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Research progress on detection methods of trace unsymmetrical dimethylhydrazine

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Abstract: Unsymmetrical dimethylhydrazine (UDMH) is an excellent liquid rocket propellant, but its toxicity is strong, which is harmful to human health and seriously pollutes the environment. However, the spectrophotometric method for detecting UDMH in the current Chinese national standards can't meet the detection requirements under high safety standards. This article not only reviews the research methods at home and abroad, but also discusses the advantages and disadvantages of these methods.

1. Introduction

There were lots of accidents caused by UDMH leakage in rocket and missile launching tests in many countries [1]. Therefore, countries have strict limits on its allowable concentrations in the environment. For example, the hygienic standard requirement of UDMH in the atmosphere of residential area in national standard of China [2] is as follows: the daily average maximum allowable concentration is 0.03 mg·m^{-3}; a single day maximum allowable concentration is 0.08 mg·m^{-3}.

In recent years, many researchers at home and abroad have carried on the research to UDMH detection methods, this review will make a brief introduction.

2. Colorimetry

The colorimetric method determines the content of the substance in the solution by comparing or measuring the color of the substance to be tested or adding a developer to obtain the color depth of the colored solution.

Mildred et al. [3] used sodium pentacyanamidoferrite (TPF) as a color reagent to measure μg-level UDMH in blood and water, it can provide early diagnosis information of exposed cases, and it can also be used to analyze air samples after modification. US military researchers used 2,4-dinitrobenzaldehyde as a derivatization reagent for UDMH, and the formed hydrazone was yellow to golden in color. When coated on porous filter paper and exposed, the limit of detection (LOD) was less than 20 ppb·hrs^{-1}, and the LOD after active suction of UDMH was less than 50 ppb·L^{-1}, but the system had a short usage time and high storage condition [4]. Li Baoting et al. [5] produced a batch of trace UDMH detection tubes with bromocresol blue as developer, which is valid for two years. The measurement range was 0.5 - 50 ppm, and the color changing range was from yellow to bluish

violet. Yao Xu et al. [6] developed a new colorimetric method for detecting UDMH based on the principle that gold nanoparticles (Au NPs) appear bluish violet by the coincidence reaction of chloric acid and UDMH. The results showed that the method performed well within the linear range of 2 M UDMH, which was 3.1% and 1.6% (n = 6), and successfully detected UDMH in water taps and lake water samples.

The colorimetric method has obvious advantages such as simple operation and equipment. It is the earliest method for determination of trace hydrazine propellants, but it also has disadvantages like low sensitivity and low quantitative detection accuracy.

3. Chromatography

Combined with some pretreatment methods, chromatography can be used to detect UDMH in complex environment.

3.1 Gas chromatography

Gas chromatography (GC) uses gas as the mobile phase, which can be separated quickly and efficiently because of its low viscosity, high mass transfer rate and high permeability. The main detectors selected when analyzing UDMH are nitrogen and phosphorus detector (NPD), mass spectrometry detector (MSD) and flame ionization detector (FID) [7].

Mazur et al. [8] used concentrators to perform chromatographic analysis on UDMH samples up to 40 μg. Sotnikov et al. [9] used NPD to analyze the hydrazone produced by UDMH and 4-nitrobenzaldehyde. The LOD was 0.03 μgŁ $^{-1}$, the relative error was less than 22% in the range of 0.06 - 0.60 μgŁ $^{-1}$, and less than 33% in the range of 0.03 μgŁ $^{-1}$. Bakaikina et al. [10] determined UDMH and its transformation in water by headspace solid phase microextraction (SPME) combined with gas chromatography-mass spectrometry (GC-MS) and gas chromatography-mass spectrometry (GC-MS/MS), the operation was easier and the degree of automation was higher. Polunin et al. [11] proposed a method for detecting UDMH on the surface of building materials, using GC-MS to analyze the more volatile conversion products, and using desorption mass spectrometry for non-volatile substances. Ul'yanovskii et al. [12] combined dispersive vortex liquid-liquid microextraction (VALLME) with gas chromatography-orbital mass spectrometry to simultaneously determine 24 compounds including UDMH, and the LOD was mostly 0.02 - 1.1 μg·L^{-1}. Within this range, the accuracy was higher than 81%, and 29 new conversion products of UDMH have been initially discovered.

In order to detect the trace amounts of UDMH gas, Cao Ye et al. [13] made it react with salicylaldehyde to generate dimethyl hydrazone and used GC-MS for quantitative analysis. Zhang Wei et al. [14] used the headspace method to determine UDMH in the simulated water sample under the best conditions, in the mass concentration range of 0.05 - 5.0 μg·L^{-1}, R^2 = 0.998. Zhang Yu et al. [15] used furfural derivatization combined with GC-MS to determine the content of UDMH in surface water. The linearity was good in the range of 0 - 100 μg·L^{-1}, and the LOD was 0.88 μg·L^{-1}. Zhang Guangyou et al. [16] used FID to determine UDMH in ambient air. When the sampling volume reached 60 L, the LOD and the limit of determination were 0.37 μg·m^{-3} and 1.2 μg·m-3. The relative standard deviation (RSD) of the measurement results of the low to high concentration of UDMH standard solutions were 3.5%, 2.6%, and 1.9%, respectively.

3.2 Liquid chromatography

Denisov et al. [17] reacted UDMH with 4-nitrobenzaldehyde to form hydrazone. Under the optimum conditions, the LOD in aqueous solution was 120 μgŁ $^{-1}$ with high performance liquid

chromatography (HPLC). Smirnov et al. [18] used glyoxal pre-column derivatization to measure UDMH and reversed-phase high performance liquid chromatography (RP-HPLC) combined with UV detector to measure its derivative 1,1-dimethylhydrazone. The measuring range of UDMH in water was 0.5 - 10000 μg·L^{-1}, after treatment with Strata SDB-L adsorption column, it was 0.01 - 20 μg·L^{-1}, and the RSD (n = 3) did not exceed 0.12 and 0.25, respectively. The contents of UDMH in natural water were determined by Osipenko et al. [19] with pre-column derivatization of phenylglyoxal and RPHPLC-MS, without pre-concentration and under optimized conditions, the LOD and quantification were 0.010 μgL $^{-1}$ and 0.030 μgL $^{-1}$. Amosov et al. [20] used 5-nitro-2-aldehyde derivatization and multi-wavelength spectrophotometry to determine the content of UDMH, MMH, hydrazine (Hy) and their main degradation products in visible light region, combined with RP-HPLC and post-column derivatized ion chromatographic separation, the LOD was below 1 μg·L^{-1}. The optimized chromatographic conditions of Han Ying et al. [21] were: C18 column separation, UV detector, acetonitrile and water as mobile phase, and the linear correlation coefficient was over 0.9994, the LOD was 0.5 μg·L^{-1}, the RSD of the simulated water sample was less than 1.69%.

Gas and liquid chromatography have low LODs and small relative errors. They can detect UDMH and its conversion products at the same time, but they also have disadvantages such as complicated operation and high requirements for the measurement environment.

3.3 Ion chromatography

Table 1: Chromatographic methods for determination of environmental UDMH.

Method	Pretreatment	Sample type	LOD	Remarks	Reference
GC-FID	4-nitrobenzaldehyde derivatization	Soil	10 μg·kg^{-1}	Simultaneous determination of derivatives and derivative reagents	[22]
GC-MS	2-nitrobenzaldehyde derivatization, methanol solution absorption	Air	9.5 ng·m^{-3}	High Sensitivity and rapid determination	[23]
HILIC-CLND	Acetonitrile removal by overnight column washing with methanol/water	Water	200 μg·L^{-1}	Simultaneous analysis of Hy and UDMH	[24]
GC-FID	Silica gel sampling with sulfuric acid furfural/sodium acetate derivative	Air	0.005 m·m^{-3}	analyze UDMH in closed environment	[25]
GC-MS	Alkali distillation / ultrasonic derivatization	Soil	0.0078 mg·kg^{-1}	Determination of trace UDMH in soil by water-sealed method	[26]
HPLC-MS	Hydrochloric acid extraction, stirring and centrifugal filtration	Soil	12.8 ng·mL^{-1}	Simultaneous-ly determine UDMH and MMH	[27]
HILIC-ESI-MS/MS	Acetonitrile extraction	Soil	1.7 μg·L^{-1}	Detection of UDMH and transformation products	[28]
RP-HPLC-UV-MS/MS	2-hydroxyquinoline formaldehyde	Water	3.7 ng·L^{-1}	No need for pre-concentration, pre-separation	[29]
IC	N/A	Water	0.005 mg·L^{-1}	First analysis by ion chromatogra-phy	[30]

Ion chromatography can analyze various organic, inorganic anions/cations and biochemical substances, and is widely used in food and pesticide residue analysis.

Smolenkov et al. [31] selected ion, ion-pair and chromatography and mass spectrometry in electrospray ionization mode to simultaneously determine UDMH and its conversion products in aqueous solutions. At the μg·L^{-1} level, up to 7 components can be determined. By using sodium octyl sulfate as the best ion pair reagent to determine the correlation coefficients of Hy, MMH, UDMH, nitrosodimethylamine and tetramethyl-2-tetrazene were all greater than 0.994. Kosyakov et al. [32] used CO_2 to extract UDMH from peat soil as samples and proposed that the extraction efficiency of UDMH could be improved by using acetone-modified SC CO_2 cosolvent.

The detection efficiency of ion chromatography is high, but because UDMH wastewater often contains metal cations, which causes great interference to the results, it is necessary to improve its

selectivity when analyzing trace amount of UDMH.

4. Electrochemical method

The electrochemical method is used to characterize and quantitative analysis components based on the relationship between electrical quantities such as potential or current and certain quantities of the substance being measured.

Ashwini et al. [33] used polymethyl methacrylate microchips to separate three types of hydrazine quickly. The LODs of Hy, MMH and UDMH were 11.9 $ng \cdot mL^{-1}$, 35.5 $ng \cdot mL^{-1}$, and 337.8 $ng \cdot mL^{-1}$. After ultrasonic-assisted headspace elimination of interference, the LODs of Hy, MMH and UDMH were increased to 6.5 $ng \cdot mL^{-1}$, 15.3 $ng \cdot mL^{-1}$ and 11.4 $ng \cdot mL^{-1}$. Kosyakov et al. [34] established a method based on the zwitterionic sulfobetaine stationary phase of high-performance liquid chromatography (Nucleodur HILIC) combined with amperometric detection, which can simultaneously determine Hy, MMH and UDMH in natural water and soil. The LOD was 0.07 - 0.13 $\mu g \cdot L^{-1}$, the analysis error of river and groundwater did not exceed 10%.

Zhang Youzhi et al. [35] designed the Metrohm 809 automatic potentiometric titrator program to analyze UDMH, which solved the key issues of titration mode selection, end point determination and titration speed, after discussing the influence of ambient temperature on the results, they also determined the optimal detection conditions. Dong Chao et al. [36] used Prussian blue (PB) as the electron transfer regulator in the oxidation process of UDMH, and the electrode used PB modified carbon paste electrode (PB/CPE). The linear range of the determination was 0.3 - 80 $mg \cdot L^{-1}$, and the LOD was 4.6×10^{-5} $g \cdot L^{-1}$. Ren Xianghong et al. [37] prepared PB/TNTs/CPE with TiO_2 nanotubes (TNTs) as the carrier of PB. The results showed that when TNTs were used as the carrier, the electron transfer efficiency and rate are improved, the linear range of the determination was 0.3 - 100 $mg \cdot L^{-1}$, and the LOD was 2.6×10^{-5} $g \cdot L^{-1}$. Hu Liming et al. [38] used perfluorinated sulfonate (Nafion) as a solvent to disperse multi-walled carbon nanotubes (MWCNTs), and prepared MWCNTs-Nafion modified glassy carbon electrodes, and used differential pulse voltammetry to determine the content of UDMH. The results showed that the electrode has good enrichment ability and electrocatalytic activity for UDMH. When its concentration was in the range of 1.3×10^{-6} - 4.0×10^{-5} $mol \cdot L^{-1}$, the oxidation peak current had a good linear relationship.

The electrochemical sensors which have low LODs and high sensitivity have been applied to the rapid detection of UDMH gas in air, but they also have disadvantages of high cost and regular maintenance.

5. Spectral method

The spectroscopy method uses the characteristic spectrum of the measured material's extranuclear electrons to transition between two specific energy levels, carries out qualitative analysis according to its characteristic wavelength, and realizes quantitative analysis according to the correlation between its characteristic spectral signal intensity and its content.

5.1 Chemiluminescence method

Chemiluminescence method has advantages of simple equipment, fast analysis, wide linear range, low LODs, high sensitivity, etc. It often combines with flow injection analysis.

Liu Quan et al. [39] used luminol-potassium permanganate system to determine trace amounts of UDMH in water. The detection linear range was 1.0×10^{-6} - 1.0×10^{-5} $g \cdot L^{-1}$, and the LOD was 1.0×10^{-7} $g \cdot L^{-1}$, the RSD for 11 parallel determinations of 1.0×10^{-5} $g \cdot L^{-1}$ UDMH was 1.9%. The team of Wu Wan'e [40] and Zhang Huitan et al. [41] used potassium bromate-luminol system to determine UDMH

in water. The linear range of measurement was 1.0×10^{-7} - 1.0×10^{-5} g·L^{-1}, and the LOD was 4×10^{-8}g·L^{-1}, the RSD for 11 parallel determinations of 5.0×10^{-7} g·L^{-1} UDMH was 2.3%.

5.2 Spectrophotometer

Spectrophotometry is the method for detecting UDMH in Chinese national standards. UDMH and TPF form a red complex in a weak acid aqueous solution. In the range of 0.01 - 1.0 mg·L^{-1}, the color depth is proportional to the concentration. However, there are many interference factors in this method, such as Hy, MMH, which will interfere with accuracy of the determination, so the selectivity and sensitivity need to be improved.

Kosyakov et al. [42] discovered a new derivatization called reagent-5-nitro-2-furaldehyde, which has high solubility in water and the formation of derivatives with obvious absorption bands. After optimizing the reaction conditions, the LODs of Hy, MMH and UDMH were 5 μg·L^{-1}, 3 μg·L^{-1} and 1.5 μg·L^{-1}. Taheri et al. [43] had established a new one-step time-weighted average for determination of UDMH in air. The daily and daytime reproducibility of UDMH were in the range of 0.082 - 0.1% and 0.091 - 0.12%, the LOD and quantitative limit were 0.002 ng mL^{-1} and 0.006 ngm L^{-1}. Han Zhuozhen et al. [44] used the solution absorption method for sampling, which simplified the steps of the GJB 2373-95 method. By comparing the real-time monitoring value of the sensor, it was considered that the method is more convenient and time-saving. Liu Weiguo et al. [45] intended to solve the problem that the determination of UDMH in propellant sewage is manual operation, which can't be detected in different places and in real-time, a flow injection analysis method for UDMH determination in sewage had been developed, the LOD was 0.1×10^{-4} gL^{-1}. Yao Xu et al. [46] improved the detection efficiency of UDMH in the air by solution absorption/spectrophotometry, analyzing its main influencing factors, and compared it with the national standard method. The results showed that two bubble tubes in series can meet the collection efficiency, and the best absorption speed was 0.2 L·min^{-1}, the standard deviation of the method was less than 5%, and comparing to the error of the national standard method was less than 2%.

5.3 Fluorescence analysis

Fluorescence analysis is a method for qualitative and quantitative analysis of samples using their fluorescence characteristics of substances, but UDMH doesn't have fluorescence characteristic, so it is necessary to use a derivatization reagent with certain fluorescence characteristics or react with it to generate fluorescent substances for derivatization and measurement.

Collins et al. [47] derivatized with o-phthalaldehyde (OPA), naphthalene-2,3-dicarbaldehyde (NDA) and anthracene-2,3-dicarbaldehyde (ADA) to determine Hy, MMH and UDMH. Through the control of pH and selection of aromatic dialdehyde, Hy, MMH and UDMH in mixed samples were distinguished and quantitatively detected. The LODs of NDA for MMH and UDMH were 120 ng·L^{-1} and 40 mg·L^{-1}, respectively. Zhang Yuqi et al. [48] fixed rhodamine B on the surface of a glass substrate to prepare a fluorescent sensing film with characteristic response to hydrazine gas. In a confined space at room temperature, when the concentration of UDMH gas gradually increased, the fluorescence emission intensity of the film decreased significantly, with an instantaneous quenching rate of 56.79% and 77.43% at 4 min. The response was reversible and selective.

5.4 Infrared spectrometry

Valakh et al. [49] used near-infrared absorption spectroscopy to determine the content of UDMH in the atmosphere. The method can diagnose the presence of up to 6 g·m^{-2} UDMH gas. Ren Manyan et al. [50] established a method for monitoring UDMH based on open optical path Fourier transform

Infrared Spectroscopy (OP-FTIR). When the concentration was below 1 ppm, the measurement error of UDMH was about 10%.

Cao Ye et al. [51] based on the infrared absorption characteristics of the three types of hydrazine and NO_2, developed a UDMH gas monitoring system that works in the near-infrared and mid-infrared bands. The near-infrared monitoring system was based on the principle of semiconductor laser absorption spectroscopy (TDLAS), which can realize online and real-time detection of hydrazine gas concentration. The mid-infrared monitoring system was based on the non-dispersive infrared absorption spectroscopy (NDIR) of the gas filter related technology (GFC), which can automatically measure the pressure and temperature of the absorption cell and control the flow and temperature, and the LOD can reach 1 ppm.

6. Conclusion

With the rapid development of aerospace industry, UDMH and various propellants are used in more and more occasions and dosages. Therefore, the improvement of UDMH detection technology has attracted the attention of scientific researchers, and more and more new methods have been used for detecting UDMH. For example, Turusova et al. [52] established a photochemical method for the determination of UDMH with the LOD 0.49 $\mu g \cdot mL^{-1}$ and the determination limit of 1.62 $\mu g \cdot mL^{-1}$.

At present, the spectrophotometric method in Chinese national standard method is not suitable for UDMH detection in the complex environment due to its complicated operation and long time-consuming. However, the chromatographic method with low LODs and high sensitivity is limited by the precision of the instrument and the complicated operation, so it is difficult to monitor UDMH in real-time; although electrochemical sensors have been widely used in UDMH concentration monitoring in various positions, they need to be overhauled regularly and there are "blank periods" in monitoring. Therefore, the detection of UDMH by spectroscopy has a larger development space and a broader prospect. In a word, the UDMH detection method with higher sensitivity, smaller relative error, shorter response time, more portable equipment, real-time monitoring, longer use time, and lower cost will surely become the future research direction, making it suitable for measuring UDMH under special environment and high safety standards.

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