

CHAPTER III

EXPERIMENTAL

3.1 Apparatus

3.1.1 Fourier-Transform NMR Spectrometer

Model AC-F 200 (200 MHz), Bruker Spectrospin

3.1.2 Fourier-Transform Infrared Spectrophotometer

Model Impact 410, Nicolet

3.1.3 Elemental Analyzer

Series II, CHNS/O Analyzer 2400, Perkin Elmer

3.1.4 Pensky-Martens Closed Flash Tester (ASTM D93)

Model ISL DMFP-93, Perzoo

3.1.5 Colorimeter (ASTM D1500)

The Fisher ASTM colorimeter

3.1.6 Apparatus for distillation (ASTM D86)

3.1.7 Cooperative fuel research council engine (ASTM D 613)

Model Wis 53186, Waukesha Motor Co., Inc.

3.2 Chemicals

Reagents were obtained from various suppliers as shown in Table 3.1.

Table 3.1 Source of chemicals

Materials	Company
Triethylene glycol, reagent grade	Fluka
1,5-pentanediol, reagent grade	Fluka
1,4-butanediol, reagent grade	Fluka
1,2-ethanediol, reagent grade	Fluka
Urea, reagent grade	Fluka
Chloroform, reagent grade	Fluka
Methylene Chloride, reagent grade	Fluka
Ether, reagent grade	Merck
Anhydrous sodium sulfate, reagent grade	Fluka
Sulfuric acid 94%, reagent grade	J.T. baker
Nitric acid 70%, reagent grade	AJAX chemicals
Ammonia solution 25%, reagent grade	AJAX chemicals
Sodium bicarbonate solution 10%, reagent grade	Fluka
Sodium carbonate solution 10%, reagent grade	Fluka
Anhydrous magnesium sulfate, reagent grade	Fluka
Dimethyl formamide(DMF) , reagent grade	Merck
Ethyl acetate , reagent grade	Fluka
Methanol , reagent grade	Fluka
Ethanol , reagent grade	Fluka
Hydrochloric acid 37 %, reagent grade	Fluka
Base diesel fuels	Oil Refinery

3.3 Synthesis of dinitrate compounds [20, 21]

3.3.1 Synthesis of triethylene glycol dinitrate

To 100 ml of dichloromethane, nitric acid (25.20 g, 0.41 moles), and sulfuric acid (14.71 g, 0.15 moles) were added. The well-agitated mixture was added slowly to triethylene glycol (15.01g, 0.10 moles) while maintaining the temperature at 0-2 °C. After the completion of addition, stirring was discontinued, and the mixture allowed to separate. The organic layer was separated and washed with water to remove nitric acid, then mixture was neutralized with 25% ammonia solution. The aqueous layer was separated, and the organic solvents was stripped off on a rotary evaporator to give 16.90 g (70.4 % yield) of triethylene glycol dinitrate as colorless liquid.

3.3.2 Synthesis of 1,5-pentane dinitrate

A reactor was charged with the mixed acids containing nitric acid (20.40 g, 25.52 % wt.), sulfuric acid (47.10 g, 57.32 %wt.) and water (14.40 g , 17.16 %wt.). The mixed acids was agitated and its temperature adjusted to 25 ± 3 °C. Then urea (2.46 g) was added to the mixed acids over a period of 1/2 hour and the temperature was allowed to rise to 32.5 ± 2.5 °C. Then , 1,5-pentanediol (14.15 g, 0.1359 mole) was slowly added to the reactor while maintaining the temperature at 32.5 ± 2.5 °C. After addition of 1,5-pentanediol, the reaction mixture was held at 32.5 ± 2.5 °C for complete separation. The agitation was stopped and the bottom acid layer was drained off from the reactor. The product was washed twice with 10 % aqueous sodium sulfate solution, once with 10% aqueous sodium carbonate solution and finally with

10% sodium sulfate solution. After the aqueous layer was separated, 18.43 g (69.8 % yield) of 1,5-pentane dinitrate was obtained as colorless liquid.

3.3.3 Synthesis of 1,4-butane dinitrate

A reactor was charged with the mixed acids containing nitric acid (20.40 g, 25.52 % wt.), sulfuric acid (47.10 g, 57.32 %wt.) and water (14.40 g, 17.16 %wt.). The mixed acids was agitated and its temperature adjusted to 25 ± 3 °C. Then urea (2.46 g) was added to the mixed acids over a period of 1/2 hour and the temperature was allowed to rise to 32.5 ± 2.5 °C. Then , 1,4-butanediol (12.25 g, 0.1359 mole) was slowly added to the reactor while maintaining the temperature at 32.5 ± 2.5 °C. After addition of 1,4-butanediol, the reaction mixture was held at 32.5 ± 2.5 °C for complete separation. The agitation was stopped and the bottom acid layer was drained off from the reactor. The product was washed twice with 10 % aqueous sodium sulfate solution, once with 10% aqueous sodium carbonate solution and finally with 10% sodium sulfate solution. After the aqueous layer was separated, 17.45 g (71.3 % yield) of 1,4-butane dinitrate was obtained as colorless liquid.

3.3.4 Synthesis of 1, 2-ethane dinitrate

A reactor was charged with the mixed acids containing nitric acid (20.40 g, 25.52 % wt.), sulfuric acid (47.10 g, 57.32 %wt.) and water (14.40 g, 17.16 %wt.). The mixed acids was agitated and its temperature adjusted to 25 ± 3 °C. Then urea (2.46 g) was added to the mixed acids over a period of 1/2 hour and the temperature was allowed to rise to 32.5 ± 2.5 °C. Then, 1,2-ethanediol (8.44 g, 0.1359 mole) were slowly added to the reactor while maintaining the temperature at 32.5 ± 2.5 °C. After addition of 1,2-ethanediol,

the reaction mixture was held at 32.5 ± 2.5 °C for complete separation. The agitation was stopped and the bottom acid layer was drained off from the reactor. The product was washed twice with 10 % aqueous sodium sulfate solution, once with 10% aqueous sodium carbonate solution and finally with 10% sodium sulfate solution. After the aqueous layer was separated, 13.50 g (65.4 % yield) of 1,2-ethane dinitrate was obtained as colorless liquid.

3.4 Determination of properties of the synthesized compounds

3.4.1 Characterization of the synthesized compounds by using instruments as follow :

1. Fourier-Transform Infrared Spectrophotometer (FTIR)
2. Fourier-Transform NMR Spectrometer
3. Elemental Analyzer (EA)

3.4.2 Determination of cetane index of base diesel fuel blended with different concentration of dinitrate compounds (0.05 and 0.10 % by weight) as follow :

1. Triethylene glycol dinitrate
2. 1,5-pentane dinitrate
3. 1,4-butane dinitrate
4. 1,2-ethane dinitrate

1. The blended base diesel fuels was obtained by blending dinitrate compounds (0.05 and 0.10 g.) with base diesel fuel (1000 g).

2. The physical properties of these blended base diesel fuels were determined as follow :

1. Mid-boiling point by ASTM D86
2. API gravity by ASTM D1298
3. Cetane Index by ASTM D976

3.4.3 Determination of cetane number of dinitrate compounds and isooctyl nitrate (blended with 0.05 and 0.10 % by weight) in base diesel fuel as follow :

1. Triethylene glycol dinitrate
2. 1,5-pentane dinitrate
3. Isooctyl nitrate

1. The blended base diesel fuels was obtained by blending dinitrate compounds (0.05 and 0.10 g.) with base diesel fuel (1000 g).

2. The cetane number (ASTM D613) of the blended base diesel fuels were determined by ASTM D613 method.