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THERMAL CHARACTERISTICS OF CONCENTRATED HYDROXYLAMMONIUM NITRATE SOLUTIONS

RONALD SASSE

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I. INTRODUCTION

Liquid propellants have been, and are, a subject of active study at the Ballistic Research and other Laboratories for a number of years. A review paper has been written¹ and several recent papers have been published² but much additional and important work is constantly appearing. One candidate system chosen for extensive study is identified as 1845 which is a homogeneous mixture of 9.626 M hydroxylammonium nitrate (HAN), 1.380 M triethanolammonium nitrate (TEAN) and 13.644 M water. This mixture of nitrate salts has the appearance of an aqueous solution but has properties more closely akin to molten salts. To formulate this system, concentrated solutions of HAN were prepared by vacuum evaporation techniques and it was soon found that HAN, at room temperature, was miscible with water in all proportions. This is a unique solubility characteristic for a nitrate salt that has allowed fundamental physical property and structure studies over the entire concentration range from dilute solutions to pure salt. The thermal response of HAN solutions and liquid propellants has been extensively studied using closed bomb techniques at low loading densities³ where typically two or less milliliters have been placed in a 50 cm³ 316 stainless steel AMINCO bomb designed to withstand 100 MPa (15 kpsi). In these closed bomb studies product gases were confined, resulting in high pressure pulses and high temperatures. Such studies offered a plethora of information, but the thermal response of HAN and propellant solutions at atmospheric pressure in open systems has received little attention and this paper attempts to fill this void.

II. EXPERIMENTAL

The boiling points of HAN solutions at various concentrations were measured at atmospheric pressure by placing a glass enclosed chromel-alumel, "type K", thermocouple into approximately 10 ml of solution and measuring response on a 10 mV recorder as heat was supplied to a 30 ml round bottom flask by a heating mantle. Temperatures were measured to within a quarter of a degree centigrade. Small sample volumes and semi-micro glassware were selected to limit any damage that might have resulted from long heating experiments leading to a "fizz" reaction, a term used to describe an exothermal excursion consuming all starting materials. To limit possible damage, heating periods were limited to about three hours. The boiling flask was left open for one set of experiments and it was fitted with a 10 cm long West reflux condenser for a second set of refluxing experiments.

III. RESULTS

A typical temperature history is shown in Figure 1 for refluxing a 12.0 M HAN solution. In these experiments a small amount of liquid, termed hold-up-volume, accumulates in the condenser and when this capacity is filled, liquid returns to the flask. A second sample of 12.48 M HAN was refluxed and the flask was weighed and analyzed before and after a 2.5 hour refluxing period. At the start of the experiment the refluxing temperature was 132°C and remained near this value during the early refluxing time period; thereafter, the temperature decreased slowly and was 129°C at the end of the experiment. Chemical analysis was performed by Madelyn Decker of our Laboratory who also provided the HAN solutions used in this study. The analyses included a Karl Fisher water determination and a basic titration using tetrabutylammonium hydroxide to measure HAN.⁴ This titration would have shown the presence of

strong acids but none were found. Neither were compounds having a large pK like NH_4^+ . Other results showing depletion of HAN and the concurrent production of water are given in Table 1. The slight increase in the weight of water shown in the table does not include the small amount required to fill the hold-up-volume of the condenser. The weight changes shown in the table are the differences between averages of the values noted.

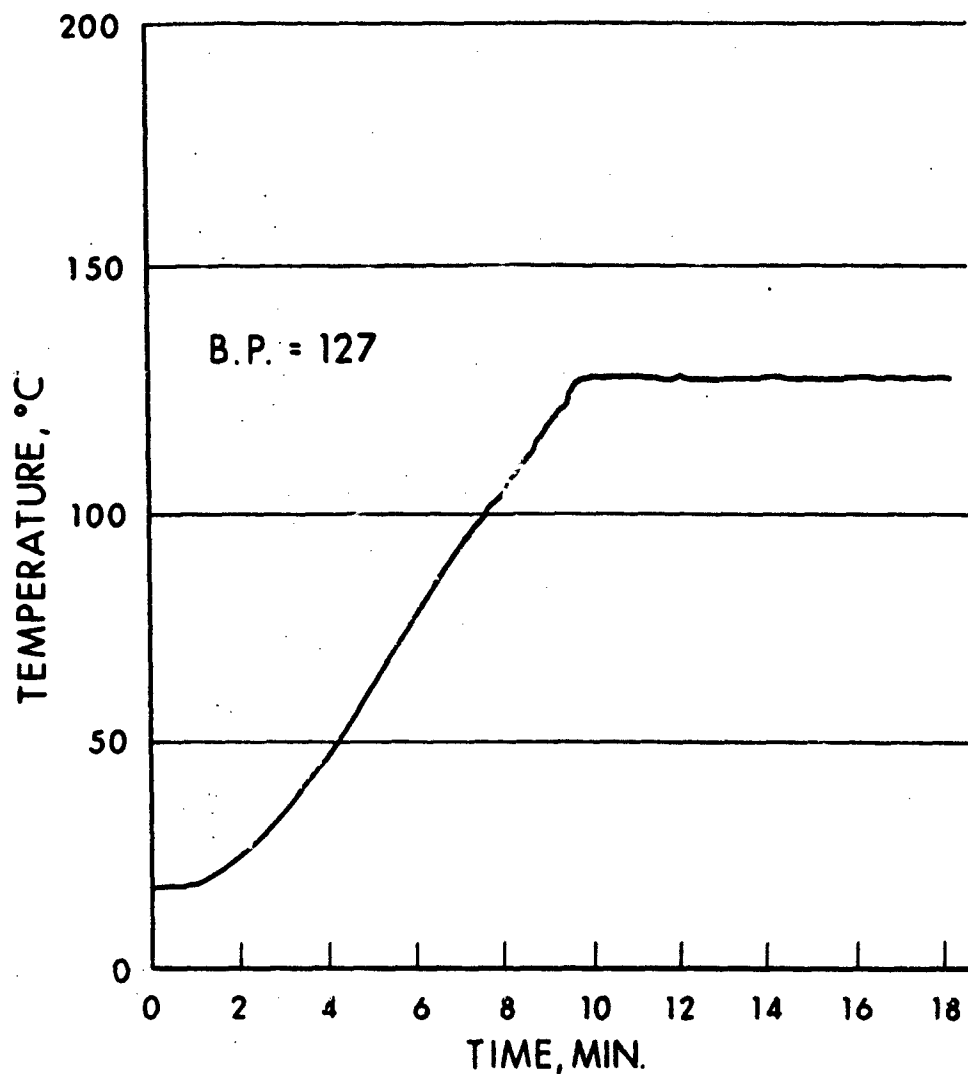


Figure 1. Temperature History of 12.48 M HAN Using a Reflux Condenser

Table 1. Thermal Decomposition of 12.48 M HAN Solution

Sample Description	Weight in Grams		
	Total	HAN	Water
Before Reflux	12.4043	9.6188	2.4317
		9.7938	2.4412
After Reflux	11.6286	9.1030	2.5697
		9.0773	2.5596
Weight Change	-0.7757	-0.6151	+0.1417

These experiments show but a 6 percent decrease in HAN concentration due to prolonged (2.5 hour) refluxing at 132-129°C with a very small amount of water production. The values presented in Table 1 suggested some thermal decomposition which could involve gas production. With this in mind, the experiment was repeated with the reflux condenser being first flushed with helium, and the gases were collected over water. A 50 cm³ buffer volume was placed between the reflux condenser and the water buret such that pure helium was initially displaced. Data are presented in Figure 2. At early times, an inordinate amount of gas was measured as a result of expansion of the vapors as they came to the vessel's temperature.

Gas samples were taken at the exit of the reflux condenser and before the gas collection system for analysis by gas chromatography. Samples were taken every 20 minutes. The gases nitrogen, N₂, and nitrous oxide, N₂O, were found in the molar ratio of 1:2, a result previously found in closed bomb studies for the "fizz reaction".⁵ Considering the total volume of gas produced, about 84 cm³, and the moles of HAN removed, 0.0064 M, one sees that that for every mole of HAN removed about 0.39 M N₂O and 0.19 M N₂ were produced; these ratios are also in accord with those previously reported.⁵

From the fact that the HAN solution did not change appreciably during refluxing, and that the refluxing temperature was nearly constant it is concluded that the boiling point was measured. It was thought that similar values could be obtained by studying an open (non-condenser) system and the results are shown in Figure 3 where the sharp change in slope was taken as the boiling point. The agreement between the two techniques was adequate. Several HAN concentrations were evaluated; boiling points for the different HAN solutions are given in Table 2. As the open system is heated longer, water vapor escapes and the HAN solution becomes more concentrated. This increase in concentration induces higher boiling points and this temperature increase promotes the rate of exothermic chemical reactions as indicated by the increasing slope before 145°C; thereafter, this process accelerates even more and the temperature history becomes asymptotic to a temperature excursion near but less than 200°C. At this temperature vigorous reactions take place and brown fumes are observed; obvious indicators of what is normally called a "fizz reaction".

This same open vessel and refluxing technique gave a boiling point of 127°C for the propellant 1845.

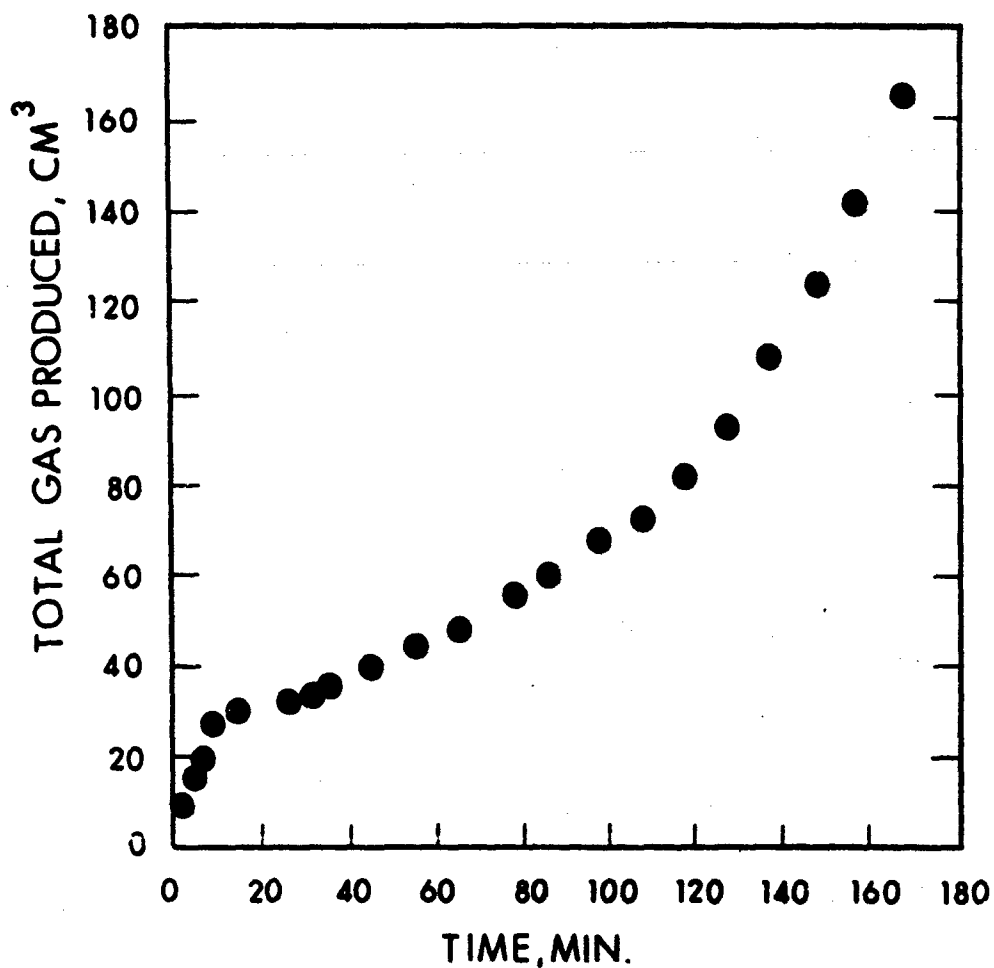


Figure 2. Total Gas Produced While Refluxing 12.48 M HAN

Table 2. Boiling Points for Different HAN Concentrations

<u>HAN</u> M	<u>Water</u> M	<u>Boiling Point</u> °C	<u>HAN</u> Mole Fraction
0.0	55.56	100	0.0000
4.0	43.72	107	0.1547
9.0	28.99	115	0.3831
12.0	20.16	127	0.5435
12.48	16.72	132	0.5848
13.0	17.21	129	0.6017
13.0	17.21	134	0.6017
15.8	8.97	145	0.7789

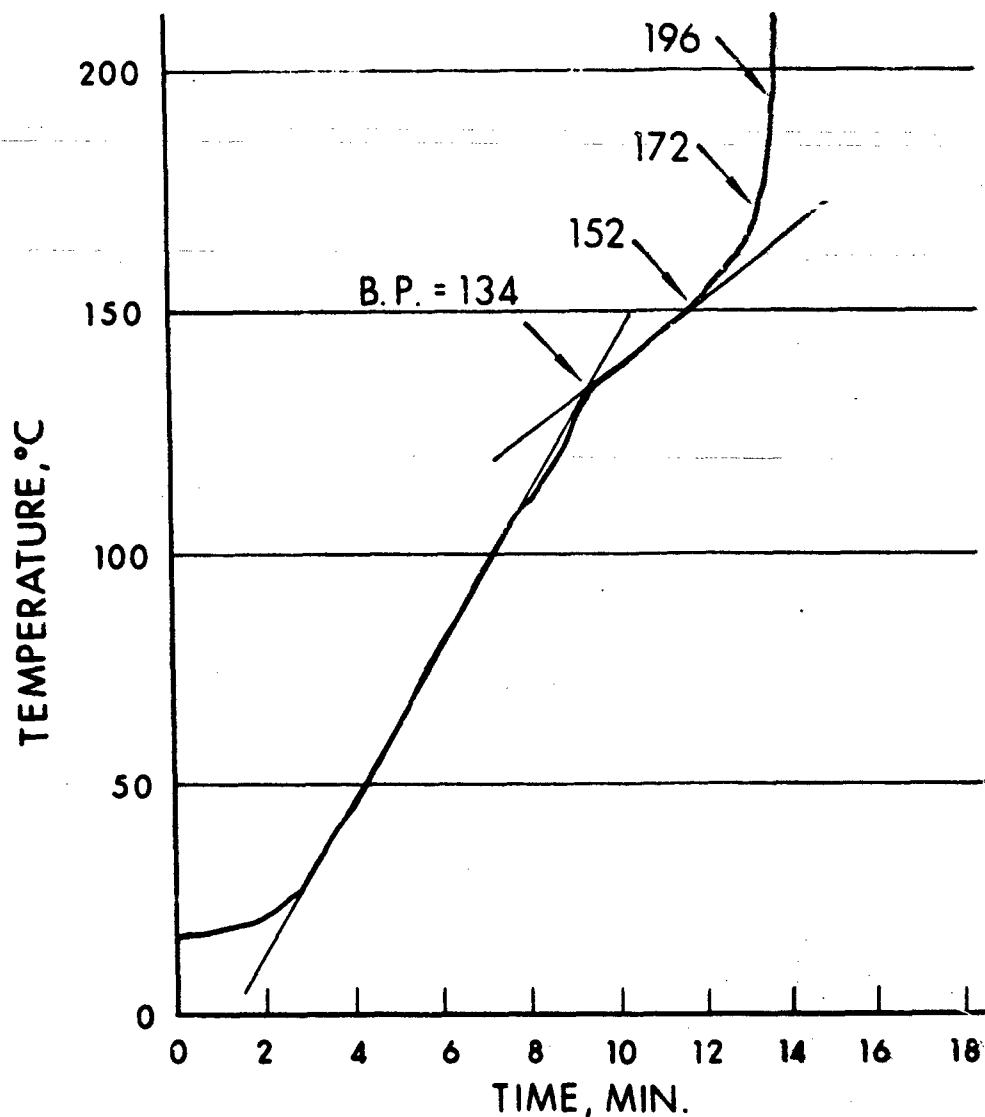


Figure 3. Temperature History of 13.0 M HAN Heated in an Open Vessel

IV. DISCUSSION

The boiling points of the HAN solutions (Table 2) have been evaluated by the Clausius-Clapeyron equation generally applicable to dilute (less than one molar) solutions. This equation relates boiling point elevation, $T_2 - T_1$, to the heat of vaporization of the solvent, ΔH_{vap} , and the mole fraction of dissolved species, X_B or the mole fraction of water X_A . Since HAN forms two ions in solution, the coefficient two appears in the equation below for the mole fraction term.

$$-\ln X_A = -\ln (1-X_B) = -\ln \left[1 - \frac{2n_2}{2n_2+n_1} \right] = \frac{\Delta H_{\text{vap}}}{R} \cdot \frac{T_2-T_1}{T_2 T_1} \quad (1)$$

where: T_1 is the boiling point of pure water and T_2 is boiling point of the solution in degrees K, R is the gas constant; $1.987 \text{ cal mole}^{-1} \text{ degree}^{-1}$, and the accepted value for the heat of vaporization for water, ΔH_{vap} is $9720 \text{ cal mole}^{-1} \text{ degree}^{-1}$. n_2 is the HAN molar concentration and n_1 is the molar concentration of water.

A plot of $\Delta T/(T_2 T_1)$ vs. $-\ln (1-X_B)$ was fitted to a straight line whose slope was equated to $R/(\Delta H_{\text{vap}})$. Linear regression gave the slope from which ΔH_{vap} for the solvent water was calculated to be $10,162 \text{ cal mole}^{-1} \text{ degree}^{-1}$ with a standard deviation of 10%; at the 95 confidence level the deviation is greater. This construction is given in Figure 4 and the straight line drawn was obtained using the accepted value of ΔH_{vap} for water. The data are limited but they are consistent with this line.

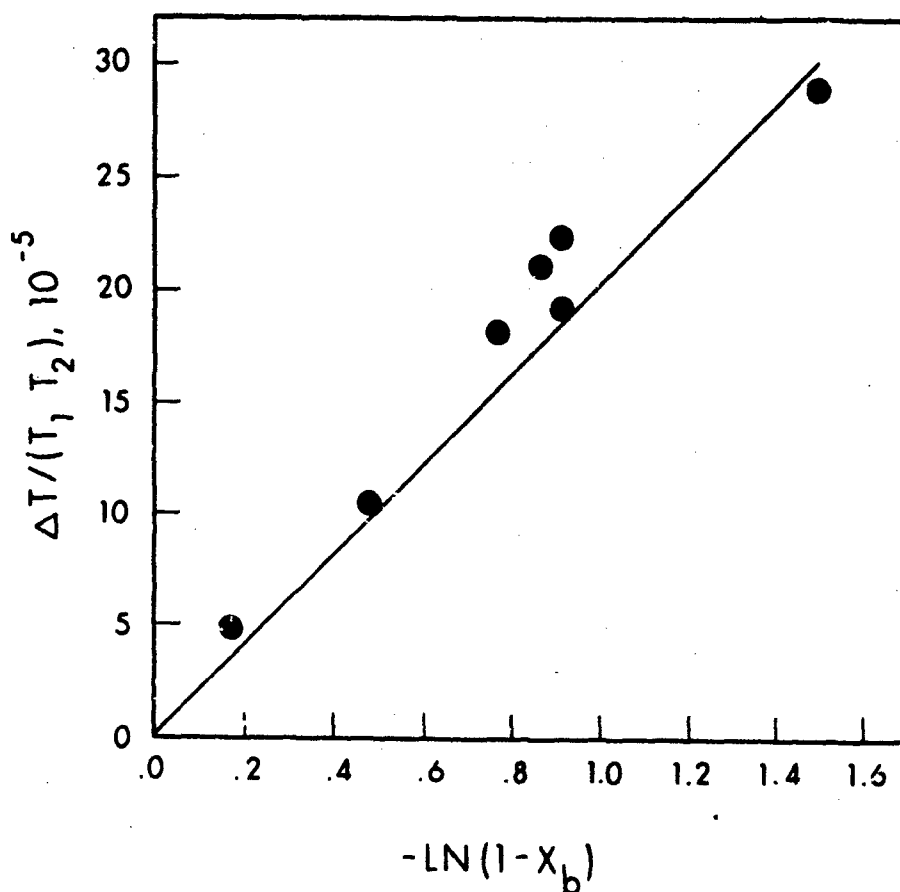


Figure 4. Evaluation of Boiling Points of Different Concentrations of HAN

It is surprising that the heat of vaporization of water can be estimated from the behavior of such concentrated solutions; however, from vapor pressure measurements of propellant solutions, in the same concentration range, ΔH_{vap} for 1845 and 1846 was found to be 9842 and 9761 cal mole⁻¹ degree⁻¹ by N.A. Messina of Princeton Combustion Research Laboratories, NJ, whose data has been previously reported.⁶ Moreover, the boiling points of these propellants were predicted, also from these vapor pressure measurements,⁶ to be 126.5 and 123.7°C. In the present study the boiling point of 1845 was measured to be 127 ± 0.25 which can be directly compared to the predicted value of 126.5°C.

The question has arisen in our Laboratory as to whether the boiling point of these solutions was measured, as opposed to the measured temperature being that for the onset of gas producing reactions. This study showed that the extent of chemical reaction was small, in that the starting materials decreased by a small amount and gaseous products were also produced to a limited amount even after prolonged refluxing. Further, the boiling point data appears to follow a linear molar relationship over the concentration range studied, and has resulted in a reasonable value for the heat of vaporization for water. Perhaps the most satisfying argument is the agreement of the predicted and measured boiling points for propellant 1845.

This author concludes that the experiments described reflect boiling phenomena and from the long refluxing experiment total gas yields are given in Figure 2. This data has been replotted as a first- or pseudo- first order reaction in Figure 5. The accelerating nature of the gas production may indicate that the concentration of some intermediate is increasing as a function of time. One possibility would be strong acid build-up but, as mentioned, none was detected. Had heating continued to accelerate the reaction, then at later times, a "fizz" reaction would have resulted. The data presented is too singular to be used as a basis for a kinetic conclusion but it is suggestive of an area worthy of further study.

The reflux and open vessel boiling experiments reflect an entirely different environment from the closed bomb work described previously and the similarity of the gas product ratio of N₂/N₂O of 1/2 was unexpected.

As a final aside and in the interests of safety, it is pointed out that in one reflux experiment an oxygen delivery tube was inserted into the hot vapor region above the boiling liquid and a "fizz" reaction soon resulted. This effect was not investigated further but it is emphasized that refluxing data were obtained in a helium atmosphere.

All of the experiments reported in this paper have been conducted at atmospheric pressure and in open vessels where gas products can escape. In contrast, under confined conditions HAN solutions have been extensively studied in closed bombs where it was found that initiation temperatures decreased, from 200 to 120°C, as HAN concentration increased from 2 to 13 M.³ These different relationships reflect vastly different transport properties between open and closed systems and the reader is cautioned that the thermal characteristics reported for HAN solutions should not be scaled or applied to any but identical conditions. Even though boiling points and initiation temperatures have been cited, they reflect experiments conducted in relatively short time periods and the experiments do not relate in any way to the

question of storage stability or to the safety of heating large volumes of material where transport properties could play a crucial role.

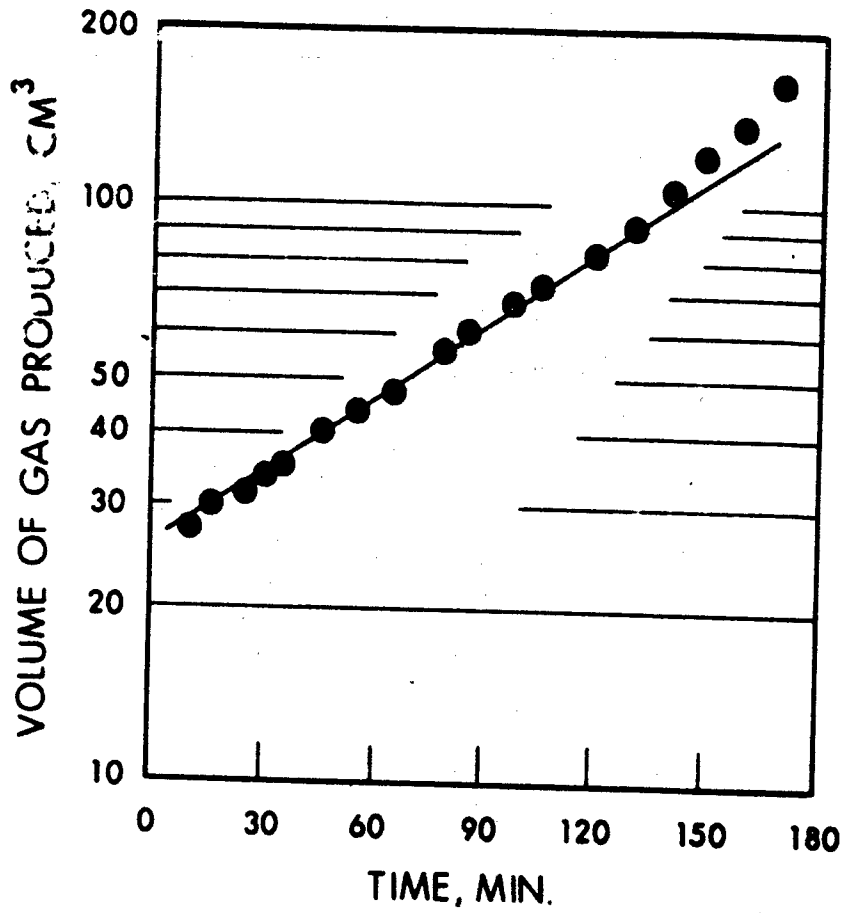


Figure 5. First Order Presentation of Gas Product Yield

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