A Thesis
Entitled

SPECTROSCOPIC MEASUREMENTS OF FLUORESCENT RADIATION EMITTED FROM SOME NATIONAL PRODUCTS

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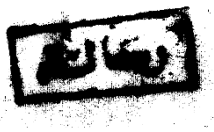

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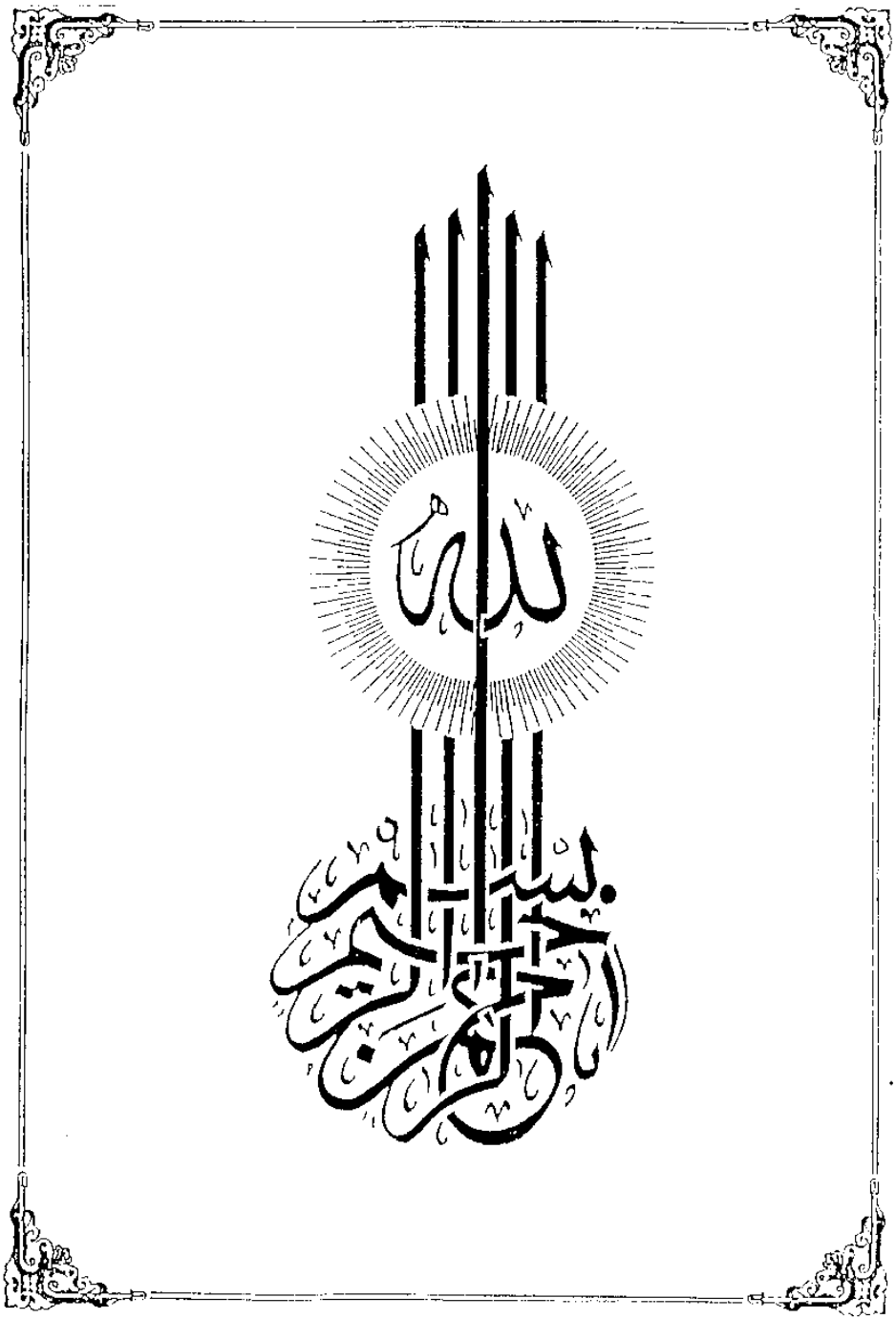

glycerol, some papers and information about that material.

At last the worker wishes to acknowledge rhône company, France for kindly supplying the di-isopropyl etner employed.

C O N T E N T S

Introduction

Spectrofluorimetry is considered to be one of the most sensitive and efficient methods of optical techniques in the detection of elements and compounds. Although this technique is a recent one, its instrumentation is developing rapidly towards optimum sensitivity to compete with that of the other optical methods.

The worker has assembled a spectrofluorimeter by which excitation and fluorescence spectra could be recorded directly. He also introduced two main modifications. A thermostatically-controlled liquid filter is inserted in the light path to prevent heat from affecting the excitation monochromator even with prolonged operation. The other modification is a shaft made for coupling with either the excitation or emission monochromator with the recorder's drive motor. By the use of such a shaft both excitation and fluorescence spectra can be automatically scanned.

The worker calculated and presented tables including correction factors for both excitation and fluorescence spectra carried out on the constructed spectrofluorimeter. The performance and sensitivity of the instrument have been determined and showed that the constructed instrument is comparable with the most powerful spectrofluorimeters. On checking the performance of the instrument, it showed com-

[illegible] limit of detection is one part per trillion of quinine sulphate.

The worker employed the constructed SFF in organic and inorganic fields of determination. In the former one he used a modified method, by which he was able to determine the gossypol (toxic pigment in cottonseed) content in Egyptian cottonseed oil down to 6×10^{-9} g/ml. In the latter one he suggested a modification to colorimetric method and applied it spectrofluorimetrically for the determination of thallium. According to such a modification the limit of detection of this element has been lowered from 3 ppm in the colorimetric method to 16 ppb in the spectrofluorimetric method prepared.

CHAPTER I

A. Historical Survey

The observation of fluorescence as a rough means of identification dates back to the nineteenth century. Then Stokes[1] published his now historic paper on fluorescence in 1852, the fluorescence of quinine, chlorophyll and other plant materials was known to him. Also, the appearance of visible fluorescence of a new organic compound was often noted in describing its properties along with colour, crystallinity, etc. Qualitative observations of visible fluorescence by direct viewing or by photography has also been in use for a great many years. It is still much used as a powerful technique in many fields, e.g., criminology, medical and biological examinations, contamination and spoilage of materials, etc. The physicist, who has studied this phenomenon as an exact science, is highly critical of others, e.g., biologists, who use instruments which yield uncorrected excitation and fluorescence spectra and who report these uncorrected values in the literature[2].

According to Parker[3] quantitative fluorimetry had been applied in a variety of fields up to about 1946, but had been regarded as unreliable by some workers. Guenfriend[4] attributed this suituation to the fact that the physicists made no attampt to standardize or simplify the instruments with which they made their measurements, each of them using an individually designed and rather complicated instrument.

Consequently, other workers including [illegible] started to use fluorescence on a wide scale without a comprehensive understanding of the phenomenon itself or its limitations. Consequently, there had been some errors in the information gathered in connection with that phenomenon. Usually such misfortune is unavoidable whenever new techniques are introduced. However, it will not take long for the different workers to master its intricacies and to use it in the proper manner.

As far back as 1935, Zscheile[4] used a photoelectric spectrophotometer to measure the fluorescence emission spectra of some plant materials. In 1943, Zscheile and Harris[5] described an improved instrument using a rubidium vacuum phototube. With the development of sensitive photomultipliers, a variety of instruments were described in the literature during the period up to 1953[6-11] where it was possible for photoelectric recording of fluorescence spectra to compete in terms of sensitivity with the less convenient and less precise photographic method.

Since Bowman, Caulfield and Udenfriend published their paper in 1953[12] in which they described a two monochromator instrument, equipment has developed to the point where complete fluorescence emission and excitation spectra of small quantities of material can be recorded automatically at the turn of a switch. Such photoluminescence measure-

ment has proved to be a powerful technique in many fields when properly applied.

Perhaps the greatest advantage of analytical photoluminescence methods over most of the others is the possibility of attaining a high degree of sensitivity. And since almost all molecules are capable of fluorescing or quenching, either directly or after suitable treatment, such methods became applicable in several fields.

However, in the inorganic field, photoluminescence has to compete with a variety of other powerful methods among which comes emission spectrography. Neverthless, every technique has its own advantages and its limitations too. Therefore, each problem must be clearly treated on its merits, regarding the facilities at the worker's disposal.

In the organic field, photoluminescence has fewer competitors, particularly when small amounts are concerned. Apart from sensitivity, it has the advantage over absorption spectrometery that two spectra are available as criteria for identification (excitation and emission spectra) instead of one. Since not all substances that can absorb light fluoresces, a luminescent substance can often be readily determined without pre-separation from other substances that are non-luminescent, or luminesce in different spectral regions. In other applications, it is often possible to convert a non-luminе-