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Experimental Studies of the Reactions of N_2O_5 With Metal Acid Anhydrides and BF_3

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-Date-

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INTRODUCTION

The major purpose of this project was the preparation, purification and determination of properties of the oxynitrates of chromium, molybdenum and tungsten. Various refinements of the methods made it possible to increase the purity and yield of the products.

The project also yielded information which will be valuable for future investigations.

A. Dinitrogen Pentoxide:

I. Preparation -

The method used to prepare N_2O_5 was based upon the reaction in the gas phase of ozone with N_2O_4 according to the following equation:

$$N_2O_4(g) + O_3(g) = N_2O_5(s) + O_2(g)$$

The system used is described as follows:

Oxygen which has been dried by running through two tubes of P_2O_5 is introduced into a Wellsbach model T23 Ozonator at a pressure of 8 psi. A voltage of 110±5 volts is applied to generate the arc which produces the ozone. The resulting mixture of ozone and oxygen is then bled into the system at 0.09 SCFM and allowed to mix with the N_2O_4 which has been dried by passing through anhydrous $CaCl_2$.

This gaseous mixture is allowed to pass through a reflux condenser packed with glass helices and then is passed into two traps surrounded by baths of a dry ice slush in methanol.

This operation is continued for approximately ten hours. The yield is between 25 and 30 grams of N_2O_5 with slight contamination by NO_2 as evidenced by a slight yellow color.

This product is then distilled from the traps into the reaction vessel by passing a mixture of ozone and dried oxygen over it while gently heating the solid to assist in sublimation. This heating is accomplished by a controlled water bath. The temperature is not allowed to exceed 75°C even though the N_2O_5 did not readily sublime even at this temperature.

II. Purification -

Purification of the N_2O_5 produced by the above method maybe easily accomplished by redistillation under an atmosphere of oxygen-ozone. Very pure N_2O_5 has been prepared this way and was condensed out in the shape of rombohydral colorless clear crystals approximately 2 mm in dimension.

III. Properties -

The N_2O_5 produced in this manner had properties similar to those already reported in the literature.

IV. Difficulties Encountered -

Only one major difficulty was encountered in the preparation of the N_2O_5 in this manner. Approximately one-third of the time the N_2O_5 produced was condensed as a white waxy like material which adhered to the glass containers and would not melt or sublime readily even at 75°C. The reported melting point of N_2O_5 is 31°C and the boiling point is reported at 47°C. This white waxy like material would slowly decompose to N_2O_4 as evidenced by the production of brown gas. It often remained at room temperature for a week before complete decomposition. This material when introduced in a reaction performs as N_2O_5. This compound will be investigated further.

B. Chromyl Nitrate:

I. Preparation -

A measured quantity of chromic anhydride was placed in a 250 ml. two necked flask fitted with a delivery tube from the N_2O_5 generator and a claisen head, condenser, adapter and receiver. While pulling a vacuum on this system the solid chromic anhydride was heated to 120°C for six hours.

During this period of drying the N_2O_5 was produced. After allowing the chromic anhydride to cool to room temperature, the N_2O_5 was transferred under dry oxygen onto the dried chromic anhydride. These two solid material were allowed to react for at least forty-eight hours at room temperature. The reaction proceeds according to the following equation:

$$CrO_3 + N_2O_5 \dashrightarrow CrO_2(NO_3)_2$$

II. Purification -

The product should be purified immediately after it is made since the dissolved impurities tend to catalyze the decomposition of the material into starting products.

Redistillation over PbO_2 led to a pure material. This distillation then yielded a clear dark red liquid when distilled in a vacuum of approximately 5 mm at a temperature range of 45-50°C. Some decomposition occured during purification and the final yield was about 65%.

III. Properties -

Chromyl nitrate is a clear dark red liquid which can be distilled and poured with ease. The boiling point is 45-50°C at 2 mm pressure. It fumes profusely in moist air and reacts instantaneously with water to produce a mixture of chromic and nitric acids. It will attack wood, paper, rubber, floor tile, and some types of enameled metal surfaces. Stainless steel is not readily attacked.

IV. Experiments performed -

(a) Preparation of a titanium complex:

A dilute solution of chromyl nitrate was reacted with titanium tetrachloride and a yellow precipitate occurred which was unstable in moist air. The carbon tetrachloride solution changed from red to a brown color. Preliminary analysis indicates that this new compound is a titanium peroxide found according to the reaction

$$TiCl_4 + CrO_2(NO_3)_2 \longrightarrow CrO_2Cl_2 + TiCl_2O_2 + N_2O_4$$

Further work on this peroxide is under way.

(b) Preparation of an antimony complex:

A dilute solution of chromyl nitrate was reacted with antimony pentachloride and a yellow precipitate occurred which was unstable in moist air. Again the carbon tetrachloride solution turned from red to brown. No reasonable analytical data have been obtained on this compound yet. Further work on this compound is in progress.

(c) The following ignition reactions were attempted:

In each case one milliliter samples of the hydrocarbon were used and 3-5 drops of chromyl nitrate.

a) Parafin oil - ignites
b) gasoline - ignites
c) 1-hexene - ignites
d) heptane - ignites

(d) One milliliter of parafin oil was added to one milliliter of a dilute carbon tetrachloride chromyl nitrate solution. No ignition occurred and no evidence of a reaction was observed.

V. Spectrum -

The spectrum of chromyl nitrate was run by the capillary film method between two sodium chloride plates on the Beckman IR 5. It agreed completely with the spectrum previously obtained by the Denver Research Institute.

VI. Difficulties encountered in the preparation of $CrO_2(NO_3)_2$ -

Attempts to prepare chromyl nitrate in a system which was not all glass failed. The ozone and N_2O_5 will attack even tygon tubing producing a sticky substance which interferes with the reaction.

If the N_2O_5 is distilled into the reaction vessel under an atmosphere of ozone the CrO_3 forms a peroxychromate which will not react with N_2O_5.

Distillation of chromyl nitrate in high vacuum produces a rapid decomposition to CrO_3 and N_2O_4 with oxygen being liberated. The distillation temperature should be no lower than 45°C and the vacuum should be regulated accordingly.

The chromyl nitrate should be stored in a container in such a manner that it does not contact even Kel-F polyfluorinated grease since it will attack this and form a dark, and hard sludge which will prevent the manipulation of any sort of joint.

C. Vanadyl Oxytrinitrate:

I. Preparation -

The vanadyl nitrate produced on this project for study purposes was made in the same manner as was chromyl nitrate.

$$V_2O_5 + 3N_2O_5 \longrightarrow 2VO(NO_3)_3$$

The V_2O_5 was dried in vacuum as was the CrO_3. The only difference was that the N_2O_5 was distilled into the reaction vessel under an atmosphere of ozone-oxygen mixture since ozone seemingly does not react with V_2O_5 to give stable peroxyvanadates.

The reaction mixture was allowed to stand for forty-eight hours after which time the vessel contained a viscous very pale yellow liquid with a boiling point of 86-91°C at 0.7 mm vacuum.

II. Purification -

Purification of vanadyl nitrate may be easily accomplished by a straight vacuum distillation since it is much more stable than chromyl nitrate. It was not found necessary to use PbO_2 to absorb the N_2O_5 decomposition products.

III. Properties -

Vanadyl nitrate is a clear, very pale yellow, viscous liquid which can be distilled and poured quite easily. The boiling point was 86-91°C at 0.7 mm pressure. It is less easily hydrolysed than chromyl nitrate; it even can be poured in air if done quickly, without too much evidence of contamination by hydrolysis products.

IV. Reactions -

a) Preparation of a BF_3 addition compound:

A dilute solution of vanadyl nitrate in anhydrous CCl_4 was treated for approximately 2 hours, with boron trifluoride. The solution became almost colorless and a pale yellow flocculent precipitate was formed. This precipitate was unstable in air and hydrolyzed readily. The ratio of reactants was $BF_3 \cdot VO(NO_3)_3$ or 1:1.

Purification was difficult since the compound dissociated in most solvents. Further work is planned on this compound.

Vanadyl nitrate was allowed to react directly with water. The solution became strongly acidic and a precipitate formed which was a polymeric form of vanadium oxide.

b) One milliliter of vanadyl nitrate was added to ten milliliters of anhydrous carbon tetrachloride. To this solution was added approximately 0.1 grams of the $N_2O_5 \cdot BF_3$ addition compound. Initially no evidence of reaction could be observed. After approximately 20 minutes a white flocculent precipitate started to form. Most of the $N_2O_5 \cdot BF_3$ solid did not dissolve. The flocculent precipitate was very hygroscopic and dissociation occurred in most solvents. Further work is planned on this compound.

V. Ignition Reactions -

Ignition reactions which were attempted with vanadyl nitrate are as follows:

a) Parafin oil - no ignition
b) Gasoline - ignition
c) 1-hexene - ignition
d) heptane - no ignition

It is apparent from these above ignition reaction that some unsaturation is necessary for ignition to occur with vanadyl nitrate. This phenomena is in agreement with its higher stability compared to chromyl nitrate.

VI. IR Spectrum -

The spectrum of vanadyl nitrate was run by the capillary film method between two sodium chloride plates on the Beckman IR 5. It was very similar to that of chromyl nitrate except that the metal oxygen bond was different. Previously a spectrum of vanadyl nitrate had been published and a comparison has been made. The comparison was favorable.

VII. Difficulties encountered in the preparation of $VO(NO_3)_3$ -

After the system had been built for the production of chromyl nitrate, no further difficulties were encountered in the preparation of vanadyl nitrate. Yields up to 85% were obtained. Passage of ozone over V_2O_5 does not seem to interfere with its reaction with N_2O_5.

D. Molybdyl Dioxydinitrate:

I. Preparation -

To prepare molybdyl nitrate the same apparatus was used as in the preparation of the other two previously described acyloxynitrates. Since MoO_3 is too stable to react with the dinitrogen pentoxide, the starting material was molybdenum dioxydichloride. The reaction then proceeds according to the following equation:

$$MoO_2Cl_2(s) + 2N_2O_5(s) \dashrightarrow MoO_2(NO_3)_2(s) + 2NO\,Cl(g)$$

The dinitrogen pentoxide was prepared in the same manner as has been previously described. It was distilled into the reaction vessel under an atmosphere of oxygen containing 6% oxone. To this condensed solid was added 125 ml. of anhydrous carbon tetrachloride. After all the N_2O_5 had dissolved, the solid MoO_2Cl_2 was added and the reaction was allowed to proceed for forty-eight hours. To assure intimate contact between the solid and solution, the slurry was stirred magnetically.

After the reaction was complete, the pale yellow precipitate was collected by decanting the excess solution of N_2O_5 in anhydrous CCl_4. The precipitate was washed with two portions of 100 ml. of anhydrous CCl_4. It was then collected in an atmosphere of dry nitrogen on a fritted glass funnel and stored in a dry ice chest in a glass stoppered bottle filled with nitrogen. Analysis indicated that the compound was $MoO_2(NO_3)_2$.

II. Purification -

No real attempt to purify the product was made since only primary experiments were to be performed. Analysis indicated that washing with anhydrous CCl_4 was sufficient for a purification procedure.

III. Properties -

Molybdyl dioxydinitrate, is a finely divided crystalline, pale yellow solid which fumes in air liberating N_2O_4 and forming the more deeply green-yellow molybdenum trioxide. No attempt was made to determine the exact melting (or decomposition) point. It was insoluble in nonpolar halogenated hydrocarbons. It was very stable when stored at low temperatures and decomposed only very slowly in an inert atmosphere, in the absence of light and at room temperature.

IV. Ignition Properties:

The only experiment performed with the molybdyl nitrate were ignition experiments. They are:

a) gasoline - ignition
b) 1-hexene - ignition
c) heptane - no ignition

The ignition properties of molybdyl nitrate are very much in line with its greater stability as compared to chromyl nitrate. They are very similar to those of vanadyl nitrate as would be expected from the greater stability of its higher oxidation state.

V. IR Spectrum -

The spectrum of molybdyl nitrate was not obtained because of its rapid rate of hydrolysis.

VI. Difficulties encountered in the preparation of $MoO_2(NO_3)_2$ -

This reaction is a two phase reaction and even after forty-eight hours of reaction time and continuous stirring, the reaction had not gone to completion. The compound is extremely sensitive to moisture.

E. Tungstyl Dioxydinitrate:

I. Preparation -

Tungstyl dioxydinitrate was prepared by the same method as molybdylnitrate. The starting material was tungsten hexachloride. The reaction proceeds according to the following equation:

$$WCl_6(s) + 4N_2O_5(s) \text{------}\rightarrow WO_2(NO_3)_2(s) + 6NOCl(g) + 3O_2(g$$

The reaction was carried out in anhydrous carbon tetrachloride. The product was a finely divided white crystalline solid.

II. Purification -

Analysis indicated that two washings with anhydrous carbon tetrachloride were sufficient to remove any byproducts.

III. Properties -

The properties are almost identical with those of molybdyl nitrate. The compound is a white finely divided crystalline precipitate.

IV. IR Spectra -

The IR spectra will be obtained on new samples which are in the process of being prepared.

F. $N_2O_5 \cdot BF_3$ Addition Compound:

I. Preparation -

For the preparation of the $N_2O_5 \cdot BF_3$ addition compound the same apparatus was used as in preparing the acylnitrates.

The N_2O_5 was prepared as previously described. After having distilled the N_2O_5 into the reaction vessel, 125 ml. of anhydrous CCl_4 was added. After complete dissolution of the N_2O_5 the reaction vessel was immersed in an ice-water bath to prevent the temperature of the reaction from going above 25°C.

A gas delivery tube was placed into the solution very near the bottom and BF_3 was bubbled through the solution for about thirty minutes. During this time a large quantity of white waxy like precipitate formed. It was collected on a buchner funnel and washed with anhydrous CCl_4.

II. Purification -

The addition compound was purified by washing three times with CCl_4.

a) Ignition test:

The $N_2O_5 \cdot BF_3$ is a white waxy like material which is insoluble in halogenated hydrocarbons, and ignites hydrocarbons[1].

b) Hydrolysis:

Hydrolysis of $N_2O_5 \cdot BF_3$ yields boric acid, nitric acid and hydrofluoric acid. It did not ignite hydrocarbons or oxygenated hydrocarbons.

The compound is a one to one addition complex of the following structure:

$NO_2^+ \ BF_3NO_3^-$

It is a powerful agent for producing nitro derivatives of hydrocarbons, especially aromatic hydrocarbons[1]. No further studies were carried out on this compound.

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1. J. Am. Chem. Soc., 80 5871, 1958.

G. Decomposition Tests:

I. Preparation of $CrO_2(NO_3)_2$ and $VO(NO_3)_3$ -

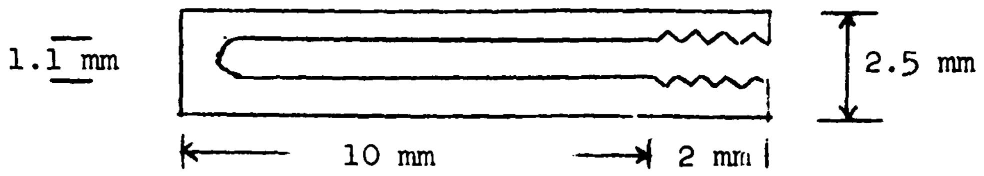
Samples of $CrO_2(NO_3)_2$ and $VO(NO_3)_3$ were prepared for use in pressure cylinders to determine the manner of the decomposition of these compounds. The $VO(NO_3)_3$ was prepared from V_2O_5 and N_2O_5 and the $CrO_2(NO_3)_2$ from CrO_3 and N_2O_5.

In the preparation of these compounds several interesting results were obtained.

In order to produce a pure product and larger yield of $CrO_2(NO_3)_2$ and $VO(NO_3)_3$ some alterations from the previous experiments were attempted. The N_2O_5 produced from N_2O_4 and O_3 was condensed in an atmosphere of ozone, O_3. It was hoped that the decomposition of N_2O_5 could be reduced in the presence of O_3. It was hoped that an atmosphere of O_3 might aid in its stabilization. Favorable results were obtained in this respect with V_2O_5 yielding pure $VO(NO_3)_3$. With CrO_3, however no $CrO_2(NO_3)_2$ was obtained. In the presence of O_3 no reaction between N_2O_5 and CrO_3 seems to take place. The O_3 seems to react with the CrO_3 surface layer giving a peroxide of chromium, a "peroxy-chromate". This compound seems to be stable. Further investigations on this compound are being planned.

II. Apparatus -

The samples were sealed in type 316 stainless steel pressure cylinders which were constructed by drilling wells into solid metal stocks. The wells were 12 mm deep and 1.1 mm in diameter. The tops of the cylinders were threaded with 0.5 inch pipe threads for a distance of 2 mm.



1/2 in. thread

Volumes of the cylinders were determined by filling them with a measured vol. of CCl_4. All cylinders measured 10 cc. To each cylinder was attached a pressure gauge with release valve attached to a T-joint. Gauges read from 0 to 60 lbs/sq. in. Gauge volumes, as measured with CCl_4, varied from 20 to 25 cc. Pipe threads were sealed with DuPont teflon tape dope.

A constant temperature water bath set at 25°C with a mercury regulator and stirred by an electric stirrer was set up to maintain the cylinders at constant temperature.

III. Procedure -

The pressure cylinders were filled in a dry box under an atmosphere of nitrogen by the use of a graduated syringe with stainless steel needle. Sample sizes were from 3 to 5 ml for $CrO_2(NO_3)_2$ and 3 to 6 ml for $VO(NO_3)_3$. The filled cylinders were placed in a 25°C water bath and readings were taken every 12 hours.

The samples with $VO(NO_3)_3$ were run for 7 days and no significant pressure readings were obtained. This compound when sealed in a stainless steel container under a dry inert atmosphere does not decompose readily.

Two samples of $CrO_2(NO_3)_2$ were run for 30 days, one sample with 3 ml and one with 4 ml. Graphs 1 and 2 show the semi-log plots for these samples with log of time (in days) vs. the reciprocal of pressure, in atmosphere. Graphs 3 and 4 are plots of pressure (psi) vs. time (days) for the same samples.

Another set of pressure cylinders was filled with $CrO_2(NO_3)_2$ to which PbO and PbO_2 was added. Some moisture or other impurities seem to have been present in the oxides since no reproducible results could be obtained.

IV. Discussion -

A continuous increase in the pressure of the $CrO_2(NO_3)_2$ cylinders is in line with the known instability of the compound. The larger (4 ml) sample shows a more rapid increase in pressure than the 3 ml sample. The 3 ml sample increased less rapidly but showed the same pressure characteristics. A comparison of the two samples must take account of the ullage present since aside from the relative comparison of the sample sizes, the effect of increased gas space above the smaller sample is important. It is known that enlarging the gas space above the sample considerably increased the decomposition rate.

In the experiment the combined volume of the gauge and cylinder was 35 cc, 4 cc were displaced by one sample and 3 cc by the other. Thus, the smaller sample had approximately 3% more gas space. Pressure results between graphs 3 and 4 therefore cannot be quantitatively compared since the larger sample gives off a larger volume of gases into a smaller gas space. As a result, the larger sample gives a greater pressure reading although presumably its rate of decomposition is less.

Graphs 1 and 2 show an interesting similarity if the shape of the graphs are compared disregarding the relative pressure reading. It is noted that on both these graphs two breaks in the slope occur, one at slightly over 2 days and another at about 8 days. The change in slope might be related to a change in the rate of decomposition as effected by a shift of equilibrium due to changes in concentration and/or partial pressures of the gas phase.

In its gas phase $CrO_2(NO_3)_2$ is in equilibrium with the starting materials N_2O_5 and CrO_3.

$$CrO_2(NO_3)_2 \rightleftharpoons CrO_3 + N_2O_5$$

In turn, N_2O_5 can decompose through several reactions

$$N_2O_5 \rightleftharpoons NO_2 + NO_3$$

$$NO_2 + NO_3 \rightarrow NO + O_2 + NO_2$$

$$NO + NO_3 \rightarrow 2\ NO_2$$

$$2NO_3 \rightarrow 2NO_2 + O_2$$

$$N_2O_4 \rightleftharpoons 2NO_2$$

The total pressure above the samples is therefore due to thepartial pressures of N_2O_5, N_2O_4, NO_2, NO_3, NO and O_2, which are interrelated by reactions which produce the equilibrium mixture. The systems become more complex since some of the gases effectively catalyze the decomposition of N_2O_5. Little is known about the state of the equilibrium mixture, the solubilities of NO_2 and O_2 in the mixture, and their partial pressures above it. The equilibrium of $N_2O_4 \rightleftharpoons 2NO_2$ in the liquid phase and its effect on the equilibrium has not been studied. The effect the precipitation of CrO_3 has on the decomposition is also unknown. Most important of all it is not known whether the decomposition occurs in the liquid or vapor phase or both. Other questions unanswered are:

what exactly is the mechanism of decomposition, and is solution and ionization occurring.

It would therefore, be impossible to try to draw conclusions at this time from the available data. It has been possible to determine the manner of decomposition. Work under way now may determine the effect of the addition of stabilizers, or the alteration of storage conditions on the stabilization of $CrO_2(NO_3)_2$.

H. Summary of Ignition Tests:

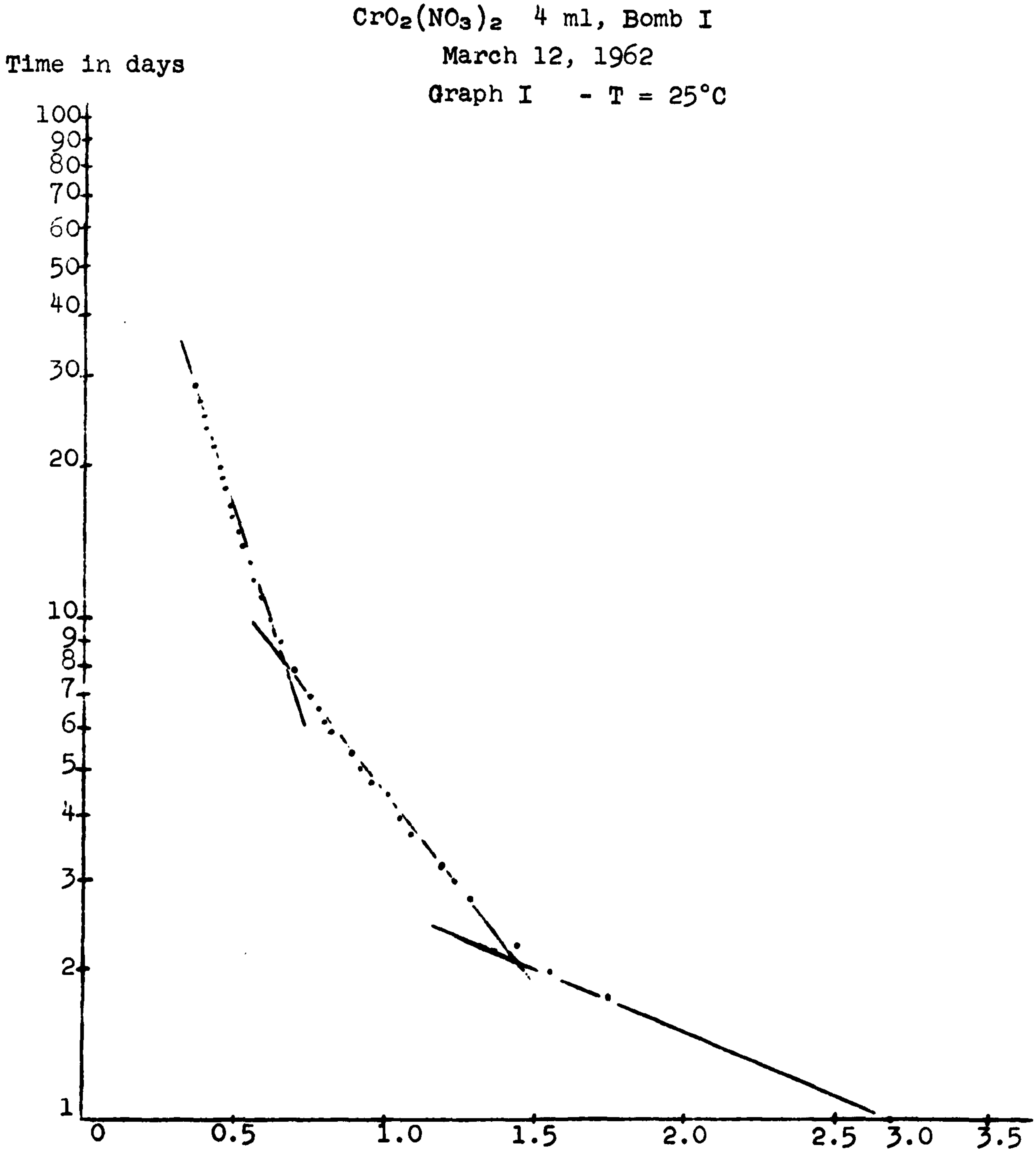
Below is a table which summarizes the ignition reaction run on the acyl nitrates:

COMPOUND	GASOLINE	HEPTANE	1-HEXENE	PARAFIN OIL
$CrO_2(NO_3)_2$	ignites	ignites	ignites	ignites
$VO(NO_3)_3$	ignites	no	ignites	sputter
$WO_2(NO_3)_2$	ignites	no	ignites	sputter
$MoO_2(NO_3)_2$	ignites	no	ignites	sputter

No ignition tests were run on the $N_2O_5 \cdot BF_3$ compound since the literature shows this compound to be a very good nitrating agent.

I. Work Being Conducted:

Experiments in which PbO_2 is added to sample cylinders containing $CrO_2(NO_3)_2$ are under way. It is hoped that this may produce a lowering of the decomposition rate. Previous tests with PbO_2 were performed but due to hydrolysis, the results could not be duplicated. It is hoped that recent results on the separation of the oxides of nitrogen by gas chromatographic means may allow the determination of the components in the gas phase. Infrared spectra on the gases are extremely complex but a combination of IR and gas chromatography may allow a detailed analysis of the spectrum.



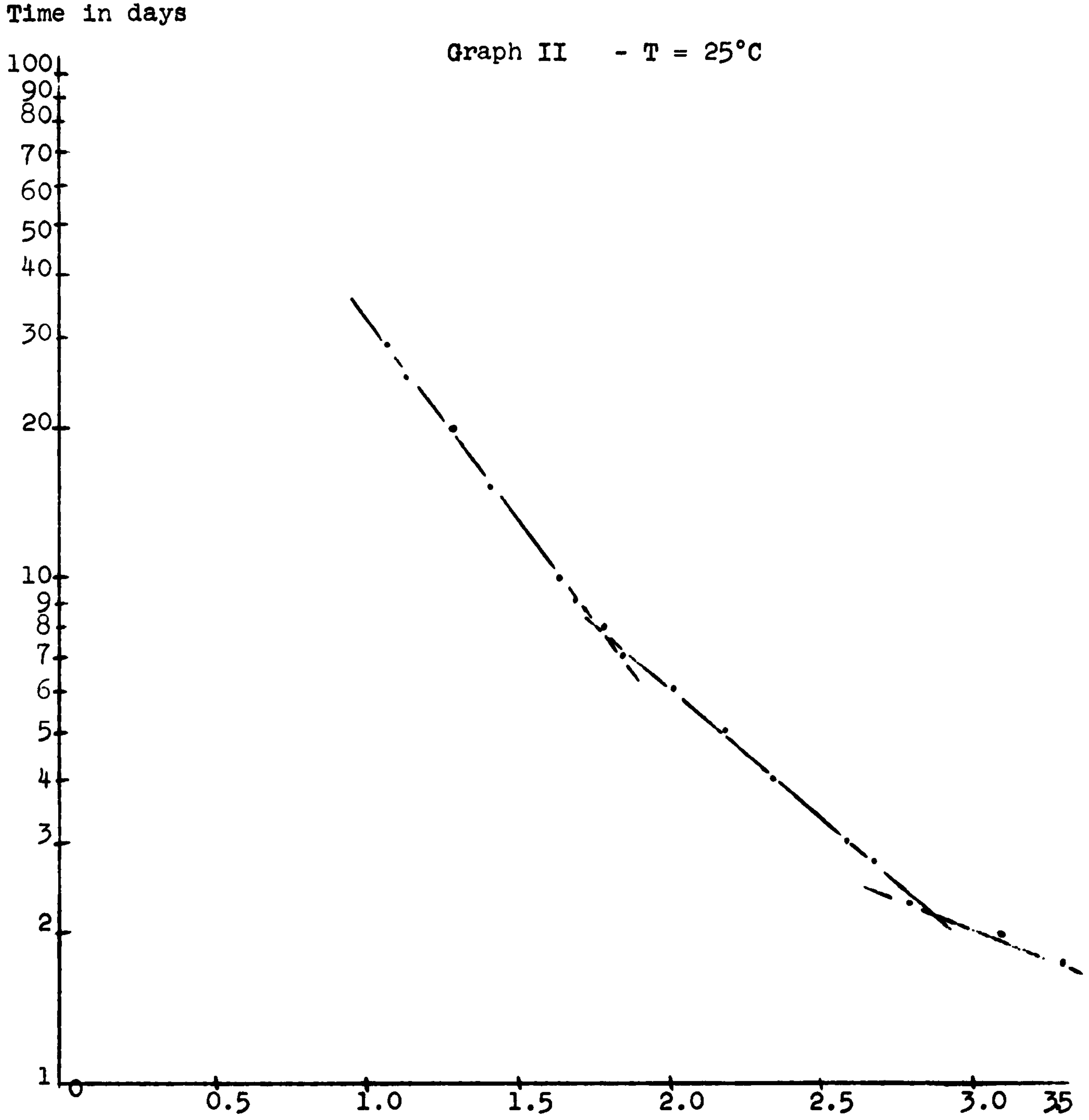
$CrO_2(NO_3)_2$ 4 ml, Bomb I
March 12, 1962
Graph I - T = 25°C

Semi-log Days vs. 1/p atms.

$CrO_2(NO_3)_2$ 3 ml, Bomb II

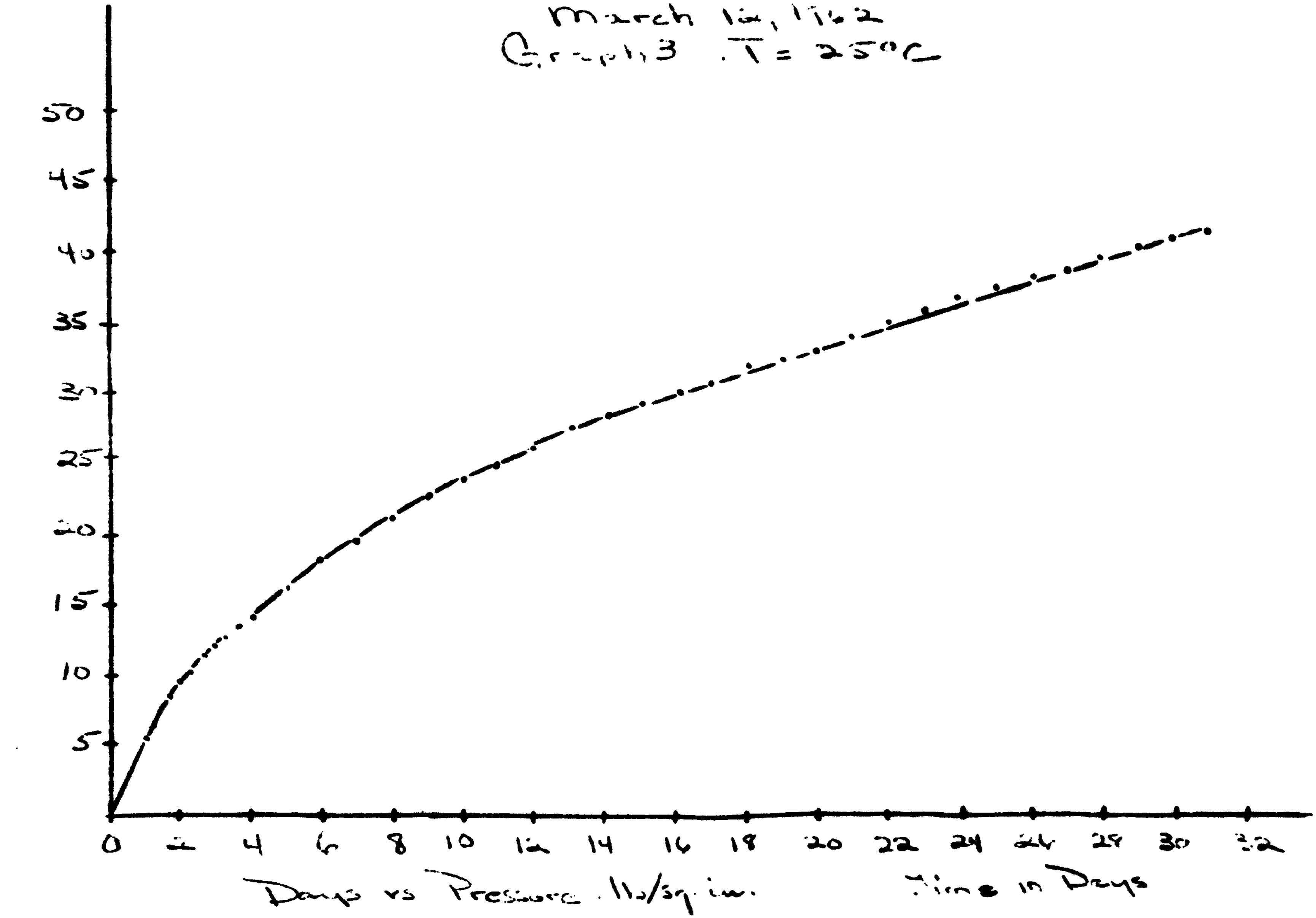
March 12, 1962

Graph II - T = 25°C



Semilog days vs. 1/p atms.

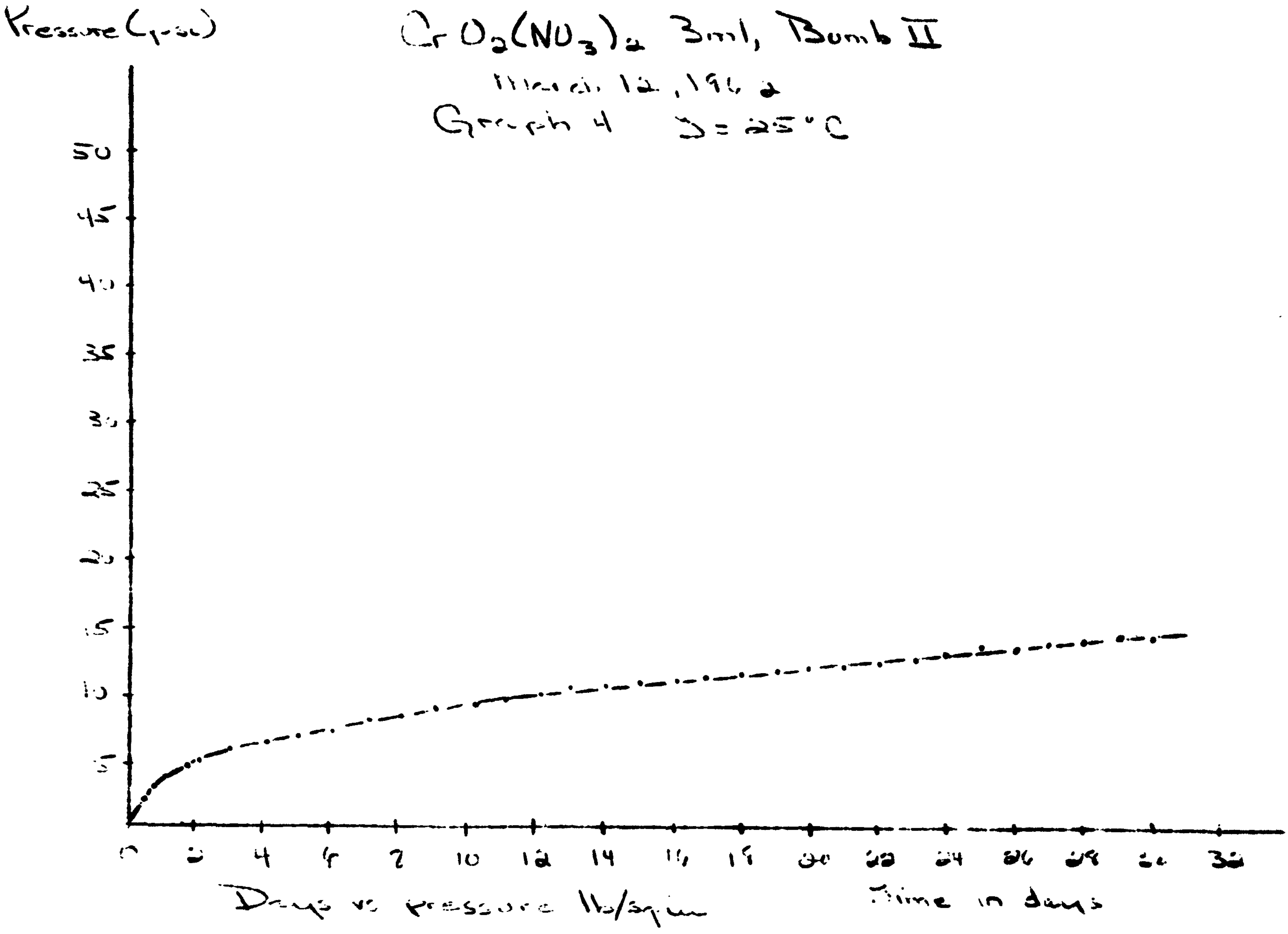
CrO$_2$(NO$_3$)$_2$ 4 ml, Bomb I
March 12, 1962
Graph 3, T = 25°C



$CrO_2(NO_3)_2$ 3ml, Bomb II

March 12, 1962

Graph 4 T = 25°C



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Additional information is presented on the decomposition of chromyl, vanadyl, and molybdyl nitrates. An interesting observation was the increased stability of chromyl nitrate over $Pb0_2$.

UNCLASSIFIED

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Vanadyl Nitrate
Tungstyl Nitrate
Molybdyl Nitrate
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