the diffusion coefficient can be expressed by the equation

$$D = A\exp(-E/RT) \qquad (11)$$

where E is the activation energy for diffusion and

$$A = e\lambda^2\frac{kT}{h}\exp(\Delta S^{\ddagger}/R) \qquad (12)$$

λ being the distance between two successive equilibrium positions.

In Table VI, the values of A, E and $\lambda[\exp(\Delta S^{\ddagger}/R)]^{1/2}$ for diffusion in the membrane are compared with values of these quantities calculated from literature data[13] on the temperature dependence of diffusion currents at the dropping mercury electrode.

It is apparent that the entropy factor is largely responsible for the difference between the diffusion coefficients of these ions in aqueous solution and in the cellophane membrane. Due to its structure, a considerable fraction of the membrane is blocked insofar as diffusion is concerned even though the diffusion is not restricted to rigorously defined pores. The smaller entropy factor is in accord with this model.

TABLE VI

ENERGY OF ACTIVATION AND ENTROPY FACTOR FOR DIFFUSION

Diffusion system	$A \times 10^3$ (cm.^{2}/sec.)	E(kcal.)	$\lambda[\exp(\Delta S^{\ddagger}/R)]^{1/2}$ (A)
Tl^+ in aqueous soln.	24	4.2	3.8
Tl^+ in cellophane membrane	3.2	4.0	1.4
Cd^{++} in aqueous soln.	41	5.0	4.9
Cd^{++} in cellophane membrane	6.6	5.2	2.0

Acknowledgment.—The authors are indebted to Professor D. D. DeFord for many valuable suggestions and discussions.

EVANSTON, ILLINOIS

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[CONTRIBUTION FROM THE RESEARCH INSTITUTE OF TEMPLE UNIVERSITY]

The Cyanogen–Oxygen Flame under Pressure

BY J. B. CONWAY[1] AND A. V. GROSSE

RECEIVED DECEMBER 20, 1957

The cyanogen–oxygen flame yields one of the highest flame temperatures obtainable by chemical means. The experimental flame temperature (4640°K.) at atmospheric pressure is in good agreement with the calculated flame temperature (4835°K.) providing additional evidence in favor of the high value for the dissociation energy of nitrogen. A technique is described for operating this flame at elevated pressures to give increased flame temperatures. Flashback velocities at various pressures are reported and are very sensitive to pressure changes. A generalized plot is presented for calculating theoretical adiabatic flame temperatures for various CO/N_2 ratios.

The equation

$$C_2N_2(g) + O_2(g) \longrightarrow 2CO(g) + N_2(g) \qquad (1)$$

represents the simplest possible stoichiometric relation describing the combustion of cyanogen. The heat of the reaction at 298°K. is equal to 126,680[2] cal. which when coupled with the fact that the reaction products are extremely stable suggests the production of very high flame temperatures by this reaction.

The reactions of cyanogen with air and oxygen have been studied for many years. Dixon[3] studied the explosion rates of various mixtures of cyanogen and oxygen at several pressures. Smithells and Ingel[4] and Smithells and Dent[5] described the structure of the cyanogen–air flame as obtained with a cone-separating apparatus and also indicated the gaseous composition of the various cones of the flame. The kinetics of the oxidation of cyanogen have been studied by Hadow and Hinshelwood[6] and Pannetier[7] discussed the mechanism by which various additives decrease the luminosity of the cyanogen–oxygen flame. The inflammability of mixtures of cyanogen and air has been studied by Pannetier and Lafitte.[8] Dolique[9] investigated the combustion of cyanogen and reported that it does not entirely follow the theoretical chemical equation. Reis[10] made a thorough study of the cyanogen–oxygen flame, analyzed the gaseous products of combustion and also analyzed the flame spectrum. A theoretical flame temperature of 4740° was reported, but no experimental flame temperature was measured. Thomas, Gaydon and Brewer[11] have reported recent work on the cyanogen–oxygen flame in which the flame temperature was measured by determining the vibrational intensity distribution of the cyanogen bands. The vibrational temperature was found to be 4800 ± 200°K. for the stoichiometric composition. The theoretical flame temperature for the stoichiometric composition was calculated to be 4850°K. using the high value (ΔH_0^0 = 226,000 cal. per mole) for the dissociation energy of nitrogen. A temperature of 4325°K. was obtained when the low value (ΔH_0^0 = 170,240 cal. per mole) was used. From these results the authors concluded that the high value is correct.

As is the case with all flames the cyanogen–oxygen flame temperature is limited by the dis-

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sociation of the reaction products. For the stoichiometric reaction described in eq. 1 the effect of dissociation on the flame temperature can be seen in the enthalpy–temperature plot shown in Fig. 1.

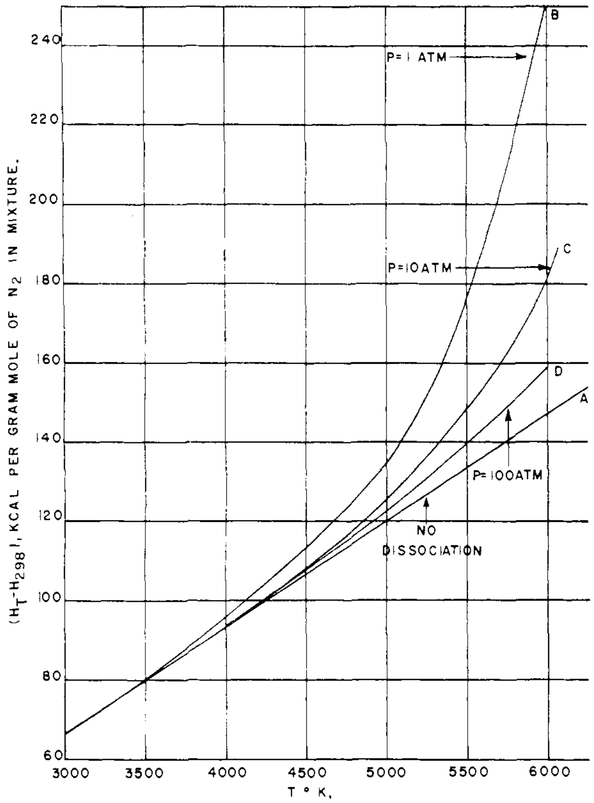

Fig. 1.—Enthalpy–temperature plot for the mixture: 2 moles of CO + 1 mole of N_2.

In curve A dissociation is neglected and it is seen that the theoretical flame temperature should be about 5200°K. With dissociation taken into account (curve B) the flame temperature (theoretical adiabatic flame temperature) at 1 atm. pressure is about 4800°K. In preparing curve A the most recent heat content data[12] for CO and N_2 were employed. In curve B the dissociation data for CO and N_2 were those of Latimer[13] corrected for some arithmetic errors which exist in the tabulated values. In addition curve B is based on calculations similar to the method employed by Ribaud[14] in that the dissociation of each gas was calculated assuming the other gas to be completely inert. The corrected enthalpies for the two gases were then added to give the enthalpy corrected for dissociation as shown in curve B. This approach is not exactly accurate but the errors involved are quite small (certainly less than 50°K.). The more accurate method has been described by Wenner[15] and when applied to this system gave values very close to those obtained using curve B.

Generally speaking the flame temperatures in chemical systems usually are increased by increasing the pressure due primarily to the suppression of dissociation of the combustion products. Such is the case in the cyanogen–oxygen system as seen in curve C and D for 10 and 100 atmospheres, respectively, although the effect is not very pronounced.

Flames in which the combustion products consist of CO and N_2 have been investigated as possible high temperature sources. A generalized plot for calculating the theoretical flame temperatures for these reactions is shown in Fig. 2 for a pressure of 1 atmosphere. For the combustion of carbon subnitride (C_4N_2) in oxygen[16] for example ($CO/N_2 = 4$ and $\Delta H_{298} = 254{,}600$ cal.) the theoretical flame temperature should be about 5300°K. (accurate calculations give 5261°K.).

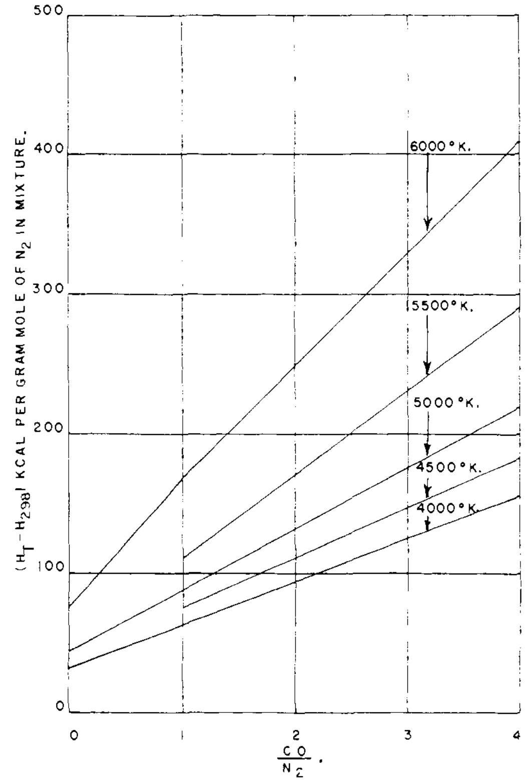

Fig. 2.—Generalized enthalpy–temperature plot for CO–N_2 mixtures.

The flame temperature at atmospheric pressure has been measured using the line-reversal method (lithium line instead of sodium line) with the sun as the comparison radiator. This technique used a two-mirror heliostat to keep the sun's image continuously focused on the slit of the spectroscope and was first employed in this Laboratory[17] to measure the temperature of the hydrogen–fluorine flame. The cyanogen–oxygen flame temperature at the stoichiometric composition (50 volume %

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(12) Thermal Properties of Gases, NBS Circular 564, 1955.

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cyanogen) was found to be 4640°K.[18] This is in good agreement with the theoretical value cited above. It is also in good agreement with the vibrational temperature (measured by the vibrational intensity distribution of the cyanogen bands) of 4800°K. reported by Thomas, Gaydon and Brewer.[11] This experimental work seems to identify the high value ($\Delta H_0^0 = 226{,}000$ cal.) of the dissociation of nitrogen as the correct value.

Experimental

Flame Characteristics.—When cyanogen was burned (torch tip was a length of stainless steel tube with a free flow area at the tip of about 0.013 cm.^{2}) in air at 226.0 cc./min. (290 cm./sec. through torch tip) the flame had three distinct zones. The outer zone was bluish and about 4 cm. in length; the middle zone was pink and had a length of about 2.5 cm.; the inner zone was violet and about 1 cm. in length. In addition, the flame was burning about 0.16 cm. off the tip, *i.e.*, it was not in contact with the surface of the tip. As oxygen was premixed with the cyanogen (cyanogen rate held at 226.0 cc./min.) the flame structure and appearance began to change. In general the pink zone got brighter and the inner zone began to disappear. When the gas composition reached 20% oxygen the top of the pink zone began to get whiter and the inner zone was almost indiscernible. At 30% oxygen the middle zone was almost entirely white except at its base where it was pinkish. At this point the white zone was about 1.5 cm. long. At 40% oxygen the flame contained only two zones. The inner zone was a very brilliant white and the outer zone was bluish. The inner zone was only about 0.6 cm. in length and the flame was burning at the tip of the torch (the flame began burning at the tip at about 10% oxygen). As the oxygen was increased still further the inner, brilliant white zone got smaller until it was only about 0.3 cm. in height. The inner zone was so brilliant that it was comparable to the brilliance of a carbon arc and it was difficult to observe this portion of the flame continuously with the naked eye.

The torch was also operated by turning on the oxygen first and gradually increasing the cyanogen rate. At 30% cyanogen (total gas about 340.0 cc./min.) the flame appeared to have two zones. The outer zone was bluish and about 2.5 cm. in length; the inner zone was violet and about 0.16 cm. in length. The flame was burning at the tip of the torch. As the cyanogen was increased the inner zone increased in brilliance until it appeared identical to the flame described above. At the stoichiometric point the flame is a very dazzling bluish white. It is very sensitive to the oxygen content in this region and even the slightly cyanogen-rich flame has an orange tinged plume which is quite noticeable. The light intensity is also appreciably diminished as the composition varies from stoichiometric.

The cyanogen was prepared by heating silver cyanide (C.P. grade) *in vacuo* and collecting the cyanogen in a liquid nitrogen trap. The product was dried and placed in a steel gas bottle for liquid phase storage (400-g. lots were prepared). The gas during use was metered using Houdry type flowmeters in connection with sulfuric acid filled manometers.

The blowoff rates for the cyanogen–oxygen flame (torch tip diameter was 0.089 cm.; free flow area was about 0.0065 cm.^{2}) at 1 atm. pressure are shown in Fig. 3. The flashback velocity was measured for the stoichiometric composition only and is seen to correspond to a total gas rate of 100.0 cc./min. (linear velocity of about 270 cc./sec. through torch tube). The critical boundary velocity gradients for blowoff and flashback at the stoichiometric composition are calculated to be 187,000 and 20,000 sec.$^{-1}$, respectively. These are much lower than the corresponding values[19] of 700,000 and 50,000 sec.$^{-1}$ for the methane–oxygen flame.

Pressure Operation.—The pressure chamber used in the flame studies at elevated pressures is shown in Fig. 4. The torch was fitted up through the bottom until the tip of the torch could be seen through the sight-glass, A. The torch was then tightened in place at the bottom stuffing-box, B. The chamber pressure was regulated by means of the needle valve, C, and the chamber was purged with argon through the inlet, D. The chamber pressure was read on the gauge, G, and the rupture disc, E, was set to break at 230 p.s.i.g. (This disc was a sheet of aluminum 0.025 cm. thick and about 7.6 cm. in diameter.)

The ignition system consisted of a central brass rod (0.16 cm. diameter) insulated from a concentric cylinder. This cylindrical device was held in place through the stuffing-box, E. A small coil (about 5 cm. long) and 0.05 cm. tungsten wire attached to the device served as the igniter. It was actuated by an 8-volt storage battery with a small coil of tungsten wire in the circuit to reduce the current sufficiently to prevent the igniter wire from burning up. With this arrangement the igniter coil became white hot when the line switch was thrown and could be operated for a few hours before being replaced. The igniter was so positioned that it was just adjacent to the tip of the torch and the rubber gasketing used in the stuffing-box allowed for a little movement of the hot wire so it could be moved directly over the torch tip when ignition was necessary. After ignition it was then moved away to prevent it from coming into direct contact with the intense flame.

In making high pressure runs it was necessary to operate with cyanogen tank pressures higher than 75 p.s.i.g. To accomplish this some cyanogen (about 70 g.) was condensed into a bomb which had a total internal volume of about 200 cc. and was designed for pressures in excess of 1000 p.s.i.g. The bomb was placed in a water-bath and before a run the water was heated to boiling. In two hours the bomb was at 100° and indicated a pressure of about 500 p.s.i.g. (the vapor pressure of cyanogen at 100°). In order to prevent condensation of cyanogen in the lines leading to the torch these lines were wound with heating wire and insulated with asbestos tape. The lines from the oxygen flowmeter (all gases were metered using Fischer and Porter Flowrators) to the torch were also heated so that both gases would be at the same temperature at the point of mixing. The lines were maintained at 100° and since the operating or metering pressure never exceeded 200 p.s.i.g. the possibility of cyanogen condensation in the line was eliminated. It also was found necessary to preheat the metering tube in the cyanogen Flowrator. This was done by placing a heating coil inside the meter-housing just adjacent to the metering tube. A thermometer placed close to this tube indicated when the temperature had reached 100°. The currents through all the heating coils were regulated with variacs.

The atmospheric pressure runs were used as guides in choosing the rates to be used at higher pressures. Since a stable flame at atmospheric pressure was obtained at 200 cc./min. of each gas it was decided to operate at high pressures with a linear velocity through the torch equal to that corresponding to a total gas rate of 400 cc. per minute at atmospheric pressure. The decision was prompted by the desire to keep the cyanogen consumption to a minimum and yet operate above the flashback velocity at atmospheric pressure. It was recognized that the flashback velocity would probably increase with pressure but it was hoped that it would not exceed the velocity corresponding to this flow rate in the pressure range to be employed. To operate at two atmospheres, for instance, the cyanogen and oxygen rates were both adjusted to 400 cc./min. measured at S.T.P. This meant that at the chamber pressure of two atmospheres the volumetric flow rate would be only 200 cc./min. of each gas measured at the chamber pressure and hence the linear velocity would be exactly the same (*i.e.*, 12.0 meters/sec.), as in the atmospheric runs.

In making a pressure run the water-bath surrounding the high-pressure bomb was turned on and the water heated to boiling. The pressure in the bomb gradually went to about 500 p.s.i.g. The argon was then turned on and the chamber was purged for about 30 seconds. The needle valve, C, was throttled until the chamber pressure increased to two atmospheres. With the argon rate set at a constant value (the argon rate was set equal to ten times the cyanogen rate) the cyanogen rate was set at 400 cc./min. at S.T.P. but metered at 150 p.s.i.g. The igniter was activated and then the oxygen rate was set at 400 cc./min. at S.T.P. but metered at 150 p.s.i.g. The flame ignited and the valve, C, was adjusted to keep the chamber pressure constant at 15 p.s.i.g. (two atmospheres). When the flame ignited the igniter was turned off and pushed out of the way. To shut down the oxygen was turned off and after the cyanogen was

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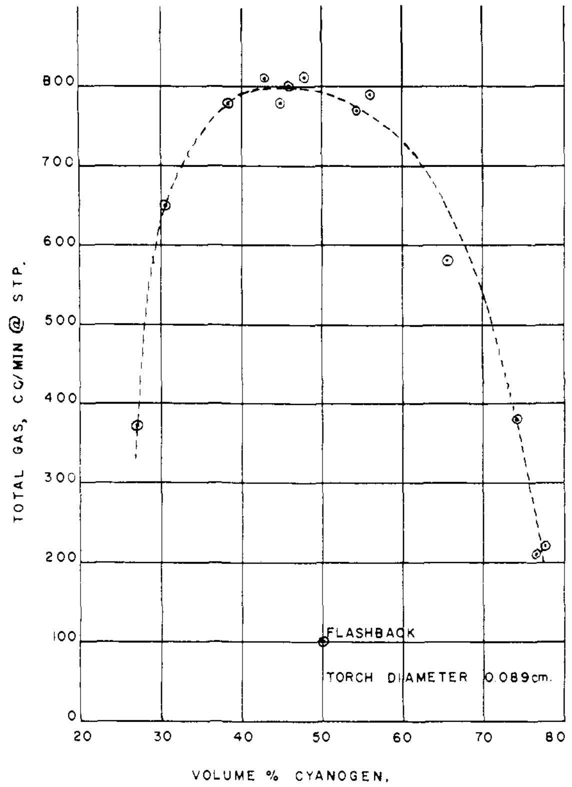

Fig. 3.—Blowoff rates for cyanogen–oxygen flame at various compositions.

turned off the valve, C, was opened to release the chamber pressure.

Runs at higher pressures were made in exactly the same way with the rates being adjusted accordingly. For instance, at three atmospheres the cyanogen and oxygen rates were set at 600 cc./min. at S.T.P. but metered at 150 p.s.i.g. In a run at 50 p.s.i.g. a flashback was obtained when operating at a total gas rate of about 1800 cc./min. at S.T.P. The velocity through the torch tube was exactly the same as that during the atmospheric run and the runs at two and three atmospheres. This indicates, therefore, that the flashback velocity at 50 p.s.i.g. is probably greater than 12 meters/sec. It was necessary, therefore, to increase the total gas rate and a stable flame was obtained at a total gas rate of 3600 cc./min. at S.T.P. The argon purge rate was 18,000 cc./min. at S.T.P. At a chamber pressure of 75 p.s.i.g. a stable flame was obtained at a total gas rate of 6000 cc./min. at S.T.P. metered at 150 p.s.i.g.

In some early runs considerable difficulty was encountered during the ignition period. After the cyanogen rate had been set and the igniter activated the flame would not ignite immediately when the oxygen was turned on and on several occasions the mixture within the pressure chamber exploded to rupture the safety disc. It is obvious that since the mixture issuing from the torch is a stoichiometric mixture of cyanogen and oxygen and hence very explosive, it is of the utmost importance to obtain immediate ignition. If this is not done a pocket of this gas forms just above the torch and eventually is ignited by the hot wire to explode within the chamber. It was not apparent why quick ignition was not obtained but it was probably due to improper positioning of the hot wire. To avoid these disturbances it was decided to purge with argon at a rate equal to ten times the cyanogen rate. This decision was based on a consideration of the explosion limits of cyanogen. The limits of inflammability of cyanogen in air are from 6.6 to 42.6% cyanogen by volume.[19] Since the inflammability limits enclose the explosion limits it was felt that the mixture within the pressure chamber would be made non-explosive by purging with an argon rate which was high enough to keep the per cent. cyanogen in the mixture below 10%. An argon purge rate equal to ten times the cyanogen rate was thought to be sufficient and was specified for all future runs. Subsequent tests showed this reasoning to be sound and no explosions were obtained when operating in this manner.

It was found possible to operate at 100 p.s.i.a. (6.8 atm.), and thereby achieve a flame temperature of 5050 ± 50°K.[20]

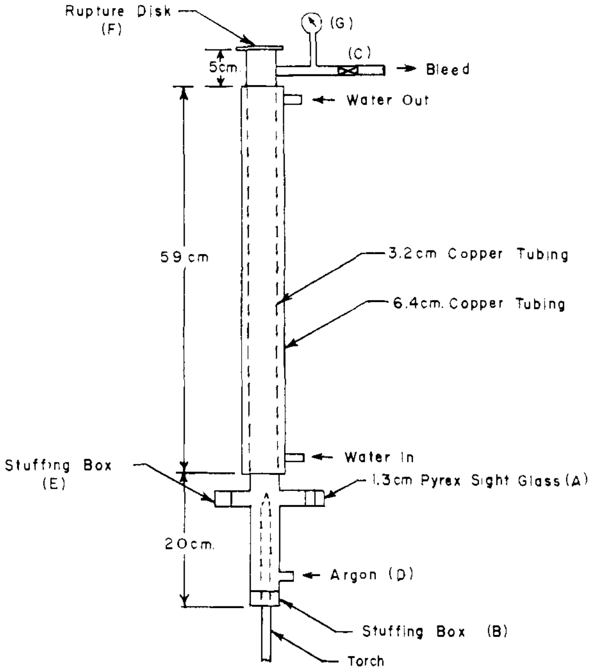

Fig. 4.—Pressure chamber for high pressure flame studies.

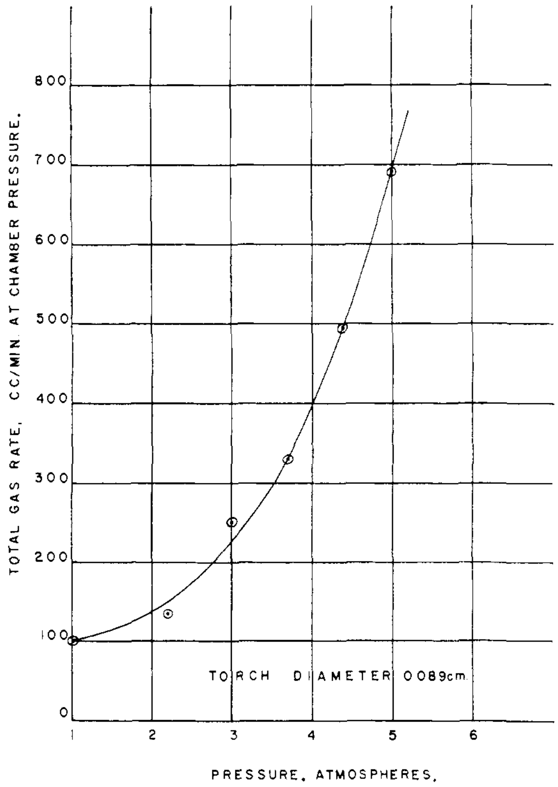

Fig. 5.—Flashback rates for cyanogen–oxygen flame at various pressures.

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(20) J. B. Conway, W. F. R. Smith, W. J. Liddell and A. V. Grosse, THIS JOURNAL, **77**, 2026 (1955).

This temperature was not measured experimentally but since the calculated flame temperature at atmospheric pressure has been confirmed by direct measurements and in view of the precision of the calculated values, it is not unreasonable to assume that this temperature is actually obtained at 100 p.s.i. (this value accounts for the reactants being preheated to 100°).

A study of the flashback velocities at various pressures yielded the data in Fig. 5. In making these measurements the rates of each gas were set to give the stoichiometric composition and the chamber pressure was gradually increased until a flashback was obtained. The gas rates were kept constant as the chamber pressure was increased. The value at 4.4 atmospheres was not determined accurately. A flashback at this pressure was obtained at 400 cc./min. but not at 800 cc./min. From visual observations it was estimated that no flashback would be obtained at 500 cc./min. These data indicate that the linear velocity through the torch corresponding to flashback is very sensitive to pressure.

Conclusions

The theoretical flame temperature for the combustion of cyanogen in oxygen is in good agreement with the experimental value providing the high value for the dissociation of nitrogen is used.

A technique is proposed for the operation of the cyanogen–oxygen flame (or other premixed flames) under pressure which provides for fairly safe (explosion-free) operation.

Blow-off rates for the cyanogen–oxygen mixtures at atmospheric pressure are presented and indicate velocity boundary gradients which are much lower than corresponding values for methane–oxygen mixtures. Flashback data at various pressures indicate that the linear velocity through the torch at flashback is very sensitive to pressure.

Acknowledgments.—The authors acknowledge the contribution of Dr. R. H. Wilson, Jr., in developing the modified line-reversal technique using the sun as the comparison radiator. The assistance of W. Smith and W. Liddell in the experimental portions of this study is also gratefully acknowledged.

PHILADELPHIA, PENNA.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, STATE UNIVERSITY OF IOWA]

Studies on the Chemistry of Halogens and of Polyhalides. XV. Iodine Halide Complexes with Acetonitrile[1]

BY ALEXANDER I. POPOV AND WILLIAM A. DESKIN

RECEIVED JANUARY 2, 1958

Slow chemical changes occurring in acetonitrile solutions of iodine halides have been investigated conductometrically and spectrophotometrically. It is shown that acetonitrile forms 1:1 complexes with iodine monochloride, iodine bromide and iodine, and that the respective formation constants are 6.9 ± 0.3, 1.40 ± 0.03 and 0.57 ± 0.02. Experimental evidence indicates that in the absence of impurities or of ultraviolet radiation there is a slow transition of the "outer complex" $CH_3CN{\cdot}IX$ to the "inner complex" $[CH_3CN{\cdot}I]^+X^-$. This slow transition is responsible for the gradual increase in conductance of the solutions and for the changes in the absorption spectra.

Introduction

When iodine or iodine halides are dissolved in polar solvents, they usually act as weak electrolytes and the resulting solutions show an appreciable electrical conductance. However, one of the puzzling aspects of such solutions is the fact that the conductance does not remain constant, but rather it tends to increase with time.[2] It also has been observed that there are time-dependent changes occurring in the absorption spectra of these solutions which roughly parallel the changes in the electrical conductance. These phenomena have been especially evident in acetonitrile solutions.

In general, this behavior has been ascribed to the reaction of the halogens either with the solvent or with a trace impurity remaining in the solvent. In recent years, however, a number of cases of similar slow change in solution properties have been reported where the solutes were much less reactive than the halogens and where a specific chemical reaction between the solvent and the solute is highly improbable.[3] Also, careful purification of the solvents did not do away with the gradual increase in conductance.[2] It was therefore decided to re-investigate the behavior of iodine and iodine halides in acetonitrile solution under carefully controlled conditions in an attempt to establish the reasons for the gradual changes in the absorption spectrum and in conductance.

Experimental Part

Iodine Halides.—Iodine monochloride was prepared by the method of Cornog and Karges.[4] It was first purified by sublimation in the usual manner. The resulting product was then introduced into a glass vacuum apparatus and further purified by several fractional crystallizations. The final product was sealed in a series of glass ampules until needed. The m.p. was 27.2°, identical with the literature value.[4]

Iodine bromide was obtained as previously described[2] and was purified by fractional crystallization until the same melting point was obtained on several repeated crystallizations. The final m.p. was 41.5°, rep. 42°.[5] Iodine of A.R. grade was purified by sublimation from a mixture of barium oxide and potassium iodide.

Solvents.—Acetonitrile and carbon tetrachloride were purified by standard methods.[2,6] The specific conductance of acetonitrile was 0.5 to 1.2 × 10^{-7} ohm^{-1} cm.$^{-1}$. The best literature value is 0.5 to 0.9 × 10^{-7} ohm^{-1} cm.$^{-1}$.[7]

Absorption Spectra.—Absorption spectra were obtained on a Cary recording spectrophotometer model 11, using stoppered silica cells of path length 1.00 ± 0.01 cm. In certain cases, 2, 5 and 10 cm. silica cells of comparable precision were used. In one series of experiments with con-

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