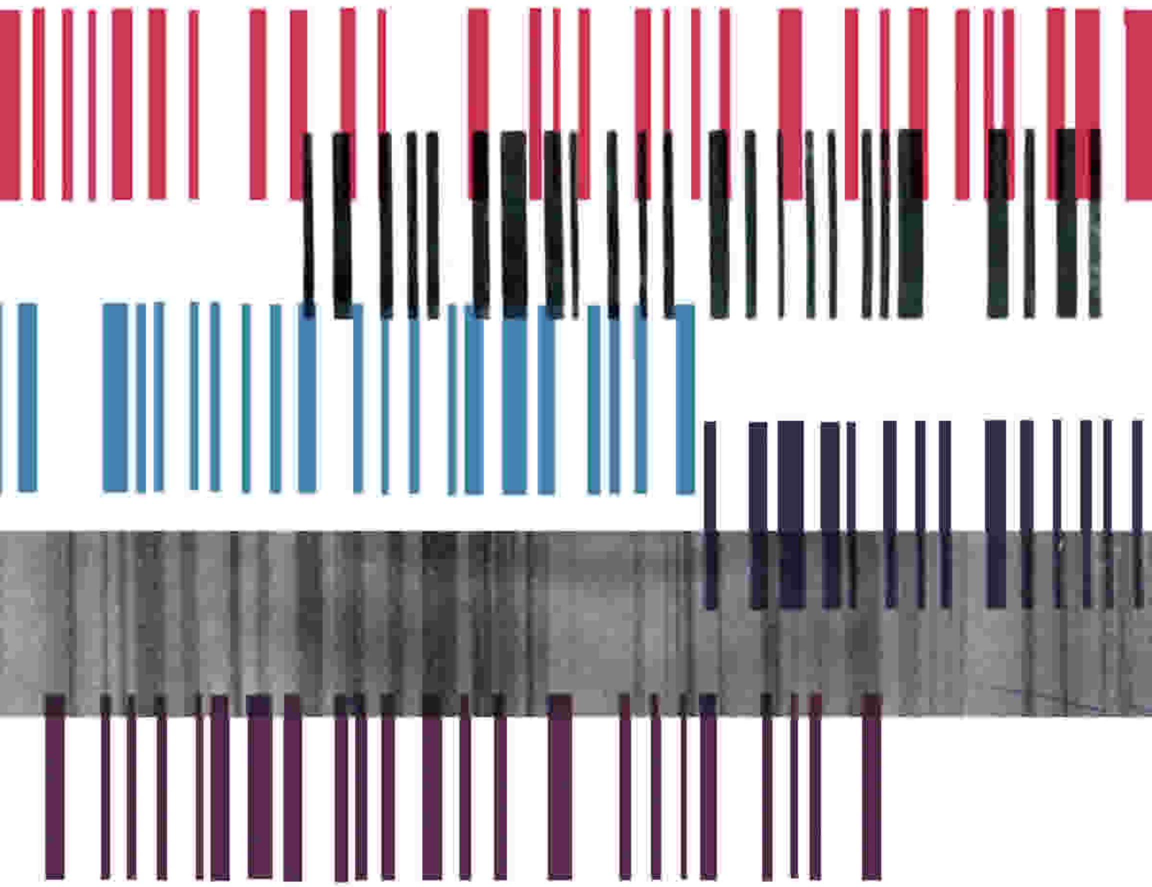

Pergamon Press

PRACTICAL HANDBOOK
ON
SPECTRAL ANALYSIS

ПРАКТИЧЕСКОЕ РУКОВОДСТВО ПО СПЕКТРАЛЬНОМУ АНАЛИЗУ

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и А. А. ЯНКОВСКИЙ

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PERGAMON PRESS

OXFORD · LONDON · EDINBURGH · NEW YORK
PARIS · FRANKFURT

PERGAMON PRESS LTD.
Headington Hill Hall, Oxford
4 & 5 Fitzroy Square, London, W.1

PERGAMON PRESS (SCOTLAND) LTD.
2 & 3 Teviot Place, Edinburgh 1

PERGAMON PRESS INC.
122 East 55th Street, New York 22, N.Y.

GAUTHIER-VILLARS ED.
55 Quai des Grands-Augustins, Paris, 6e

PERGAMON PRESS G.m.b.H.
Kaiserstrasse 75, Frankfurt am Main

Distributed in the Western Hemisphere by
THE MACMILLAN COMPANY · NEW YORK
pursuant to a special arrangement with
Pergamon Press Limited

First English edition 1964

Library of Congress Catalogue Card Number 63-16858

This is an edited translation of the original Russian *Prakticheskoye rukovodstvo po spektral'nomu analizu*, published in 1960 by Izdatel'stvo Akademii nauk B.S.S.R., Minsk

MADE IN GREAT BRITAIN

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FOREWORD

In 1959 the 21st Congress of the Communist Party of the Soviet Union and the June General Meeting of the Central Committee of the Soviet Union, set new tasks for speeding up technical progress in industry and building construction and for raising the quality of the materials produced.

In raising the quality of production great importance is attached to setting-up comprehensive and continuous controls over production processes, starting with the raw material and ending with the finished goods. It is particularly important to control the chemical composition in production, which governs many technical characteristics of the goods produced.

Emission spectral analysis is an up-to-date method for controlling the chemical composition of various materials, and has found wide use in industry and in various scientific investigations.

In White Russia spectral analysis is widely used in the iron and steel, metalworking, engineering, instrument-manufacture industries, etc., to analyse ferrous and non-ferrous metals and alloys. Spectral analysis methods are used in geological investigations to determine the composition of White Russian minerals, in particular for minute concentrations of elements in the potash salts of the Starobino deposits. Spectral analysis is now being used successfully in medicine to determine the minute concentration content of elements formed in blood and tissues during various illnesses. Such methods are used in biology, agriculture and criminology.

In view of the development and rapid growth of spectral analysis laboratories, the inadequacy of the literature on spectral analysis is keenly felt. Handbooks published earlier are now regarded as collectors' pieces.

The purpose of the present handbook is to give a short account of the main problems in methods for carrying out the spectral analysis of the materials encountered in practice in industrial laboratories.

Unlike previous publications, this book deals both with visual and photographic methods of spectral analysis. The future of photo-electrical methods is indisputable, but their introduction into industrial spectral analysis laboratories requires time and does not exclude the further development and use of visual and spectrographic methods.

The methods presented in this book, are selected on the basis of data given in the literature and from practical experience in this field in works laboratories in the U.S.S.R. and in particular in the White Russian council of national economy.

Naturally, it is impossible to present in one volume the whole of the theoretical and experimental data and the diverse procedures described in the literature. In view of this, in many cases, instead of delving deeply into the physical essentials of the processes being considered, the authors have simply summarized practical data available. For a detailed study of the principles of spectral analysis more fundamental handbooks should be referred to. Of these, the main ones are listed in the literature references, where reference is also made to papers in journals, monographs, etc. giving fuller data on concrete methodological problems.

The authors wish to thank T. M. Zhbanovaya, L. I. Kiselevskii, M. A. Krivosheyevaya, P. A. Naumenkov, G. V. Ovechkin, Ye. N. Paltarak and A. M. Tokarevaya, who kindly commented on the manuscript of the book. The authors will also be glad to receive readers' comments.

INTRODUCTION

Spectral Analysis and Its Possibilities

Spectral analysis is a physical method for determining the chemical composition of matter. It is based on the study of the spectral composition of light emitted, absorbed or reflected by the material being investigated. By the term "spectral analysis" we shall denote atomic emission spectral analysis.

Atomic emission spectral analysis has a number of advantages over other industrial methods of determining the chemical composition of materials.

A distinguishing feature of spectral analysis is its high sensitivity, since it is possible to determine individual chemical elements in amounts totalling millionths of a milligramme. Only a small amount of material is required, so that the end-product can be analysed and then used in service.

By analysing samples on the basis of their emission spectra it is possible to determine simultaneously almost all the chemical elements in various solids, liquids and gases.

Using up-to-date Russian equipment spectral analysis takes several minutes only. Thus it is possible, for instance to determine the composition of a metal during the melting process. By using high-speed electronic computers it is obviously possible to control not only the composition of the metal during the melting process, but also the process itself.

Spectral analysis laboratories do not require expensive or scarce reagents. The photographic methods require ordinary photographic reagents; with the visual and photo-electric methods the need for chemical reagents completely disappears.

Spectral analysis is more accurate than chemical analysis for determining small concentrations of material, and slightly less accurate when evaluating large concentrations.

Much time, material and work can be saved by using spectral analysis in the national economy and in scientific investigations.

At up-to-date establishments up to 90 per cent of all analyses of metals and alloys are carried out by spectral analysis methods. Chemical methods are mainly used for sulphur and carbon analyses.

In the analysis of complex materials by spectral methods the results of the assessment of the individual elements may be distorted by the presence of additional impurities, the so-called "third-body" effect (cf. p. 43), in the species being studied. At present methods are being developed for minimizing or allowing for these effects. The difficulties that restrict the use of spectral analysis methods are provisional and can eventually be overcome.

Production of Spectra

Light is made up of electro-magnetic radiations of definite wavelength. The wavelength of light is measured in angstroms (Å) (1Å = 10^{-8}cm). Red rays have a wavelength of about 6500 Å, green rays of 5300 Å, violet rays of 4100 Å (Table 1). Rays of various wavelengths are present in the radiation from most light sources.

The radiation spectrum of any light source can be produced very simply by means of a triangular transparent glass prism (Fig. 1). On passing through

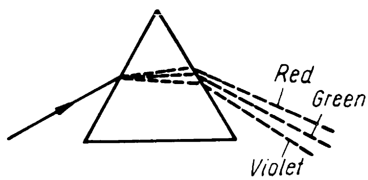

FIG. 1. Splitting of light into a spectrum by means of a prism.

the prism the light forms a band of colour which is the optical spectrum of the source. The action of the prism is based on its different refraction of light to various wavelengths. The red rays, of long wavelength, are only refracted slightly, the green rays are refracted more strongly, and the violet rays (short wavelength) are refracted even more strongly. Thus pencils of colour issue from the prism at different angles and we see light that is split up according to its wavelengths.

The human eye can detect only a narrow spectral colour range. By means of special instruments it can be shown that beyond the red region there is an infrared region and beyond the violet region there is an ultraviolet region. In order to work in this part of the spectrum quartz prisms are used, since glass only transmits visible light together with a very small proportion of the ultraviolet rays. Most spectral analysis instruments contain a glass or a quartz prism.

Light can also split up into a spectrum by other methods. Nowadays increasing use is being made of instruments containing a diffraction grating instead of a prism, i.e. a glass or metal plate on which a large number of parallel equidistant grooves have been ruled by means of a diamond point (the grooves being 1 μ or less apart).

The very narrow apertures of the diffraction grating act each as independent sources when light falls on them, and they radiate the light in all directions.