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ANALYSIS OF HYDROXYLAMMONIUM
NITRATE BASED LIQUID PROPELLANTS

RONALD SASSÉ

SEPTEMBER 1990

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13. ABSTRACT (Maximum 200 words) Chemical analysis of hydroxylammonium nitrate (HAN) based liquid propellants have matured over the past twenty years, and various techniques have been offered to meet particular requirements at particular times. This has resulted in a plethora of technology built by several authors, and it now seems prudent to summarize those methods selected by our Laboratory to best characterize propellant.			
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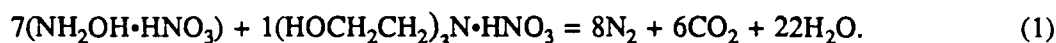
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I wish to thank Charles Leveritt and Josephine Wojciechowski of the BRL for creating a LOTUS spread sheet incorporating the two density models. With such a display, data could be more readily compared and evaluated.

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1. INTRODUCTION

Liquid propellants (LP) have been, and continue to be, a subject of active study at the Ballistic Research Laboratory (BRL) and other laboratories for a number of years. Two candidate systems chosen for extensive evaluation are identified as LP-1845 and LP-1846—both of which are homogeneous mixtures of hydroxylammonium nitrate (HAN), triethanolammonium nitrate (TEAN), and water. The propellants are formulated to be stoichiometric with respect to the combustion products, carbon dioxide, nitrogen, and water. This leads to Equation 1,



An added constraint is that the propellant be 11 mol in nitrate; this condition was met by multiplying Equation 1 by 11/8. The result is:



Such a mixture must be diluted to a liter; the required water will depend on the displaced volume of the dissolved salts. This is not a trivial matter, for the salts are very hygroscopic and cannot be weighed using standard techniques. Instead, by accurately determining the density of several HAN and TEAN solutions, and relating density to concentration (known to 2-3 parts per 1,000 by titration) a model was developed to predict the density of liquid propellant. This work is described in two recent reports (Sassé et al. 1988; Sassé 1988), and the density of LP-1845 at 20° C was calculated to be 1.46939. These methods, which will be described later, predict that the concentrations of water in Equation 2 are increased on both the right and left side of the equation by 14.000 mol water. Results are given in Table 1 and the values listed as LP-1845 def. are the molar concentrations of LP-1845 conforming to the above idealized definition. Propellant 1846 is defined as the above formulation (Equation 2) diluted with water such that it is 20 weight-percent in water.

Earlier in the program, in about 1973, Eli Freedman (Freedman and Travis 1981) considered several different fuel components for HAN-based propellants using known or estimated densities. He offered several propellant compositions on a weight-percent basis. His values for 1846 and 1845 are often used, and in some cases rounded off, but the table has been reproduced in Nathan Klein's more

accessible and unclassified report (Decker et al. 1987) which is employed here to compare the ideal defined propellant to the weight-percent values used in every past, present and future procurement action. By establishing a weight-percent standard, as we have done in the early part of the BRL program, we avoided the horrendous problem of correcting our specifications every time a slightly better density estimate was made. The compositions for 1845 and 1846 are given in Table 1 under the listing weight-percent. Differences between the two approaches are small, but the weight-percent specification values do not exactly conform to stoichiometric definition. In many reports, these two approaches have been stated to be equal where in fact they are but nearly equal.

Table 1. Chemical Composition of Liquid Propellant

<u>TYPE</u>	Propellant Composition						<u>DENSITY,</u> g/cm3 @20° C
	<u>HAN,</u>		<u>TEAN,</u>		<u>WATER,</u>		
	wt.-%	mol	wt.-%	mol	wt.-%	mol	
1845 def	62.884	9.625	19.850	1.375	17.266	14.000	1.46937
1845 wt.-%	63.23	9.62	19.96	1.38	16.81	13.64	1.47261
1846 def	60.806	9.180	19.194	1.312	20.000	16.090	1.44998
1846 wt.-%	60.79	9.09	19.19	1.30	20.02	15.93	1.44983

Acceptable deviations from target concentrations have been set at ± 0.5 weight-percent with respect to the concentrations of HAN, TEAN, and water. These are the current limits tolerated in purchasing and contracting actions. Furthermore, an excess nitric acid limit in HAN is being considered, at about 0.1 weight-percent, as well as a lower boundary representing some limit on the degree of acid deficiency or free amine allowed. Clearly, we have to become more precise and perhaps even include a lower limit for allowable ammonium ion concentration which has never been explicitly specified.

1.1 Density. Densities were measured at 20.00° C using a DMA 55 density meter made by Anton Parr with a precision of 0.0062%. The instrument employs a hollow oscillator and relates mass to frequency. It was soon evident that taking samples of propellant from an open beaker showed the measurable effects of evaporation which was eliminated by employing a 30 cm³ storage syringe allowing several aliquot additions. Glassware and tubing were oven-dried before use.

The densities of HAN and TEAN solutions were measured as a function of molar concentration and this data was used to form a predictive model for estimating the density of propellant (Sassé et al. 1988; Sassé 1988). Also, these values were used to develop weight-percent relationships. A small error appears in the equations of these reports, and the proper equations are given below. They are incorporated in a BRL-generated LOTUS 1-2-3 spread sheet that performs much of the required arithmetic which is available on request.

$$\rho_{\text{TEAN}} = 0.99823 + 0.0546873 \text{ mol (TEAN)} \quad (3)$$

$$\rho_{\text{TEAN}} = 0.99823 + 0.0034015 \text{ (wt.-% TEAN)} \quad (4)$$

$$\rho_{\text{HAN}} = 0.99935 + 0.04630 \text{ mol (HAN)} - 0.0004007M(\text{HAN})^2 \quad (5)$$

$$\rho_{\text{HAN}} = 1.00083 + 4.5813 \times 10^{-3} \text{ (wt.-% HAN)} + 2.4609 \times 10^{-5} (\text{wt.-% HAN})^2 \quad (5)$$

The model is described (Sassé 1988) and from concentration values it subtracts the volume of TEAN from a particular propellant solution and then calculates the density of the remaining HAN solution. The density of the propellant is then the weighted average of the two parts. An example is given in the Appendix. David Cawfield (1990) of Olin Chemical Corporation of Tennessee used the same tables of experimental data to derive a similar model in terms of the volumes of constituents. The two models give almost the same result, differing by but 0.12%. Therefore, one can use either model. The ratio between the measured and calculated densities was 1.004 ± 0.007 among 21 lots of propellant.

1.2 Analytical Procedures. In recent times, analytical reports have been published by the same author where conflicting statements have been presented among a collection of reports, or various authors have recommended different approaches. This situation reflects progress as opposed to

controversy; however, it seems prudent at this time to identify the analytical methods currently used by BRL. Such methods exclude dynamic production-related controls which should be considered separately.

2. HAN-TEAN ANALYSIS

Early attempts to titrate LP with aqueous base gave blended endpoints near a pH of 10 for the combined HAN and TEAN concentrations. This is the direct result of the respective pK's being too close in value where the equivalence points for titrating HAN and TEAN individually are at pH's of 8.8 and 9.7, respectively. This situation was avoided by adding a small amount of acetone that quantitatively reacts with HAN to form an oxime and nitric acid. Then nitric acid and TEAN could be titrated with base, yielding two distinct endpoints, one near a pH of 5 and the other near a pH of 10. The procedure is as follows:

Samples of 0.25 to 0.30 grams of LP are diluted with
50 ml of distilled water to which 2.0 ml of acetone
are added. Titrations are performed with 0.25 to
0.30 mol of NaOH.

In a series of replicate samples, it is beneficial to calculate the individual HAN-to-TEAN ratios such that statistics can be performed. An advantage of this approach is that these ratios are independent of any standardization error of the titrant. Such data are given in Table 2.

3. ANALYSIS OF EXCESS STRONG ACID IN LP

Strong acid, if present in moderate amounts in LP, can be titrated directly with strong aqueous base. The resulting typical shaped curve can be interpreted routinely. Such is the case for solutions more acidic than about 0.20 weight-percent. At lesser acid concentrations the titration curve is not developed. Therefore, to analyze samples containing a small amount of excess acid, a spiking technique was adopted where 1 to 4 ml of 0.25 to 0.30 mol nitric acid was added which allowed the development of a shaped curve from which the first and second derivatives can be extracted. This method, first described in a liquid propellant conference (Sassé 1990), provided the added benefit of detecting any degree of acid deficiency (a condition of excess free amine). Obviously, HAN titrations

must be corrected for this degree of excess acid or excess amine. Typical titration graphs are shown in Figure 1 for the titration of pure HAN and also a HAN sample spiked with 4.00 ml of 0.25 mol HNO_3 . In the figure, the titrant volumes were adjusted to equal equivalents. The analytical procedure follows:

Samples are prepared by weighing 28 grams of LP and diluting with 40 ml of distilled water. Samples are spiked with 1 to 4 ml of 0.25 to 0.30 mol HNO_3 and titrated with 0.2 to 0.3 mol NaOH .

Analysis for one LP lot having a density of 1.44519 ± 0.00014 at 20.00°C , is given in Table 2. Deviations reflect the precision of the titration using an unbiased estimate for error where sample population was taken at N rather than N-1.

Table 2. HAN and TEAN Analysis of LP-1846

SAMPLE MASS, g	HAN, wt.-%	TEAN, wt.-%	HAN/TEAN, ratio
0.2994	61.2150	20.0451	3.0539
0.3246	61.2755	19.9200	3.0761
0.3064	61.1151	20.1117	3.0388
0.3116	61.3617	19.9005	3.0834
0.3131	61.2155	19.8350	3.0862
0.3223	61.1544	19.6815	3.1072
0.3221	61.2751	20.0476	3.0565
0.3404	61.2039	19.9052	3.0748
MEAN VALUES, wt.-%: 61.2270		19.9308	3.0721
STANDARD DEVIATIONS: ($\pm$)		($\pm$)	($\pm$)
Unbiased		0.0769	0.0216

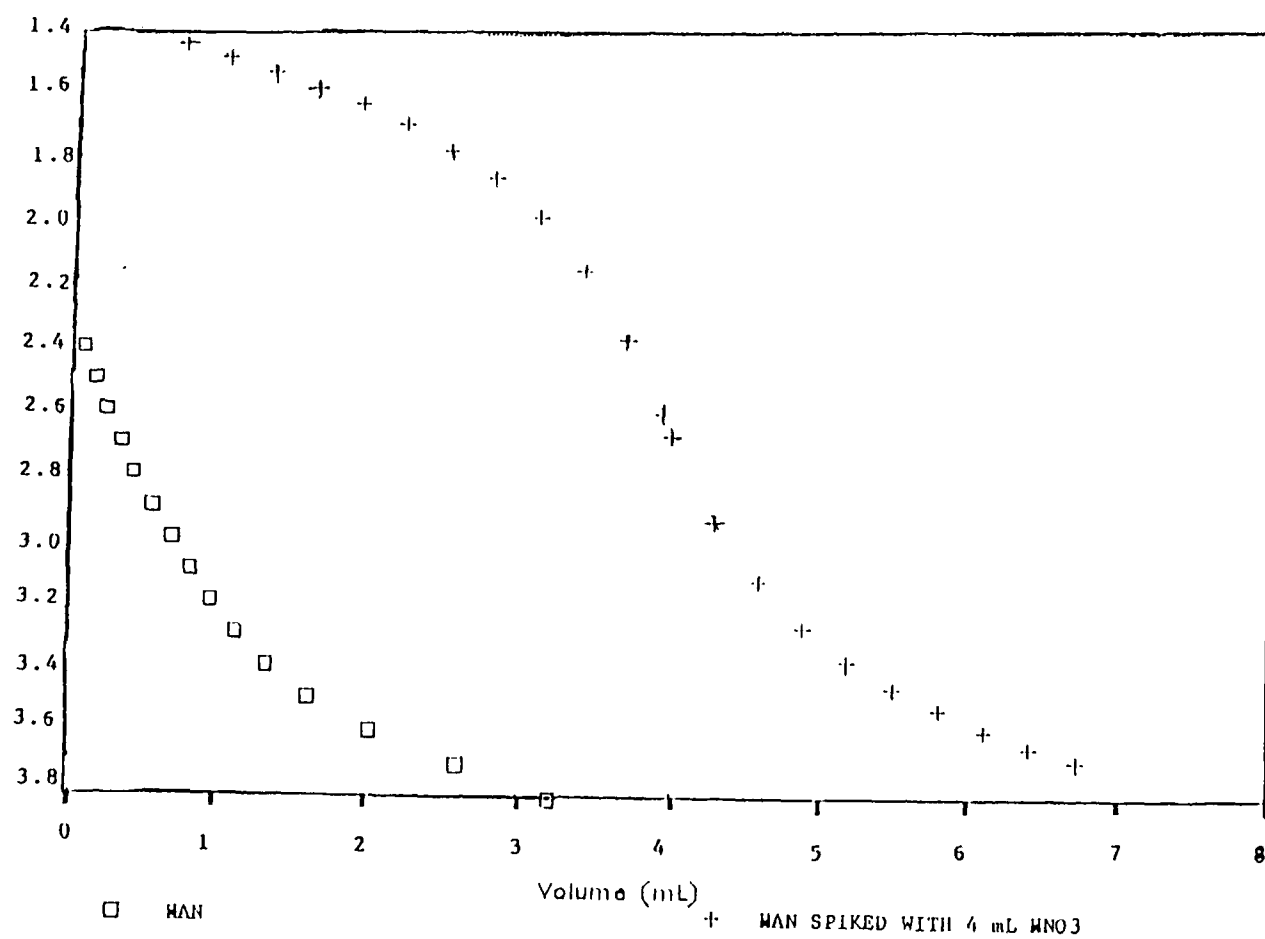


Figure 1. Titrations.

Table 3. Excess HNO_3 in LP-1846

SAMPLE MASS, g	Acid, wt.-%
35.8778	0.0030
35.9070	0.0016
35.8933	0.0024
35.9157	0.0014
35.9635	0.0042
MEAN VALUE, wt.-%: 0.0025	
STANDARD DEVIATION ^a : (±)	
Unbiased 0.00289	

^aQuoted precisions, shown in Tables 2 and 3, do not include the estimate for the precision of the standard base, which in this example was $0.2730(\pm)0.0021$ mol. No protocol has been established but it is suggested that a minimum of five samples be analyzed for qualification requirements.

As described, this spiking technique was successful in determining either excess acid or excess amine in LP and, obviously, this same method can be, and was, applied to make similar measurements for either HAN or TEAN solutions.

4. DETERMINATION OF TOTAL NITRATE

Total nitrate concentration is measured by ultraviolet spectrophotometer at 302 nm using 1.0-cm cells. The extinction coefficient was measured to be 7.225 l/mol-cm by Richard Biddle (1985) of Morton Thiokol Corporation, over the concentration range to 0.13 mol. In the narrow concentration range of 0.11 to 0.13 mol, Biddle also reported an extinction coefficient of 7.163 l/mol-cm. In neither case were individual values given nor were deviations reported. Thus, it is possible that the absorbance of nitrate is not a linear function of concentration or the results reflect his equipment.

Using a PERKIN-ELMER Model 3840 Lambda diode array visible/ultraviolet spectrophotometer at 302 nm, ten KNO_3 solutions to 0.1 mol resulted in an absorbance to concentration relationship in 1-cm cells of:

$$\text{abs} = 0.018 + (6.587 \pm 0.092) (\text{NO}_3^-). \quad (7)$$

The difference in the measured extinction coefficients may be due to the different instruments used and each experimenter should calibrate his own equipment.

Propellant samples were diluted to 1:100 parts by weight and 13 different propellant lots were analyzed. The ratios of the measured ultraviolet nitrate to the titration values for the sum of HAN, TEAN, and nitric acid were calculated. This ratio was 1.010 ± 0.014 , thus the titration values as well as the total ultraviolet nitrate-based values seem consistent.

5. WATER ANALYSIS

Karl Fisher methods were used to analyze water using Hydranal as titrant and acidic ethanol as solvent. The solvent was prepared using absolute ethanol made 10% in glacial acetic acid. One hundred ml were placed in a titration cell and the Hydranal was standardized with weighed 20 μ l additions of water. Propellant weighed samples of 100 μ l were analyzed. The cell was recharged with solvent for every two analyses. Results of seven aliquots of one propellant gave a water content of 20.18 ± 0.19 wt.-%.

6. ACCOUNTABILITY

The sum of the weight-percents of HAN, TEAN, nitric acid, and water, all determined by titration methods, was $99.83 \pm 0.95\%$ when combining the data for seven propellant lots. With the similar objective of accountability, the ultraviolet nitrate values were compared to the titration values and for seven propellant lots which resulted in a ratio of 101.55 ± 1.45 . The fact that we have accounted for the entire sample leads confidence to the procedures and tactics employed.

Evidence of accountability is also offered by physical measurement where the chemical composition, as determined by titration, was used to calculate density which was compared to

measured values. As stated earlier, the agreement was excellent being accurate to about a half of one percent.

7. OTHER METHODS

Some FTIR results have been published (Klein and Wong 1987); however, routine analytical methods have yet to be developed.

Metals in liquid propellant are generally analyzed by inductively coupled plasma spectroscopy (ICP), although a subset of metals, to include iron, can be adequately analyzed by atomic absorption spectroscopy. Methods are standard for either analysis and "qualification" limits have been set such that no metal impurity can exceed one part per million.

Ammonia is an expected by-product of HAN production and it can be measured in HAN solutions by direct, basic titration. However, in propellant, the pK's for ammonia and TEAN are too close to one another to allow this type of analysis. Instead, the standard micro Kjeldahl was performed by two BRL contractors, Olin Chemical Corporation. (Barnett, Dotson, and Leistra 1988) and ICT (Hansen, Backof, and de Greiff 1989), where ammonia was steamed distilled from basic propellant, collected, and titrated.

8. CONCLUSION

The analytical methods described have been polished over the last decade. They have been used to study and qualify over 15 Mg of LP. New methods may be developed in the future that may be more novel or convenient, but it is not clear that improved accountability will be achieved.

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APPENDIX: CALCULATIONS

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In order to separate the constitutive density effects of TEAN and HAN solutions, a liter of propellant consisting of two parts is considered: one of pure TEAN and the other a solution of HAN. These volume elements are noted as V_{TEAN} and V_{HAN-H_2O} .

To estimate the concentration of HAN in this idealized division, the volume of TEAN is subtracted from the propellant volume. Equations (3), (8), and (9) are offered to achieve this result. From the relationship:

$$M_1 V_1 = M_2 V_2 , \quad (8)$$

where $M_1 V_1$ represents HAN in the propellant and $M_2 V_2$ represents HAN in its subdivided volume element.

Expansion first yields,

$$M_1 V_1 = M_2 [V_1 - V_{TEAN}] ; \quad (9)$$

and further expansion yields,

$$M_1 V_1 = M_2 \left[V_1 - \frac{M_{TEAN}(MW_{TEAN})}{\rho_{TEAN}} \right] , \quad (10)$$

where M_{TEAN} is the number of moles of TEAN in a liter of propellant or 1.357 mol/l and MW is the molecular weight of TEAN 212.2022.

Substitution gives:

$$9.625(1000) = M_2 \left[1000 - \frac{1.357(212.2022)}{1.328} \right] . \quad (11)$$

Thus, the concentration of HAN in the divided system is M_2 or 12.2899 mol/l, and it as a volume of $V_1 - V_{TEAN}$ or 783.164 cm^3 .

The density of HAN at this concentration is given by applying the second order Equation 5 and is 1.50875. The density of propellant $\rho_{1,2}$ is then assumed to be the weighted average of its constituents or:

$$\rho_{1,2} V_{1,2} = \rho_1 V_1 + \rho_2 V_2 ; \quad (12)$$

expansion yields

$$\rho_{1845} = \rho_{HAN-H_2O} \left[\frac{V_{HAN-H_2O}}{V_{1,2}} \right] + \rho_{TEAN} \left[\frac{V_{TEAN}}{V_{1,2}} \right] ; \quad (13)$$

and substitution yields

$$\rho_{1845} = 1.50785 \left[\frac{783.164}{1000} \right] + 1.328 \left[\frac{1000 - 783.164}{1000} \right] ; \quad (14)$$

and, thus, the density of LP-1845 is calculated to be 1.46937 g/cm^3 .

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