

## Chemical incompatibility of nitrocompounds

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### Abstract

Chemical incompatibilities are found to be more qualitative than quantitative. However, many incidents resulting from inadvertent mixing have convinced many that quantitative determination of incompatibility is more helpful. In this study, calorimetric methods such as heat-flow calorimetry (DSC) and accelerating rate calorimetry (ARC) were utilized to quantify chemical incompatibility. Initial exothermic onset temperature, enthalpy change, heat evolution thermogram, and adiabatic runaway behavior data, were used to characterize the incompatibility effects. The test data revealed that aromatic nitrocompounds are generally more incompatible with base than acid, whereas inorganic ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) exhibits the opposite trend. For aromatic nitrocompounds and base mixtures, initial exothermic onset temperatures were reduced by about 40°C to 240°C. Severity of incompatibility could be accounted for by an unstable nitroquinonoid formation, inductive effect and mesomeric effect of substituted functional groups in the benzene ring. Incompatibilities of ammonium nitrate and various acids, e.g. hydrochloric acid, were also investigated. © 1997 Elsevier Science B.V.

*Keywords:* Chemical incompatibility; Nitrocompound

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### 1. Introduction

Chemical incompatibility can result in a potentially hazardous mixture which could possibly lead to accidents by thermal or mechanical impact during chemical processing, transportation or storage. In particular, mischarge or overcharge in reactor, contamination in storage tanks or bulk-shipping tanks could possibly lead to a violent reaction because of the enormous quantity involved. The diversities of incompatibility, as

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suggested by the U.S. Coast Guard (USCG), were reactivity with water (and other chemicals), self-reactive, and flammable hazards. Such considerations closely resemble those of the NFPA's classification. Both methods use non-quantified descriptions such as maybe, mild, moderate and vigorous. On the other hand, the Dow Chemical binary mixture rating system, based on adiabatic temperature rise and gas evolution rate determined by isoperibolic calorimeter [1] provides empirical criteria. Another chemical incompatibility was defined if, in the course of the experiment, more than a 25°C rise was observed. All these differences revealed the need for more detailed studies. In fact, effectively designing a quantitative compatibility test scheme is essential for hazard assessment and more thorough understanding of incompatibility [2].

Nitrocompounds and related nitrates are used as explosives, because of their characteristically exothermic decomposition with abrupt gas evolution and shock wave. Many accidents caused by nitrocompounds or nitrates have been reported [3,4]. A previous work studying the thermal instability of seven nitrobenzyl alcohol by thermoanalytical techniques (TG, DSC) and adiabatic calorimetry (ARC) was reported [5]. Another investigation concluded the typical onset temperature (360°C in DTA) and enthalpy change (380–410 KJ mol<sup>-1</sup>) of nitro group decomposition [6]. Grewer and Rogers [7] also indicated that exothermic decomposition of mixtures in nitrobenzene and nitrobenzoic acid with additives could result in a secondary reaction or a catalytic decomposition in a nitration process.

In this study, a more detailed study regarding mixing incompatibility of nitrocompounds was characterized by thermal analysis data, e.g. initial exothermic onset temperature, enthalpy change, heat evolution thermogram and adiabatic runaway behavior. In general, this study attempts to:

- identify the incompatibility trends of nitrocompounds with acids and alkalies;
- establish a quantitative approach for incompatibility hazard characterization;
- analyze the thermal instability and incompatibility of nitrocompounds and isomers by inductive or mesomeric effects of different substituted groups.

## 2. Experimental

### 2.1. Samples

Twenty-four nitrocompounds, various mineral acids, and alkalies were purchased from Merck. Appropriate quantities of samples were weighed into the testing cell and sealed, the residues were weighed at the end of the decomposition to confirm that no material loss had occurred.

### 2.2. Differential scanning calorimeter (DSC)

The dynamic temperature scanning (4°C min<sup>-1</sup>) was executed on a Mettler TA4000 system coupled with a DSC25 measuring cell [8]. The system was connected to an IBM compatible PC where the data were evaluated and stored. A disposable high pressure crucible (ME-26732) was used for acquiring thermogram. A standard aluminum crucible

(ME-27331) was then used for taking heat capacity ( $C_p$ ) measurements, which were used to calculate adiabatic temperature rise and thermal inertia ( $\phi$ ) in ARC tests. Sensitivity of exothermic onset temperature was chosen to be  $0.2 \text{ W g}^{-1}$  (equivalent to a signal-to-noise ratio around 5).

### 2.3. Accelerating rate calorimeter (ARC)

This study also utilized a microprocessor controlled accelerating rate calorimeter (ARC), manufactured by Columbia Scientific Industries of Austin, Texas. The detailed performance and theory of the ARC instrument can be found elsewhere [9]. The adiabatic temperature and pressure behavior of decomposition were investigated for different weight percent ratios. Sample bombs made of s.s. 304 and Hastelloy C were used.

## 3. Results and discussion

Thermal instability of nitrocompounds with acids, bases and other chemicals are presented and assessed by activating or deactivating various substituted functional groups. Comparing DSC curves and initial decomposition temperatures detected by ARC provides a basis for assessing the compatibility of nitrocompounds with the additives.

### 3.1. Instabilities of nitrocompounds

DSC thermograms provide thermal stability information such as initial decomposition temperature and decomposition enthalpy. Table 1 summarizes the DSC investigations of decomposition of nitrocompounds. In this study, data of nitrobenzene and 1-chloro-4-nitrobenzene correspond exactly to those in Grewer and Rogers [7]. Also, the nitro group's decomposition energy is consistent with Grewer's study [6]. However, Table 1 reveals that the onset temperature is markedly influenced by adjacent functional groups. On the basis of initial decomposition temperature, DSC data indicate that:

1. Thermal stability—1-chloro-4-nitrobenzene > nitrobenzene > nitromethane > nitrotoluene (2-nitrotoluene) > nitroaniline (2-nitroaniline) > nitrobenzoic acid (2-nitrobenzoic acid) > dinitrobenzene (2,4-dinitrobenzene) > nitrophenol (2-nitrophenol) > dinitrotoluene (2,4-dinitrotoluene) > 2,4-dinitrophenol > 2,4,6-trinitrophenol.
2. Effect of activating groups to nitrocompounds— $-\text{NO}_2$  (ortho,meta) >  $-\text{OH}$  >  $-\text{COOH}$  >  $-\text{NH}_2$  >  $-\text{CH}_3$  >  $-\text{Cl}$ .
3. Effect of mononitrocompounds activating position—ortho- > meta- > para-.
4. Activating influence of numbers of nitrogroup—trinitro- > dinitro- > mononitro-.

### 3.2. Mixing incompatibility of 2-nitrotoluene with other chemicals

Table 2 lists the onset temperatures of the decomposition in DSC for a mixture of 2-nitrotoluene with various substances. As revealed in the table, mixtures of 2-

Table 1  
Thermal stability of nitrocompounds

Compounds	$T_{\text{onset}}$ (°C)	$-\Delta H$ (J g <sup>-1</sup> )	$-\Delta H$ (kJ mol NO <sub>2</sub> <sup>-1</sup> )
Ammonium nitrate	250	1577	126
Nitrobenzene	380	2757	339
Nitromethane	320	5292	323
2-Nitrophenol	250	2481	345
3-Nitrophenol	310	2269	316
4-Nitrophenol	270	2155	300
2-Nitrotoluene	290	2404	329
3-Nitrotoluene	310	2070	284
4-Nitrotoluene	320	2322	318
2-Nitroaniline	280	2225	307
3-Nitroaniline	300	2269	314
4-Nitroaniline	310	2026	279
2-Nitrobenzoic acid	270	1894	297
3-Nitrobenzoic acid	300	1899	298
4-Nitrobenzoic acid	310	1934	304
1,2-Dinitrobenzene	280	3310	259
1,3-Dinitrobenzene	270	3488	293
1,4-Dinitrobenzene	350	3701	311
2,4-Dinitrotoluene	250	3574	298
3,4-Dinitrotoluene	280	3987	333
2,6-Dinitrotoluene	290	3451	288
1-Chloro-4-nitrobenzene	395	2283	360
2,4-Dinitrophenol	240	3598	331
2,4,6-Trinitrophenol	220	5130	391

$T_{\text{onset}}$ : onset temperature;  $\Delta H$ : enthalpy change.

nitrotoluene with acetic acid, acetic anhydride, aniline, ethanol and sodium chloride have a compatible stability which is approximately the same as a pure compound. However, the stabilities with mineral acid are lower. The decomposition begins at temperature about 50°C lower in hydrochloric or sulfuric acid than for 2-nitrotoluene alone. Note that a lower exothermic peak appears at 130–160°C in the nitric acid mixture. This

Table 2  
DSC experimental data of 2-nitrotoluene with some substances

Additives	Mass proportion (mg mg <sup>-1</sup> )	$T_{\text{onset}}$ (°C)	$-\Delta H$ (J g <sup>-1</sup> )
—	—	290	2404
6N HCl	(2.32/3.59)	240	2945
6N H <sub>2</sub> SO <sub>4</sub>	(2.36/4.36)	240	2587
6N HNO <sub>3</sub>	(2.78/2.7)	160/240 (2 peaks)	1614 + 2693
NaCl	(4.03/0.21)	290	2034
Acetic acid	(2.42/1.94)	280	2482
Acetic anhydride	(2.22/2.4)	290	2546
Ethanol	(2.06/2.02)	260	2277
Aniline	(2.29/2.84)	280	2365

effect was assessed to be a decomposition of reaction mixtures in the nitration of aromatic compounds or a secondary nitration step proposed by Grewer and Rogers [7]. Our further studies indicated that this exothermic peak cannot be found in a fully stirred C-80 mixing experiment, though being able to be observed only in an unstirred DSC crucible or an ARC test bomb. We, therefore, conclude that this lower exothermic peak is due to the delayed mixing (or nitration) caused by the super-heated water solution in DSC cell, not because of a secondary nitration reaction. For a well mixing condition such an exothermic peak is no longer observed.

According to the Coast Guard chart, nitrocompounds are compatible with mineral acid such as sulfuric acid, hydrochloric acid and nitric acid [10]. However, this study shows that if incompatibility is defined as a 50°C lowering of the onset temperature, 2-nitrotoluene is not compatible with these acids.

### 3.3. Incompatibility of nitrocompounds with HCl or NaOH

Table 3 displays the exothermic reaction or decomposition of nitrocompounds with 6N HCl. Most of the aromatic nitrocompounds decompose at about 240°C and are not markedly influenced by the relative quantity of acid and the adjacent functional group.

Table 3  
DSC experimental data of nitrocompounds with 6N HCl

Compounds	Mass proportion (mg mg <sup>-1</sup> )	$T_{\text{onset}}$ (°C)	$\Delta T_{\text{diff}}$ (°C)	$-\Delta H$ (J g <sup>-1</sup> )
Ammonium nitrate	3.41/2.41	40/170 (2 peaks)	-210	478 + 2463
Nitrobenzene	3.93/3.77	240	-140	2622
Nitromethane	3.86/2.02	120	-200	5386
2-Nitrophenol	3.48/2.45	220	-30	2694
3-Nitrophenol	2.12/7.37	220	-90	3085
4-Nitrophenol	2.04/6.49	210	-60	2618
2-Nitrotoluene	2.32/3.59	240	-50	2945
3-Nitrotoluene	1.88/5.59	230	-80	2579
4-Nitrotoluene	2.91/1.71	240	-80	2464
2-Nitroaniline	3.52/6.54	190	-90	2319
3-Nitroaniline	3.75/7.13	210	-90	2760
4-Nitroaniline	2.02/3.97	200	-110	2558
2-Nitrobenzoic acid	2.47/2.56	230	-40	1894
3-Nitrobenzoic acid	1.96/1.72	240	-60	2006
4-Nitrobenzoic acid	1.75/2.04	240	-70	2042
1,2-Dinitrobenzene	2.6/4.42	190	-90	3034
1,3-Dinitrobenzene	3.32/2.85	240	-30	4008
1,4-Dinitrobenzene	2.1/4.81	240	-110	4214
2,4-Dinitrotoluene	2.3/1.63	240	-10	3668
3,4-Dinitrotoluene	2.69/2.33	240	-40	3801
2,6-Dinitrotoluene	2.44/2.34	240	-50	3928
1-Chloro-4-nitrobenzene	3.24/2.32	280	-115	2190
2,4-Dinitrophenol	2.47/2.68	220	-20	4261
2,4,6-Trinitrophenol	3.56/2.9	200	-20	4712

$\Delta T_{\text{diff}}$ : temperature difference of  $T_{\text{onset}}$  between Table 3 and Table 1.

However, for ammonium nitrate and nitromethane, the incompatibility effects are quite significant.

Table 4 summarizes the influence of sodium hydroxide on the incompatibility of nitrocompounds. Onset temperature differences ranged from 40°C (2-nitrophenol) to 240°C (nitromethane). Further experiments indicated that the decomposition of nitrocompounds remain unaffected by the amount and concentration of sodium hydroxide. The onset decomposition temperature of 4-nitrotoluene and NaOH mixtures with weight ratios of 1:1.5 to 1:6 were measured to be 140°C. Also, potassium hydroxide and sodium hydroxide yielded almost the same results.

Effects of sodium hydroxide on the instability of a nitrocompound mixture were further investigated by ARC. Fig. 1 presents adiabatic temperature changes as a function of time of 2-nitrotoluene. Fig. 2 shows the adiabatic time to maximum rate (TMRad) vs temperature plot. The lower initial onset temperature and the shorter of the TMRad indicate the more incompatible nature of the mixtures.

### 3.4. Incompatibility of nitromethane and $NH_4NO_3$ with HCl and NaOH

Nitromethane has found extensive applications as a commercial solvent, a reactive intermediate and high energy fuel. The detonation potential of nitromethane under shock

Table 4  
DSC experimental data of nitrocompounds with 6N NaOH

Compounds	Mass proportion (mg mg <sup>-1</sup> )	$T_{onset}$ (°C)	$\Delta T_{diff}$ (°C)	$-\Delta H$ (J g <sup>-1</sup> )
Ammonium nitrate	4.64/2.26	320	+70	1848
Nitrobenzene	2.02/5.56	220	-160	2569
Nitromethane	2.96/7.64	80	-240	4896
2-Nitrophenol	3.19/2.06	210	-40	2632
3-Nitrophenol	2.05/7.29	150	-160	2720
4-Nitrophenol	1.99/6.3	230	-40	2314
2-Nitrotoluene	2.25/2.15	200	-90	2321
3-Nitrotoluene	1.84/6.64	220	-90	2797
4-Nitrotoluene	1.82/3.72	140	-170	2112
2-Nitroaniline	2.2/6.6	200	-80	2335
3-Nitroaniline	2.2/6.4	150	-150	2565
4-Nitroaniline	4.67/1.37	220	-90	2070
2-Nitrobenzoic acid	3.02/7.12	180	-90	1835
3-Nitrobenzoic acid	2.41/4.15	190	-110	2124
4-Nitrobenzoic acid	3.91/4.74	190	-120	1925
1,2-Dinitrobenzene	2.52/7.34	120	-160	3310
1,3-Dinitrobenzene	2.91/1.82	120	-150	3889
1,4-Dinitrobenzene	2.47/6.69	120	-230	3547
2,4-Dinitrotoluene	2.82/2.33	80	-170	3923
3,4-Dinitrotoluene	2.35/5.12	120	-160	4122
2,6-Dinitrotoluene	2.72/2.91	120	-170	4360
1-Chloro-4-nitrobenzene	3.06/2.33	200	-195	2443
2,4-Dinitrophenol	2.57/5.67	160	-80	4339
2,4,6-Trinitrophenol	2.16/7.52	80	-140	5463

$\Delta T_{diff}$ : temperature difference of  $T_{onset}$  between Table 4 and Table 1.

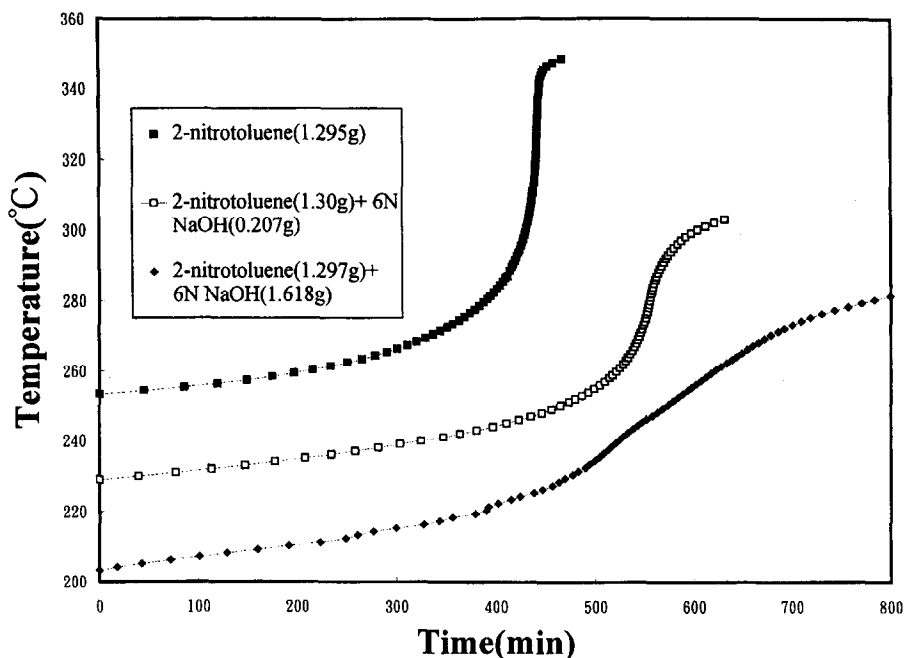


Fig. 1. Adiabatic runaway of 2-nitrotoluene with 6N NaOH in ARC.

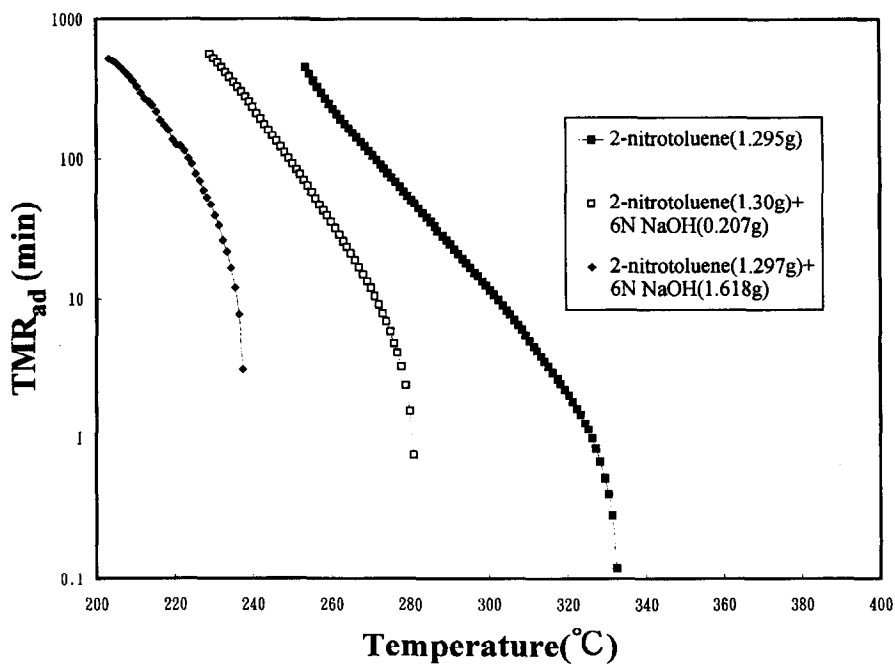


Fig. 2. TMRad of 2-nitrotoluene with 6N NaOH.

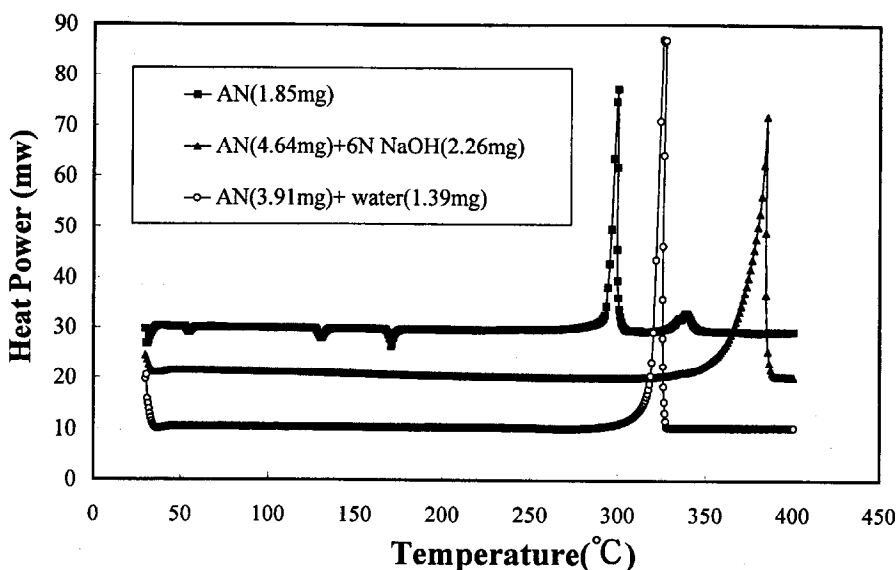


Fig. 3. DSC thermogram of ammonium nitrate (AN).

or strong heating has been known for decades. Table 3 indicated that, in the presence of 6 N HCl, there exists the explosion or detonation hazard resulting from incompatibility because of the onset temperature lowering of 200 °C than the pure state.

Ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) can be deflagrated in a confined space at elevated temperatures. Fire and explosion incidents have been subsequently caused by ammonium nitrate due to its self-reactive nature and incompatibility. Besides being used as a blasting agent and fertilizer, it is also utilized to produce nitrous oxide ( $\text{N}_2\text{O}$ ) in the gas industry by pyrolysis [11].

Delayed decomposition in a higher temperature region, as shown in Fig. 3, reveals that  $\text{NH}_4\text{NO}_3$  is compatible with pure water ( $\text{H}_2\text{O}$ ) and sodium hydroxide (NaOH). This inhibited decomposition by water because of the steady-state approximation of nitronium ion was verified directly from DSC thermogram [12]. Fig. 4 reveals the incompatible mixing of  $\text{NH}_4\text{NO}_3$  with nitric acid, sulfuric acid and hydrochloric acid. In particular, the mixture of  $\text{NH}_4\text{NO}_3$  with hydrochloric acid, catalyzed by chloride ion, evolves heat at a much lower temperature around 40 °C [13].

### 3.5. Decomposition intermediate of nitrocompounds and their mixtures with NaOH and HCl

Thermal stability of seven nitrocompounds determined by thermoanalytical techniques (TG, DSC) and by ARC indicated that ortho-isomers are less stable than meta- and para- ones. Nevertheless, the relative stabilities of meta and para isomers were not compared. Also, no apparent correlation could be established regarding the isomers stability as well as no clear discussions or explanation of incompatibility of nitrocompounds mixtures were conducted.



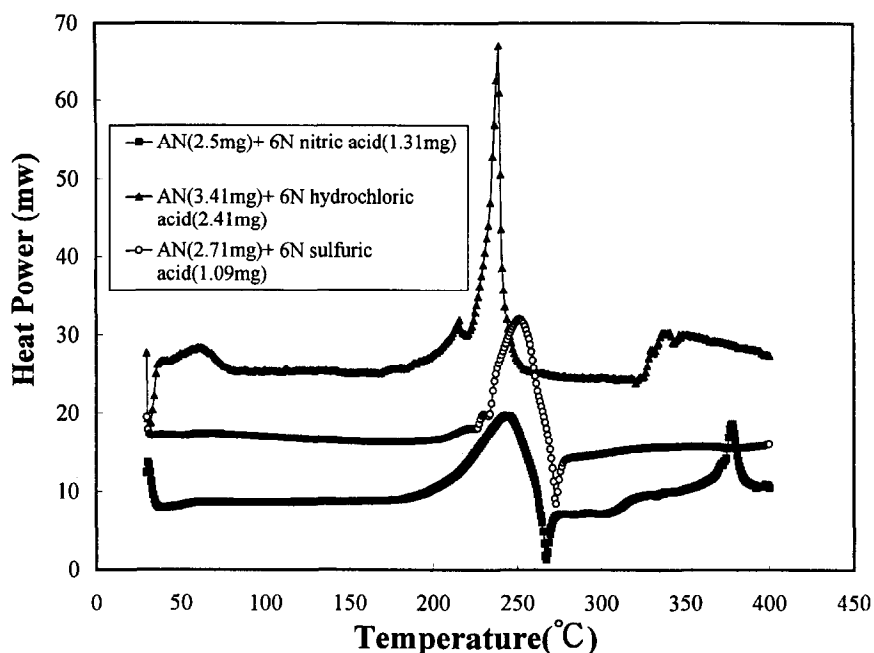
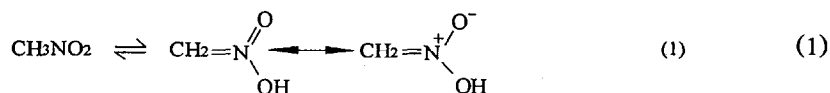


Fig. 4. DSC thermogram of ammonium nitrate (AN) with acids.

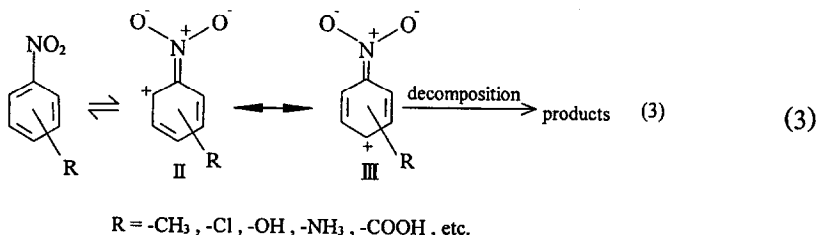
The kinetics of thermal decomposition of nitromethane was investigated by Makovsky and Lenji [14] with static or flow methods at various temperatures (310–480 °C) and low (4–400 mmHg) and high pressures (12–20 atm; 40 atm). They proposed that rupturing of the N–O bond is the initial step of the decomposition of nitromethane. From the experimental data in this study, nitromethane is not compatible with 6 N NaOH and 6 N HCl. Also, a tautomeric form (isonitro structure) of nitrocompound was proposed. Moreover, a pseudo-ionic structure accounted for the lower stability of nitromethane in 6 N HCl or 6 N NaOH as shown below:



Thermal instability of nitrocompounds can be attributed to the nitro group ( $-\text{NO}_2$ ). The N–O bond lengths and the C–N–O angles are 1.23 Å and 118 °C, respectively. This identity arises from the intrinsically mesomeric structure of the group, which is presented in the valence–bond resonance structure as:

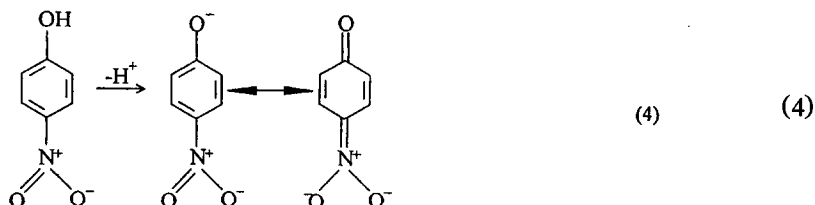


Stability of nitrobenzene can be assessed by the dissociation of N–O bonding, while instability of substituted nitrocompounds can be explained by the conjugation between the nitro group and aromatic ring. Decomposition reactivity developed in the intermediates can be related to the structures as:

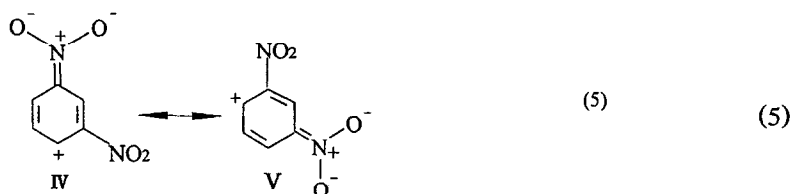


Formation of intermediate II or III and the dissociation of N–O bonding, which control the rate-determining-step of decomposition or instability, are significantly affected by the inductive and mesomeric effects of substituents. Instability of nitrotoluene and stability of 1-chloro-4-nitrobenzene, compared with nitrobenzene, are attributed to the inductive effect of -CH<sub>3</sub> and -Cl groups, respectively. Mesomeric effect of -OH, -NH<sub>2</sub>, and -COOH groups conducts the instability of nitrophenols, nitroanilines, and nitrobenzoic acids, respectively. The lower onset temperature of decomposition can be associated with the lower potential energy of intermediate according to Arrhenius equation. However, meta-substituted aromatics cannot yield quinonoid forms. Stability of mono-substituted nitrobenzene isomer can be verified in the order of para- > meta- > ortho-, thereby confirming the finding of others [5,15]. Ullrich and Grever [15] demonstrated that the stabilities of the nitrobenzene, diazonium chlorides, and the nitro group possess greater sensitivity than the chlorine substituent. Zeman [16] concluded that the primary thermolysis of 2,4-dinitrotoluene and 2,6-dinitrotoluene should be the hydrogen transfer from methyl group onto the oxygen of the ortho-nitrogroup. However, homolysis of C–NO<sub>2</sub> bond of 3,5-dinitrotoluene is supposed to occur [16]. The presence of a relatively unequal, potential energy of structure II and III is proposed owing to the unequal stability of ortho- and para-substituted nitrobenzene.

The nitrophenols are stronger acids than phenols. Due to the electron-attractive inductive effect of the nitro group, the 4-nitrophenol (and 2-nitrophenol) can be stabilized by the 4-nitrophenoxide (and 2-nitrophenoxide) ion resonance structure, which leads to a lower decomposition temperature than nitrobenzene.



Substitution of a second or third nitro group at the meta-position has the effect of lowering potential energy of the intermediate, thereby leading to the formation of:



The unusual behavior of 1,4-dinitrobenzene may be expressed by the structure VI which possesses a 70°C decomposition temperature higher than that of 1,3-dinitrobenzene with resonance form of IV and V.



These resonance structures account for the instability of 1,3-dinitroaromatic, 2,4-dinitroaromatic and 2,4,6-trinitroaromatic compounds.

#### 4. Conclusions

This study presents an analytical approach to thermal instability and incompatibility assessments of nitrocompounds. Application of calorimetry techniques is a promising alternative to generate data necessary for chemical incompatibility analysis. Thermal-induced instability of nitrocompounds is characterized by N–O bond dissociation of isonitro structure or acid-quinonoid influenced by substituted functional groups. Further study would be required to verify the decomposition mechanisms of nitrocompounds and their incompatible mixtures, e.g. intermediate identification, ESR (electron spin resonance), thermal-induced decomposition gas analysis, and UV spectrometry.

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