

# **ILLEGIBLE DOCUMENT**

**THE FOLLOWING  
DOCUMENT(S) IS OF  
POOR LEGIBILITY IN  
THE ORIGINAL**

**THIS IS THE BEST  
COPY AVAILABLE**

A NEW UNIMOLECULAR REAGENT FOR NITROGEN DIOXIDE ANALYSIS

by

TIEN-YOU CHENG

B.S., Tung Hai University, 1974

M.S., Tsing Hua University, 1976

---

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemistry

KANSAS STATE UNIVERSITY

Manhattan, Kansas

1981

Approved by:



Major Professor

SPEC  
COLL  
LD  
2668  
.74  
1981  
C44  
c. 2

TABLE OF CONTENTS

Page

LIST OF FIGURES.....iii

CHAPTER

I. INTRODUCTION..... 1

II. EXPERIMENT

    Part I Synthesis of N-(4-aminophenylsulfonyl)-N'  
            (1-naphthyl)-ethlenediamine..... 7

    Part II Nitrite ion determination..... 10

    Part III Nitrogen dioxide determination..... 16

III. RESULTS AND DISCUSSION..... 26

LITERATURE CITED..... 40

ACKNOWLEDGMENTS..... 42

## LIST OF FIGURES

Figure	Page
1. Scheme for the synthesis of N-(4-aminophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	6
2. Absorbance curve for the reagent in $3.34 \times 10^{-5}$ M nitrite ion solution.....	12
3. Calibration curve for nitrite ion concentration.....	14
4. A motor-driven injection nitrogen dioxide gas dilution system.....	20
5. A permeation nitrogen dioxide gas dilution system.....	22
6. Calibration chart of flowmeter.....	24
7. NMR spectrum of p-acetaminobenzene sulfonyl chloride... 30	30
8. NMR spectrum of N-(4-acetamidophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	30
9. NMR spectrum of N-(4-aminophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	32
10. C-13 NMR spectrum of N-(4-acetamidophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	34
11. C-13 NMR spectrum of N-(4-aminophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	34
12. IR spectrum of p-acetaminobenzene sulfonyl chloride....	36
13. IR spectrum of N-(4-acetamidophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	36
14. IR spectrum of N-(4-aminophenylsulfonyl)-N'(1-naphthyl)-ethylenediamine.....	38

## Chapter I

### INTRODUCTION

Industrial poisoning from the oxygen compounds of nitrogen is a hazard in many industries. They may be found in the atmospheres of chemical plants manufacturing nitric acid and may occur in the welding, metal cleaning and nitration purposes industries. Nitrogen dioxide is the most toxic of the various oxides of nitrogen.

The fumes of nitrogen dioxide are extremely dangerous because of their insidious character. The bad feature of this type of poisoning is that little warning is given to the workers for the nitrogen dioxides fail to set up defensive respiratory reflexes. A concentration of 20 - 25 ppm could irritate the eye, while 150 ppm may bring about strong local irritations, especially in the respiratory organs (1). The generally allowable concentration of daily eight-hours exposures is 5 parts of nitrogen dioxides per million parts of air (2).

Many methods have been developed for the detection of nitrogen dioxide. An early method, although tedious, involves the conversion of nitrogen dioxide to nitrite and the nitration of phenoldisulfonic acid (3, 4). Another approach to this trace analysis is the Griess method (5), which utilizes the nitrite formed from nitrogen dioxide to diazotize sulfanilic acid which in turn couples with 1-naphthylamine to form an azo dye. Many variations of this method have been developed by changing the