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Development and Validation of HPLC Method for DAAF and its Applications in Quality Control and Environmental Monitoring

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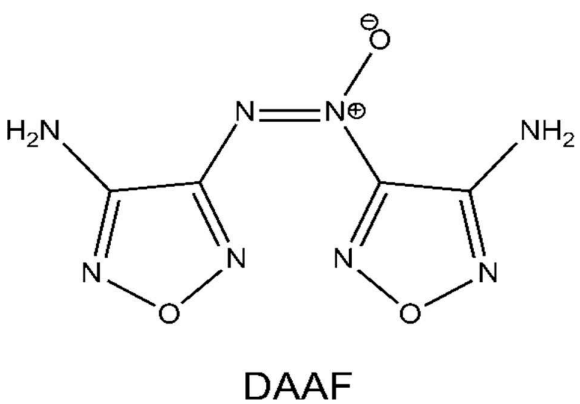
Abstract: 3,3-Diamino-4,4-azoxyfurazan (DAAF) as a promising energetic material, has many potential applications in both civil and military areas. High-performance liquid chromatography (HPLC) is an important analytical method for quality control and environmental monitoring of DAAF. In this study, chromatographic conditions of DAAF were optimized to develop an analytical method based on HPLC-DAD. DAAF and its byproducts were separated within 13 min with DAAF's retention time of 8.690 min and the resolution of all peaks were above 1.5 with high number of theoretical plates ($N > 54259$) under optimum condition of HPLC. Meanwhile, the method was validated and showed good performance for quantitative analysis of DAAF with LOD of 0.19 ng and linearity range from 0.84 to 211.16 $\mu g\,mL^{-1}$. Furthermore, the method was applied to the analysis of the purity of DAAF for quality control of different preparation stages and trace DAAF in wastewater for environmental monitoring around explosive manufacturing.

Keywords: DAAF · HPLC · Analytical method · Quality control · Environmental monitoring

1 Introduction

Insensitive energetic materials based on furazan have received much attention due to their favorable properties including high energy density, good safety, and high nitrogen content during the last decade [1–3]. Recently, 3,3-diamino-4,4-azoxyfurazan (DAAF) has been regarded as a promising energetic material [4–6] shown in Figure 1, and widely studied to investigate its various properties for both civil and military applications. DAAF has good thermal stability, high positive enthalpy, and high density of active oxygen with a small critical diameter (< 2 mm), high detonation velocity of 7.98 km s^{-1} and detonation pressure of 30.6 GPa at a density of 1.69 $g\,cm^{-3}$ [7]. Meanwhile, it is much simpler, greener, and more eco-friendly for the synthesis of DAAF [8]. Further, DAAF is expected to be used in place of TATB in insensitive booster explosives and RDX in melt cast explosives [9]. Besides, DAAF displays many potential applications in rocket propellants, fuels, and detonators [10, 11].



DAAF

Figure 1. Molecular structure of DAAF.

Analytical method of identification and quantification is one of very important topic of energetic materials during their researching and developing process [12, 13]. The determination of energetic materials has many benefits in many areas including quality control for the synthesis, environmental protection around the factory, and the prevention of terrorist attacks in public security [14–16]. Although DAAF is being synthesized and widely investigated about its potential applications [17, 18], there is a few reports about the quantitative analysis of DAAF. Sivabalan studied cyclic voltammetric response of DAAF by using glassy carbon electrode [19]. However, the sensitivity of above-mentioned electro-analysis is at ppm level for DAAF with linear calibration plots between 20 and 90 ppm.

There is a continuing need for developing new method to improve the sensitivity of detecting DAAF. High-performance liquid chromatography (HPLC) has much more sensitivity and reliability than electro-analysis, so HPLC is a very

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important tool for the analysis of energetic materials [20–22]. However, there is only one literature about the HPLC method of DAAF at present. The HPLC method reported by Qiu [23] was only used to investigate the solubility of DAAF with much higher purity (>0.99). Chromatographic conditions were not optimized and validated, and analytical parameters including precision, sensitivity, and accuracy were also not reported in that method so that it would be not suitable for the analysis of complicated DAAF samples in various applications such as quality control and environmental monitoring.

In this study, we studied chromatographic conditions in detail including mobile phase, flow rate, column temperature, and separating time, to develop a reliable method based on HPLC for DAAF. Meanwhile, the method was validated by investigating the sensitivity, linearity, precision, accuracy, etc., and it displayed much better performance for quantitative analysis of DAAF. Furthermore, the method was applied successfully to the analysis of the purity of DAAF during the preparation, and trace DAAF in explosive wastewater.

2 Experimental Section

2.1 Instrumentation

All chromatographic experiments were performed on a 1260 infinity HPLC system (Agilent, Waldbronn, Germany) equipped with a quaternary high-pressure pump, a micro vacuum degasser, an auto-sampler, a column compartment and a diode array detector (DAD). UV-Vis spectra were recorded on a Cary-100 spectrophotometer (Varian, Palo Alto, USA). The pH of solution used in HPLC experiments was measured on a DELTA 320 pH meter (Mettler Toledo, Greifensee, Switzerland). Ultrapure water was purified by a Millipore-Q system (Millipore, Bedford, MA, USA) with the resistivity of 18.2 MΩ. Samples were weighed by an XPE26 electronic scale (Mettler Toledo, Greifensee, Switzerland) with a precision of 0.001 mg.

2.2 Reagents and Chemicals

DAAF was synthesized in our laboratory according to the reported literature [24], and the reference material of DAAF was obtained by the recrystallization with the purity of 99.83 %. HPLC grade methanol, acetonitrile, and 2-propanol were purchased from Merck Chemicals (Darmstadt, Germany). Disposable syringes were purchased from Fisher Scientific (Waltham, USA). The filter of 0.46 μm was purchased from AS One (Osaka, Japan). All of solutions were prepared with HPLC grade solvents or twice-distilled water unless otherwise stated.

2.3 HPLC Method

Chromatographic analysis was operated on an Agilent 1260 infinity HPLC. A Zorbax Eclipse Plus-C18 column (4.6 mm I. D.×150 mm, 5 μm) from Agilent Technologies was used for separation of DAAF and its byproducts. Chromatographic conditions were optimized by investigating several separating factors including mobile phase, separating time, and so on. Finally, the optimum method was obtained for quantitative analysis of DAAF, which was as follows: The mobile phase consisted of acetonitrile and water; Gradient elution mode was used with the content of acetonitrile increasing linearly from 15 % to 85 % between 0 to 16 min, and then keeping the value of 85 % until 20 min; The re-equilibrating time of the column for next injection was 4 min; The elution was at a flow rate of 1.0 ml min^{-1}; The temperature of column oven was 30 °C; The injection volume was 5.0 μL; The signal of DAD was detected at 225 nm. The HPLC analysis, data acquisition, and processing were controlled by Agilent ChemStation software.

2.4 Sample Preparation

The solution of DAAF was prepared with the concentration of 0.2 mg mL^{-1} in CH_3CN and then used for the optimization of chromatographic condition. All the solution containing DAAF was filtered through a 0.46 μm membrane before HPLC analysis.

The external standard method was used for quantitative determination of DAAF. A standard solution of DAAF was prepared by dissolving the reference material of DAAF (10.56 mg) in CH_3CN (50 mL). Working standards at different concentration of 0.85-8.44-21.12-42.23-63.35-84.46-126.70-168.92-211.16 μg mL^{-1} were prepared by appropriate dilution in mobile phase, just before HPLC analysis.

2.5 Method Validation

The performance of HPLC method was estimated by studying main validation parameters such as selectivity, linearity, limit of detection and quantitation, accuracy, precision, and robustness.

The blank samples without DAAF were injected into HPLC to test the matrix effect, and then the selectivity of the method was evaluated by analyzing the interferences of blank samples for the separation of DAAF.

The calibration curve was constructed by plotting peak area (y) of DAAF against its concentration (x) using several concentration levels, and then followed linear regression analysis to evaluate the linearity.

To investigate the sensitivity, the limit of detection (LOD) was estimated by a signal-to-noise ratio of 3. The limit of quantification (LOQ) was determined by calculating a signal-to-noise ratio of 10.

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The accuracy was determined by the percent recovery method. Four different concentrations of standard DAAF (12.67, 25.34, 50.68, and 101.36 $\mu g\,mL^{-1}$) were added into the samples, respectively. Then, the actual concentration was determined to calculate the recovery of HPLC method (n = 6).

To evaluate the precision, peak areas of DAAF were recorded in the same day with six consecutive injections (n = 6) for intra-day precision and over three different days with eighteen injections (n = 18, six injections for each day) for inter-day precision.

The stability of HPLC method was determined by analyzing peak area of DAAF by each interval of 6 h during continuous running of 24 h.

2.6 Real Sample Analysis

HPLC method was used to investigate the purity of DAAF for quality control of different preparation process including synthesis, refinement, and nano-crystallization. Refined DAAF was prepared by solvent/non-solvent method with DMF as a solvent and water as a non-solvent. Nano-DAAF was prepared by rapid and low-temperature crystallization process [5].

Further, the method was used to measure the concentration of remnant DAAF in explosive wastewater. DAAF (50 mg) was washed in the water (5 mL) by different methods such as oscillation, vortex, and ultrasound. Then, these samples were centrifuged and the wastewater was injected into HPLC for the analysis of trace DAAF.

3 Results and Discussion

3.1 Optimization of Chromatographic Conditions

3.1.1 The Type of Organic Solvents

Gradient elution mode of HPLC was used for the separation of DAAF because isocratic elution mode was not suitable for separating DAAF containing several byproducts of different polarities from the synthesis. Under gradient elution, the effect of different types of organic solvents such as acetonitrile (CH_3CN) and methanol (CH_3OH) on the separation of DAAF was investigated by combining with water to form the mobile phase in HPLC. As shown in Figure 2, the baseline drift (about 16 mAU) of HPLC for CH_3OH was more serious than that for CH_3CN because there was an overlap between the absorption of CH_3OH (180–205 nm) and that of DAAF (190–500 nm). The baseline of HPLC for CH_3CN was much smoother than that for CH_3OH and it was more beneficial for the quantitative analysis of DAAF, so CH_3CN was selected as an organic solvent in the mobile phase.

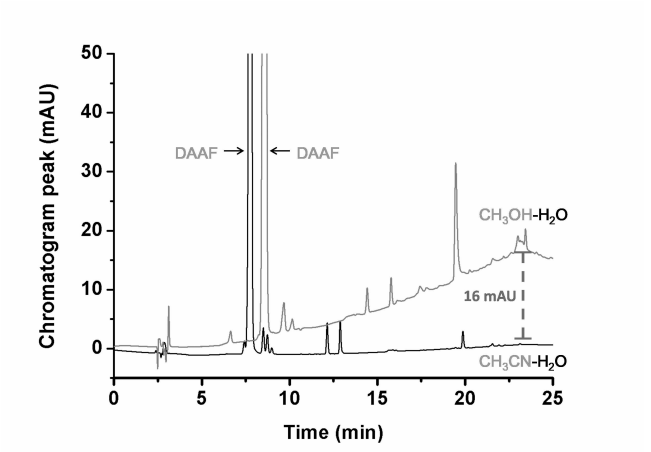

Figure 2. The baseline drift of HPLC in different mobile phase containing CH_3OH (upper) and CH_3CN (down).

3.1.2 Detection Wavelength

The wavelength of HPLC detector is an important parameter to influence the sensitivity of detecting DAAF. Although the wavelength of 236 nm was used to analyze DAAF in the reported literature [23], maximum absorption of DAAF in pure CH_3CN was recorded at 230 nm by UV-Vis absorption spectrophotometer. In this study, *in-situ* spectrum of DAAF in the mobile phase was obtained by a diode array detector (DAD) of HPLC and maximum absorption was found at 225 nm in Figure 3. Further, we investigated the effect of different detection wavelengths on the signal of DAAF, and peak area/height was measured at different wavelengths including 220, 225, 230, 236, 240 nm in Table 1. Peak area and height of DAAF at 225 nm were 2337.91 and 462.49, respectively, both of which were highest among all tested wavelengths. The result was attributed to the maximum absorption of DAAF in the mobile phase at 225 nm. Hence,

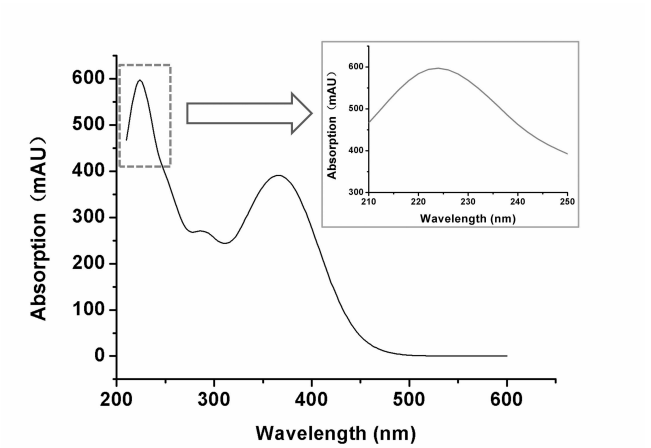

Figure 3. *In-situ* spectrum of DAAF from a diode array detector of HPLC.

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Table 1. Peak area and height of DAAF under different detection wavelengths.

Wavelength	220 nm	225 nm	230 nm	236 nm	240 nm
Peak area	2288.44	2337.91	2236.25	1975.12	1811.74
Peak height	452.74	462.49	442.47	390.81	358.47

225 nm was proved to be an optimum wavelength for the analysis of DAAF.

3.1.3 Flow Rate

The flow rate of HPLC often has an important effect on the separation of targeted samples, so we investigated the separating behavior of DAAF under different flow rate from 0.6 to 1.5 ml min^{-1}. As shown in Figure 4A, peak 1 was not separated from DAAF, and the resolution between peak 2 and 3 was only 0.8 under the flow rate of 0.6 ml min^{-1}, which was lower than required resolution of 1.5. The separation of peak 1 from DAAF was more and more obvious, and the resolution was increased to 2.35 under flow rate of 1.0 ml min^{-1}. The separation of peak 3 and 4 became bad with the resolution of 1.08 when the flow rate was enlarged continually to 1.2 ml min^{-1}. Peak 4 disappeared and was included in peak 3 when the flow rate was 1.5 ml min^{-1}. Therefore, 1.0 ml min^{-1} was used as the optimization of flow rate for DAAF.

3.1.4 Injection Volume

The injection volume of DAAF was optimized to improve the separation between DAAF and its byproducts. There was no separation between peak 1 and DAAF shown in Figure 4B when the injection volume of DAAF with the concentration of 0.2 mg ml^{-1} was enhanced from 10 to 40 μL. Meanwhile, all peaks became much wider from 0.08 to 0.29 and the symmetry went worse from 0.84 to 2.96 as the injecting volume increased from 5 to 40 μL. Moreover, injection volume (less than 5 μL) was too smaller to maintain

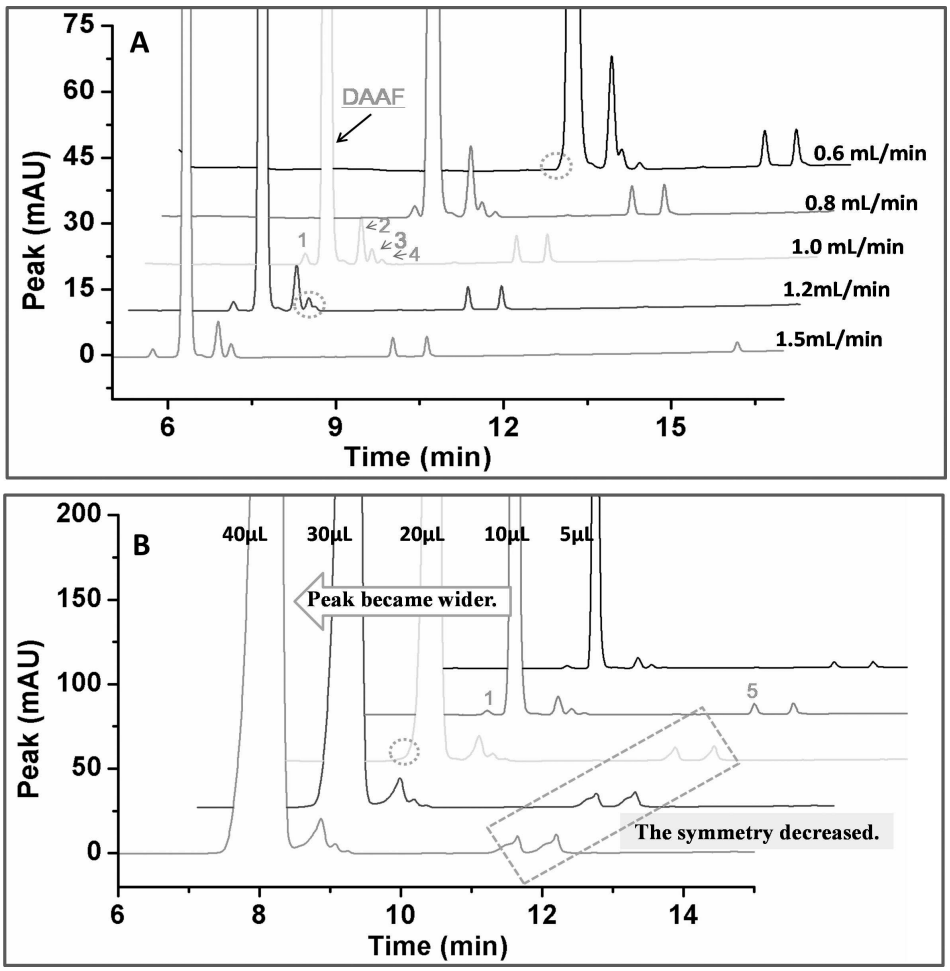

Figure 4. Chromatographic analysis of DAAF under different flow rate (A) and different injection volume (B).

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high peak area of DAAF, which was not good for the quantitative analysis. Hence, 5 μL was chosen as the optimal injection volume for DAAF.

3.1.5 Column Temperature

We investigated the effect of different column temperatures on separation efficiency and retention time of DAAF in Table 2. The symmetry of peak 1 became better from 0.84 to 0.95, when the column temperature was increased from 25 to 30 °C. It displayed much worse resolution between peak 3 and 4 when the column temperature was higher than 30 °C. The resolution of peak 3 and 4 was 1.07 at 35 °C and it went down to <1.0 under much higher temperature of 40 or 45 °C. Therefore, optimum temperature of HPLC column was set at 30 °C for the analysis of DAAF.

Table 2. Symmetry and resolution of DAAF under different temperatures.

Temperature (°C)	25	30	35	40	45
The symmetry	0.84	0.95	0.95	1.08	1.04
The resolution	1.39	1.24	1.07	0.86	0.75

3.1.6 The Content of CH_3CN

The content of CH_3CN in mobile phase is also an important factor for the separation and detection of DAAF, so we evaluated the effect of the content of CH_3CN at the starting/ending stage of gradient elution mode on the determination of DAAF. Firstly, we investigated different content of CH_3CN at the starting stage including 10%, 15%, 20%, 25%, and 30% with the same content of CH_3CN (90%) at the ending stage in Figure 5A. Peak 4 was not separated from peak 3 when the content of CH_3CN was only 10%. The separation between peak 3 and 4 became much better as

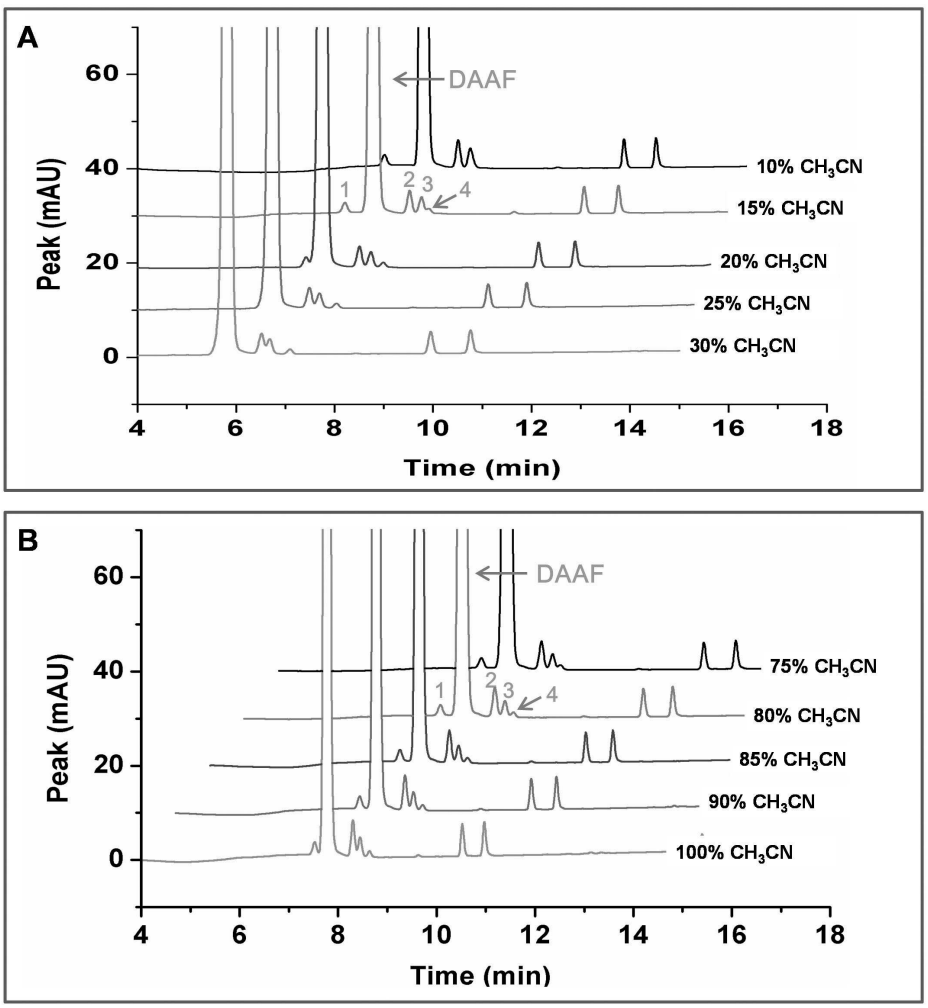

Figure 5. Chromatographic analysis of DAAF under different content of CH_3CN at the starting stage (A) and the ending stage (B).

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the content of CH_3CN was increased from 10% to 15%. The resolution of DAAF and peak 1 was 3.07 for the CH_3CN content of 15% and it got much worse with the resolution of 1.24 as the increase of the content of CH_3CN to 20%. Peak 1 was hidden in DAAF when the content of CH_3CN was increased to 25% and even 30%. Hence, 15% was used as the optimal starting content of CH_3CN for DAAF.

Secondly, different content of CH_3CN at the ending stage including 75%, 80%, 85%, 90%, and 100% was studied on the basis of 15% CH_3CN content at the starting stage. As shown in Figure 5B, the resolution of peak 3 and 4 was bad when the content of CH_3CN was 75%. The resolution of peak 3 and 4 became better from 0.98 to 1.16 with the increase of CH_3CN content from 80% to 85%. Peak 1 was not effectively separated from DAAF as the CH_3CN content increased continually to 90% and even 100%. Therefore, the optimum content of CH_3CN was 85% at the ending stage.

3.1.7 Separating Time of HPLC

It is necessary to optimize the separating time of HPLC because it would waste too much time under longer separating time and samples would be not separated effectively under too shorter time. Firstly, the running time of gradient profile was investigated by changing the time from 10 to 20 min in Figure 6A. Peak 1 was not seen in the chromatogram of DAAF when the running time was short (10 min). Peak 1 appeared gradually from DAAF as the time was prolonged to 16 min. The resolution and symmetry of peak 1 and DAAF was 2.56 and 1.00, respectively, under the running time of 16 min. However, the resolution and symmetry of peak 3 and 4 became much worse when the time increased from 16 to 18 min. The symmetry of peak 4 at the running time of 18 min was 0.51, which was much worse than that at the running time of 16 min (0.98). It was worst for the running time of 20 min. So, the running time of gradient profile was optimized to be 16 min.

The equilibration time of column before next analysis is very important for the reproduction and reliability of the determination of DAAF. We investigated the equilibration time by measuring the area and retention time of DAAF after different equilibration time from 1 min to 6 min in Figure 6B. The retention time of DAAF was shortened from 8.424 to 8.407 min with the increase of equilibration time from 1 to 4 min. The retention time kept the constant value when the equilibration time increased continually to 6 min. Meanwhile, peak area of DAAF became stable and the reproduction was good when the equilibration time was above 4 min. Hence, the optimal equilibration time was set to 4 min

3.1.8 Optimal Condition of HPLC

The optimum condition for the analysis of DAAF was obtained after investigating several parameters including organic solvents, flow rate, column temperature, CH_3CN content, and separating time. The column was a Zorbax Eclipse Plus-C18 column (4.6 mm I.D.×150 mm, 5 μm) with a flow rate of 1.0 ml min^{-1}. The temperature of column was set at 30 °C with injection volume of 5.0 μL and detection wavelength of 225 nm. The mobile phase consisted of CH_3CN and H_2O with the content of CH_3CN increasing linearly from 15% to 85% during 16 min and keeping 85% from 16.01 to 20 min. The re-equilibrating time of the column was 4 min. As shown in Figure 7, the separation of all peaks in DAAF was finished before 13 min with DAAF's retention time of 8.690 min and the resolution of all peaks was above 1.5 with high number of theoretical plates (N > 54259) under optimum condition of HPLC.

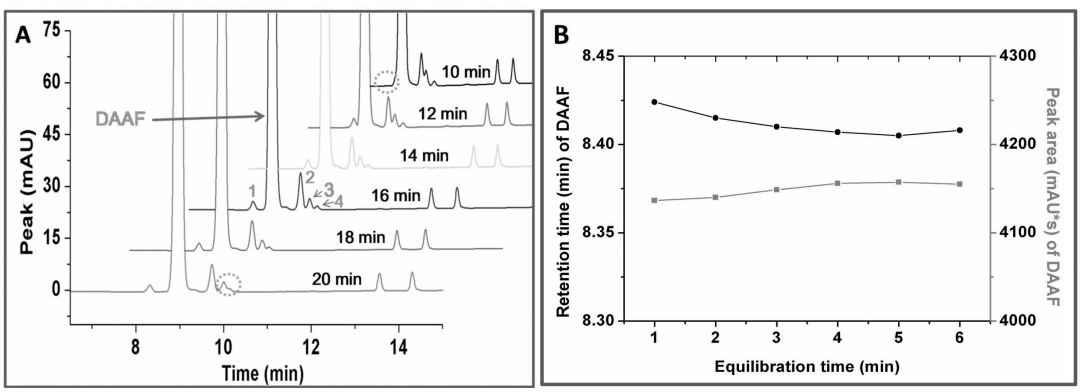

Figure 6. Chromatographic analysis of DAAF under different separating time (A), and retention time (●) and peak area (■) of DAAF under different equilibration time (B).

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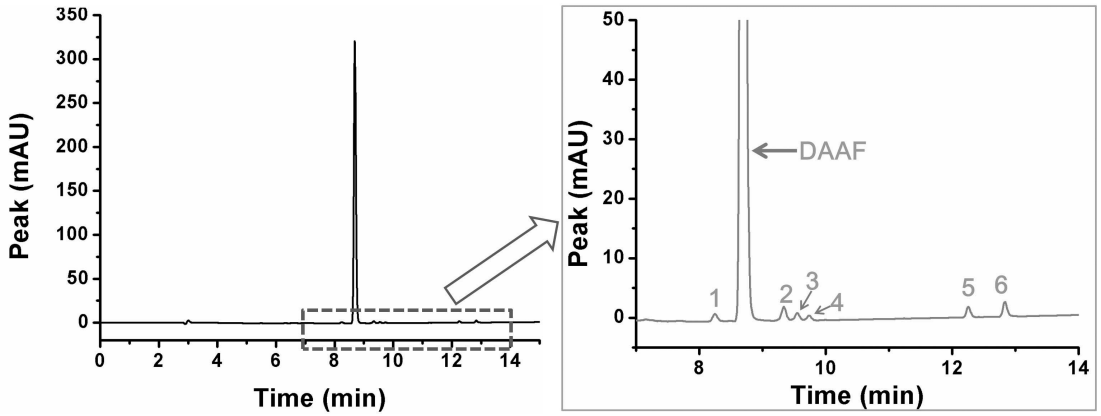

Figure 7. The chromatogram of DAAF under optimum condition of HPLC.

3.2 Method Validation

3.2.1 The Selectivity Towards Interferences

The solvent of CH_3CN without DAAF was used as a blank sample in order to verify the selectivity of HPLC method. CH_3CN was filtered through a 0.46 μm membrane and then injected into HPLC for the analysis. Meanwhile, the sample of CH_3CN containing DAAF was analyzed by HPLC-DAD after the same pretreatment. As shown in Figure 8, retention time of DAAF and its byproducts were located from 8.257 to 12.846 min, and there were no interferences found from blank sample during the separating time of DAAF. Hence, the analytical method was highly selective toward the analysis of DAAF.

3.2.2 Linearity, Sensitivity and Accuracy

The reference material of DAAF was used to prepare different concentrations of standard solutions at nine levels, and then they were injected into HPLC for quantitative analysis of DAAF. The regression analysis was operated by plotting peak area (y) versus corresponding concentration (x) to yield a regression equation ($y = 11.5793x + 0.1743$), and it displayed good linearity during wider range from 0.84 to 211.16 μg/mL with good correlation coefficient ($R^2 = 0.9996$) in Figure 9.

Limit of detection (LOD) and limit of quantification (LOQ) for DAAF were analyzed under the optimum condition of HPLC. LOD was evaluated with S/N ratio of 3, and it was 0.19 ng (38 ng/mL). Meanwhile, LOQ with the value of 0.63 ng (127 ng/mL) was obtained with S/N ratio of 10. So, the method displayed more sensitivity and wider linearity range than reported electro-analysis with the sensitivity at ppm and linearity range between 20 and 90 ppm [19].

Further, the accuracy was assessed by calculating the recovery of adding different concentrations of standard DAAF with three replicates for each level. The recoveries were from 98.24% to 102.72% in Table 3, and it was indicated that the method was highly accurate.

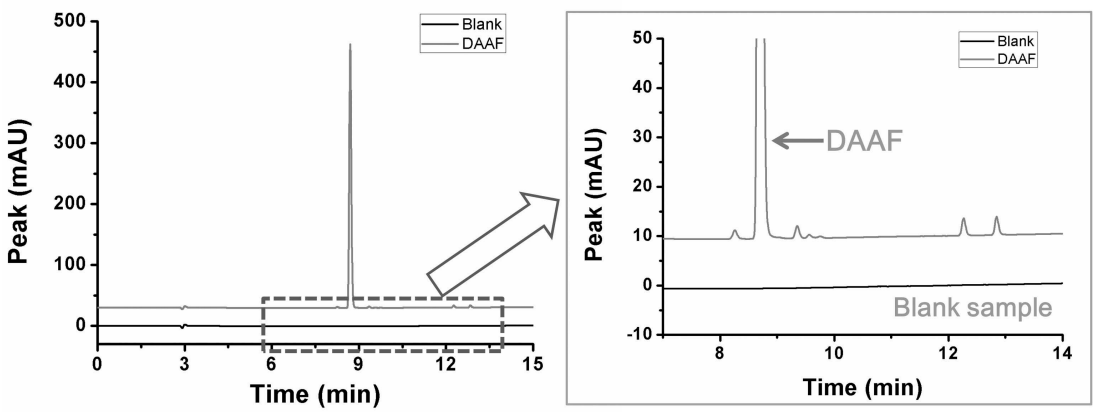

Figure 8. The chromatogram of blank sample without/with DAAF.

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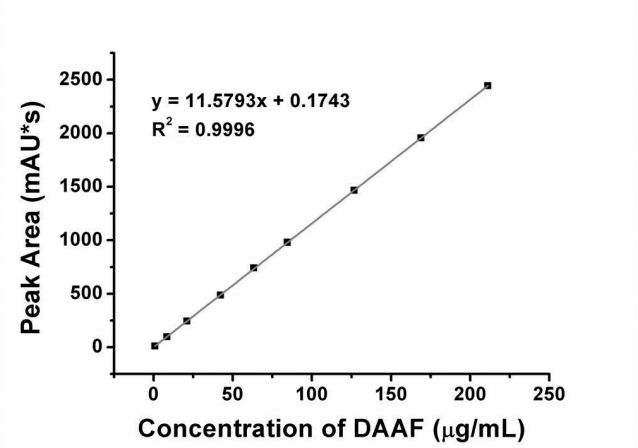

Figure 9. The regression analysis of peak area of DAAF versus its concentration.

Table 3. The recovery of DAAF after adding different standard solutions.

Samples	Added (μg/mL)	Found (μg/mL)	Recovery	RSD (n = 3)
DAAF 1	12.67	12.53	98.73 %	0.90 %
DAAF 2	25.34	25.82	101.91 %	0.40 %
DAAF 3	50.68	52.06	102.72 %	0.87 %
DAAF 4	101.36	99.58	98.24 %	0.65 %

3.2.3 The Precision

The inter-day and intra-day precision was determined in terms of relative standard deviation (RSD%). To evaluate intra-day precision, peak area was recorded with six consecutive injections per day. Inter-day precision was examined by 18 injecting analysis over three consecutive days (six injections for each day). RSD of intra-day precision was from 0.12 % to 0.18 % with the inter-day precision of 0.57 % in Table 4. So, the method was demonstrated to have adequate precision for quantitative analysis of DAAF.

3.2.4 The Stability

The standard solution of DAAF with the concentration of 0.2 mg ml^{-1} was used to test the stability of DAAF in the method. Peak area of DAAF was recorded at 1, 6, 12, 18, and 24 h, respectively, and the injection was carried out in triplicate at each time point. As shown in Table 5, there was no obvious change toward the peak area of DAAF with good RSD of 0.21 % during 24 h, so the method was enough stable for the analysis of DAAF in the long run.

Table 5. Peak area of DAAF under different time.

Time	Peak area			Mean	RSD
1 h	2351.2	2351.9	2352.6	2351.9	0.21 %
6 h	2355.8	2356.5	2356.3	2356.2	
12 h	2358.0	2360.9	2362.7	2360.5	
18 h	2362.1	2361.7	2362.8	2362.2	
24 h	2362.5	2363.9	2366.0	2364.1	

Table 6. Robustness of the method under different conditions.

Parameter	Setting	t_R (min)	R	N
Types of Column	Plus-C18	8.711	3.23	53081
	SB–C18	8.608	3.30	68346
	SB-phenyl	9.307	6.83	85868
Injecting volume (μL)	3	8.717	3.30	73676
	5	8.711	3.23	69924
	7	8.712	3.08	63038
Column temp. (°C)	28	8.782	2.63	67380
	30	8.713	3.19	65483
	32	8.669	2.60	63648

3.2.5 The Robustness

Robustness of the method was investigated to measure the resolution (R), retention time (t_R), and theoretical plates (N) of DAAF by changing the separation conditions of HPLC in Table 6. Although different types of column including Zorbax Eclipse Plus-C18, Zorbax Eclipse StableBond-C18 (SB–C18), and Zorbax Eclipse StableBond phenyl-C18 (SB-phenyl) had slight effect on the retention time of DAAF, all of their resolutions were above 3.23 with high theoretical plates ($\geq$ 53081). There was no obvious change in retention time and good resolution ($\geq$ 3.08) was observed with high

Table 4. The inter-day and intra-day precision of the method.

Day	Peak area (n = 6)						Intra-day precision	Inter-day precision
1	2180.1	2183.6	2190.8	2185.2	2188.4	2182.7	0.18 %	0.57 %
2	2196.8	2201.8	2199.1	2198.6	2199.5	2205.0	0.13 %	
3	2217.4	2213.3	2216.6	2210.7	2212.1	2214.7	0.12 %	

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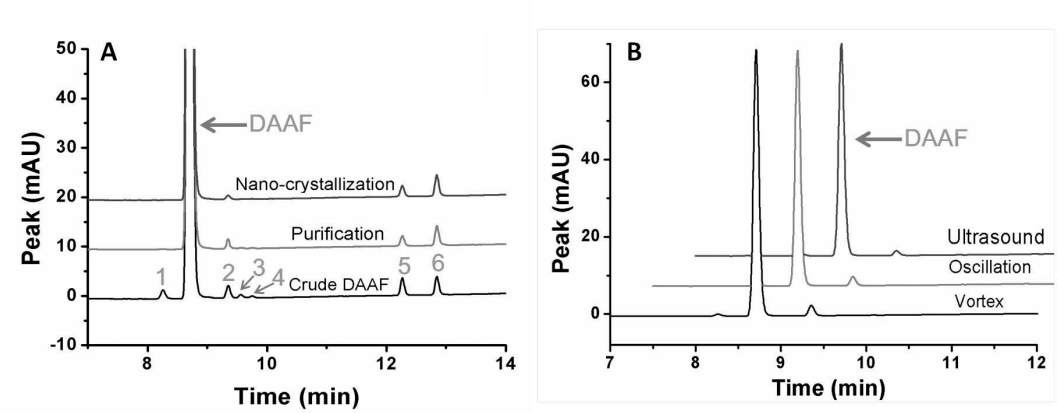

Figure 10. Chromatographic analysis of high purity of DAAF (A) and trace DAAF in wastewater (B).

theoretical plates ($\geq$63038) when the deviation of injection volume was 5 ± 2 μL. Good resolution and high theoretical plates were also obtained under column temperature of 30 ± 2 °C.

The slight variations in the types of column, injection volume, and column temperature did not led to considerable differences in the resolution and theoretical plates of DAAF. Thus, the method exhibited good robustness for DAAF.

3.3 Sample Analysis

3.3.1 Quality Control of Different Stages

The change of the purity of DAAF was analyzed by above-established method for quality control of different preparation stages including the synthesis, refinement, and nano-crystallization in Figure 10A and Table 7. DAAF from the synthesis had low purity of 93.38%, and the purity was increased to 97.20% after the refinement by using DMF as a solvent to remove some byproducts in DAAF. Higher purity of 99.64% was observed when refined DAAF was further nano-crystallized, and it was indicated that nano-crystallization resulted in great increase of the purity of DAAF from the synthesis.

Table 7. The determination of high purity of DAAF and trace DAAF in wastewater.

Different stages	Synthesis	Refinement	Nano-crystallization
The purity of DAAF	93.38%	97.20%	99.64%
Washing methods	Ultrasound	Oscillation	Vortex
Content of DAAF ($\mu g\,mL^{-1}$)	22.87	25.49	29.48

3.3.2 Environmental Monitoring of Wastewater

This method was also used to evaluate trace DAAF in washing wastewater for environmental monitoring around the explosive manufacturing. Three treatment methods including oscillation, vortex, and ultrasound were combined with water to wash DAAF with the production of wastewater, and chromatographic analysis was shown in Figure 10B. Then, these samples were centrifuged and the wastewater was injected into HPLC for the analysis of remanent DAAF. The concentration of DAAF in wastewater ranged from 22.87 to 29.48 $\mu g\,mL^{-1}$ through different washing methods in Table 7. The results indicated that the method had the capability of trace analysis of DAAF in environmental monitoring.

4 Conclusion

An analytical method based on HPLC-DAD was developed and validated for the analysis of DAAF in this study. Several conditions of chromatographic separation were optimized, and all of DAAF and its byproducts were separated and detected with high efficiency, resolution, and symmetry. Meanwhile, the method was proved to be sensitive, precise, accurate, robust, and reproducible through the validation. Further, potential applications of the method in real samples were evaluated, and it was capable of determining both the purity of DAAF during different preparation stages and trace DAAF in explosive wastewater. Overall, the method can be applied for the quality control of DAAF during the manufacture and environmental monitoring of DAAF in the wastewater.

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