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A Study on Consumption and Characterization of Stabilizer Content of NEPE Propellant via FTIR

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Abstract: Nitrate ester plasticised polyether (NEPE) propellants, a kind of high-energy solid propellant, were tested for their stabilizer content and Fourier transform infrared spectrum (FTIR) to study their ageing performance. On the one hand, a stabilizer consumption equation containing the ageing temperature, stabilizer content, and ageing time was established by analyzing the data of stabilizer content of NEPE aged at high temperatures (328, 333, 338, 343, 348, and 353 K). On the other hand, it was found that the second derivative spectral absorbance of 1598 cm^{-1} has a high linear correlation to the stabilizer content of NEPE propellant, and an equation, which can be used to calculate the stabilizer content of NEPE with FTIR data, was established. Thereafter, to prove the universality and accuracy of these two equations, FTIR testing of NEPE aged at room temperature (298 K) was carried out. Accordingly, the two other equations for stabilizer consumption at room temperature were derived by the FTIR data and the stabilizer consumption equation, respectively. They were consistent and the universality and accuracy of these equations were proven, and the feasibility of non-destructive monitoring methods for NEPE propellants via FTIR was experimentally verified.

Keywords: NEPE propellant · stabilizer content · infrared spectrum

1 Introduction

With the increasing energy density and specific impulse in solid rocket motors, improved solid propellants are always required. Many researchers have already studied the physical, chemical, mechanical, thermal, and combustion properties of such solid propellants [1–5]. Nitrate ester plasticised polyether (NEPE) propellants, a new generation of high-energy solid propellants, have become a research focus: they make use of polyethers, such as polyethylene glycol (PEG) or ethylene oxide–tetrahydrofuran (PET) co-polyether, as a binder. Additionally, a mixed nitrate (usually nitroglycerin (NG) and 1,2,4- butanetriol trinitrate (BTTN) is involved, serving as a plasticiser and an energetic component [6–9].

Currently NEPE propellants have been widely used in rockets and missiles. The long-term performance of solid rocket motors is mainly determined by the propellant storage stability. Therefore, studies of NEPE storage performance have been significant in avoiding economic losses caused by performance failures or premature retirements. During the ageing process of the NEPE propellant, NG decomposes and releases nitrous oxides and consequently accelerates the degradation of the nitrate ester [10–14]. As a consequence, changes in the physical, chemical, mechanical, ballistic, and thermal properties of the propellants occur. These changes also alter the propellant performance [15–18], therefore, stabilizers are needed to react with the products of decomposition such as NO, NO_2, HNO_2, and HNO_3 to stop the catalytic action. The propellant will have a much longer service life, and early decomposition or even explosion during storage will be avoided [19,20]. In summary, research on stabilizers to improve NEPE propellant storage performance and predict service life is important.

There are already several reports on the NEPE propellant stabilizers. M.A. Bohn [21,22] deduced a propellant stabilizer consumption equation from a dynamic model including zero-order and first-order reactions. Afterward, the research was subsequently further improved, *i.e.*, the stabilizer content was calculated via the reaction rate in kinetic models, and the storage life was then inferred.

W. Stephens [23] analysed the infrared spectrum of solid propellants aged nearly 8.5 years using Fourier transform infrared spectroscopy. The results showed that the infrared spectra of the solid propellants were sensitive to the binder matrix microstructure responses. The wavenumber associated with macroscopic properties were found, which made it possible to predict the storage life of the solid propellants by infrared spectroscopy and correlation analysis.

R.A. Fifer [24] examined solid propellant surface composition via Fourier transform infrared photoacoustic spectroscopy (FTIR-PAS). It was found that FTIR-PAS was suitable for the characterization of the composite propellant surfaces. Thus, FTIR-PAS can be incorporated into both quality

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Table 1. Accelerated ageing schedule.

Ageing temperature [K]	Ageing time [day]															
328	0	40	80	120	150	180	220	240	270	290	320	350	380	410	430	
333	0	30	60	90	120	150	180	210	230	250	270	290	310	330	350	370
338	0	20	40	60	80	100	120	130	140	150	160					
343	0	15	30	45	55	65	75	80	85	90	95	98	101	104		
348	0	10	20	25	30	34	38	41	44	47	50					
353	0	4	6	8	10	12	14	16	18	20	22					

control and quality assurance monitoring of solid propellants.

Nevertheless, the stabilizer consumption equation has been deduced in theory only, and the non-destructive detection of propellants has not yet been achieved in engineering practice. Here, FTIR and stabilizer content tests of NEPE propellant aged at high temperatures were carried out. Two equations describing the stabilizer consumption rate at different temperatures and the relationship between stabilizer content and infrared spectrum, respectively, were derived. Thereafter, verification testing was also carried out to show that these two equations were universal and accurate. The feasibility of a non-destructive test for propellant stabilizer content was also experimentally verified.

2 Experimental Section

2.1 Accelerated Ageing Test

The main equipment used for the high-temperature accelerated ageing test was an AHX2863 safety oven, which consists of the box body, a heating temperature controller, thermostatic apparatus, and sample frame. The propellant sample measured 160 mm×30 mm×10 mm, was sealed with aluminium foil, placed into the ageing chamber, and subjected to accelerated ageing at six temperatures (328, 333, 338, 343, 348, and 353 K). Table 1 summarises the accelerated ageing schedule.

2.2 Stabilizer Content Test

The stabilizer content was determined by gas chromatography according to the GJB-770B-2005 standard method 216.1 [25]. Samples were prepared according to GJB-770B-2005 standard method 101.1 and extracted according to standard method 102.1 [25]. flame ionization detector was used in gas chromatograph. The quantitative method was internal standard method. Diethyl phthalate was selected as internal standard substance. The component content was calculated by analyzing The area under the peak of standard solution and solution to be measured. Each sample was tested twice, and The stabilizer content was obtained by taking an average value.

2.3 FTIR Test of Propellants Aged at High Temperature

The infrared spectra of the propellants aged at high temperature were obtained by a horizontal attenuated total reflection (HATR) approach. The model of infrared spectrometer used was an 60SXR (Thermo Nicolet Corporation). ZnSe was used as the crystalline material of the probe (light range 4000 to 650 cm^{-1}). The refractive index was 2.42, the infrared reflection angle was 45°, the infrared spectral resolution was 2 cm^{-1}, and the frequency range was 4000 to 650 cm^{-1}. The FTIR test was performed on slices of the propellants.

2.4 FTIR Test of Propellants Aged at Room Temperature

The NEPE propellant cartons were aged at 298 K from 0 to 11 years, and the NEPE propellant samples used in the test was cut from propellant cartons. To prevent the propellant from being destroyed, the infrared spectrometer WQF-510 model (Beijing Beifen-Ruili Analytical Instrument Group) has been improved, and a constant pressure fibre attenuated total reflection (ATR) probe which can be used in infrared spectrum analysis was developed [26]. The infrared spectra of NEPE propellants aged at room temperature were obtained by using this fibre. FTIR of the inner and outer of the propellant were tested at the same time.

3 Results and Discussion

3.1 Deduction of the Stabilizer Consumption Equation

The stabilizer of the propellant samples was 2-nitrodiphenylamine, and the initial content thereof was 0.5%. To provide a dimensionless relationship between ageing time and stabilizer content, the data was normalized. The initial value of stabilizer content is considered as 1. The stabilizer content v. ageing time at each ageing temperature is shown in Figure 1.

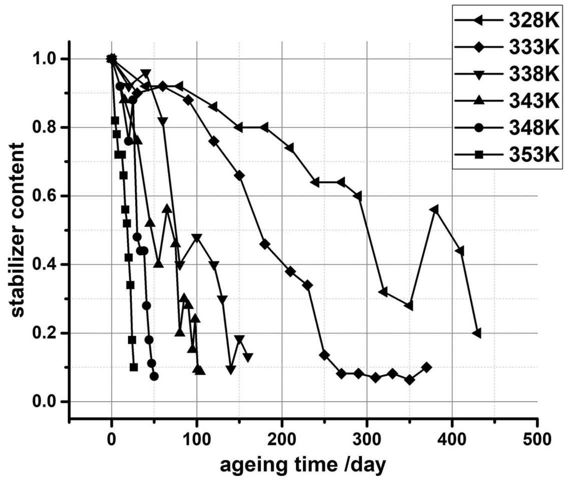

Figure 1. Stabilizer content *v.* ageing time.

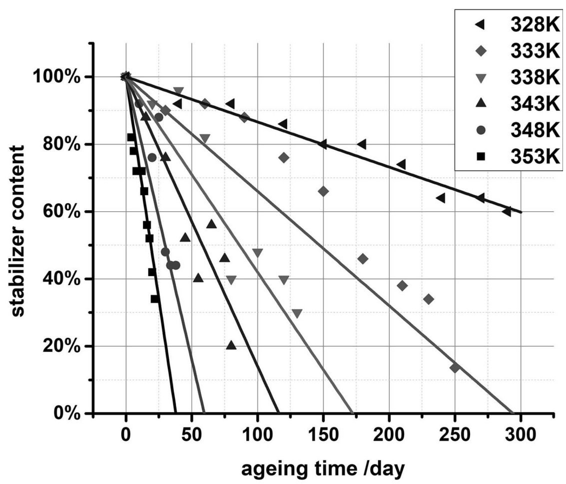

Figure 2. Fitting lines: stabilizer content and ageing time.

Table 2. Coefficients *b* and R^2.

T	353 K	348 K	343 K	338 K	333 K	328 K
b	−0.0264	−0.0162	−0.0086	−0.0058	−0.0034	−0.0013
R^2	0.9516	0.8081	0.8736	0.8775	0.9363	0.9674

As shown in Figure 1, the stabilizer content and the ageing time are linearly related; however, because of the failure of NEPE propellants in the late stage of ageing, the consumption rate of stabilizer content changed. The stabilizer content tested at this period was meaningless. Therefore, it was necessary to remove the data where the stabilizer content was unstable at the end of the ageing period. The fitting results are shown in Figure 2.

The fitting equation for the stabilizer content and ageing time is as follows:

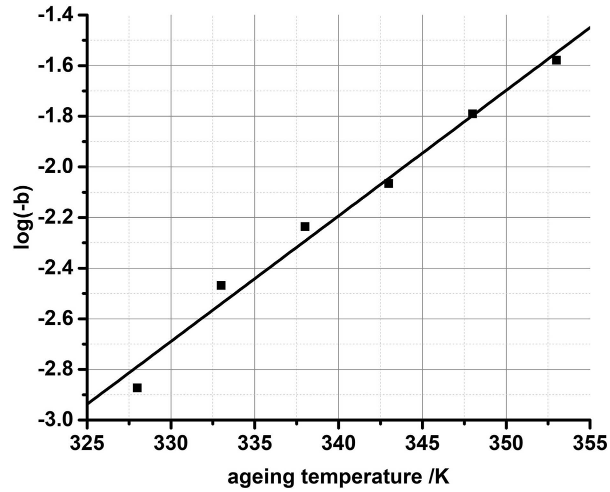

Figure 3. Logarithm of the coefficient b as function of ageing temperature.

$$\alpha = a + b\tau \quad (1)$$

where α is the stabilizer content, τ is the ageing time (day), and a and b are fitting coefficients; because the initial stabilizer content of the NEPE was known to have been 1, $a = 1$. The values of b and coefficient of determination R^2 are listed in Table 2.

As shown in Table 2, there is a significant linear relationship between ageing temperature and log (−b). Next, ageing temperature and log (−b) were fitted. Figure 3 shows ageing temperature v. log (−b):

The fitting results are described by Equation 2:

$$log(-b) = -19.05 + 0.0496T \quad (2)$$

In this equation, T is the ageing temperature (K). The linear correlation coefficient r is 0.9923, which indicated significant correlation. Consequently, the fitting results can be deduced as follows:

$$b = -10^{-19.05+0.0496T} \quad (3)$$

So, Equation 1 can be written as:

$$\alpha = 1 - 10^{-19.05+0.0496T}\tau \quad (4)$$

Equation 4 is the stabilizer consumption equation.

3.2 Characterization of Stabilizer Content via FTIR

Vibrating bonds in functional groups absorb energy at a frequency that corresponds to the vibrational frequency of the bond. These frequencies are expressed as wave-

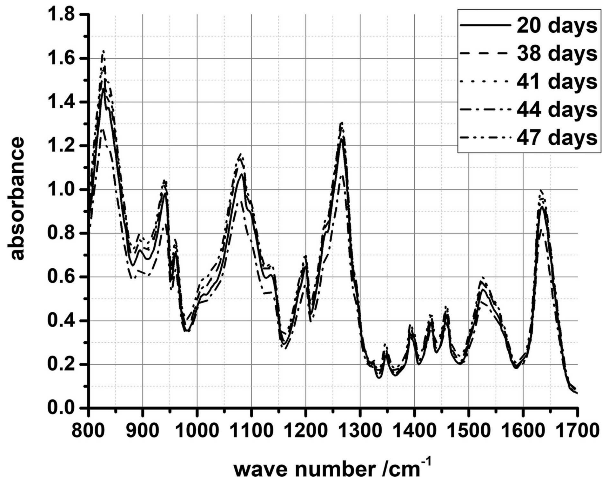

Figure 4. Infrared spectra of NEPE aged at 348 K.

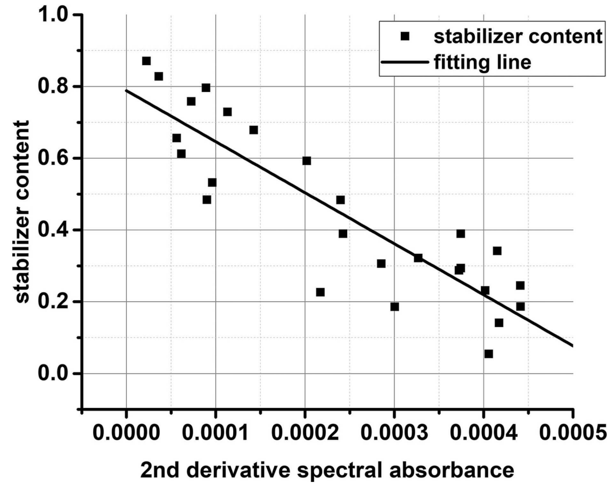

Figure 5. Fitting line of second derivative absorbance value of 1598 cm^{-1} and stabilizer content.

Table 3. Coefficients *a* and *b*.

Ageing temperature [K]	Ageing time [days] Stabilizer content					
348	20	38	41	44	47	
	0.679	0.390	0.342	0.294	0.245	
343	30	75	85	90	95	
	0.729	0.322	0.231	0.186	0.141	
338	40	80	120	140	160	
	0.796	0.593	0.389	0.288	0.185	
333	60	120	180	270	330	
	0.828	0.656	0.484	0.226	0.055	
328	40	150	240	290	320	430
	0.871	0.758	0.613	0.532	0.484	0.306

numbers. If the frequency of the radiation matches the vibrational frequency, the bond will absorb the radiation and the amplitude of the vibration will increase. Each type of bond vibrates at a characteristic wavenumber, which makes infrared spectroscopy useful when trying to identify functional groups. The infrared spectra of NEPE aged at 348 K are shown in Figure 4.

Second derivative spectroscopy is the technique of recording the second derivative of the spectrum with respect to the wavelength. It offers a distinct advantage over conventional spectroscopy [27]. The second derivative spectra enhanced and enlarged the minor difference in the original infrared spectra, and the overlapped peaks can be more clearly recognized for easier comparison and analysis. Currently second derivative spectroscopy has been widely used to quantitatively detect components in liquid, solid and gas mixture [28–30].

This technique was used to identify the relationship between the characteristic wavenumber and functional groups in NEPE propellant, and Equation 5 was used to obtain the second derivative spectrum [31], The ageing time and the stabilizer content are listed in Table 3.

$$\left[\frac{d^2y}{dx^2}\right]_i = \frac{1}{1001(d\sigma)^2} \left[22(y_{i-6}+y_{i+6})+11(y_{i-5}+y_{i+5})+2(y_{i-4}+y_{i+4}) - 5(y_{i-3}+y_{i+3})-10(y_{i-2}+y_{i+2})-13(y_{i-1}+y_{i+1})-14y_i\right] \tag{5}$$

The correlation between the stabilizer content of propellant and the second derivative absorbance of each wave number was measured by using the Pearson correlation coefficient. The results show that the maximum correlation coefficient was −0.882, and the characteristic second derivative absorption band was 1598 cm^{-1}. Thus, the second derivative spectral absorbance of 1598 cm^{-1} can be used to describe the stabilizer content of NEPE in a quantitative sense. In Figure 5, the X-axis delineates the second derivative absorbance value of 1598 cm^{-1}, and the Y-axis delineates the stabilizer content of the propellant.

The fitting equation is:

$$\alpha = 0.788 - 1.42^{*}10^{3}x \tag{6}$$

Equation 6 can be used to calculate the stabilizer content of NEPE, where α is the stabilizer content and x is second derivative absorbance value of 1598 cm^{-1}.

The band at 1598 cm^{-1} was attributable to N–H bond stretching in this aromatic organic compound [32]. It indicated that the 2-nitrodiphenylamine in the NEPE propellant reacted with the NO_x decomposed from nitric acid ester during ageing. As a result, when the ageing time was prolonged, the stabilizer content decreased, and second derivative absorbance of 1598 cm^{-1} increased. This meant that it was reasonable to characterise the stabilizer content of NEPE propellant with a second derivative absorbance of 1598 cm^{-1}.

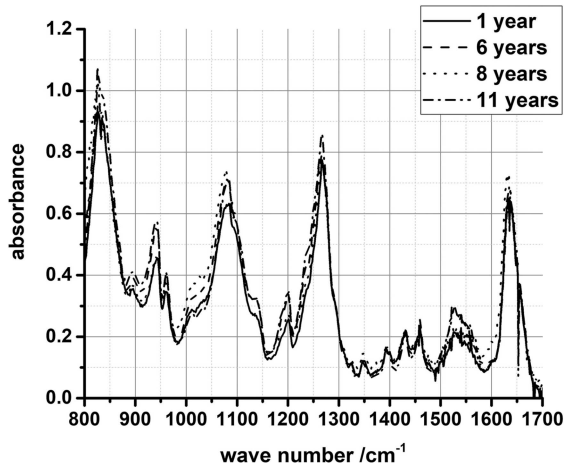

Figure 6. Infrared spectral data after different ageing times.

Table 4. Stabilizer content after room temperature ageing.

Ageing time [year]	Second derivative spectral absorbance	Stabilizer content
0	−3.290E-04	1.26
1	−3.276E-04	1.25
2	−2.817E-04	1.19
3	−2.726E-04	1.18
4	−1.965E-04	1.07
5	−2.031E-04	1.08
6	−3.403E-04	1.20
7	−2.067E-04	1.08
8	−1.966E-04	1.07
9	−1.711E-04	1.03
10	−3.501E-04	1.28
11	−7.342E-05	0.90

3.3 Verification Test

To prove the universality and accuracy of Equations 3 and 5, FTIR testing of NEPE propellant aged at room temperature was carried out. The composition of the propellant used in this test is the same as the previous one (see Figure 6).

FTIR of the inner and outer of the propellant were analyzed. The results showed that the chemical composition of inner grain was same as that of the surface of propellant. The second derivative spectral absorbance of 1598 cm^{-1} of the FTIR data were extracted, and the stabilizer content value was calculated from Equation 5. The results showed that the stabilizer content of inner and the outer surface of the propellant were almost the same. After that, the stabilizer content were obtained by taking the average value. The data processing results are shown in Table 4.

The fitting results are described in Equation 7:

$$\alpha = -0.000050\tau + 1.23 \tag{7}$$

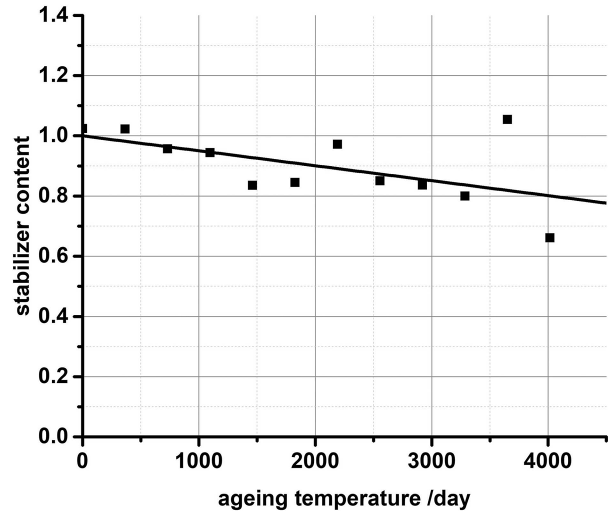

Figure 7. The stabilizer content as a function of the ageing time at room temperature.

The two FTIR test methods are different and therefore a correction factor was required. It is known that the initial stabilizer content is 1, so the initial stabilizer content was corrected to 1 and the equation corrected to the following form:

$$\alpha = -0.000050\tau + 1 \tag{8}$$

The stabilizer contents, as a fitting line of ageing time of the NEPE propellants, are shown in Figure 7.

Equation 8 is the fitting equation, which can be used to describe the stabilizer consumption in propellants aged at room temperature. At the same time, another equation playing the same role can be deduced from the stabilizer consumption equation. Substituting T = 298 K into Equation 4, the equation can be deduced as follows:

$$\alpha = -0.000054\tau + 1 \tag{9}$$

The effects of Equations 8 and 9 were consistent and a comparison thereof was made:

As seen in Figure 8, the stabilizer consumption rate at room temperature (as calculated from Equation 4) is almost the same as that from Equation 8. This suggests that the law governing stabilizer consumption is the same under both room temperature, and accelerated, ageing conditions. Thus, it can be concluded that not only are the accuracy of the two equations proven, but these two equations are also applicable to those propellants aged at room temperature. Moreover, the results also suggested that FTIR can be used to determine the stabilizer content of NEPE propellants. This approach contributes to the development of non-destructive monitoring of NEPE propellants.

3.4 Uncertainty Analysis

The tolerance of measurement of stabilizer content measured by gas chromatography is 0.01 %. In order to evaluate the uncertainty of stabilizer content in equation 4, a Kline McClintock uncertainty analysis was applied. In general, if n measurements x_n are being made, each with a measurement tolerance of ω_n, and a function F is calculated using the measured values, then the uncertainty or tolerance in the calculation can be determined as Equation 10 [33]:

$$\omega_F = \sqrt{(\frac{\partial F}{\partial x_1})^2\omega_1^2 + (\frac{\partial F}{\partial x_2})^2\omega_2^2 + \ldots + (\frac{\partial F}{\partial x_n})^2\omega_n^2} \tag{10}$$

After calculation and nondimensionalization, Figure 9 shows (ω_α/α) v. ageing time at 298 K.

It should be noted that ω_α/α is a function of ageing time and temperature. It increases with the increase of ageing time and temperature. But it can still keep high accuracy for a long period of ageing time.

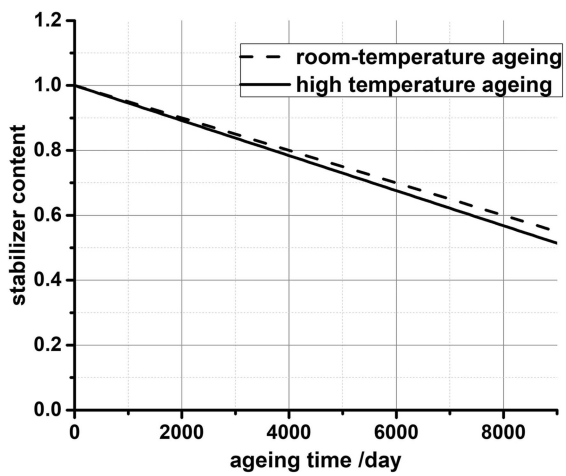

Figure 8. Comparison of the two equations.

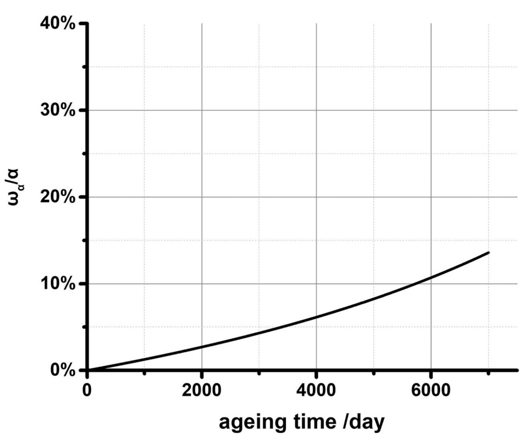

Figure 9. Uncertainty analysis of stabilizer consumption equation at room temperature.

4 Conclusion

FTIR and stabilizer content testing of NEPE propellant aged at different temperatures was carried out. By analysing the data from these tests, the following conclusions were drawn:

1) The change rule of stabilizer content of NEPE propellants aged at different temperature was revealed. The stabilizer consumption equation containing ageing temperature, stabilizer content, and ageing time was established.
2) The relationship between the stabilizer content of NEPE propellant and its infrared spectrum is revealed. The equation, which can be used to calculate the stabilizer content of NEPE propellant with its own infrared spectral data, was established.
3) The universality and accuracy of the two equations were proven by verification test. Using these two equations, the ageing time of NEPE propellant can be predicted by its infrared spectrum.
4) The feasibility of using FTIR to test the stabilizer content of NEPE propellants was proven, and this approach contributes to the development of non-destructive monitoring of NEPE propellants.

Symbols and Abbreviations

NEPE	Nitrate ester plasticised polyether
FTIR	Fourier transform infrared spectrum
α	stabilizer content
τ	ageing time
a	fitting coefficient
b	fitting coefficient
T	ageing temperature
x	second derivative absorbance value
R^2	coefficient of determination

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