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**AGING CHARACTERISTICS OF PBXN-106E AS A
FUNCTION OF VARIOUS STABILIZERS AND THE
MIXED ENERGETIC NITROPLASTICIZER BDNPA
TO BDNPA**

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RESEARCH AND TECHNOLOGY DEPARTMENT

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An aging study of (1) the PBXN-106E formulation with various stabilizers and the mixed energetic nitroplasticizer bis(2,2-dinitropropyl)acetal (BDNPA) and bis(2,2-dinitropropyl) formal (BDNPF) at 25°C and 60°C/30%RH for a 9-month period and (2) BDNPA/BDNPF at 104°C for 1, 2, and 3-week periods has been completed. The methods and conditions utilized are discussed. The analysis data include: (1) morphological properties, (2) density, (3) shore "A," (4) high performance liquid chromatography (HPLC), (5) off-gas, (6) impact, (7) percent extract, (8) mechanical properties, (9) acid number, and (10) vacuum thermal stability. The PBXN-106E aging study was undertaken to: (1) evaluate the use of tetrakis [methylene(2,3-di-tert-butyl)-4-hydroxyhydrocinamate]methane (irganox 1010), 2,4-bis(N-octylthio)-6-(4-hydroxy-3,5-di-tertbutylanilino)-1,3,5-triazine (irganox 565), tris(2,4-di-tert-butylphenyl-phosphite (irgafos 168), 2,2'-methylene-bis-(4-methyl-6-tert-butyl phenol) (AO-2246, cyanox), N-methyl-p-nitroaniline (MNA), and 2-nitrodiphenylamine				
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19. (2-NDPA) as possible stabilizer replacements for the phenyl- β -naphthylamine (PBNA) currently in use and (2) to assess the effectiveness of the Aerojet (AJ) acetal/formal (A/F) plasticizer clean-up procedures (e.g., Al_2O_3 /silica gel and/or degassing) on material manufactured at the AJ Plant #1 in October-November 1980. Although this material passed all the required specification tests, it was found to be totally stabilizer (PBNA) depleted after 1 year of storage. PBX mixes prepared from this material also resulted in formulations with very poor aging characteristics.

An additional aging study was completed on several of the A/F samples with added stabilizers (e.g., PBNA, MNA, AO-2246, and mixtures of MNA/2-NDPA). Several A/F samples acquired from the Naval Ordnance Station, Indian Head (NOS/IH) and the Naval Weapons Station, Yorktown (NWS/YK) were used as control or reference samples. Acid number determinations were obtained on (1) unstabilized samples, (2) stabilized samples, and (3) stabilized samples which were aged at 104°C for 1, 2, and 3-week periods.

The data obtained from the PBXN-106E stabilizer study indicate that the majority of the cured mixes containing PBNA or a mixture of MNA and 2-NDPA were less porous than the mixes formulated with either cyanox or mixtures of irgafos and irganox. The total gas volume evolved by the investigated mixes is similar. The rate of nitrogen evolution appears to determine the "time to the fissure" and is mainly responsible for porosity and fissure formation due to the limited solubility of nitrogen in this PBX formulation. The extent of cure, moisture content, acidity, type of stabilizer, and specific impurities also influence the amount and rate or timing of the off-gas reactions.

The data obtained from the 9-month aging study at 25°C indicate no appreciable depletion in any of the assayed components (HMX, RDX, BDNPF, BDNPA, stabilizer), however, data obtained from the 9-month aging study at 60°C/30%RH show noticeable stabilizer depletion in the mixes formulated with either cyanox or mixtures of irgafos and irganox. These data indicate that PBNA and the tested (or certain) mixtures of MNA and 2-NDPA appear to be the most efficient stabilizers for the PBXN-106E formulation.

The presence of a relatively high level of volatiles, due to the use of less rigorous conditions in the final degassing step during processing, appears to be the primary cause of the instability of the BDNPA/BDNPF manufactured at AJ in October-November 1980. Treatment of this material with alumina, silica-gel, and/or degassing removes the bulk of these impurities. The treated samples were also found to contain less acidic components than the untreated samples. PBNA and mixtures of MNA/2-NDPA were found to be the most efficient stabilizers for this nitroplasticizer at ambient temperature.

The results of the aging test at 104°C using the various samples of A/F also show that PBNA and mixtures of MNA/2-NDPA are the most effective stabilizers for this nitroplasticizer. The data suggest that this test (104°C for 1, 2, and 3-week periods) would be a useful addition to the A/F specification as an added indication of initial purity and long-term stability.

EXECUTIVE SUMMARY

This report describes an aging study of PBXN-106E formulated with (1) various stabilizers and (2) the mixed nitroplasticizer bis(2,2-dinitropropyl)formal (BDNPF) and bis(2,2-dinitropropyl)acetal (BDNPA).

The PBXN-106E stabilizer study was undertaken to evaluate the use of irganox 565, irganox 1010, and irgafos 168 (obtained from Ciba Geigy), cyanox (AO-2246), N-methyl-p-nitroaniline (MNA), and 2-nitrodiphenylamine (2-NDPA) (see Nomenclature) as possible replacements for phenyl- β -naphthylamine (PBNA), a suspected carcinogen.

A concurrent study of the PBXN-106E mix formulated with BDNPF/BDNPA manufactured by a "batch process" at Aerojet Plant #1 in October-November of 1980 was also undertaken. The acid number of this plasticizer approached the specification limit after 1 year of storage at ambient temperature and was found to be PBNA depleted. PBX mixes prepared with this material resulted in formulations with very poor aging characteristics. The PBXN-106E mixes used in this study were prepared with BDNPF/BDNPA manufactured at Aerojet Plants #1 and #2 as well as nitroplasticizer obtained from the Naval Ordnance Station, Indian Head (NOS/IH). The NOS/IH acetal/formal was used as the "control sample" since previous mixes containing this mixture resulted in formulations with good aging characteristics. The PBX mixes were characterized via morphological and mechanical properties testing, as well as chemical and off-gas analysis.

An additional aging study was completed on several of the acetal/formal samples with added stabilizers (e.g., PBNA, MNA, AO-2246, and mixtures of MNA/2-NDPA). Several acetal/formal samples acquired from NOS/IH and the Naval Weapons Station, Yorktown (NWS/YK) were used as control or reference samples. Acid number determinations were obtained on (1) unstabilized samples, (2) stabilized samples, and (3) stabilized samples which were aged at 103°C for 1, 2, and 3-week periods.

The data obtained from the PBXN-106E stabilizer study indicate that the majority of the cured mixes containing PBNA or a mixture of MNA/2-NDPA were less porous than the mixes formulated with either cyanox or mixtures of irgafos and irganox. The rate of nitrogen evolution appears to determine the "time of fissure" and is mainly responsible for porosity and fissure formation due to the limited solubility of nitrogen in the PBXN-106E formulations. The extent of cure, moisture content, acidity, type of stabilizer, and specific impurities also influence the amount and rate or timing of the suspected off-gas reactions (Figure 1).

The data obtained from the nine month aging study at 25°C indicate no appreciable depletion in any of the assayed components (HMX, RDX, BDNPF, BDNPA, stabilizer), however, data obtained from the nine month aging study at 60°C/30%RH show noticeable stabilizer depletion in the mixes formulated with either cyanox or mixtures of irgafos and irganox. PBNA and mixtures of MNA and 2-NDPA appear to be the most efficient stabilizers for the PBXN-106E formulation.

The presence of a relatively high level of volatiles, due to the use of less rigorous conditions in the final degassing step during processing appears to be the primary cause of the unstable BDNPA/BDNPF manufactured at Aerojet in October-November 1980. Treatment of this material with alumina, silica-gel, and/or degassing removes the bulk of these impurities. The treated samples were also found to contain less acidic components than the untreated samples (Table 8). PBNA and mixtures of MNA/2-NDPA were found to be the most efficient stabilizers for this nitroplasticizer at ambient temperature.

The results of the aging test at 104°C using the various samples of acetal/formal also show that PBNA and mixtures of MNA/2-NDPA are the most effective stabilizers for this nitroplasticizer. The data suggest that this test (104°C for 1, 2, and 3 week periods) would be a useful addition to the A/F specification as an indication of the materials long term stability.

FOREWORD

↓ This report describes an aging study of (1) the PBXN-106E composition formulated with various stabilizers and the unstabilized and reworked Aerojet (AJ) mixed nitroplasticizer bis(2,2-dinitropropyl)formal (BDNPF) and bis(2,2-dinitropropyl)acetal (BDNPA) and (2) the unstabilized and reworked Aerojet nitroplasticizer with various stabilizers. These studies were undertaken to (1) evaluate the usefulness of various stabilizers in the PBXN-106E formulations, (2) assess the effectiveness of the Aerojet clean-up procedures on the reworked acetal/formal (A/F) manufactured by batch process at their Plant #1, and (3) determine the most effective stabilizer for the PBXN-106E formulation and the energetic nitroplasticizer A/F. The PBXN-106E mixes were characterized via morphological and mechanical properties as well as off-gas, shore "A," % extract, vacuum thermal stability, impact, and chemical analysis. The A/F samples were characterized via acid number.

The identification of commercial materials and/or manufacturers implies neither criticism nor endorsement by the Naval Surface Weapons Center.

Approved by:

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CONTENTS

<u>Chapter</u>		<u>Page</u>
1	INTRODUCTION	1
2	EXPERIMENTAL	3
3	RESULTS AND DISCUSSION	11
	AEROJET ENERGETIC NITROPLASTICIZER BDNPF/BDNPA	11
	MORPHOLOGY/MECHANICAL PROPERTIES/CUBE CRACKING	11
	OFF-GAS STUDY	11
	PBXN-106E AGING STUDY AT 25° AND 60°C/30%RH	13
	ACETAL/FORMAL AGING STUDY	13
	NOMENCLATURE	15
<u>Appendix</u>		<u>Page</u>
A	ACCELERATED AGING TEST PROCEDURE	A-1

ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	SUSPECTED OFF-GAS GENERATING REACTIONS	12

TABLES

<u>Table</u>		<u>Page</u>
1	SAMPLE PREPARATION MATRIX.	4
2	ZERO-TIME TEST DATA.	5
3	OFF-GAS TEST DATA OF PBXN-106E SAMPLES	6
4	AGING STUDY (25°C) OF PBXN-106E MIXES.	8
5	AGING STUDY (60°C/30%RH) DATA.	9
6	ACETAL/FORMAL ACID ASSAY	10

CHAPTER 1

INTRODUCTION

The Insensitive Munitions Advanced Development-High Explosives (IMAD-HE) Program, formerly the Explosives Advanced Development (EAD) Program, has completed an aging study of PBXN-106E formulated with (1) various stabilizers and (2) various samples of the mixed nitroplasticizer bis(2,2-dinitropropyl)formal (BDNPF) and bis(2,2-dinitropropyl)acetal (BDNPA).

The PBXN-106E stabilizer study was undertaken to (1) evaluate the use of 2,4-bis(n-octylthio)-6-(4-hydroxy-3,5-di-tertbutylanilino)-1,3,5-triazine (irganox 565), tetrakis[methylene(3,4-di-tert-butyl)-4-hydroxyhydro-cinnamate]methane (irganox 1010), tris(2,4-di-tert-butylphenyl)-phosphite (irgafos 168)--all obtained from Ciba Geigy, 2,2'-methylene-bis-(4-methyl-6-tert-butyl phenol) (cyanox, AO-2245), N-methyl-p-nitroaniline (MNA), and 2-nitrodiphenylamine (2-NDPA) (see Nomenclature) as possible replacements for phenyl-β-naphthylamine (PBNA) a suspected carcinogen and (2) determine the most efficient stabilizer for this system.

A concurrent study of the PBXN-106E mix formulated with BDNPF/BDNPA manufactured by a "batch process" at Aerojet Plant #1 in October-November of 1980 was also undertaken to determine the reason for the poor aging characteristics of PBX mixes formulated with this plasticizer. The acid number of this plasticizer approached the specification limit after one year of storage at ambient temperature and was also found to be PBNA depleted. The mixes evaluated in this study were formulated with BDNPF/BDNPA manufactured at Aerojet Plants #1 and #2 as well as nitroplasticizer obtained from the Naval Ordnance Station, Indian Head (NOS/IH). These mixes were characterized via morphological and mechanical properties testing as well as chemical and off-gas analysis (see Nomenclature).

An additional aging study (104°C for 1, 2, and 3-week periods) was completed on several of the acetal/formal samples with added stabilizers (e.g., PBNA, MNA, AO-2245, and mixtures of MNA/2-NDPA). This study was undertaken to determine (1) the most efficient stabilizer for the energetic plasticizer acetal/formal and (2) if this type of test could be used as an indication of the long-term stability of BDNPF/BDNPA.

CHAPTER 2

EXPERIMENTAL

A series of 14 PBXN-106E mixes (three pans, one 4" cube each) were processed at the Naval Surface Weapons Center, White Oak (NSWC/WO) using several stabilizers and the various grades of unstabilized and reworked acetal/formal obtained from Aerojet Plants #1 and #2. Reference or control acetal/formal was obtained from NOS/IH and from the Naval Weapons Station, Yorktown, Virginia (NWS/YK) since previous mixes made with these materials resulted in formulations with good aging characteristics.

A listing of these compositions can be found in Table 1. Zero-time test data were acquired on all 15 test mixes. The data (Table 2) include mechanical properties at 25°C, morphology (i.e., number of voids), and time to fissure at 70°C. Four-inch cubes of all 15 mixes were initially stored at 60°C. If no fissuring was observed, the temperature was raised to 70°C with weekly x-rays taken. Units displaying evidence of cracking and/or fissuring were withdrawn from further testing. The density values were obtained in-house by air pycnometer and at NOS/IH by mercury displacement.

Based on the initial morphological and cube cracking results, five of the mixes were not expected to survive a 60°C off-gas study. The nine remaining mixes were selected to evaluate the effects of plasticizer and stabilizer on off-gas composition and rate. One gram samples, 4 mm high by 6 mm in diameter, were stored for 3 days over molecular sieves, then sealed under vacuum in 10 ml glass bulbs. Triplicate samples of each mix were stored at ambient temperature for 2 weeks and at 60°C for 4- and 8-week periods. Following sample withdrawal from storage, the tip of each bulb was broken under vacuum and the gas mixture introduced into a GC/MS system. The resulting gas species produced as a function of storage time were qualitatively and quantitatively analyzed (Table 3). More emphasis was placed on the PBNA stabilized mixes since the results of the cube cracking tests, as well as the zero-time porosity data, indicated that PBNA is the preferred stabilizer for this system. A comparison of various plasticizer lots in conjunction with the same stabilizer was also useful in further characterizing the various PBXN-106E mixes.

TABLE 1. SAMPLE PREPARATION MATRIX*

<u>MIX NO.</u>	<u>BONPA/BONPF (A/F) SAMPLE</u>	<u>STABILIZER ADDED*</u>
1	UNSTABILIZED A/F, AJ PLANT #1, UNTREATED	AO-2246
2	UNSTABILIZED A/F, AJ PLANT #1, UNTREATED	PBNA
3	UNSTABILIZED A/F, AJ PLANT #1, DEGASSED	AO-2246
4	UNSTABILIZED A/F, AJ PLANT #1, DEGASSED	PBNA
5	UNSTABILIZED A/F, AJ PLANT #1, Al ₂ O ₃ /SILICA-GEL	AO-2246
6	UNSTABILIZED A/F, AJ PLANT #1, Al ₂ O ₃ /SILICA-GEL	PBNA
7	UNSTABILIZED A/F, AJ PLANT #1, Al ₂ O ₃ /SILICA-GEL/DEGASSED	AO-2246
8	UNSTABILIZED A/F, AJ PLANT #1, Al ₂ O ₃ /SILICA-GEL/DEGASSED	PBNA
9	UNSTABILIZED A/F, AJ PLANT #2, UNTREATED	PBNA
10	UNSTABILIZED A/F, AJ PLANT #2, REWORKED	PBNA
11	A/F, NOS/IH	PBNA
12	UNSTABILIZED A/F, AJ PLANT #1, Al ₂ O ₃ /SILICA-GEL/DEGASSED	CG#1
13	UNSTABILIZED A/F, AJ PLANT #1, Al ₂ O ₃ /SILICA-GEL/DEGASSED	CG#2
14**	A/F, NOS/IH BENCHMARK	PBNA
15	A/F, NOS/IH	MNA/2-NDPA

Note: *See Nomenclature

** Mix #14 - PBXN-106 contains toluene diisocyanate (TDI) as the cross linking agent. All other mixes (PBXN-106E) contain polymethylene polyphenylisocyanate (PAPI) as the cross linking agent.

TABLE 2. ZERO-TIME TEST DATA*

MIX NO.	RDNPA/BDMPF (A/F)	STABILIZER	VOID CONTENT	DENSITY** AIR	MECHANICAL PROPERTIES (25°C)				TIME TO FISSURE AT 70°C** (WEEKS)	
					SH		IN			SHORE "A"
					PSI	EH %	PSI	PSI		
1	AJ, UNTREATED	AO-2246	HIGH	1.59	1.613	33	10.1	549	42	7
2	AJ, UHTREATED	PBNA	MEDIUM	1.65	1.638	35	10.1	626	51	2
3	AJ, DEGASSED	AO-2246	MEDIUM/HIGH	-	-	37	9.2	608	49	2
4	AJ, DEGASSED (DEG)	PBNA	LOW	1.66	1.654	39	11.2	579	51	3
5	AJ, Al ₂ O ₃ , SG	AO-2246	HIGH	1.61	1.617	40	10.4	678	49	9
6	AJ, Al ₂ O ₃ , SG	PBNA	LOW	-	-	41	10.6	620	52	4
7	AJ, Al ₂ O ₃ , SG, DEG	AO-2246	HIGH	-	-	38	11.0	621	45	9
8	AJ, Al ₂ O ₃ , SG, DEG	PBNA	LOW	-	-	42	12.5	622	49	5
9	AJ, PLANT NO. 2	PBNA	MEDIUM	-	-	37	10.0	672	49	2
10	AJ, PLANT NO. 2 REWORKED	PBNA	LOW	-	-	37	9.6	729	51	5
11	NOS/IH	PBNA	LOW	-	-	39	9.3	674	52	6
12	AJ, Al ₂ O ₃ , SG, DEG	CG#1	HIGH	-	-	32	10.5	574	48	3
13	AJ, Al ₂ O ₃ , SG, DEG	CG#2	HIGH	-	-	34	10.3	569	47	7
14	NOS/IH	PBNA	MEDIUM/HIGH	-	-	36	9.3	600	48	4
15	NOS/IH	MNA/2-NDPA	LOW	-	-	40	12.2	560	53	-

*See Nomenclature

**CC/GM

***Five weeks at 60°C, prior to raising the temperature to 70°C

TABLE 3. OFF-GAS TEST DATA OF PBXN-106E SAMPLES

MIX NO.	PBXN-106E SAMPLE	2 WEEKS AMBIENT CC/GM x 10 ³ (STP)					4 WEEKS 60°C CC/GM x 10 ³ (STP)					8 WEEKS 60°C CC/GM x 10 ³ (STP)				
		N ₂	NO	CO ₂	N ₂ O	AA*	N ₂	NO	CO ₂	N ₂ O	AA*	N ₂	NO	CO ₂	N ₂ O	AA*
1	AJ, UNTREATED AC-2246	18.3	-	5.4	0.4	0.3	42.2	-	50.6	4.0	32.4	43.9	16.2	69.7	5.6	28.3
2	AJ, UNTREATED PBNA	7.8	-	7.4	0.6	1.3	27.9	12.1	47.8	4.5	19.5	29.8	24.0	56.4	5.8	36.5
3	AJ, DEGASSED AO-2246	14.7	-	18.1	0.1	1.4	32.8	-	62.1	2.9	20.2	33.0	-	80.5	5.1	24.8
6	AJ, Al ₂ O ₃ /SG PBNA	6.4	-	20.9	0.1	0.6	21.1	-	59.9	2.8	20.3	24.1	21.3	75.4	4.4	17.4
8	AJ, Al ₂ O ₃ /SG/ DEG, PBNA	6.7	-	25.4	0.1	0.1	18.8	-	67.5	3.2	20.6	20.6	21.4	84.2	5.1	13.6
10	AJ, PLANT#2 REWORK, PBNA	6.8	-	12.3	0.1	0.3	17.8	-	54.5	2.4	13.6	22.9	18.8	69.5	4.4	15.0
11	NOS/IH, PBNA	5.7	-	13.7	0.1	-	17.0	-	66.1	2.7	15.6	20.6	20.6	64.0	4.4	20.9
13	AJ, Al ₂ O ₃ /SG/ DEG, CG#2	17.1	-	16.9	0.1	-	32.7	-	66.7	2.9	-	35.3	-	86.7	5.4	-
14	NOS/IH PBNA	8.4	-	2.8	0.3	-	26.9	-	23.3	14.4	2.9	39.4	4.2	32.8	25.8	3.8
15	NOS/IH MNA/2-NDPA	9.9	-	0.9	0.1	0.2	24.2	-	37.3	2.5	6.1	24.9	-	55.7	3.9	6.6

*AA = acetaldehyde

Based on the cube cracking results and porosity data, several of the mixes were not expected to survive the 60°C/30%RH aging without fissuring and were excluded. These include mix numbers 2, 3, 4, 6, 9, 12, and 14. Among the AO-2246 stabilized mixes (mix #1, 5, and 7), only the one sample containing nitroplasticizer which had been degassed/ Al_2O_3 /silica gel treated was selected (mix #7). Six mixes were chosen for long term aging at 25°C and 60°C/30%RH. The initial (0-time) and nine month data include mechanical properties, density, shore "A," % extract, % stabilizer concentration, high performance liquid chromatography (HPLC), vacuum thermal stability, and 50% impact point. These data can be found in Tables 4 and 5.

Various grades of acetal/formal((1) AJ unstabilized and treated (Al_2O_3 , silica-gel, degassed), (2) AJ stabilized (PBNA, AO-2246, MNA, 2-NDPA), and (3) control samples (NWS/YK, NOS/IH)) were analyzed for acidic components. These samples were also selected for an additional aging study at 104°C for 1, 2, and 3 week periods (see Table 6). The aging conditions of one week at 104°C approximate a ten year aging period at ambient temperature.

TABLE 4. AGING STUDY (25°C) OF PBXN-108E MIXES

MECHANICAL PROPERTIES										STABILIZER CONCENTRATION				
-----------------------	--	--	--	--	--	--	--	--	--	--------------------------	--	--	--	--

MIX NO.	AGE	SM (PSI)	EM (%)	SR (PSI)	ER (%)	YM (PSI)	SHORE "A"	% EXTRACT	% CYANOX	% PBNA	% CG #2	% MNA/2-NDPA
7	0-TIME	38	11.0	37	11.7	621	45	93.00	0.18			
	9 MONTHS	46	9.6	44	10.5	794	50	93.04	0.17			
8	0-TIME	42	12.5	38	14.4	622	49	93.00		0.14		
	9 MONTHS	48	10.3	46	11.5	754	51	93.07		0.14		
10	0-TIME	37	9.6	34	11.9	729	51	93.03		0.18		
	9 MONTHS	38	10.0	36	11.9	686	48	93.07		0.19		
11	0-TIME	39	9.3	36	10.7	674	52	93.02		0.16		
	9 MONTHS	42	10.1	40	11.5	685	47	93.13		0.16		
13	0-TIME	34	10.3	33	11.5	569	47	93.41			0.22	
	9 MONTHS	37	10.7	36	11.7	577	47	93.47			0.21	
15	0-TIME	40	12.2	39	13.2	560	51	93.03				0.06/0.13
	9 MONTHS	45	11.8	43	12.7	598	50	93.05				0.06/0.14

* No chemical changes observed in any of the other assayed components (e.g., RDX, HMX**, BDNPF, BDNPA) during the nine month aging study.

** Present as an "impurity" in the RDX lots.

TABLE 6. AGING STUDY (60°C/30%RH) DATA*

MIX NO.	AGING CONDITION	MECHANICAL PROPERTIES						SHORE "A"	DENSITY (GM/CC)	VTS-100°C (CC/GM/48 HRS)	IMPACT-50% HT (CM)	STABILIZER CONCENTRATION			
		SM (PSI)	EM (%)	SR (PSI)	ER (%)	YM (PSI)	AO-2246 (%)					PBMA (%)	CGP2 (%)	MMA/2-NBPA (%)	
7	0-TIME	29	18.8	29	19.4	333	47	1.61	-0.15		38.0	0.18			
7	9 MONTHS	32	17.0	32	17.3	395	35	1.64	+0.26		38.8	0.002			
8	0-TIME	31	20.8	31	21.8	331	46	1.63	-0.06		38.0	6.14			
8	9 MONTHS	34	17.0	34	17.0	403	35	1.66	+0.75		35.2	0.001			
10	0-TIME	24	16.8	23	18.2	344	45	1.63	0.0		37.3	0.18			
10	9 MONTHS	28	17.7	28	18.5	353	48	1.64	+0.66		36.6	0.002			
11	0-TIME	--	----	--	----	----	45	1.63	+0.05		36.6	0.16			
11	9 MONTHS	--	----	--	----	----	39	1.64	+0.68		38.0	0.002			
13	0-TIME	28	17.3	28	17.3	340	45	1.62	+0.24		38.0		0.22		
13	9 MONTHS	27	17.7	27	17.7	308	34	1.47**	+0.75		36.6		0.07		
15	0-TIME	--	----	--	----	----	53	1.62	+0.29		37.3			0.06/0.13	
15	9 MONTHS	--	----	--	----	----	48	1.64	+0.05		35.2			0.07/0.14	

* All mixes were tested at ambient conditions. No chemical changes were observed in any of the other assayed components (e.g., RDX, HMX, BNPF, BNPA) during the nine month aging period.

** Excessive voids noted in this sample.

TABLE 6. ACETAL/FORMAL ACID ASSAY

BDNPA/BDNPF SAMPLES	A/F AS RECEIVED	ADDED STABILIZER**	A/F PLUS STABILIZER**	ACID NUMBER*		
				AGING AT 104°C		
				1 WEEK	2 WEEK	3 WEEK
A/F used in mix #'s 1, 2	5.74	A0-2246 PBNA	5.49 5.46	5.60 3.76	8.36 4.77	----- -----
A/F used in mix #'s 3, 4	0.27	A0-2246 PBNA	0.24 0.20	0.37 0.17	1.79 0.40	----- 1.67
A/F used in mix #'s 5, 6	0.41	A0-2246 PBNA	0.30 0.10	0.41 0.008	2.34 0.17	----- 2.39
A/F used in mix #'s 7, 8	0.29	PBNA	0.17	0.08	0.18	1.03
A/F used in mix # 9	2.37	MNA PBNA	2.25 2.19	1.77 1.01	3.89 1.17	----- -----
A/F used in mix # 10	0.22	MNA/2-NDPA PBNA	0.11 0.03	0.11 0.03	0.08 0.08	0.11 0.11
A/F used in mix #'s 11, 14, 15	0.06	PBNA	***	0.09	0.08	0.07
A/F, NBR #38019, NWS/YK lot # 20-118-002	0.06	PBNA	***	0.08	0.08	0.06
A/F, AJ, lot # 13F-162-004 reworked, Al ₂ O ₃ clean up	0.74	PBNA MNA/2-NDPA	0.51 0.60	0.47 0.47	3.98 2.03	5.65 7.41
A/F, AJ, lot # 2J-176-001, first article material**	0.05	PBNA	***	0.36	0.08	0.08
A/F, AJ, lot # H-118-001**	0.03	PBNA	***	0.19	0.22	2.16

Samples returned to ambient temperature before testing.

*Milligrams of KOH/gram of acetal/formal

**Stabilizers added at NSWC/MO; 0.25%

***Stabilizer added by manufacturer - control samples.

CHAPTER 3

RESULTS AND DISCUSSION

AEROJET ENERGETIC NITROPLASTICIZER BDNPF/BDNPA

The presence of a relatively high level of volatiles, due to the use of less rigorous conditions in the final degassing step during processing, appears to be the primary cause of the poor aging characteristics or anomalous behavior of the BDNPF/BDNPA nitroplasticizer manufactured by "batch process" at Aerojets' Plant #1 in October-November of 1980. As indicated in Table 5, treatment of this material with alumina/silica-gel removes the bulk of the impurities. Combining this treatment with degassing removes even more of these impurities but does not appear to be cost effective in terms of the benefits gained.

MORPHOLOGY/MECHANICAL PROPERTIES/CUBE CRACKING

The zero-time test results of the fourteen PBXN-106E mixes can be found in Table 2. All of the mixes formulated with AO-2246, CG#1, and CG#2 showed numerous voids. The majority of the mixes containing PBNA and MNA/2-NDPA stabilizers exhibited considerably less porosity. The 70°C fissure data indicate that the highly porous mixes had the "longest time to fissure" since the presence of voids aids gas diffusion. Of the low porosity mixes, those formulated with the aluminum oxide/silica-gel treated material and the NOS/IH produced nitroplasticizer had the "longest time to fissure."

OFF-GAS STUDY

Off-gas results of nine PBXN-106E mixes can be found in Table 3. It appears that the high porosity characteristic noted in the zero-time mixes formulated with AO-2246, CG#1, and CG#2 is mainly caused by gas evolution during processing and cure. Off-gas analyses after two weeks of ambient storage indicate that the AO-2246 and the CG#2 stabilizer promote nitrogen evolution at an early stage in the aging process. The drastic reduction noted in both the nitrogen and carbon dioxide evolution rates between the four and eight week periods has also been observed for urethane based crosslinked double based propellants formulated with hexamethylene diisocyanate (HMDI). Both nitrogen and carbon dioxide mainly originate from urethane bonds and unreacted isocyanate. Nitrogen is chiefly responsible for porosity and fissure formation since it is fifty times less soluble than carbon dioxide and other more polar gases in most propellant and explosive formulations. The "time to fissure" is further determined by diffusion path (e.g., diameter, porosity) as well as mechanical properties. Probable mechanisms for the formation of nitrogen and carbon dioxide, based on the hydrolysis and oxidation of isocyanate and urethane groups, are cited in Figure 1. The

SUSPECTED OFF-GAS GENERATING REACTIONS

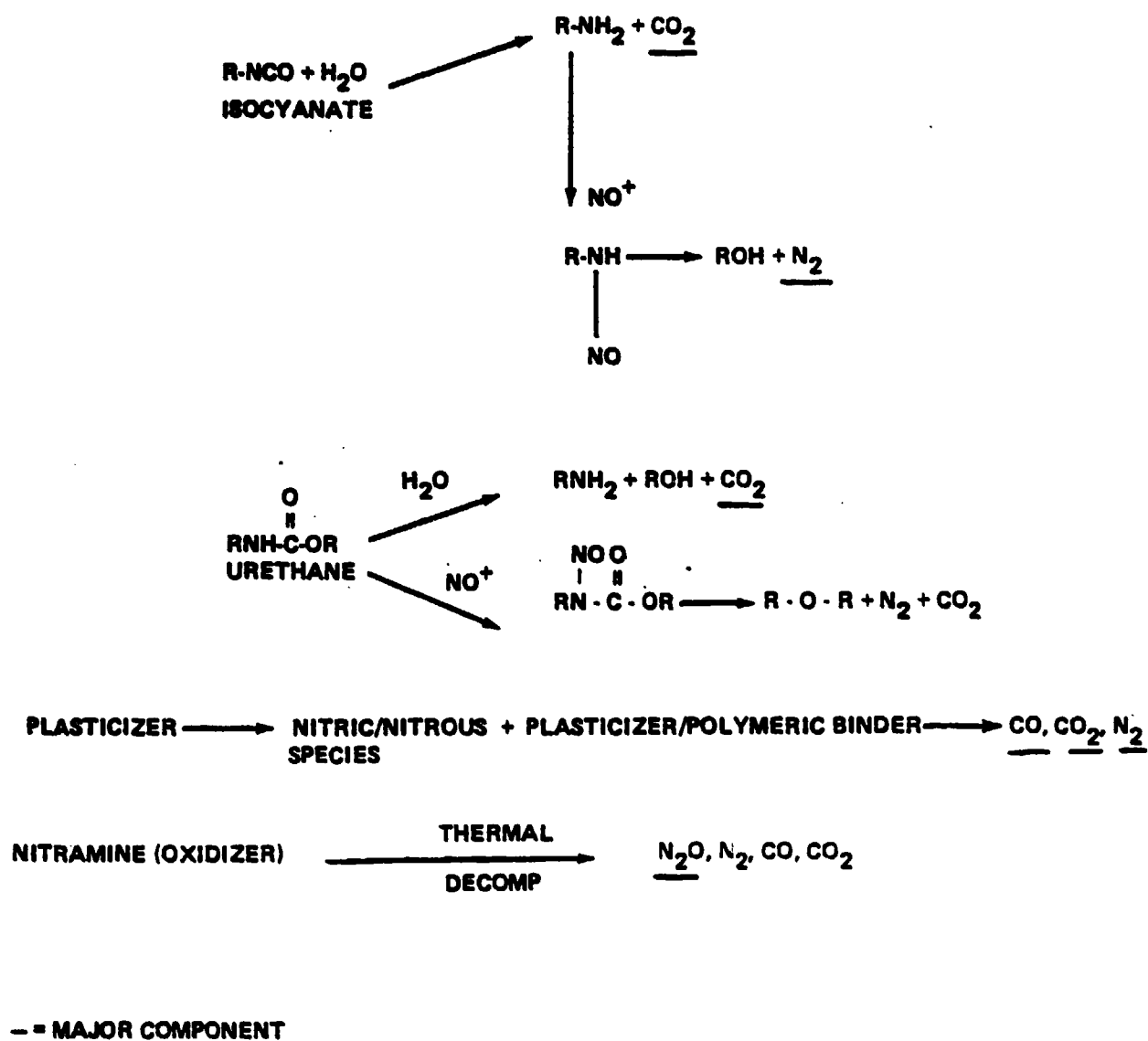


FIGURE 1. SUSPECTED OFF-GAS GENERATING REACTIONS

extremely high crosslink density of the PBXN-106E mixes (0.09 m mole/gram as compared to 0.024 - 0.030 m mole/gram for optimized propellant formulations) together with suspected excess isocyanate favor the evolution of both gases. The extent of cure, moisture content, acidity, type of stabilizer, and specific impurities also influence the amount and rate or timing of these reactions. It is assumed that the oxygen observed in the off-gas mixtures was dissolved in the zero-time propellant and is not a side reaction product. The carbon monoxide and nitric oxide, as well as portions of nitrogen and carbon dioxide are the results of oxidative degradation reactions involving mainly the energetic plasticizer (Figure 1). Acetaldehyde (AA) represents an intermediate oxidation product which may decrease in concentration with progressive aging due to further oxidation to form CO, CO₂, and water. In general, data (Table 3) obtained after 4 and 8 weeks also indicate that most gas mixtures which are relatively low in nitrogen exhibit a larger volume of nitric oxide. Nitrous oxide is not detected in the PBXN-10E mixes evolving the largest volumes of CO₂.

PBXN-106E AGING STUDY AT 25° AND 60°C/30%RH

Based on the morphological data (cube cracking and porosity) five mixes were selected for long-term (9 months) aging at 25°C and 60°C/30%RH. The conditions of 9 months at 60°C/30%RH approximate 9 years at 23.9°C, or 11 years at 21.1°C in real time storage. The results of these studies can be found in Tables 4 and 5. The data show no physical aging trends (e.g., fissuring and/or cracking) were noted after storing for 9 months at 25°C. Besides suspected post cures for mixes 7 and 8 during the first 2 weeks of aging, no significant changes were observed in the mechanical properties, shore "A," % extract or stabilizer concentration after the 9 month aging period (Table 4). The presence of voids in some of the test mixes could be responsible for the variations noted in the initial mechanical properties and hardness (shore "A") measurements. The reproducibility of the % extract data tends to support these conclusions. Decreases in stabilizer concentration (Table 5) were observed in four of the PBXN-106E mixes containing AO-2246, PBNA, and CG#2 after the 9-month period at 60°C/30%RH. However, no significant decrease was noted in mix #15. No changes were noted in any of the other HPLC assayed components, e.g., RDX, HMX, BDNPF, and BDNPA. The results of these studies indicate that PBNA and mixtures of MNA and 2-NDPA are the most efficient stabilizers for the PBXN-106E system.

ACETAL/FORMAL AGING STUDY

The detailed BDNPF/BDNPA test data of four control samples as well as various grades of nitroplasticizer used in the PBXN-106E study can be found in Table 6. The data show that PBNA and MNA/2-NDPA are the most efficient stabilizers for this energetic nitroplasticizer. The acid number values of the control samples, with the exception of the AJ sample from NWS/YK, lot #H-118-001, 12/6/83, remained within the specification limit (less than 0.5 milligram KOH/gram sample--see Appendix A), even after 3 weeks at 104°C. Of the various grades of nitroplasticizer used in this study, only the reworked material containing PBNA and MNA/2-NDPA were still within the specification limit after 2 weeks of aging at 104°C. In general, the addition of stabilizer (0.25%) to the unstabilized nitroplasticizer decreased the acid number value. This decrease continued even after 1 week of aging at 104°C. The results of this aging test indicate that

(1) PBNA and MNA/2-NDPA are the most effective stabilizers for this energetic nitroplasticizer and (2) this test (104°C for 1, 2, and 3-week periods) would be a useful addition to the acetal/formal specification¹ as a further indication of initial purity and long-term stability.

¹Weapons Specification, Bis(2,2-Dinitropropyl)Acetal-Bis(2,2-Dinitropropyl)Formal, Mixture of; WS 1141A, (Code Index 10001) 16 Sep 1963.

NOMENCLATURE
PBX COMPOSITION

<u>Ingredients</u>	<u>Use</u>	<u>PBXN-106*</u>	<u>PBXN-106E</u>
RDX Type B Class A	explosive component	60.0	60.0
Type B Class E	explosive component	15.0	15.0
Phenyl-beta-naphthylamine (PBNA)	stabilizer	0.250	---
Ferric Acetylacetonate (FeAA)	catalyst	0.025	---
Bis(2,2-dinitropropyl)acetal/ Bis(2,2-dinitropropyl)formal (50/50)	energetic nitroplasticizer	18.548	17.75
1,1,1-tris(hydroxymethyl)propane	polymer	0.485	0.56
Polyoxyethyleneglycol	polymer	4.500	4.24
2,4-toluene diisocyanate (TDI)	cure agent	1.192	----
PAPI-135	cure agent	---	2.20

*Baseline or benchmark sample

NOMENCLATURE (Cont.)

High Performance Liquid Chromatography Data

HPLC Conditions:

Isocratic elution

Detector: 254 nm

Cartridge: Radial - PAK A with RCM-100 Radial Compression Module

Flow rate: 0.5 to 2.0 ml/minute

Mobile phase: methanol:water (v,v)

Chart speed: 0.5 cm/minute

Compound	Mobile Phase (MeOH:H ₂ O)	Flow Rate (ml/min)	Retention Time (R _t) (minutes)
HMX	70:30	1.0	3.3
RDX	70:30	1.0	4.8
PBNA	70:30	1.0	27.3
BDNPF	70:30	1.0	8.1
BDNPA	70:30	1.0	9.7
MNA	70:30	2.0	2.9
2-NDPA	70:30	2.0	15.3
Cyanox (A0-2246)	90:10	0.5	12.0
Irganox 565	100:0	2.0	6.7
Irganox 1010	100:0	2.0	3.3
Irgafos 168	100:0	2.0	6.0

NOMENCLATURE (Cont.)

Mechanical Properties Test - procedures as stated in the JANNAF CPIA publication on Mechanical Properties, # 21, Solid Propellant Mechanical Behavior Manual.

Designations: S_m = stress at maximum
 S_r = stress at rupture
 E_m = elongation at maximum
 E_r = elongation at rupture
 Y_m = Young's Modulus

Impact Sensitivity Test

The apparatus consists of a hardened 2.5 ky steel weight, free falling from a height of up to 3.2 meters, onto a hardened tool steel "striker" pin which transmits the shock to the test sample. The explosive sample rests without restraint on 5/0 grade, flint sandpaper, which rests without restraint on a hardened tool steel anvil, which in turn is rigidly supported on the machine base. Approximately 35 mgs of sample is used per shot. A rigidly designed series of 25 trials, known as the "Bruceton up-and-down" method, is run and the simple "Bruceton method" manipulation of the number of "fires" and "no-fires" for each of the carefully selected drop heights provides the mean height for a "fire." A calibrated microphone "noisemeter" decides whether a "fire" or a "no-fire" occurs in a particular trial. This mean height, called the "50% Point" or "50% Impact height" is then reported in cm.

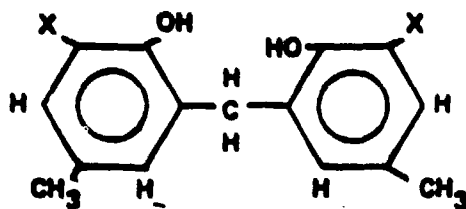
Vacuum Thermal Stability Test

Vacuum thermal stability tests at 100°C were determined on the PBXN-166E samples in accordance with the procedures detailed in (a) NAVORD OD44811, Vol. 1, of 1 January 1972, and (b) The Vacuum Thermal Stability Test for Explosives, NOLTR 70-142, of 28 October 1970. All the samples tested passed the interim qualification criteria of 2.0 cc/gm (corrected) for 48 hrs. (less 1st hr.) at 100°C. The negative (-) cc/gm gas evolved is due to condensate formed on the cold part of the tube (distillation process) and not to the formation of decomposition gases such as CO₂, CO, N₂O, NO₂, etc. which do not condense at room temperature.

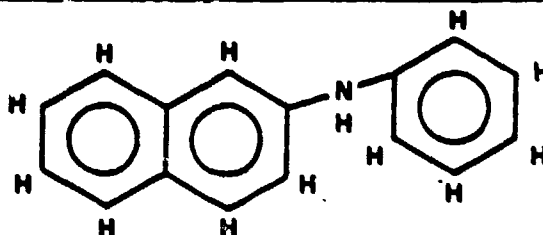
NOMENCLATURE (Cont.)

CYANOX (AO - 2248)

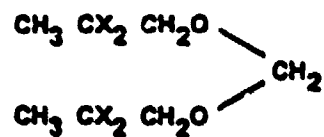
X = T-BUTYL



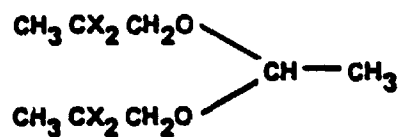
2,2' - METHYLENE BIS (4-METHYL-6-T-BUTYLPHENOL)

FBNA

PHENYL-BETA-NAPHTHYLAMINE

BDNPF

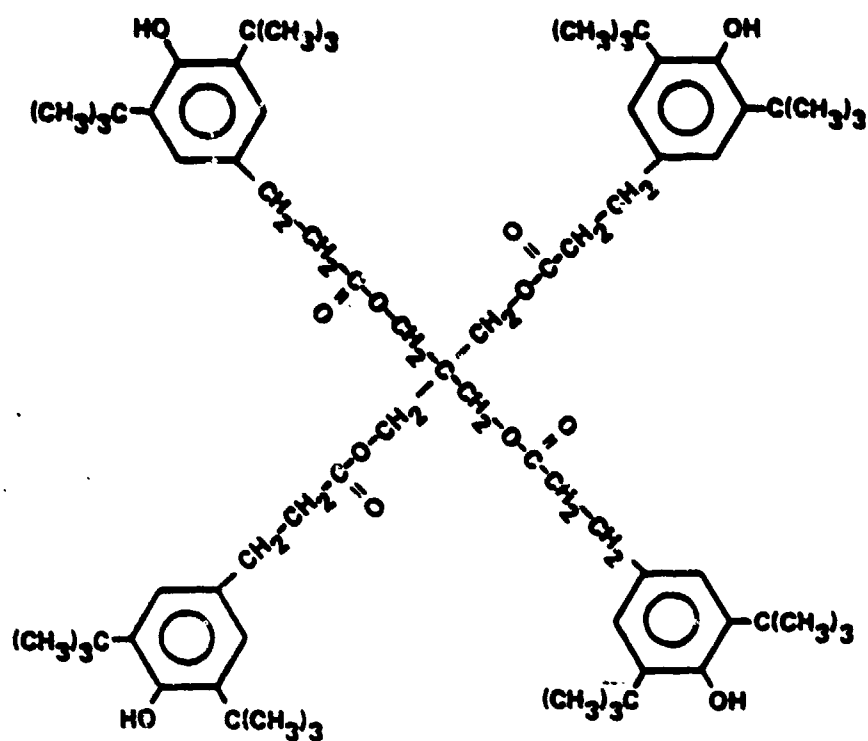
BIS (2,2-DINITROPROPYL) FORMAL

X = NO₂BDNPA

BIS (2,2-DINITROPROPYL) ACETAL

X = NO₂

NOMENCLATURE (Cont.)

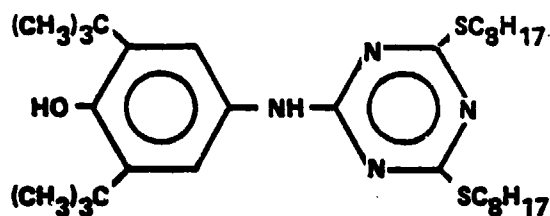
IRGANOX 1010**TETRAKIS [METHYLENE (3,5-DI-TERT-BUTYL-4-HYDROXYHYDROCINNAMATE)] METHANE**

CG #1 - IRGANOX 565 / IRGAFOS 168 (2/1, W:W)
 CG #2 - IRGANOX 1010 / IRGAFOS 168 (2/1, W:W)

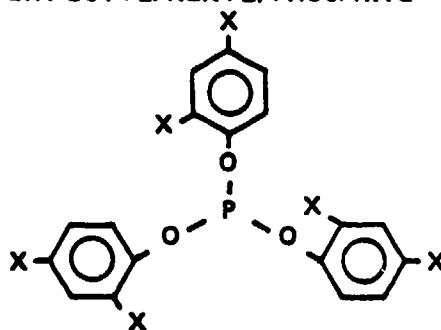
NOMENCLATURE (Cont.)

IRGANOX 566

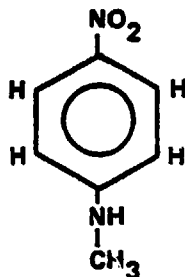
2,4-BIS (N-OCTYLTHIO)-6-(4-HYDROXY-3,5-DI-TERT-BUTYLANILINO)-1,3,5-TRIAZINE

IRGAFOS 168 -TRIS (2,4-DI-TERT-BUTYLPHENYL) PHOSPHITE

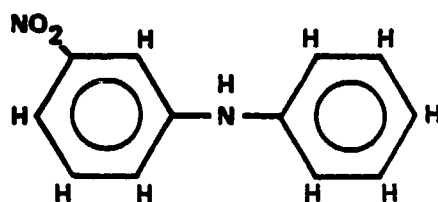
X = T-BUTYL

MNA

N-METHYL-PARA-NITROANILINE

2-NDPA

2-NITRODIPHENYLAMINE



APPENDIX A

ACCELERATED AGING TEST PROCEDURE

Accelerated aging test procedure* - acid number determination

Apparatus - analytical balance (samples weighed to nearest 0.005 gram), 50 ml analytical burette, 125 ml Erlenmeyer flask, magnetic stirrer and stirring bar, oil bath maintained at $104^{\circ} \pm 0.5^{\circ}\text{C}$.

Chemicals - a 1% phenolphthalein solution in 60% methanol and 40% distilled water, a 0.02N methanolic potassium hydroxide (KOH) solution, a 1:1 v/v solution of methanol/chloroform (ACS grade), and distilled water.

Aging Procedure - Two hundred grams of the test material shall be weighed into a 125 ml Erlenmeyer flask. The mouth of the flask shall be covered with aluminum foil and the flask immersed in an oil bath to a point above the level of the sample. The temperature of the oil bath shall be maintained at $104^{\circ} \pm 0.5^{\circ}\text{C}$ (220°F) for seven days or 168 hours. After heating, remove the sample from the bath and cool to room temperature.

Acid Number Determination - Draw approximately 10 grams of the BDNPF/BDNPA from the flask and weigh it to the nearest 0.005 grams into a 125 ml Erlenmeyer flask. To the flask, add 25 ml of a 1:1 v/v solution of methanol/chloroform. Stir until the entire sample is in solution. Add 25 ml of distilled water and approximately 20 drops of the 1% phenolphthalein solution. With continuous stirring, titrate with 0.02N methanolic KOH to a 30 second pink end point in water layer. Run a blank with all the reagents omitting the BDNPF/BDNPA.

$$\text{Calculation - Acid Number (mg KOH/gm sample)} = \frac{(V_2 - V_1) (N) (56.11)}{W}$$

Where: V_1 = volume of methanolic KOH solution used in the sample titration, ml

V_2 = volume of methanolic KOH used in blank titration, ml

W = weight of sample in grams

N = normality of KOH

*According to Aerojet, this procedure approximates a 10 year aging period at ambient temperature.

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