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PHYSICAL PROPERTIES OF NORMAL BUTYL ESTERS OF 2,4-D, 2,4,5-T, AND "ORANGE"

Richard A. Hansen

AUGUST 1965

UNITED STATES ARMY
BIOLOGICAL LABORATORIES
FORT DETRICK

U.S. ARMY BIOLOGICAL LABORATORIES
Fort Detrick, Frederick, Maryland

TECHNICAL MEMORANDUM 74

PHYSICAL PROPERTIES OF NORMAL BUTYL ESTERS OF 2,4-D, 2,4,5-T, AND "ORANGE"

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Physical Sciences Division
DIRECTORATE OF BIOLOGICAL RESEARCH

Project 1C014501B71A01

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ABSTRACT

Physical properties of normal butyl esters of 2,4-dichlorophenoxyacetate, 2,4,5-trichlorophenoxyacetate and their equal mixture by weight (denoted as "Orange") are reported. Summaries of data from Dow Chemical Company, Edgewood Arsenal (Army Chemical Center), and U.S. Army Biological Laboratories are also given. Recently acquired data on viscosities as a function of temperature, flow rates as a function of pressure, theoretical calculations of thermal conductivities, specific heats, surface tensions, and freezing points are also reported.

Samples obtained from Dow Chemical Company were of commercial (93 to 96%) purity. Methods and reliability of data from sources other than from the author are not known and such data are reported for the reader's convenience only.

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I. INTRODUCTION

In the storage, transport, and dissemination of organic liquids such as the herbicides employed to defoliate broad leaf plants and trees, knowledge of such properties as the temperature-viscosity correlation, specific heat, freezing point, surface tension, thermal conductivity, and whether its flow characteristics are Newtonian or non-Newtonian is of primary importance to certain uses of these materials.

Transport and dissemination of defoliants by airborne systems indeed demands factual data of such properties on which to base design and modifications of wing tanks, dissemination procedures, nozzle orifice sizes, pressures required, and a multitude of similar points of usage. The acquisition and report of such data are the subject of this paper.

Tables listing data on 2,4-dichlorophenoxyacetate (2,4-D) and 2,4,5-trichlorophenoxyacetate (2,4,5-T) acquired by other investigators are given in the appendixes. The information listed therein, having been made readily, although informally, available, has been included in this report for the convenience in having most of such data on physical properties of these materials under one cover. An exhaustive literature survey has not been made and neither the accuracy nor the methods used to obtain the data listed in the appendixes are known. Sources of these data are Dow Chemical Company, Edgewood Arsenal, and the U.S. Army Biological Laboratories.

II. MATERIALS

Samples of the materials investigated were procured by Crops Division from Dow Chemical Company on 26 July 1962.

The material denoted hereafter as 2,4-D is a normal butyl ester of 2,4-dichlorophenoxyacetate, approximately 95% pure. The order number was 761152 and the lot number 13GW148. The material hereafter denoted as 2,4,5-T is a normal butyl ester of 2,4,5-trichlorophenoxyacetate, approximately 93% pure.

The material known herein as "Orange" is an equal mixture by weight of 2,4-D and 2,4,5-T; the estimated purity is 94%. The only physical property of Orange determined earlier is a value of 1.278 at 25 C for the specific gravity.

III. METHODS

A. VISCOSITY-TEMPERATURE[2]

All viscosity data on 2,4-D and 2,4,5-T were determined with a Höppler "falling ball" viscometer in a controlled temperature chamber. Temperatures are equilibrium values and were measured by a copper-constantan thermocouple affixed to the dropping tube. A Pace heated thermocouple reference junction (100 C) and a Sanborn low level recorder provided a reference temperature and recorded the thermocouple voltages. Times of fall of the ball between marks on the dropping tube were determined by stopwatch. The instrument was calibrated by silicon oil standards under conditions identical to those existing for the materials under investigation.

Viscosity data on Orange were obtained both with the Höppler and with a Brookfield viscometer which employs a rotating cylinder. In using the Brookfield instrument, the sample was held in a constant temperature vessel of sufficient size to avoid container effects. Temperatures were measured by thermocouples with care taken to assure viscosity measurements on the sample at the equilibrium temperature. Data were also obtained by D.B. Coulson in our laboratories by the Cannon method at 25 C.

B. FLOW RATE-PRESSURE[3]

Flow rates at various pressures, using filtered samples at ambient temperatures, were determined by using an extrusion rheometer to extrude 40 ml of the materials through either a two- or six-inch length of stainless steel tubing of 18, 20, or 22 gauge.

Pressures were from 0 to 80 psig and average shear rates from 0 to 40×10^4 reciprocal seconds. Flow was laminar since Reynolds numbers were not greater than 35 in any run. Total time of extrusion was determined by stopwatch between the 10- and 50-ml graduations on a graduated cylinder as it filled.

C. SURFACE TENSION[4]

All data on surface tensions are those of G.W. Trout and L.H. Solecki of Physical Sciences Division and were determined by the "drop weight" method. In this method a drop of the material is made to fall from the tip of a vertically held capillary tube of known dimensions, taking care that the drop is made to detach itself as slowly as possible and that adsorption equilibrium is attained between the drop and its environment before detachment. Since the organic liquids of this report are not considered to be highly volatile, no additional precautions in this regard were taken.

In the "drop weight" method, the surface tension is determined by $\gamma = (mg/r)(F)$ where m is the mass of the drop in grams; g is the coefficient of acceleration in cgs units; r is the radius of the capillary tip in centimeters; and F is a drop-weight correction factor. The radius of the capillary tip is a constant equal to 0.2422 cm and the correction factor (F) is 0.2655 as calibrated for the capillary used with water and benzene. More detailed explanations plus tables of the correction factor (F) are available in standard references.[6]

Determinations were made at 25 and 35 C for 2,4-D and Orange and at 35 and 40 C for 2,4,5-T. Equilibrium temperatures were attained by enclosing the apparatus in a glass chamber immersed within a constant temperature bath held at the required temperature.

D. THERMAL CONDUCTIVITY (THEORETICAL)

The treatment reported here is that of J.F.D. Smith, recommended by McAdams in his book on heat transmission.[6] Briefly, Smith's equation is empirical, based on water, paraffin alcohols, hydrocarbons, petroleum fractions, and other liquids.[7] It is recommended for estimating the thermal conductivities of nonmetallic liquids at 86 F and one atmosphere pressure, where measured values are not available.

The equation is as follows:

$$K = 0.00266 + 1.56\ (C_p - 0.45)^3 + 0.3\ (\rho/m)^{1/3} + 0.0242(\mu/\rho)^{1/9}$$

where K is the conductivity in BTU/(hr)(ft)^2 (deg F/ft); C_p is the specific heat; ρ is the specific gravity relative to water; m is the average molecular weight; and μ is the viscosity in centipoise at 86 F.

E. SPECIFIC HEAT

The specific heat was determined by the method of mixtures. A known weight of material was heated to an equilibrium temperature in the vicinity of 100 C and then mixed with a known weight of the same material that was in equilibrium at ambient temperature (about 26 C) in an aluminum cup placed in a calorimeter. The equilibrium temperature of the mixture was recorded and the resulting heat balance equation solved for the specific heat.

F. FREEZING POINT

Attempts to determine a freezing point by observation of a lag in the rate of temperature depression due to latent heat formation were unsuccessful because the materials supercooled readily. Similarly, attempts to determine a melting point by observation of a lag in the rate of temperature rise or elevation were also unsuccessful because the presence of impurities obscured what small degree of latent heat formation exists in these material

Therefore, the material was placed in a constant temperature chamber, allowed to attain equilibrium and then seeded with crystals of the same material. A temperature level was determined at which the entire sample crystallized or "froze" rapidly following seeding, and a higher temperature level was determined at which the sample remained liquid over a 24 hour period. The "freezing point" was arrived at by bracketing the point, i.e., successively raising the equilibrium temperature of the chamber and sample in smaller and smaller increments until a point was reached at which the sample, following seeding, slowly crystallized over approximately an eight-hour period, and above this point remained liquid.

IV. RESULTS

A. VISCOSITY-TEMPERATURE

The viscosity-temperature relations are reported in numerical form in Table 1 and graphically in Figure 1 for 2,4-D. Available data from sources listed in Appendix A for 2,4-D are also plotted in Figure 1. The correlation is excellent even though the samples undoubtedly differ slightly in the degree and kind of impurities. Since 2,4-D supercools readily, difficulties were encountered at approximately -20 C when the sample froze or changed phase rapidly and erratically. The phase change did not occur in all instances. An empirical equation may be fitted to the curve shown in Figure 1 as follows:

$$\ln \eta = \frac{6.551 \times 10^{13}}{T^{5.378}}$$

where $\ln \eta$ is the natural log of the viscosity in centipoise and T is the absolute temperature in degrees Kelvin.

TABLE 1. VISCOSITY AS A FUNCTION OF TEMPERATURE FOR 2,4-D

Method or Source	Temperature, C	Viscosity, centipoise
Höppler	49.3	8.3
	47.8	8.7
	46.6	9.2
	42.6	10.9
	36.4	14.2
	35.6	14.6
	35.0	14.6
	27.8	22.4
	24.0	28.4
	11.6	63.1
	6.8	96.8
	6.5	96.8
	1.7	156
	-5.5	326
	-14.7	984
	-18.5	2114
Army Chemical Center	25	27.2
	35	16.2
	50	8.6
Dow Chemical Company	20	38.0
	30	22.4
	40	14.7
	50	10.5
	60	8.2
USABL	28	26.0

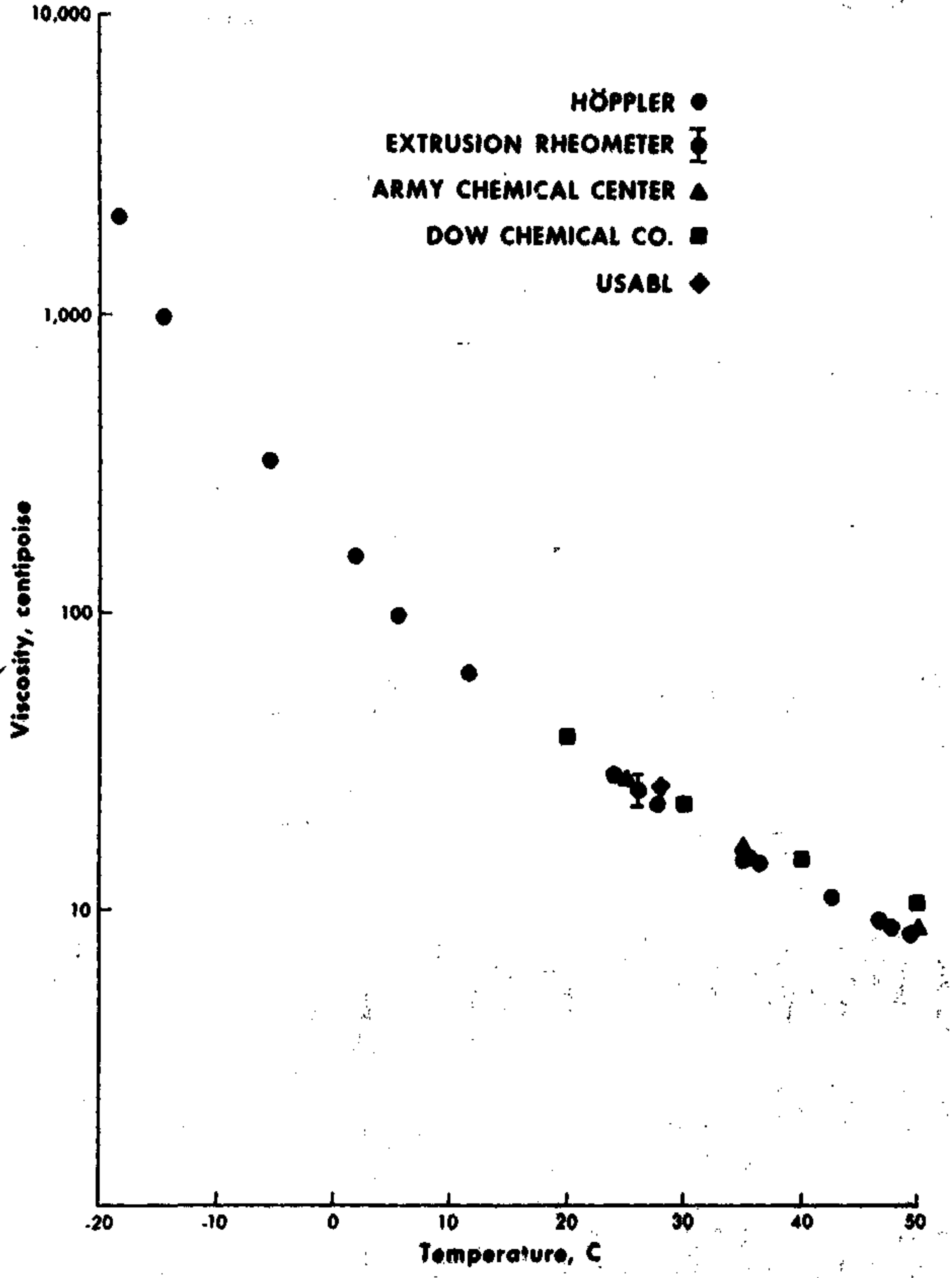

Figure 1. Viscosity Versus Temperature of 2,4-D.

Table 2 and Figure 2 show the data for 2,4,5-T. The empirical equation fitting the curve of Figure 2 is as follows:

$$\ln \eta = \frac{3.876 \times 10^{14}}{T^{5.637}}$$

TABLE 2. VISCOSITY AS A FUNCTION OF TEMPERATURE FOR 2,4,5-T

Method	Temperature, C	Viscosity, centipoise
Höppler	50.4	16.5
	46.8	19.7
	41.8	26.5
	37.4	34.4
	36.1	37.3
	26.1	69.5
	10.4	322
	-1.8	1869

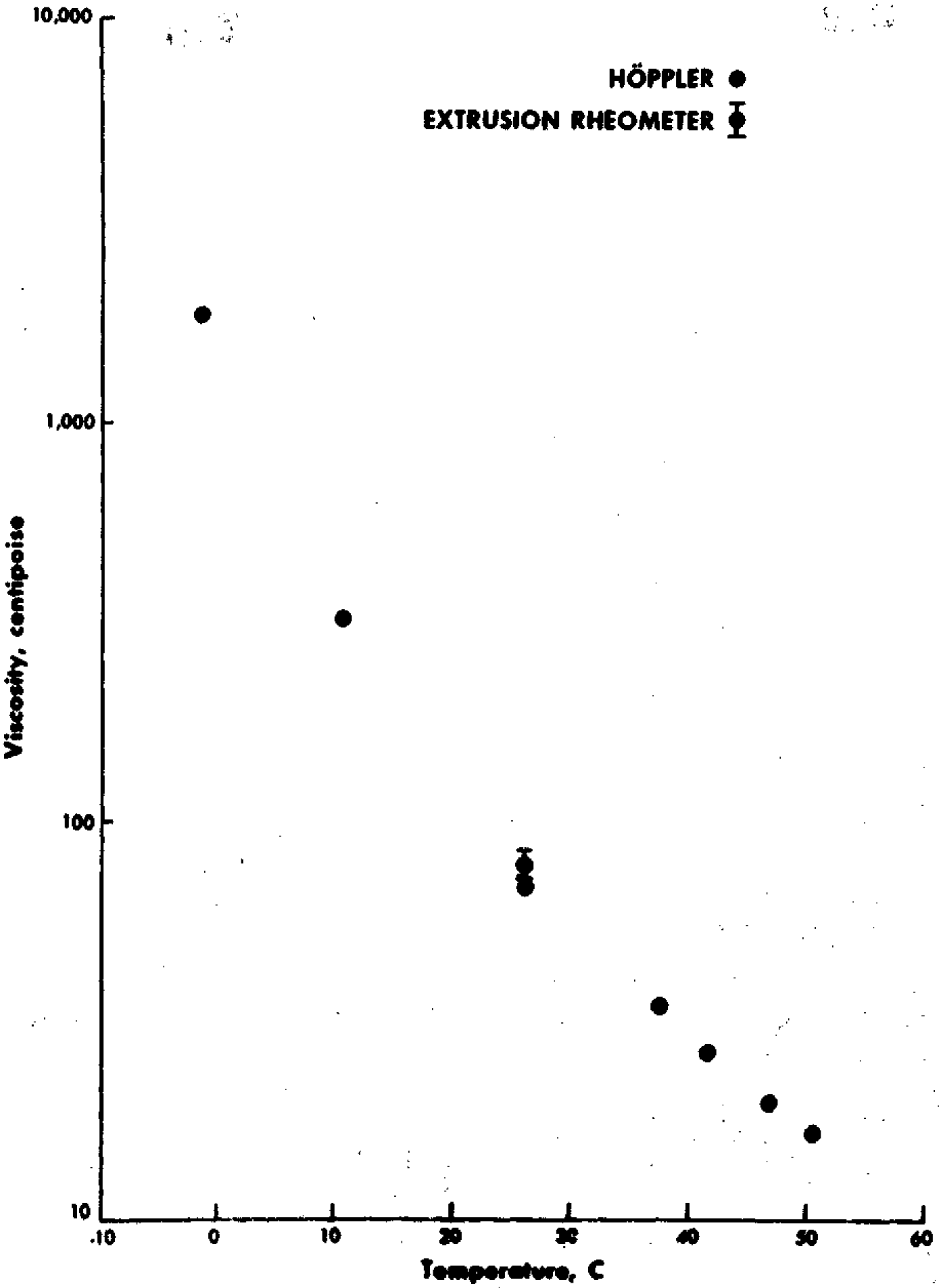

Figure 2. Viscosity Versus Temperature for 2,4,5-T.

Viscosity-temperature data for Orange is shown in Table 3 and Figure 3. Dow Chemical data and a point at 25 C obtained by the Cannon method by D.B. Coulson are also plotted in Figure 3. The empirical equation fitting the curve is:

$$\ln \eta = \frac{1.080\times10^{14}}{T^{5.436}}$$

TABLE 3. VISCOSITY AS A FUNCTION OF TEMPERATURE FOR ORANGE

Method or Source	Temperature, C	Viscosity, centipoise
Höppler	18.1	70.8
	17.3	74.6
	13.8	102
	5.9	220
	3.9	262
	1.6	333
	-3.9	800
Brookfield	-1.2	540
	-4.0	730
	-7.5	1270
	-10.9	1910
	-11.1	2180
	-13.4	3200
	-16.0	4700
	-16.8	5100
	-18.0	7200
	-20.0	9200
	-21.7	16100
	-23.5	20000
	-23.6	25300
	-25.5	45200
Dow Chemical Company	20	62.0
	30	34.5
	40	19.3
	50	13.2
	60	10.0
Cannon	25	45.4

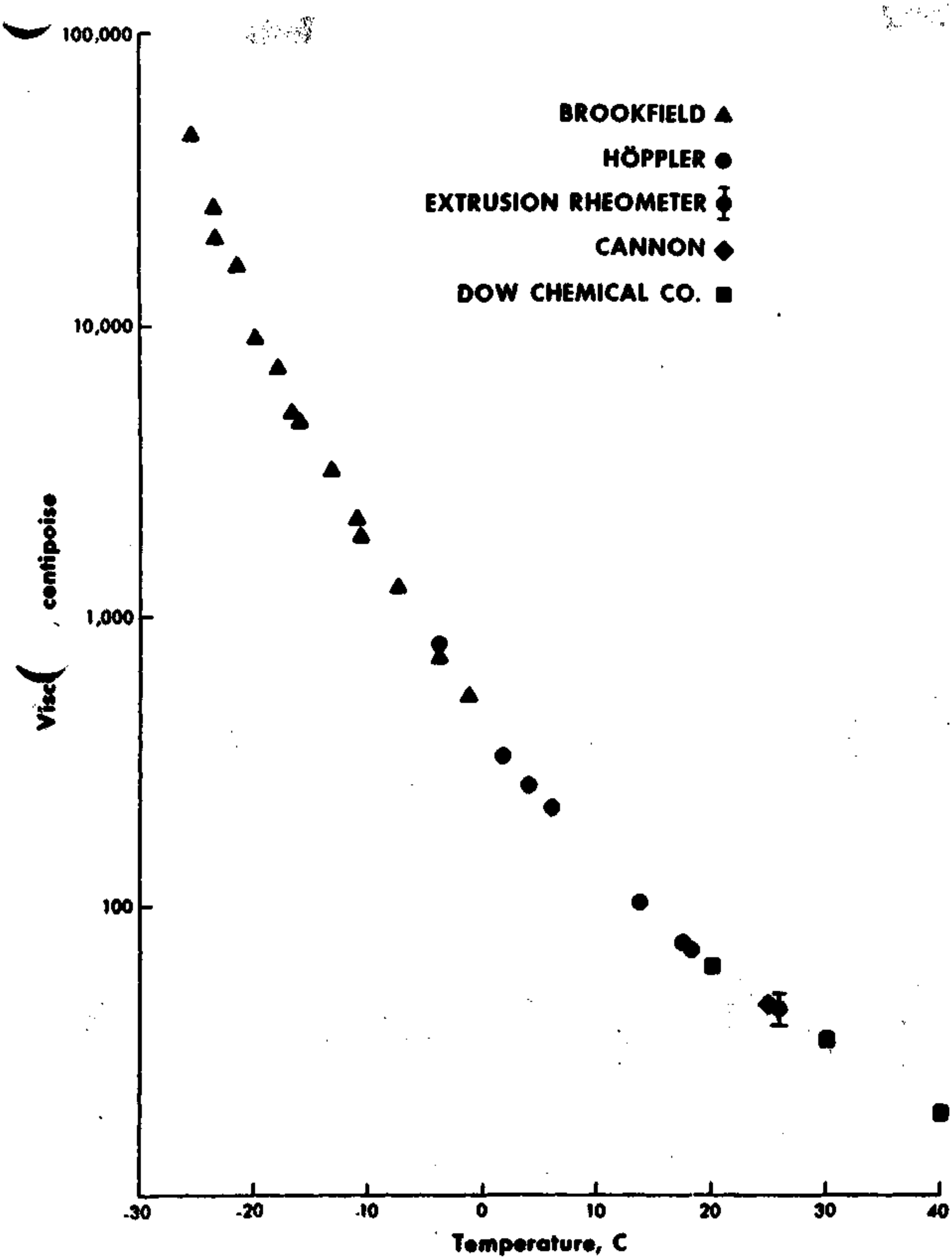

Figure 3. Viscosity Versus Temperature for Orange.

B. FLOW RATE-PRESSURE

Extrusion rheometer data for all materials are reported in Tables 4, 5 and 6. Table 4 gives data not only for needles of 6-inch length but also for needles of 2-inch length. All data were obtained at ambient temperatures (about 26 C). Data reported in the viscosity column in all three tables give the viscosity in centipoise calculated from the equation for laminar flow through a cylindrical tube of finite length as follows from Poiseuille's formula:[8]

$$\eta = \frac{4.43r^4Pt}{Vl} \times 10^7$$

where η is the viscosity in centipoise, r is the radius of the inner surface of the tube or needle in inches, P is the gauge pressure in lb/in^2, t is the time in seconds for 40 ml of sample to flow through, V is the volume of sample in milliliters, and l is the tube or needle length in inches. Viscosity data obtained in this manner are of academic value in determining correct functioning of the system but are of less value in determining viscosity per se. Other systems are not only more accurate but also their temperature stability and range may be controlled more easily.

Calculations and data from the determinations indicate that within each needle, the acceleration and deceleration end effects were negligible; consequently graphing the flow rate Q or V/t in ml/sec versus the pressure P in psig should give straight lines passing through the origin for each set of data if the liquid is Newtonian. Data are shown graphically in Figures 4, 5, and 6 for all three materials. The data were obtained with needles of all three diameters and meet the criteria stated above for that characteristic of Newtonian liquids. Only data obtained with the 6-inch-long needles are plotted. The conclusion therefore is that these materials are Newtonian.

C. SURFACE TENSION

Surface tension data are reported in Table 7 for all three materials. Little variation with temperature in the range from ambient to 10 or 15 C above ambient was noted. Surface tensions exhibited the normal inverse relationship to temperature increase.

The radius of the capillary tip used in all determinations was 0.2422 cm so that the surface tension in each case is equal to 1.074 m x 10^3 dynes/cm. During these trials, 2,4,5-T froze at 25 C; therefore, data are reported for temperatures of 35 and 40 C for this material.

TABLE 4. EXTRUSION RHEOMETER DATA FOR 2,4-D

	P, psig	t, sec	Q, 10^{-1} cm^3/sec	Pt, psig-sec	η, centipoise
			6-Inch Needles		
22 gauge	80	327	1.24	26,160	23.0
	60	440	0.92	26,400	23.2
	40	670	0.60	26,800	23.6
	20	1357	0.28	27,140	23.9
20 gauge	80	75	5.32	6,000	25.4
	60	100	4.00	6,000	25.4
	40	151	2.48	6,040	25.5
	20	300	1.32	6,000	25.4
18 gauge	80	25	16.00	2,000	24.9
	60	33	12.12	1,980	24.6
	40	51	7.84	2,040	25.4
	18	114	3.52	2,052	25.5
			2-Inch Needles		
22 gauge	81	129	3.10	10,449	27.5
	61	173	2.31	10,553	27.8
	42	250	1.60	10,500	27.6
	21	535	0.75	11,235	29.5
20 gauge	81	28	14.28	2,268	28.8
	59	38	10.52	2,242	28.5
	40	55	7.28	2,200	27.9
	20	110	3.64	2,200	27.9
18 gauge	41	19	21.04	779	29.0
	30	25	16.00	750	28.0
	19	42	9.52	798	29.7

TABLE 5. EXTRUSION RHEOMETER DATA FOR 2,4,5-T

	P, psig	t, sec	Q, 10^{-5} cm^3/sec	Pt, psig-sec	η, centipoise
			<u>6-Inch Needles</u>		
22 gauge	81	1110	3.60	89,910	79.1
	61	1475	2.72	89,975	79.2
	40	2310	1.72	92,400	81.3
	22	4202	0.96	92,444	81.4
20 gauge	80	232	17.24	18,560	78.5
	60	312	12.84	18,720	79.2
	40	473	8.44	18,920	80.0
	20	934	4.28	18,680	79.0
18 gauge	80	76	52.64	6,080	75.6
	60	101	39.60	6,060	75.3
	40	150	26.68	6,000	74.6
	20	307	12.04	6,140	76.3

TABLE 6. EXTRUSION RHEOMETER DATA FOR ORANGE

	P, psig	t, sec	Q, 10^{-5} cm^3/sec	Pt, psig-sec	η, centipoise
			<u>6-Inch Needles</u>		
22 gauge	80	588	6.80	47,040	41.4
	60	796	5.04	47,760	42.0
	40	1230	3.24	49,200	43.3
	20	2640	1.52	52,800	46.5
20 gauge	80	133	30.08	10,640	46.0
	59	182	21.96	10,738	46.4
	40	281	14.24	11,240	48.6
	20	549	7.28	10,980	47.4
18 gauge	80	44	90.8	3,420	42.5
	60	59	67.6	3,540	44.0
	38	95	42.0	3,610	44.9
	20	183	22.0	3,660	45.5

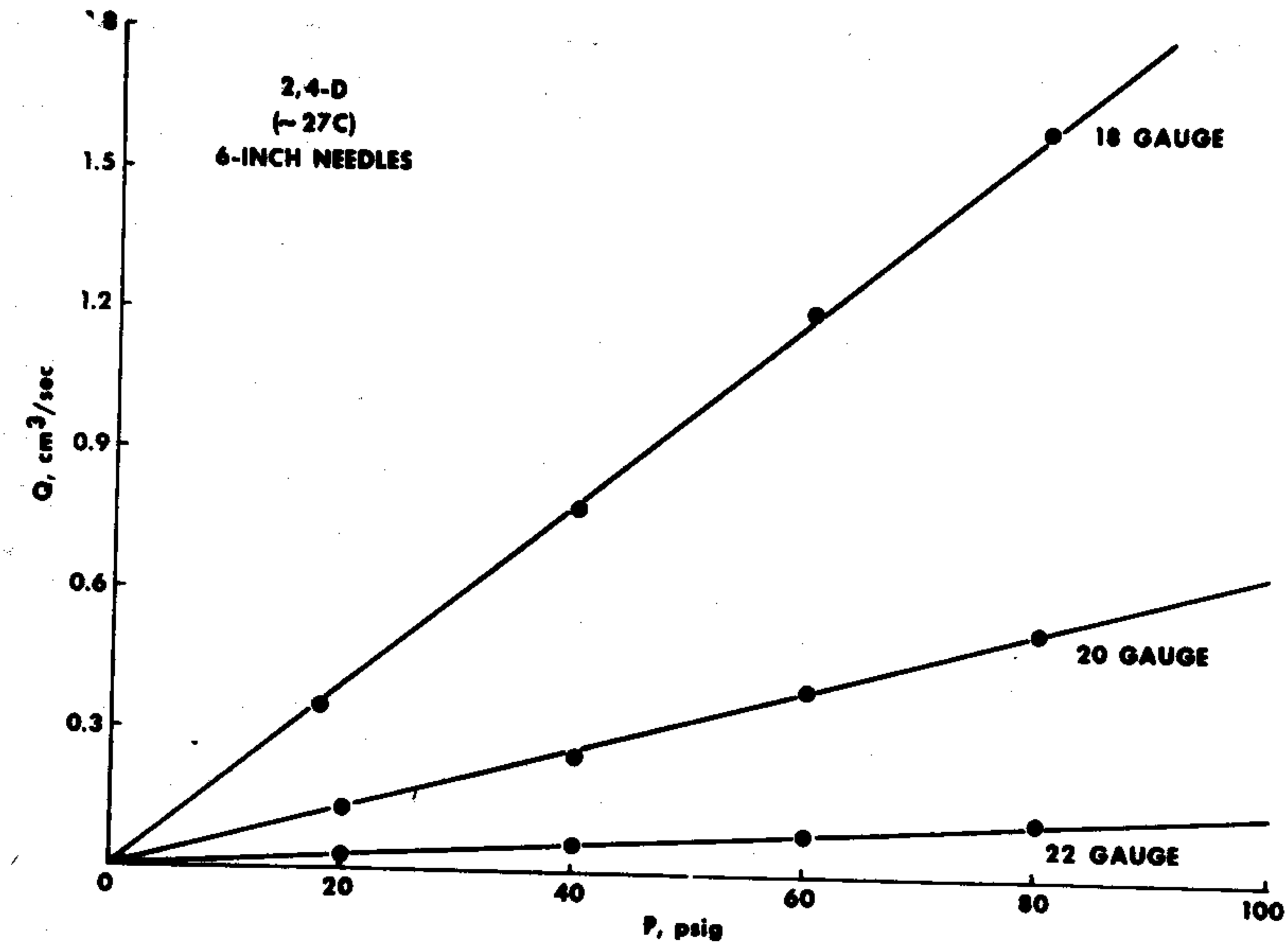

Figure 4. Flow Rate Versus Pressure for 2,4-D.

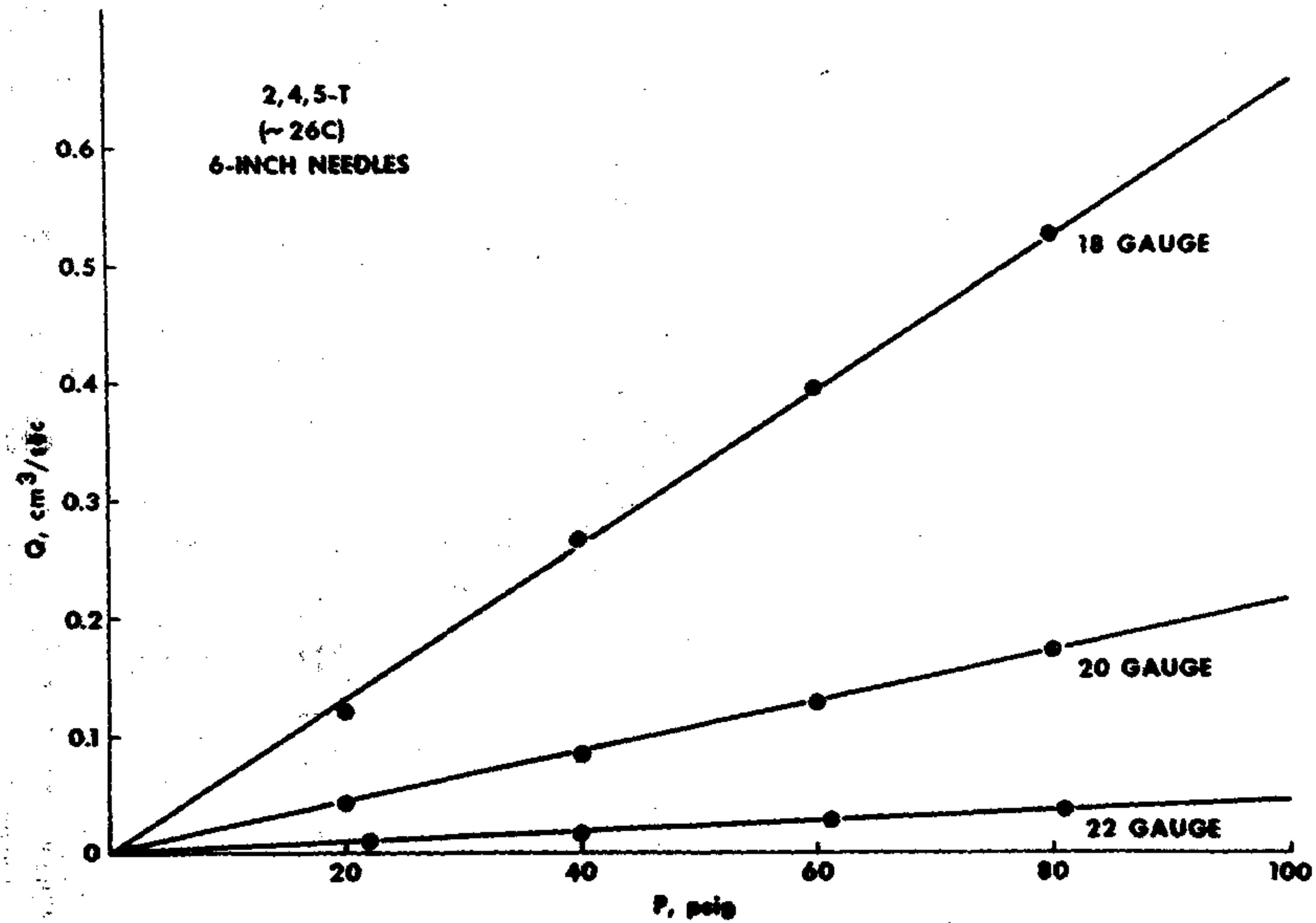

Figure 5. Flow Rate Versus Pressure for 2,4,5-T.

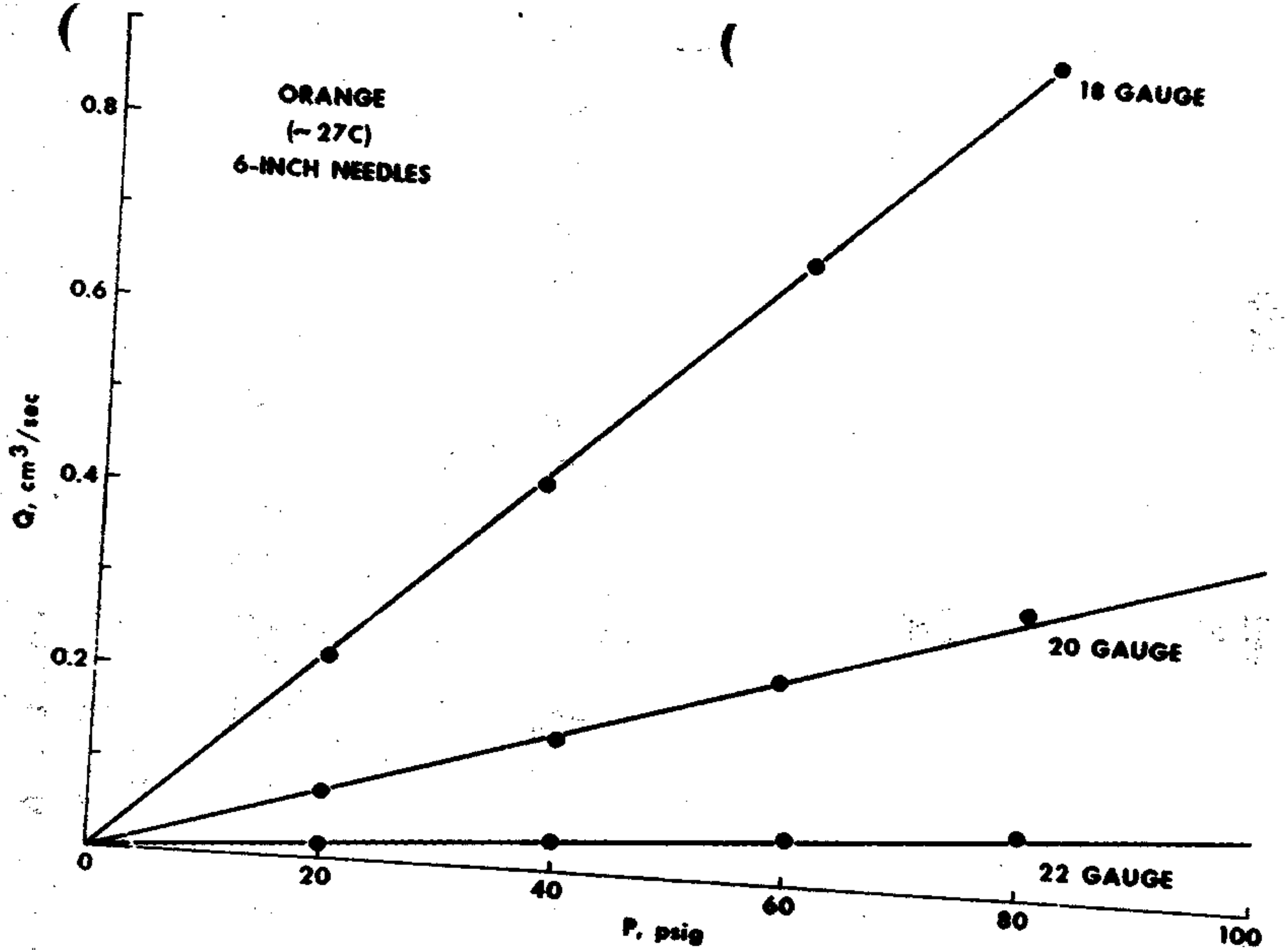

Figure 6. Flow Rate Versus Pressure for Orange.

TABLE 7. SURFACE TENSION DATA FOR 2,4-D; 2,4,5-T; AND ORANGE

Material	T, C	ρ, g/cm^3	m, g	Surface Tension, dynes/cm
2,4-D	25	1.24	0.03325	35.71
			0.03322	35.68
			0.03327	35.73
	35	1.23	0.03247	34.87
			0.03248	34.88
			0.03252	34.93
2,4,5-T	35	1.32	0.03348	35.96
			0.03349	35.97
			0.03348	35.96
	40	1.31	0.03305	35.50
			0.03315	35.60
			0.03310	35.55
Orange	25	1.28	0.03350	35.98
			0.03348	35.96
			0.03348	35.96
	35	1.27	0.03285	35.28
			0.03283	35.26
			0.03284	35.27

D. THERMAL CONDUCTIVITY (THEORETICAL)

Smith's empirical equation, reported earlier, gives the following values for the thermal conductivities:

for 2,4-D 0.0789 BTU/(hr)(ft^2)(deg F/ft)
or 3.26×10^{-4} cal/(cm^2)(sec)(deg C/cm)

for 2,4,5-T 0.0846 BTU/(hr)(ft^2)(deg F/ft)
or 3.49×10^{-4} cal/(cm^2)(sec)(deg C/cm)

and for Orange, a first approximation would be an average of the values for 2,4-D and 2,4,5-T. This would be

$$0.0817 \text{ BTU/(hr)(ft}^2\text{)(deg F/ft)}$$
$$\text{or } 3.37\times10^{-4} \text{ cal/(cm}^2\text{)(sec)(deg C/cm)}.$$

E. SPECIFIC HEAT OF 2,4,5-T, AND ORANGE

Seven trials with the method of mixtures described earlier gave a value of 0.289 for the specific heat of 2,4,5-T with an average deviation of 0.019. Eight trials gave a value of 0.342 with an average deviation of 0.017 for the specific heat of Orange.

F. FREEZING POINT OF ORANGE

The freezing point value determined by the bracketing method was 7.4±0.5 C. The freezing point in the case of an impure material is questionable since, due to impurities, the "point" was a temperature range over which a change in state occurred.

V. CONCLUSION

The physical properties reported here are those determined using impure samples of the materials as they were commercially available. Such values, therefore, are to be regarded as approximate values only when speaking of the properties of the pure materials since the degree of purity has an appreciable effect upon thermal properties of such materials. These values are, however, sufficiently accurate to serve as a source of data upon which to base designs of equipment and systems employing these materials.

An additional property of these materials noted is that they all chemically attack such materials as brass, Lucite, and epoxy cement. Such reactions are probably due to the small amounts of the material not yet converted from the normal acid form.

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APPENDIX A

PROPERTIES OF 2,4-D OBTAINED FROM OTHER SOURCES

1. Name — n-butyl 2,4-dichlorophenoxyacetate
2. Code — CD-143
3. Formula — $C_{12}H_{14}Cl_2O_3$
4. Molecular Weight — 277
5. Melting Point* — 9 C
6. Freezing Point** — 12.6 C (K_{FP} = 7.6 C/mole)
7. Density or Specific Gravity** — 1.2358 (28 C or 82.4 F)
8. Heat of Vaporization** — 68.5 cal/g
9. Heat of Fusion** — 17 cal/g
10. Specific Heat (Liquid)** — 0.283 cal/g C
11. Boiling Point** — 317 C at 760 mm of Hg (K_{BP} = 10 C/mole)
    157 C at 2 mm of Hg
    146-7 C at 1 mm of Hg
12. Vapor Pressures**

T (C)	25*	147	189	205	226	248	265	317
P (mm of Hg)	0.004*	1	10	20	50	100	150	760

13. Surface Tension***

T (C)	25	35	50
S.T. (dynes/cm)	34.3	33.4	32.8

14. Refractive Index** — 1.5230 at 28 C
    1.5340 at 24 C
15. Critical Pressure** — 231 atmospheres
16. Critical Temperature** — 605 C
17. Critical Volume** — 0.116 liters/mole

* Dow Chemical Co. data.
** USABL data.
*** Edgewood Arsenal data.

APPENDIX B

PROPERTIES OF 2,4,5-T DETERMINED BY CROPS DIVISION

1.	Name	n-butyl 2,4,5-trichlorophenoxyacetate
2.	Formula	$C_{12}H_{13}Cl_3\ O_3$
3.	Molecular Weight	311
4.	Density or S.G.	1.327 at 26 C
5.	Melting Point	32 to 33 C
6.	Boiling Point	162 to 165 C at 1 mm of Hg

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13. ABSTRACT

Physical properties of normal butyl esters of 2,4-dichlorophenoxyacetate, 2,4,5-trichlorophenoxyacetate and their equal mixture by weight (denoted as "Orange") are reported. Summaries of data from Dow Chemical Company, Edgewood Arsenal (Army Chemical Center) and U.S. Army Biological Laboratories, are also given. Recently acquired data on viscosities as a function of temperature, flow rates as a function of pressure, theoretical calculations of thermal conductivities, specific heats, surface tensions, and freezing points are also reported.

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