is kept moving as before but the pen-drop mechanism is switched off. The pen then indicates values of magnetization without actually recording them. The field strength is turned by hand to the required value and the pen watched until it settles at a constant value. The mechanism is switched on and a point is recorded. This is considerably more accurate than attempting to use a fast sweep rate. Unlike the direct integration technique, the effect of unbalance in the coils may be minimized by reducing the sweep rate, to zero if necessary, at the time of taking a reading.

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Specific heat of Apiezon T grease from 1 to 350° K

E. F. WESTRUM, Jr.†, CHIEN CHOU†, D. W. OSBORNE‡, and H. E. FLOTOW‡

Establishment of thermal contact among various parts of calorimetric assemblies which must be dismantled to facilitate loading is often achieved by use of a low vapour-pressure grease. Apiezon T grease is one of the commercially available greases that we have employed for this purpose.‡ It has shown satisfactory adhesive, film-strength, and thermal conduction properties as a thin film (usually < 0·1 mm thick) between metallic and other mating surfaces from below 1 to 350° K. Another advantage of Apiezon T is that it is soluble in common organic solvents (e.g. benzene and carbon tetrachloride), whereas silicone greases that are often used for thermal contact are difficult to remove.

Although in most experiments it is customary to utilize the same quantity of such greases in measurements both on the full and the empty calorimeter, in some instances it is convenient to add an excess and to make appropriate adjustment for its specific heat. For this reason, and also because there was an irreproducibility of a few hundredths of a percent in the heat capacity of an empty calorimeter above 200° K, it was considered desirable to examine the heat capacity of this grease.

Determinations of the specific heat of a 32·54 g sample of Apiezon T were made at the University of Michigan with Calorimeter W-7 in the Mark I cryostat from 5 to 350° K by means of the adiabatic techniques previously described.[1] Independent determinations on a 0·2542 g sample of Apiezon T from a different tube were made at Argonne National Laboratory with an isothermal-type calorimeter[2] in the range 1·3–22° K. For the latter measurements the grease was spread in a thin layer on the outside of the calorimeter.

In the region of overlap the two sets of data agree within the estimated experimental errors. The smoothed results at selected temperatures are presented in Table 1. The estimated probable error from 1 to 15° K is 2 per cent, and it decreases above this temperature to about 0·2 per cent from 25 to 205° K and from 320 to 350° K. Below 4·2° K the data are adequately represented by the equation:

$$C \times 10^6 = 2{\cdot}034T + 5{\cdot}065T^3 + 0{\cdot}3547T^5 - 0{\cdot}01012T^7$$

expressed in cal/(deg g).

Over most of the temperature range the specific heat curve is of the normal sigmoid shape to be

TABLE 1. SPECIFIC HEAT OF APIEZON T, IN cal/(deg g)

T(°K)	$C \times 10^3$	T(°K)	$C \times 10^3$	T(°K)	$C \times 10^3$†
1	0·00744	25	32·00	180	252·0
2	0·0546	30	41·45	190	265·5
3	0·207	35	50·84	200	281·1
4	0·530	40	59·96	210	(306)
5	1·035	45	68·83	220	(362)
6	1·73	50	77·46	230	(410)
7	2·60	60	93·91	240	(447)
8	3·64	70	109·4	250	(476)
9	4·81	80	124·3	260	(498)
10	6·11	90	138·5	270	(516)
11	7·50	100	151·8	280	(526)
12	8·98	110	164·8	290	(524)
13	10·53	120	177·6	300	(517)
14	12·14	130	190·3	310	(505)
15	13·80	140	202·7	320	502·2
16	15·51	150	214·9	330	509·2
18	19·02	160	227·1	340	518·0
20	22·65	170	239·3	350	525·9

† The values in parentheses in this column are in the anomalous region.

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§ We used a sample of Apiezon T grease produced by Metropolitan-Vickers Electrical Co. Ltd. This product is currently made by Associated Electrical Industries Ltd (England), and distributed by Shell Companies.

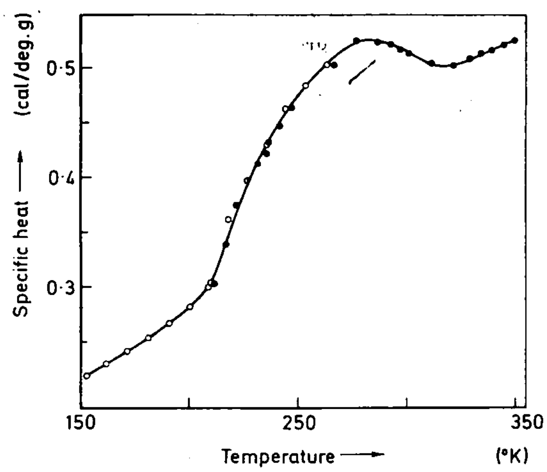

Figure 1. Two series of specific heat determinations on Apiezon T in the anomalous region

expected for a hydrocarbon of high molecular weight. Between 205 and 320° K a broad anomaly occurs as shown in Figure 1. In this region the experimental values were not reproducible to better than 2 per cent, and considerable variation in the time required to reach equilibrium was noted. The origin of the anomaly is unknown, and if it is not characteristic of the hydrocarbon components of the grease it may occur in some additive such as a hydrated metal soap employed to modify the properties of the grease. For precision calorimetry which includes the range between 205 and 320° K it is obviously desirable to minimize the amount of Apiezon T used for thermal contact. We usually employ less than 50 mg.

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A cryostat for an RÉ 1301 E.P.R. spectrometer

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OUR industry produces an instrument for studying electron paramagnetic resonance spectra which is used in many laboratories; the RÉ 1301. The instrument has good technical features and is reliable in operation but has the considerable drawback of not being adaptable to low temperature measurements.

In using the RÉ 1301 instrument it is relatively simple to cool the specimen to 77° K either with a small Dewar of glass, quartz, or plastic, with the lower end drawn out to a 'finger' which is in the resonator cavity, or with a glass tube which goes across the resonator and has nitrogen vapour passed through it. However, a special cryostat is necessary for lower temperatures (down to 4·2° K).

A helium cryostat designed for low temperature measurements with the RÉ 1301 e.p.r. spectrometer is described below. The waveguide system of the spectrometer does not have to be changed when using the cryostat.

Construction

The construction is partly based on the cryostat used in studying frozen free radicals.[1]

The cryostat consists of the following three main parts: (1) The outer vacuum jacket 8 (in the drawing) with the resonator 17 inside; (2) The nitrogen can 9 with heat shield 12 and (3) The helium can 10. All three jackets are mounted on flanges 5 to 7 in the upper part of the cryostat. The lifting mechanism 1 is fixed on flange 5 and is connected to bellows 2 by which the helium space can be raised and lowered.

The helium can has an upper neck 3 with a guide tube 4 for the shaft and a lower tubular tail 11 ending in the quartz ampoule 13. The 8 mm diameter quartz ampoule is inserted into the ground end of the tubular tail with a hot setting rubber which achieves vacuum tightness. With this form of helium can either a specimen can be immersed directly in the liquid helium by means of the shaft, or a specimen can be deposited onto the quartz ampoule through the outer window 14 and this is then lowered into the resonator cavity. The ampoule is centered relative to the opening in the resonator by three centering screws 22 on the bellows and the conical Teflon plug 16 pressed into the resonator opening. The cryostat has window 15 for observing the centering.

The cryostat has an external detachable getter 21 to pump the vacuum space.

We used standard resonator and waveguides which are in the spectrometer assembly and are rectangular (the specification is 3425 43 sb). Resonator 17 and waveguides 18 are connected by 1 mm Teflon washers 19 which act as thermal barriers. The waveguides are soldered directly to the walls of the outer case.

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