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Viscosity of Helium, Oxygen, Helium-Oxygen, Helium-Steam, and Oxygen-Steam Mixtures at High Temperatures and Pressures

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The need for the viscosity of gases arises in at least three important phases of chemical engineering: fluid mechanics, heat transfer, and kinetics. Because the experimental determination of the viscosity of a gas is difficult and expensive, the practicing engineer must usually obtain the viscosity data he needs from the published literature. The viscosity of pure component gases at atmospheric pressure is readily available in the literature over wide temperature ranges. In addition to the pure component data, several charts and nomographs may be used to obtain a reliable estimate of the viscosity of a pure component when actual data have not been measured.

Viscosities of some of the more common gases have been measured at elevated pressures. Many charts and nomographs have been prepared which correlate the viscosity of gases as functions of temperature and pressure. More often, however, the engineer is concerned with determining the viscosity of gaseous mixtures at elevated pressures. The variation of the viscosity of gaseous mixtures with pressure has received little attention experimentally or theoretically. Boyd (1) measured the viscosity of hydrogen-nitrogen mixtures at elevated pressures. Clauson (2) has recently studied methane-nitrogen mixtures with a Rankine capillary at pressures up to 5000 p.s.i.a. These data plus the viscosity data on air at elevated pressures measured by Nasini and Pastonesi (10) and Kestin and Pilarczyk (6) represent all the data available on gaseous mixtures at elevated pressures.

This investigation was conducted for the Reactor Experimental Engineering Division of Oak Ridge National Laboratory. The pure components and mixtures investigated were of particular interest to the homogeneous reactor project.

The first phase of this investigation was the determination of the viscosity of helium and oxygen at elevated pressures over a temperature range of 200° to 450°C. The second phase was the determination of the viscosity of helium-oxygen, steamhelium, and steam-oxygen mixtures at elevated pressures and temperatures.

While no reliable method of predicting the viscosity of these mixtures from the pure component data is available, the data as presented should be of use to the practicing engineer. Possibly later the data on pure component and mixture viscosity will also be of value in testing the reliability of methods of predicting the viscosity of mixtures at elevated pressures.

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APPARATUS

Description. The viscometer, shown in Figures 1, 2, and 3, consisted essentially of three parts, the gas feed and preheat section, the viscometer itself, and the gas bleed and flow measurement section.

Stainless steel pressure tubing, Type 304, 1/4 inch in outside diameter and 3/32 inch in inside diameter, was used throughout the apparatus with the exception of the capillary tube, and the capillary tube pressure taps. The feed and preheat section for permanent gases consisted merely of pressure tubing running from a supply tank either through a pressure regulator or directly into a coiled tube immersed in a salt bath. The regulator used was a diaphragm-type balanced pressure controller manufactured by the Victor Equipment Co. It was found to throttle gas pressures from tank pressure down to any lower pressure with no noticeable fluctuations in output pressure.

The preheater coil consisted of three sections of the pressure tubing 25 feet long coiled on 2-inch centers connected in parallel. Preliminary calculations showed that even under the laminar conditions maintained in the coil, the gas would be heated to virtually the bath temperature under the most extreme conditions of temperature encountered in this work. The



Figure 1. Feed and preheat sections of the viscometer

preheater bath itself was a section of large pipe with a welded bottom. The cylinder thus formed was embedded in insulating cement and the entire apparatus was enclosed in a sheet metal shell. It was then filled with a eutectic mixture of potassium nitrate and sodium nitrite salts which was obtained from the American Cyanamid Corp. under the commercial name Aeroheat. Heat was supplied to the bath by three 1000-watt immersion heaters, connected in parallel through a variable transformer. Control was maintained by a Wheelco Potentiotrol to $\pm 0.5^{\circ}$ C. from the set point. Temperature in this and other sections of the apparatus was measured with a Rubicon Type B, high-precision potentiometer.

The preheater was attached to the viscometer with high pressure. The pressure tubing and the fine capillary of the viscometer were joined by an Aminco through type connector. All tubing between the preheater and the viscometer was jacketed and heated electrically by nichrome resistance wire. Control was manual using a variable transformer.

The viscometer itself was constructed from type 304 stainless steel tubing, roughly 17.5 feet long, 1/4 inch in outside diameter and 1/32 inch in inside diameter. The inside diameter as given by the manufacturer was 1/32 inch + 0.010 - 0.000. The inside wall was smooth but not necessarily of constant diameter. At about 9 inches from one end and 15 inches from the other end, 0.030-inch holes were drilled through one wall of the tube and short lengths of the capillary tubing were Heliarc welded over the holes. A wire just smaller than the diameter of the capillary was pushed into each end of the tube to assure that no burrs remained on the inside from the drilling.

The capillary tube was then placed in a loop of 2-inch pipe as shown in Figure 2. The lower section of the loop contained heaters with a total capacity of 3000 watts. An agitator was in-



Figure 2. Details of viscometer

stalled to provide circulation in the loop and special high temperature pipe insulation was placed around the entire apparatus. Water was used as the bath liquid for low temperature calibration runs and Aeroheat was used for higher temperatures.

Temperature control of the viscometer was maintained by a Brown electronic potentiometer recorder controller. The temperature of the bath fluid was checked at various points along the capillary tube. Maximum variation of the temperature was determined to be $\pm 1^{\circ}$ C. from the control point. During all runs the temperature of the bath fluid was measured at the midpoint of the capillary using a Chromel-Alumel thermocouple and the Rubicon potentiometer. This bath temperature was taken to be the gas temperature in the capillary tube. No actual measurement of the temperature of the gas stream was attempted.

A Model 200 Barton differential pressure gage was used to determine the pressure drop across the capillary tube. The welded pressure taps of the capillary tube were connected to this gage with standard pressure tubing. Valves and a bypass were included in the lead lines. The gage was a standard 0 to 20 inches of water meter which could operate at static pressures up to 2500 p.s.i. The gage was sensitive enough that a vernier was added so that the pressure drop could be read to the nearest 0.01 inch of water.

At the exit from the viscometer a tee was installed, one side led to a pressure gage and the other side to the bleed and flow measurement apparatus. The pressure gage was a 16-inch Bourdon gage manufactured by the U. S. Gage Co. The scale was divided in 10-pound increments and readings could be estimated to the nearest pound.

The bleed and flow measurement apparatus as shown in Figure 3 consisted of a needle valve, a water-jacketed orifice, and a wet-test gas meter. The orifice was water-jacketed to maintain it at a constant temperature as thermal effects in the expanding gas caused an unjacketed orifice to contract and diminish the flow rate. The helium-oxygen mixtures were prepared by changing helium and oxygen in the proper proportions into standard helium cylinders. The cylinders were charged 2 months in advance to allow sufficient mixing time. Samples of each cylinder were analyzed with a portable Burrell gas analysis apparatus.

Several modifications were required before the apparatus could be used to determine the viscosity of the steam-helium and steam-oxygen mixtures. Pure gas (helium or oxygen) from the supply tank was passed through the regulator and then into



Figure 3. Bleed and flow measurement section

the saturator as shown in Figure 1. The saturator consisted of a salt bath, preheating coils, and the saturator vessel. The salt bath contained Aeroheat, and it was controlled to within $\pm 0.3^{\circ}$ C. of the set point by a Brown electronic potentiometer recorder controller. The preheating coils consisted of 40 feet of standard stainless 304 pressure tubing. The saturator vessel was a standard Aminco type 347 stainless steel pressure vessel. It was filled to a depth of 4 inches with distilled degassed water and with steel packing in the form of 1/4-inch rings.

The gas was preheated in the coil, and then bubbled through water in the Aminco autoclave. The gas became saturated with vapor as it bubbled through the water, the composition depending upon the temperature and total pressure. The mixture thus generated was passed through a heated tube into the preheater, and then to the viscometer.

This saturator was used in an earlier investigation to determine the saturation composition of the steam-helium-water system (8).

Change in the viscometer itself was limited to the leads to the Barton gage. The standard pressure tubing leads to the Barton gage as illustrated to the left of the gage in Figure 2 were removed. These leads were replaced with 1/4-inch double extrastrong stainless steel pipe as is illustrated to the right of the gage in Figure 2. Both leads were heated up to the cross as is shown in Figure 2. The unheated parts of the pressure leads and the Barton gage itself were filled with distilled degassed water. Thus, when a steady state was reached during a run, a water meniscus was present in the 1/4-inch tees of both of the lead lines.

The orifice and bleed sections were essentially the same as for permanent gases, except that the valve was located after the orifice and the entire section, excepting the valve and the pressure gage, was wrapped with Nichrome resistance wire.

The steam-gas mixture, after expansion to atmospheric pressure, was passed through an ice trap where most of the water was condensed. The remaining water was removed in a gas drying tube filled with Drierite and the dried gas was metered in the wet test meter.

The temperatures of the five sections of the apparatus (two preheat lines, two lead lines, one orifice) were controlled manually. Only the section between the final preheat bath and the viscometer had to be maintained at a very precise temperature. The others had to be maintained hot enough to prevent condensation inside the lines.

Calibration of Elements. The thermocouples used were Chromel-Alumel and were made by mercury arc welding under a layer of oil. The couple used for measuring the temperature in the viscometer was previously used by Luker (δ) for P-V-T studies. It was calibrated to $\pm 0.2^{\circ}$ C.

The pressure gage was calibrated both by the manufacturer

and in the laboratory. It was found to be accurate to ± 2 p.s.i., the precision with which the gage could be read.

The Barton gage was calibrated by connecting a high pressure water manometer in parallel and introducing a small pressure differential into the system. The water manometer was read with a cathetometer to the nearest 0.01 cm. of water. The gage used was found to be correct as read. It was precise to ± 0.03 inch of water.

Different wet-test meters were used. All were calibrated by displacing a volume of air through the meter with water at a known temperature. It was found that the amount of water in the meter was very critical. Therefore instead of relying on the leveling needle on the meter, the amount of water in the meter was fixed by maintaining the gross weight constant. Using this technique, the precision of the wet-test meters used was $\pm 1\%$.

The gas analyzer was checked by analyzing air for its oxygen content. This analysis gave between 20.6 and 21.0% of oxygen for several analyses.

Because the diameter of the capillary tube could not be accurately measured, the procedure followed in this and most other experiments with capillary viscometers was to calibrate the apparatus using a gas of known viscosity and obtain the term L/D^4 as a calibration constant. The gas selected for calibration purposes was nitrogen because its viscosity has been very precisely measured at 25°, 50°, and 75° C. from 1 to 1000 atm. (9). Calibration runs were made at 50° C. Summarized in Table I are the results of the calibration runs.

To clarify these results two viscometers of identical construction were used in this study. Viscometer 1 was destroyed by an explosion, so the second viscometer was constructed to complete the study. The calibration constant was checked periodically during the investigation. Only once during the study was a significant change noted in the calibration constant.

| Table | I. Calibratio | on Constant | ts, $L/D^4 \times 10^4$ | ⁴ Cm. ⁻³ |
|------------|---------------|-------------|-------------------------|--------------------------------|
| Viscometer | No. of | Mean | Standard | % |
| Number | Runs | Value | Deviation | Deviation |
| 1 | 19 | 532 | 2.6 | 0.48 |
| 2 | 13 | 784 | 5.2 | 0.66 |

| Table II. Viscosity of Helium | | | | | | | |
|-------------------------------|-----------|----------|------------------------------|--|--|--|--|
| Temp., | Pressure, | Reynolds | Viscosity | | | | |
| °C. | P.S.I.A. | No. | Centipoise X 10 ⁴ | | | | |
| 307 | 1027 | 47 | 320 | | | | |
| 304 | 1032 | 39 | 318 | | | | |
| 305 | 1289 | 45 | 320 | | | | |
| 304 | 1810 | 70 | 321 | | | | |
| 304 | 1902 | 79 | 322 | | | | |
| 244 | 1867 | 81 | 300 | | | | |
| 197 | 1853 | 88 | 275 | | | | |
| 360 | 1841 | 72 | 335 | | | | |
| 409 | 1819 | 59 | 355 | | | | |
| 409 | 1804 | 86 | 355 | | | | |
| 405 | 300 | 14 | 349 | | | | |
| 406 | 300 | 20 | 350 | | | | |
| 353 | 303 | 22 | 318 | | | | |
| 352 | 300 | 27 | 318 | | | | |
| 381 | 307 | 20 | 337 | | | | |
| 326 | 311 | 21 | 321 | | | | |
| 291 | 312 | 22 | 308 | | | | |
| 267 | 309 | 22 | 301 | | | | |
| 221 | 309 | 23 | 286 | | | | |
| 178 | 309 | 25 | 264 | | | | |
| 177 | 1,19 | 16 | 263 | | | | |
| 218 | 120 | 14 | 283 | | | | |
| 253 | 120 | 13 | 296 | | | | |
| 55 | 312 | 48 | 213 | | | | |
| 79 | 312 | 47 | 222 | | | | |
| 287 | 1030 | 43 | 305 | | | | |
| 286 | 1088 | 43 | 306 | | | | |
| 266 | 569 | 40 | 301 | | | | |
| 281 | 563 | 39 | 307 | | | | |
| 309 | 542 | 35 | 322 | | | | |

| | Table III. Visc | osity of Oxyge | n |
|--------------|-----------------|----------------|------------------------------|
| Temperature. | Pressure. | Revnolds | Viscosity. |
| °C. | P.S.I.A. | No. | Centipoise X 10 ⁴ |
| 297 | 1461 | 445 | 350 |
| 297 | 1461 | 403 | 351 |
| 270 | 1401 | 338 | 328 |
| 197 | 1430 | 357 | 305 |
| 221 | 1439 | 340 | 316 |
| 274 | 1451 | 759 | 334 |
| 325 | 1446 | 419 | 356 |
| 352 | 1456 | 443 | 367 |
| 375 | 1462 | 454 | 376 |
| 399 | 1462 | 400 | 387 |
| 426 | 1478 | 388 | 397 |
| 451 | 1501 | 385 | 405 |
| 294 | 1409 | 422 | 344 |
| 293 | 737 | 341 | 341 |
| 324 | 732 | 319 | 348 |
| 297 | 752 | 414 | 336 |
| 323 | 756 | 345 | 346 |
| 56 | 1472 | 1300 | 264 |
| 80 | 1466 | 1350 | 272 |
| 202 | 772 | 308 | 296 |
| 243 | 763 | 307 | 314 |
| 276 | 760 | 299 | 332 |
| 321 | 755 | 275 | 348 |
| 354 | 751 | 261 | 359 |
| 449 | 748 | 167 | 398 |
| 415 | 743 | 171 | 385 |
| 192 | 1886 | 728 | 303 |
| 230 | 1882 | 463 | 335 |
| 258 | 1876 | 493 | 331 |
| 298 | 1871 | 480 | 343 |
| 430 | 1828 | 231 | 367 |
| 375 | 1816 | 417 | 371 |
| 330 | 1813 | 460 | 355 |

The purity of the gases used was stated by the manufacturer to be better than 99.6%. The helium in particular was better than 99.95% pure.

EXPERIMENTAL PROCEDURE

Operating the viscometer using a noncondensable gas was a very simple procedure. The three heated parts, the inlet lines, the preheat bath, and the viscometer itself, were brought to the desired temperature. While the apparatus was heating, a vacuum pump was connected in place of the orifice and the entire system was evacuated. The gas was then permitted to flow into the apparatus from the supply tank. When the pressure reached the desired level, the bypass valve on the Barton gage was closed and the orifice was placed at the outlet of the tube

Gas flow was started by opening the valve upstream from the orifice. Immediately a pressure difference could be observed on the Barton gage. After 10 to 20 minutes, the system reached steady state. During the run, the time was recorded whenever the wet-test meter recorded a full revolution. This gave the time necessary for 0.1 cubic foot of gas to pass through the meter. Other data recorded were the Barton gage reading, the pressure, the temperature in the viscometer, the temperature of the wet-test meter, and the barometric pressure. Runs were continued to allow a minimum of 0.2 cubic foot of gas to flow through the apparatus at steady conditions. A steady state was said to exist if neither the pressure differential nor the time for 0.1 cubic foot varied more than 1% during the course of a run. Other data, such as the temperatures in the preheat bath and inlet, were observed regularly but not recorded.

Running the viscometer with the steam mixtures was hardly as simple as the aforementioned procedure. First, the lead lines and Barton gage were filled with water. The preheat system was revised according to Figure 1. The orifice was selected and installed. Water was charged into the saturator by forcing it under pressure of the other gaseous component to be used from a second bomb. In so doing, the entire system became filled

Table IV. Viscosity of Helium-Oxygen Mixtures in Centipoises

| | 28.3 Mole | % Helium | | | 46.9 Mole | % Helium | | | 72.0 Mole | % Helium | |
|---------------|-----------------------|-----------------|-------------------------------------|---------------|-----------------------|-----------------|-------------------------------------|---------------|-----------------------|-----------------|-------------------------------------|
| Temp., °C. | Pressure, P.S.I.A. | Reynolds No. | Viscosity, Cp. X 10 ⁴ | Temp., °C. | Pressure, P.S.I.A. | Reynolds No. | Viscosity, Cp. X 10 ⁴ | Temp., °C. | Pressure, P.S.I.A. | Reynolds No. | Viscosity, Cp. X 10 ⁴ |
| 195 | 1873 | 419 | 304 | 239 | 1007 | 348 | 328 | 410 | 1947 | 111 | 390 |
| 224 | 1867 | 430 | 318 | 212 | 1010 | 200 | 314 | 369 | 1939 | 109 | 386 |
| 269 | 1821 | 374 | 336 | 282 | 1012 | 142 | 345 | 341 | 1932 | 114 | 364 |
| 309 | 1811 | 353 | 353 | 313 | 1013 | 125 | 354 | 310 | 1931 | 122 | 348 |
| 352 | 1810 | 335 | 369 | 342 | 1019 | 122 | 366 | 277 | 1928 | 127 | 336 |
| 379 | 1799 | 197 | 380 | 363 | 1016 | 127 | 374 | 245 | 1914 | 166 | 320 |
| 444 | 1793 | 191 | 396 | 428 | 1029 | 117 | 395 | 212 | 1905 | 169 | 308 |
| 415 | 1786 | 193 | 393 | 309 | 663 | 156 | 354 | 190 | 1899 | 175 | 297 |
| 385 | 1777 | 199 | 379 | 423 | 659 | 96 | 394 | 190 | 1283 | 144 | 296 |
| 383 | 1290 | 221 | 378 | 391 | 657 | 96 | 386 | 220 | 1278 | 131 | 318 |
| 351 | 1286 | 228 | 366 | 355 | 654 | 103 | 365 | 256 | 1273 | 129 | 324 |
| 325 | 1281 | 234 | 354 | 329 | 650 | 101 | 362 | 303 | 1247 | 118 | 341 |
| 297 | 1278 | 238 | 344 | 282 | 640 | 101 | 337 | 338 | 1237 | 110 | 360 |
| 272 | 1270 | 241 | 336 | 254 | 635 | 105 | 324 | 434 | 1222 | 94 | 396 |
| 446 | 1266 | 202 | 400 | 224 | 632 | 111 | 314 | 370 | 1190 | 72 | 371 |
| 414 | 1256 | 205 | 389 | | | | | 406 | 1182 | 67 | 388 |
| 260 | 1243 | 240 | 328 | | | | | 334 | 1178 | 71 | 354 |
| 221 | 1237 | 248 | 314 | | | | | 431 | 675 | 72 | 393 |
| 202 | 1229 | 253 | 306 | | | | | 394 | 670 | 74 | 378 |
| 198 | 660 | 196 | 396 | | | | | 364 | 664 | 76 | 364 |
| 225 | 660 | 191 | 313 | | | | | 340 | 660 | 78 | 358 |
| 269 | 656 | 182 | 328 | | | | | 305 | 655 | 82 | 341 |
| 293 | 650 | 177 | 339 | | | | | 284 | 651 | 83 | 332 |
| 321 | 648 | 167 | 348 | | | | | 252 | 645 | 85 | 320 |
| 345 | 642 | 160 | 360 | | | | | 220 | 640 | 87 | 305 |
| 445 | 636 | 145 | 392 | | | | | 192 | 633 | 86 | 296 |
| 408 | 627 | 149 | 381 | | | | | | | | |

with steam at the saturation pressure. The pressure was then increased to the desired total pressure by introducing the inert gaseous component.

After charging was completed, the heaters on the various sections of the apparatus were turned on and brought to the desired temperature along with the preheater bath. When the desired temperature was reached, the outlet valve was opened and the steam-gas mixture started to flow from the system.

As a pressure differential appeared on the Barton gage, an unbalanced condition was produced in the leads to the gage. This condition caused a small amount of water to flow from the cool section of the low pressure lead line into the heated section where it vaporized. Simultaneously an equivalent amount of water vapor flowed from the heated high pressure line into the cooled section where it condensed. During the equalization the needle of the Barton gage showed marked oscillation. After an hour or more the pressure differential became constant.

After the pressure drop across the capillary became constant, the efflux was directed into the recovery system, and a run was started. Data taken were similar to those taken for permanent gases except that the weight of water condensed in the traps was also recorded. Runs were continued until 0.2 cubic foot of the gaseous part of the mixture had passed through the wettest meter. The steady state obtained with this technique was not as satisfactory as with permanent gases. Some cycling was evident in most cases.

Table V. Effect of Composition on the Viscosity (Centipoise X 10⁴) of Helium-Oxygen

| Mixtures | at | 300. | · C. | |
|----------|----|------|------|--|
| | | | | |

| Pressure, Atm. | - | _ | | | |
|----------------|-----|------|------|------|-----|
| | 100 | 72.0 | 46.9 | 28.0 | 0 |
| 45 | 335 | 363 | 368 | 364 | 363 |
| 85 | 336 | 367 | 372 | 368 | 368 |
| 125 | 338 | 371 | | 372 | 372 |

RESULTS AND DISCUSSION

The experimental results obtained are tabulated in Tables II to VII, and they are presented graphically in Figures 4 to 10.

Before discussing the results, the experimental uncertainty of the data should be considered. For the noncondensable gases studied, the experimental measurements had the following minimum accuracy:

| $\pm 1^{\circ}$ C. |
|--------------------------|
| ± 0.03 inch of water |
| ±2 p.s.i. |
| $\pm 1\%$ |
| |

In the discussion of the apparatus, the calibration constants for the two viscometers were shown to have average deviations of ± 0.48 and $\pm 0.66\%$ respectively.



(12)

(16)

(3)

- Data of this work
- Trautz and Binkele ×
- Trautz and Zink
- Trautz and Heberling (13)(14)
- + Trautz and Hesseini I Wobser and Muller (17)
- I- Johnston and Grilly





Calculation of the viscosity from the above measurements using Poiseuille's law with the calibration constant which had the larger average deviation, could produce a maximum error of $\pm 3\%$.

However, probably the best way to arrive at a realistic value of the precision of the data is to consider the deviations in the calibration constants. For the first viscometer, the maximum deviation of the calibration constant from the mean value was -0.94%. For the second viscometer the maximum deviation from the mean value was -1.0%. Thus, from the calibration constant data, it may be concluded that the precision of the data was probably $\pm 1\%$. The accuracy of the data is dependent upon the accuracy of the data used for calibration purposes. The data of Michels and Gibson (9) which were used for the calibration show a precision in the order of 0.1%; however, it is possible the accuracy is poorer. A reliable estimate of the probable accuracy of the results presented for noncondensables is $\pm 1.5\%$.

Figure 4 shows the viscosity isobars of pure helium at 125 and 20 atm. Also plotted on this graph are the results of six investigations of the viscosity of helium at 1 atm. The viscosity of helium was found to increase approximately 4% as the pressure increased from 1 to 125 atm. This was somewhat greater than was found by Kestin and Pilarczyk (6) who observed an increase of 0.4% from 1 to 51 atm. at 20°C. Actually the increase was so small that it could not be determined very precisely with the apparatus used.

The viscosity of oxygen is plotted in Figure 5. Isobars were determined at 50 and 100 atm. Results of three investigations at 1 atm. and the smoothed isobars of Kiyama and Makita (7) up to 100 atm. are shown also. The two investigations at elevated pressures appear to agree very well, although they are in different temperature ranges.

Isobars of the viscosity of three helium-oxygen mixtures are shown in Figures 6, 7, and 8. Unfortunately, there are no data available at 1 atm. for comparison. The results tabulated in Table V show the effect of composition on the viscosity of these mixtures. The viscosity of these mixtures is substantially independent of the helium content up to the maximum helium content investigated.

The viscosity data for steam, steam-helium, and steamoxygen mixtures are tabulated in Tables VI and VII. The previous discussion of experimental uncertainties is also applicable to these data. Several other uncertainties are encountered in measuring the viscosities of condensables. In particular, it was impossible to observe the water vapor meniscus visually in the lead lines to the Barton gage. However, the technique used in establishing a steady reading on the Barton gage is believed to be a satisfactory method of equalizing the water levels in the gage lead lines. Also, it can be seen from the tabulated data that the composition varied somewhat from run to run for the steam-gas mixtures. The most probable accuracy for these data on condensables is $\pm 3\%$.

As shown in Table VII the viscosity of steam was measured to assure that the apparatus could be used for condensable



gases. The viscosity of steam was determined to be 260×10^4 and 264×10^4 centipoises at 280° C., 710 p.s.i.a. and 320° C., 708 p.s.i.a. These viscosities compare favorably with the smoothed data of Hawkins, Solberg, and Potter (261×10^4 and 267×10^4 , respectively) as currently presented in the steam tables (5). The data showed rather large discrepancies when compared to the smoothed data of Timroth (11) (196 $\times 10^4$ and 214 $\times 10^4$, respectively). From the above comparisons, the viscometer was judged to function adequately for condensable gases. It was also concluded that the viscosity of the steam-

| Table | VI. | Viscosity of Steam-Oxygen Mixtures |
|-------|-----|------------------------------------|
|-------|-----|------------------------------------|

| Temp., | Pressure, | Reynolds | Viscosity, | Mole Fraction |
|--------|-------------|----------|----------------------|---------------|
| °C. | P.S.I.A. | No. | Cp X 10 ⁴ | Oxygen |
| 404 | 1997 | 214 | 330 | 0.364 |
| 383 | 1995 | 614 | 308 | 0.288 |
| 369 | 1989 | 606 | 310 | 0.269 |
| 363 | 2000 | 632 | 302 | 0.276 |
| 348 | 1999 | 655 | 300 | 0.334 |
| 331 | 1993 | 598 | 309 | 0.312 |
| 310 | 1996 | 623 | 29 0 | 0.256 |
| 277 | 989 | 375 | 247 | 0.262 |
| 326 | 1000 | 406 | 270 | 0.264 |
| 302 | 999 | 434 | 253 | 0.236 |
| 378 | 1000 | 113 | 301 | 0.301 |
| 392 | 9 90 | 118 | 303 | 0.325 |
| 283 | 993 | 427 | 267 | 0.320 |
| 304 | 985 | 376 | 254 | 0.240 |
| 327 | 985 | 408 | 261 | 0.222 |
| 350 | 980 | 416 | 272 | 0.249 |

| Table VII. | Viscosity o | f Steam and | Steam-Heli | um Mixtures |
|---------------|-----------------------|-----------------|-------------------------------------|-------------------------|
| Temp., °C. | Pressure, P.S.I.A. | Reynolds No. | Viscosity, Cp. X 10 ⁴ | Mole Fraction Helium |
| 280 | 710 | 262 | 260 | 0. |
| 320 | 708 | 272 | 337 | 0. |
| 342 | 1988 | 573 | 251 | 0.273 |
| 343 | 2010 | 953 | 255 | 0.230 |
| 315 | 2018 | 725 | 247 | 0.279 |
| 335 | 2028 | 792 | 263 | 0.258 |
| 361 | 2018 | 701 | 265 | 0.288 |
| 396 | 2009 | 612 | 255 | 0.285 |
| 358 | 2000 | 465 | 253 | 0.345 |
| 321 | 2011 | 537 | 248 | 0.252 |
| 329 | 2010 | 398 | 254 | 0.356 |
| 351 | 1997 | 145 | 259 | 0.374 |
| 315 | 994 | 518 | 244 | 0.209 |
| 342 | 991 | 517 | 246 | 0.199 |
| 293 | 996 | 283 | 240 | 0.254 |
| 384 | 989 | 317 | 255 | 0.310 |
| 370 | 1020 | 312 | 254 | 0.157 |





gas mixtures should be compared to the steam data of Hawkins and others (5).

Figure 9 shows measurements of the viscosity of a 70 mole % steam-30 mole % oxygen mixture at 1000 and 2000 p.s.i.a. as a function of temperature. Also shown are the respective pure component data. The results are considerably more scattered than the data for permanent gases. In general the scattering in the data is very low as compared with most measurements on pure steam. The viscosity of the mixture was determined to be lower than the viscosity of either of the pure components as is illustrated in Figure 9. The steam viscosity data of Timroth (17) are, however, lower than the viscosity of this mixture. As shown in Figure 9, least squares regression lines were fitted to the mixture data. The equations for these lines are:

$$\mu = 0.319t + 190 \text{ at } 2000 \text{ p.s.i.a}$$

 $\mu = 0.452t + 122 \text{ at } 1000 \text{ p.s.i.a.}$

Figure 10 shows the viscosities of a 30 mole % helium-70 mole % steam mixture as well as that of the pure components. The same comments are applicable for this mixture as were presented for the steam-oxygen mixture. The pressure dependency of the viscosity of this mixture is very similar to that of helium. This is of particular interest, because the helium-oxygen mixture was essentially independent of the helium content, and the viscosity of the steam-oxygen mixture showed greater pressure dependence than oxygen.

The equations for the least squares regression lines for the steam-helium mixtures shown in Figure 10 are:

$$\mu = 0.105t + 219$$
 at 2000 p.s.i.a

 $\mu = 0.169t + 190 \text{ at } 1000 \text{ p.s.i.a.}$

When more accurate steam viscosity data are available, the viscosity of these two steam-gas mixtures should be investigated more thoroughly to determine whether these mixtures actually exhibit a minimum as the data presented indicate.

NOMENCLATURE

D = diameter of capillary

L = length of capillary

 $t = \text{temperature}, \circ \mathbf{C}.$

 μ = viscosity, centipoise X 10⁴

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Polymers from the Unsaturated Bisacetals of Pentaerythritol

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 $\mathbf{P}_{ ext{entaerythritol}}$ is finding many new uses as a raw material in resins (1, 4). Some of the interesting thermosetting resins developed recently have been prepared from pentaerythritol or other polyhydroxy compounds and acrolein. Rothrock (7) prepared air drying coatings by a reaction of polyhydroxy compounds with unsaturated aldehydes like acrolein in the presence of strong acid catalysts. Schulz and Wagner (9, 10, 12) described a poly(methyl methacrylate)-like resin having good mechanical properties prepared by condensing pentaerythritol with acrolein and removing the water of acetalization. By using milder catalysts and lower reaction temperatures (10), they were able to isolate the unsaturated bisacetal of pentaerythritol in this condensation. In all of this initial work, the resins were obtained in one stage by introducing stoichiometric quantities of the two reactants and distilling off the by-product water without isolating the intermediate compounds.

Orth (5, 6) demonstrated the advantages of carrying out these

reactions in two stages. In the first stage the unsaturated bisacetal of pentaerythritol was prepared and in the second stage this acetal was polymerized with a polyhydroxy compound to give the resin. If the acetalization and poly addition were run in two steps the following advantages were claimed.

Two polyhydroxy compounds of different reactivity could be used; the slower reacting compound could be fully reacted in the first stage before introducing the faster reacting compound in the second stage.

Lower shrinkage would occur on curing the resin.

Reduced water absorption of the resin would take place.

Mechanical properties of the resin would be improved.

Raw materials of technical quality could be employed in the first stage without affecting the yield, and the product of the first stage could be readily purified.

The general equation for the formation of the resin by the two-stage process is shown below.

+ 2H,O1

