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PREPARATION OF NITRO COMPOUNDS VI PREPARATION INFRARED
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WEAPONS SYSTEMS RESEARCH LABORATORY

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TECHNICAL MEMORANDUM

WSRL-0294-TM

PREPARATION OF NITRO COMPOUNDS
VI. PREPARATION, INFRARED AND ULTRAVIOLET SPECTRAL
CHARACTERISATION OF NITRATION PRODUCTS OF DIPHENYLAMINE

M. MULAR and A. VOM BERG

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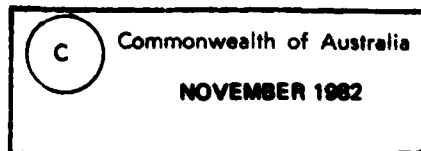
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S U M M A R Y

A variety of nitro- and nitroso- compounds of diphenylamine have been prepared. Although literature methods of preparation were chosen, some of the published methods were modified in order to obtain a reasonable yield of the desired product. Most literature preparations gave low yields, and some of the methods were difficult to reproduce. All compounds studied in this work were characterised by their melting points and infrared and ultraviolet spectra.



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1. INTRODUCTION

Diphenylamine (DPA) is used extensively as a stabiliser in single base gun propellants. It reacts readily with oxides of nitrogen evolved during the decomposition of nitrocellulose to form a variety of nitro- and nitroso-derivatives(ref.1-5). The process considerably slows the autocatalytic degradation of nitrocellulose. High performance liquid chromatographic (HPLC) analysis of the reaction products of DPA found in propellant extracts should provide valuable information which could be used to predict the service life of gun propellants(ref.2-5). In order to conduct such analyses it is necessary to synthesise some of the nitro- and nitroso- compounds which have been found in aged propellants stabilised with DPA for use as reference standards. This Memorandum describes the syntheses and spectroscopic (infrared and ultraviolet) characterisation of eight of these compounds, and the spectral characterisation of five other (commercially available) compounds. Methods described in the literature were used for the syntheses but several were modified to improve the yield. Some literature papers were found to be incorrect in their reference to the original synthesis of a particular compound - upon checking the original paper cited, no mention of the compound could be found.

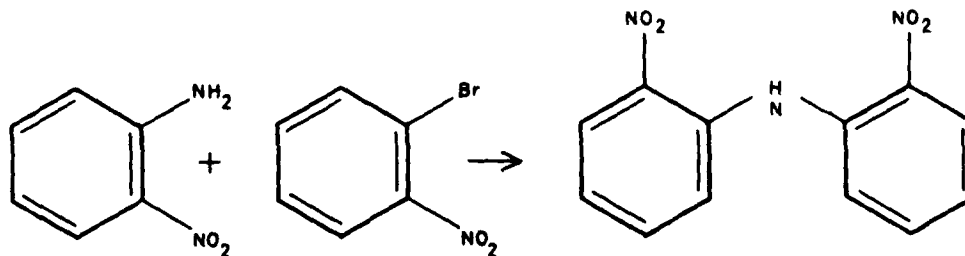
Table 1 lists all the compounds studied in this work, their source, method of purification, and comparison of melting points with those described in the literature. DPA, 2-nitrodiphenylamine, 4-nitrodiphenylamine, 2,4-dinitrodiphenylamine and 2,4'-dinitrodiphenylamine (Table 1) are commercially available and therefore were not synthesised.

2. DETAILS OF PREPARATION

2.1 2,2'-Dinitrodiphenylamine(ref.6)

Asquith(ref.6) reported the preparation of 2,2'-dinitrodiphenylamine by heating o-nitro-bromobenzene with o-nitroacetanilide in nitrobenzene for 16 hours at 180° in the presence of anhydrous potassium carbonate and cuprous bromide. Removal of excess nitrobenzene by steam distillation followed by hydrolysis of the crude reaction product with hydrochloric acid gave the desired compound in 67% yield. In this work, several attempts by this method gave only low yields (10%) of the desired compound.

The following procedure was developed:



A mixture of o-nitroaniline (1.2 g), o-nitrobromobenzene (2.0 g), anhydrous potassium fluoride (1.0 g) and anhydrous potassium carbonate (0.5 g) in nitrobenzene (25 mL) was heated in a covered nickel crucible at 150°C for

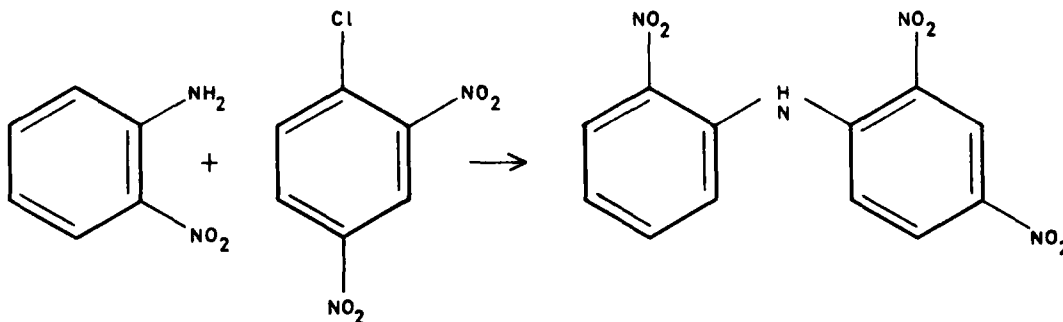
48 hours. The nitrobenzene was removed by steam distillation and the solid residue extracted with hot absolute ethanol. The ethanolic solution was evaporated under vacuum and the dark orange residue was dissolved in boiling ethanol containing a little decolourising charcoal, filtered and allowed to cool. Orange crystals were obtained. Recrystallisation from absolute ethanol gave 2,2'-dinitrodiphenylamine as orange, needle-like crystals (0.45 g, 20% yield) m.p. 164 to 165°, lit(ref.6) m.p. 172°, lit(ref.7) m.p. 166 to 167°.

2.2 2,2'4-Trinitrodiphenylamine(ref.8,9)

Bell(ref.8) reported the preparation of 2,2'4-trinitrodiphenylamine using a general procedure by passing an equimolar solution of 2,4-dinitrochlorobenzene and 2-nitroaniline in dimethylformamide through a caustic soda/alumina column. In this laboratory, such a procedure resulted only in intractable tars. (Bell also reported the formation of tars in other preparations eg preparation of 2,4- and 2,6-dinitrodiphenylamine).

Vorozhtsov(ref.9) described a general procedure for the preparation of 2,4-dinitrophenyl derivatives of amines by heating the amine with 2,4-dinitrochlorobenzene in the presence of potassium fluoride for 10 min to 2.5 hours at 100° to 130°.

In this work, the following procedure was used:

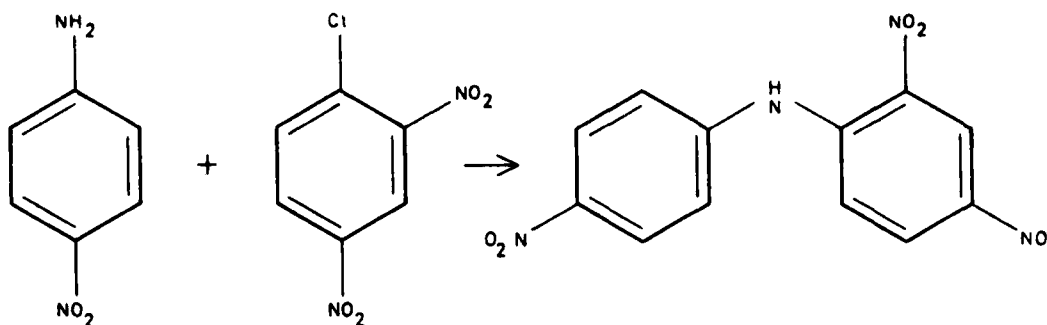


A mixture of o-nitroaniline (1.0 g), 2,4-dinitrochlorobenzene (2.0 g) and anhydrous potassium fluoride (1.5 g) was heated at 150° for 48 hours. The solidified reaction mass was pulverised, washed well with water (100 mL), hydrochloric acid (5%, 50 mL) and again with water (200 mL).

The residue was extracted with hot ethanol. Upon cooling the extract, yellow crystals were obtained. Recrystallisation from absolute ethanol gave 2,2',4-trinitrodiphenylamine as brilliant yellow crystals (0.22 g, 10%) m.p. 185 to 186°, lit(ref.10) m.p. 183 to 186°.

2.3 2,4,4'-Trinitrodiphenylamine(ref.8,11)

The procedure described by Bell(ref.8) again proved unsuccessful in this laboratory and gave only intractable tars. The general procedure described by Sharnin(ref.11) and Vorozhtsov(ref.9) was followed:

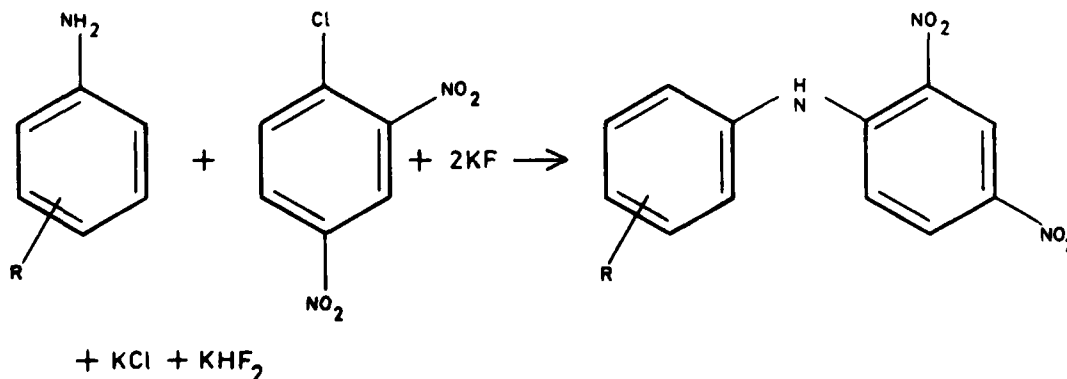


A mixture of p-nitroaniline (1.0 g), 2,4-dinitrochlorobenzene (2.0 g) and anhydrous potassium fluoride (1.5 g) was heated at 150° for 48 hours. The solidified reaction mass was pulverised, washed well with water (100 mL), hydrochloric acid (5%, 50 mL) and again with water (200 mL). The residue was extracted with hot ethanol. Upon cooling the extract, yellow crystals were obtained. Recrystallisation from absolute ethanol gave 2,4,4'-trinitrodiphenylamine as yellow crystals (0.22 g, 10%) m.p. 184 to 186°, lit(ref.1,7,10) m.p. 181 to 189°.

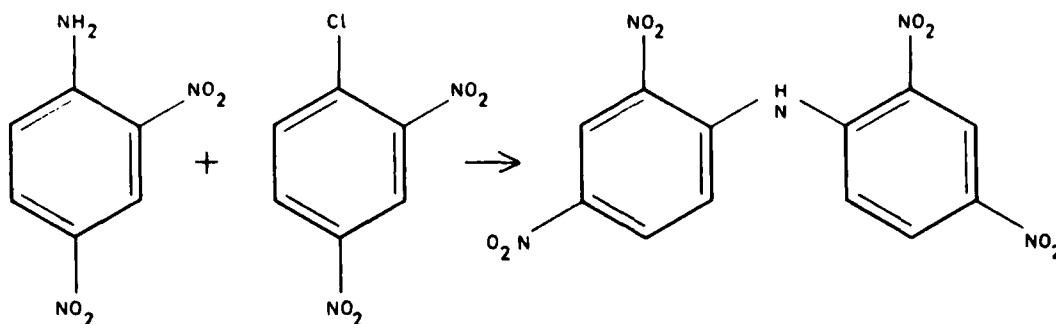
2.4 2,2',4,4'-Tetranitrodiphenylamine(ref.9,12)

Hoffman(ref.12) describes the conversion of dinitrodiphenylamine into tetranitrodiphenylamine by heating with nitric acid. He obtained three different products depending upon the conditions used. In this work, nitration of 2,4'-dinitrodiphenylamine with nitric acid gave a good yield of the hexanitrodiphenylamine (see Section 2.5 below) and none of the tetra-derivative.

Vorozhtsov(ref.9) obtained 2,2',4,4'-tetranitrodiphenylamine in 97% yield by heating a mixture of 2,4-dinitroaniline, 2,4-dinitrochlorobenzene and potassium fluoride for 2.5 hours at 120 to 130°, according to the general equation:

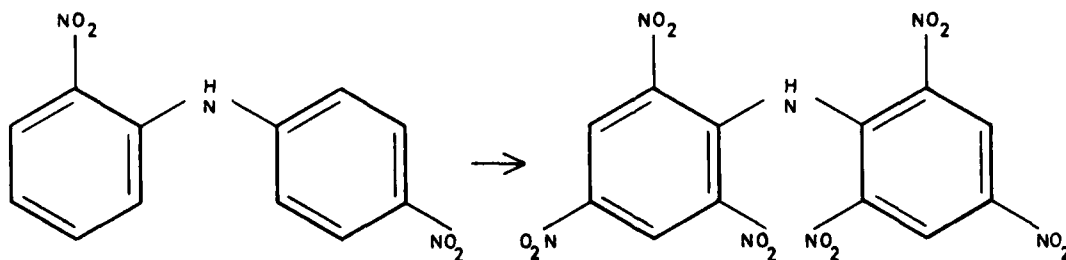


In this work, using the above conditions, none of the desired product was obtained and only starting materials were recovered. The following procedure was used:



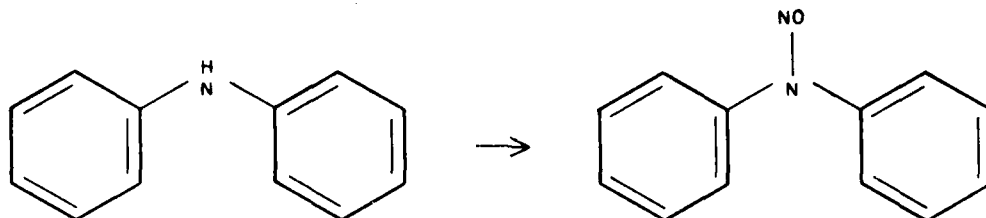
A mixture of 2,4-dinitroaniline (1.8 g), 2,4-dinitrochlorobenzene (2.0 g) and anhydrous potassium fluoride (1.2 g) was heated at 150° for 48 hours. The solidified reaction mass was pulverised, washed well with water (100 mL), hydrochloric acid (5%, 50 mL) and again with water. It was then washed with cold ethanol (300 mL) until no further red colour was extracted. The residue was then extracted with hot ethanol (200 mL), which upon cooling gave yellow-green crystals. Recrystallisation from absolute ethanol gave 2,2',4,4'-tetranitrodiphenylamine (1.03 g, 30%) as chlorine-green crystals, m.p. 200 to 201°, lit(ref.1,9) m.p. 199°.

2.5 2,2',4,4',6,6'-Hexanitrodiphenylamine(ref.12)



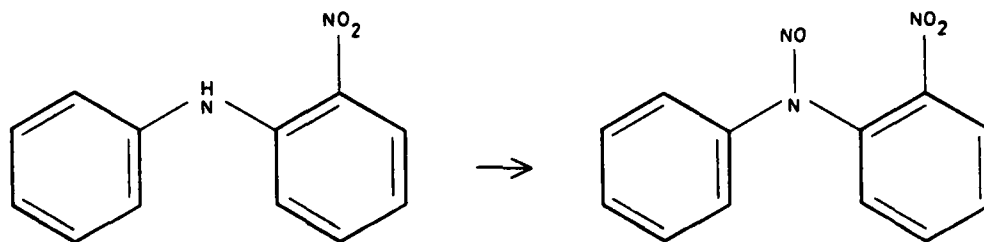
A solution of 2,4'-dinitrodiphenylamine (1.0 g) in nitric acid (70%, 50 mL) was heated at 40° for 1½ hours and then allowed to stand overnight. The yellow precipitate was filtered and washed with cold water until the washing was acid-free. The product was then dried at 100° for 2 hours. 2,2',4,4',6,6'-Hexanitrodiphenylamine (1.66 g, 98%) was obtained as yellow crystals, m.p. 234° (decomp), lit(ref.12) m.p. 240 to 244° (decomp), lit(ref.10) m.p. 235 to 236° (decomp).

2.6 N-nitrosodiphenylamine(ref.13-16)



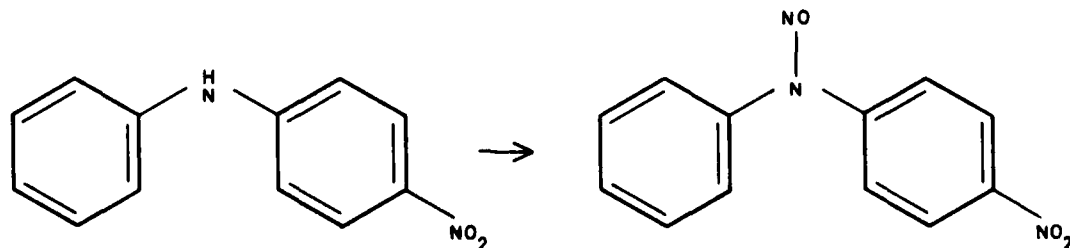
A sample of N-nitrosodiphenylamine was kindly provided by R.M. Kempson and had been obtained in 55% yield as pale yellow crystals m.p. 66.5 to 67°, lit(ref.13) m.p. 68°.

2.7 2-Nitro-N-nitrosodiphenylamine(ref.17)



A concentrated aqueous solution of sodium nitrate (10 mL) was added to a mixture of 2-nitrodiphenylamine (2.0 g) in glacial acetic acid. The mixture was stirred vigorously and then allowed to stand until all effervescence ceased. During this reaction the initial dark red solution changed to colourless and buff coloured granules began to crystallise. Water was added to fully precipitate the crystals which were filtered and then recrystallised from absolute ethanol to give 2-nitro-N-nitrosodiphenylamine (0.9 g, 39%) as buff coloured crystals m.p. 99 to 100°, lit(ref.17) m.p. 99 to 100°.

2.8 4-Nitro-N-nitrosodiphenylamine(ref.1,17)



A solution of 4-nitrodiphenylamine (2.0 g) in glacial acetic acid was treated in the same manner as outlined above. The crude product was heated in ethanol containing a little decolourising charcoal. Recrystallisation from ethanol gave 4-nitro-N-nitrosodiphenylamine (1.6 g, 70%) as cream coloured crystals m.p. 127 to 129⁰, lit(ref.1) m.p. 130 to 134⁰.

3. SPECTROPHOTOMETRIC DATA

The compounds studied in this work were characterised by comparison of their melting points, ultraviolet and infrared spectra with those published in the literature.

3.1

The ultraviolet spectra were determined on a Unicam SP700 Spectrophotometer in absolute ethanol using 0.5 cm cells. The results are summarised in Table 2.

3.2

The infrared spectra were recorded on a Perkin-Elmer 521 Grating Spectrophotometer using the potassium bromide disc technique. A brief discussion of the infrared data is given in generalised form in Table 3(ref.10,18). The recorded spectra are shown in figures 1 to 13.

4. DISCUSSION

Some of the nitro-derivatives of diphenylamine were difficult to prepare - problems were experienced in reproducing literature methods. In general yields were low. When mild reaction conditions were used only starting materials were recovered and when reaction conditions were severe only intractable tars were obtained (as shown by thin layer chromatography). Freshly prepared solutions of all compounds studied in this work showed only one component by HPLC, however, on standing for several months the solutions of nitro- and nitroso- derivatives showed evidence of compound instability (appearance of other peaks by HPLC). The nitro- and nitroso- derivatives of diphenylamine were subsequently stored in a refrigerator.

5. CONCLUSIONS

Eight nitro- and nitroso- derivatives of diphenylamine have been synthesised by (modified) literature methods, and characterised by their melting points, ultraviolet and infrared spectra. The experimental conditions employed ie concentration of reagents, use of solvents, degree of heating and time of reaction are important parameters which were often modified in order to achieve useful yields of the desired compounds.

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TABLE 1. MELTING POINTS OF COMPOUNDS STUDIED

Compound	Source	Method of Purification	Obtained*	Melting Point Literature
Diphenylamine	BDH	Recrystallised from isopropanol	53-53.5°	52.8-54.1°(ref.1)
2-Nitrodiphenylamine	Aldrich	Recrystallised from absolute ethanol	75.5-76°	75-75.5°(ref.1)
4-Nitrodiphenylamine	Aldrich	-	132-135°	132-133°(ref.1)
2,2'-Dinitrodiphenylamine	This work	Recrystallised from absolute ethanol	164-165°	166-167°(ref.7) 172-172.5°(ref.10)
2,4-Dinitrodiphenylamine	Hopkin & Williams	-	156-159°	156-159°(ref.6)
2,4'-Dinitrodiphenylamine	Hopkin & Williams	-	220-222°	219-223°(ref.10)
2,2',4'-Trinitrodiphenylamine	This work	Recrystallised from absolute ethanol	185-186°	183-186°(ref.10)
2,4,4'-Trinitrodiphenylamine	This work	Recrystallised from absolute ethanol	184-186°	181-189°(ref.10)
2,2',4,4'-Tetranitrodiphenylamine	This work	Recrystallised from absolute ethanol	200-201°	199°(ref.1)
2,2',4,4',6,6'-Hexanitrodiphenylamine	This work	-	234° dec	243-244° dec(ref.1) 234-236° dec(ref.10)
N-nitrosodiphenylamine	This work	Recrystallised from petroleum spirit	66.5-67°	66.5-67°(ref.1)
2-Nitro-N-nitrosodiphenylamine	This work	Recrystallised from absolute ethanol	99-100°	99-100°(ref.17)
4-Nitro-N-nitrosodiphenylamine	This work	Recrystallised from absolute ethanol	127-129°	130-134°(ref.1)

* All melting points (°C) obtained are corrected: they were done on a Koffler hot stage microscope.

TABLE 2. ULTRAVIOLET SPECTRAL DATA

Compound	λ_{max}^a (posn of max in abs. alcohol) nm		E_{mol}^b (molar extinction coefficient) $\times 10^{-4}$	
	Obtained	Lit(ref.1)	Obtained	Lit(ref.1)
Diphenylamine	285	285	2.05	2.03
2-Nitrodiphenylamine	420	422	0.63	0.66
4-Nitrodiphenylamine	387	390	2.09	2.12
2,2'-Dinitrodiphenylamine	406	417-23	0.51	0.94
2,4-Dinitrodiphenylamine	352	349-51	1.67	1.70
2,4'-Dinitrodiphenylamine	409	405	1.47	1.39
2,2',4-Trinitrodiphenylamine	378	370-75	1.31	1.43
2,4,4'-Trinitrodiphenylamine	376	365-67	1.35	2.16
2,2',4,4'-Tetranitrodiphenylamine	396	401-02	1.27	2.27
N-nitrosodiphenylamine	294	295	0.56	0.57
2-Nitro-N-nitrosodiphenylamine	none	none	-	-
4-Nitro-N-nitrosodiphenylamine	316	317-20	1.36	1.37

^a Other maxima were observed in many cases - the figure quoted is convenient for spectrophotometric determination.

^b $E_{\text{mol}} = \frac{A}{lC}$ where A = absorbance, l = pathlength (cm)

C = concentration (moles/L)

TABLE 3. INFRARED SPECTRAL DATA

Absorption Bands (microns)	Comments
2.9-3.0	Weak band due to N-H stretching found in all the compounds containing the NH group. Absent in the N-nitroso compounds.
3.2	Weak band due to C-H stretching, found in all the compounds.
6.2-6.4	Two strong bands caused by skeletal in-plane vibrations of the phenyl ring.
6.5-6.6	Strong band found in all the compounds containing NO ₂ . Caused by asymmetrical NO ₂ stretching vibrations.
6.6-6.7	Strong band due to skeletal in-plane vibration of the phenyl ring.
6.7-6.8	Medium to strong band found in the N-nitroso compounds, due to N=O stretching.
6.9	Medium band caused by skeletal in-plane vibration.
6.9-7.0	Weak band due to C-H stretching.
7.4-7.5	Strong band found in all the compounds containing NO ₂ . Caused by asymmetrical NO ₂ stretching vibrations.
7.6-7.8	Strong band caused by C-N stretching of the aromatic amine - only medium intensity in the N-nitroso compounds.
8.3-10.0	A series of bands, varying in intensity, caused by various substitutions in the aromatic ring and the C-H in-plane deformation modes of the remaining hydrogen atoms.
8.7-9.3	Medium band caused by different C-N modes.
10.8-10.9	Medium band found in 2,4-dinitrodiphenylamine, 2,2',4-trinitrodiphenylamine, 2,4,4'-trinitrodiphenylamine and 2,2',4,4'-tetranitrodiphenylamine. It occurs when the 2,4- positions are occupied.
11.7-12.0	Medium to strong band found in all the compounds containing NO ₂ , due to the C-N stretching mode. N-Nitrosodiphenylamine shows a weak band.
12.7-14.1	One, two or three bands appear in this region for each compound. This region would be expected to contain one or two bands that result from various patterns of phenyl substitution. Also, a band due to NO ₂ appears to be present. The exact assignments are difficult because of the marked effect of the NO ₂ groups in causing shifting of the phenyl groups from their normal position(ref.10,18).

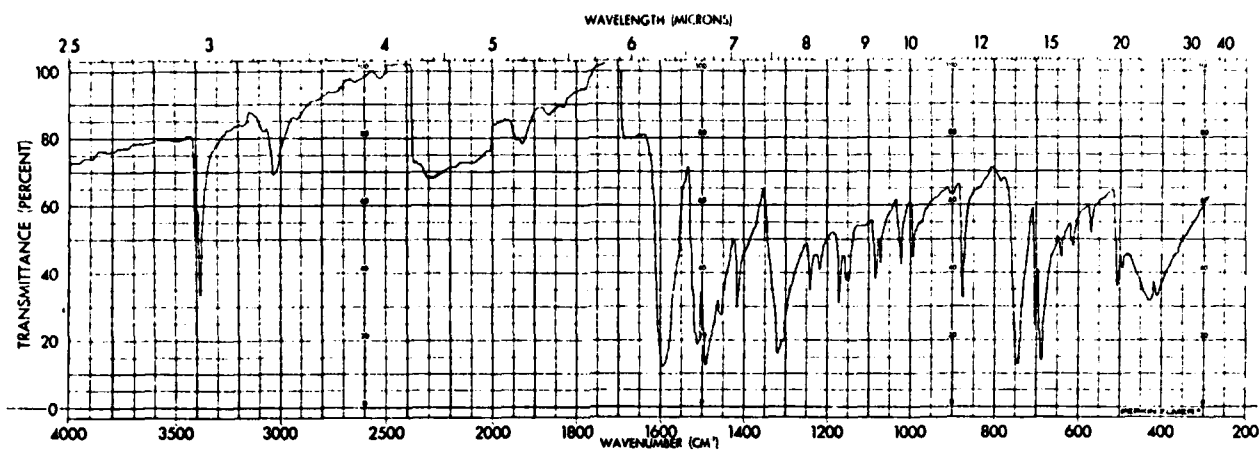


Figure 1. Infrared spectrum - Diphenylamine

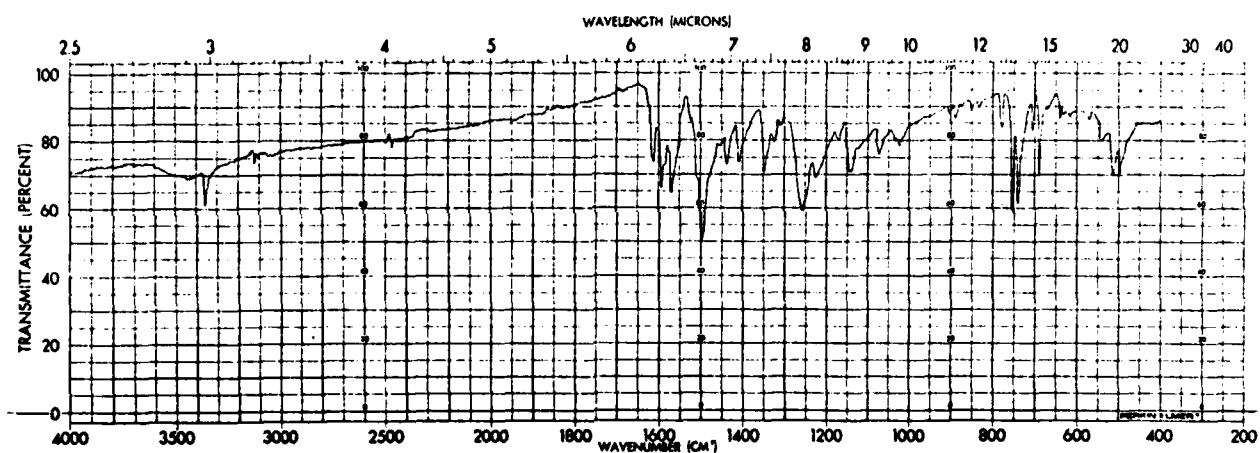


Figure 2. Infrared spectrum - 2-Nitrodiphenylamine

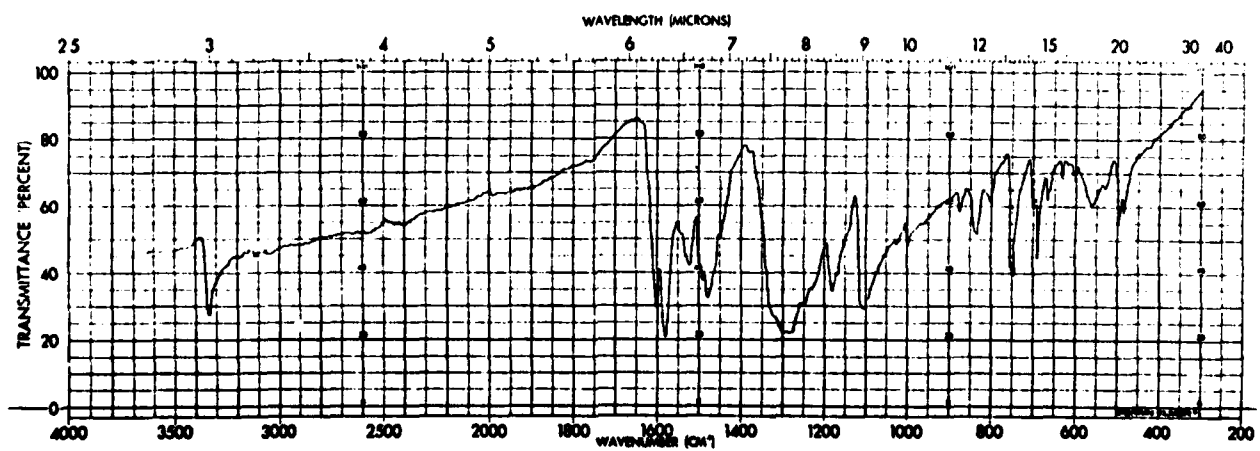


Figure 3. Infrared spectrum - 4-Nitrodiphenylamine

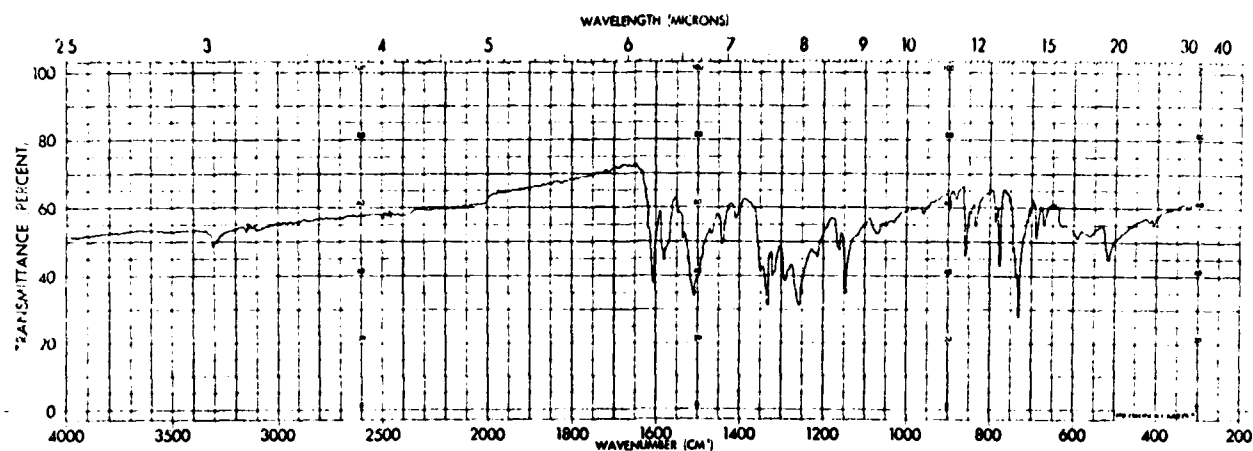


Figure 4. Infrared spectrum - 2,2'-Dinitrodiphenylamine

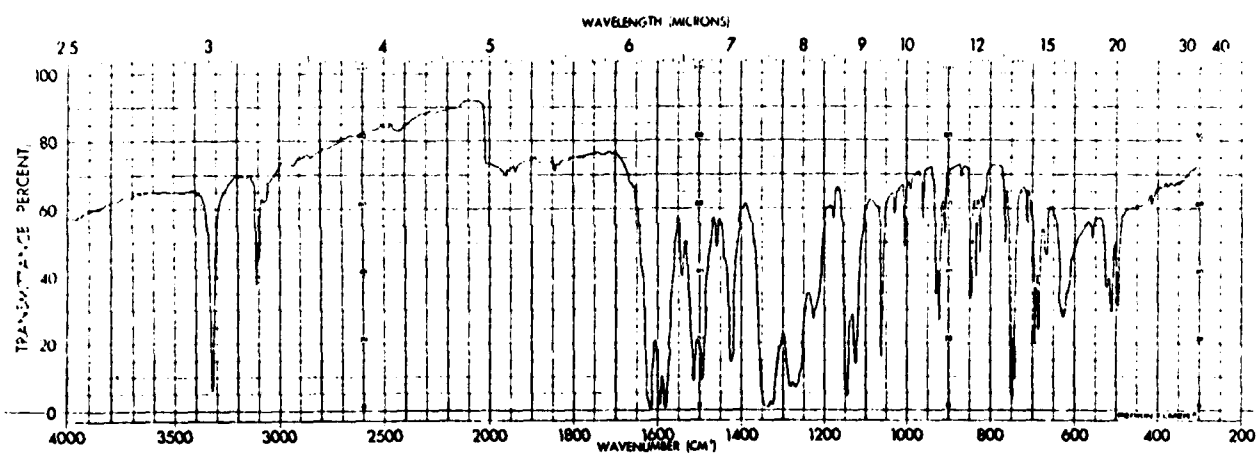


Figure 5. Infrared spectrum - 2,4-Dinitrodiphenylamine

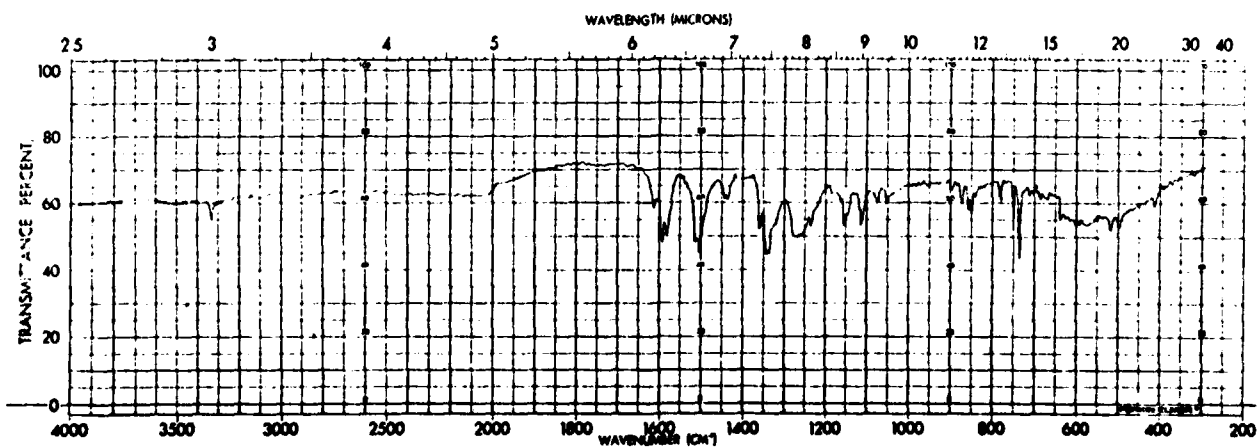


Figure 6. Infrared spectrum - 2,4'-Dinitrodiphenylamine

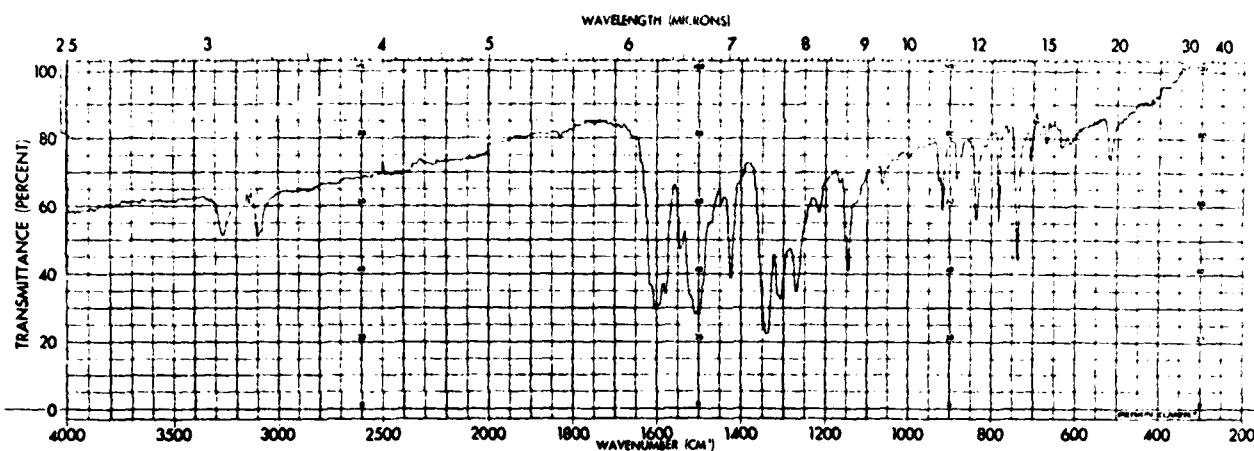


Figure 7. Infrared spectrum - 2,2',4-Trinitrodiphenylamine

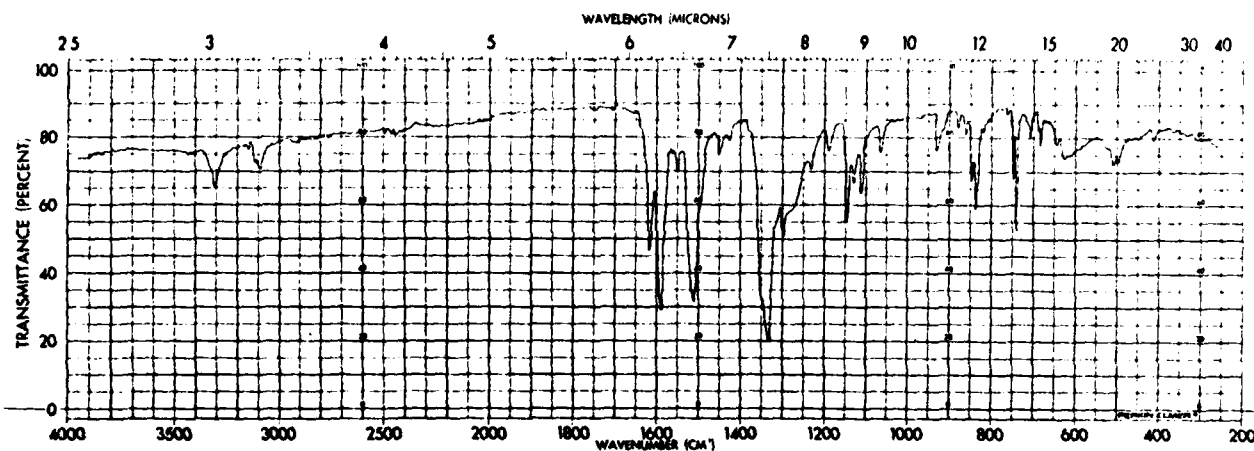


Figure 8. Infrared spectrum - 2,4,4'-Trinitrodiphenylamine

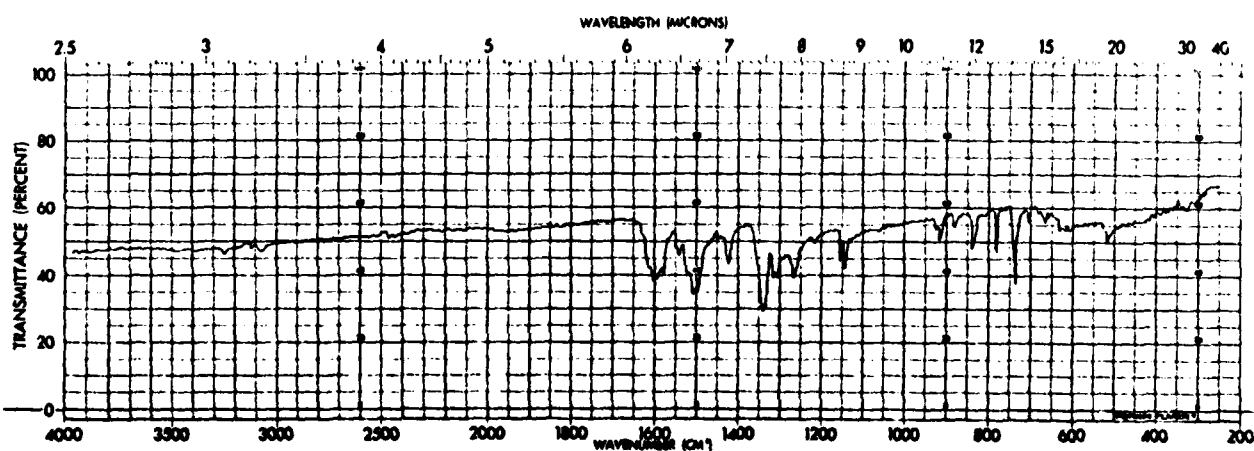


Figure 9. Infrared spectrum - 2,2',4,4'-Tetranitrodiphenylamine

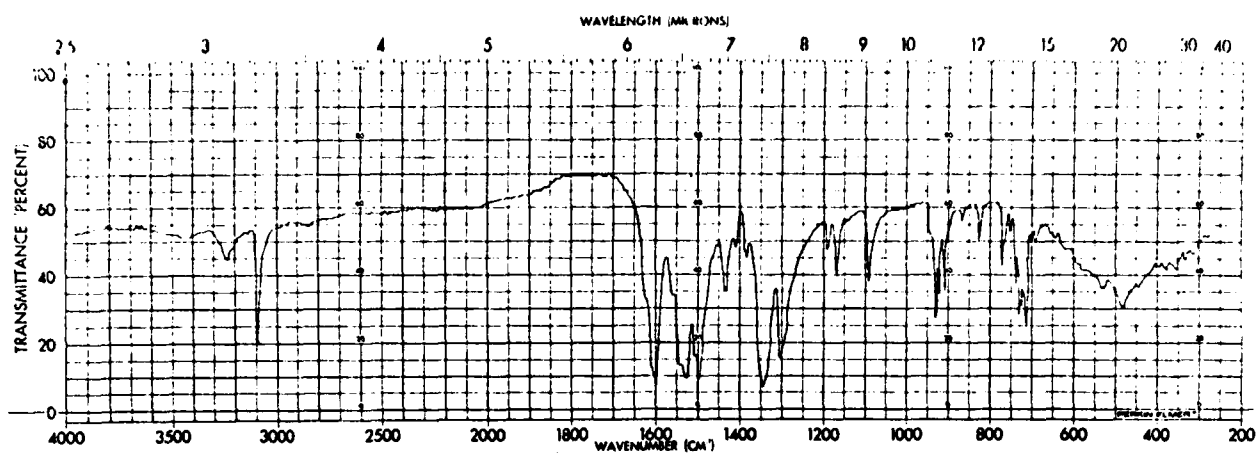


Figure 10. Infrared spectrum - 2,2',4,4',6,6'-Hexanitrodiphenylamine

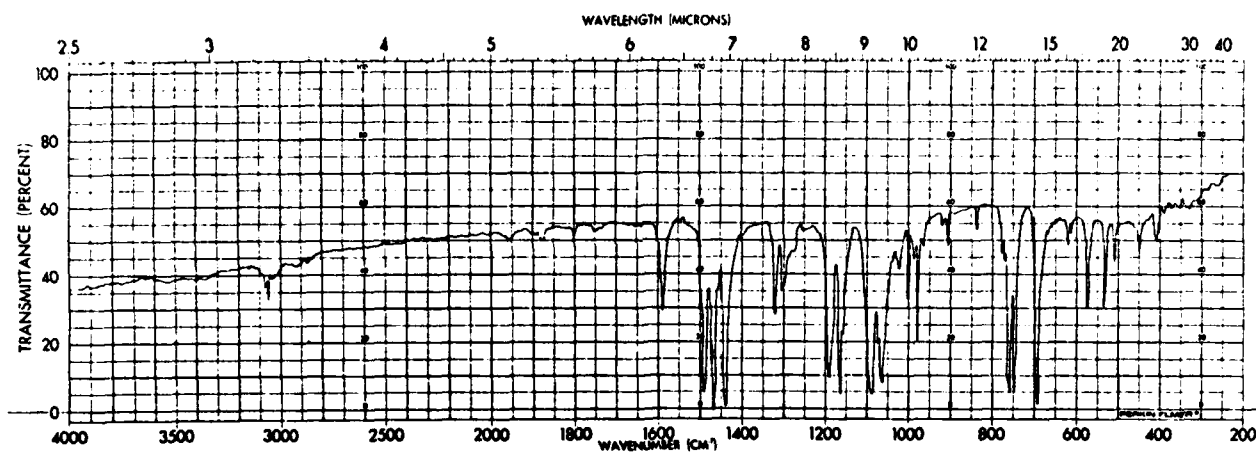


Figure 11. Infrared spectrum - N-Nitrosodiphenylamine

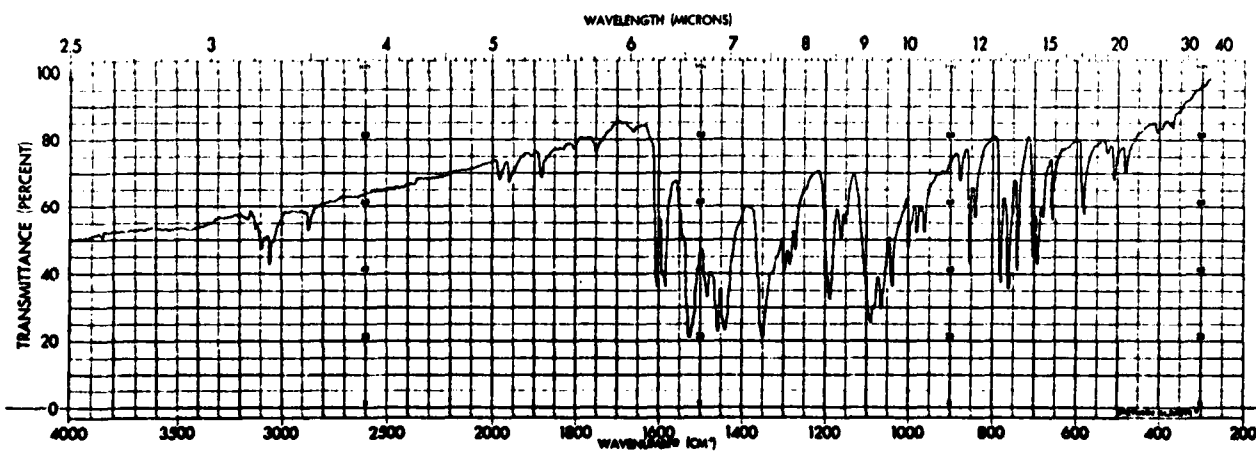


Figure 12. Infrared spectrum - 2-Nitro-N-nitrosodiphenylamine

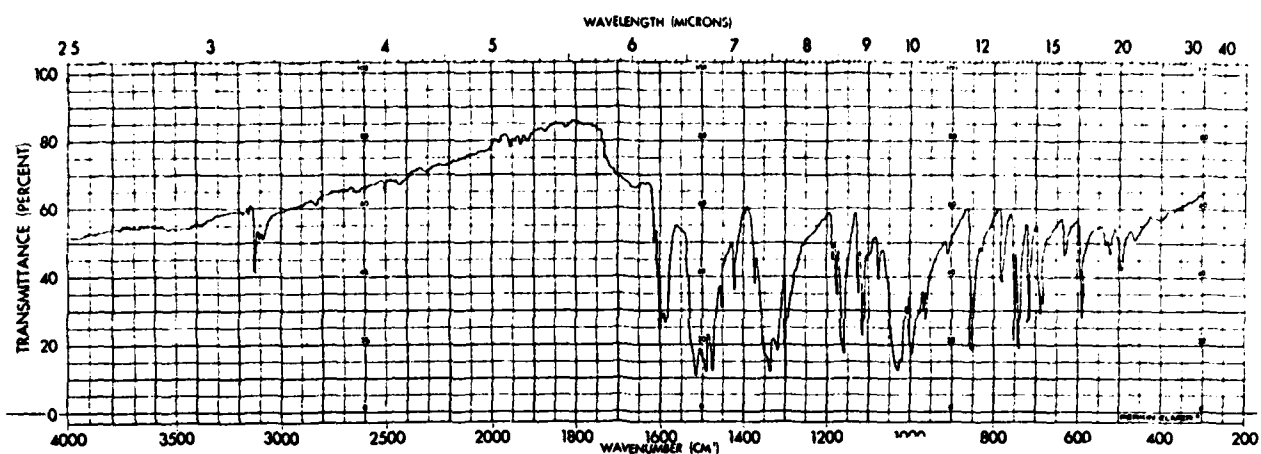


Figure 13. Infrared spectrum - 4-Nitro-N-nitrosodiphenylamine

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16 SUMMARY OR ABSTRACT:

(if this is security classified, the announcement of this report will be similarly classified)

A variety of nitro- and nitroso- compounds of diphenylamine have been prepared. Although literature methods of preparation were chosen, some of the published methods were modified in order to obtain a reasonable yield of the desired product. Most literature preparations gave low yields, and some of the methods were difficult to reproduce. All compounds studied in this work were characterised by their melting points and infrared and ultraviolet spectra.

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