MASTER

FEFO DEGRADATION IN LX-09

S. T. Switzer

QUALITY DIVISION

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ABSTRACT

A study of FEFO degradation in several 1969 neat FEFO library samples and FEFO extracted from library LX-09 lots prepared using these samples was made by gas chromatography.

INTRODUCTION

FEFO is the liquid explosive in LX-09 HE. It is therefore important to study its degradation patterns after prolonged contact with the HMX and DPNPA components of LX-09. It is also necessary to search for neat FEFO break-up trends in library samples. Then, by comparing these two sets of data, it can be determined whether FEFO spontaneously degrades over the years or whether its breakup is noticeable only after the prolonged contact with other LX-09 components. This study found the latter case to be true.

EXPERIMENTAL

APPARATUS

All chromatograms were determined by FID on a Perkin Elmer 3920 chromatograph, using matched 1/8-inch x 1 m columns packed with OV3. Helium carrier was employed at a flow rate of 35 cc/min. Area percents for all peaks were determined by computer.

SAMPLE PREPARATION

Neat FEFO/LX-09 library samples were selected such that matched sets by FEFO raw material lot were available. Neat FEFO samples were purified by distillation (to remove most of the ethyl acetate used in packaging) before chromatograms were determined.

FEFO was extracted from powdered LX-09 samples by shaking 500 mg of each lot with 10 mls ether on an automatic shaker for 1-1/2 hours. Each sample was then filtered through a medium porosity fritted glass crucible and the filtrate analyzed by GC for FEFO components(1).

Duplicate extractions of each LX-09 lot showed reproducibility within 2% pure FEFO.

PROCEDURE

A temperature program of 40 to 230 C at 16°/min, with the oven remaining at 40 C for four minutes (to allow time for the ether peak to elude) and 230 C for up to 30 minutes, was used in each of the 20 to 30 μ l injections of LX-09 extracted FEFO.

(1) C. S. MacDougall, *Component Assay of LX-09*, MHSMP-76-17U (January-March 1976).

The 0.15 μ l samples of neat FEFO were injected for each run, using a temperature program of 80 to 230 C at 16°/min, with the oven remaining at 230 C for eight minutes.

Additional testing of selected neat FEFO lots using the 40 to 230 C program showed no significant difference from values obtained in the 80 to 230 C program.

Several chromatograms were run on each neat FEFO lot and LX-09 extraction to determine averages and show reproducibility within 0.5 and 1% pure FEFO, respectively.

The ethyl acetate used in packaging neat FEFO for shipment and the ether peak (resulting from ether used for extractions) in LX-09 extracted samples were disregarded.

DISCUSSION

All LX-09 extracted FEFO was prepared following the standard method outlined in this report, with one attempted exception; LX-09 pellets, rather than powder, were used for the first set of extractions. However, those chromatograms showed an unusually large high boiler percentage, implying that these impurities were selectively extracted while the pure FEFO and low boiling impurities were not. Once this was established, pre-ground LX-09 was used for all extractions.

Tables I, II, and III show individual studies on percents FEFO, high boiling impurities, and low boiling impurities, respectively, for each of the library neat and library LX-09 extracted FEFO samples. Original (1969) acceptance values for neat FEFO samples are also listed as reference guides.

Library neat samples show no marked change from original FEFO lots, although each library neat percent FEFO is slightly lower than, and every library neat percent low boiler is slightly higher than the original. Changes in percent high boiler fluctuate slightly in both directions. However, none of these three components show a difference of more than one percentage point from the original analysis.

Percentages for library LX-09 extracted FEFO components compared with library neat sample values show more noticeable changes, varying from 0.15 to 14.66 points difference. LX-09 extracted FEFO low boiling components and pure FEFO percentages, especially, exhibit marked deviation from library neat sample values, with a tendency of the former to increase while the latter decreases. High boiler values for lots 941-1 and 700-3 are unusually large due to the presence of one high boiler each, appearing after much longer elution times than normally experienced.

Table IV shows the samples grouped into three categories, based on their changes in component percentages. Component percentages for LX-09 extracted FEFO compared with library neat FEFO in the first grouping are within experimental error of being the same.

The second grouping shows moderate differences in the two sets of values. This is most noticeable in both the increase in LX-09 extracted FEFO's low boiling impurities and the decrease in pure FEFO from library neat FEFO percentages.

Percentage differences in the last grouping are very large, showing the same increase in low boiling impurities as pure FEFO percentages decrease. The high boiling impurities increase is due to the presence of a new, very large high boiler in each of the LX-09 extracted samples.

Lots within each category have basically the same shape chromatograph, distinctly different from the other two.

CONCLUSION

Based on the very small changes of library neat FEFO samples component percentages as compared with original acceptance data, it can be assumed that there is minimal break-down of neat FEFO over the seven year period considered.

FEFO extracted from library LX-09 lots, however, shows a varying degree of break-down, with the pure FEFO peak generally decreasing as the low boiling impurities increase. This implies FEFO fragmentation into smaller, and consequently, lower boiling components. Reasons for this severe fragmentation diversity have not yet been determined, but may be due to such factors as storage conditions, FEFO association with different impurities in different lots, and/or the presence of RDX.

Table I. Comparison of Percent Pure FEFO in
Original, Library Neat and Library
LX-09 Extracted FEFO.

<u>LX-09 Lot No.</u>	<u>FEFO Lot No.</u>	<u>Original* FEFO</u>	<u>Library Neat FEFO</u>	<u>Δ From Original</u>	<u>Library LX-09 Extracted FEFO</u>	<u>Δ From Library Neat</u>
940-2	10	96.27	95.45	-0.82	91.03	- 4.42
700-3	11	96.47	95.52	-0.95	80.86	-14.66
700-9	12	96.48	95.74	- 0.74	96.42	+ 0.68
940-3	13	96.6	95.71	-0.89	91.31	- 4.40
941-1	17	96.6	96.46	-0.14	87.30	- 9.16
700-8	53	98.42	97.59	-0.83	97.27	- 0.32
700-11	54	97.75	97.66	-0.09	93.96	- 3.70
701-2	57	98.34	98.15	-0.19	97.85	- 0.30

**Samples studied from 5/6/69 to 10/29/70.*

Table II. Comparison of Percent High Boiling Components in Original, Library Neat and Library LX-09 Extracted FEFO

<u>LX-09 Lot No.</u>	<u>FEFO Lot No.</u>	<u>Original* FEFO</u>	<u>Library Neat FEFO</u>	<u>Δ From Original</u>	<u>Library LX-09 Extracted FEFO</u>	<u>Δ From Library Neat</u>
940-2	10	2.40	2.70	+0.30	3.60	+0.90
700-3	11	2.41	2.90	+0.49	9.72	+6.82
700-9	12	2.56	2.59	+0.03	2.10	-0.49
940-3	13	2.6	2.52	-9.98	3.08	+9.56
941-11	17	2.3	2.01	-0.29	6.81	+3.80
700-8	53	1.28	1.79	+0.61	2.27	+0.48
700-11	54	2.07	1.78	-0.29	2.60	+0.82
701-12	57	1.30	1.29	-0.01	1.46	+0.17

**Samples studied from 5/6/69 to 10/29/70.*

Table III. Comparison of Percent Low Boiling Components in Original, Library Neat and Library LX-09 Extracted FEFO

<u>LX-09 Lot No.</u>	<u>FEFO Lot No.</u>	<u>Original* FEFO</u>	<u>Library Neat FEFO</u>	<u>Δ From Original</u>	<u>Library LX-09 Extracted FEFO</u>	<u>Δ From Library Neat</u>
940-2	10	1.33	1.85	+1.85	5.37	+3.52
700-3	11	1.13	1.58	+0.45	9.43	+7.85
700-9	12	0.97	1.68	+0.71	1.50	-0.18
940-3	13	0.8	1.77	+0.97	5.61	+3.84
941-1	17	1.1	1.53	+0.43	5.88	+4.35
700-8	53	0.29	0.62	+0.33	0.47	-0.15
700-11	54	0.18	0.56	+0.38	3.45	+2.89
701-12	57	0.36	0.56	+0.20	0.71	+0.15

**Samples studied from 5/6/69 to 10/29/70.*

Table IV. Library LX-09 Lot Grouping Based on Similar Comparison with Neat Samples

<u>LX-09 Lot No.</u>	<u>High Boiler</u>	<u>Low Boiler</u>	<u>Pure FEFO</u>
700-8/53 ^a	+0.48	-0.15	-0.32
700-9/12 ^a	-0.49	-0.18	+0.68
701-2/57 ^a	+0.17	+0.15	-0.30
940-2/10 ^b	+0.90	+3.52	-4.42
940-3/13 ^b	+0.56	+3.84	-4.40
700-11/54 ^b	+0.82	+2.89	-3.70
941-1/17 ^c	+3.80	+4.35	-9.16
700-3/11 ^c	+6.82	+7.85	-14.66

^aLX-09 extractions components showing virtually no difference from neat FEFO components.

^bLX-09 extractions components showing moderate difference from neat FEFO components.

^cLX-09 extractions components showing marked differences from neat FEFO components.