

XXXVII.—*On Heptane from Pinus Sabiniana.*

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IN a paper read to the California Pharmaceutical Society on December 13, 1871, and reprinted in the *Pharmaceutical Journal* for March 30, 1872, Mr. William Wenzell, of San Francisco, describes as new and under the name of *Abietene** a hydrocarbon obtained by distilling the terebinthinate exudation of a coniferous tree, *Pinus sabiniana*, Dougl. This tree is indigenous to California, and grows on the dry slopes of the foot-hills of the Sierra Nevada and on the hills along the

* The name *Abietin* was originally applied by Caillot to a resin obtained from the turpentine of *Abies picea* and *Abies balsamea*.

coast; it appears to be pretty plentiful, and is known locally as the nut pine or Digger pine, names suggested by the circumstance that the Digger Indians rely to some extent on its fruit for food.

In order to procure the exudation, the tree during winter is notched and guttered at a convenient height from the ground, and the resin on distillation yields the liquid hydrocarbon. The crude oil is met with in San Francisco as an article of commerce, and during the last few years has acquired some reputation under the names of "abietene," "erasine," "aurantine," "theoline," &c., as a substitute for "benzoline" or "petroleum benzene" for removing grease-spots, paint-stains, &c., from clothing. It is a nearly colourless mobile liquid of powerful aromatic smell, recalling that of oil of oranges. The crude oil, according to Mr. Wenzell, boils almost entirely between 100° and 105° , and on redistillation the liquid is obtained of the constant boiling point of 101° . The portion boiling above 105° leaves on evaporation a small quantity of a resinoid body possessing the strong penetrating odour of the original product. The liquid is highly inflammable and burns with a brilliant white smokeless flame. Its vapour is powerfully anæsthetic, and it has been used with success as an insecticide.

No analysis or vapour-density determination of this liquid is given in the communication above referred to, nor does Mr. Wenzell offer any opinion as to its real nature. He contrasts its characters with those of terebene (spirits of turpentine), the hydrocarbon obtained from *Pinus palustris*, *P. sylvestris*, &c., and points out the very striking differences observed in their physical and chemical properties. Abietene has a specific gravity of 0.694 at 16.5° , whilst that of terebene is 0.840 at about the same temperature. Oil of turpentine boils at 160° , whilst the boiling point of abietene is 101° . Hydrochloric acid gas is but slightly dissolved by abietene, whereas oil of turpentine absorbs it with avidity. Nitric acid acts violently on oil of turpentine; it has little or no action on abietene in the cold.

Abietene was evidently sufficiently remarkable to merit further investigation. From its general behaviour towards reagents, it seemed not unlikely that it would prove to be a paraffin, and the boiling point and specific gravity appeared to indicate *normal heptane*. The occurrence of heptane, or indeed of any other paraffin, playing the part of oil of turpentine in the vegetable kingdom was hitherto unheard of. The only natural source of this hydrocarbon was petroleum or some of the fossil-fish oils obtained in Greenland, Switzerland, and other places. Menhaden oil contains considerable quantities of heptane, and it is met with among the products of the destructive distillation of cannel and shale.

Through the kindness of Mr. H. B. Brady, F.R.S., I was able to obtain, through Dr. Squibb, of New York, about a couple of gallons

of abietene from Mr. Wenzell. The crude oil fully answered the description already given of it by the latter gentleman. It was almost colourless, and had the strong persistent odour of oil of oranges. It began to boil slightly below 100° , and the greater part distilled below 101° . The residual portion darkened slightly, and on evaporation left a small quantity of resinoid matter of a brown colour, which had the peculiar smell of the liquid in a very high degree. This led to the supposition that the odour of the crude oil might be due in great part to the resin in solution, and further examination proved this to be the case. On agitating it for some time with oil of vitriol the acid became brown, and on again distilling the hydrocarbon was found to have lost its smell of orange oil.

Two determinations of its boiling point were made by the aid of a thermometer capable of being read to 0.01° C. The first observation gave 98.27° at 755.6 mm., the second 97.93° at 746.9 mm. The mercurial columns in both cases were wholly immersed in the vapour, and the barometric pressures are corrected and reduced to 0° C. Assuming the validity of the well-known expression—

$$T = t + 0.0375(b - 760),$$

where t is the observed boiling point and b the barometric pressure, the boiling point, T , under the standard pressure becomes respectively—

I		98.43°
II		98.42

The first determination was made on about three litres of the liquid, the second on about 250 c.c., after a second treatment with a mixture of nitric and sulphuric acids; in both cases the liquid boiled as constantly as distilled water. In all I obtained about seven litres of the perfectly pure substance from seven and a half litres of the crude oil.

That abietene is actually heptane is proved by the following analyses and determinations of vapour-density:—

I. 0.3500 gram hydrocarbon gave 1.0754 gram CO_2 and 0.5050 gram H_2O .

II. 0.3000 gram hydrocarbon gave 0.9215 gram CO_2 and 0.4336 H_2O .

Calculated for C_7H_{16} .		Found.	
		I.	II.
C	 83.97	83.85	83.78
H	 16.03	16.03	16.06
	100.00	99.88	99.84

Two determinations of its vapour-density, made in a modified form of Hofmann's apparatus,* gave the following results:—

I. Weight of liquid	144.5 mgm.
Volume of vapour (corrected for expansion of glass, error of meniscus, &c. . .	90.39 c.c.
Temperature of vapour.....	99.90° C.
Barometer (corrected and reduced)....	757.6 mm.
Height of mercury in tube (reduced) ..	387.8 „
II. Weight of liquid.....	76.9 mgm.
Volume of vapour (corrected)	69.77 cc.
Temperature	99.60° C.
Barometer	749.0 mm.
Height of mercury in tube	493.4 „

	Found.	
Calculated for C_7H_{16} .	I.	II.
49.90	50.07	49.94

I. *The Physical Characters of Heptane from Pinus Sabiniana.*

The possession of so large a quantity of perfectly pure heptane has induced me to study some of the physical characters of this hydrocarbon. These observations have included the determination of its specific gravity, and the measurement of its thermal expansion, refractive index, optical activity, viscosity, and superficial tension.

Specific Gravity.—Three determinations of specific gravity made with different bottles gave the following results:—

0.68848 at 14.98°
 0.68855 „ 14.87
 0.68858 „ 14.87

compared with water at the same temperatures respectively. The weighings were made by the method of vibrations and the weights are reduced to a vacuum. Reducing these results by the aid of Rossetti's tables for the expansion of water, and by means of the formula for the thermal expansion of heptane given below, the specific gravity at 0°, compared with water at 0°, becomes respectively—

0.70057
 0.70055
 0.70058

Mean .. 0.70057

* The apparatus here referred to allows of the determination of a vapour-density with almost as much exactitude as that of any other physical constant; it was designed more especially with a view of controlling the purity of some liquids employed in a research involving the determination of a number of thermal expansions.

Thermal Expansion.—The rate of expansion of the heptane was determined by heating the hydrocarbon in dilatometers properly calibrated and graduated. The volume of the hydrocarbon at 0° was obtained by immersing the dilatometers in melting snow. The instruments were then placed in a large copper bath fitted with plate-glass sides, and holding several litres of water which could be kept in constant motion by stirrers worked by a small hydraulic engine. The water was heated by steam and the temperature ascertained by thermometers capable of being read to 0·01° C.

Two series of observations were made. The first set gave the following results:—

Temperature.*	Observed volume.	Calculated volume.	Temperature.	Observed volume.	Calculated volume.
0·00	3290·1	3290·4	53·71	3516·3	3516·9
9·04	3326·1	3326·0	62·80	3560·1	3559·9
17·27	3359·2	3359·1	72·12	3606·2	3605·7
26·96	3398·9	3399·1	80·68	3649·5	3649·5
35·55	3435·9	3435·7	87·32	3684·2	3684·6
44·96	3477·2	3477·1	93·70	3719·9	3719·4

Combining the results by the method of equation of conditions we obtain the formula—

$$3290\cdot361 + 3\cdot895352t + 0\cdot00432087t^2 + 0\cdot00003183t^3,$$

by means of which the numbers in the third column of the above table are calculated.

The second series made in a different dilatometer gave the following data:—

Temperature.	Observed volume.	Calculated volume.	Temperature.	Observed volume.	Calculated volume.
0·00	2879·6	2879·3	53·71	3076·4	3077·0
9·04	2910·5	2910·6	62·81	3114·5	3114·5
17·27	2939·3	2939·5	72·13	3155·2	3154·7
26·97	2974·2	2974·5	80·68	3193·6	3193·2
35·54	3006·4	3006·2	87·27	3223·8	3224·1
44·96	3042·3	3042·3	93·71	3255·3	3255·4

* These temperatures are expressed in degrees of the air thermometer in order to make them uniform with my other observations of thermal expansion. The conversion from the ordinary thermometric values is effected by a table compiled from Regnault's and Recknagel's comparisons of the air and mercurial thermometers (see Wullner's *Lehrbuch der Physik*). To convert the degrees of the air thermometer into those of the mercurial thermometer add 0·08° at 10°, 0·16° at 25°, 0·16° at 65°, 0·1 at 80°.

These numbers afford the expression—

$$2879\cdot302 + 3\cdot432882t + 0\cdot00247831t^2 + 0\cdot00003964t^3,$$

by the aid of which the numbers in the third column of the above table are computed.

Dividing through by the first terms respectively and correcting for the expansion of the glass of the dilatometers ($0\cdot00002130$ for 1° in Series I, and $0\cdot00002303$ in Series II) the formulæ become—

$$\text{I. } 1 + 0\cdot00120517t + 0\cdot00000133841t^2 + 0\cdot00000009701t^3.$$

and

$$\text{II. } 1 + 0\cdot00121529t + 0\cdot00000088819t^2 + 0\cdot000000013787t^3.$$

The mean formula is—

$$1 + 0\cdot00121023t + 0\cdot0000011133t^2 + 0\cdot00000001174t^3,$$

by the aid of which the following table showing the relative volumes of heptane from *Pinus sabiniana* at every 5° between 0° and 100° is calculated:—

Tempera- ture.	Volume.	Difference.	Tempera- ture.	Volume.	Difference.
0	100000	—	55	107188	712
5	100608	608	60	107916	728
10	101222	614	65	108659	743
15	101844	622	70	109420	761
20	102474	630	75	110198	778
25	103114	640	80	110995	797
30	103763	649	85	111812	817
35	104423	660	90	112650	838
40	105094	671	95	113509	859
45	105778	684	100	114390	881
50	106476	698			

The volume at the boiling point $98\cdot43^\circ$ is $1\cdot14111$: hence the specific gravity at this temperature is $0\cdot61393$, which gives for heptane the specific volume $162\cdot54$. Kopp's values ($C = 11$; $H = 5\cdot5$) afford the number 165 .

Refractive Index.—A careful set of observations of the index of refraction of heptane from *Pinus sabiniana* was made for me by Mr. John I. Watts in the Physical Laboratory of the Owens College by one of Elliott's horizontal goniometers.

Five concordant determinations of the angle of the prism employed gave as a mean result—

$$\theta = 59^\circ 12' 20''.$$

The same number of observations of the angle of minimum deviation gave—

$$\delta = 27^{\circ} 21' 44''.$$

The source of light was a sodium flame. The temperature of the liquid was 17.6° .

Inserting these numbers in the well-known formula—

$$\mu = \frac{\sin. \frac{1}{2}(\delta + \theta)}{\sin. \frac{1}{2} \theta}.$$

we obtain 1.3879 as the refractive index for D.

The *specific refractive energy*, $\frac{\mu - 1}{d}$, where d is the specific gravity of the liquid at 17.6° , is 0.565; whence the *molecular refractive energy* is 56.4, in close accordance with the computed number 55.8, calculated by Landolt's values, C = 5, H = 1.3.

Optical Activity.—It is well known that the various terpenes are optically active, often to a very remarkable extent: hence a special interest seemed to be attached to the question of the behaviour of the heptane of *P. sabiniana* towards polarised light. Through the kindness of Professor Gamgee, Mr. J. H. Poynting was able to make a number of observations on the hydrocarbon by means of an excellent Laurent's polarimeter belonging to the Physiological Laboratory of the Owens College. These observations seem to indicate that the heptane does actually rotate the plane of the polarised ray, although to a very slight extent only, a column 200 mm. long exhibiting a rotatory power of $+6.9'$. This amount is so small, that I was disposed to attribute it to the possible presence of impurities, despite the careful rectification to which the hydrocarbon had been subjected: the original exudation might have contained small quantities of a terpene, or a minute trace of the resinous matter which it has been stated the crude oil leaves on evaporation might still be contained in the distilled hydrocarbon. The liquid with which Mr. Poynting experimented was accordingly agitated with a mixture of concentrated nitric and sulphuric acids at frequent intervals for several days, but the acids remained perfectly colourless, and the hydrocarbon after redistillation showed precisely the same rotatory power as before, viz., $+6.9'$. I am inclined to believe, however, that an optically active terpene is actually present in crude abietene, although not to a greater extent than 0.5 per cent.: the liquid residue left on distilling the crude oil boiled between 140° and 160° , and showed a rotatory power of $-12^{\circ} 23.5'$: this liquid, it should be noted, had not been treated with acids. The resin itself, in alcoholic solution, appears to be optically inactive.

Viscosity.—The capillary transpiration of the heptane from *Pinus sabiniana* was determined by a method identical in principle with

that already employed by Poiseuille, Graham, and others; this consists in noting the time required for a given volume of the liquid at some particular temperature and under a uniform pressure to flow through a cylindrical capillary tube of known dimensions. The capillary tube employed was 85.4 mm. in length and 0.2105 mm. in internal diameter: it was sealed, at right angles, to a long cylindrical bulb, containing between two marks etched on the narrow tube above and below the bulb 14.7755 c.c. The open end of the capillary tube was connected with a receiver and the whole was so arranged that it could be immersed in a large bath fitted with plate-glass sides and holding several gallons of water. The temperature of the bath, and therefore of the hydrocarbon contained in the bulb and tubes, could be raised to any desired point below about 80° by heating a coil of copper tube affixed to the side of the bath through which the water from the bath continually circulated. The liquid in the bath was kept in constant agitation by means of stirrers worked by a small hydraulic engine; and the temperature was ascertained by a thermometer capable of being read to 0.01° C. The hydrocarbon was drawn into the bulb by the aid of the air-pump, and as soon as it had acquired the temperature of the bath, it was forced through the capillary tube by the pressure of a confined volume of air kept at a nearly constant tension of 111.2 mm. of mercury by the weight of a column of water of uniform height; and the times at which the level of the liquid passed the two marks, as observed through a telescope, were noted on a chronometer. This apparatus has been employed by Mr. John Muir, student in the Laboratory of the Yorkshire College, in the determination of the viscosity coefficients of a number of liquids; a detailed description of it is reserved for a communication containing the results of his experiments.

Two independent series of observations were made with heptane with the following results:—

Series I.

Temperature.	Time (seconds).	V.	η .	Temperature.	Time (seconds).	V.	η .
6.72°	931.7	7313	.005268	35.07°	611.1	11151	.003454
10.27	794.3	8579	.004490	40.16	582.2	11704	.003292
15.31	749.3	9094	.004236	44.75	560.4	12159	.003168
20.05	710.9	9585	.004019	49.70	534.7	12745	.003023
25.06	673.8	10113	.003810	54.84	512.0	13309	.002895
29.95	642.9	10599	.003635				

Series II.

Temperature.	Time (seconds).	V.	η .	Temperature.	Time (seconds).	V.	η .
7.09°	845.0	8064	.004777	30.06°	639.6	10654	.003616
10.31	792.9	8594	.004482	35.03	611.6	11141	.003457
15.30	749.3	9094	.004236	39.16	585.2	11645	.003300
20.15	713.0	9557	.004031	45.37	555.7	12262	.003142
25.09	673.6	10116	.003808	49.30	538.0	12666	.003042

The transpiration times are corrected for any slight irregularities in the pressure (these never exceeded 0.4 mm. of mercury), and for the change in the dimensions of the bulb and capillary tube by heat.

If Q be the volume of the liquid flowing through the tube in the time t , p the pressure under which it is driven expressed in millimetres of water, l the length of the tube, and r its radius in millimetres, then the volume, V , in cubic millimetres flowing in one second of time through a tube 1 mm. long, 1 mm. radius, and under a pressure of 1 mm. of water, on the assumption that Poiseuille's laws hold good under these conditions, is given by the formula—

$$V = \frac{Q \cdot l}{t \cdot p \cdot r^4}.$$

The numbers so obtained are given in the third column of the above tables.

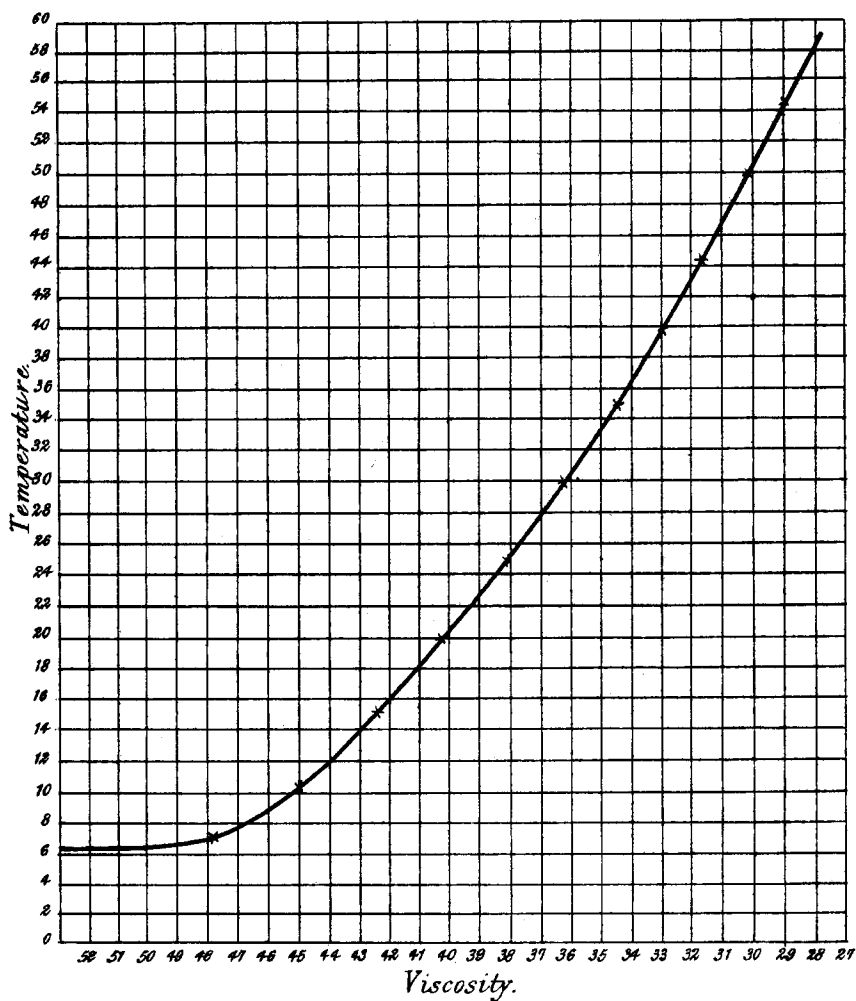
If all the particles of the moving liquid are flowing in parallel straight lines, with velocities proportional to their distances from a plane parallel to these lines, then of any two contiguous layers, the motion of that which moves the more rapidly will be retarded by a force (viscosity) dependent, *ceteris paribus*, on the nature of the liquid, and varying with its temperature. The coefficient of viscosity in absolute measure is the force expressed in dynes thus exerted on a liquid surface one square centimetre in area, lying in a plane of uniform velocity, when the difference of the velocities of two layers one centimetre apart is one centimetre per second.

The viscosity coefficients, η , of heptane for the particular temperatures given in the table, are directly obtained from the values of V in absolute measure by the expression—

$$\eta = \frac{\pi \cdot 98.1}{8 \cdot V},$$

by means of which the numbers in the last columns of the above tables are calculated. These results are graphically represented in the accompanying figure.

It will be noticed that the viscosity suddenly diminishes at some



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point between 8° and 10° . After the latter temperature has been reached the rate of diminution is very regular, and may be represented by an expression of the form—

$$\eta = a + bt + ct^2.$$

Combining all the observations in both series of experiments made at temperatures above 15° by the method of equation of conditions, we obtain the formula—

$$\eta = 0.005003 - 0.00005501t + 0.0000003061t^2,$$

which gives results in close accordance with the observed mean values, as the following comparison between the experimental and computed values shows:—

t° .	η .		Differ- ence.	t° .	η .		Differ- ence.
	Observed.	Calcu- lated.			Observed.	Calcu- lated.	
15.31	.004236	.004233	.000003	39.66	.003300	.003303	.000003
30.10	.004025	.004021	.000004	45.06	.003155	.003145	.000010
25.07	.003809	.003816	.000007	49.50	.003032	.003031	.000001
30.01	.003625	.003628	.000003	54.84	.002895	.002907	.000012
35.05	.003456	.003451	.000005	98.43	—	.002554	—

Surface Tension.—The superficial tension of the heptane from *P. sabiniana* was determined (1) by observations on the rise of the hydrocarbon in a cylindrical capillary tube of known diameter, and (2) by measurements of the form of an air-bubble placed under a plate of glass held under and parallel to the surface of the liquid.

In the first series of observations the apparatus was so arranged that the relation of the surface tension to the temperature could be ascertained from observations on the rise of the liquid in the same tube at different temperatures. A detailed description of the apparatus is reserved for a communication giving the results of an extended series of observations on the surface tension of liquids with special reference to their chemical relations. The measurements were made by the aid of a cathetometer belonging to my colleague, Professor Rücker, and reading to 0.02 mm., and each result is the mean of some six or eight observations. Diameter of tube 0.2531 mm.

Temperature.	Height from the outside level of the liquid to the lowest point of the meniscus in the capillary tube.	Calculated.	Difference.
	Observed.		
7·0	49·62 mm.	49·76	+0·14
15·6	48·36	48·20	-0·16
23·8	46·70	46·71	+0·01
30·2	45·58	45·54	-0·04
38·4	43·99	44·05	+0·06
45·5	42·72	42·76	+0·04
54·6	41·12	41·11	-0·01
60·4	40·10	40·05	-0·05
69·5	38·44	38·39	-0·05
76·8	36·99	47·07	+0·08

No correction has been made for the increase in the diameter of the tube with the temperature. The correction for a difference of temperature of 100° would not affect the fourth place of decimals in the value of the diameter. Combining the observations by the method of least squares, we find that the height of the liquid in the capillary tube may be accurately represented by the expression—

$$h = 58.032 - 0.1818t.$$

The values calculated by this formula are given in the third column of the above table.

The above formula for a tube 1 mm. in radius becomes—

$$H = 6.4581 - 0.0230067t.$$

If the constant of capillarity (a) be considered as half the weight in milligrams of a column of liquid, of which the area of the base is 1 square millimetre, and of which the height is that of a column of liquid in a tube 1 mm. radius, then—

$$a = \frac{H \cdot s}{2},$$

in which s is the specific gravity of the liquid. This for heptane at 0° is—

$$a = \frac{6.4581 \times 0.70057}{2} = 2.2621 \text{ mgm.}$$

Neglecting the angle of capillarity, which, as the observations with the air-bubble show is certainly not very far from 180° , the surface tension in C. G. S. units at 0° is—

$$T = 2.2621 \times 9.81 = 22.19,$$

and its relation to the temperature is expressed by—

$$T = 22.19(1 - 0.003563t).$$

The observations with the air-bubble were made practically in the manner described by Quincke (*Pogg. Ann.*, **139**, 1). A rectangular trough $2\frac{1}{2}$ inches broad, 3 inches long, and 1 inch deep, made of plane parallel plates of glass, cemented together at the edges, was levelled before the cathetometer and filled with the heptane, and another plate of glass was then immersed in the liquid to a depth of a few millimetres, and carefully adjusted until it was parallel with the surface. A bubble of air about 30 mm. in diameter was then blown under the glass by means of a bent capillary tube, and its depth (K), together with that of the point where its tangent plane was vertical (k), was determined by the cathetometer.

The value of the superficial tension in C. G. S. units is given by the expression—

$$T = \frac{1}{2}(K - k)^2gs.$$

In the first set of observations ten readings of K and k were taken. The temperature was 15° .

$$K = 3.622 \text{ mm.}$$

$$k = 1.083$$

$$g = 9.81$$

$$s \text{ at } 15^\circ = 0.6882.$$

$$\text{Hence } T = 21.80.$$

In the second set five readings of K and k were taken on another bubble. The temperature was 10° .

$$K = 3.452 \text{ mm.}$$

$$k = 0.960.$$

$$s = 0.6922.$$

$$\text{Hence } T = 21.12.$$

These numbers agree very well with those deduced from the height of the liquid in the capillary tube.

The method of measurement was scarcely accurate enough to afford any very reliable value of the angle of capillarity. This is given by the formula—

$$\text{Cos. } \theta = \frac{K^2}{(K - k)^2} - 1.$$

Combining all the observations, we find the angle of capillarity of heptane 167° . It is worthy of note that the heptane from *Pinus sabiniana* has the lowest surface tension of any liquid yet measured.

Since the superficial tension of a liquid entirely vanishes at the critical point, that is at the temperature at which the gaseous and liquid states become continuous, the expression $1 - .003563t$ may afford us an approximation to the critical point of heptane, if we assume that the rate of diminution in capillarity is represented by this

formula at high temperatures. We thus find the critical point of the heptane to be 281° .

Whether the heptane from *Pinus sabiniana* is absolutely identical with that boiling at about the same temperature obtained from petroleum and shale oil remains to be established. The existing evidence, so far as it goes, seems to indicate that the hydrocarbons are really isomeric.

In May, 1874, Professor Schorlemmer, to whom I am indebted for a number of organic preparations for determination of certain of their physical constants, kindly sent me a quantity of normal heptane from petroleum, which he believed to be practically free from the isomeric boiling at 90° , and from octane. I found it to boil constantly between 98.1° and 99.1° under a pressure of 765 mm. Its specific gravity at 0° compared with water at 4° was 0.7299.

In May, 1877, Professor Schorlemmer sent me a second sample of petroleum heptane which had been prepared with special care. This I found to boil between 98.1° and 98.6° under a barometric pressure of 750.3 mm.

A determination of its vapour-density afforded the following data:—

Weight of hydrocarbon.....	150.7 mgm.
Volume of vapour	91.28 c.c.
Temperature	99.8° C.
Pressure	381.3 mm.
Calculated.	Observed.
49.9	50.1

Two determinations of its specific gravity at 0° compared with water at 4° gave—

I	0.7301
II	0.7301

numbers which are almost identical with that afforded by the first sample, and much higher than that of the heptane from *P. sabiniana*, viz., 0.70057.

On the other hand, the hydrocarbon from *P. sabiniana* has precisely the same specific gravity as that of the heptane obtained by heating azelaic acid with caustic baryta. Dale found that the latter body boils at $98-99^{\circ}$, and has a specific gravity of 0.6851 at 17.5° ; Schorlemmer, who procured the same hydrocarbon in larger quantity, observed 0.6840 at 20.5° . Reducing these observations by means of the formula given on page 301, they become respectively—

I	0.6992
II	0.7000

If, as Schorlemmer believes, the heptanes from azelaic acid and petroleum are probably identical, the difference in their specific gravities is at present inexplicable. We are now engaged on a joint investigation on the chemical constitution of the heptane from *P. sabiana*, which may incidentally throw light on this question.
