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**Compositional Analysis of PBX 9503 -
A TATB/HMX/Kel-F 800 Formulation**

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MASTER

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**Process Development
Endeavor No. 205**

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A compositional analysis technique for PBX 9503, a TATB/HMX/Kel-F 800 formulation, was developed. Liquid chromatographic HMX analyses were made on a LiChrosorb SI-60 column (25 cm) using tetrahydrofuran as the mobile phase and a UV detector for quantitation. Kel-F 800 was separated from the HMX and TATB by nitric acid reflux. The TATB content was obtained by a gravimetric method.

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COMPOSITIONAL ANALYSIS OF PBX 9503 -
A TATB/HMX/KEL-F 800 FORMULATION

INTRODUCTION

This report describes a method for the analysis of PBX 9503 of the approximate composition 80% TATB, 15% HMX and 5% Kel-F 800. PBX 9503 contains 0.2% dye which was not analyzed in this study but is included in the TATB percentage.

EXPERIMENTAL

In each of four 30 ml volume bottles was accurately weighed a 1-g sample of PBX 9503 molding powder, Lot No. 0024-185-01. Approximately 25 ml of cyclohexanone was added and the bottles were sealed with Teflon-lined screw caps. The sealed bottles were placed in a shaker and agitated vigorously for one hour. The bottles and contents were then centrifuged for 10 minutes. The contents were transferred onto a 5 μ m Teflon filter disc using a Millipore Pyrex filter holder. The filter disc, a petri dish, and cover had previously been weighed. Acetone was used to complete the TATB transfer from the bottles. The TATB was washed with approximately 15 ml of acetone, then the filter holder top was removed and TATB washed from the top onto the filter disc while applying vacuum. The residue was then air-dried. The TATB and filter disc were transferred into the small petri dish, covered and placed in a 100 C vacuum oven for one hour. The petri dish, filter disc, contents and cover were then removed from the oven and placed in a desiccator to cool to ambient temperature before weighing. The TATB content was determined from the TATB remaining on the filter (Table I). A sample of TATB only was extracted and the recovery is shown in Table II.

Table I. Analysis of PBX 9503, Lot No. 0024-185-01

<u>Sample No.</u>	<u>TATB (%)</u>	<u>HMX (%)</u>	<u>Kel-F 800 (%)</u>
I	80.38	14.46	5.01
II	80.50	14.41	5.09
III	80.38	14.33	5.15
IV	<u>80.44</u>	<u>14.44</u>	<u>5.05</u>
Average ^a	80.42 $\pm$ 0.09	14.41 $\pm$ 0.09	5.08 $\pm$ 0.09

^aThe averages are expressed as 95% confidence intervals.

Table II. Analysis of TATB Sample

<u>Sample No.</u>	<u>TATB (%)</u>
I	99.96
II	99.95
III	99.97
IV	<u>99.99</u>
Average	99.96 $\pm$ 0.03

The Kel-F was determined by the following procedure. In each of four 250 ml flasks, 2 g samples of PBX 9503 were weighed. The weights of sample and flask were recorded. One hundred ml of 70% HNO₃ was added to each flask fitted with a condenser. Using a hot plate, the HNO₃ was refluxed for four hours. A pre-cleaned and dried 60-ml medium fritted filter was weighed for each sample. The Kel-F that remained in the flasks was filtered. Each flask was rinsed with 25 ml of 60 C nitric acid and the acid filtered. Each flask was washed with 25 ml of distilled water and the water filtered. The flasks, filters and contents were dried in a 100 C vacuum oven for approximately 16 hours. The Kel-F is softened, thus allowing the acid and water to be removed by vacuum. The flasks and filters were then placed in a desiccator to cool to ambient temperature. The flask and filters were weighed and the Kel-F percentage determined as shown in Table I. A sample of pure Kel-F only was analyzed using the above procedure and these results are given in Table III.

Table III. Analysis of Kel-F 800 Sample

<u>Sample No.</u>	<u>Kel-F 800 (%)</u>
I	100.5
II	100.1
III	100.2
IV	<u>99.81</u>
Average	100.17 $\pm$ 0.13

HMX was determined by liquid chromatography using the following conditions. A liquid chromatograph fitted with a LiChrosorb SI-60 (25 cm length and 4.6 mm inner diameter) column and a carrier of THF were used. The peaks were detected using a 254 nm UV detector and areas integrated. Fig. 1 shows a typical chromatograph used in determining the HMX percentage. Note that Kel-F 800 is not detected at 254 nm.

Fig. 1. Typical Chromatogram

Samples (1 g) of PBX 9503 were weighed in each of four 125-ml flasks. One hundred ml of THF were pipetted into each flask and the sample dispersed in an ultrasonic bath for approximately one minute. An accurate reference standard was prepared in a 125-ml flask using approximately the same amount of HMX used in the PBX 9503 and 100 ml of THF. The samples were injected through a prefilter to remove TATB solids. The percentages calculated from the integrated peaks are shown in Table I.

DISCUSSION

Nine lots of PBX 9503 were analyzed by this procedure and 95% confidence intervals were calculated for the three components in each lot. For TATB the interval widths ranged from 0.16 to 0.40% with an average width of 0.22%. The HMX interval widths ranged from 0.08 to 0.64% with an average width of 0.34% and the Kel-F 800 interval widths ranged from 0.10 to 0.60% with an average width of 0.32%.

The Kel-F 800 and HMX analyses are very accurate, but the TATB contents have built-in errors. The impurities found in the various lots of TATB are extracted by solvents. The solvent cyclohexanone selected to extract the HMX and Kel-F 800 from the TATB will also extract some of the impurities. The cyclohexanone cannot be saturated with the impurities due to the unavailability of the impurities. If the various impurities were available, the solubility would vary depending on the impurities present in the TATB.

A second source of error is filtration of TATB. Particles less than 5 m pass through the filter, thus lowering the TATB content. Even with the built-in errors on the TATB, the closure for the nine lots was less than 0.5%.

The HMX method by liquid chromatography is advantageous in determining the homogeneity of the molding powder granules by varying the sample size.

CONCLUSION

A technique was developed for the compositional analysis of PBX 9503. The HMX analysis was done by liquid chromatograph. A gravimetric method was used to obtain the Kel-F and TATB analysis.

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