Microwave-assisted Extraction for the Chromatographic Analysis of Propellant

Xiaoyu Zhou, Ran Xiong [a], Bin Zhang, Yan Luo
The 63981 unit of PLA. China
[a] Bxiongran@163.com

Keywords: Propellant, UAE, SDWP, Stabilizer, Gas chromatography.

Abstract. In order to timely and accurate understanding of propellant in the process of storage, security, need for stabilizer determination of propellant, the microwave-assisted extraction (UAE) and solvent-dissolve-water-precipitation extraction (SDWP) were used in this paper for chromatographic analysis of propellant, comparing the result of the test, the error is small, get the microwave-assisted extraction can be used in the gas phase chromatography determination of propellant stabilizer experiment, this method shortens the test time, improve the test efficiency.

1. Introduction

Propellant is a major energy source for launching projectiles or propelling the rocket movement. It is mainly stored in the warehouse, and its storage security is directly related to wartime use. In the process of storage, the nitrifying cellulose will automatically decompose, which can delay or inhibit the reaction of the reaction when adding the tranquilizer, thus improving the chemical stability of propellant. As a result, the stabilizer can display prolong the storage life of propellant, regular inspection of stabilizer in the propellant, can grasp the quality situation of propellant, so as to guide the management of ammunition properly, use and storage. The microwave-assisted extraction (UAE) and solvent-dissolve-water-precipitation extraction(SDWP) were used in this paper from many traditional powder sample pretreatment method, compare the result of the microwave-assisted extraction to the feasibility of the application of chromatographic analysis for propellant.

2. Test method

2.1 Sample preparation

A single base propellant: dense texture, with a glass slide; A double - based propellant: dense texture, with a slide of glass; A three - base propellant: dense texture, with slides blown into a flower; Some propellant: the texture is more dense, use glass to scrape the flake. Powder sample pieces: the principle of combustion layer thickness is not more than 0.5mm medicine take whole grain, grain burning layer thickness is greater than 0.5mm medicine grain processing into small piece of 2-3mm, 3mm and 2mm double screen, take on the 2mm sieve sieve.

2.2 Instruments and Reagents

Reagent: propanone: analysis of pure GB686-78; Petroleum ether: pure HG3-1006-76, boiling point 60 ℃ ~ 90 ℃;

Instrument: gas chromatograph: sp-2304 gas chromatograph, as shown in Fig.1; Ultrasonic cleaning instrument: KH - 3200B type ultrasonic cleaner, as shown in Fig.2.

Fig.1 Gas chromatograph 3420A

Fig.2 Ultrasonic cleaner

2.3 The chromatographic conditions

Carrier gas: high purity hydrogen, flow rate 200mL/min ~ 240mL/min;

Column temperature: 155 ℃ ~ 160 ℃;

Gasification temperature: 250 ℃.

Bridge current: 180mA ~ 200mA;

Chromatographic separation column: 3mm diameter, 500mm stainless steel column.

2.4 Soak

Solvent-dissolve-water-precipitation extraction: Said to take the crushed sample 1.5g~2g (called a quasi to 0.001g), in the 50ml of the clean with triangle in a bottle, quantitative add fluid tube with a pipette or add 15ml volume for (single-base medicine for 4:6, double-base medicine for 3:7) acetone - petroleum ether mixture, after soaking 2.5h ~ 4h, can be determined.

Microwave-assisted extraction: Said in the crushed sample 1.5g~2g (called a quasi to 0.001g), in the 50ml of the clean with triangle in a bottle, add about 80 ml of acetone ultrasound 20min (power density is about 0.2w.cm-2), after transfer into 100ml volumetric flask and constant volume of water, the filtrate is the sample solution (about 0.5h) sample preparation time, can be determined.

2.5 Standard solution preparation

The standard solution is prepared by 1.4.

2.6 Calibration

The instrument was normal and the baseline was flat, the standard solution of 6μL was taken with a microsyringe and the chromatograph was injected to measure the peak height of the stabilizer. The peak height difference of 3 times is not greater than 5%.

2.7 Determination

After the calibration, the sample was injected with a microsyringe 4μL, injected with the chromatograph, and measured the peak height of the tranquilizer. The difference of the peak height was not greater than 5%. After testing 2 ~ 3 samples, the calibration is done

3. Results calculation and analysis

3.1 Calculation formula

Using formula (1) to calculate the content of the tranquilizer:

$$C_i = \frac{W_0 \times H_i}{W_i \times H_0} \times C_0 \times 100\% \quad (1)$$

Type:

Ci——The content of the tested sample, %;

C0——The content of the standard propellant, %

Hi——The peak of the tested sample;

H0——The peak of the standard propellant;

Wi——The quality of the tested samples, g;

W0——The quality of the standard propellant, g;

3.2 Error provision

Each sample was prepared with two bottles of leaching fluid, the result was accurate to 0.01%, the difference between the two measurements was not more than 0.1%, and the sample results were averaged.

3.3 Test results and analysis

The single-base, double-base, sanki-base and propellant were used to gas chromatography determination of propellant stabilizer test, and the formula (1) is used for calculation, test results and calculation results are shown in table 1

Table 1. the test data of propellant tranquilizers for gas chromatography

Sample	Stabilizer content (%) (UAE)	Stabilizer content (%) (SDWP)	Error
Single-base	1.50	1.58	-5.1%
Double-base	2.65	2.54	4.3%
Sanki-base	1.40	1.33	5.3%
Propellant	3.15	3.08	2.3%

Through the content of the stabilizer in the powder in the table 1 shows that the UAE and the SWDP to get the error were within 6%, the error is small, thus, the UAE can be used for the powder of chromatographic analysis.

4. Conclusion

1)The UAE was uesd to chromatographic analysis of propellant, the test data compared with the SWDP, the error is small, thus, the UAE can be used for chromatographic analysis of propellant.

2)The extraction time of the UAE was 0.5h, the SWDP requires 3 hours, so the UAE can shorten the extraction time of propellant, so as to improve the efficiency of the analysis of propellant.

References

[1] General equipment department general equipment protection department. Gunpowder test [M]. Beijing: National defense industry press, 2000.

[2] General equipment department general equipment protection department. General theory of ammo detection [M]. Beijing: National defense industry press, 2000.

[3] Li dongyang et al. Analysis design and test of ammunition storage reliability [M]. Beijing: National defense industry press, 2013.

[4] Sample pretreatment techniques for chromatographic analysis of energic materials [J]. Energic materials, 23 (3): 279-284.