PHASE BEHAVIOR OF ACETONE-ETHANE
AND ACETONE-PROPANE SYSTEMS

A Thesis
Presented in Partial Fulfillment of the Requirements
for the Degree Master of Science

By
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SUMMARY

Critical locus curves were determined for the acetone-ethane and acetone-propane systems. Both curves exhibit a point of maximum pressure.

These curves, when compared with those for the acetone-n-heptane (1), acetone-n-octane, and acetone-n-decane systems (2), indicate that acetone forms azeotropes in the critical region with members of the normal paraffin hydrocarbon series between n-pentane and n-octane.

The shape of the curves is dependent on the difference in volatility of the components. When the difference is small the critical locus will possess a minimum critical temperature and may possess, in addition, a minimum as well as a maximum critical pressure point. Beyond a certain difference in volatility the locus curve is characterized by a maximum critical pressure point only. This is in agreement with the hypothesis of Kay and Warzel (3).
STATEMENT OF THE PROBLEM

It was desired to determine the critical locus curves of the acetone-ethane and acetone-propane systems. This was to be done by determining the crown of the P-T border curves of a series of mixtures of each of the binary systems.
INTRODUCTION

The increasing use of high pressure in the chemical industries has resulted in the need for a better understanding of the phase behavior of mixtures in the critical region.

The chemical nature of the components of a mixture determines its phase behavior. So long as the components of a binary system are of similar chemical nature the critical locus curve varies from a straight line to a curve with a maximum pressure point depending on the difference in volatility of the components. When there is a marked difference in the chemical nature of the components and the difference in their volatility is relatively small the critical locus curve possesses a point of minimum critical temperature and may, in certain instances, also show a point of minimum critical pressure as well as a point of maximum critical pressure.

In order to learn more about the factors which affect the shape of the critical locus curve a research program was initiated to determine the critical locus curves of binary systems composed of acetone and the normal paraffin hydrocarbons. Previous to this investigation Congoliere (1) had studied the acetone-n-heptane system and McAdams (2) the acetone-n-octane and acetone-n-decane systems.
EQUIPMENT

P-V-T Apparatus

The apparatus used for the determination of the pressure-temperature border curves was developed by Kay (4). A diagram of the apparatus assembled for operation is shown in Figure 1.

A small sample of known composition was confined over mercury in a thick-walled glass capillary tube, 10, which was surrounded by a jacket, 7, and heated by the condensing vapors of a liquid vaporized in the side-arm boiling flask, 4, by an electric heater, 3. A mercury layer, 6, prevented contamination of the boiling liquid by the rubber stopper, 5, supporting the jacket. A glass tube, 8, held in place by asbestos strips, 9, served to reduce heat losses from the jacket.

A pressure regulating assembly connected by a rubber hose, 20, through the side-arm condenser, 16, was used to control the temperature in the jacket. A vacuum pump attached at bulb 24 served to reduce the pressure in the system to any desired value, as read by the mercury manometer, 22. Bulb 23 could be opened to atmospheric pressure. Three 5-gallon bottles, 25, were used as surge tanks to increase the volume of the system. The temperature of the bath was measured by an iron-constantan thermocouple, 13, the cold junction of which was immersed in an ice bath, 19, in conjunction

4
with a sensitive potentiometer. The radiation shield, 12, was not used.

Pressure was applied to the sample by compressed nitrogen from the cylinder, 34, through the compressor block, 2, in which the sample tube was held. Pressure adjustments were made by adding high pressure nitrogen through the needle valves, 31, or by releasing gas through the exhaust valve, 32. The surge tanks, 33, served to stabilize the pressure by increasing the volume of the system. The pressure was measured by means of a gage, 27, graduated in two psi intervals over a range of 0-1500 psi. The pressure gage was calibrated with the dead weight gage, 29. Heavy walled copper tubing, 26, was used to connect the pressure system.

To bring about rapid equilibrium once the temperature and pressure of the sample were set, a steel ball, floating on the mercury in the capillary tube, was agitated by moving a permanent magnet up and down the jacket by hand. The electromagnet, 11, and pulley set-up, 14 and 15, were not used.

A metal tray, 1, upon which the compressor block is mounted, retained any mercury spilled during assembly or disassembly of the apparatus. A suction flask was used to remove the spilled mercury.
Compressor Block and Experimental Tube

Figure 2 is a cross-sectional view of the compressor block, showing its construction and the method of holding the experimental tube. The compressor block consisted of two steel cylinders, A and B, connected with a steel tube, D, and was filled with mercury. Pressure was applied to the mercury surface in leg B by compressed nitrogen and transmitted through the mercury to the sample in the experimental tube seated in the front leg, A. The connecting tube, D, also acted as a safety check valve. A steel ball, E, which floated in the mercury, did not interfere with the flow of mercury unless the experimental tube burst, in which case the sudden surge of mercury would drive the steel ball into the valve seat, F, and prevent any further loss of mercury.

A diagram of the pyrex experimental tube with approximate dimensions is shown in Figure 3. The closed end section of 330 mm long precision bore tubing served as the working section in which all measurements were made. The 3 mm section was of standard pyrex capillary tubing. The 13 mm bulge served as the seal in the stuffing box of the compressor block. The section of 18 mm pyrex tubing served as a gas reservoir for the loading of the gases.

Gas Microburette

The gas microburette was used to precisely measure
FIGURE 2. COMPRESSOR BLOCK.
FIGURE 3. EXPERIMENTAL TUBE

ALL DIMENSIONS IN MILLIMETERS
and load pure samples of a desired gas into the experimental tube.

The burette consisted of a measuring section of 2.54 mm diameter precision bore capillary tubing graduated in 1 mm divisions for a total length of 50 cm. The delivery tip of the burette was reduced to a straight section with an external diameter of approximately 1.5 mm, so as to permit its insertion into the end of the experimental tube as shown in Figure 4. The U-shaped section between the delivery tip and the precision bore measuring section was quite flexible in its manipulation in the mercury pot. The strength of this capillary and its flexibility insured little strain upon insertion in the experimental tube or the gas collecting tube as shown in Figure 4. The calibrated measuring section of the burette was surrounded by a water jacket to stabilize the temperature of the gas sample. Air was slowly bubbled through the water bath in order to remove gradients in its temperature, which was measured with a thermometer supported from a stopper at the top of the jacket.

The driving mechanism of the gas microburette was a stainless steel bellows, which was extended or compressed by means of a threaded rod and sleeve arrangement. The bellows was capable of a maximum volume change of 70 cc.
PROCEDURE

Purification Procedure

The two hydrocarbons, ethane and propane, were used as received from Phillips Petroleum Company. The purity of each was stated to be 99.9 mole per cent or better.

Reagent grade acetone was further purified by distilling with $P_2O_5$. Out of a 1000 cc charge, the first 200 cc were discarded, the next 500 cc kept as pure sample, and the remaining 300 cc discarded as waste.

Tube Preparation

The experimental tube was prepared by cleaning and washing with chromic acid cleaning solution and water, and then drying with reagent grade acetone and filtered air. Before placing the tube on the loading apparatus a 1/16 inch diameter steel ball was placed in it. The steel ball was cleaned by stirring with a pair of tweezers in a small beaker of acetone. The ball was then passed over a flame to remove the acetone and dropped into the dry experimental tube. The tube was then sealed on the apparatus as shown in Figure 5.

Procedure for Loading Acetone

The experimental tube and flask, Figure 5, were mounted on a ringstand such that the sample tube was inclined downward. Stopcock $E$ was lubricated with mercury and a mercury seal was used on top of the stopcock.
The flask was then connected to a vacuum pump through appropriate dry ice-acetone traps. The flask and experimental tube were evacuated for one to two hours. Stopcock E was then closed and the vacuum line removed. The flask was then partially filled with distilled mercury. To retain the vacuum and prevent air from entering the system, the stem F of stopcock E was filled with mercury. Then the stopcock was cracked to allow atmospheric pressure to force part of the mercury into the flask and closed again while there was still a small amount of mercury remaining in the stem as a seal against the atmosphere.

The acetone to be loaded was then placed in a simple Hemple distilling apparatus with a drying agent. The sample was refluxed for a few minutes and the first few drops to distill were discarded. The side arm of the distillation column was then placed in stem F of the loading flask. As the sample distilled into the stem, stopcock E was cracked and atmospheric pressure forced the sample into the flask. Care was taken to maintain a liquid seal against the stopcock at all times. When the desired amount of sample was loaded, the distillation apparatus was removed and the vacuum train again attached. The system from the stopcock through the cold trap was reevacuated and the vacuum pump kept running during the rest of the loading operation.
With the sample in the evacuated flask on top of the mercury the de-gassing procedure was begun. Experimental tube A was cooled at tip H causing some of the sample to distill into the sample tube. When the tube was filled to a length of perhaps 50 mm the tip was heated driving the liquid sample back into the flask. Then stopcock E to the vacuum system was opened. Any volatile gases which had been forced out of the experimental tube during the distillation along with a small amount of the sample in the vapor phase were swept into the cold trap. Stopcock E was again closed and the entire process repeated six times. On the seventh filling the desired amount of acetone was allowed to remain in the experimental tube. The loading apparatus was then removed from the mounting rack and tipped quickly into a vertical position with the tube tip on the bottom. This caused the mercury in the flask to occupy the void space above the sample and sealed the sample in the tube.

The sample was then cooled in liquid nitrogen to prevent vaporization and the stopcock opened to the vacuum train. The excess sample in the flask was then distilled into the cold trap. The experimental tube above the cold sample in the flask was warmed to vaporize any sample trapped along the wall above the tip. The vacuum caused these pockets of vaporized sample to
rise and be displaced by mercury. After all the unused sample had been distilled into the cold trap the vacuum system was disconnected from the loading flask and air was admitted.

The tube was then marked with a file where the glass seal had been made and was broken free from the loading flask. The loaded experimental tube was then ready to place in the compressor block.

Procedure for Loading Propane and Ethane

The gas microburette was used to measure and introduce precisely known amounts of propane and ethane into the experimental tube. The acetone was loaded into the sample tube first as described earlier.

The sample tube or experimental tube was usually already in the compressor block when the gases were to be loaded. The liquid sample was frozen in the tip of the tube by means of an open pyrex cooler containing liquid nitrogen. With the liquid sample frozen in the tip of the tube, the pressure was released from the compressor block and the screw collar O and sleeve N, Figure 2, removed. The mercury pot, Figure 4, and gasket were then screwed tightly on the front leg of the compressor block after which the experimental tube was partially raised out of the front leg of the block while pouring mercury in the back leg to maintain the mercury seal. A cork was placed tightly in the back leg of the block,
and the mercury pot filled with mercury. The experimental tube, with the sample continually frozen in its tip by the open cooler, was raised until its open end rested on the bottom of the pot and then clamped in place.

The gas collecting tube, Figure 4, was thoroughly cleaned with chromic acid cleaning solution, rinsed with distilled water, and dried with acetone and filtered air. The open bulb section of the gas collecting tube was placed slightly below the surface of the mercury in the mercury pot and clamped into place as shown in Figure 4. With the stopcock closed, a suction line was attached to the upper end of the gas collecting tube from a water aspirator. The stopcock was opened, allowing the mercury to rise slowly inside the open bulb of the collecting tube until it was above the stopcock. The suction line was removed and the collecting tube lowered further into the mercury and clamped in place.

A gas sample bulb was cleaned with acetone and dried. Its capillary tip was scratched with a file, inserted under the mercury surface into the collecting tube, and broken, releasing the gas into the tube.

The U-tube of the microburette was cleaned with acetone and placed into the mercury pot after its entire capillary was filled with mercury. After any residual air was displaced from the tip by forcing mercury from it, the tip was placed under the gas collecting tube and the tube
lowered until the tip of the burette protruded into the gas sample. Gas was then drawn into the burette, flushed throughout the burette and expelled to the atmosphere. A sample large enough to almost fill the calibrated section of the burette was then drawn in the same manner and mercury drawn in from the pot, behind the sample.

The level of the mercury in the pot was adjusted to the height of the zero mark of the burette by raising or lowering the gas collecting tube in the mercury pot. This was accomplished with the aid of a cathetometer in order to insure atmospheric pressure on the sample. Air was allowed to slowly bubble through the water bath to eliminate temperature gradients within the bath. The mercury at the top of the section containing the gas sample was then brought to the zero mark of the burette by rotating the fine adjustment knob on the burette.

The water bath temperature, barometric pressure, and length of gas sample were read and recorded.

The delivery tip of the burette was then placed inside the experimental tube and the gas sample was discharged into the large gas bulb. This procedure was repeated until the desired amount of gas was in the experimental tube.

The gas sample in the large bulb was then transferred into the upper capillary portion of the tube by
vigorously tapping. Care was taken at all times to prevent air from entering the tube.

After all the gas had risen into the capillary section, it was frozen in the upper part of the tube by lowering the liquid nitrogen cooler, which was keeping the previous sample frozen in the tip of the capillary tube. To prevent displacement of some of the previous sample into the large gas bulb by the new gas sample, several inches of mercury were left frozen between the two samples. This mercury was easily displaced after pressure was applied to the system.

The gas microburette and the gas collecting tube were removed from the mercury pot. The bulb of the experimental tube was inserted into the compressor block, and the mercury removed from the pot. The mercury pot was then removed, along with the cork in the back leg of the compressor block. The remaining fittings were placed on the experimental tube and the tube clamped into position.

With the entire sample frozen in the tip of the experimental tube, the depth of the mercury below the top of the back leg mark or reference point was determined. Then the fittings were connected and enough pressure applied to the system to hold the sample within the capillary section of the experimental tube.

With pressure on the experimental tube, the open
cooler was removed and the mercury and sample thawed. The apparatus was then ready for experimental observations.

**P-T Experimental Procedure**

The first step in the start up of the P-V-T apparatus was to adjust the pressure gage so that it read zero at atmospheric pressure unless some propane or ethane were in the sample, in which case enough pressure was constantly maintained on the sample to keep it condensed in the end of the tube. This was done by opening valve 32, referring to Figure 1, to the atmosphere, and adjusting the scale of the gage. Then the exhaust valve was closed, the vacuum pump turned on, and the pressure in the jacket lowered to correspond to the desired temperature. Before the electric heater was turned on, a pressure above the estimated critical pressure was applied to the sample in order to keep the sample from vaporizing and escaping from the tube.

The electric heater was then turned on. When the ring of condensing vapors had risen to the top of the glass insulating jacket, the powerstat was adjusted so that the condensation ring would go no higher. To aid in this a stream of air was blown against the site of condensation. When the temperature in the jacket had become steady, the bubble and dew points were determined for that temperature by lowering the pressure until
they appeared. The temperature was considered steady when potentiometer readings became constant.

The method for determination of the bubble, dew, and critical points depended upon visual observation of the sample. The bubble point was taken when, after vigorous agitation, a bubble about the size of a pin point was visible at the top of the tube. The dew point was taken as that point where a minute amount of liquid was last observed between the mercury meniscus and the wall of the tube after vigorous agitation. The critical point was taken as the point where the meniscus between the liquid and vapor phase could no longer be observed and the sample appeared smoky to reflected light and brown to transmitted light.
RESULTS

The pressure and temperature relations at the liquid-vapor phase boundaries of five different mixtures of acetone and ethane and of five different mixtures of acetone and propane were determined in order to define the pressure-temperature-composition diagrams of the two binary systems. The experimental data for these two systems are represented by the curves in Figures 6 and 7. Tabulated experimental data are given in the appendix.

The acetone used in this investigation was found to have a critical temperature of 235.01 degrees centigrade and a critical pressure of 683.5 pounds per square inch.
Figure 6: Critical Locus and Border Curves for the System Acetone-Ethane

Compositions are in mole per cent ethane.
FIGURE 7. CRITICAL LOCUS AND BORDER CURVES FOR THE SYSTEM ACETONE–PROPANE
DISCUSSION OF RESULTS

The P-T critical locus curves of both systems studied exhibit a point of maximum pressure with no points of minimum pressure or temperature. This type of curve indicates that the intermolecular forces are not of the type or magnitude tending to form an azeotrope.

The critical loci of the two systems are shown in Figure 9 along with the critical loci of the acetone-n-heptane, acetone-n-octane, and acetone-n-decane systems. These compiled data show that critical loci of binary systems of acetone with the normal paraffin hydrocarbons is following a pattern in accordance with the hypothesis of Kay and Warzel (3). The data of Poettman and Katz (5) show that binary systems of carbon dioxide with the paraffin hydrocarbons exhibit similar behavior. The work of Warzel (6) and Fisch (7) indicates that the ammonia-hydrocarbons systems may also follow this pattern of behavior.

The reported compositions are probably accurate within 0.1 mole per cent. Possible sources of error are in reading the length of the column of sample, calibration of the measuring tubes, and in assuming that the perfect gas law holds over the small pressure and temperature range from the known value to the values at which the gas samples were measured.

The estimated accuracy of the pressure measurements
is within 1.0 psia. Sources of error are in reading the pressure gage, which was marked in increments of 2.0 psia, and from the pressure gage taking a "set" when it was necessary to leave pressure on the sample to prevent its expansion and subsequent loss.

The temperature measurements are estimated to be accurate within 0.05 degrees centigrade. Possible sources of error are in the observation of the sample, temperature gradients in the condensing vapors, and calibration of the thermocouple and potentiometer.

The values for the critical pressure and temperature of acetone as determined in this investigation differed slightly from those reported in the literature (11). Values for the critical pressures and temperatures of ethane and propane were literature values (11).
CONCLUSIONS

The results of this investigation show that the acetone-ethane and acetone-propane binary systems follow the general pattern of phase behavior exhibited by systems which do not form azeotropes.

From the data of this investigation and from that of other systems, (1), (2), (5), (6), (7), (8), (9), (10), a general pattern for the shape of the critical locus curves of binary mixtures of a series of homologous compounds with a compound which forms azeotropes with some members of the series is indicated. This pattern is illustrated in Figure 8. Curves 1 through 8 represent the vapor pressure curves of the homologous compounds. Curve A represents the vapor pressure curve of the azeotropic agent, a compound which forms non-ideal solutions with the members of the homologous series due to its difference in chemical nature. Binary systems of the azeotropic agent with members of the series are characterized by the formation of azeotropes where the critical temperature of the series member is not too different from the critical temperature of the azeotropic agent. When azeotropes are formed in the critical region, the critical loci will appear as shown by the broken lines C₂B, C₃B, ... C₆B. As the difference in critical temperatures between the compounds of the homologous series and the azeotropic
FIGURE 8. PREDICTED BEHAVIOR OF THE CRITICAL LOCUS CURVES OF A SERIES OF HYDROCARBONS WITH ANOTHER COMPONENT.
agent become greater, the tendency to form an azeotrope no longer exists, and critical locus curves with maximum pressure points are formed as illustrated by curves C\textsubscript{1}B and C\textsubscript{3}B. The height of the maximum pressure point seems to depend on the difference between the critical temperatures of the two compounds in the binary, the maximum pressure becoming greater as the difference between critical points becomes greater.

These relations refer only to systems in which minimum boiling azeotropes are formed. Although no experimental data are available on systems forming maximum boiling azeotropes, it is possible that for these systems critical loci with maximum temperature points would appear.
RECOMMENDATIONS

Although the critical loci of the binary acetone-paraffin hydrocarbon systems determined thus far exhibit the expected behavior, it is recommended that the acetone-n-hexane and acetone-n-pentane systems be studied to determine if they also follow the predicted trend.

Methods for predicting critical locus curves should be investigated. The field of statistical mechanics seems to be the most logical area for new developments in this difficult work.
LITERATURE CITED


APPENDIX I

CALIBRATIONS

Thermocouple Calibration

The iron-constantan thermocouple was calibrated at three known temperatures and a calibration curve of observed temperature vs. correction was prepared. The points of known temperature used for calibration were the ice point and the condensing points of naphthalene and water.

Pressure Gage Calibration

The pressure gage was calibrated with a dead weight gage tester. A known weight was placed on the pan of the dead weight gage and the pressure was adjusted until the nitrogen pressure balanced the dead weight gage. The calculated pressure of the dead weight gage was recorded at the observed pressure of the pressure gage. A calibration chart was prepared of correction vs. the observed gage reading.

Sample Tube Calibration

An equation for the volume of any size sample in the sample tube was obtained in the usual manner of successive weighings of various length mercury columns down to and including the empty tube. The volume is represented by the equation \( v = 0.00320 \ L - 0.014013 \), where \( v \) is volume in cc and \( L \) is length in mm from the tube tip. This includes the volume of the steel ball which was assumed to be a perfect sphere.
APPENDIX II
SAMPLE CALCULATIONS

Loading Acetone

A sample calculation sheet for an acetone loading
is given in Table I. Items 1 through 3 are self-explanatory.
Item 4 is the temperature of the bath surrounding the
calibrated experimental tube and is taken as the sample
temperature. Item 5, the top of the mercury meniscus,
item 6, the bottom of the mercury meniscus, and item 8,
the tube tip, are read with a cathetometer. Item 7 is the
difference between items 5 and 6. Item 9 is the difference
between items 8 and 5. Item 10 is the calculated volume
of the acetone not including the meniscus correction.
It is calculated by the equation which was obtained by
the tube calibration. Item 11 is calculated by assuming
the mercury meniscus forms a segment of a sphere and
finding the difference between this volume and the volume
of a cylinder the length of the meniscus height. Item
12 is obtained by adding items 10 and 11. Item 13 was
obtained from a plot of acetone density vs. temperature
at atmospheric pressure. Item 14 is found by multiplying
item 12 by item 13.

Loading Gases

A sample calculation sheet for the loading of gases
and calculation of sample composition is given in Table
II. Items 1 through 3 are self-explanatory. Items
4 and 5 are experimental data. Item 6 is a correction

34
### TABLE I

**SAMPLE CALCULATION SHEET FOR LOADING ACETONE**

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to reduce the barometer reading to standard temperature and gravity. Item 7 is the difference between items 5 and 6. Item 8 is item 7 changed to psia. Item 9 corrects for the difference in mercury capillary pressure between the sample loading tube and the mercury pot. Item 10 is the difference between items 8 and 9. Item 11 is calculated by assuming the perfect gas law holds over the range from a known reference point to the pressure and temperature conditions of the sample. Item 12 is experimental data. Item 13 is obtained by multiplying item 12 by the volume per unit length which was obtained by assuming the sample loading tube was a perfect right cylinder. Item 14 is calculated by multiplying item 13 by item 11. Item 15 is the weight of gas added to the sample in previous runs. Item 16 is the sum of items 14 and 15. Item 17 is the weight of the gas plus the weight of the acetone in the sample. Items 18 and 19 are self-explanatory.

P-T Data

A sample calculation sheet for a typical run to determine a point on the P-T border curve is given in Table III. Items 1-4 are self-explanatory. Item 5 is the type of point being determined. Bubble points are designated by B, dew points by D, and critical points by C. Items 6 and 7 are experimental data. Item 8 is a correction to reduce the barometer reading to
standard gravity and temperature. Item 9 is the difference between items 7 and 8. Item 10 is experimental data. Item 11 is the gage correction read from the pressure gage calibration curve. Item 12 is item 10 minus item 11, Item 13 is item 9 changed to psia. Item 14 is the sum of items 12 and 13 where item 13 is item 9 changed to psia.

A cathetometer is used to read items 15, 16, and 18. Items 16 and 18 remain constant throughout a run. Item 15 is read for each point. Item 17 is the difference between items 15 and 16. Item 19 is measured while loading. Item 20 is the difference between items 18 and 19. Item 21 is the difference between items 16 and 20. Item 22 is the vapor pressure of mercury at the sample temperature. Item 23 is item 21 corrected to standard temperature. Item 24 is the same correction for item 17. Item 25 is the sum of items 22, 23, and 24. Item 26 is item 25 changed to psi. Item 27 is item 14 minus item 26.

Item 28, the reading of the potentiometer, is experimental data. Item 29 is taken from the reference chart for iron-constantan thermocouples. Item 30 is also calculated from this chart. Item 31 is item 29 plus item 30, or item 28 converted into a temperature reading. Item 32 is from the calibration curve for the thermocouple. Item 33 is item 32 minus item 31.
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### APPENDIX III

**EXPERIMENTAL DATA**

**TABLE IV**

**P-T-x EXPERIMENTAL DATA**

**Acetone-Ethane system**

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### 66.1 Mole % Ethane

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### 82.7 Mole % Ethane

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77.9 Mole % Propane

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