ASSAY OF PBX 9501

C. S. MacDougall

QUALITY DIVISION

MASTER

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Normal Process Development
Endeavor No. 105

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SUMMARY

An infrared spectrophotometric method is used for determining Estane in PBX 9501. In this method a 1 g sample of 9501 is extracted with 1,2-dichloroethane. The mixture is mechanically shaken for one hour, centrifuged, and an aliquot of the supernatant is transferred to a 0.50 mm NaCl cell. The IR spectrum at 1717 cm^{-1} is recorded, and the percent Estane calculated from the measured peak height. The HMX remaining from the dichloroethane extraction is determined gravimetrically, and the nitroplasticizer is calculated by difference.

INTRODUCTION

The planned utilization of PBX 9501 in weapons systems necessitated the development of assay capabilities for HMX, Estane and the nitroplasticizer in this material. A standard IR spectrophotometric determination is used in the Estane determination. The HMX is determined by a gravimetric method. The nitroplasticizer is calculated by difference. This report concerns the transfer of technology to the Quality Chemical Laboratory and describes the accuracy and precision of the methods.

EXPERIMENTAL

APPARATUS

Spectrophotometric analyses are performed using a Perkin-Elmer 521 Grating Infrared Spectrophotometer. Other instruments of equal competence may be used. Estane standards are prepared by dissolving purified stock Estane in 1,2-dichloroethane.

PROCEDURE

Estane

A 1 g sample of explosive is weighed into a 125 mL Erlenmeyer. Twenty mL of 1,2-dichloroethane are added, and the mixture is mechanically agitated for one hour. After the Estane is extracted, the mixture is centrifuged. The supernatant is then measured spectrophotometrically at a wave length of 1717 cm^{-1} against a reference containing 15 mg Estane in 20 mL of 1,2-dichloroethane. The height of the peak from the sample is compared with those peaks produced by standards, and the composition of the explosive is calculated from the measured Estane.

The calibration curve is prepared with purified stock Estane solutions in 1,2-dichloroethane. Fig. 1 shows a typical calibration curve. Beers Law is obeyed over the concentration interval measured. Settings of the spectrophotometer are as follows:

Scan Drive	10
Scan Time	4
Suppression	0

Fig. 1. Standardization of Estane

Expansion	5X
Attenuator Speed	1100
Slit Program	1000
Gain	3.5
Source Current	0.82 amps
Scale Position	Variable, set median concentrated standards near A = 0,5
Cell Thickness	0.50

A test sample of PBX 9501 was repeatedly analyzed for the Estane (Table I). The precision is excellent, with a relative standard deviation of 0.4 percent. Unfortunately, an assay on the test material was not available, but as will be shown later in this paper, the results are well within the specifications of the PBX 9501.

HMX

The HMX from the centrifugate of the Estane extraction is washed onto a glass-fritted crucible with 1,2-dichloroethane saturated with HMX, and the residue is weighed.

The test 9501 sample was repeatedly analyzed for HMX (Table II). A relative standard deviation of 0.13 percent is obtained on the HMX determination. Table III shows a comparison between an experimental assay of the test 9501 and the theoretical composition as determined by the specifications. As can be seen, all measurements compare very well with the specifications, indicating good accuracy of the methods.

CONCLUSIONS

The assay methods for PBX 9501 have been adapted to operations in the Quality Chemical Laboratories and are ready for qualification of the material as received by Pantex.

ACKNOWLEDGEMENT

The author wishes to thank L. M. Boyles for performing the analyses listed in this report.

Table I. Analysis of PBX 9501 for Estane

<u>Run Number</u>	<u>Estane (%)</u>
1	2.43
2	2.44
3	2.44
4	2.45
5	2.46
6	2.45
7	2.45
8	2.46
9	2.46
10	2.46
11	2.43
12	2.43
13	2.46
14	2.46
Average	2.45 $\pm$ 0.01

Table II. Analysis of PBX 9501 for HMX

<u>Run Number</u>	<u>HMX (%)</u>
1	95.04
2	95.15
3	94.80
4	94.97
5	94.89
6	95.11
7	94.93
8	94.94
9	94.83
10	94.71
11	94.90
12	94.71
13	94.74
14	94.77
15	94.79
16	94.93
17	94.91
18	94.75
19	94.92
Average	94.88 $\pm$ 0.12

Table III. Composition of PBX 9501

<u>Component</u>	<u>Measured</u>	<u>Theoretical</u>
HMX	94.88	95.0 ± 0.3
Estane	2.45	-
Nitroplasticizer	2.67	-
Estane/Nitroplasticizer	0.92	1.00 ± 0.10