Determination of Ethyl Centralite Stabilizer in a Double-Base Propellant by Gas Chromatography: Linearity, Accuracy, Precision

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Linearity, accuracy and precision of the gas chromatography determination of ethyl centralite contents in double-base propellant samples, were investigated. Different kinds of calibration curves were generated and the linear detector range for ethyl centralite was determined. By comparing of different calibration procedures the single point calibration was chosen as an optimal calibration method of the gas chromatography determination of ethyl centralite contents in double-base propellants, but some revisions related to the preparation of ethyl centralite standards and samples were needed. The accuracy of the results and the precision of the gas chromatography method were verified. This method was applied for the determination of the stabilizer contents in the double-base propellant, Plamen D, which was artificially aged until the stabilizer content becomes equal to zero.

Key words: explosive materials, double base propellant, stabilizer, ethyl centralite, gas chromatography.

Used symbols

GC	– gas chromatography;
HPLC	– high-performance liquid chromatography;
AA	– acetanilide;
FID	– flame ionization detector;
R^2	– correlation coefficient;
X_n	– content of CI in mass %, measured after "n" repeating GC injection of calibration;
X -average measured	– average measured values of the content of CI in mass % in the calibration sample;
X -actual	– known actual values of the content of CI in mass % in the calibration sample.

Introduction

ETHYL centralite, in the further text Centralite I (CI) is used for the stabilization of double-base powders and propellants. Monitoring of available stabilizers is an important element in determining safe storage life times of powders and propellants [1].

Different instrumental methods [2, 3, 4] have been applied to determine the content of the stabilizer in powders and propellants. Chromatography methods such as high-performance liquid chromatography (HPLC) and gas chromatography (GC) were used as leading methods for determining the stabilizer contents in powders propellants.

In our country, the procedure for determination of stabilizer CI contents in double-base powders and propellants by HPLC, is described in SNO 8069/91 standard [4], which is related with chemical stability of powders and propellants.

The costs of powder and propellant sample analyses by the HPLC method are 30 % higher then the costs of the same samples analyses by the GC method, because of an expensive high purity solvent acetonitrile is used as a mobile phase for HPLC. The mobile phase in GC is a cheap gas, nitrogen.

The Department for Explosive Materials Examination, has an extensive experimental experience in the Capillary Gas chromatography method determination of CI contents in double-base powders and propellants [5].

Therefore, the final aim of this work is the application of the Capillary GC method (besides the HPLC method) for monitoring CI consumption during storage and artificial ageing of double-base powders and propellants, as well as its implementation into stability surveillance programs. After examination of linearity, accuracy, and precision of the GC method, it will be used for the determination of the stabilizer, ethyl centralite, consumptions in the double-base propellant, Plamen D, artificially aged until the stabilizer content became equal to zero.

In general, an analytical instrumental method must include various validation studies but for beginning, it is usually not necessary to perform all of them. Many researchers focus on linearity, accuracy, and precision studies [6]. Results of validation studies may indicate that a change in the procedure is necessary, which may then require revalidation.

Linearity study

A linearity study verifies that the concentrations of sample solutions are in a concentration range where the detector response is linearly proportional to the concentration. When the detector sensitivity varies significantly between the components, or when sensitivity varies with the amount of a component, good quantitative results require the use of a calibration calculation for each component of interest. Prior to the analysis of samples, the standard calibration curves of the signal response of the GC instrument versus the analyte concentration are generated, by the analysis of a series of standard solutions over a concentration range appropriate for the samples to be analyzed. It can be the single-point calibration or the

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multilevel calibration procedure [9]. There is no way, other than experiment or experience, to know which one of calibration procedures is optimal for the analysis.

The method of single-point calibration requires a preparation and the analysis of only one calibration standard, which must contain known amounts of the component to be calibrated and the internal standard, too. The graph of the measured area of the analyte (examined components) versus the analyte concentration, obtained by the single level calibration method, is a straight line passing from the origin to the calibration point. The single level calibration procedure requires the linear function of the analyte peak areas and its concentrations, in the whole range of analyte concentrations. The multi-level calibration curve is used in the case when the range of the analyte concentration is wide, if a method is non-linear and for the determination of the linear range of instruments [10-12]. The multi-level calibration procedure requires a preparation and the analysis of a series of calibration standards. Each of them must contain known amounts of the component to be calibrated, in concentrations expected in samples, and the constant amounts of the internal standard as well. Then, the calibration curve is generated by the mathematical description of the experimental results. The aim of this investigation was to test suitability of the single-point calibration procedure of the Gas Chromatography determination of ethyl centralite content in double-base propellants.

Determine accuracy

The accuracy of the method is the closeness of the measured value to the true actual value of the sample [6].

Determine precision

The precision of the analytical method is the amount of scatter in the results obtained from multiple analyses of a homogeneous sample. Each of samples is independently prepared according to the method procedure. To be meaningful, the precision study must be performed using the exact sample and the standard preparation procedures that will be used in the final method [6].

Experiment

In the Military Technical Institute, different kinds of calibration curves of ethyl centralite were examined and described in [13]. This article results from [13]. Namely, after the examination of the linearity and the accuracy of the GC method [13], this method was used for the determination of the stabilizer, ethyl centralite, consumptions in samples of double-base propellant, Plamen D, artificially aged until the stabilizer content became equal to zero. The samples of the double-base propellant, Plamen D, were used for the determination of the GC method precision.

Linearity study

The concentrations of CI in the calibration standard solutions covered the whole concentration range of CI expected for the powder and the propellant samples.

Non-linear calibration curve (eight-level):
Eight calibration standard solutions [13] with known concentrations of CI were prepared (0.04 mg/mL to 1.4 mg/mL). The non-linear calibration curve is generated by the GC analysis of a series of eight calibration standard solutions. All calculations related to the sample mass of 2 g.

Linear calibration curve (eight-level):
Then the concentrations of CI in the calibration standard solutions were corrected, in order to achieve linearity. The liinear calibration curve is generated by the analysis of eight calibration standard solutions with the concentrations of CI (0.04 mg/mL to 0.7 mg/mL) [13].

The range of the CI linear detector was determined. The mass of the sample was also corrected with respect to the concentration of CI in the calibration standards and it was 1 g.

Linear calibration curve (three-level):
The linear calibration curve is generated by the analysis of three calibration standard solutions with the concentrations of CI (0.03 mg/mL to 0.5 mg/mL) in the detector linear range [13]. All calculations related to the mass of the sample, 0.7 g.

Single-point calibration

The single-point calibration was carried out using only one calibration standard with the concentration of CI (0.5 mg/mL) that was a little higher than the maximal concentration of CI expected for propellant samples [13]. It is the maximal concentration of CI in calibration standards of the three-level linear calibration curve. All calculations related to the mass of the sample, 0.7 g and 3.57 mass %CI

Determine accuracy

In order to determine the accuracy of the gas chromatography determination of CI content in double-base propellants, the calibration samples of CI with the known CI concentrations have been prepared. The same procedure for the accuracy determination was used and described in literature [13-15]. The CI concentrations in the calibration sample solutions covered the whole concentration range of CI expected for propellants samples, from relatively high initial values to low values at the end of the ageing period. Each of the calibration samples was in triplicate injected in GC from the same volumetric flask, so there were three results of the CI content measurements. The accuracy was investigated for all types of calibration procedures.

Determine precision

The precision of the GC method was investigated by measuring the CI content in the samples of the double-base propellant, Plamen D (NGR-176, PP-1), artificially aged in the thermo-block at 100ºC for the times up to the consumption of the stabilizer to the relative average CI concentration, about 2 mass %.

The precision study was performed using the same sample and standard preparation procedures that were used in the final method. It was the single-point calibration procedure with the calibration standard concentration of 0.5 mg/mL. The mass of the propellant samples was about 0.7 g.

The internal standard method was used for the CI quantitative determination using acetanilide (AA) as the internal standard. The internal standard solution was prepared by dissolving 1g of AA, weighted to an accuracy of ±0.1 mg, in 100 mL dichloromethane.

In the 50 mL volumetric flask, the calibration standard solution was prepared by weighting 0.0250 g of Centralite I (GC purity) to an accuracy of ±0.1 mg. Then, 2 mL of the internal standard solution AA was added and the content of the volumetric flask was diluted to the mark with dichloromethane. A sample of the standard solution was injected in the gas chromatograph. The injection standard solution size was 3 µL.

To determine the precision of the GC method, six of the

propellant samples, Plamen D, were independently prepared and analyzed by the same procedure described in [13-15].

The samples of propellants cut to the dimension 2 mm x 2 mm x 2 mm and put into six 50 mL volumetric flasks weighted about 0.7 g each with an accuracy of ±0.1 mg. The samples were being extracted for 48 hours with 40 mL of dichloromethane.

Then 2 mL of the internal standard solutions AA were added into each of six flasks. Finally, the content of each volumetric flask was diluted to 50 m with dichloromethane. The GC injection sample solution size was 3 µL.

Chromatographic conditions

Chromatographic conditions were the same for the analysis of the calibration standards and the calibration samples that contained the known concentration of CI as well as for the analysis of double-base propellants samples.

The CI stabilizer content was measured by the capillary gas chromatography [13] on the Hewlett Packard 5890A Model Gas Chromatograph equipped with the flame ionization detector (FID). Data were processed using the Hewlett Packard Model 3396A integrator. Nitrogen was used as a carrier gas at 20 mL·min^{-1} and hydrogen was obtained by the Hewlett Packard Model 7525 hydrogen generator. Hydrogen was used at 50 mL·min^{-1} and compressed air at 380 mL·min^{-1}. A 5 m x 0.53 mm internal diameter capillary column DB-1 with the dimethylpolysiloxane stationary phase and 2.65 µm film thickness was used. The column temperature was programmed from 100°C to 200°C at 10°C/min. The injector temperature was 200°C (split injector) and the detector temperature was 300°C. The injected standard and sample volumes were 3 µL.

GC determination of the CI content in the artificially aged samples of thePlamen D propellant

The content of CI was determined in the Plamen D propellant samples, artificially aged at 90°C for the times up until the stabilizer content became equal to zero, by the same GC procedure as the procedure described for the determination of precision.

These aging test were run on 30 g samples using pyrex tubes (150 mm long by 25 mm), capped with loosely ground glass stoppers. The entire tube was placed inside the cavity of the heating block to ensure uniformity of heating and to avoid condensation at the top of the tube.

Results and discussion

Linearity study

This GC method uses the internal standard AA, so the data are recorded as peak area ratios (ethyl centralite area/internal standard area) versus concentration ratios (ethyl centralite concentration/internal standard concentration) for the calibration standards analyzed by the method.

Non-linear (eight-level) calibration curve and accuracy

The results of the non-linear calibration are shown in Fig.1. Eight points of the calibration curve represents the results for eight calibration standards with the constant concentration of internal standard acetanilide, AA (1 mg/mL) and the increasing concentrations of CI (0.04; 0.2; 0.4; 0.6; 0.8; 1.0; 1.2 i 1.4 mg/mL).
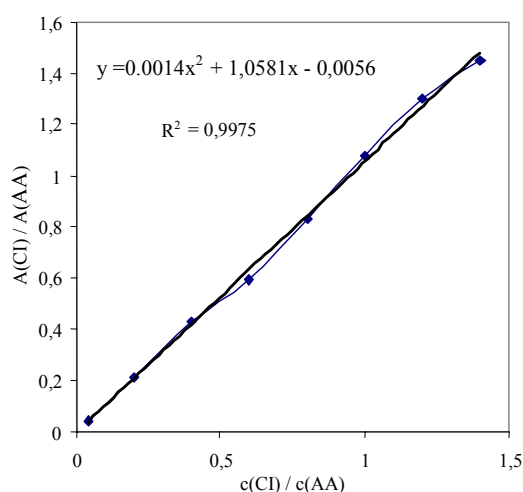

Figure 1. Non-linear eight-level calibration curve for the calibration standards with the maximum concentration of CI, 1.4 mg/mL

Table 1 presents the results of the determination of the GC method accuracy for the calibration sample with a known relative average concentration using the non-linear calibration procedure.

Table 1. Actual and measured CI concentrations in the calibration sample (non-linear eight-level calibration) and the values of relative average errors

X_1, mass% CI	1.59
X_2, mass% CI	1.45
X_3, mass% CI	1.45
Xaverage measured, mass% CI	1.50
Xactual, mass% CI	1.50
Relative error (%)	0

where: X_n – is the CI content in mass %, measured after "n" repeated GC injections of the calibration sample; *Xaverage measured* – average measured values of the CI content in mass % in the calibration sample; *Xactual* – known actual values of the CI content in mass % in the calibration sample.

In an ideal theoretical situation, an equivalent response would be obtained at each concentration of CI, and the data points would form a straight line with a zero slope. But in a practical situation, there are many reasons for the deviation from linearity.

Some common causes of non-linearity are column overload, detector overload, component degradation on the column and component adsorption on the column (an otherwise linear curve misses the origin) [17].

By analyzing of the response of the detector versus the concentration plot (Fig.1), the deviation from linearity was observed at CI concentrations higher than 0.5 mg/mL. But the correlation coeficient had a satifactory value R^2=0.9975 and the relative error (Table 1) was 0 %. However, the aim of every GC analyst is to the avoid non-linear calibration procedure. It can be used only in the case when optimal chromatographic conditions are achieved and there are no other ways to compensate for non-linearity.

Linear eight-level calibration curve (max. CI concentration, 0.7 mg/mL) and accuracy

In order to obtain better linearity of the calibration procedure, new eight CI calibration standard solutions were prepared. The CI concentrations in the calibration standard solutions covered the whole CI concentration range expected for powder and propellant samples (0.2 to 3.5 mas%), but the maximum concentration was twice lower than the previous one (0.7 mg/mL). The results of the linear

eight-level calibration are shown in Fig.2.

Eight points of the calibration curve represent the results for eight calibration standards with the constant concentration of the internal standard acetanilide, AA (1 mg/mL) and the increasing concentration of CI (0.04; 0.1; 0.2; 0.3; 0.4; 0.5; 0.6 and 0.7 mg/mL).
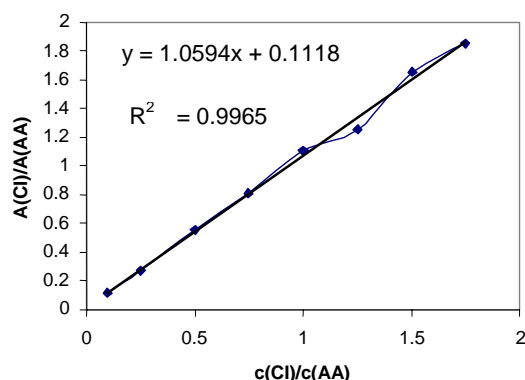

Figure 2. Linear eight-level calibration curve for the calibration standards with the maximum concentration of CI, 0.7 mg/mL

Table 2 represents the results of the GC method accuracy determination by using the linear eight-level calibration procedure in the calibration sample (mass 1g) with relatively high, average and low CI concentration.

Table 2. Actual and measured CI concentrations in the calibration sample (linear eight-level calibration) and the values of relative average errors

X_1 MASS% CI.	0.04	0.35	1.97	2.87
X_2 MASS% CI.	0.05	0.34	2.01	3.11
X_3 MASS% CI.	0.05	0.34	2.11	3.01
X AVERAGE MEASURED, MASS% CI.	0.05	0.34	2.03	3.00
X ACTUAL, MASS% CI.	0.1	0.5	2	3.00
RELATIVE ERROR (%)	95	32	1.5	0.00

By analyzing the curve (Fig.2.), the deviation from linearity was observed at CI concentrations between 0.4 mg/mL and 0.5 mg/mL. The y-intercept had a bad effect on the fit results in the lower part of the curve, so the accuracy was smaller in the samples with low and average CI concentration. Therefore, linearity was determined again, with CI concentration in the range from 0.04 mg/mL to 0.5 mg/mL.

Linear three-level calibration curve (max CI concentration 0.5 mg/mL) and accuracy

The CI concentrations were not in a wide range, so the calibration curves was generated by the analysis of a series of only three standard solutions over a concentration range appropriate for the propellants samples to be analyzed. The results of the linear three-level calibration procedure are shown in Fig.3.

The correlation coefficient is satisfactory R^2 =0.9999, so the linearity study verifies that the calibration solutions are in a concentration range where the analyte response is linearly proportional to the concentration. The linear detector range of CI was determined and it was from 0.03 mg/mL to 0.5 mg/mL.

Table 3 represents the results of the GC method accuracy determination by using the linear three-level calibration procedure in the calibration samples (mass 0.7 g) with relatively high, average and low CI concentration.
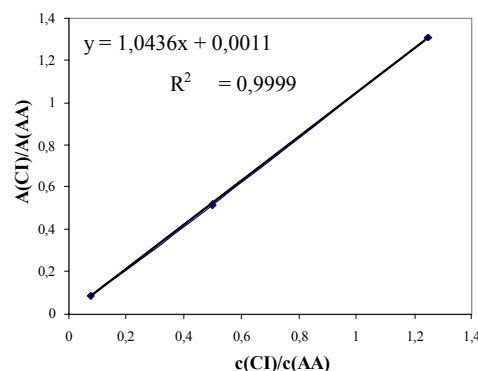

Figure 3. Linear three-level calibration curve for the calibration standards with the maximum CI concentration, 0.5 mg/mL

Table 3. Actual and measured CI concentrations in the calibration samples (mass 0.7 g) and the values of relative average errors - linear three-level calibration

X_1 mass% CI.	0.23	1.43	2.8
X_2 mass% CI.	0.22	1.45	2.91
X_3 mass% CI.	0.22	1.42	2.81
Xaverage measured, mass% CI.	0.22	1.43	2.84
X*actual*, mass% CI.	0.21	1.43	2.86
Relative error (%)	4.8	0	0.7

The actual and measured values were found to be in good agreement as verified by the calculated relative average errors.

Single-point calibration (CI concentration in the calibration standard was 0.5 mg/mL) and accuracy

One of the aims of this investigation was to test suitability of the single-point calibration for the determination of the CI content in propellants samples. The highest CI concentration calibration standard from the linear three-level calibration curve was used for the single-level calibration. The results of the single-point calibration procedure are shown in Fig.4.
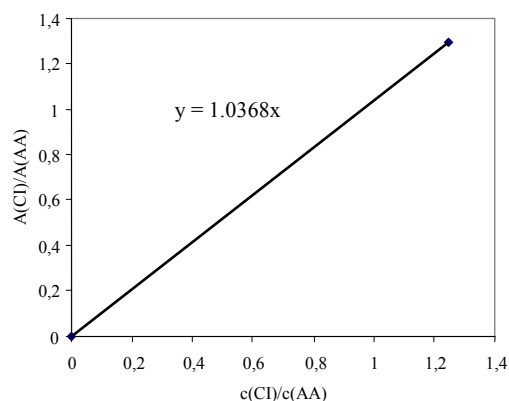

Figure 4. Single-point calibration (CI concentration in the calibration standard was 0.5 mg/mL)

The graph of the measured area of the analyte (CI) versus the concentration of the analyte (CI), obtained by the single-point calibration procedure is a straight line passing from the origin to the calibration point. The correlation coefficient (R^2) in the single point calibration procedure could not be a linearity criterion, because in this situation it had ideal values $R^2 = 1$.

Table 4 represents the results of the GC method accuracy determination by using the single-point calibration

procedure in calibration sample (mass 0.7 g) with relatively high, average and low CI concentration.

Table 4. Actual and measured CI concentrations in the calibration samples (mass 0.7g) and the values of relative average errors, single-point calibration

X_1, mass% CI.	0.23	1.44	2.8
X_2, mass% CI.	0.23	1.42	2.9
X_3, mass% CI.	0.23	1.47	2.88
Xaverage measured, mass% CI.	0.23	1.44	2.86
Xactual, mass% CI.	0.21	1.43	2.86
Relative error (%)	9.52	0	0

The actual and measured values were found to be in good agreement as verified by the calculated relative average errors which had the value of 0 mass % for high and average concentrations and the satisfactory values for low concentrations. Therefore, the accuracy of the GC results in the whole CI concentration range in double-base propellants was verified.

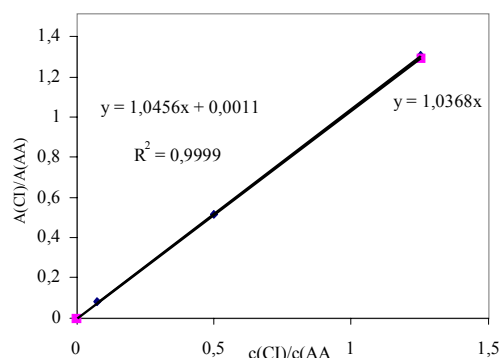The results of the single-point and the three-level calibration procedure are shown in Fig.5.

Figure 5. Three-level and single-point calibration procedures

Fig.5 shows that the single-point calibration standard is in the linear detector range. The straight line passing from the origin to the calibration point overlaps with the linear three-level calibration curve. So, the calibration standard of CI concentration, 0.5 mg/mL, could be used for quantitative determination of the CI content by the GC method in the samples of propellants.

The aim of this investigation was to test suitability of the single-point calibration of the gas chromatography determination of the ethyl centralite content in double-base propellants.

By these experimental results the single-point calibration was verified as an optimal calibration method of the GC determination of the ethyl centralite content in double-base propellants, but some revisions related to the preparation of ethyl centralite standards and samples were needed. The CI concentration in the calibration standard has to be in the linear detector range, so it was decreased and the mass of samples was decreased from 2 g to 0.7 g. Decreasing of the concentration of the calibration standard and the sample by diluting require three time higher volumes of an expensive high purity organic solvent dichloromethane and the costs of analyses by the GC method are higher.

There is a possibility to increase the experimental error by decreasing the concentration of the calibration standard and the sample by the method of aliquot and it also needs adding of a new amount of solvent.

Decreasing of the sample mass was chosen as a compromising solution because it was very simple and it was the cheapest way. The advantages of using the single-

point over the three-point calibration are shorter time of analysis, lower amounts of gasses and lower amounts of expensive solution dichloromethane, so the overall cost is smaller.

Investigation of the precision of the GC determinations of the CI contens in double-base propellants

The statistical analysis of the results of measuring the CI content in the samples of Plamen D, double-base propellant, was carried out and shown in Table 5.

Table 5. Statistical analysis of the results of measuring the CI content in Plamen D double-base propellant samples

Num. of samples	Plamen D NGR-176, PP-1	
	CI, mass%	
1	X_{11}	1.86
	X_{12}	1.84
	X_{13}	1.86
2	X_{21}	1.84
	X_{22}	1.85
	X_{23}	1.86
3	X_{31}	1.82
	X_{32}	1.81
	X_{33}	1.88
4	X_{41}	1.89
	X_{42}	1.88
	X_{43}	1.83
5	X_{51}	1.82
	X_{52}	1.88
	X_{53}	1.84
6	X_{61}	1.81
	X_{62}	1.78
	X_{63}	1.78
$\overline{X}$	1.84	
δ	0.033	
$\dfrac{t \cdot \delta}{\sqrt{n}}$	0.017	
$\overline{X} \pm \dfrac{t \cdot \delta}{\sqrt{n}}$	1.84 ± 0.017	

where: $X_{i,j}$ – is the concentration of the CI in i-th sample by j-th measuring; $\overline{X}$ - average value; δ –standard deviation, $\dfrac{t \cdot \delta}{\sqrt{n}}$ - confidence coefficient and $\overline{X} \pm t \cdot \delta / \sqrt{n}$ - confidence interval of measurements for the 95% confidence coefficient and eighteen repeated measurements where $t = 2.101$.

The statistical analysis of the results of measuring the CI contents in double-base propellants by gas chromatography verified the precision of the GC method.

GC determination of the CI content in the artificially aged samples of the Plamen D propellant

At the end of investigation, this GC method was applied for monitoring of the consumption of ethil centralite in the Plamen D double-base propellant NGR-176, PP-1 within a storage period at 90°C, for the times up until the stabilizer content became equal to zero. The results are shown in Fig.5. and can be used for determining a safe storage life of propellants.

Further investigations aim at the verification of the GC method, through parallel GC and HPLC determinations of the ethil centralite content in aged double-base powders and propellants.

Conclusion

− Eight calibration standard solutions with the known CI concentrations were prepared;

- Multi-level (non-linear and linear) and single-point calibration curves were generated;
- Linear detector range was determined for CI (0.03 do 0.5 mg/mL);
- By comparing of different calibration curves of ethyl centralite, the single-point calibration was chosen as an optimal calibration method of the GC determination of ethyl centralite content in double base propellants, but some revisions related to the preparation of ethyl centralite standards and samples, were needed.
- The calibration standard of the CI concentration, 0.5 mg/mL, was used for quantitative determination of the CI content by the GC method in the propellant samples; The CI content in that calibration standard is about 3.6 mass%, calculated for a mass of 0.7 g of the examined propellant samples. It was a value higher than the highest expected value of the CI content in double base powder and propellants.
- Masses of the propellant samples were decreased from 2 g to 0.7 g;
- The GC analysis of the calculated relative average errors values of the determination of the ethyl centralite content in the calibration samples verified the accuracy of the results in the whole concentration range of ethyl centralite in double-base powders and propellants.
- The statistical analysis of the results of measuring the ethyl centralite content in the Plamen D double-base propellants samples with a relatively average concentration of ethyl centralite verified the precision of the GC method.
- This GC method is fast, linear, accurate and precise; therefore, this instrumental method can be used for the determination of the CI content and for monitoring the consumption of ethyl centralite during storage and artificial ageing of double-base powders and propellants.

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Gasno-hromatografsko određivanje sadržaja stabilizatora etil centralita u dvobaznom raketnom gorivu – linearnost, tačnost, preciznost

Istraživane su linearnost, tačnost i preciznost gasno-hromatografskog određivanja sadržaja etil centralita u uzorcima dvobaznog raketnog goriva. Generisane su različite vrste kalibracionih krivih i određen je linearni opseg rada detektora za etil centralit. Poređenjem različitih kalibracionih postupaka, kalibracija kroz jednu tačku je izabrana kao optimalan kalibracioni postupak gasno-hromatografskog određivanja sadržaja etil centralita u dvobaznim raketnim gorivima, ali potrebne su neke izmene u pripremi kalibracionog standarda etilcentralita i ispitivanih uzoraka. Verifikovane su tačnost rezultata i preciznost gasno-hromatske metode.
Gasno-hromatografska metoda je primenjena za određivanje sadržaja stabilizatora, u uzorcima dvobaznog raketnog goriva, Plamen D, ubrzano starenog do potpunog utroška stabilizatora.

Ključne reči: eksplozivni materijali, dvobazno raketno gorivo, stabilizator, etil centralit, gasna hromatografija.

Détermination du contenu de stabilisateur de l'éthyle centralite dans le propergol bibasique par la chromatographie à gaz - linéarité, exactitude, précision

Linéarité, exactitude et précision de la détermination par la chromatographie à gaz du contenu de l'éthyle centralite dans les échantillons du propergol bibasique ont été examinés. On a réalisé différents types des courbes de calibrage et déterminé la portée linéaire du détecteur pour l'éthyle centalite. Les différents procédés de calibrage ont été comparés et on a choisi le calibrage à travers un point comme le procédé optimal dans la détermination citée. Cette détermination est faite par la chromatographie à gaz, mais certains changements dans la préparation de la norme de calibrage de l'éthyle centralite et des échantillons examinés sont nécessaires. L'exactitude et la précision de la méthode chromatographique à gaz ont été vérifiées. Cette méthode est appliquée pour la détermination du contenu du stabilisateur dans les échantillons du propergol bibasique, Plamen D, vieilli artificiellement jusqu'à ce que le contenu du stabilisateur devienne égal à zéro.

*Mots clés***: matières explosives, propergol bibasique, stabilisateur, éthyle centralite, chromatographie à gaz.**