

PRELIMINARY SPECTROPHOTOMETRIC DETERMINATION OF THE DYE CONTENT IN ORANGE SMOKE GRENADES

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Abstract

A protocol for determination of the content of dye in a mixture for orange smoke grenades was proposed. The protocol was based on spectrophotometric determination of the dye 1,4-dihydroxyanthraquinone in DMSO solutions. The Beer's law is obeyed in the concentration range of $(0.5 - 1.2) \times 10^{-5}$ mol/l dye with molar absorptivity 7.3×10^3 L/mol.cm. The protocol is very fast and show good accuracy (recovery 98.6 - 103.6 %) and precision (RSD 3.8%). The protocol was applied in model and real samples.

Keywords: 1,4-dihydroxyanthraquinone, spectrophotometry, smoke grenades, dye determination

Introduction

Smoke grenades are used for several purposes. The primary use is the creation of smoke screens for camouflage and the signalling of aircraft. Various colors smoke grenades are produced (red, yellow, green, blue, orange, white) and all use very brightly colored dyes. The main components of a colored smoke composition are dye, oxidant and sugars. The potassium chlorate is used as an oxidant. The mixtures are usually produced by mixing exactly weight components. However, to respond to the modern demands of quality control a determination of actual composition of the mixture should be applied [1]. It imposes a development of protocols for determination of the components of the smoke grenades mixtures. The methods reported in the literature are mainly based on gas chromatography or HPLC [2-6]. However, they used expensive instruments and needs qualified personal. The dye used to produce orange smoke grenades in a Bulgarian factory is 1,4-Dihydroxyanthraquinone. A low cost, easy to implement and with good accuracy and precision method is needed. The dye absorbs in the visible region and spectrophotometric method could be studied if appropriate solvent is found [4].

This study presents the results of development of a protocol for determination of dye content in orange smoke grenades by spectrophotometry.

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Materials and Methods

Reagents and instrumentation

Orange dye (1,4-dihydroxyanthraquinone, trade name Solvant Orange 86) was used as obtained. Sugar, KClO_3 , acetone and dimethyl sulfoxide (DMSO) were at p.a. grade. The spectra were acquired by spectrophotometer Carry 100 Varian.

Methods

Preparation of model mixtures: Exactly weighed quantities of orange dye, KClO_3 and powdered sugar were thoroughly mixed to obtain the different compositions containing from 46 to 54 % dye. KClO_3 and sugar were added to the mixture in a constant ratio 1:1. The mixture was dissolved in DMSO to obtain a solution with a concentration of the dye 3×10^{-3} mol/L. The working solutions were prepared daily by dilution with DMSO. The spectra were acquired at room temperature and DMSO was used as a reference.

Results and Discussion

Solubility of the studied dye 1,4-dihydroxyanthraquinone in acetone and DMSO was studied. The both solvents dissolved completely the dye in the studied concentration range. Due to good solubility, stable solutions, lower volatility and lower toxicity DMSO was chosen for further studies. The spectra of model mixtures in DMSO are presented in Fig. 1.

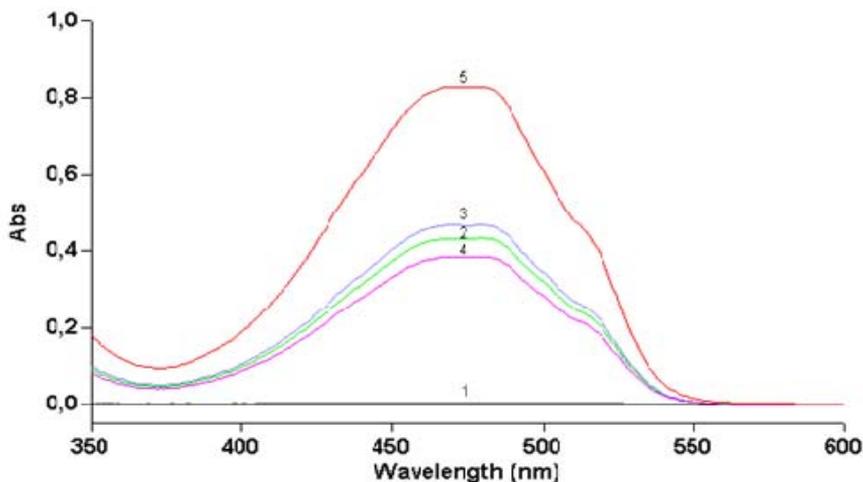


Fig. 1. UV-Vis spectra of: (1) sugar + KClO_3 in DMSO; (2) sugar + dye in DMSO; (3) KClO_3 + dye in DMSO; (4) dye + sugar + KClO_3 (the concentration of colored component in the solutions (2) - (4) is 3×10^{-6} mol/L; (5) Dye in DMSO

As it can be seen from Figure 1, the 1,4-dihydroxyanthraquinone absorbed photons at $\lambda_{\text{max}} = 475 \text{ nm}$ (curve 5). The sugar and KClO_3 don't absorb photons in the studied spectral region (curve 1). The matrix components didn't influence the absorbance of the dye (curves 2, 3 and 4). The absorbance at 475 nm depended on the concentration of 1,4-dihydroxyanthraquinone in the solution (curve 3 and 5). The Beer's law was obeyed in the studied concentration range $(0.5-1.2) \times 10^{-5}$ mol/L of dye in DMSO. The equation of linear curve

was obtained by least squares method: $A = -0.009 + 7.3 \times 10^3 \times C$ with regression coefficient $r = 0.9979$ ($n = 5$). The molar absorptivity of the studied dye was 7.3×10^3 L/mol.cm at $\lambda_{\max} = 475$ nm.

The following protocol for the determination of 1,4-dihydroxyanthraquinone in smoke grenade mixtures was developed: calibration by one point external standard method with standard mixture containing 50 % dye, 25 % KClO_3 and 25% sugar. From this mixture a stock solution with 3×10^{-3} mol/L of the dye in DMSO was prepared. Diluted solution (5×10^{-5} mol/L) was used for absorbance measuring and calculation of coefficient of the analytical function. The solution of the real sample was prepared with the concentration of the dye around 5×10^{-5} mol/L in DMSO. The absorbance of both solutions was measured at 475 nm.

The accuracy and precision of the developed protocol for spectrophotometric determination of the orange dye were studied in model mixtures containing varied concentrations of the dye from 46% to 54% and KClO_3 and sugar in 1:1 ratio. The results are presented in Table 1.

Table 1. Results from determination of dye content in model mixtures for orange smoke grenades using the proposed protocol

Dye percentage in model mixtures	obtained percentage*	recovery, %
45.95 %	46.30 ± 1.2 (n=3)	100.7
47.93 %	49.61 (n=2)	103.5
50.00 %	49.99 ± 3.8 (n=6)	100.0
51.88 %	52.02 (n=2)	101.2
53.85 %	53.04 (n=1)	98.5

* Number of samples is presented in the brackets

The developed protocol was applied for determination of orange dye (Solvent Orange 86) content in a real sample of mixture for smoke grenades. The following results are obtained (50.0 ± 3.8 %) ($n = 6$, $P = 95\%$).

Conclusion

A protocol for determination of the content of dye in a mixture for orange smoke grenades was proposed. The protocol was based on spectrophotometric determination of the 1,4-dihydroxyanthraquinone in DMSO solutions. The protocol is very fast and show good accuracy (recovery between 98.6 and 103.6 %) and precision (RSD 3.8%). It could be applied for determination of other dyes in smoke grenades mixtures and the study is currently in the progress.

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