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THE SYNTHESIS OF SOME NEW POLYNITRO-2,3-DIMETHYLNAPHTHALENES

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Abstract—The polynitration of derivatives of 2,3-dimethylnaphthalene has been investigated.

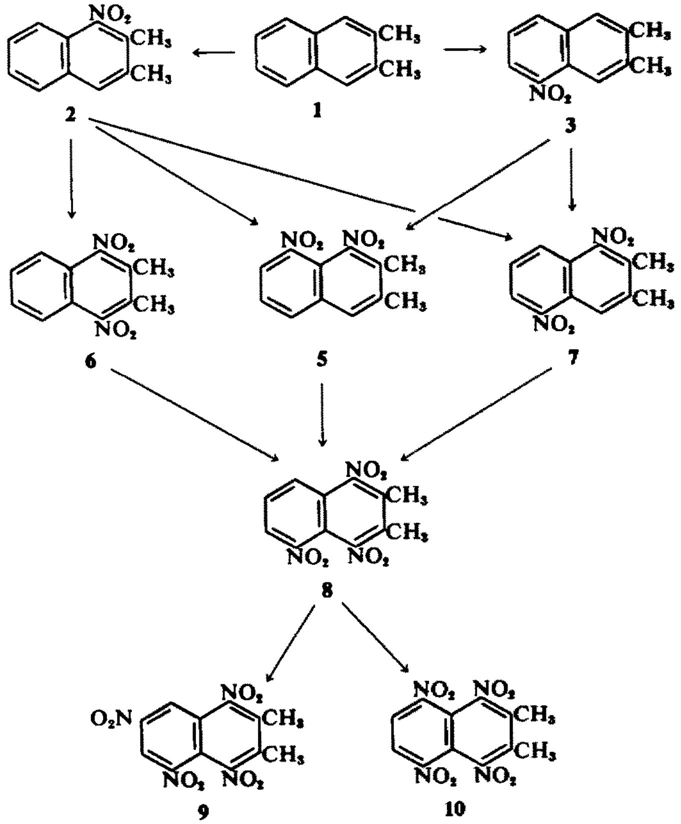
The nitration of methylbenzenes has been extensively studied, but relatively few investigations have been concerned with the nitration of methylnaphthalenes.

The mononitration of 1- and 2-methylnaphthalene has been reported in some detail,[1,2] as has the mononitration of a number of the isomeric dimethylnaphthalenes.[3] The synthesis of polynitrodimethylnaphthalenes has been limited to the preparation of dinitro derivatives,[4-7] except in the case of 2,6-dimethylnaphthalene from which a tetranitro derivative has been prepared.[7] The lack of information on synthetic routes to polynitrodimethylnaphthalenes is surprising since such compounds might be expected to exhibit explosive properties similar to the structurally similar polynitrotoluenes, and might be expected to be intermediates in the preparation of thermally stable explosives[8] having applications in space research.[9,10] In view of this, we have studied the nitration of methylnaphthalenes and dimethylnaphthalenes, and we now report on the synthesis of some new polynitro-2,3-dimethylnaphthalenes. Prior to this work, the only reported derivatives of 2,3-dimethylnaphthalene (1) were 1-nitro-2,3-dimethylnaphthalene (2),[3] 5-nitro-2,3-dimethylnaphthalene (3),[3] 6-nitro-2,3-dimethylnaphthalene (4)[3] and 1,8-dinitro-2,3-dimethylnaphthalene (5).[5]

Nitration of **1** with nitric acid in acetic anhydride gave a product mixture from which 1,4-dinitro-2,3-dimethylnapthalene (**6**), 1,5-dinitro-2,3-dimethylnaphthalene (**7**) and **5** were separated by fractional crystallisation and column chromatography. Undoubtedly the dinitro-compounds are formed via the intermediacy of **2** and **3** as shown in Scheme 1. In accord with this, the nitration of **2** in acetic anhydride yielded a mixture of **5**, **6** and **7**. The ratio of the isomers in the latter product mixture, as determined by NMR spectroscopy, was *ca* 1:1:1. It may be inferred from this ratio that the deactivating effect of the nitro group in the methyl substituted ring counterbalances to some extent the activating influence of the methyl groups, with the overall result that the rate of nitration in the methylated ring is similar to that for the non-methylated ring. Obviously, the presence of a nitro group in the non-methylated ring as in **3** would deactivate this ring towards nitration relative to the methylated ring and, as was to be expected, nitration of **3** with nitric acid/acetic anhydride gave a product mixture comprised solely of **5** and **7**. The NMR spectrum of the product mixture showed that the isomer ratio was *ca* 60:40, indicating that nitration occurred at the 4-position in **3** in preference to the 1-position. This presumably arises because the 4-position is "*meta*" to the nitro group in **3** and as such is a favoured site for nitration. Obviously electronic effects have a greater influence than steric effects in determining the preferred site of nitration since the 1-position in **3** is less sterically hindered than the 4-position.

Conversion of **5**, **6** and **7** into 1,4,5-trinitro-2,3-dimethylnaphthalene (**8**) can be achieved by nitration with the stoichiometric amount of nitric acid dissolved in sulphuric acid. As far as could be determined by NMR spectroscopy, **8** is the sole product in each of the reactions. Nitration of **8** in a mixture of nitric acid and sulphuric acid gives 1,4,5,7-tetranitro-2,3-dimethylnaphthalene (**9**) and a small yield of 1,4,5,8-tetranitro-2,3-dimethylnaphthalene (**10**). **9** can be isolated by fractional crystallization but it was not possible to isolate **10**. However, this latter compound can be synthesised by an indirect route (see below). The various steps in the conversion of 2,3-dimethylnaphthalene into the tetranitrocompounds **9** and **10** are summarised in Scheme 1.

The synthesis of polynitro-2,3-dimethylnaphthalenes having nitro substituents in the non-methylated ring only, or having a greater number of nitro groups in the non-methylated ring than in the methylated ring can be achieved via the intermediacy of 5-amino or 5-acetamido derivatives of 2,3-dimethylnaphthalene. For instance, 5-amino-8-nitro,2,3-dimethylnaphthalene (**11**) is obtained on treatment of **3** with alkaline hydroxylamine, and can be diazotised and treated with sodium

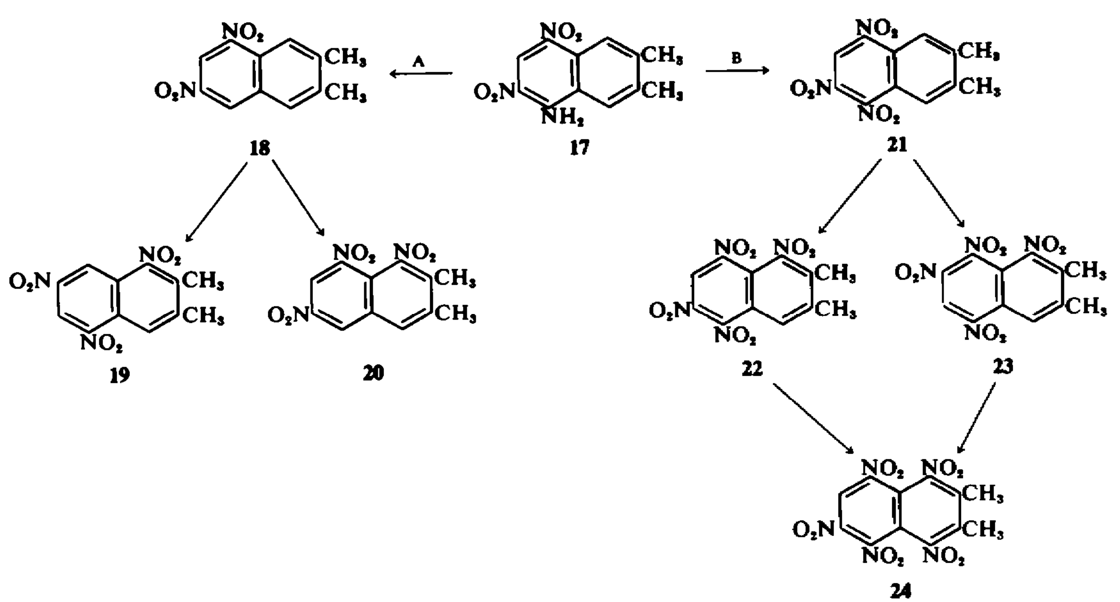


SCHEME 1

nitrite to yield 5,8-dinitro-2,3-dimethylnaphthalene (**12**). Nitration of this latter compound using the required amount of nitric acid for mononitration results in substitution in the methylated ring to give 1,5,8-trinitro-2,3-dimethylnaphthalene (**13**) as the only product. Nitration of **13** with excess of mixed acid takes the reaction through to the tetranitro stage, and here 1,4,5,8-tetranitro-2,3-dimethylnaphthalene (**10**) is the only product.

Reaction of 5-amino-2,3-dimethylnaphthalene (**14**) with a 1 : 1 mixture of acetic anhydride/acetic acid yields 5-acetamido-2,3-dimethylnaphthalene (**15**). The acetamido group in this latter compound activates the nonmethylated ring such that nitration using nitric acid/acetic anhydride results in substitution in the non-methylated ring only. Thus, dinitration of **15** gives 5-acetamido-6,8-dinitro-2,3-dimethylnaphthalene (**16**) as the only detectable product. Hydrolysis of **16** results in the formation of 5-amino-6,8-dinitro-2,3-dimethylnaphthalene (**17**), an intermediate which can be used to prepare tri-, tetra- and pentanitro-derivatives of 2,3-dimethylnaphthalene in which the number of nitro groups in the nonmethylated ring exceeds that in the methylated ring (see Scheme 2).

Route A in Scheme 2 is initiated by removing the amino group from **17** via diazotisation to yield 5,7-dinitro-2,3-dimethylnaphthalene (**18**). Nitration of the latter compound in mixed nitric/sulphuric acid using the stoichiometric amount of nitric acid for conversion to trinitro products gave a 30:70 mixture of 1,5,7-trinitro-2,3-dimethylnaphthalene (**19**) and 1,6,8-trinitro-2,3-dimethylnaphthalene (**20**). **19** and **20** were separated by column chromatography, and their structures assigned from their NMR and mass spectra. The compounds are not distinguishable from each other by their NMR spectra but are distinguishable by their mass spectra. Thus, the $(M-NO_2)^+$ ion gives rise to the base peak in the spectrum of **20**, a feature characteristic of polynitronaphthalenes having nitro groups in adjacent *peri*-positions,[11] whereas the M^+ ion gives rise to the base peak in the spectrum of **19**, a feature characteristic of polynitronaphthalenes having only one of the *peri*-positions occupied.[11] It is noteworthy that nitration of **18** occurred preferentially at the 4-position to give **20** as the major product. This is probably because the 4-position is a "*meta*" position in relation to the nitro groups at the 5- and 7-positions and as such is favoured for nitration over the 1-position which is an "*ortho*/*para*" position.



SCHEME 2

The synthesis of 5,6,8-trinitro-2,3-dimethylnaphthalene (21) was achieved by the conversion of the amino group of 17 into a nitro group, via diazotisation and treatment with sodium nitrite (route B of Scheme 2). The trinitro compound (21) is of interest since the three nitro groups are in the non-methylated ring, leaving two highly activated sites vacant in the methylated ring. Nitration of 21 in nitric acid/sulphuric acid using the stoichiometric amount of nitric acid for conversion to the tetranitro stage gave a mixture of 1,5,6,8-tetranitro-2,3-dimethylnaphthalene (22) and 1,5,7,8-tetranitro-2,3-dimethylnaphthalene (23). The nitration of 21 in excess of nitric acid/sulphuric acid yielded 1,4,5,6,8-pentanitro-2,3-dimethylnaphthalene (24) as the sole product. Undoubtedly, the formation of this latter compound proceeds via the intermediacy of the tetranitro derivatives 22 and 23 as shown in Scheme 2.

EXPERIMENTAL

Unless otherwise stated, column chromatography was carried out with Merck silica gel (0·05–0·2 mm) using 400 × 20 mm columns, and elution with 1 : 1 benzene-petroleum (b.p. 60–80°). Mass spectral molecular weights were determined with an AEI MS9 mass spectrometer. NMR spectra were recorded on a Perkin-Elmer R10 spectrometer (tetramethylsilane as internal reference). The structures of the nitration products were assigned from NMR and mass spectra. In particular, the following criteria were used in the assignments from the NMR spectra: (a) a slightly broadened methyl resonance indicated coupling to an *ortho* proton at either the 1- or 4-positions in 2,3-dimethylnaphthalene, (b) a sharp methyl resonance indicated a nitro group in either the 1- or 4-positions.

Nitration of 2,3-dimethylnaphthalene (1). Nitric acid (*d* 1·42; 1 ml) was added dropwise during 0·5 h to a solution of 1 (1 g) in acetic anhydride (10 ml) maintained in an ice-bath so as to keep the temperature below 10°. The mixture was then maintained at 20° for 2 h, and poured into water (50 ml). The precipitated product was filtered off, washed with water, and dried *in vacuo* to give a pale yellow solid (0·98 g). The first crop from recrystallisation of this solid from petroleum (b.p. 60–80°) gave 1,8-*dinitro-2,3-dimethylnaphthalene* (5) (0·3 g), m.p. 243–5° (lit,[5] 245°). The remaining crops were combined and column chromatographed to give three fractions. Evaporation to dryness of the first fraction gave 1,4-*dinitro-2,3-dimethylnaphthalene* (0·26 g, 17%), m.p. 197°. (Found: C, 58·4; H, 4·2; N, 11·1. $C_{12}H_{10}N_2O_4$ requires C, 58·5; H, 4·1; N, 11·4%); τ ($CDCl_3$) 2·31 (4H, s) and 7·58 (6H, s); *m/e* 246 (M^+). Evaporation of the second fraction to dryness gave 1,5-*dinitro-2,3-dimethylnaphthalene* (0·35 g, 22%), m.p. 160°. (Found: C, 58·5; H, 4·6; N, 11·3. $C_{12}H_{10}N_2O_4$ requires C, 58·5; H, 4·1; N, 11·4%); τ ($CDCl_3$) 1·5–2·5 (4H, m), 7·49 (3H, s) and 7·65 (3H, s, broadened); *m/e* 246 (M^+). Evaporation of the third fraction to dryness gave 5 (0·08 g).

Nitration of 1-nitro-2,3-dimethylnaphthalene (2). Nitric acid (*d* 1·42; 0·5 ml) was added dropwise during 0·5 h to a solution of 2 (1 g) in acetic anhydride (10 ml), the temperature being maintained below 10°. After standing at 20° for 1 h, the solution was poured into cold water (50 ml). The precipitate was filtered off, washed with water, and dried *in vacuo* to give a pale yellow solid (0·97 g). The NMR spectrum of a sample of the product showed it to be a 1 : 1 : 1 mixture of 5, 6 and 7.

Nitration of 5-nitro-2,3-dimethylnaphthalene (3). Nitric acid (*d* 1·42; 0·5 ml) was added dropwise during 0·5 h to a solution of 3 (1 g) in acetic anhydride (10 ml), the temperature being maintained below 10, in an ice-bath. After standing at 20° from 1 h, the solution was poured into water (50 ml). The precipitate was filtered off, washed with water, and dried *in vacuo* to give a pale yellow solid (1·06 g). The NMR spectrum of a sample of the product showed it to be a *ca* 60 : 40 mixture of 5 and 7.

Preparation of 1,4,5-trinitro-2,3-dimethylnaphthalene

(**8**). A solution of **5** (2 g) in sulphuric acid (*d* 1·84; 30 ml) was prepared, and nitric acid (*d* 1·42; 0·5 ml) added with stirring. The mixture was allowed to stand at 20° for 2 h, and poured on to crushed ice (50 g). The precipitate was filtered off, washed with water, and dried *in vacuo* to give **8** (1·9 g, 81%), m.p. 175°. (Found: C, 49·3; H, 3·3; N, 14·5. $C_{12}H_9N_3O_6$ requires C, 49·5; H, 3·1; N, 14·4%); τ ($CDCl_3$) 1·75–2·3 (3H, m), 7·5 (3H, s) and 7·55 (3H, s); *m/e* 291 (M^+). Using the same procedure, **7** (2 g) gave **8** (2·0 g, 88%). There was no evidence for any other compound in the NMR spectrum of the crude product. Using the same procedure **6** (2 g) gave **8** (1·6 g, 68%). There was no evidence for any other compound in the NMR spectrum of the crude product.

Nitration of **8**. Nitric acid (*d* 1·42; 1·5 ml) was added to a solution of **8** (1 g) in sulphuric acid (*d* 1·84; 30 ml). The mixture was stirred for 1 h, and then poured on to crushed ice (50 g). The precipitate was filtered off, and dried *in vacuo* to give a white solid (1·05 g). The NMR spectrum of a sample of the product in $CDCl_3$ showed that it was a *ca* 95:5 mixture of 1,4,5,7-*tetranitro*-2,3-*dimethylnaphthalene* (**9**) and 1,4,5,8-*tetranitro*-2,3-*dimethylnaphthalene* (**10**). τ (**9**) 1·07 and 1·22 (2H, ABq, J_{AB} 2 Hz), 7·37 (3H, s) and 7·44 (3H, s); (**10**) 1·80 (2H, s) and 7·42 (6H, s). Recrystallisation of the product mixture from glacial acetic acid gave pure **9** (0·97 g, 84%), m.p. 279°. (Found: C, 42·9; H, 2·5; N, 16·2. $C_{12}H_8N_4O_8$ requires C, 42·9; H, 2·4; N, 16·7%); *m/e* 336 (M^+). Evaporation of the mother liquor to dryness gave a residue from which it was not possible to isolate **10**, since column chromatography of the mixture using various stationary phases resulted in rapid decomposition in each case.

Preparation of 5,8-dinitro-2,3-dimethylnaphthalene (**12**). A soln of **3** (1·1 g) in 95% ethanol (10 ml) was added to a soln of hydroxylamine hydrochloride (3 g) in 95% ethanol (50 ml), and the mixture maintained at 60° whilst a solution of potassium hydroxide (10 g) in methanol (30 ml) was added dropwise, with stirring, during 1 h. The mixture was stirred for a further 2 h, and poured into water (300 ml). The precipitate was filtered off, washed with water and dried. Recrystallisation from 95% ethanol gave 5-*amino*-8-*nitro*-2,3-*dimethylnaphthalene* (**11**) (0·7 g, 54%), m.p. 188°. (Found: C, 66·4; H, 5·5; N, 13·0%. $C_{12}H_{12}N_2O$ requires C, 66·7; H, 5·6; N, 13·0%). **11** (1 g) was added to a mixture of sulphuric acid (*d* 1·84; 4 ml) and glacial acetic acid (25 ml), and cooled to 5° in an ice-bath. The cooled mixture was added to a soln of sodium nitrite (5 g) in sulphuric acid (*d* 1·84; 5 ml). After standing for 0·5 h, the red diazonium mixture was poured into a mixture of sodium nitrite (10 g), sodium bicarbonate (20 g) and water (200 ml). The brown precipitate (0·7 g) was filtered off, dried, and chromatographed to give **12** (0·06 g, 5%), m.p. 177°. (Found: C, 58·2; H, 4·4; N, 11·3. $C_{12}H_{10}N_2O_4$ requires C, 58·5; H, 4·1; N, 11·4%); τ ($CDCl_3$) 1·82 (2H, s), 2·01 (2H, s) and 7·48 (6H, s, broadened); *m/e* 246 (M^+).

Nitration of **12**. A solution of nitric acid (*d* 1·42; 0·25 ml) in sulphuric acid (*d* 1·84; 25 ml) was prepared, and a portion (0·75 ml) added to a soln of **12** (0·03 g) in sulphuric acid (*d* 1·84; 3 ml). The mixture was stirred for 2 h, and poured on to crushed ice (20 g). The precipitate was filtered off, washed with water, and dried *in vacuo* to give 1,5,8-*trinitro*-2,3-*dimethylnaphthalene* (**13**) (0·025 g, 70%), m.p. 170°. (Found: C, 49·2; H, 3·5; N, 14·1. $C_{12}H_9N_3O_6$ requires C, 49·5; H, 3·1; N, 14·4%); τ ($CDCl_3$) 1·67 (1H, s), 1·88 (2H, s), 7·38 (3H, s, broadened) and 7·48 (3H, s); *m/e* 291 (M^+).

Nitration of **13**. Nitric acid (*d* 1·42; 0·05 ml) was added to a soln of **13** (0·02 g) in sulphuric acid (*d* 1·84; 5 ml). The mixture was stirred for 1 h, and poured on to crushed ice (20 g). The precipitate was washed with water and dried *in vacuo* to give **10** (0·02 g, 87%), m.p. 160°. (Found: C, 42·3; H, 2·1; N, 16·1. $C_{12}H_8N_4O_8$ requires C, 42·9; H, 2·4; N, 16·7%); τ ($CDCl_3$) 1·80 (2H, s) and 7·42 (6H, s); *m/e* 336 (M^+).

Preparation of 5,7-dinitro-2,3-dimethylnaphthalene (**18**). A soln of **3** (8·5 g) in 95% ethanol (150 ml) was refluxed with hydrazine hydrate (15 ml) in the presence of 10% Palladium on charcoal (0·1 g) for 10 h. Hot filtration, followed by cooling, gave 5-*amino*-2,3-*dimethylnaphthalene* (**14**) (7·0 g, 96%), m.p. 65°. (Found: C, 84·3; H, 7·5; N, 8·1. $C_{12}H_{13}N$ requires C, 84·2; H, 7·6; N, 8·2%). **14** (7 g) was added to a mixture of glacial acetic acid (25 ml) and acetic anhydride (25 ml). Cooling resulted in the separation of 5-acetamido-2,3-dimethylnaphthalene (**15**) which was filtered off, washed with water, and dried (8·0 g, 92%), m.p. 162°. (Found: C, 77·7; H, 6·8; N, 6·5. $C_{14}H_{15}NO$ requires C, 77·8; H, 7·0; N, 6·6%). **15** (8 g) was dissolved in acetic anhydride (100 ml), and nitric acid (*d* 1·50; 4 ml) added dropwise, the temp being maintained below 10° in an ice-bath. The mixture was poured into water (500 ml) and the product filtered off and dried. Recrystallisation from 95% ethanol gave 5-*acetamido*-6,8-*dinitro*-2,3-*dimethylnaphthalene* (**16**) (4·6 g, 41%), m.p. 190°. (Found: C, 55·0; H, 4·2; N, 13·7. $C_{14}H_{13}N_3O_5$ requires C, 55·5; H, 4·3; N, 13·9%). **16** (4·6 g) was refluxed for 16 h in 95% ethanol containing 70% sulphuric acid (15 ml). Cooling and neutralising the reaction mixture with N sodium hydroxide (300 ml) resulted in the precipitation of 5-*amino*-6,8-*dinitro*-2,3-*dimethylnaphthalene* (**17** , which was filtered off and dried (3·0 g, 76%), m.p. 150°. (Found: C, 55·0; H, 4·2; N, 16·0. $C_{12}H_{11}N_3O_4$ requires C, 55·2; H, 4·2; N, 16·1%). **17** (0·6 g) was added to a mixture of sulphuric acid (*d* 1·84; 2 ml) and glacial acetic acid (15 ml), the resulting mixture cooled to 5° in an ice-bath, and then added to a solution of sodium nitrite (1 g) in sulphuric acid (*d* 1·84; 5 ml). After standing for 0·5 h, the red diazonium solution was poured into a soln of ferrous sulphate (10 g) and ethylene glycol (20 ml) in water (30 ml). The brown precipitate was filtered off, dried and column chromatographed to give **18** (0·08 g, 14%), m.p. 156°. (Found: C, 58·2; H, 4·5; N, 11·3. $C_{12}H_{10}N_2O_4$ requires C, 58·5; H, 4·1; N, 11·4%); τ ($CDCl_3$) 1·10 (2H, s), 1·60 (1H, s), 2·08 (1H, s), 7·43 (3H, s, broadened) and 7·47 (3H, s, broadened) (singlet at 1·10 splits into an ABq on addition of C_6D_6); *m/e* 246 (M^+).

Nitration of **18**. Nitric acid (*d* 1·42; 0·12 ml) was added to a soln of **18** (0·5 g) in sulphuric acid (*d* 1·84; 20 ml). The mixture was stirred at 20° for 2 h, and then poured on to crushed ice (50 g). The precipitate was filtered off, washed with water, and dried *in vacuo* to give a pale yellow solid (0·4 g). Column chromatography of the solid yielded two fractions. Evaporation of the first fraction to dryness gave 1,5,7-*trinitro*-2,3-*dimethylnaphthalene* (**19**) (0·095 g, 16%), m.p. 169° (yellow needles). (Found: C, 49·3; H, 3·5; N, 14·6. $C_{12}H_9N_3O_6$ requires C, 49·5; H, 3·1; N, 14·4%); τ ($CDCl_3$) 1·05 and 1·21 (2H, ABq, J_{AB} 2 Hz), 1·46 (1H, s), 7·35 (3H, s, broadened) and 7·54 (3H, s); *m/e* 291 (M^+) (base peak at *m/e* 291). Evaporation of the second fraction to dryness gave 1,6,8-*trinitro*-2,3-*dimethylnaphthalene* (**20**) (0·232 g, 39%), m.p. 178° (white crystals). (Found: C, 49·6; H, 3·3; N, 14·7. $C_{12}H_9N_3O_6$ requires C, 49·5; H, 3·1; N, 14·4%); τ ($CDCl_3$) 1·05 and 1·21 (2H, ABq, J_{AB} 2 Hz), 1·84 (1H, s), 7·35

(3H, s, broadened), 7·46 (3H, s); *m/e* 291 (M^+) (base peak at *m/e* 245).

Preparation of 5,6,8-*trinitro*-2,3-*dimethylnaphthalene* (**21**). **17** (0·9 g) was added to a mixture of sulphuric acid (*d* 1·84; 1·5 ml) and glacial acetic acid (15 ml), and cooled to 5° in an ice-bath. The mixture was added to a soln of sodium nitrite (1 g) in sulphuric acid (*d* 1·84; 4 ml), and allowed to stand for 0·5 h. The red diazonium solution was poured into a mixture of sodium nitrite (7 g), sodium bicarbonate (15 g), and water (150 ml) to give a buff coloured precipitate, which was filtered off and dried. The product (0·3 g) was chromatographed on a column of silica gel (350 × 20 mm) using benzene as eluent to give **21** (0·15 g, 18%), m.p. 152°. (Found: C, 50·2; H, 3·6; N, 13·9. $C_{12}H_9N_3O_6$ requires C, 49·5; H, 3·1; N, 14·4%); τ ($CDCl_3$) 1·22 (1H, s), 1·61 (1H, s), 2·35 (1H, s), 7·40 (3H, s, broadened) and 7·45 (3H, s, broadened); *m/e* 291 (M^+).

Nitration of **21**. A solution of nitric acid (*d* 1·42; 2·5 ml) in sulphuric acid (*d* 1·84; 25 ml) was prepared, and a portion (0·4 ml) added to a soln of **21** (0·2 g) in sulphuric acid (*d* 1·84; 6 ml). The mixture was maintained at 35° for 1 h, and then poured on to crushed ice (10 g). The resulting precipitate was filtered off, washed with water, and dried *in vacuo* to give a white solid (0·2 g). Elemental analysis showed that the product was a mixture of tetranitro-2,3-dimethylnaphthalenes. (Found: C, 42·9; H, 2·3; N, 16·3. $C_{12}H_8N_4O_8$ requires C, 42·9; H, 2·4; N, 16·7%). The NMR spectrum of a sample of the product in $CDCl_3$ showed that it was a *ca* 70:30 mixture of 1,5,7,8-*tetranitro*-2,3-*dimethylnaphthalene* (**23**) and 1,5,6,8-*tetranitro*-2,3-*dimethylnaphthalene* (**22**); τ (**23**) 1·23 (1 H, s), 2·18 (1H, s), 7·35 (3H, s, broadened) and 7·43 (3H, s); τ (**22**) 1·40 (1H, s), 1·60 (1H, s), 7·43 (3H, s, broadened) and 7·54 (3H, s). All attempts to separate the mixture by column chromatography failed due to decomposition of the products on the various stationary phases used.

Nitric acid (*d* 1·42; 1 ml) was added to a soln of **21** (0·13 g) in sulphuric acid (*d* 1·84; 10 ml). The mixture was stirred at 50° for 2 hr, then poured on to crushed ice (50 g). The resulting precipitate was filtered off, washed with water, and dried *in vacuo* to give 1,4,5,6,8-*pentanitro*-2,3-*dimethylnaphthalene* (**24**) (0·05 g, 34%), m.p. 175°. (Found: C, 37·2; H, 2·4; N, 17·8. $C_{12}H_7N_5O_{10}$ requires C, 37·8; H, 1·8; N, 18·4%); τ (CD_3COCD_3) 0·81 (1H, s), 7·29 (3H, s) and 7·36 (3H, s); *m/e* 381 (M^+).

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