

Transport Properties of Some Organic Heat Transfer Fluids

Thermal Conductivity of Biphenyl, Phenyl Ether, Dowtherm A, and Santowax R

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BECAUSE of their low vapor pressure and high resistance to pyrolysis, certain high boiling organic substances such as biphenyl, phenyl ether, and their mixtures have long been in use as heat transfer fluids.

Interest in these substances has recently been renewed, as they seem to offer advantages over other fluids as coolants and moderators in nuclear reactors (8, 14), or even as working fluids in heat engines of a nuclear power plant (16).

A substance that has found wide application as a high-temperature heat transfer fluid is the eutectic mixture of 73.5% phenyl ether and 26.5% biphenyl, commonly known under various trade names—e.g., Dowtherm A, Thermex, Diphyl, VOT. It possesses a notable practical advantage over its two constituents—a melting point (at about 12° C.) below normal room temperature.

Fairly comprehensive thermodynamic data for Dowtherm A can be found in manufacturer's publications. Apart from individual points of unspecified origin, thermal conductivity of this substance has been investigated experimentally by only three previous observers, whose results do not agree too well (4, 12, 23). Thermal conductivity values found in data compilations (7, 18, 20) are considerably at variance with the former experimental values.

Data on thermal conductivity of the constituents of Dowtherm A, biphenyl, and phenyl ether are even scarcer.

When, at the request of the Atomic Energy Research Establishment, Harwell, the thermal conductivity of Santowax R—a mixture of *o*-, *m*-, and *p*-terphenyl—was studied, accurate information was gathered on the thermal conductivity of Dowtherm A and its constituents, from their melting points to their atmospheric boiling points.

DISCUSSION OF METHOD

A vertical coaxial cylinder apparatus with guard rings was chosen for this investigation.

Heat is generated in an inner emitting cylinder and conducted radially through a fluid-filled annulus to a coaxial receiving cylinder. From the measured surface temperature of both emitting and receiving cylinders, the amount of heat conducted radially through the annulus, and the dimensions of the apparatus, the thermal conductivity can be computed from the following equation.

$$k = -Q \ln(r_2/r_1) / 2\pi L (t_2 - t_1)$$

where

- k = thermal conductivity, cal./cm. sec. ° C.
- Q = radial heat flow through annulus under steady-state conditions, cal./sec.
- r_1, r_2 = radii of emitting and receiving cylinders, respectively, cm.
- t_1, t_2 = temperatures of emitting and receiving surfaces, respectively, ° C.
- L = length of emitting cylinder, cm.

EXPERIMENTAL

Apart from the modifications described below, the apparatus and experimental procedure were the same as used for work on heavy water (24).

Filling Device. As the melting points of three of the substances studied were above room temperature, provision had to be made for filling the autoclave and maintaining the substance under test in the liquid phase in every part of the apparatus. Further, a small excess pressure above atmospheric had to be applied when the temperature rose

close to, or exceeded, the atmospheric boiling point. The bellows assembly shown in Figure 1 was used to prevent the pressurizing gas from being dissolved in the substance under test. The device designed for this purpose (Figure 1) consisted of an outer steel vessel, 1, heated by electrothermal heating tapes wound on its outer surface. The current to the heater was so adjusted that the temperature inside the vessel, indicated by the resistance thermometer, 2, was a few degrees above the melting point of the substance under test. Inside vessel 1 there was a closed bellows assembly, 3, the inside of which could be pressurized by admitting nitrogen through the pressure control valve, 5. End stops were provided to protect the bellows from excessive movement and strain. The degree of expansion or compression of the bellows was indicated by a tubular extension attached to the bottom sealing plate. An air-cooled gland, 4, sealed the inside of the bellows assembly from the atmosphere.

Temperature Measurement and Calibration of Thermocouples. In addition to the initial careful calibration of all thermocouples, the two thermocouples located in the center of the emitter and receiver, respectively, were

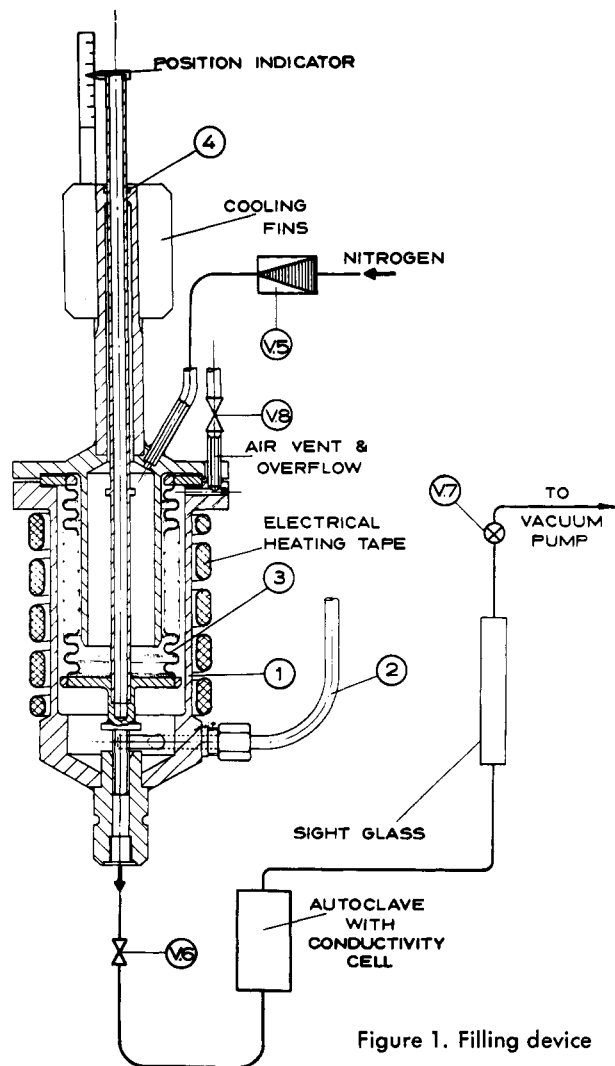


Figure 1. Filling device

frequently compared *in situ* by switching off the emitter and guard ring heaters and allowing the conductivity cell to reach an isothermal state. Particularly careful attention was given during such tests to the control of uniformity of temperature along the walls of the thermostat.

Conductivity Cell. The dimensions of the cell at 20° C. were given by the manufacturers (Pitter Gauge and Precision Tool Co., Woolwich) as:

Emitting cylinder (silver). Average diameter 3.1892 cm., maximum deviation not over 0.00025 cm.

Receiving cylinder (Hidural 5, Langley Alloys Ltd.). Average diameter 3.3401 cm., maximum deviation not over 0.0001 cm.

The dimensions of the cylinders at the temperatures of the experiments were calculated from the known dimensions at 20° C., using the coefficient of thermal expansion of Hidural 5 (a high conductivity copper alloy) quoted by Langley Alloys, Ltd. (13) and of silver given by Owen and Yates (15).

Materials Investigated. With the exception of Santowax R, the materials tested were standard laboratory reagents of known specification and their purity was not tested further. The manufacturers' specifications were:

Biphenyl (Hopkin and Williams, Ltd.). Crystallizing point 68.7° C., minimum distillation range 2.5° C. maximum, initial and final boiling point to include 256° C.

Phenyl ether (B.D.H. laboratory reagent). Very slightly tinted crystalline solid or clear liquid, melting point 26–7° C., 5% alcoholic solution clear, vapor phase chromatograph assay on the last batch revealed only 1 peak.

The eutectic mixture of 73.5% phenyl ether and 26.5% biphenyl was prepared from the individual constituents obtained from the above suppliers.

Santowax R (Monsanto Chemicals, Ltd.). A sample was supplied by Atomic Energy Research Establishment, Harwell, which had earlier analyzed the material and found the following composition (2): biphenyl 1%, *o*-terphenyl 8%, *m*-terphenyl 52% *p*-terphenyl and higher polyphenyls 39%.

Filling of Autoclave. Where the substance was solid at room temperature, the autoclave and the pipe connections were heated to a few degrees above the melting point of the substance under test and evacuated to about 0.5 mm. of Hg. Valves 6 and 7 were then closed. After the bellows assembly had been removed from the filling device, vessel 1 was heated and filled with a representative sample of the test substance. During the filling with Santowax R great care was taken to keep the mixture liquid and thus avoid fractional crystallization.

A temporary lid was fitted to vessel 1 and the space above the level of the liquid was then evacuated. On opening valve 6 the liquid filled the autoclave by its own static head. Completion of filling was indicated by the appearance of the liquid level in the short glass sight tube preceding valve 7. Air was then admitted and the bellows assembly attached to vessel 1. During this stage further material was added if the liquid level had fallen below a certain mark.

To reduce the amount of air trapped, the bellows assembly, before refitting to vessel 1, was elastically compressed to its safe minimum length. After it had been inserted into vessel 1 and the lid secured and sealed, it was allowed to expand. The excess fluid displaced from the vessel was allowed to escape through the overflow connection. After the overflow line had been closed and nitrogen gas admitted through control valve 5, the autoclave was pressurized to a chosen value and the apparatus was ready for the experiment.

Possible Errors and Corrections to Thermal Conductivity Measurements. When measuring thermal conductivity it is essential that heat exchange between the emitting and receiving surface by radiation and by convection be either negligible or accurately determined.

Convection. Absence of convection in thermal conductivity measurements with coaxial cylinders can be assumed if the product of Grasshof and Prandtl numbers (Rayleigh's criterion) for the given operating conditions does not exceed about 1000. Largely because of the narrow annulus used, Rayleigh's criterion was not exceeded in these experiments and consequently the data reported here are not affected by convection.

The absence of convection is also shown by comparing determinations made at nearly the same temperatures but with different temperature differences across the annulus—e.g., experiments 8 and 9 for biphenyl, 10 and 11 for Santowax R. Such comparisons also directly confirm the absence of appreciable radiation.

Radiation. To determine the heat exchange by radiation between the emitting and receiving cylinders, not only the optical properties of the respective metals must be known, but also those of the fluids investigated. Even under unfavorable conditions, heat transferred by radiation forms only a small fraction of that transferred by conduction, and, because of the properties of the liquids investigated and the low emissivities of the highly polished emitter and receiver surfaces, radiative heat exchange is further reduced by internal absorption in the fluid-filled annulus.

Considering the least favorable case, the experiment at the highest temperature (392° C.) and with the largest temperature difference of 3° C. across the annulus, the heat transmitted by radiation would amount to 0.7% of the heat conducted through the annulus, if the fluid under test were completely transparent to thermal radiation. Should any of the fluids, however, possess appreciable absorption bands at wave lengths between 5 and 9 microns, the amount of heat exchange by radiation would be less.

To clarify this point the infrared absorption spectra of the four substances—biphenyl, phenyl ether, Dowtherm A, and Santowax R—were obtained in the wave length region 2 to 6 microns with a Grubb-Parson S/3/A double-beam spectrometer. These measurements were made by the Spectroscopic Analysis Group of the establishment. The spectrograms, with the relevant experimental details, are shown in Figure 2. All four substances have strong absorption bands, particularly in the middle region of the spectral range studied, a region which coincides with the location of the maxima of black body radiation for the temperature range of these experiments. The average absorption for any of these substances is not less than 60%, and the amount of heat transferred directly by radiation must therefore be considerably less than the previous estimate of 0.7%. In view of the tolerance level dictated by other experimental errors, no corrections for radiative heat transfer were applied.

Errors through Axial Heat Flow. Because of the difficulty in obtaining an accurate relative calibration between the guard ring thermocouples and the emitter and receiver thermocouples, some small unbalance of $\pm 1 \mu\text{v.}$ (equivalent to $\pm 0.02^\circ \text{C.}$) had to be accepted, which could lead to an uncorrected axial heat flow. Calculations showed that in the extreme case—i.e., both guard ring temperatures either above or below the emitter temperature—the axial heat flow caused by a temperature difference of about 0.02°C. could amount to about 0.8% of the radial heat flow.

Temperature Drop in Walls of Conductivity Cell. Because of the location of the thermocouples somewhat below the emitting and receiving surfaces, the measured temperature difference between them included the temperature drops through the metal layers between the thermocouples and the surfaces of the cylinders. The values were computed with the aid of the known thermal conductivity of Hidural 5 (13) and pure silver (1), the materials of the receiver and emitter, respectively, and the known linear dimensions of the conductivity cell. The order of this correction is 0.2% of the measured temperature difference.

RESULTS

The experimental results have been compiled in Table I and plotted in Figures 3 to 6 to facilitate comparison with the work of previous authors. The numbers in the first column refer to the chronological order of these tests. The conductivity values in Table I represent the arithmetic mean of 3 to 7 individual determinations made under the same conditions during a total time of 50 to 120 minutes. The standard deviation of individual points from the mean value was in all cases less than 1%.

None of the liquids investigated showed any abnormal behavior within the temperature range of these tests, and the results for all four substances could be adequately correlated by linear equations between temperature and thermal conductivity. The constants required were obtained from a least squares analysis. The proposed equations are given in Table I.

In the absence of other published data on the thermal conductivity of Santowax R, the first substance studied, it was felt worthwhile to confirm the reliability of the apparatus by a direct comparison with a test fluid of well known thermal conductivity before proceeding with these determinations. Although the method employed in this research is an absolute one and therefore does not depend on a calibration, it was felt desirable to prove the absence of any changes in the apparatus—e.g., in the alignment

of the coaxial cylinders—that might have taken place during prolonged periods of operation at high temperatures. The substance chosen for this independent check was toluene for which reliable observers (6, 17, 19, 22) have reported results which agree within less than 2%. Two determinations were made of sulfur-free toluene (B.D.H. laboratory reagent) at 49.7° and 90.7° C. Results agreed well (better than 1%) with average values obtained from the data of four previous authors. A similar check was carried out after the tests on phenyl ether and Dowtherm A were completed. A point obtained at 33.7° C. again agreed closely with previous observations.

On the basis of these tests, irreversible changes in the geometry of the cell—e.g., misalignment or distortion of the cylinders—can be excluded as a possible source of error in this work. Combining all other remaining errors, it was estimated that a total accuracy to $\pm 1.5\%$ can be claimed, an estimate which is supported by the results on toluene obtained in this research.

DISCUSSION

Biphenyl. The thermal conductivity of biphenyl was measured by Hilpert and Schmidt (10), who used a horizontal plate apparatus heated from above. As shown in Figure 3, their results are 12 to 18% lower, but the temperature coefficient, $-dk/dt$, is only slightly higher than the one found in this research.

The data by Briggs (3), who used a simplified coaxial cylinder apparatus, are in good agreement with those of this investigation, with regard to not only the numerical values of thermal conductivity but also its temperature coefficient. On the whole, the conductivity values determined by Briggs are about 2% higher than the present ones.

Cecil, Koerner, and Munch (4) used a hot wire cell with a 0.5-cm. annulus. They found that in the temperature range 98° to 217° C. the thermal conductivity of biphenyl was 31.6×10^{-5} cal./cm. sec. ° C. and was independent of temperature. Their data were cited by McEwen (14) and Stone (21). A zero temperature coefficient of thermal conductivity of a nonassociated liquid is most unlikely and has not been found by other workers on this or similar liquids. It seems probable that in view of the comparatively large annulus of their cell, their results were affected by convection. Similar considerations apply also to their work on Dowtherm A.

Phenyl Ether. The only earlier measurements on this substance are due to Hilpert and Schmidt (10), who studied both biphenyl and phenyl ether. The results of these observers are again between 12 and 18% lower than those of this work and their temperature coefficient is slightly higher. There is insufficient detail in the article to permit an analysis of the method and test procedure, and therefore no explanation of this discrepancy is offered.

As is seen in Figure 4, one point at 35° C. (11) is in good agreement with this research.

Eutectic Mixture of 26.5% Biphenyl and 73.5% Phenyl Ether (Figure 5). The eutectic mixture of biphenyl and phenyl ether has been widely employed in the chemical industry as a high-temperature heat transfer fluid in preference to either of its constituents on account of its low freezing point.

The first extensive experimental investigation of the thermal conductivity of this substance was due to Kerzhentsev and Vargaftik (12), who measured this property between 20° and 360° C. using a vertical, hot-wire cell with an annulus of 0.0385 cm. Within this temperature range they found a linear relationship between thermal conductivity and temperature. In comparison with the present results, Kerzhentsev's data are between 4 and 6% lower but their temperature coefficient is the same.

Woolf and Sibbitt (23), using a coaxial cylinder apparatus without guard rings and employing thermistors as thermom-

Table I. Experimental Thermal Conductivity Values Compared with Proposed Correlation Equations

Expt. No.	Temp., ° C.	Pres- sure, P.S.I.	Temp. Diff., ° C.	No. of Indi- vidual Detns.	Thermal Con- ductivity, Cal./Cm. Sec. ° C.		$k_c - k_{ex}$
					Exptl., k_{ex}	Calcd., k_c	
Biphenyl $10^4 \times k = 3.55 - 0.0035 t$ cal./cm. sec. ° C.							
5	92.0	60	3.46	4	3.25	3.23	-0.02
6	128.8	60	3.95	5	3.12	3.10	-0.02
1	164.3	Atmos.	4.09	5	2.97	2.98	+0.01
2	199.5	Atmos.	4.12	6	2.85	2.86	+0.01
7	211.2	60	4.31	4	2.80	2.82	+0.02
4	244.2	60	4.15	5	2.69	2.70	+0.01
3	244.3	60	4.26	4	2.68	2.70	+0.02
8	305.7	60	4.06	9	2.50	2.49	-0.01
9	308.4	60	7.06	1	2.52	2.48	-0.04
Phenyl Ether $10^4 \times k = 3.53 - 0.0037 t$ cal./cm. sec. ° C.							
6	56.7	Atmos.	3.59	4	3.30	3.32	+0.02
5	79.0	Atmos.	3.65	5	3.25	3.24	-0.01
1	129.4	Atmos.	3.91	5	3.07	3.05	-0.02
2	188.5	Atmos.	4.83	5	2.82	2.84	+0.02
4	219.8	Atmos.	4.32	4	2.73	2.72	-0.01
3	238.0	Atmos.	4.61	6	2.65	2.65	0.00
Dowtherm A $10^4 \times k = 3.47 - 0.0033 t$ cal./cm. sec. ° C.							
1	47.1	Atmos.	3.38	4	3.32	3.32	0.00
2	53.8	Atmos.	6.94	5	3.32	3.30	-0.02
3	96.5	Atmos.	3.67	4	3.16	3.15	-0.01
4	153.3	Atmos.	3.90	4	2.94	2.97	+0.03
5	212.5	Atmos.	4.39	6	2.73	2.77	+0.04
6	250.7	Atmos.	4.40	5	2.68	2.64	-0.04
Santowax R $10^4 \times k = 3.52 - 0.0023 t$ cal./cm. sec. ° C.							
6	155.4	Atmos.	2.35	3	3.16	3.16	0.00
7	155.6	60	2.43	4	3.15	3.16	+0.01
1	189.0	Atmos.	2.48	6	3.11	3.09	-0.02
5	208.6	Atmos.	2.48	3	3.04	3.04	0.00
2	235.4	Atmos.	2.55	5	3.00	2.98	-0.02
10	273.6	68	2.67	4	2.86	2.89	+0.03
11	276.0	Atmos.	5.35	3	2.87	2.89	+0.02
3	297.1	Atmos.	2.59	7	2.85	2.84	-0.01
4	241.8	Atmos.	2.74	3	2.74	2.74	0.00
8	345.2	60	2.87	5	2.73	2.73	0.00
9	391.5	60	2.94	4	2.63	2.62	-0.01

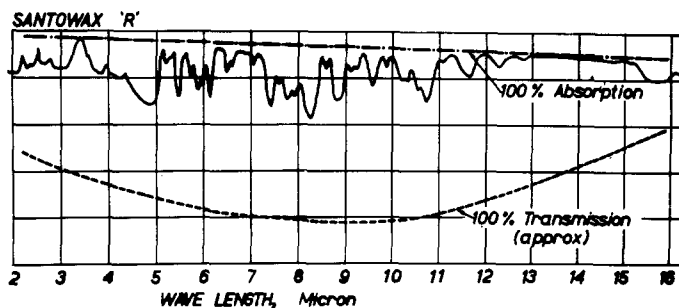
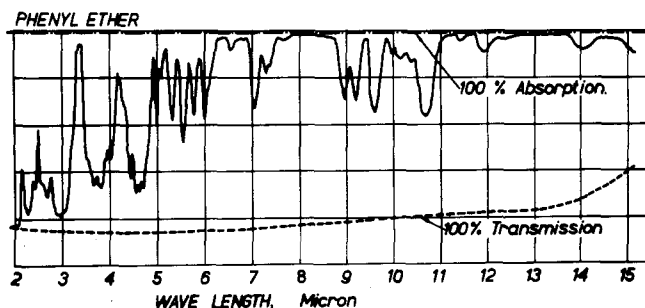
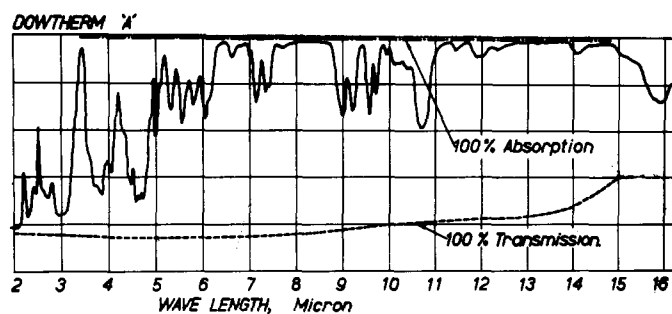
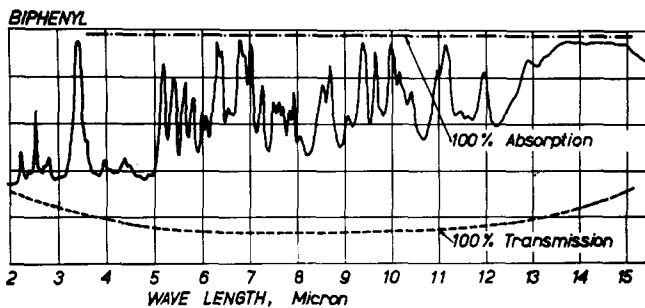


Figure 2. Absorption spectra of substances studied
Liquid film 0.3 mm. thick

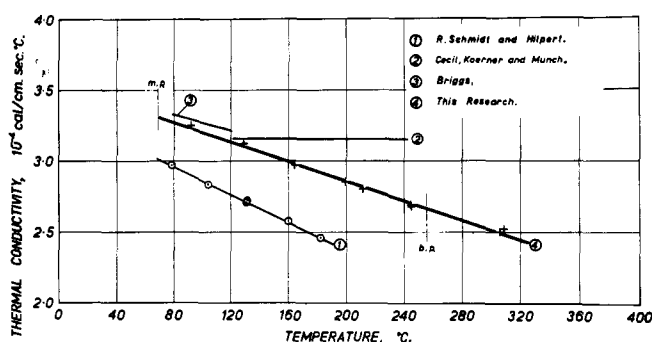


Figure 3. Thermal conductivity of biphenyl

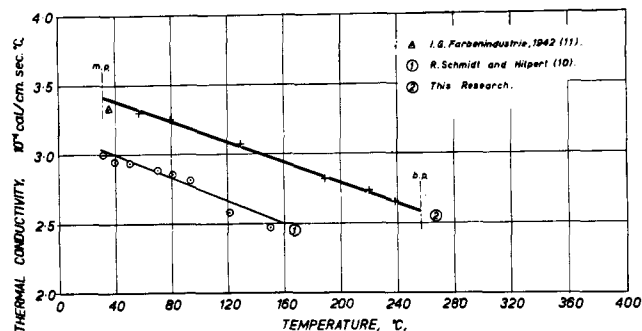


Figure 4. Thermal conductivity of phenyl ether

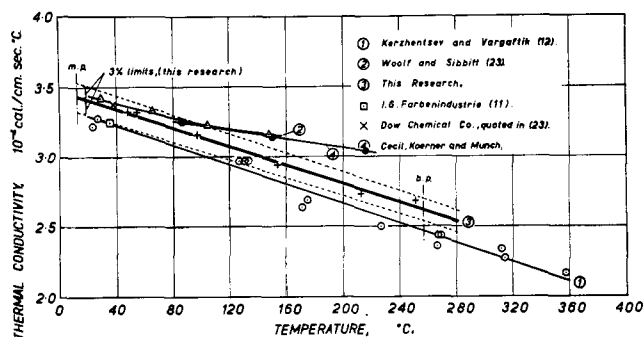


Figure 5. Thermal conductivity of Dowtherm A

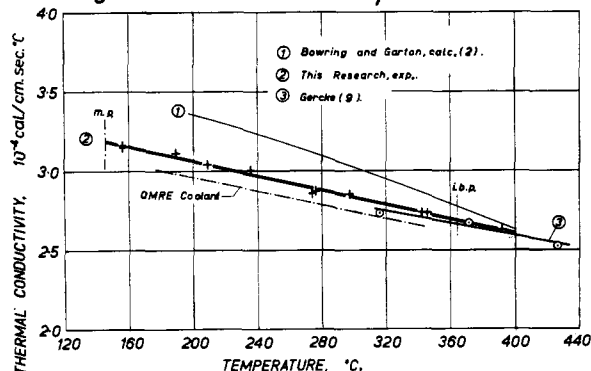


Figure 6. Thermal conductivity of Santowax R

eters, investigated the thermal conductivity of several organic liquids, including Dowtherm A. Their results are in good agreement with this work at the lower end of their temperature range—i.e., between 30° and 60° C.—where their data are about 1.5% higher. Because of a somewhat smaller temperature coefficient of the thermal conductivity exhibited in the work of Woolf and Sibbitt the discrepancy increases with rising temperature, and at 150° C.—i.e., at the upper end of the temperature range studied by them—their values are almost 6% higher than those reported here.

Cecil, Koerner, and Munch (4) obtained data at 86°, 150°, and 217° C. Their value at 86° C. agrees well with this research but the discrepancy increases with increasing temperature to about 11% at 217° C.

Two individual values, one at 35° C. quoted by I.G.

Farbenindustrie (11), and one at 38° C., quoted by Woolf and Sibbitt and attributed to the manufacturer, the Monsanto Chemical Corp., both lie within the 3% confidence limits of this investigation.

It is usually rather difficult to suggest probable causes for the discrepancies between the data of different observers, as not all essential details for a full analysis are reported. This applies to a lesser extent to the data of Woolf and Sibbitt, who also investigated the thermal conductivity of carbon tetrachloride, a substance for which reliable values have been established through the work of previous investigators. Comparing the data of Woolf and Sibbitt with those of Riedel (17), Challoner and Powell (6), and Cecil and Munch (5), all of whom agree within 1.5% of an average value of 2.50×10^{-4} cal./cm. ° C. sec. for the

thermal conductivity of carbon tetrachloride at 25° C., it was found that Woolf's value was almost 9% higher. Further, Woolf's data on Aroclor 1248 (23) were criticized by Cecil and Munch, who studied the same substance and found values almost 15% lower. From an analysis of the apparatus and the test procedure employed by Woolf and Sibbitt, it seems possible that the effect of axial heat losses in their unguarded conductivity cell was underestimated—an error which would always lead to higher thermal conductivity values than the correct ones and, further, would tend to increase with a rising difference between the cell and ambient temperatures. Doubt must therefore be expressed as to the validity of Woolf and Sibbitt's claim of an accuracy of 2.5%.

It proved impossible to explain the discrepancy between the results of Kerzhentsev and Vargaftik (12) and this work. Although these authors carried out experiments on other fluids using a conductivity cell identical to the one used for their work on Dowtherm A, the results of that study were published in another paper not obtainable by the authors of this report. However, the excellent agreement between the present data on toluene and the best published values (6, 17, 19, 22) on this substance seems to support the validity of the data given in Table I.

Thermal Conductivity of Toluene,
10⁻⁴ Cal./Cm. Sec. ° C.

Temp., ° C.	This research	Averaged from (6, 17, 19, 22)
33.7	3.18	3.16
49.7	3.06	3.06
90.7	2.80	2.78

Data on the thermal conductivity of Dowtherm A were also found in two general papers on the use and properties of high temperature heat exchange fluids. As the values quoted there differ considerably from those of this research, and compilations of property data of this kind are likely to find their way to the practical engineer who cannot spare the time to study the specialized literature, it was thought necessary to review them briefly and to point out the magnitude of error.

Schulze (20) in a general article on high-temperature heat transfer fluids reported certain physical properties of Diphyl between 50° and 350° C. From his tabulated values of the density, ρ , the specific heat, c_p , and the thermal diffusivity, α , the thermal conductivity, k , was computed from $k = \alpha c_p \rho$. The values thus obtained were practically constant over the entire temperature range. At 50° C. his value of 3.23×10^{-4} cal./cm. sec. ° C. agrees fairly well with that of this research and other previous observers; at elevated temperatures—i.e., in the range of practical applications—the error becomes appreciable if a constant value of the thermal conductivity is assumed. At 300° C., for example, the thermal conductivity found in this research is 24% lower than Schulze's recommended value. The discrepancy would be worse still if the data by Kerzhentsev and Vargaftik were compared. Schulze did not mention the method by which the thermal conductivity values were obtained, but stated that they were determined by the manufacturers, Farbenfabriken Bayer, Leverkusen. It follows that certain derived quantities contained in that author's compilation—viz., thermal diffusivity and Prandtl number—must also be in error.

In a paper similar in scope to that by Schulze, Rottenburg (18) gave an extensive compilation of the thermodynamic and transport properties of organic, inorganic, and metallic substances, suitable as high-temperature heat exchange media. The thermal conductivity values of Dowtherm A, taken from the Dowtherm Handbook (7), display an unusual trend. The thermal conductivity rises at first with increasing temperatures up to about 250° C. where it reaches a maximum and then falls slowly with a further increase in temperature. This trend was not

confirmed in any of the three experimental studies reported here. There is also considerable numerical disagreement with the present experimental data which culminates in a discrepancy of 73% at 300° C. Even for technical heat transfer calculations this is excessive.

Santowax R (Figure 6). In an article on organic coolant technology, Gercke (9) quoted values of unspecified origin of the thermal conductivity of Santowax R which agree fairly well with this work but seem to display a somewhat smaller temperature coefficient. In the same article experimental values were given for the thermal conductivity of a reactor coolant similar in composition to Santowax R (OMRE: 17% biphenyl; 45.3, 29.2, 5.6% *o*-, *m*-, *p*-terphenyl, respectively; 2.9% intermediates and high boiling compounds). The temperature coefficient of these data is the same as found for Santowax R in this research but the absolute values are generally about 3% lower than for Santowax R.

Bowring and Garton (2) reported thermal conductivity values for Santowax R which were computed with the aid of an equation proposed by Vargaftik (22) for the thermal conductivity of nonassociating liquids. As is seen in Figure 6, the agreement is only moderate and the deviation between the experimental and the computed values progressively increases with falling temperature.

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The infrared spectrograms were obtained by the Spectroscopic Analysis Group of this establishment; M.T. Dupree assisted during assembly of the experimental apparatus and carried out some of the measurements.

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