

LA-UR-19-21405

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Title: Development of a Solvent-Free Process to Prepare DNAN

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Intended for: WP-2749 Final Report

Issued: 2019-02-21

REPORT DOCUMENTATION PAGE

*Form Approved
OMB No. 0704-0188*

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1. REPORT DATE (DD-MM-YYYY) 02/21/2019	2. REPORT TYPE SERDP Final Report	3. DATES COVERED (From - To)
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4. TITLE AND SUBTITLE The Development of a Solvent-Free Process to Prepare DNAN	5a. CONTRACT NUMBER
	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S) Chavez, David E. Mash, Eugene	5d. PROJECT NUMBER WP-2749
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Los Alamos National Laboratory MS C920 Los Alamos, NM 87545	8. PERFORMING ORGANIZATION REPORT NUMBER LA-UR-19-21405
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9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Strategic Environmental Research and Development Program 4800 Mark Center Drive, Suite 17D03 Alexandria, VA 22350-3605	10. SPONSOR/MONITOR'S ACRONYM(S) SERDP
	11. SPONSOR/MONITOR'S REPORT NUMBER(S) WP-2749

12. DISTRIBUTION/AVAILABILITY STATEMENT
DISTRIBUTION STATEMENT A. Approved for public release: distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
The objective is the scale-up of an additive-free, solvent-free dinitration of anisole, from the previously demonstrated one gram scale to a 500 gram scale, for the production of DNAN using the equipment and facilities available at the Los Alamos National Laboratory for manipulation and synthesis of explosive materials. The overall process would involve the stepwise scale-up of the process from 1 gram to larger scales to demonstrate the applicability of this process towards advancement to larger scale process level production of DNAN. The data collected will allow for a case to be made on whether the process developed can be a suitable method for the development into a more extensive project. These data include reaction times, temperatures, conversion, yields and reproducibility. Overall the effort relates to the SERDP SON by developing a potential pathway to prepare DNAN using environmentally benign methods.

15. SUBJECT TERMS
Solvent-Free Process, DNAN

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES 11	19a. NAME OF RESPONSIBLE PERSON David Chavez
a. REPORT	b. ABSTRACT	c. THIS PAGE			19b. TELEPHONE NUMBER (Include area code) 505-665-2742
UNCLASS	UNCLASS	UNCLASS	UNCLASS		

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The Development of a Solvent-Free Process to Prepare DNAN

David E. Chavez, Eugene Mash

Abstract

Objectives: Additive-free, solvent-free chemical reactions are generally more environmentally benign than alternative processes that involve these reaction components. 2,4-Dinitroanisole (DNAN) is of interest to the military and to industry as a replacement for trinitrotoluene (TNT) in melt-cast explosives. As anisole is a widely used feedstock chemical, the development of an efficient, additive-free, solvent-free method for its dinitration could lead to adoption of a new “green” synthetic route for DNAN manufacture.

Technical Approach: Our goal is the scale-up (from the previously demonstrated one gram scale to a 500 gram scale) of the additive-free, solvent-free dinitration of anisole using the equipment and facilities available at the Los Alamos National Laboratory.

Results: Through a series of simple optimization process, we have been successful at scaling demonstrating the development of a solvent-free process for the synthesis of 2,4-dinitroanisole using a batch process. The overall method involves the use of a mixture of propionic anhydride and 98% nitric acid to form the reactive nitrating species, propionyl nitrate. This nitrating reagent is then added to a highly concentrated solution mixture of anisole and propionic anhydride slowly over time to produce the desired 2,4-dinitroanisole product. This batch process could be scaled-up to the 270g scale. Flow-reaction methods should allow for larger scale production of DNAN.

Benefits: This work has addressed the SERDP SEED Statement Of Need regarding environmentally benign methods for the synthesis of DNAN. Having achieved our goal, the next step will be to partner with a commercial manufacturer of explosive materials to scale the synthesis up to production levels.

Objective

Our objective is the scale-up of an additive-free, solvent-free dinitration of anisole, from the previously demonstrated one gram scale to a 500 gram scale, for the production of DNAN using the equipment and facilities available at the Los Alamos National Laboratory for manipulation and synthesis of explosive materials. The overall process would involve the stepwise scale-up of the process from 1 gram to larger scales to demonstrate the applicability of this process towards advancement to larger scale process level production of DNAN. The data collected will allow for a case to be made on whether the process developed can be a suitable method for the development into a more extensive project. These data include reaction times, temperatures, conversion, yields and reproducibility. Overall the effort relates to the SERDP SON by developing a potential pathway to prepare DNAN using environmentally benign methods.

Background

2,4-Dinitroanisole (DNAN) is of interest to the military and to industry as a replacement for trinitrotoluene (TNT) in melt-cast explosives.¹ DNAN has been prepared by dinitration of anisole,²⁻⁴ by further nitration of *p*-nitroanisole or *o*-nitroanisole,⁵⁻⁷ and by nucleophilic aromatic substitution of 1-chloro-2,4-dinitrobenzene using basic methanol.⁸ The latter process is presumably used in industry, since 1-chloro-2,4-dinitrobenzene is found as an impurity in manufactured DNAN.^{1,9} Major problems with large-scale industrial nitrations of chlorobenzene are (1) the use of forcing, potentially dangerous reaction conditions (required to produce 1-chloro-2,4-dinitrobenzene); and (2) disposal of strongly acidic reaction mixtures when recycling of the mixtures is no longer practical. Our work addresses these two issues.

Anisole is a widely used feedstock, presumably made industrially by reaction of phenol with methanol in the vapor phase.¹⁰ In contrast with chlorobenzene, which is less reactive than benzene toward nitration, anisole is much more reactive. Thus, the reaction conditions required to dinitrate anisole should be milder (and less hazardous) than those required for dinitration of chlorobenzene.

The environmental impact of disposal of large volumes of dilute spent sulfuric acid from industrial nitration reactions¹¹ calls for the development of an efficient and environmentally benign methods for nitration that do not involve sulfuric acid. We have demonstrated the viability of a new “green” synthetic route for DNAN manufacture from anisole that involves no catalysts, additives, or solvents. The development of an efficient and environmentally benign method for dinitration of anisole might lead to adoption of a new “green” synthetic route for DNAN manufacture.

We became interested in DNAN syntheses when we were asked to prepare stable isotope-labeled versions of DNAN for use in studies of the metabolism and disposition of this compound in the environment.¹² Given the commercial availability of [¹³C₆]-anisole and [¹⁵N]-nitric acid, synthetic routes to DNAN starting from anisole were most appealing. Dinitration procedures include treatment of anisole with nitronium tetrafluoroborate,² urea nitrate-sulfuric acid,³ urea nitrate-polyphosphoric acid,⁴ and an acidic montmorillonite clay impregnated with anhydrous cupric nitrate (claycop).¹³ The claycop procedure seemed most amenable to synthesis of both ¹³C-labeled and ¹⁵N-labeled DNAN (via the use of ¹⁵N-enriched claycop). We first performed the claycop dinitration of unlabeled anisole according to the published procedure,¹³ which involved addition of a solution of anisole (10 mmol) in CCl₄ (30 mL) to a mixture of claycop (4.8 g, Sigma Aldrich) and acetic anhydride (15 mL, 16 mmol), followed by addition of ~6 equivalents of fuming nitric acid. Acetic anhydride and nitric acid mixtures generate acetyl nitrate¹⁴ *in situ*, and many examples of aromatic nitration with acetyl nitrate have been reported.^{15,16} While this work was in progress, a synthesis of DNAN by treatment of anisole with propanoyl nitrate generated *in situ* over zeolite H β catalyst was reported.¹⁷ To determine whether claycop (or any other additive or catalyst) was required, a solution of anisole (10 mmol) in CCl₄ was added to an ice-cold mixture of acetic anhydride (53 mmol) and 100% nitric acid (47 mmol). An aqueous workup and flash silica gel column chromatography afforded DNAN as a light yellow solid in 85% yield. Subsequent uses of this additive-free nitration method for syntheses of the required

^{13}C -labeled and ^{15}N -labeled versions of DNAN were successful.¹² Then, in a further application of Occam's Razor, we wondered if a solvent such as CCl_4 was necessary for this reaction. Solvent-free nitration would be a more environmentally benign process. Results from our initial survey appear in Table 1 and are discussed below.

Table 1. Nitrations of Arenes **1a-f**

		Yields (%) ^a			
Arene	R	2a-f	3a-f	4a-f	5a-f
1a	OCH_3	(82 ^b)			
1b	OCH_2CH_3	81		9	
1c	CH_3	5	5	44	36
1d	CH_2CH_3	9	2	45	40
1e	Cl	3		59	14
1f	Br			68	10

^aYields based on isolated products. Ratios of products in mixtures determined by ^1H NMR analysis.

^bYield after crystallization.

The electron-rich, highly activated arenes anisole and phenetole, the weakly activated arenes toluene and ethylbenzene, and the weakly deactivated arenes chlorobenzene and bromobenzene were subjected to nitration on a 10 mmol scale with ~ 5 equivalents of acetyl nitrate, generated *in situ* and in the absence of additives and solvent. Reaction of anisole (**1a**) with acetyl nitrate gave a mixture of 2,4-dinitroanisole and 4-nitroanisole as determined by ^1H NMR analysis. Crystallization from 20% EtOAc/hexanes afforded 1.62 g (8.2 mmol, 82% yield) of DNAN as colorless needles. Reaction of phenetole (**1b**) under similar conditions gave comparable results.

With the less reactive arenes toluene and ethylbenzene, more complex mixtures of mono- and dinitrated products were obtained. Treatment of toluene (**1c**) with ~ 5 equivalents of acetyl nitrate under the same conditions used for anisole and phenetole, followed by an extractive aqueous workup, gave an oil that was subjected to gravity column chromatography on silica gel. Elution with 5% ethyl acetate/hexanes gave an inseparable 1.2:1 mixture of 4-nitrotoluene and 2-nitrotoluene as determined by ^1H NMR analysis, in 80% combined yield. Further elution of the column with 7% ethyl acetate/hexanes and with 10% ethyl acetate/hexanes gave 2,6-dinitrotoluene and 2,4-dinitrotoluene, each in 5% yield. Reaction of ethylbenzene (**1d**) gave

similar results. As this was a preliminary survey to gauge the feasibility of solvent-free nitration, no attempts were made to optimize the reaction conditions for production of dinitrated products.

Halobenzenes gave predominantly mononitrated products. Treatment of chlorobenzene (**1e**) with ~5 equivalents of acetyl nitrate under the same conditions used for anisole and phenetole, followed by an extractive aqueous workup, gave an oil that was a 21:5:1 mixture of 1-chloro-4-nitrobenzene, 1-chloro-2-nitrobenzene, and 1-chloro-2,4-dinitrobenzene, respectively, in 76% combined yield as an oil. A similar treatment of bromobenzene (**1f**) gave a 7:1 mixture of 1-bromo-4-nitrobenzene and 1-bromo-2-nitrobenzene in 78% combined yield as an oil.

Materials and Methods

Nitration of Anisole using Propionic Anhydride. (Ratio of nitric acid to anisole 2.4:1). 98% Nitric acid (3.0 g, 47 mmol, 2.0 mL) was added dropwise manually using a pipette with stirring to propionic anhydride (6.9g, 54 mmol, 7.0 mL) in 50 mL round bottomed flask an ice bath. After 10 min, a solution of anisole (2.16 g, 20 mmol) in propionic anhydride (2.0 mL) was added dropwise manually using a pipette and the reaction mixture allowed to attain room temperature. After stirring overnight, the reaction mixture was poured into water (50 mL) in a 200 ml beaker and stirred 1 hour. The solid was collected by filtration using Whatman 40 filter paper, using a porcelain Buchner funnel and air dried to provide 3.65 g of a 12:1 mixture of 2,4-dinitroanisole and 4-nitroanisole as determined by ¹H NMR analysis. This weight and ratio correspond with an 87% yield of 2,4-dinitroanisole.

As the reaction was scaled-up, the reaction vessel was changed to a jacketed flask (100 mL – 5 L), and a -20-120 °C VWR recirculating bath, which was used to cool the reaction mixture to the desired temperature. Chemglass addition funnels (50 mL -500 mL) were used to add 98% nitric acid and the propionic anhydride/anisole mixture to the reaction vessel over the desired amount of time.

Results and Discussion

The project's proof of concept was met to a large degree using the methods developed, the only exception being that the original intent of the project was to synthesize 2,4-dinitroanisole (DNAN) on a 500 g scale, whereas it was scaled up to a 250 g scale. The overall process involved a stepwise scale-up method involving an initial synthesis phase where DNAN was initially prepared at the 2 gram scale followed by synthesis of DNAN at the 10 g, 30 g, 90 g, and 270 g, scale. In order to abide the safety protocols developed for the scale-up of explosive materials, the DNAN prepared at the 2 g and 10 g scales were subjected to small scale sensitivity testing, which includes differential scanning calorimetry, drop-weight impact, friction, electrostatic discharge and vacuum thermal stability testing.

During the initial synthesis phase, the reaction was performed as described in the Materials and Methods section, scaled down such that 2 g of total product was produced. The chemistry proceeded smoothly as previously described with the reaction times and products yields identical to those reported in the Materials and Methods section. The prepared DNAN was characterized by ^1H NMR to determine the isomer ratio, which was the same as that previously reported (12:1 DNAN: 4-nitro-anisole). The small scale sensitivity data is included in the appendix of this report.

The sensitivity data was required prior to scaling up to the 10 g level. With satisfactory sensitivity data in hand, we proceeded to perform the nitration on the 10 g scale. At the 10 g scale, the initial formation of the propionyl nitrate (from 98% nitric acid and propionic anhydride) could be performed in about 15 minutes at 8-10 °C. The subsequent addition of anisole in propionic anhydride is quite exothermic. On the 10 g scale, the dropwise addition of the anisole/propionic anhydride solution required 2.5 hours in order to maintain the overall reaction temperature at or below 10 °C. Upon complete addition, the reaction was allowed to stir overnight and then poured into water. The total yield was 18 g (88%), very similar to the results obtained on the 2 g scale. Additionally, the isomer ratio was identical to the 2 g scale (12:1 DNAN: 4-nitro-anisole). Once again, small scale sensitivity testing was required prior to scaling up to the next level (30 g). The sensitivity data collected were nearly identical to the original small scale sensitivity data collected at the 2 g scale. The data is included in the appendix of this report.

Upon scaling up to the 30 g scale, the addition rates for both the formation of the propionyl nitrate and the subsequent addition of anisole/propionic anhydride began to increase significantly, but particularly for the latter. In order to keep the reaction temperature between 8-10 °C for the propionyl nitrate, the addition rate was extended to 1 hour. Subsequent addition of the anisole/propionic anhydride solution required a 6 hour addition time. Stirring the reaction overnight followed by quenching of the reaction by pouring into water and stirring for an hour provided DNAN in essentially identical yield (54.1 g) and isomer ratio (12:1 DNAN: 4-nitro-anisole) as the previous trials.

Due to the ineffectiveness of the chiller at the 30 g scale, we chose to move to a higher capacity recirculating bath to increase the cooling efficiency for the reaction. Overall, the reaction at the 90 g scale proceeded similarly with respect to addition times. Here again, the yields (162 g) and isomer ratios (12:1 DNAN: 4-nitro-anisole) were within the same range as the previous experiments.

The 270 g scale-up was somewhat more challenging from a heat dissipation perspective. The formation of the propionyl nitrate occurred at 8-10 °C, requiring an addition rate such that the reaction was complete in 3 hours. We extended to addition time of the anisole/propionic anhydride solution to 18 hours, or overnight in order to effectively remove the heat of this exothermic reaction effectively, while maintaining the temperature at 10 °C or lower. The reaction was then allowed to stir for an additional 24 hours. The reaction quenching and isolation procedure was similar as that previously described. Once again, the reaction provided a similar yield (487 g) to our previous trials and the isomer ratio was identical to the other smaller scale trials (12:1 DNAN: 4-nitro-anisole).

Given the length of time to complete the reaction for the 270 g scale, we did not pursue the 500 g scale up of the reaction.

Conclusions and Implications for Further Research

The project set out to develop a method to reduce the amount of byproducts and waste from the synthesis of DNAN by investigating solvent free nitration protocols. We have been successful in dramatically reducing the amount of solvent and nitrating species from literature reports to create an effective process for producing DNAN. The ideal ratio of nitrating reagent (propionyl nitrate) to DNAN is 2:1, and our process has been successful at achieving a successful nitration method using a ration of 2.4:1. The main challenge with this methodology is the exothermic nature of the nitration process, which becomes a significant challenge under near solvent-free conditions. We have demonstrated that a batch process for the preparation of DNAN can be successful. In order to make significant progress on a larger scale, transition to a flow reaction process would be ideal. This methodology is perfect for addressing the heat removal challenges presented by batch process. Additionally, we have already demonstrated that the chemistry is successful. The next step would be to partner with an industry partner with expertise on flow processes to transition the technology developed in this project to the next level. Aromatic nitration reactions are among the most common of industrial chemical reactions. For example, over 3B pounds of nitrobenzene are produced annually in the US. As described in the EPA's IMPQCE Series document, Toxic Pollutant Identification: Nitrobenzene/Aniline Manufacturing, most industrial nitration reactions utilize mixtures of nitric and sulfuric acids in continuous flow processes. Considerable time, energy, and expense are involved in recycling both the nitric and sulfuric acids. When acid recycling is no longer possible, wastes are disposed through wastewater treatment plans, which again require considerable time, energy, and expense. The catalyst- and solvent-free process under development has the potential to greatly reduce (in some processes by >90%) the amount of nitric acid used and to eliminate the use of sulfuric acid altogether.

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Appendices

1. Small Scale Sensitivity Data Phase I
2. Small Scale Sensitivity Data Phase II

Small Scale Sensitivity Data Phase I

Test	DNAN (2,4-dinitroanisole)	Reference
Impact (Type 12) H₅₀ (cm)^a	>320	PETN (0601-02 L-298) 10.0
Friction Load₅₀ (N)^b	>360	PETN (0601-02 L-298) 78.9
Spark Screen** or Til* (J)^c	*0.125	PETN (0601-02 L-298) *0.025
DSC (10°C/min ramp in N₂ atmosphere)		
Onset of Decomposition (°C)	None	PETN (94-01B) 165
Peak Exotherm (°C)	None	PETN (94-01B) 205
Vacuum Stability (ml/g) (Gas evolved after 48hr @ 100°C)	0.21	N/A
a. Drop height resulting in a “go” in 50% of samples obtained through a Neyer D-optimal testing method. b. Force resulting in a “go” in 50% of samples obtained through a Neyer D-optimal testing method. c. TIL test: Highest level at which 20 consecutive No-Go results are observed. Screen test: 13 No-Go results at 0.25 J.		

Small Scale Sensitivity Data Phase II

Test	2,4-dinitroanisole (DNAN)	Reference
Impact (Type 12) H₅₀ (cm)^a	>320	PETN (0601-02 L-298) 17.2
Friction Load₅₀ (N)^b	>360	PETN (0601-02 L-298) 64.7
Spark Screen** or Til* (J)^c	*0.0625	PETN (0601-02 L-298) *0.0625
DSC (10°C/min ramp in N₂ atmosphere)		
Onset of Decomposition (°C)	None	PETN (94-01B) 167
Peak Exotherm (°C)	None	PETN (94-01B) 206
Vacuum Stability (ml/g) (Gas evolved after 48hr @ 100°C)	0.17	N/A
a. Drop height resulting in a “go” in 50% of samples obtained through a Neyer D-optimal testing method. b. Force resulting in a “go” in 50% of samples obtained through a Neyer D-optimal testing method. c. TIL test: Highest level at which 20 consecutive No-Go results are observed. Screen test: 13 No-Go results at 0.25 J.		