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Compositional Analysis Technique for HNS/Kel-F 800

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Process Development
Endeavor 101

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COMPOSITIONAL ANALYSIS TECHNIQUE
FOR HNS/KEL-F 800

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ABSTRACT

A compositional analysis procedure for the plastic-bonded explosive consisting of HNS and Kel-F 800 is presented. The Kel-F is determined gravimetrically after extraction of the HNS with fuming nitric acid. The HNS content is calculated by difference.

INTRODUCTION

The described method has been employed successfully in the evaluation of HNS/Kel-F 800 compositions wherein the Kel-F content was ≤ 7 percent.

Approximately 1.5 g of the explosive mixtures is introduced into a weighed Erlenmeyer flask with stirring bar and covered with 100 ml of fuming nitric acid. The contents are digested over a boiling water bath for 90 minutes and suction filtered over a medium porosity crucible. The glassware and bar are given 3 successive rinses in water and hot DMF before drying under vacuum in a steam oven for 2 hours at 100 - 105 C.

The gain in weight of the glassware and bar is attributed to Kel-F, which is insoluble in nitric acid. The Kel-F content is established by

dividing this weight by the sample weight. The HNS content is determined by difference.

EXPERIMENTAL

Materials and Equipment

Analytical Balance, Ainsworth,
Type 24N
Sintered Glass Crucible,
Medium Porosity, 60 ml
Capacity
Vacuum Oven
Suction Filter Equipment
Erlenmeyer Flask, 250 ml
Condenser, Liebig
Anti-bump Magnetic Bar
Hot Plate; Water Bath
Nitric Acid (Fuming)
DMF, Reagent Grade
HNS, Lot No. 7312-07C-003
Kel-F 800, Lot No. 557

PROCEDURES

The digestion and analysis of HNS/Kel-F compositions are promoted in thoroughly cleaned, dried, and accurately weighed utensils. These may be stored in a desiccator until needed.

Weigh the Erlenmeyer flask (250 ml), the sintered glass crucible, medium porosity, 60 ml capacity, and stirring bar to the nearest 0.0001 g.

Into the Erlenmeyer flask introduce approximately 1.5 g weighed to the nearest 0.0001 g of the HNS/Kel-F composition. Position the flask over a water bath within an exhaust hood, drop the stirring bar in the flask and cover the contents with 100 ml of fuming nitric acid. Set the condenser in place and digest the mixture over the boiling water bath for 90 minutes.

Remove the condenser and carefully suction filter the flask contents over the previously tared sintered glass crucible. Move the crucible over an auxiliary suction flask, free of nitric acid, and rinse the crucible, flask and bar with distilled water. Follow this with a 90 ml hot DMF rinse and a final distilled water rinse.

Dry the glassware and bar in an evacuated steam oven for 2 hours at 100 to 105 C. Remove the pieces and cool in a desiccator to ambient. Reweigh the cooled flask, crucible, and bar to the nearest 0.0001 g. The Kel-F content is calculated from the combined gain in weight of the three items using the following calculation:

$$\% \text{ Kel-F} = \frac{\text{Gain in Weight}}{\text{Sample Weight}} \times 100$$

The HNS percentage composition is determined by difference.

RESULTS AND DISCUSSIONS

The literature states that polychlorotrifluoroethylene (Kel-F) is

"...unaffected by bases and acids (including fuming oxidizing agents) over the complete range of concentration and temperatures."

To confirm the effect of fuming nitric acid on Kel-F during the analysis procedure 4 Kel-F samples were digested in 100 ml of fuming nitric acid at 90 C for 90 minutes (see Table I). In each case some swelling was observed along with a slight gain (0.0325 percent) in weight. This change in mass is too insignificant to warrant correcting the Kel-F values obtained during the analysis of HNS/Kel-F compositions.

Table I. Digestion of Kel-F in Fuming Nitric Acid (Kel-F 800, Lot No. 557)

Sample	Kel-F Actual (g)	Kel-F Recovered (g)	Recovery (%)
1	0.5521	0.5522	100.02
2	0.5065	0.5069	100.08
3	0.9948	0.9950	100.02
4	0.9969	0.9970	100.01
Mean	-	-	100.03
Standard Deviation	-	-	0.03

Eight HNS/Kel-F compositions, with a Kel-F content ranging from 4.0 to 7.4 percent, were carefully formulated and subsequently analyzed by the described procedure. The compositions and the corresponding analyses are tabulated in Table II. The HNS percentage compositions were established by difference. These values are listed in Table III.

Table II. Kel-F Recovery from Known HNS/Kel-F Mixtures by Extraction of the HNS With Nitric Acid

<u>Sample Size (g)</u>	<u>Kel-F Actual (g)</u>	<u>Kel-F Found (g)</u>	<u>Deviation (g)</u>	<u>Kel-F Actual (%)</u>	<u>Kel-F Found (%)</u>	<u>Kel-F Recovery (%)</u>
1.6212	0.0761	0.0780	+0.0019	4.69	4.81	102.49
1.6203	0.0662	0.0673	+0.0011	4.09	4.15	101.66
1.5070	0.0731	0.0742	+0.0011	4.85	4.92	101.50
1.5127	0.0898	0.0935	+0.0037	5.94	6.18	104.12
1.5179	0.0911	0.0921	+0.0010	6.00	6.07	101.10
1.5045	0.0924	0.0938	+0.0014	6.14	6.23	101.51
1.5174	0.1124	0.1154	+0.0030	7.41	7.60	102.67
1.4984	0.1063	0.1048	-0.0015	7.09	6.99	98.59
Mean	-	-	-0.0019	-	-	-
Standard Deviation	-	-	0.0009	-	-	-

Table III. HNS Recovery (By Difference) from Known HNS/Kel-F Mixtures Through Nitric Acid Extraction

<u>Sample Size (g)</u>	<u>Kel-F Recovered (g)</u>	<u>HNS Actual (g)</u>	<u>HNS Found (g)*</u>	<u>Deviation (g)</u>	<u>HNS Recovery (%)</u>
1.6212	0.0780	1.5451	1.5432	0.0019	99.87
1.6203	0.0673	1.5541	1.5520	0.0011	99.82
1.5070	0.0742	1.4339	1.4328	0.0011	99.92
1.5127	0.0935	1.4229	1.4192	0.0037	99.74
1.5179	0.0921	1.4268	1.4258	0.0010	99.92
1.5045	0.0938	1.4121	1.4107	0.0014	99.90
1.5174	0.1154	1.4050	1.4020	0.0030	99.78
1.4984	0.1048	1.3921	1.3938	0.0017	100.12
Mean	-	-	-	0.0019	99.87
Standard Deviation	-	-	-	0.0009	-

*By difference

Two LX-15 formulations (Lot Nos. 0128-170-02 and 0129-170-01) were analyzed. The Kel-F percentage content found, along with the com-

position of the two formulations are listed in Table IV. The HNS content, established by difference, are listed in Table V.

Table IV. Analysis of HNS/Kel-F Formulations - Kel-F Content Established Through Nitric Acid Extraction of the HNS

Sample	Sample Size (g)	Kel-F Formulated (g)	Kel-F Found (g)	Deviation (g)	Kel-F Recovery (%)
1*	1.5140	0.1056	0.1060	+0.0004	100.38
2*	1.5007	0.1047	0.1051	+0.0004	100.38
1**	1.5251	0.0784	0.0785	+0.0001	100.13
2**	1.4989	0.0770	0.0758	-0.0012	98.44
3**	1.4984	0.0770	0.0763	-0.0007	99.09
4**	1.4985	0.0770	0.0759	-0.0011	99.51
Mean	-	-	-	0.0006	99.65
Standard Deviation	-	-	-	0.0004	-

*Kel-F/HNS Lot No. 0128-170-02; % Composition - Kel-F 6.98, HNS 93.02

**Kel-F/HNS Lot No. 0129-170-01; % Composition - Kel-F 5.14, HNS 94.86

Table V. Analysis of HNS/Kel-F Formulations - HNS Content (By Difference) Established Through Nitric Acid Extraction

Sample	Sample Size (g)	HNS Formulated (g)	Kel-F Found (g)	HNS Difference (g)	HNS Deviation (g)	HNS Recovery (%)
1*	1.5140	1.4083	0.1060	1.4080	0.0003	99.97
2*	1.5007	1.3959	0.1051	1.3956	0.0003	99.97
1**	1.5251	1.4467	0.0785	1.4466	0.0001	99.99
2**	1.4989	1.4218	0.0758	1.4231	0.0013	100.09
3**	1.4984	1.4214	0.0763	1.4221	0.0007	100.05
4**	1.4985	1.4214	0.0759	1.4226	0.0012	100.08
Mean	-	-	-	-	0.0005	100.03
Standard Deviation	-	-	-	-	0.0006	-

*Kel-F/HNS Lot No. 0128-170-02; % Composition - Kel-F 6.98, HNS 93.02

**Kel-F/HNS Lot No. 0129-170-01; % Composition - Kel-F 5.14, HNS 94.86

The gravimetric analysis of HNS/Kel-F compositions by extraction of the HNS with hot fuming nitric acid gives analytical values which agree with the quantitative compositions of the formulations. This analytical method is superior to previously employed analytical procedures where-in the Kel-F was extracted with boiling organic solvents namely n-

butyl acetate and methyl-iso-butyl ketone.

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